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On combination of floating-zone recrystallization with other purification methods

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Information on combination of floating-zone recrystallization with other purification methods (Czochralski, Verneil, distillation) is summarized.

Обобщены сведения о совмещении бестигельной зонной рекристаллизации с другими методами очистки (методами Чохральского, Вернейля, дистилляцией).

The floating-zone recrystallization is an effective purification method of various substances. During development of this method, the use of liquid zones with incomplete melting and eccentric arrangement was proposed to increase productivity [1]. Recently, this embodiment of floating-zone melting takes an interest in connection with possibility to combine it with other purification methods. An initial material for floating-zone melting often has form of scrap or powder and it must be shaped (for example, by melting) to a rod, desirably of a round section. Other methods of crystal growth may be used as a preliminary operation to shape the rod. A publication that summarizes separate data about these developments seems to be useful.

A crystallization method was proposed to purify substances where a crystal is subjected to floating-zone melting in the course of its forming by pulling the crystal from melt (Czochralski method), Fig. 1 [2]. The crystallization purification of substance by a combined method occurs as during the crystal forming by Czochralski as well as during the floating-zone recrystallization.

The floating-zone melting can be combined with Verneil method to prepare pure single crystals from refractory powders (Fig. 2) [3]. The growing crystal *1* moves relatively to the zone heater (or zone heaters) and is subjected to additional purification by floating-zone recrystallization.

Later, it was proposed to increase the purification efficiency by substitution of the crystal pulling from melt by distillation with condensation in a temperature gradient [4], Fig. 3. To that end, a distillation device is used that provides the rod-shaped condensate by pulling the condensate from condensation zone [5]. It is known that distillation may be a more effective purification method from several admixtures for many substances in comparison with recrystallization [6] and that it is expedient to use a complex of purification methods to increase the purification efficiency [7]. Moreover, the distillation with condensation in temperature gradient provides an increased purification efficiency in comparison with simple distillation [8].

In the device shown in Fig. 3, a temperature gradient along the vapor duct is established a result of heat radiation from crucible. At the initial position, the seed crystal is placed so that its bottom butt has the predominant condensation temperature of

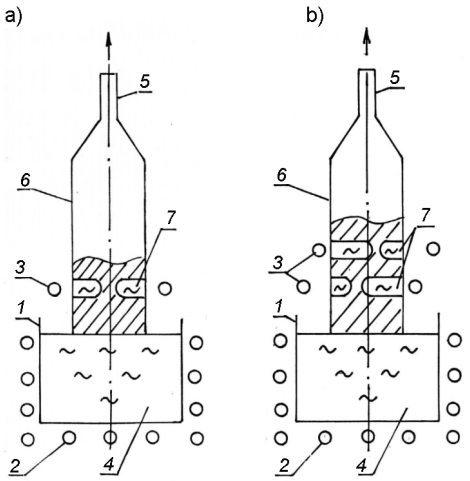

Fig. 1. Scheme of floating-zone melting combined with the crystal pulling from the melt: a) with one liquid zone; b) with two eccentric zones: *1*, crucible; *2*, crucible heater; *3*, zone heater; *4*, melted initial material; *5*, seed crystal; *6*, pulled crystal; *7*, liquid zone.

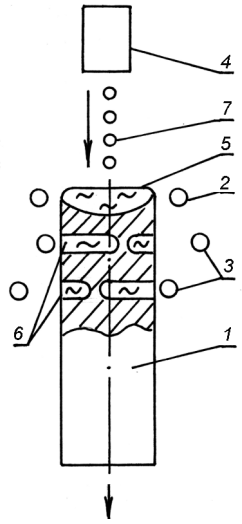

Fig. 2. Scheme of floating-zone melting in combination with Verneil method: *1*, crystal; *2*, main heater; *3*, additional zone heater; *4*, dosing device for initial powder; *5*, melt; *6*, liquid zones; *7*, initial powder.

main component. The vapors of the substance to be purifired move along the vapor duct and are condensed on the crystal bottom butt. (The baffles prevent the direct passing of vapors from material in crucible to crystal without repeated impacts with the duct wall). The low-volatile components condense in the duct below the level of the predominant condensation temperature of main basic component while volatile components go away through the gap between the crystal and the vapor duct. As a result, the pure condensate grows on the crystal bottom butt. The growing crystal is moved relatively of zone heater (or zone heaters) and is subjected to supplementary purification by floating-zone recrystallization.

Thus, the schemes shown demonstrate a combination possibility of the floating-zone recrystallization with other purification methods (Czochralski, Verneil, distillation) to increase of purification efficiency. It is to note that in these methods, the purification from impurities connected with crucible material occurs, too, and that the shown schemes permit to realize the floating-zone melting with melt feeding in crucible. The combined methods have a lower power requirements since the crystal exposed to floating-zone melting did not get cold in previous process.

It is to note also that there are equations of impurity distribution along the crystals

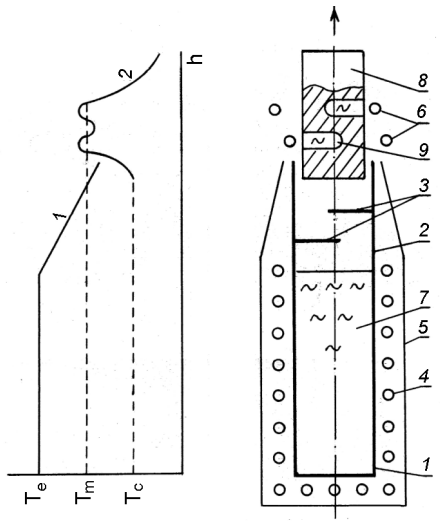

Fig. 3. Scheme of floating-zone melting in combination with distillation: *1*, crucible; *2*, vapor duct; *3*, bafflers; *4*, crucible heater; *5*, heat shield; *6*, zone heaters; *7*, initial material; *8*, crystal; *9*, liquid zone. Curve *1* is the temperature distribution along the crucible and vapor duct; curve *2*, the temperature distribution along the crystal. T_e is the evaporation temperature, T_m is the melting temperature, T_c is the predominant condensation temperature of main component.

grown by various directional recrystallization methods [9] or using the distillation [10, 11]. However, the calculation of impurity distribution in the crystal for combined methods remains still an unsolved problem.

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Про сполучення безтигельної зонної плавки з іншими методами очистки

О.І.Кравченко

Узагальнено відомості про сполучення безтигельної зонної плавки з іншими методами очистки (методами Чохральського, Вернейля, дистиляцією).