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Effect of argon flow on the quality of Czochralski silicon crystal

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Abstract The effects of argon gas flow on the axial temperature gradient near the interface, the oxygen concentration, and the radial oxygen uniformity was investigated for 8-inch CZ silicon growth. As argon flow rate was increased, the temperature gradient was increased in the crystal near the crystal/melt interface and the oxygen content in the crystal was decreased. But the radial oxygen uniformity was deteriorated. It was found that argon flow is one of the important growing parameters to affect the quality of crystals such as oxygen content and uniformity.

쵸크랄스키 실리콘 단결정의 특성에 미치는 아르곤 유동의 영향

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요 약 8인치 쵸크랄스키 실리콘 단결정 성장에 있어서, 계면에서의 온도기울기, 산소 농도 및 반경방향의 산소농도 분포에 미치는 아르곤 가스 유동의 영향을 조사하였다. 아르곤의 유입량을 증가시킴에 따라 계면 근처 결정내 온도기울기가 증가하였으며, 결정내 산소농도는 감소하였다. 한편, 반경방향의 산소농도 균일성은 악화됨이 관찰되었다. 실험 결과를 종합하여 볼 때, 아르곤 유동이 산소 농도 및 균일 분포성 등의 결정특성에 중요한 영향을 미치는 성장 공정변수임을 확인할 수 있었다.

1. Introduction

The quality of silicon crystals required by the semiconductor industry becomes high because it is crucial to high-density integration for advanced devices. Therefore, more tight oxygen content and defect controls are also demanded in growing crystals. Although the growing related crystal quality is mainly determined by hot zone design, the changes of hot zones is often difficult due to great expenses. It would be desirable if the quality of crystals can be controlled by growth parameters without the change of hot zone parts. Argon flow is thought to be one of the important growing parameters that affect the quality of crystals, however its effect has not been well understood. The Czochralski growing chamber is filled with an inert gas (i.e., argon) because the growing environment has to be free of reactive gases, and this gas is swept out of the chamber by the vacuum system during the process.

The axial temperature gradient (G) in the crystal near the crystal/melt interface has been reported to be an important factor determining the type of grown-in microdefects in Czochralski silicon crystals [1-3]. Because this axial temperature gradient is mostly predetermined by hot zone structure, the appropriate hot zone design is prerequisite for obtaining the crystal quality needed. But, once the hot zone structure is set, only a few growing parameters can be changed. The argon flow is believed to increase the axial temperature gradient in the crystal, but the quantitative results have not been reported.

Oxygen is one of the most important impurities to be tightly controlled in a silicon crystal grown by Czochralski method because device yield is significantly influenced by oxygen precipitates formed during the device process. It has been reported that the argon gas flow and the chamber pressure can influence the oxygen content in the crystal significantly, however, their effects are dependent on the hot zone structure [4]. For example, the lower chamber pressure was found to increase the oxygen concentration in the

open-type hot zone (without a heat shield) but to decrease it in the gas-controlled hot zone (with a heat shield). More clarification is in need of further investigation.

As mentioned, argon flow is believed to be one of the important growth parameters that control the quality of silicon crystals. Although the influence of argon flow on the crystal quality may be smaller than those of other growing parameters such as pull rate, it can be the very useful growth parameter that helps with obtaining the required quality of crystals. In this study the effects of argon flow rate and chamber pressure on the temperature gradient near the crystal/melt interface and the oxygen concentration distribution were investigated with different flow rates and pressure.

2. Experimental procedures

A commercial grower (Mitsubishi N3) was used for growing B-doped <100> oriented Si crystals. 110 kg high purity polycrystal silicon was charged in a 24-inch quartz crucible for each run and a heat shield was applied. The diameter of ingot was kept as about 8 inch (200 mm) during the growth by ADC (automatic diameter control) system. The growing chamber was in vacuum and argon gas was flown down on the melt surface followed by being swept out in order to expel SiO and prohibit the oxidation inside of the grower.

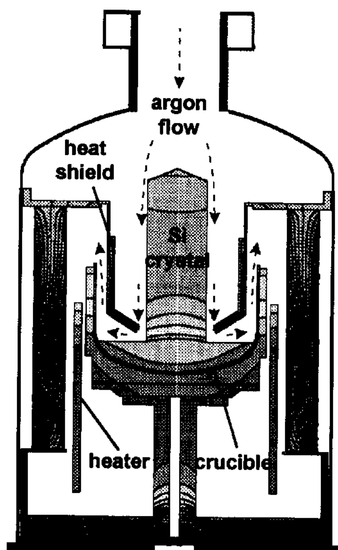

Fig. 1. Schematic diagram of the argon gas flow path near the growing crystal.

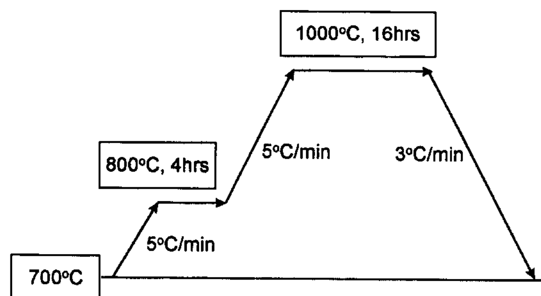

Fig. 2. Heat treatment process for the specimens observed by X-ray topography method (in N_2 atmosphere).

The argon flow path near the growth interface is schematically shown in Fig. 1. The argon flow rate and chamber pressure were varied on purpose.

Slugs were taken from CZ grown ingots and their oxygen concentrations were measured by a Fourier transform infrared spectrometer (QS-300, Bio-Rad). Oxygen radial gradient (ORG), which is used as a standard to quantify the radial oxygen concentration homogeneity, was determined as follows:

ORG (%)
= [(average concentration at 4 positions of the edge)-(concentration at the center)]
×100 %/(concentration at the center) (1)

Some ingots were sectioned vertically and prepared for observing the axial OiSF (Oxidation induced Stacking Fault) distributions by X-ray topography method. Two step heat treatment was carried out in the atmosphere of N_2, as shown in Fig. 2.

3. Results and discussion

3.1. *Temperature Gradient in the Crystal Near the Interface*

It has been reported that the type and distribution of defects formed during the growth are closely related to V/G (pull rate/axial temperature gradient in the crystal near the interface) [1, 3, 5]. For instance, OiSF ring shrinks to zero as V/G decreases to the critical value. In this research, therefore, G values were evaluated indirectly by observing the OiSF ring disappearance with the reduced pull rate (V). Figure 3 shows one typical result that OiSF ring shrinks to zero as the pull rate decreases.

The effect of argon gas flow on G has not been well

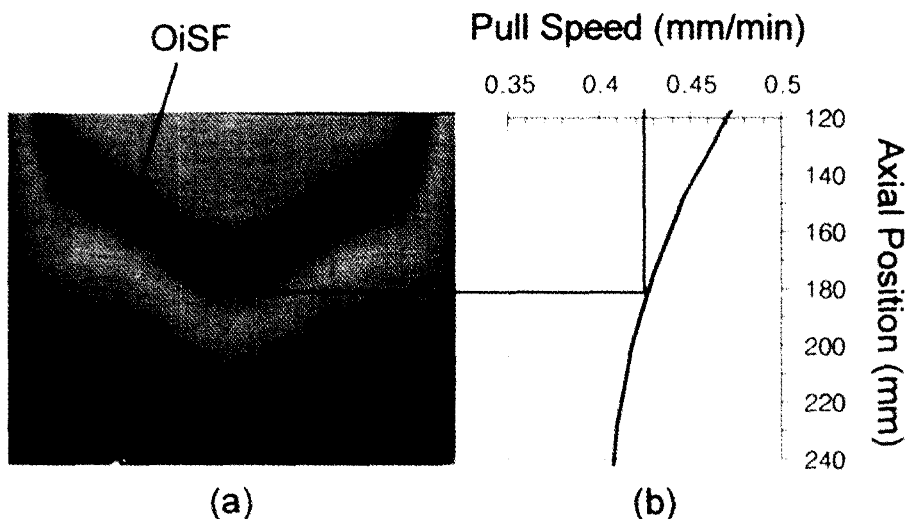

Fig. 3. Relationship between pull rate reduction and OiSF disappearance in 8-inch Si crystal pulling (a) X-ray topograph of the vertically sliced specimen (b) pull speed vs. axial position.

Table 1
Effects of argon flow rates and chamber pressure on the pull rate which OiSF ring shrinks to zero and calculated G

	Flow rate (l/min)	Pressure (Torr)	Critical pull rate (mm/min)	Calculated G [5] (K/mm)
Case I	30	30	0.40~0.41	3.08~3.15
Case II	45	15	0.41	3.15
Case III	60	15~20	0.42~0.43	3.23~3.31

known, but it is expected to increase G by increasing cooling rate of the crystal surface near the interface. The comparison of three argon flow conditions is shown in Table 1. As indicated, G was increased by an increase in argon flow rate (with a decrease in pressure). Although the maximum increase is about only 0.02 mm/min in pull rate (0.16 K/mm in G), this amount can be important in actual growing industry. It is confirmed that argon flow can be used as a useful growing parameter for achieving the required quality of crystals. The used critical V/G for OiSF disappearance is 0.13 $mm^2min^{-1}K^{-1}$ [5].

3.2. *Oxygen Incorporation into the Crystal*

3.2.1. Oxygen Concentration

The oxygen transport phenomenon into the crystal is complicated but is closely related to the melt convection near the crystal/melt interface [6-8]. If the dissolution rate of oxygen from the crucible is not changed, the oxygen concentration in the melt near the interface is known to be mainly determined by the balance of the outward flow from the crystal center, and the inflow from the melt surface. Figure 4 shows the influence of argon flow rate and chamber pressure on the axial oxygen concentration profile. Since the oxygen content can be greatly affected by the melt temperature, power input for body growing was kept as about 67 kW. Oxygen content in the ingot was initially high and became stabilized to show only small variations. When three different cases of argon flow rate/chamber pressure are compared, flow rate increase tends to decrease the oxygen concentration.

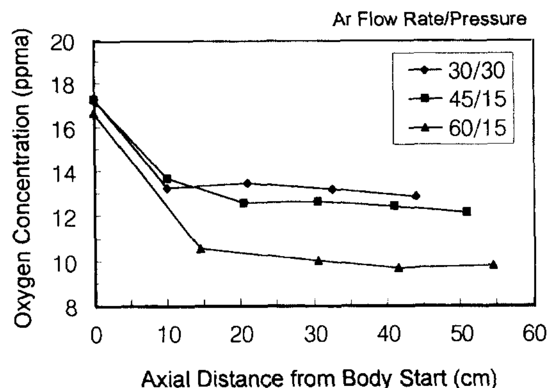

Fig. 4. Oxygen concentration profile in the crystal measured along the growth direction on center line.

In the hot zone with a heat shield (Fig. 1) the argon flow rate is accelerated at the gap between the crystal

surface and the inside surface of heat shield, and the collision is mainly occurred around the meniscus area. As a result, the cooling of this part can be very high when the argon gas flow is intense (high Ar flow rate and low chamber pressure). And the inflow action from the surface melt (below the heat shield) into the melt below the crystal can be promoted due to the increased temperature difference between them. The oxygen content at the melt surface is always lower than that in the bulk melt because of the SiO evaporation at the surface. When argon flow rate is increased, the evaporation of SiO can be partly reduced due to the surface eddy by the argon flow. However, the oxygen concentration incorporated into the crystal will be eventually decreased because the increased inflow action enables more surface melt to be supplied to the growth interface.

3.2.2. Radial Oxygen Uniformity

It was observed that the oxygen concentration in crystal was decreased as the argon flow was intensified, but the radial homogeneity of oxygen concentration was found to be deteriorated, as shown in Fig. 5. This is assumed also due to the promoted inflow of the surface melt into the growth interface. For a low argon flow rate, the radial oxygen concentration was relatively uniform. As the flow rate was increased, the overall oxygen level was decreased but the relative content reduction was larger at the periphery area in which oxygen-depleted surface melt can be easily introduced. As a result, the radial oxygen uniformity was worsened.

The axial oxygen content and ORG are summarized together in Fig. 6. The oxygen concentration measurement was made on the center of wafer. For both oxygen content and ORG comparison, specimens were taken from the position that is about 20 cm from the body start of crystals. Recalling Machida et al.s research [4], the obtained result is not consistent with it. The hot zone configuration used in this study can be classified as gas controlled type from Machida *et al.* And an increase in argon flow rate and a decrease in pressure were expected to increase the oxygen content. This discrepancy is believed due to different flow patterns in the melt, which were originated by different hot zone configurations and charge sizes. It is confirmed that the oxygen incorporation into the crystal is greatly influenced by the change of flow pattern near the interface.

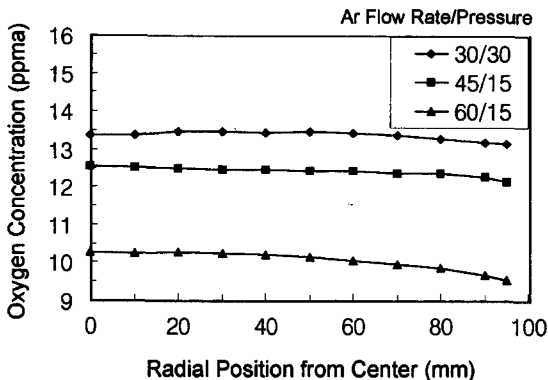

Fig. 5. Radial oxygen concentration distributions in relation to argon flow rate/pressure (at 20 cm axial distance from body start).

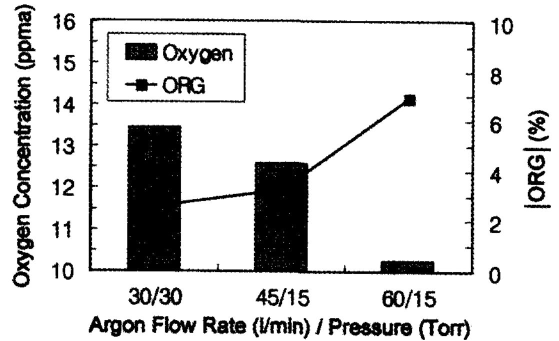

Fig. 6. Effect of argon flow on the oxygen content and the radial uniformity at 20 cm axial distance from body start.

4. Summary and conclusions

The effects of argon flow on G, oxygen concentration, and ORG were investigated with the constant hot zone configuration, crystal/crucible rotation, and power input. The obtained results are summarized as follows:

1) An increase in argon flow rate was found to increase the axial temperature gradient (G) in the crystal near the interface probably due to the increased cooling rate around the area.

2) The overall oxygen concentration in the crystal was reduced by increasing the flow rate, but ORG was increased. And these are thought to be due to that the inflow of the surface melt into the interface was promoted by intensified argon flow.

3) In conclusion, argon flow is believed to be an important growing parameter to control the quality of crystals such as oxygen content and uniformity.

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