To the issue of melting zone height control in the technology of electron-emitting crucible-less zone melting of silicon

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Abstract

The new approach to the evaluation of the height of the melting zone during the crucible-less zoned melting process of silicon is substantiated. This new approach takes into account the experimentally established specifics of the brightness field formation.

Key words: *electron-emitting melting, melting of silicon, melting zone.*

Introduction

Zone melting as one of substance crystalline modification methods is now widely used in research and technologies aimed at purification of crystals, metals, semiconductors, organic substances and to create materials with given distribution of additives. Zone melting can be carried out in a crucible (container) or with the help of a crucibleless method. Exclusively crucibleless zone melting should be used for materials with the melting temperature of more than 1500 K (e.g. for silicon – 1685 K) as in liquid phase they are chemically active.

The technology of zone melting consists in creation and displacement along the sample of a narrow melted zone that can be accomplished with the help of different methods [1]. Electron beam crucibleless zone melting has become widely used nowadays. It is being accomplished in a vacuum chamber and melting zone is formed under the influence of the electron beam. The most important parameters of zone melting, stipulating its quality are as follows: its height, shape of melting zone and crystallization section, steadiness of displacement velocity and temperature distribution along its surface. Nowadays zone melting has been formed into powerful branch of production of materials with prescribed properties but in spite of constant theoretical base development and technological process improvement, the problem of control of its geometric parameters remains actual.

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The use of television information – measuring systems for the first time allowed to obtain reliable experimental results on temperature distribution on the surface of the liquid phase after complete melt-through and on melting zone geometry [2]. But the ambiguous nature of the surface brightness field of the liquid phase, in particular, the impact of third-party sources radiation, reflected from the melting zone surface, which is the cause of systematic error, doesn't allow us to get the necessary precision of melting zone height measurement.

The aim of this paper is to justify the new approach to control the height of melting zone, which would take into account the experimentally established peculiarities of field brightness formation on its surface.

Features of melting zone brightness field formation

Melting zone shape in Earth conditions, is primarily determined by the influence of gravity, surface tension forces, power and stability of the electron beam, and even defects or their absence in the source of electrons and focusing field and is characterized by the zone height $h_з$ and shape factor.

Shape factor determines asymmetrical profile of the melting zone, and if the sample itself doesn't have the relevant defect, indicating the presence of defects of electron source and focusing field.

During previous research a large amount of experimental data has been accumulated, making it possible to make a number of important conclusions about the nature of the melting zone brightness field formation, in particular to clarify the concept of its height. In general, the height of the zone $h_з$ is calculated as the difference between the coordinates of the points that define the upper and lower boundaries of the zone, for example, along the axis Y.

In real conditions, melting zone boundary as shown in Fig. 1, is shaped as a complex line. Fragments of the upper and lower melting zone boundaries (for silicon) are shown in more details in Fig. 2.

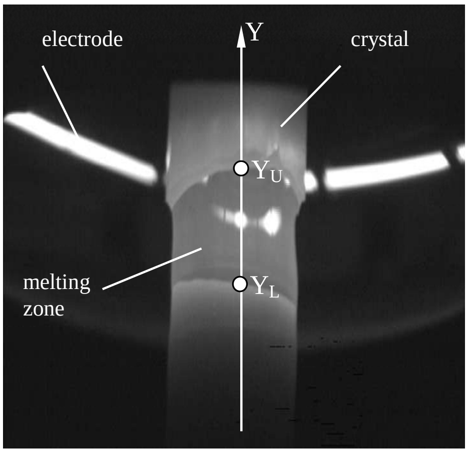
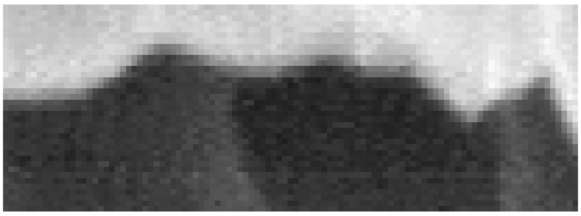
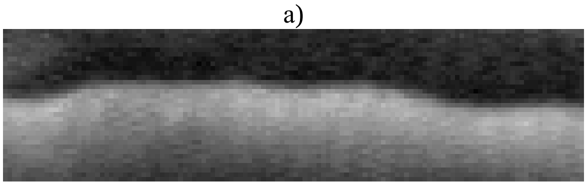


Figure 1 – Melting zone image with significant height variance across its perimeter



a)



b)

a) upper melting area, b) lower solidification area

Figure 2 – Melting and solidification area close-ups

So one can talk either about determining the height along a given coordinate or defing the average and extreme values. Zone boundaries can be defined as the coordinate of the lower Y_L or upper Y_U boundaries of melting zone for a fixed coordinate or the average of all Y coordinates along the perimeter (accordingly, zone height is $h_z=Y_L-Y_U$).

Thus, for each crystal there is some maximum height value h_{max}, under which zone can be still stable. So, h_{max} shall be referred to the critical parameters which are subject to constant monitoring. Stability of zone height is one of the main conditions under which for example, a high degree of purification can be achieved. To achieve a high degree of purification, sometimes a method of programmable height change can be used, which is that in the last cycles zone height is maintained lower compared to the first ones. In addition, all other random changes in the value of h_z lead to deterioration of crystal quality.

Modern electronic current stabilization means provide the possibility to create and maintain stable and symmetric temperature field on melting zone surface. But in the course of melting local deformation of the electrode or local emission reduction capacity may occur, resulting in additional temperature gradients, thus, in the breach of circular symmetry of temperature field. This asymmetry leads to skewness of crystallization zone, and it is known that its shape can also influence the quality of melting. Since this defect can not be corrected during melting, an electrode is subject to change. Thus, timely detection of asymmetric temperature field by measuring melting zone height is an extremely important task.

Spatial and temporal fluctuations of the electron beam also lead to local disturbance of field temperature distribution on crystal surface, which negatively affects the quality of melting. In particular, [3] states that the asymmetry of the temperature field adversely affects the distribution of impurities in the crystal crosssection, increasing their concentration in some areas and decreasing it in the other ones.

To compensate for the impact of possible temperature field asymmetry in some zone melting devices crystal is rotating around its axis. Thus the combined effect of rotating and linear movements leads to equalization of temperature field, accompanied by changes in the concentration along a spiral. Detailed analysis of the brightness field of on the surface in the liquid phase along the axis Y (Fig. 1) revealed the presence of a legitimate nature of temperature distribution, due to electron beam structure. To investigate the correlation in the distribution of temperature on the melting zone surface numeric series of temperature values were formed – in the presence (number 1), and in the absence (number 2) of rotary motion that showed practically the same distribution of temperature across all vertical cross sections (Fig. 3).

To determine the coefficient of correlation between temperature distributions in different modes the following formula [4] was used:

$$r_{xy}=\frac{\sum_{i=1}^{N}x_{1i}x_{2i}-N\bar{x}_1\bar{x}_2}{\sqrt{\left(\sum_{i=1}^{N}x_{1i}^2-N\bar{x}_1^2\right)\left(\sum_{i=1}^{N}x_{2i}^2-N\bar{x}_2^2\right)}}\,, \quad (1)$$

where x_{1i}, x_{2i} are signal value at the i point of the cross section, N is number of cross section points.

Tests have been carried out at 200 rpm of crystal, provided that N=80. Average correlation coefficient r_{xy}=0.923 has been obtained, indicating the presence of correlation in the distribution of temperature on the welding zone surface in the presence and in the absence of rotational motion.

The second problem is connected with the effect of electrode radiation reflection into input aperture, detected in the course of experiments (Fig. 4).

This effect is observed only in the liquid phase when the zone surface area is a reflector of complex shape. The picture changes when electrode is moving and due to changes in the zone shape.

Thus, the algorithm for zone height measuring, based on determination of the distance between sites with the set contrast on the ordinate, can lead to

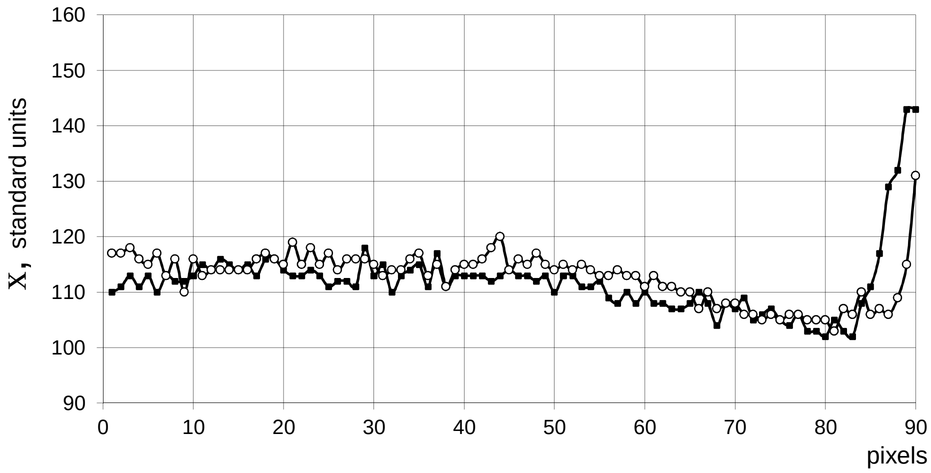
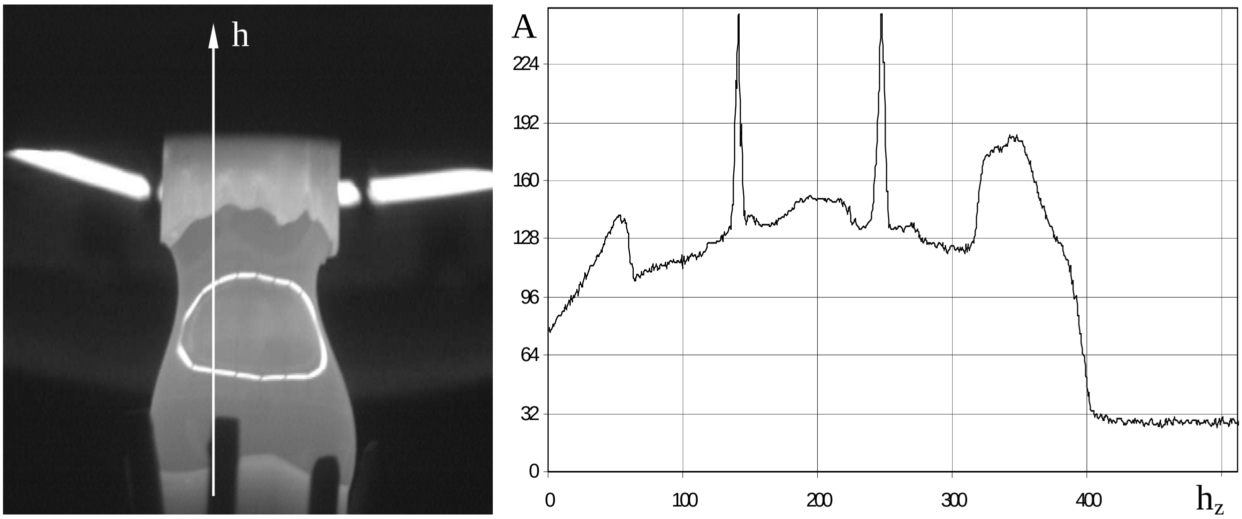


Figure 3 – To the issue of correlation ratio defining among the temperature distributions



a) b)

a) overall view of the zone, b) vertical signal breakup

Figure 4 – Electrode reflection on the surface of a melted zone

significant errors due to the emergence of additional contrasting sections on this ordinate. It is possible to avoid these errors, using amplitude filtering within the zone, focusing on the fact that the signal, formed by electrode image area, is much greater than the signal formed by adjacent areas (Fig. 4). But the practical implementation of the algorithm has been recognized as inappropriate, as the process of zone melting is accompanied by rapid and random changes of reflected flow.

We have suggested to apply adaptive algorithm, which provides that the height analysis is conducted in y direction only if there are two contrasting areas. With the emergence of the additional contrasting area algorithm provides countermotion of the upper and lower markers of the three points that are outside the melting zone. Meanwhile at each step sign and quantity of signal change is determined. As soon as the preset threshold of signal change is achieved, the coordinates of the markers are fixed and $h_з$ is calculated. In this way, the impact of reflected electrode image is completely eliminated.

It must be mentioned that the presence of temperature gradients that was above considered as a negative factor that generates fluctuations of impurities and thus prevents obtaining crystals with uniform concentration, in its turn could be the component of programmable techchnologies of production of materials with predetermined properties.

To justify the methods, used to determine melting zone height, the pecularities of liquid phase formation and melting process have been taken into account.

Fig. 5 presents conventional malting zone image and correspondent values of signals, generated by pixels of photoelectric converter, which are located on both sides near its upper interrface.

Values of signals A_i, formed by pixels that are "located" in the melting zone interface are in the range of $A_p<A_i<A_т$, where A_p, $A_т$ are signals, generated by pixels, "located" in images of liquid and solid phases areas.

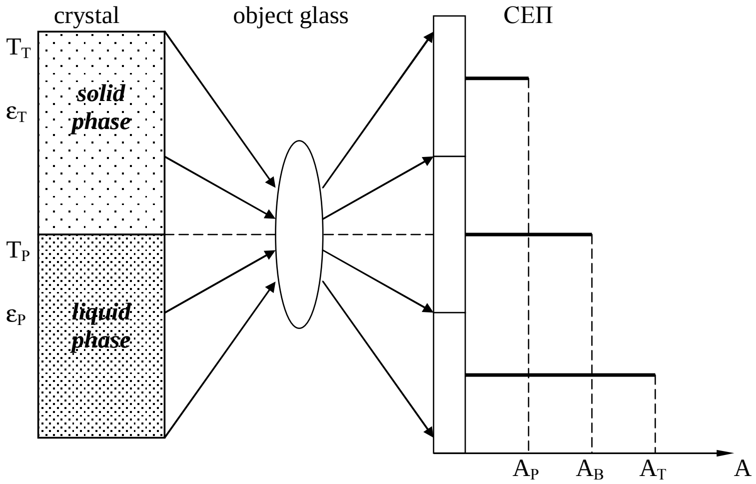


Figure 5 – Relative signal levels on zone interface

Since directly at the melting zone interface, the temperatures of solid and liquid phases are almost identical, the differences in its brightness are stipulated mainly by differences in coefficients of radiating capacity of surfaces in solid ε_T and liquid ε_P phases.

A_B signal, generated by pixel, located on the upper welding zone boundary image is coincidental and is constantly changing in the course of melting. At any given time its value is in the range of $A_P<A_B<A_T$, where A_P and A_T are average pixel signals values, located entirely within the image of liquid and solid phases correspondingly. A_H signal of the melting zone lower boundary is generated similarly. Such uncertainty of coordinates limits leads to errors in the course of melting zone height measurement. This increase in the accuracy of measurement is impossible without research on signal generation character within the melting zone boundaries and the development of appropriate techniques and algorithms. It is clear that the form of crystallization section where the mixing of substances in the liquid phase, takeas place and which actually determines the uniformity of distribution of impurities, in some way characterizes the melting zone shape, and thus the value of its height. Actually between solid and liquid phases, which are characterized by constant process parameters in terms of luminous flow formation, thre is the area, the brightness of which varies from a maximum value (at ε_T=0.64) to the minimum (at ε_P=0,46) during the transition from solid phase into the liquid and vice versus. The width of the area is stipilated by natural mechanisms of liquid phase formation and crystallization from melting. Abrupt changes in the dynamics of the process and temperature gradients also significantly influence the shape and position of the crystallization area, creating conditions for the deterioration of crystal quality due to emergence of local areas, supesaturated with impurities.

Pecularities of melting zone brightness fields must be taken into account while developing the methodology, aimed at the enhancement of the accuracy of coordinates measurement. Since we are talking about increasing the accuracy of the melting zone boundaries coordinates, light signalling characteristic should be considered in the range from a minimum A_P value, which corresponds to the brightness of the liquid phase at the interface to the maximum A_P value, corresponding to solid phase brightness on the same boundary.

Fig. 6 displays the distribution of brightness (signal) in the lower boundary of the melting zone, which proves that the site at the phase intherface has a width of about 0.3 mm. (In monographs [1, 3] it is stated that the width of the crystallization area is in the range of 0.15–0.35 mm). From Fig. 6, which shows changes in the signal on the interface of solid and liquid phases it can be concluded that brightness changes are of a linear character in the transitional area.

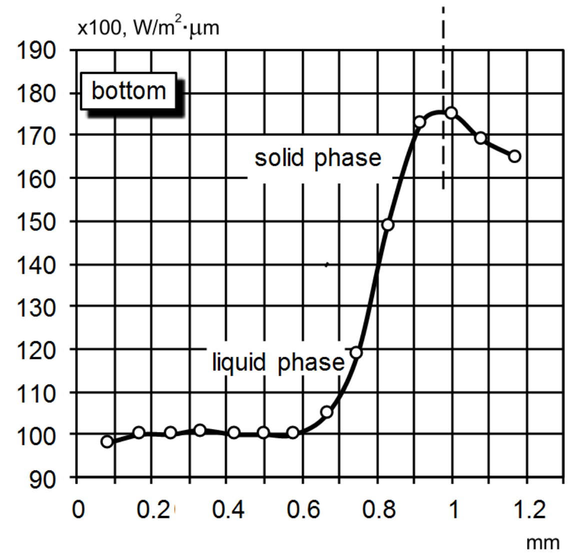


Figure 6 – Brightness distribution over melting zone surface (lower boundary)

Fig. 7 illustrates the adaptive algorithm for determining the height of the melting zone during melting at a speed of melting zone displacement of 100 mm per hour. Melting zone height has been calculated in pixels as the difference between the mean values of the coordinates Y_L and Y_U within the rectangular piece.

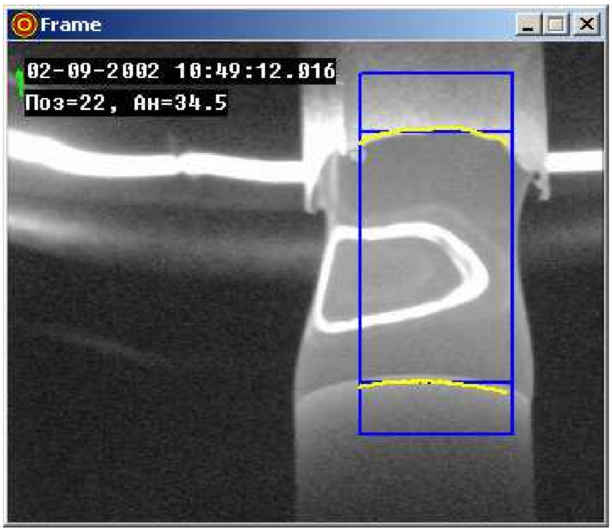


Figure 7 – Screenshot of the working window; h_z=146 pix, V=100 mm per hour

The abovementioned features of signal generation have been considered while improving the algorithm for adaptive search of melting zone boundaries, which could provide melting zone height control in the course of melting at the level of up-to-date requirements.

Conclusions

The new approach to measuring the melting zone height in the process of melting has been grounded. It takes into account the experimentally established peculiarities of formation of brightness field on the surface and at the interface which creates conditions for improving existing and developing more effective methods to control melting zone height.

It has been established that the algorithm of zone height measurement, based on determination of the the distance between sites in the y direction with the set contrast, can lead to significant errors due to the emergence of the additional contrasting areas on this ordinate, such as reflected electrode image.

An adaptive algorithm has been developed and tested which with the appearance of additional contrast areas provides counter-movement of the upper and lower markers from the initial points that are outside the melting zone and thus eliminates the reflected image impact on the measurement result.

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До питання контролю висоти зони плавлення в технології електронної емісії безтигельної зонної плавки кремнію

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Обґрунтовано новий підхід до контролю висоти зони розплаву в процесі безтигельної зонної плавки кремнію, який враховує експериментально встановлені особливості формування поля яскравості.

Ключові слова: *зона розплаву, електронно-променева плавка, плавлення кремнію.*