DOI: 10.31319/2519-2884.48.2026.7
UDC 669.112.22

Bashev Valerii, Doctor of Physical and Mathematical Sciences, Professor of the Department of Condensed State Physics
Tomina Anna-Mariia, Candidate of Technical Science, Associate Professor of the Department of Condensed State Physics
Dorozhka Tatyana, Postgraduate Student, Department of Condensed State Physics
Fokin Vladislav, Postgraduate Student, Department of Condensed State Physics
Dniprovsky State Technical University, Kamianske

Башев В.Ф., д.ф.-м.н., професор, ORCID: 0000-0002-3177-0935, e-mail: bashev_vf@ukr.net
Томіна А.-М.В., к.т.н., доцент, ORCID: 0000-0001-5354-0674, e-mail: an.mtomina@gmail.com
Дорожка Т.М., здобувач третього (доктор філософії) рівня вищої освіти,
e-mail: tndorozhka@gmail.com
Фокін В.І., здобувач третього (доктор філософії) рівня вищої освіти, e-mail: oms@dstu.dp.ua
Дніпровський державний технічний університет, м. Кам’янське

FEATURES OF THERMAL COOLING MODES IN SPLAT-QUENCHING AND MICROWIRE CASTING

The article examines the features of thermal cooling regimes of a melt during splat quenching and micro-wire casting in glass insulation under ultra-high cooling rates. On the basis of heat-conduction equations and similarity-theory criteria, a quantitative evaluation of heat-transfer coefficients and cooling rates at the contact and free surfaces of rapidly solidified films, as well as of micro-wires with various diameters cooled in air and water, is carried out. It is established that during splat quenching, simultaneous non-equilibrium crystallisation occurs on both surfaces of the film, while the achieved degrees of supercooling (≈250—350 K) and cooling rates are sufficient for the formation of metastable structures. It is shown that a decrease in film thickness and micro-wire diameter leads to a sharp increase in both the heat-transfer coefficient and the cooling rate.

***Keywords**: splat quenching; cooling rate; heat transfer; non-equilibrium crystallisation; rapidly solidified films; glass-coated microwires.*

У статті досліджено особливості теплових режимів охолодження розплаву під час splat-загартування та виготовлення мікродроту в скляній ізоляції за умов надзвичайно високих швидкостей тепловідводу. На основі рівнянь теплопровідності та критеріальних співвідношень теорії подібності проведено кількісний аналіз коефіцієнтів тепловіддачі й швидкостей охолодження для контактної та вільної поверхонь швидкоохолоджених плівок, а також мікродротів різного діаметра при охолодженні в повітрі та воді. Установлено, що під час splat-загартування відбувається одночасна нерівноважна кристалізація з обох поверхонь плівки, а досягнуті ступені переохолодження (≈250—350 K) і швидкості охолодження є достатніми для формування метастабільних структур. Показано, що зменшення товщини плівки та діаметра мікродроту зумовлює різке зростання коефіцієнта тепловіддачі й швидкості охолодження. Аналіз температурних полів і розподілу переохолодження по перерізу плівок дозволив якісно та кількісно пояснити спостережувані зміни структурної морфології. Встановлено, що при литті мікродроту в скляній ізоляції, незважаючи на нижчі швидкості охолодження порівняно зі splat-загартуванням, аморфна скляна оболонка сприяє підвищенню рівня нерівноважного формування структури матеріалу.

***Ключові слова**: splat-загартування; швидкість охолодження; теплопередача; нерівноважна кристалізація; швидкоохолоджені плівки; мікродріт у скляній ізоляції.*

Problem's formulation

In the methods of splat quenching (SQ) and glass-insulated microwire casting (GIC), the cooling rate (CR) of the melt reaches values up to 10^8 K/s, due to which a wide range of metastable structural states is formed in non-equilibrium alloys, including micro- and nanocrystalline, as well as amorphous alloys, with unique complexes of physicochemical properties [1, 2]. At the same time, the high level of physical, mechanical, chemical, and other properties of rapidly cooled SQ materials has stimulated the development of high-performance technological varieties of SQ suitable for industrial and microelectronics applications [1, 2]. The formation of samples under conditions of non-equilibrium cooling requires technological modes of crystallization in which the required cooling rate of the material would be realized, as this determines both the morphology and a number of physicochemical properties of films and cast microwires in glass insulation [3, 4]. However, methods for direct experimental determination of CR in such samples are practically impossible, and the complexity of CR estimation is associated with establishing the dependence of the heat transfer coefficient on the parameters that determine it [5—8]. Additionally, the simultaneous non-equilibrium crystallization observed on both contact and free surfaces [7, 8] makes it difficult to predict structural morphology, limiting the controlled formation of materials with the desired set of mechanical, thermal, and physical properties. Therefore, there is a practical need for theoretical and computational approaches that allow reliable calculation of heat transfer coefficients, cooling rates, and the resulting microstructural evolution in rapidly solidified films and microwires [5, 6, 9].

Analysis of recent research and publications

Recent studies of SQ and glass-insulated microwire casting have shown that extremely high cooling rates allow the formation of a wide range of metastable structural states, including micro- and nanocrystalline and amorphous alloys with unique physicochemical properties. These non-equilibrium cooling techniques provide materials with enhanced physical, mechanical, and chemical characteristics, making them attractive for industrial and microelectronics applications. At the same time, the complexity of heat transfer processes and the simultaneous crystallization at contact and free surfaces create challenges in predicting and controlling the structural evolution of rapidly cooled films and microwires. Experimental determination of cooling rates is technically difficult due to the influence of multiple parameters such as sample dimensions, thermophysical properties of the cooling medium, and processing conditions. Consequently, recent research has focused on developing theoretical and computational models to estimate cooling rates and heat transfer coefficients, enabling a more accurate prediction of microstructure formation and optimization of processing conditions for high-performance materials [4, 8, 9].

Formulation of the study purpose

The purpose of this study is to develop a theoretical and computational framework for estimating heat transfer coefficients and cooling rates during splat quenching and glass-insulated microwire casting. The work aims to improve the prediction of non-equilibrium crystallization processes and the resulting microstructural evolution in rapidly solidified films and microwires, enabling controlled formation of metastable states with desired physicochemical and mechanical properties.

Presenting main material

In the methods of splat quenching and GIC, the cooling rate of the melt reaches values up to 10^8 K/s, due to which a wide range of metastable structural states is formed in non-equilibrium alloys, including micro- and nanocrystalline, amorphous alloys, with unique complexes of physicochemical properties. At the same time, the high level of physical, mechanical, chemical and other properties of rapidly cooled materials has stimulated the development of high-performance technological varieties of SQ, suitable for use in industrial and microelectronicscale [1, 2]. In the process of forming samples under conditions of non-equilibrium cooling, it is necessary to create such technological modes of crystallization in which the required cooling rate of the material would be realized. The latter determines, first of all, the formation of both morphology and a number of physicochemical properties of CR from the liquid state of films and cast microwire in glass insulation [3, 4]. Methods for direct experimental determination of CR in such samples are practically impossible, therefore there is a practical need to obtain calculated relationships using the provisions of the general theory of heat transfer (HT) [5, 6]. In the process of HT, heat transfer mainly occurs through the collision of the melt and the cooling medium, which is described by Newton's equation:

$$q=\alpha(T_P-T_C), \tag{1}$$

where q is specific heat flux; α is heat transfer coefficient; $(T_P$-$T_C)$ is the difference between the temperature of the wall and the cooling medium.

To describe the thermal processes in the method of SQ a sample droplet on the inner heat-conducting surface of a cylinder that rotates rapidly (~8000 rpm), the model of one-sided heat flow through the contact surface “film-cylinder” is usually used using the well-known Fourier equation of heat conductivity. To simplify the calculations, it is usually assumed in this equation that the heat flow through the free surface of the film can be neglected [1]. However, there are experimental results from which it follows that in some cases of HT, simultaneous crystallization can be observed on the contact and free surfaces [7, 8]. In the latest work, the authors, when obtaining metallic glass $Fe_{76,1}Nb_{3,0}Cu_{1,0}Si_{13,8}B_{6,1}$ computer modeling of surface crystallization processes due to heat removal through the free surface was carried out depending on the quenching modes. It was found that depending on the nature of crystal nucleation and growth rate, it is possible to realize the entire variety of surface crystallization phenomena observed when obtaining metallic glass. Therefore, under these conditions, the estimation of the CR of the free surface of the film and the CR core is of particular interest. The complexity of the CR estimation is primarily associated with establishing the dependence of the heat input coefficient α from the parameters that determine it. It has been experimentally established [1, 7, 8] that α depends on the diameter of the CR and the size of the quenching film, the thermophysical properties of the cooling medium, the temperature pressure $(T_P$-$T_C)$. The properties of the medium can be described by the following basic parameters: coefficient of thermal expansion β [K^{-1}], thermal conductivity λ [W/m·K], specific heat capacity C_P [J/kg·K], density ρ, dynamic and kinematic viscosity μ and $\nu=\dfrac{\mu}{\rho}$ [m^2/s], a — thermal conductivity [kg/m·s] and a common parameter that depends on the shape, structure of the surface and its dimensions. Obtaining such experimental dependences is practically impossible due to the significant number of parameters that affect the process under study. Therefore, in this work, the relations obtained using the similarity theory [5] and experimental dependences were used. Three thermophysical complexes can describe all parameters that affect the thermal conductivity coefficient: the Nusselt number (Nu), the Grashof number (Gr), and the Prandtl number (Pr):

$$Nu=\frac{\alpha L}{\lambda};\ Gr=\frac{\beta g L^3(T-T_C)}{\nu^2};\ Pr=\frac{\nu}{a},\ a=\frac{\lambda}{C_P\rho}, \tag{2}$$

where L is geometric size characteristic of a body of this configuration; g is acceleration due to gravity. It is convenient to determine the heat transfer coefficient from the criterion equation:

$$Nu=F(Gr,Pr). \tag{3}$$

For the average heat transfer during air cooling, the last equation has the form:

$$Nu=0.037Re^{0.8}Pr^{0.43}\left(\frac{Pr_1}{Pr_2}\right)^{0.25}, \tag{4}$$

where Pr_1, Pr_2 is Prandtl numbers, which are calculated at film and ambient temperatures.; Re is Reinold’s number which is proportional of relation forces inertia to forces friction. Transforming equation (4) gives the following expression:

$$\alpha=0.037\left(\frac{Vd}{\nu}\right)^{0.8}0.7^{0.43}\left(\frac{0.7}{0.72}\right)^{0.25}\frac{\lambda}{d}. \tag{5}$$

In [9], the value of the coefficient was experimentally obtained at the interface between the metal in the form of a thermocouple junction with a diameter of 50 microns and running water, which is 1 cal/cm s·K (Fig. 1, 2). However, the technical limitations of the experiments did not allow us to determine the dependence of the heat transfer coefficient on the smaller diameter of the CR and the change in the cooling fluid. Due to the technical complexity of the experiments on the determination α there is a need for its theoretical calculations.

For practical results of convective coefficients of infinite cylinders (CR case), the following formula can be used [6]:

$$\alpha_1 = A_1 \left(\frac{T - T_C}{d^5} \right)^{1/8}, \tag{6}$$

where d is the diameter of the CR; A_1 is coefficient that includes the physical parameters of the environment:

$$A_1 = 1.18 \left(\frac{\beta g \nu}{a} \right)^{1/8} \frac{\lambda_m}{\nu_m^{1/4}}. \tag{7}$$

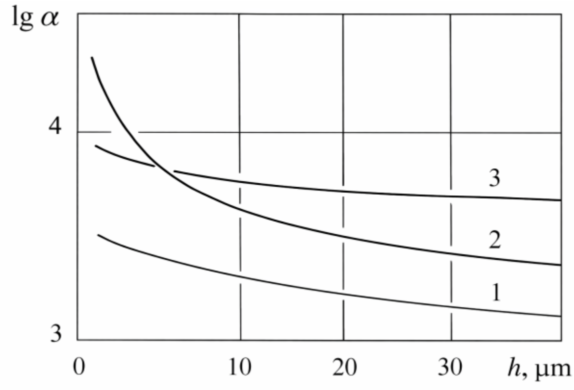

Fig. 1. Dependence of the coefficient α on the free surface from the thickness Fe—20 at.% C film and linear speed of rotation of the cylinder:1—70 m/s; 2—500 m/s; 3—1000 m/s

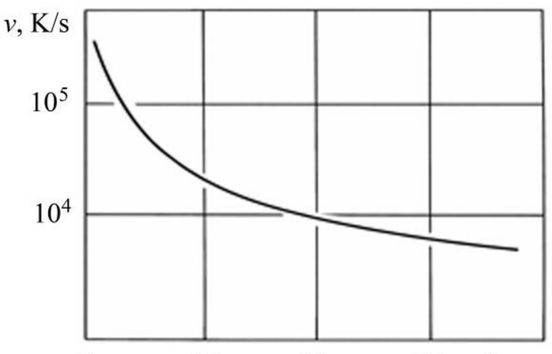

Fig. 2. Cooling rate of the free surface Fe—20 at.% C film (speed of rotation of the cylinder—70 m/s)

The work also calculated the heat transfer coefficients of CR during cooling in various cooling media. The following equation was used to describe the cooling process of CR in air [6]:

$$\alpha_1(d) = \lambda_1 \frac{0.26 \left(\omega \frac{d}{v^1} \right)^{0.6}}{d}, \tag{8}$$

where λ_1, v_1 is thermal conductivity and viscosity of air; ω, d is pulling speed and diameter of the CR.

The results of calculations of heat transfer coefficients during cooling of CR in water and air are shown in Fig. 3. Naturally, the coefficient when cooled with water is much larger, therefore the Nusselt criterion was calculated from the following equation:

$$Nu(d) = 0.25\left(\omega \frac{d}{\nu_2}\right)^{0.8} Pr_2^{0.43}\left(Pr_2 / Pr_3\right)^{0.25}, \tag{9}$$

where λ_2, ν_2 is thermal conductivity and viscosity of water; Pr_2, Pr_3 is Prandtl number of water at saturation temperature and at the former environment accordingly.

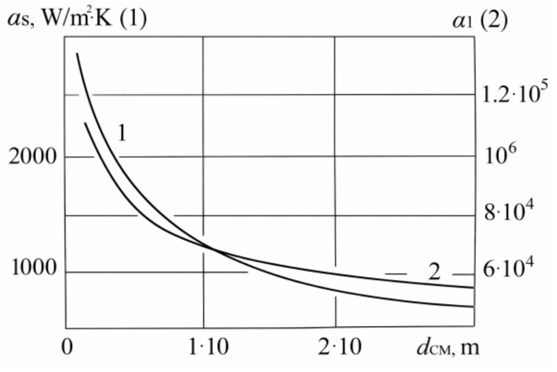

Fig. 3. Dependence of the heat coefficient recoil when cooling CR in air (1) and water (2)

For CRs of large radii, the coefficient varies slightly depending on the insulation thickness and corresponds to experimental values. However, with decreasing CR thickness, it increases sharply and for ultrathin samples reaches ~10 W/m K (Fig. 3, 4).

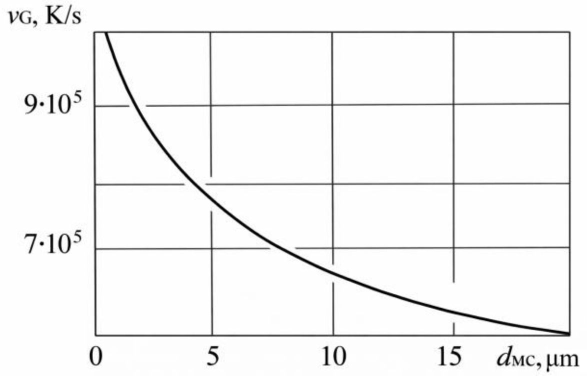

Fig. 4. Dependence of the CR. CR from the core diameter (water cooling)

The formation of crystals on a heat-conducting substrate (Fig. 5) has several structural zones: columnar (at the interface with the substrate) and a zone of equilibrium crystals. From the above microstructures it is clear that with the advancement of the former to the center of the film, second-order axes begin to form. Qualitative features of structure formation under conditions of large SQ can be explained by the analysis of crystallization kinetics and thermal cooling modes: first of all, the value of the thermal parameter Bio (*Bi*). Naturally, on the surface of the film at the interface with the heat-conducting substrate, the undercooling is greater than in its inner layers. Therefore, the conditions for crystal nucleation near the substrate are favorable due to the additional possibility of the seeding action of the substrate itself. Increasing SH eliminates diffusion (concentration) undercooling and the directions of the nuclei that arise in the depth of the liquid may no longer coincide with the direction of external heat removal (Fig. 6).

Fig. 5. Microstructure (×300) of steel, obtained from wedge-shaped castings

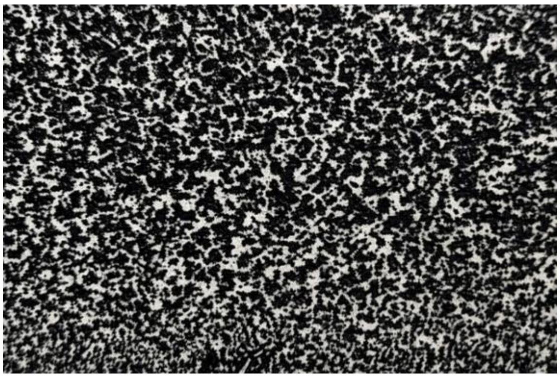

Fig. 6. Microstructure (×1000) of steel films obtained at CR (10^6 K/s)

In order to quantitatively explain the emerging change in structural morphology, the problem of one-dimensional cooling of a medium with a constant temperature on the contact and free surfaces of a film with a thickness of. It is taken into account that the cooling process through the contact and free surfaces is independent. The general solution of the Fourier differential equation:

$$\frac{\partial\theta}{\partial\tau}=a\frac{\partial^2\theta}{\partial x^2} \tag{10}$$

was found as the sum of two opposite processes. It is assumed that the boundary conditions are: a) x=0,
$\left(\frac{\partial\theta}{\partial x}\right)_{x=0}=0$; b) $x=\delta$, $\left(\frac{\partial\theta}{\partial x}\right)_{x=\delta}=-\frac{\alpha}{\lambda}\theta_{x=\delta.}$ Initial conditions: at loss temperature $\theta=\theta_0$. The general solution of the Fourier equation, taking into account the boundary conditions, has the well-known form [6]:

$$\theta=\sum_{n=1}^{n\to\infty}\frac{2\theta_0 sin\mu_n}{\mu_n+sin\mu_n\cos\mu_n}cos\left(\mu_n\frac{x}{\delta}\right)exp\left(-\frac{\mu_n^2 a\tau}{\delta^2}\right), \tag{11}$$

where $\mu_1,\mu_2,...\mu_n$ is solution of the equation ($ctg\mu_n=\frac{\mu_n}{Bi}$, $Bi=\frac{\alpha\delta}{\lambda}$); *a, λ* is thermal conductivity and thermal conductivity films respectively. Using dimensionless quantities: $\frac{x}{\delta}=X\,(0\le X\le 1)$, $\frac{\theta_n}{\theta_0}=\theta$, $F=\frac{a\tau}{\delta^2}$ (Fourier number), the temperature distribution across the cross section of films of different thicknesses for the contact and free surfaces was calculated (Fig. 7, 8), tabl. 1.

Table 1. Thermophysical parameters used in the calculations of the rapid cooling process of a liquid iron film

Film thickness, δ, 10^{-6} m	Heat transfer coefficient (contact surface), α_K, W/m²·K	Heat transfer coefficient (free surface), α_{CB}, W/m²·K	Thermal conductivity a, m²/s	Thermal conductivity, λ, W/m·K	Biot number, *Bi*	Initial temperature, *T*, K
10	10^5	10^4	5	30	0,033	1810
100	10^5	10^4	5	30	0,33	1810

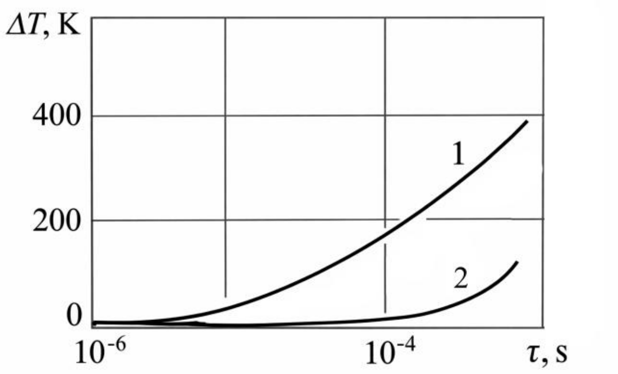

Fig. 7. Dependence of temperature distribution over the cross-section of the iron film (δ = 10 μm): 1, 2 — for contact and free surface accordingly

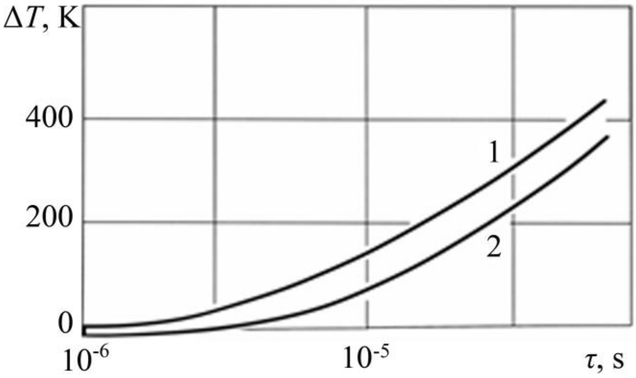

Fig. 8. Dependence of temperature distribution of Fe film (δ = 100 μm): 1, 2 — for contact and free surface accordingly

The dependences of this distribution for the contact and free surfaces of the iron film with the heat transfer coefficients, respectively, are given: 10^5 and 10^4 W/m²·K (*Bi* = 0,333) qualitatively reflect the observed changes in the structural morphology of the film: approximately at a third of the distance from the free surface, the supercooling value, which is the sum of two cooling processes (Fig. 9, 10), has the smallest value, the latter can be the cause of deviation from the direction of external heat removal. Experimental confirmation of structural changes in the morphology of films by the above calculations of thermophysical cooling processes gives reason to consider the proposed approach to determining the features of non-equilibrium cooling of films by the splat method to be quite correct.

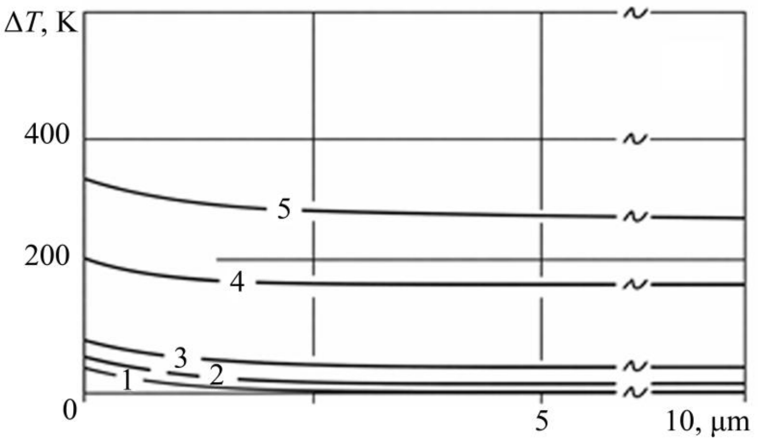

Fig. 9. Distribution of total undercooling in a Fe—film with a thickness 10 μm over time

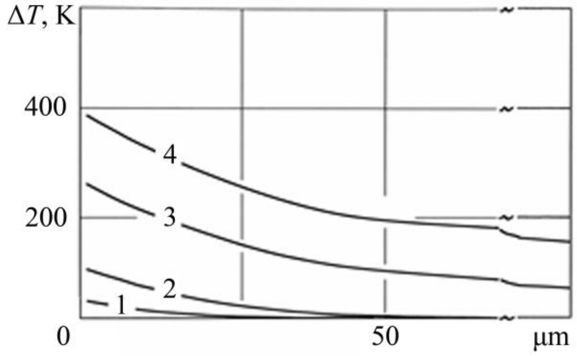

Fig. 10. Distribution of total undercooling in a Fe—film with a thickness 10^2 microns over time

Conclusions

Features and analysis of thermal cooling regimes in the splat quenching process showed that simultaneous non-equilibrium crystallization occurs on both (contact and free) sides of the film. The degrees of undercooling (350 and 250 K, respectively) and the cooling rates achieved in this case are sufficient for non-equilibrium formation of the film structure. The recorded features of non-equilibrium quenching from the melt in the splat cooling process can have a practical solution for creating interlayer amorphous-crystalline structures with the necessary set of improved physical properties of the SQ-films. A quantitative assessment of the maximum level of cooling rates in the process of casting a microwire in glass insulation is given. Although this level is significantly lower than that achieved in the process of splat-cooling, the possibility of more non-equilibrium formation of the substance in the case of CR is due to the influence of the amorphous substrate in the form of glass insulation. The amorphous state in thin CRs with a cast iron core Fe—20 at.% C confirms the implementation of an increased, compared to splat-hardening, level of non-equilibrium formation of the microwire in combination with the cooling rates specified in the work and the increased degree of supercooling of the liquid core of the CR.

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ОСОБЛИВОСТІ ТЕПЛОВИХ РЕЖИМІВ ОХОЛОДЖЕННЯ ПРИ SPLAT-ГАРТУВАННІ ТА ЛИТТІ МІКРОДРОТУ

Реферат

У статті досліджуються особливості теплових режимів охолодження розплавів під час splat-гартування та лиття мікродротів у скляній ізоляції при надзвичайно високих швидкостях охолодження, які досягають 10^8 К/с. Встановлено, що зазначені умови сприяють формуванню широкого спектра метастабільних структурних станів, зокрема мікро- та нанокристалічних, а також аморфних сплавів, які проявляють унікальні фізико-хімічні властивості. Однак, проблема прямого експериментального визначення швидкості охолодження у тонких плівках та мікродротах технічно ускладнена визначенням залежності тепловіддачі від низки таких параметрів, як товщина зразка, теплофізичні властивості як охолоджуючого середовища, так і зразка, а також режими гартування. Крім того, одночасна нерівноважна кристалізація на контактній і вільній поверхнях ускладнює точне передбачення формування морфології структури. У зв’язку з цим слід підкреслити практичну значущість застосування теоретичних і обчислювальних методів для оцінки коефіцієнтів тепловіддачі, швидкостей охолодження та особливостей зміни мікроструктури. На основі рівнянь теплопровідності та критеріїв подібності проведено кількісний аналіз тепловіддачі й швидкості охолодження плівок і мікродротів різного діаметра у повітрі та воді. Доведено, що зменшення товщини плівки або діаметра мікродроту призводить до помітного зростання коефіцієнта тепловіддачі і швидкості охолодження, що створює умови для утворення метастабільних структур. У процесі лиття скляної ізоляції, де швидкості охолодження дещо нижчі порівняно із ГРС, аморфна скляна оболонка сприяє підвищенню рівня нерівноважного формування структурного стану матеріалу з-за більшого переохолодження розплаву. Результати моделювання температурного поля та розподілу переохолодження у плівках різної товщини дали змогу пояснити структурні зміни морфології залежно від режимів тепловідведення. Встановлено, що на контактній поверхні плівки переохолодження є вищим, що створює сприятливі умови для зародження і росту кристалів. Запропонований метод оцінки коефіцієнтів тепловіддачі і швидкостей охолодження дає змогу прогнозувати характер морфологічних змін і фізико-хімічні властивості швидкоохолоджених матеріалів, що має важливе практичне застосування для отримання ГРС-покриттів і мікродротів із заданими експлуатаційними характеристиками.

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Надійшла до редколегії 19.02.2026
Прийнята після рецензування 20.03.2026
Опублікована 26.03.2026