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The judgment of the All-melted-moment during using electron beam melting equipment to purify silicon

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Abstract. Experiment has proved that the rate of impurity removal depends on the pressure and the temperature of the vacuum chamber during using electron beam to smelt silicon, and the amount of removed-impurity depends on time when other conditions are the same. In the actual production process, smelting time is a decisive factor of impurity removal amount while pressure and temperature of the vacuum chamber is certain due to a certain melting power. To avoiding the influence of human control and improving the quality of production, thinking of using cooling water temperature to estimate the state of material during metal smelting is considered. We try to use the change of cooling water temperature to judge that when silicon is all melted and to evaluate the effectiveness of this method.

1. Introduction

Electron beam melting, referred to as EBM, is a method by changing the kinetic energy of high-speed electron beam to heat energy to smelt metal [1, 2] in a vacuum environment (10^{-3} Pa). This method is usually used to smelt metal and alloy of high melting point, heat-resisting material and high-quality alloy for special purpose [3, 4]. It also can be used to smelting silicon to remove P and metal impurity, such as Al, Fe and so on [5].

Shi S et al. researched the mechanism of volatile impurities transfer during using electron beam to smelt silicon, and they have proved that the rate of impurity removing depending on pressure and temperature of vacuum chamber during using electron beam to smelt silicon. At this stage, the removal of impurity is sensitive to smelting time when pressure and temperature is certain. This research is mainly aimed at Fe, Al and Ca and so on and it has been proved that electron beam melting equipment has purification effect generally [6-8]. But the research did not judged when silicon is all melted definitely. It is hard to detect temperature of molten pool since the chamber of electron beam melting equipment has an environment of high temperature and high vacuum. In actual production process, we usually judge the moment of all-melted by manually watching, but this method is volatility especially disturb the removal of impurity at initial stage of smelting [9] when the impurity is volatilize quickly. In addition, electron beam melting equipment has high energy consumption. So for

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the stability of product and reducing energy consumption, it is important to judge when the silicon is all melted.

Considering cooling water can reflect the temperature of molten pool, in our experiment, we try to use the change of cooling water temperature to judge the time when silicon is all melted and evaluate the effectiveness of this method.

2. Experiment

During production, the main equipment is EBS-500 electron beam furnace and accessories are vacuum system include mechanical, diffusion and roots pump, electronic control system and cooling system. We use the silicon on the bottom of crucible after the preparation of mono-crystalline silicon using Czochralski method as raw material, which is n-type semiconductor.

Table 1. Main steps and parameters.

Step	Load material	Increasing vacuum	Preheat electron gun	Electron beam emission	Smelting	Solidification
Parameter	50-60kg each ingot	0.5Pa for Electron gun 0.2Pa for smelting	Vacuum:0.5Pa Up to 1000mA In 30min	Vacuum:0.2Pa Up to 250kW	20min	Electron beam attenuation

During the process smelting, vacuum degree of chamber will be influenced by gas from silicon. And electron gun will be shut down when the pressure of chamber is higher than security line. This phenomenon often occurs when purifying silicon whose purity is poor. We can know from the change in temperature of cooling water that the temperature of chamber reduce quickly. And on the bottom of molten pool, there can be liquid silicon curdling because of a lot of heat being taken away by cooling water. After turn on electron gun, silicon will be all welt at a time, but we can not make sure when it is. In our experiment, we collect temperature value of cooling water displayed in electronic control system each 10 second in the process of melting and smelting.

3. Result

3.1. Judge the moment of all-melted

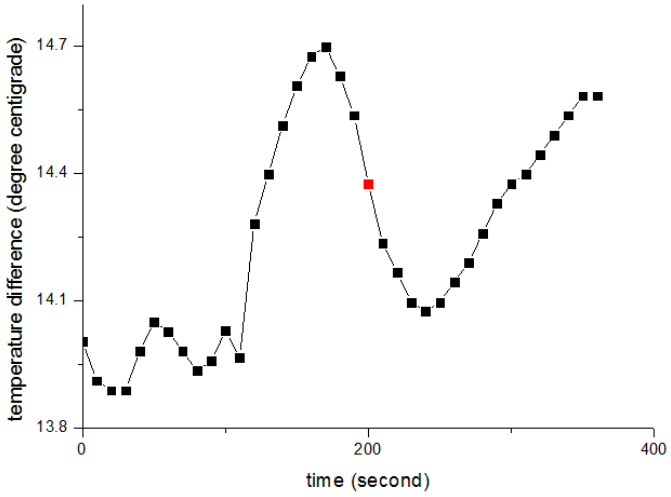


Figure 1. The change of cooling water temperature difference nearby the moment of all-melted.

We draw figure 3.1.1 through the cooling water temperature nearby the moment of all-melted, which is one of a series, the red point means all-melted observed manually. Through the similarity we can know that the change of cooling water temperature difference is fluctuant before the moment of all-melted. The reason is that solid-liquid transition occurs repeatedly at the bottom of molten pool because a lot of heat is absorbed by cooling water when the silicon is almost melted, on the contrary, the heat absorption and release of this process influence the temperature of cooling water. We can also know that the cooling water temperature difference is always increase after silicon is all melted because the temperature of melt is increase fast and continually, so if we can find the similarity of this process, the similarity can be the gist of all-melted.

Figure 3.1.2 is drawn through the temperature difference of whole smelting progress which is one of a series, the red point means all-melted moment watched manually, the green point means electron gun being shut down and yellow point means the power of electron gun reaching up to 250kW again.

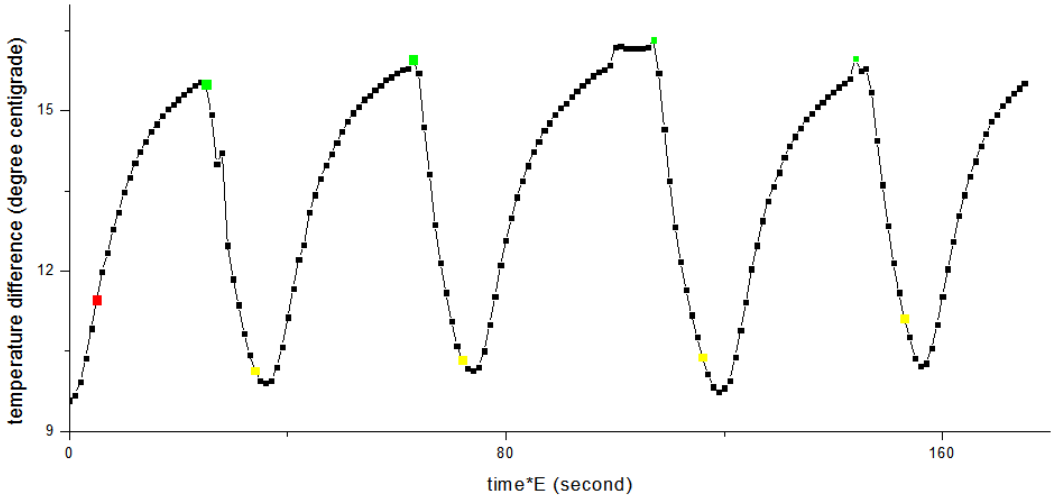
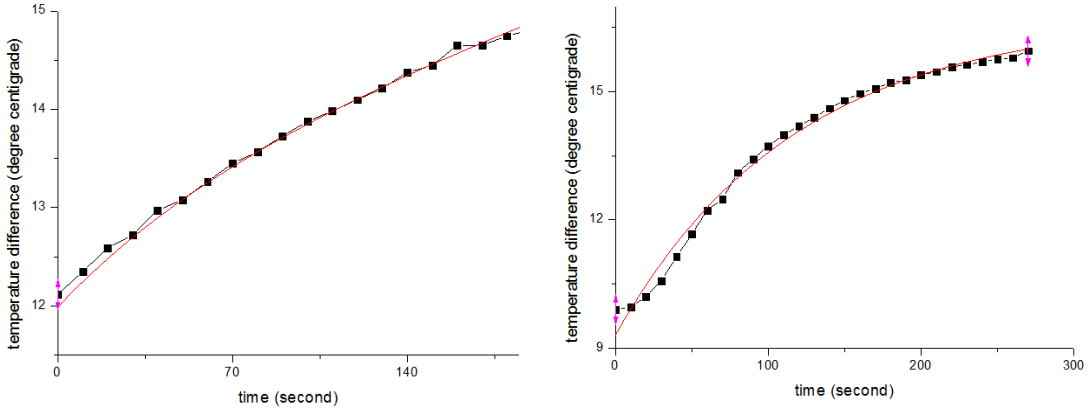


Figure 2. The change of temperature difference of whole smelting progress.

We know that the impact of electron beam make the melt temperature and the cooling water temperature difference increase. Since the melt temperature will not increase infinitely, it will remain stable after a while, which is the same for the cooling water temperature difference. This phenomenon shows in figure 3.1.2 before the third time electron gun is shut. When electron gun is shut down, the melt temperature and the cooling water temperature difference fall quickly, and the decrease lasts for a short time after the power of electron gun being up to 250kW again. So we can speculate that there is a small amount of melt solidifying again during the electron gun is shut. After turn on electron gun, the heat offered by electron beam is lower than the heat absorbed by cooling water for a short time which result in the decrease of cooling water temperature difference. As the power of electron gun increasing, the former counteract the latter and the speed of temperature decrease is getting slow. Finally the heat offered by electron beam is higher than the heat absorbed by cooling water and the solid silicon melt again. So the change of cooling water temperature difference is decreasing first and then increasing.

Now we fit the curve from the red point to the first green point and the result is showed in figure 3.1.3.



3.1.3-a 3.1.3-b

Figure 3. The fitted curve of the change in cooling water temperature difference

Through the fitted curve we can know that from one point which is after the red point, the curve is upward as exponential function, and the function expression is:

$$y = A_1 \exp(-x / B_1) + A_2 \exp(-x / B_2)$$

The value of A_1, B_1, A_2, B_2 depends on the environment of production, such as the in-flowing cooling water temperature, the melt quality and so on. We can draw the same conclusion if we fit any other curves which is similar to this process

In the situation of the electron gun being shut down, we fit the curve from the moment when the cooling water temperature difference begin to increase to the moment when the electron gun is shut again, and the result is showed in figure 3.1.3-b. We can know from the fitted curve that from one moment the curve is upward as exponential function, and the function expression is:

$$y = A_1 \exp(-x / B_1) + A_2 \exp(-x / B_2)$$

The value of A_1, B_1, A_2, B_2 depends on the environment of production. We can draw the same conclusion if we fit any other curves which is similar to this process.

In conclusion, when the cooling water temperature difference begin to rise as exponential function no matter it is in the process of melting or smelting, the moment of beginning is the all-melted-moment.

3.2. The evaluation of effectiveness

After smelting, the content of B is still the same and the p-n type of product is unstable. The reason is that the purification make donor impurity and acceptor impurity balance each other out and the small difference of smelting time will lead to the change of p-n type. Combing with figure 4.1, the logical relation curve of electrical resistivity and smelting time, we can know the degree of P removal [10, 11].

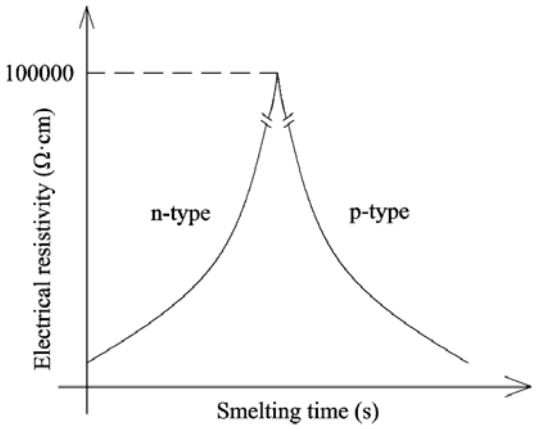
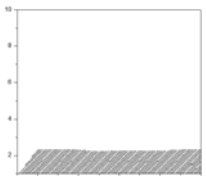
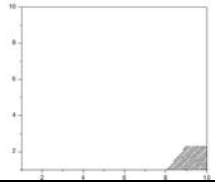
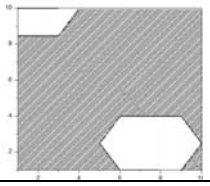


Figure 4. The logical relation curve of electrical resistivity and smelting time.

In the actual production process, the time of electron gun being shut down can be 1-6 times even more and the influence of total time errors can not be ignored. If we use the method above to calculate the smelting time and combine with the scatter-gram of product's electrical resistivity, we can evaluate the effectiveness of this method. We fit all the curves which is rising, calculate the smelting time, draw the scatter-gram and gather them to table 4.1.

Table 2. Smelting time and scatter-gram of product's electrical resistivity.

Smelting time (s)	1030	930	840
product' s electrical resistivity(the shadow means n-type)			

We can know from figure 4.1 that around the balance of donor impurity and acceptor impurity, the product will be p-type if the smelting time is longer slightly and will be n-type if the smelting time is shorter slightly. Combine figure 4.1 and table 4.1 we know that, the product is mainly n-type whose melting time is shorter relatively and is mainly p-type whose smelting time is longer relatively, which means this method of judging the all-melted-moment is efficient.

4. Conclusion

Through the experiment and analysis we draw a conclusion that during the process of using electron beam melting equipment to purify silicon, if the change of cooling water temperature difference is upward as exponential function at a moment, and the function expression is:

$$y = A_1 \exp(-x / B_1) + A_2 \exp(-x / B_2)$$

The silicon is all melted at this moment and the value of A_1, B_1, A_2, B_2 depends on the environment of production, such as the in-flowing cooling water temperature、the melt quality and so on. Using this method to calculate the smelting time and combing smelting time with figure 4.1 and scatter-gram of product's p-n type, we can deem that the method by using the change rule of cooling water temperature difference to judge the all-melted-moment is efficient.

Acknowledgments
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