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Short Communication

"The Thermal Wave" in Technology of Crystal Growth from the Melt

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It was found that the temperature fluctuations at the interface crystal-melt are the main reason for the formation of single crystals inhomogeneity grown by the Czochralski method. To reduce the heterogeneity of the layered method is proposed to reduce temperature fluctuations in the melt through the creation of artificial heat wave formed by the modulation of the heater temperature setting of growing single crystals. This paper describes the experimental technique to measure the temperature directly in the field of crystal growth of gallium arsenide from the melt. We investigated the possibility of special control actions for decreasing the temperature fluctuations at the crystallization front. These actions relate to the modification of the thermal and kinetic control parameters normally used in the Czochralski method of crystal growth, such as heater temperature, as well as crystal and crucible rotation rates. Unsteady low energetic thermal control actions (thermal waves, induced by periodic changes of the heater temperature) are able to eliminate temperature fluctuations near the crystal / melt interface and may be a potential tool for the growth of striation-free gallium arsenide single crystals.

Keywords: Crystal growth, Analytical solution, Czochralski crystal growth.

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1. INTRODUCTION

The Czochralski crystal growth is the most common used technique for growing crystals [1, 2]. Fluctuations are often encountered in this process and these are believed to be the cause of inhomogeneities in the crystals that are undesirable in many applications. A number of theoretical and experimental studies were carried out in an attempt to reduce or control the fluctuations in the Czochralski crystal growth process [3-5].

2. CZOCHRALSKI CRYSTAL GROWTH

The Czochralski crystal growth is the most common used technique for growing crystals. Gallium arsenide is placed in a fused quartz crucible, and the crucible is heated up so that the melts. As the raw materials is melting, a seed crystal is attached to a rod and placed on a shaft. When the material is melted, the seed crystal is lowered into the melted material in such a way that the tip penetrates below the surface of the melted gallium arsenide. The melt is held at a temperature slightly above the melting point. The shaft is then rotated counterclockwise while the crucible is rotated clockwise. Due to the rotation of the shaft, the crystal seed is slowly pulled away from the molten in a form of ingot. The diameter of the obtained crystal is maintained by carefully controlling the temperature and rotating speeds of the crucible and the rod [2].

In this work, we investigated die possibility of using special control actions for decreasing the temperature fluctuations at the crystallization front. These actions relate to the modification of the thermal and kinetic control parameters normally used in the Czochralski method of crystal growth, such as heater temperature, as well as crystal and crucible rotation rates. These control actions can be defined as "low-energetic", because they do not change the energy of the system significantly.

The results of previous theoretical research into these methods have not produced a model suitable for practical use. This is due to the complexity of the processes which occur during Czochralski crystal growth as a result of the interactions between convective heat and mass transfer[6-7].

The purpose of the present work was to assess the influence of some of the control actions specific to the creation of "thermal waves" within the melt, in particular to investigate novel effects of controlling the speed of rotation of the crystal and crucible and low frequency vibrational movements of the crystal and melt on the uniformity of properties of GaAs single crystals grown by the Czochralski method.

3. EXPERIMENTS AND RESULTS

The system for measuring the temperature distribution during crystal growth is shown in Fig. 1. A special method was used for fixing thermocouples into the sub-crystalline area which were attached to a graphite "silk" which floated on the surface of the melt. This method allowed the measurement of the temperature at a constant distance from the crystallization front during the whole crystal pulling process.

An experimental investigation was also made concerning the temperature change in the sub-crystalline region across the crystal using co- and counter-rotation of the crystal and the crucible containing the melt and accelerated rotation of the crucible and melt. These actions caused considerable changes in the degree of temperature fluctuations and were much less sensitive in comparison with the heating control. These actions and others with a similar influence on the process of single crystal growth can be classified as "low energetic" actions because the change in power of the heater unit was less than 1 %.

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3.1 Mathematical Modeling and Experimental Investigation of Crystal Growth Processes

Mathematical modeling was carried out using the well known general hydrodynamics model of Czochralski crystal growth. This model included the mass and heat transfer in the crucible on the basis of the dynamic Navier-Stokes equations (Boussinesque approach) [1].

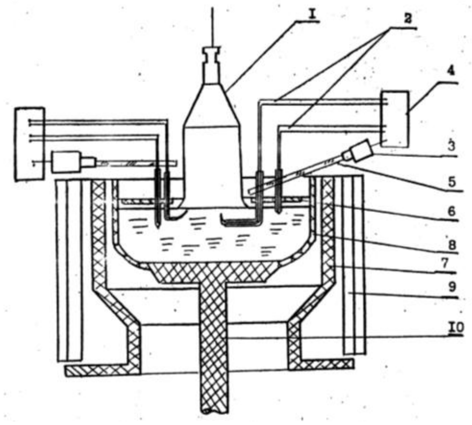

Fig. 1 – Scheme of temperature observation during crystal growth. 1 – crystal, 2 – thermocouples, 3 – optical pirometer, 4 – holder for thermocouples, 5 – optical suit, 6 – graphit silk on the melt surface, 8 – crucible, 9 – graphite baffles, 10, 7 – heater-crucible, 3 – melt of GaAs, 4, 5 – thermocouple holders, 7 – quartz capillary, 8 – crystal, 9 – seed, 10 – crucible rotation chuck

A cylindrical crucible radius $\underline{R_c} = 7$ cm which was filled by the GaAs melt to a height of $H = 4$ cm and crystal radius $R_s = 3.8$ cm, was considered. The temperature, of the crucible wall was uniform and, in the general case, could be time-dependent. No-slip conditions for radial velocity and free conditions for angular velocity were assumed at the liquid encapsulated Czochralski (LEC) interface. The crucible and crystal were rotated at constant angular velocities $w_c = 16$ r.p.m. and $W = 6$ r.p.m., respectively.

3.2 Crystal Growth Process Employing Dynamic Speed Control of the Crystal and Crucible Rotation

The growth conditions were: temperature gradient near the crystal surface – (25-30) °C·cm^{-1} and in the melt – (16-18) °C·cm^{-1}. The rate of crystal rotation was controlled linearly from 5 to 20 r.p.m. and the crucible rotation rate was varied from 3 to 15 r.p.m. over 60 min. The speed of crystal growth was 8mm / min. The process of crystal growth under these parameters was alternated over a 60 min period. After growth, the boules were cut up into wafers of 1 mm thickness and prepared for examination in an optical microscope using a standard chemical / mechanical polishing technique.

The results of crystal homogeneity (measured via the defect EL2 distribution on the wafer section) with variations in crystal and crucible rotation rates are presented in Fig. 2. The number of waters which did not show a marked dependence on the ratio of crystal / crucible rotation speed was minimal. This implies that control of the crystal and crucible rotation rate could be used for optimizing the crystal growth process in terms of homogeneity. Higher radial crystal homogeneity could be correlated with the crystal and crucible speed rotation, with best results achieved when the shape of the crystallization front was almost fiat [1, 8-10].

Fig. 2 – Result of etching a longitudinal section of GaAs crystal. Lower section without heal changing, upper section with heating wave (x 50)

The temperature of the melt was monitored at a position 1.5 mm from the crystal-melt boundary over the entire crystal growth period. Control of the temperature changes near to the crystallization front during the experiments using crystal and crucible rotation provided an estimate of the temperature fluctuations under which the various parts of the crystal were formed. The experimental resultsindicate an unclear minimum dependence. The character of the dependence may be related to flow interactions, causing natural and forced convection in the melt. This may lead to the formation of a stable flow under the region of the crystal at a certain point in time.

4. CONCLUSIONS

Unsteady low energetic thermal control actions (thermal waves, induced by periodic changes of the heater temperature) are able to eliminate temperature fluctuations near the crystal / melt interface and may be a potential tool for the growth of striation-free GaAs crystals.

It was established that the dependence of optical uniformity on amplitude and frequency of vibration has a critical characteristic which can be used in the optimization of technological parameters of crystals with high uniformity.

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