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Development of Silicon Growth Techniques from Melt with Surface Heating

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Abstract

The work contains literary and own data on the history of the development of silicon growing technologies with volumetric and surface heating of the melt. The advantages and disadvantages of technologies with surface heating are discussed. Examples are given of the implementation of such processes in the 60-70s of the last century and the reasons for the termination of the relevant works. Principal solutions for the realization of the crystal growth process with electron beam heating of the melt surface realized in KEPP EU (Latvia) are described. Differences in the management of the growing process of crystals with a constant diameter in comparison with the Czochralski method are discussed. The geometric and electro-physical properties of the obtained crystals are presented. Possible application of such crystals and the immediate tasks of technology development are described.

Introduction

The historical review of the silicon crystal growth methods goes beyond the goal of this paper, but in my opinion, since some historical data support the ideas set forth they should be illustrated in Table 1.

Tab 1. Silicon crystal growth history

Year	1950	1956	1962	1967	1972	1973	1974	1976	1980	1984	1986	1988	1990	1992	1997	2005	current time
CZ diameter, mm [1]	12	25	40	50	65	78			100	125		150		200	300	400	450
CZ charge, kg [1]	0,05	0,4	1,2	2,5	6	12			24	38		65		110	200	400	350
FZ diameter, mm [2, own data]		10	40				65	78	100		125		150				200
FZ/pydestal diameter [3,4]			40		42											60	no data
Skull silicon diameter,mm [5, own data]				40	45												see below
DSS size, cm [6,7]										32x32					60x60	75x75	115x115
DSS charge, kg [6,7]										50					170	450	1200

1. The influence of heating methods on the properties of crystals and the development of processes

The fundamental difference between the CZ and DSS methods from the FZ process, both conventional and pedestal, as well as from the process of growing from the skull, is most clearly illustrated by diagrams of numerical simulation of these processes shown in Fig. 1. In Fig. 1 shows the principal difference in the sizes of the surface heating zones in the FZ and electron-beam processes with a nominal diameter of 10-20 mm and volumetric heating of the melt in CZ and DSS, where the typical size The heating zone is measured in hundreds of Millimeters and the surface area in modern systems is about 1 Square meter.

Obviously, in the case of volumetric heating, the systems are characterized by more uniform thermal fields favorable for crystal growth. At the same time, a large number of

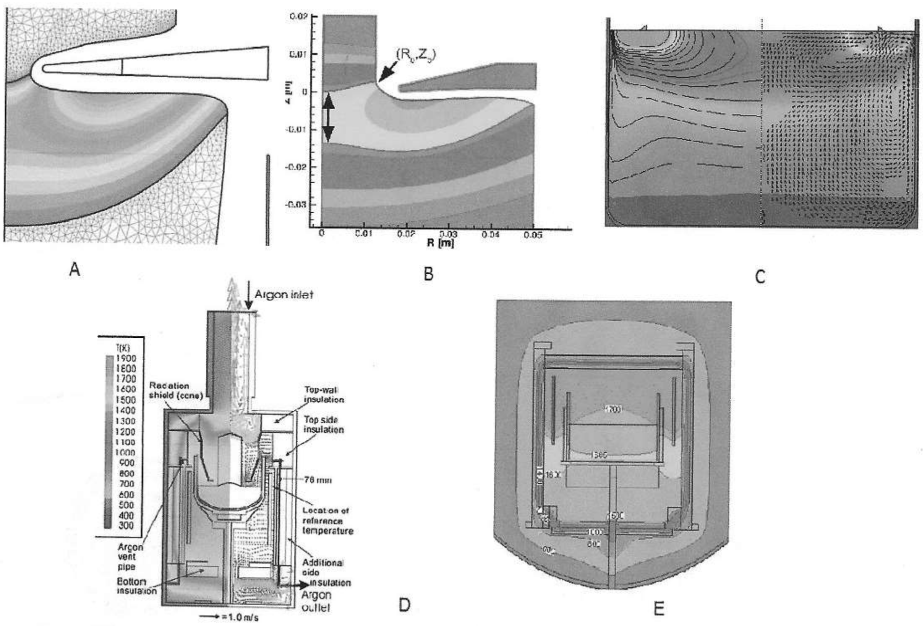

Fig 1. Area of heating in different crystal growing techniques:A- FZ [8]; B -pydestal [4]; C – Electron beam [own data]; D- CZ [9]; E – DSS [10]

different tools, mainly graphite, are used in mass heating methods, which leads to inevitable contamination of the melt in the process.

Much more pure methods of surface heating has a number of own shortcomings. Both FZ processes, employing either induction or electron-beam heating, require a polycrystalline silicon rod complying with a number of special requirements, both in terms of geometric features and process technology: deposition is carried out at a low rate. The low rate of rod deposition in the Siemens-process provides for its high density and the lack of amorphous phase inclusions. Such inclusions contribute to rather inhomogeneous rod melting and can lead to the formation of dislocations. Adherence to the technology of growing rods makes them very expensive - no less than 2 times more expensive than the lump polysilicon of appropriate quality used in the process of growing from the crucible. In addition, high stresses, exceeding a certain value, provide to the crack formation in the course of processing are not acceptable. Non-availability of the adequate diameter rod techniques hindered the development of the FZ process in the late 1960s, early 1970s and early 1990s. The high price of the above rods stipulates the high price of the FZ single crystals, limits their use and thereby the outcome of the equipment used in process.

The high cost of developing equipment extends to the low quantity of produced units, thus increasing the price of equipment. As a result, the second significant component of the cost price is added, which additionally raises the price and limits the use of FZ single crystals. The price advantage obtained in connection with the elimination of consumables is not enough to compensate for the increased costs of raw materials and depreciation, as a result, the use of silicon is limited to special detectors and power electronics. Constantly increasing demands on loads increase the requirements for the diameter of the plates (Fig. 2).

Taking into account the current trend and in accordance with the estimates [11], the need for plates with a diameter of 300 mm should be expected by 2020-25. To solve this problem, it is extremely important to produce high-purity rods of the appropriate diameter (250-350 mm). In the industry demand for 300-millimetre plates for high-power thyristors has appeared, but polycrystalline rods for the production of 300 mm single crystals and equipment and technologies to enable the production of corresponding single crystals are absent.

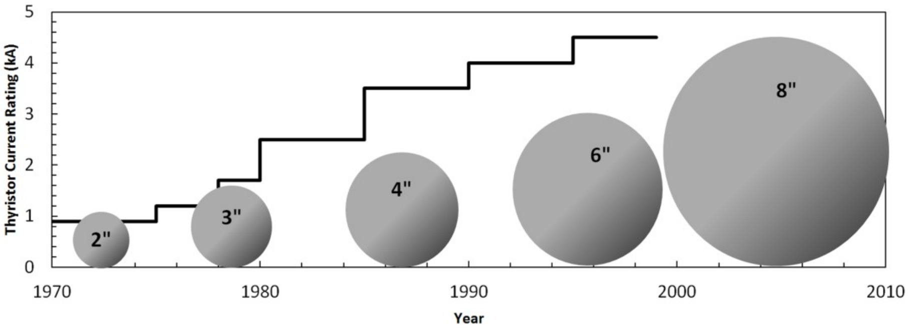

Fig 2. Growth of thyristor current and wafers diameter [11]

2. Growing crystals with electron beam heating

A slightly different situation with the processes of electron beam melting. Such processes were used in the growth of single crystals with dislocations from the pedestal [4] in a vacuum. At the same time, because of the problems of organizing the optimal heat distribution, as well as the significant evaporation of silicon, its condensation on the surfaces of the vacuum chamber, rigging, and entry into the melt and move to the crystallization surface, such processes have not developed. On the other hand, a number of processes with the use of a skull are patented at different times, the schemes of which are shown in Fig. 3.

Although these schemes suffer from a number of drawbacks, silicon single crystals were obtained as a result of their implementation. The circuit in Fig. 3a significantly benefits from zero requirements to the size and shape of the raw material, with the exception of the feeding rod. Obviously, the main problem of the process lies in the difficulty of heating the central region of the melt, which is cooled by a copper crucible and tends to joint to a growing crystal. In order to eliminate the above problem, we developed a furnace with reduced heat output from the bottom of the melt and established the scheme of the process shown in Fig. 4.

The presence of thermal insulation contributes to improving the energy efficiency of the process. In the case of a 200 mm rod growth, the whole process consumes up to 25 kWh / kg, including melting of the formation and growing crystals. (The energy consumption of the vacuum pump and cooling system is not included). The first crystals with a diameter of about 100 mm were obtained in 2011. In 2014, the diameter of rods grown in stable operation reached 170 mm, and in 2016 - 220 mm.

The architecture of the equipment provides for a fixed molten bath enabling to reduce melt mixing associated with the rotation of a large-diameter molten bath. The heating homogeneity is ensured by the motion of the focal spots of the electron beams. However, the

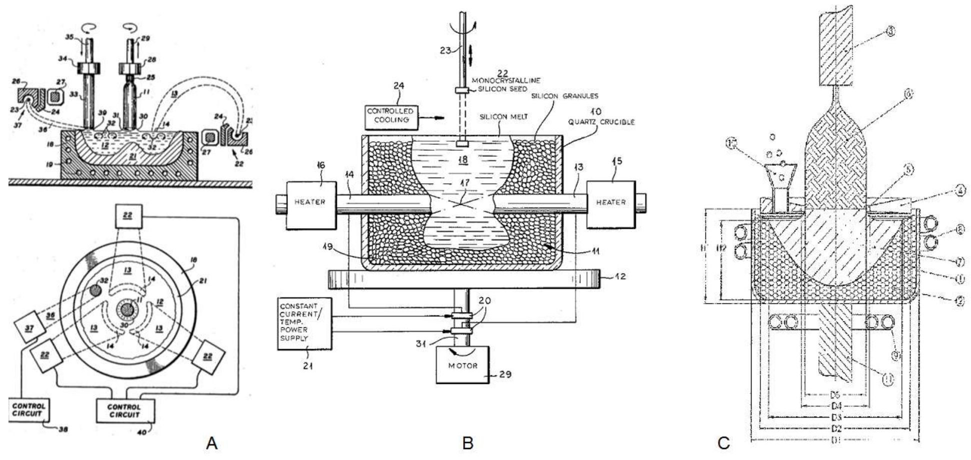

Fig 3. Different types of skull pulling processes: A – from cold crucible [5]; B – with inside current heating [12]; C – with multiple induction heating [13]

problem related to crystal diameter controlling turns up. As the crystal grows the level of the melt decreases and the controllable meniscus removes from the video camera. An attempt to use a laser distance meter encountered the problem of reflected rays scattering, since there is a wave exists on the surface of the melt.

Nevertheless, this does not imply any engineering faults but natural property of the process. The fact is that the electron beam creates pressure on the melt surface and any beam displacement, as a consequence, forms a wave on the surface. In addition, the electron beam forms a luminous trace and the melt meniscus near the skull surface features almost the same brightness as the growing crystal surface, Fig. 5C.

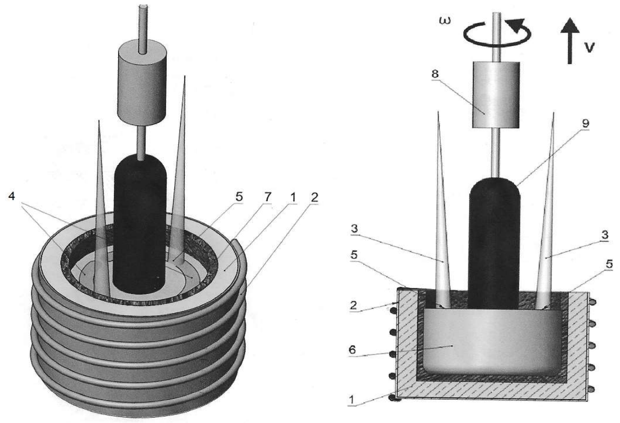

Fig 4. Electron beam pulling process developing at KEPP EU: 1-cold crucible; 2-insulation; 3-electron beam; 4- way of focal spot on melt surface; 5-focal spots; 6- melt; 7-container; 8- seed holder; 9 - pulling crystal

As a result, the camera periodically "loses" the object of observation. We managed to solve this problem in principle [14], by taking arrangements to regulate the diameter by speed of pulling proportionally to a diameter change, however, the control over the melt level has not been solved yet. As a result of the operations carried out, an industrial-scale plant designated to grow silicon polycrystalline rods (Fig. 5a) has been developed and the techniques that enables to grow rods up to 220 mm in diameter have been mastered. In addition, the principal feasibility of growing crystals up to 300 mm (12") in diameter has been shown. The maximum mass of the crystals obtained is 80 kg at a charge of 100 kg. The results of the impurity content control in crystals (N 3-2) and melt residues (N 5-2) in comparison with the high purity FZ single silicon (N-HR) are shown in Fig. 6.

Fig 5. Electron beam pulling process: A- puller; B- 6”; 9”; 12” ingots archived on puller shown at A; C- view of process

3. Increasing the purity of silicon grown using electron beam heating

Within the level of ICP MS capabilities it is shown that the obtained rod purity (sample No. 3-2) is practically the same as the purity of the reference sample of high purity FZ silicon (N-HR). The remedial works aimed to eliminate impurities detected in the melt residuals are currently being carried out within the framework of the Research No. 2.3

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The analysis of the impurities detected proves the need to clean the vacuum chamber surface prior to carrying out the process of obtaining clean rods as well as to form and maintain the condensed pure silicon layer thereon. The formation of a group of metallic impurities similar by composition to stainless steel indicates the exposure of the vacuum chamber and equipment steel parts to irradiation by primary or secondary electrons. In such a case, the most dangerous surfaces are those that may be irradiated by primary electrons, trajectories of which are slightly deviated from the axis of the main stream. Due to relatively small number of such electrons, they leave no traces on the steel surface but lead to the residual gases saturation in the vacuum chamber with the vapours of metals detected. Screening of these surfaces by other materials, for example, titanium, allows detecting the places of irradiation defined as a source of contamination. If the discharge of electron-beam heater materials is considered to be the source of contamination, these metals will be removed simultaneously with the electrode materials. To remove electrode materials, a gas-dynamic window has been developed in order to separate the gas-discharge and process chambers space by the membrane. On the side of the vacuum chamber the micro quantities of inert gas are supplied to the membrane, while on the side of the gas-discharge chamber a region of increased pressure of a mixture of this inert gas with the gases from the discharge chamber carrying electrode vapours is formed. The gas mixture from the high-pressure region is evacuated by means of an additional vacuum pump, carrying away the electrode

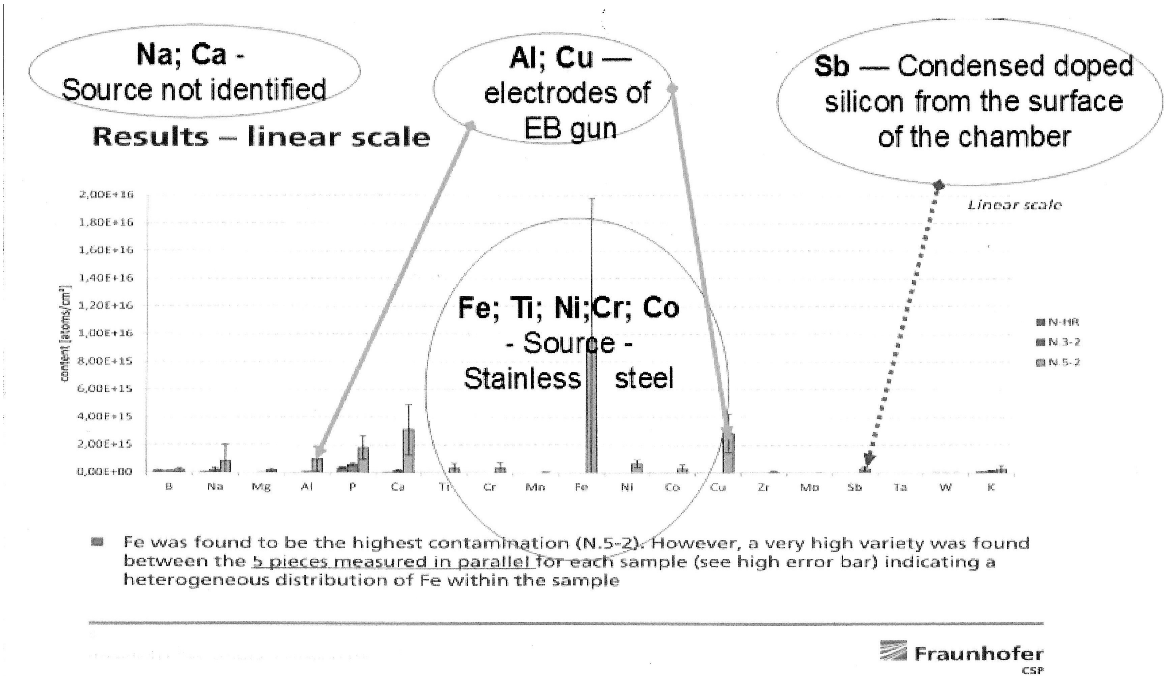

Fig. 6. Results of impurities control in high resistivity FZ sample (N-HR), sample from bottom part of electron beam pulled rod (EBR) (N 3-2) and residues of melt (N 5-2)

Conclusions

1) The advantages and disadvantages of the silicon crystal growth techniques employing local surface heating have been analysed.

2) For the first time in the world the equipment and technique have been developed to make the silicon rods growth from the melt heated by an electron-beam feasible. The availability of methods to obtain polycrystalline rods up to 300 mm in diameter has been exemplified to be followed by single crystals growth in the FZ process. Moreover, the purity of the rods at the control level of modern ICP MS corresponds to the purity of the standard FZ silicon.
3) The sources of impurities introduced in the course of growth process have been identified by traces thereof in the melt residues after the process. The remedies for elimination have been outlined to ensure the quality warranty of the rods produced.

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