
SYMPOSIUM M

Progress In Bulk Semiconductor Crystal Growth

Symposium M was well attended and most of the papers were concerned with Si. Dr. W. Lin's (AT&T Bell Labs, Allentown) paper was concerned mainly with those factors controlling the chemical uniformity of large diameter crystals within the context of nonstandard Czochralski growth techniques. These techniques are (a) use of a double crucible, (b) application of a magnetic field, and (c) continuous growth. The double crucible process refers to the use of a smaller crucible immersed into liquid silicon contained in a larger crucible. Pulling is done from the smaller of the two and melt connection is made through a capillary. This results in a Si melt of constant composition, thus minimizing segregation. In addition, convection is also reduced because pulling is effectively being done from a smaller liquid volume. Application of a magnetic field decreases thermal gradients and thus decreases convection. Continuous growth is carried out by use of two crucibles. Poly Si is continuously fed into one and this melt is continuously transferred via a transfer tube into the crucible from which growth is done. The volume and composition of the growth melt is thus maintained constant. Thermal asymmetries and convection also are minimized because the volume of liquid is maintained constant during growth of very large crystals. Oxygen in Si derives mainly from the SiO₂ crucibles and it is transferred to the melt-solid interface mainly by convection. The above techniques, particularly the application of a magnetic field, will minimize oxygen incorporation.

Dr. T. Abe (Shin-Etsu Handotai, Japan) discussed the prevailing models of swirl formation in Si and the role impurity doping plays on their formation. B and C impurities give rise to swirl defects, while N significantly reduces swirl concentration. Al and Ga, because of their tendency to deoxidize Si through the formation of oxides, give rise to swirl-free crystals.

Dr. Zhu Li-huei (Electronic Material Research Institute, China) described a reduced pressure (4-15 Torr Argon) Czochralski puller he has been using for growth of 3-4" diameter, 10-20 kg Si crystals. The design of the heating system and the controlled flow of argon over the surface of the melt before exiting resulted in improved control over thermal gradients (and thus convection). Crystals grown in this setup were swirl-free with reduced C contamination and a homogeneous distribution of oxygen.

Dr. C. P. Khattak (Crystal Systems, Salem, MA) described how the Heat Exchange Method, used successfully for the growth of 8" diam. sapphire crystals, was adapted for the production of 45 kg, 32 cm² Si crystals for photovoltaic applications. Crucial for this result was the solution of the SiO₂ crucible problem to produce crack-free crystals.



FRANK WANG (left) and JACK WERNICK

Dr. G. Jacob (CNET, Grenoble, France) argued that native defects in GaAs play the most important role regarding dislocation generation during crystal growth for he found no correlative influence of dopants on mechanical behavior and dislocation generation during growth. Dislocation free Ga₉₉In₀₁As crystals were produced and he further argued that this material should be the one used technologically for producing GaAs-based devices.

Dr. T. Fukuda (Optoelectronics Joint Research Laboratory, Japan) described an automated computer controlled 4" diameter GaAs Czochralski unit for growth of constant diameter crystals for high speed IC devices. Optical fibers are used for sensing and feedback for control. Two superconducting solenoids on the outside of the chamber generate a magnetic field normal to the growth axis. It was shown that the application of a magnetic field decreases the thermal oscillations at the melt-solid interface to near zero and extremely good chemical homogeneity is obtained; there is little evidence of growth striations. EPDs are $\approx 3000/\text{cm}^2$ over a major portion of the diameter. The EL2 trap concentration also decreased. A technique for melt purification prior to growth was also described. At the start of a run, GaAs is synthesized in-situ with excess As and B₂O₃ at a pressure of 70 atmospheres. The pressure is then reduced to 28 atmospheres followed by a further reduction to 1 atmosphere. "Boiling" or bubbling of excess As occurs and it appears that C as CO and Si as SiO is removed by this action.

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small, components can be easily fabricated, and hardening takes place at ordinary temperatures. The annual production of ordinary cement is 1,000 million tonnes per year, and with steel it is the most important material in the construction industry; yet our understanding of cement properties is nothing like as advanced as that of steels. Although there has been an increasing interest amongst materials scientists in cement research in recent years, I believe that we are only at the beginning of an exciting growth area; some of the properties of the new low porosity cements, e.g., Young's modulus of 50 GPa, flexural strength of 150 MPa, and fracture toughness of 1 kJ/m^2 are comparable with those for plastics, some ceramics and some metals, and although, no doubt, validation under service condition will take time, such materials are likely to replace certain plastics, ceramics, and metals for some low temperature applications in the future.

I want to return now to the point I made earlier that materials science and technology, and I find it difficult to draw a clear distinction between the two, is really an enabling technology, aimed at providing the engineer with the materials and processes to turn a design into hardware, and providing the engineer with new opportunities. To bring

this about, it is absolutely essential for the engineer and materials scientist to work closely together and to be aware of each other's needs and opportunities. This theme was emphasized in the talks we heard yesterday in the session on "The Role of Government, Industries, and Universities in the Support and Performance of Materials Research." It is also a central theme in the conference program of this and previous MRS conferences. It is this aspect of bringing scientists and engineers together in symposia concerned with current technological problems and developments, which makes these conferences so stimulating and important. The Boston conference has now established itself as an important annual event in materials science and technology. I am very honored and grateful not only for the award but also for inviting my wife and me to this conference, and I am only sorry I cannot stay for the whole week. On a personal note, I am very pleased to have the opportunity of meeting so many of my old friends and colleagues again, and it is a matter of considerable satisfaction that amongst your contributors and symposia organizers are several members and ex-members of my Oxford department, and members of the AEA. I want to thank you once again, Mr. President, and members of the MRS, for honoring me in this way, and my wife, for giving me the support which enabled me to do the work which has led to this honor.

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Professor R. A. Brown's (MIT) paper was concerned with an analysis of meniscus-defined crystal growth. The shape of the crystals and stability of the processes for EFG, floating zone, and LEC growth are dictated by the interaction between heat transfer from the melt, crystal, and surrounding with the shape of the molten zone, which is dictated by surface tension. In each system the temperature fields in the melt, crystal and die (for EFG) and the shapes of the melt/solid and melt/gas interfaces are calculated by a new finite-element algorithm for solving simultaneously two-dimensional heat transfer models in each phase and the Young-Laplace equation for meniscus shape. Calculations gave predictive results and the applications of the models are only limited by our knowledge of the high temperature thermophysical properties of the material in question.

Dr. E. R. Weber (Berkeley) reviewed the diffusion behavior of several 3-d elements (Co, Ni, Cu) in Si and contrasted their behavior with that of Au. Cu and Ni enter

Si interstitially and diffuse rapidly. The well known diffusion data for Au suggest that during the initial diffusion stages, it diffuses via an interstitial mechanism and then changes over to a substitutional mechanism, thus accounting for the observed decrease in diffusivity at long times. Neutron activation, EPR and DLTS measurements were used to confirm the two well known deep donor and acceptor levels and he showed that Fe, as an accidental impurity, leads to Au-Fe pairs. Two new deep levels are formed which can be ascribed to these pairs. Suitable heat treatments for Si:Au devices were obtained that eliminate the device instabilities that arise from the presence of Au-Fe pairs.

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