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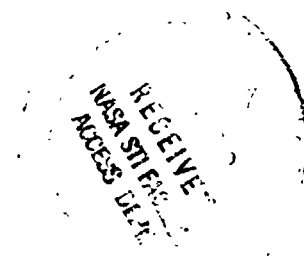
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SOLIDIFICATION (CRYSTAL GROWTH) IN THE PRESENCE
OF GRAVITATIONAL FORCES

(NAS 8-30537)

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FINAL REPORT COVERING THE PERIOD
NOVEMBER 30, 1977 - AUGUST 31, 1978



SUMMARY

The study of "Crystal Growth in the Presence of Gravitational Forces" resulted in a total of eighteen scientific publications which contributed significantly to the advancement of our experimental and theoretical understanding of gravitational effects on crystal growth and dopant segregation. Moreover, this research program served directly and indirectly as a comprehensive ground-based study to optimize crystal growth experiments conducted in space. Thus, the design of the semiconductor growth experiments M-562 (Skylab III-IV) and MA-06⁰ (ASTP) leaned heavily on the results of this investigation as did major portions of the theoretical interpretation of these experiments. For the convenience of the reader, copies of six pertinent publications resulting from this program are attached. (See list of publications on pages 3 and 4.)

PROGRESS REPORT COVERING THE PERIOD
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(comprising three no-cost contract extensions)

During the analysis of the growth experiments conducted in space, as yet unexplained ridge formations on the resolidified semiconductor crystals were observed. In view of the potential of this effect for zero-gravity processing of systems requiring confinement and/or stabilization, it was decided to investigate to the extent possible the surface tension behavior of doped and undoped InSb melts. An investigation of the temperature and composition dependence of InSb melts was moreover deemed desirable since several theoretical studies implied and/or predicted the presence of significant surface tension induced convective flows for unconfined InSb melts.

During the indicated time period we conducted surface tension determinations of InSb melts making use of the sessile-drop technique covering the temperature range from 530 to 880°C. The surface tension of liquid InSb (undoped) and its dependence on temperature was thus determined making use of the Kozakevitch-Urbain approach and it was found, through a linear regression of the data, that the temperature dependence of σ is given by:

$$\sigma = 392 - (T-530) \times (7 \times 10^{-3}) \pm 10 \text{ dyne/cm} \quad 530 \leq T \leq 880^\circ\text{C}$$

The study showed that $d\sigma/dT$ for intrinsic InSb is significantly less (by almost one order of magnitude) than reported by Lazarev and Dashevskii (-0.007 ± 0.018 dyne/cm/°C). It was also found that the addition of dopant elements at concentration levels of 10^{19} did not significantly effect either the absolute value of σ or its temperature dependence. Subjecting the ambient of the InSb melts to partial oxygen pressures, which are equivalent to unintentional oxygen contaminations in actual growth systems, did not change the σ values to beyond the error limits of the experimental technique.

On the basis of the surface tension data obtained, it must be concluded that surface tension induced convective flow velocities in InSb under reduced gravity conditions range from zero to at most 1 cm/sec. Accordingly, no convective interference with dopant segregation can be expected during growth in space since the momentum boundary layer (at the crystal melt interface) associated with any Marangoni-type convective flows would, at the given growth rate, be significantly larger than the predicted diffusion boundary layer thickness.

In view of the fact that, contrary to a theoretical study by S.V. Bourgeois and L.W. Spradley, the experimental investigation of a Te-doped InSb crystal

grown in space indicates steady-state segregation and thus the absence of any convective interference due to Marangoni-type flows in the melt. The apparent discrepancy between theory and the experiment must, on the basis of our investigation, be attributed to erroneous $d\sigma/dT$ values used by Bourgeois and Spradley.

LIST OF PUBLICATIONS

1. Kim, K.M., Witt, A.F. and Gatos, H.C., "A Crystal Growth Method Based on Controlled Power Reduction Under Stabilizing Gradients", J. Mat. Sci. 6 (7) 1036 (1971).
2. Kim, K.M., Witt, A.F., and Gatos, H.C., "Crystal Growth from the Melt under Destabilizing Thermal Gradients", J. Electrochem. Soc. 119, 1281 (1972).
3. Gatos, H.C., and Witt, A.F., "The Problem of Chemical Inhomogeneities in Electronic Materials", presented at Second Cairo Solid State Conference, April 1973.
4. Kim, K.M., Witt, A.F., and Gatos, H.C., "Segregation Behavior in a Stationary Vertical Zone with Converging Interfaces - Pressure Induced Segregational Effects", J. Electrochem. Soc. 121 (3) 448-51 (1974).
5. Carruthers, J.R., and Witt, A.F., "Transient Segregation Effects in Czochralski Growth," presented at International Spring School on Crystal Growth - II, Japan, 1974, in Crystal Growth and Characterization, ed. by R. Ueda and J.B. Mullin, North-Holland Publishing Co., 1975.
6. Witt, A.F., Gatos, H.C., Lichtensteiger, M., Lavine, M., and Herman, C.J., "Steady State Growth and Segregation Under Zero Gravity: InSb", presented at the Third Space Processing Symposium - Skylab Results, Marshall Space Flight Center, Huntsville, Alabama, April 28-May 1, 1974.
7. Gatos, H.C., and Witt, A.F., "The Problem of Chemical Inhomogeneities in Electronic Materials", Recent Advances in Science and Technology of Materials, Vol. I, ed. by A. Bishay, Plenum Press, 1974, p. 59.
8. Gatos, H.C., and Witt, A.F., "Indium Antimonide Crystal Growth, Experiment M-562", NASA Final Report, NASA Contractor Report, NASA Cr-120558, September 1974, Alabama.

9. Witt, A.F., Gatos, H.C., Lichtensteiger, M., Lavine, M., and Herman, C.J., "Crystal Growth and Steady State Segregation Under Zero Gravity: InSb", J. Electrochem. Soc. 122 (2) 276 (1975).
10. Gatos, H.C., Witt, A.F., Lichtensteiger, M., Lavine, M., and Herman, C.J., "Crystal Growth and Segregation Behavior Under Zero Gravity Conditions: InSb", in "Skylab Science Experiments", Science and Technology Series, Vol. 38, American Astronautical Society, 1975, p. 7.
11. Witt, A.F., "Crystal Growth Experiments on ASTP - An Overview", presented at European Space Agency Conference, Frascati, Italy, April 6-8, 1976; Proceedings of the European Space Agency Conference, Frascati, Italy, ESTEC, The Netherlands, 1976.
12. Gatos, H.C., Herman, C.J., Lichtensteiger, M., and Witt, A.F., "Quantitative Determination of Zero Gravity Effects on Crystal Growth from the Melt (Experiment MA-060)", presented at European Space Agency Conference, Frascati, Italy, April 6-8, 1976; Proceedings of the European Space Agency Conference, Frascati, Italy, ESTEC, The Netherlands.
13. Gatos, H.C., and Witt, A.F., "Interface Markings in Crystals: Experiment MA-060", presented at NASA Space Processing Symposium, Marshall Space Flight Center, May 26-27, 1976; Apollo-Soyuz Test Project: Preliminary Science Report, National Technical Information Service, Springfield, Va., 1976, p. 25/1 - 25/8.
14. Witt, A.F., "Phenomena in Czochralski Growth of Semiconductors", presented at First European Conference on Crystal Growth, Zurich, Switzerland, September 12-18, 1976; published in Proceedings, North-Holland Publishing Company, 1977.
15. Martin, E.P., Witt, A.F., and Carruthers, J.R., "On the Application of a Heat-Pipe to Czochralski Growth", presented at ICCG-5, July 18-22, 1977; published in Extended Abstracts, ICCG-5, 1978.
16. Kim, K.M., Witt, A.F., Lichtensteiger, M., and Gatos, H.C., "Quantitative Analysis of the Thermohydrodynamic Effects on Crystal Growth and Segregation Under Destabilizing Vertical Thermal Gradients: Ga-Doped Germanium", J. Electrochem. Soc. 125 (3) 475 (1978).
17. Martin, E.P., Witt, A.F., and Carruthers, J.R., "Application of a Heat-Pipe to Czochralski Growth, Part I: Growth and Segregation Behavior of Ga-Doped Ge", J. Electrochem. Soc. 126 (2) 284 (1979).
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