Novel Processes for Modular Integration of Silicon-Germanium MEMS with CMOS Electronics

Carrie Wing-Zin Low

Electrical Engineering and Computer Sciences
University of California at Berkeley

February 28, 2007

Novel Processes for Modular Integration of Silicon-Germanium MEMS
with CMOS Electronics

By

Carrie Wing-Zin Low

B.S. (University of California, Berkeley) 2001
M.S. (University of California, Berkeley) 2004

A dissertation submitted in partial satisfaction of the

requirements for the degree of

Doctor of Philosophy

in

Engineering - Electrical Engineering and Computer Sciences

in the

Graduate Division

of the

University of California, Berkeley

Committee in charge:

Professor Tsu-Jae King Liu, Co-chair
Professor Roger T. Howe, Co-chair
Professor Richard M. White
Professor Oscar D. Dubón, Jr.

Spring 2007

The dissertation of Carrie Wing-Zin Low is approved:

Co-chair _____ Date _____

Co-chair _____ Date _____

_____ Date _____

_____ Date _____

University of California, Berkeley

Spring 2007

Novel Processes for Modular Integration of Silicon-Germanium MEMS
with CMOS Electronics

Copyright © 2007

by

Carrie Wing-Zin Low

Abstract

Novel Processes for Modular Integration of Silicon-Germanium MEMS

with CMOS Electronics

By

Carrie Wing-Zin Low

Doctor of Philosophy in Engineering - Electrical Engineering and Computer Sciences

University of California, Berkeley

Professor Tsu-Jae King Liu, Co-chair

Professor Roger T. Howe, Co-chair

Equipment control, process development and materials characterization for LPCVD poly-SiGe for MEMS applications are investigated in this work. In order to develop a repeatable process in an academic laboratory, equipment monitoring methods are implemented and new process gases are explored. With the dopant gas BCl_3, the design-of-experiments technique is used to study the dependencies of deposition rate, resistivity, average residual stress, strain gradient and wet etch rate in hydrogen-peroxide. Structural layer requirements for general MEMS applications are met within the process temperature constraint imposed by CMOS electronics. However, the strain gradient required for inertial sensor applications is difficult to achieve with as-deposited films.

Approaches to reduce the strain gradient of LPCVD poly-SiGe are investigated. Correlation between the strain gradient and film microstructure is found using stress-depth profiling and cross-sectional TEM analysis. The effects of film deposition

conditions on film microstructure are also determined. Boron-doped poly-SiGe films generally have vertically oriented grains -- either conical or columnar in shape. Films with conical grain structure have large strain gradient due to highly compressive stress in the lower (initially deposited) region of the film. Films with small strain gradient usually have columnar grain structure with low defect density. It is also found that the uniformity of films deposited in a batch LPCVD reactor can be improved by increasing the deposited film thickness, using a proper seeding layer, and/or depositing the film in multiple layers. The best strain gradient achieved in our academic research laboratory is 1.1×10^{-6} μm^{-1} for a ~3.5 µm thick film deposited at 410°C in 8 hours, with a worst-case variation across a 150 mm-diameter wafer of 1.6×10^{-5} μm^{-1} and a worse-case variation across a load of twenty-five wafers of 7×10^{-5} μm^{-1}. The effects of post-deposition annealing and argon implantation on mechanical properties are also studied. While the as-deposited film can achieve the desired mechanical properties, post-deposition processing at elevated temperatures can degrade the strain gradient.

<div style="text-align:right">

_____

Professor Tsu-Jae King Liu, Co-chair   Date

</div>

<div style="text-align:right">

_____

Professor Roger T. Howe, Co-chair   Date

</div>

To Mom and Dad

Table of Contents

List of Figures

List of Tables

Acknowledgement

I would first like to thank my research advisor, Professor Roger Howe, for encouraging me to go to graduate school and introducing me to the world of MEMS. His broad knowledge and endless enthusiasm about the MEMS field provided a tremendous amount of energy throughout the course of my graduate work. Professor Tsu-Jae King Liu has been providing insightful technical advice at all stages of my research. I am especially thankful to have Professor Liu as my official co-advisor after Professor Howe's departure to Stanford University; the last chapter of my research would have been impossible without her support. In addition, Professor Oscar Dubón's input on materials science was a great supplement to this work. I would also like to thank Professor Richard White for serving in my qualifying exam and thesis committee.

The work presented here is a collaborative effort with many individuals and groups. UC Berkeley Microfabrication Laboratory staff, including Patrick Wehrly, Jimmy Chang, Danny Pestal Bob Hamilton, Bill Flounders and Katalin Voros, provided tremendous support in maintaining the SiGe furnace and making custom modifications for my research. Dr. Andrea Franke provided professional guidance and shared her friendship during the first phase of my poly-SiGe study. Prof. Sherif Sedky of the American University in Cairo provided insight on the excimer laser annealing work. Dr. Yasuo Kunii of Hitachi Kokusai Electric, Inc. shared his experience in the BCl_3 development work. Dr. Uthara Srinivasan and Brian Bush provided valuable discussions on anti-stiction coating. Xin Sun's assistance on CMOS testing during the weekend was greatly appreciated. In addition, the TEM analysis was conducted at the National Center

Ruth Gjerde in the Graduate Office was a great source of support and information. The administrative support from Tom Parsons, Lindy Manly, John Huggins, Jukka-Pekka Vainio and other BSAC staff were deeply appreciated.

Last but not least, I would like to thank my family, especially my husband Andy Mai, for their love and support through the years.

Chapter 1: Introduction

1.1 Process strategies for MEMS and CMOS integration

The ability to integrate MEMS and IC technology is highly desirable for high performance MEMS devices. There are two main challenges in integration: standard metallization of electronic circuits limits the post process temperature to be below 450°C, and the large topography after the MEMS process limits the compatibility with further lithography steps. Using silicon as the MEMS structural material, most of the integration processes have mixed fabrication of both MEMS and CMOS steps to overcome the temperature limit of the CMOS and the topography problem created by the MEMS structures. Some examples include Analog Devices' iMEMS® process [1.1], Sandia National Laboratory's embedded MEMS process [1.2] and UC Berkeley's SOI process [1.3]. All these mixed fabrication processes are specialized and foundry services are limited.

To take advantage of the low manufacturing cost of foundry services, modular integration of MEMS and CMOS electronics has received substantial interest. In addition, modular integration allows separate development and optimization of the MEMS and the CMOS modules. Modular integration can have the MEMS steps first or the CMOS steps first.

For MEMS-first modular integration, single crystal or epitaxial silicon surface is required for the CMOS module. Stanford's and Bosch's wafer-scale encapsulation process using epitaxial silicon has the potential for MEMS-first modular integration [1.4], [1.5]. The schematic of the wafer-scale encapsulation process is shown in Figure 1.1. The

starting material is an SOI wafer. The buried oxide serves as the sacrificial material, and the MEMS structures are defined by lithography and deep reactive ion etching. A layer of gasket oxide is deposited as the sacrificial material between the MEMS structures and the capping layer. The gasket oxide is patterned and removed from the MEMS anchor regions and the circuit areas. Then 10 μm of silicon is grown in an epitaxial reactor at 1000°C. Polycrystalline silicon is deposited over the oxide, serving as the capping layer; single crystal silicon is grown where the gasket oxide is removed. Etch holes are defined and the structure is released with vapor HF. The etch holes are then sealed with thermal oxide. The thermal oxide is removed from the metal contact area and the single crystal silicon area. CMOS process can be potentially done on the single crystal silicon area after the MEMS process. In this approach, the MEMS structures are made out of the single crystal silicon device layer of the SOI wafer, and high-quality inertial sensors and RF resonators can be built. Also, the 10 μm thick encapsulation layer can withstand conventional back-end packaging process, such as dicing and injection molding. However, for the CMOS module, the electronic circuits cannot be placed directly on top of the MEMS area due to the selective epitaxial growth. Also, controlling the quality of the epitaxial silicon is very challenging for the circuitry areas.
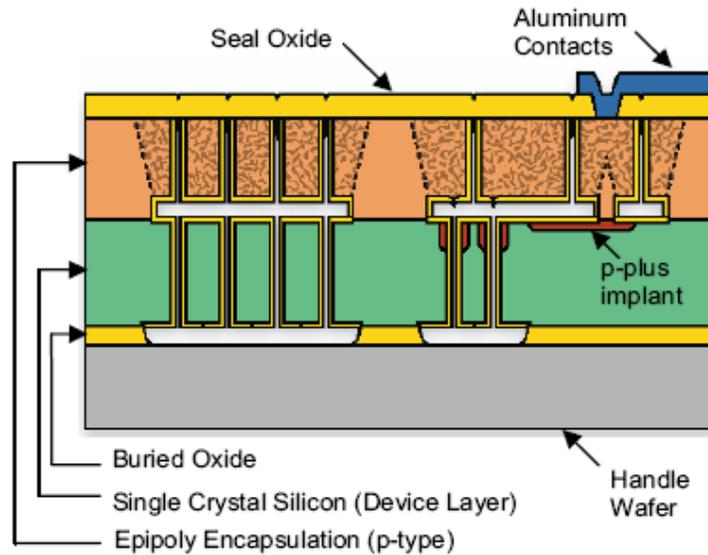
Figure 1.1 Schematic of wafer-scale encapsulation for MEMS-first integration [1.5]

For CMOS-first modular integration, low process temperature materials are used for the MEMS steps to overcome the temperature limit imposed by the foundry CMOS. For example, Texas Instruments has a commercially successful process to make digital micromirror displays using a Ti-Al alloy [1.6]. IBM is developing a copper-based MEMS process for RF switches and resonators [1.7]. Aluminum nitride (AlN) is being explored as the structural material for RF filters and resonators at UC Berkeley [1.8]. Amorphous silicon (a-Si:H) has been demonstrated for bimorph thermal actuator application by University of Waterloo[1.9]. Polycrystalline-silicon-germanium (poly-SiGe) is another low-temperature surface micromachining material. Compared to Ti-Al, Cu, AlN and a-Si:H films, poly-SiGe has similar properties and process as the conventional surface micromachining material polycrystalline-silicon (poly-Si). Also, poly-SiGe can be used for adaptive optics [1.10], RF resonators [1.11] and inertial

sensors applications [1.12]. Figure 1.2 is a schematic of a SiGe MEMS resonator built on top of foundry CMOS electronics. After the completion of the CMOS steps, contacts are opened and vias to the electronics are first made. Sacrificial material can be either silicon dioxide or pure germanium. Poly-SiGe is used as the structural material. In this approach, MEMS devices are built directly on top of the circuitry, reducing interconnect resistance and saving valuable die area. Since there is a temperature constraint on the MEMS process, the mechanical properties of poly-SiGe are not as good as the single crystal silicon used in the wafer-scale encapsulation process discussed above. The Young's modulus and quality factor of poly-SiGe are slightly lower than those of single crystal silicon or poly-Si. With the temperature limitation, achieving the specifications of the low residual stress and strain gradient for inertial sensor applications are the main challenges for poly-SiGe. This work studies the control of the desired poly-SiGe materials properties with the temperature constraint.
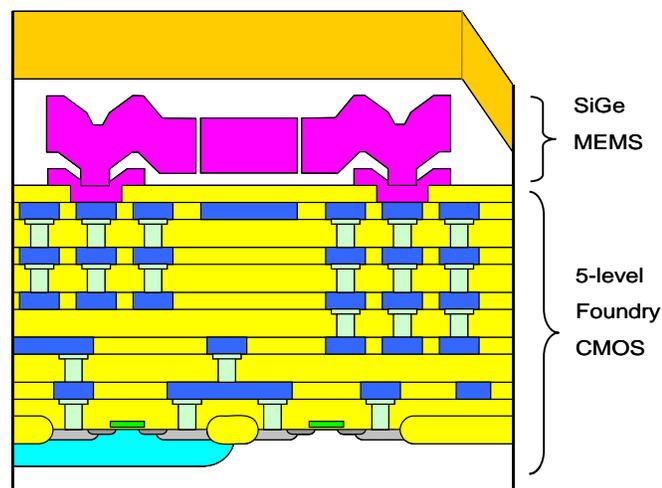
Figure 1.2 Schematic of modular integration of MEMS and CMOS with poly-SiGe (courtesy of R. T. Howe and B. L. Bircumshaw)

1.2 SiGe research in IC and MEMS

Silicon-germanium has been studied extensively as the base material for heterojunction bipolar junction transistors [1.13], [1.14]; as the gate, source/drain or channel material for CMOS devices [1.15]-[1.17]; and as the absorption material for optical or thermal electronics [1.18]-[1.20]. Recently, poly-SiGe has been investigated as an alternative structural material for surface micromachining. Poly-SiGe has materials properties that are similar to those of poly-Si. In contrast to poly-Si, poly-SiGe can be deposited and crystallized at very low temperatures with good stability, which makes it promising for post-CMOS integration of MEMS [1.12], [1.21], [1.22]. This modular approach to MEMS integration is an attractive route to higher-performance and lower-cost microsystems.

Several approaches to depositing poly-SiGe for MEMS applications have been investigated by various research groups: atmospheric- or reduced-pressure chemical vapor deposition (APCVD or RPCVD) [1.23], low-pressure chemical vapor deposition (LPCVD) [1.21], [1.24], [1.25], plasma enhanced chemical vapor deposition (PECVD) [1.24], [1.26], [1.27] and pulsed laser deposition (PLD) [1.28]. The deposition rate for the APCVD or RPCVD processes is about 4 nm/min at 520ºC, which is too low to be economical at CMOS compatible temperatures. Films deposited by PLD have high particle density and require addition annealing for crystallization. Poly-SiGe LPCVD and PECVD processes are well established and most promising for use in manufacturing. The deposition rate for the PECVD process is about 100 nm/min at 450ºC, which is about 6× higher than that of the LPCVD process at the same temperature. On the other hand,

PECVD films typically have very high hydrogen content. The hydrogen evolves with excimer laser annealing and leaves small pores in the film [1.29]. While the LPCVD process has lower deposition rate, it usually has a large batch size for higher throughput and lower cost. Also, excimer laser annealing does not result in pores in LPCVD film [1.30]. Another major advantage of LPCVD process is its conformal coverage of all surfaces, which can also be used for planarization and gap filling. While PECVD and LPCVD poly-SiGe processes are both promising for post-CMOS integration, recent research has focused on pushing down the thermal budget, fine tuning the materials properties and developing a robust process for large volume production.

1.3 Desired MEMS properties for poly-SiGe

The desired SiGe properties for MEMS applications are very different from those of electronic device applications. In general, a film thicker than 2 μm is needed for lateral capacitive sensing. For post CMOS processing, the deposition temperature of poly-SiGe is limited to below 450°C. Deposition rate and crystallinity of the film can be improved with higher germanium content. However, the etch selectivity of a pure germanium sacrificial layer to a poly-SiGe structural layer for H_2O_2 etching decreases with increasing germanium content in the poly-SiGe film. A germanium content of 60% is desired for reasonable deposition rate and crystallinity with adequate resistance to H_2O_2 etching. In order to have good electrical connection to the electronics, the desired resistivity is below 10 mΩ-cm for RF MEMS applications. For inertial sensor applications with long suspension length, low residual stress and strain gradient are required. To avoid buckling of a clamped-clamped beam, a small tensile residual stress is desired. However, with

6

special design, films with compressive stress can also be used. Low strain gradient is the most critical requirement for inertial sensor applications. The typical strain gradient specification for inertial sensors is less than 1×10^{-5} μm^{-1}, which results in less than 5 μm tip deflection for a 1 mm long beam. In addition to the above materials requirements, developing a high throughput, high yield and repeatable process is critical for large volume production.

1.4 Overview of dissertation

This work presents the materials and the process development of LPCVD poly-SiGe. This dissertation is organized in the following chapters:

Chapter 2 reviews the LPCVD poly-SiGe reactor. The deposited thin-film materials properties and the robustness of the process heavily depend on the condition of the reactor. The configuration, the design, the operation and the process monitoring of the reactor are discussed.

In Chapter 3, the development and the challenges of using new process gases are described. Boron trichloride (BCl_3) has been successfully developed as a better p-type dopant gas to replace diborane (B_2H_6); disilane (Si_2H_6) is investigated as a silicon precursor; germyl silanes (($H_3Ge)_xSiH_{4-x}$) are reviewed as the potential single-source silicon and germanium precursors.

Chapter 4 describes the process development to achieve the desired materials properties for RF MEMS and inertial sensor applications. The focus is on optimizing the strain gradient of the film, which is the most challenging materials property for inertial sensor applications. Uniformity and repeatability of the process are also discussed.

In Chapter 5, the effects of post-deposition processing on the materials properties are investigated. Also, a study of the CMOS thermal budget limits is presented.

Chapter 6 summarizes the main contributions of this work and suggests future directions.

References

[1.1] T. A. Core, W. K. Tsang and S. J. Sherman, "Fabrication technology for an integrated surface-micromachined sensor," *Solid State Technology*, Oct. 1993, pp. 39-47

[1.2] J. H. Smith, S. Montague, J. J. Sniegowski, J. R. Murray, R. P. Manginell and P. J. McWhorter, "Characterization of the embedded micromechanical device approach to the monolithic integration of MEMS with CMOS," *SPIE*, vol. 2879, pp. 306-314, 1996

[1.3] T. J. Brosnihan, "An SOI based, fully integrated fabrication process for high-aspect ratio microelectromechanical systems," Ph.D. Thesis, Dept. of ME, University of California at Berkeley, 1998

[1.4] R. N. Candler, W.-T. Park, H. Li, G. Yama, A. Partridge, M. Lutz and T. W. Kenny, "Single wafer encapsulation of MEMS Devices," *IEEE Transcations of Advanced Packaging*, vol. 26, no. 3, 2003, pp. 227-232

[1.5] W.-T. Park, R. N. Candler, S. Kromnueller, M. Lutz, A. Partridge, G. Yama and T. W. Kenny. **"**Wafer-scale film encapsulation of micromachined accelerometers," in *Proc. 12th International Conference on Solid-State Sensors, Actuators and Microsystems (Transducers 03)*, June 2003, pp. 1903-1906

[1.6] P. F. Van Kessel, L. J. Hornbeck, R. E. Meier and M. R. Douglass, "A MEMS-based projection display," *Proc. of IEEE*, vol. 86, no. 8, 1998, pp. 1687-1704

[1.7] C. V. Jahnes, J. Cotte, J. L. Lund, H. Deligianni, A. Chinthakindi, L. P. Buchwalter, P. Fryer, J. A. Tornello, N. Hoivik, J. H. Magerlein and D. Seeger, "Simultaneous fabrication of RF MEMS switches and resonators using copper-based CMOS

interconnect manufacturing methods," *17th IEEE Micro Electro Mechanical Systems Conference (MEMS-04)*, Maastricht, The Netherlands, Jan. 25-29, 2004

[1.8] G. Piazza, "Piezoelectric aluminum nitride vibrating RF MEMS for radio front-end technology," Ph.D. Thesis, Dept. of EECS, University of California at Berkeley, 2005

[1.9] S. Chang and S. Sivoththaman, "Development of a low temperature MEMS process with a PECVD amorphous silicon structural layer," *J. Micromech. Microeng.* vol. 16, 2006, pp. 1307-1313

[1.10] B. C.-Y Lin, T.-J. King and R. S. Muller, "Poly-SiGe MEMS actuators for adaptive optics," *Photonics WEST*, sponsored by SPIE, The International Society for Optical Engineering, Conference 6113, Paper 6113-28, San Jose, CA, January 25, 2006

[1.11] E. P. Quévy, A. San Paulo, E. Basol, R. T. Howe, T.-J. King, and J. Bokor, "Back-end-of-line Poly-SiGe disk resonators," *19th IEEE Micro Electro Mechanical Systems Conference (MEMS-06)*, Istanbul, Turkey, Jan. 2006

[1.12] A. Witvrouw, A. Mehta, A. Verbist, B. Du Bois, S. Van Aerde, J. Ramos-Martos, J. Ceballos, A. Ragel, J. M. Mora, M. A. Lagos, A. Arias, J. M. Hinojosa, J. Spengler, C. Leinenbach, T. Fuchs and S. Kronmüller, "Processing of MEMS gyroscopes on top of CMOS ICs," in *Proc. 52nd IEEE International Solid-State Circuits Conference*, San Francisco, CA, February 6-10, 2005, pp. 88-89

[1.13] S. S. Iyer, G. L. Patton, S. S. Delage, S. Tiwari, J. M. C. Stork, "Silicon-germanium base heterojunction bipolar transistors by molecular beam epitaxy," in *Proc. International Electron Devices Meeting*, New York, 1987, pp. 874-876

[1.14] J. D. Cressler and G. Niu, *Silicon-Germanium Heterojunction Bipolar Transistors*, Artech House, 2003

[1.15] N. Kistler and J. Woo, "Symmetric CMOS in fully-depleted silicon-on-insulator using P$^+$-polycrystalline SiGe gate electrodes," in *Proc. International Electron Devices Meeting*, 1993, pp. 727-730

[1.16] H. Takeuchi, W.-C. Lee, P. Ranade, and T.-J. King, "Improved PMOSFET short-channel performance using ultra-shallow $Si_{0.8}Ge_{0.2}$ source/drain extensions," in *Proc. International Electron Devices Meeting*, 1999, pp. 501-504

[1.17] T.-J. King and K. C. Saraswat, "Polycrystalline silicon-germanium thin-film transistors," *IEEE Transactions on Electron Devices*, vol 41, no. 9, 1994, pp. 1581-1591

[1.18] S. Sedky, P. Fiorini, K. Baert, L. Hermans and R. Mertens, "Characterization and optimization of infrared poly SiGe bolometers," *IEEE Transactions on Electron Devices*, vol 46, no. 4, 1999, pp. 675-682

[1.19] M. Strasser, R. Aigner, M. Franosch and G. Wachutka, "Miniaturized thermoelectric generators based on poly-Si and poly-SiGe surface micromachining," *Sensors and Actuators A*, vol 97-98, 2002, pp. 535-542

[1.20] P. Van Gerwen, T. Slater, J. B. Chévrier, K. Baert and R. Mertens, "Thin film boron-doped poly-crystalline silicon$_{70\%}$-germanium$_{30\%}$ for thermopiles," *Sensors and Actuators A*, vol. 53, 1996, pp. 325-329

[1.21] A. E. Franke, J. M. Heck, T.-J. King and R. T. Howe, "Polycrystalline silicon germanium films for integrated microsystems," *IEEE/ASME Journal of Micro-electromechanical Systems*, vol. 12, pp. 160-171, Apr. 2003

[1.22] C. W. Low, B. L. Bircumshaw, T. Dorofeeva, G. Solomon, T. -J. King and R. T. Howe, "Stress stability of poly-SiGe and various oxide films in humid environments," in *Proc. Stability of Thin Films and Nanostructures Symposium, Materials Research Society Meeting*, Boston, MA, Nov. 29 - Dec. 3, 2004

[1.23] S. Sedky, P. Fiorini, M. Caymax, S. Loreti, K. Baert, L. Hermans and R. Mertens, "Structure and mechanical properties of polycrystalline silicon germanium for micromachining applications," *IEEE/ASME Journal of Micro-electromechanical Systems*, vol. 7, no. 4, pp. 365-372, Dec. 1998

[1.24] T. Fuchs, C. Leinenbach, S. Kronmueller, f. Laermer, T, Thomas, K. Robb, H. Seidel and W. Frey, "Industrial applications of poly-silicon-germanium as functional MEMS material," in *Proc. Electrochemical Society SiGe Materials, Processing, and Devices Symposium*, Honolulu, HI, Oct. 3-8, 2004, pp. 1001-1013

[1.25] C. W. Low, M. L. Wasilik, H. Takeuchi, T.-J. King and R. T. Howe, "In-situ doped poly-SiGe LPCVD process using BCl_3 for post-CMOS integration of MEMS devices," in *Proc. Electrochemical Society SiGe Materials, Processing, and Devices Symposium*, Honolulu, HI, Oct. 3-8, 2004, pp. 1021-1032

[1.26] T. Van der Donck, J. Proost, C. Rusu, K. Baert, C. Van Hoof, J.-P Celis and A. Witvrouw, "Effect of deposition parameters on the stress gradient of CVD and PECVD poly-SiGe for MEMS applications," in *Proc. SPIE Conference*, San Jose, CA, USA, Jan. 28-29, 2004, pp. 8-18

[1.27] A. Mehta, M. Gromova, P. Czarnecki, K. Baert and A. Witvrouw, "Optimization of PECVD poly-SiGe layers for MEMS post-processing on top of CMOS," in *Proc.*

13th International Conference on Solid-State Sensors, Actuators and Microsystems (Transducers 05), Seoul, Korea, June 5-9, 2005, pp. 1326-1329

[1.28] S. Sedky, I. El Defrar and O. Mortagy, "Pulsed laser deposition of boron doped $Si_{70}Ge_{30}$," in *Proc. Materials Research Society Meeting*, San Francisco, CA, 2006

[1.29] S. Sedky, M. Gromova, T. Van der Donck, J.-P. Celis, A. Witrouw, "Characterization of KrF excimer laser annealed PECVD Si_xGe_{1-x} for MEMS post-processing," Sensors and Actuators A, vol. 127, 2006, pp. 316-323

[1.30] S. Sedky, R. T. Howe and T.-J. King, "Pulsed Laser Annealing, a Low Thermal Budget Technique for Eliminating Stress Gradient in Poly-SiGe MEMS Structures," *IEEE/ASME Journal of Micro-electromechanical Systems*, vol. 13, no.4, pp.669-675, Aug. 2004

Chapter 2: LPCVD Poly-SiGe Technology

Low pressure chemical vapor deposition (LPCVD) is an industry standard deposition technique commonly used to form poly-Si, silicon dioxide and silicon nitride films. Chemical vapor deposition involves the thermal decomposition of source gases to form a solid thin film directly on the wafer substrate. LPCVD has the advantage of high throughput, conformal step coverage and good uniformity. In this section, the configuration, the design, the operation and the process monitoring of the LPCVD poly-SiGe reactor are presented.

2.1 LPCVD Reactor Overview

In-situ doped poly-SiGe films were deposited in a Tystar hot-wall horizontal LPCVD reactor, approximately 125 cm in length and 23 cm in diameter (Figure 2.1). This reactor is configured to accommodate various process conditions and hardware modifications in an academic research environment. The operating pressure range of the furnace is 100 mTorr – 2000 mTorr and the operating temperature range is 300°C – 450°C. There are four channels for precursor gases and two channels for dopant gases with various flow ranges. Process gases can be introduced into the tube via the gas ring or the injector. Unreacted gases are pumped out to the exhaust. This furnace is capable of processing both 100 mm-diameter and 150 mm-diameter wafers. Wafers are placed vertically in wafer boats at the center of the reactor. There is a computer connected to the reactor for process control and recipe management. The furnace normally runs in an automatic mode. It can also be operated manually for setup verification or trouble

shooting. Since hazardous chemicals are used, there are multiple safety interlocks implemented in the software and the hardware.
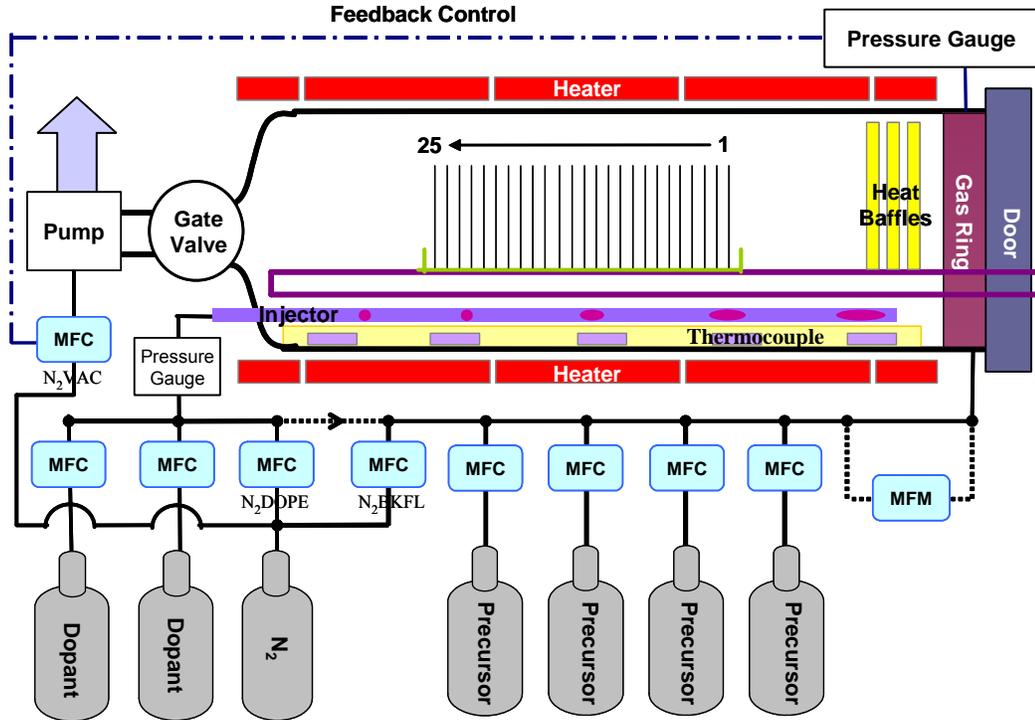
Figure 2.1: LPCVD reactor schematic

2.2 Process gases

The details of all the process gases are listed in Table 2.1 below. Pure silane (SiH_4) or disilane (Si_2H_6) and germane (GeH_4) are available as the gaseous silicon and germanium precursor gases, respectively. Boron trichloride (BCl_3), diborane (B_2H_6) and phosphine (PH_3) can be used as the dopant gases. The dopant gases are diluted to target the desired doping concentration in the film. Most of the precursor and dopant gases are reactive and are health or fire hazards. Toxic and flammable gases are stored in exhausted gas cabinets, each with a sprinkler and a gas leak detector. The N_2 bottle is hooked up to

three lines since it has three roles in the furnace: N_2Dope – to monitor the injector condition and to prevent deposition in the injector for undoped film processes; N2Vac – to reduce the pumping efficiency for pressure control; N_2BKFL – to flush out toxic gases and bring the tube to atmospheric pressure.

TABLE 2.1 Summary of process gases (Hazards information from MSDS of Matheson Tri-Gas, Inc.)

| Gas | Conc. | Range (sccm) | Function | Route | Hazards (NFPA rating) | | | Status |
					Health	Fire	Reactivity	
SiH_4	100%	200	Silicon precursor	Gas ring	2	4	3	Active
Si_2H_6	100%	200	Silicon precursor	Gas ring	1	4	2	Active
GeH_4	100%	200	Germanium precursor	Gas ring	3	4	2	Active
BCl_3	1% in He	50	Boron dopant	Injector/gas ring	3	0	2	Active
B_2H_6	10% in H_2	100	Boron dopant	Injector/gas ring	4	4	3	Inactive
PH_3	50% in H_2	10	Phosphorous dopant	Injector/gas ring	4	4	2	Inactive
N_2	100%	100	Injector maintenance	Injector/gas ring	1	0	0	Active
N_2	100%	2000	Pressure control	Pump	1	0	0	Active
N_2	100%	5000	Flush and backfill	Gas Ring	1	0	0	Active

As shown in Figure 2.1, process gases can be introduced into the reactor either through the gas ring located at the door (load) end of the tube or through the multi-pore injector located beneath the wafer boats. Silicon and germanium precursor gases are introduced from the door end through the gas ring; the dopant gases are introduced from the pump side via the injector. Introducing the dopant gases via the gas ring is also feasible. During deposition, reaction gases are consumed faster at the gas inlet and their partial pressures are depleted down the stream. The depletion effect across the load is more pronounced for gases introduced via the gas ring. The multi-pore injector helps to reduce the cross-load depletion effect by injecting gas at multiple pores along the load. The pores have increasing diameter along the line of gas flow to compensate the pressure loss along the stream. Since the injector pores are small, the pressure inside the injector is fairly high. Silicon and germanium precursor gases should not be introduced through the injector because the injector is at the deposition temperature and the injector pores will

quickly clog due to the high SiGe deposition rate inside the injector. On the other hand, the pores in the gas ring do not clog readily because they have large orifices and the gas manifold temperature is lower than the deposition chamber temperature.

The dopant gas can also clog up the injector but by a different mechanism. B_2H_6 can easily decompose into a solid polymer B_xH_y in the furnace operating temperature range [2.1]. The polymer slowly builds up inside the injector. After a certain threshold, the dopant gas flow can no longer be approximated as uniform, and the deposition results in cloudy film having high resistivity. An injector change is required at this point. The B_2H_6 doping process was terminated after the alternative boron dopant gas BCl_3 was successfully demonstrated.

The gas line for phosphine (PH_3) is also inactive. For post-CMOS SiGe deposition, low thermal budget is the essential requirement. Phosphine retards the deposition rate and extra annealing is required to activate the dopant [2.2]. With the precursor gases SiH_4, Si_2H_6 and GeH_4 introduced via the gas ring and BCl_3 introduced via the injector, there remain one precursor gas channel and one dopant gas channel available in the reactor for advanced process experiments.

2.3 Process recipes

Process recipes are stored in the control computer. Process temperature, process pressure, gas flow rates and deposition time are the variables in the recipe. The recipe has a pre-programmed process sequence. A typical deposition recipe consists of the following steps: pump/purge cycles after wafer loading, leak check, process parameters (temperature, gas flow rate and pressure) stabilization, deposition, and finally

17

pump/purge cycles to flush out the unreacted process gases and bring the pressure up to atmospheric pressure. Multiple depositions with different process parameters can be programmed into one recipe. If the process parameters are out of tolerance, the recipe will go to an abort sequence, which shuts off all the toxic gases and flushes the tube with nitrogen. The process can be re-directed to normal mode manually after trouble-shooting.

A standby recipe is loaded if the furnace is not running a deposition. The standby recipe consists of a 5-minute Si_2H_6 coating step at 450°C for conditioning purpose. After the coating step, the tube is flushed and held in N_2 ambient.

2.4 Wafer placement

About 50 wafers can be loaded vertically in the SiGe reactor. Wafers can be placed in either open wafer boats or caged wafer boats. Figure 2.2 shows both wafer boat configurations. Boats with different length and wafer spacing are readily available. Process gases can reach the wafers in open boat from all directions, whereas the gases can only enter through the slots of the caged boat. For mass transport-limited deposition, the deposition rate tends to be higher towards the wafer edge for open boat configuration due to diffusion effects. In such a case, caged boat can improve the cross-wafer uniformity significantly. If the deposition is surface reaction-limited, the uniformity is about the same for both wafer boat configurations. In this case, the deposition rate is significantly lower for wafers sitting inside the caged boat due to the loading effect of the wafer boat surface. The surface area of the caged boat is about the same as that of the wafers sitting inside. Process gases are consumed by the deposition on the wafer boat surface.
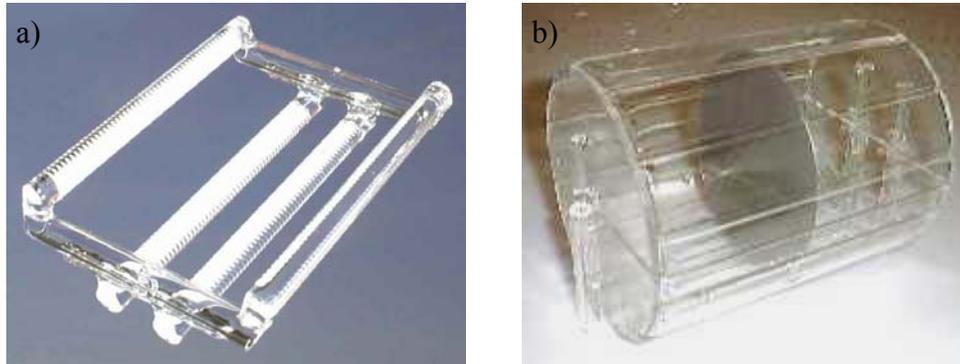
Figure. 2.2 a) Open boat; b) Caged boat [2.3]

Since an LPCVD system is a batch reactor, the throughput can be increased by using the maximum number of process wafers. However, there are tradeoffs between throughput and uniformity. The number of wafers in the reactor is limited by the length of the temperature flat zone and by gas transport. Process temperature has less fluctuation at the center of the tube. The process gases are consumed as they travel down the tube, resulting in a higher deposition rate for wafers sitting near the gas inlet. There is also a limit on wafer spacing. If wafers are placed too close to each other, diffusion transport limitations could result in higher deposition rate at wafer edges.

Both 100 mm-diameter and 150 mm-diameter wafers can be placed on wafer boats sitting on the cantilevers. However, the 100 mm-diameter wafers are placed below the center axis in the 230 mm-diameter reactor and there is more open space for gas flow on the top of the wafer. By comparison, 150 mm-diameter wafers are nearly centered in the reactor, resulting in better cross-wafer deposition uniformity.

The placement of the wafers in the boat and the placement of the boat on the cantilevers both affect the characteristics of the deposited film. In order to achieve

reproducible results, consistency in wafer placement is necessary. Wafers sitting at the leading and trailing edges of the load usually have worse uniformity due to vortexes. Therefore, dummy wafers should be placed at the edges.

2.5 Quartz tube

The process tube is made of quartz. Since the reactor is a hot-wall system, deposition occurs on the quartz wall as well as on the wafers. The deposited SiGe film has good adhesion to the quartz wall. The film is compressive and the stress applied on the quartz wall increases as the film gets thicker. Also, poly-SiGe and quartz have different thermal expansion coefficients. The quartz tube will eventually crack due to stress and thermal cycling. In industry, the quartz tube is pulled out and cleaned regularly because it is very expensive to ruin a full load of wafers that have gone through many process steps. In an academic research laboratory, the cost of changing the quartz-ware is lower compared to that of regular cleaning. In this case, the quartz tube stays in the furnace until it cracks. Tube cracking is not a safety hazard, since the tube operates at low pressure during deposition, the toxic and flammable process gases cannot leak out unless the pump fails at the same time. Also the reactor is enclosed in an exhausted gas cabinet. In addition, the tube usually cracks during loading and unloading when temperature and pressure change significantly.

The quartz tube usually cracks near the door where there is a greater temperature gradient and the deposited film is thicker. A liner can be used to increase the lifetime of the tube. The liner is an extra piece of quartz cylinder inserted inside the tube that can significantly reduce the deposition on the tube. Since the vacuum is held by the tube,

process does not go down with small cracks on the liner wall unless it collapses. Recent year's process record shows that the lifetime of the quartz tube is about 200 hours of deposition, which corresponds to roughly 100 μm of film thickness.

A 5 minute leak monitor step is set up in all recipes to monitor the rate of the pressure rise in the tube. The furnace is hard-pumped in the previous step. The pump is then turned off for 5 minutes and pressure rise is measured. The initial intention for this monitor step was to correlate the rate of pressure rise and the quartz tube lifetime. No strong correlation between the rate of pressure rise and the quartz tube lifetime was found, but the origin of the pressure rise was determined.

The leak monitor step is set up in two different ways. In the standby recipe, the leak monitor was done quite early in the process sequence, before the temperature stabilization. For the deposition recipe, the leak monitor was done after the temperature stabilization. It turns out that the leak rate for the standby recipe is usually about 10 mTorr/min and always <1 mTorr/min for the deposition recipe. The standby recipe is usually loaded after users remove their wafers. The rising pressure is caused by moisture outgasing after loading. For the deposition recipe, the leak monitor was done after the quartz ware was baked out for more than an hour. If the standby recipe is run after the door is closed for a few hours, the rate of rise goes down significantly.

The pressure sensor is not good enough to measure the rate of pressure rise due to the real leaking since it is designed to measure the deposition pressure in the 100 mTorr range. The small leak rate results in an oxygen content in the poly-SiGe film in the order of 1×10^{19} cm^{-3}, compared to 1×10^{18} cm^{-3} in an industrial reactor with a N$_2$ load-lock chamber.

2.6 Operation control

2.6.1 Pressure control

The pressure in the furnace is controlled by the pump, the pressure gauge and the N_2Vac line using feedback. The base pressure can reach <1 mTorr while the pump is running at its full power and there is no gas flowing in the furnace. Typical rate of rise is 4 mTorr/min when the pump is turned off and the quartz tube is in good condition. Flowing process gases increase the pressure, yet the pressure is still typically below the desired process pressure. The process pressure is measured by the pressure gauge located near the door. To adjust the pressure, a controlled amount of nitrogen (N_2Vac line) is introduced to the pump to reduce its efficiency. For a particular amount of process gas flow, the minimum achievable process pressure is set by the pumping efficiency; the maximum achievable process pressure is set by the upper limit of N_2Vac flow used to reduce the pumping speed. A particular feedback setting can accommodate a range of pressure with a fixed total gas flow rate. With proper feedback setting, the usual settling time for the pressure is about 1 minute and the process is capable of pressure change during deposition.

2.6.2 Temperature control

The temperature control system includes a five-zone resistor coil heater and two sets of thermocouple tubes mounted inside and outside of the reactor. The two outer zones of the heater are called the guard zones. Since heat is lost faster at the door end and the pump end of the tube, the two guard-zone heaters run at higher powers than those in

the center flat zone. The center flat zone has three heaters, which makes temperature gradient control possible. The manufacturer's nominal operating temperature of this particular furnace is 600°C for the best temperature control. However, the desired deposition temperature for poly-SiGe is in the range of 400°C – 450°C and even lower for pure poly-Ge. For the low temperature range, the heater is only running at 20 – 30% of its full power. The feedback control between the heater and the thermocouple is digitized, and small changes can vary the temperature significantly at the low temperature range. As a result, the temperature profile during deposition is approximately a sinusoidal function with a peak-to-peak amplitude of 6°C and a period of 25 minutes. A typical temperature profile of a 425 °C deposition is shown in Figure 2.3. The temperature profiles recorded by the five thermocouples are labeled in the plot. The pump side and the door side temperatures have the most fluctuations. It usually takes an hour for the temperature to settle within ± 5°C of the set point.

Temperature calibration can help to stabilize the temperature faster. During the calibration session, the heater power for a particular temperature is stored in memory for future reference. This can significantly reduce the adjustment time during temperature stabilization. Since the heater condition changes over time, temperature calibration should be done regularly, especially after a power shutdown.
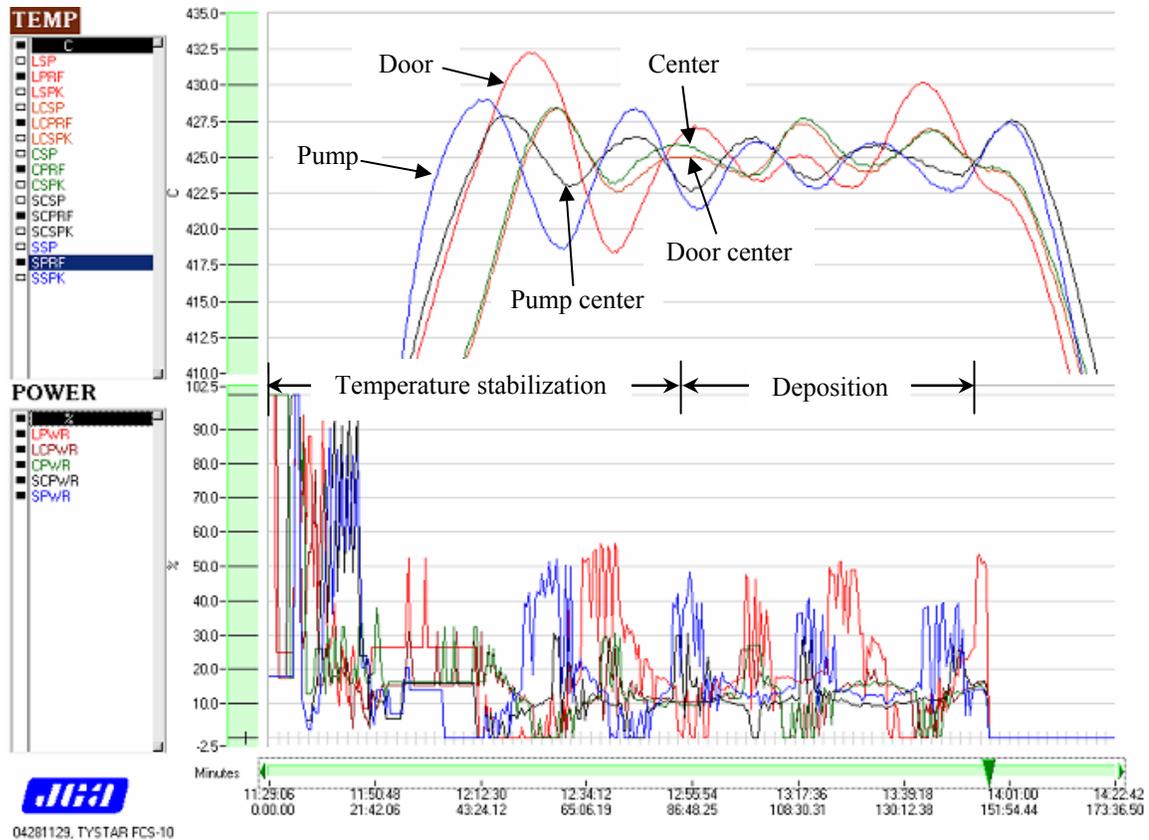
Fig. 2.3 Temperature and power profiles of a 425 °C deposition

2.6.3 Gas flow rate control

The flow rate of each gas is controlled by individual mass flow controllers (MFC). The MFC range for each gas is showed in Table 2.1 in sccm. The manufacture's specification is ±1% output error the for gas flow within 5% to 95% of the full range.

Figure 2.4 shows the schematic of a mass flow controller. It can be separated into two main components: a mass flow meter (MFM) and a proportional controller. The mass flow meter divides the flow between a heated sensing tube, where the mass flow is actually measured, and a flow bypass, where the majority of flow passes. Mass flow meters use the thermal properties of a gas to directly measure the mass flow rate. The

resistors wrapped around the sensing tube serve as both the heating and sensing elements. As the gas flow through the heated sensing tube, it absorbs some heat, and creates a temperature difference along the stream. The temperature difference between the two resistors is measured by a Wheatstone bridge so that mass flow in the sensor tube can be determined. Since each gas molecule has a specific ability to pick up heat, each MFC is calibrated to a particular gas or gas mixture. The other main component, the proportional controller, consists of a variable displacement solenoid valve and the control electronics. The controller drives the valve to the correct position so that the measured flow equals the desired flow set point.
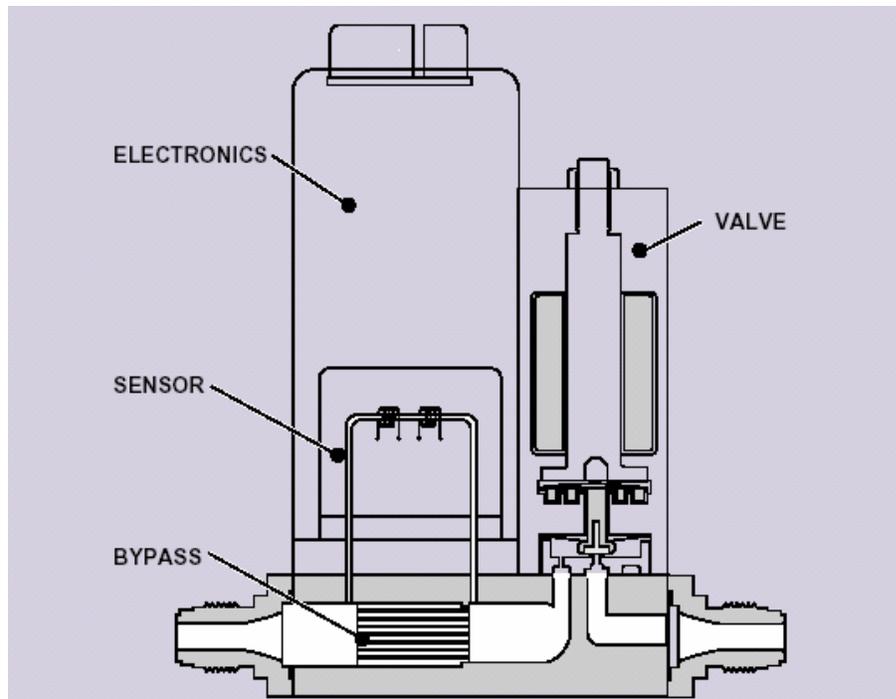
Fig. 2.4 Mass flow controller schematic [2.4]

2.7 Process monitor

2.7.1 Automatic process condition monitor

The furnace control computer has the capability to monitor the real-time process condition. The computer acquires data from the reactor every 30 seconds. Temperature, pressure, gas flow rates, gas valve conditions are recorded and archived in the computer. Figure 2.3 is a typical example of temperature and heater power for a deposition. The data acquisition runs 24 hours a day. This archival data is very useful for trouble shooting aborted depositions and maintaining statistical process control.

2.7.2 Process logbook

In additional to the automatic process monitoring, a process logbook (Appendix C) has been manually maintained since 2002 to better sustain the process. The process logbook contains the process condition for all depositions, problem reports and maintenance notes. Historical information of the reactor has been studied for failure analysis, design improvement and new process qualification.

2.7.3 Injector condition monitor

As discussed in Section 2.2, injector clogging caused by dopant gas B_2H_6 is a main challenge for uniformity and repeatability control. As shown in Figure 2.1, the injector is hooked up to the dopant gases and N_2Dope line. Dopant gas is used during deposition and N_2Dope is used during standby. A pressure gauge is mounted at the upstream of the injector to monitor the clogging condition. When there is some gas flowing through the injector, pressure will build up and it can be measured by the

pressure gauge. As the injector clogs, the pressure reading will go up. The reading of the pressure gauge depends on several factors:

- The gas flow rate through the injector

- The temperature of the tube

- The pressure of the tube

- The clogging condition of the injector

For injector monitoring purposes, the pressure gauge reading vs. the clogging condition is of highest interest. The simplest approach is to keep all other factors constant and make the clogging condition the only dependence of the pressure gauge reading. However, it is important to know how small fluctuations of other factors could affect the pressure gauge reading before taking the simplified approach. A monitor recipe can be chosen after identifying critical parameters in injector pressure gauge reading. The control limit for injector change can be determined by comparing a good injector and a clogged injector. To identify critical parameters in injector pressure gauge output, full factorial design is used because the experiment is neither time consuming nor expensive.

Flowing either B_2H_6/H_2 mixture or N_2dope generates a pressure inside the injector; therefore the injector condition monitoring could be done during either deposition or standby. For the interest of reducing B_2H_6 usage, N_2 is chosen as the monitoring gas. Since the N_2Dope MFC has full range of 100 sccm, outputting $10 - 90$ sccm of N_2 will be accurate. If $10 - 90$ sccm of N_2 is the only gas flow in the furnace, the pressure of the tube can be controlled between 100 to 900 mTorr. The pressure of the tube acts as an external load to the injector and therefore affects the injector pressure gauge reading. The operating temperature of the furnace is in the range of $300 - 450$ °C.

Since the gas flow in the injector is heated up inside the furnace, the temperature of the tube influences the pressure of the injector.

Based on the hardware limits, low, medium and high values are chosen for the gas flow rate, the tube pressure and the temperature. For the clogging condition, a new injector and a clogged injector are used in the experiment for comparison. The $3^3 \times 2$ full factorial design is summarized in Table 2.2. For each clogging condition, four replications were done at the center point where N_2Dope flow rate = 45 sccm, tube pressure = 500 mTorr and temperature = 400 °C.

TABLE 2.2 Full factorial design to identify critical parameter for injector condition monitoring

Variables	Settings		
N_2Dope flow rate (sccm)	10	45	90
Tube pressure (mTorr)	100	500	900
Temperature (°C)	350	400	450
Injector condition	New		Clogged

Multivariate analysis of variance (MANOVA) [2.5] in Table 2.3 shows that the N_2Dope flow rate and the clogging condition are the most statistically significant factors with large F ratio and small p-value. The interaction term of N_2Dope flow and the clogging condition is also very significant. The tube pressure does not matter, and the temperature term has a small contribution.

TABLE 2.3 Effect tests of parameter for injector pressure gauge reading

source	DF	Sum of Squares	Mean Square	F Ratio	Prob. > F
N_2Dope flow rate (sccm)	1	261.83	261.83	2552.7	< 0.0001
Tube pressure (mTorr)	1	0.2341	0.2341	2.2823	0.1373
Temperature (°C)	1	0.8639	0.8638	8.4220	0.0055
Injector condition	1	58.115	58.115	566.59	< 0.0001
N_2Dope (sccm) × Tube pressure (mTorr)	1	0.0027	0.0027	0.0266	0.8711
N_2Dope (sccm) × Temperature (°C)	1	0.1432	0.1431	1.3956	0.2432
Tube pressure (mTorr) × Temperature (°C)	1	0.0043	0.0043	0.0416	0.8392
N_2Dope (sccm) × Injector condition	1	7.5486	7.5486	73.594	< 0.0001
Tube pressure (mTorr) × Injector condition	1	0.00004	0.00004	0.0004	0.9835
Temperature (°C) × Injector condition	1	0.0659	0.0659	0.6423	0.4268

Parameter estimations in Table 2.4 show that the pressure gauge output increases with the N_2Dope flow rate and the clogging condition. The parameter of the interaction term of the N_2Dope flow rate and the clogging condition are also positive. Therefore, for the same clogging condition, high N_2Dope flow rate gives better sensitivity of the pressure gauge reading.

TABLE 2.4 Parameter estimates for injector pressure gauge reading

Term	Estimate	Std. Error	t Ratio	Prob. > |t|
Intercept	0.7442	0.4389	1.70	0.0963
N_2Dope flow rate (sccm)	0.0672	0.0013	50.5	< 0.0001
Tube pressure (mTorr)	0.0002	0.0001	1.51	0.1373
Temperature (°C)	0.0031	0.0011	2.90	0.0055
Injector condition	0.9842	0.0413	23.8	< 0.0001
(N_2Dope (sccm) - 45) × (Tube pressure (mTorr) - 500)	-6.649e-7	0.0000	-0.16	0.8711
(N_2Dope (sccm) - 45) × (Temperature (°C) - 400)	0.0000	0.0000	1.18	0.2432
(Tube pressure (mTorr) - 500) × (Temperature (°C) - 400)	-6.667e-7	0.0000	-0.20	0.8392
(N_2Dope (sccm) - 45) × Injector condition	0.0114	0.0013	8.58	< 0.0001
(Tube pressure (mTorr) - 500) × Injector condition	-0.0000	0.0001	-0.02	0.9835
(Temperature (°C) - 400) × Injector condition	-0.0009	0.0011	-0.80	0.4268

Knowing the parameter dependence of the pressure gauge reading, the regular monitoring of the injector condition can be simplified. A standby step is chosen for run-to-run monitoring. In this particular step:

- N_2Dope flow rate = 90 sccm

- Tube pressure ≈ 110 mTorr

- Temperature = 350 – 450°C

This is the standby condition the furnace should be in before the user loads the deposition recipe. All users are required to record injector condition data for every run. The nitrogen flow rate in this step is controlled by a mass flow controller, which is relatively reliable. The tube pressure has a small fluctuation due to variations in pumping efficiency. However, the tube pressure is the least significant factor for the injector pressure reading. Although the temperature set point is 350°C for this step, actual

temperature can vary from 350°C to 450°C because a 450°C coating is the prior step and it takes sometime for the tube to cool down. To make the regular monitoring user friendly, waiting for the temperature to stabilize to 350°C is not required. To choose the threshold for injector change, a new injector and a clogged injector are compared at various temperatures with 90 sccm of N_2 flow and 110 mTorr of tube pressure.

Comparing a new and a clogged injector in Figure 2.5 for the simplified monitoring condition, the control limit for an injector change is chosen as 9 Torr. This control limit is rather conservative and adds to the workload of the maintenance staff, but it is desirable for process stability.
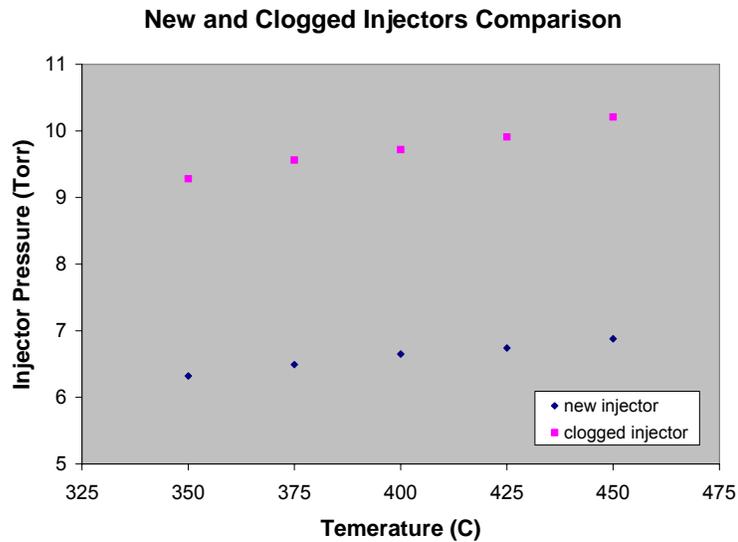
Figure 2.5: Pressure reading for a new injector and a clogged injector

Historical data of injector pressure since the injector monitoring was set up are shown in Figure 2.6. Before February 2004, B_2H_6 was the only boron dopant gas available. The data have a periodical pattern: injector pressure rises with B_2H_6 doped process usage and drops after the injector change. On average, injector change was done after ~ 20 hours of deposition. In some cases, when the deposition is not critical and some

users could not wait for the injector change, they used the injector above the control limit.

Injector Monitoring
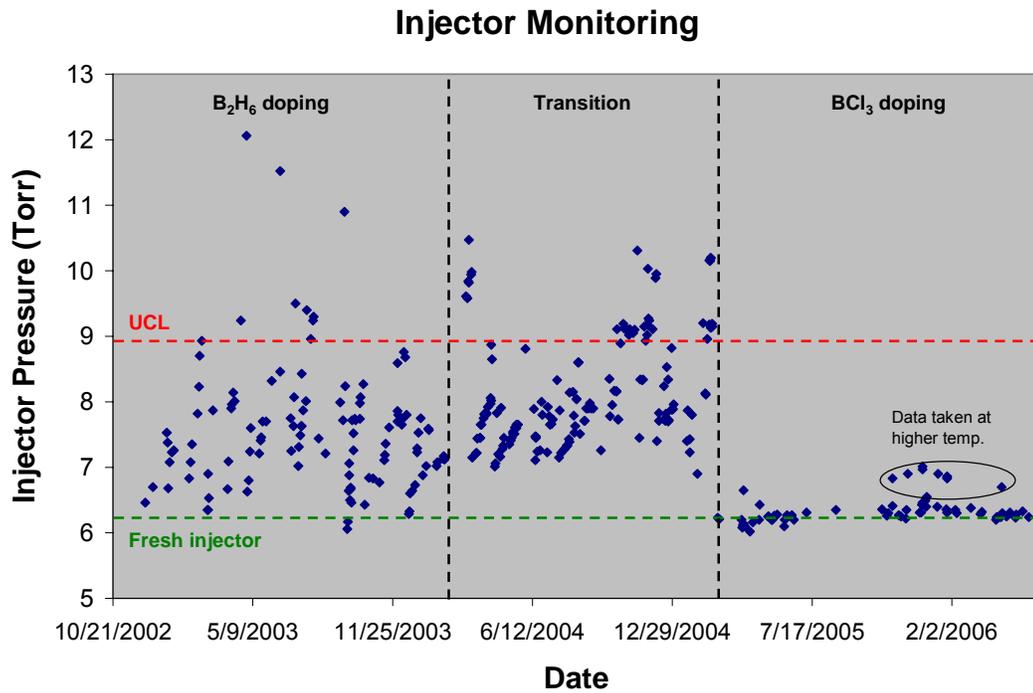
Figure 2.6 Historical data for injector pressure

Single-use quartz injector is not conducive for frequent changes due to the high risk of cracking. Stainless steel injectors have been used for a few years. Since a stainless steel injector is more expensive than a quartz injector, stainless steel injectors have been cleaned and reused over and over. The clogged injector can be drilled out in the machine shop and re-installed in the furnace. This cleaning method is not very satisfactory, as there is always some residue left after the drilling. It can be seen that the initial pressure of a "freshly clean" injector rises over time.

Pulling out the injector so frequently is a maintenance issue and process repeatability is still not guaranteed, due to the small drift of the injector condition over time. Also, contamination is always a concern for injectors coming back from the

machine shop.

BCl$_3$ doping was investigated since February 2004 [2.6]. Details of the BCl$_3$ doping process development will be presented in Chapter 3. The same technique has been used to monitor the injector pressure over time. Since BCl$_3$ is more thermally stable compared to B$_2$H$_6$ and it does not form a solid polymer inside the injector, injector clogging does not seem to be an issue (see Fig. 2.6). The B$_2$H$_6$ bottle was removed permanently from the reactor in March 2005 and a quartz injector was installed for the BCl$_3$ line. The injector pressure has been very stable and the injector lifetime is the same as other quartz-ware in the furnace.

2.7.4 MFC monitor

The SiGe film is deposited using SiH$_4$ and GeH$_4$. The deposition rate and the thin-film's mechanical properties depend strongly on the germanium content, which in turn depend on the outputs of the SiH$_4$ and GeH$_4$ mass flow controllers. The output gas flow rate could drift throughout the lifetime of the MFC. The most important attribute of an MFC for achieving run-to-run repeatability is not the accuracy, but the consistency. It is necessary to monitor the performance of the MFCs for process control.

If the MFC is taken out of the reactor, its output can be tested with nitrogen instead of the actual gas the MFC is calibrated to, such as SiH$_4$ or GeH$_4$. With the electronic set point entered and the input line hooked up to the nitrogen bottle, the nitrogen output will be regulated by the MFC, which can then be quantified with a trusted mass flow meter (MFM) calibrated to nitrogen. As discussed before, the mass flow measurement depends on the specific heat of the gas. Since nitrogen has a different

specific heat than the gas that the MFC is calibrated to, a correction factor (CF) should be used to convert the actual nitrogen output from the MFC being tested:

$$N_2 \text{ flow through a MFC calibrated to gas } A = \frac{set\ point}{CF} \tag{2.1}$$

Correction factor for the gases used in the SiGe reactor are listed in Table 2.5 below.

TABLE 2.5 N_2 equivalent correction factor (data from Unit Instruments application note)

Gas	Correction factor (CF)
SiH_4	0.603
Si_2H_6	0.321
GeH_4	0.591
1% BCl_3 in He $\approx$ pure He	1.399
N_2	1

Measuring the MFC with the external MFM is not a convenient way to monitor the performance of the MFC regularly. Pulling out the MFC from the gas line involves running a few pump/purge cycles and switching some valves in order to prevent contamination and to ensure safety. To track the MFC performance easier, an *in-situ* mass flow verification system is implemented with a mass flow meter installed at the downstream of all the MFCs in the reactor, as shown in Figure 2.1. The black dashed line shows the connection of the MFM loop. The MFM is bypassed during regular deposition to minimize its usage and it is activated only for the MFC monitoring purpose by some valve switching. All of the dopant gases are re-routed so that they can go through the MFM and enter the tube via the gas ring. Individual gas coming out from the MFC can flow through the MFM for calibration. The mass flow meter is calibrated to primary standard with N_2. In this case, the correction factor is used again to covert the actual gas flow:

$$gas\ X\ flow\ through\ a\ MFM\ calibrated\ to\ N_2 = MFM\ output \times CF \qquad (2.2)$$

For the monitoring recipe, all gases are directed to flow through the MFM individually. Three set points are chosen for each gas to check linearity. The range of the MFM is chosen to be 500 sccm of N_2, which accommodates most of the ranges of SiH_4, Si_2H_6, GeH_4 and N_2Dope and N_2BKFL as listed in Table 2.1. The BCl_3/He MFC range is too small for the MFM to resolve. To get around this problem, 90 sccm of N_2Dope is flowing together with the BCl_3/He mixture so that the total gas flow rate falls into the measurable range of the MFM. After the gas flow rate quantification, the MFM is flushed and cleaned with nitrogen flow.

MFC monitoring data using the internal MFM loop since November 2005 are presented in Figures 2.7 – 2.11. Both the SiH_4 and GeH_4 MFCs were pulled out of the reactor and measured with an external MFM in February 2005. The external MFM and the internal MFM give similar reading for the GeH_4 output. During the subsequent 9-month period, the SiH_4 MFC experienced a downward drift in gas output for some unknown reason, but the SiH_4 MFC output has stayed roughly constant since November 2005. Since consistency is more important than accuracy, the SiH_4 MFC was not changed out. It can be seen that the output of the Si_2H_6 MFC is slowly drifting higher. The Si_2H_6 flow rate is not very critical for process control because it is mainly used for standby coating and amorphous-Si seeding layer. The BCl_3/He mixture and N_2 MFCs are fairly constant. It can be seen that most of the data do not match the number calculated with Equation 2.2. The MFCs or the MFM might not be perfectly accurate, but consistency is more critical.

With the regular monitoring, it is possible to achieve run-to-run repeatability with drifting MFCs. The gas flow rate in the deposition recipe could be corrected to accommodate the change in the MFC output. For a more sophisticated system, feedback could be implemented for self-correction.
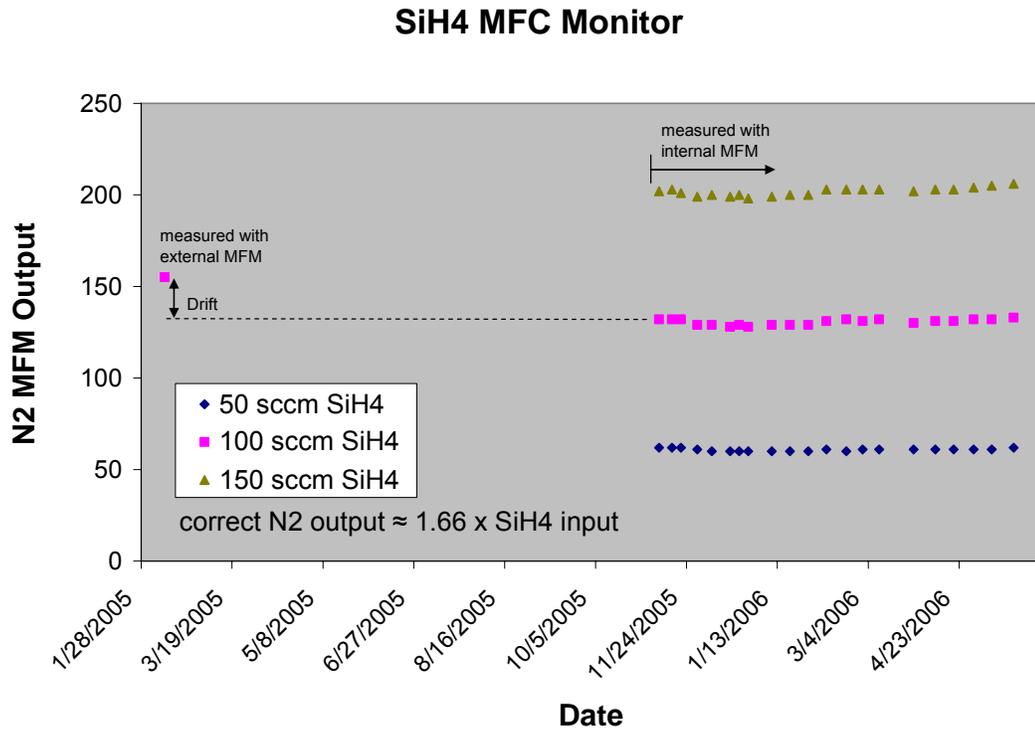
Figure 2.7 SiH$_4$ MFC monitoring data

GeH4 MFC Monitor

measured with
internal MFM →

measured with
external MFM

◆ 50 sccm GeH4
■ 100 sccm GeH4
▲ 150 sccm GeH4

correct N2 output ≈ 1.69 x GeH4 input

N2 MFM Output

Date
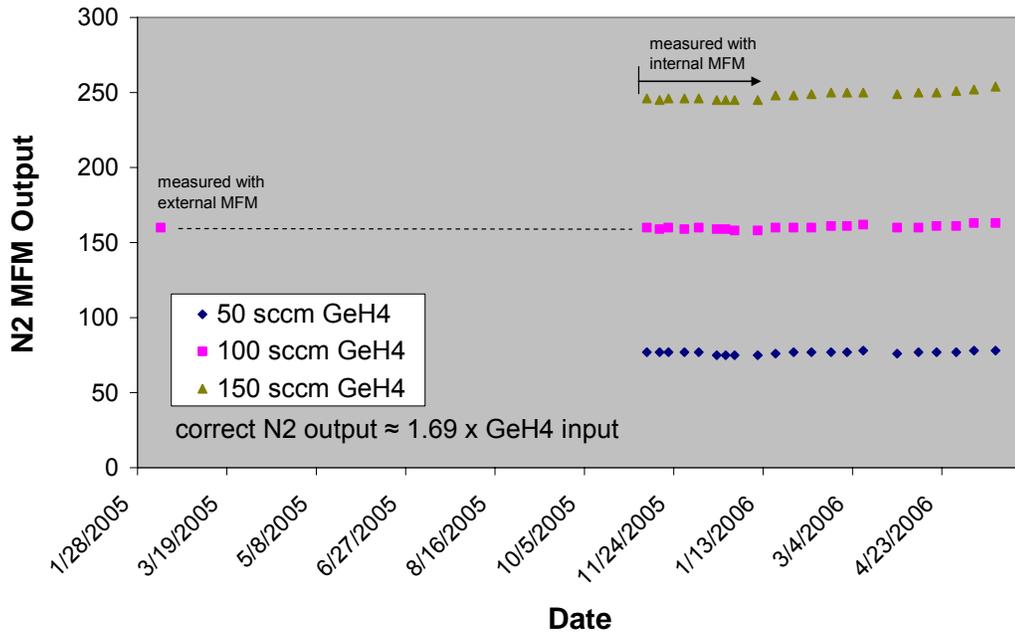
Figure 2.8 GeH_4 MFC monitoring data

Si2H6 MFC Monitor

measured with
internal MFM →

◆ 50 sccm Si2H6
■ 100 sccm Si2H6
▲ 150 sccm Si2H6

correct N2 output ≈ 3.12 x Si2H6 input

N2 MFM Output

Date
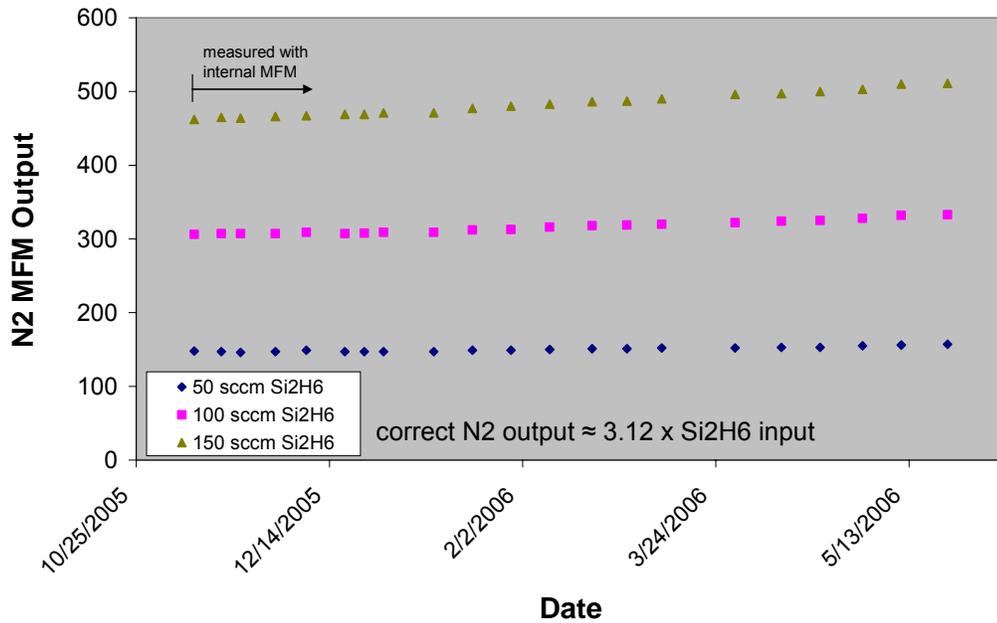
Figure 2.9 Si_2H_6 MFC monitoring data

BCl3 MFC Monitor
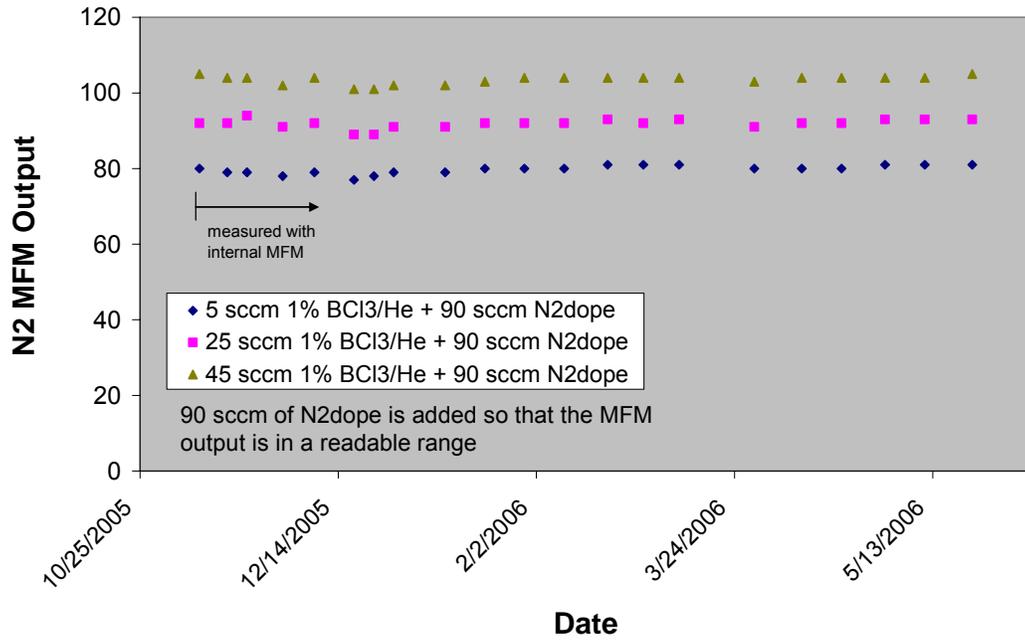
Figure 2.10 BCl$_3$ MFC monitoring data

N2 MFCs Monitor
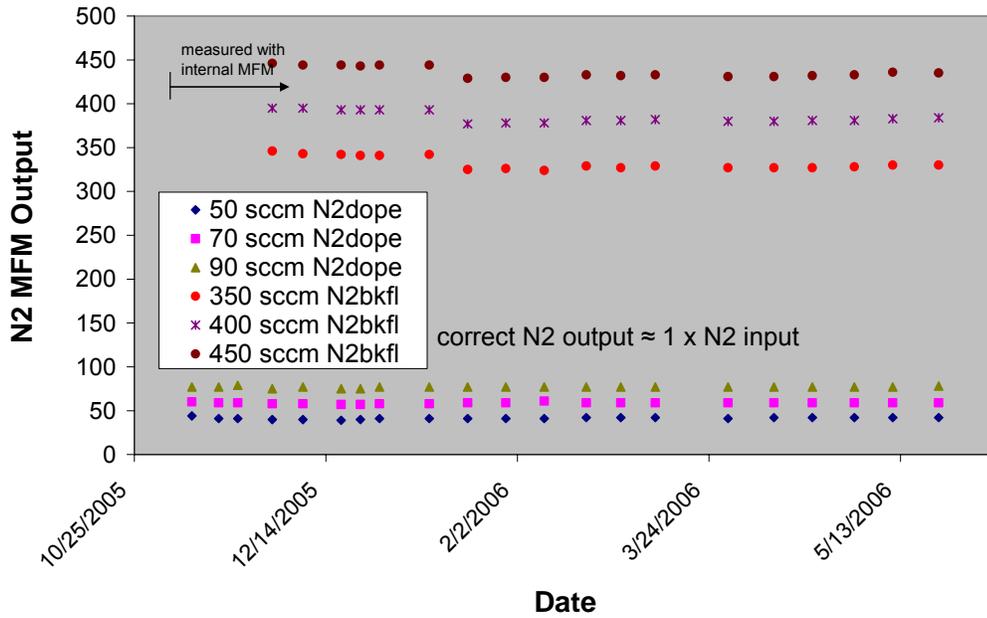
Figure 2.11 N$_2$Dope and N$_2$BKFL MFCs monitoring data

37

2.8 Summary

The initial design of the LPCVD poly-SiGe reactor was very similar to that of an LPCVD poly-Si reactor. Some special modifications were implemented for the poly-SiGe reactor because of the usage of multiple precursor gases and different dopant sources. With appropriate maintenance and regular process monitoring, the poly-SiGe reactor has fairly good performance in process uniformity and repeatability for academic research purposes.

References

[2.1] L. H. Long, "The mechanisms of thermal decomposition of diborane and of interconversion of the boranes, a reinterpretation of the evidence", *J. Inorg. Nucl. Chem*, vol. 32, 1970, pp. 1097-1115

[2.2] Y.-C. Jeon, T.-J. King and R. T. Howe, "Properties of phosphorous-doped poly-SiGe films for microelectromechanical system applications", *J. Electrochemical Society*, vol. 150(1), 2003, pp. H1-H6

[2.3] http://www.amquartz.com/wafercarriers.htm

[2.4] http://www.celerity.net/support/technical_data/technical_articles/2000_june.pdf, "MFC Mass Flow Controller", *European Semiconductor Magazine*, pp. 3 (2000)

[2.5] D. C. Montgomery, *Introduction to Statistical Quality Control*, 4th edition, John Wiley & Suns, Inc., 2001, pp. 571-672

[2.6] C. W. Low, M. L. Wasilik, H. Takeuchi, T.-J. King and R. T. Howe, "In-situ doped poly-SiGe LPCVD process using BCl_3 for post-CMOS integration of MEMS devices," in *Proc. Electrochemical Society SiGe Materials, Processing, and Devices Symposium*, Honolulu, HI, Oct. 3-8, 2004, pp. 1021-1032

Chapter 3: Investigation of Advanced Process Gases

Silane (SiH_4), germane (GeH_4), diborane (B_2H_6) and phosphine (PH_3) are the conventional precursor and dopant gases for SiGe deposition with LPCVD, PECVD and UHV-CVD processes. LPCVD poly-SiGe MEMS technology has unique challenges in achieving good process control with large batch sizes and limiting the thermal budget for post-CMOS processing. In an effort to improve the process control and deposition rate, advanced process gases have been investigated. This chapter discusses the developments and challenges of using boron trichloride (BCl_3) as a dopant gas, disilane (Si_2H_6) as a silicon precursor and germyl silanes ($(H_3Ge)_xSiH_{4-x}$) as single-source silicon and germanium precursors.

3.1 Boron trichloride (BCl_3) as the boron dopant gas

For MEMS applications, low resistivity is one of the requirements for the poly-SiGe structural layer. Ion implantation is not an attractive option since dopant activation with high temperature annealing increases the thermal budget for the post-CMOS process. Also, the mechanical properties would be hard to control with the non-uniform dopant distribution. *In-situ* doping does not have these disadvantages, but there are other process challenges. B_2H_6 and PH_3 are the conventional boron and phosphorous sources for *in-situ* doping. As mentioned in Section 2.2, dopant gas B_2H_6 clogs up the injector and makes the process difficult to control; PH_3 doping retards the deposition rate and requires post-deposition annealing to improve dopant activation. Recently, a high-throughput LPCVD process was developed using BCl_3 as the dopant source for epitaxial

SiGe growth [3.1, 3.2]. Also, BCl₃ is being studied for *in-situ* doping of poly-Si [3.3].

These results initiated the development of the BCl₃ doping process for poly-SiGe [3.4].

3.1.1 BCl₃ mixture concentration

To investigate the feasibility of using BCl₃ for the poly-SiGe process, the BCl₃

doped epi-SiGe process was studied [3.1, 3.2]. The epi-SiGe system is a vertical LPCVD

furnace, capable of processing fifty 200 mm-diameter wafers. A summary of the epitaxial

process conditions and data are listed in Table 3.1 below. Pure gas partial pressure is

shown instead of flow rate to normalize the difference in system size and pumping.

Hydrogen is used as the carrier gas in the epi-SiGe system.

TABLE 3.1 Summary of the BCl₃ doped epi-SiGe process [3.1, 3.2]

Temp. (°C)	Pressure (mTorr)	SiH₄ (mTorr)	GeH₄ (mTorr)	BCl₃ (mTorr)	Doping (cm^{-3})	Resistivity (mΩ-cm)	Ge content	Dep. Rate (nm/min)
500	228	45.6	0.912	0	NA	NA	10%	0.5
500	228	45.6	2.28	0	NA	NA	20%	1.5
500	228	45.6	4.1	0	NA	NA	30%	3
470	228	39.5	2.7	1.5×10^{-4}	4×10^{18}	NA	26.7%	NA
470	228	39.5	2.7	1.7×10^{-3}	8×10^{19}	1	26.7%	0.6
470	228	39.5	2.7	4.6×10^{-3}	1×10^{20}	NA	26.7%	NA

Although there are significant differences between the reactors and the processes,

the target doping concentration for the poly-SiGe film is in the same order of magnitude

as that of the epi-SiGe film. To install a BCl₃ bottle to the poly-SiGe reactor, the gas

concentration and the MFC range need to be specified. To roughly match the doping

level of 8×10^{19} cm⁻³, the gas flow rate of pure BCl₃ can be calculated as:

BCl_3 *flow rate in the poly reactor*

$$= \frac{BCl_3 \; pressure \; in \; the \; epi \; reactor}{SiH_4 \; pressure \; in \; the \; epi \; reactor} \times SiH_4 \; flow \; rate \; in \; the \; poly \; reactor$$

$$= \frac{1.7 \times 10^{-3} \; mTorr}{39.5 \; mTorr} \times 100 \; sccm$$

$$= 0.0043 \; sccm$$

(3.1)

The volume of epi-SiGe reactor is about 6× greater than the volume of the poly-SiGe reactor. Also, 1000 sccm of pure SiH_4 and 42 sccm of 0.1% BCl_3 are used for the epitaxial deposition. Assuming 100 sccm of SiH_4 flow in the poly-SiGe reactor, the calculation in Equation 3.1 is in good agreement with the epi-SiGe reactor's BCl_3 flow rate. The pure BCl_3 flow rate of 0.0043 sccm is very difficult to control with a mass flow controller. If the dopant gas is diluted, a larger flow rate can be used. In this case, flowing 4.3 sccm of 0.1% BCl_3 would be reasonable to control with a 10 sccm range MFC.

As a comparison, 5% of BCl_3 diluted in He is used in the epi-SiGe reactor. The gas is further diluted down to 0.1% in H_2 in the system before getting into the deposition chamber. Since the poly-SiGe reactor does not have the capability to dilute the dopant gas in the system, using a 0.1% concentration gas bottle would be more convenient. However, this simplified approach limits the flexibility of adjusting the dopant concentration if the doping level does not come out as expected. To keep the cost of installation low, 0.1% BCl_3 diluted in He was first used as the dopant gas. Preliminary results of the BCl_3 doped process using the 0.1% concentration bottle are summarized in Table 3.2.

TABLE 3.2 Summary of the BCl$_3$ doped poly-SiGe process with 0.1% concentration bottle

Temp. (°C)	Pressure (mTorr)	SiH$_4$ (mTorr)	GeH$_4$ (mTorr)	BCl$_3$ (mTorr)	Doping (cm^{-3})	Resistivity (mΩ-cm)	Ge content	Dep. Rate (nm/min)
425	400	247	148	5.0×10^{-3}	1.4×10^{17}	790	70%	8.6
425	400	236	141	2.3×10^{-2}	5.6×10^{17}	264	70%	8.9
425	400	227	136	4.1×10^{-2}	5.4×10^{18}	93.6	70%	8.8
425	400	225	136	4.1×10^{-2}	5.2×10^{18}	100	70%	8.9
425	400	165	99	1.4×10^{-1}	2.2×10^{19}	32	70%	3.1

The resistivity of the poly-SiGe films deposited using the 0.1% concentration BCl$_3$ bottle is much higher than desired. The last run listed in Table 3.2 has a dopant concentration closer to the desired range, but the deposition rate is significantly lower than in other runs. In this case, the SiH$_4$ and GeH$_4$ flow rates are scaled down by 5× so that the BCl$_3$ partial pressure is increased. Comparing results in Table 3.1 and Table 3.2, the offset in boron incorporation between the epi-SiGe and poly-SiGe films is about two orders of magnitude. These results show that neglecting the mismatches between the epi-SiGe and the poly-SiGe processes in Equation 3.1 is not a valid assumption. The difference in dopant incorporation of the two systems comes from several sources: the deposition temperature of the poly-SiGe film is much lower; the oxygen contamination level of the poly-SiGe system is an order of magnitude higher; the poly-SiGe film is deposited on an oxide surface, whereas the epi-SiGe film is deposited on an ultra-clean Si surface; the germanium content of the poly-SiGe film is much higher, and the deposition rate of the poly-SiGe film is more than 10× faster than that of the epi-SiGe.

Since the BCl$_3$ doped epi-SiGe process was developed for the same reason that dopant gas B$_2$H$_6$ does not yield satisfactory process stability, a comparison of the results for both dopant gases in the epi-SiGe process and then scaling with the B$_2$H$_6$-doped poly-SiGe process can lead to a more accurate calculation of the appropriate BCl$_3$ bottle

concentration for the poly-SiGe process. Table 3.3 and Table 3.4 show the results of the

B_2H_6 doped epi-SiGe and poly-SiGe processes, respectively.

TABLE 3.3 Summary of the B_2H_6 doped epi-SiGe process [3.5]

Temp. (°C)	Pressure (mTorr)	SiH₄ (mTorr)	GeH₄ (mTorr)	B₂H₆ (mTorr)	Doping (cm⁻³)	Resistivity (mΩ-cm)	Ge content	Dep. Rate (nm/min)
550	228	45.6	1.52	3.8×10^{-2}	8.0×10^{19}	2	22%	7
550	228	45.6	15.2	2.0×10^{-3}	1.5×10^{19}	7	60%	60

TABLE 3.4 Summary of the B_2H_6 doped poly-SiGe process

Temp. (°C)	Pressure (mTorr)	SiH₄ (mTorr)	GeH₄ (mTorr)	B₂H₆ (mTorr)	Doping (cm⁻³)	Resistivity (mΩ-cm)	Ge content	Dep. Rate (nm/min)
450	600	275	160	16.4	1.2×10^{19}	37	65%	15

Comparing the results of the B_2H_6 doped epi-SiGe and poly-SiGe processes, there

is also an offset in dopant incorporation for the two processes. To recalculate the

appropriate BCl_3 bottle concentration for the poly-SiGe process, one approach is to take

the ratio of the BCl_3 and B_2H_6 doped processes with the same boron concentration:

$$
\begin{aligned}
BCl_3 \; & \textit{pressure in the poly reactor} \\
&= \frac{BCl_3 \; \textit{pressure in the epi reactor}}{B_2H_6 \; \textit{pressure in the epi reactor}} \times B_2H_6 \; \textit{pressure in the poly reactor} \\
&= \frac{1.7 \times 10^{-3} \; mTorr}{3.8 \times 10^{-2} \; mTorr} \times 16.4 \; mTorr \\
&= 0.73 \; mTorr \quad (\textit{assume } 600 \; mTorr \; \textit{process pressure}, 1.2 \times 10^{19} \; cm^{-3} \; \textit{boron conc.}) \\
&\textit{or } 0.49 \; mTorr \quad (\textit{assume } 400 \; mTorr \; \textit{process pressure}, 1.2 \times 10^{19} \; cm^{-3} \; \textit{boron conc.})
\end{aligned}
\tag{3.2}
$$

Assuming that the process pressure is 400 mTorr and the gas flow rates of SiH₄, GeH₄

and BCl₃ are 100 sccm, 60 sccm and 10 sccm, respectively; the BCl₃ concentration can

be calculated as:

$$
\frac{10 \; sccm \; BCl_3 \; mixture \times BCl_3 \; concentration}{100 \; sccm \; SiH_4 + 60 \; sccm \; GeH_4 + 10 \; sccm \; BCl_3 \; mixture} \times 400 \; mTorr = 0.49 \; mTorr
\tag{3.3}
$$

$$
\Rightarrow BCl_3 \; concentration = 2\% \quad (\textit{for } 1.2 \times 10^{19} \; cm^{-3} \; \textit{boron concentration})
$$

The calculations in Equations 3.2 and 3.3 neglect the effects of temperature and

deposition rate on boron incorporation in the film.

The other approach to estimate the appropriate BCl$_3$ bottle concentration is to extrapolate information from Table 3.2, where all the data are for the BCl$_3$ doped poly-SiGe process. A curve fitting for data in Table 3.2 yields the relationship:

$$boron\,concentraton = 5 \times 10^{17}\,e^{34.1 \times BCl_3\,pressure}$$
$$\Rightarrow 0.099\,mTorr\,BCl_3\,pressure\,yields\,1.2 \times 10^{19}\,cm^{-3}\,boron\,concentraton \qquad (3.4)$$
$$or\,0.165\,mTorr\,BCl_3\,pressure\,yields\,1.0 \times 10^{20}\,cm^{-3}\,boron\,concentraton$$

Again, assuming 400 mTorr process pressure and the gas flow rates of SiH$_4$, GeH$_4$ and BCl$_3$ mixture are 100 sccm, 60 sccm and 10 sccm, respectively, the BCl$_3$ concentration can be calculated as:

$$\frac{10\,sccm\,BCl_3\,mixture \times BCl_3\,concentration}{100\,sccm\,SiH_4 + 60\,sccm\,GeH_4 + 10\,sccm\,BCl_3\,mixture} \times 400\,mTorr = 0.099\,mTorr$$
$$\Rightarrow BCl_3\,concentration = 0.42\% \quad (for\,1.2 \times 10^{19}\,cm^{-3}\,boron\,concentration) \qquad (3.5)$$
$$or\,BCl_3\,concentration = 0.7\% \quad (for\,1.0 \times 10^{20}\,cm^{-3}\,boron\,concentration)$$

The two approaches above both yield numbers larger than 0.1% BCl$_3$ concentration for the desired doping level although calculation with Equations 3.4 and 3.5 is more reliable. Since the target resistivity for poly-SiGe film is less than 10 mΩ-cm, the boron doping concentration should be in the range of 1.0×10^{20} cm^{-3}. According to Equation 3.5, a 1% concentration would be appropriate. If the boron doping level is slightly off target with the 1% concentration BCl$_3$ mixture, the gas flow rate can be adjusted to accommodate. Further experiments justified that the 1% concentration is appropriate for LPCVD poly-SiGe deposition.

It should be noted that pure BCl$_3$ is a liquid at room temperature with a vapor pressure of 988 Torr. Pumping the gas into the reactor was once a concern. However, when BCl$_3$ is diluted with a gas that liquifies at much higher pressure, the overall liquification pressure of the mixture is higher than that of pure BCl$_3$. With lower BCl$_3$

concentration in the mixture, liquification pressure of the overall mix is higher. The inert gas helium is used to dilute BCl_3. The mixture of 0.1% or 1% BCl_3 balanced in He is in gas phase at the maximum pressure limit of the tank. The gas mixture was made with gravimetric blending for which each individual gas is weighted while pouring them into cylinder.

3.1.2 Experimental details

The epi-SiGe reactor that first demonstrated the BCl_3 doped process is a vertical LPCVD system with all the process gases introduced via one gas inlet and an open boat for automatic loading [3.2]. The epi-SiGe reactor is capable of processing fifty 200 mm-diameter wafers and across-load uniformity is within ± 5% for resistivity and ± 2% for film thickness.

The initial test of the BCl_3 doped poly-SiGe process used a similar configuration as the epi-SiGe reactor. SiH_4 and GeH_4 were introduced at the gas ring located at the load side of the tube. The BCl_3/He mixture was also introduced through the gas ring for these experiments, even though introducing BCl_3 via a multi-pore injector located at the bottom of the wafer boats is another option. Twenty-five 4"-diameter and twenty-five 6"-diameter wafers placed in open boats at the center of the reactor were used per load. To investigate the feasibility of the process, the deposition rate, crystallinity, dopant incorporation, resistivity, residual stress, strain gradient, as well as effects of thickness on electrical and mechanical properties were characterized [3.4].

The process conditions of various depositions are summarized in Table 3.5. Poly-SiGe films were deposited onto Si wafers coated with ~2 μm thick low-temperature

(450°C) LPCVD SiO_2. A ~5 nm thick undoped amorphous Si (a-Si) seeding layer was deposited first using 100 sccm of Si_2H_6 to promote adhesion of SiGe to SiO_2. The a-Si layer was deposited at 300 mTorr for 15 minutes at various temperatures. For the poly-SiGe deposition, the process pressure was held constant at 400 mTorr. Temperature, BCl_3 partial pressure, and deposition time were varied. The SiH_4 to GeH_4 gas flow ratio was held constant in order to target a 70% Ge content. The flow rates of the two gases however were reduced in some cases to increase the partial pressure of BCl_3. The high Ge content was chosen to ensure crystallinity for consistent resistivity measurement with boron doping. It should be noted that the selectivity of pure Ge to $Si_{30}Ge_{70}$ for H_2O_2 etching is degraded to 10:1 [3.6]. High peroxide etching selectivity is desired for integrated MEMS applications because the use of pure Ge as the conformal sacrificial layer eliminates the need to passivate the underlying CMOS.

Experimental data was collected with five 4"-diameter wafers that were placed in slots 3, 8, 13, 18, and 23 of the wafer boats, counting from the gas inlet side. A four-point probe instrument was used to measure the sheet resistance. The films were patterned and etched for the thickness measurement using a stylus-based profiler. Wafer curvature was measured before and after SiGe deposition (backside SiGe film removed) to determine the average residual stress of the film. A cantilever beam array was patterned and released for strain gradient measurement. The strain gradient was calculated as the reciprocal of the radius of curvature of the cantilevers with various lengths. Resistivity, thickness, and strain gradient were measured at various points on each wafer, and average numbers are reported here. Ge content, B and Cl concentrations were determined by secondary ion mass spectroscopy (SIMS) with wafers at the gas inlet side of the load. The

47

crystallinity of selected films was determined by transmission electron microscopy.

3.1.3 Results and discussion

Overall results of average deposition rate, resistivity, residual stress, Ge content, and B doping level, along with uniformity of the BCl_3 and B_2H_6 doped poly-SiGe processes are summarized in Table 3.5. Both B_2H_6 and BCl_3 doped SiGe films have similar Cl concentration, all below 2×10^{16} cm^{-3}, which indicates Cl incorporation is not a problem for the BCl_3 doping process.

Table 3.5 Results summary for BCl$_3$ doped poly-SiGe process verification

| Run # | Deposition Recipe | | | | | | | Results | | | | | | | | | | | |
| | a-Si | SiGe layer | | | | | | Entire Load | | | | | | | | Gas Inlet Side (near door) | | |
	Temp (°C)	Temp (°C)	SiH$_4$ (sccm)	GeH$_4$ (sccm)	BCl$_3$/He (sccm)	BCl$_3$ conc.	Time (min)	Avg. DR (nm/min)	DR XW unif.	DR XL unif.	Avg. Res. (mΩ-cm)	Res. XW unif.	Res. XL unif.	Avg. stress (MPa)	Stress stdev	Ge cont.	B. conc. (cm^{-3})	Res. (mΩ-cm)
T22	450	350	0	100	16.5	0.1%	60	7	2%	3%	70	7%	12%	-240	26	100%	8.0×10^{18}	65
T2	400	400	100	60	9.2	0.1%	60	4.4	3%	5%	673	32%	68%	-268	21	74%	3.4×10^{18}	180
T4	400	400	100	60	16.5	0.1%	60	4.5	2%	2%	175	21%	60%	-301	13	74%	1.0×10^{19}	58
T5	425	425	100	60	1.8	0.1%	60	8.6	3%	8%	893	4%	10%	-74	16	70%	1.4×10^{17}	790
T1	425	425	100	60	9.2	0.1%	60	8.9	2%	6%	412	7%	25%	-61	16	70%	5.6×10^{17}	264
T6	425	425	100	60	16.5	0.1%	60	8.8	2%	6%	199	10%	41%	-100	17	70%	5.8×10^{18}	93.6
T11	425	425	100	60	16.5	0.1%	60	8.9	2%	6%	221	12%	41%	-94	26	70%	5.2×10^{18}	100
T14	425	425	100	60	16.5	0.1%	120	8.7	1%	6%	131	13%	46%	-70	18	70%	6.2×10^{18}	54
T12	425	425	100	60	16.5	0.1%	180	8.8	2%	7%	110	13%	48%	-34	9	70%	6.0×10^{18}	46
T17	425	425	80	48	16.5	0.1%	90	7.6	2%	8%	180	12%	46%	-70	11	70%	6.9×10^{18}	77
T18	425	425	50	30	16.5	0.1%	120	5.7	2%	9%	142	13%	50%	-139	19	68%	9.3×10^{18}	58
T9	425	425	20	12	16.5	0.1%	180	3.1	NA	NA	45	NA	NA	-99	8	64%	2.2×10^{19}	32
T3	450	450	100	60	9.2	0.1%	60	13.4	2%	8%	655	5%	20%	-15	9	67%	3.3×10^{17}	472
T19	425	425	100	60	3.6	1%	60	8.5	3%	8%	69	17%	64%	-130	18	73%	2.8×10^{19}	21
T20	425	425	100	60	9.2	1%	60	8.4	3%	9%	9.3	15%	60%	NA	NA	73%	1.5×10^{20}	3.6
T23	425	425	100	60	9.2	1%	60	8.4	3%	10%	9.2	16%	63%	-157	13	NA	NA	3.5
T21	425	425	100	60	16.5	1%	60	8.5	3%	12%	2.7	9%	40%	-163	14	73%	3.1×10^{20}	1.6
R3*	400	400	100	60	60 B$_2$H$_6$	10%	60	7.9	2%	3%	0.87	2%	3%	-240	28	NA	NA	0.90
R1*	425	425	100	60	60 B$_2$H$_6$	10%	60	9.2	2%	9%	7.2	7%	86%	-108	34	72%	4.0×10^{19}	16
R2*	450	450	100	60	60 B$_2$H$_6$	10%	60	13.1	2%	13%	65.7	6%	66%	-36	16	NA	NA	119

*B$_2$H$_6$ was introduced from the pump side through a multi-pore injector. Caged boats were used. Thirty 4"-diameter and fifteen 6"-diameter wafers were used per load.

49

3.1.3.1 Deposition rate

Comparison of Runs T5, T1, T6, T19, T20, and T21 with BCl_3 partial pressure as the only difference, the deposition rate does not depend strongly on the BCl_3 flow rate for the doping range studied here. Also, depositions with identical parameters but various deposition times (Runs T11, T14, and T12) show the same deposition rate, which suggests that there is no incubation period at the beginning of the deposition.

An Arrhenius plot of deposition rate is shown in Figure 3.1 for 100 sccm SiH_4, 60 sccm GeH_4 depositions at 400 mTorr for both BCl_3 and B_2H_6 processes. The deposition temperatures of these runs are rather low and the processes are believed to be operating in the surface reaction-limited regime. The activation energies of the BCl_3 and the B_2H_6 doped processes are extracted to be 0.94 eV and 0.42 eV, respectively. They are of the same order of magnitude as the undoped poly-SiGe process reported before [3.7]. For the 425°C and 450°C depositions, the deposition rates are similar for both dopant gases. B_2H_6 process has a much higher deposition rate at 400 °C. The resistivity of the 400 °C B_2H_6 recipe is very low, and boron incorporation for this film is estimated to be on the order of 1×10^{21} cm^{-3}. B_2H_6 is known to enhance deposition for poly-Si, but the temperature effect and the doping effect cannot be distinguished in Figure 3.1.
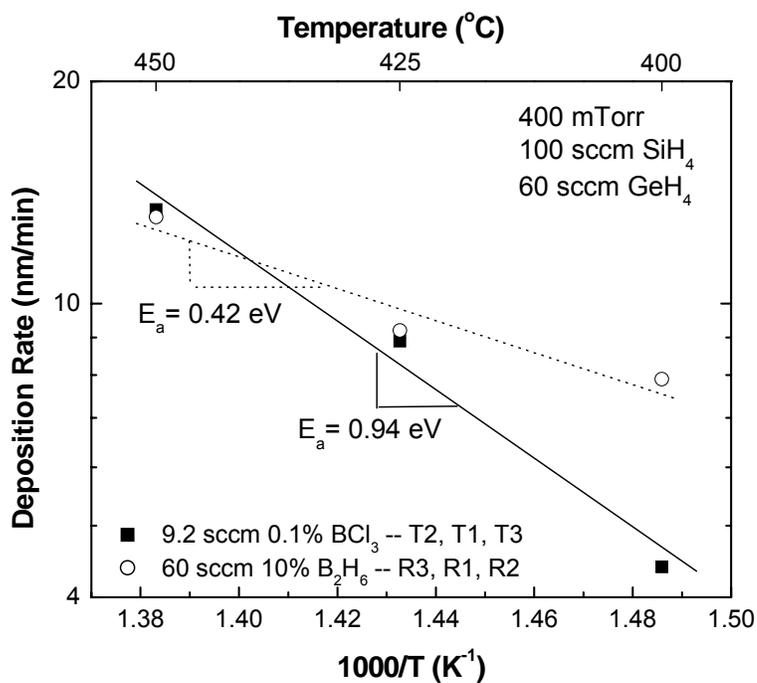
Figure 3.1 Arrhenius plot of deposition rate

3.1.3.2 Crystallinity

Cross-sectional TEM images for films of different thicknesses are shown in Figure 3.2. These two films have exactly the same recipe except the deposition times are 1 hour and 3 hours for the thin film and the thick film, respectively. Both films have vertically-oriented grain structure with finer grains at the bottom. The two TEM images are shown on the same scale. As the film gets thicker, the grains grow significantly larger.
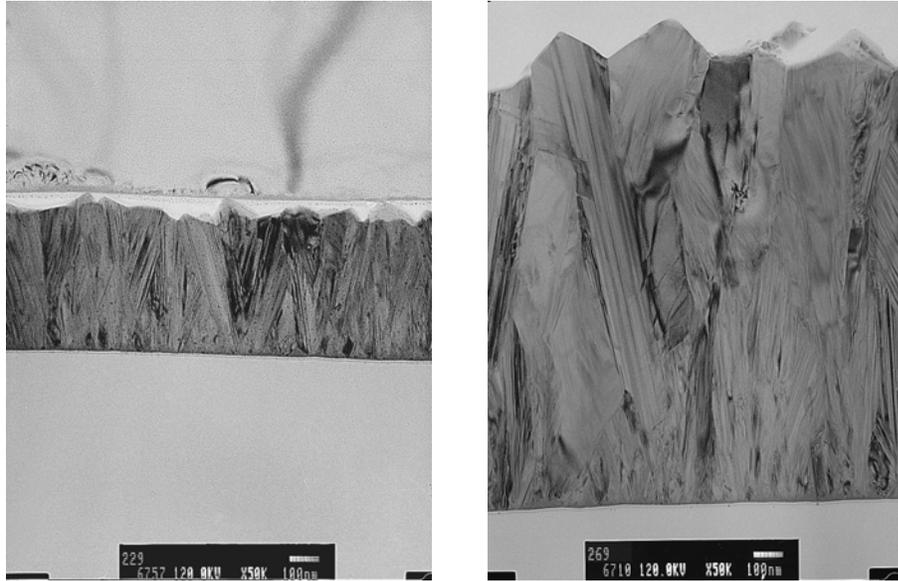
Figure 3.2 Cross-sectional TEM images: a) Run T6 - 0.5 um film; b) Run T12 - 1.5 um film

3.1.3.3 Dopant incorporation

Figure 3.3 plots boron concentration *vs.* partial pressure for all 425°C poly-SiGe depositions. As a comparison, data for epi-SiGe deposited at 470°C [C.1, C.2] is also shown. As discussed previously, the difference in dopant incorporation of the two systems comes from several sources: the deposition temperature, the oxygen contamination level, the deposition substrate, the germanium content and the deposition rate. All of the above differences result in more than two orders of magnitude offset in boron incorporation between the films.

A data point from the B_2H_6 process is also shown in Figure 3.3. A high B_2H_6 partial pressure is required to achieve similar doping levels in the film while other deposition conditions are identical. The consumption of B_2H_6 is mainly due to the

decomposition inside injector rather than the disassociation on the wafer surface. Comparing the three B_2H_6 depositions (R3, R1 and R2) at various temperatures provides further evidence of the temperature instability of B_2H_6. Resistivity is found to be an order of magnitude higher for an increment in temperature of 25°C with the same B_2H_6 flow rate.
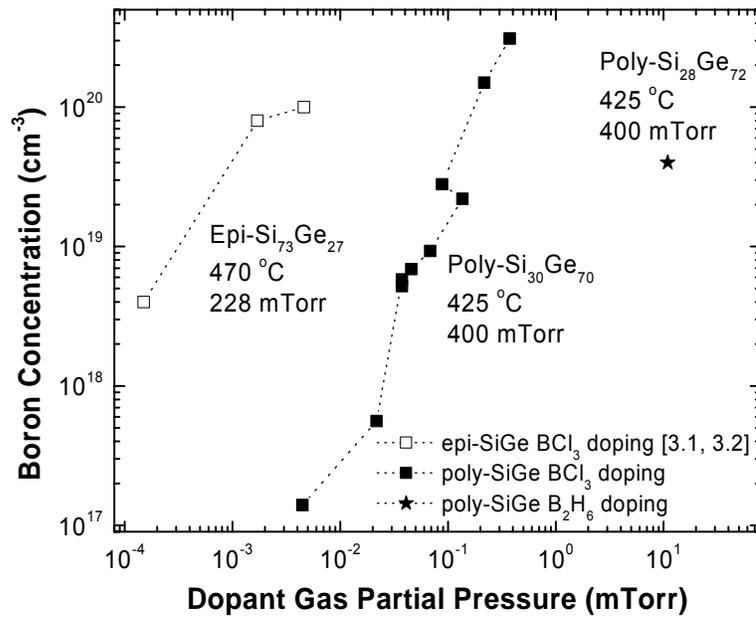
Figure 3.3 Boron concentration *vs.* dopant gas partial pressure

Resistivity vs. boron concentration for the BCl_3 doped process is plotted in Figure 3.4 for ~70% germanium content films deposited at 425°C with similar thicknesses. As expected, resistivity decreases linearly with boron doping. The resistivity of poly-SiGe is more than 10× higher than that of single crystalline films due to carrier trapping at the grain boundaries.
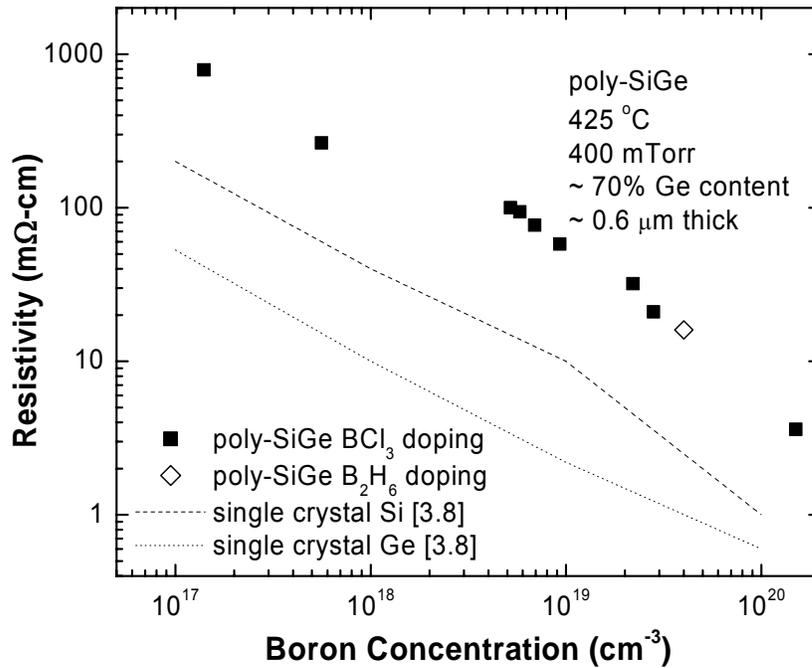
Figure 3.4 Resistivity *vs.* boron concentration

A comparison of BCl₃ runs (T1, T2, and T3) with same deposition conditions except for temperatures shows that higher temperature gives lower doping level and higher resistivity. This phenomenon could be explained by less efficient boron incorporation as the deposition rate goes up with temperature.

Resistivity *vs.* film thickness is plotted in Figure 3.5(a) for runs having the same deposition conditions but different deposition times. Wafer positions are also labeled on the graph. For wafers from the same run, higher resistivity and lower deposition rate are observed at the gas outlet due to the gas depletion effect. Comparing wafers at the same position from different runs, thicker films are found to have lower resistivity. This might be a result of furnace annealing and/or crystal growth during deposition. A two-hour 425°C anneal in N₂ ambient was done for several 1-hour deposition films to discriminate

the two effects. There is no change in resistivity after the annealing stage. As shown in Figure 3.2 earlier, thicker films have larger grains, which is consistent with this lower resistivity observation since carrier trapping is more significant with higher grain boundary density [3.9].
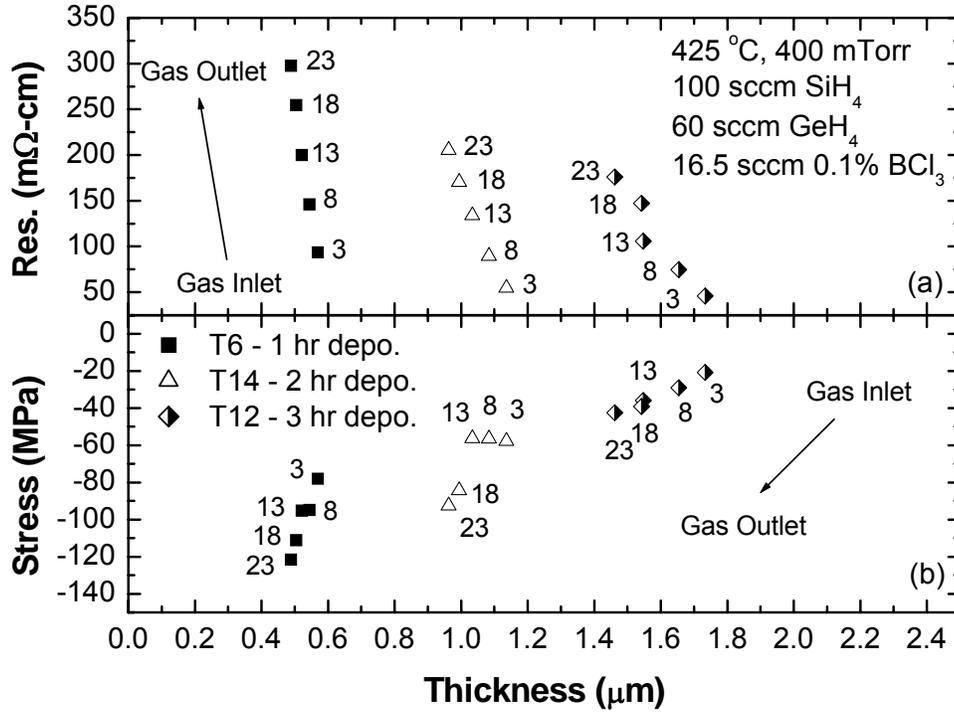
Figure 3.5 a) Resistivity and b) Stress *vs.* Film thickness

3.1.3.4 Stress

Figure 3.5(b) shows the average residual stress vs. film thickness. Thicker films have less compressive stress. Furnace annealing at 425 °C for 2 hours does not cause significant change in residual stress. This reduction in residual stress magnitude with thickness allows the growth of thick films without peeling off. However, the origins of

the residual stress and the reasons for its decrease with film thickness are not completely understood.

Comparing films with similar thickness and boron doping as the only process variable, we can see that the average residual stress is more compressive as boron doping increases, as shown in Figure 3.6. This possibly indicates the boron atoms are not residing in substitutional lattice sites, but interstitial lattice sites or grain boundaries. A data point from the B_2H_6 process shows that the average stress for B_2H_6 doped film is less compressive for the same level of doping. Further investigation on crystallinity might be able to explain this phenomenon. However, 0.6 μm films are too thin for most MEMS applications, and the 1.7 μm thick BCl_3 doped film with 6.0×10^{18} cm^{-3} doping yields reasonably low residual stress of -21 MPa.
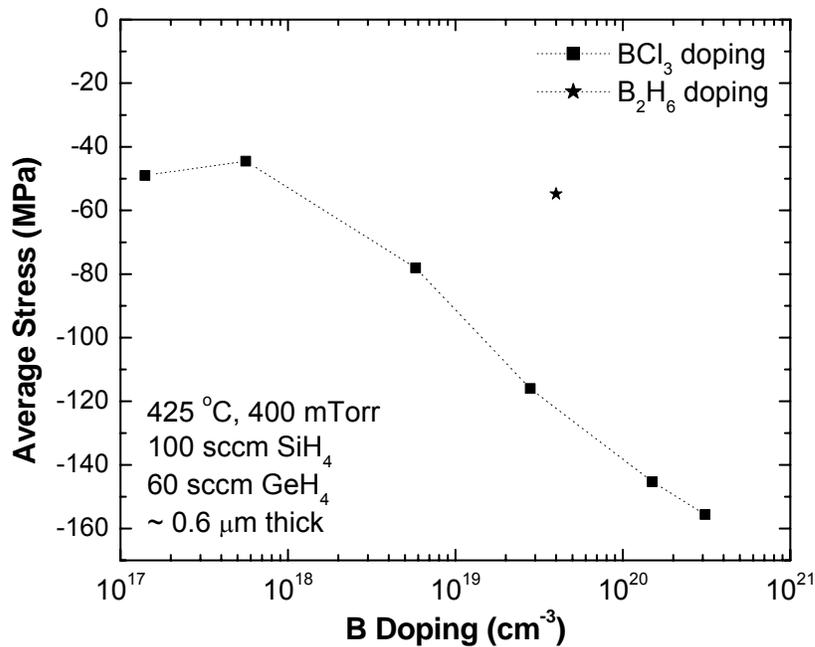
Figure 3.6 Average stress vs. doping

3.1.3.5 Strain gradient

Cantilever beam arrays from the thicker films were released for strain gradient measurement. Films with thickness less than 1 μm are too thin for reliable strain gradient measurements. As shown in Figure 3.7, the error increases as the film gets thinner and strain gradient is smaller for thicker film.
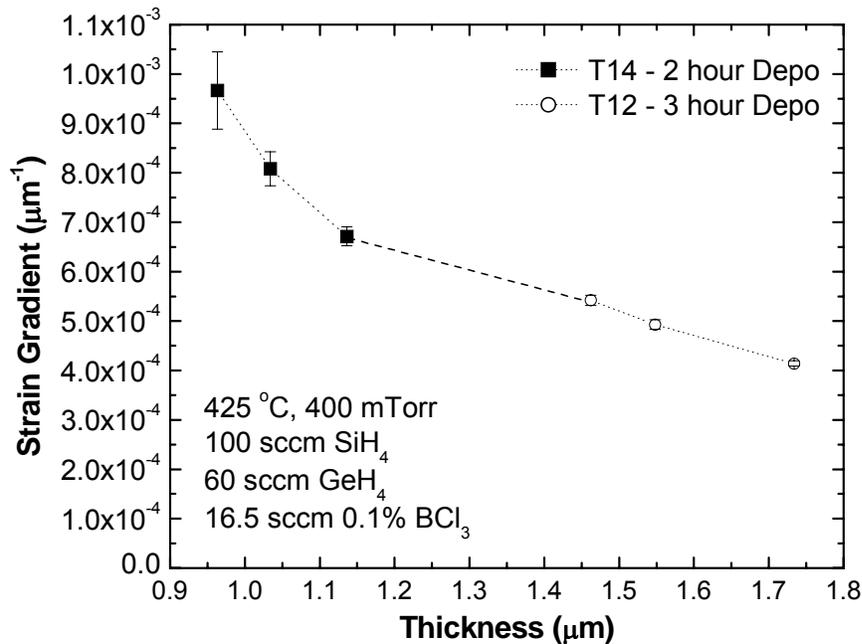
Figure 3.7 Strain gradient vs. thickness

3.1.3.6 Process uniformity

Process uniformity summarized in Table 3.5 is defined as the normalized standard deviation across the wafer and across the load. A few B_2H_6 doped depositions done in the same LPCVD reactor are listed at the bottom of Table 3.5 for comparison. The dopant gas B_2H_6 is introduced from the pump side via a multi-pore injector located underneath the wafer boats to minimize the depletion effect. Caged boats were used in the B_2H_6 doped process. As a comparison, the BCl_3 process provides similar uniformity within a

wafer and better cross load uniformity in most cases with open boats and without using a gas injector.

The wafer flat region has lower resistivity for the BCl_3 process, which is due to the fact that the 100 mm-diameter wafers are placed below the center axis in the 230 mm-diameter reactor and there is more open space for gas flow on the top of the wafer flat. As a comparison, 150 mm-diameter wafers, which are nearly centered in the reactor, have better cross-wafer uniformity in resistivity.

Deposition rate and resistivity of selected 425°C runs are plotted vs. wafer position in Figure 3.8 to demonstrate the cross load uniformity of the BCl_3 process. Wafers at the gas inlet side have higher deposition rate and lower resistivity, which suggests a gas depletion effect. Increasing the gas flow rate together with using an injector to introduce BCl_3 could result in better cross-load uniformity. A 425 °C B_2H_6 deposition is also shown in Figure 3.8 as a reference. Cross-load variation in deposition rate and resistivity is similar to the BCl_3 process, despite the fact that B_2H_6 is introduced via a multi-pore injector. Cross-load resistivity of the B_2H_6 shows the opposite trend compared to the BCl_3 process, because B_2H_6 was introduced from the pump side.
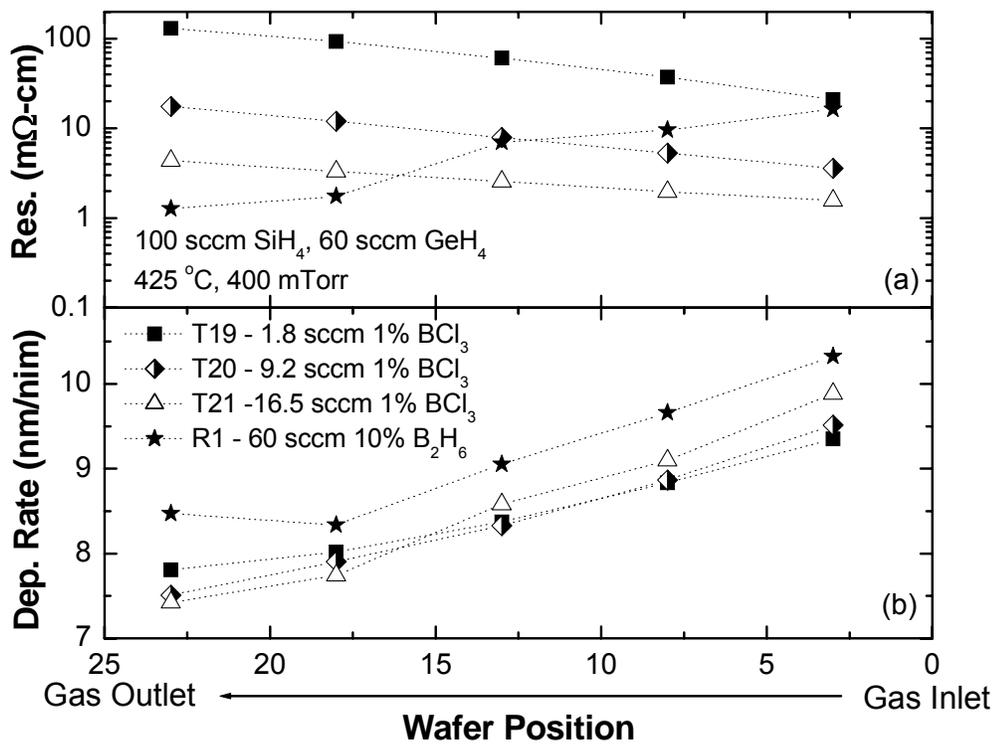
Figure 3.8 Deposition rate and resistivity across load

Runs T6 and T11, as well as Runs T20 and T23 have exactly the same deposition parameters, but they were separated by a few other depositions. The results show that the BCl_3 process is reproducible. Moreover, results of other similar depositions are reasonably consistent, which indicates that the process is operating in a relatively stable manner. In comparison, the decomposition of B_2H_6 causes periodic clogging of the injector, which results in large run-to-run variation unless the injector is changed approximately every 20 hours of deposition. Decomposition of BCl_3 is rather slow compared to B_2H_6, and injector clogging is not anticipated to be a problem. On the other hand, introducing B_2H_6 through the gas ring to avoid injector clogging is not an option

since large across load variation in film thickness and resistivity [3.11] resulted from its tendency to decompose at low temperature.

3.1.3.7 Furnace configuration optimization

All of the above experiments had the BCl_3 mixture introduced via the gas ring together with SiH_4 and GeH_4 in order to match the configuration of the epi-SiGe system and to minimize conflict with the existing B_2H_6 process for initial test. As discussed in Chapter 2, the poly-SiGe reactor is fairly flexible to accommodate new processes. To further improve the resistivity uniformity, different hardware configurations have been investigated.

As mentioned before, the cross-load resistivity uniformity of the BCl_3 doped poly-SiGe process could be further improved using an injector. A few BCl_3-doped depositions were done to study the improvement in uniformity with the injector. Since B_2H_6 is known to clog up the injector, a second injector was installed in the furnace to isolate the clogging problem. The second injector can be installed parallel to the B_2H_6 injector from the rear (pump) side or opposite to the B_2H_6 injector from the front (door) side. The two-injector configuration was setup temporarily with a few additional hand valves for manually switching between the lines. The rear injector configuration is identical to the existing B_2H_6 injector. The front injector is easier to install and remove, but it was later found out that the dopant gas leaked out near the door, making the front injector configuration very similar to the gas ring configuration. Sheet resistance data across the load of 50 wafers are shown in Figure 3.9. With the rear injector, the cross-load uniformity is improved by an order of magnitude.
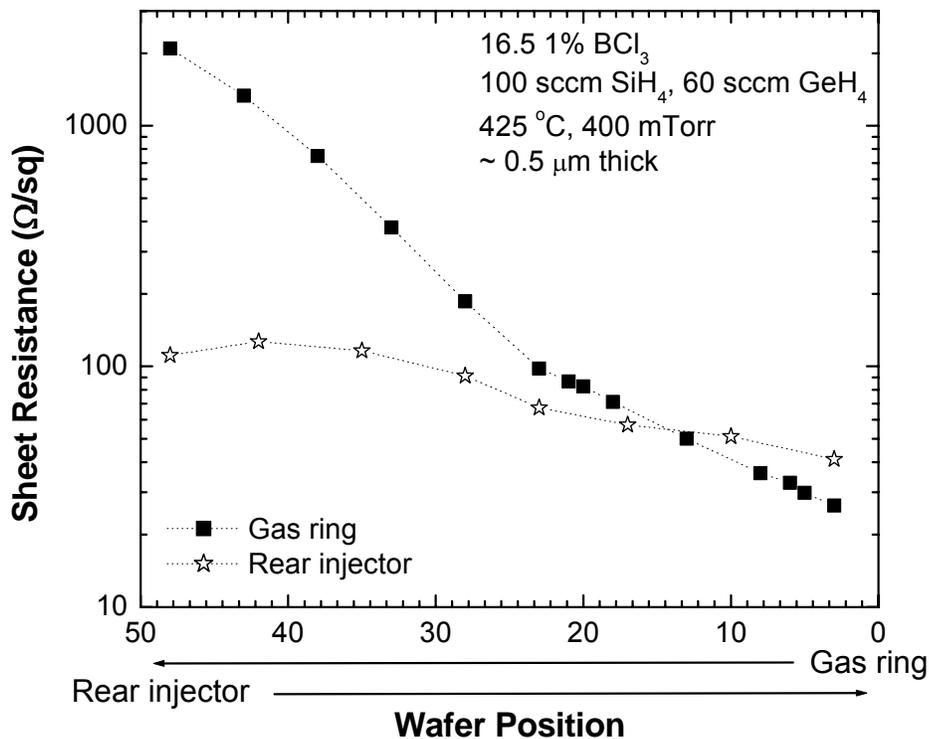
Figure 3.9 Cross-load uniformity of sheet resistance with gas ring and injector

When the BCl_3/He mixture is introduced via the gas ring, it comes out through multiple orifices along the circumference of the ring so cross wafer uniformity is not a problem. When the BCl_3/He mixture is injected from the bottom of the furnace using the injector, the cross load uniformity is improved whereas the cross wafer uniformity is sacrificed due to gas depletion. The depletion effect is more pronounced when the dopant gas flow rate is small. Figure 3.10 shows the variation in sheet resistance for both 100 mm-diameter and 150 mm-diameter wafers with 3.6 sccm of BCl_3 flow rate. The cross wafer uniformity of sheet resistance is not terribly bad for the 100 mm-diameter wafers at slots 1-25, but there is about 3× variation across the 150 mm-diameter wafers at slots 26-50. Increasing the BCl_3 flow rate can minimize the depletion effect and improve the cross

wafer uniformity. Figure 3.11 shows data for a deposition with 16.5 sccm of BCl_3 flow rate and cross wafer uniformity in sheet resistance is significantly improved.

Cross Wafer Uniformity
(with 3.6 sccm BCl_3)
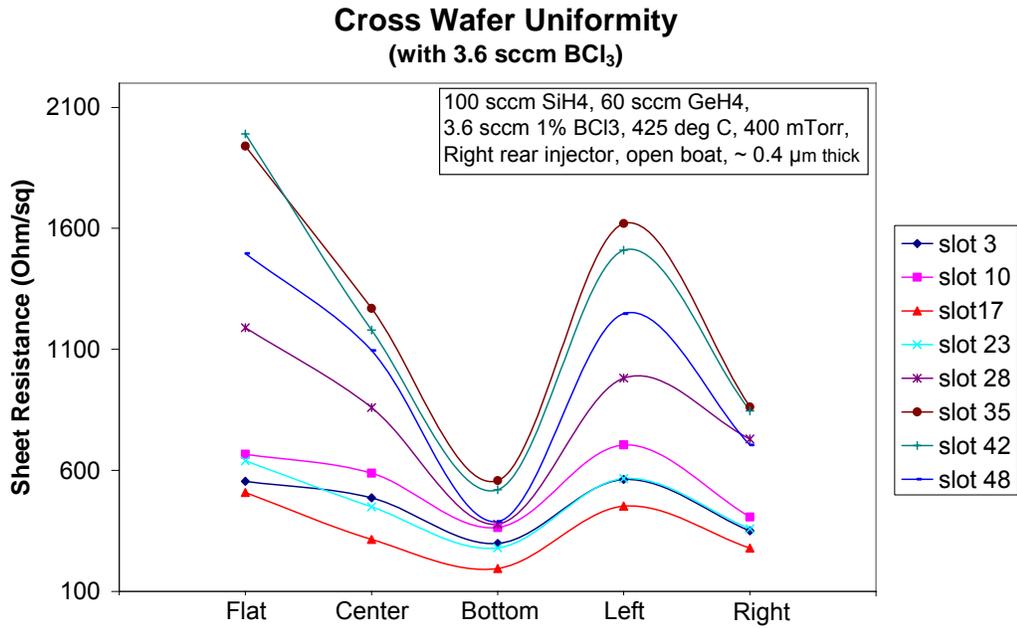
Figure 3.10 Cross wafer uniformity of sheet resistance with low BCl_3 flow rate

Cross Wafer Uniformity
(with 16.5 sccm BCl_3)
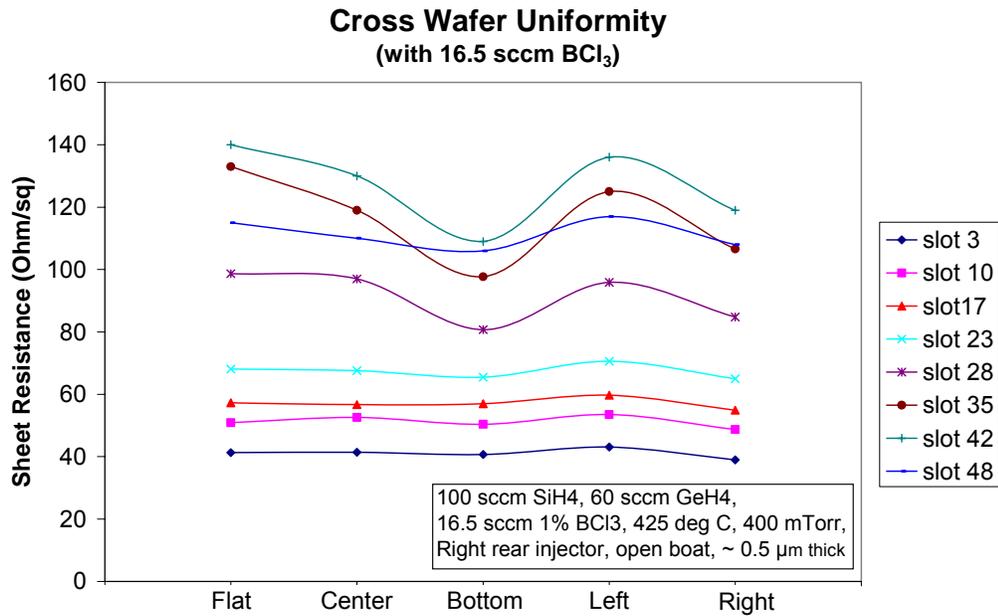
Figure 3.11 Cross wafer uniformity of sheet resistance with high BCl_3 flow rate

62

The experiments above show that using the rear injector with large dopant gas flow rate would be the optimal configuration to yield the best uniformity in resistivity. It is also confirmed that the injector does not get clogged up with the BCl_3 doping process. However, it is a concern if both B_2H_6 and BCl_3 are available in a reactor with two injectors. During the deposition with one dopant line, the injector for the other dopant line is at standby with no gas flow. Deposition on the standby injector will build up material at the small pores and eventually clog them up. Flowing a small amount of N_2 at the standby injector could prevent deposition on the pores, but this approach would involve significant hardware modification.

3.1.4 Comparison of the two boron dopant gases

To conclude the investigation of BCl_3 as a new dopant gas, a comparison with B_2H_6 is made in Table 3.6 for the study of the deposition and properties of LPCVD poly-$Si_{30}Ge_{70}$. The desired doping level for poly-$Si_{30}Ge_{70}$ is achieved with 1% BCl_3 concentration. The better uniformity and higher doping efficiency for the BCl_3 process is clearly advantageous over the B_2H_6 process for poly-SiGe films, but the small degradation in deposition rate and increase in residual stress are drawbacks. Tradeoffs between residual stress and resistivity are also involved in optimizing boron concentration for the BCl_3 doped process studied here. Both residual stress and resistivity can be improved by increasing the film thickness. Thicker films also have advantages in strain gradient and grain crystallinity, and thicker films are desired for better electro-mechanical performance in electrostaticcally driven MEMS applications. The initial BCl_3 doped process shows better process uniformity and repeatability than the more mature

B$_2$H$_6$ doped process. Based on these results, the B$_2$H$_6$ process was abandoned. Further development with the BCl$_3$ doped poly-SiGe process with the desired properties for MEMS applications will be discussed in Chapter 4.

TABLE 3.6 Comparison of BCl$_3$ and B$_2$H$_6$ as dopant gases for poly-SiGe process

Comparison	BCl$_3$ doping	B$_2$H$_6$ doping
Deposition rate	Similar	Similar
Crystallinity	Similar	similar
Dopant incorporation	+ + +	- - -
Residual stress	-	-
Strain gradient	Similar	similar
Uniformity	+ +	- -
repeatability	+ +	- -
Safety	Toxic and corrosive	Toxic and flammable
Shelf life	24 months	6 months
Cost	Negligible compared to GeH$_4$	Negligible compared to GeH$_4$

3.2 Disilane (Si$_2$H$_6$) as the silicon precursor

3.2.1 Literature review

Disilane (Si$_2$H$_6$) has been used as the silicon source for low temperature poly-silicon deposition due to its higher reactivity compared to silane (SiH$_4$) [3.11]. Disilane fragments into SiH$_4$, SiH$_3$ and SiH$_2$ in the gas phase. SiH$_3$ and SiH$_2$ decompose readily to form silicon at lower temperatures than SiH$_4$. Disilane has also been previously used as the silicon source for poly-SiGe deposition [3.10, 3.12], but the higher cost of using disilane was a concern.

Since lowering the thermal budget is an important consideration for SiGe post foundry-CMOS integration, anything that helps to increase the deposition rate other than temperature would be worth considering for this goal. The benefit of Si$_2$H$_6$ over SiH$_4$ is re-investigated to understand how much improvement can be gained with Si$_2$H$_6$ in reducing the thermal budget and how this change impacts the physical properties of the film. The cost of using disilane should not be a significant factor if there is a large

improvement in the process. LPCVD systems have large batch sizes so the additional cost is spread over many wafers and might become insignificant.

Data from previous poly-SiGe depositions using Si_2H_6 as the silicon source are summarized in Table 3.7 [3.12]. The poly-SiGe deposition was done in the same reactor in 2002. At that time, 100 mm-diameter wafers and caged boats were used.

TABLE 3.7 SiGe deposition with Si_2H_6 as the silicon precursor with 100 mm-diameter wafers, caged boat [3.12]

Temp. (°C)	Press. (mTorr)	Si_2H_6 (sccm)	GeH_4 (sccm)	PH_3 (sccm)	B_2H_6 (sccm)	Dep. Rate (nm/min)	Resistivity (mΩ-cm)	Stress (MPa)	% Ge by RBS
350	300	25	175	0	0	1.2	NA	NA	NA
400	300	25	175	0	0	1.8	NA	NA	66
450	300	25	175	0	0	4.0	NA	NA	NA
450	300	10	190	5	0	6.6	1300	-180	79
450	300	15	185	5	0	7.0	50000	-270	72
500	300	15	185	5	0	9.0	130	-170	74
500	300	25	175	5	0	9.4	120	-170	68
550	300	15	185	5	0	10.0	180	NA	80
550	300	25	175	5	0	9.6	170	-100	78
550	300	35	165	5	0	8.2	200	-50	75
550	300	50	150	5	0	10.5	50	-180	65
350	300	25	175	0	40	10.5	7800	NA	58
400	300	25	175	0	40	13.0	5400	NA	54
450	300	25	175	0	40	22.0	5.5	NA	50

In the interest of lowering the thermal budget or increasing the deposition rate, the numbers in Table 3.7 are encouraging. The deposition rate with Si_2H_6 and GeH_4 is about $2\times$ higher than with SiH_4 and GeH_4 for the same total gas flow rate. The resistivity with PH_3 doping is a lot higher than desired, but there is room for improvement in the B_2H_6 doped process. The average residual stress is in a reasonable range. There is no information on process uniformity. Since Si_2H_6 is more reactive, more GeH_4 flow is needed to achieve a similar germanium content as compared against the SiH_4 and GeH_4 process. To obtain similar film thickness, the cost of using Si_2H_6 as the silicon source is slightly higher, mainly because more GeH_4 is used.

3.2.2 Experimental results and discussion

Based on the results above, a few depositions were done to explore the Si_2H_6 process by finding out the deposition rate and basic material properties. The experiments were done during the development of the BCl_3 doped poly-SiGe process using SiH_4 and GeH_4 (Chapter 4). To make the comparison easier, the same furnace geometry was used with Si_2H_6 replacing SiH_4 for the deposition. There are twenty-five 150 mm-diameter wafers sitting in an open boat per load. The process conditions and the results are summarized in Table 3.8.

TABLE 3.8 SiGe deposition with Si_2H_6 as the silicon precursor with 150 mm-diameter wafers, open boat

Temp. (ºC)	Press. (mTorr)	Si_2H_6 (sccm)	GeH_4 (sccm)	BCl_3 (sccm)	Dep. Rate (nm/min)	Resistivity (mΩ-cm)	Stress (MPa)	% Ge by SIMS	B conc. (cm^{-3})
425	600	25	175	12	12.7	Not uniform	NA	NA	NA
375	600	25	175	12	7.7	Amorphous	-386	58	2.8×10^{19}
400	600	25	175	12	NA	Not uniform	NA	58	2.5×10^{19}
400	300	15	185	18	9.4	Amorphous	-248	63	3.9×10^{19}
425	300	15	185	18	11.2	3.1	-163	62	4.9×10^{19}
425	350	25	175	18	NA	Not uniform	NA	55	3.1×10^{19}
425	350	15	150	18	NA	Amorphous	NA	59	3.9×10^{19}

Data in Table 3.8 show there is indeed an improvement in deposition rate with Si_2H_6 compared to data shown in Table 4.2 with SiH_4 at similar temperatures, but there are tradeoffs between uniformity and crystallinity. High deposition temperature and high deposition pressure result in poor uniformity – the film at the center of the wafer is thinner with very high resistivity. In this case, the reaction is in the mass transportation limited regime since disilane is very reactive. The gas reacts on the wafer edge before it diffuses to the wafer center. In the extreme case, the wafer looks darker than usual, especially at the edge. Gas-phase nucleation happens here. Silicon particles form in the gas phase and reach the wafer surface, creating a porous film with high particle density. Lowering the deposition temperature, the deposition pressure, and the disilane/germane

gas flow ratio can push the deposition into the surface-reaction limited regime. However, with lower temperature and lower germanium content, the film is amorphous. Although the boron concentration is quite high, the film is not conductive. This set of experiment shows that the process window with disilane is narrow. The deposition condition has to be carefully chosen to get a uniform and conductive film. Reducing the total gas flow rate, increasing the wafer spacing or use of a caged boat could increase the process window.

The benefits of changing the source gas from SiH_4 to Si_2H_6 are not being maximized due to the effect on Ge content in the film. The Si_2H_6 partial pressure needs to be reduced to keep the same Ge content, which reduces the deposition rate and makes the net gain in deposition rate small. Also, the increase in deposition rate using Si_2H_6 is not worth the negative impact on process control. Other variables such as tube pressure and total gas flow may offer better means to achieve higher deposition rates with the SiH_4 and GeH_4 process.

3.3 Germyl silanes $((H_3Ge)_x SiH_{4-x})$ as the silicon germanium precursors

3.3.1 Literature review

As a promising material for modular CMOS and MEMS integration, the major challenges for LPCVD poly-SiGe in manufacturing are achieving good control of mechanical properties and lowering the SiGe deposition thermal budget.

The thin-film mechanical properties and the deposition rate depend strongly on germanium content. However, there are difficulties in controlling the germanium content in a LPCVD reactor using SiH_4 and GeH_4. As discussed in Chapter 2, mass flow

controllers are used to control the SiH_4 and GeH_4 gas flow rates. MFC drift has been an issue and it is a concern for run-to-run repeatability. Also, since SiH_4 and GeH_4 have different consumption rates, the wafers closer to the gas inlet have higher germanium content. This in combination with the loading effect results in a deposition rate that is higher at the gas inlet side than at the exhaust side of the furnace.

While the above difficulties could be overcome with sophisticated engineering solutions like MFC recalibration and precursor injection, the new family of precursor germyl silanes $((H_3Ge)_xSiH_{4-x})$ for SiGe deposition that has been developed recently can potentially be a simpler approach to solve these issues [3.13, 3.14]. As shown in Figure 3.12, this family of germyl silanes has direct Si-Ge bonds. The SiGe film compositional control is defined by the Si:Ge ratio of the precursor molecule rather than the precision of the gas delivery system with the binary precursor gases such as SiH_4 and GeH_4. Because the optimal germanium content for MEMS applications is between 50% and 70%, H_3GeSiH_3 and $(H_3Ge)_2SiH_2$ are of particular interest. Figure 3.13 shows the Arrhenius plot of the epitaxial deposition rate for various precursor gases [3.14]. The germyl silane molecules provide high deposition rates at low temperatures relative to disilane. The SiGe deposition rate achieved with the germyl silane precursors is expected to be greater than that achieved with SiH_4 and GeH_4. Therefore, germyl silane precursors could facilitate further reductions in the thermal budget for fabrication of MEMS on CMOS, to minimize any detrimental impact on CMOS reliability.

H₃SiGeH₃   (H₃Ge)₂SiH₂   (H₃Ge)₃SiH   (H₃Ge)₄Si
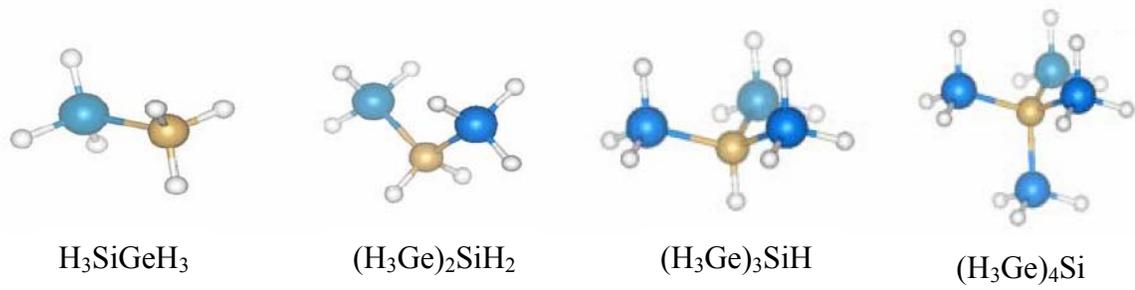
Figure 3.12 Structures of the germyl-silanes [3.13]
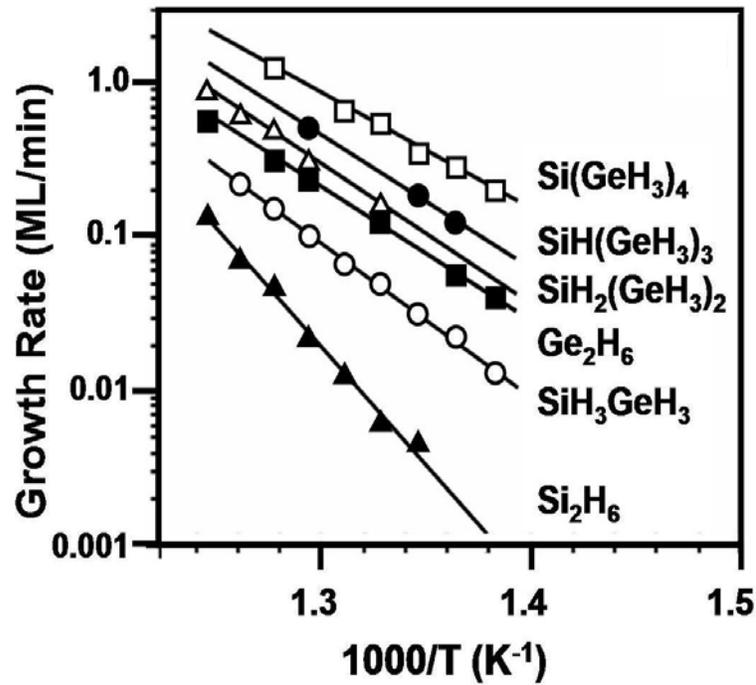
Figure 3.13 Temperature dependence of the first epitaxial layer growth rates for various precursors on Si (100) [3.14]

3.3.2 Experimental plan

The germyl silane precursors are being developed commercially [3.15] and collaborations are underway to test the deposition with these precursors with the LPCVD

system. While the germyl silane precursors have the potential benefits of precise germanium content control, run-to-run reproducibility, higher deposition rate and lower deposition temperature, there are also some unknowns. Since the germyl silane molecules have large molecular weight with high reactivity, the process should be carefully designed so that the deposition is in the surface reaction limited regime. There will be tradeoffs between uniformity and deposition rate like that seen for Si_2H_6 deposition.

Most of the germyl silane precursors are liquids at room temperature, except that H_3GeSiH_3 is in gas phase. The vapor pressures at 22ºC for $(H_3Ge)_2SiH_2$, $(H_3Ge)_3SiH$ and $(H_3Ge)_4Si$ are 55 Torr, 10 Torr and 1 Torr, respectively. If these liquid phase precursors are used, heating of the gas bottle and the delivery line will likely be needed to ensure sufficient gas is being pumped into the reaction chamber. To find out if heating would be needed for the LPCVD system, the gas flow and pressure stability can be verified manually once the precursor is hooked up to the reactor. Diluting the liquid with another gas or using a bubbler might be alternatives to heating, if lower concentration is necessary for better uniformity control.

To simplify the installation, the gas-phase precursor pure H_3GeSiH_3 will be tested first. Initial depositions should be targeted to understand the basic process and material properties. Cross wafer and cross load process uniformity, deposition rate, germanium content, resistivity, crystallinity and average residual stress should be characterized. Based on the results of the initial depositions, the process can be fine tuned to once the tradeoffs among deposition rate, uniformity and crystallinity are understood. For the LPCVD reactor in the UC Berkeley Microlab, the adjustable process parameters and their range are listed in Table 3.9.

TABLE 3.9 Adjustable process parameters with H_3GeSiH_3 in the Berkeley Microlab's LPCVD poly-SiGe reactor (Tystar20)

Parameters	Process space
Gas flow rates	$10 - 90\%$ MFC range of H_3GeSiH_3 $10 - 4500$ sccm of N_2 dilution
Temperature	$300 - 450°C$ Temperature ramping across the load
Tube pressure	$200 - 800$ mTorr
Wafer placement	Wafers can be placed at every slot or further apart form each other Open boat or caged boat can be used

Since the deposition will result in 50% germanium content SiGe film with relatively high deposition rate, an amorphous film might result from deposition temperatures below $425°C$. A thin crystalline seeding layer generated with SiH_4 and GeH_4 can be used to help to form a fully crystalline seed for the H_3GeSiH_3 main deposition.

3.4 Summary

The investigation of new process gases discussed in this chapter involves a good amount of background study, hardware modification and process verification. Boron trichloride (BCl_3) has been proven to be a better boron dopant source compared to diborane (B_2H_6), resulting in a more stable and better controlled process. Further development and characterization with the BCl_3 doped process will be discussed in the next Chapter. As a silicon precursor, disilane (Si_2H_6) can improve the deposition rate and lower the thermal budget of the process; however there is significant drawback in the process control due to its high reactivity. The process space with Si_2H_6 is narrow and the cost of using disilane will be higher since more germane will be needed to get the desired germanium content. As single-source silicon and germanium precursors, germyl silanes ((H_3Ge)$_x$$SiH_{4-x}$) have the potential of providing higher deposition rate and producing

uniform germanium content film across a large batch. Experiments beyond the scope of this thesis are needed to understand germyl silanes' pros and cons in LPCVD applications once these gases are available for laboratory experiments.

References

[3.1] Y. Kunii, Y. Inokuchi, A. Moriya, H. Kurokawa and J. Murota, "In situ B doping of SiGe(C) using BCl_3 in ultraclean hot-wall LPCVD", *Appl. Surface Sci.*, vol. 224, pp. 68-72, 2004

[3.2] Y. Kunii *et al.*, private communication, 2004

[3.3] B. Caussat, E. Scheid, B. de Mauduit and R. Berjoan, "Influence of dopant concentration and type of substrate on the local organization of low-pressure chemical vapour deposition in situ boron doped silicon films from silane and boron trichloride", *Thin Solid Films*, vol. 446, pp. 218-226, 2004

[3.4] C. W. Low, M. L. Wasilik, H. Takeuchi, T.-J. King and R. T. Howe, "In-situ doped poly-SiGe LPCVD process using BCl_3 for post-CMOS integration of MEMS devices," in *Proc. Electrochemical Society SiGe Materials, Processing, and Devices Symposium*, Honolulu, HI, Oct. 3-8, 2004, pp. 1021-1032

[3.5] A. Moriya, M. Sakuraba, T. Matsuura and J. Murota, "Doping and electrical characteristics of in situ heavily B-doped $Si_{1-x}Ge_x$ films epitaxially grown using ultraclean LPCVD", *Thin Solid Film*, vol. 343-344, pp. 541-544, 1999

[3.6] B. L. Bircumshaw, M. L. Wasilik, E. B. Kim, Y. R. Su, H. Takeuchi, C. W. Low, A. P. Pisano, T.-J. King and R. T. Howe, "Hydrogen peroxide etching and stability of p-type poly-SiGe films," *17th IEEE Micro Electro Mechanical Systems Conference (MEMS-04)*, Maastricht, The Netherlands, Jan. 25-29, 2004, pp. 514-519

[3.7] M. Cao, A. Wang and K. C. Saraswat, "Low pressure chemical vapor deposition of $Si_{1-x}Ge_x$ films on SiO_2, characterization and modeling", *J. Electrochem. Soc.*, vol. 142(5), pp. 1566-1572, 1995

[3.8] S. M. Sze, *Physics of Semiconductor Devices*, 2nd ed., p. 32-33, John Wiley & Sons, New York, 1981

[3.9] N. C. C. Lu, C. Y. Lu, M. K. Lee, C. C. Shin, C. S. Wang, W. Reuter and T. T. Sheng, "The effect of film thickness on electrical properties of LPCVD polysilicon films", *J. Electrochem. Soc.*, vol. 131(4), pp. 897-902, 1984

[3.10] A. E. Franke, "Polycrystalline Silicon-Germanium Films for Integrated Microsystems," Ph.D. Thesis, Dept. of EECS, University of California at Berkeley, pp. 118-119, 2000

[3.11] T. Kamins, *Polycrstalline silicon for integrated circuits and displays*, 2nd edition, Kluwer Academic Publishers, pp. 44, 48 and 52, 1998

[3.12] Y-C. Jeon, T.-J. King, R. T. Howe, "Properties of phosphorus-doped poly-SiGe films for microelectromechanical systems", *J. Electrochem. Soc.*, vol. 150(1), pp. H1-H6, 2003

[3.13] C. J. Ritter, C.-W. Hu, A. V. G. Chizmeshya, J. Tolle, d. Kelewer, I. S. T. Tsong and J. Kouvetakis, "Synthesis and fundamental studies of $(H_3Ge)_xSiH_{4-x}$ molecules: precursors to semiconductor hetero- and nanostructures on Si", *J. Am. Chem. Soc.*, vol. 127(27), pp. 9855-9864, 2005

[3.14] C.-W. Hu, J. Tolle, A.V.G. Chizmeshya, J. Menéndez, I. S. T. Tsong, C. Ritter and J. Kouvetakis, "Low-temperature pathways to Ge-rich $Si_{1-x}Ge_x$ alloys via single-source hydride chemistry", *Appl. Phys. Lett.*, vol. 87, pp. 181903, 2005

[3.15] M. D. Stephens, C. J. Ritter and M. A. Pikulin, "The utility of novel single-source germyl silanes", in Proc. *Proc. Electrochemical Society SiGe & Ge Materials, Processing, and Devices Symposium*, Cancun, Mexico, Oct. 29 – Nov. 3, 2006

Chapter 4: LPCVD Poly-SiGe Process Optimization

For MEMS applications, poly-SiGe's low resistivity, low wet-etch rate in heated hydrogen peroxide (H_2O_2) solution, low tensile stress and low strain gradient are the desirable properties. The electrical and mechanical properties of poly-SiGe films depend strongly on the deposition process conditions. This chapter describes the process development to achieve the optimal poly-SiGe film for RF MEMS devices and inertial sensors.

The experimental details and thin film characterization methods are presented first. The results of all the depositions are summarized in section 4.3. Each set of experiments is described individually in the follow sections. Then the results of all experiments are reviewed together for microstructural properties study, and the characteristic microstructure for achieving low strain gradient film is elucidated.

4.1 Experimental details

Boron-doped poly-SiGe films were deposited in a Tystar hot-wall horizontal LPCVD reactor as described in Chapter 2. Pure SiH_4 and GeH_4 were used as the silicon and germanium precursor gases, respectively. 1% BCl_3 diluted in He was used as the dopant gas. SiH_4 and GeH_4 were introduced through a gas ring located at the load side of the tube. The BCl_3/He mixture was introduced from the pump side through a multi-hole injector located beneath the wafer boats. Twenty-five 150 mm-diameter wafers were placed in open boats at the center of the reactor per load. Poly-SiGe films were deposited onto silicon substrates coated with a 2 μm-thick LPCVD SiO_2. A very thin (<5 nm)

amorphous-silicon seed layer was deposited first using Si_2H_6 to promote the adhesion of poly-SiGe to the oxide.

Experimental data were collected with five 4"-diameter wafers that were placed in slots 3, 9, 15, and 21 of the wafer boats, counting from the gas inlet side. A four-point probe instrument was used to measure the sheet resistance. The films were patterned and etched for the thickness measurement using a stylus-based profiler. Wafer curvature was measured before and after SiGe deposition (with backside SiGe film removed) to determine the average residual stress of the film. A cantilever beam array was patterned and released for strain gradient measurement. Resistivity, thickness, and strain gradient were measured at various points on each wafer, and average numbers are reported here. The crystal orientation of selected films was studied by X-ray diffraction. Transmission electron microscopy (TEM) analysis was used extensively to understand the film's microstructure and its correlation with the deposition condition and mechanical properties.

4.2 Characterization methods

4.2.1 Transmission electron microcopy

Transmission electron microcopy analysis is commonly used to obtain crystallographic information from specimens that are thin enough to transmit electrons [4.1]. A stream of electrons produced by the electron gun is focused to a small and coherent beam by a set of condenser lenses and aperture. The beam then strikes the specimen and part of it is transmitted. The transmitted part is focused by a set of

objective lenses and the image of the specimen is passed onto the image plane for the user to see.

While the theory and operation of the transmission electron microscope are fairly simple, the challenge of TEM analysis is in the sample preparation. The thickness of the specimen should be less than a few thousand angstroms for the electron beam to penetrate through. The sample preparation is time consuming and requires patience. Cross-sectional TEM analysis is mainly used for this dissertation. Figure 4.1 illustrates the general procedure for cross-sectional TEM specimen preparation. First, the wafer with the thin film is cleaved into a few 3 mm by 3 mm pieces. Two pieces are glued together with the thin films facing each other using epoxy (M-bond 610). Two dummy pieces are glued on the sides as mechanical supports. The sandwich is then cut into a few slides approximately 500 μm thick using a diamond saw. The slide is chopped into a disk using a slurry disk cutter. The sample then goes to the dimpler for mechanical grinding and polishing. Once the center region of the sample is thinned down to about 20 μm, the disk is mounted in an ion-milling machine where the specimen is further sputter thinned by ion bombardment until a hole appears. The edge near the hole is thin enough for imaging under the electron microscope.

Top view TEM specimen can be prepared using similar techniques with the bonding and slicing steps skipped. A disk can be cut from the wafer and the sample is mechanically polished and ion milled to final thickness from the backside.

Figure 4.1 Cross-sectional TEM sample preparation method

4.2.2 X-ray diffraction

X-ray diffraction (XRD) measurement is a non-destructive method that provides information on crystallinity and texture of bulk solids and thin films. Monochromatic X-ray is used to determine the inter-planar spacing of the material. Material composition

and crystal orientation can be obtained from the X-ray spectra. When the Bragg condition for constructive interference is obtained, a diffraction peak is produced and the relative peak height is proportional to the number of grains in a preferred orientation.

For a poly-SiGe thin film on a single-crystal silicon substrate, the X-ray spectra contain diffraction peaks for both the thin film and the substrate. The diffraction peak positions can be calculated with Bragg's Law [4.2]:

$$\lambda = 2d \sin \theta \qquad (4.1)$$

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \qquad (4.2)$$

where h, k and l are the Miller indices for the direction and a is the lattice constant for a cubic material. The lattice constant of Si, Ge and $Si_{1-x}Ge_x$ are listed below:

$$\text{Si: } a = 5.43 \, \overset{\circ}{A} \qquad (4.3)$$

$$\text{Ge: } a = 5.66 \, \overset{\circ}{A} \qquad (4.4)$$

$$Si_{1-x}Ge_x: \ a(x) = (5.43 + 0.20x + 0.027x^2) \, (\overset{\circ}{A}) \ [4.3] \qquad (4.5)$$

$$Si_{40}Ge_{60}: \ a = 5.56 \, \overset{\circ}{A} \qquad (4.6)$$

The peak position shifts with different germanium content. However, it is difficult to calculate the germanium content from the peak position since peak shift can also be induced by stresses in the film. The diffraction peaks of the poly-SiGe sample are listed in Table 4.1.

TABLE 4.1 XRD 2θ angle calculation

Material	Direction	d (Å)	2θ (°)
Si	<200>	2.72	33.96
Si	<400>	1.36	69.14
Ge	<200>	2.83	31.59
Ge	<400>	1.42	65.96
$Si_{40}Ge_{60}$	<111>	3.21	27.77
$Si_{40}Ge_{60}$	<220>	1.97	46.14
$Si_{40}Ge_{60}$	<311>	1.68	54.71
$Si_{40}Ge_{60}$	<222>	1.61	57.36
$Si_{40}Ge_{60}$	<400>	1.39	67.31
$Si_{40}Ge_{60}$	<331>	1.28	74.30

4.2.3 Strain gradient measurement

Minimization of the strain gradient, or the out-of-plane curvature of a released beam, is a critical requirement for inertial sensor applications in which the lateral dimensions of mechanical structures are in the range of hundreds of microns. A typical target value of strain gradient is 1×10^{-5} μm^{-1} for inertial sensor applications, which would yield 1.25 μm tip deflection of a 500 μm long cantilever beam. The dependence of stress and strain gradient on film microstructure and deposition conditions is well-understood for poly-Si films [4.1], [4.4]. The mechanical properties of poly-SiGe can be studied with similar techniques used for poly-Si.

The released cantilever beam shown in Figure 4.2(a) is commonly used for strain gradient measurement. Strain gradient is calculated as [4.5]:

$$\frac{1}{\rho} = \frac{2\Delta z}{L^2} \tag{4.7}$$

where ρ is the radius of curvature of the beam, Δz is the out-of-plane deflection of the tip and L is the length of the cantilever beam.

Figure 4.2 Strain gradient measurement: a) cross-sectional schematic of a released cantilever beam; b) stress depth profile before release; c) stress depth profile after release, but before bending

The strain gradient of the film can also be calculated as [4.5]:

$$\frac{1}{\rho} = \frac{12M}{EWH^3} = \frac{12}{EH^3} \int_{-n}^{H-n} \sigma(z) \, z \, dz \tag{4.8}$$
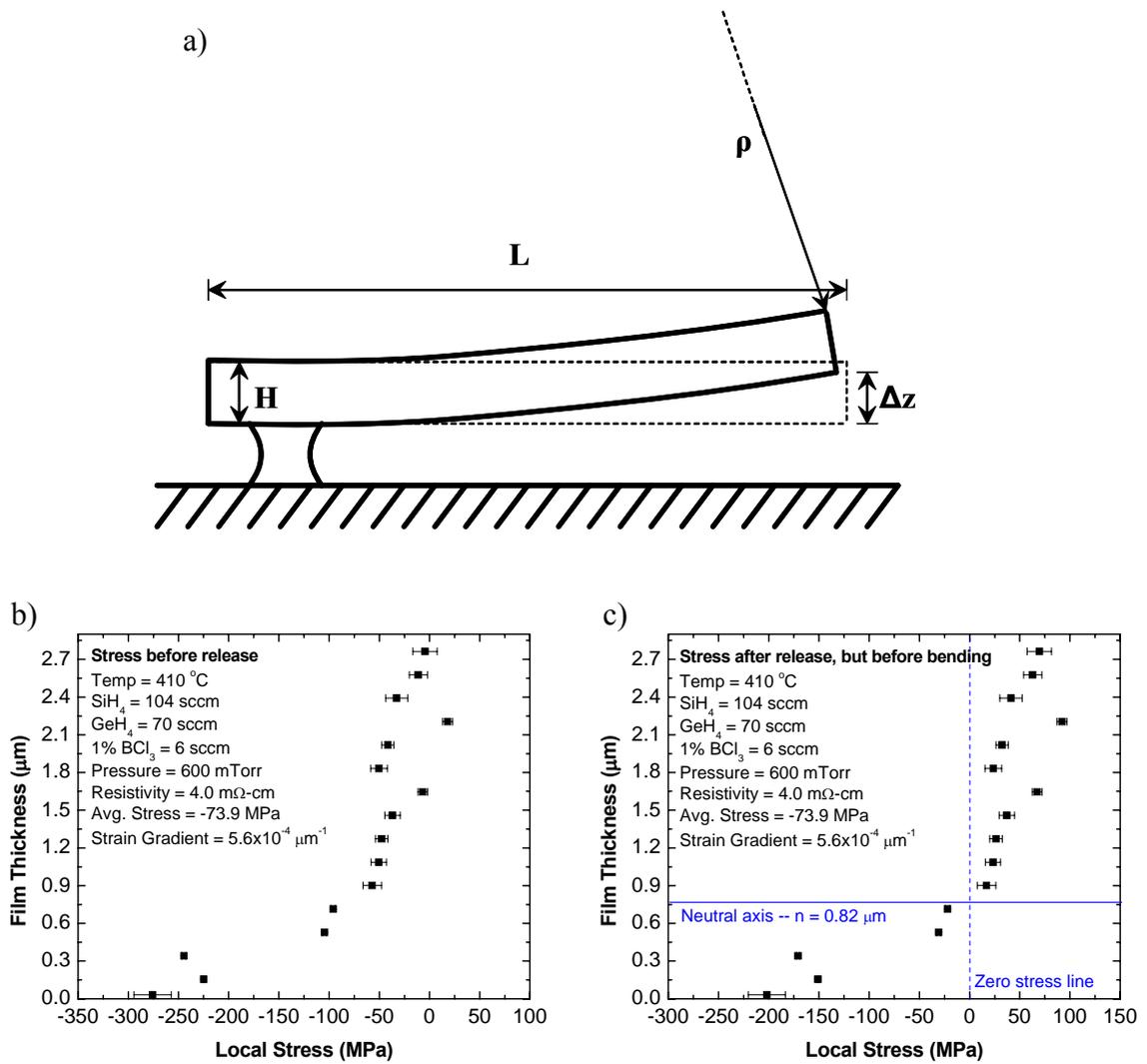
where M is the bending moment, E is the Young's Modulus, W is the width of the cantilever beam, H is the film thickness, n is the position of the neutral axis, σ(z) is the stress depth profile in the film and z is the distance from the neutral axis. Strain gradient

81

is related to stress gradient with the Young's Modulus as a proportionality factor, assumed to be 140 GPa for poly-SiGe.

Stress *vs.* depth profiles $\sigma(z)$ for Equation (4.8) were generated by incrementally etching (thinning) an unpatterned poly-SiGe film on the front side of the wafer and then measuring the change in wafer curvature, in an iterative manner [4.1]. Initial wafer curvature was measured prior to any etching of the poly-SiGe film using a reactive ion etcher. After every 0.16 μm of etching, the wafer was taken out from the etcher for wafer curvature measurement. The stress of the thin etched layer was determined from the change in wafer curvature. This process was repeated until the poly-SiGe film was completely etched away. A typical stress profile of a film before release is shown in Figure 4.2(b). To find the neutral axis in Equation (4.8), the stress profile is shifted by the amount of average residual stress and the position of the neutral axis is at the intersection of the zero stress line and the shifted stress profile, as shown in Figure 4.2(c). Error bars displayed on the stress curve indicate the measurement uncertainty. Note that the systematic error is significantly larger at the bottom of the film because of the cumulative effect of cross-wafer etch-rate non-uniformity, which reduces the validity of the assumption of uniform film thickness for the stress measurement toward the bottom of the film. Nonetheless, the general shape of the stress profile is still valid and the two methods of strain gradient measurement yield similar results with ~20% discrepancy.

4.3 Overall experimental data

The average deposition rate, resistivity, average residual stress and strain gradient, along with cross-wafer (XW) and cross-load (XL) deposition uniformity, are summarized

in Table 4.2. Uniformity is reported as normalized standard deviation for deposition rate and resistivity. Standard deviation is reported for average residual stress since its value can be both positive and negative. Average strain gradient and its best value within each run are reported in Table 4.2. Further statistical analysis of the strain gradient is discussed later in the chapter.

The experimental study was done in a series of design-of-experiments (DOE) runs. The 1st DOE was performed to understand the process space and characterize the deposition rate, resistivity, average residual stress, strain gradient and wet etch rate in hydrogen-peroxide solution. The results of the 1st DOE showed that the structural layer requirements for general MEMS applications can be met within the process temperature constraint imposed by CMOS electronics, but the strain gradient requirements for inertial sensor applications remain a major challenge. The rest of the experiments were all designed to achieve low strain gradient with good uniformity. The ramping experiment explores the option of ramping down the temperature and germanium content during deposition for grain control. The 2nd DOE looked into the effect of varying the dopant gas flow rate and the process pressure. In addition, the effects of film thickness and the initial seed layer were studied with the best recipe from the 2nd DOE. Also, multiple layer deposition was used to create fine-grain microstructure. Low strain gradient can be achieved with several of the approaches described above. All recipes which yield films with absolute strain gradient $\leq 1 \times 10^{-4}$ μm^{-1} are highlighted in Table 4.2. Strain gradient uniformity study has focused on these highlighted recipes.

Table 4.2 Experimental data for all runs

Run #	Si$_2$H$_6$ seed	SiGe seed Temp (°C)	Press. (mT)	SiH$_4$ (sccm)	GeH$_4$ (sccm)	*BCl$_3$ (sccm)	Time (min)	SiGe Deposition Temp. (°C)	Press. (mT)	SiH$_4$ (sccm)	GeH$_4$ (sccm)	*BCl$_3$ (sccm)	Time (min)	Avg. thn (μm)	Avg. DR (nm/min)	DR XW unif	DR XL unif	Avg. Res. (mΩ·cm)	Res. XW unif	Res. XL unif	Avg. stress (MPa)	Stress XL stdev (MPa)	Avg. strain gradient (μm^{-1})	Best Strain gradient (μm^{-1})	H$_2$O$_2$ ER (nm/min)
DOE1-1	Yes	None						410	600	120	50	6	381	1.8	4.7	3%	9%	6.9	37%	67%	-228	24	6.60e-4	4.09e-4	1.3
DOE1-2	Yes	None						410	600	104	70	6	335	2.6	7.8	1%	11%	5.2	31%	24%	-92	16	6.74e-4	5.13e-4	18
DOE1-3	Yes	None						410	600	112	60	12	318	2.1	6.5	2%	12%	2.4	18%	18%	-168	8	5.88e-4	1.84e-4	2.2
DOE1-4	Yes	None						410	600	120	50	18	421	2.6	6.2	2%	14%	0.96	11%	13%	-161	26	-5.22e-6	-9.05e-6	0.78
DOE1-5	Yes	None						410	600	104	70	18	250	2.0	7.9	2%	14%	1.8	14%	15%	-155	17	6.06e-4	2.66e-4	4.9
DOE1-6	Yes	None						425	600	112	60	6	302	2.2	7.2	2%	12%	7.3	38%	23%	-120	26	8.24e-4	5.87e-4	0.33
DOE1-7	Yes	None						425	600	120	50	12	290	1.8	6.1	2%	13%	3.1	17%	19%	-171	27	9.74e-4	6.37e-4	0.39
DOE1-8	Yes	None						425	600	112	60	12	302	2.2	7.4	1%	14%	3.2	21%	22%	-119	21	7.95e-4	6.03e-4	1.1
DOE1-9	Yes	None						425	600	104	70	12	230	2.4	9.9	1%	14%	3.8	28%	25%	-73	19	5.10e-4	3.26e-4	4.6
DOE1-10	Yes	None						425	600	112	60	18	256	2.5	8.2	2%	15%	2.1	14%	17%	-97	21	5.38e-4	3.89e-4	1.6
DOE1-11	Yes	None						440	600	120	50	6	185	1.9	7.6	1%	12%	9.8	43%	21%	-126	25	5.09e-4	3.41e-4	1.1
DOE1-12	Yes	None						440	600	104	70	6	135	1.7	12.5	2%	14%	19	59%	31%	-53	21	4.68e-4	4.05e-4	8.2
DOE1-13	Yes	None						440	600	112	60	12	218	2.0	9.3	1%	15%	5.5	34%	26%	-104	24	4.42e-4	3.67e-4	0.78
DOE1-14	Yes	None						440	600	120	50	18	265	2.0	7.6	2%	14%	2.6	17%	17%	-131	27	5.30e-4	3.52e-4	0.056
DOE1-15	Yes	None						440	600	104	70	18	182	1.9	10.8	1%	15%	3.3	27%	23%	-70	18	4.87e-4	4.07e-4	2.33
Ramp-ref	Yes	None						430	600	140	60	30	230	1.9	8.4	2%	14%	1.4	7%	9%	-135	21	6.40e-4	4.86e-4	NA
Ramp-SiH$_4$	Yes	None						430	600	140-190	60	30	30*11	2.6	7.9	2%	14%	1.3	7%	10%	-126	14	3.80e-4	2.74e04	NA
Ramp-temp	Yes	None						430-380	600	140	60	30	30*11	2.5	7.6	2%	12%	1.0	5%	6%	-164	25	3.87e-4	2.86e04	NA
DOE2-t1	Yes	None						410	1200	150	50	30	60	0.61	10.2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
DOE2-t2	Yes	None						410	900	150	50	30	60	0.58	9.7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
DOE2-t3	Yes	None						410	700	150	50	30	60	0.55	9.2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
DOE2-t4	Yes	None						410	600	140	60	30	60	0.52	8.7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
DOE2-t5	Yes	None						410	350	140	60	30	60	0.38	6.4	NA	NA	1.3	NA	NA	NA	NA	NA	NA	NA

Table 4.2 Experimental data for all runs (continued)

Run #	SiGe seed							SiGe Deposition								Results									
	Si₂H₆ seed	Temp (°C)	Press. (mT)	SiH₄ (sccm)	GeH₄ (sccm)	*BCl₃ (sccm)	Time (min)	Temp (°C)	Press. (mT)	SiH₄ (sccm)	GeH₄ (sccm)	*BCl₃ (sccm)	Time (min)	Avg. thn (μm)	Avg. DR (nm/min)	DR XW unif	DR XL unif	Avg. Res. (mΩ-cm)	Res. XW unif	Res. XL unif	Avg. stress (MPa)	Stress XL stdev (MPa)	Avg. strain gradient (μm^{-1})	Best Strain gradient (μm^{-1})	H₂O₂ ER (nm/min)
DOE2-1	Yes	None	None	None	None	None	None	410	600	140	60	15	230	1.6	6.4	3%	14%	1.6	17%	19%	-238	14	1.71e-4	6.22e-6	NA
DOE2-2	Yes	None	None	None	None	None	None	410	600	140	60	30	230	1.8	7.7	2%	15%	0.8	5%	10%	-204	9	7.49e-5	6.85e-6	NA
DOE2-3	Yes	None	None	None	None	None	None	410	600	140	60	45	230	1.9	8.3	3%	13%	0.7	3%	6%	-167	6	2.81e-4	8.26e-5	NA
DOE2-4	Yes	None	None	None	None	None	None	410	350	140	60	15	360	1.8	5.1	2%	12%	1.5	8%	12%	-182	25	6.5e-4	4.73e-4	NA
DOE2-5	Yes	None	None	None	None	None	None	410	350	140	60	30	340	2.0	5.9	2%	10%	0.8	3%	3%	-176	13	3.87e-4	3.11e-4	NA
DOE2-6	Yes	None	None	None	None	None	None	410	350	140	60	45	313	2.0	6.3	3%	9%	0.7	2%	1%	-174	10	4.71e-4	3.81e-4	NA
ThickDepo	Yes	None	None	None	None	None	None	410	600	140	60	35	480	3.8	8.0	2%	14%	0.7	7%	5%	-157	11	2.5e-5	-7.9e-7	NA
LayerStack	Yes	None	None	None	None	None	None	410	350	140	60	30	85*4	1.9	5.7	3%	10%	1.7	9%	6%	-220	13	-1.21e-4	-7.46e-5	NA
SiGeSeed-t1	No	410	300	47	20	35	20	None	None	None	None	None	None	0.05	2.5	NA	NA	1.5	NA	NA	NA	NA	NA	NA	NA
SiGeSeed-1	No	410	300	47	20	35	20	410	600	140	60	35	260	2.2	8.3	2%	13%	0.9	6%	6%	-182	17	4.65e-4	2.57e-4	NA
SiGeSeed-2	Yes	410	350	140	60	35	20	410	600	140	60	35	230	1.87	7.5	3%	14%	0.8	5%	5%	-216	20	9.35e-5	5.17e-7	NA
Ge	Yes	None	None	None	None	None	None	350	300	0	100	12	287	2.2	7.7	2%	4%	5.0	4%	4%	-83	6	NA	NA	387

85

*BCl₃ is diluted in helium (1% BCl₃ / 99% He)

4.4 1st design-of-experiments

4.4.1 Experimental design

For the LPCVD process, the process variables include temperature, pressure, silane-to-germane gas flow ratio, dopant gas flow rate, and load size. In this section, the LPCVD poly-SiGe process parameter space is explored with a 15-run DOE [4.6]. SiH_4-to-GeH_4 gas flow ratio, BCl_3 gas flow rate, and temperature are the input variables; deposition rate, resistivity, average residual stress, strain gradient and H_2O_2 etch rate are functions of the input variables.

All depositions targeted a poly-SiGe film thickness of 2 μm. For the design of experiments, a face-centered central composite design was used. Deposition temperature (410°C, 425°C, or 440°C), SiH_4 to GeH_4 ratio (104/70, 112/60 or 120/50 sccm/sccm), and BCl_3 flow rate (6, 12 or 18 sccm) were chosen as input variables. It should be noted that initial design intended to have the summation of SiH_4 and GeH_4 flow rates as a constant – 200 sccm. However, the SiH_4 MFC experienced an electronic drift prior to this experiment, so that the actual SiH_4 gas flow rate was later found out to be ~80% of the design value. The process pressure was kept constant at 600 mTorr in each recipe. The process details for each deposition run are summarized in Figure 4.3. The lower value of temperature was set by the amorphous-to-polycrystalline transition temperature, and the upper value was set by thermal budget limits imposed by foundry CMOS electronics. The upper and lower values of SiH_4/GeH_4 flow ratio were set by the crystallinity requirement and hydrogen-peroxide etch rate, respectively. High-germanium-content films have lower amorphous-to-polycrystalline transition temperature. However, the wet etch rate is higher for germanium-rich films, which is not desirable for micro-machining processes using

86

pure germanium as the sacrificial material [4.7]. The lower value of BCl_3 flow rate was set by resistivity considerations, while the upper value was set by the maximum flow rate of the mass flow controller (MFC).
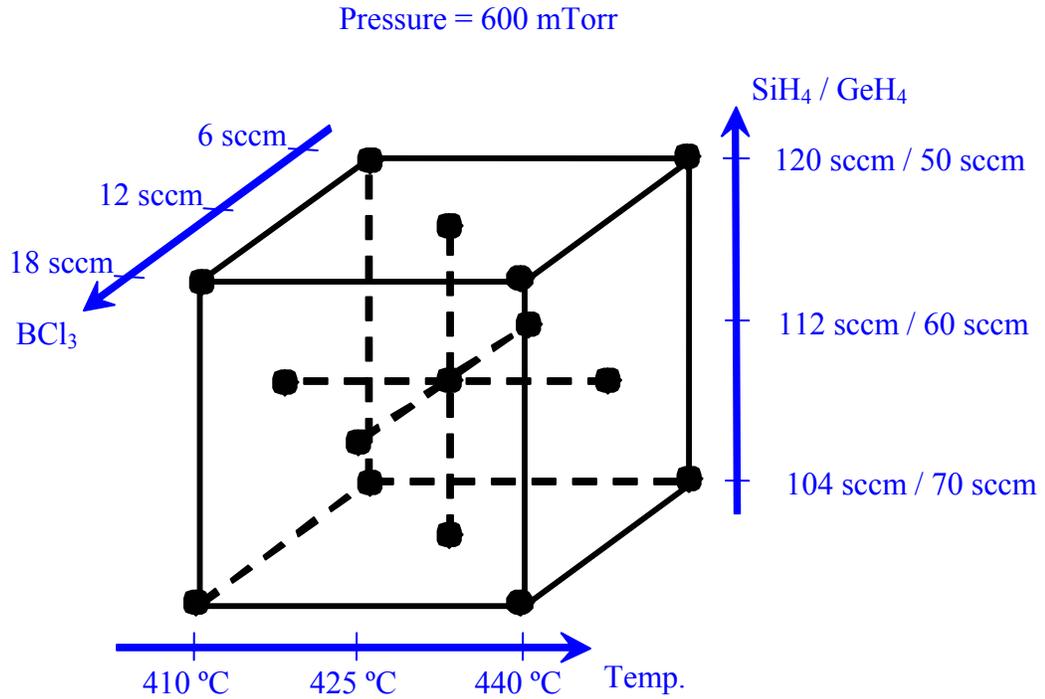
Figure 4.3 1st design of experiments input parameter values

4.4.2 Results and interpretation

The experimental data for the 1st design of experiment can be found in Table 4.2. The deposition rate, resistivity, and wet etch rate fall within reasonable ranges. Depending on the application, a recipe can be chosen to meet specific requirements. The average residual stress is compressive for each of the recipes although a small tensile stress is desired for some applications. The strain gradient is higher than desired for inertial sensor applications.

Since a LPCVD system is a batch reactor, cross-load uniformity is an important manufacturing consideration. The wafer-to-wafer uniformity and within-wafer uniformity reported here are for the Tystar reactor in an academic research laboratory; it is not surprising that they do not meet specifications for high volume production. However, uniformity is expected to be significantly better for the sophisticated LPCVD systems used within production environments. In our research tool, the cross-wafer uniformity of deposition rate is better than 3%. Due to the different consumption rates of SiH_4 and GeH_4, the germanium content in films deposited onto wafers closest to the gas inlet is about 3 atomic percent higher than for films deposited onto the wafers closest to the exhaust [4.7]. This gradient in germanium content, in combination with the loading effect, results in a deposition rate that is higher at the gas inlet side than at the exhaust side. To improve the cross-load uniformity, an injector can be used for the precursor gases. The dopant gas is introduced via an injector located at the bottom of the reactor. Thus, the film resistivity is lower in the regions of the wafers closer to the injector. Due to gas depletion effects, recipes utilizing low BCl_3 flow rate tend to have worse cross-wafer uniformity in resistivity. Since the injector design was not optimal, cross-load uniformity of resistivity depends on the wafer position relative to location of the injector holes. Improved injector design, higher BCl_3 flow rate, and *in-situ* wafer rotation as in a vertical furnace should all enhance the uniformity of film resistivity.

To deduce general trends, the average values for deposition rate, resistivity, residual stress, strain gradient, and wet etch rate were analyzed using the JMP™ statistical software package [4.8]. Confidence intervals for the output observables *vs.* input factors are shown in Figure 4.4. The deposition rate increases with temperature, but

decreases with SiH_4/GeH_4 ratio and shows no dependence on dopant gas flow rate. The film resistivity mainly depends on BCl_3 flow rate. The average residual stress becomes less compressive with increasing temperature and decreasing SiH_4/GeH_4 ratio. Average residual stress was previously reported to become more compressive when boron doping is increased by orders of magnitude in Chapter 3. In this experiment, the boron doping variation range is small, and no significant trend is found for the average residual stress *vs.* dopant concentration. For the strain gradient data, the error bar is larger than the slope of the trend in Figure 4.4. Further investigation of the strain gradient will be discussed in the next section. Wet etch rate mainly depends on the germanium content in the film and thus increases inversely with SiH_4/GeH_4 ratio as expected.
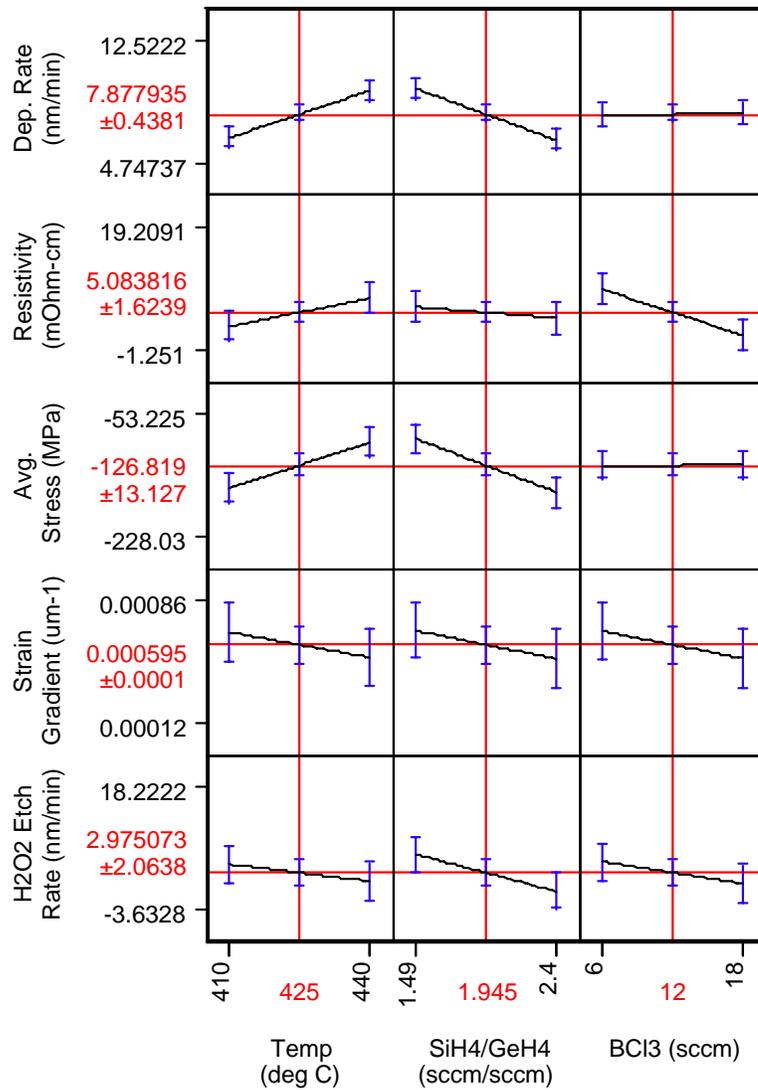
Figure 4.4: Values and confidence intervals for various responses

4.4.3 Mechanical properties study

The stress profiles of all the deposition conditions along with some TEM images are presented in Figure 4.5 over the next few pages. The majority of the deposited films have upward curvature upon release, because the compressive stress at the bottom of the film is usually significantly higher than in the rest of the film. The slope of the stress

profile (hence the strain gradient) depends on the film deposition conditions. Similar results have been found for APCVD and PECVD poly-SiGe films [4.9], [4.10].

a)

DOE1-1

Temp = 410 $^{\circ}$C
SiH$_4$ = 120 sccm
GeH$_4$ = 50 sccm
1% BCl$_3$ = 6 sccm
Pressure = 600 mTorr
Avg. Stress = -200.6 MPa

Film Thickness (μm)
Local Stress (MPa)

0.5 μm

b)

DOE1-2

Temp = 410 $^{\circ}$C
SiH$_4$ = 104 sccm
GeH$_4$ = 70 sccm
1% BCl$_3$ = 6 sccm
Pressure = 600 mTorr
Avg. Stress = -73.9 MPa

Film Thickness (μm)
Local Stress (MPa)

c)

DOE1-3

Temp = 410 $^{\circ}$C
SiH$_4$ = 112 sccm
GeH$_4$ = 60 sccm
1% BCl$_3$ = 12 sccm
Pressure = 600 mTorr
Avg. Stress = -158.5 MPa

Film Thickness (μm)
Local Stress (MPa)

0.5 μm

92

d)

e)

f)

g)

DOE1-7
Temp = 425 °C
SiH$_4$ = 120 sccm
GeH$_4$ = 50 sccm
1% BCl$_3$ = 12 sccm
Pressure = 600 mTorr
Avg. Stress = -138.5 MPa

Film Thickness (µm)

Local Stress (MPa)

h)

DOE1-8
Temp = 425 °C
SiH$_4$ = 112 sccm
GeH$_4$ = 60 sccm
1% BCl$_3$ = 12 sccm
Pressure = 600 mTorr
Avg. Stress = -93.9 MPa

Film Thickness (µm)

Local Stress (MPa)

i)

DOE1-9
Temp = 425 °C
SiH$_4$ = 104 sccm
GeH$_4$ = 70 sccm
1% BCl$_3$ = 12 sccm
Pressure = 600 mTorr
Avg. Stress = -50.8 MPa

Film Thickness (µm)

Local Stress (MPa)

94

j)

DOE1-10
Temp = 425 °C
SiH_4 = 112 sccm
GeH_4 = 60 sccm
1% BCl_3 = 18 sccm
Pressure = 600 mTorr
Avg. Stress = -76 MPa

Film Thickness (μm)

Local Stress (MPa)

k)

DOE1-11
Temp = 440 °C
SiH_4 = 120 sccm
GeH_4 = 50 sccm
1% BCl_3 = 6 sccm
Pressure = 600 mTorr
Avg. Stress = -104.9 MPa

Film Thickness (μm)

Local Stress (MPa)

l)

DOE1-12
Temp = 440 °C
SiH_4 = 104 sccm
GeH_4 = 70 sccm
1% BCl_3 = 6 sccm
Pressure = 600 mTorr
Avg. Stress = -28.8 MPa

Film Thickness (μm)

Local Stress (MPa)

95

m)

n)

o)

Figure 4.5 Stress profiles and TEM images for DOE1 recipes.

96

Based on the stress profiles shown above, the average residual stress is expected to decrease (become less compressive) with increasing film thickness since the compressive stress gradually releases along the film thickness. The average strain gradient is also expected to decrease with increasing film thickness. As discussed previously, there is a thickness variation across the load for each deposition run. Figure 4.6 shows that the average residual stress becomes less compressive with increasing film thickness. Figure 4.7 shows that the strain gradient decreases with increasing film thickness, as expected. From Figure 4.4 it can be seen that the average residual stress and the strain gradient each varies with germanium content. The small variation (3 atomic percent) in germanium content across the load is a secondary effect for the observed dramatic changes in average residual stress and strain gradient.
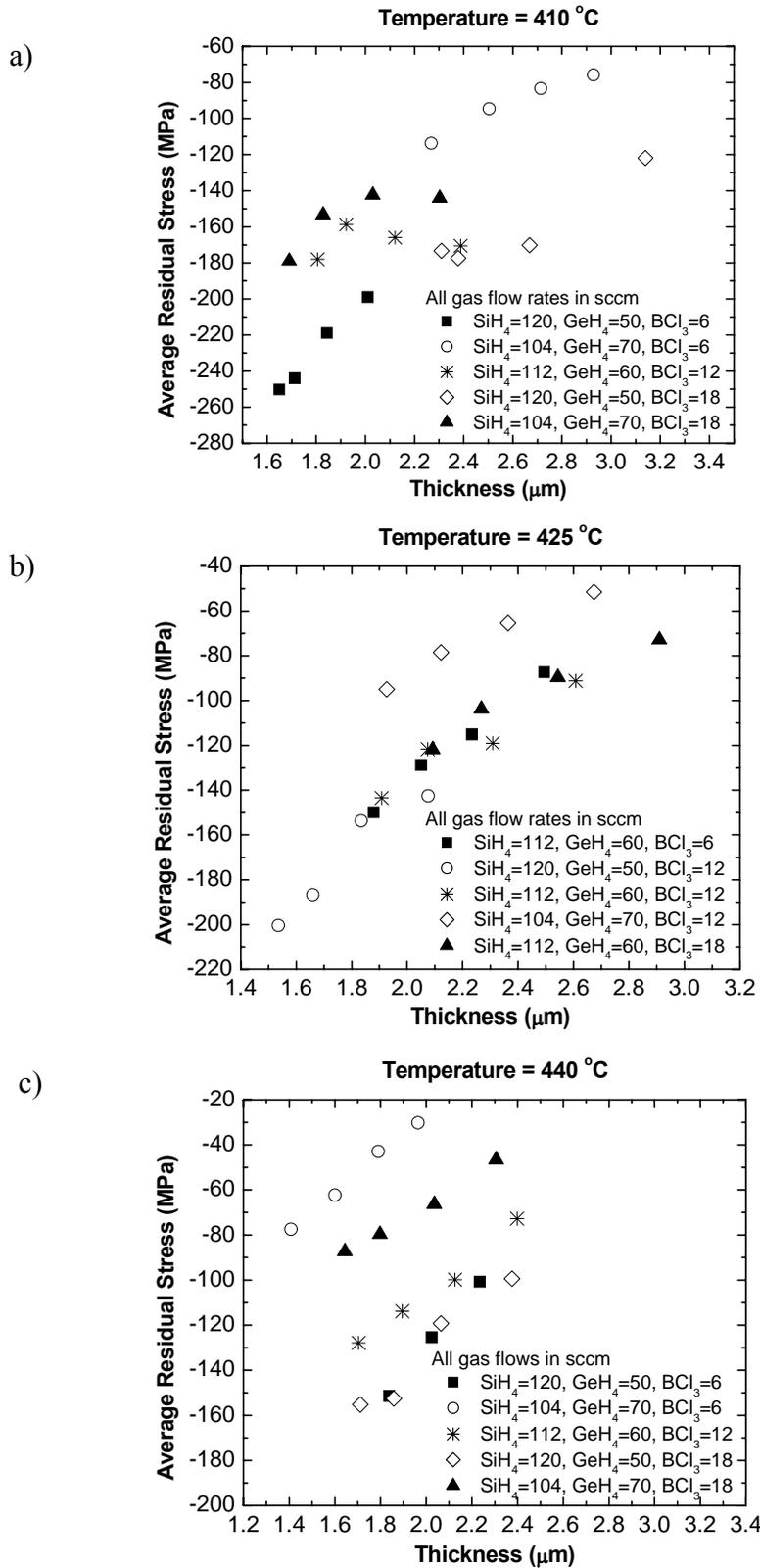
Figure 4.6 Average residual stress vs. film thickness for films deposited at various temperatures: a) 410°C; b) 425°C; c) 440°C
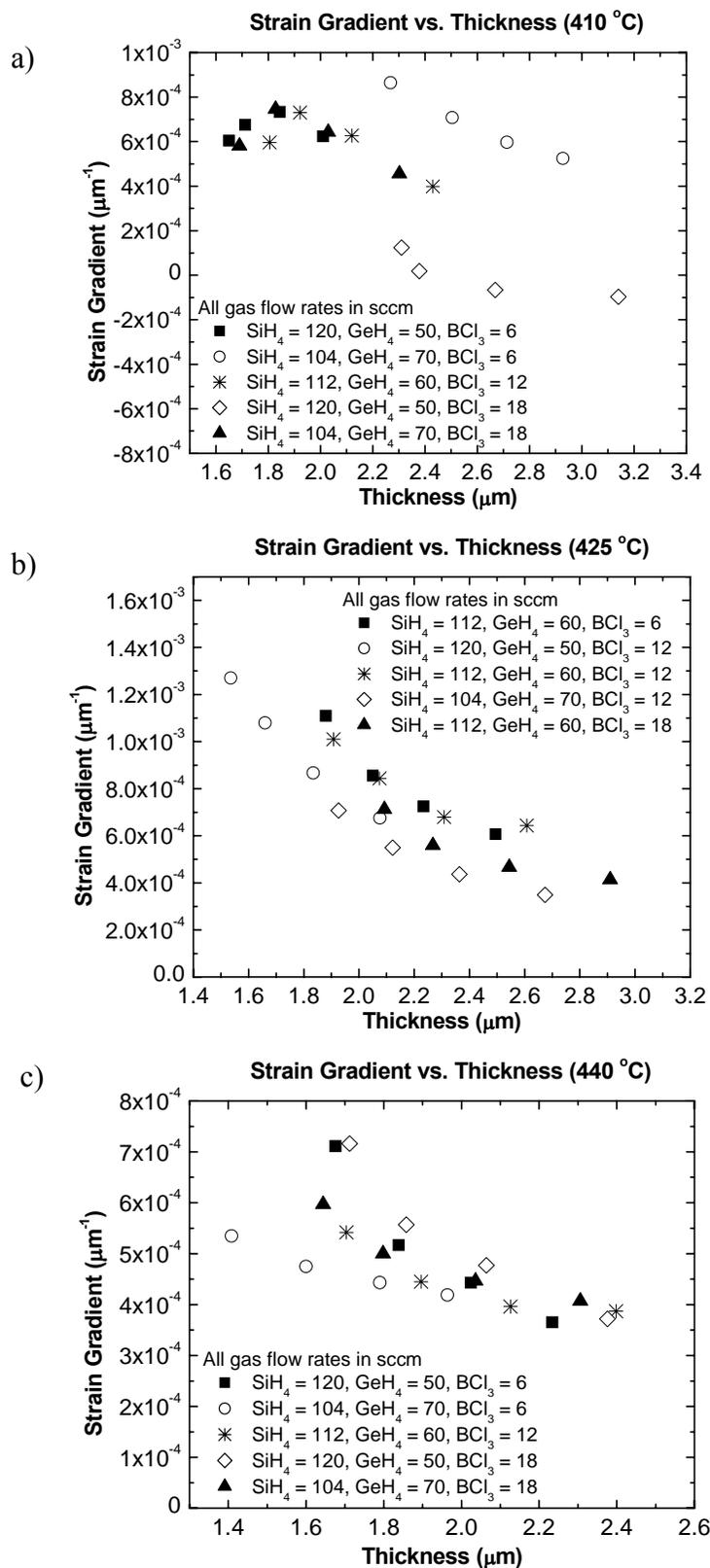
Figure 4.7 Strain gradient vs. film thickness for films deposited at various temperatures: a) 410°C; b) 425°C; c) 440°C

Figure 4.8 shows the correlation of strain gradient with resistivity, for 2-μm-thick films. Five measurements were taken from each wafer. It should be noted that the dopant injector is not well-optimized for uniformity; so there is variation in film resistivity across the wafer. Since the deposition rate does not depend on dopant concentration, the film thickness is fairly uniform across the wafer. For films deposited at 410°C, the strain gradient increases with resistivity, but there is no significant correlation seen for films deposited at 425°C or 440°C.

a)

Strain Gradient vs. Resistivity (410 $^{\circ}$C, ~ 2 μm)

b)

Strain Gradient vs. Resistivity (425 $^{\circ}$C, ~ 2 μm)

c)

Strain Gradient vs. Resistivity (440 $^{\circ}$C, ~ 2 μm)
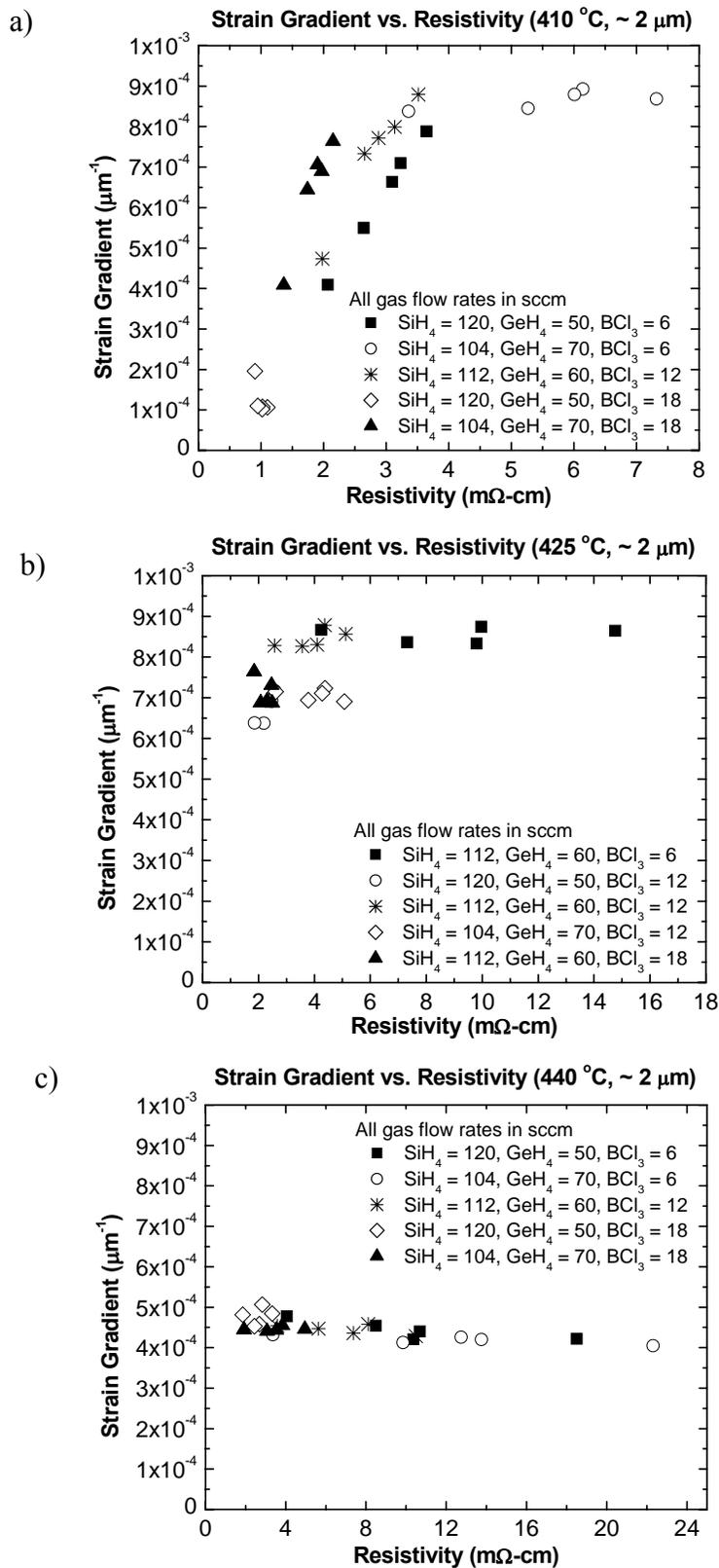
Figure 4.8 Strain gradient vs. resistivity for 2-μm thick films deposited at various temperatures: a) 410°C; b) 425°C; c) 440°C

101

The microstructure of selected films was studied by transmission electron microscopy (TEM). The images are shown next to the stress profile in Figure 4.5. Comparing the films deposited with Recipes DOE1-1 and DOE1-4, they are both deposited at 410°C but with different BCl_3 flow rates. The more heavily doped film has a thinner amorphous layer at the bottom of the film. This is consistent with previous reports that *in-situ* boron doping enhances the crystallinity of poly-SiGe [4.11]. In addition, the more heavily doped film has a vertically uniform grain structure through its thickness. The microstructure depth profiles correlate well with the stress depth profile measurements shown to the left. At the oxide-substrate interface, the film is amorphous and hence has highly compressive stress. Furthermore, the film with lower boron concentration has a conical grain structure and the variation in grain size along the film thickness results in a larger stress gradient.

The cross-sectional TEM image of films deposited at 440°C (Recipe DOE1-13 and DOE1-14) are also shown in Figure 4.5(m) and Figure 4.5(n). There is no significant difference in the microstructures for these two films although they have different doping level and germanium content. Due to the higher deposition temperature, the film is polycrystalline at the oxide substrate interface. As the film grew, the average grain size increased, so that the grains are conical. The stress profile shown to the left indicates that the highest compressive stress is located at the bottom of the film where the average grain size is smallest, which results in an upward curvature of the released film. As seen in Figure 4.8(b) and Figure 4.8(c), the strain gradients of the films deposited at 425°C and 440°C do not depend on the boron concentration. Thus it is very likely that the thermal effect is more significant for crystallinity compared to the boron doping effect. The

440°C recipes yield films with lower strain gradient compared to the 425°C recipes, possibly because the stress is relieved by *in-situ* annealing during the higher temperature deposition.

Comparing the TEM images of DOE1-3 and DOE1-13 in Figure 4.5(c) and Figure 4.5(l), the only difference in deposition condition is the temperature. Since higher temperature enhances crystallization, the crystal seeding of DOE1-13 starts earlier and has a higher density. The growth rate of the crystals also increases with temperature and the film in DOE1-13 is rougher at the surface. The microstructures of both recipes are conical in shape and have high strain gradient.

Revisiting Figure 4.8(a), it seems very promising to reduce the strain gradient by increasing the boron doping for films deposited at low temperature (near the amorphous-to-polycrystalline transition temperature). For a closer examination of the strain gradient *vs.* resistivity trend, all of the data for films deposited at 410°C are plotted in Figure 4.9. Figure 4.9(a) shows the data for two recipes yielding a linear correlation between strain gradient and resistivity. The improvement in strain gradient with decreasing resistivity is mainly due to crystallinity enhancement by boron doping. Figure 4.9(b) shows the data for two recipes that do not yield a linear correlation between strain gradient and resistivity. Since these recipes yield films with relatively high resistivity, this suggests that there exists a threshold of minimum boron doping required for crystallinity enhancement. Moreover, this threshold doping level depends on the germanium content: films with higher germanium content have better crystallinity for a given deposition temperature, and the boron doping effect is not as pronounced. Figure 4.9(c) shows the data for the recipe that yields the lowest strain gradient; the released films can have either

positive or negative out-of-plane curvature. The significant cross-load variation makes it difficult to control strain gradient via doping. The variation from wafer to wafer is due to cross-load variations in film thickness and germanium content. The smaller variation in strain gradient across a wafer is a result of microstructure non-uniformity. Since the deposited film consists of a single columnar-grain layer, local variations [4.12], [4.13] in microstructure makes strain-gradient control challenging in the range of 1×10^{-5} μm^{-1} and lower.
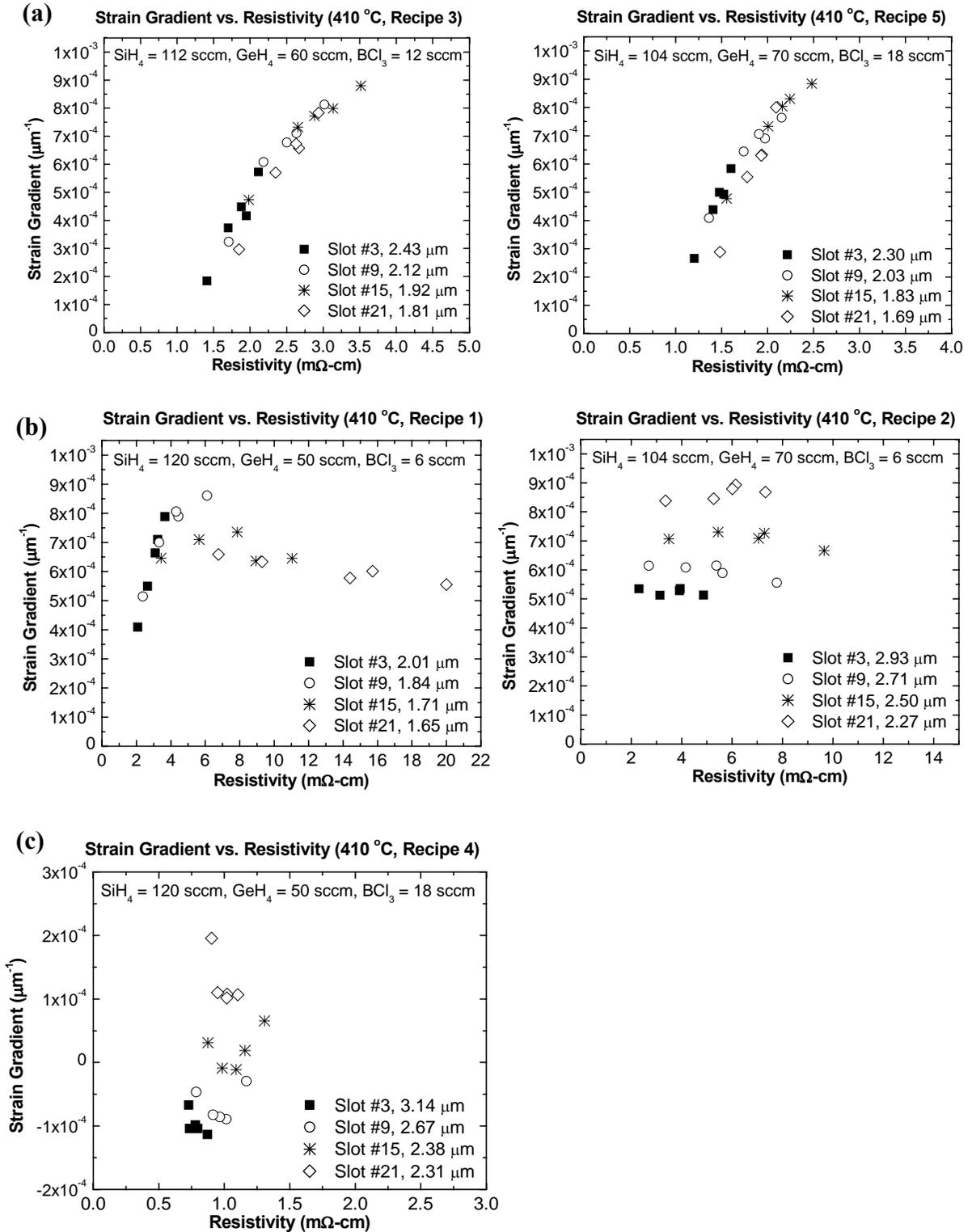
Figure 4.9 Strain gradient vs. resistivity for films deposited at 410 °C, showing: a) linear correlation; b) non-linear correlation; c) minimum strain gradient

4.4.4 Summary

The design of experiments method has been used to investigate deposition of *in-situ*-boron-doped poly-SiGe films by LPCVD. Films with low resistivity and slow wet-etch rate (in heated H_2O_2 solution) can be achieved at reasonable rates at low temperatures suitable for post-CMOS MEMS integration. Within the process space explored, all of the films have compressive residual stress; so designers must be aware of the potential for buckling of released clamped-clamped poly-SiGe beams. The minimum achievable strain gradient for a ~2 μm thick single layer of poly-SiGe is at least an order of magnitude higher than desired for inertial sensor applications. The large stress gradient is due to highly compressive stress in the lower portion of the film formed at the beginning of the deposition process. For films deposited at low temperature (near the amorphous-to-polycrystalline transition temperature), crystallinity can be enhanced by *in-situ* boron doping. As a result, films with higher boron doping develop a more columnar microstructure and hence a lower strain gradient. Strain gradient control in the range of 1×10^{-5} μm^{-1} remains a challenge for single step deposition that is ~2 μm thick due to local variations in single-layer columnar microstructures.

4.5 Ramping experiment

4.5.1 Experimental setup

The results of DOE1 show that the initially deposited amorphous region has higher compressive stress compared to the crystalline region, resulting in a positive stress profile within the film thickness. Also, films with conical microstructures have large strain gradients due to variations in grain size. Starting the deposition at high temperature

and high Ge content can enhance initial crystallization and the amorphous region at the oxide surface can be reduced.

To understand how the germanium content and temperature variation could affect the microstructure, a set of ramping experiment was performed. This set of experiment consists of a reference recipe (Ramp-ref), a SiH_4 flow ramp-up recipe (Ramp-SiH_4) and temperature ramp-down recipe (Ramp-temp) as listed in Table 4.2. The SiH_4-to-GeH_4 ratio is increased or the process temperature is decreased during the deposition in this experiment. All recipes have constant pressure, GeH_4 and BCl_3 flow rates. Higher BCl_3 flow rate 30 sccm was used to improve the resistivity uniformity. The reference recipe has constant temperature at 430°C and constant SiH_4 flow rate at 140 sccm. The SiH_4 flow ramp-up recipe has a constant temperature at 430°C. It has a step time of 30 minutes and the SiH_4 flow rate is ramped up from 140 sccm by +5 sccm at each step until reaching 190 sccm. The temperature ramp-down recipe has a constant SiH_4 flow rate at 140 sccm. It has a step time of 30 minutes and the temperature is ramp down from 430°C by -5°C at each step till 380°C. For both ramping recipes, the vacuum was not broken between steps to ensure continuous grain growth.

The process conditions of all three depositions are shown in Figure 4.10-12. As discussed in Chapter 2, the process temperature has sinusoidal fluctuations at constant set point and this phenomenon can be seen again in Figure 4.10 for the reference recipe. The process pressure and the gas flow rates are very stable. For the SiH_4 flow ramp-up deposition shown in Figure 4.11, the SiH_4 MFC can quickly follow the input value and has a step response. The temperature ramp-down deposition has the process temperature following the set point with some oscillation, as shown in Figure 4.12.

107
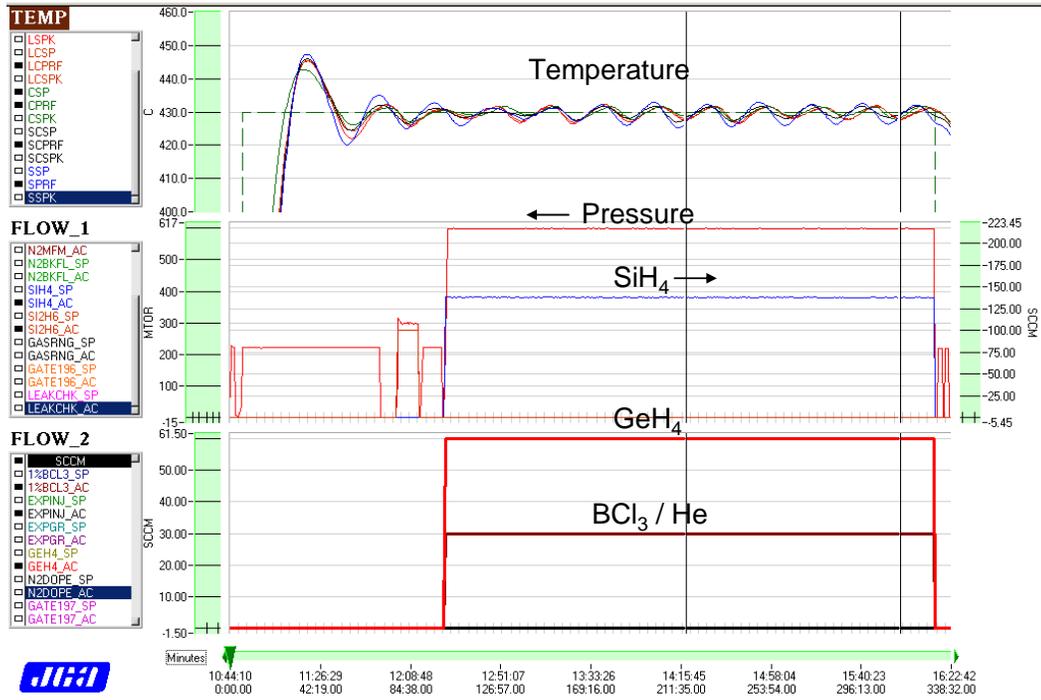
Reference Run

Figure 4.10 Process conditions of the reference deposition (Ramp-ref)

SiH$_4$ Ramp Up
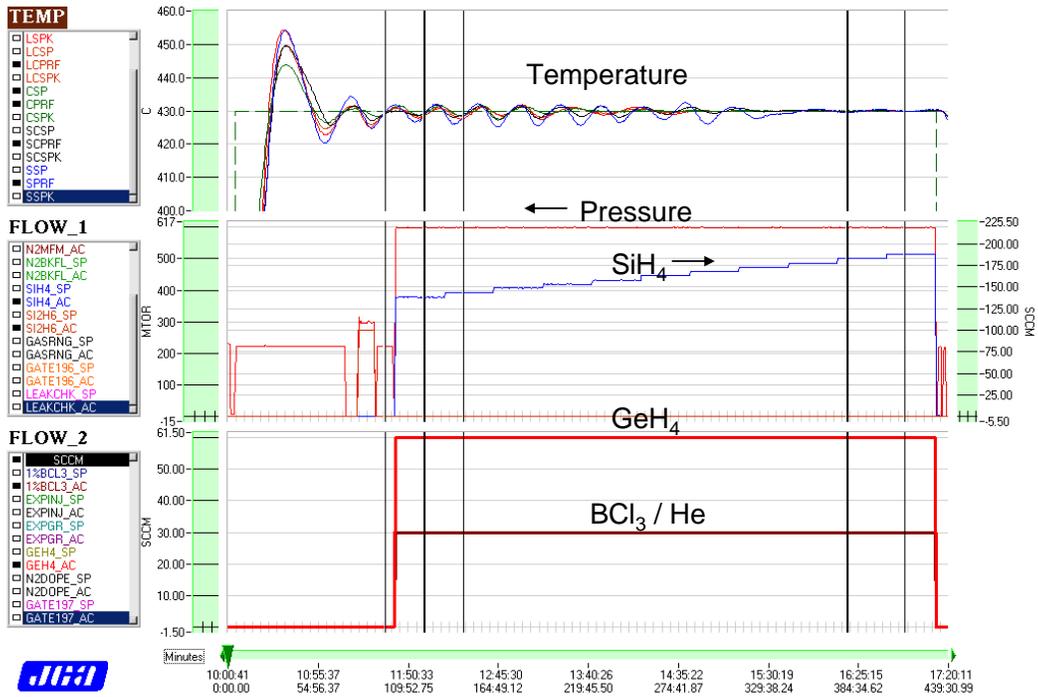
Figure 4.11 Process conditions of the SiH$_4$ flow ramp-up deposition (Ramp-SiH$_4$)

108
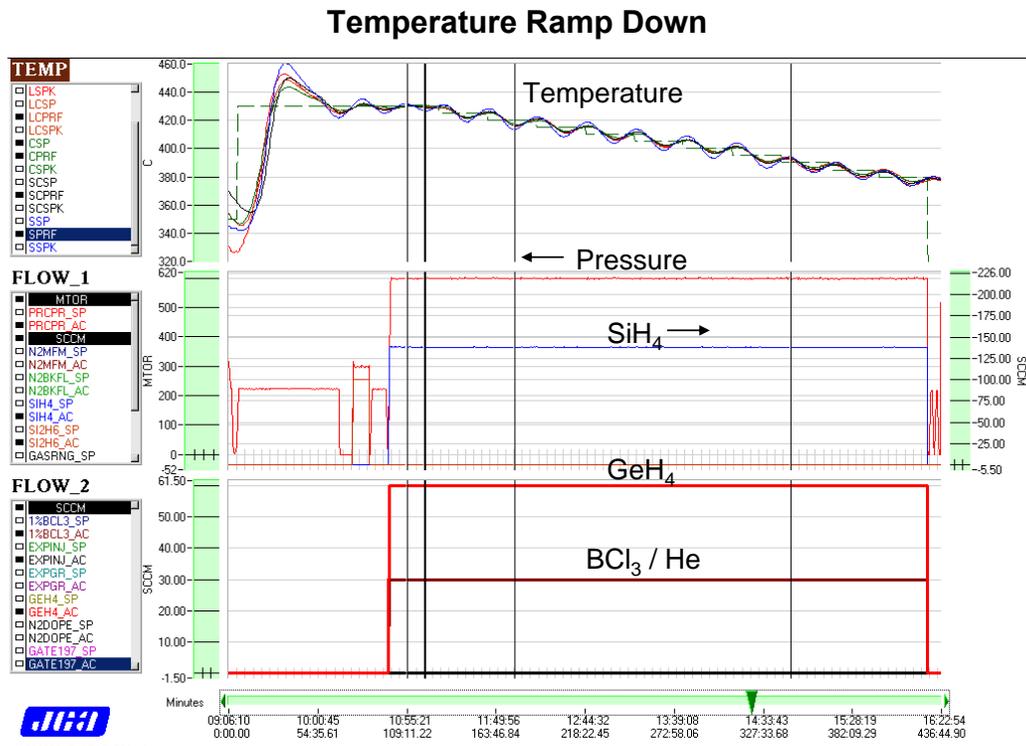
Figure 4.12 Process conditions of the temperature ramp-down deposition (Ramp-temp)

4.5.2 Results and interpretation

The data of the ramping experiments are listed in Table 4.2. All of the depositions targeted film thickness of 2 μm. The deposition rate for the two ramping recipes was underestimated and films are thicker than expected. Comparing to DOE1, resistivity uniformity is improved with high BCl_3/He flow rate. Since the stress and strain gradient vary with film thickness, wafers with 2.3 μm thick film from these three runs are being compared for mechanical properties. The reference run (Ramp-ref), the SiH_4 flow ramp-up run (Ramp-SiH_4) and the temperature ramp-down run (Ramp-temp) have average residual stress of -109 MPa, -140 MPa and -183 MPa. The result for average residual stress is as expected. Low germanium content and low temperature films have higher

compressive stress. Since all recipes have the same starting layer, the larger compressive stress is from the low germanium content or low temperature top layers.

The strain gradients for the 2.3 μm thick film from each recipe have no significant differences; all are around 4.5×10^{-4} μm^{-1}. The stress profile and the cross-sectional TEM images are shown in Figure 4.13. Considering the measurement errors, the stress profiles of the three recipes do not show a significant difference. Also, the microstructures of the three recipes have similar conical texture.

It should be noted that the last few layers of the SiH_4 flow ramp-up run and the temperature ramp-down run would give amorphous films if they were deposited directly on oxide. Since the grain growth is continuous, the low Ge content or low temperature layers follow the "footprint" of the existing polycrystalline grain structure and continue to be polycrystalline. The surface roughness of the temperature ramp-down recipe is significantly lower than the reference recipe due to the low processing temperature later in the deposition.
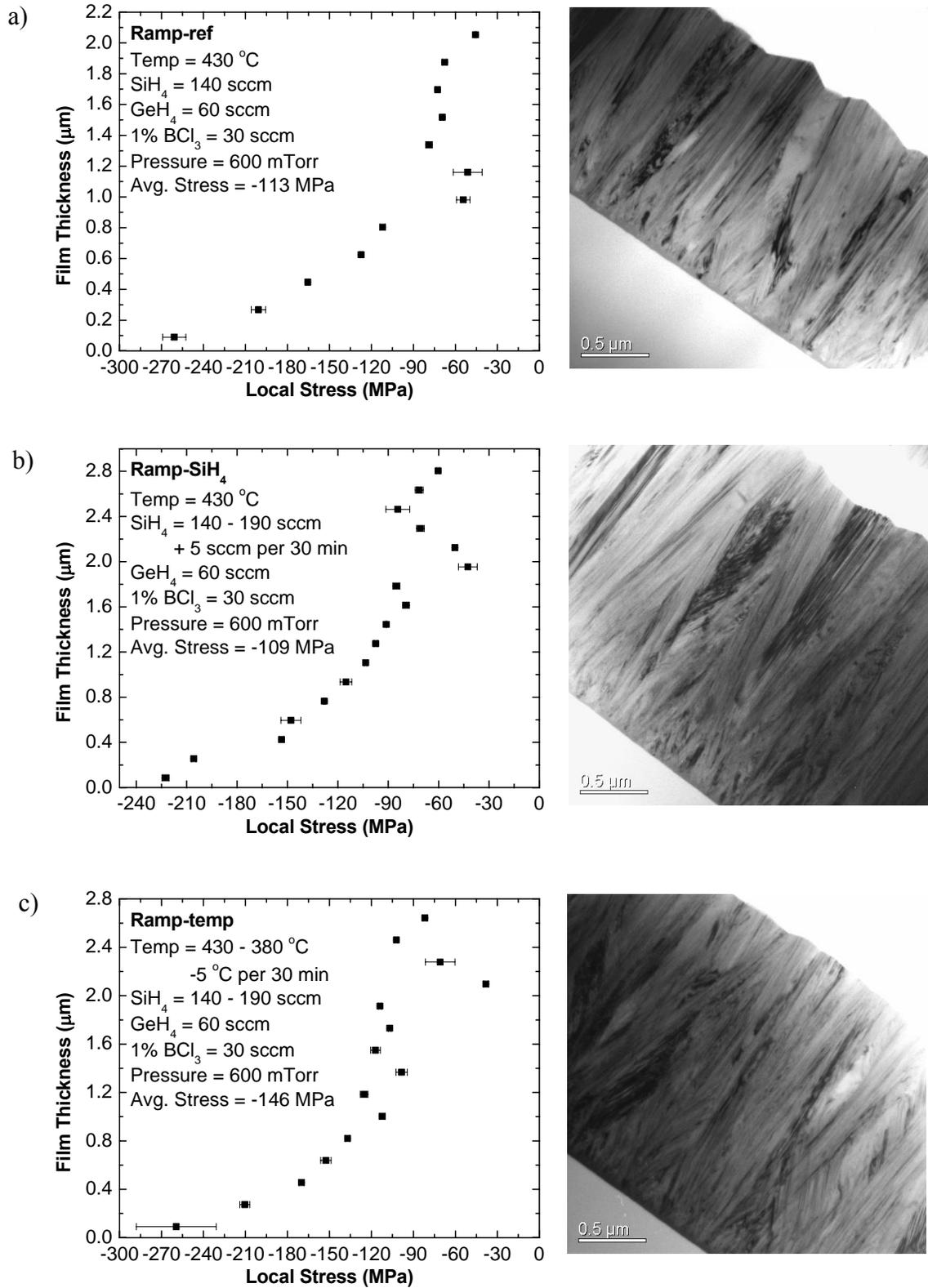
Figure 4.13 Stress profiles and cross-sectional TEM images for the ramping experiments

4.5.3 Summary

The set of ramping experiments show that ramping up the SiH_4 flow rate or ramping down the process temperature during the deposition increases the average compressive stress in the film and does not improve strain gradient. The lower germanium content or lower temperature deposition slows down the deposition rate and lateral diffusion rate becomes significant. All films in this experiment have conical microstructures with high strain gradient. The temperature ramp down recipe is desired for reducing thermal budget without sacrificing the deposition rate or resistivity, but temperature control is problematic with the Tystar furnace at low deposition temperatures.

4.6 2nd design-of-experiments

4.6.1 Experimental setup

The 1st DOE shows that recipes utilizing low BCl_3 flow rate tend to have worse cross-wafer uniformity in resistivity due to gas depletion effects. Also, at low deposition temperature (410°C), strain gradient decreases with resistivity. The BCl_3 mass flow controller was re-calibrated from 20 sccm range to 50 sccm range after DOE1. Higher BCl_3 gas flow rate of 15 sccm, 30 sccm and 45 sccm were used in DOE2, in order to look into improvement in resistivity uniformity and strain gradient with higher doping levels.

Variation in pressure is also explored in DOE2. Higher process pressure enhances deposition rate, but film thickness uniformity will be sacrificed if the deposition is so fast that it is no longer limited by the surface reaction rate. DOE1 used 600 mTorr process pressure. This process pressure results in reasonable deposition rate with good film

thickness uniformity. A few short test runs (DOE2-t1 through DOE2-t5 listed in Table 4.2) were performed to understand the process pressure range for DOE2. The test runs show that the deposition rate increases with process pressure, but films deposited at pressure above 700 mTorr are very rough with significant color variation across the wafer. Gas phase nucleation happened in these cases. With high process pressure, some nucleation happens before the gas molecules reach the wafer surface [4.14]. The clusters formed in gas-phase nucleation coat the wafer surface later. Diffusion is limited on the wafer surface for these clusters and the film on the wafer is porous and has poor uniformity. The process pressures for DOE2 were chosen as 350 mTorr and 600 mTorr.

The six depositions of the 2nd DOE with BCl$_3$ flow rate and process pressure as variables are listed in Table 4.2. A deposition temperature of 410°C and germanium content of ~60% were chosen based on the results from DOE1. All depositions targeted film thickness of 2 μm. Reducing the strain gradient is the main goal for this set of experiments.

4.6.2 Results and interpretation

The results are also summarized in Table 4.2. Higher BCl$_3$ flow rate enhances the deposition rate. Higher BCl$_3$ flow rate also reduces the gas depletion effects and improves the cross wafer resistivity uniformity. Lower process pressure decreases the deposition rate, but improves the cross wafer resistivity uniformity.

The strain gradient for the films deposited at 600 mTorr (DOE2-1, DOE2-2, DOE2-3) is relatively low, which is consistent with the results from DOE1. Figure 4.14(a) plots the strain gradient against the resistivity for the 600 mTorr runs. For recipe

DOE2-1 with 15 sccm of BCl_3 flow, the strain gradient decreases with resistivity and this follows the trend from Figure 4.8(a). However, strain gradient becomes independent of doping after resistivity < 1 mΩ-cm. Recipe DOE2-2 gives the lowest strain gradient, but there is a significant amount of variation.

Films deposited at 350 mTorr (DOE2-4, DOE2-5, DOE2-6) have relatively high strain gradient. The relationship between strain gradient and resistivity is plotted in Figure 4.14(b). For the 350 mTorr depositions, doping does not help reduce the strain gradient as much as for the 600 mTorr depositions. Lower pressure gives lower deposition rate; so crystallinity enhancement by boron doping is less significant.

a)

Strain Gradient vs. Resistivity (~ 2 μm)

b)

Strain Gradient vs. Resistivity (~ 2 μm)

Figure 4.14 Strain gradient vs. resistivity for DOE2: a) 600 mTorr depositions (DOE2-1, DOE2-2 and DOE2-3); b) 350 mTorr depositions (DOE2-4, DOE2-5 and DOE2-6)

115

As discussed before, from the same run, there is film thickness variation across the load due to the gas depletion effect with wafers closer to gas inlet having a thicker film. Strain gradient vs. film thickness are plotted in Figure 4.15. For the 600 mTorr depositions, strain gradient decreases with increasing film thickness, but the slope of the trend becomes smaller after a certain thickness for recipes DOE2-2 and DOE2-3. As discussed in the 1st DOE, films with strain gradient below 1×10^{-4} μm^{-1} have significant variation across the wafer due to local variation in microstructure. For the 350 mTorr depositions, strain gradient is almost independent of film thickness.
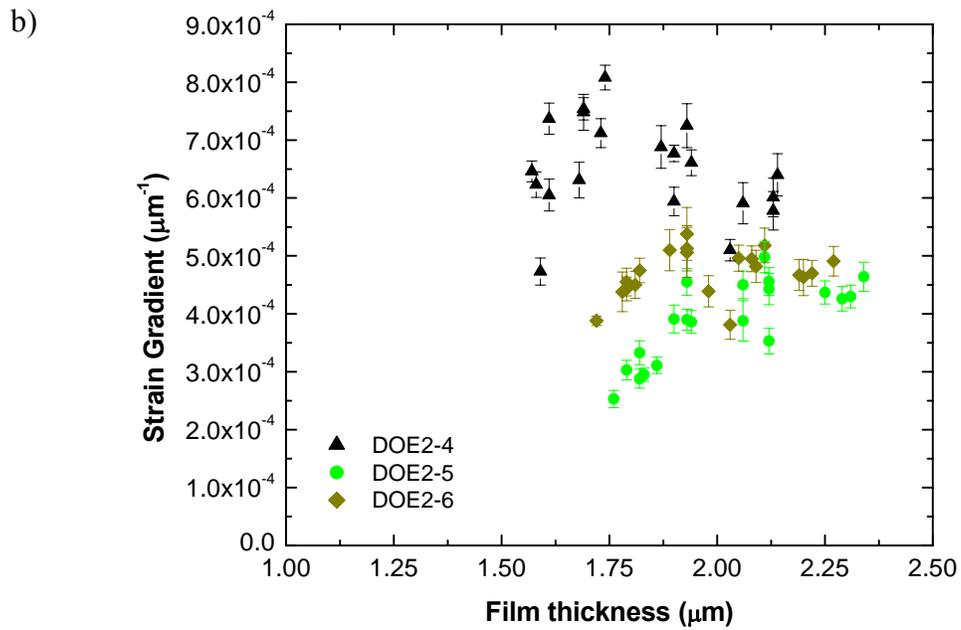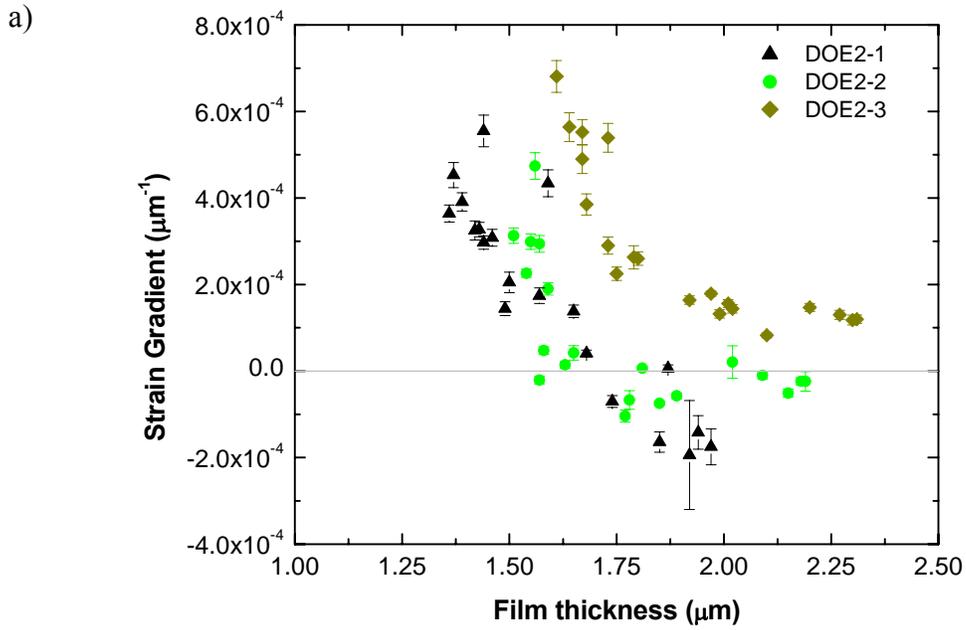
Figure 4.15 Strain gradient vs. film thickness for DOE2: a) 600 mTorr depositions (DOE2-1, DOE2-2 and DOE2-3); b) 350 mTorr depositions (DOE2-4, DOE2-5 and DOE2-6)

117

Stress profile and TEM images for each recipe are shown in Figure 4.16. The 600 mTorr depositions have uniform stress profiles along the film thickness, whereas the 350 mTorr depositions have positive stress profiles, corresponding to large strain gradients. Comparing the TEM images of DOE2-1, DOE2-2 and DOE2-3, there are no significant differences in their microstructures. They all have ~200 nm amorphous layers at the bottom. In each film, there are a few grains with very low defect density and others have twinning defects. Recipe DOE2-2 has the lowest strain gradient in this set of experiment and its microstructures have the lowest defect densities. Figure 4.15(a) shows that strain gradient decreases with film thickness for the 600 mTorr depositions. The amorphous portion of the film has higher compressive stress compared to the columnar portion and this contributes to a positive strain gradient. Thicker films consist of a large columnar portion; so the effect of the amorphous portion is reduced.

The film deposited at 350 mTorr (DOE2-5) has a much thinner amorphous region. Also, the low pressure film has a conical texture with twinning defects in all grains. The slope of the stress is roughly constant and the strain gradient does not have a strong dependence on film thickness. In this case, the strain gradient is related to the variation in grain size in the film.

a) DOE2-1
Temp = 410 °C
SiH$_4$ = 140 sccm
GeH$_4$ = 60 sccm
1% BCl$_3$ = 15 sccm
Pressure = 600 mTorr
Avg. Stress = -208 MPa

b) DOE2-2
Temp = 410 °C
SiH$_4$ = 140 sccm
GeH$_4$ = 60 sccm
1% BCl$_3$ = 30 sccm
Pressure = 600 mTorr
Avg. Stress = -187 MPa

c) DOE2-3
Temp = 410 °C
SiH$_4$ = 140 sccm
GeH$_4$ = 60 sccm
1% BCl$_3$ = 45 sccm
Pressure = 600 mTorr
Avg. Stress = -148 MPa

d) DOE2-4
Temp = 410 °C
SiH₄ = 140 sccm
GeH₄ = 60 sccm
1% BCl₃ = 15 sccm
Pressure = 350 mTorr
Avg. Stress = -166 MPa

e) DOE2-5
Temp = 410 °C
SiH₄ = 140 sccm
GeH₄ = 60 sccm
1% BCl₃ = 30 sccm
Pressure = 350 mTorr
Avg. Stress = -159 MPa

f) DOE2-6
Temp = 410 °C
SiH₄ = 140 sccm
GeH₄ = 60 sccm
1% BCl₃ = 45 sccm
Pressure = 350 mTorr
Avg. Stress = -151 MPa

Figure 4.16 Stress profiles and cross-sectional TEM images for DOE2

120

Test run DOE2-t4 has similar parameters as run DOE2-2, except for a shorter deposition time. The TEM images from these two runs are put together for comparison in Figure 4.17. Two samples from different boat locations are being studied for the initial grain growth process from run DOE2-t4 with 60 minutes of deposition time. Figure 4.17(a) is a sample from a wafer closer to the gas outlet (slot #15) and Figure 4.17(b) is a sample from a wafer closer to the gas inlet (slot #3). The wafer at the gas outlet has a thinner film and there is still some amorphous region being exposed at the top surface. The wafer closer to gas inlet has a thicker film and the top surface is completely crystallized. The crystal seeds are spaced out about 100 nm apart. The crystals grow vertically and expand laterally. Once the neighboring crystals meet, the amorphous region is covered up. Figure 4.17(c) is a sample from DOE2-2 with 230 minutes of deposition time. We can see that the thickness of the amorphous region for DOE2-t4 and DOE2-2 is about the same. Longer deposition time or *in-situ* annealing at the deposition temperature does not crystallize the amorphous region.
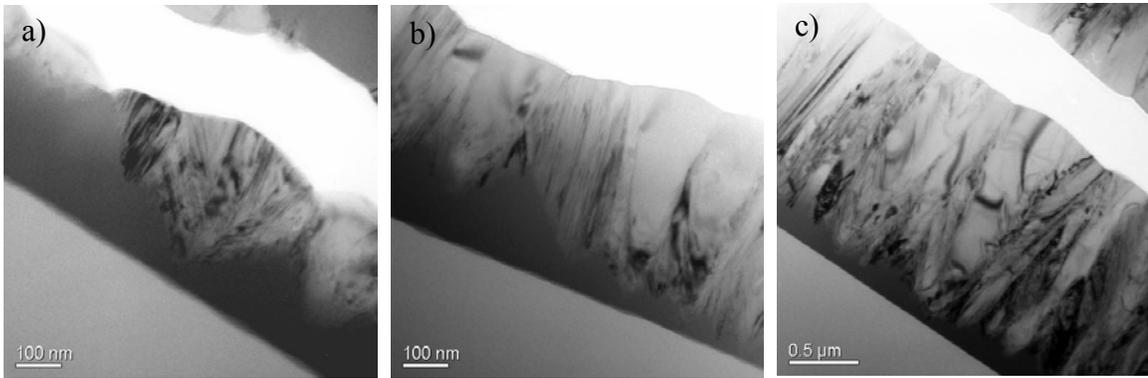
Figure 4.17 TEM images for recipe 410 °C, 600 mTorr, 140 sccm SiH_4, 60 sccm GeH_4 and 35 sccm BCl_3: a) film deposited for 60 minutes at wafer slot #15; b) film deposited for 60 minutes at wafer slot #3; c) film deposited for 230 minutes at wafer slot #9

To further understand the formation of the film deposited by DOE2-2, a special TEM sample was prepared by double-wedge technique for top view imaging at various depths. The double-wedge TEM analysis is courtesy of Dr. Erdmann Spiecker of the National Center for Electron Microscopy at Lawrence Berkeley National Laboratory. Depth profile quantification is still being studied. Images in Figure 4.18 are taken at the same magnification from the bottom to the top of the film. More pictures are taken at the lower portion (the first 0.5 μm) of the film where the grain evolution occurs. These images clearly show the grain growth process during the deposition. Near the sacrificial oxide layer, the SiGe film has a transition zone from amorphous to polycrystalline. The sparse crystalline seeds start among the amorphous region. As the deposition goes along, the seeding density and crystal size increase. Eventually, the film becomes fully crystalline and the grains reach their final lateral size once the film reaches 0.4 μm in thickness.

122
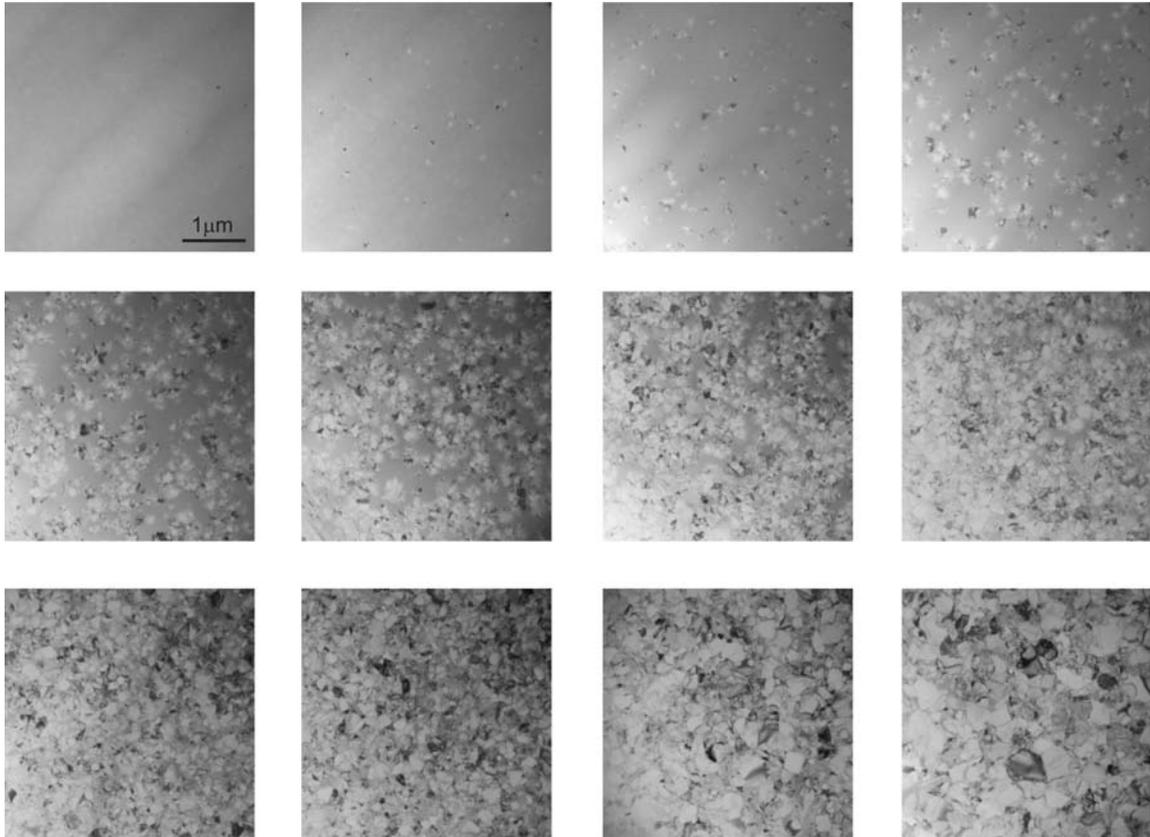
Figure 4.18 Top view TEM images for film deposited with recipe DOE2-2 at various depths (Courtesy of Dr. Erdmann Spiecker)

4.6.3 Summary

The 2nd DOE confirms the low strain gradient result from the 1st DOE. The optimal recipe for low strain gradient film is found to be: 410 °C, 600 mTorr, 140 sccm SiH$_4$, 60 sccm GeH$_4$ and 35 sccm BCl$_3$. The low strain gradient film consists of a thin amorphous region at the oxide interface and columnar crystalline microstructure with very few defects. The films consist of a single layer microstructure and low range strain gradient $< 1 \times 10^{-4}$ μm^{-1} is very sensitive to small variation in microstructure. The thickness variation of the amorphous region results in large variation in strain gradient.

123

Increasing the overall film thickness will increase the polycrystalline portion of the film and the effect of the bottom amorphous layer will be minimized. Also, thicker films result in beams that are stiffer for out-of-plane bending, which reduces the impact of the strain gradient. Multilayer depositions with separate Si_2H_6 nucleation might create several layers of microstructures. The randomness of microstructure could be averaged out, resulting in better strain gradient uniformity. The thick film deposition and the layer stack experiments will be discussed in the following sections.

4.7 Thick deposition

Using the optimal recipe from DOE2 with a longer deposition time, thicker films are being studied. This experiment targets film thickness of 4 μm, whereas all previous experiments targeted film thickness of 2 μm. The results of the thick deposition (ThickDepo) are summarized in Table 4.2. Figure 4.19 shows a SEM image of a released cantilever beam array. The strain gradient of this film is very small and the tip deflection of the cantilever beam is hardly visible. The strain gradient vs. thickness for the thick deposition is plotted together with results from DOE2 in Figure 4.20. As expected, the strain gradient and its uniformity are improved as the film thickness increases. The strain gradient reaches the range of 1×10^{-5} μm^{-1}. The variations across the load and across the wafer are also significantly smaller.
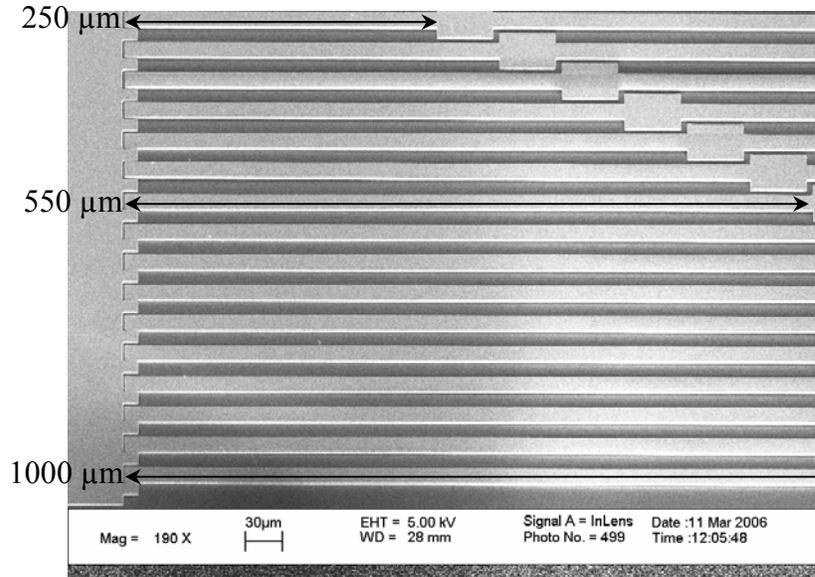
Figure 4.19 SEM image of released cantilever beam array for Recipe ThickDepo.
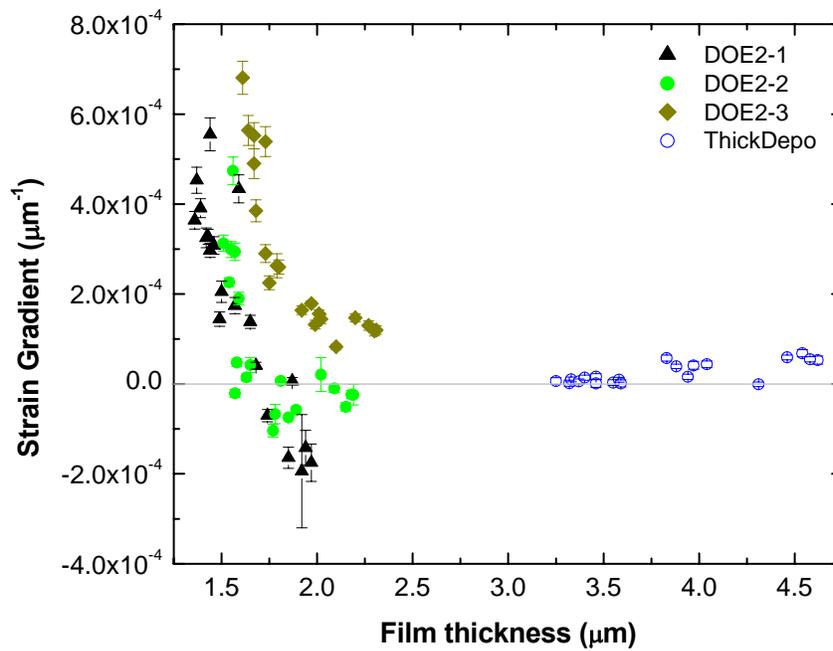
Figure 4.20 Strain gradient vs. film thickness plot

The stress profile and the cross sectional TEM image for the thick deposition are shown in Figure 4.21. The stress profile through the film thickness is relatively uniform. The film consists of a large portion of columnar microstructure and the defect density within each grain is very low. This thick film has similar deposition condition as DOE2-2, except for slightly higher BCl_3 flow rate and longer deposition time. Comparison of the stress profile and the microstructure of this thick film with DOE2-2 shown in Figure 4.16(b) can be made. The thicker film has slightly lower (less compressive) stress, especially for the stress in the lower portion of the film. Some of the stress might be released due to *in-situ* annealing during the long deposition. In both cases, the thickness of the amorphous region is similar, but the thicker film has taller columnar grains. Thus, a larger portion of the thicker film consists of columnar crystalline structures.
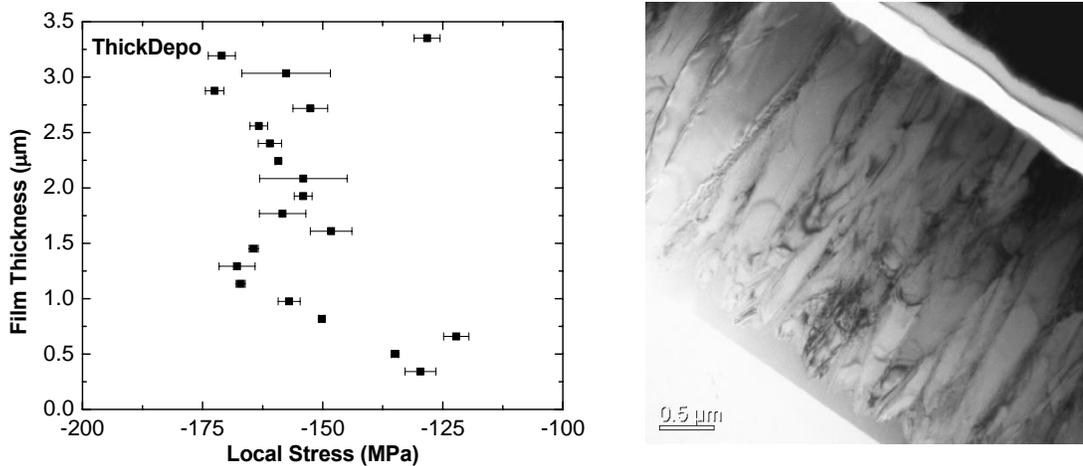
Figure 4.21 Stress profile and cross sectional TEM image for recipe ThickDepo

4. 8 Multiple-layered film deposition

Fine-grained poly-Si films with low strain gradient have been demonstrated to be sufficiently reproducible for high-volume production [4.4]. If the average grain size in a poly-SiGe film is comparable to its thickness, there can be considerable variation in mechanical properties from beam to beam, which is not acceptable for high-volume manufacturing processes. The average grain size can be limited by depositing the film in multiple steps to create a layered stack, to average out random variations in grain microstructure and modify the stress-*vs.*-depth profile.

As a proof-of-concept experiment, Recipe LayerStack in Table 4.2 consists of four 85-minute depositions of Recipe DOE2-5. To ensure grain growth interruption from layer to layer, the vacuum was broken in-between the depositions by opening the furnace door. The disadvantage of this approach is that the temperature has to re-stabilize and temperature overshoot occurs during the stabilization. As a result, more processing time is required and hence the thermal budget is larger. With a more sophisticated LPCVD reactor, it should be possible to simply flow O_2 in-between depositions to avoid the need to open the door, so that the furnace temperature can remain stable throughout the film deposition process and therefore process throughput would not be affected significantly.

The stress distribution within the layered film and the cross sectional TEM image are shown in Figure 4.22. The stress profile of the layered film also consists of four regions, each very similar to the stress profile for the bottom quarter of the film shown in Figure 4.16(e) for recipe DOE2-5. Every layer within the film is very similar to the bottom quarter of the film shown in Figure 4.16(e). Overall, the stress distribution with the multiple-layered film is more uniform as compared to a single-layered film, so that

the absolute value of the strain gradient is smaller. Due to unintentional heating during temperature stabilization, the earlier deposited layers were annealed so that their amorphous regions are partially crystallized, resulting in a downward curvature (negative strain gradient) of the released cantilever beam. This fine-grained layered-stack film ends up with a strain gradient of -1.2 $\times 10^{-4}$ μm^{-1}. Finer grains and more uniform stress distribution can be achieved with more layers. To avoid having a negative strain gradient, a fully crystallized film should be used because of its better thermal stability.
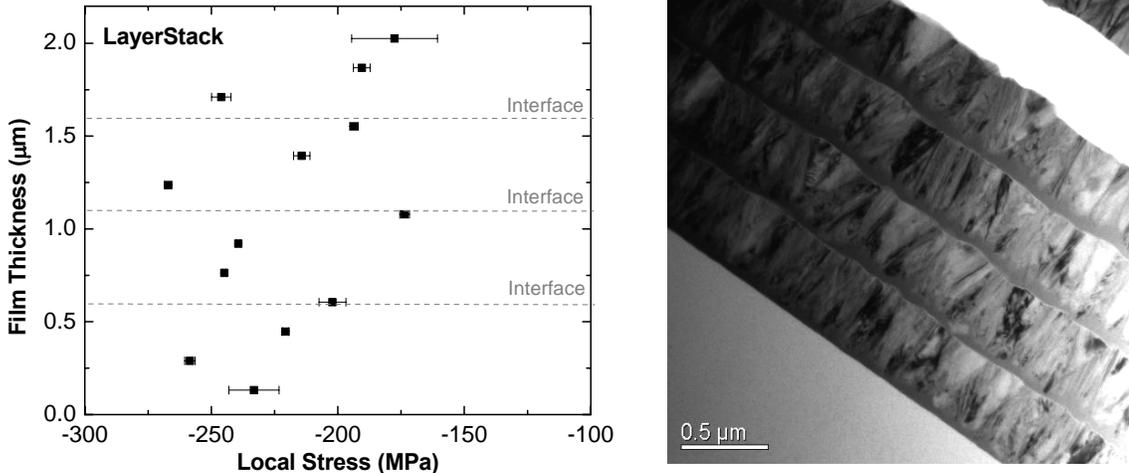
Figure 4.22 Stress profile and cross sectional TEM image for Recipe LayerStack

4.9 Seeding layer experiments

Previous TEM images show that the SiGe film starts out amorphous at the oxide interface for deposition temperature lower than 440 °C with Si_2H_6 seeding. For low strain gradient film, the thickness variation of this amorphous region causes a uniformity problem. This section discusses methods to minimize the amorphous region and its effects on strain gradient.

In the interest of lowering the thermal budget, a 410°C deposition temperature is being studied here. All of the previous experiments show that films deposited at 410°C have an amorphous starting layer, but eventually crystallize. In the earliest stage of film formation, the nuclei are spaced far apart. Before they can diffuse on the surface to find low energy crystal lattice sites, they are pinned to the substrate by subsequently absorbed atoms (adatoms). Eventually, these adatoms form clusters serving as crystal seeds and subsequently adatoms can attach to crystal seeds, resulting in their growth. To initialize the crystallization earlier in the deposition, a lower deposition-rate seeding layer could be used.

A quick test on this crystallization hypothesis was done with a low gas flow rate and low pressure at 410°C (SiGeSeed-t1 listed in Table 4.2). In this recipe, low pressure and low SiH_4 and GeH_4 flow rates are used to reduce the deposition rate. BCl_3 flow rate stays high to enhance crystallization. At low deposition rates, gas molecules have more time to settle down at low energy crystal lattice sites on the wafer surface before the next gas molecules are adsorbed. The cross sectional TEM image is shown in Figure 4.23. Fully crystallized films are achieved at the oxide interface at 410°C with ~60% germanium content.
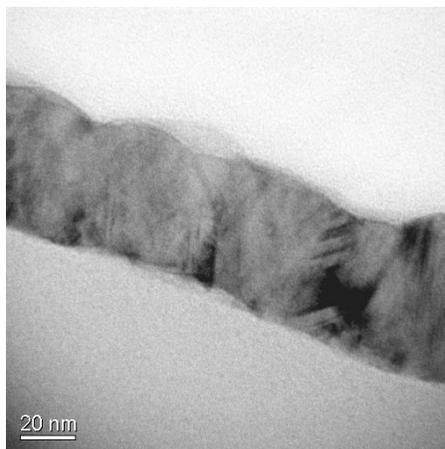
Figure 4.23 Cross sectional TEM image for film deposited with recipe SiGeSeed-t1

The above recipe (SiGeSeed-t1) is used as the seeding layer for deposition SiGeSeed-1 (listed in Table 4.2). After the thin crystallized seeding layer deposition, the most optimal recipe from DOE2 was used for the main deposition. The vacuum was not broken between the seeding layer and the main deposition. The process pressure and gas flow rates are ramped up immediately in the process recipe. This recipe was intended to grow a columnar microstructure without an amorphous region at the oxide interface.

The stress profile and cross sectional TEM image of recipe SiGeSeed-1 is shown in Figure 4.24. The film is indeed fully crystallized, but the texture is conical rather than columnar. The initial low pressure and low gas flow rates seeding recipe enhances crystalline seeding due to the resultant low deposition rate and high boron concentration. Fine crystal grains formed during the initial stage of film deposition compete for lateral growth, resulting in a conical grain structure and high strain gradient. Since there is variation in grain size through the film thickness, there is also variation in the stress distribution. At the bottom of the film where the randomly oriented grains compete to

130

grow, higher compressive stress is developed. This film has high strain gradient. During the grain growth, grains oriented with the fastest growing plane survive and the film consists of conical structures.
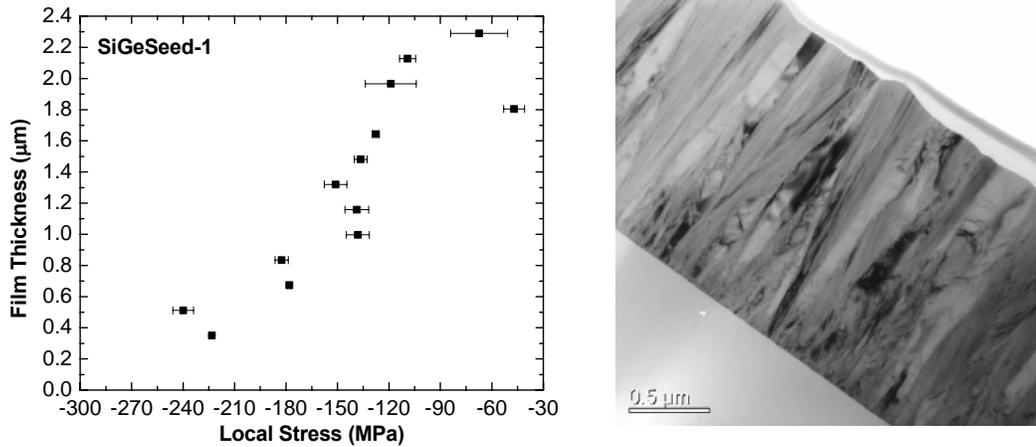
Figure 4.24 Stress profile and cross sectional TEM image for recipe SiGeSeed-1

The above experiment shows that fine grain seeding results in conical microstructures with high strain gradient. In order to achieve film with low strain gradient and good uniformity, the amorphous region should be suppressed while keeping a columnar texture. The 2nd DOE shows that films deposited at 350 mTorr have thinner and more uniform amorphous regions, with sparse crystal seeding. The next seeding experiment SiGeSeed-2 combines recipes DOE2-5 and DOE2-2. Recipe DOE2-5 was used for the initial 20 minute seeding and recipe DOE2-2 was used for the main deposition. The process pressure jumps from 350 mTorr to 600 mTorr after the seeding layer without breaking vacuum. This recipe was designed to deposit a film with a very

thin amorphous region and columnar crystal structures. The deposition time targeted 2 μm film thickness.

The results of the SiGeSeed-2 deposition are listed in Table 4.2. The stress profile and the cross sectional TEM image are shown in Figure 4.25. As expected, this film has auniform stress distribution. The texture is columnar, with an initial amorphous region of about 0.1 μm in thickness. The defect density within each grain is low.
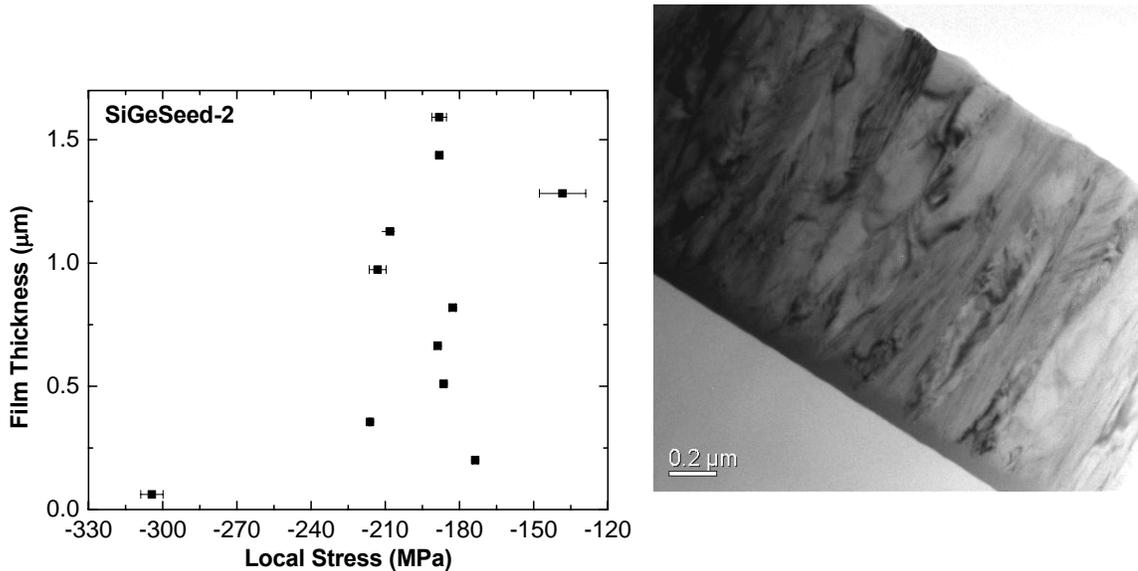
Figure 4.25 Stress profile and cross sectional TEM image for recipe SiGeSeed-2

Columnar microstructure is a characteristic feature for films with low strain gradient. The film deposited by recipe SiGeSeed-2 has a low strain gradient and the strain gradient uniformity is significantly better than other film with similar thicknesses as shown in Figure 4.26. Comparing the TEM images for SiGeSeed-2 with those of recipes DOE2-1, DOE2-2 and DOE2-3 in Figure 4.16, the main difference is in the amorphous region. Films deposited by recipe SiGeSeed-2 have thinner and more uniform amorphous

regions. For the recipes in DOE2, the variations in amorphous region results in large variations in strain gradient.
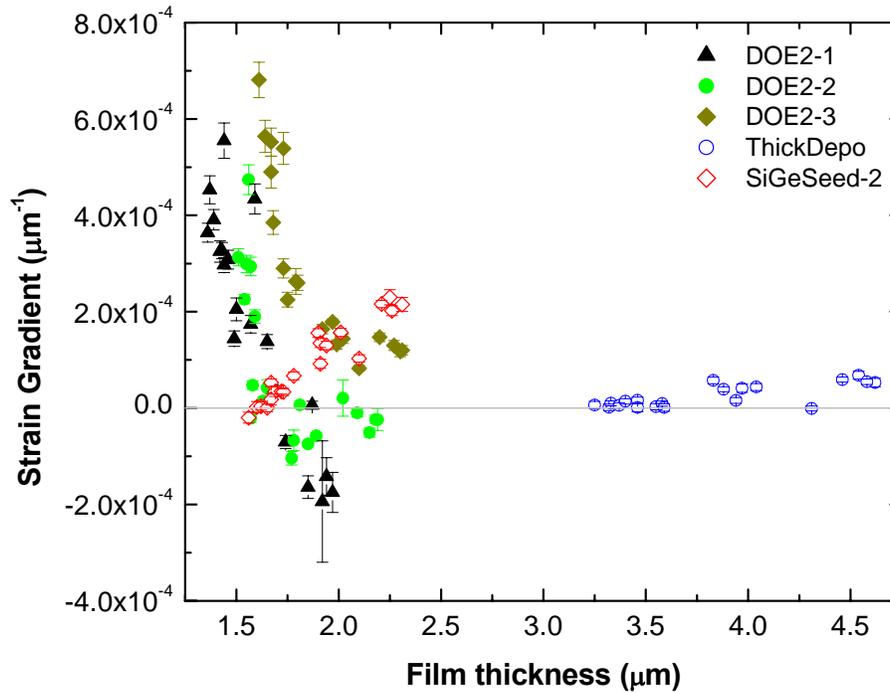
Figure 4.26 Strain gradient vs. film thickness for various recipes

4.10 Structure properties study

The previous sections provided a large amount of information on deposition conditions, microstructures and the resulting strain gradient in the film. This section reviews all the experiments and presents the correlation between the strain gradient and film microstructure as well as the effects of film deposition conditions on film microstructure [4.15].

4.10.1 Strain gradient and film microstructure

Grains within boron-doped poly-SiGe films generally have vertical orientation, with either conical or columnar shape. Films with low strain gradient are highlighted in light yellow in Table 4.2. A few TEM images are presented again in Figure 4.27 and Figure 4.28 for comparison. Films with a strain gradient larger than 4.5×10^{-4} μm^{-1} generally have conical grain structure with many twins and other defects (Figure 4.27); in contrast, films with positive strain gradient less than 1×10^{-5} μm^{-1} (Figure 4.28) generally have columnar grain structures with few defects within a single grain.
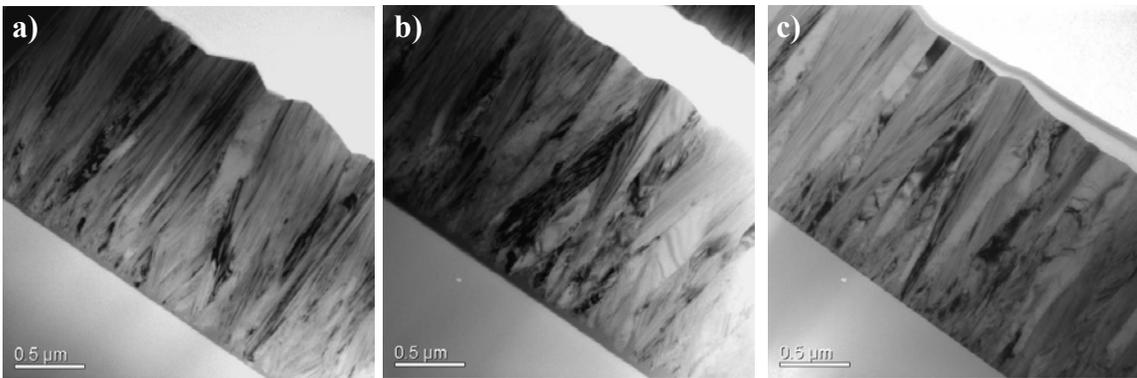
Figure 4.27 X-TEM images of as-deposited poly-SiGe films with strain gradient $>4.5 \times 10^{-4}$ μm^{-1}, deposited with: a) Recipe Ramp-ref; b) Recipe DOE2-5; c) Recipe SiGeSeed-1. (ref. Table 4.2.)
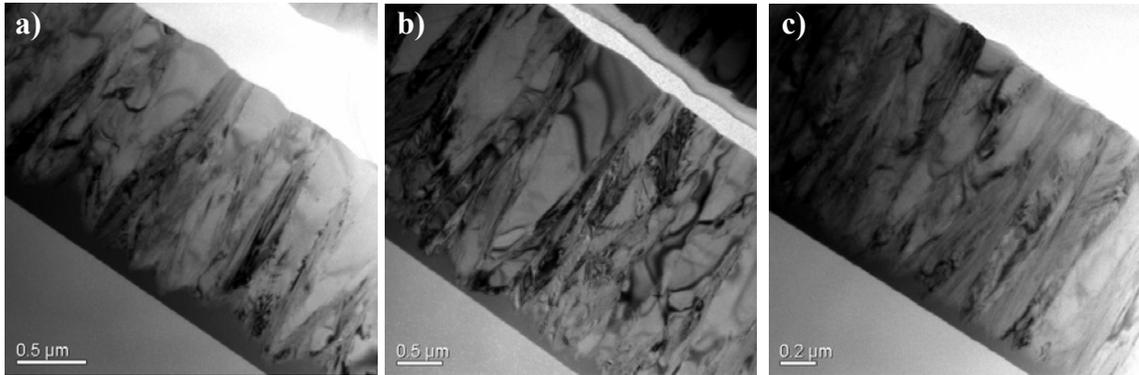
Figure 4.28 X-TEM images of as-deposited poly-SiGe films with positive strain gradient $<1\times10^{-5}$ μm^{-1}, deposited with: a) Recipe DOE2-2; b) Recipe Thick-depo; c) Recipe SiGeSeed-2. (ref. Table 4.2)

The strain gradient, which can also be interpreted as the stress-*vs.*-depth distribution, is strongly correlated with the film microstructure [4.16]. Films that have a large strain gradient usually start out with fine grains during the initial stage of deposition. As the deposition proceeds, these fine grains grow vertically and compete with each other for lateral growth. Defects are formed during the competition, and the surviving grains develop into conical structures. As a result, the compressive stress is larger in the lower portion of the deposited film as compared to the upper portion, as shown in Figure 4.29(a). This positive stress gradient causes the film to bend upward upon release.

Films that have a low strain gradient start out as an amorphous layer with sparse crystalline seeds. This structure results in large grain size because of the large spacing between the seeds (>100 nm spacing), which reduces lateral grain growth competition and hence results in fewer defects within the grains. To achieve the lowest strain gradient,

the spacing between seeds should match the final lateral grain size, in the range of 100

nm to 200 nm. In such a case, the residual stress remains approximately uniform

throughout the film thickness, as shown in Figure 4.29(b). The thin amorphous layer at

the bottom of the film has slightly higher compressive stress than the crystalline upper

portion of the film, which results in a small positive strain gradient. The stress of the thin

amorphous region is difficult to measure accurately for the stress-*vs.*-depth profile due to

the cumulative effect of etching non-uniformity, which leads to a significant uncertainty

in the residual thickness as it is thinned down to the amorphous region.
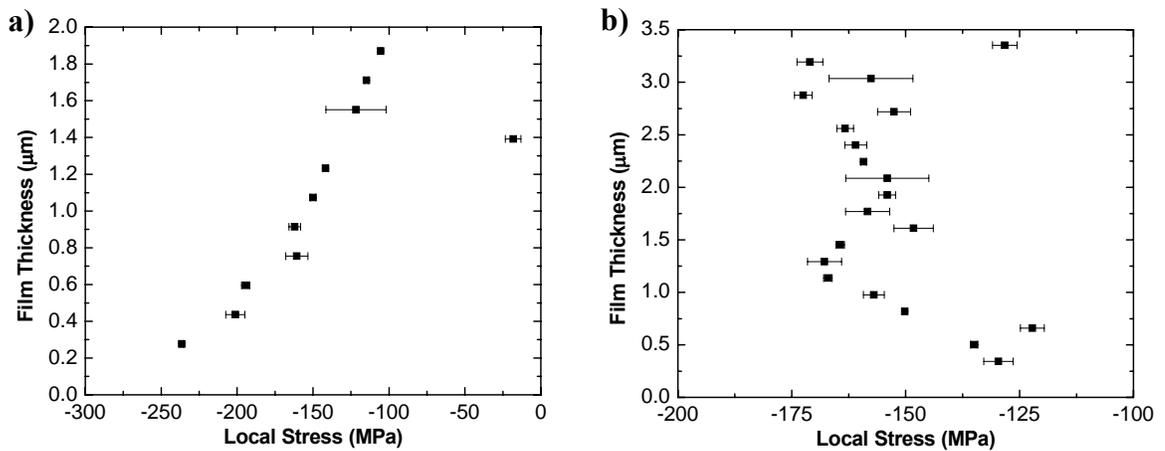
Figure 4.29 Stress-*vs.*-depth profiles: a) film with large strain gradient, shown in Figure
2.27(b); b) film with small strain gradient, shown in Figure 4.28(b)

To further understand grain growth competition, the grain orientations for all

samples shown in Figure 4.27 and 4.28 were analyzed with conventional XRD. The

measurement gives an average of crystal orientation of the entire film thickness. Data are

shown in Figure 4.30. Since the XRD equipment is not dedicated to thin film

measurement, the silicon substrate gives a strong <400> signal at 69º. It should be noted that the absence of dual peaks indicates that the SiGe alloy is homogeneous.

XRD
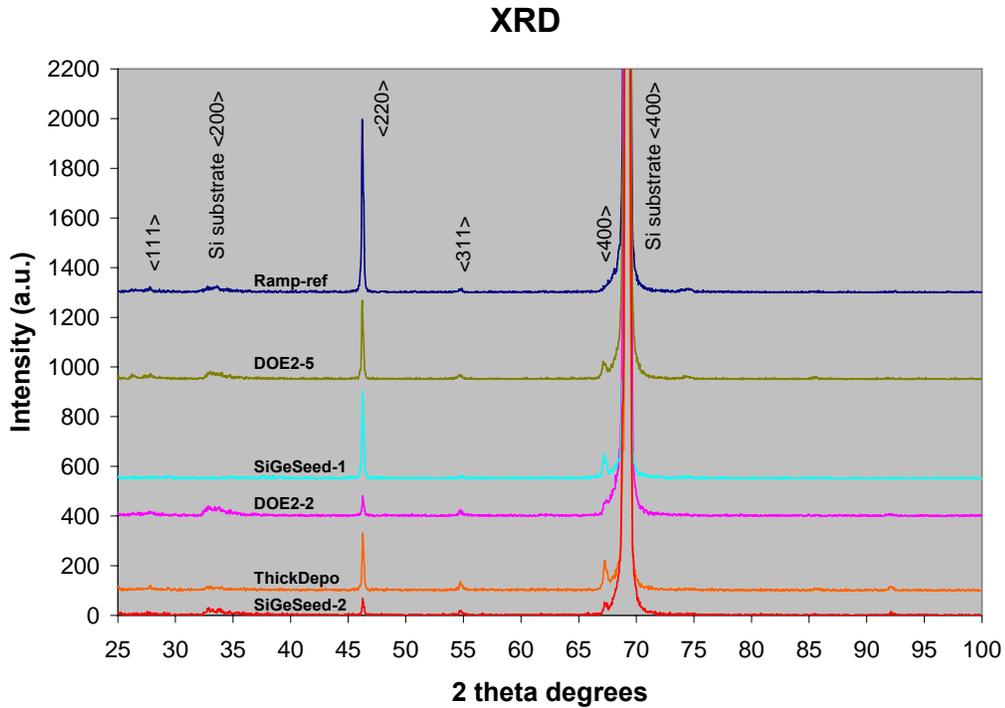
Figure 4.30 XRD data for films shown in Figure 4.27 and Figure 4.28

The big picture is that samples with large stress gradient have strong <220> orientation and samples with small stress gradient have less preference in grain orientations. Since the oxide substrate is amorphous, grains should start with random orientations. For the low strain gradient films without much grain growth competition, the final grain orientation is also random. For high strain gradient films with grain growth competition, orientation <220> is favored.

There is also a correlation between the pronounced <220> texture and grains with high density twinning defects in the high strain gradient films. The <220> direction is the only crystal direction which contain two {111} planes that are the twin-planes. In a

<220>-oriented grain there are two different potential twin planes parallel to the growth direction. This means that twins once formed stay in the grain: *i.e.* they extend in the newly formed part of the grain during growth. Also multiple twinning can take place.

4.10.2 Film Microstructure and Deposition Conditions

The film microstructure is determined by its deposition conditions. The effects of deposition temperature, deposition pressure, boron doping level, film thickness, seeding layer, and multiple-layered deposition are summarized in this section.

Deposition Temperature. Comparing the films shown in Figure 4.27(a) (Recipe Ramp-ref) and Figure 4.28(a) (Recipe DOE2-2), the only difference in processing condition is the deposition temperature. A higher deposition temperature results in a thinner amorphous region at the oxide interface and finer initial grains. Although the volume of the highly compressive amorphous region is suppressed, grain-size evolution during deposition is a more significant factor, resulting in a larger strain gradient.

Deposition Pressure. Deposition pressure is the only variable for the films shown in Figure 4.27(b) (Recipe DOE2-5) and Figure 4.28(a) (Recipe DOE2-2). In both cases, the films start out as an amorphous layer with sparse crystalline seeds. Since the deposition rate decreases as the process pressure goes down, adatoms have a better chance to form clusters and crystal seeds at low pressure. As a result, crystal seeds form earlier. For a fixed deposition temperature, a lower deposition rate also results in more lateral diffusion for the adatoms and hence more lateral grain growth. Thus, the grains are more conical in shape; thus films deposited at lower pressure have larger strain gradient.

Boron Doping Level. Experiments in DOE1 indicated that boron doping enhances crystallinity for films deposited near to the amorphous-to-crystalline transition temperature, i.e. if the initial amorphous region is minimal, the strain gradient can be reduced by increasing the boron doping concentration. Higher doping levels are explored in DOE2. Figure 4.31 shows the relationship between strain gradient and resistivity. X-TEM images are also shown for selected cases. The thickness of the amorphous region at the lower oxide interface remains approximately constant as the boron doping level exceeds a certain threshold, beyond which the strain gradient in the film is determined by other factors, such as grain size and defect density.

Film Thickness. The films shown in Figure 4.28(a) (Recipe DOE2-2) and Figure 4.28(b) (Recipe ThickDepo) have significantly different thicknesses. Since the boron concentration is not a significant factor at high doping levels, deposition time is the main difference between these two films. In both cases, the thickness of the amorphous region is similar, but the thicker film has taller columnar grains. Thus, a larger portion of the thicker film consists of columnar crystalline structures.
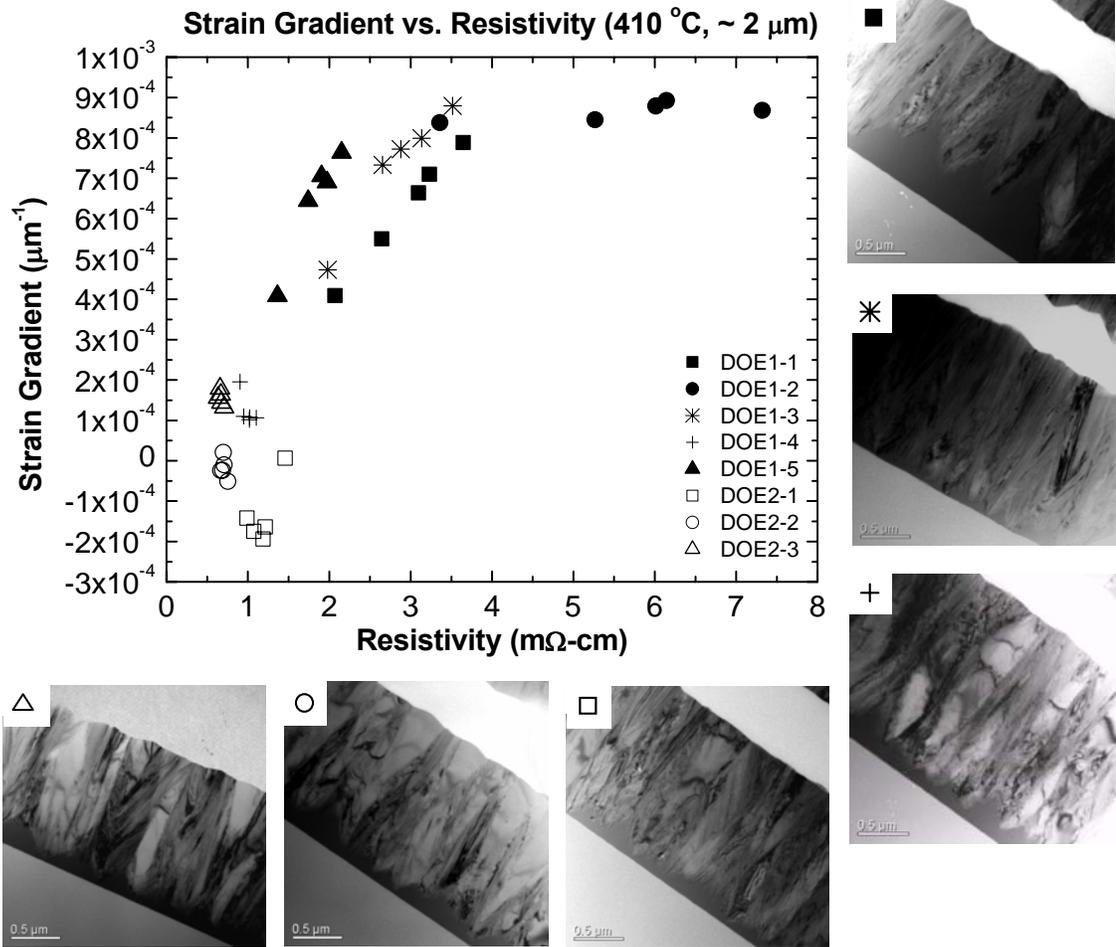
Figure 4.31 Relationship between strain gradient and resistivity, and film microstructure for films deposited near to the amorphous-to-polycrystalline transition temperature.

Seeding Layer. The films shown in Figure 4.27(c) (Recipe SiGeSeed-1), Figure 4.28(a) (Recipe DOE2-2) and Figure 4.28(c) (Recipe SiGeSeed-2) were deposited using similar main deposition conditions but different seeding layers. It should be noted that vacuum was not broken between the seeding and the main deposition steps, so that grain growth was not interrupted.

The low pressure and low gas flow rates used for SiGe seed-layer deposition in Recipe SiGeSeed-1 enhance crystal seeding due to the resultant low deposition rate and high boron concentration. Fine crystal grains formed during the initial stage of film deposition compete for lateral growth, resulting in a conical grain structure and high strain gradient as shown in Figure 4.27(c).

As discussed above, lower deposition pressure enhances initial crystal seeding as well as lateral grain growth. Recipe SiGeSeed-2 combines a low pressure deposited SiGe seed layer with a high pressure deposited main layer. The resulting film (Figure 4.28(c)) has a thinner amorphous layer (compared to the film shown in Figure 4.28(a) and columnar grains.

Multiple-layered film deposition. Section 4.8 described the generation of fine-grained poly-SiGe by multiple layer deposition. Grain growth can be interrupted by breaking the vacuum between depositions. The grain size and the stress distribution can be controlled by the number of deposition steps. Since the partially amorphous layer generated by Recipe DOE2-5 was use for each of the depositions, the final film ended up with a negative strain gradient due to *in-situ* annealing of the earlier deposited amorphous regions. A fully crystallized film such as the one generated by recipe SiGeSeed-1 has better thermal stability and therefore it should be used for each layer deposition to avoid having a negative strain gradient for the multiple-layered film deposition.

4.10.3 Uniformity

Since a LPCVD system is a batch reactor, cross-wafer and cross-load uniformities are important manufacturing considerations. The film thickness, resistivity, and average

residual stress are fairly uniform for all of the deposition recipes studied in this work. The strain gradient, however, is very sensitive to deposition process variations. Achieving low strain gradient with good uniformity is a major challenge for high volume manufacturing of poly-SiGe inertial sensors. Within the limitations of a horizontal LPCVD system in an academic laboratory, we are able to study the sensitivity of strain gradient to deposition process variations.

Figure 4.32 presents the stress-gradient variation data for all deposition runs yielding films with absolute strain gradient $\leq 1 \times 10^{-4}$ μm^{-1}. For each run, the strain gradient data was collected from four wafers across the load, and five locations on each wafer. For each location on a wafer, more than ten measurements of cantilever beam tip deflection were used to determine the strain gradient. The variation represents the range of these measurements for the same location.

At first glance, it would seem that larger variation is seen for negative strain gradient as compared with positive strain gradient. However, this is due to limitations in measurement accuracy rather than process uniformity issues, because there is not much room for the cantilever beams to bend downward so that only the very short beams could be measured. Also, tip deflection is difficult to measure for a curled-down beam.

Films with low strain gradient always have a thin amorphous region and large columnar grains. The amorphous region contributes a small positive strain gradient due to its higher compressive stress as compared to the crystalline region of the film. Although the amorphous region is necessary to ensure proper crystal seeding to form columnar grains, variations in the thickness of this region result in variations in strain gradient.

142
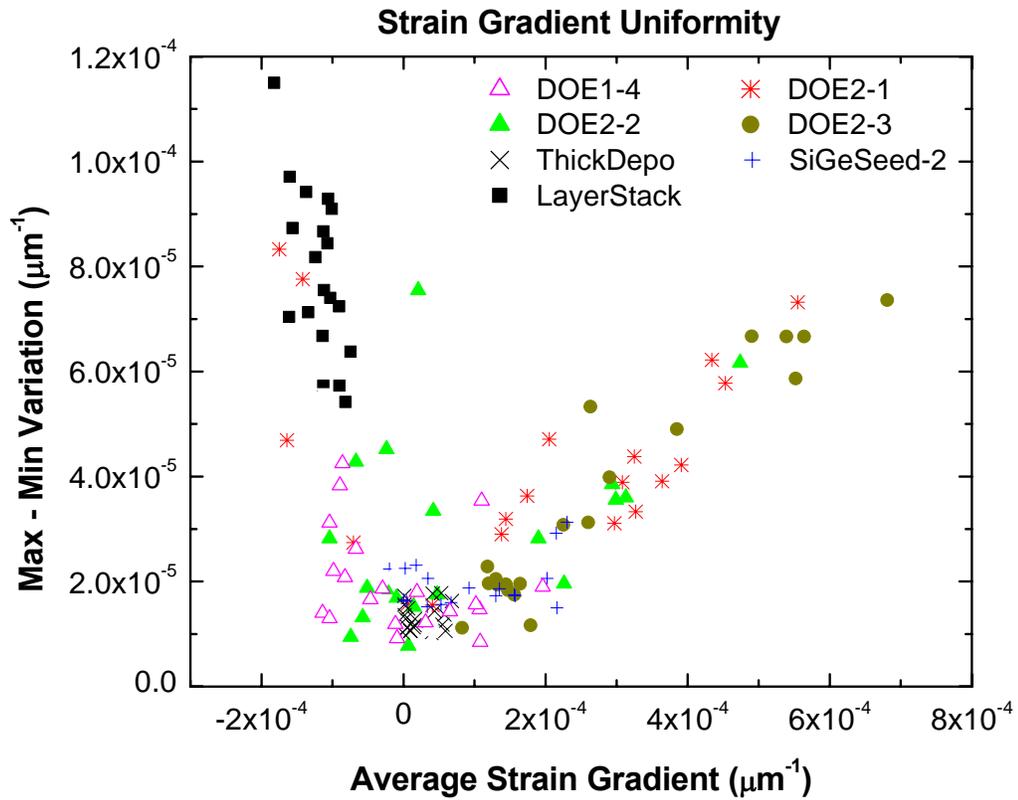
Figure 4.32 Variation in strain gradient vs. the average strain gradient.

A simple approach to minimize the effect of the lower amorphous region is to grow a thicker columnar crystalline layer. As shown in Figure 4.32, Recipe ThickDepo yields the best results for strain gradient and uniformity, due to its large film thickness. The average film thickness is 3.8 μm for Recipe ThickDepo whereas it is approximately 2 μm for the other recipes. Among the 2 μm deposition recipes, Recipe SiGeSeed-2 achieves the best strain gradient and uniformity. In this case, the additional low pressure deposited SiGe seed layer makes the initial amorphous layer thinner and more uniform, which significantly improves the uniformity of the strain gradient. Increasing the volume ratio of the crystalline region to the amorphous region is the key for improving stress-

gradient uniformity for single-layered columnar films. The combined use of the low pressure SiGe seed layer and a long deposition time should further improve the results.

Recipe LayerStack, the multiple-layer deposition process, was intended to yield a film with lower and more uniform strain gradient. Indeed, the strain gradient is improved as compared to Recipe DOE2-5. Unfortunately, the negative curvature results in a large measurement error, so that it is not possible to confirm that uniformity is improved.

4.11 Summary

The deposition of *in-situ*-boron-doped poly-SiGe films has been investigated with the design-of-experiments technique. At a CMOS compatible deposition temperature of 410°C, films with low resistivity and low wet-etch rate in heated H_2O_2 solution can be achieved with a reasonable deposition rate. The films with 60% germanium content generally have compressive residual stress so that careful design is required to prevent buckling of released clamped-clamped beams.

Strain gradients in LPCVD poly-SiGe films have been studied extensively using cantilever-beam tip deflection measurements, stress-*vs.*-depth profiling, and microstructure analysis using cross-sectional TEM. Films with strain gradients meeting the specification of 1×10^{-5} μm^{-1} for inertial sensor applications always have a thin initially deposited amorphous layer and thick columnar grains. The uniformity of strain gradient across a wafer and across a wafer load can be improved with a thinner amorphous region and thicker crystalline region. Alternately, uniformity can also be improved with a multiple-layered deposition process.

In our academic research laboratory, the as-deposited poly-SiGe films can achieve strain gradient below 7×10^{-5} μm^{-1} across a load of twenty-five 150mm-diameter wafers, with less than 1.6×10^{-5} μm^{-1} variation within a single wafer for certain slots within the load and a best case of only 1.1×10^{-6} μm^{-1}. This result is for ~3.8 μm-thick films deposited at 410°C for 8 hours, which meets the thermal process budget constraint imposed by CMOS electronics [4.17]. With tighter process control within a production environment, the strain gradient and its uniformity can be further improved.

References

[4.1] P. A. Krulevitch, "Micromechanical Investigations of Silicon and Ni-Ti-Cu Thin Films", Ph.D. Thesis, University of California, Berkeley (1994)

[4.2] W. L. Bragg, "The diffraction of short electromagnetic waves by a crystal", *Proceedings of the Cambridge Philosophical Society*, **17**, pp. 43-57 (1912)

[4.3] Dismukes, J.P., L. Ekstrom, E.F. Steigmeier, I. Kudman and D.S. Beers, "Thermal and Electrical Properties of Heavily Doped Ge-Si Alloys up to 1300°K", *J. Appl. Phys.* **35** ,1964, 2899. (b)

[4.4] K. Nunan, G. Ready, P. Garone, G. Sturdy and J. Sledziewski, "Developing a Manufacturable Process for the Deposition of Thick Polysilicon Films for Micro Machined Devices", *Proceedings of IEEE/SEMI Advanced Semiconductor Manufacturing Conference*, pp. 357-366 (2000)

[4.5] S. D. Senturia, *Microsystem Design*, Kluwer Academic Publishers, pp. 201-238 (2001)

[4.6] C. W. Low, T.-J. King Liu and R. T. Howe, "Characterization of polycrystalline silicon-germanium film deposition for modularly integrated MEMS applications," *IEEE/ASME Journal of Micro-electromechanical Systems*, vol. 16, no.1, pp.68-77, Feb. 2007

[4.7] B. L. Bircumshaw, M. L. Wasilik, E. B. Kim, Y. R. Su, H. Takeuchi, C. W. Low, A. P. Pisano, T.-J. King and R. T. Howe, "Hydrogen Peroxide Etching and Stability of P-type Poly-SiGe Films," *17th IEEE Micro Electro Mechanical Systems Conference (MEMS-04)*, Maastricht, The Netherlands, Jan. 25-29, 2004, pp. 514-519

[4.8] D. C. Montgomery, *Introduction to Statistical Quality Control*, 4th edition, John Wiley & Suns, Inc., pp. 571-672, 2001

[4.9] T. Van der Donck, J. Proost, C. Rusu, K. Baert, C. Van Hoof, J.-P Celis and A. Witvrouw, "Effect of deposition parameters on the stress gradient of CVD and PECVD poly-SiGe for MEMS applications," in *Proc. of the SPIE Conference*, San Jose, CA, USA, Jan. 28-29, 2004, pp. 8-18

[4.10] Molfese, A. Mehta and A. Witvrouw, "Determination of stress profile and optimization of stress gradient in PECVD poly-SiGe films," *Sensors and Actuators A*, vol. 118, pp. 313-321, 2005

[4.11] S. Sedky, A. Witvrouw, A. Saerens, P. V. Houtte, J. Poortmans and K. Baert, "Effect of *In Situ* Boron Doping on Properties of Silicon Germanium Films Deposited by Chemical Vapor Deposition at 400 °C", *Journal of Materials Research*, Vol. 16, No. 9, pp. 2607-2612 (2001)

[4.12] D. Mirfenderski, M. Ferrari and A. Der Kiureghian, **"**Analysis of microfabricated textured multicrystalline beams. I. Homogenization approach," in *Proc. Smart Materials Fabrication and Materials for Micro-Electro-Mechanical Systems, Mater. Res. Soc*, Pittsburgh, PA, 1992, pp. 91-96

[4.13] D. Mirfenderski, A. Der Kiureghian and M. Ferrari, **"**Analysis of microfabricated textured multicrystalline beams. II. Probabilistic approach," in *Proc. Smart Materials Fabrication and Materials for Micro-Electro-Mechanical Systems, Mater. Res. Soc*, Pittsburgh, PA, 1992, pp. 97-101

[4.14] S.-M. Sun, S. L. Girshick and M. R. Zachariah, "The role of total pressure in gas-phase nucleation: A diffusion effect," *Journal of Chemical Physics*, vol. 118, no. 2, pp. 736-745

[4.15] C. W. Low, T.-J. King Liu and R. T. Howe, "Study of poly-SiGe structural

properties for modularly integrated MEMS," in *Proc. Electrochem. Soc. SiGe and Ge: Materials, Process, Devices Symp.* Cancun, Mexico, Nov. 3, 2006

[4.16] C. V. Thompson, "Structure evolution during processing of polycrystalline films," *Journal of Annu. Rev. of Mater. Sci.,* vol. 30, pp. 159-190, 2000

[4.17] H. Takeuchi, A. Wu, X. Sun, R. T. Howe and T. –J. King, "Thermal budget limits of quarter-micron foundry CMOS for post-processing MEMS devices," *IEEE Trans. Electron Devices*, vol. 52, pp. 2081-2086, 2005

Chapter 5: Post-Deposition Processing of Poly-SiGe Films

Various post-deposition processes have been studied to look into the effects of post-processing on mechanical properties of poly-SiGe structural films and the performance of the underlying CMOS electronics. The goal of post processing is to improve the poly-SiGe film structural properties while keeping a low thermal budget for the CMOS electronics. Annealing is widely used in poly-Si and poly-SiGe structural films to lower the resistivity, stress, strain gradient and improve quality factor [5.1] – [5.4]. The changes in structural properties of poly-SiGe have been studied with a large variety of annealing methods, including furnace annealing, rapid thermal annealing, flash lamp annealing and excimer laser annealing. This chapter also discusses ion implantation as an alternative for modifying the mechanical properties of the poly-SiGe film without increasing the thermal budget. Finally, the results of an investigation into the CMOS thermal budget allowance will be presented.

5.1 Furnace annealing

As discussed in Chapter 4, LPCVD poly-SiGe films with low strain gradient always have a thin amorphous region at the lower oxide interface. Post-deposition annealing in a nitrogen ambient can be used to crystallize this amorphous region. An atmospheric pressure furnace was used to anneal a few unpatterned wafers from deposition ThickDepo (ref. Table 4.2). The nitrogen flow rate was set to 3000 sccm during the annealing to prevent oxidation. Annealing temperature and time are the variables in the recipe. In the annealing furnace, the temperature has to stabilize before

149

loading the wafers. It takes about 5 minutes to reach temperature set point again after loading. Once the annealing is done, the process temperature ramps down immediately and the furnace door opens. A minimum annealing time of 30 minutes was used to minimize the error in thermal budgets.

Various annealing times and temperatures were explored for this film, as listed in Table 5.1. In all cases, the annealing temperature was higher than the deposition temperature (410°C). We can see that there is no significant change in resistivity, but the average residual stress is reduced and the strain gradient becomes more negative. Annealing at 600°C is not compatible with advanced CMOS devices [5.5], but this high-temperature annealing magnifies the result for this study. The strain gradient of the as-deposited film is on the order of 1×10^{-5} μm^{-1}. Recipes FA-b and FA-c result in negative curvature of the released cantilever beams.

TABLE 5.1 Summary of post-deposition furnace annealing (FA).

Recipe	Temp. (°C)	Time (min.)	Resistivity (mΩ-cm)	Stress (MPa)	Strain Gradient (μm^{-1})
As-deposited	NA	NA	0.6	-150	4.7×10^{-5}
FA-a	430	30	0.6	-147	2.7×10^{-5}
FA-b	430	180	0.6	-144	-1.52×10^{-4}
FA-c	600	30	0.6	-115	-4.4×10^{-4}

The stress profile and cross-sectional TEM analyses of the as-deposited and annealed films are shown in Figure 5.1-4. Crystallization of the lower amorphous portion can be clearly seen for the 600°C-annealed film (Figure 5.4) as compared to the as-deposited film (Figure 5.1). In contrast, a change in the film microstructure is not readily apparent for the 430°C-annealed films. In all cases, no apparent changes are observed for the upper crystalline portion of the film.

Comparing the stress profile in Figure 5.4 against the one in Figure 5.1, the stress distribution within the upper crystalline portion remains the same, whereas the stress within the lower portion changes dramatically from compressive to tensile after the 600°C annealing, resulting in the large negative shift in the strain gradient. This is consistent with the cross-sectional TEM analyses of the film microstructure. The amorphous region of the as-deposited film is not densely packed. Upon high-temperature annealing, voids and defects are removed, resulting in tensile stress in this region. The crystallization of the amorphous region makes the average stress less compressive and changes the strain gradient towards the negative direction. The resistivity of the film does not change significantly. The boron concentration in the film is about 1×10^{21} cm^{-3}. Probably there is no additional dopant activated by the annealing. Also the amorphous region in the as-deposited film is relatively thin compared to the film thickness and it does not reduce the overall resistivity significantly after crystallization.

The stress distribution does not show a significant change after 30 minutes of annealing at 430°C (FA-a) compared to the as-deposited film, consistent with the cross-sectional TEM analyses of the film microstructure. The reduced variability in the stress distribution as compared to that of the unannealed film can be attributed to differences in measurement accuracy and position of the wafer within the furnace. Although not clearly visible, a small part of amorphous region got crystallized by the low temperature annealing, and this contributes to a small change in strain gradient. With longer annealing time, the change is more significant.
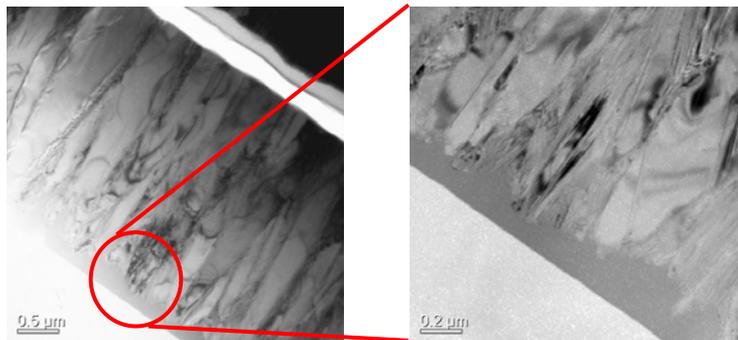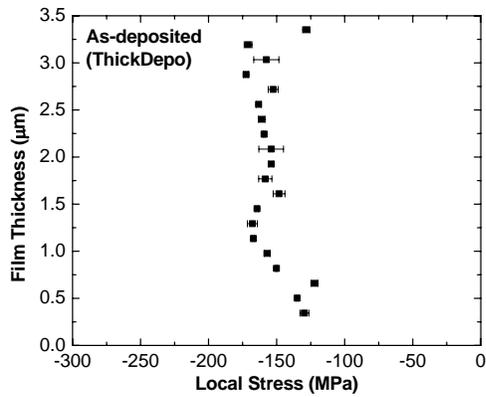
Figure 5.1 Stress profile and cross-sectional TEM image for the as-deposited film (film deposited by Recipe ThickDepo)
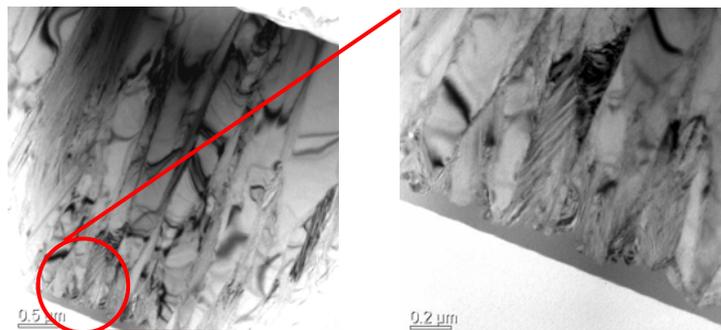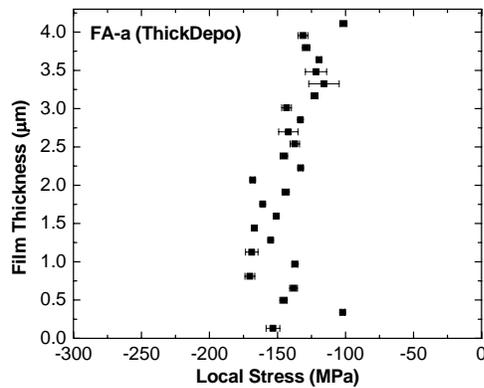
Figure 5.2 Stress profile and cross-sectional TEM image for FA-a (film deposited by Recipe ThickDepo)

152
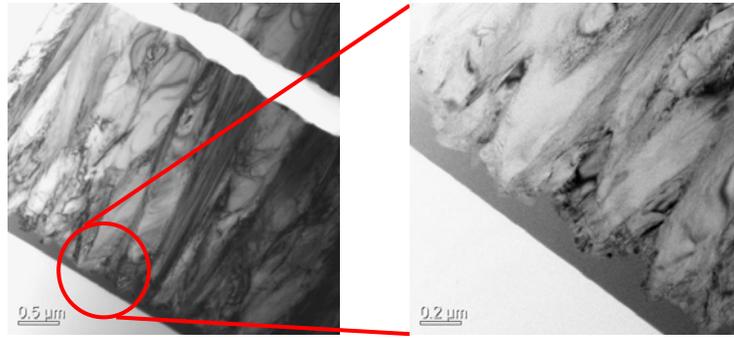
**Figure 5.3** Cross-sectional TEM image for FA-b (film deposited by Recipe ThickDepo)
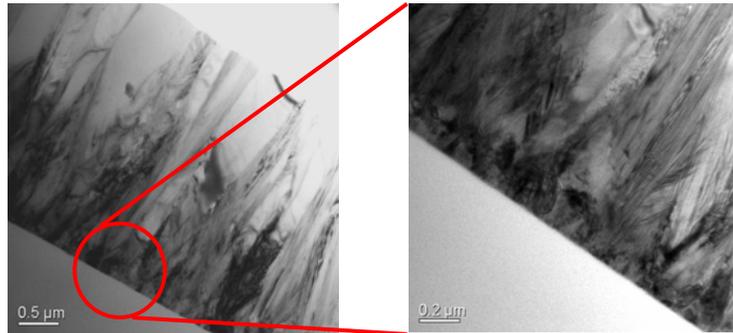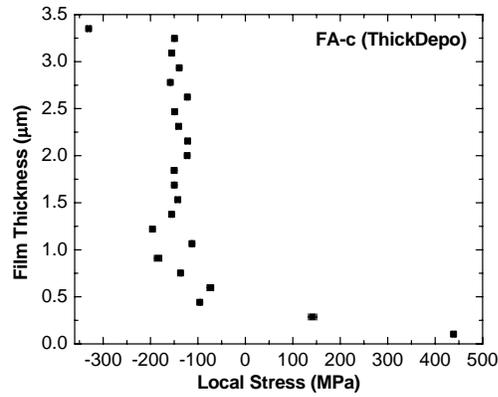

Figure 5.4 Stress profile and cross-sectional TEM image for FA-c (film deposited by Recipe ThickDepo)

Furnace annealing at elevated temperature (higher than the deposition temperature) causes the amorphous layer to crystallize and thereby changes the strain

153

gradient in the negative direction. Thus, caution is advised when post-processing poly-SiGe films at temperatures higher than the deposition temperature for a long period of time.

5.2 Rapid thermal annealing

The rapid thermal annealing (RTA) tool is a single wafer system using lamp illumination for rapidly heating the wafer. The temperature of the wafer can be ramped up in a few seconds. The process temperature of the RTA tool is controlled by a thermocouple with a feedback system. With appropriate setting, the wafer temperature can stabilize to the set point in a few seconds. A water cooling system is attached to the process chamber so that the wafer temperature can be dropped down by hundreds of degree Celsius in a few seconds after heating.

Wafers from Recipe ThickDepo (ref. Table 4.2) were also used for the RTA study. The annealing temperatures were chosen to be 410°C, 430°C, 470°C, 510°C and 550°C. The recipe was adjusted so that the temperature does not overshoot, but it takes 10 seconds to reach the set point. One minute of annealing time was used in each recipe. Nitrogen flow was used during the annealing so that the film does not oxidize.

The results of the annealing are listed in Table 5.2 for comparison. There is no significant change in resistivity, but the changes in stress and strain gradient are dramatic. It is interesting that the results of all RTA runs are very similar to those of the furnace annealing at 600°C for 30 minutes (listed in Table 5.2). The film was deposited at 410°C for hours and annealing at 410°C for 1 minute should not change the properties of the

film. This observation suggested that the temperature the film experienced during the RTA section might be a lot higher than the set point.

TABLE 5.2 Summary of post-deposition rapid thermal annealing (RTA).

Recipe	Temp. (°C)	Time (min.)	Resistivity (mΩ-cm)	Stress (MPa)	Strain Gradient (μm^{-1})
As-deposited	NA	NA	0.6	-150	4.7×10^{-5}
RTA-a	410	1	0.6	-112	-4.6×10^{-4}
RTA-b	430	1	0.6	-110	-3.3×10^{-4}
RTA-c	470	1	0.5	-118	-3.5×10^{-4}
RTA-d	510	1	0.5	-102	-3.6×10^{-4}
RTA-e	550	1	0.6	-115	-4.4×10^{-4}

Stress profile and TEM analysis for the as-deposited film, recipe RTA-b and RTA-e are shown in Figure 5.1, Figure 5.5 and Figure 5.6, respectively. Both of the RTA conditions fully crystallized the bottom amorphous regions of the films and the stress of those regions become less compressive. The stress profile and the microstructures of the RTAed films are very similar to those of the 600°C furnace annealed film (shown in Figure 5.4). Comparing RTA and furnace annealing at 430°C, furnace annealing for longer time does not create visible change in the amorphous region, as shown in Figures 5.2 and 5.3 in the previous section. This observation suggests that there is a temperature discrepancy between the RTA chamber and the annealing furnace. The annealing furnace has similar a temperature control as the poly-SiGe deposition furnace; therefore, the annealing temperature and the deposition temperature should be consistent.

The results of this RTA experiments are not very helpful for understanding how a reduced thermal budget would change the properties of the film, because of the uncertainty in annealing temperatures. It is confirmed again that crystallization of the initially amorphous region creates a dramatic change in the residual stress and this affects the strain gradient of the entire film significantly.

155
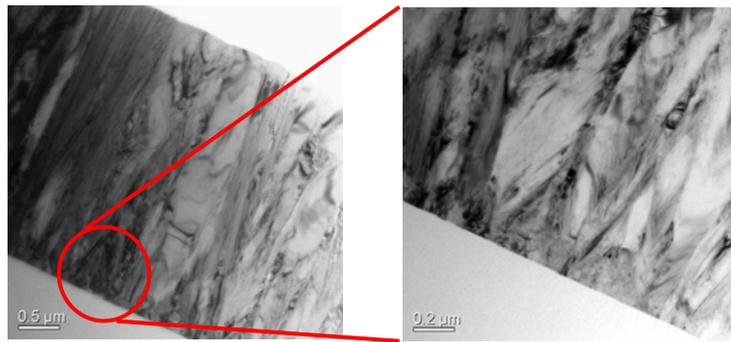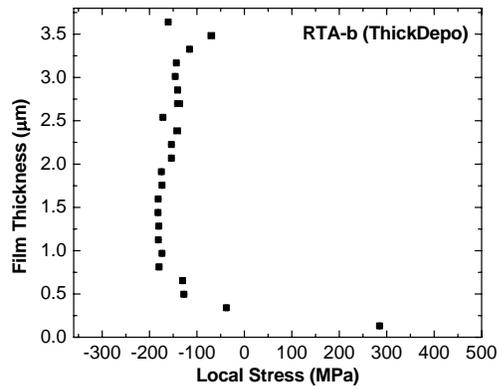
Figure 5.5 Stress profile and cross-sectional TEM image for RTA-b (film deposited by Recipe ThickDepo)
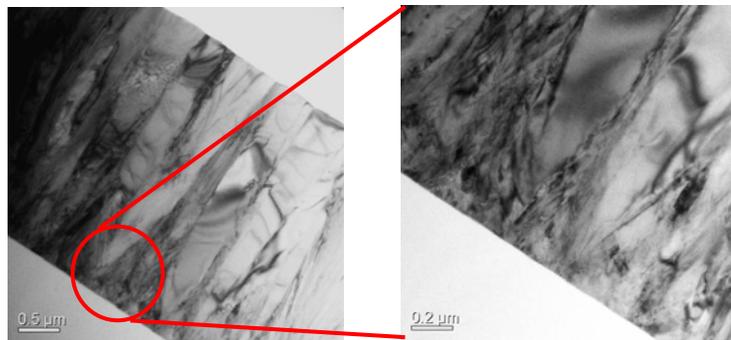

Figure 5.6 Cross-sectional TEM image for RTA-e (film deposited by Recipe ThickDepo)

5.3 Flash lamp annealing

Flash lamp annealing (FLA) is an advanced rapid thermal annealing process that allows the wafer to be held at process temperature for just a few milliseconds and then to be cooled down rapidly. Flash lamp annealing has been investigated in the IC industry for ultra-shallow junction formation [5.6] - [5.8]. The effect of flash lamp annealing on poly-SiGe film was studied with the support of Mattson Technology, the manufacturer of the FLA tool.

Flash lamp annealing of a few SiGe samples was done in a demo tool at Mattson Technology. The temperature distribution across the substrate is close to a Gaussian distribution over the sample area, but the sample holder makes the edge of the sample slightly hotter. In this experiment, the samples were preheated to an intermediate temperature of 220°C. A capacitor bank is discharged through the heating lamp to achieve the additional temperature jump on the top side of the sample. The duration of the annealing is on the order of 1 ms. The temperature jump could be measured by the additional increase in backside temperature. The backside temperature can be measured by a radiometer if it is above 760°C. The annealing temperatures for the poly-SiGe films were chosen to be at 500°C, 600°C and 700°C. In this case, the temperature cannot be measured by the radiometer, so is roughly predicted by the lamp power.

Poly-SiGe films from depositions DOE1-3 and DOE1-13 (ref. Table 4.2) were used in this experiment. The two depositions recipes have different process temperature but identical process pressure and gas flow rates. The results of the annealing are listed in Tables 5.3a and 5.3b. The sample size for the demo tool is restricted to 12 mm by 12 mm. After the annealing, the lithography, etch and release steps were done at die level. In this

case, the average residual stress cannot be measured with the change in wafer curvature. The strain gauge test structure defined by the mask did not survive after the release. Therefore the average residual stress is not available for these annealed films. We can see that for both DOE1-3 and DOE1-13 films, flash lamp annealing does not change the resistivity significantly. Also, only the 700°C annealing changes the strain gradient towards the negative direction.

TABLE 5.3a Summary of post-deposition flash lamp annealing (FLA) for deposition DOE1-3.

Recipe	Temp. (°C)	Time (ms.)	Resistivity (mΩ-cm)	Stress (MPa)	Strain Gradient (μm^{-1})
As-deposited	NA	NA	2.6	-168	5.8×10^{-4}
FLA-a	500	1	2.8	NA	5.9×10^{-4}
FLA-b	600	1	2.3	NA	5.8×10^{-4}
FLA-c	700	1	2.3	NA	-2.92×10^{-5}

TABLE 5.3b Summary of post-deposition flash lamp annealing (FLA) for deposition DOE1-13.

Recipe	Temp. (°C)	Time (ms.)	Resistivity (mΩ-cm)	Stress (MPa)	Strain Gradient (μm^{-1})
As-deposited	NA	NA	6.1	-100	3.8×10^{-4}
FLA-d	500	1	6.8	NA	3.8×10^{-4}
FLA-e	600	1	5.4	NA	3.2×10^{-4}
FLA-f	700	1	5.0	NA	5.8×10^{-5}

The textures of the films before and after annealing are compared by TEM analysis. For reference, the stress profiles and the TEM images for the as-deposited films are shown in Figures 5.7 and 5.10. Both DOE1-3 and DOE1-13 depositions result in conical textures with amorphous starting layers and the compressive stress gradually decreases along the film thickness. Since DOE1-13 is a higher temperature deposition, the amorphous region is thinner.

Comparing Figure 5.7 with Figure 5.8, we can see that flash lamp annealing at 600°C of the DOE1-3 film does not change the microstructure so that the strain gradient of the film remains the same. Figure 5.9 shows the film after 700°C FLA and the bottom amorphous region is completely crystallized. Similar to furnace annealing and RTA

discussed in previous sections, this crystallization of the bottom amorphous layer changes the local stress from compressive to tensile. A negative bending moment is generated at the bottom of the film by the crystallization and the strain gradient changes towards the negative direction. Figure 5.11 shows similar results with the DOE1-13 film annealed at 700°C. Since the as-deposited amorphous region for deposition DOE1-13 is thinner, the negative bending moment generated by the crystallization is also smaller. As a result, the change in strain gradient after the annealing is less significant compared to the DOE1-3 film.

It should be noted that the crystallized region at the bottom of the FLA-ed film looks different from the furnace annealed film (Figure 5.4) or RTA-ed film (Figure 5.6). The FLA results in fine grained polycrystalline structures that are distinguishable from the as-deposited crystalline structures, whereas the furnace annealing or the RTA "extends" the grain growth from the original crystalline structures. In all cases, the surface roughness remains the same after the annealing. This indicates that the flash lamp annealing does not involve melting or re-solidification.

The flash lamp annealing changes the stress and the strain gradient in a similar fashion as the furnace annealing and the rapid thermal annealing, by crystallizing the amorphous region at the bottom of the SiGe film. Since the duration of the heat pulse is in the millisecond range, crystallization does not occur for temperatures below 700°C. The effects of the flash lamp annealing on the underlying CMOS electronics will be studied in a later section.
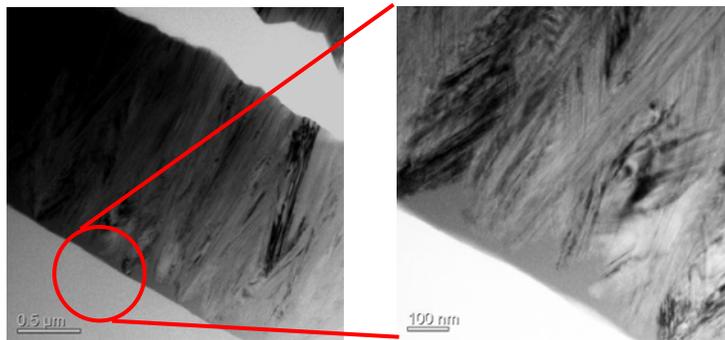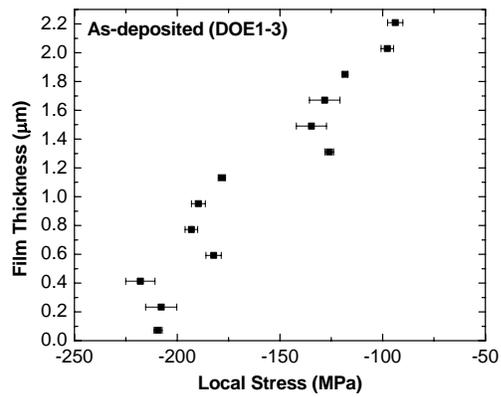
Figure 5.7 Stress profile and cross-sectional TEM image for the as-deposited film (film deposited by Recipe DOE1-3)
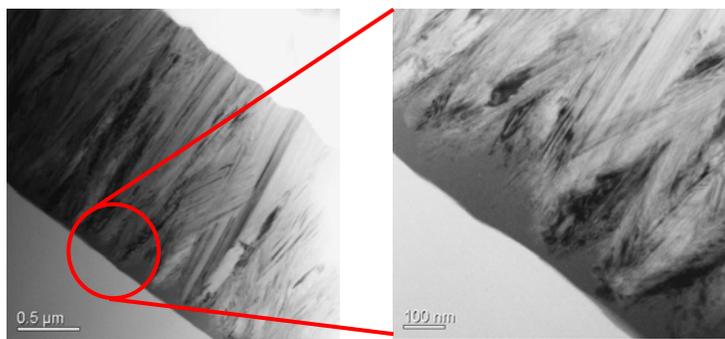

Figure 5.8 Cross-sectional TEM image for FLA-b (film deposited by Recipe DOE1-3)
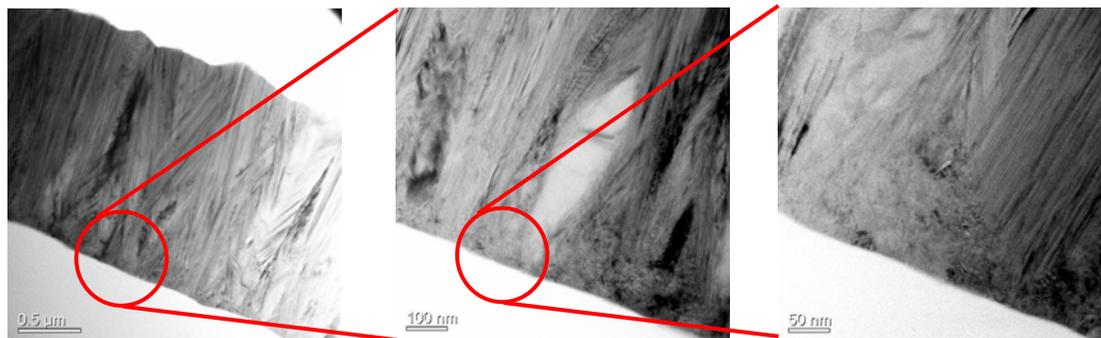
**Figure 5.9** Cross-sectional TEM image for FLA-c (film deposited by Recipe DOE1-3)
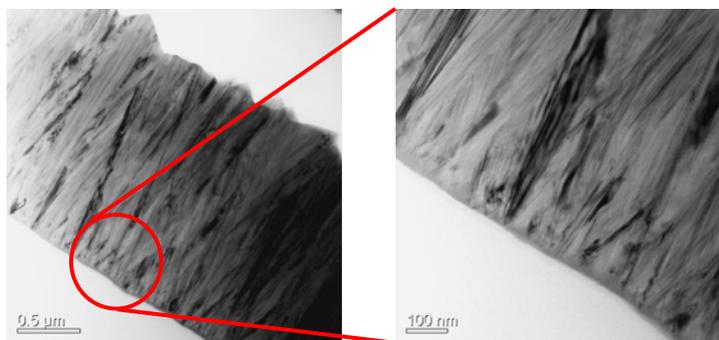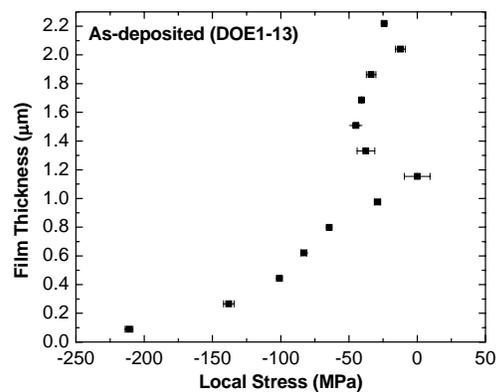
Figure 5.10 Stress profile and cross-sectional TEM image for the as-deposited film (film deposited by Recipe DOE1-13)
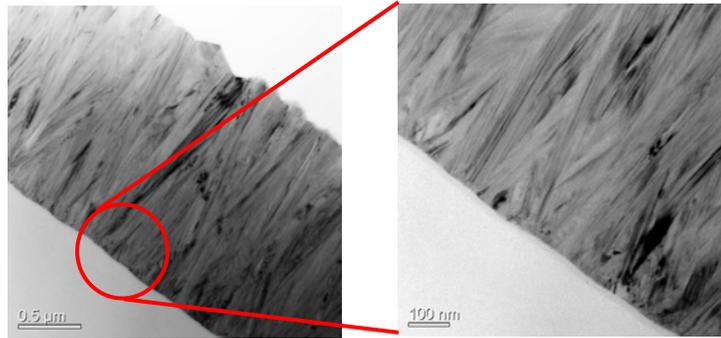
Figure 5.11 Cross-sectional TEM image for FLA-f (film deposited by Recipe DOE1-13)

5.4 Excimer laser annealing

The excimer lasers are commonly used in material processing research due to its high energy density. They can be used as energy sources for surface annealing or material ablation. Excimer laser annealing (ELA) of poly-SiGe had been previously studied by other researchers [5.9] – [5.11] and the author [5.12]. Pulsed-laser annealing can be used to tune the stress and the strain gradient of the poly-SiGe film. The laser energy locally heats up the SiGe film, and therefore the thermal budget seen by the underlying CMOS is not increased. This section briefly reviews the work done by the author for a Master's project [5.12].

Excimer is short for "excited dimmer". The laser used in the experiments has KrF as the gaseous lasing media, excited by means of electrical discharge. The diatomic molecule KrF has very short lifetime and dissociates to release the energy through ultraviolet photons at the wavelength of 248 nm. The laser excitation is pulsed with a duration time of 38 ns. The laser beam output has a fluence range of 200 – 800 mJ/cm^2.

This fluence level allows the radiation to penetrate the top most portion of the poly-SiGe film, down to a depth of ~0.3 μm.

The laser energy melts the top region of the film, and the melted region re-solidifies upon cooling. This melting and re-solidification process densifies the affected region and results in tensile stress locally. Most of the single layer as-deposited poly-SiGe films have compressive stress with a positive stress profile (positive strain gradient). After the excimer laser annealing, the strain gradient of the film will be worsened by the tensile stress on the top region of the film. However, if excimer laser annealing is applied to a film with negative strain gradient, the strain gradient of the annealed film will change in the positive direction toward zero.

The results of the excimer annealing study are summarized in Table 5.4. A bi-layer deposition recipe was chosen to generate a film with negative strain gradient with the stress tuning technique [5.13]. The bottom layer was deposited at higher temperature with higher germanium content compared to the top layer. After the ELA, the resistivity of the film remains the same, whereas the residual stress and the strain gradient change in the positive direction. The texture of the as-deposited and ELA-ed films can be compared with the cross-sectional TEM images shown in Figures 5.12 and 5.13.

TABLE 5.4 Summary of post-deposition excimer laser annealing (ELA).

Recipe	Energy (mJ/cm^2)	Resistivity (mΩ-cm)	Stress (MPa)	Strain Gradient (μm^{-1})
As-deposited	NA	1.4	-7	-1.23×10^{-4}
ELA-a	200	1.4	7	-2.05×10^{-5}
ELA-b	400	1.4	40	5.90×10^{-4}
ELA-c	600	1.4	72	1.07×10^{-3}

The melted depth is about 250 nm for 600 mJ/cm^2 fluence. The tensile stress of this melted thin region is a few hundreds of MPa and creates a large positive bending

moment, therefore the strain gradient changes significantly after annealing. Since the melting and re-solidification involves reflow, the surface roughness of the film decreases after the annealing.
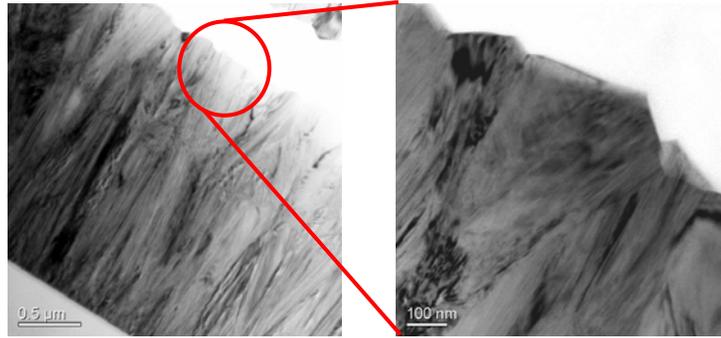

Figure 5.12 Cross-sectional TEM image for the as-deposited film in the ELA experiment
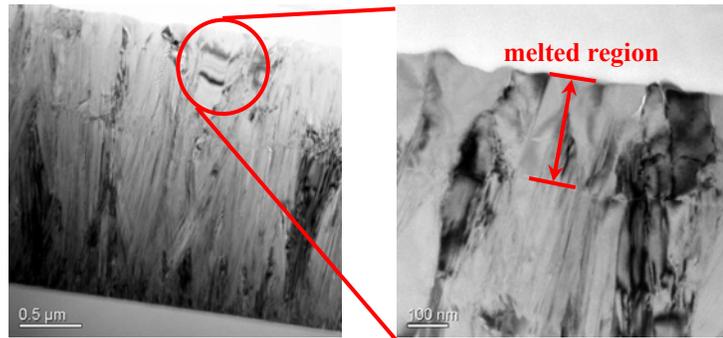
Figure 5.13 Cross-sectional TEM image for ELA-c

Excimer laser annealing has the lowest thermal budget of all the annealing method discussed above, because the heating is limited to the top region of the poly-SiGe film and the underlying CMOS won't be affected. However, the mechanical properties of the poly-SiGe film are very sensitive to the laser energy and achieving low strain gradient with good uniformity is very difficult.

5.5 Argon implantation

Ion implantation and machining have each been used to modify the stress in thin films [5.14], [5.15]. Argon implantation is a low-cost and high-throughput process that is readily available in the IC industry. Therefore, the effect of argon implantation (AI) on the strain gradient was studied in this work, for different doses and acceleration energies. Multiple-layered poly-SiGe films deposited by Recipe LayerStack (ref. Table 4.2) were used in this study. The results are summarized in Table 5.5.

TABLE 5.5 Summary of post-deposition argon implantation (AI) for deposition LayerStack.

Recipe	Energy (keV)	Dose (cm^{-2})	Resistivity (mΩ-cm)	Stress (MPa)	Strain Gradient (μm^{-1})
As-deposited	NA	NA	3.1	-229	-1.17×10^{-4}
AI-a	30	1×10^{14}	4.7	-201	-4.97×10^{-5}
AI-b	65	1×10^{14}	5.9	-198	1.25×10^{-5}
AI-c	100	1×10^{14}	5.7	-189	1.32×10^{-4}
AI-d	100	1×10^{13}	3.4	-207	-1.97×10^{-4}
AI-e	100	1×10^{12}	3.1	-203	-1.44×10^{-4}
AI-f	180	1×10^{16}	1.9	-162	6.25×10^{-4}

As an extreme case, Figures 5.14 and 5.15 show the stress profiles and cross-sectional-TEM for the as-deposited film and a film implanted with 1×10^{16} cm^{-2} Ar$^+$ at 180 keV. The implantation amorphizes the top portion (~ 0.3 μm) via damage to the crystalline structure, and thereby relieves the compressive stress within this portion of the film. The implant also causes a small drift of the stress in the position direction in the middle region of the film compared to Figure 5b. Though not apparent from the X-TEM image, some argon ions penetrate the film beyond the amorphized region, which may possibly account for the small amount of stress relaxation in the middle region of the film. Overall, the stress profile after argon implantation has a positive slope (increasing from the bottom of the film to the top of the film) and results in a strain gradient of 6.25×10^{-4} μm^{-1}, whereas the as-deposited film has a strain gradient of -1.2×10^{-4} μm^{-1}.
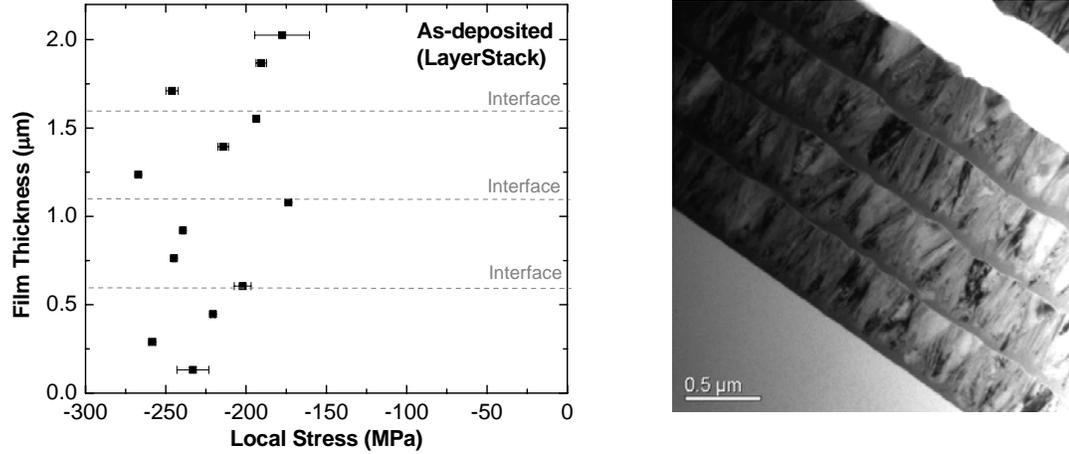
Figure 5.14 Stress profile and cross-sectional TEM image for the as-deposited film (film deposited by Recipe LayerStack)
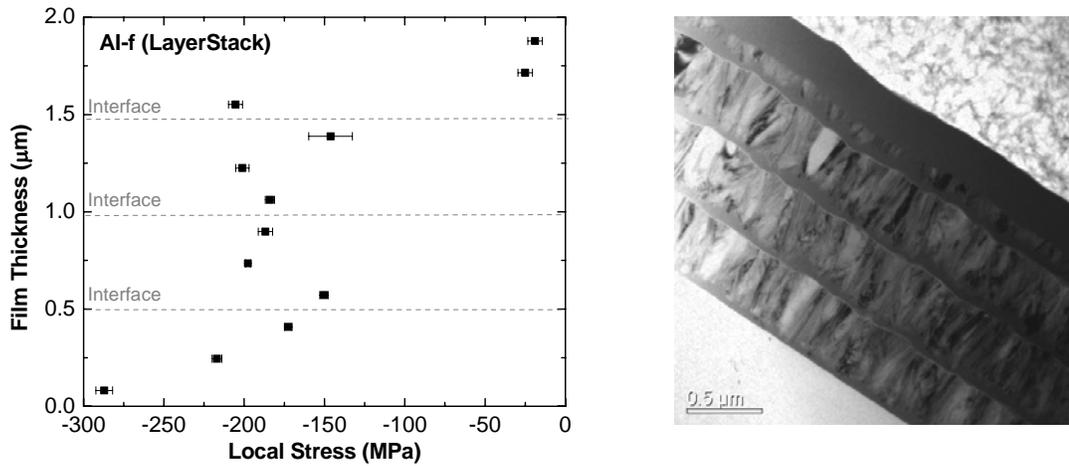

Figure 5.15 Stress profile and cross-sectional TEM image for AI-f (film deposited by Recipe LayerStack)

The amorphization thickness and the stress distribution within the film can be modified with the implant dose and acceleration energy. Thus the strain gradient can be tuned by ion implantation. Figure 5.16 shows the relationship between the strain gradient and the argon implantation conditions. X-TEM images are inserted next to most of the data points, and clearly show the upper amorphized region created by the implantation. For a given dose, higher acceleration energy results in thicker amorphized region. Amorphization does not occur for dose $\leq 1 \times 10^{13}$ cm^{-2} at 100 keV. The lowest strain gradient is achieved with 1×10^{14} cm^{-2} dose and 65 keV acceleration energy.
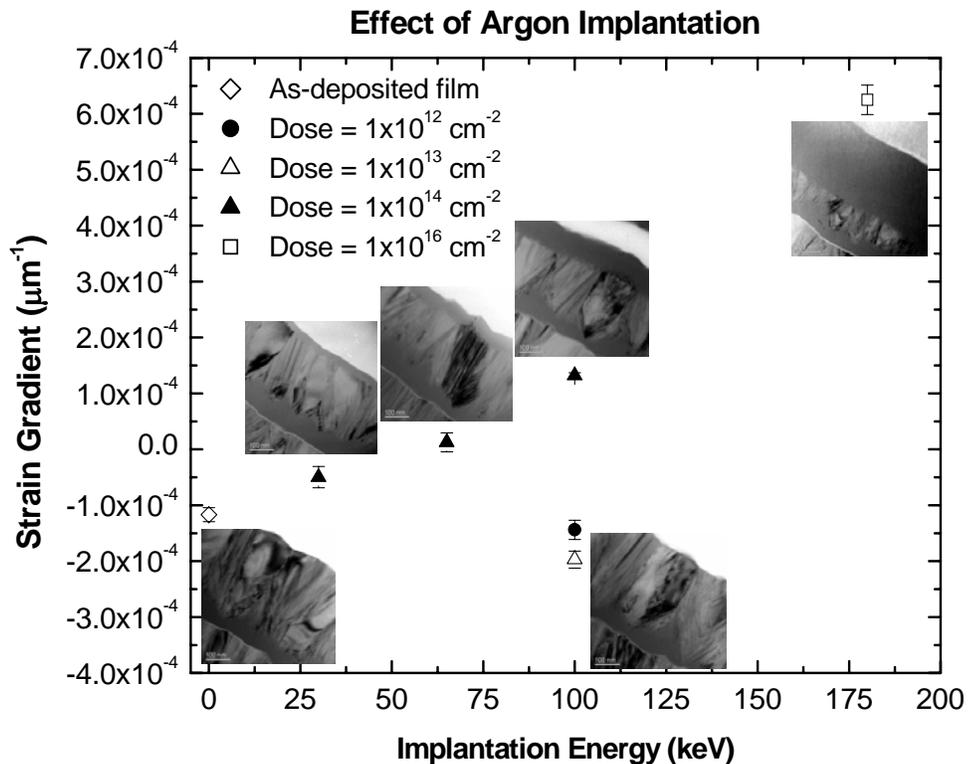

Figure 5.16 Correlation of strain gradient with post-deposition argon implantation conditions.

It should be noted that the implanted film remains electrically conductive (resistivity < 10 mΩ-cm) even though the upper portion is amorphous. However, the resistivity is lower for the case with maximum dose and energy and this is not well-understood. No increase in wet-etch rate in heated H_2O_2 solution is seen for the implanted film.

Among all the post-processing methods discussed above, ion implantation is the lowest thermal budget method. It also has the advantage being a standard low-cost and high throughput process in IC manufacturing and has well controlled process uniformity and repeatability.

5.6 CMOS thermal budget limitations

Integration of SiGe MEMS on CMOS had been demonstrated before. Franke *et al.* first demonstrated SiGe MEMS resonators on 3 μm gate-length CMOS circuitry made in the UC Berkeley Microfabrication Laboratory [5.3]. Witvrouw *et al.* demonstrated a SiGe MEMS gyroscope over 0.35 μm foundry CMOS circuitry [5.16]. Takeuchi *et al.* studied the thermal budget limits of 0.25 μm foundry CMOS circuitry by rapid thermal annealing and furnace annealing [5.5]. In this work, 0.13 μm and 0.25 μm foundry CMOS are studied with the thermal budget generated by the actual MEMS film depositions.

5.6.1 Processing of the MEMS layers

Since there were a limited amount of 0.13 μm and 0.25 μm foundry CMOS chips provided by collaborators, the CMOS thermal budget test were done at the die level. As

shown in Figure 5.17, a thin layer of pure germanium was first deposited as the passivation for the sacrificial release. Then 2 μm of oxide was deposited as the sacrificial layer. Lastly, 2 μm of SiGe structure layer was deposited. The thermal history the CMOS chips experience for the depositions includes:

Ge deposition – 3 hr 30 min. at 350°C

Oxide deposition – 4 hr 30 min. at 400°C

SiGe deposition – 5 hr 45 min. at 410°C

There is temperature fluctuation of ± 20°C during stabilization for each deposition. The CMOS chips were split into three groups. Some had no further thermal processing, some were flashed lamp annealed at 700°C for 1 ms and some were rapid thermal annealed at 430 °C for 1 minute.
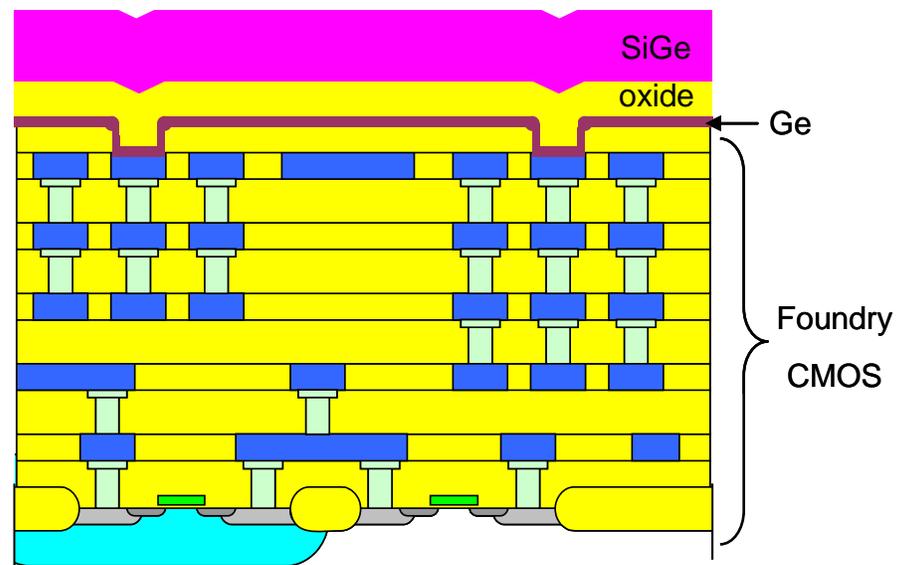

Figure 5.17 Schematic of MEMS layers processing on foundry CMOS

In order to do electrical measurement of CMOS after the MEMS processing, the MEMS films need to be stripped off to re-expose the metal bond pads for probing. The SiGe film was stripped off using a reactive ion etch at 60°C. The oxide sacrificial layer was removed in HF solution (50 ml 49% HF + 200 ml DI water) at room temperature for 10 minutes. The germanium layer was removed in 30% H_2O_2 solution at 80°C for 5 minutes.

5.6.2 Metal contact damage

During the film removal steps, metal contact damage is observed. Figure 5.18 shows the conditions of the metal contact along the film removal steps. Since pre-measurement was done on the CMOS electronics, there are probe marks left on the metal contacts. The residue around the bond pads become visible after the removal of the SiGe film. By the end of the film removal processes, the residual is clearer and there is almost no metal left on the contact.
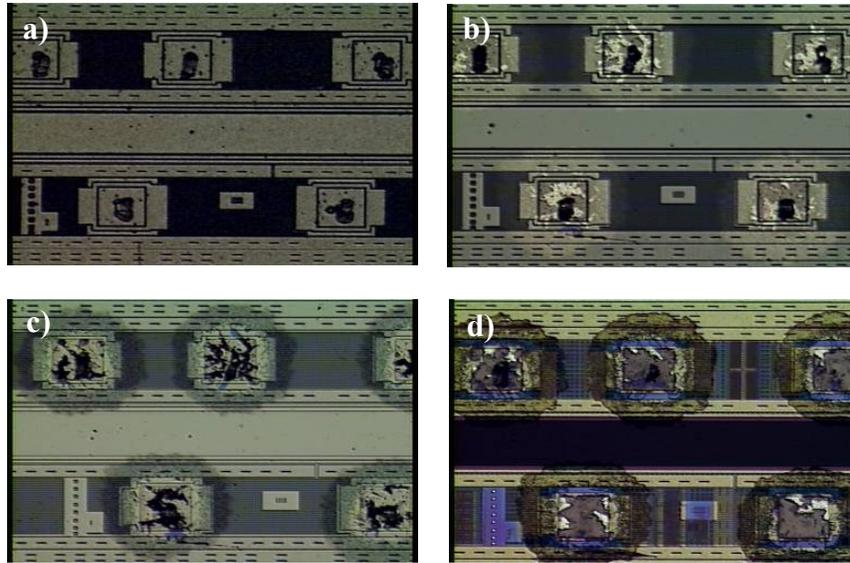
Figure 5.18 CMOS metal contacts: a) after all depositions; b) after SiGe film removal; c) after oxide film removal; d) after Ge film removal

The chips with no further thermal processing after the depositions and the ones with furnace annealing or flash-lamp annealing have similar metal contact damage. The chips that went through the rapid thermal annealing have more damage, as shown in Figure 5.19. Film delamination happened right after the RTA step. The MEMS layers and the CMOS passivation peeled off and the metal contacts were exposed. The metal contacts were completely damaged during the film removal processes.
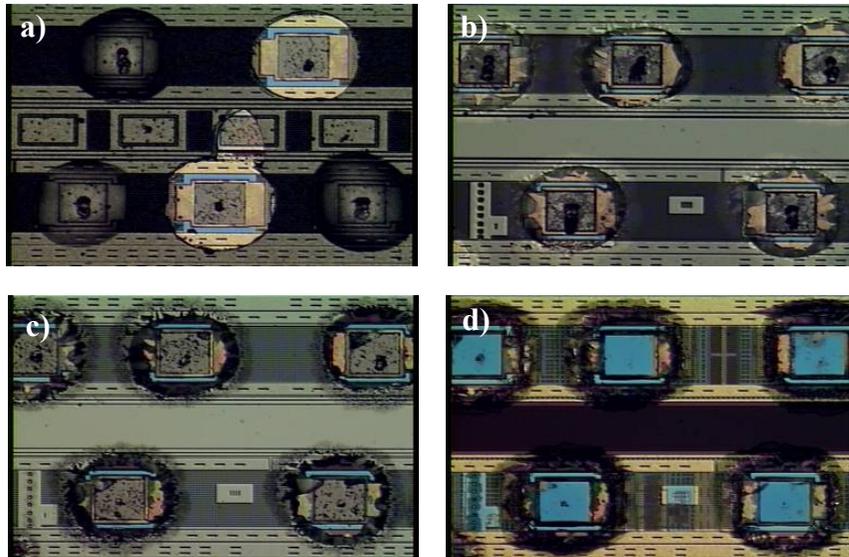
Figure 5.19 CMOS metal contacts: a) after all depositions and rapid thermal annealed at 430°C; b) after SiGe film removal; c) after oxide film removal; d) after Ge film removal

In order to understand the origin of the metal contact damage, etch experiments were done on CMOS chips with no film deposited on them. Figure 5.20(a) shows that the peroxide etch does not attach the metal layer. Figure 5.20(b) shows that the HF etch attaches the metal, but does not create the residual damage around the bond pads as seen before. Therefore the bond pad damage shown in Figures 5.18 and 5.19 is not caused by HF attack through the germanium passivation.
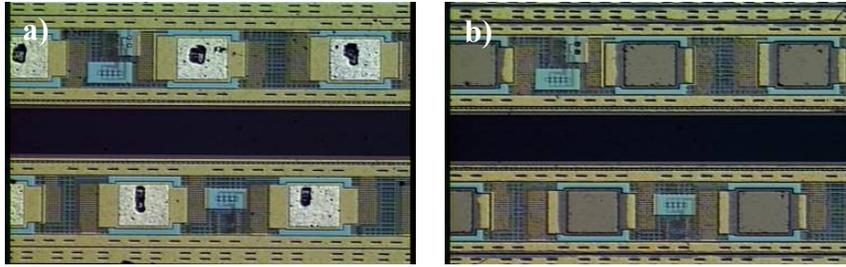
Figure 5.20 CMOS metal contacts: a) without depositions and etched in 30% H_2O_2 solution at 80°C for 5 minutes; b) without depositions and etched in HF solution (50 ml 49% HF + 200 ml DI water) at room temperature for 10 minutes

The damage of the metal contact is likely due to Al and Ge reaction. The eutectic point of Al-Ge is at 420°C [5.17]. During temperature stabilization for the LTO and SiGe depositions, the temperature can easily exceed 420°C. Aluminum diffuses into the germanium layer. After the film removal, the Al-Ge residue becomes visible around the metal contact. A thin layer of TiN should be used between aluminum and the germanium layers. TiN is being used as diffusion barrier for Si and Al, also SiGe and Al. In addition, TiN can be removed using H_2O_2 solution [5.18].

5.6.3 Film delamination after RTA

Figure 5.19(a) shows that the film delaminates after the 430 ºC RTA. However, no delamination is observed with furnace annealing or flash lamp annealing. Previous experiments show that RTA has a more dramatic effect on mechanical properties compared to long furnace annealing at the same temperature. It is believed that the actual

RTA temperature is higher than specified. The poor adhesion is likely due to the change in stress after the RTA.

As described in Figure 5.17, Ge, SiO_2 and SiGe layers are deposited on foundry CMOS chips sequentially. The stress of individual film after each thermal process is summarized in Table 5.6. For these measurements, thermal budget of the sequential depositions is generated by furnace annealing at comparable temperature and duration. The germanium layer has the most significant stress change since the sequential depositions and annealing step are at higher temperatures compared to its deposition temperature.

TABLE 5.6 Stress of individual thin film after each thermal process step

Thin Film	Stress after Ge dep.	Stress after oxide dep.	Stress after SiGe dep.	Stress after RTA
Ge	-220 MPa	NA	-144 MPa	5 MPa
SiO_2	NA	-46 MPa	-30 MPa	-31 MPa
SiGe	NA	NA	-215 MPa	-137 MPa

RTA experiments were also done with different thin film stacks to check their adhesion. The results are listed in Table 5.7. The single or double-layer films show no delamination after the RTA, whereas the SiGe, SiO_2 and Ge tri-layer stack delaminates on either silicon substrate or CMOS chips due to the larger mismatch in stress.

TABLE 5.7 Adhesion of various thin film stacks after RTA at 430 °C for 1 minute

Film stack	Delamination
Ge on Si substrate	No
SiO_2 on Si substrate	No
Ge and SiO_2 on Si substrate	No
SiGe and SiO_2 on Si substrate	No
SiGe, SiO_2 and Ge on Si substrate	Yes
SiGe, SiO_2 and Ge on CMOS chips	Yes

5.6.4 Electrical measurements

Electrical measurements on the same device were made before and after the process and compared. Transistors, Kelvin test structures for via resistance and metal resistance were measured for the 0.13 μm technology. Previous results show that the an increase in via resistance limits the thermal budget for 0.25 μm CMOS technology [5.5]. In this study, none of the 0.25 μm via structures were measurable before the processing. Only the transistor was tested for the 0.25 μm technology. The CMOS test results are summarized in Table 5.8. Due to the metal contact damage, post-process measurement is affected by the contact resistance significantly, especially for the 2-terminal devices. Isolating the failure caused by the thermal process metal contact damage is difficult.

Table 5.8 CMOS test summary

Process	Technology	Chip ID	Device	Survive?	Failure analysis
Depositions FLA @ 700 ºC for 1 ms Film removals	0.13 μm	MOS1	NMOS	Yes	
			PMOS	Yes	
		MOS2	NMOS	Yes/No	Large gate leakage
			PMOS	No	Punch through
		Kel1	via1	Yes	
			via2	Yes	
			via3	Yes	
			via4	No	Damaged metal line
			via5	Yes	
			via6	No	Damaged metal line
			via7	No	Damaged metal line
		Kel2	via1	Yes	
			via2	Yes	
			via3	No	Damaged metal line
			via4	No	Damaged metal line
			via5	No	Damaged metal line
			via6	No	Damaged metal line
			via7	No	Damaged metal line
		Met1	m1	Yes	
			m2	Yes	
			m3	Yes	
			m4	Yes	
			m5	No	Bad contact
			m6	Yes	
			m7	Yes	
			m8	Yes	
		Met2	m1	No	Bad contact
			m2	No	Bad contact
			m3	No	Bad contact
			m4	No	Bad contact
			m5	No	Bad contact
			m6	No	Bad contact
			m7	No	Bad contact
			m8	No	Bad contact
	0.25 μm	Die1	NMOS	Yes	
			PMOS	Yes	
		Die2	NMOS	Yes	
			PMOS	Yes	
		Die3	NMOS	No	
			PMOS	Yes	
Depositions RTA @ 430 ºC for 1 min Film removals	0.13 μm	MOS3	NMOS	No	No gate control
			PMOS	Yes	
		Kel3	via1	Yes	
			via2	Yes	
			via3	No	Damaged metal line
			via4	Yes	
			via5	No	Damaged metal line
			via6	No	Damaged metal line
			via7	No	Damaged metal line
		Met3	m1	No	Bad contact
			m2	No	Bad contact
			m3	No	Bad contact
			m4	No	Bad contact
			m5	No	Bad contact
			m6	No	Bad contact
			m7	No	Bad contact
			m8	No	Bad contact
	0.25 μm	Die4	NMOS	No	
			PMOS	No	

176

		MOS4	NMOS	Yes	
			PMOS	Yes	
		Kel4	via1	Yes	
			via2	Yes	
			via3	Yes	
			via4	Yes	
			via5	No	Damaged metal line
			via6	No	Damaged metal line
Depositions	0.13 μm		via7	No	Damaged metal line
		Met4	m1	Yes	
Film removals			m2	Yes	
			m3	Yes	
			m4	Yes	
			m5	No	Bad contact
			m6	No	Bad contact
			m7	No	Bad contact
			m8	No	Bad contact
	0.25 μm	Die5	NMOS	Yes	
			PMOS	Yes	

In summary, the group of samples that did not go through additional annealing has the best survival rate. Most of the devices could not survive rapid thermal annealing. Film delamination after the RTA exposes the devices to the HF solution during the sacrificial oxide removal. Also, the actual temperature of the RTA tool is believed to be higher than the set point as discussed in section 5.2. The performance of the flash lamp annealing group falls in-between that of the unannealed devices and the RTA-ed devices.

The performance of the surviving 0.13 μm technology transistors is plotted in Figure 5.21. The threshold voltage, on-current, off-current, sub-threshold swing and transconductance of the NMOS devices increase after the process. The threshold voltage of the PMOS devices becomes more negative and the on-current, off-current and sub-threshold swing decrease. The change in transconductance is inconsistent. The NMOS performance does not degrade as much as the PMOS. For the PMOS, the I_{on} change is smaller compared to the change in V_t, but the I_{off} change is consistent with the change in V_t. The degradation in PMOS performance is a bigger concern.

177

The performance of the Kelvin via and metal electromigration structures for 0.13 μm technology are plotted in Figure 5.22 and Figure 5.23. Resistance increases for all devices after processing. Most of the dead devices have visible broken metal lines due to the film removal process. There are also many devices not measurable due to contact damage.
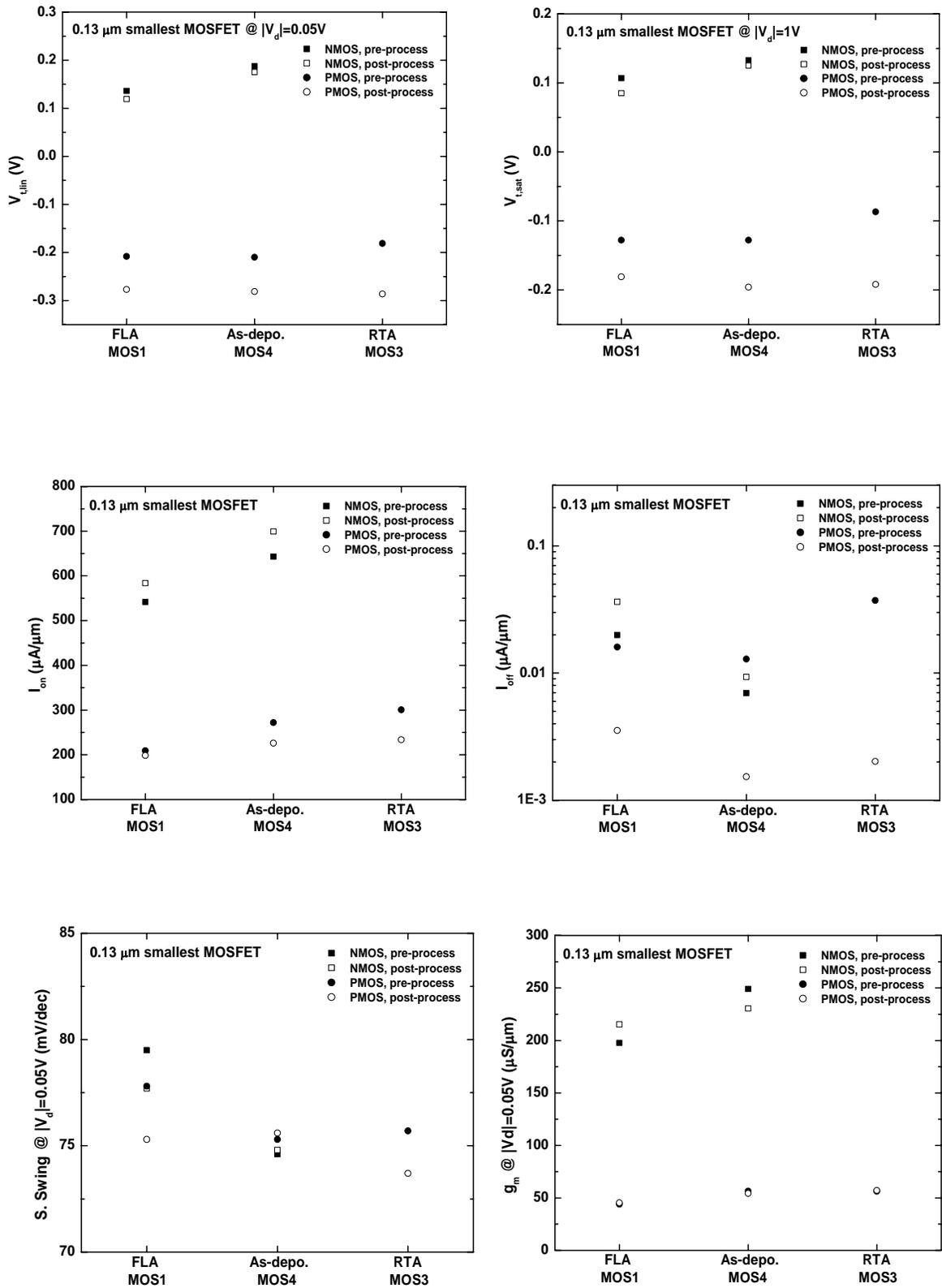
Figure 5.21 0.13 μm technology transistor performance before and after post-processing.
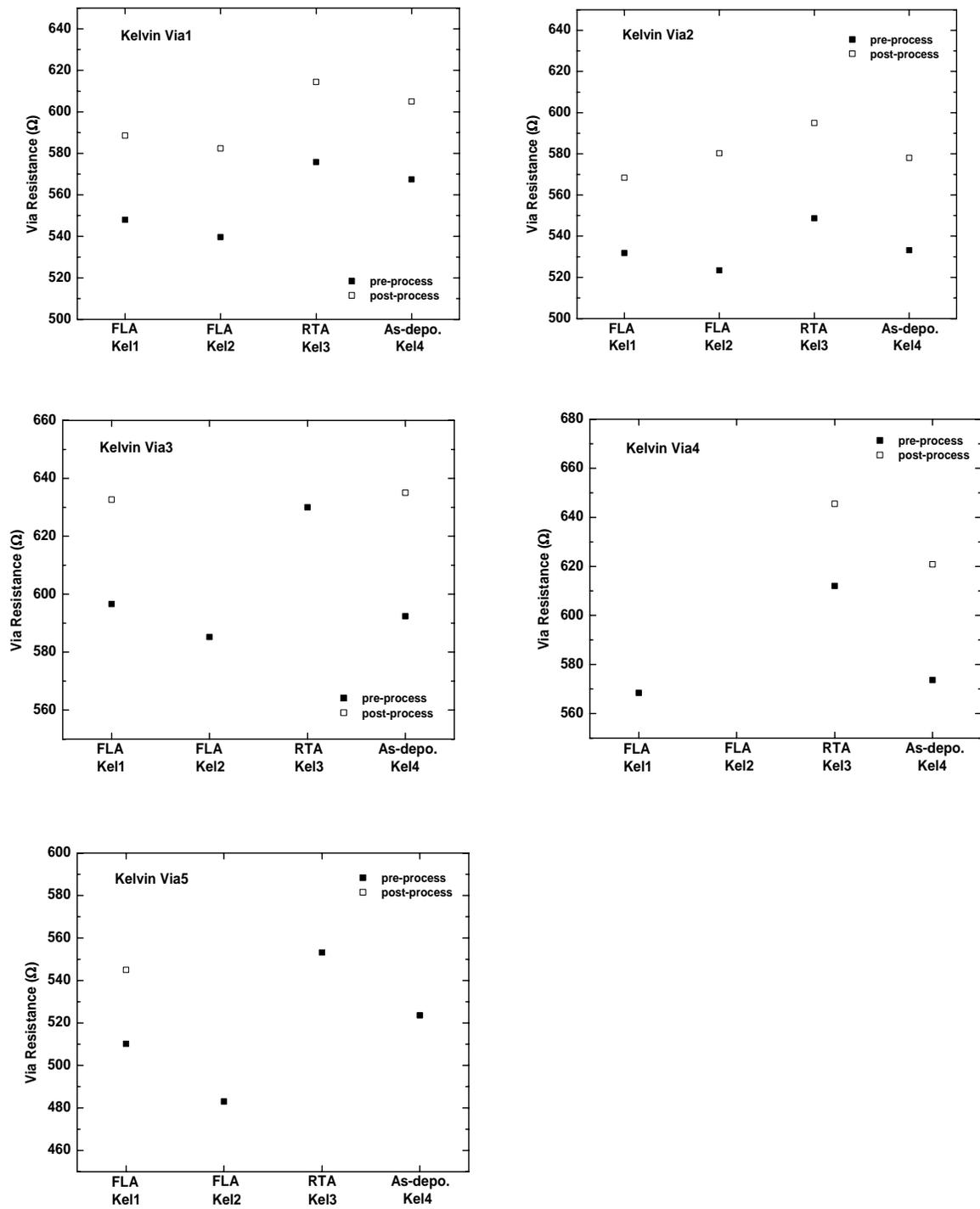
Figure 5.22 0.13 μm technology Kelvin via resistance before and after post-processing.
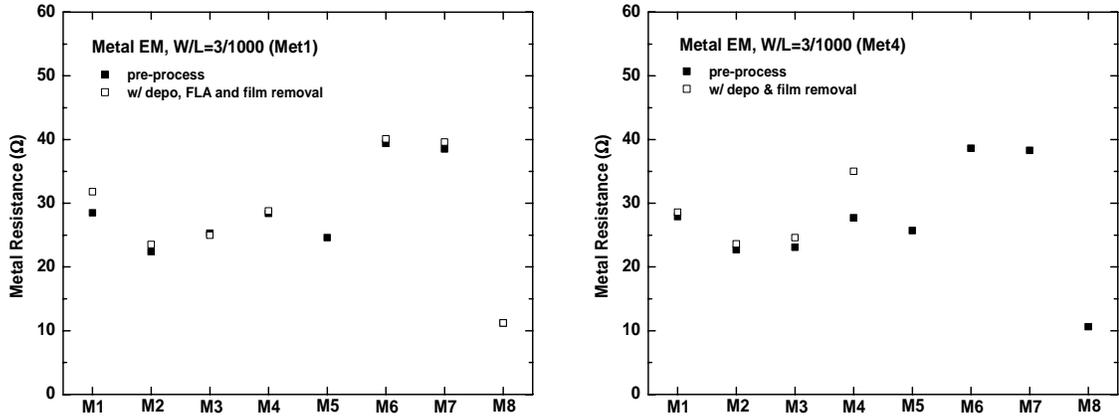
Figure 5.23 0.13 μm technology metal electromigration test structure resistance before and after post-processing

The performance of the 0.25 μm technology transistors are plotted in Figure 5.24. The threshold voltage of the NMOS moves in the negative direction. The threshold voltage of PMOS moves in the positive direction. The relative change is less than 3%. The on-current change doesn't show the same trend for all annealing conditions. The off-current increases after annealing, which is consistent with the threshold voltage shift.
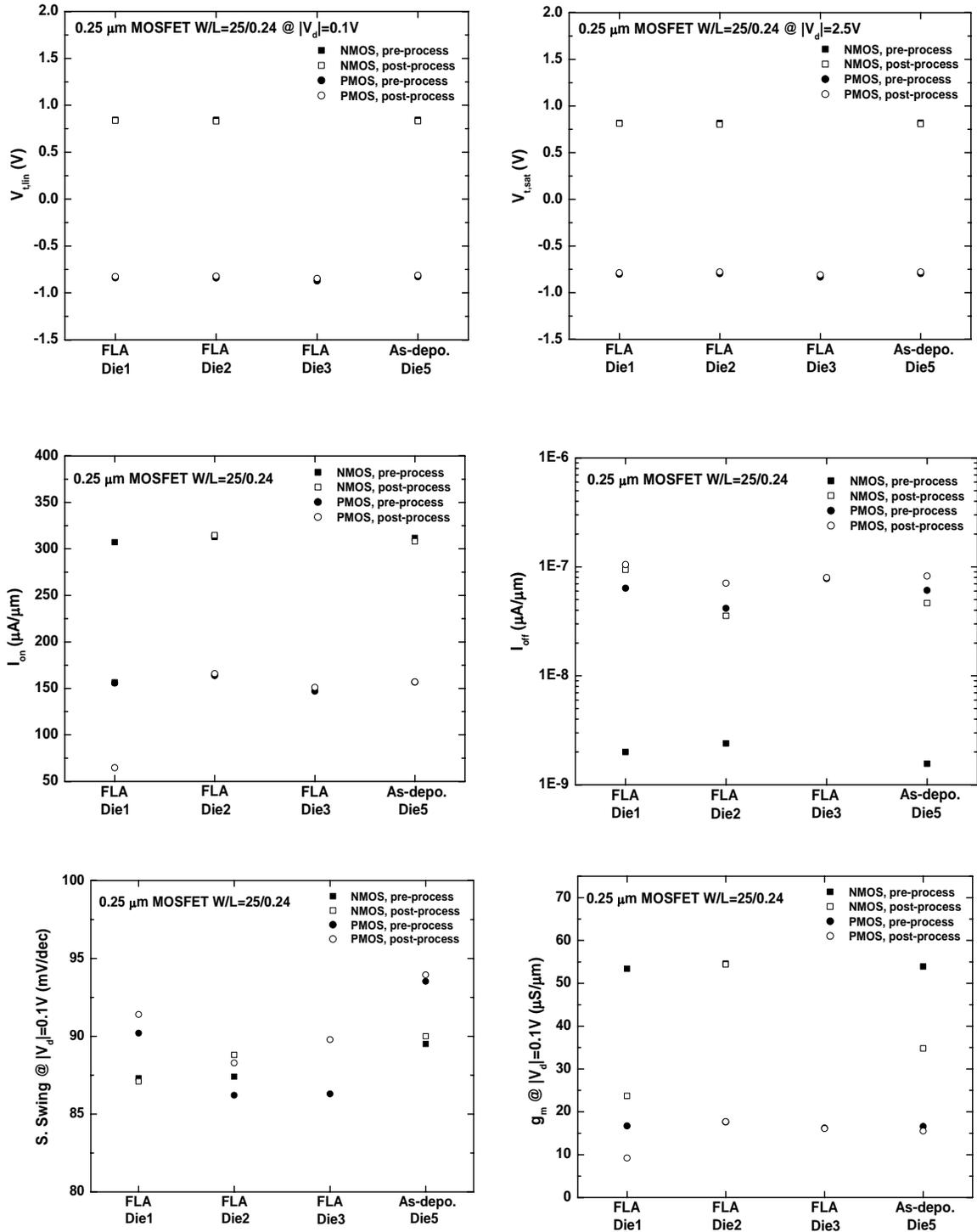
Figure 5.24 0.25 μm technology transistor performance before and after post-processing.

5.7 Summary

A variety of post-deposition processes are discussed in this chapter. Annealing the poly-SiGe film at elevated temperature can crystallize the bottom amorphous region. The top polycrystalline region can be melted and re-crystallized with an excimer laser. High dose and high energy argon ion implantation can be used to amorphize the top crystalline region. All of these post-processes can alter the film mechanical properties. With the appropriate combination of deposition and post-processing, the desired film properties can be achieved. However, the as-deposited film can also have the desired mechanical properties, as described in Chapter 4. Post-deposition processing of poly-SiGe is not recommended for tuning the mechanical properties, because of additional sources of variability. Also, caution is advised for back-end processes such as anti-stiction coating and encapsulation at elevated temperature.

The CMOS thermal budget limit study with the film depositions and removals was inconclusive. Without a Ti/TiN barrier layer, inter-diffusion between the aluminum and the germanium passivation layer damages most of the electrical contacts. Thus, the cause of the apparent degradation in device performance cannot be definitively attributed to the thermal post-processing. Another experiment conducted with equivalent thermal budget furnace annealing is needed to eliminate apparent degradation due to metal contact damage. A full process with film depositions and removals can then be repeated with the Ti/TiN barrier layer deposited first.

References

[5.1] P. A. Krulevitch, "Micromechanical investigations of silicon and Ni-Ti-Cu thin films," Ph.D. Thesis, Dept. of ME, University of California at Berkeley (1994)

[5.2] K. Nunan, G. Ready, P. Garone, G. Sturdy and J. Sledziewski, "Developing a manufacturable process for the deposition of thick polysilicon films for micro machined devices," in *Proceedings of IEEE/SEMI Advanced Semiconductor Manufacturing Conference*, pp. 357-366 (2000)

[5.3] A. E. Franke, Ph.D. thesis, "Polycrystalline silicon-germanium films for integrated Microsystems," Ph.D. Thesis, Dept. of EECS, University of California at Berkeley, 2000

[5.4] S. A. Bhave, B. L. Bircumshaw, W. Z. Low, Y.-S. Kim, A. P. Pisano, T.-J. King and R. T. Howe, "Poly-SiGe: A high-Q structural material for integrated RF MEMS," in *Proc. Solid-State Sensors and Actuators Workshop*, Hilton Head Island, SC, 2002, pp. 34-37

[5.5] H. Takeuchi, A. Wu, X. Sun, R. T. Howe and T. –J. King, "Thermal budget limits of quarter-micron foundry CMOS for post-processing MEMS devices," *IEEE Trans. Electron Devices*, vol. 52, pp. 2081-2086, 2005

[5.6] T. Gebel, M. Voelskow, W. Skorupa, G. Mannino, V. Privitera, F. Priolo, E. napolitani and A. Carnera, "Flash lamp annealing with millisecond pulses for ultra-shallow boron profiles in silicon," *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, vol. 186, issue 1-4, pp. 287-291, 2002

[5.7] H. Wirth, D. Panknin, W. Skorupa and E. Niemann, "Efficient p-type doping of 6H-

SiC: Flash-lamp annealing after aluminum implantation," *Applied Physics Letters*, 74, issue 7, pp. 979-981 (1999)

[5.8] T. Ito, K. Suguro, T. Itani, K. Nishnohara, K. Matsuo and T. Saito, "Improvement of threshold voltage roll-off by ultra-shallow junction formed by flash lamp annealing," *Proceedings of 2003 Symposium on VLSI Technology*, pp. 53-54, 2003

[5.9] S. Sedky, J. Schroeder, T. Sands, R. T. Howe, T. -J. King, "Pulse Laser Annealing of Silicon Germanium Films", *Material Research Society Symposium Proceedings*, 741, J4.1.2-6, p. 61-66, 2002

[5.10] S. Sedky, R. T. Howe, and T.–J. King, "Pulse laser Annealing, a low thermal budget technique for eliminating stress gradient in poly-SiGe MEMS structures", *Journal of Microelectromechanical Systems*, vol. 13(4), pp. 669-675, 2004

[5.11] S. Sedky, M. Gromova, T. Van der Donck, J.-P. Celis and A. Witvrouw, "Characterization of KrF excimer laser annealed PECVD Si_xGe_{1-x} for MEMS post-processing," *Sensors and Actuators A*, vol. 127, pp. 316-323, 2006

[5.12] C. W. Low, M.S. report, "Excimer laser annealing of silicon germanium for MEMS applications," Dept. of EECS, University of California at Berkeley, 2004

[5.13] B. C.-Y. Lin, T.-J. King and R. T. Howe, "Optimization of poly-SiGe deposition processes for modular MEMS integration," in *Proc. Materials Research Society Meeting, Symposium A*, Boston, MA, December 1-5, 2003, pp. A2.4.1-6

[5.14] T. G. Bifano, H. T. Johnson, P. Bieden and R. Krishnamoorthy Mali, "Elimination of stress-induced curvature in thin-film structures", *IEEE J. MEMS*, vol. 11, no. 5, pp. 592-597, 2002

[5.15] W. Shi, H. Zhang, S. Wang, G. Zhang and Z. Li, "Modifying residual stress and

stress gradient in LPCVD Si_3N_4 film with ion implantation," *13th International Conference on Solid-State Sensors, Actuators and Microsystems* pp. 824-827, 2005

[5.16] A. Witvrouw, A. Mehta, A. Verbist, B. Du Bois, S. Van Aerde, J. Ramos-Martos, J. Ceballos, A. Ragel, J. M. Mora, M. A. Lagos, A. Arias, J. M. Hinojosa, J. Spengler, C. Leinenbach, T. Fuchs and S. Kronmüller, "Processing of MEMS gyroscopes on top of CMOS ICs," in *Proc. 52nd IEEE International Solid-State Circuits Conference*, San Francisco, CA, February 6-10, 2005, pp. 88-89

[5.17] B. Predel, Landolt-Bornstein, Group IV Physical Chemistry - *Phase Equilibria, Crystallographic and Thermodynamic Data of Binary Alloys, Volume 5 - Electronic Materials and Semiconductors*, Springer – Verlag, 1998

[5.18] M.-A. N. Eyoum, PhD thesis, "Modularly integrated MEMS technology", Dept. of EECS, University of California at Berkeley, 2006

Chapter 6: Conclusion

6.1 Contributions of this work

The development of LPCVD poly-SiGe for MEMS applications started in UC Berkeley about a decade ago. A few generations of researchers have contributed to the fundamental understanding of the technology. As the technology has matured, interest for commercialization has grown. Most of the work done in this thesis is driven by industrial interests.

Reducing the thermal budget is an important consideration for post-CMOS integration. However, meeting both specifications for thermal budget and materials properties is challenging, especially for the strict strain gradient requirement for inertial sensor applications. There have been some efforts to improve strain gradient with post-deposition annealing. It is found that the post-deposition annealing adds extra variables to the process in addition to the extra thermal budget in most cases. Developing as-deposited films with the desired materials properties is preferred. Since temperature has an exponential effect on thermal budget, reducing the deposition temperature is an on-going effort for poly-SiGe development. Table 6.1 summarizes the materials development of as-deposited LPCVD poly-SiGe films to date. The latest results of PECVD film are also listed for comparison. Stress balancing with multiple-layer depositions was used by Bhave [6.2], Lin [6.3] and Mehta [6.4] for strain gradient optimization. Comparing the results for LPCVD films, a recipe with the lowest thermal budget and the lowest strain gradient is developed in this thesis work. Comparing the best recipes for the LPCVD and the PECVD systems, the mechanical properties of the films

187

are comparable. Although the deposition rate of the best LPCVD recipe is an order of magnitude lower than that of the best PECVD recipe, a lower deposition temperature of 410°C was used in the LPCVD recipe. Microcrystalline SiGe deposited at 400°C or lower is under development using the PECVD system, but the desired materials properties have not been achieved yet [6.4]. Excimer laser annealing of PECVD SiGe deposited at 210°C is another low thermal budget approach for controlling mechanical properties [6.5]. Excimer laser annealing does not increase the thermal budget of the underlying CMOS since the thermal treatment is localized to the topmost MEMS layer. However, the excimer laser annealing step adds extra variables to the process. Also, compromise has to be made between electrical conductivity and strain gradient.

TABLE 6.1 Summary of materials development of poly-SiGe (as-deposited films)

Method	Leading researcher	Date	Temp. (°C)	Time (min)	Thn. (µm)	Ge cont. (%)	Res. (mΩ-cm)	Stress (MPa)	Best strain gradient (µm^{-1})
LPCVD	Franke [6.1]	2000	450	180	3.1	67	1.8	10	1.9×10^{-4}
LPCVD	Bhave [6.2]	2002	425		3	68/65/62			1.8×10^{-4}
LPCVD	Lin [6.3]	2003	425		3.9	69/65	0.55	-36	1.1×10^{-5}
LPCVD	Low	2006	410	480	3.5	60	0.65	-157	1.1×10^{-6}
PECVD	Mehta [6.4]	2005	450	40	4	65/56	1.0	20	3.5×10^{-6}

For the interest of high volume manufacturing, this thesis work furthers the understanding of the sensitivity of materials properties to process variations and improves the process stability with new process gases and hardware modifications. Having a stable and efficient dopant gas significantly reduces the maintenance effort and improves the process repeatability. *In-situ* control of the SiH_4 to GeH_4 gas flow ratio is also an important aspect of process monitoring.

In the interest of minimizing the strain gradient for inertial sensor applications, extensive materials analysis was performed to understand the correlations among the

deposition condition, the microstructure, and the mechanical properties. Boron-doped poly-SiGe films generally have vertically oriented grains, either conical or columnar in shape. Films with small strain gradient usually have columnar grain structure with low defect density. The uniformity of films deposited in a batch LPCVD reactor can be improved by increasing the deposited film thickness, using a proper seeding layer, and/or depositing the film in multiple layers.

6.2 Recommendations for future work

As the poly-SiGe MEMS technology is being transferred to industry, a more robust process is required for high volume manufacturing. Better equipment and tighter process control are necessary to generate high yield. Developing in-line measurement methods for film thickness and germanium content will be important for statistical process control.

For fundamental research, it would be interesting to study the process with the newly developed single-source silicon and germanium precursors $SiGe_2H_8$ and $SiGeH_6$. These precursors have the potential of providing higher deposition rate and producing uniform germanium content film across a large batch.

While a deposition process that gives as-deposited low strain gradient film has been developed with large columnar microstructures, long term repeatability has not been proven. Fine-grained poly-Si films have been demonstrated with reproducibly low strain gradients. The grain size of poly-Si is control by *in-situ* PH_3 or O_2 doping [6.6]. Therefore, additional work can be done to investigate the feasibility of depositing fine-grained poly-SiGe films with average grain size approximately an order of magnitude

smaller than the film thickness, to achieve uniformly low strain gradient. Boron-doped poly-SiGe films always have conical or columnar microstructures. Phosphorus-doped poly-SiGe films can have fine-grained microstructures, but the deposition rate is retarded [6.1]. Carbon doping might provide another option to generate fine-grained microstructures. Carbon is commonly used in epi-SiGe for bandgap engineering and strain compensation, where SiH_3CH_3 is the carbon precursor used in the CVD system. Carbon might be able to serve as an impurity to break up the grain formation and result in fine-grained microstructures.

References

[6.1] A. E. Franke, "Polycrystalline silicon-germanium films for integrated Microsystems", Ph.D. Thesis, Dept. of EECS, University of California at Berkeley, 2000

[6.2] S. A. Bhave, B. L. Bircumshaw, W. Z. Low, Y.-S. Kim, A. P. Pisano, T.-J. King and R. T. Howe, "Poly-SiGe: a high-Q structural material for post-CMOS integrated RF MEMS," in *Proc. Solid-State Sensor, Actuator, and Microsystems Workshop*, Hilton Head Island, SC, 2003, pp. 34-37

[6.3] B. C.-Y. Lin, T.-J. King and R. T. Howe, "Optimization of poly-SiGe deposition processes for modular MEMS integration," in *Proc. Materials Research Society Meeting, Symposium A*, Boston, MA, December 1-5, 2003, pp. A2.4.1-6

[6.4] A. Mehta, M. Gromova, P. Czarnecki, K. Baert and A. Witvrouw, "Optimization of PECVD poly-SiGe layers for MEMS post-processing on top of CMOS," in *Proc. 13th International Conference on Solid-State Sensors, Actuators and Microsystems (Transducers 05)*, Seoul, Korea, June 5-9, 2005, pp. 1326-1329

[6.5] S. S. Sedky, O. Mortagy and A. Witrouw, "Low thermal budget techniques for controlling stress in SiGe deposited at 210°C," in *Proc. Materials Research Society Meeting*, San Francisco, CA, January, 2006

[6.6] K. Nunan, G. Ready, P. Garone, G. Sturdy and J. Sledziewski, "Developing a Manufacturable Process for the Deposition of Thick Polysilicon Films for Micro Machined Devices," *Proceedings of IEEE/SEMI Advanced Semiconductor Manufacturing Conference*, pp. 357-366, 2000

Appendix A: SAM Coating of Poly-SiGe for Stiction Reduction

The large surface-to-volume ratio of MEMS devices makes them vulnerable to adhesion upon contact. The interfacial forces between surfaces include capillary, van der Waals, and electrostatic attractions. Capillary force causes stiction of the structure to the substrate during the sacrificial layer wet etch – so-called "release stiction"; van der Waals and electrostatic attraction cause surfaces permanently to adhere to each other during device operation – "in-use stiction". Overcoming these interfacial forces is essential for the successful fabrication and operation of MEMS devices.

A.1 SAM overview

Various techniques have been investigated to achieve low adhesion energy [A.1]. Surface modification using hydrophobic self-assembled monolayers (SAM) is one of the most successful strategies, as it addresses both release and in-use stictions. SAM coatings are conformal with dense and stable structures. In addition, the ability to tailor both the head and the tail groups of the constituent molecules gives a large variety of feasible coating materials.

Self-assembled monolayers (SAM) are molecular assemblies that are formed spontaneously by the immersion of the appropriate substrate into a solution of an active surfactant in an organic solvent. The molecule consists of three main parts. The first part is the head group, which chemisorbs at all of the surface sites, resulting in a close-packed monolayer. The second part is the alkyl chain, where van der Waals interactions between chains contribute further to the ordering of the monolayer. The third part is the terminal

group. For anti-stiction purposes, a methyl terminal group makes the surface hydrophobic.

Poly-Si has been a conventional MEMS material for more than a decade and various SAM coatings for poly-Si have been investigated [A.2 – A.6]. Poly-SiGe is a promising material for the modular integration of MEMS and CMOS, due to its low process thermal budget and its good electrical and mechanical properties [A.7]. Poly-SiGe MEMS processing shares many similarities with poly-Si processing; stiction is unfortunately also a problem with poly-SiGe. In this work, the feasibility of SAM coating on poly-SiGe is studied. OTS and 1-octadecene SAM were applied to poly-Si, poly-SiGe and poly-Ge surfaces for comparison. Effectiveness of the coating was measured by the water contact angle. N_2 ambient annealing was applied to SAM coated films for thermal stability study.

A.2 Experimental details

Poly-Si, poly-SiGe and poly-Ge films used in this SAM study were deposited in conventional LPCVD reactors. The poly-SiGe film has approximately 68% germanium content. All films have surface roughness less than 3 nm rms so that topography does not affect the coating and the contact angle measurement significantly.

Alkyltrichlorosilane-based monolayer OTS [$CH_3(CH_2)_{17}SiCl_3$] and alkene-based monolayer 1-octdecene [$CH_3(CH_2)_{15}CH=CH_2$] were studied in this experiment. Both precursor molecules contain a straight 18-carbon chain and a hydrophobic CH_3 tail group. Both molecules bind to the substrate only at one end, but with different mechanisms. The chlorosilane molecules of the OTS react with water to form silanols, which then condense

to form siloxane polymers with the elimination of water [A.2]. The alkene-based SAM abandons the chlorosilane chemistry and adopts a free radical reaction of a primary alkene [1-octadecene, $CH_3(CH_2)_{15}CH=CH_2$] to bind the precursor molecule to a hydrogen terminated silicon surface with a Si-C bond [A.4].

The coating procedure of OTS and 1-octadecene are listed in Tables A.1 and A.2. A hexadecane and carbon tetrachloride mixture with 6:4 volume ratio was used as the OTS solvent. The OTS concentration was approximately 1mM. The 1-octadecene solution was prepared with 10% volume of 1-octadecene with 90% volume of hexadecane as the solvent. All chemicals used in the SAM coating process were standard solvent grade except hexadecane and chloroform were anhydrous (Aldrich Chemical Co.).

TABLE A.1 OTS coating procedure

Purpose	Procedure	Duration
	Acetone rinse	5 min.
	IPA rinse	5 min.
Cleaning	DI wafer rinse	5 min.
	UVO light*	5 min.
	HF etch	5 min.
Oxidation	UVO light*	5 min.
SAM coating	IPA rinse	5 min.
	OTS mixture	60 min.
Cleaning	IPA rinse	5 min.
	DI water rinse	5 min.

* Poly-SiGe and poly-Ge films did not get the UVO light treatment since their oxides form readily in air.

TABLE A.2 1-octadecene coating procedure

Purpose	Procedure	Duration
	Acetone rinse	5 min.
Cleaning	IPA rinse	5 min.
	DI wafer rinse	5 min.
H termination	HF rinse and dry	5 min.
SAM coating	IPA rinse	5 min.
	1-octadecene mixture @ 180ºC with N_2 purge	30 min.
	Petroleum ether rinse	5 min.
Cleaning	IPA rinse	5 min.
	DI water rinse	5 min.

The thermal stability of the coatings in N_2 ambient was tested using a rapid thermal annealing system. Temperature was ramped up to the set point from room temperature in 30 sec, the set temperature stayed constant for 5 minutes and then slowly cooled down to room temperature in about an hour.

The effectiveness of the SAM coatings and thermal stability were evaluated with static water contact angle measurement. Data were taken with DI water (resistivity > 18 $M\Omega$) according to the sessile droplet method. Droplet size was approximately 4 μl.

A.3 Results and discussion

A.3.1 Film characterization

Water contact angle data given in Figure A.1 and Table A.3 confirms that well-packed monolayers are formed on all poly-Si, poly-SiGe and poly-Ge surfaces. Data for uncoated samples are also listed for reference. Samples exposed to ambient humidity for 5 days show similar water contact angles, which indicates the SAM coatings do not degrade in ambient at room temperature.
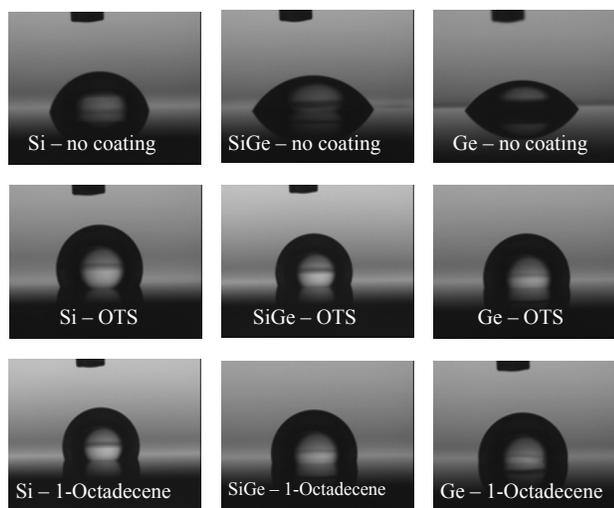
Figure A.1 Images of water droplet on various surfaces

TABLE A.3 Water contact angle data for poly-Si, poly-SiGe and poly-Ge surfaces

Coating	Poly-Si	Poly-SiGe	Poly-Ge
None	71.3º	60.5º	54.7º
OTS	116.4º	114.3º	112.3º
1-octadecene	108.8º	100.9º	93.5º

The contact angle data indicate that all surfaces become hydrophobic after the SAM coating. However, having large water contact angle is not sufficient to show that the head group of SAM is chemically bonded to the substrate.

The data show that the contact angle decreases with germanium content for both SAM coatings. In the case of OTS on oxidized poly-Si, absorption takes place through the hydrolysis of the Si-Cl bonds to form Si-OH groups; the OH groups interact with OH groups on the oxidized surface, forming Si-O-Si bonds to the substrate through condensation reaction. Si-O-Si bonds are also form between adjacent head groups, creating a cross-linked network at the surface [A.2]. If a similar reaction happens on oxidized poly-SiGe or poly-Ge surfaces with the formation of Si-O-Si and/or Ge-O-Ge

bonding networks, the larger cell dimension of germanium would reduce the packing density of OTS molecules, resulting in smaller water contact angles.

For the case of 1-octadecene on poly-Si, the SAM molecule bonds directly to the hydrogen terminated silicon [A.4]. By analogy with this reaction, one would expect that hydrogen terminated germanium surfaces would be required for the bonding to form at the substrate. It is well known that germanium surfaces are less stable compared to Si surfaces and that a native germanium oxide layer forms readily in air [A.8]. It has been observed that an HF dip makes poly-Si hydrophobic, whereas poly-SiGe and poly-Ge remain hydrophilic. Therefore, it is unclear that if 1-octadecene are chemically bonded to germanium. It has been reported that 1-octadecene could coat substrates other than hydrogen terminated silicon without chemical bonding and still result in large water contact angles [A.4].

A.3.2 Thermal stability

Thermal stability of the SAM coatings has been investigated in N_2 ambient since most MEMS packaging processes contain steps at elevated temperatures. The resulting water contact angle data of the films after heating are summarized in Figure A.2 and Figure A.3. We found that OTS coating survives to higher temperatures, consistent with results reported on poly-Si surface [A.2, A.4, A.9, and A.10].
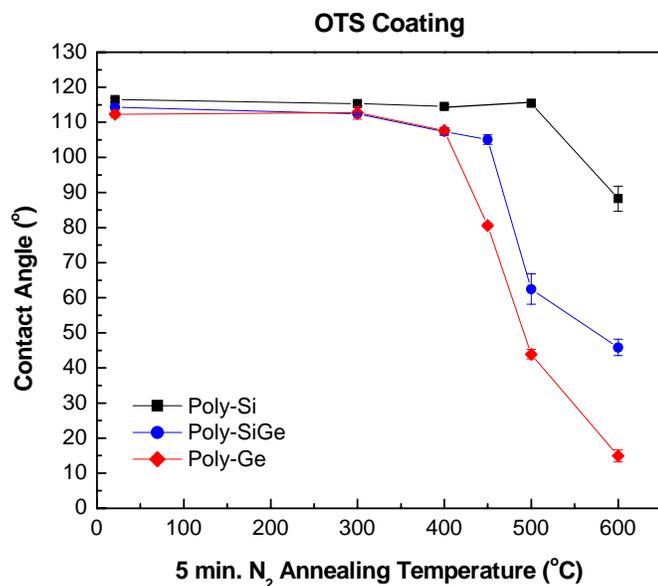
Figure A.2 Water contact angle measurements on OTS SAM-coated Si, SiGe and Ge to assess thermal stability in N_2 ambient.
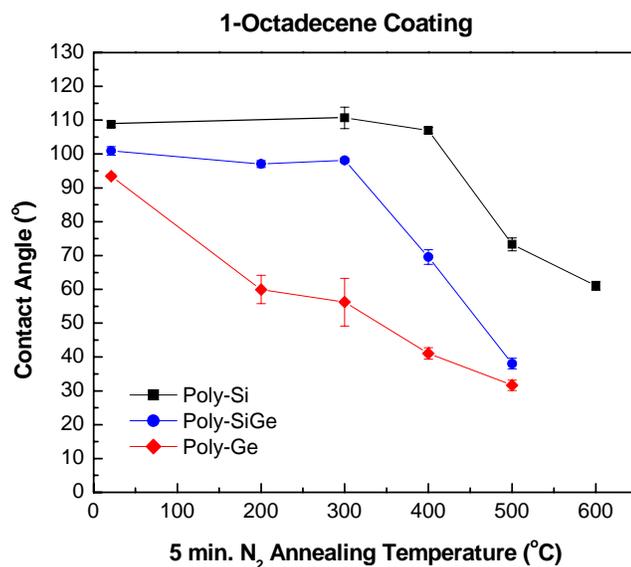
Figure A.3 Water contact angle measurements on 1-octadadecene SAM-coated Si, SiGe and Ge to assess thermal stability in N_2 ambient.

There are three possibilities for the OTS decomposition mechanism: cleavage of the Si-O or Ge-O head group bond, cleavage of the Si-C bond, and cleavage of the C-C bond. Thermal stability of OTS coating on oxidized silicon surface has been well-studied with high resolution electron energy loss (HREEL) spectrum [A.9]. HREEL spectrum shows that the siloxane head groups remain on the surface until about 827°C. Si-C modes are hard to detect due to the presence of the Si-O-Si symmetric stretch. If Si-C bond cleavage were occurring to a significant extent, the entire chain would be desorbed completely. Gradually decrease in water contact angle upon heating suggests that the decomposition of OTS coated Si surface begin with the cleavage of C-C bonds at 467°C. As a result of the C-C bond cleavage, a shorter alkyl radical is left on the surface and reactions with molecular hydrogen in the ambient yield CH_3 group at the tail. Although the chain length has been significantly reduced, the monolayer is still reasonably well-ordered at a temperature slightly higher than the C-C bond cleavage point.

If OTS coatings on poly-SiGe or poly-Ge are analogous, we would expect them to have similar thermal stability as poly-Si. However, the data show that thermal stability decreases with increased germanium content. Since germanium oxide is known to be unstable at elevated temperature [A.8], the Ge-O bond cleavage might happen before the C-C bond cleavage.

The desorption mechanism of 1-octadecene monolayer on silicon surface is very different from OTS [A.10]. HREEL spectrum shows the presence of Si-H groups following annealing to 377 °C. This suggests the desorption of the alkyl monolayers occurs through β-hydride elimination. At higher temperatures, the entire chain decomposed with SiC vibrational modes evident.

From Figure A.3, we can see the SAM coating on poly-SiGe follows the trend of poly-Si, but break down starts at a lower temperature. On the other hand, poly-Ge breaks down at a significantly lower temperature compared to poly-Si. As discussed before, the germanium oxide could not be eliminated before the 1-octadecene coating. The head group of the SAM might not be able to bond to the germanium surface with the Ge-C bond. Upon heating, the SAM molecules become disordered due to the lack of chemical bonding at the head group. For the case of coating on poly-SiGe, there should exist some Si-C bonds. However, the packing density of the monolayer should be lower compared to that of poly-Si surface, which results in smaller water contact angle and worse thermal stability.

A.4 Summary

Water contact angle measurement shows that self-assembled monolayer coatings on unpatterned poly-SiGe surfaces have hydrophobic properties and reasonable thermal stability. The existence of germanium oxide at the surface degrades the packing density and thermal stability of the SAM coating. Comparing OTS and 1-octadecene coatings, OTS monolayer gives higher water contact angle and better thermal stability on poly-SiGe surface. Due to the existence of germanium oxide at the poly-SiGe surface, alkyltrichlorosilane-based monolayer is believed to be a better coating material for poly-SiGe than alkene-based monolayer.

Further experiments should be done with cantilever beam array to characterize the release and in-use stiction of the SAM-coated poly-SiGe surfaces. HREEL spectroscopy

should also be used to further study the correlation between the germanium content and the desorption mechanism.

References

[A.1] R. Maboudian, R. T. Howe, "Critical review: Adhesion in surface micromechanical structures," *J. Vac. Sci. Technol. B* vol. 15(1), pp. 1-19, 1997

[A.2] U. Srinivasan, M. R. Houston, R. T. Howe, and R. Maboudian, "Alkyltrichlorosilane-based self-assembled monolayer films for stiction reduction in silicon micromachines," *J. MEMS*, vol. 7(2), pp. 252-260, 1998

[A.3] B. H. Kim, T. D. Chung, C. H. Oh and K. Chun, "A new organic modifier for anti-stiction," *J. MEMS*, vol. 10(1), pp. 33-40, 2001

[A.4] W. R. Ashurst, C. Yau, C. Cararo, C. Lee, G. J. Kluth, R. T. Howe, R. Maboudian, "Alkene based monolayer films as anti-stiction coatings for polysilicon MEMS," *Sensors and Actuators A*, vol. 91(3), pp. 239-248, 2001

[A.5] T. M. Mayer, M. P. de Boer, N. D. Shinn, P. J. Clews, and T. A. Michalske, "Chemical vapor deposition of fluoroalkylsilane monolayer films for adhesion control in microelectromechanical systems," *J. Vac. Sci. Technol. B,* vol. 18(5), pp. 2433-2440, 2000

[A.6] W. R. Ashurst, C. Carraro, R. Maboudian and W. Frey, "Wafer level anti-stiction coatings for MEMS," *Sensors and Actuators A,* vol. 104(3), pp. 213-221, 2003

[A.7] A. E. Franke, J. M. Heck, T. –J King, and R. T. Howe, "Polycrystalline silicon germanium films for integrated Microsystems," *J. MEMS*., vol. 12, pp. 160-171, 2003

[A.8] K. Prabhakaran, F. Maeda, Y. Watanabe, and T. Ogino, "Thermal decomposition pathway of Ge and Si oxides: observation of a distinct difference," *Thin Solid Films*, vol. 369, pp. 289-292, 2000

[A.9] G. J. Kluth, M. M. Sung, and R. Maboudian, "Thermal behavior of alkylsiloxane self-assembled monolayers on the oxidized Si(100) surface", *Langmuir*, vol. 13, pp. 3775-3780, 1997

[A.10] M. M. Sung, G. J. Kluth, R. W. Yauw, and R. Maboudian, "Thermal eehavior of alkyl Monolayers on silicon surfaces," *Langmuir*, vol. 13, pp. 6164-6168, 1997

Appendix B: Stress Stability of LPCVD Poly-SiGe and SiO$_2$ Films

Reliability specifications typically require MEMS structural layers to have long-term material stability. In past research, results from the analysis of wafer curvature over time appeared to indicate that poly-SiGe films experience a stress drift in humid environments [B.1]. This observation created a major challenge to the plausibility of poly-SiGe MEMS technology. In order to understand the stress-drift phenomenon, multi-layer thin film stress is modeled [B.2] with the same methodology used to derive the Stoney Equation [B.3]. Results show that the residual stress of poly-SiGe films is, in fact, stable in ambient conditions. The apparent residual stress drift of the poly-SiGe films reported in Ref. [B.1] was caused by the unstable low temperature LPCVD oxide on the backside of the wafers.

B.1 Experimental details

The average residual stresses of various thin films were determined with wafer curvature measurements before and after thin film deposition using a Tencor FLX-2320. Long term average residual stress monitoring was done with various layer stacks as shown in Figure B.1. Poly-Si, poly-SiGe, and poly-Ge, as well as various oxides, were deposited and removed from single crystal silicon (SCS) wafers under different conditions as summarized in Table B.1. Initial wafer curvature measurements were taken from the bare Si wafer for the single layer stacks (Figure B.1a & Figure B.1b), and from the oxidized wafer before poly-Si, poly-SiGe or poly-Ge deposition for the bi-layer stacks (Figure B.1c).

Poly-Si, poly-SiGe or poly-Ge	Various Oxides

a) Poly-Si, SiGe or Ge on silicon   b) Oxide on silicon   c) Poly-Si, SiGe or Ge on oxide
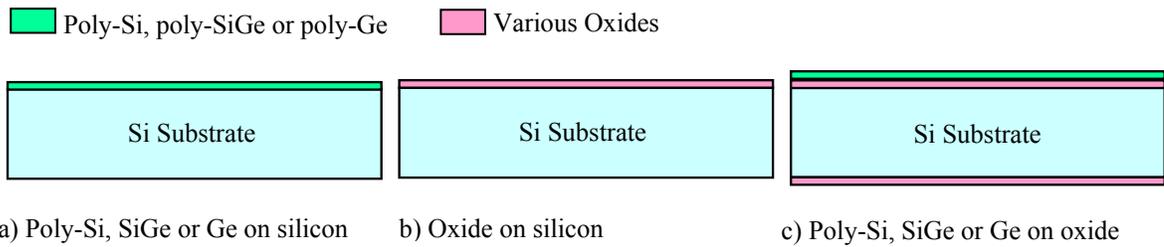
Figure B.1 Layer stacks for stress monitoring

TABLE B.1 Deposition and removal conditions of the various thin films

Film	Deposition method	Removal method
Poly-Si (0.6 µm)	LPCVD @ 620ºC	RIE @ 60ºC
Poly-SiGe (0.2 – 1 µm)	LPCVD @ 400 – 450ºC	RIE @ 60ºC
Poly-Ge (0.4 µm)	LPCVD @ 350ºC	RIE @ 60ºC
Dry thermal oxide (1200 Å)	Thermally growth @ 1050ºC	HF solution @ 21ºC
Wet thermal oxide (1600 Å)	Thermally growth @ 1050ºC	HF solution @ 21ºC
LPCVD oxide (2 µm)	LPCVD @ 450ºC	HF solution @ 21ºC
PECVD oxide (0.5 µm)	LPCVD @ 390ºC	NA (single side deposition)

B.2 Results and discussions

Wafers used in Ref. [B.1] to monitor the stress stability had a poly-SiGe film deposited on top of a 2 µm LPCVD oxide, as shown in Figure B.1c. This layer stack is commonly used in MEMS: the thick oxide serves as a sacrificial layer and the poly-SiGe serves as the structural layer. For comparison purposes, the results reported in Ref. [B.1] have been reproduced in this work, as plotted in Figure B.2. The measured stresses of poly-SiGe and poly-Ge on LPCVD oxide become more tensile over time, but all poly-Si, poly-SiGe and poly-Ge films on thermal oxide or SCS are stable. These results indicate a problem with the LPCVD oxide. It should also be noted that poly-Si films on LPCVD oxide are more stable than poly-SiGe and poly-Ge films on the same oxide. This is because, during the poly-Si deposition, the LPCVD oxide is annealed at 620 °C.
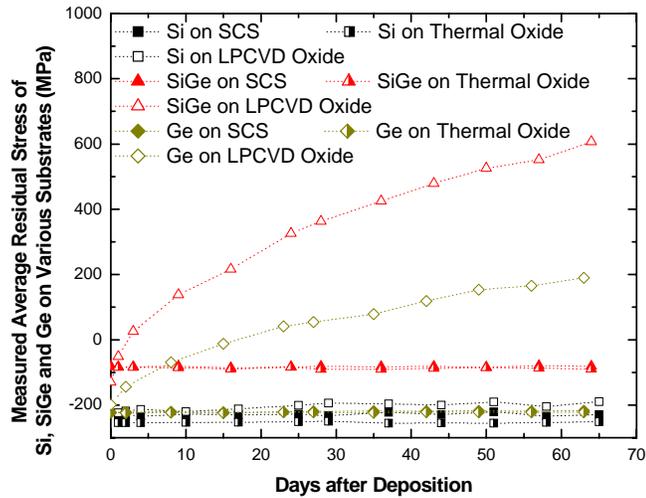
Figure B.2 Stress stability of poly-Si, SiGe and Ge on various substrates.

Further experimentation was done with the poly-SiGe on LPCVD oxide wafers (Figure B.3). If the backside poly-SiGe films of two similar wafers are removed at different times, the drift profiles and absolute stresses of the wafers are nearly identical, with an offset in the x-axis. When the backside poly-SiGe and LPCVD oxide films are both removed, the stresses of the topside poly-SiGe and LPCVD oxide films become stable. This suggests that the stress drift reported in Ref. [B.1] is due solely to the instability of the LPCVD oxide film exposed to the ambient on the backside of the wafer.
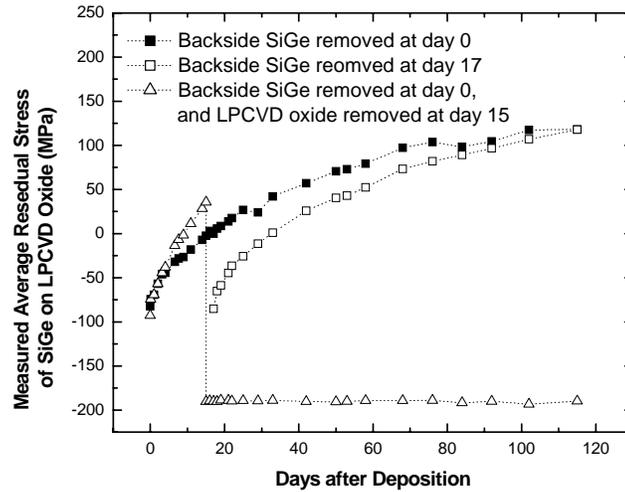
Figure B.3 Stress stability of poly-SiGe on LPCVD oxide

Low-temperature (450°C) LPCVD oxide films are known to be porous and of poor quality [B.4]. In this investigation, different experiments were done with LPCVD oxide wafers to characterize its stability (Figure B.4). The residual stress of the as-deposited LPCVD oxide wafer becomes more compressive over time. The "drift" rate slows over time. The backside oxide removal date does not affect the measurement. Putting the LPCVD oxide films in a desiccated environment slows down the stress drift. Annealing the films at 615°C for five hours results in a tensile film that becomes more compressive more slowly than unannealed films. The above facts suggest that the absorption of ambient water into the LPCVD oxide films is the major cause of the observed stress drift. As water is absorbed, the films become more compressive. The 615°C N_2 annealing appears to densify the oxide and decreases the diffusion constant. It is also shown in Figure B.4 that if a wafer is annealed 136 days after deposition in N_2 at 425°C for an hour, the resultant stress was approximately that of a fresh oxide film. The

stress drift of this 425 ºC annealed wafer is faster than a monitor wafer with the backside also etched on day 136. Since the 425ºC anneal is lower than the deposition temperature of 450ºC, it is unlikely that the anneal rearranges or densifies the oxide molecules; rather, the anneal most likely drives out the absorbed water. Ultimately, the stress drift of the 425ºC annealed wafer is faster than that of the control wafer on day 136 due to a larger moisture gradient.
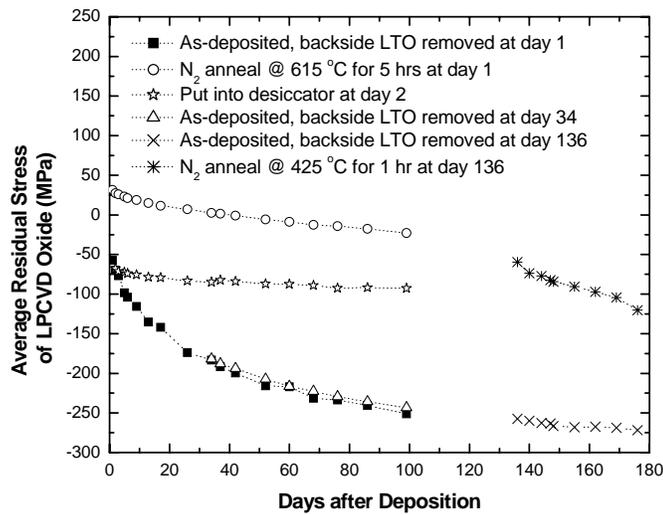
Figure B.4 Stress stability of LPCVD oxide

Returning to the data for poly-SiGe on LPCVD oxide wafers in Figure B.3, as the backside oxide films became more compressive, the poly-SiGe film appeared to become more tensile. Data in Figure B.3 also indicates that poly-SiGe is an effective barrier to moisture. The backside LPCVD oxide did not drift until it was exposed to the ambient after the poly-SiGe layer was removed. Finally, the stress of the frontside LPCVD oxide did not drift under the poly-SiGe cap.

Figure B.5 shows the stress stability of different oxide films. The stress of the 450ºC LPCVD oxide becomes more compressive while the stresses of the 1050ºC dry and wet thermal oxides are nearly stable. The stresses of TEOS-based PECVD oxides deposited at 390ºC also become more compressive, but at a much higher rate than the LPCVD oxide films. Also, the TEOS-based PECVD oxide films begin to saturate within 30 days of being exposed to the ambient. Their remarkably high stress drift rate is expected since TEOS-based oxides are generally more porous [B.5] and the diffusion constant of water is much higher. In contrast, silane-based PECVD oxide films were found to have better stress stability in humid environments (data not presented here).
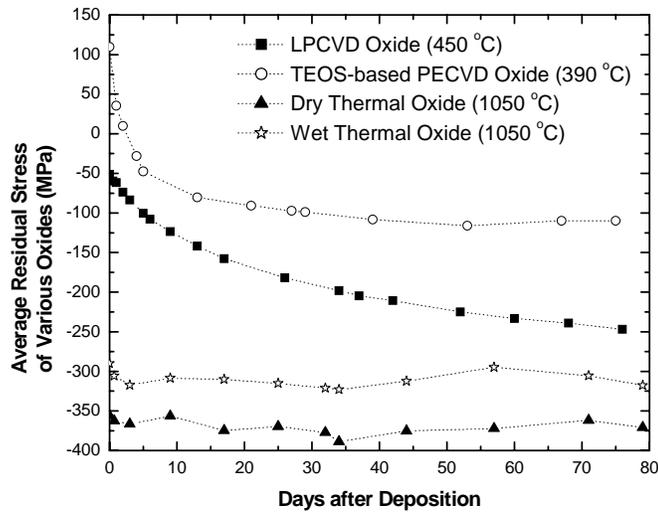
Figure B.5 Stress stability of various oxides

B.3 Modeling thin film stress

We can now turn to the stress analysis of n thin films on the frontside and m thin films on the backside of a single-crystal substrate wafer. In this case, the thin films experience nearly ideal biaxial stress, or plane stress. Moreover, the residual stresses in

the films are uniform over the wafer, not varying with direction or position. We shall assume that the thin films are linear, isotropic materials. This is reasonable for amorphous and poly-crystalline materials. The single-crystal substrate, on the other hand, is anisotropic. However, proper choice of the Young's modulus will minimize the error introduced by this assumption [B.6].

To proceed, we assume that there exists a neutral axis in the substrate whose position is unaffected by the existence of the films. Further, it is assumed that the deflections are small and the shear forces are negligible. Finally, we shall assume the residual stress in the thin films is small compared to the substrate stiffness, allowing us to neglect the contraction/extension of the wafer caused by the thin films. The material and geometric constants of the substrate and thin films are listed in Table B.2. Figure B.6 illustrates the setup for two frontside and two backside films.

TABLE B.2 Material and geometric constants

	Substrate	Film f1	...	Film fn	Film b1	...	Film bm
Young's Modulus	E_s	E_{f1}	...	E_{fn}	E_{b1}	...	E_{bm}
Thickness	H	h_{f1}	...	h_{fn}	h_{b1}	...	h_{bm}
Residual Stress	$\sigma_s = 0$	σ_{f1}	...	σ_{fn}	σ_{b1}	...	σ_{bm}
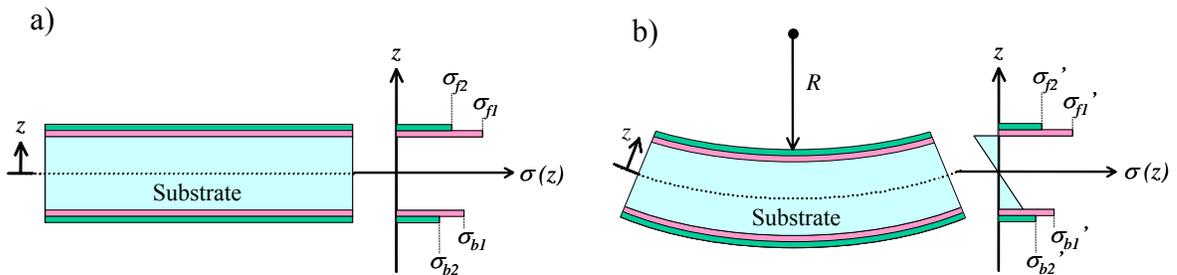
Figure B.6 Cross-sectional views of a substrate with thin films on both sides for $n = 2$ and $m = 2$: a) before release; b) after release.

For uniform, constant biaxial stress, the constitutive relation relating stress, σ, to strain, ε, is:

$$\sigma = \frac{E}{1-\upsilon}\varepsilon = E'\varepsilon \tag{B.1}$$

where E' and υ are the biaxial elastic modulus and Poisson's ratio of the material, respectively. Conceptually, we imagine the wafer clamped so that, despite the thin film residual stresses, there is no deflection of the wafer or stress-relaxation of the thin-films. This is the "Before Release" state. Next, we imagine the wafer released from its clamps and deforming to an equilibrium state: the "After Release" state.

The system is in static equilibrium after release. Therefore, the net moment per unit length of the system after release, M_{ar}, must equal the net moment before release, M_{br}. Before release, the moments are due only to the thin films:

$$M_{br} = \sum_{i=1}^{n} \int_{-H/2}^{H/2+F(i)} \sigma_{fi} z\, dz - \sum_{j=1}^{m} \int_{-H/2-B(j)}^{-H/2} \sigma_{bj} z\, dz \qquad F(i) = \sum_{i=1}^{n} h_{fi}, \ B(j) = \sum_{j=1}^{m} h_{bj}$$

$$M_{br} \approx \frac{H}{2}\left[\sum_{i=1}^{n} \sigma_{fi} h_{fi} - \sum_{j=1}^{m} \sigma_{bj} h_{bj}\right] \tag{B.2}$$

After release, the substrate develops a balancing moment via pure bending, M_{ar}:

$$M_{ar} = \int_{-H/2}^{H/2} \sigma_{s}' z\, dz = \int_{-H/2}^{H/2} E_{s}' \frac{z^2}{R}\, dz = \frac{E_{s}'}{12R} H^3 \tag{B.3}$$

where R is the radius of curvature of the wafer. Recall that, for static equilibrium after release, we require $M_{ar} = M_{br}$. Hence, equating (B.2) and (B.3):

$$\frac{1}{6}\left(\frac{E_{s}'}{R}\right)H^2 = \sum_{i=1}^{n} \sigma_{fi} h_{fi} - \sum_{j=1}^{m} \sigma_{bj} h_{bj} \tag{B.4}$$

211

The Tencor FLX-2320 measures the change in the radius of curvature of a substrate caused by the stress of a thin film. The stress of the thin film is calculated with a simplified version of Eq. (B.4):

$$\sigma_{measured} = \frac{1}{6}\left(\frac{E_s{}'}{R}\right)\frac{H^2}{h_f} = \sigma_f \tag{B.5}$$

which is appropriate for single layer thin films as shown in Fig. 1a & b.

For the bi-layer stacks shown in Fig. 1c, Eq. (4) can be simplified to:

$$\sigma_{measured} = \frac{1}{6}\left(\frac{E_s{}'}{R}\right)\frac{H^2}{h_{SiGe}} = \sigma_{SiGe} + \sigma_{ox(f)}\frac{h_{ox(f)}}{h_{SiGe}} - \sigma_{ox(b)}\frac{h_{ox(b)}}{h_{SiGe}} \tag{B.6}$$

If the stresses of the frontside oxide and the backside oxides are cancelled out, Eq. (B.6) would be equivalent to Eq. (B.5) and the measured stress would be the true SiGe film stress. However, the backside oxide absorbs moisture and becomes more compressive; whereas the stress of the frontside oxide is constant under the SiGe cap. Therefore, the measured stress is not the true stress of SiGe film once the backside oxide starts to absorb moisture. An apparent stress drift of the SiGe film is observed and qualitative agreement with the oxide stress change is found.

B.4 Summary

The average residual stresses of poly-Si, poly-SiGe, poly-Ge, and thermal oxide are stable in ambient conditions. Poly-SiGe remains a promising material for modular MEMS integration. LPCVD and TEOS-based PECVD oxides absorb moisture and become more compressive in a humid environment. Due to their high deposition rates, LPCVD and PECVD oxides are often used as MEMS sacrificial layers. However, the

stress drift in sacrificial materials is not anticipated to affect the mechanical properties of

the MEMS structure layers since they are eventually removed.

References

[B.1] B. L. Bircumshaw, M. L. Wasilik, E. B. Kim, Y. R. Su, H. Takeuchi, C. W. Low, A. P. Pisano, T.-J. King and R. T. Howe, "Hydrogen peroxide etching and stability of p-type poly-SiGe films," *17ᵗʰ IEEE Micro Electro Mechanical Systems Conference (MEMS-04)*, Maastricht, The Netherlands, Jan. 25-29, 2004, pp. 514-519

[B.2] C. W. Low, B. L. Bircumshaw, T. Dorofeeva, G. Solomon, T.-J. King and R. T. Howe, "Stress stability of poly-SiGe and various oxide films in humid environments", *Proceedings of Stability of Thin Films and Nanostructures Symposium*, Materials Research Society, Boston, MA, Nov. 29 – Dec. 3, 2004

[B.3] G. Stoney, "The tension of metallic films deposited by electrolysis", *Proc. Roy. Soc London*, vol. A82, pp. 172, 1909

[B.4] M. Madou, *Fundamentals of Microfabrication*, 2ⁿᵈ ed, CRC Press, p. 302, 2002

[B.5] K. Kwok E. Yieh, S. Robles and B. C. Nguyen, "Surface related phenomena in integrated PECVD/Ozone-TEOS SACVD processes for sub-half micro gap fill: electrostatic effects", *J. Electrochem. Soc.*, vol. 141(8), pp. 2172-2177, 1994

[B.6] S. D. Senturia, *Microsystem Design*, Kluwer Academic Publishers, pp. 201-238, 2001

Appendix C: Tystar20 Logbook

The process logbook was created to monitor the LPCVD reactor (Tystar20) and sustain the process. This logbook is a more comprehensive summary of the reactor than the Microlab wand system record, as it includes the process conditions, fault reports, and maintenance comments. Users are required to enter pre-deposition standby conditions, deposition recipe, and process comments in the Wand system. Standby information including temperature, process pressure (PRCPR), N2dope flow rate, and injector pressure, are recorded at the standby mode to track the injector condition. Deposition information includes temperature, pressure, gas flow rates, and deposition time. Users can also enter observations in the comment log. When there is a problem with the reactor, users enter a fault report in the Wand system and equipment staff will diagnose the problem and enter a maintenance comment. Failure analysis, design improvement and new process qualification have been studied based on the historical information of the reactor recorded in the logbook.

It should be noted that process conditions entered by users are sometimes incomplete and contradictory. Some of the process conditions can be retrieved in the furnace condition recording computer inside the Microlab. However, the computer does not have a record of the injector pressure, and the injector condition has to be monitored manually.

User	Date	Standby conditions				Deposition conditions								Comments
		Standby Temp (°C)	Standby inj. Press (Torr)	Standby PRCPR (mTorr)	Standby N2DOPE	Inj. Press (Torr)	Time (min)	Temp (°C)	SiH4 (sccm)	B2H6 (sccm)	BCl3 (sccm)	GeH4 (sccm)	Press (mTorr)	
Wehrby	12/5/2002	New injector												
christop	12/6/2002		6.46	102		3.9	670	425	100	60		60	400	Uniform +-2.5%, 10 Ohm/sq
christop	12/6/2002					4	270	425	100	60		58	400	uniform
meyoum	12/11/2002					3.~	60	450	100	60		60	600	Low R, uniform
meyoum	12/13/2002					3.~	60	450	100	60		60	600	Low R, uniform
christop	12/16/2002													
Christop	12/17/2002		6.7			4.6	240	425	100	60		58	400	uniform
Wehrby	12/18/2002	New injector												
Christop	12/18/2002					3.5	30	425	100	60		58	400	coat
Christop	12/19/2002													
Blakelm	1/6/2003		7.53			3.77	67	425	100	60		70	300	
							159	425	100	60		58	600	
Blakelm	1/7/2003		7.38				134	425	100	60		70	300	
Blakelm	1/8/2003		6.68	101	89.5		201	425	100	60		70	300	
							53	425	100	60		58	600	
blakelm	1/10/2003		7.08	101	89.5		171	450	100	60		70	400	
Sny	1/16/2003		7.26	102	89.3	4.82-4.85	90	450	100	60		58	600	
Wlow	1/16/2003					4.7	150	400	100	60		25	800	uniform
Wlow	1/17/2003					6.29	30	450	85	80		90	600	Not uniform, more than 100% resistance variation
Wehrby	1/17/2003	GeH4 tank changed												
Takeuchi	1/21/2003													

High injector pressure during depo. Resistivity 10X higher, also cross wafer variation. Change injector

User	Date													Comments
Wehrby	1/22/2003					4.13	150	400	100	60		50	800	uniform
Wlow	1/22/2003					5.08	30	450	85	80		90	600	Bottom part of wafer has 40% less resistivity
Wlow	1/22/2003					4.4		450	100	60		60	600	
Meyoum	1/22/2003													—

bob 1/24/2003: Tyeda20's process tube cracked yesterday. Not unexpected given the hours of deposition. We will rebuild the tube with new quartzware which we modified. We have increased the length of our liner and decreased the diameter of our heat baffles to protect the area where failure have occurred. This, to extend quartzware life time. These mods should have no effect on overall dimensions or the placement of the injector and should not effect process and do not present a delay. The tube was used for 731 hours during the period 07/25/02 through 01/27/03. New tube, new injector, new liner.

User	Date													Comments
Wehrby	1/27/2003	New injector												
Challew2	1/28/2003													

Hazy film and high resistivity. Change injector

User	Date													Comments
Meyoum	2/2/2003					~10.9	60	450	100	60		60	600	Film is very hazy and non-uniform. Resistivity is high
Meyoum	2/2/2003						60	450	100	60		60	600	
meyoum	2/2/2003						60	450	100	60		60	600	
Wehrby	2/4/2003													Calibration from 300-500C
Jimmywgmc	2/5/2003													
Yanw	2/6/2003													
Blakelm	2/6/2003		6.~	101	89.3	4.~	150	400	100	60		30	600	Good amorphous film

Name	Date	Temp	V1	V2	V3	V4	V5	V6	V7	V8	V9	V10	Comments
Blakeln	2/7/2003		6.83	101	89.3	3.95	210	380	100	60	30	600	Good amorphous film
Blakeln	2/9/2003		7.08	101	89.5	4.22	210	390	100	60	30	600	Good amorphous film
Wlow	2/11/2003		7.35	101	89.5	4.54	150	400	100	60	50	800	Very uniform film 1% R variation across wafer load
Wlow	2/12/2003		8.06	102 (after)	89.3	4.64 --	120	425	100	60	50	800	Uniform looking, 15% R variation
Megumn	2/13/2003		11.2 (?)			5.3		450	100	100	100	600	
Blakeln	2/19/2003		7.82	101	89.4	4.81	150	395	100	60	30	600	No standby between runs
Blakeln	2/19/2003		N/A	N/A	N/A		150	395	100	60	30	600	
Blakeln	2/21/2003		8.23	101	89.5	5.22	150	425	100	60	70	400	No standby between runs.
Blakeln	2/21/2003		N/A	N/A	N/A	5.46	53	425	100	60	58	600	New genus recipe, process aborted during depo. Ge tank empty.
Megumn	2/22/2003		8.7				60	450	60	60	60	600	
Wehrby	2/24/2003												Replace Ge tank.
Megumn	2/24/2003					5.9	60	450	100	60	60	600	Film is uniform, but resistivity is very high
Equavy	2/25/2003		8.93	101	89.2	0.2	300	425	0	0	0	400	Annealing run
wlow	2/26/2003												I was performing a pressure/flow test through the injector when the screen locked up.
Wehrby	2/27/2003												Reset tystar20 computer.
Suy	2/27/2003					3.~	410	425	100	20	70	400	B2H6 tank pressure down to 4.5 psi
Megumn	2/28/2003					N/A	35	375	0	0	120	600	First wafer is fine, and second is not uniform. Event out of standby recipe!
bob	3/3/2003												New door, new injector, new B2H6 tank. New gas flange has been installed. Base pressure and rate of rise are good.
Wehrby	3/4/2003												Replace B2H6 tank.
Equavy	3/5/2003		6.35	101	89.4	0.2	300	400	0	0	0	400	Thermal annealing
Wlow	3/6/2003	350.1	6.35	101	89.3	6.89	150	400	100	60	25	800	Same recipe as before, wafers changes from amorphous
Megumn	3/6/2003	350	6.9			3.99	50	325	100	60	60	600	
suy	3/7/2003	350	6.53	101	89.3	4.09	60	450	100	60	58	600	Adjusted boat position. Load boat 15.5 in, Center boat: 24 in, Pump boat 32.875 in to the back of the door
Wlow	3/7/2003	372.5		101	89.3		150	400	100	60	25	800	Injector pressure gauge got turned off
Wehrby	3/10/2003												Found power strip power on bottom failed. Replaced power strip with spare.
Megumn	3/11/2003				89		30	400	0	60	180	300	
Zaiya	3/11/2003												
Zaiya	3/12/2003				0.4	4.8			100, 650m	60, 650m	48, 650m	400, 650m	
Suy	3/13/2003	350	7.87	101	89.8	5.29	60	450	100	60	58	600	
Zaiya	3/14/2003					5.78			100	60	25	600	
Zaiya	3/16/2003					3.6			100	20	25	400	
Zaiya	3/18/2003												Found tystar20 in manual mode. Placed tystar20 in auto mode.
Wehrby	3/18/2003												Replaced quartz tube and liner (enabled 176 hr, 53 hr of depo). Installed new TC sheath and split TC cover. Base pressure 12 mT. Rate of rise 15 mT/min cold. Have turned on the firing circuit. After reaching operating temp, base pressure is 7 mT, and rate of rise is 6 mT/min.
Suy	3/18/2003												Quartz tube is broken, did not do a run
Wehrby	4/3/2003												1st time: process stopped because SiH4 low (actually not true) 2nd time: stopped at DTEQ. Cannot run boatout recipe

Name	Date												Notes
Wehrly	4/3/2003												New injector
Wlow	4/3/2003	349.5	6.67	101	89.5	4.12	150	400	100	60	50	800	No standby between runs.
Meyoum	4/3/2003					4.11	60	400	100	60	60	600	
Yanw	4/3/2003						660	375	0	0	105	600	Nucleation: 450C, 300mT, 180 sccm disilane, 2min
Blakeim	4/4/2003		7.09	101	89.3		150	425	100	60	70	400	
Meyoum	4/4/2003												
Meyoum	4/5/2003					4.41	60	425	100	60	60	600	
lingang	4/5/2003						140	425	100	60	65	400	
Wehrly	4/8/2003												Replace Ge tank.
Meyoum	4/8/2003		7.9	100	89	4.47		425	0	60	200	900	Sheet resistance 1.6 Ohm/sq
Wlow	4/9/2003	350.9	7.96	101	89.6	5.05	150	400	100	60	35	600	Resistivity not uniform. Could be at transition of amorphous and poly.
even	4/10/2003												Pump got shut off. Process aborted to hold step. Pump control switch hits cooling H2O pipe when access door is open. Pump restarted OK.
wlow	4/11/2003	346	8.14	101	90	5.15	150	400	100	60	35	800	
Lingang	4/13/2003	350	8.01	101			350	350	0	0	200	300	Standard nucleation
Zhijn	4/22/2003												
Lingang	4/22/2003	355	9.24	101		5.13		350	0	60	200	300	
Wehrly	4/29/2003												Injector pressure 9.2. New injector
Zhijn	4/30/2003					4.16	350		200	0	20	400	
Zhijn	4/30/2003						350		100		85	400	
Lingang	4/30/2003	350	12.06	101	89.5								Requested injector change, didn't do a run
Wehrly	5/1/2003												Injector pressure 12.09. New injector
Wlow	5/1/2003	371	6.63	102	89.9	3.9	150	400	100	60	50	800	No standby between runs.
Jimmygmac	5/2/2003												Calibration from 300-450C and 300C
Lingang	5/3/2003	330	6.8	101	90	4.3	280	425	100	60	65	400	
Lingang	5/3/2003						280	425	100	60	65	400	No standby between runs.
yanw	5/3/2003												The computer locked up when inputting parameters. Have reset tystar20 and reloaded configuration and standby recipe.
Wehrly	5/5/2003												
Suy	5/5/2003		7.24	101	90	4.34	60	425	100	60	70	400	6" wafer
Lingang	5/6/2003	330	7.6	101	90		60	300	100	60	200	300	Depo in't press. not recorded
Wehrly	5/13/2003												Found TCU green light out and red light on. Tried to reset several times and clean edge card connector and tighten up chips and it was still on. Replace TCU broad with spare. TCU now functions OK. Center zone profile TC signal reads 2.7 mvolts with a voltmeter which is about 270 C, but when it is plugged in reads 32 C. Have removed center TC and tystar20 now read normal and controlling. Have started calibration.
Wehrly	5/14/2003												Rematched DCS-30 to FCS 10 temps. Reset TCU and reset software reset Temps now read normal and controlling. Have started calibration.
wehrly	5/15/2003												Replace Germane tank.
Jimmygmac	5/16/2003												Temp calibration finished at 300 C.
Yanw	5/18/2003	300	7.21	101	89.7	0.21	600	375	0	0	105	600	Nucleation temp 400, Si2H6 180, pre 300, time 10 min
Jimmygmac	5/20/2003												calibration
Blakeim	5/20/2003		7.41	101	89.5	4.56	150	425	100	60	70	400	

Name	Date												Comments
Blakelin	5/20/2003						50	425	100	60	58	600	No standby between runs.
Linquag	5/21/2003	350	7.46	101	90		4	300	0	60	200	300	
Equerv	5/23/2003		7.7	102	90		120	350	0	60	200	300	Expected thickness 2 um.
Linquag	5/28/2003		7.7	101	89		290	425	100	60	65	400	
Equerv	5/29/2003							350	0	60	200	300	Can't record standby parameters.
Equerv	6/5/2003		8.32	102	89.9		120	350	0	60	200	300	Nucleation 5 min
bob	6/11/2003												The IH80 mechanical pump has frozen. Protocols from Edwards for rotating the pump did not free the rotor. The pump will be replaced.
Webcity	6/13/2003												Replace mechanical pump. Pumped down to base pressure of 11 mT, rate of rise is 12 mT/min.
jimmyspmc	6/16/2003												
Msyoum	6/16/2003							350	0	60	200	300	Furnace stated at idle mode, no standby info.
Blakelin	6/17/2003	350	8.46	112	89.4	4.8		425	100	60	70	400	No standby between runs.
Blakelin	6/17/2003						330	425	100	60	58	600	
Wlow	6/17/2003	444	11.52	101	90	6.54	150	400	100	60	35	800	Ge tank empty, process aborted.
Mcnail	6/18/2003												Replace Ge tank.
Blakelin	6/18/2003												The 6" boat has lots of particles.
Webcity	6/20/2003												New injector
wlow	6/20/2003												Ran the standby recipe for Si2H6 coating, and found the furnace in special hold mode. Checked history, the process failed the leak check.
bob	6/20/2003												Tystar20 has been taken out of "special hold" and returned to service.
Msyoum	6/23/2003												Found furnace in idle mode, no standby info. (enabled 277 hr, 83 hr of depo)
Webcity	6/30/2003												Change quartz tube. Quartz tube broken
wlow	7/2/2003												Cannot run recipe. Error message "MFS/TCU I/O error or manual mode prevents run".
Webcity	7/2/2003												Found 460 #2 in manual. Placed it into auto. Started standby recipe.
Wlow	7/2/2003	441.2	7.75	112	89.8	4.43	60	450	100	60	58	600	Excellent uniformity
Wlow	7/3/2003	347.6	7.25	112	89.3	4.40-4.50	280	425	100	60	65	400	Very uniform
Wlow	7/6/2003	350.3	7.63	112	89	4.55	150	400	100	60	35	800	No standby between runs. Cloudy wafer, resistivity not uniform.
Wlow	7/16/2003					4.71	150	400	100	60	25	800	No standby between runs. Resistivity has 10% variation across wafer, seand 40% variation across load
Wlow	7/16/2003					5.6	30	450	85	80	90	600	Resistivity not uniform, varies in 4 orders of magnitude.
Wlow	7/7/2003	350	8.07	112	89.3	4.85-4.91	150	400	100	60	35	800	No standby between runs. 10% variation in resistivity.
Wlow	7/7/2003					5.00-5.07	150	400	100	60	25	800	No standby between runs. Resistivity 100x higher than expect. 100% variation across wafer.
Wlow	7/7/2003					5.20-5.23	150	400	100	60	35	800	
Nmpc	7/8/2003							450	100	60	60	600	Film is cloudy
Msyoum	7/8/2003					5.3-5.8	240	425	100	60	60	400	No standby info between runs. Film has uniform resistivity. Process aborted during APBG.
Wlow	7/9/2003	348	9.5	112	89.3								Furnace monitoring, didn't do a run. Request injector change.
Webcity	7/11/2003												Get cloudy film. New injector

Name	Date												Comments
Wlow	7/13/2003	352.2	7.02	112	89.2		450	30	85	80	90	600	Forgot to check injector pressure during depo. Resistivity 4X higher than expect. 20% variation across
Wlow	7/13/2003					4.39-4.41	400	150	100	60	40	800	No standby between runs. Film has excellent uniformity in R. 2% variation across wafer and load.
Wlow	7/13/2003					4.38-4.44	400	150	100	60	40	400	No standby between runs. 10% R variation across wafer, 40% across load.
Wlow	7/14/2003	348.2	7.31	112	89.3	4.41-4.54	400	60	100	60	25	800	Film is cloudy. R varies in orders of magnitude.
Wlow	7/14/2003					4.55-4.61	400	90	100	60	25	800	No standby between runs. Film is cloudy.
Wlow	7/16/2003	349.5	7.40	112	89.4	4.62-4.68	400	150	100	60	50	800	Good film. 5% variation of R.
Shlim	7/17/2003	347.8	7.63	112	89		350	240	0	0	200	300	Cloudy film
shlim	7/18/2003	344.8	7.63	112	89.4		350	240	200 (disilane)	0	200	300	Good film
Wlow	7/18/2003	445.5	8.43	114	89.2	4.73	400	150	100	60	50	800	Good film 5% variation in R
equevy	7/20/2003		7.87	112	89.6		350	120		60	200	300	Nucleation 400C, 300mT, Si2H6 200, 5 min. Expect 2 run film
Equevy Wehrly	7/23/2003	Ge tank changed.											Abort. Ge tank empty
Wlow	7/24/2003	351.9	8.01	112	89.6	5.01	400	150	100	60	50	800	Excellent film
Wlow	7/24/2003					5.10-5.26	400	210	100	60	50	800	No standby between runs. Excellent film.
Megeum	7/25/2003	450	9.4		90		350	120		60	200	300	Nucleation 350C, SiH4 200, 300 mT, 5 min. Abort during nucleation step.
megeum	7/25/2003					4.8	350	150		60	200	300	No standby info. Nucleation 350C, SiH4 200, 300 mT, 5 min.
Jimmygemac	7/26/2003	Modify recipe and now users can deposit polysilicon in tystar20. Phosphine is the dopend gas.											
Bean	7/26/2003												No standby info. "buffer queue error" during backfill of boat but Jimmy thought computer crashed
Bean	7/28/2003						500						No standby info. Abort after 6 hr of depo. Jimmy found SiH4 out of tolerance.
Bean	7/29/2003						500						Jimmy restarted the process and let it run for the remaining time. 7 hr total.
Bean	7/30/2003						615				0	375	No standby info. Abort after 45 min of depo. Failed 3 times after about 20 mins etch due to low phosphine flow. The film looks metallic as expected.
Jimmygemac	7/30/2003	Tried to restart Bean's process. The process aborted due to PH3 low alarm after 15-20 mins depo. Move recipe to wait step. Will check PH3 line. Tystar20 is down for doped poly process.											
Wehrly	7/30/2003	E2H6 tank changed.											
Wehrly	7/30/2003	Replaced quartz cover for the lifting fork.											
ehhim	7/31/2003	8.96					450		100	60	58	600	

220

Name	Date												Notes
Equavy	7/31/2003						120	350	0	60	200	300	Event out of standby, nucleation, 400C, 300mT, Si2H6 200, 5 min, expect 2um film, film looks beautiful
Equavy	8/3/2003		9.24	112	89.6		140	425	100	60	60	400	nucleation, 400C, 300mT, Si2H6 200, 10 min, expect 2 um film, film looks beautiful
Equavy	8/4/2003		9.3	111	89.6	6.09		425	100	60	60	400	nucleation, 400C, 300mT, Si2H6 200, 10 min, film looks a bit cloudy. This run has very different Ge content with the previous.
bob	8/6/2003												Tested GeH4 MFC. 0-200: 25 sccm = 58 mT, 50 sccm = 90 mT, 100 sccm = 140 mT. 0-50: 25 sccm = 61 mT, 50 sccm = 94 mT.
Prof. King	8/6/2003												Standard SiGe process is down due to phosphorus out-diffusion by the doped poly process. Dope poly process should be shut off.
jimmygme	8/6/2003												get rid off poly-Si recipe
Wehdy	8/11/2003												New injector
wlow	8/11/2003	435	7.44	114	89.7	4.33-4.38	90	450	100	60	58	600	Standard MEMS exchange recipe for monitoring the furnace. Eight wafers were placed at the two ends (skipping the first slot) of both 4" boats. Resistivity is pretty much uniform. 20% variation across wafer. 5% variation across metal boat to the load side of the non-metal boat. The pump side of the non-metal boat has significantly low R, half of other wafers
bob	8/12/2003												There is a ~5% discrepancy between the 0-50 and 0-200 sccm Germane MFC at 50 sccm. A freely calibrated 0-200 sccm MFC has been procured and will be installed to fix this
wehdy	8/13/2003												Changed GeH4 into lo and hi MFCs. GeH4LO 25sccm=61mtorr, 50sccm=94mtorr, GeH4HI 25sccm=56mtorr, 50sccm=85mtorr, 100sccm=130mtorr.
takeuchi	8/15/2003												recipe aborted due to GeH4Hi leak
bob	8/19/2003												The recipe switch for delivery of dopant gases to the gas ring and injector is not working. Dopant can only be delivered to the injector. Jimm has traced the failure to one of the pneumatic valves that switch gas delivery.
wehdy	8/20/2003												Changed GeH4HI MFC. Flowed GeH4 into lo and hi MFCs. GeH4LO 25sccm=61mtorr, 50sccm=94mtorr, GeH4HI 25sccm=61mtorr, 50sccm=144mtorr.
Wehdy	8/20/2003												Changed out tumpo on injector line. PH3 now flows.
wehdy	8/21/2003		7.21	112	89.8	4.26	280	425	100	60	60	400	nucleation, 400C, 300mT, Si2H6 200, 10 min, expect 2.2 um
equavy	8/21/2003												Changed quartzware (not broken, enabled 213 hr, 66 hr of depo), new tube new cantilever sheaths and gas ring. Reinstalled rear piping, TC, heat baffles and boats. Lowest base pressure is 22 mT. Rate of rise is 35 mT/min.
Wehdy	9/5/2003												Base pressure is at 11 mT and rate of rise is at 15 mT/min cold. Have turned on the firing circuits.
Wehdy	9/8/2003												Pump is making noise. It has been checked out and filled with Di water and oil. Base pressure and rate of rise are good. Tystar20 needs dummy wafers and a coating run.
Wehdy	9/8/2003												Performing dummies maintenance.
Wehdy	9/9/2003												Maintenance: Coating run has been completed. Calibration has been completed at 400, 450, 500 C.
Wehdy	9/10/2003					4.22	60	350	0	60	200	300	nucleation, 400C, 300mT, Si2H6 200, 5 min
equavy	9/10/2003												The 2 wafers placed on the load side of the load boat (1 dummy in) were a little cloudy and there was a dark spot (unclouded on the lower quadrant closest to me as
ehkim	9/11/2003	350	7.99	107	89.8	4.65	60	425	100	60	60	400	nucleation, 400C, 300mT, Si2H6 200, 5 min
gruidai	9/12/2003					4.71	60	425	100	60	60	400	nucleation, 400C, 300mT, Si2H6 200, 5 min

Name	Date													Comments
suy	9/15/2003	350	7.72	110	89.7	0.16	60	425	100	0	100	60	400	film looked real bad, parts fot he wafer are very dull, only the center looks shiny, perhaps reaction was really
Meyoum	9/16/2003					4.8	90	425	100	60	100	60	600	trilayer
Meyoum	9/16/2003						120	350	0	60			300	
Meyoum	9/16/2003						90	425	100	60			600	
Meyoum	9/17/2003	350	10.9		89.9	5.14	90	425	100	60	100	60	600	trilayer
Meyoum	9/17/2003						120	350	0	60			300	
Meyoum	9/17/2003					5.16	90	425	100	60			600	
suy	9/18/2003	350	8.24	111	90.2	0.03	60	425	100	0	100	60	300	Nucleation 400C, 300mT, Si2H6 100, 20 min. The center boat cover has a ~1" long piece broken off.
Wehrly	9/19/2003	Injector pressure near 9. New injector												
wbw	9/21/2003	330	6.06	111	90.1	3.65	330	400	100	60	100	50	800	Inucleation, 400C, 300 mT, Si2H6 200, 5 min.
Wehrly	9/22/2003	Replace broken boat cover.												
Wehrly	9/22/2003	GeH4 tank change.												
wbw	9/22/2003	330	6.17	110	89.9	3.74-3.91	440	400	100	60	100	50	800	nucleation, 400C, 300 mT, Si2H6 200, 5 min. There is a ring at the bottom of wafers closer to the pump.
ekim	9/23/2003	390	6.64	111	89.6	5.52	30	425	100	0	100	60	400	undoped SiGe with N2Dope flow if 60 through the injector.
chanyj	9/24/2003	330	6.88	112	89.6	0.05	270	450	0	0	0	0	300	nucleation, 450 C, 300 mT, Si2H6 200, 1 min. Didn't realize there is no input for Si2H6 in depo. Ended up Bilayer deposition. Nulceation, 400C, 300mT, Si2H6 200, 5 min. Three wafers are at the center boat. The
wbw	9/24/2003	449	7.06	112	89.6	3.97-4.01	150	400	100	60	100	50	800	one at the load end is fine. The ones in the middle and at the pump end have rings at the bottom again. Injector flow issue.
wbw	9/24/2003					4.03-4.08	150	400	100	60	100	45	800	
marport	9/25/2003	Germane and disilane usage will no longer be logged manually at the end of your runs. The usage will be determined from flow sensors automatically.												
ekbro	9/25/2003	was not able to run the process.												
bnanh	9/25/2003	The injector was cleaned out about a week ago, and there appear to be problems already. Perhaps the injector cleaning/replacement process is not working properly.												
Meyoum	9/25/2003	350	6.5	0	89.6	0.02	120	350	0	0	100	180	300	nucleation, 350 C, 300 mT, Si2H6 100, 10 mins. Film is nice and uniform.
ekim	9/26/2003	Please flow 60 sccm of N2Dope when running undoped poly-SiGe process so that the injector doesn't get clogged. Jimmy wrote up a new recipe especially for this purpose with a variable flow rate input for N2Dope: UDSIGEA.020												
Meyoum	9/26/2003	347	6.67	111	89.9	3.6	120	350	0	60	0	180	300	nucleation, 350 C, 300 mT, Si2H6 100, 10 mins. Film is nice and uniform.
yanw	9/26/2003	445	6.69	111	89.9	0.02	360	375	0	0	0	180	300	nucleation, 400 C, 300 mT, Si2H6 200, 5 mins. Film is nice and uniform.
Meyoum	9/27/2003	350	6.46	112	89.9	3.45	90	425	100	60	100	60	600	nucleation, 425 C, 400 mT, Si2H6 100, 10 mins. Film is nice and uniform.
Blaheim	9/27/2003					4.75	483	425	100	60		65	400	No standby info. Bilayer run.
Blaheim	9/27/2003					4.8	28	425	100	60		58	600	
yanw	9/28/2003	350	7.72	112	90.2	0.03	300	375	0	0	0	180	300	Continue depo. No nucleation. Film looks fine.
Wehrly	9/29/2003	Replace injector due to the ring at the bottom of the wafer.												
Wehrly	9/30/2003	Replace GeH4 tank.												

222

user	date												comments
ehhim	9/30/2003	349	7.52	112	90		30	450	100	60	58	600	Monitor run. LTO wafers are cloudy. Didn't use a nucleation step.
ehhim	9/30/2003	445	7.26	111	89.3		120	425	100	60	60	400	Ran SiGE10la
griddal	10/2/2003	345.6	7.74	112	89.9		60	375			180	300	
suy	10/3/2003	330	7.72	111	89.7		60	350	0		180	300	nucleation, 350C, 300mT, Si2H6 100, 20 min
bob	10/6/2003		4.28							60			The mechanical pump is making noise. Change out the pump. Test the new pump with standby recipe without problem.

Jimmygmac 10/6/2003 Qualified pump.

| suy | 10/9/2003 | 350 | 7.74 | 123 | 89.7 | | 120 | 400 | 90 | 60 | 70 | 400 |

flow test for the new mechanical pump

at standby recipe, step: WAIT, temp = 350 C
manual mode: PRECHH4 = ON
    Gate 196 = ON (although actual status is OFF)
    Gate 197 = ON (although actual status is OFF)
all other gases off

N2VAC	N2DOPE	PRCPR
0	90	123
500	90	464
1000	90	751

N2VAC	N2DOPE	PRCPR
0	0	9
500	0	409
1000	0	701

suy 10/9/2003

N2VAC	N2DOPE	GEH4	PRCPR (didn't check gate valve 196, 197)
0	0	LO25	61
0	0	LO50	94
0	0	HI25	58
0	0	HI50	90

suy	10/9/2003	330	7.98	123	89.7		120	425	110	60	50	400	Nucleation 300 mTorr, 100 Si2H6, 400C, 20 min.
megoruns	10/9/2003		4.54				20	350	0	60	180	300	standby info not available. Nucleation 350C, 15 min, 300 mTorr, 100 Si2H6
suy	10/10/2003	330	8.07	123	89.9		120	425	125	60	35	400	
Wehzhy	10/13/2003	SiH4 tank changed.
suy	10/14/2003	330	8.27	122	89.6		120	425	105	60	55	400	nucleation 375C, 5 min, 300 mTorr, 90 Si2H6
griddal	10/14/2003		5.15				30	375	198	0	181	294	
Wehzhy	10/16/2003	Injector pressure 8.3. New injector
ehhim	10/16/2003	330	6.43	122	89.8		240	425				400	completed MX R1739 - approx. 4 um doped SiGe dep. Completed undoped SiGe multilayer run - recipe

Name	Date	Event												Notes
ehim	10/22/2003		350	6.84	123	89.6	5.84	70	425	110	0	60	425	Use UDSIGEA recipe . 60 sccm of N2 dope flow through injector
Equerv	10/22/2003						3.84	12	350	0	60	200	300	no standby info
wlow	10/22/2003						4.17	90	400	100	60	40	800	No standby info. Nucleation 400C, SiH4 200, 300 mT, 10 min.
gudai	10/23/2003		350		300 (?)	89.9	5.51	60	353.4	0	0	181	300	resistivity of C3 is nice and uniform. 8.8 ohm/sq; resistivity of C8 is not uniform. top 8.8 ohm/sq, bottom 23.6 ohm/sq; resistivity of C13 is worse. top 1.1 ohm/sq, bottom 6.23 ohm/sq
ehim	10/28/2003		330	6.83	123			70	425	90	0	70	400	nucleation 400 C, 10 min, 300 mTorr, 100 Si2H6
Wehrty	11/3/2003	Si2H6 tank changed.												UDSIGEA. Center 4" boat cover broken.
suy	11/6/2003		330	6.77	122	89.7	4.08	120	400	0	60	90	600	nucleation 400C, 20 min, 600 mTorr, 100 Si2H6. Center boat cover was missing, borrowed form trystar19.
Wehrty	11/7/2003	Replaced boat cover.												
Meyoum	11/7/2003						4.25	90	425	100	60	60	400	
Meyoum	11/7/2003							120	350	0	60	180		trilayer
Meyoum	11/7/2003						4.38	90	425	100	60	60	400	
Wehrty	11/10/2003	Ge tank changed.												
Meyoum	11/13/2003		330	7.11	122	89.9	3.73	30	425	100	40	60	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Meyoum	11/14/2003		330	7.19	122	90	3.17	90	425	100	45	60	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Meyoum	11/14/2003						3.87	120	350	0	45	170	300	no standby info
Meyoum	11/14/2003						3.92	90	425	100	45	60	400	no standby info
Meyoum	11/15/2003		330	7.36	122	89.7	0.003	90	425	100	45	60	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Meyoum	11/15/2003						3.8	120	350	0	0	170	300	no standby info
Meyoum	11/15/2003							90	425	100	45	60	400	no standby info
Meyoum	11/20/2003		330	7.61	121	89.9	1.59	40	425	125	10	35	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Meyoum	11/20/2003						2.05	40	425	120	15	40	400	no standby info
Meyoum	11/21/2003						2.036	40	425	120	20	40	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Meyoum	11/21/2003						2.36	40	425	110	25	40	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
wlow	12/2/2003		346.2	7.7	122	89.9	0.1	60	425	100	0	55	400	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
wlow	12/2/2003		336	7.86	122	89.7	0.1	80	425	100	0	42	400	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
wlow	12/2/2003		454.5	8.59	123	90.1	0.1	100	425	100	0	30	400	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
chengj	12/3/2003		330	7.8	120	90	6.22	180	450	180 (Si2H6)	0	0	300	amorphous Si
Meyoum	12/8/2003		330	7.65	121	90.2	2.45	110	425	115	20	45	400	Nucleation 425C, Si2H6 100, 400 mT, 10 min.
Meyoum	12/8/2003						3.65	240	350	0	45	170	300	no standby info
chengj	12/9/2003		330	7.74	118	90	7.46	180	450	150 (Si2H6)	20	0	300	amorphous Si
chengj	12/9/2003		330	7.74	118	90	7.8	270	450	149 (Si2H6)	19.7	0	300	amorphous Si

224

Name	Date												Notes
Meyoum	12/11/2003	350	8.76	118	90.2		100	425	115	15	45	400	Nucleation 425C, Si2H6 100, 400 mT, 10 min.
Meyoum	12/11/2003					5.29	120	425	110	60	45	400	Nucleation 425C, Si2H6 100, 400 mT, 10 min.
Meyoum	12/12/2003					3.29	40	425	100	15	45	400	no standby info
Meyoum	12/12/2003	350	8.68	122	92.3		120	425	115	15	45	400	Nucleation 425C, Si2H6 100, 400 mT, 10 min.
Meyoum	12/13/2003					3.39	30	350	0	30	170	300	Nucleation 350C, Si2H6 100, 300 mT, 15 min.
Meyoum	12/13/2003	350	7.8	120	90	2.18	30	350	0	0	170	300	Nucleation 350C, Si2H6 100, 300 mT, 15 min.
chanyj	12/15/2003					5.65	70	450	100	59.4	50.2	800	Nucleation 450C, Si2H6 150, 300 mT, 2 min.
Wendy	12/17/2003												New injector
suy	12/18/2003	350	6.29	118	89.7	3.sth	90	450	100	58	60	600	Monitor run.
wbw	12/19/2003	353.4	6.33	118	89	3.81	170	450	100	60	70	400	Nucleation 450C, Si2H6 100, 300 mT, 10 min. Bi-layer
wbw	12/19/2003						80	425	100	60	58	600	Nucleation 450C, Si2H6 150, 300 mT, 2 min.
chanyj	12/20/2003	350	6.6	118	90	3.73	60	450	110	50	40	850	Nucleation 400C, Si2H6 100, 300 mT, 15 min. Bi-layer.
bob	12/22/2003												GeH4 tank change.
wbw	12/23/2003	349.2	6.64	118	89.7	3.99-4.05	150	400	100	60	50	800	Nucleation 450C, Si2H6 150, 300 mT, 2 min.
wbw	12/23/2003					4.10-4.16	120	400	100	60	37	800	No standby info. Nucleation 425C, Si2h6 100, 300 mT, 15 min. Bi-layer.
chanyj	12/27/2003	350	6.73	117	89.6	4	90	450	100	60	50	800	Nucleation 400C, Si2H6 100, 300 mT, 20 min. Didn't get GeH4 charged.
wbw	12/27/2003					4.22-4.29	150	425	100	60	50	800	Nucleation 450C, Si2H6 100, 300 mT, 10 min. Bi-layer. 1st layer depo was set to be 150 min, but recipe aborted at 140 min. Standby info affected by Jimmy's
wbw	12/27/2003					4.33-4.38	120	425	100	60	25	800	Nucleation 450C, Si2H6 100, 300 mT, 10 min. Bi-layer.
chanyj	12/29/2003	330	7.23	118	89.9	0.43	180	400	105	0	45	800	Nucleation 425C, Si2H6 100, 300 mT, 15 min. Saw "power fail" alarm and cleared. Cantilever cracked when door opens. Wafer boat got stuck inside the tube.
jimmyepmac	12/30/2003												Check GeH4 flow manually. OK.
wbw	12/30/2003	346	7.29	2	0	4.5-4.54	140	450	100	60	70	400	
wbw	12/30/2003	346.5	7.53	118	89.9	4.54-4.59	120	425	100	60	50	600	
wbw	1/1/2004					4.8-4.86	140	450	100	60	70	400	
wbw	1/1/2004					4.8-4.92	120	425	100	60	50	600	
wbw	1/4/2004	349.4	7.75	118	89.7	4.77-4.9	130	425	100	60	58	400	
wbw	1/4/2004					4.77-4.91	150	400	100	60	35	800	
Wendy	1/5/2004												Adjusted cantilever section. Closed tystar20. Tystar20 is ready for wafer removal
Jimmyepmac	1/6/2004												Checked "Power fail" error. There might be a power glitch for the building. If it was less than 3 sec, the process will resume.
Wendy	1/7/2004												Standby injector pressure 8.1. Replaced injector.
chanyj	1/7/2004	330	6.88	120	90	0.35	190	410	105	0	45	800	Nucleation 400C, Si2H6 100, 300 mT, 20 min. Didn't get GeH4 charged again.
suy	1/12/2004	330	7.02	121	90	4.45	90	450	100	60	58	600	Monitor run.
Meyoum	1/12/2004					4.32	120	425	115	60	45	400	No standby info. Nucleation 425C, Si2H6 100, 400 mT, 10 min.
wbw	1/13/2003	352.1	7.23	118	89.9	4.37-4.45	150	425	100	60	55	400	
wbw	1/13/2004					4.48-4.55	180	400	100	60	40	800	Nucleation 425C, Si2H6 100, 300 mT, 15 min. Bi-layer. "Power fail" happened a few times, always after I
Wendy	1/13/2004												Checked connections on back of CPU board and they were tight. Probably what is happening is that the keyboard when pressed is giving a bounce on a certain key combination making the CPU think it had a power fail.

Name	Date												Notes
Equavv	1/15/2004	350	7.59	118	90.1		60	350	0	60	180	300	Nucleation 400C, Si2H6 200, 300 mT, 1 min.
Meynum	1/16/2004	350	7.57	117	89.9	4.77	120	425	100	60	60	400	Nucleation 400C, Si2H6 100, 400 mT, 10 min.
Wehdiv	1/22/2004	New injector											
wlnw	1/27/2004	350.1	7.02	121	90	4.39-4.44	90	450	100	58	60	600	Monitor run. Didn't get charge for GeH4.
wlnw	1/27/2004	Standby recipe got aborted during Si2H6 coating. Error message "TF N2DOPE NE".											
memport	1/28/2004	Check GeH4 charging program. Charge showed up after one day.											
Jimmygrmc	1/28/2004	Found N2dope MFC drifted ~4 sccm. When asked for 10 sccm during coat step, it flows ~14 sccm. When shut off, it reads 4 sccm.											
Wehdiv	1/28/2004	Diagnosed N2dope MFC, could not find any fault. Adjusted N2 pressure at top of TB5 and MFC now reshifting from 9.8 to 9.9 with 10 sccm for five minutes. Set N2dope to 90 and reading was 89.9 to 89.8 for five minutes. Tpster20 is up and ready for use.											
Meynum	1/28/2004	350	7.08	121	89.7		120			15	40		Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Wehdiv	2/2/2004	Germane tank changed.											
Equavv	2/5/2004	350	7.13	121	89.9	3.95	60	350	0	60	180	300	Nucleation 400C, Si2H6 200, 300 mT, 5 min.
chenyj	2/6/2004	330	7.17	121	90	3	120	400	105	15	45	800	Nucleation 400C, Si2H6 100, 300 mT, 15 min. Didn't get charge for GeH4 usage. Film looks hazy.
chenyj	2/7/2004	330	7.12	120	90	1.48	180	400	105	10	45	800	Nucleation 400C, Si2H6 100, 300 mT, 20 min.
memport	2/9/2004	RUMS shows no GeH4 usage on 2/6/2004. The control software has been changed so that GeH4 and Si2H6 usage is always displayed.											
wlnw	2/10/2004	Check chenyj's run on 2/6/2004 on Jon Goldman. GeH4 flow was 45 sccm. Jon Goldman doesn't match with RUMS?!											
Wehdiv	2/12/2004	Install BCl3 (0.1% in He) tank.											
wlnw	2/13/2004	PH3 line has been replaced with BCl3 line. BCl3 comes in through gas ring. We are using the old PH3 MFC. 1 sccm of PH3 = 1.836 sccm of BCl3. Rear injector is removed for initial test. A dummy stainless steel injector is installed in the front to check corrosion and iron combination due to possible reaction with Cl2 and HCl. This injector is not hooked up. Changed out caged boat (7.3", 7.3" and 7.8" in length, 1.5 wafers) to open boat. Two 4" and two 6". Each boat is 4.7" in length, holds 13 wafers. The front of the 1st 4" boat is 20.5" away front inner side of front door. 1st 6" is 30.5" away from door.											
wlnw	2/13/2004						120	425	100	18.4	50	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. Coating run. No standby info. Heat baffle got dark after this run.
wlnw	2/13/2004	Recipe cannot pass leak check. Quartz tube broken. (enabled 551 hr, 194 hr of depo).											
Wehdiv	2/18/2004	Replace quartz tube, liner, TC sheath, split TC sheath, cantilever sheaths. Base pressure 5 mT, rate of rise 4 mT/min.											
wlnw	2/19/2004	The front of the 1st 4" boat is 22.5" away front inner side of front door. 1st 6" is 32" away from door.											
wlnw	2/19/2004						120	425	100	18.4	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. Coating run. No standby info. Heat baffle look fine.
wlnw	2/19/2004	348	off	0	0	NA	60	425	100	9.2	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. BC13 test.
wlnw	2/20/2004	454.2	off	0	0	NA	60	400	100	9.2	60	400	Nucleation 400C, Si2H6 100, 300 mT, 15 min. BC13 test.
wlnw	2/20/2004	353	off	0	0	NA	60	450	100	9.2	60	400	Nucleation 450C, Si2H6 100, 300 mT, 15 min. BC13 test.
wlnw	2/24/2004	350.3	off	0	0	NA	60	400	100	16.5	60	400	Nucleation 400C, Si2H6 100, 300 mT, 15 min. BC13 test.
wlnw	2/24/2004	452.3	off	0	0	NA	60	425	100	16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. BC13 test.

226

User	Date												Notes
wlow	2/24/2004	350.3	off	0	0	NA	60	400	100	16.5	60	400	Si2H6 nucleation 400C, Si2H6 100, 300 mT, 15 min. Ge nucleation: 350C, GeH4 100, BCl3 16.5, 300 mT,
jimmywgmac	2/26/2004												Perform clean maintenance after Microlab power failure last night.
Wehdy	3/3/2004												Remove fixed injector and bagged it. Install clean near injector on tystar20.
bob	3/4/2004												Yijian reported hazry film produce by tystar19, could not reproduce good result with the same recipe. B2H6 cylinder is 8 months old and ~100 psi. It began at 1800 psi. We know there is some decay of B2H6 and a recently replaced quartz injector in tystar19 clogged prematurely. B2H6 tank will be changed every 6 months.
Wehdy	3/5/2004												Replace B2H6 tank
srw	3/9/2004	350	9.61	283	89.4	1.89	210	425	115	15	45	400	Nucleation 400 C, Si2H6 100, 400 mT, 15 min. Sheet resistance from 4ptprb: I=0.453mA, V=1.15-3. From the SEM, film thickness 1.15 um (too thick), and surface is quite rough.
changj	3/11/2004	330	9.58	284	89.3	2.06	180	400	105	20	45	400	There are lots of particles in the furnace after the power failure on Feb. 25, 2004. Cannot do anything to the tube. The best we can do is to clear all the dummies.
wlow	3/12/2004												After restoring the B2H6 process with a newly clear injector, injector pressure is found to be very high (9.5 Torr), PRCPR is also high (283 mTorr). This is because the standby recipe got changed during the BCl3 experiment. There is 200 sccm of N2BKFL during standby in addition to 90 sccm of N2DOPE. Jimmy will get rid of the N2BKFL and this problem can be cleaned.
wlow	3/12/2004	352	9.84	288	90	NA	10	450	200	18	60	800	Nucleation 450C, Si2H6 200, 800 mT, 60 min. Switch to open boat for BCl3 depo. This is a coating run.
wlow	3/12/2004	NA	NA	NA	NA	NA	180	425	20	16.5	12	400	Nucleation 425 C, Si2H6 100, 300 mT, 15 min. BCl3 run, Some particles on wafers.
wlow	3/12/2004												This is a follow up of the particle problem. The dummy wafers were coated with a layer of particles. I trashed all of them. The deposition after the dummy change still has particles, at least 20 particles per wafer are visible. The particles are caused by the power failure a few weeks ago. The furnace had been used after that. However, since most people use caged boat configuration, they don't see significant amount of particles on wafers. I am using open boat for BCl3 doping, and it is a big problem. There should be more particles on the wall. The boats (caged and open), cantilevers, heat baffles should be cleaned as soon as possible. The injector might also need a change. I will do two more not so critical runs to collect more particles to the dummy wafers tomorrow.
wlow	3/12/2004	NA	NA	NA	NA	NA	60	425	100	2	60	400	Nucleation 425 C, Si2H6 100, 300 mT, 15 min. BCl3 run, Some particles on wafers. Forgot to record standby info.
wlow	3/13/2004	NA	NA	NA	NA	NA	60	425	100	16.5	60	400	Nucleation 350 C, Si2H6 150, 300 mT, 20 min. Ge nucleation 100 GeH4, 16.5 BCl3, 300 mT, 10 min. BCl3 run, Some particles on wafers. Forgot to record standby info.
wlow	3/13/2004	450	10.47	289	89.6	NA	60	425	100	6.5	60	400	Nucleation 425 C, Si2H6 100, 300 mT, 15 min. BCl3 run. Switch back to caged boat after depo.
changj	3/13/2004	330	9.82	291	89.3	2.08	15	410	105	0	45	400	Nucleation 410 C, Si2H6 100, 400 mT, 10 min.
najeem	3/16/2004												Clean dummy wafers, wipe off cantilevers and wall
ethin	3/16/2004		9.94				60	450	100	60	58	600	Monitor run.
changj	3/17/2004	330	9.98	286	89.5	2.18	90	325	0	0	80	600	Nucleation 400 C, Si2H6 100, 300 mT, 20 min.
changj	3/17/2004												Before I started my process, I saw the dummy wafers (6" boat)are covered by particles. But the surface of my wafer after deposition seems ok.
jimmywgmac	3/18/2004												Particles will be fixed in the film. The user observe no particles on his film, the problem is clear for now. Will monitor the tube regularly.
jimmywgmac	3/18/2004												Turn off N2BKFL at standby
changj	3/18/2004	330	7.15	121	89.3	0.07	120	410	110	0	40	400	Test GeH4 and B2H6.
changj	3/23/2004	330	7.21	118	89.5								Nucleation 410C, Si2H6 100, 400 mT, 10 min.

User	Date													Comment
cherryj	3/24/2004	350	7.22	118	89.5	4.36	180	415	110	60		40	400	Nucleation 415, Si2H6 100, 400 mT, 10 min.
cherryj	3/24/2004	350	7.22	118	89.5	NA	1	450	100	0		0	800	Nucleation 450, Si2H6 200, 800 mT, 20 min. Coating after switting to open boat
wbw	3/25/2004	349.8	7.44	117	89.4	NA	180	425	100		16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. BCl3 test.
wbw	3/25/2004	NA	NA	NA	NA	NA	180	425	100			60	400	
Wendy	3/29/2004													GeH4 tank change. 7 psi can barely finish a deposition with 60 sccm GeH4 for 3 hr.
cherryj	3/29/2004	350	7.45	118	89.5	4.38	120	410	110	55		40	400	Nucleation 410C, Si2H6 100, 400 mT, 10 min.
cherryj	3/29/2004	350	7.45	118	89.5	3.12	120	415	110	30		40	400	Nucleation 415C, Si2H6 100, 400 mT, 10 min.
cherryj	3/30/2004	350	7.65	121	89.6	2.04	120	420	110	15		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
cherryj	3/31/2004	350	7.65	121	89.6	NA	120	420	110	5		40	400	Process aborted at NTEQ. Nucleation 420C, Si2H6 100, 400 mT, 10 min.
cherryj	3/31/2004													Pressure too high and process aborted at NTEQ.
bob	3/31/2004													The B2H6 supply to tystar20 will be pumped to vacuum temporarily while we service the pre-diborane valve on tystar19. Tystar20 remains up and useable without B2H6. This work should be completed by noon today and diborane restored.
Wendy	4/1/2004													Tystar20 is being used to pump out the diborane gas line for tystar19.
cherryj	4/3/2004	330	7.75	121	89.5	3.5	120	420	110	5		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
cherryj	4/4/2004	330	7.81	118	89.5	NA	150	325	0	0		80	400	Nucleation 400C, Si2H6 100, 400 mT, 20 min.
cherryj	4/5/2004	330	7.82	118	89.4	NA	150	420	100	3		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
cherryj	4/6/2004	330	7.82	118	89.4	0.05	30	325	0	0		80	400	Nucleation 400C, Si2H6 100, 400 mT, 4 min.
cherryj	4/6/2004	330	7.82	118	89.4	0.4	120	420	110	1		40	400	Nucleation 400C, Si2H6 100, 400 mT, 10 min.
cherryj	4/7/2004	330	7.81	118	89.4	NA	120	425	115	5		35	400	Nucleation 425C, Si2H6 100, 400 mT, 7 min.
cherryj	4/7/2004	330	7.83	118	89.4	NA	120	430	120	0		30	400	Nucleation 430C, Si2H6 100, 400 mT, 5 min.
cherryj	4/7/2004	330	7.82	118	89.4	1.03	120	430	120	5		30	400	Nucleation 430C, Si2H6 100, 400 mT, 7 min.
cherryj	4/7/2004													Report particle problem.
Maysam	4/9/2004	330	7.92	121	90	4.5	90	350	0	60		170	300	Nucleation 350C, Si2H6 100, 300 mT, 15 min.
wbw	4/12/2004	NA	NA	NA	NA	NA	120	425	100		16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min
wbw	4/12/2004													Building power failed for 20 minutes.
wbw	4/12/2004	NA	NA	NA	NA	NA	90	425	80		16.5	48	400	Thick coating run was done before depo to recover from power failure. Nucleation 425C, Si2H6 100, 300 mT, 15 min.
wbw	4/13/2004	353.9	8.06	118	89.9	NA	60	400	100		16.5	60	400	Si seed 450C, Si2H6 100, 300 mT, 15 min; Ge nuc 350C, GeH4 100, 300 mT, 16.5 BCl3, 300 mT
wbw	4/13/2004	336.5	7.97	118	89.7	NA	120	425	50		16.5	30	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min
wbw	4/14/2004	346.6	8.02	118	90.1	NA	60	425	100		16.5	60	400	Si seed 450C, Si2H6 100, 300 mT, 15 min; Ge nuc 350C, GeH4 100, 300 mT, 16.5 BCl3, 300 mT
wbw	4/14/2004	443	8.87	121	89.7	NA	60	350	0		16.5	100	400	Nucleation 450C, Si2H6 100, 300 mT, 15 min
wbw	4/14/2004													No particle in all of my wafers from the deposition during the past few days.
suoyi2	4/15/2004	430	8.65	121	90.1	5.23	60	450	100	60		58	600	Nucleation 450C, Si2H6 100, 400 mT, 10 min. Lots of particles in 6" dummies.
suoyi2	4/15/2004													Report particle problem.
wbw	4/16/2004													Particle problem are likely caused by the dirty injector. Request injector change.
Wendy	4/16/2004													Injector changed due to particle problem.

228

User	Date													Notes
srw2	4/19/2004	350	7.01	121	90	4.37	30	450	100	60		58	600	check particle problem
srw2	4/20/2004	350	7.06	121	90	4.23	60	425	100	60		70	400	Nucleation 425C, Si2H6 100, 400 mT, 10 min
Equavy	4/22/2004	NA	7.83	NA	NA	NA	12	350	0	60		180	300	Nucleation 350C, Si2H6 200, 300 mT, 1 min
Equavy	4/23/2004	350	NA	NA	NA	NA	12	350	0	60		180	300	Nucleation 350C, Si2H6 10, 300 mT, 1 min
chanyj	4/24/2004	350	7.2	121	89.9	0.12	120	420	110	0		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min. Process aborted.
Jimmygmac	4/26/2004													Previous process was aborted due to SiH4 out of tolerance. Restart the process with no problem. Will monitor MFC.
bob	4/26/2004													Clean dummy wafers.
srw2	4/27/2004	350	7.16	121	89.9	4.3	38	425	100	60		70	400	Nucleation 425C, Si2H6 100, 400 mT, 10 min.
wbw	4/28/2004	447.3	7.91	122	89.9	4.3	60	425	100	60		60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min
bob	4/30/2004													The BCl3 cylinder has been changed to 1%. With a setpoint of 20 psi, the cylinder pressure is 750 psi.
wbw	4/30/2004	353.5	7.28	121	90.1	NA	60	425	100	60	9.2	60	400	Dummy run for BCl3 line clean out. Nucleation 425C, Si2H6 100, 300 mT, 15 min.
wbw	4/30/2004	NA	NA	NA	NA	NA	60	425	100	60	9.2	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min.
wbw	4/30/2004	NA	NA	NA	NA	NA	60	425	100	60	16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min.
wbw	4/30/2004	NA	NA	NA	NA	NA	60	425	100	60	3.6	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min.
wbw	5/1/2004	350.5	7.33	121	89.9	NA	60	425	100	60	9.2	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min.
wbw	5/1/2004	NA	NA	NA	NA	4.52	60	450	100	60		60	400	Nucleation 450C, Si2H6 100, 300 mT, 15 min.
wbw	5/1/2004	NA	NA	NA	NA	4.36	60	400	100	60		60	400	Nucleation 400C, Si2H6 100, 300 mT, 15 min.
chanyj	5/2/2004	350	7.45	121	90	NA	120	420	110	0		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Equavy	5/10/2004	NA	7.35	NA	NA	4.04	120	350	0	60		180	300	Nucleation 350C, Si2H6 200, 300 mT, 5 min.
chanyj	5/12/2004	350	7.41	121	90	NA	25	420	110	0		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
chanyj	5/13/2004	350	7.45	121	90	NA	120	420	110	10		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
chanyj	5/15/2004	350	7.51	121	90	1.97	120	430	120	15		30	400	Nucleation 430C, Si2H6 100, 400 mT, 10 min.
chanyj	5/15/2004	350	7.54	121	90	NA	15	430	120	10		30	400	Nucleation 430C, Si2H6 100, 400 mT, 7 min.
Equavy	5/17/2004	NA	7.51	NA	NA	4.74	200	350	0	60		180	300	Nucleation 350C, Si2H6 100, 300 mT, 1 min.
Wehriby	5/18/2004													GeH4 tank change.
yanw	5/18/2004	350	7.61	111	90.1	NA	80	420	110	0		40	400	Did not run the process
chanyj	5/20/2004	350	7.6	121	90	NA	80	420	110	0		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
chanyj	5/20/2004	350	7.65	121	90	NA	15	420	110	0		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Wehriby	5/21/2004													SiH4 tank changed.
chanyj	5/22/2004	350	7.65	121	90	NA	90	325	0	0		80	400	Nucleation 400C, Si2H6 100, 400 mT, 10 min.
chanyj	5/22/2004	350	7.65	121	90	NA	15	420	110	0		40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Wehriby	6/2/2004													Front flange has been hooked up from the gas flange to allow front injection.
wbw	6/2/2004	NA	NA	NA	NA	NA	20	425	100	60	16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 2 min. Switch to open boat, install front injector. Coating run.
wbw	6/2/2004	448.8	8.81	118	89.9	NA	60	425	100	60	16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. Front injector test.
wbw	6/4/2004													Jon Goldman computer has not been recording data for all bank 5 furnaces since mid-May. Rebooting the computer doesn't work. Recipe cannot be loaded from computer to furnace.
wehriby	6/9/2004													Jon Goldman computer is fixed by computer support group via windows 2000.
wehriby	6/10/2004													Gazing switching function does not work.

Name	Date													Comments
Wehdry	6/14/2004	Chasing now switches back and forth. Removed capped front injector port and installed ultratorr adapter.												
srw2	6/14/2004	330	7.89	121	89.9	4.7	40	425	100	60	70	400		Nucleation 425C, Si2H6 100, 400 mT, 10 min.
srw2	6/15/2004	Regrust injector change for long depo.												
Wehdry	6/16/2004	Injector changed.												
Equwvy	6/16/2004	330	7.11	117	89.9	4.37	125	425	100	60	40	400		Nucleation 400C, Si2H6 200, 400 mT, 5 min.
chenyi	6/16/2004	330	7.47	121	90	0.18	15	420	110	0	40	400		Nucleation 420C, Si2H6 100, 400 mT, 10 min.
chenyi	6/17/2004	330	7.47	121	90	0.18	90	325	0	0	80	400		Nucleation 400C, Si2H6 100, 400 mT, 20min.
chenyi	6/17/2004	330	7.45	121	90	0.34	15	420	110	0	40	400		Nucleation 400C, Si2H6 100, 400 mT, 20min.
wbw	6/19/2004	347.3	7.24	117	89.9	0.19	60	425	100	60	60	400	16.5	Nucleation 425C, Si2H6 100, 300 mT, 15 min. Front injector test. No difference with gas ring.
chenyi	6/19/2004	Found standby process was aborted, could not find reason from process history.												
jimmygemc	6/22/2004	Suspected that the standby recipe was corrupted. Deleted the recipe and downloaded from DCS30 PC. Ran the recipe and it finished without problem.												
blahelm	6/23/2004	348	7.26	117	89.9	4.98	485/60	425	100	60	65/58	4000/600		No nucleation layer. Bi-layer run. Didn't break vacuum in between.
srw2	6/25/2004	Tried to run the coating part of the standby recipe last night, came in this morning and found the process is aborted. Checked process history, it seems PRCCPR was high and triggered the abort sequence.												
Wehdry	6/25/2004	Ran standby recipe with no problem. Process pressure and rate of rise are good. Base pressure is good.												
srw2	6/25/2004	330	8	117	89.9	5.11	270	425	100	60	70	400		Nucleation 425C, 400 mT, Si2H6 100, 10 min.
chenyi	6/26/2004	330	7.8	121	90	NA	120	420	110	15	40	400		Nucleation, 420C, 400 mT, Si2H6 100, 10 min.
Wehdry	7/2/2004	Injector changed.												
Equwvy	7/3/2004	349	7.23	121	90	NA	32	350	0	60	180	300		Nucleation 350C, 300 mT, Si2H6 200, 1 min.
yuw	7/4/2004	440	7.92	121	90	0.37	360	NA	NA	NA	NA	600		Nucleation 375C, 400 mT, Si2H6 100, 8 min.
wbw	7/5/2004	Found tystar20 in ABPG mode. DH shows after 2 minutes of standby coating, process was aborted with error "IFPRCPR NE". After 2 minutes into ABRT, step went to ABPG with message "PRCPR OK". Furnace was at N2BKFL=199, PRCPR=231, Temp=450, injector pressure=8.71. Exited out of abort mode and reloaded the recipe. Same thing happened.												
Jimmygemc	7/6/2004	Found that the process aborted due to PRCPR too high. The initial N2VAC shooted up which caused the problem. Adjusted the pid constants for pressure control on MFS460. Tried the recipe twice and the pressure stabilized within one minute.												
Equwvy	7/6/2004	420	7.78	121	89.9	4.44	300	425	100	60	40	400		Nucleation 400C, 400 mT, Si2H6 200, 5 min.
Equwvy	7/7/2004	349.7	7.65	118	90.1	NA	5	300	0	60	180	300		No nucleation
Equwvy	7/9/2004	330	7.66	119	89.9	NA	5	300	0	60	180	300		No nucleation
chenvi	7/9/2004	330	NA	121	90	NA	120	420	110	15	40	400		Nucleation 420C, 400 mT, Si2H6 100, 10 min.
wbw	7/11/2004	352.8	7.73	118	89.9	3.36	60	425	100	60	60	400	16.5	Nucleation 425C, 300 mT, Si2H6 100, 15 min. Left rear injector test.
wbw	7/11/2004	Got a change of $11134290.12 for Si2H6. Only used 100 sccm Si2H6 for 15 minutes.												
vorus	7/13/2004	RUMS software was rebooted and checked by Tim D. Charges are OK now.												
Equwvy	7/12/2004	NA	NA	NA	NA	NA	300	425	100	60	40	400		Nucleation 400C, 400 mT, Si2H6 200, 5 min. Found furnace in abort mode. Had to run standby recipe twice to get the coating step. Usual problem, computer gauges pressure too high and abort the recipe.
chenyi	7/16/2004	330	NA	121	90	NA	120	410	110	40	40	400		Nucleation 410C, Si2H6 100, 400 mT, 10 min.
chenyi	7/17/2004	330	8.33	121	90	NA	105	420	110	30	40	400		Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Wehdry	7/20/2004	GeH4 tank change.												

Name	Date												Note
Wehdry	7/20/2004	Injector pressure 8.5. Injector changed.											
Equevy	7/20/2004	366	7.15	118	89.9	4.05	30	350	0	60	180	300	No nucleation.
chanyi	7/20/2004	330	NA	121	90	NA	8	420	100	0	40	400	Nucleation 420C, Si2H6 200, 400 mT, 10 min.
chanyi	7/21/2004	330	NA	120	90	NA	90	325	0	0	80	400	Nucleation 400C, Si2H6 100, 400 mT, 15 min.
chanyi	7/21/2004	330	7.87	120	90	NA	9	420	110	0	40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Equevy	7/23/2004	349	7.22	121	90	NA	30	350	0	60	180	300	No nucleation.
chanyi	7/24/2004	350	NA	121	90	1.95	120	420	110	15	40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
blahelin	7/26/2004	330	7.26	121	90	NA	27	400	100	60	40	800	No nucleation. GeH4 charge did not show up.
Equevy	7/28/2004	350	7.28	121	89.6	4.12	12	350	0	60	180	400	No nucleation.
chanyi	7/31/2004	350	7.33	121	90	2	90	420	110	15	40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Equevy	8/1/2004	330	7.33	118	90	NA	20	350	0	30	180	300	No nucleation.
Equevy	8/3/2004	330	7.43	118	89.9	NA	60	330	0	30	180	300	Nucleation 350C, Si2H6 200, 300 mT, 5 min.
blahelin	8/4/2004	330	7.39	118	89.8	5.03/5.13	485/45	425/425	100/100	60/60	65/53	400/600	No nucleation. Bilayer process.
Equevy	8/4/2004	350	8.14	118	89.7	0.06	20	350	0	0	180	300	Nucleation 350C, Si2H6 200, 300 mT, 5 min.
chanyi	8/6/2004	330	NA	120	90	0.2	60	420	112	0	37	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
chanyi	8/6/2004	350	NA	121	90	NA	90	420	112	0	37	300	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Equevy	8/9/2004	330	8.15	121	89.9	5.27	150	425	100	60	40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
chanyi	8/9/2004	330	NA	121	90	NA	60	420	112	0	37	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Wehdry	8/9/2004	Injector pressure 8.5. Injector changed.											
Equevy	8/10/2004	390	7.79	121	89.7	4.61	150	425	100	60	40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
Equevy	8/11/2004	349	7.53	121	89.9	NA	45	350	0	60	180	300	Nucleation 350C, Si2H6 100, 300 mT, 1 min.
Equevy	8/12/2004	330	7.63	121	90	4.73	300	425	100	60	40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
Equevy	8/14/2004	351	8.04	121	89.9	NA	5	300	0	60	180	300	No nucleation.
Equevy	8/14/2004	330	8.04	118	89.9	5.04	300	425	100	60	40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
wlnw	8/16/2004	Verify that the front injector is leaky. Plugged up the front injector port with a rod, pumped out the line, and then flow N2 to build up some pressure. The pressure dropped immediately to zero once the N2 flow was turned off.											
chanyi	8/16/2004	330	8.6	121	90	NA	120	420	110	15	40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
chanyi	8/17/2004	330	8.6	121	90	NA	180	325	0	0	80	400	Nucleation 400C, Si2H6 100, 400 mT, 20 min.
chanyi	8/17/2004	330	8.6	121	90	4.3	150	420	110	40	40	400	Nucleation 420C, Si2H6 100, 400 mT, 10 min.
Wehdry / bob	8/19/2004	Injector pressure 8.63. Injector changed. The manifold was inspected and cleaned.											
Wehdry	8/19/2004	Tystar20 front injector for the dopant line has been capped off. Tystar20 line that went to the front injector now has been hooked up to the right rear injector. This will allow switching of the one injector or the other when the gas ring is on. As hooked up at present the left injector can only be used by manually switching it.											
elshim	8/19/2004	347.1	7.51	117	89.6	NA	20	425	100	60	70	400	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
wlnw	8/19/2004	NA	NA	NA	NA	NA	60	425	100	3.6	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. No standby info. Test right rear injector.
chanyi	8/19/2004	NA	NA	NA	NA	NA	90	420	110	30	40	400	NA
wlnw	8/20/2004	NA	NA	NA	NA	NA	60	425	100	16.5	60	400	Nucleation 425C, Si2H6 100, 300 mT, 15 min. No standby info. Test right rear injector.
wlnw	8/20/2004	NA	NA	NA	NA	NA	60	350	0	16.5	100	300	Nucleation 450C, Si2H6 100, 300 mT, 15 min. No standby info. Test right rear injector.
chanyi	8/20/2004	330	NA	121	90	0.22	180	325	0	0	80	400	Nucleation 400C, Si2H6 100, 400 mT, 20 min.
elshim	8/25/2004	351.7	7.71	118	89.9	NA	25	425	100	60	70	400	Nucleation 425C, Si2H6 100, 300 mT, 10 min.

jimmygmac	8/25/2004	Make N2dope a variable in recipe SiGeBCl3											
elzkim	8/26/2004	348.9	7.71	118	99.9	NA	25	425	100	60	70	400	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
chenyi	8/28/2004	330	7.9	121	90	3.8	150	420	110	40	40	400	Nucleation 420C, Si2H6 100 k 400 mT, 10 min.
whvw	8/28/2004	Tried to load the new SiGeBCl3 recipe in the furnace, got error "recipe/configuration mismatch. Must re-edit". This happened to all recipes.											
jimmygmac	8/30/2004	Deleted all recipes. Download configuration files and other recipes from DCS30. The recipe can only load once without problem. After a few recipe loadings, FCS10 displays strange messages. The FCS10 may need to be hard rebooted, soft rebooted doesn't help.											
Welzhy	8/30/2004	Reloaded configuration. Two recipes b toutb.020 and sig gevaub.020 were able to be downloaded into tystar20 without problems. Loaded each one into the tystar20 and had no config mismatch. When standby recipe was loaded in and ran one time, the recipe would load correctly the first time then all other recipes including the standby recipe would give a config mismatch. If configuration is loaded again the recipes will work. Will continue to monitor.											
Welzhy	8/30/2004	Jimmy came in and loaded in three more recipes with no problems. Loaded them in two more times with no problems. Found fan stopped on right side of unit. Started fan manually.											
chenyi	8/30/2004	Standby recipe was aborted.											
Welzhy	8/31/2004	Leak check and rate of rise are good. Started standby recipe and started running without problems thru the 2nd temp step.											
chenyi	8/31/2004	330	7.9	121	90	0.22	190	325	0	0	80	400	No nucleation.
chenyi	8/31/2004	Screen was frozen and would not respond to my command when I pressed "Menu" and later it became completely black. There is an alarm "No Auto?on the backside of the furnace.											
Welzhy	8/31/2004	Clear alarm and FCS-10 front pannel.											
Welzhy	9/1/2004	GeH4 leak change.											
blakelin	9/2/2004	330	7.98	121	89.9	4.52	30	350	60	60	180	300	No nucleation.
chenyi	9/3/2004	Standby recipe was aborted.											
jimmygmac	9/3/2004	Check the tube and could not repeat the problem. The reason may by that some time the MFC opens up faster than the gate valve after the leak check step. Adjust the pressure check step after the leak check step.											
chenyi	9/3/2004	330	7.91	121	90	NA	140	325	0	0	80	300	Nucleation 425C, Si2H6 100, 300 mT, 45 min.
chenyi	9/4/2004	330	7.9	121	90	NA	70	420	30	110	40	300	Nucleation 420C, Si2H6 100, 300 mT, 10 min. / Nucleation 425C, Si2H6 100, 300 mT, 15 min. Right
whvw	9/4/2004	NA	NA	NA	NA	NA	60	425	100	3.6 (+26 n2dope)	60	400	rear injector. No improvement in XW uniformity with n2dope.
whvw	9/4/2004	"Si2H6 leak" alarm in unit CVD460-196 was on a few times. In all cases, Si2H6 was not supposed to be running.											
chenyi	9/6/2004	A SiGe coated sample was etched in piranha. The recipe was the same before, but piranha never hurt.											
Welzhy	9/7/2004	Cannot repeat Si2H6 leak problem.											
chenyi	9/7/2004	330	7.9	121	90	3.2	60	420	30	110	40	300	Nucleation 420C, Si2H6 100, 300 mT, 5 min.
chenyi	9/9/2004	330	NA	121	90	NA	90	420	30	110	40	300	Nucleation 400C, Si2H6 100, 300 mT, 5 min.
mwasilik	9/14/2004	chenyi's recipe is close to the "edge" with respect to certain responses and eventually yields results past the sigma values.											
Welzhy	9/16/2004	Injector pressure 8.31. Injector changed.											
chenyi	9/18/2004	330	7.26	121	90	NA	30	325	0	0	80	300	Nucleation 400C, Si2H6 100, 300 mT, 3 min.
chenyi	9/19/2004	330	NA	121	90	NA	10	420	110	0	40	300	Nucleation 400C, Si2H6 100, 300 mT, 5 min. Quartz tube cracked.
chenyi	9/19/2004	After I loaded the wafer and boat completely went in, I heard a sharp sound followed by gas leakage. One of the string was found loose. The process stayed at idle when I returned to check. The tube was found broken later. (enabled 711 hr, 208 hr of depo)											
Welzhy	9/24/2004	Install new quartzware and injector. Base pressure and rate of rise are good.											
gprohask	9/29/2004	Install new BCl3 MFC. 20 sccm full range calibrated to He. Pum ped/purged then hard pumped and leak checked Leak check OK.											
Eguevvv	9/30/2004	445	8.35	121	89.9	4.97	150	425	100	60	40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
Welzhy	9/30/2004	Patrick Welzhy decides to retire after ten years of service in the Microlab. Bob Prohaska will be the next technician for all the LPCVD furnace.											

232

wbw	10/1/2004	350	7.78	118	89.6	NA	60	425	140		12	60	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min. 6" wafers only, open boats 28" from door.
chenyi	10/2/2004	350	NA	121	90	NA	120	420	110	0		40	300	No nucleation.
chenyi	10/2/2004	350	NA	121	90	0.09	45	325	0	0		80	300	No nucleation.
chenyi	10/3/2004	350	NA	121	90	NA	120	420	110	0		40	300	No nucleation.
vidya	10/4/2004	350	7.95	117	89.5									
Equervy	10/5/2004						150	425	100	60		40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
Meyoum	10/5/2004					0.21	230	425	100	60	18	60	400	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Equervy	10/7/2004	Computer frozen while entering recipe. Communication lost with the furnace, alarm in the back. Problem solve later.												
Equervy	10/7/2004	350	8.17	118	89.2	4.72	72	350	0	60		180	300	No nucleation.
chenyi	10/10/2004	The boat-in/out step in software seemed to be messed up. Sometimes when I pressed "event" after I loaded my wafers, the boat entered only half-way and then came out automatically with "boatout" showing on the CRT screen. Today the boat stopped movement before the door is closed, both manual and auto modes did not make door close successfully. Finally I used my hands to push the boat in.												
chenyi	10/10/2004	350	8.16	121	90	NA	10	420	110	0		40	300	Nucleation 400C, Si2H6 100, 300 mT, 3 min.
chenyi	10/10/2004	350	8.16	121	90	NA	90	325	0	0		80	300	Nucleation 400 C, Si2H6 100, 300 mT, 3 min.
chenyi	10/10/2004	350	8.16	121	90	NA	15	420	110	0		40	300	Nucleation 400C, Si2H6 100, 300 mT, 3 min.
reprobank bob	10/11/2004	The drive pulley was loosed on the shaft. It has been tightened.												
bob	10/11/2004	GeH4 tank changed												
Equervy	10/11/2004	350		121	90	NA	300	425	100	60		40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
blahaim2 bob	10/11/2004	450	9.11	118		4.38	165	425	115	40		60	400	No nucleation.
bob	10/12/2004	Injector pressure 8.82. B2H6 injector changed.												
Equervy	10/12/2004	350	7.73	118	89.2	NA	7	300	0	60		180	300	No nucleation.
Equervy	10/13/2004	350	7.73	118	89.3	NA	300	425	100	60		40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
Equervy	10/14/2004	350					120	425	100	60		40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
chenyi	10/16/2004	350	8.89	121	90	4.89	120	420	110	50		40	300	Nucleation 400C, Si2H6 100, 300 mT, 3 min.
chenyi	10/20/2004	350	9.19	121	90		60	430	110	0		40	400	Nucleation 400C, Si2H6 100, B2H6 100, 400 mT, 3 min.
blahaim2	10/21/2004	350	9.12	117	89.3	0.5	90	400	100	0		20	800	Nucleation 400C, Si2H6 100, 400 mT, 3 min.
blahaim2	10/22/2004	350	9.13	118	89.4	0.5	120	400	100	0		35	800	Nucleation 400C, Si2H6 100, 400 mT, 3 min. Took 1 hr 45 min to stabilize to 400 C.
blahaim2	10/24/2004	350	9.1	117	89.5	0.18	0	0	0	0		0	0	Nucleation 400C, Si2H6 100, 400 mT, 2 hr.
blahaim2	10/25/2004	350	9.11	118	89.6	0.49	0	0	0	0		0	0	Nucleation 400C, Si2H6 200, 800 mT, 4 hr.
blahaim2	10/28/2004	350	9.02	117	89.2	0.49	150	410	150	0		10	800	No nucleation.
blahaim2	10/29/2004	350	9.11	118	89.6	0.49	300	410	150	0		10	800	No nucleation.
wbw	10/30/2004	Found furnace running standby recipe, leg2 step. Set temp is 450 C, but center temp is only 289 C, stuck at this step for more than a day. Changed set temp to 300 C, and put furnace on hold.												
jimmygmac	11/2/2004	Reset tru, firing package and fbs10. Temp started to respond. Ran the standby recipe without problem. Done tru calibration.												
Equervy	11/3/2004	Communication failure with DCS30. No monitoring.												
jimmygmac	11/3/2004	Turned off DCS30 and restarted. Went into the tru calibration program and disconnected the communication. DCS30 is recording furnace info now.												
Equervy	11/3/2004	350	9.05	118	89.6		45	350	0	60		180	300	No nucleation.
Meyoum	11/3/2004	350					240	400	100	60	18	45	300	Nucleation 425C, Si2H6 100, 400 mT, 15 min.
Equervy	11/5/2004	350	9.1	119	89.8		150	425	100	40		40	400	No nucleation.
wbw	11/8/2004	S2H6 ran out. Standby recipe aborted at coat step.												

Performing injector maintenance. (BCl3 injector is changed by error).

Maintenance notes from bob (09-nov-2004 14:12:46) :

The SS injector on tystar20 has been changed. The installed injector was first measured with a capillary gauge to assure the orifices were open at to specification. The 0-100 torr injector pressure gauge was rezeroed (added +25 torr to zero). The following pressure correlations were measured (clogged B2H6 injector reading):

bob   11/9/2004

N2DOPE sccm	Injector Pressure/torr (Temp 450C)
10	3.1
20	4.5
40	6.67
60	8.36
80	9.84
100	11.17

To assure the injectors meet spec. I am ordering additional, new injectors and have ordered a set of carbide drills to match the orifices.

tystar20 is in standby and ready for use.

blahblim2   11/9/2004

330	10.31	118	89.3	7.58	48.5/35	42.5/42.5	100/100	60/60	65/58	400/600	Bilayer deposition. No nucleation. Deposition injector pressure taken at 2nd layer.

Maintenance notes from bob (10-nov-2004 15:19:27) : (BCl3 injector is changed by error again)

The injector installed 11/09 was removed and inspected. There is no about it is 100% open -- unobstructed and the orifices are sized correctly. A spare injector, freshly drilled and with an open bore was installed. The following N2DOPE pressures were recorded (clogged B2H6 injector reading):

bob   11/10/2004

N2DOPE sccm	Pressure Temp = 350C
20	5.9
40	7.57
60	9.44
80	11.06
100	12.5

Performing injector maintenance.

Maintenance notes from bob (10-nov-2004 17:17:08) :

It is likely I have removed and been changing the wrong injector. I have changed the other injector and here are the results:

bob   11/10/2004

N2DOPE sccm	mtorr	temp 350C
20	3.22	
40	4.76	
60	6.00	
80	7.88	
100	8.03	

													notes
elim	11/12/2004	347.8	7.45	118	89.4	4.77	150	100	60	40	400		No nucleation.
Equery	11/13/2004				89.5	5	392	425	100	60	40	400	SiGevario recipe. No nucleation.
wblw	11/13/2004	347.5	8.34	117	89.5								Injector monitor. No de position.
Equery	11/16/2004	351	8.33	117	89.2	5	300	0	60	180	300		No nucleation.
Equery	11/17/2004	330	8.34	121	89.5		392	425	100	60	40	400	No nucleation.
liman	11/18/2004	GeH4 tank change.											
bob	11/19/2004	Si2H6 tank change.											
bob	11/19/2004	Install quartz injector for BCl3 line.											
wblw	11/19/2004	New quartz injector standby conditions: temp=350, prepr=118 n2dope=89.6, init pressure=7.6											

234

User	Date												Comment
wblw	11/19/2004	330	9.15		121	89.6							Si2H6 coating with standby recipe. Pressure reading for B2H6 injector. No deposition.
wblw	11/19/2004	349.2	7.6 quartz		118	89.6	0.53	60	410	150	6	50	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for Si2H6 problem. Rerun OK.
wblw	11/19/2004													Process got into SHLD for Si2H6 leak. Exited out of SHLD and ran the recipe again, process completed without problem
bob	11/19/2004													The Si2H6 problem is cleared w/o understanding the cause. Will monitor.
wblw	11/21/2004	348.9	8.93		117	89.6	0.5	60	440	130	18	70	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for Si2H6 flow error, reran w/o problem.
wblw	11/22/2004													Found backup thermocouple running.
bob	11/22/2004													The center TC defaulted to the backup TC for unknown reasons. Exchanging the t20 TC for the t19 did not help. In the process of checking wiring the problem cleared. Next tystar visit we'll trace through the wiring harness.
even	11/22/2004													Verified tc connections, also found spike tt's were loose in the spring holder. I tightened all the springs so tt's were tight against tube and not easy to move. I noticed a big change in the spike reading for zone 1 and 5.
bob	11/22/2004													TC connections for zone 1 and 5 were switched by mistake.
wblw	11/22/2004													Preform thermal calibration.
wblw	11/23/2004	350.6	9.02		117	89.4	0.53	300	425	140	12	60	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
wblw	11/24/2004	351.7	9.17		117	89.4	0.5	300	425	140	6	60	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for Si2H6 problem twice, 3rd rerun passed.
bob	11/24/2004													The Si2H6 cylinder is unregulated and currently delivery pressure is 40 psi. It is possible the recipe tolerances are resulting in a flow fault. Because the run eventually succeeded we'll wait on addressing this issue until Jimmy is back and can review the DCS30 logs.
wblw	11/24/2004	439.8	10.03		118	89.6	0.5	180	440	130	18	70	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for Si2H6 flow error, reran w/o problem.
wblw	11/25/2004	353.2	9.27		117	89.5	0.53	380	410	150	6	50	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for Si2H6 problem. Rerun OK. Nucleation 440C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for Si2H6 flow error, reran w/o problem.
wblw	11/26/2004	353.3	9.24		117	89.5	0.53	185	440	130	6	70	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min. Recipe aborted at NUCL for SiH4 problem. Then aborted again 6 min into depo for SiH4 problem. Resumed process manually with 179 min of depo. 30 psi SiH4 left after depo.
wblw	11/26/2004	357.3	7.71 quartz		118	89.6		120	325	0		80	300	No deposition. Quartz injector monitor. Pressure stays after 24 hr 25 min of BCl3 deposition.
chenyj	11/29/2004	330			121	90		120	325	0		80	300	Nucleation 400C, Si2H6 100, 300 mT, 3 min.
jimmygmc	11/29/2004													checked the DCS30 PC data log. found that for the past few runs, the Disilane (Si2H6) did not turn off in time when running 100sccm. It turned down to 3 sccm and gradually to off. The trial time took over 6 minutes. I suspect that the cylinder pressure may be too high (40psi). Usually, the MFC needs only 18 psi for normal operation. Since the leak alarm was generated by the MFC460, it is not possible to modify the recipe tolerance to eliminate the problem.
bob	11/29/2004													tystar20 remains down for the time being while we replace a defective purge-gas valve. Tystar20 should be available 11/30, 10 AMish following restoration of the SiH4 and removal of the BCl3 test injector.
bob	11/30/2004													Removed the qtz. BCl3 injector, bagged and placed on top of the tystarbank5 pump cabinet for Carrie's review. Installed an injector "stub" in lieu of this injector. Installed recipe SIGESTNC.020.

Name	Date	V1	V2	V3	V4	V5	V6	V7	V8	V9	V10	V11	Comments
changyj	12/1/2004	350	9.11	121	90		120	400	110	50	40	300	Nucleation 400C, Si2H6 100, 300 mTorr, 2 min.
elisio	12/1/2004	350	7.8 (?)				90	350	0	60	180	300	No nucleation
elisio	12/1/2004												Process doesn't seem to progress. It is in SHLD step.
bob	12/3/2004												There is an issue with stable disilane delivery. The mass flow controller is suspect and will be replaced on Monday. In the meantime, t20 is in use; however, disilane delivery maybe a problem.
elisio	12/3/2004	330	8.4(?)			5.52							No nucleation.
whw	12/5/2004	349.1	9.89	118	89.6	5.21	150	400	100	60	40	400	B2H6 injector monitor. No deposition.
bob	12/6/2004												The disilane MFC has been changed. The new MFC was tested in normal mode. It seems to work well
blahblm2	12/6/2004	330	9.95	117	89.5	NA	27	400	100	60	40	800	No nucleation.
rprohask	12/7/2004												Injector pressure 10 Torr at 350C. B2H6 injector changed.
Bguwvy	12/7/2004	330	7.4	117	89.3	4.62	150	435	100	60	40	400	Nucleation 400C, Si2H6 200, 400 mT, 5 min.
blahblm2	12/10/2004	330	7.71	121	89.7	B54.0/24.1/16/100/30		425	00/11.5/10 60/40/40	60	6.5/45/65	400	Trilayer depo. Nucleation 400C, Si2H6 100, 400mm T, 5 min.
Bguwvy	12/10/2004	350	7.83	118	89.5	NA	38	350	0	60	180	300	No nucleation.
Bguwvy	12/10/2004												B2H6 bottle depleted. Furnace went to SHLD step after 38 min of deposition. I switch the furnace to PMFC to finish the deposition and put it back to standby.
liman	12/15/2004												Plastic pump purge line has been replaced with stainless steel.
bob	12/16/2004												Install new quartz injector for BCl3 line. The previous quartz injector was somehow broken sitting on top of the pump cabinet.
whw	12/17/2004	347.1	7.68 quartz	117	89.3	0.5	60	440	150	60	6	600	No deposition. Quartz injector monitor.
whw	12/17/2004	345.6	7.73	117	89.3	0.5	60	410	130	70	18	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min.
whw	12/17/2004	406.7	8.24	117	89.3	0.5	60	440	140	60	18	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min.
whw	12/19/2004	345.6	7.71	117	89.5	0.5	300	425	150	70	18	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
whw	12/20/2004	357.9	7.82	117	89.5	0.5	420	410	150	70	12	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min.
whw	12/21/2004	344.7	7.73	117	89.3	0.5	45	350	0	100	12	300	Nucleation 350C, Si2H6 100, 300 mT, 20 min.
whw	12/21/2004	452.2	8.53	117	89.5	0.46	257	440	150	50	6	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min.
whw	12/22/2004	351.1	7.74	117	89.4	0.43	250	410	130	70	18	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min.
whw	12/23/2004	350.9	7.7	117	89.4	0.4	265	440	150	50	18	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min.
whw	12/23/2004	430.7	8.34	117	89.5	0.39	290	425	150	50	12	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
whw	12/24/2004	350.2	7.72	117	89.4	0.41	135	440	130	70	6	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min. 9604 sccm of GeH4 consumes 6.5 psi of the bottle.
takeuchi	12/27/2004												S2H6 has been added as a variable in recipe SiGeBCl3, deposition step.
whw	12/27/2004	NA	NA	NA	NA	0.47	5	425	25 Si2H6	175	12	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min. Recipe check for Si2H6.
bob	12/28/2004												GeH4 tank change.
whw	12/28/2004	341.7	7.88	117	89.4	0.51	230	425	130	70	12	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
whw	12/28/2004	456.5	8.82	117	89.3	0.51	335	410	130	70	6	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min.
whw	12/29/2004	346.5	7.88	117	89.6	0.47	60	425	25 Si2H6	175	12	600	Nucleation 425C, Si2H6 100, 300 mT, 10 min.
whw	12/29/2004	NA	NA	NA	NA	0.25	287	350	0	100	12	300	Nucleation 350C, Si2H6 100, 300 mT, 20 min.
whw	12/30/2004	355.7	7.89	117	89.3	0.51	318	410	140	60	12	600	Nucleation 410C, Si2H6 100, 300 mT, 10 min.
whw	12/31/2004	355.6	7.96	117	89.4	0.51	218	440	140	60	12	600	Nucleation 440C, Si2H6 100, 300 mT, 10 min.

Performing BCl3 quartz injector monitor after 59 hours of BCl3 doped deposition.

N2DOPE (sccm)	Injector Pressure (torr)	Temp (deg C)	PRCPR (Torr)
10	2.29	363.5	22
20	3.37	363.3	39
30	4.23	363	54
40	4.95	362.7	67
50	5.59	362.1	78
60	6.2	361.3	89
70	6.72	360.6	98
80	7.24	359.5	110
90	7.71	358.5	117

wblw 12/31/2004

bob 1/5/2005 A new B2H6 cylinder is connected to tystar20. It is hard pumping overnight and will be turned on 1/6 by 9 am. T20 will be up tomorrow.

The new B2H6 cylinder is installed. The quartz injector used by Carrie Low over the holidays has been removed. This quartz injector broke during removal due to mechanical force.

bob 1/6/2005 A dummy quartz stub has replaced it and the valve on the dopant line to this dummy injector has been closed. The B2H6 stainless injector was removed and replaced with a cleaned injector. the current injector pressure is: 90 sccm N2slope - 7.03 Torr.

jimmygmac 1/18/2005 Found the standby recipe at special hold step. Check the history and found the reason was pressure high. Check the pump and found it is off. Equip staff please check the pump. If it is OK, please restart the pump. Tystar200 is down.

memel 1/18/2005 Restarted the pump. Ready for use.

equerv 1/19/2005	350	7.4	119	89.9	340	425	100	60	400	Nucleation 400C, Si2H6 200, 5 min.
chenjj 1/20/2005	350	7.87	120	90	110	400	110	35	300	Nucleation 400C, Si2H6 100, 3 min.
chenjj 1/23/2005	350	7.43	120	90	120	325	0	0	300	Nucleation 400C, Si2H6 100, 2 min.
Equerv 1/23/2005	330	7.23	118	89.2	5	300	0	60	300	No nucleation.
chenjj 1/26/2005	330	7.8	121	90	110	420	110	35	300	Nucleation 400C, Si2H6 100, 3 min.

bob 2/3/2005 Mavis requested to install a new quartz injector for the BCl3 line.

Magnum 2/3/2005 330   6.9   101   89   Recipe aborted due to SiH4 error.

jimmygmac 2/3/2005 Found that tystar1 &12 flow SiH4 without problem, but both tystar19 and 20 could not flow SiH4. Traced the gas line and could not found any valve closed. The recipe is set to wait Lab member can unload her wafers. Tystar20 is down for any process that uses SiH4. Equip staff please double check the gas lines.

wblw 2/3/2005 Standby recipe aborted due to Si2H6 flow error.

bob 2/4/2005 There is a definite problem with SiH4 flow on t20. At 2000 sccm flow. The supply was exhausted in ~ 3 minutes. Tystar19 was tested and could still flow SiH4. We will replace the SiH4 mfc for tystar20 later this afternoon and see if this is a fix. tystar20 remains down and will hopefull be up by the end of today.

bob 2/4/2005 A new SiH4 mfc was installed on t20. There is an issue with the SiH4 flow. We cannot complete rep-airs today and need to take the SiH4 circuit offline on Monday to make further checks. t20 is down and locked for the weekend.

rgrobask 2/7/2005 There seems to be something amiss with the 460. A new mfc does not work under the control of the 460 but does work perfectly under control of a test electronics package. Substituting a new cable from 460 to sih4 mfc results in _still_ wrong, but different, behavior.

bob 2/8/2005 The tystar20 SiH4 flow issue has been traced to the delivery line. We will open the line for inspection in the morning.

bob 2/9/2005 The flow issue with t20 has been located and fixed Tomorrow, (2/0905) I will check the GeH4 and SiH4 mfc's for flow values. If all is well the tube should be back available by the afternoon.

bob 2/10/2005 Found the gas ring line hooked up to the front injector port. Clogged port caused gas flow problem. The SiH4 and GeH4 mfcs were removed and the calibration measured and recorded. Both are correct. Previous SiH4 flows were likely 75% of set values. Carrie Low will compare previous runs to compare results. Carrie is also looking into ways to monitor flow and ratios for SiH4 and GeH4.

237

Perform MFC test with N2 flow meter.

Person	Date	MFC	Electronic input	N2 output (sccm)	correction factor	conversion (sccm)
wlow	2/10/2005	SiH4 200 (before)	100%	250	0.6	150
		SiH4 200 (before)	50%	125	0.6	75
		SiH4 200 (now)	100%	320	0.6	192
		SiH4 200 (now)	50%	155	0.6	93
		GeH4 200 (now)	100%	330	0.57	188.1
		GeH4 200 (now)	50%	160	0.57	91.2
		GeH4 200 (new spare)	100%	345	0.57	196.65
		GeH4 200 (new spare)	50%	165	0.57	94.05

Person	Date	Note
wlow	2/10/2005	Standby recipe aborted twice with error message "IF PRCPR.NE". Jon Goldman is not collecting data and I cannot find out what went wrong.
jimmygmac	2/11/2005	Reboot DCS30 PC. Will monitor the pressure.
wlow	2/11/2005	A follow up on the standby recipe abort problem: I was watching the furnace when the process aborted 2 minutes into the Si2H6 coating step. The pressure could not be achieved and N2vac kept changing values to adjust the pressure. It always got too high or too low. Also tested the PRCPR at manual mode. Flow 100 sccm N2 dope, no other gas. PRCPR cannot be settled at 300 or 400 mTorr in 5 minutes. After 10 minutes, it could reach the set point. If the PRCPR setpoint is 600 mTorr, it could be achieved faster. Some problem with the PRCPR and N2vac feedback control?
jimmygmac	2/11/2005	Adjusted the pressure control constant offset 0, reset 2, delay 0. The pressure control can reach 300 and 900 mTorr in a minute.

Person	Date	MFC		Electronic input		N2 output	correction factor					conversion	Notes
elisio	2/11/2005	348	9.2	118	90	4.51	1.50	425	100	60	20	400	Nucleation 400C, Si2H6 200, 5 min.
chengj	2/13/2005	330	9.2	120	90		120	325	0	0	80	300	Nucleation 400C, Si2H6 100, 3 min.
blakelin	2/14/2005	350	8.13	112	89.9	NA	660	410	150		18	600	Nucleation 410 C, Si2H6 100, 300 mT, 10 min. Film color not uniform. Some dots as big as 5 mm in diameter that have different color at the lower right corner of each wafer.
Egusey	2/15/2005	350	8.11	112	89.2	5.65	340	425	100	60	40	400	No nucleation.
chengj	2/17/2005	330	8.96	120	90	NA	110	420	110	35	40	300	Nucleation 400 C, Si2H6 100, 3 min.
wlow	2/20/2005	356.6	9.18	112	89.9	0.53	60	375	25 Si2H6	0	12	600	Nucleation 375C, 300 mT, Si2H6 100, 15 min. Uniform but high R film.
wlow	2/20/2005	456.5	10.16	112	89.9	0.57	70	425	0	0	0	600	Furnace anneal. Flow 100 sccm N2.
wlow	2/21/2005	353.3	9.17	112	89.9	0.53	60	440	130	0	18	600	Nucleation 440 C, 300 mTorr, Si2H6 100, 10 min. Repeat DOE-15 to identify SiH4 MFC problem.
wlow	2/21/2005	454.5	10.15	112	89.2	0.54	60	400	25 Si2H6	0	12	600	Nucleation 400 C, 300 mT, Si2H6 100, 12 min. Cloudy film, Large R variation.
wlow	2/22/2005	345	9.13	112	89.9	0.57	60	440	150	0	6	600	Nucleation 440 C, 300 mT, Si2H6 100, 10 min. Repeat DOE-11 to identify SiH4 MFC problem.
wlow	2/22/2005	458.1	10.2	112	89.9	0.28	60	400	15 Si2H6	0	18	300	Nucleation 400 C, 300 mTSi2H6 100, 12 min. Uniform film, high R.
wlow	2/22/2005	457.2	10.19	112	89.9	0.27	60	425	15 Si2H6	0	18	300	Nucleation 425 C, 300 mT, Si2H6 100, 10 min. Uniform film, low R. Pressure varied from 250-350 mTorr.
wlow	2/22/2005												Pressure varied from 250-350 mTorr in the previous depo. Lower limit of the pressure is reached for the particular gas flows. Need to adjust PIN setting with Jimmy.
wlow	2/23/2005	343.4	9.14	112	89.9	0.3	60	425	25 Si2H6	0	18	350	Nucleation 425 C, 300 mT, Si2H6 100, 10 min. Cloudy film.
wlow	2/23/2005											175	GeH4 tank pressure reaches a hard zero. 6 psi barely covers a 1-hr depo with 175 sccm.

User	Date													Comment
rgrohmk	2/24/2005													GeH4 tank changed.
rgrohmk	2/24/2005													Tested pump throughput with N2. The corresponding flow and pressure: 100-121, 200-211, 500-418, 1000-712, 1500-978, 2000-1236.
wlmw	2/24/2005	336	9.19	112	89.9	0.3	60	425	15 Si2H6	0	18	150	350/500	Nucleation 425 C, 300 mT, Si2H6 100, 10 min. Pressure cannot be stabilized at 300 mTorr. Manually change setpoint to 350 mTorr.
bob	2/24/2005													Jimmy will adjust the PIN setting to find the right Px value for the pressure feedback.
blahblim2	2/24/2005	330	9.15	111	89.9	NA	115	425	130		12	70	600	Nucleation 425 C, 300 mT, 100 Si2H6, 10 min. Process aborted after 1 hr 6 min into depo. History shows SiH4 flow problem.
bob	2/25/2005													SiH4 delivery pressure to T20 is 23 psi - normal. Manually tested gas flow and no problem can be found a this time. Will monitor. With the agreement of lab members, the following modifications have been made to tystar20: 1) the injector line formerly used for B2H6 has been routed to the quartz injector. 2) the SS injector has been removed and the tubulation plugged off on the tube. 3) the valve used for dopant -> gas ring has been capped at the rear gas shelf and no longer in use. The He/BCl3 1% is now the only boron source for tystar20 (B2H6 remains available for tystar19). The mfc used for He/BCl3 remains a 0-20 sccm. The configuration file for the FCS10 remains as before as does the definition table on the rear MFS2 (460 controller).
bob	2/28/2005													To be done at a future date: 1) Change He/BCl3 1% mfc from 0-20 sccm to 0-50 sccm. 2) Change the disilane mfc from 0-100 sccm to 0-200 sccm. 3) Add series mfm for insitu ratio/flow checks of SiH4, S2H6, GeH4. 4) Update FCS10 config so it is up to date. 5) Update the MFS2 (460 controller) definition table. 6) Update recipes
wlmw	3/1/2005													Did manual gas flow test to verify the injector pressure gauge connection. The injector pressure gauge is now reading the BCl3 quartz injector pressure. Should choose "gas ring off" option to flow BCl3, opposive to before.
wlmw	3/1/2005													The furnace got into a strange "STBY" mode, cannot get it back to normal with serval attempts. Checked the tube configurations. it was ok. Found all the recipes were corrupted. Down loaded b.tourb, sigestnc, and sigebcl3 into the fcs10. Tried to download more recipes, but the fcs10 alarmed for power failure, and all the recipes previously downloaded corrupted. Tried again, and everything repeated. Now there are only the above 3 recipes in the fcs10. Suspected that the ram of fcs10 is not working right. Some ram may be malfunction which limited the number of recipes. Tystar20 is running standby recipe now. The temperature recovers to 350C. Equipment staff should contact Tystar for further diagonsis.
jimmygmac	3/1/2005													The backup battery voltages we're tested on t17-20. T20 showed 3.74 volts, a bit lower than the rest which were slightly over 4 volts. A call is in to Tystar asking if this might account for recipe corruption. New batteries will be sought in any case.
rgrohmk	3/2/2005													Jimmy has successfully re loaded four recipes and t20 is operational. The symptoms observed are believed to be faulty RAM and the folks at Tystar have agreed to send a set of replacement chips which will be installed upon arrival. For now t20 is up and running.
blahblim2	3/4/2005	330	6.23	112	89.9	2.18	115	425	130		12	70	600	Nucleation 425 C, 300 mT, 100 Si2H6, 10 min.
blahblim2	3/6/2005	330	6.21	112	89.9	0.57	180	410	130		0	70	600	Nucleation 410 C, 300 mT, 100 Si2H6, 10 min.
Meyoum	3/7/2005													When I opened the furnace to load my wafers, I realized that the tube was broken. There were lots of the broken pieces everywhere inside the furnace. I left the tube on BTOUB recipe.
jimmygmac	3/7/2005													Found that the quartz liner was broken. The tube is still holding vacuum. The leak rate is 3 mtorr/min. Wait for the equip staff to change it. Tystar20 is down.
rgrohmk	3/14/2005													Changed the liner, along with new TC sheath, injector and cantilever cover.
jimmygmac	3/22/2005													Did thermal calibration. The tube failed leak check when running standby recipe.
rgrohmk	4/1/2005													Changed quartz tube, reused other quartzware from last time. Leak rate 2 mT/min. (231 hr of depo)
jimmygmac	4/5/2005													Thermal calibration 300-450 C.

Name	Date	330	6.2	112	112(?)	112	89.9	6.2(?)	60	9(?)	150	410	18	50	600	Notes
wlow	4/7/2005															Jon Goldman is not recording data. "TCU communication error" on screen.
jimmy/gmac	4/8/2005															Reset the TCU. DCS30 is recording data again.
fuwe(?)	4/7/2005	330	6.2	112	112			NA	60		150	410	18	50	600	Nucleation 410 C, 300 mT, 100 Si2H6, 2 min.
blakelim	4/8/2005	330	6.08	112	112		89.9	NA	30		130	425	12	70	600	Nucleation 425 C, 300 mT, 100 Si2H6, 10 min.
blakelim	4/9/2005	330	6.12	112	112		89.9	2.02/NA	115/386		130/130	425/410	12 and 6	70/70	600/600	Nucleation 425/410 C, 300 mT, 100 Si2H6, 10 min. 4 um bi-layer.
wlow	4/10/2005	353.6	6.14	112	112		89.9	2.06	60		140	425	12	60	600	Nucleation 425 C, 300 mT, 100 Si2H6, 10 min. DOE recipe 8, redo test run for MFC calibration.
wlow	4/10/2005	451.5	6.65	115	115		89.9	2.57	60		103	440	18	70	600	Nucleation 440 C, 300 mT, 100 Si2H6, 10 min. DOE recipe 15 with adjusted SiH4 flow for MFC cal.
jimmy/gmac	4/11/2005								10		0	400	0	180	300	Nucleation 400 C, 300 mT, Si2H6 100, 10 min.
wlow	4/12/2005	351.4	6.1	112	112		89.9	1.42	60		119	440	6	50	600	Nucleation 440 C, 300 mT, 100 Si2H6, 10 min. DOE recipe 11 with adjusted SiH4 flow for MFC cal.
jimmy/gmac	4/13/2005	350							6		0	400	0	180	300	Nucleation 400 C, 300 mT, Si2H6 100, 10 min.
Megnum	4/19/2005	350	6.02	112	112		89.9		10		150	440	12	50	600	Nucleation 440 C, 300 mT, Si2H6 100, 10min.
jimmy/gmac	4/22/2005	350							16		0	400	0	180	300	Nucleation 400 C, 300 mT, Si2H6 100, 60 min. 2 etr runs, 1kA undoped Ge
Megnum	4/23/2005	330	6.16	112	112		89.9		5		140	425	12	60	600	Nucleation 425 C, 300 mT, Si2H6 100, 1 min. Recipe aborted since gas ring was set to on (cannot flow
Megnum	4/25/2005							2.13	140		140	425	12	60	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
Equevy	5/2/2005		6.2						125		115	425	20	55	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
Equevy	5/3/2005	330	6.43						125		115	425	20	55	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
Equevy	5/14/2005								72		0	350	12	100	300	No nucleation.
Equevy	5/15/2005		6.25						80		0	350	12	100	300	No nucleation.
blakelim2	5/16/2005	330	6.25	112			89.9		20+10		0	350	12	180	300	No nucleation.
equevy	5/17/2005								285		0	425	20	50	600	No nucleation.
equevy	5/17/2005								30		0	350	12	100	300	No nucleation.
bob	5/18/2005															Tystar5 shutdown because of a brief power outage, ~12 noon. The bank has been restarted, the pumps turned on and the standby recipes loaded. The tystar20 MFS-1 (460 controller) is giving an error: FLOWCNTRL ID. I have not seen this error before. We cannot reset it I have a call into Tystar for guidance on a fix. Tystar20 is down until this problem is cleared.
jimmy/gmac	5/18/2005															Talked to Tystar engineer and obtained flow ID. Checked the ID with MFS460 dip switches. Found one switch was set wrong. Reset the dip switch and the problem went away.
equevy	5/20/2005	330	6.2	118					24		0	350	12	100	300	No nucleation.
equevy	5/20/2005	330							12		0	350	12	100	300	No nucleation.
equevy	5/21/2005	330	6.2	118			89.9		470		120	425	20	50	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
bob	5/23/2005															GeH4 bottle changed.
blakelim2	5/24/2005	350	6.26	112			89.5	2.82/NA	485/535		1100/110	425/425	20/20	70/60	400/600	No nucleation. Bilayer process.
Megnum	5/28/2005	350	6.28	112			89.9	2.02	100		130	425	10	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
equevy	6/7/2005	350	6.1	118					60		115	425	20	55	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
equevy	6/8/2005	350	6.2	119				2	16		0	350	12	100	300	No nucleation.
equevy	6/8/2005	350							16		0	350	12	100	300	No nucleation.
equevy	6/9/2005	350							410		120	425	20	50	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
Megnum	6/11/2005	350	6.27	112			89.5	2.74	660		120	410	18	50	600	Nucleation 410 C, 300 mT, Si2H6 100, 2 min.

User	Date	V1	V2	V3	V4	V5	V6	V7	V8	V9	V10	Note
Megpoum	6/11/2005											The SiGe film on my wafers turned out to be very hazy and non-uniform. I am not sure why is this, I am trying to identify if this is due to a furnace problem or a process issue. The sheet resistance of the film was pretty low.
Megpoum	6/13/2005											I had 5 patterned wafers and 2 SiO2 test wafers distributed throughout boat 6' boats. Patterned wafers were more hazzy than non patterned wafers. This indicates that maybe it's due to a pre-furnace cleaning process. I used SVC14 and Di-water since I had SiGe films underneath. I am still trying to find the best wet cleaning process prior to the furnace.
Megpoum	6/18/2005	330	6.27	112	89.5		140	425	12	60	600	Nucleation 300 C, 300 mT, Si2H6 100, 10 min.
equvvy	6/21/2005	330	6.2	118	89.2		20	350	12	100	300	No nucleation
equvvy	6/22/2005	330					120	425	20	50	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
Megpoum	7/9/2005	330	631	112	2.78		25	410	18	30	600	Nucleation 410 C, 300 mT, Si2H6 100, 5 min. Process aborted.
Megpoum	7/9/2005											I found the furnace aborted after 25min of deposition. When checking the history to find out the problem, the ABORT step says Pressure High. My wafers are still in the furnace.
bob	7/11/2005											Tystar20 has been reset and Marie will remove her wafers. Jimmy Chang needs to review the history file for this tube to learn why the recipe aborted. Marie requests a monitor run once tystar20 is back up and running.
jimmygmac	7/11/2005											I found that the process ended after the nucleation step. Si2H6 flowed 5 minutes and pressures stayed at 300 mtorr. after that the dcs30 pc show not process gas flow the reason could be that the furnace was disabled. re-run the process with the process parameter. process went thru nucleation and 5 minutes into deposition without problem. pressure control was within 1 minute to the set point. could not repeat the problem. in the future, please do not remove wafers or run any other recipe. it will delete all the alarm infor in the file. tystar20 is up for use.
bob	7/12/2005											The keyboard for tystar20 has been replaced. Tystar20 is ready to use. Performing GeH4 maintenance. The LOW GeH4 was tested at several flows. It seems to be working correctly.
Megpoum	8/20/2005	330	635	115	89.5	2.25	360	425	105	12	70	600 — Nucleation 425 C, 300 mT, Si2H6 100, 5 min.
Megpoum	8/20/2005						120	410	125	12	50	600 — Continuous runs. Nucleation 410 C, 300 mT, Si2H6 100, 3 min.
Megpoum	8/20/2005					2.23	100	410	115	12	60	600 — Continuous runs. Nucleation 410 C, 300 mT, Si2H6 100, 3 min.
Megpoum	8/20/2005					2.23	80	410	105	12	70	600 — Continuous runs. Nucleation 410 C, 300 mT, Si2H6 100, 3 min.
bob	8/17/2005											tystar20 is aborting SiGe recipes. The process pressure and gas control has been checked and does not seem to be the issue. t20 is enabled in my name pending review by Jimmy Chang.
Megpoum	10/18/2005					2.2	170	410	125	12	50	600 — No nucleation.
Megpoum	10/18/2005					2.2	130	410	105	12	70	600 — No nucleation.
bob	10/18/2005											Tystar20 is operating correctly and Marie has completed her run. we do not know why two previous run attempts aborted but it is likely the shutoff of the toxic gases for the addition of an mfm may have been the cause. Tystar20 is up.
Megpoum	10/25/2005	330	636	115	89.5	2.2	240	410	135	12	40	600 — No nucleation
Megpoum	11/28/2005							410	130	12	45	600 — Recipe aborted
Megpoum	11/28/2005											Marie's recipe was aborted 5 minutes into deposition. Her recipe: SiH4=130, GeH4=45, BCl3=12, 410 deg C, 600 mTorr. Record computer shows SiH4 and BCl3 flow were fine. GeH4 flow was 45 sccm, but setpoint was 0. PRCPR was 138 mTorr, but setpoint was 0. N2VAC was 0, didn't respond for pressure control.
wlsw	11/28/2005											Test gas flow manually, SiH4, BCl3, PRCPR and N2VAC all respond to input value. But GeH4(low) and GeH4(high) cannot flow. Jimmy found there was not compress air signal sent to the GeH4 prevalve.

User	Date												Comment
jimmyggmac	10/31/2005												found that a valve in the gas cabinet was turned off. turned on the valve and the gas flow without problem. equip staff please turn on the valve after finishing jobs. tystar20 is up for use.
megonum	11/1/2005	330	6.26	115	89.5	2.22	210	410	130	12	45	600	No nucleation
Megonum	11/3/2005	330	6.3	115	89.5	2.2	360	425	105	12	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 5 min.
Megonum	11/3/2005	330		115	89.5	2.22	210	410	130	12	45	600	No nucleation.
tystar	11/8/2005												Henry Heidbreder of Tystar Co. is here to change the definition table and update the furnace. MFM loop is hooked up. When GasRing = on, all gases (dopant and precursor) can run through the MFM loop for MFC calibration. BC13 MFC has been updated from 20 to 50 sccm full range. Remove GeH4(lo), PH3 and B2H6 channels. EXPTN1 and EXPGR are available for new dopant and precursor. Modify PIN setting for temperature control.
wlw	11/8/2005												GeH4 tank is empty. N2 is running overnight through the BC13 line after the MFC change. Temperature calibration is in progress.
wlw	11/9/2005												Temperature calibration did not finish. Edit standby and deposition recipe. Add a 5-min leak monitor step to recipes.
bob	11/9/2005												GeH4 bottle changed. BC13 is back on.
wlw	11/9/2005	436	6.83	115	87								Leak monitor - door open right before.
wlw	11/9/2005	356	6.41	115	87								Leak monitor - door open right before.
wlw	11/9/2005												Run MFC monitor manually.
wlw	11/9/2005	NA	10				10	430	140	30	60	600	Leak monitor - door kept close for a few hours. Check recipe. Nucleation 430 C, 300 mT, Si2H6 100, 5 min.
jimmyggmac	11/9/2005												Data record in DCS30 do not match the definition table. N2Dope is off by 10x, PH3, GeH4(low), GeH4(high) are still there. N2MFM, BCL3, etc do not show properly.
jimmyggmac	11/10/2005												Start temperature calibration for the range of 300 - 450 C.
wlw	11/14/2005												Temperature cannot stabilize at 300 C over the weekend. Restart calibration for the range of 350 - 400 C.
jimmyggmac	11/16/2005												End zones temperature are off by a few degree. Center zones temperature are good to 0.5 deg C.
wlw	11/16/2005												Change configuration in DCS30. Now gas channels show up properly.
wlw	11/16/2005												Run MFC monitor manually.
Megonum	11/17/2005					2.1	120	425	105	12	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 2 min.
Megonum	11/18/2005	330	6.27	115	87	2.1	240	425	105	12	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 10 min.
Megonum	11/18/2005					2.1	130	410	130	12	45	600	No nucleation
wlw	11/18/2005												Run MFC monitor manually.
wlw	11/21/2005												Some old dummy wafers have very thick deposition and stick on the quartz boats. Take all boats out and clean in Si etcher.
wlw	11/21/2005	343.8	6.25	115	87		60	430	140	30	60	600	Nucleation 430 C, 300 mT, Si2H6 100, 10 min.
wlw	11/22/2005												Set up recipes: SIGEINONU (deposition w/o nucleation), MULLA (12-layer deposition), MONVAR (MFC monitor for regular users)
wlw	11/22/2005												While running standby recipe, "no auto" alarm in the back. Silence alarm and reset computer. Process seemed to proceed, but aborted after some time. Cannot display history on the screen.
jimmyggmac	11/23/2005												no auto alarm comes on when the FCS10 has power failure. if it is reset for long time, over a few seconds, the alarm will show up. since all the alarm messages were erased, could not know the cause. restart the coating (standby) recipe and it finished without problem. this problem is cleared for now.
wlw	11/28/2005	349.9	6.22	115	87		230	430	140	30	60	600	Nucleation 430 C, 300 mT, Si2H6 100, 10 min.
wlw	11/29/2005	349.5	6.35	115	87		30*11	430	140-190	30	60	600	Nucleation 430 C, 300 mT, Si2H6 100, 10 min. SiH4 ramping experiment (+5 sccm per layer), 11 layer deposition.

242

User	Date											Notes
wlw	11/30/2005	NA	NA	NA	NA	30*11	430-380	140	30	60	600	Nucleation 430 C, 300 mT, Si2H6 100, 10 min. Temperature ramping experiment, (-5 deg C per layer). 11 layer deposition.
wlw	11/30/2005											Run MFC monitor manually.
Magnum	12/1/2005	330	6.9	115	87	80	425	105	12	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 5 min.
Magnum	12/1/2005	NA	NA	NA	NA	60	410	130	12	45	600	No nucleation
wlw	12/2/2005											Modify BTOUTB recipe and set gas ring to off so that the MFM line is inactive for the 5000 sccm N2BKFL.
wlw	12/8/2005											Run MFC monitor manually.
wlw	12/10/2005											Center zone temperature off calibration. Set temperature is 450 C and actual temperature is ~160 C.
jimmygmac	12/12/2005											Found that the TCU was locked up. Reset TCU all temperature read normal except the center zone. The center zone temp readout was fluctuated. There was a "B" symbol next to the temp readout out. It indicated that the TC was not working. Swapped the center zone t/c with other zone t/c. Found that the t/c was working. It is suspected that the t/c block has a bad contact, or we have a electronic problem. Tystar20 is down until equip staff fixed the t/c problem.
wlw	12/15/2005											Created recipe MONCON for MFC monitor. There are three set values for each gas.
peetsl	12/16/2005											Bad connections at both the TCU board and TC block. Connection re-establish and temperature now reading correctly from TC.
wlw	12/16/2005											Start temperature calibration for the range of 300 - 450 C. Started TCU calibration yesterday for 350-450 deg C and actual temp at 450 deg C (within 0.5 deg C). Temperature calibration should be done, but people said it started alarming since last night.
wlw	12/17/2005											I disconnected the TCU section on the pull-down menu communication -> disconnect, then file -> exit DCS30 gives error "TCU communication failure". The furnace is operational, but DCS30 cannot capture data.
wlw	12/17/2005					60	410	150	30	50	1200	Nucleation 410 C, 300 mT, Si2H6 100, 10 min. The temperature stabilization for the standby recipe (450 deg C) takes forever. The temperature has to be good to within 1 deg C. The tolerance in the recipe is 5 deg C for the center
wlw	12/18/2005					60	410	150	30	50	700	zones and 10 deg C for end zones. This starts to happen after the TCU communication failure error. I ran a deposition recipe SiGeBCl3 with set temperature 410 deg C. It can
						60	410	140	30	60	700	stabilize within an hour and the tolerance is correct.
wlw	12/18/2005											Run MFC monitor with recipe MONCON.
wlw	12/18/2005	350.5	6.31	116	87	60	410	150	30	50	900	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
jimmygmac	12/19/2005											Reset TCU. DCS30 now talking with tystar20 and collecting data. the standby recipe was corrupted. the tolerance was set to default. Reload the recipe.
wlw	12/19/2005											Standby recipe aborted 3 times. It always happened at the beginning of the Si2H6 coating step. I tested the Si2H6 flow manually and couldn't see any problem.
wlw	12/20/2005	360	6.31	116	87	60	410	150	30	50	700	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
						60	410	140	30	60	700	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
jimmygmac	12/21/2005											Found that the reason for abort was N2DOPE instead of Si2H6. Ran the standby recipe twice with recipe and once manually, not problem with the process.
wlw	12/21/2005	348	6.43	119	87	60	410	140	45	60	350	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
wlw	12/22/2005	449.7	6.97	119	87	230	410	140	30	60	600	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
Magnum	12/22/2005	330	6.46	119	87	120	425	102	12	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 5 min.
Magnum	12/22/2005					100	410	130	12	45	600	No nucleation. Second layer of bi-layer.
wlw	12/22/2005	458.6	7.01	119	87	230	410	140	45	60	600	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
wlw	12/23/2005											Run MFC monitor with recipe MONCON.
jimmygmac	12/23/2005											Tystar20 lost all the configuration when a lab member entering the recipe. Reset the FCS10 using cs command. Down load configuration file from DCS30. MFS460 #2 alarmed for BAD RAM. Reset it and the alarm cleared. Down load all the process recipes needed.
wlw	12/26/2005	349.9	6.45	116	87	313	410	140	45	60	350	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
wlw	12/26/2005	350.2	6.49	121	89	250	410	140	15	60	600	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
wlw	12/27/2005	349	6.4	119	89	340	410	140	30	60	350	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
wlw	12/28/2005	351.7	6.55	119	89	360	410	140	15	60	350	Nucleation 410 C, 300 mT, Si2H6 100, 10 min.
wlw	12/28/2005											Run MFC monitor with recipe MONCON.

243

User	Date											Notes
wlow	1/10/2006											Run MFC monitor with recipe MONCON.
wlow	1/10/2006											I used the MONCON recipe to run weekly MFC monitor. The "gas ring" switch has a bad connection. The dopant gas went through the injector path rather than the MFM path. This was noticed with the reading on the injector pressure gauge and the MFM reading. The actual value of the gas ring follows the setpoint on the screen. I had to switch the "gas ring" on and off a few times in the RM page to get it to actually turn on.
peatal	1/11/2006											What was thought to be a simple bad connection turns out to likely be a malfunction in the electronics in the MFS-460. Currently in communication with Tystar for help with trouble shooting mfs 460 I/O board components. Tystar20 is down for now.
peatal	1/12/2006											swapped out MFS-460 I/O board with a replacement. MFS now correctly controls the gassing solenoid. Malfunction most likely due to bad open collector transistor Q1 (2N6038) on the MFS-460 I/O board. Looking into replacing. Tystar20 is up for use.
wlow	1/12/2006	350	64	117	89	20	410	47	35	20	300	No nucleation. Pressure not stable
wlow	1/12/2006											1/12/2006, recipe SiGeNoNu. Pressure jumps between 150 - 530 mTorr for the following recipe: 410 deg C, 47 sccm SiH4, 20 sccm GeH4, 35 sccm BCl3, 300 mTorr, 20 min. The data is record in DCS30 on
jimmygmac	1/13/2006											As a reference, 350 mTorr was fine in the past for 140 sccm SiH4, 60 sccm GeH4, 15-45 sccm BCl3. tystar furnace pressure control parameters are optimized for high gas flow (>200sccm total) and high pressure (300 mtorr and above). in order to run low gas flow (<100sccm total) or low pressure, the control parameter needs to be changed again. will talk to the super user about the gas flows and show equip engineer how to adjust the parameters. tystar20 is still up for most of processes.
jimmygmac	1/13/2006											adjusted the pressure control parameters according the tube use. new values are: reset 20, delay 0.5, offset 0.0 Super user is test both high and low pressure deposition and ok with it. tystar20 is up
wlow	1/13/2006	448	6.9	120	89	20	410	47	35	20	300	No nucleation.
wlow	1/13/2006					15	430	47	35	20	300	No nucleation.
wlow	1/20/2006											Run MFC monitor with recipe MONCON.
wlow	1/20/2006											Start TCU calibration for300-450 deg C.
wlow	1/25/2006	348.6	6.36	120	89	15	350	0	12	100	300	No nucleation. Process aborted at the beginning of the deposition, because BCl3 flow was higher than setpoint. Restarted the process manually and went OK.
wlow	1/26/2006	349	6.31	120	89	8	300	0	6	50	300	No nucleation. 56 sccm of N2 flow during deposition.
wlow	1/26/2006	451	6.86	120	89	15	350	0	0	100	300	No nucleation. 12 sccm of N2 flow during deposition.
wlow	1/26/2006	449.7	6.83	120	89	8	300	0	0	50	300	No nucleation. 62 sccm of N2 flow during deposition.
wlow	1/30/2006											Run MFC monitor with recipe MONCON.
wlow	2/7/2006											Created recipe SiGeSeed. SiH4, GeH4, BCl3, Si2H6, temperature and pressure are all variables in nucleation step. Main deposition follows nucleation immediately. Gas flow rates and pressure in the main deposition are variables, but temperature has to be the same as nucleation.
wlow	2/7/2006	349.9	6.35	120	89	5	410	140	35	60	600	Nucleation 410 C, 300 mT, SiH4 47, GeH4 20, BCl3 35, 5 min. Test SiGeSeed recipe.
wlow	2/7/2006											Activate BNTLK interlock for steps with toxic gas flow for all recipes. Increase safety factor for potential leak.
wlow	2/9/2006	350.1	63	117	89	260	410	140	35	60	600	Nucleation 410 C, 300 mT, SiH4 47, GeH4 20, BCl3 35, 20 min.
wlow	2/9/2006											Run MFC monitor with recipe MONCON.
bob	2/10/2006											GeH4 bottle change. New pressure 137 psi.
wlow	2/20/2006											Run MFC monitor with recipe MONCON.

User	Date											Comment
wbw	2/28/2006											Power outage for about an hours. Inspec furnace and found no particle problem.
wbw	3/1/2006	349.7	638	120	89	480	410	140	35	60	600	Nucleation 410 C, 300 mT, Si2H6 100, 10 min. An earth quake happened during depo - 3.4M in Orinda. Everything is fine.
wbw	3/1/2006											Run MFC monitor with recipe MONCON.
wbw	3/10/2006											Run MFC monitor with recipe MONCON.
heikam	3/15/2006	350.4	629	117	89							
wbw	3/17/2006	351.6	632	151	89	130	410	47	35	20	300	no nucleation
wbw	3/17/2006											Standby pressure a lot higher than normal. Base pressure test with no gas flow and pump running at full power, 61 mT. Rate of rise - no change in 5 minutes. (As a reference, Tystar19 has a base pressure of 0 mTorr and rate of rise of 6 mTorr in 2 minutes.) Open the tube and found quartzware close to the door turned blue/black. The color should be grey with SiGe coating. One of the cantilever has a crack at the pump end. Run standby recipe and leak check failed. Total 185 hours of deposition. These have been much ordered from LP Glass and are expected to arrive tomorrow. All the other quartzware on tystar20 has been replaced so that upon arrival the sheath can be installed, tube can be leak checked and temperature calibration can commence. Tystar20 will most likely be available wednesday.
pestal	3/27/2006											
pestal	3/29/2006											Thermocouple has been installed, tube has passed leak check, heaters are on. Tube ready for coating and calibration by process staff.
jimmygmac	3/29/2006											started standby recipe (coating). temp is still low (~50C). Will check tomorrow morning to see the status.
wbw	3/29/2006											Run MFC monitor with recipe MONCON.
wbw	3/30/2006											Press and gas flow test to confirm setup. Injector's orientation is wrong. Start temperature calibration.
pestal	3/30/2006											Injector orientation has been corrected.
jimmygmac	3/30/2006											finished coating. temp/heater calibration done 300-450C. tystar20 is running standby recipe and is up for use.
wbw	3/30/2006											Cannot open the door with boatout recipe. N2BKFL, PRCPR, BNTLK, GNTLK and BOATSPD are all correct on the display. Also verify the N2 flow with the MFM loop. It seems like the tube is not vented - loosening the cable and pull on the door does not work. Also try reseting the boatloader. Out of idea...
equery	4/1/2006	350				200	425	100	30	70	600	Nucleation 425 C, 300 mT, Si2H6 100, 0.5 min
equery	4/1/2006	330				72	350	0	12	100	300	No nucleation
pestal	4/3/2006											vented tube and found boatloader stuck. Cable had slipped off pulley near tube door end was caught, inhibiting boatloader from being manually or automatically pulled out. Everything has been resituated and boatloader is working fine. Tystar20 has been left pumping down and is ready for use.
wbw	4/6/2006	350.5	62	117	89	8.5*4	410	140	30	60	350	Nucleation 410 C, 300 mT, Si2H6 100, 10 min. 4 layer stacks, open door between depositions.
wbw	4/7/2006	357.4	624	120	89	45	350	0	12	100	300	Nucleation 350 C, Si2H6 100, 300 mT, 20 min. Pure Ge deposition to cover CMOS.
wbw	4/7/2006											Cannot open door automatically with bottomth recipe. I have to pull in on the door and press "boat out" on the key broad to get the door out.
pestal	4/7/2006											Boatloader in-limit flag found too far "in," no longer closing the auto-in switch. When the boatloader was commanded to open, the flag, on the way out, would then close the auto-in switch causing the boatloader to think it needed to pull itself closed. Adjusted in-limit flag to keep the auto-in switch closed when boatloader was all the way in. Boatloader now functions as it should. Tystar20 is available for use.
blahshin2	4/8/2006	330	623	120	89	35	425	140	18	60	600	Nucleation 425 C, Si2H6 100, 300 mT, 10 min.
wbw	4/9/2006	347.8	623	120	89	20/230	410	140	35	60	350/600	Nucleation 410 C, Si2H6 100, 300 mT, 10 min. Bilayer deposition, no interruption in between.
equery	4/10/2006	330				300	410	140	35	60	600	Nucleation 410 C, Si2H6 100, 300 mT, 5 min.
wbw	4/10/2006	362.9	624	120	89	10	450	20	0	0	1200	No nucleation. Helf Erik Garnett on Pt catalyst nanowire grow test. Used 4" caged boat.
heikam	4/10/2006					57	450	130	30	70	600	Nucleation 450 C, Si2H6 100, 300 mT, 10 min.
wbw	4/10/2006											Run MFC monitor with recipe MONCON.

245

User	Date											Comments
blahblah2	4/12/2006	350	626	120	90	115	425	130	12	70	600	Nucleation 425 C, Si2H6 100, 300 mT, 10 min.
blahblah2	4/12/2006	350	626	120	90	70	425	140	18	60	600	Nucleation 425 C, Si2H6 100, 300 mT, 10 min.
egarrett	4/13/2006	330	624	120	89	15	450	100	0	0	600	No nucleation. Try to grow nanowire w/ Pt catalyst
egarrett	4/13/2006											One of the 6" open boats is broken.
pestal	4/14/06											Broken wafer boat replaced.
marycancam	4/14/2006	442	6.7	120	89	180	440	130	18	70	600	Nucleation 440 C, Si2H6 100, 300 mT, 10 min.
blahblah2	4/15/2006	352	63	117	89	180	410	130	12	70	600	Nucleation 410 C, Si2H6 100, 300 mT, 10 min.
wbw	4/20/2006											Run MFC monitor with recipe MONCON.
egarrett	4/21/2006	350.1	625	120	89	15	450	100 Si2H6	10	0	300	No nucleation. Try to grow nanowire w/ Pt catalyst. Also try to deposit poly-Si on Ag catalyst nanowire. (Ag removed)
egarrett	4/26/2006	349.9	631	120	89	15	450	100 Si2H6	5	0	300	No nucleation.
wbw	4/27/2006											Tystar20 started doing temperature stabilization for the standby coating since last night. The center zone has a "B" next to the actual temperature. All other zones are at ~400 C, where the setpoint is 450 C. the "B" next to the temp reading means that the thermal couple in the tube is not working. the spike t/c is used as a backup instead since the spike t/c fluctuates more, it takes longer to stabilize the temp.
jimmygmac	4/28/2006											tried resetting TCU, and FCS10, no effect. tried securing the t/c connection, no effect. equip staff please check the t/c and connections to find out that there is any open circuit. Tystar20 is usable, but it is better wait for the equip staff to fix the t/c. Just like last time, this problem is caused by a faulty connection in the J1 connector coming out of the back of the TCU. Last time I was able to fix the bad connections using spare pins in the connector, but seeing as there are no more spare pins, I think it's due time to do a complete rebuild of the connector. This will be done when the Tystar Tech comes up some time this week (possibly next week.) For now, the "B" is gone and Tystar20 is available for use.
pestal	5/1/2006											Run MFC monitor with recipe MONCON.
wbw	5/1/2006	330	623	120	89	130	350	0	12	100	300	Nucleation 350 C, Si2H6 100, 300 mT, 20 min.
blahblah2	5/4/2006											Center zone thermocouple has bad connection. Temperature jumps from 35 deg C to 500 deg C. The heater is working hard to respond to the false temperature reading.
wbw	5/4/2006											Unplugged unstable center T/C to force TCU to use the "back up" center T/C. Tystar20 can be used in this state, temperature stabilization may take longer. This faulty T/C connection will be fixed permanently Tuesday.
pestal	5/4/2006											
egarrett	5/5/2006	350.1	628	120	89	90	450	100 Si2H6	10	0	300	No nucleation. Nanowire experiment.
tystar	5/9/2006											Henry Heitbrink of Tystar Co. fixed the TC problem.
equery	5/11/2006	330				72	350	0	12	100	300	No nucleation.
wbw	5/11/2006											Run MFC monitor with recipe MONCON.
blahblah2	5/14/2006	330	633	120	89	120	425	140	12	60	600	Nucleation 425 C, Si2H6 100, 300 mT, 10 min.
equery	5/16/2006	330				300	410	140	45	60	600	Nucleation 410 C, Si2H6 100, 300 mT, 5 min.
jimmygmac	5/18/2006											the center tfc showed "B" after the temp reading. it means the profile tfc is malfunction. either the t/c itself or the connection needs to be checked. the temp control is using the spike t/c outside the tube now. tystar20 is still usable.
wbw	5/19/2006											Edit SiGeNoNu recipe, add N2BKFL as a variable. Pressure can reach 2000 mTorr with 1500 sccm of N2BKFL for Erik Garnett's nanowire experiment.
egarrett	5/23/2006	353.3	624	120	89	90	450	100 Si2H6	5	0	300	No nucleation. Recipe aborted about 5 min short.
egarrett	5/23/2006											Furnace aborted during post-deposition purge/pump step. Cannot unload wafers until problem is resolved. Wafers left in and furnace disabled. the set time was 1:30:00, but it aborted about 5 minutes short. on the alarms history, the tempc faults were the cause of abort check on found that the deposition was not finished.
jimmygmac	5/23/2006											DCS30 computer, there were serveral spikes on the temp. monitor the status and found that the "B" showed up on Center and SC zones. this is an old problem that has not been fixed.

User	Date	Comment
pstal	5/23/2006	Same faulty connector problem. Working to get parts to rebuild connector problem. Also, fixed boat loader problem caused by bootloader in-limit flag being too far in, which caused the bootloader to be forced in when it was trying to move out by retipping boot-in switch. Can process stuff run a test run to see if TC connections are stable please?
wlow	5/23/2006	Run MFC monitor with recipe MONCON.
jimmygmc	5/24/2006	checked the DCS pc and found that there were still a few temp spike readings. temp1, temp1c, temp2c... tempc are ok, but tempc and tempc are jumping up and down. tystar20 still has temp problem.
pstal	5/25/2006	The suspicious connector has been completely rebuilt. Watching stability of temperature readings over night to determine if this connection is the culprit or if we are dealing with a misbehaving TCU board.
wlow	5/26/2006	Microlab account inactive. No more babysitting for Tystar20.
pstal	5/31/2006	spiking temperatures have gotten better but have not disappeared. Odd nature of the spikes makes me believe it is either a communication problem, or a TCU board problem.
pstal	5/31/2006	Swapped out TCU Board, tube will need to sit a while to collect that to see if spikes are gone. Tube will also need calibration.
pstal	5/31/2006	after TCU board change, temperature reading stability is significantly better. All tystar20 needs now is calibration.