

JECMA5 19 (10) 1007-1149 (1990)

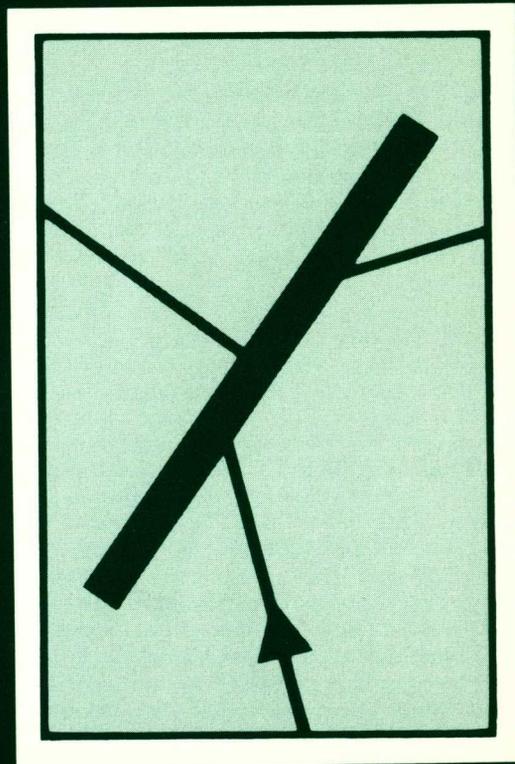
ISSN 0361-5235

# Journal of ELECTRONIC MATERIALS

Volume 19, Number 10

October 1990

SPECIAL ISSUE ON LOW TEMPERATURE  
SILICON EPITAXY



**TMS**  
Minerals • Metals • Materials



**IEEE**

A Joint Publication of  
The Minerals, Metals & Materials Society  
and the  
Institute of Electrical and Electronics Engineers

K7871  
566

# Foreword

The papers appearing in this special issue were presented at a symposium sponsored by the Electronic Device Materials Committee of TMS, held during the 1990 Annual Meeting in Anaheim, California. The symposium received contributions representing a wide spectrum of interests in the silicon epitaxy research community; covering the topics of growth techniques, hetero-structural growth, structural characterization and device performance. Of particular significance in this collection is that research on materials grown by molecular beam epitaxy as well as chemical vapor deposition techniques are presented in a unified fashion for the first time. Therefore, this volume is dedicated to the integration of the materials science issues pertaining to the growth and device performance of silicon based epitaxial structures. The editors gratefully acknowledge the assistance of the numerous reviewers, with a special acknowledgment to Drs. J. Regolini and J. Sturm for their assistance in this symposium. Last, but not least, we also wish to thank all the authors for their contributions on this important subject.

*Guest Editors:*  
Martin L. Green  
AT&T Bell Laboratories  
Murray Hill, NJ 07974

Krishna Rajan  
Materials Eng.  
Department  
RPI  
Troy, NY 12180

Rafael Reif  
Dept. of Electrical  
Engineering  
MIT  
Cambridge, MA 02139

## SPECIAL ISSUE PAPERS

Dislocation Configurations in Si/Si <sub>x</sub> Ge <sub>1-x</sub> Strained Layer Heterostructures <i>Krishna Rajan</i>	1009
Oxygen and Carbon Incorporation in Low Temperature Epitaxial Si Films Grown by Rapid Thermal Chemical Vapor Deposition (RTCVD) <i>M. L. Green, D. Brasen, M. Geva, W. Reents, Jr., F. Stevie and H. Temkin</i>	1015
NMOSFET Characteristics on a Double Hetero-Epitaxial Si/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> /Si Structure Fabricated by a New MBE Growth Method <i>H. Hanafusa and H. Ogata</i>	1021
Very Low Defect Remote Hydrogen Plasma Clean of Si (100) for Homoepitaxy <i>B. Anthony, T. Hsu, L. Breaux, R. Qian, S. Banerjee and A. Tasch</i>	1027
The Influence of Defects on Device Performance of MBE-Grown Si Homojunction and Strained Si <sub>1-x</sub> Ge <sub>x</sub> /Si Heterostructures <i>D. X. Xu, G. D. Shen, M. Willander, J. Knall, M.-A. Hasan and G. V. Hansson</i>	1033
Defect Microstructure in Single Crystal Silicon Thin Films Grown at 150° C–305° C by Remote Plasma-Enhanced Chemical Vapor Deposition <i>T. Hsu, B. Anthony, L. Breaux, R. Qian, S. Banerjee, A. Tasch, C. Magee and W. Harrington</i>	1043
In Situ Temperature Measurement by Infrared Absorption for Low-Temperature Epitaxial Growth of Homo- and Hetero-Epitaxial Layers on Silicon <i>J. C. Sturm, P. V. Schwartz and P. M. Garone</i>	1051
Silicon Molecular Beam Epitaxy: Highlights of Recent Work <i>John C. Bean</i>	1055
Low Thermal Budget Solid Phase Epitaxial Growth of CaF <sub>2</sub> on Si (111) Substrates <i>R. Singh, R. P. S. Thakur, A. J. Nelson, S. C. Gebhard and A. B. Swartzlander</i>	1061
Low Temperature, Defect-Free Silicon Epitaxy Using a Low Kinetic Energy Particle Process <i>Tadashi Shibata and Tadahiro Ohmi</i>	1065
Reduced Pressure and Temperature Epitaxial Silicon CVD: Kinetics and Applications <i>J. L. Regolini, D. Bensahel and J. Mercier</i>	1075
Heavily P-Doped (>10 <sup>21</sup> cm <sup>-3</sup> ) Si and SiGe Films Grown by Photo-CVD at 250° C <i>A. Yamada, Y. Jia, M. Konagai and K. Takahashi</i>	1083
Remote Plasma-Enhanced CVD of Silicon: Reaction Kinetics as a Function of Growth Parameters <i>B. Anthony, T. Hsu, L. Breaux, R. Qian, S. Banerjee and A. Tasch</i>	1089
Low-Temperature Epitaxy Using Si <sub>2</sub> H <sub>6</sub> <i>F. Mieno and Y. Furumura</i>	1095
The Kinetics of Dislocation Glide in SiGe Alloy Layers <i>C. G. Tuppen and C. J. Gibbings</i>	1101
In Situ STM Characterisation of Epitaxial Layers in a 3-Inch MBE System <i>R. Butz and H. Wagner</i>	1107
Three Dimensional Devices Fabricated by Silicon Epitaxial Lateral Overgrowth <i>G. W. Neudeck, P. J. Schubert, J. L. Glenn, Jr., J. A. Friedrich, W. A. Klaasen, R. P. Zingg and J. P. Denton</i>	1111
Delta Doping Superlattices in Silicon <i>H. P. Zeindl, E. Hammerl, W. Kiunke and I. Eisele</i>	1119
Selective Silicon Epitaxial Growth on a Submicrometer WSi <sub>2</sub> Grating: Application to the Permeable Base Transistor <i>P. A. Badoz, D. Bensahel, L. Guérin, P. Perret, C. Puissant and J. L. Regolini</i>	1123

## REGULAR ISSUE PAPERS

Rapid Thermal Chemical Vapor Deposition of Germanium on Silicon and Silicon Dioxide and New Applications of Ge in ULSI Technologies <i>M. C. Öztürk, D. T. Grider, J. J. Wortman, M. A. Littlejohn, Y. Zhong, D. Batchelor and P. Russell</i>	1129
Grain Growth Mechanism in Boron Doped Polycrystalline Silicon <i>S. Kalainathan, R. Dhanasekaran and P. Ramasamy</i>	1135
Selective Area Growth of GaAs with Semiconductor-Metal-Semiconductor Structures Using Pulsed-Mode Operation of MOCVD <i>Chang-Hee Hong, Chang-Tae Kim and Young-Se Kwon</i>	1141
Shallow, Small Area, TiSi <sub>2</sub> Contacts to n <sup>+</sup> and p <sup>+</sup> Silicon <i>A. H. Perera and J. P. Krusius</i>	1145

# In Situ Temperature Measurement by Infrared Absorption for Low-Temperature Epitaxial Growth of Homo- and Hetero-epitaxial Layers on Silicon

J. C. STURM, P. V. SCHWARTZ and P. M. GARONE

Department of Electrical Engineering, Princeton University, Princeton, N.J. 08544

The use of infrared transmission to measure silicon wafer temperature in a lamp-heated susceptor-free reactor is described. The relevant temperature range is 400 to 800° C, and the accuracy is on the order of a few degrees centigrade. The method is then applied towards the growth of silicon and silicon-germanium alloy layers on silicon substrates. For silicon-germanium layers typical of those used in heterojunction bipolar transistors, no change in absorption compared to that of the silicon substrates is observed.

**Key words:** Si-Ge, temperature measurement, low temperature epitaxy

## I. INTRODUCTION

A goal of many researchers in the epitaxial growth field is the development of low-temperature (<800° C) epitaxial growth processes on silicon. In this temperature range the growth rate of chemical vapor deposition (CVD) processes is usually controlled by surface reaction processes, with the result that the growth rate is a strong function of temperature. Typical silicon activation energies of ~2.0 eV give an order of magnitude change in growth rate from 700 to 800° C. In the case of Si<sub>1-x</sub>Ge<sub>x</sub> alloy films on silicon, in addition to growth rate, temperature will also affect the final composition.<sup>1</sup> The change in film thickness and composition will affect the critical thickness for strain stability, which in turn will strongly affect the defect density in the final film and interfaces.<sup>2</sup>

One approach to such low-temperature growth involves the use of lamp heating and no susceptor as is common in rapid thermal processing.<sup>3</sup> In this rapid thermal processing or Limited Reaction Processing (LRP) approach, temperature measurement is difficult since there is no susceptor from which the temperature may be measured with a thermocouple (assuming ideal thermal contact between the susceptor and the wafer). In such susceptor-free reactors, one typically would use a pyrometer to measure the temperature, usually at 5 μm (to avoid interference from the tungsten halogen lamps used to heat the wafer) for work in the 600° C range.<sup>4</sup> This in practice has two significant drawbacks which make it difficult if not impossible to obtain an accurate measure of the absolute temperature. First, quartz is opaque at 5 μm, and special windows are required to view the wafers. This is inconvenient when conventional quartz reactor tubes are used. Second, accurate knowledge of the temperature requires knowledge of the emissivity. In the 400 to 800° C range, silicon emissivity is a strong function of temperature, wavelength,<sup>5</sup> doping, surface layers

such as field oxides, etc. In this paper a new non-invasive optical approach to silicon temperature measurement is described, and then applied towards the epitaxial growth of silicon and silicon germanium alloys on silicon substrates. The method relies on the temperature dependence of the infrared absorption of silicon.

## II. INFRARED ABSORPTION IN SILICON

Infrared absorption in silicon can proceed in general by two processes: band to band absorption and free carrier absorption. Band to band absorption is an indirect process in silicon, and hence requires phonons, whose population is a strong function of temperature due to Bose-Einstein statistics. Further, it is well known that the bandgap in silicon is a decreasing function of temperature ( $E_G = 1.12$  eV at 25° C, 0.96 eV at 400° C, and 0.80 eV at 800° C).<sup>6</sup> Therefore one expects a strong increase in band to band absorption with increasing temperature for wavelengths near the bandgap. The free carrier concentration in silicon is also a strong function of temperature since the bandgap decreases with temperature. For example, the intrinsic carrier concentration ( $n_i$ ) is  $2 \times 10^{17}$  cm<sup>-3</sup> at 600° C and greater than  $10^{18}$  cm<sup>-3</sup> at 800° C.<sup>6</sup> Therefore, for moderately doped substrates (< $10^{17}$  cm<sup>-3</sup>) one expects a strong temperature dependence of free carrier as well as band to band absorption.

We have adapted a quartz-walled system for rapid thermal CVD for the in situ measurement of infrared transmission at 1.3 and 1.55 μm (Fig. 1).<sup>7</sup> Semiconductor lasers are modulated and coupled into an optical fiber, whose output is then focussed through a hole in the reflector walls onto the wafer. A detector under the wafer detects the transmitted signal through another hole in the reflector walls. Two key points for this technique are (1) lock-in amplifiers are used to recover the transmitted signals independent of any lamp interference or infrared emission by the wafer itself, and (2) the transmitted signal at any temperature  $T$  is always

(Received February 20, 1990; revised June 4, 1990)

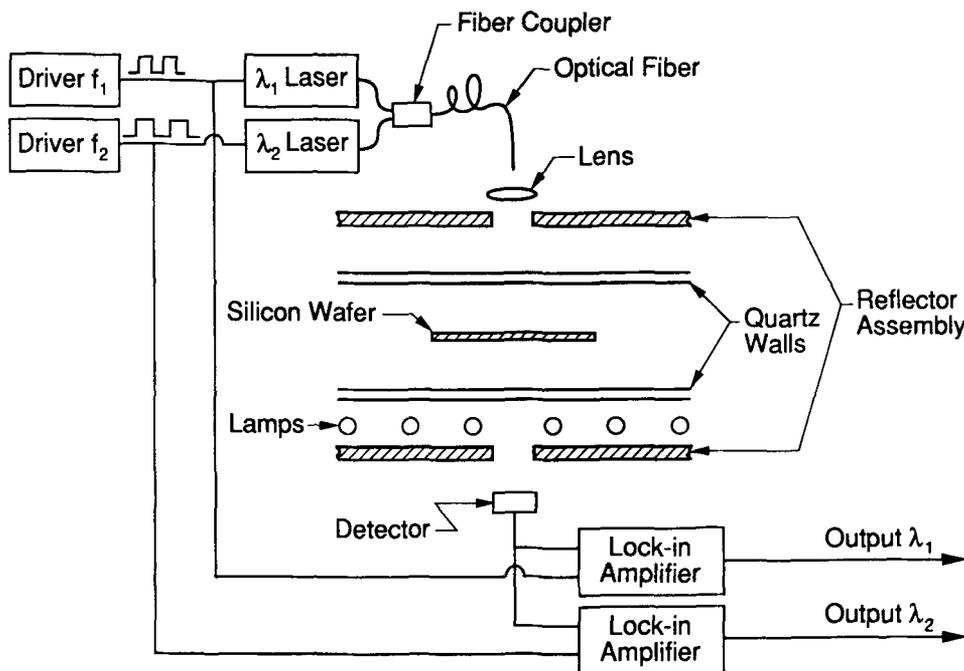


Fig. 1 — Schematic of the Limited Reaction Processing chamber adapted for in situ infrared transmission measurement.

normalized (divided) by the room temperature signal for that wafer. Since moderately doped wafers have negligible absorption at room temperature, the room temperature signal depends only on factors such as laser power, detector efficiency, scattering from the rough wafer backside, surface reflection, etc. Since it is easily shown that these factors all have negligible temperature dependence, the normalized signal  $t(T)$  depends only on optical absorption, and one curve (Fig. 2) should describe all wafers of similar thickness. In Fig. 2, squares and triangles represent data measured in the chamber of Fig. 1. The temperature was measured by a thermocouple integrally welded to the wafer. The circles represent independent data of absorption measured in a spe-

cially adapted conventional furnace where the temperature was measured by conventional thermocouple techniques. The two sets of data are in good agreement, giving confidence in the results. As can be seen, the higher energy photons ( $1.3 \mu\text{m}$ ) have higher absorption as would be expected from band to band absorption. Further work is needed to determine how heavy substrate doping affects these curves.

### III. APPLICATION TO EPITAXIAL GROWTH

When growing silicon in the reaction-rate limited regime, temperature control on the order of a few degrees centigrade is required for accurate control of the film thickness (e.g. 5%). The required sensitivity and laser stability can be found from the slope of  $t(T)$  in Fig. 2. For example, at  $750^\circ\text{C}$ , one degree resolution requires detecting a 3% relative change in the  $1.5 \mu\text{m}$  signal, easily done with simple electronics. Near  $600^\circ\text{C}$ , one degree resolution would require under 1% relative resolution on the  $1.55 \mu\text{m}$  signal, but only 4% resolution on the  $1.3 \mu\text{m}$  signal. Therefore, for highest resolution it is advantageous to operate at the shortest wavelength that gives a detectable signal. Over the range of  $500$  to  $800^\circ\text{C}$ , temperature resolution on the order of one degree is indeed possible. Indeed, the biggest problem to date is the calibration of the absolute temperature scale in Fig. 2 due to thermocouple uncertainties (typically 1%). No dependence of the transmission on the process gases (typically 6 Torr of hydrogen and 60 mTorr of dichlorosilane) has been observed.

The optical transmission through a wafer depends on the wafer thickness, which changes during

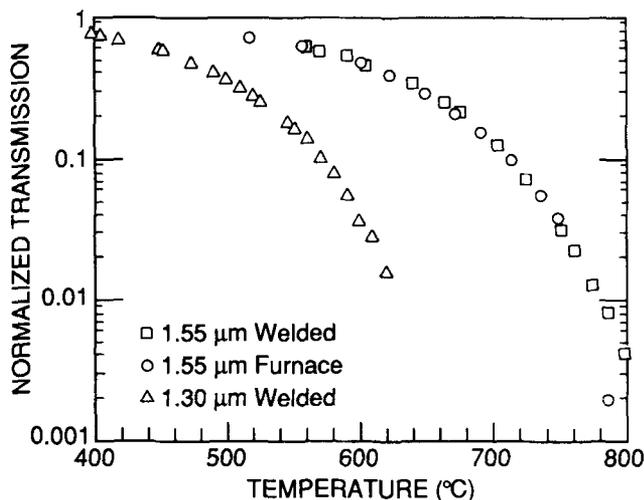


Fig. 2 — Optical transmission divided by room temperature transmission at 1.3 and  $1.55 \mu\text{m}$  at elevated temperature for a  $450\text{-}\mu\text{m}$  thick wafer ( $p$ -type,  $20\text{--}50 \text{ ohm-cm}$ ).

epitaxial growth. The thickness dependence of the transmitted signal will then limit the application of this technique to epitaxial growth. Because of the rough wafer backside and extra absorption, multiple internal reflections may be ignored, and one may model the thickness dependence of transmission by a single  $e^{-\alpha \cdot d}$  term, where  $\alpha$  is the absorption coefficient and  $d$  is the wafer thickness. Using this relationship, Fig. 3 shows the expected effect of a  $\pm 50 \mu\text{m}$  change in the transmission through a  $450 \mu\text{m}$  wafer. Over the complete temperature range of interest this causes a  $\pm 12^\circ\text{C}$  error in extracted temperature, assuming no correction for the thickness change is made. Therefore, the growth of less than  $5 \mu\text{m}$  of epitaxial silicon would cause less than a one degree change in inferred temperature. Using a wafer with a welded thermocouple for temperature measurement, we have measured normalized transmission vs temperature before and after the growth of  $2 \mu\text{m}$  of epitaxial silicon. As expected from the above argument, the normalized transmission was indeed unchanged within the repeatability of our system (a few degrees). We now use this method routinely for the growth of all epitaxial silicon samples in our reactor at  $800^\circ\text{C}$  or less. At present, our upper temperature limit of  $800^\circ\text{C}$  is due to the small signal received by the detector. Higher temperatures will require increased laser power (currently  $\sim 1 \text{ mW}$ ), improved detection electronics, reduced scattering from the wafer backside, or switching to a wavelength with lower absorption. Although many (greater than 50) process runs are possible without visible deposition on the quartz tube, according to the above thickness arguments, wall deposition on the order of microns should have little effect on the technique, especially if this thickness changes little during the growth cycle for a single wafer.

As explained earlier, an important motivation for our work is the growth of  $\text{Si}_{1-x}\text{Ge}_x$  structures on silicon. In this case temperature control is even more important than in the case of silicon growth be-

cause the growth temperature affects the film composition for fixed gas flows. For example, the same gas flows that yield a composition of  $\text{Si}_{0.77}\text{Ge}_{0.23}$  at  $625^\circ\text{C}$  yield a composition of  $\text{Si}_{0.87}\text{Ge}_{0.13}$  at  $700^\circ\text{C}$ .<sup>1</sup> Changes in germanium fraction can exponentially affect the equilibrium critical layer thickness for misfit dislocation formation. For layers grown metastable beyond the equilibrium critical thickness, the amount beyond the equilibrium critical thickness is a driving force for misfit dislocation propagation. Further, the kinetics of relaxation (dislocation nucleation and propagation) are strong functions of temperature.

Since  $\text{Si}_{1-x}\text{Ge}_x$  alloy films have a bandgap lower than that of silicon, for a given epitaxial layer thickness, higher absorption would be expected than for a silicon film, making the  $5 \mu\text{m}$  upper thickness limit of silicon homoepitaxy overly optimistic for this heteroepitaxial case. However, the current main application of  $\text{Si}_{1-x}\text{Ge}_x$  alloys is as the base layer in heterojunction bipolar transistors (HBT's). In such structures, 30% and 50 nm appear to be reasonable upper limits for the germanium mole fraction  $x$  and layer thickness, respectively. To test the usefulness of the absorption technique for temperature measurement of such structures, normalized transmission was first measured on a silicon substrate with a welded thermocouple used for absolute temperature measurement.  $54 \text{ nm}$  of  $\text{Si}_{0.67}\text{Ge}_{0.33}$  was then grown on both sides of the wafer (total thickness  $108 \text{ nm}$ ). The transmission vs temperature was measured again, using the same normalization value, with the results shown in Fig. 4. The two sets of points (before and after SiGe growth) are the same within the error of our measurements. Therefore one may use optical absorption (with silicon absorption calibration) for the growth of  $\text{Si}_{1-x}\text{Ge}_x$  with less than 33% Ge and less than  $108 \text{ nm}$  thickness. The upper limits of allowable germanium fraction as a function of thickness have not yet been determined.

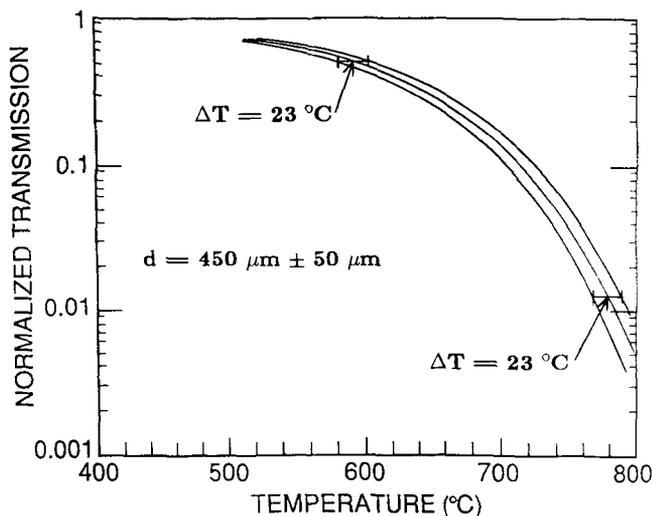


Fig. 3 — Effect of a  $\pm 50 \mu\text{m}$  change in wafer thickness on normalized transmission and extracted temperature.

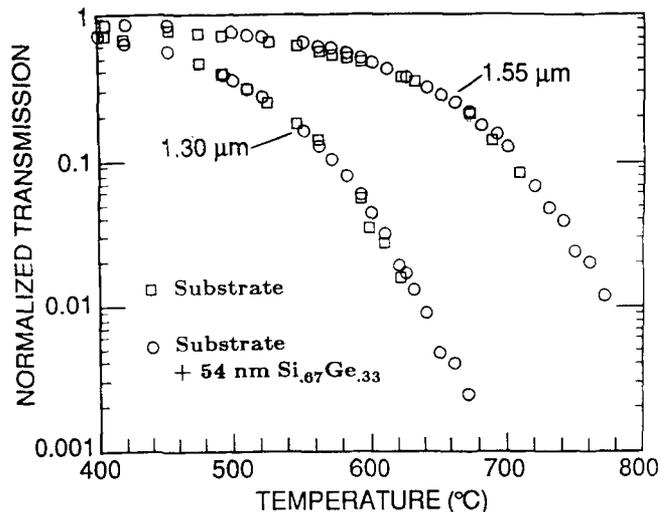


Fig. 4 — Normalized transmission vs temperature before and after the growth of a  $\text{Si}_{0.67}\text{Ge}_{0.33}$  layers  $54 \text{ nm}$  thick on both sides of a silicon substrate.

#### IV. CONCLUSION

A new technique for the in-situ measurement of temperature during epitaxial growth on silicon has been described and demonstrated. The technique relies on the temperature dependence of infrared absorption, and offers a method to measure silicon wafer temperature from 500 to 800° C, with an absolute accuracy on the order of a few degrees. No knowledge of emissivity is necessary. The method also is well suited to the growth of silicon-germanium alloy layers on silicon for heterojunction bipolar transistor applications.

#### ACKNOWLEDGEMENTS

The support of ONR (N00014-88K-0396), NSF (ECS-86157229) and Texas Instruments is greatly appreciated.

#### REFERENCES

1. P. M. Garone, J. C. Sturm, P. V. Schwartz, S. A. Schwarz and B. A. Wilkens, *Appl. Phys. Lett.* **56**, 1275 (1990).
2. J. C. Bean, L. C. Feldman, A. T. Fiory, S. Nakahara and I. K. Robinson, *J. Vac. Sci. Technol A-2*, 436 (1984).
3. C. A. King, J. L. Hoyt, D. B. Noble, C. M. Gronet, J. F. Gibbons, M. P. Scott, T. I. Kamins and S. S. Laderman, *IEEE Elec. Dev. Lett. EDL-10*, 159 (1989).
4. R. E. Sheets, in *Rapid Thermal Processing*, Proc. Symp. Mat. Res. Soc. Vol. 52, eds, T. O. Sedgwick, T. E. Seidel and B-Y. Tsaur 191 (1986).
5. T. Sato, *Jpn. J. Appl. Phys.* **6**, 339 (1967).
6. C. D. Thurmond, *J. Electrochem. Soc.* **122**, 1133 (1975).
7. J. C. Sturm, P. V. Schwartz and P. M. Garone, *Appl. Phys. Lett.* **56**, 961 (1989).