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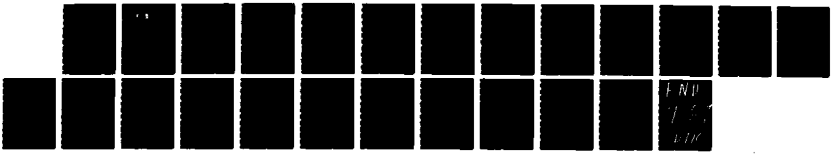

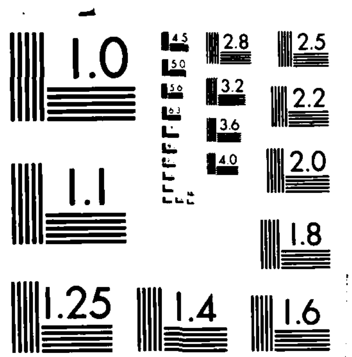
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The objective of this program is to carry out basic research on the phenomenon of surface-energy-driven secondary grain growth (SEDSGG) in thin films on amorphous and single crystal substrates. Means of enhancing grain boundary mobility, such as ion bombardment, doping, and rapid thermal annealing are investigated. Theoretical models for SEDSGG are developed. The role of surface patterning in SEDSGG is also studied.

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"Single Crystal Films of Semiconductors on Amorphous Substrates
Via a Low Temperature Graphoepitaxy"

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Cambridge, MA 02139

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TABLE OF CONTENTS

1.0 Introduction

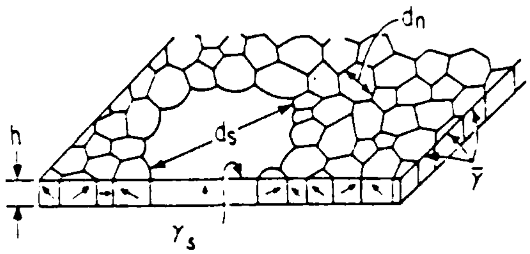
As the title of this grant indicates, the long-range motivation of this research effort is the development of a technology for producing single-crystal films on amorphous substrates. In response to suggestions from our sponsor in the latter part of this past year, and because of some important phenomena we have observed (see Section 3.0), we are now emphasizing the acquisition of basic understanding of the energetics and kinetics of grain growth in ultrathin films rather than developments aimed at short-term demonstrations of single-crystal films on amorphous substrates. By ultrathin films we mean films with thicknesses in the sub-500Å range, in which surface energy driving forces play a significant role. Our effort has also been expanded to include investigations of ultrathin films on single-crystal substrates, in recognition of new insights we have gained on the possible role of surface-energy-driven grain growth in some types of heteroepitaxy. Until the initiation of our research program there was very little research in the domain of ultrathin films or enquiries into the role of surface energy anisotropy in morphological and structural changes. We are confident that our basis studies will provide the underpinnings for achieving the long-range technological objective: new film configurations for advanced electronic systems.

In Section 2-8 of this report we describe briefly the knowledge we have gained about grain growth using a variety of approaches, and summarize our current views in Section 9.

2.0 Surface-Energy-Driven Grain Growth - Basic Concepts

The phenomenon of surface-energy-driven grain growth is depicted schematically in Fig. 1. If a polycrystalline film is sufficiently thin, normal

Secondary Grain Growth



$$\Delta\gamma = \gamma_s - \bar{\gamma}$$

The driving force due to surface energy anisotropy

$$\Delta F = \frac{2A\Delta\gamma}{Ah} = \frac{2\Delta\gamma}{h}$$

To get large grains with uniform texture
decrease the film thickness

Figure 1. Schematic depiction of surface-energy-driven grain growth (SEDGG). A grain with minimum surface energy, γ_s, is shown growing into a matrix of normal grains with average surface energy $\bar{\gamma}$.

grain growth will tend to produce columnar grains in which the boundaries are perpendicular to the surface plane. Normal grain growth, which is driven by the reduction of grain boundary energy, occurs if the grain boundaries are able to move. However, normal grain growth tends to cease when the columnar grain diameters are approximately twice the film thickness. Among the columnar grains some are oriented such that their surfaces have minimum energy. These grains can grow further to consume their neighbors, becoming large secondary grains with a specific crystallographic texture. This is the phenomenon we call surface-energy-driven secondary grain growth

(SEDSGG). Our studies of this phenomenon have been the most extensive to date and have focused on thin films of semiconductors as well as a few metals. The driving force for SEDSGG includes a term due to surface energy minimization, as depicted in Fig. 1, and also a term due to grain boundary energy.

$$\Delta F \cong \frac{2\Delta\gamma}{h} + \frac{\gamma_{gb}}{h} \qquad (1)$$

In this equation, $\Delta\gamma$ is the difference between the surface energy of the growing secondary grain, γ_s, and the average surface energy of neighboring normal grains, $\bar{\gamma}$. The grain boundary energy is γ_{gb} and h is the film thickness.

In our experimental and theoretical work to date we have focused on SEDSGG in films on amorphous substrates. If these substrates are planar, γ_s has a minimum value for grains with restricted textures, but there is no restriction on in-plane or azimuthal orientations. On patterned or single-crystal substrates, however, γ_s has minimum values for grains with 3-dimensionally constrained orientations. That is, secondary grains will differ in surface energy depending on their in-plane orientation relative to the single-crystal or patterned substrate. It is likely that in many cases of heteroepitaxy that occur by Volmer-Weber growth, orientation is achieved not at the stage where discrete islands exist on the substrate, but at the stage where islands coalesce. In this case, the achievement of epitaxy should be considered a form of surface-energy-driven secondary grain growth. This viewpoint has not been clearly expressed previously in the literature. Under this research grant we are applying our knowledge and experience with

SEDSGG on amorphous substrates to fundamental studies of the initial stages of heteroepitaxy.

Figure 2 illustrates how surface-energy-driven secondary grain growth (SEDSGG), in conjunction with patterning of an amorphous substrate surface, can lead to a film with a specific in-plane orientation as well as a specific axis perpendicular to the film. Growth to impingement of three-dimensionally oriented grains can lead to single-crystal films.

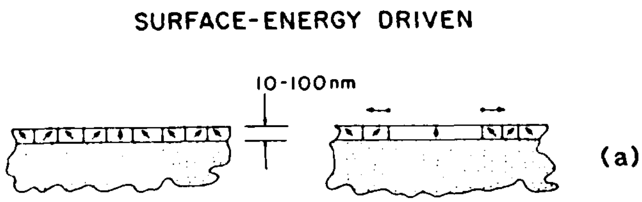


Figure 2(a). Schematic cross section of a film undergoing SEDSGG. The grain with minimum interfacial energy by virtue of its orientation grows by consuming grains with other orientations.

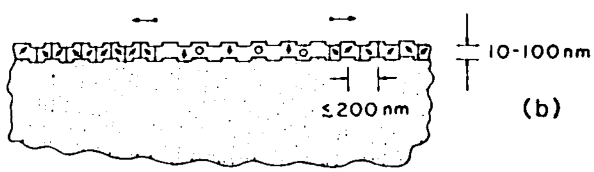


Figure 2(b). SEDGG in conjunction with surface patterning (solid-state-graphoepitaxy). A grain of minimum interfacial energy is one that is oriented relative to the surface pattern as well as the substrate normal.

We are hopeful that the basic knowledge acquired under this research grant of the SEDSGG phenomenon on planar and patterned surfaces will ultimately enable us to provide guidelines for a technology capable of achieving device-quality single-crystal films on amorphous substrates. Such a technology should enable new film configurations for advanced electronic systems to be achieved.

The phenomena observed in thin and ultrathin films are more complex than depicted in Figs. 1 and 2. For example, the top surfaces of polycrystalline films are not planar as depicted. Instead, grooves form at grain boundaries. Some of the most startling phenomena we have observed were in films so thin (150 Å) that they formed a network, but were not entirely continuous. Despite the discontinuous nature of the films, extremely large grains with a specific crystallographic texture were formed by SEDSGG. This is discussed in Section 3.

A major component of our efforts is on means of enhancing grain boundary mobility in films of Si and Ge. Such studies provide new information on how grain boundaries move in covalently bonded materials, as well as offering hope that SEDSGG can be applied at moderate or low temperatures. These studies are described in Sections 4 to 6.

3.0 SEDSGG in Ultrathin (≲150Å) Films

In ultrathin films of Ge, about 150Å thick, secondary grains many micrometers in diameter were observed after annealing, despite the presence of a high density of voids in the film. One would normally expect that such voids would pin grain boundary motion and thereby suppress formation of large secondary grains. We attribute these somewhat surprising results in part to very high surface-energy driving forces. Presumably, in such ultrathin films the driving force is sufficiently high to overcome the inhibitory effects of the voids. The anomalous secondary grain growth in ultrathin Ge films is similar to secondary grain growth observed in 150Å-thick films of Au by an earlier graduate student, Chee Wong.

Ultrathin films, in which surface energy is a major factor in the energetics, is an exciting new area of materials science. Because of the potential for providing significant new knowledge, as well as new technology, we have, over the last 5 months, focused a large portion of our efforts on this area. The initial observations were made on Ge films that were capped with SiO_2, except in one case where the ultrathin condition occured as a result of ion-beam thinning. (Capping was used due to the tendency of Ge films to oxidize on annealing, and because of certain problem of contamination.) Our subsequent efforts have been to achieve secondary grain growth in uncapped films where the driving force would be even larger. We have solved the contamination and oxidation problems but so far the tendency of Ge films to agglomerate has held us up. We believe this problem will be solved over the next month.

4.0 Ion-Beam-Enhanced Grain Growth

In February 1987 graduate student Harry Atwater completed his PhD thesis, which was a broad study of the enhancement of grain boundary motion by means of ion bombardment. His study of ion-beam-enhanced grain growth (IBEGG) was the first of its kind and may open up new fields of study and engineering. He studied Ge, Si, and Au films less than 1000Å thick. Ion beams in the 40 - 100 keV range were employed, resulting in an ion damage profile whose peak is approximately in the center of the thin film. Concurrent with ion bombardment, samples were annealed at 500 - 1000 °C for Ge and Si, and at room temperature for Au. The temperature was chosen so that ion damage is annealed dynamically. IBEGG was characterized by varying the ion dose, ion energy, ion flux, ion species, temperature, and thin film deposition conditions. The effect of these parameters on grain size and microstructure was analyzed both qualitatively and quantitatively using transmission electron microscopy (TEM). A transition state model was developed to describe the motion of grain boundaries during ion bombardment.

The model accounts for the dependence of IBEGG on all experimental parameters. An atomistic picture of the jump rate at grain boundaries during IBEGG was proposed. Monte-Carlo simulation of ion range and defect production was performed using the TRIM code and a modified Kinchin Pease formula. The calculated defect yield per incident ion was correlated with enhanced grain growth, and used to estimate the number of atomic jumps at the grain boundary per defect generated at the boundary for a given driving force, a quantity which is approximately constant for a given film material. The IBEGG and thermal growth rates have been related to their respective point defect populations. That is, the grain growth rate appears to depend only on the concentration of vacancies and interstitials, irrespective of whether they are created thermally or by ion bombardment. We consider this a rather important finding.

The grain growth observed in Ge under ion bombardment was normal, that is, a monomodel distribution with random orientation was obtained. Only at the highest doses (10^{16} cm^{-2}) was there a hint of secondary grain growth. To pursue this point we plan to utilize low energy bombardment as discussed in Section 10.

5.0 The Effect of Dopants on Grain Growth in Silicon

In our studies of ion-beam-enhanced grain growth we arrived at the conclusion that grain boundary mobilities can be greatly enhanced due to injection of point defects at the boundaries. We have reached the same conclusion from separately funded studies of the effect of dopants on grain growth in polycrystalline silicon films.

We have observed that doping with electron donors, specifically phosphorous and arsenic, leads to significant enhancement of the rates of both normal and secondary grain growth in silicon films. We have also observed that doping with boron has little or no effect on either sort of grain growth. However, codoping with boron as well as with an acceptor leads to compensation of grain boundary mobility enhancement. Over the past year we have carried out extensive studies of normal grain growth in silicon in order to develop an understanding of the Fermi energy dependence of grain boundary mobility.

We chose to study normal grain growth in order to avoid effects due to surfaces. This allows more direct determination of grain boundary mobilities. However, the mechanisms of grain boundary motion (but not the driving force) should be the same for both normal and secondary grain growth. Normal grain growth is driven by the reduction of grain boundary energy alone. Phosphorous and arsenic are known to segregate to silicon grain boundaries and therefore almost certainly reduce grain boundary energies. The effect of P and As on grain growth must therefore be specifically due to enhancement of grain boundary mobilities.

P, As, and B doping affect the rates of self diffusion and oxidation in similar ways to their effects on grain boundary mobility. In these cases, kinetic enhancement has been ascribed to changes in point defect concentrations. Similarly, we have related measured grain boundary mobilities to the total vacancy concentration. Vacancies in silicon can be neutral or have a single negative, double negative or single positive charge. While the concentration of neutral vacancies remains fixed, the concentration of negatively charged vacancies increases with increasing

Fermi energy. While the concentration of positively charged vacancies increases with decreasing Fermi energy, the net effect on the total vacancy concentration is negligible. Because the energies for changed vacancies are known, it is possible to calculate the total vacancy concentration as a function of electron concentration and temperature.

When we assume that grain boundary motion occurs through both a diffusive process (e.g. grain boundary dislocation climb) and a non-diffusive process (e.g. grain boundary dislocation glide), we find that the diffusive process scales with the total vacancy concentration. Calculations of grain growth rates predicted using this model are shown as lines and are accompanied by experimental points in Figure 3.

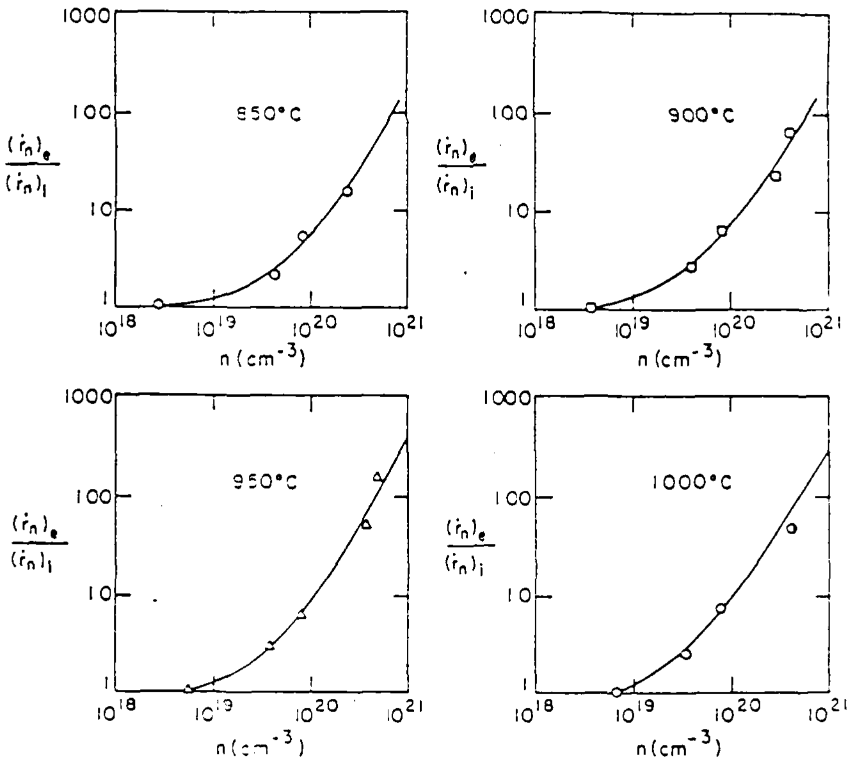


Figure 3. Ratios of the extrinsic and intrinsic normal grain growth rates as a function of electron concentration n and annealing temperature. Lines indicate theoretical predictions and points represent experimental results.

From these results we conclude, as before, that increased concentrations of point defects cause increases in grain boundary mobilities. In the case of ion-beam-enhanced grain growth, point defects are generated athermally due to atomic collision while here vacancies are generated due to the presence of dopants. We will continue to investigate these and other means of stimulating grain boundary motion.

6. Grain Growth by Rapid Thermal Processing

In the past year we completed a brief investigation of rapid thermal annealing (RTA) of doped polycrystalline silicon on amorphous SiO_2. We carried out these studies in order to determine the time dependence of secondary grain growth at very short times. In the work described in section 5, we observed that the rate of secondary grain growth in doped polysilicon was not constant and in fact saturated at very short times. By carrying out RTA experiments we were able to establish that SEDSGG occurred at a constant rate only for the first few seconds of an anneal. During this time, however, the rate of growth was quite high (approximately 700Å/sec at 1100 °C and 4500Å/sec at 1200 °C). The origin of saturation of grain growth is not completely clear at this time. However, through cross-sectional electron microscopy we have observed grain boundary grooves, suggesting that saturation probably occurs due to groove formation.

Our experiments on rapid thermal annealing of polysilicon also yielded an unanticipated new insight. A number of groups have been investigating homoepitaxial transformation of polycrystalline silicon films deposited on single-crystal substrates. When these results are compared to our results we find virtually identical time, temperature and dopant concentration dependencies. This comparison suggests that homoepitaxial transformation of polycrystalline silicon also occurs through a surface-energy-driven grain growth process. This experimental result, as well as theoretical analyses to be described below, have encourged us in our decision to investigate epitaxy which occurs via grain boundary motion.

7.0 Basic Mechanisms of Heteroepitaxy

As previously mentioned, over the past year we have focused attention on the issue that surface-energy-driven secondary grain growth can lead to conventional heteroepitaxy as well as graphoepitaxy. In our experiments so far, we have investigated SEDSGG on planar and patterned amorphous substrates. In the case of planar substrates, surface energy minimization constrains the orientation of a grain relative to rotations out of the plane of the film but not relative to rotations in the plane of the film. For this reason, SEDSGG on planar amorphous substrates leads to grains with restricted textures but random in-plane or azimuthal orientations. In order to constrain 3-dimensionally the orientations of secondary grains, it is necessary that the surface energy be anisotropic for rotations in the plane of the film. This anisotropy can be provided by artificial surface relief or by a single crystal substrate.

We anticipate that in some cases, epitaxy occurs despite the initial nucleation and growth of discrete, randomly oriented islands. In such a system, epitaxial alignment occurs when islands coalesce, at which point grain boundary motion and grain growth can occur. Epitaxy due to grain growth driven by surface energy minimization can be promoted by lattice matching but does not require lattice matching. In our review of the literature on epitaxy we have identified a number of "anomalies" which can be explained using the model outlined above.

We have initiated experimental research on grain growth in polycrystalline films on single crystal substrates. We are currently investigating Au on Silicon and CaF_2 on Silicon.

8.0 Theoretical Developments

We have extended the theory of surface-energy-driven grain growth to allow production of distributions of secondary grain sizes and orientations. This theory has also been extended to allow analysis of homoepitaxy and heteroepitaxy. A manuscript describing these developments is in preparation.

Analysis of distributions of secondary grain sizes and orientations requires understanding not only of those factors which promote grain growth but also those which inhibit grain growth. If a phenomena such as grain boundary grooving inhibits grain growth, there exists a minimum surface energy difference between neighboring grains required for boundary motion to occur. Such a requirement can actually promote increased selectivity for growth of secondary grains. If fewer grains can grow, then those which do grow will attain larger final sizes and have more restricted orientations. These factors can be quantitatively analyzed given knowledge of the orientation dependence of the surface energy, i.e. given a Wulff plot for the energy of the grain/substrate interface. The orientation dependence of surface energies near energy minima can be predicted by a variety of means.

In addition to analytic modeling of secondary grain growth, we are also (under separate funding), i) developing analytical models of 2-D grain growth, ii) developing analytical models for 2-D particle coarsening (including coarsening driven by surface energy minimization) and iii) developing computer models for microstructural evolution in thin films, including nucleation and growth to impingement, normal grain growth and secondary grain growth.

9.0 Current Perspectives on Surface-Energy Driven Secondary Grain Growth

In thin films, especially ultrathin films, surface energy can have dominant importance in phase stabilization and in driving kinetic processes. In this program we are concerned with understanding and controlling microstructural evolution in thin films. We feel that surface-energy-driven grain growth is a process of great importance both during film formation and during subsequent processing. It can be the principal process controlling the final structure of films on planar or patterned amorphous substrates as well as single crystal substrates.

Surface-energy-driven secondary grain growth can be controlled by modifying the driving force for growth or by modifying the grain boundary mobility. Competing processes such as grain boundary grooving (which is also surface-energy-driven) can impede grain growth due to energetic reasons but may in some cases promote orientation selectivity. High surface energy anisotropy also promotes selectivity. Orientation of secondary grains can be three-dimensionally constrained due to surface topography on amorphous substrates or due to interface energy minimization on single crystal substrates. In all cases, the total driving force for SEDSGG increases with decreasing film thickness. This has been clearly confirmed in our experiments on germanium.

Grain boundary mobilities can be increased by generation of point defects. This has been demonstrated in experiments using ion bombardment and electronically active dopants in silicon.

Through the combination of experimental and theoretical analyses discussed above, we feel that we should continue to explore the use of ion enhancement. We will also continue work on ultrathin films both on amorphous and single crystal substrates. In addition to continued study of capped films, we will investigate SEDSGG in uncapped films. This will further allow us to investigate the role of grain boundary grooving while also providing surfaces with higher energies.

10.0 Future Plans

We will continue to emphasize the investigation of secondary grain growth in ultra-thin films. We believe that information gained from studies in this unique domain will provide significant new information on how grain boundaries are induced to move, and, in addition, may provide the basis for technological breakthroughs in semiconductor films on amorphous substrates and heteroepitaxy.

The investigation of ion-beam-enhanced grain growth will be redirected to emphasize low energy (<5KeV) ion bombardment. This work will be done with the ion gun recently installed in the UHV deposition system. This gives us the capability to deposit films under high cleanliness conditions and then to immediately ion bombard these films without breaking vacuum. Because we will be using low energy ions, we can work with ultrathin films (~150Å). In addition, we can balance sputtering and deposition rates to maintain constant film thickness. This will enable us to go to higher doses than previously possible, and should help suppress film beading and surface grooving effects. We are hopeful that such low energy bombardment will further expand our knowledge of grain boundary motion and defect removal. It may also provide a low temperature means of inducing secondary grain growth.

The studies of dopant effects or grain growth have provided us with a wealth of information. This work will be finished up and a PhD thesis submitted.

The investigation of SEDSGG on single-crystal substrates and of the early stages of heteroepitaxy will be continued through the efforts of a senior graduate student. We may seek a second source of funding so that this work can be expanded.

11.0 Publications Under Grant

1. Journal Articles

C.V. Thompson, "Secondary Grain Growth in Thin Films of Semiconductors: Theoretical Aspects", J. Appl. Phys. 58, 763 (1985).

H.-J. Kim, C.V. Thompson, "Compensation of Grain Growth Enhancement in Doped Silicon Films", Appl. Phys. Lett., 48, 399 (1986).

C.C. Wong, H.I. Smith and C.V. Thompson "Surface-Energy-Driven Secondary Grain Growth in Thin Au Films", Appl. Phys. Lett., 48, 335 (1986).

H.J. Frost and C.V. Thompson "The Effect of Nucleation Conditions on the Topology and Geometry of Two-Dimensional Grain Structures," to be published in Acta. Metallurgica.

S.M. Garrison, R.C. Cammarata, C.V. Thompson, and H. I. Smith, "Surface-Energy-Driven Grain Growth During Rapid Thermal Annealing (<10s) of Thin Silicon Films", J. Appl. Phys., 61, 1652 (1987).

J. Palmer, C.V. Thompson, and H.I. Smith, "Grain Growth and Grain Size Distribution in Thin Germanium Films on SiO_2," (submitted to J. Appl. Phys.).

2. Published Conference Proceedings

C.C. Wong, H.I. Smith and C.V. Thompson, "Room Temperature Grain Growth in Thin Au Films." Japan Institute of Metals, 4th International Symposium on Grain Boundary Structure and Related Phenomena, p. 641 (JIMIS-4), Minakami Spa, Japan, November 25-29, 1985.

H.-J. Kim and C.V. Thompson, "The Effect of Dopants on Grain Boundary Mobility in Silicon." Japan Institute of Metals, 4th International Symposium on Grain Boundary Structure and Related Phenomena, p. 495 (JIMIS-4), Minakami Spa, Japan, November 25-29, 1985.

H.J. Kim and C.V. Thompson, "The Effects of Dopants on Surface-Energy-Driven Grain Growth in Ultrathin Si Films", Proc. fall meeting of the Mat. Res. Soc., Boston, MA, Dec. 2-6, 1985.

C.V. Thompson and H.I. Smith, "Secondary Grain Growth in Thin Films", Proc. fall meeting of the Mat. Res. Soc., Boston, MA, Dec. 2-6, 1985.

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H.I. Smith, M.W. Geis, C.K. Chen, and C.V. Thompson, "Crystalline Films on Amorphous Substrates by Zone Melting and Surface-Energy-Driven Grain Growth in Conjunction with Patterning," Mat. Res. Soc. Symp. Proc., 53, 3 (1986).

R.C. Cammarata, C.V. Thompson, S.M. Garrison, and H.I. Smith, "Secondary Grain Growth During Rapid Thermal Annealing of Doped Polysilicon Films," to be presented at Spring MRS meeting.

3. <u>Theses</u>

J.E. Palmer, "Secondary Grain Growth in Ultra Thin Germanium Films on Silicon Dioxide," M.S. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, August, 1985.

C.C. Wong, "Secondary Grain Growth and Graphoepitaxy in Thin Au Films," Ph.D Thesis, Department of Materials Science & Engineering, Massachusetts Institute of Technology, February, 1986.

S.M. Garrison, "The Kinetics of Secondary Grain Growth in Rapidly Thermal Annealed Thin Silicon Films," M.S. Thesis, Department of Materials Science and Engineering, Massachusetts Institute of Technology, June, 1986.

H.A. Atwater, "Ion Beam Enhanced Grain Growth in Thin Films," Ph.D. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, February, 1987.

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