Investigation of Temperature Field and Melt Flows in Large-Diameter CZ Silicon Modelling Experiments with Impact of Magnetic Fields

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Abstract

Physical simulation of Czochralski silicon crystal growth (CZ) is presented. The hydrodynamics and heat transfer in CZ for Re and Gr criteria being equal to the values of these criteria in the silicon crystal growth process and so implementing the real boundary conditions for the heat flows both on the walls and bottom of the crucible and on the melt free surface were studied. Maximum approximation to large diameter silicon single crystal growth is the main achievement of the presented method. The results of the hydrodynamics and heat transfer simulation for a 300-mm silicon crystal pulled from a 700-mm crucible are discussed.

Introduction

Semiconductors are an integral part of the modern electronic industry. The mostly widespread nowadays is single-crystalline silicon (Si), which is used as a basic raw material for the production of integrated circuits. The main bulk of silicon for electronic industry (more than 90%) is produced by means of ingot pulling from the melt - the Czochralski (CZ) method. The main problem of the technology is unstable melt convection that leads to the fluctuations of the growth rate and, subsequently, to the inhomogeneity of dopant concentration along the axis and diameter of the growing crystal.

The fluid flow is turbulent in large CZ growth systems, such as 100 kg in a 22-in. (or larger) crucible. As crystal growth proceeds, the ratio of the free melt surface to the melt in contact with the crucible wall increases, and the axial oxygen level tends to decrease unless corrective steps are taken. The radial oxygen gradient, or uniformity, depends on the oxygen distribution at the crystal-to-melt interface boundary layer. That distribution in turn, is influenced by the fluid flow in the melt under the crystallization interface. Crystal rotation, which drives a centripetal flow under the crystal interface helps to achieve radial uniformity of oxygen and the dopant.

To improve the technological parameters of the growth process in the context of continuous transition to the larger diameters of the crystal it is especially necessary to carry out scientific investigations of the melt behaviour that dramatically affects the quality of the CZ-grown silicon crystals.

In the recent years there are many reports dealing with a numerical simulation of the CZ melt flow and temperature field. These works are commonly based on the numerous turbulence models (since the turbulent nature of the melt flow in the crucible is now widely approved) and has to be verified in that or other way by experimental investigations.

Experimental investigations of the flows in the crucible are restricted by opacity of the environment. That is why the model experiments simulating the growth conditions and allowing flow visualization and/or invasive measuring methods are carried out. Earlier experiments [1] were carried out with the crucible rotation. Free convection was studied later.
by several research teams [2-7]. Most of the experiments were carried out with transparent liquids at room temperature. Schematic measurements of velocity had been carried out in some cases only [6, 7]. In some investigations, direct measurements in the silicon melt were carried out [8], however, the environment aggression and the complexity of such experiments complicate or, in general, make impossible the measurements of velocity in the melt. For this reason, it is necessary to carry out more experiments under conditions, and with boundary conditions, more approximated to the growth of silicon single crystals of larger diameter.

The main attention of the current work has been paid to the development of a proper (adequate) physical model of hydrodynamics and heat transfer processes in a large-scale Czochralski single silicon growing apparatus.

1. Experimental setup

The proposed physical model advises to use a low-temperature eutectic alloy InGaSn (melting point - 10.35 °C and the Prandtl number value is close to that that in liquid silicon) as a modelling liquid. The model provides the boundary conditions on the surface of crystal, melt and crucible sidewall. The water-cooled crystal model allows to simulate and control the heat losses through the crystal. Radiative heat losses from the melt surface in real processes are considered through additional cooling of the free surface in the model. The values of all parameters in the model within a separate experiment are kept constant, so a quasi-stationary in time situation is simulated. The process adequacy is fulfilled by satisfying the process geometrical proportions (the ratio of crystal and crucible diameters, the crucible shape, melt level) as well as by the similarity of physical processes in the melt due to equality of the main similarity criteria both in the model and in the real process for a silicon 300 mm diameter single crystal grown from a 700-mm crucible.

The main similarity criteria are Gr (similarity of free convection phenomena), Re (similarity of velocity field driven by forced convection flows, - crystal/crucible rotation), Ha (similarity of MHD effects under the impact of steady magnetic fields), K and ε (similarity of the electromagnetic force distribution in the melt, see [9]). For more details on the similarity criteria for the current experimental model see [10]. Fig.1 schematically illustrates the experimental stand. A standard silica crucible (1) of 500 mm in diameter is filled with InGaSn eutectic (2) as high as 140 mm. A resistive heater (3) arranged on the crucible outer side surface heats the melt. A water-cooled crystal model, 160 mm in diameter, (4) is a hollow cylinder with 1-mm thick walls made of stainless steel.

A 3% HCl layer on the melt free surface prevented its oxidation. To cool the melt surface (to simulate the radiation heat flux), an additional heat exchanger - a water-cooled spiral (5) was placed in the HCl solution. Temperature of the water cooling the crystal and the spiral was kept 10°C; and the flow rate was adjusted so that the ratio of the heat losses through the crystal to those through the spiral was ~1/4. Heater power was about 3 kW.

The setup was supplied with a combined inductor (bore diameter - 700 mm) generating magnetic fields. Two sets of coils (6) were used to generate a steady axial magnetic field (uniform, CUSP-field). Another three coils (7) ensured a longitudinal alternating field (pulsating or three-phase travelling). The rates of crystal and crucible rotation ranged within 0 - 25 rpm for the crystal and 0 -7 rpm for the crucible.

The temperature in the melt was measured in a quasi-stationary regime with the system parameters being constant. A multi-channel probe consisting of 32 Cu-NiCu
thermocouples (9) and arranged in the vertical plane was used to measure the temperature field. The thermocouples were arranged in rows at different depths enveloping in such a way a half-section of the melt.

A 12-bit PC controller (Keithley DAS-1801HC) was used for probe sampling. It allowed sampling from all 32 channels for 1 second and registered both the mean temperature distribution in the melt and the temperature field fluctuations. After measuring, in about 20 minutes for each regime, the data from all 32 sampling points were interpolated throughout the domain of measurements. So a picture illustrating the temperature field variations in time related to the non-stationary convective melt flows was obtained.

2. Results of the experiments

2.1. General case – no magnetic fields

The mean temperature distribution in the melt for the non-rotating crystal and crucible can be found in Fig.2. In this case, the motion of liquid represents a single vortex. The value of temperature drop between the hottest and coldest zones in the melt is about 10 K. The shape of isotherms testifies that the heated liquid at the walls ascends and, when reaches the surface, partially cools and moves deep down the crucible and cools more near the crystal. A cold jet of liquid directed to the crucible bottom forms along the central axis. The analysis of the temperature pulsations value in the melt shows their maximum in the subcrystal zone.

Fig.2 illustrates a temperature distribution when the crystal and the crucible counter-rotate. In this case, the value of temperature drop between the crucible wall and the crystal comes up to 22 K. Respectively, the radial temperature gradient in the subcrystal zone increases (about 0.85 K/cm, if compared to 0.34 K/cm in the absence of rotation). This fact and also the shape of isotherms in this regime testify the suppression of the radial convective heat transfer in the crucible.

2.2. Influence of the CUSP field

The experiments in the CUSP field were carried out when neither the crystal nor the crucible rotated, and when both of them rotated in opposite directions. The value of magnetic field induction varied within 0 - 40 mT. Fig.3a,c shows that under corresponding regimes the value of temperature drop in the melt practically does not change under the applied field (see Fig.2a,c). Fig.3b,d demonstrates a decrease of temperature pulsations in the melt subject to the CUSP-field influence. Here one can see the values of root-mean square scatter of temperature pulsations in the subcrystal zone (curve ▲) and in the rest of the melt averaged across the section (curve ▼). The obtained results show that in the absence of rotation the value of pulsations in the subcrystal zone about twice exceeds the mean value across the section even at the maximum induction value. When the crystal and the crucible rotate in opposite directions, the temperature fluctuations are less suppressed in the subcrystal zone than without rotation, and this phenomenon has been registered in the rest of the melt. This fact proves the main peculiarity of the CUSP-field effect: the melt keeps moving in the subcrystal zone with the non-stationary flows being suppressed at the crucible periphery.

2.3. Influence of the alternating travelling magnetic field

The travelling field with a 50 Hz frequency was generated within the melt volume by a three-phase inductor (7, Fig.1) for a current phase shift in the windings being p/3. A volume
Fig. 2. Temperature distribution in the melt: (a) – no crystal and crucible rotation; (b) – crystal (15 rpm) and crucible (-5 rpm) counter-rotate.

Fig. 3. Temperature distribution in the crucible under the CUSP-field: (a) – n = 0, nc = 0, (c) – n = 15 rpm, nc = -5 rpm, (b, d) – temperature pulsation value (standard deviation) versus CUSP-field induction value; (b) – n = 0, nc = 0; (d) – n = 15 rpm, nc = -5 rpm.

Fig. 5. Temperature distributions in the melt under the impact of traveling magnetic field (TMF), A=5000A: (a) no rotation, TMF “upwards”; (b) no rotation, TMF “downwards”; (c) n = 15 rpm n= -5 rpm, TMF “upwards”; (d) n = 15 rpm n= -5 rpm, TMF “downwards”;

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force directed either upwards or downwards along the vertical axis dependent of the phase shift between the edge coils is induced under the field influence at the melt periphery. So it is possible to drive ordered meridional melt flows of different directions. Fig.4 shows a field of velocities in the melt occurring under the travelling field running "upwards" measured in the melt under isothermal conditions in the absence of crystal and crucible rotation. The distribution was obtained by measuring two velocity components by a conductive anemometer-type probe with a local steady magnetic field at more than 100 reference points in the crucible section. In this case, a strong enough meridional vortex (~10 cm/s - max. value) appears in the melt with its center at the crucible periphery. The change of travelling field directions results in the change of vortex sign, with the motion pattern qualitatively being the same as in Fig.4.

The temperature field in the melt was measured when the crystal and the crucible rotated and when neither of them rotated, for two directions of the travelling field. The results of temperature distribution measurements under the field with the current load (ampere-turns) being A = 5000 A are illustrated in Fig.5. The temperature distributions related to the regime of free convection under the travelling field influence for different field directions are shown in Fig.5a,b. Additional melt stirring obviously decreases temperature drops in the liquid metal, i.e., the temperature field equalizes. If the field runs "upwards" (a), the temperature drop between the crucible wall and the melt center decreases by ~ 2 K compared to the regime without field (Fig.2a). Probably, in case of a strong enough stirring the temperature distribution should correspond to the temperature field in a solid body yet as if with a lower thermal conductivity coefficient. If the field runs "downwards" and no rotation (Fig.5b), the cold melt is pushed out of the subcrystal zone along the melt surface to the crucible periphery, decreasing in such a way the temperature drop along the crucible radius - a "hot point" in the melt shifts along the side surface toward the crucible bottom. Increase of crystal and crucible rotation rates (Fig.5c,d) increases the temperature vertical gradient in the melt. The most interesting from the viewpoint of crystal growth conditions is the case with the field running "upwards" (Fig.5c) - the pattern of temperature distribution is close to that of a solid body, the lower temperature gradient should decrease the negative influence of non-stationary heat convection, and the presence of an appropriate radial temperature gradient at the crystallization front should contribute to a stable single crystal growth.

3. Concluding remarks

The experimental investigation of temperature distributions in the model of Czochralski melt was carried out. The designed laboratory furnace ensures adequate simulation of the hydrodynamics and heat transfer in the melt, including CZ simulation in magnetic fields. Temperature distributions under the impact of axially-radial (CUSP) and alternating travelling magnetic fields were obtained. A multichannel measurement technique that allows acquiring of spatial instant distributions of temperature field in the melt has been developed.

The experimental stand allows investigations with the basic criteria of the process (the Reynolds, Grashof numbers) being equal to the actual values of these criteria during real growth of single crystals. The facility allows simulating in a crucible of a diameter up to 500
mm, considering the radiation heat loss from the melt free surface and implementing the real boundary conditions for the heat flows on the wall and bottom of the crucible.

References

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