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ЛАБОРАТОРНАЯ ТЕХНИКА

TUBE FOR OBTAINING CRYSTALS IN A LABORATORY FURNACES

© 2011 B. Cabric[a], N. Danilovic[a], A. Janicijevic[b]

[a] *Faculty of Sciences, PO Box 60, 34000 Kragujevac, Serbia, e-mail: bcabric@kg.ac.rs*

[b] *Faculty of Technology and Metallurgy, P.O. Box 494, 11000 Belgrade, Serbia*

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A model of an air-cooled tube with a number of movable plugs, installed in a laboratory tube furnace is presented. The setup (modular "crystallization shelf-comb") allows easy regulation and simultaneous crystallization tests of a series of crystallization parameters in crucible columns, enabling rapid acquisition of crystals. The relationship between the crystallization parameters has been derived and numerically analyzed. This method can also be applied in crucible or chamber furnaces.

In previous paper [1] we have described a model of an air cooled tube with string of movable spheres ("crystallization lattice bridge") installed in a laboratory tube furnace, with the aim of regulating the crystallization fronts and rates in columns of crucibles. In this paper, we describe the improvement of the interior of the cooler, i.e. an air-cooled tube with a series of radial holes ("crystallization shelf-comb") (Fig. 1). The improved cooler is simple to build and handle, features regulation paths and rates of air flow better, and can also be applied in crucible or chamber furnaces. The cooler allows simultaneous crystallization tests ("comb") of different crystallization parameters, with the purpose of obtaining crystals.

The procedure involves first an increase of the voltage until the substances are completely melted. Then, while a constant furnace voltage is maintained, a small airflow is introduced through the cooler and crystallization starts on the surface of the melts for the lower row (Fig. 2) and at the bottom of the melts for the upper row. With the increase of the airflow, the crystallization front reaches the bottom and the surface of the melt [2, p. 271; 3, p. 596].

The shapes of crystallization fronts in each crucible are regulated by the plug front, i.e. the plug head ("crystallization seals"). The crystallization rate interval in each crucible is regulated by the cross-section of the airflow a (see equation (2) of [4]) and the plug height δ_p. The crystallization rate in the crucibles below the cooler can also be regulated by the distance of the plug front from the surface of the melt b. The temperature gradient is regulated by the distance of the cooler from the furnace wall c. Different temperature gradients in the crucibles can be simultaneously tested using an inclined cooler.

The rate of melt solidification depends upon extracting the latent heat of solidification. For a time interval t a crystal layer of thickness δ is formed (Fig. 1). During the formation of an elementary crystal layer of thickness $d\delta$ per unit area, the amount of heat released is $L\rho d\delta$ (L denotes the latent heat of solidification and ρ the crystal density); the latter is being extracted through the cooler for a time interval dt. On this basis the following equation may be written [4]

$$L\rho d\delta = \frac{\Delta T}{1/\alpha + \delta_p/k_p + \delta/k} dt, \qquad (1)$$

where ΔT denotes the difference between the temperature of the melt and that of the air stream, α is the coefficient of heat transfer from the cooler wall to the

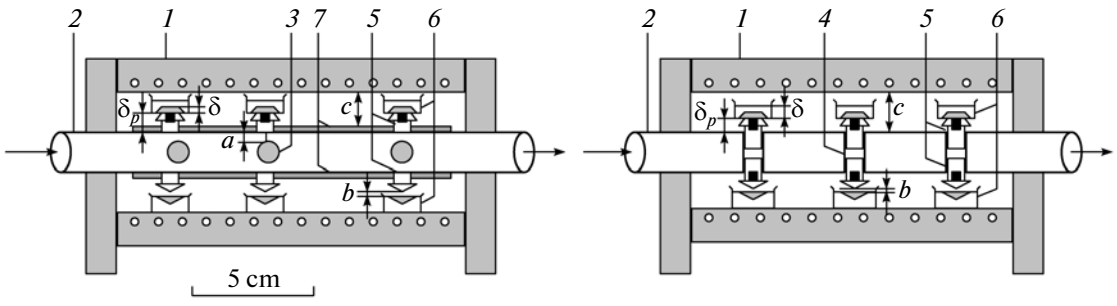

Fig. 1. Crystallization tube in a tube furnace: (*1*) laboratory tube furnace, (*2*) air cooled tube ("crystallization shelf-comb"), (*3*) radial holes in a horizontal position ("crystallization thresholds"), (*4*) radial holes in a vertical position ("crystallization sockets"), (*5*) movable cold plugs, (*6*) columns of crucibles, and (*7*) slide bars.

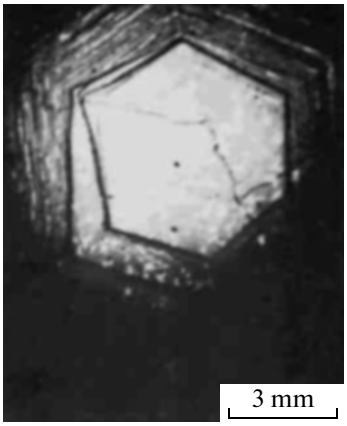

Fig. 2. Single crystal plates of $BaNi_2(PO_4)_2$ (newly synthesized compound with layered crystal structure [3]) formed on the surface of the melt below the plug head.

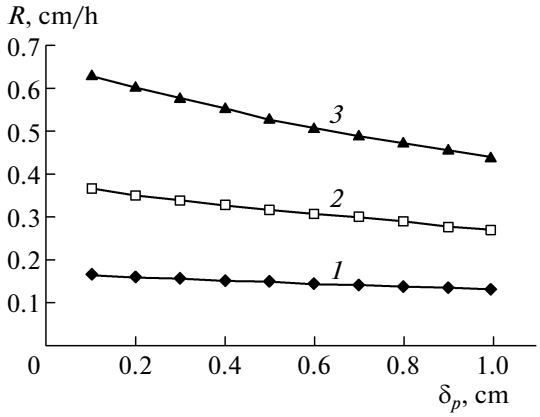

Fig. 3. Crystallization rate as a function of the height of the plug ρ_p, in the case of tin (Sn): L = 58200 J/kg, ρ = = 7300 kg/m^3, k_p = 0.756 W/mK, k = 59.8 W/mK, δ = = 2 cm, *1* – ΔT = 10 K, α = 20 W/m^{2}K; *2* – ΔT = 15 K, α = 30 W/m^{2}K; *3* – ΔT = 20 K, α = 40 W/m^{2}K.

air stream, δ_p denotes the plug height, k_p designates the heat conductivity of the plug and k is the heat conductivity of the crystal.

Transforming equation (1) we obtain

$$R = \frac{d\delta}{dt} = \frac{\Delta T}{L\rho(1/\alpha + \delta_p/k_p + \delta/k)}. \quad (2)$$

The quotient $d\delta/dt$ denotes the rate of crystal layer growth, which is usually represented by the symbol R.

In accordance with equation (2), we obtained numerical values of crystallization rate R in function of the height of the plug δ_p (Fig. 3).

Plugs and plug heads of various shapes and dimensions can be mounted and thus tested simultaneously. By varying the internal and external shape and dimensions of the cooler, a set of crystallization tubes-combs can be modeled for tests in a wider range of crystallization parameter and substances. Plugs can be mounted on several rings and slipped over the cooler ("cylindrical crystallization comb"), and installed in a tube furnace in a vertical position ("grafted crystallization tree").

A cooler can be twisted into the shape of the letter "U" ("crystallization horseshoe") and installed in a crucible or chamber furnace. Several different coolers (a family group of "crystallization horseshoe") or a planar air cooler (cold board) with a matrix of movable plugs ("crystallization plug-board") can be installed in a chamber furnace. This increases the number of simultaneous crystallization tests of different crystallization parameters and substances, enabling fast studies of obtaining single crystals, using a low-budget, simple to install and regulate, portable ("pocket") device and laboratory furnaces.

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