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Effect of lamp power and mirror position on interface shape of silicon molten zone during infrared convergent heating

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ABSTRACT

The effects of lamp power and mirror position on the feed-melt and crystal-melt interfaces during the growth of a silicon single crystal were examined using the mirror-shifting-type infrared convergent-heating floating-zone (IR-FZ) method. The lamp power used to form the molten zone was carefully changed. The positions of the mirrors along a horizontal plane were also systematically shifted from positions close to the molten zone to more distant positions as compared with the conventional mirror position. The solid–liquid (S–L) interfaces at both the feed-melt and the crystal-melt sides were examined carefully at the different mirror positions under the different lamp powers using a feed rod of 15 mm in diameter, and it was found that the convexities (*h*/*r*) of the S–L interface shapes at both sides decreased with increasing lamp power. It was also found that the interface shapes were independent of the mirror position. A lower lamp power was enough to maintain the stable molten zone during the crystal growth at the closer mirror position. Therefore, effective heating was realized at the closer mirror position.

Introduction

Silicon single crystals are the most important manufactured crystals at present, and they are widely used as substrates for various device applications such as large-scale integration (LSI), solar cells, etc.[1, 2] The ability to grow bulk single crystals reduces the manufacturing cost. There are two major manufacturing methods for the growth of bulk silicon single crystals. The Czochralski (CZ) method is more widely used because dislocation-free, larger-diameter crystals can be grown[3] at lower cost. However, control of the segregation of dopants and suppression of contamination during the growth process by this method are very difficult.[1] It is also well-known that contamination of impurities such as oxygen and carbon from the crucible and the heater often occur in CZ silicon growth.

The second method is the radio frequency-heating floating-zone method (rf-FZ). Because this is a crucible-free zone-melting method,[4] the contamination level of the grown crystal is lower than that of the crystal grown by the CZ method, and the segregation of the dopant along the growth direction can be controlled easily.[1,5] However, the inhomogeneity of the dopant distribution along the radial direction is more prominent, and it needs to be homogenized in order to improve the reliability of device performance.[6] Also, although a high-purity and dislocation-free single crystal can be easily grown, the manufacturing cost is much higher than that of the CZ method.[1,7] With this method, the curvature of the interface between the melt zone and the grown crystal and the radial temperature gradient are important factors for increasing the diameter of the grown crystal and also for controlling the defects of the grown crystal.[1,8]

The infrared convergent-heating floating-zone (IR-FZ) method is one of the most powerful techniques for the growth of single crystals, as noted by Koohpayeha et al.[9] High-purity and dislocation-free single crystals can be grown by this method[10] because it too is a crucible-free zone-melting method. Therefore, segregation of the dopant can be controlled by this method. However, in spite of these advantages, the IR-FZ method is not a popular method like the CZ and the rf-FZ methods. The growth of large-diameter crystals is very difficult because unexpected contact between the feed and the grown crystal often occurs, disturbing the stability of the molten zone as the diameter of the grown crystal increases.[11] The maximum reported diameter of silicon grown by the IR-FZ method was approximately 15 mm.[10]

Maintaining a stable molten zone is very important for successful crystal growth by the IR-FZ method, and the stability of the molten zone and the quality of the grown crystal are strongly related to the shape of the S–L interfaces.[12, 13, 14]

Higuchi and Kodaira[11] reported that the interface shape could be controlled by varying the rotation rate of the seed crystal during the IR-FZ growth of rutile. Some authors have reported that the heat reservoir has an effect on the interface shape during growth by the IR-FZ method.[8, 15, 16] In other studies, the convexity of the interface was decreased by adjusting the tilting angles (*θ*) of the ellipsoidal mirrors.[12, 17] It has also been reported that the quality and the grown diameter of rutile single crystal were improved with decreasing crystal side convexity.[13, 14] These results indicate that the diameter of the silicon single crystal by the IR-FZ method might also be improved by controlling the interface shape.

In the conventional IR-FZ method, the layout of the heating lamp and the ellipsoidal mirrors are in the same horizontal plane, and thus the focus position of each mirror is also usually fixed at the rotation center of the feed and the grown crystal. However, it is not always necessary to maintain the focus position at the rotation center in order to form a molten zone. In this study, therefore, the mirror position was shifted systematically from a position close to the molten zone to a more distant position along the same horizontal plane in order to examine the effects of mirror position on the solid–liquid (S–L) interfaces during the growth of silicon using the IR-FZ method. We also examined the effect of lamp power on the silicon growth.

Concept of mirror position

An image furnace (model FZ-T-10000-H-TY-1, Crystal Systems Corporation) with four ellipsoidal mirrors was modified to realize the change in mirror position. A schematic illustration of the newly modified mirror-shifting-type image furnace is shown in Fig. 1. Four 1.5 kW halogen lamps used as radiation sources were set up in front of the respective ellipsoidal mirrors, which could be shifted horizontally by using screw gauges. As the mirror position was shifted, the focus position of the convergent heating was also shifted. In the conventional IR-FZ method, on the other hand, the focus position of each ellipsoidal mirror is not movable. In this experiment, the conventional mirror position and the corresponding focus position are defined as the zero (0) mm position. The more distant position is defined as a positive (+) shift, whereas the closer position is defined as a negative (−) shift.

Experimental

A silicon polycrystalline rod (>11N) of 15 mm in diameter was used as the feed rod. The seed rods were also polycrystalline silicon material of 20 mm in diameter. A small amount of iron (~0.15 g) was put on the top of the seed crystal in order to characterize the molten zone shape. After inserting the quartz tube, the sample area was evacuated up to 4×10^{-3} Pa. The feed rod and the seed rod with iron powder were heated in high-purity argon gas (>99.9995%) at a flow rate of 0.5 L/min. The iron-doped molten zone was stirred for approximately 30 min to mix the iron with the silicon. For all growth experiments, the growth rate and the feeding rate were 5 mm/h and 10 mm/h, respectively. The upper shaft and lower shaft rotation speeds were 4 rpm and 50 rpm, respectively. When the growth length was approximately 20 mm, the molten zone was quenched by quickly stopping the rotation of the shafts and turning off the lamps. The quenched samples were cut through the center along the growth direction and were polished to a mirror-like surface. Most of the iron remained in the quenched molten zone without dissolving into the grown crystal since the segregation coefficient of iron into silicon ($\sim 8 \times 10^{-6}$) is much smaller than unity. The molten zone shape was characterized by the iron distribution obtained by an electron probe microanalyzer (EPMA) (model JXA-8200, JEOL Ltd.).

In the experiments, the mirror position was shifted from −2 mm, which was the position closest to the melt zone while avoiding contact, to +4 mm in 2-mm steps. The applied lamp powers were varied from 4.2 to 5.0 kW.

Results and Discussion

Figures 2(a)–(c) show the iron distribution mapping images of the quenched molten zone for different lamp powers at the zero (0) mm position. The shape of the S–L interface at both the feed side and the grown crystal side was found to be significantly affected by the lamp power. At the lower lamp power, the stability of the molten zone was disturbed by the contact of the feed with the grown crystal. At the higher lamp power, on the other hand, the melt drop easily occurred. The stable molten zone was realized in the range of lamp power between 4.4 – 5.0 kW. Similar behavior has also been reported in the growth of $La_{2-x}Sr_xCuO_4$ (LSCO) single crystals.[18]

To characterize the effect of lamp power on the molten zone shape quantitatively, several parameters were evaluated, as shown in the schematic in Fig. 3(a). The convexities at both the feed side (h_F/r_F) and the crystal side (h_C/r_C) decreased as the lamp power increased, from 0.69 to 0.45 and from 0.58 to 0.43, respectively, as shown in Figs. 3(b) and (d), respectively. In the IR-FZ growth of silicon crystal, no experimental data on convexity has been reported so far. The differences of convexity in the IR-FZ growth of silicon crystal as a function of lamp power are significant as compared with previously reported differences as a function of rotation rates[11] and mirror-tilting angles[17] in the IR-FZ growth of rutile. The gap between the feed and the grown crystal and the molten zone length (L) both increased with the increase of the lamp power, as shown in Fig. 3(c), although the change of L was much smaller than that of the gap. The increase of L did not disturb the stability of the silicon melt because this length is smaller than the theoretical limit of the molten zone length of 16 mm given by Heywang.[19] During the crystal growth, h_F, h_C, and the gap, which are significantly affected by the lamp power, are not observable in real time. To characterize the effect of mirror position on these parameters, the effect of lamp power must be excluded. The minimum gap situation can be realized using a lamp power slightly higher than that at which contact of the feed with the grown crystal occurs. The minimum gap situation, therefore, is considered to be ideal for investigating the effect of mirror position on the interface shape while excluding the lamp power effects. In the larger gap situation, it was very difficult to obtain reliable data due to the lamp power effects with good reproducibility.

Figures 4(a)–(d) show the iron distribution mapping images of the quenched sample at the different mirror positions using a feed rod of 15 mm in diameter, and the effect of mirror position on several parameters are shown in Fig. 5. As shown in Fig. 5(a), all the observed gap values were less than 1.3 mm. The minimum gap situation was found to be realized through this experiment in comparison with the results shown in Fig. 3(c). The molten zone length and the feed side and crystal side convexities which are shown in Figs. 5(a) and (b), respectively, were also found to be almost independent of the mirror position. As shown in Fig. 5(c), however, the lamp power required to form a stable molten zone under minimum gap conditions was found to be dependent on the mirror position. The lower lamp power was enough to maintain the stable molten zone at the closer mirror position. This means that more effective heating is realized at the closer mirror position than at the conventional mirror position.

Conclusions

The effects of mirror position and lamp power on the S–L interface shape during silicon crystal growth by the floating zone method using a mirror-shifting-type image furnace were studied using a feed rod of 15 mm in diameter. The characteristic parameters for the molten zone shape during the crystal growth were affected more by the lamp power than by the mirror position. In the closer mirror position, however, more effective heating was realized.

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Figure captions

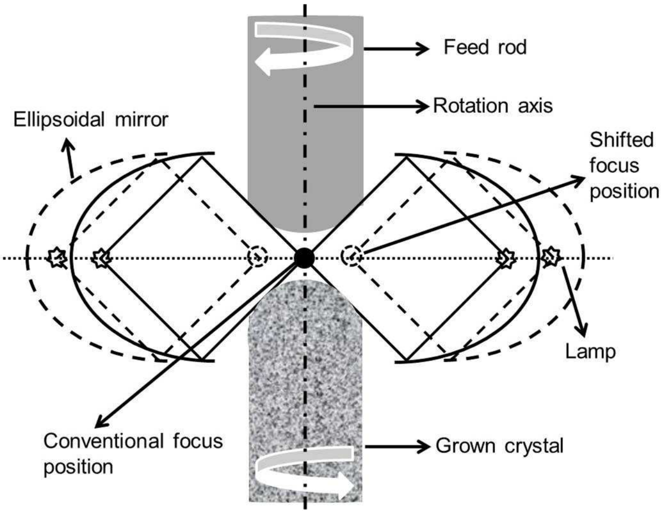
Fig. 1 Schematic illustration of mirror position. The filled circle indicates the conventional focus position of the ellipsoidal mirrors, defined as the zero (0) mm position, and the dashed lines with open circles show the more distant positions of the ellipsoidal mirrors, defined as plus (+) directions.

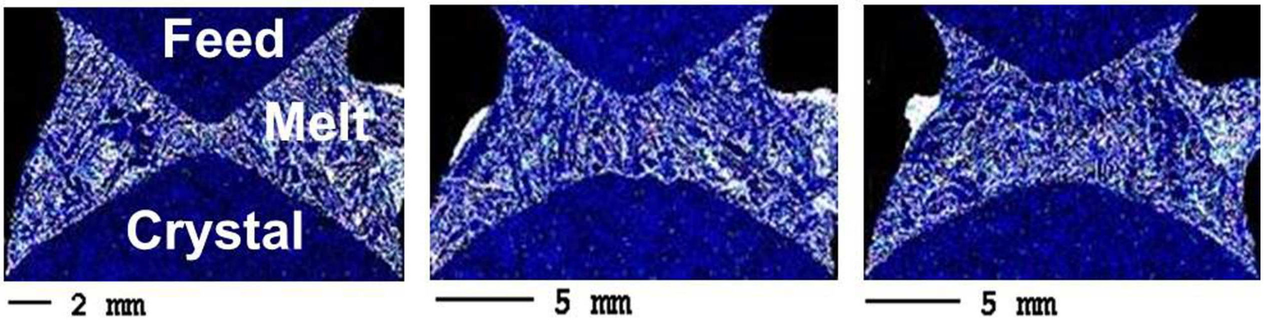
Fig. 2 EPMA mapping images for iron distribution of the quenched molten zones for (a) 4.4 kW, (b) 4.7 kW, and (c) 5.0 kW lamp power at mirror position of 0 mm.

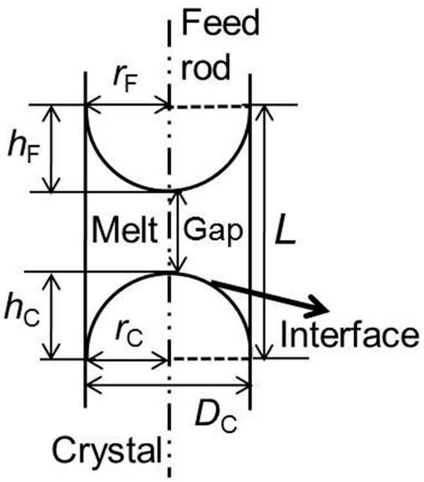
Fig. 3 Lamp power dependences of the parameters characterizing the molten zone shape: (a) schematic illustration showing the definition of convexity (h/r), gap and zone length (L), (b) feed-side convexity (h_F/r_F), (c) gap and zone length, L (mm) and (d) crystal-side convexity (h_C/r_C).

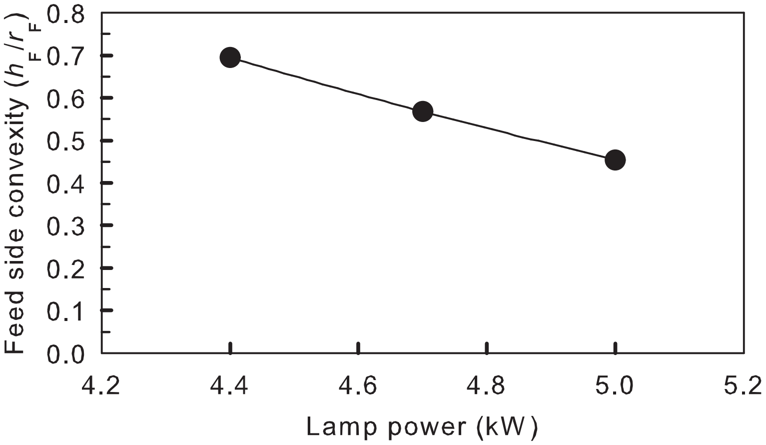
Fig. 4 Iron distribution EPMA mapping images of the quenched melt prepared at different mirror positions, (a) −2 mm, (b) 0 mm, (c) +2 mm, and (d) +4 mm.

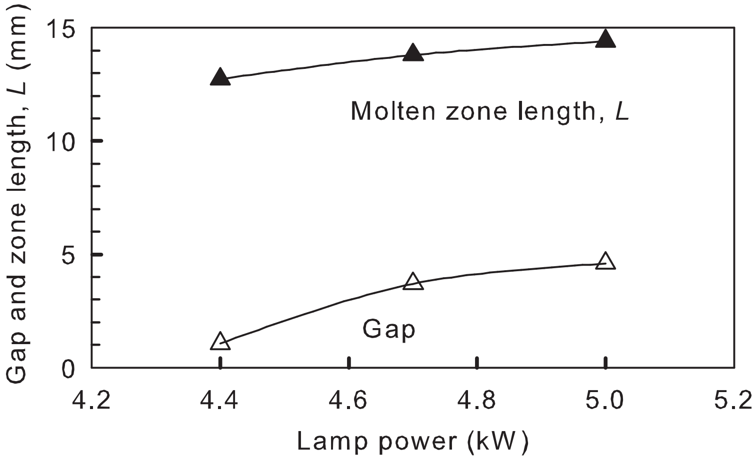
Fig. 5 Mirror position dependences of parameters characterizing the molten zone shape: (a) gap and zone length, L (mm), (b) feed-side convexity (h_F/r_F) and crystal side convexity (h_C/r_C) and (c) required lamp power for the growth. The vertical dotted lines indicate the conventional mirror position.

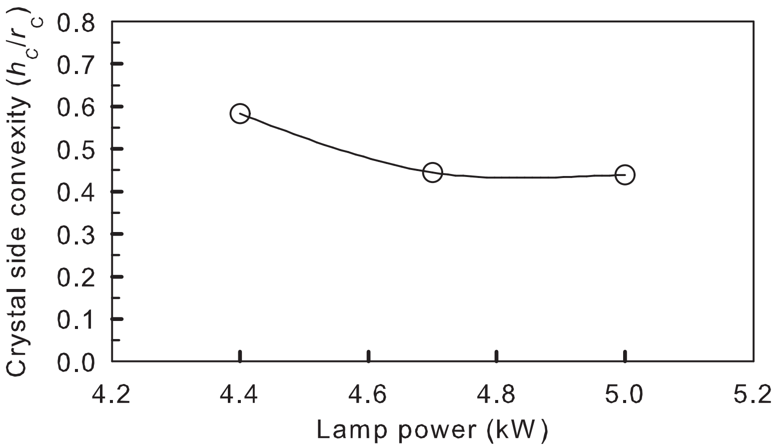

Feed rod
Rotation axis
Ellipsoidal mirror
Shifted focus position
Lamp
Conventional focus position
Grown crystal

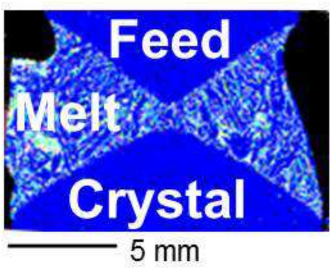

Feed
Melt
Crystal
2 mm
5 mm
5 mm

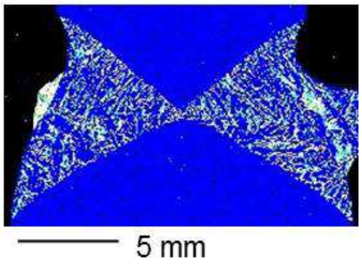

Feed
rod
r_F
h_F
Melt
Gap
L
h_C
r_C
Interface
D_C
Crystal

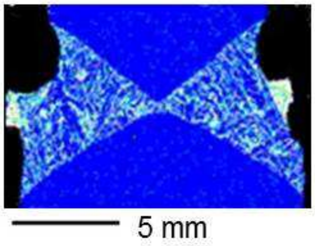

Feed side convexity (h_F/r_F)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
4.2
4.4
4.6
4.8
5.0
5.2
Lamp power (kW)

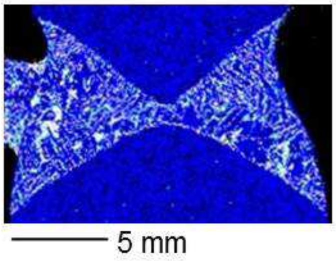

Gap and zone length, L (mm)
15
10
5
0
Molten zone length, L
Gap
4.2
4.4
4.6
4.8
5.0
5.2
Lamp power (kW)

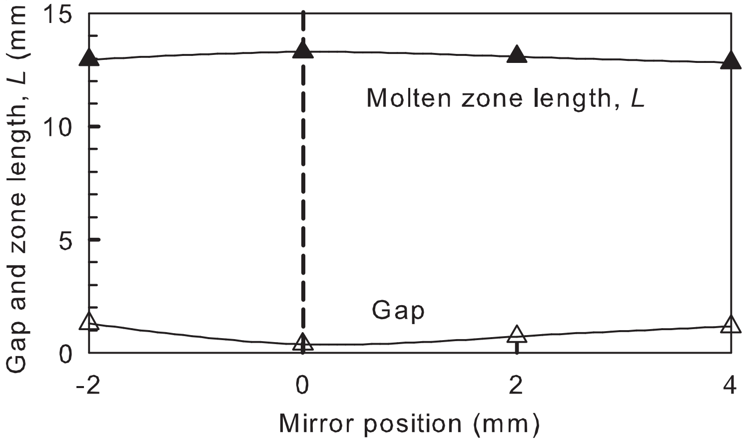

Crystal side convexity (h_c/r_c)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
4.2
4.4
4.6
4.8
5.0
5.2
Lamp power (kW)

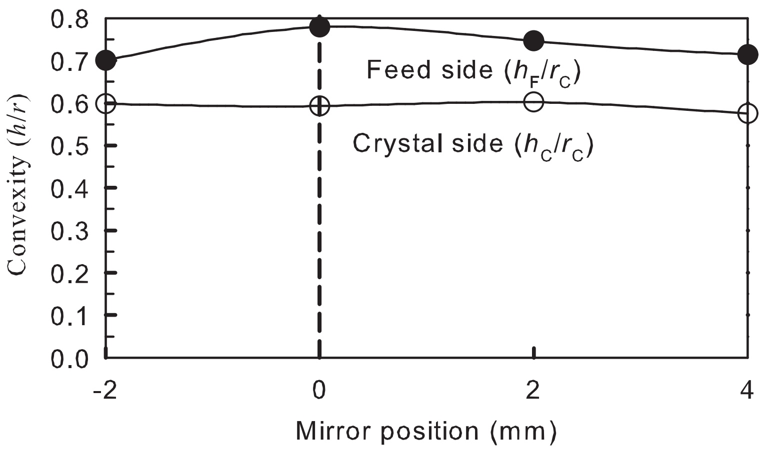

Feed
Melt
Crystal
5 mm

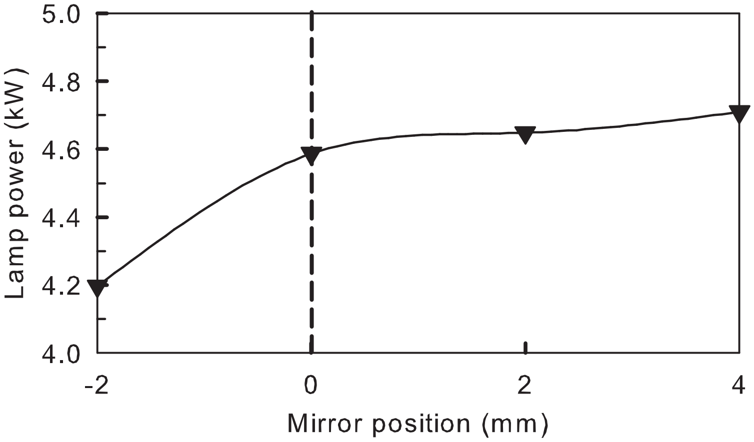

5 mm


5 mm


5 mm


Gap and zone length, L (mm)
15
10
5
0
-2
0
2
4
Mirror position (mm)
Molten zone length, L
Gap


0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
Convexity (h/r)
Feed side (h_F/r_C)
Crystal side (h_C/r_C)
-2
0
2
4
Mirror position (mm)


5.0
4.8
4.6
4.4
4.2
4.0
Lamp power (kW)
-2
0
2
4
Mirror position (mm)