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Design and Calculation for Test Tube with the Aim of Regulation Simultaneous Crystallization Tests

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ABSTRACT

A design for an air-cooled test tube, with a series of modular and movable rings (cylindrical "crystallization comb"), installed in a laboratory crucible furnace is presented. The setup allows easy regulation simultaneous crystallization tests of a series of different crystallization rates in several columns (matrix) of test tubes, enabling fast studies of obtaining crystals. This low-budget, portable device (*i.e.* adjustable airstream with more simple control options), can also be applied in tube and chamber furnaces. The relations between the crystallization rate and parameters of air-cooled test tube are given and numerically analyzed.

Keywords: Technique for Crystal Growth; Design and Calculation; Test Tube—Cylindrical Crystallization Combs; Crystallization Clusters—Obtaining Crystals from Melt

In a previous paper [1] we have described a design for air-cooled test tube (bilateral "crystallization comb"), installed in a laboratory chamber furnace with the aim of regulating the crystallization fronts and rates in a two columns of crucibles. In this paper, we describe the development and improvement of the exterior of the cooler, *i.e.* modular air-cooled test tube (cylindrical "crystallization comb") with several columns of test tubes ("crystallization cluster") (**Figure 1**), and a numerical study of control options of matrix of new crystallization parameters (**Figures 2** and **3**). The improved cooler is simpler to build and handle, features regulation of different crystallization fronts and rates in several columns of test tubes [with the help of telescopic movable tube/sieve—see (5) in **Figure 1(a)**] or rings—see (9) in **Figure 1(b)**]. This stationary and adaptive device, with operating body air (gas), allows easy regulation simultaneous crystallization tests of different crystallization parameters and substances, enabling fast studies of obtaining single crystals from substances with unknown crystallization parameters, using a laboratory furnaces.

By the assumption that the liberated latent heat of solidification is equal to the heat removed by the air stream through the cooler, the following expression for the matrix of crystallization rates R_{ij} in the test tubes (see **Figure 1(b)**) is derived [4]:

$$R_{ij} = \frac{\Delta T_j}{\lambda_c \rho_c \left(1/\alpha_i + \delta_j/k_j + \delta_c/k_c\right)} \quad (1)$$

where i and j denote the indices of internal rings and external rings/plugs, c is the crystallization substance index, ΔT denotes the difference between the temperature of the melt and that of the air stream, λ is the latent heat of solidification, ρ designates the crystal density, α is the coefficient of heat transfer from the cooler wall to the air stream, k designates the heat conductivity.

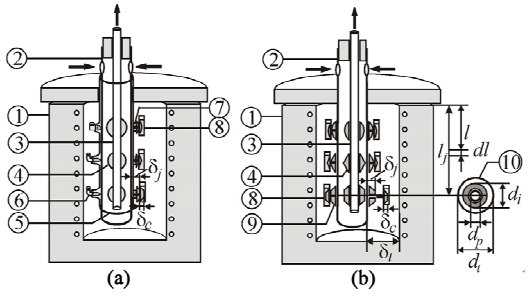

Figure 1. Air-cooled test tube (cylindrical "crystallization comb") in a crucible furnace: (1) laboratory crucible furnace, (2) air-cooled test tube (cylindrical "crystallization comb"), (3) pipe ("draining shuttle"), (4) modular and movable rings, (5) telescopic movable tube with mounting holes (crystallization test sieve), (6) curved Tamman test tubes [2,3] ("crystallization tests comb"), (7) mounting plugs, (8) test tubes ("crystallization cluster"), (9) rings with radial mounting holes ("junction rings"), and (10) cross section of the air-cooled test tube.

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The coefficient of heat transfer from the test tube wall to the air stream can be calculated using the following expression (p. 152 of [5]):

$$\alpha_i = \left[4.13 + 0.23\left(\frac{\theta}{100}\right)\right]\frac{w_{i0}^{0.75}}{d_t^{0.25}} \quad (2)$$

where θ is average temperature of the airstream in ˚C (up to 1000˚C), and

$$w_{i0} = w_i \frac{273}{273+\theta}$$

where w_i is average velocity of the airstream next to the ring i (0˚C, 1.013 bar) in m/s.

On the basis of the continuity and the cross section of the airstream, the following expression for the velocity of the airstream next to the ring i, w_i, we obtain:

$$w_i = w_t\left(\frac{d_t^2 - d_p^2}{d_t^2 - d_i^2}\right) \quad (3)$$

where w_t denotes an average velocity of the airstream in the test tube without ring, d_t and d_p is the diameter of the test tube and the pipe respectively, d_i is diameter of the ring—see (10) in **Figure 1(b)**.

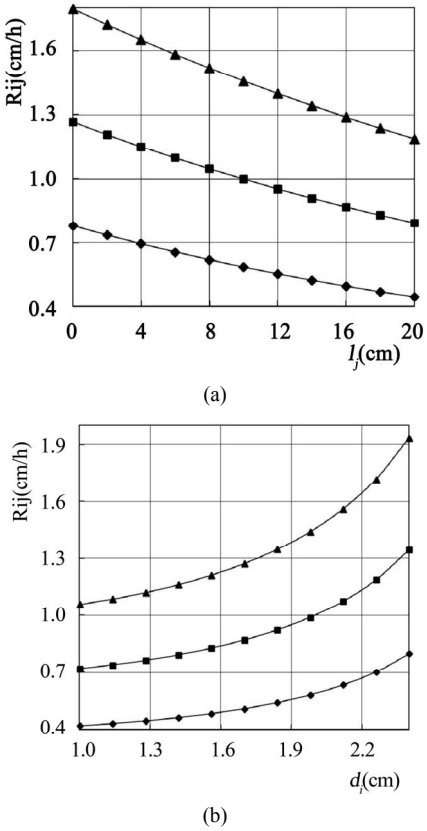

Figure 2. Crystallization rate R_{ij} as a function of the position of the ring along the test tube, l_j, and the diameter of the ring, d_i, when ΔT_0 = 150˚C: –♦– w_t = 0.6 m/s; –■– w_t = 1.2 m/s, –▲– w_t = 2 m/s. (a) d_i = 2 cm; (b) l_j = 10 cm.

Based on the fact that the heat removed from the test tube wall is equal to the heat accepted by the air stream, we have derived the following expression (integral equation) for the difference between the temperature of the melt and that of the air stream ΔT_j at the point l_j:

$$\Delta T_j = \Delta T_0 - \frac{4}{w_t d_t \rho_a c_a}\int_0^{l_j} \alpha_t \Delta T_l dl \quad (4)$$

where ΔT_0 denotes the difference between the temperature of the melt and that of the air stream at the point l_j = 0, (**Figure 1(b)**), α_t is the coefficient of heat transfer from the test tube without ring to the air stream [Equations (2) and (3) when $d_i = d_p$], ΔT_l denotes the difference between the temperature of the melt and that of the air stream at the point l, ρ_a and c_a designates density and heat capacity of the air stream, respectively.

In accordance with Equations (1)-(4), the authors obtained the numerical values of crystallization rate, R_{ij}, as function of l_j and d_i (**Figure 2**), and w_t and ΔT_0 (**Figure 3**),

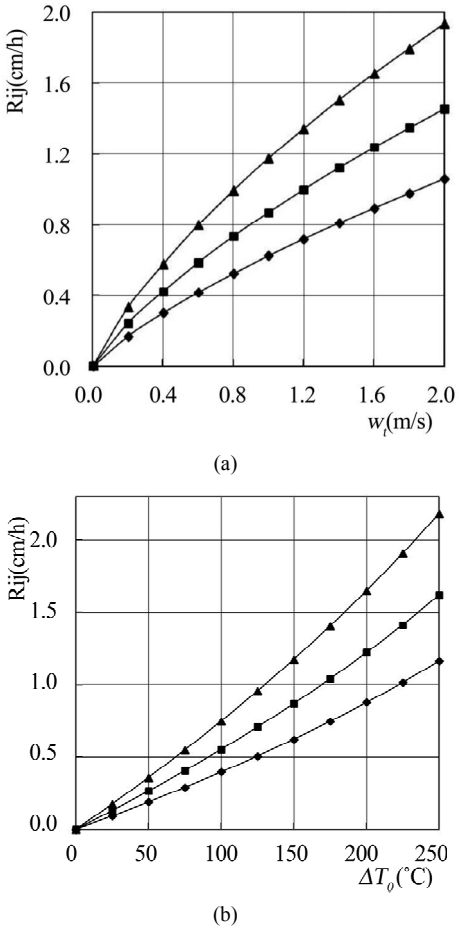

Figure 3. Crystallization rate R_{ij} as a function of the velocity of the airstream in the test tube, w_t, and difference between the temperature of the melt and that of the air stream at the point l = 0 – ΔT_0, when l_j = 10 cm: –♦– d_i = 1 cm, –■– d_i = 2 cm, –▲– d_i = 2.4 cm. (a) T_0 = 150˚C; (b) w_t = 1 m/s.

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in the case of bismuth: T_{melt} = 271˚C, λ_c = 52300 J/kg, ρ_c = 9800 kg/m^3 and k_c = 7.2 W/mK. In all numerical calculation is taken that: d_t = 3 cm, d_p = 1 cm, δ_j = 0.5 cm, k_j = 0.756 W/mK (pyrex *i.e*. borosilicate glass, softening point ≈ 600˚C), δ_c = 0 cm (**Figure 1(b)**), ρ_a = 0.682 kg/m^3 and c_a = 1.035 kJ/kgK.

The shape of crystallization front in each test tube can be regulated by the plug or ring front (**Figure 1**). The crystallization rate in each test tube can be regulated by the position of the ring along the test tube (**Figure 2(a)**) and by the diameter of the ring (**Figure 2(b)**); and/or by the velocity of the air stream and the difference between the temperature of the melt and that of the air stream at the point l_j = 0—**Figures 3(a)** and **(b)**, respectively. The crystallization rate in tests tubes can also be regulated by the thickness of the ring δ_j (Equation (1)). Different crystallization rates in the test tubes in one ring ("wreath") can be simultaneous tested using asymmetric ring. The temperature gradient is regulated by the distance air-cooled test tube from the furnace wall δ_t. Different temperature gradients in the test tubes in one ring can be simultaneous tested using air-cooled test tube dislocation to the axis of the furnace*, i.e.* asymmetric position of the key.

Tamman test tubes (Figure 3.1.-8 of [2]; Figure 3.1-2. of [3]), plugs and rings of various numbers, shapes and dimensions can be mounted and thus simultaneously tested (fineness of the comb). By varying the internal and external shapes and dimensions of the cooler, a set of "crystallization combs/keys" can be modeled for tests over a different range of crystallization fronts and rate intervals. The air cooled test tube can be installed into a tube furnace in a horizontal position ("crystallization shelf"—Figures 16 and 17 in [6]) or vertical position ("crystallization corncob"—Figure 13 in [6]). Several different air-cooled test tubes (a family group of "crystallization keys/combs") can be installed in the chamber furnace (Figure 11 in [6]. This increases the number of simultaneous crystallization tests (comb) of different crystallization parameters, using a low-budget, modular, easy to install and remove, portable ("pocket") device, *i.e.* airstream with more simple control options. This provides a way ("crystallization cluster") for fast studies of obtaining crystals from substances with unknown crystallization parameters, and the economic expansion application of laboratory furnaces.

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