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Metallurgy

Digitalization of the thermoplastic beryllium oxide slurry forming process using ultrasonic activation

[1] Zhapbasbayev U.K., [1*]Ramazanova G.I., [2] Retnawati H., [3] Sattinova Z.K.

[1] *Satbayev University, Almaty, Kazakhstan*
[2] *Yogyakarta State University, Indonesia*
[3] *L.N. Gumilev Eurasian National University, Astana, Kazakhstan*

** Corresponding author email: g.ramazanova@satbayev.university*

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ABSTRACT
This paper presents the results of the digitalization of the thermoplastic beryllium oxide slurry forming process using ultrasonic activation. Ceramics made from beryllium oxide (BeO) using ultrasound-assisted forming exhibit more intense sintering and, in comparison to ceramics formed without ultrasound, have reduced shrinkage (by 2.4-4.3%) and sintering temperature (by 50-180°C). The forming processes occurring during ultrasonic treatment resulted in the homogenization of the thermoplastic suspension and dense packing of BeO powders in the casting. Ultrasound activation alters the rheology of the thermoplastic slurries. These changes are attributed to processes of slurry mass dispersion and mass exchange at the phase boundary of the suspension. Ultrasound activation also enhances casting properties. During the cooling-solidification process under the influence of ultrasound, the density and strength of the castings increase due to the effective compensation of shrinkage. Shrinkage compensation is carried out according to the classical scheme by supplying a liquid suspension. For hot casting with ultrasound of thermoplastic beryllium oxide slurries, it is advisable to use compositions with a binder content of 11.0-11.7% by weight since these compositions provide better shrinkage compensation and, consequently, a denser casting.

Keywords: forming process, thermoplastic slurry, beryllium oxide, viscoplastic state, casting solidification.

Information about authors:

Zhapbasbayev Uzak Kairbeekovic	*Doctor of Technical Sciences, Professor, Head of the Research and Production Laboratory "Modeling in Energy", Satbayev University, 22 Satpaev Street, 050013 Almaty, Kazakhstan. Email: uzak.zh@mail.ru*
Ramazanova Gaukhar Izbasarovna	*Candidate of physical and mathematical sciences, Leading Researcher. Research and Production Laboratory "Modeling in Energy", Satbayev University, 22 Satbayev str., 050013 Almaty, Kazakhstan. E-mail: g.ramazanova@satbayev.university*
Retnawati Heri	*Doctor, Professor, Universitas Negeri Yogyakarta (Yogyakarta State University), 55281, Jl. Colombo No.1, Yogyakarta, Indonesia. E-mail: heri_retnawati@uny.ac.id*
Sattinova Zamira Kanaevna	*Candidate of physical and mathematical sciences, Associated Professor, L.N. Gumilev Eurasian National University, 2 Satpayev str., 010008 Astana, Kazakhstan. E-mail: sattinova.kz@gmail.com*

Introduction

High thermal conductivity beryllium oxide slurries within the technological temperature and solid phase concentration range exhibit thixotropic properties and a high dependence on viscosity and yield stress on temperature [[1], [2], [3], [4], [5]]. The nature of the changes in structural-mechanical properties is determined by the ratios of the solid phase (BeO) and binders and occurs in all stages of the forming process [[1], [5]].

The analysis of volume-phase relationships revealed that the density increase during the cooling process of the liquid state for casting systems amounts to 5-6% [1,6]. The interval of the solid-plastic state accounts for 70-80% of the temperature shrinkage. This highlights the need for special attention to shrinkage compensation at this stage. Varying the ratio of solid phase and binders does not provide an approximation of the rheological properties of beryllium oxide - thermoplastic binder to traditionally hot molding technologies [[1], [5]].

Ultrasound activation leads to a significant change in the rheology of thermoplastic casting systems [[1], [3], [4], [5]]. Varying the intensity and temperature-time parameters of ultrasound treatment enhances the effectiveness of its impact on the rheology of thermoplastic slurries. The mechanism of such influence is related to the control of the solid-liquid phase interface and the parameters of absorption layers [[1], [3], [4], [5]]. The most favorable conditions in terms of deformational behavior of casting systems are achieved in the temperature range of 63-68°C and an ultrasound treatment time of 7-10 minutes [[1], [3], [4], [5]].

The change in the properties of the slurry mass under the influence of ultrasound affects the microstructure and properties of ceramics. The samples obtained with ultrasound have a more homogeneous structure and higher structural-mechanical and electrophysical parameters [[1], [3], [4], [5]].

The method of hot casting of beryllium oxide ceramics has been developed mainly empirically [[1], [6], [8]]. Conducting a detailed analysis of this method will allow a more reasonable approach to the ceramic molding process. This work presents some results of modeling the process of beryllium oxide ceramic forming using the hot casting method.

Problem Statement

The forming process of a thermoplastic slurry takes place in a die (Fig. 1). The flat cavity has a thickness of 2h = 0.0015 m, a width of B = 0.03 m, and a length of L = 0.071 m. The cooling circuit of the die, illustrated in Figure 1, comprises three segments. The cooling contour of the die, illustrated in Figure 1, comprises three segments. Liquid slurry with an initial temperature of t_0 = 80°C flows into the cavity.

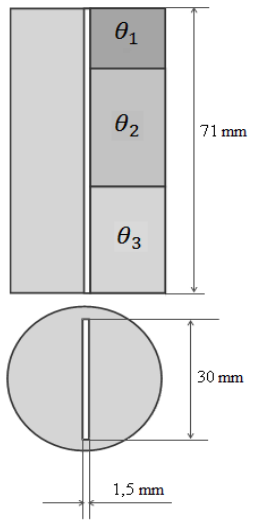

Figure 1 - Diagram of a flat cavity die

Rheological Model of the Thermoplastic Slurry

The composition of the thermoplastic slurry comprises a solid phase (beryllium oxide powder) and a liquid phase (organic binder) [[1], [3], [4], [5]]. The liquid phase is composed of paraffin, beeswax, and oleic acid in a ratio of 0.82:0.15:0.03. The particle size distribution of the beryllium oxide powder is provided in Table 1. The organic binder has a mass fraction ω within the range of 0.095 to 0.117.

Table 1 - Characteristics of Beryllium Oxide Powder [1]

Bulk Density, $\rho_0 \cdot 10^3$ kg/m³		**0.75**
Specific surface area, S·10⁻³ m²/kg		**1.72**
Particle size distribution by fractions of BeO particles	Particle Size Fraction, µm	%
	Up to 1.4	35.2
	1.4-4.2	52.7
	4.2-7.0	9.6
	7.0-9.8	1.7
	9.3-12.6	0.4
	12.6-15.4	0.3
	15.4-18.2	0.1

This composition of BeO powder (see Table 1) demonstrates satisfactory casting properties of the slurry with a variation in the mass fraction of the binder from ω = 0.095 to ω = 0.117. In the case of increasing the finer fractions of BeO powder in the composition, there is a higher demand for binder content ω. On the other hand, an increase in the coarser fractions of BeO powder leads to the discoloration of ceramics, indicating the presence of micro-pores and cracks [1].

Within the range of shear rate variation from 0.005 to 1200 1/s, the thermoplastic slurry can be classified as viscoplastic liquids according to Schwedoff-Bingham [[1], [5]]. The effective molecular viscosity μ_{eff} of this fluid takes the following form [[7], [8], [9], [10], [11], [12]]:

$$\mu_{\text{eff}} = \begin{cases} \mu_p + \tau_0 |\dot{\gamma}|^{-1}, & \text{if } |\tau| > \tau_0 \\ \infty, & \text{if } |\tau| \le \tau_0 \end{cases} \quad (1)$$

Here τ_0 is the yield shear stress, μ_P is the plastic viscosity, $\boldsymbol{\tau} = \mu_{\text{eff}} \boldsymbol{S}$ is the shear stress tensor, $\boldsymbol{S} \equiv \sqrt{2 S_{ij} S_{ij}}$ is the strain rate tensor, $S_{ij} = \frac{1}{2}\left(\frac{\partial U_i}{\partial x_j} + \frac{\partial U_j}{\partial x_i}\right)$ is the second invariant of the strain rate tensor. The Schwedoff-Bingham model is a simple viscoplastic fluid model that linearly relates the yield shear stress to the viscosity [[7], [8], [9], [10], [11], [12]].

Ultrasonic treatment influences the rheological properties of the slurry. The plastic viscosity (μ_p) and the yield stress (τ_0) of the slurry are dependent on the temperature (t) and the binder mass fraction (ω). Experimental data for the binder mass fraction ω = 0.117 after ultrasonic treatment are expressed by empirical relationships [[1], [3], [4], [5]]:

$$\mu_p(t) = 293.626 \cdot \exp(-0.058 \cdot t), \text{ Pa·s} \quad (2)$$

$$\tau_0(t) = 11.4 + 11.41 \cdot \exp\left(-\frac{t-70.05}{5.47}\right), \text{ Pa} \quad (3)$$

The density of the thermoplastic slurry can be expressed in standard form as:

$$\rho = \frac{\rho_{mb} \cdot \rho_{cb}}{(1-\omega)\rho_{cb} + \omega \cdot \rho_{mb}} \text{ kg/m}^3 \quad (4)$$

where ρ_{mb} and ρ_{cb} are the densities of beryllium oxide and binder, respectively; ω is the relative mass fraction of the binder.

The temperature dependence of the binder density at ω = 0.117 is as follows:

$$\rho_{cb}(t) = 0.852 + 0.073 \cdot \cos(0.056 \cdot t - 1.44), \text{ kg/m}^3 \quad (5)$$

The density of beryllium oxide is ρ_{mb}=3020 kg/m³. In the temperature range from 80 to 40 °C, the density of the binder increases from 779.7 to 901.0 kg/m³, and the density of the slurry also increases from 2245.7 to 2355.3 kg/m³.

During the molding process, the slurry mass cools and solidifies within the mold cavity. The change in the aggregate state initiates in the near-wall area of the cavity and gradually encompasses the entire cavity.

According to experimental data, the phase transition occurs within a temperature range of 54 to 40°C. The binder remains in an amorphous state. Within the range of aggregate state change, the slurry transitions from an amorphous viscous-plastic state to an amorphous solid-plastic state [[1], [3]]. The heat of phase transition, released per unit mass of the slurry, is determined by the enthalpy change ΔH.

The apparent heat capacity method [[13], [14], [15], [16], [17],[18], [19], [20], [21]] determines changes in the heat capacity and enthalpy of the slurry during the phase transition. The heat capacity of the slurry increases due to the latent heat associated with the phase transition within the temperature range [[14], [15], [21]]:

$$c_p = c_s \cdot \left(1 - \alpha(\bar{t})\right) + c_l \cdot \alpha(\bar{t}) + H_{1\to 2} \frac{d\alpha}{dt} \quad (6)$$

where c_s and c_l are the heat capacities of the slurry in solid and liquid states, respectively; $\alpha(\bar{t}) = 0$, $\alpha(\bar{t}) = 1$ for the slurry in solid and liquid states, respectively; $\bar{t}$ is the dimensionless temperature of the slurry. For the binder mass fraction ω=0.117 the function $\alpha(\bar{t})$ takes the following form:

$$\alpha(\bar{t}) = 5.714 \cdot \bar{t} - 2.857$$

The apparent heat capacity method is advantageous in that the phase transition zone's location is not known beforehand and is determined by the calculation of the thermoplastic slurry's temperature [[19], [20], [21]].

The dependency of the thermal conductivity of the slurry on temperature for ω = 0.117 is given by [[1], [5]]:

$$\lambda = 1.6 + 4.8 \cdot \exp(-0.017 \cdot t), \text{ W/(m·}^0\text{C)} \quad (7)$$

Formulas (2)-(7) express the properties of the thermoplastic slurry during the hot casting process.

Mathematical Model

The OZ axis of the Cartesian coordinate system is directed along the axis of the flat mold cavity, while the OY axis is perpendicular to it (Fig. 1). The casting process occurs along the OZ axis. Intensive water circulation takes place in the cooling contours. The temperature of the slurry at the wall of each part of the cavity is considered equal to the temperature of the cooling liquid.

The molding process occurs with a change in the aggregate state and rheological properties of the slurry.

The system of equations for the motion of the thermoplastic slurry using the Schwedoff-Bingham rheological model (1) can be written in the following form:

$$\rho u \frac{\partial u}{\partial z} + \rho v \frac{\partial u}{\partial y} = -\frac{dp}{dz} + \frac{\partial}{\partial y}\left(\mu_{eff} \frac{\partial u}{\partial y}\right) + \rho g \quad (8)$$

$$\frac{\partial \rho u}{\partial z} \quad \frac{\partial \rho v}{} = 0 \quad (9)$$

The energy equation, considering the enthalpy of the phase transition by the apparent heat capacity method, can be expressed as follows:

$$\rho u c_p \frac{\partial t}{\partial z} + \rho v c \frac{\partial t}{\partial y} = \frac{\partial}{\partial y}\left(\lambda \frac{\partial t}{\partial y}\right) + \mu\left(\frac{\partial u}{\partial y}\right)^2 \qquad (10)$$

Here, *z* and *y* are the longitudinal and transverse coordinates, *u* and *v* are the components of the velocity, $p, \rho, t, c_p, \mu_p, \lambda$ are the pressure, density, temperature, heat capacity, viscosity, and thermal conductivity of the slurry, respectively.

The system of equations (8)-(10) describes the slurry molding process in a flat mold cavity. The rheological properties of the slurry are expressed by formulas (1)-(7).

The pressure gradient in the motion equation is determined from the conservation of mass flow rate within the mold cavity [22]:

$$\int_S \rho u dS = \rho_o u_o S \qquad (11)$$

where S_0 is the cross-sectional area of the cavity.

The problem is solved with boundary conditions as follows:

at z = 0, at the inlet: $u = u_0,\ v = 0,\ t = \theta$ (12)

at z > 0, on the axis: $\frac{\partial u}{\partial y} = — = v = 0$ (13)

at z>0, y=y$_1$, on the wall: $v = 0,\ \frac{\partial u}{\partial y}\ \ 0$ (14)

The water temperature in the first, second, and third cooling zones is denoted as θ_1, θ_2, θ_3, respectively. The boundary conditions for temperature on the wall are as follows:

at $0 \le z < l_1$, $y = y_1$, $t = \theta_1$;
at $l_1 \le z < l_2$, $y = y_1$, $t = \theta_2$; (15)
at $l_2 \le z < l_3$, $y = y_1$, $t = \theta_3$

The numerical method [22] is used to solve the system of equations (1)–(11) with boundary conditions (12)–(15). The discretization grid consists of cells with sides Δz_i, and Δy_j. The second-order accuracy Crank-Nicholson scheme is applied to solve the momentum equation (8) and the energy equation (10). A two-layer second-order scheme is used to solve the equation (9). The method of splitting is used to determine the pressure gradient from the mass flow conservation condition (11).

Discussion of Computational Data

Fig. 2 illustrates the temperature and density distributions within three thermal contours of a flat mold cavity with a thickness of $2h$ = 0.0015 m, a casting speed u_0=0.05 m/min, and an initial slurry temperature of 80 °C. In the first cooling contour, the wall temperature is θ_1 = 80 °C. The temperature field demonstrates a decrease from 80 to 78 °C (Fig. 2a), and the density increases from 2245 kg/m³ to 2260 kg/m³ (Fig. 2b).

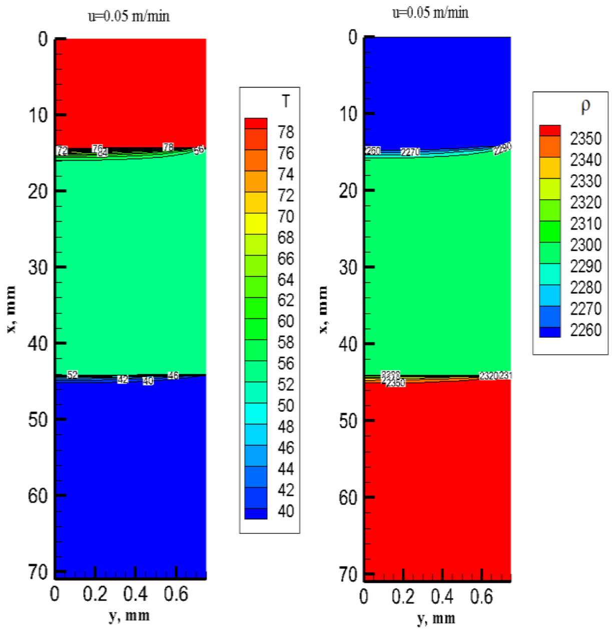

Figure 2 – Temperature and density distribution of the thermoplastic slurry in the flat mold cavity at u_0 = 0.05 m/min, ω=0.117

In the second cooling contour, the wall temperature is θ_2=56 °C. With decreasing temperature, the dynamic viscosity, $\mu_p(t)$, density $\rho(t)$, and yield stress, $\tau_0(t)$ all experience an increase. The temperature of the slurry drops from 78 to 52 °C (Fig. 2a), leading to an elevation in the slurry density from 2250 to 2300 kg/m³ (Fig. 2b).

At the beginning of the second cooling contour, there exists a transitional region where the temperature sharply drops from 78 to 56 °C, while the density increases from 2250 to 2290 kg/m³. Subsequently, the rate of temperature reduction slows down from 56 to 52 °C due to the heat release from the phase transition (Fig. 2a). An observable density increase of the thermoplastic slurry can be noted, from 2290 to 2300 kg/m³ (Fig. 2b).

In the third cooling contour, the wall temperature reaches θ_3=40 ^{0}C, leading to further cooling of the slurry mass and a decrease in temperature within the region transitioning from 52 to 40 °C (Fig. 2a). This causes a rise in density from 2290 to 2350 kg/m³ (Fig. 2b).

In this area, the transition from a viscous-plastic state to a solid-plastic state occurs, resulting in the solidification of the thermoplastic slurry (Fig. 2).

Doubling the casting speed to 0.1 m/min under otherwise identical conditions has a negligible impact on the structural transformation of the slurry (Fig. 3). This is attributed to the rapid cooling rate within the flat mold cavity.

Experimental data [[1], [5]] indicate that an increase in the transition region between different states can lead to slurry shrinkage and the formation of cracks and voids, subsequently reducing the casting's strength. The smaller the transition area between aggregate states, the less the slurry will shrink. Ultrasonic treatment, by improving rheological properties, leads to a reduction in the transition area and consequently decreases the shrinkage of the beryllium oxide casting.

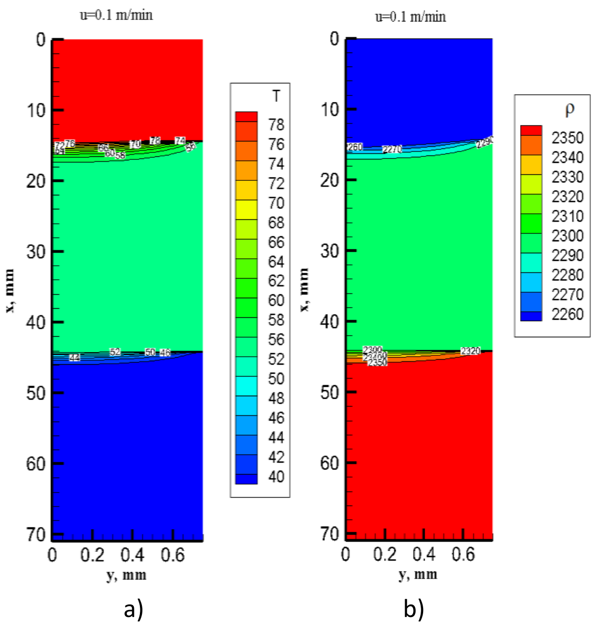

Figure 3 - Temperature and density distribution of the thermoplastic slurry in the flat mold cavity at u_0 = 0.1 m/min, ω=0.117

Comparison of Calculation with Experiment

Table 2 shows the experimental data of casting ability and mechanical strength of the casting as a function of binder content and casting speed [1].

The calculated data (Fig. 4) were obtained at two experimental casting speeds in the flat mold cavity: u_0=0.185 m/min and u_0=0.165 m/min for the binder mass fraction ω=0.117.

Table 2 - Dependency of Ceramic Strength on Casting Speed in the Flat Mold Cavity

Binder content in the slurry, mass fraction	The viscosity of slurry at T_0=75 °C, Pa·s	Casting ability of slurry, mm	Casting speed, mm/min	Mechanical strength of casting in bending, MPa
0.117	4.17	89	165	8.17

In the first cooling contour, the cooling water temperature is θ_1=75 °C, in the second it's θ_2=59 °C, and in the third, it's θ_3=45 °C. The liquid slurry flows into the flat mold cavity at an initial temperature of t_0=75 °C (Fig. 1).

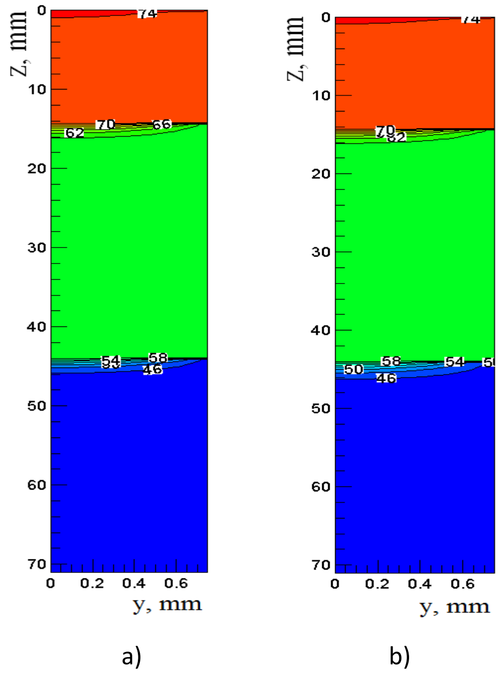

Figure 4 - Temperature field distributions depending on casting speed: a) u_0= 0.185 m/min; b) u_0= 0.165 m/min

As can be observed from Fig. 4, the transition regions between different cooling contours occupy minor intervals. The casting speed ensures a nearly uniform temperature distribution across the cavity's cross-section. Such temperature distribution leads to the homogeneity of rheological and thermophysical properties of the slurry over the cavity cross-section. In this case, the shrinkage of the thermoplastic slurry will be uniform. Consequently, cracks and voids, which could lead to a reduction in the strength of the beryllium oxide casting, do not form.

The slurry mass solidifies within the mold cavity.

This indicates that the beryllium oxide ceramic product has taken a structural shape.

Conclusion

Ultrasonic treatment improves the rheological properties and increases the flowability of thermoplastic beryllium oxide slurry in the molding cavity. Empirical formulas for plastic viscosity and shear stress of the slurry as a function of temperature were obtained. The phase transition of thermoplastic beryllium oxide slurry occurs within a certain temperature range. The latent heat is accounted for by an increase in heat capacity within the temperature range of the phase transition.

Calculation results show the entire molding stage of thermoplastic beryllium oxide slurry. Comparative calculations with experimental data established the conditions for molding thermoplastic slurry with ultrasonic activation, allowing to obtain solidified castings of beryllium oxide. The speed and temperature conditions of the molding slurry are important for determining the internal shrinkage of the casting during the cooling-hardening process of beryllium oxide and are a topic for future research.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

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Ультрадыбысты қолдану арқылы бериллий оксидінің термопластикалық шликерін қалыптау процесін цифрландыру

[1]Жапбасбаев Ұ.Қ., [1*]Рамазанова Г.І., [2] Retnawati H., [3]Саттинова З.Қ.

[1] Сәтбаев университеті, Алматы, Қазақстан
[2]Йогьякарта мемлекеттік университеті, Индонезия
[3] Л.Н. Гумилев атындағы Еуразия ұлттық университеті, Астана, Қазақстан

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ТҮЙІНДЕМЕ
Жұмыста ультрадыбысты қолдану арқылы бериллий оксидінің термопластикалық шликерін қалыптау процесін цифрландыру нәтижелері берілген. Ультрадыбысты қолдану арқылы алынған бериллий оксиді BeO керамикасы қарқынды күйежентектелумен сипатталады және ультрадыбыссыз қалыпталған керамикамен салыстырғанда шөгуі (2,4÷4,3%) және күйежентектелу температурасы (50÷180 °C) төмен. Ультрадыбыстық өңдеу кезіндегі қалыптау процестері термопластикалық шликердің гомогенизациялануына және құймадағы BeO ұнтақтарының тығыз орналасуына әкеледі. Ультрадыбыстық белсендіру термопластикалық шликердің реологиясын өзгертеді. Бұл өзгерістер шликер массасының ұсақталу процестеріне, сондай-ақ суспензияның фазалар бөліну шекарасындағы масса алмасуына байланысты. Ультрадыбыстық белсендіру құймалардың қасиеттерін де жақсартады. Ультрадыбыс әсерінен салқындау–қату процесі кезінде шөгуді тиімді компенсациялау есебінен құймалардың тығыздығы мен беріктігі артады. Шөгудің өтемі (компенсациясы) сұйық суспензияны жңберу арқылы классикалық схемаға сәйкес жүзеге асырылады. Бериллий оксидінің термопластикалық шликерін ультрадыбыс арқылы ыстық құю үшін байланыстырғыш салмағы бойынша 11,0-11,7% құрамда болған жөн. Өйткені бұл құрам шөгудің жақсы компенсациясы болуға және тиісінше тығызырақ құйма алуға қол жеткізеді.

Түйін сөздер: қалыптау процесі, термопластикалық шликер, бериллий тотығы, тұтқыр-пластикалық күй, құйманың қатаюы.

Авторлар туралы ақпарат:

Жапбасбаев Ұзақ Қайырбекұлы

Техника ғылымдарының докторы, профессор, "Энергетикадағы модельдеу" ғылыми-өндірістік зертханасының меңгерушісі, Сәтбаев университеті, Сәтбаев көшесі, 22 үй, 050013, Алматы, Қазақстан. Email: uzak.zh@mail.ru

Рамазанова Гауһар Ізбасарқызы	*Физика-математика ғылымдарының кандидаты, жетекші ғылыми қызметкер, «Энергетикадағы модельдеу» ғылыми-өндірістік зертханасы, Сәтбаев университеті, Сәтбаев көшесі, 22 үй, 050013, Алматы, Қазақстан. Email: g.ramazanova@satbayev.university*
Retnawati Heri	*Доктор, профессор, Йогьякарта мемлекеттік университеті, 55281, Дж. Коломбо №1, Йогьякарта, Индонезия. E-mail: heri_retnawati@uny.ac.id*
Саттинова Замира Қанайқызы	*Физика-математика ғылымдарының кандидаты, қауымдастырылған профессор, Л.Н. Гумилев атындағы Еуразия ұлттық университеті, Сәтбаев көшесі, 2 үй, 010008, Астана, Қазақстан. Email: sattinova.kz@gmail.com*

Цифровизация процесса формования термопластического шликера оксида бериллия с ультразвуковой активизацией

[1] Жапбасбаев У.К., [1*] Рамазанова Г.И., [2] Retnawati H., [3] Саттинова З.К.

[1] Satbayev University, Алматы, Казахстан
[2]Государственный университет Джокьякарты, Индонезия
[3] Евразийский национальный университет имени Л.Н. Гумилева, Астана, Казахстан

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АННОТАЦИЯ

В работе представлены результаты цифровизации процесса формования термопластичного шликера оксида бериллия с использованием ультразвуковой активации. Керамика из оксида бериллия BeO, формованная с применением ультразвука, отличается более интенсивным спеканием и имеет по сравнению с керамикой, формованной без ультразвука, меньшую усадку (на 2,4÷4,3%) и температуру спекания (на 50÷180 °C). Процессы формования, протекающие при ультразвуковой обработке, приводят к гомогенизации термопластичного шликера и плотной упаковке порошков BeO в отливке. Ультразвуковая активация изменяет реологию термопластичных шликеров. Эти изменения обусловлены процессами диспергации шликерной массы, а также массообменном на границе раздела фаз суспензии. Ультразвуковая активация улучшает также свойства отливок. В процессе охлаждения-затвердевания под действием ультразвука происходит увеличение плотности и прочности отливок за счет эффективной компенсацией усадки. Компенсация усадки идет по классической схеме за счет подпитки жидким шликером. Для горячего литья с ультразвуком термопластичного шликера оксида бериллия целесообразно использовать составы с содержанием связки 11,0-11,7% вес. Поскольку на этих составах достигается лучшая компенсация усадки и соответственно более плотная отливка.

Ключевые слова: процесс формования, термопластичный шликер, оксид бериллия, вязкопластичное состояние, затвердевание отливки.

	Информация об авторах:
Жапбасбаев Узак Каирбекович	*Доктор технических наук, профессор, заведующий научно-производственной лабораторией "Моделирование в энергетике", Satbayev University, ул. Сатпаева 22, 050000, Алматы, Казахстан. Email: uzak.zh@mail.ru*
Рамазанова Гаухар Избасаровна	*Кандидат физико-математических наук, ведущий научный сотрудник. Научно-производственная лаборатория "Моделирование в энергетике", Satbayev University, ул. Сатбаева, 22, 050013 Алматы, Казахстан. E-mail: g.ramazanova@satbayev.university*
Retnawati Heri	*Доктор, профессор, Государственный университет Джокьякарты, 55281, Jl. Коломбо №1, Джокьякарта, Индонезия. E-mail: heri_retnawati@uny.ac.id*
Саттинова Замира Канаевна	*Кандидат физ.-мат. наук, Ассоциированный профессор, Евразийский национальный университет имени Л.Н. Гумилева, ул. Сатпаева, 2, 010008 Астана, Казахстан. E-mail: sattinova.kz@gmail.com*

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