Letters

A Crystal Growth Method Based on Controlled Power Reduction Under Stabilising Thermal Gradients

A crystal growth method from the melt is presented in which the solidification isotherm is made to advance by controlled reduction of the power input to the heater system (involving no mechanical motion) under conditions of stabilising thermal gradients (absence of thermal convection) [1]. The absence of mechanical motion in the growth system eliminates the problem of random and periodic mechanical perturbations which introduce chemical and crystalline heterogeneities in the growing materials [2]; furthermore, design and construction problems are significantly simplified. Using vertical growth conditions with the crystal-melt interface advancing upwards, stabilising thermal gradients are established and, thus, thermal convection, a major cause of non-uniform dopant segregation, is virtually eliminated.

A schematic diagram of the system as used for single crystal growth of tellurium doped indium antimonide is shown in fig. 1. In our experiments a seed, 3 cm long, machined and etched to 1.3 cm in diameter was fitted in a quartz tube 10 cm long. The tube was positioned inside the circular heater on a water cooled metal block with the seed contacting the cooled surface. Polycrystalline ingot material was placed on top of the seed and the outer large diameter quartz tube, providing for an inert ambient atmosphere, was mounted. After flushing with hydrogen, the furnace was brought to the melting point temperature. The polycrystalline ingot and part of the seed were thus melted and the system was allowed to reach thermal equilibrium. Crystal growth was initiated and sustained by employing automatic power input control and reducing the thermocouple control signal by means of a motor driven bucking voltage placed in series with the thermocouple. Single crystals up to 5 cm long were obtained.

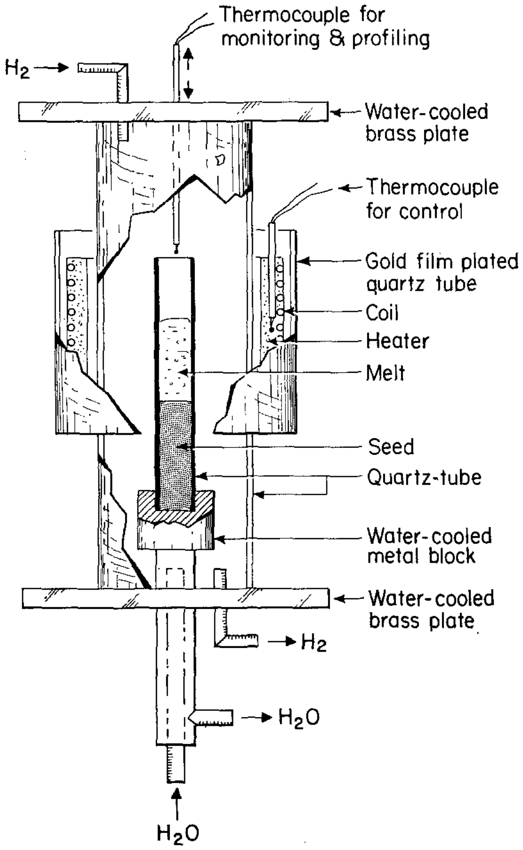

Figure 1 Schematic representation of crystal growth arrangement.

Crystal growth under continuous reduction of input power proceeds with a smooth advancement of the solidification isotherm in direction of the melt. It was found that step-wise manual power reduction at fixed intervals in all instances resulted in abrupt changes in growth rate and thus, in chemical heterogeneities and extensive twinning. The water cooled bottom plate provides for excellent control of vertical thermal gradients and permits the establishment of a flat solidification isotherm.

In the vertical growth configuration with the seed at the bottom, there are no thermal convection currents which are normally present in Czochralski-type growth arrangements where the temperature in the melt increases downwards. Thermal convection in the melt has two adverse effects; it results in temperature fluctuations at the growth interface and thus, in non-uniform dopant segregation. It furthermore tends to reduce thermal gradients in the melt and thus renders heavily doped growth systems more susceptible to the adverse effects of constitutional supercooling [3]. By eliminating thermal convection the present growth method provides a convenient technique for the preparation of homogeneous single crystals over a wide range of dopant concentration since growth interface deterioration due to consitutional supercooling can be retarded or completely avoided. In the absence of convection currents the transport of dopant impurities in the melt is strictly diffusion controlled (diffusion controlled normal freezing) and their distribution in the melt may be conveniently and quantitatively treated by means of the Burton-Prim and Slichter relationship [4].

A number of crystals grown were cut along the growth axis and the dopant distribution was examined with high resolution etching [5]. In all instances the portions of the crystals grown by the present method exhibited no detectable chemical heterogeneities as shown in fig. 2. Several crystals about 4 cm long were grown from melts heavily doped with Sn(10^{19} to 10^{20}/cm^3). No interface degradation due to consitutional supercooling was observed except in the last few millimeters. The same melt composition resulted in virtually instantaneous interface decay when single crystal growth was attempted under optimised conditions in a Czochralski-type growth arrangement.

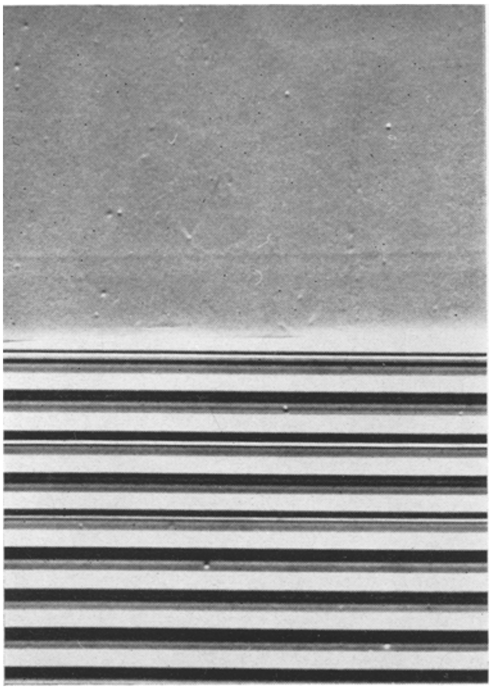

Figure 2 Longitudinal section of a tellurium doped InSb single crystal. The lower striated region is the seed grown by rotational pulling (Czochralski). The upper region, in which the dopant is homogeneously distributed, was grown with the present method (× 400).

In summary, the presently described method makes possible crystal growth in the absence of convection currents in the melt and thus minimises chemical heterogeneities in the crystals; it allows the establishment of steep temperature gradients in the vicinity of the growth interface and thus the convenient growth of heavily doped crystals with delayed interference from constitutional supercooling; it permits the growth of crystals with uniform diameter; it simplifies the design and construction of crystal growth apparatus and lends itself readily to automation.

Acknowledgements

The authors are grateful to Mr Clifford Herman for his expert assistance and advice and to the National Aeronautics and Space Administration for financial support.

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Received 23 March
and accepted 28 March 1971

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