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Modeling of ceramic products molding process

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Results of experiments and calculations of the motion and heat exchange of the BeO slurry in the annular cavity are presented. The slurry is a highly concentrated structured system where the mineral phase is BeO powder and liquid phase is an organic binder. The obtained temperature field determines the transition of the slurry from liquid (viscous-plastic) to solid-plastic state. Calculated data of the isotherms of solidification zone of the beryllia thermoplastic slurry are in a good agreement with the experimental results.

Keywords*:* thermoplastic slurry, molding, solidification

INTRODUCTION

The development of new areas of science and directions of technology increases requirements for advanced properties and quality of ceramic fabrications. Products of complex configuration from new non-metallic materials (high thermal conductivity, oxygen-free, superconducting, etc.) are becoming more and more popular. In spite of using isostatic pressing technology of hot casting under pressure [1, 2] remains the basis for the obtaining long-length, multi-channel, complex shaped ceramic fabrications from non-plastic powders.

Nowadays technology of slurry molding (extrusion) is very relevant in connection with intensive development of Metal Injection Moulding (MIM) technology [3, 4], where same physical processes take place.

While a lot of attention has been paid to improving of the technology and the creation of the new equipment last years, up to now there remains an unsolved problem of obtaining fault free products by this method. As a result, in practice it does not often achieve the desired quality of moldings and obtaining of acceptable products, which makes this process less profitable. Obtaining of ceramic fabrications by hot molding from dispersion materials with anomalous physical properties, such as BeO is particularly complicated. In this case, the difficulties of obtaining the products of high quality are caused firstly by thermal properties of beryllium oxide, in particular, its unique thermal conductivity [5, 6]. Clearly, it is impossible to eliminate technological limitations and problems without the development based on all experience and knowledge of theoretical representations about regularities and mechanisms of regulation of the thermal regime of the casting on the forming process of molding.

The results of experimental research and the generalization of them by calculations of mathematical model of the thermoplastic slurry of beryllium oxide molding process are presented in this paper.

EXPERIMENTAL

Experimental research of the effect of casting regimes on the temperature field in the zone of solidification of the molding was made on the experimental bushing (Fig.1), by measuring the temperature using a thermocouple installed on the different levels by the height of crystallizer. Experimental bushing is structurally closed to the production plant and it is designed for casting of circular tube with the outer diameter 0.02 m and the inner diameter 0.012 m. Material of mandrel and crystallizer is steel of grade X18H10T. The total height of the cylindrical part of the annular cavity is H = 0.028 m, the height of the hot zone of the annular cavity is h_1=0.008 m, the height of the cold zone of the annular cavity is h_2=0.02 m. Water with the temperature t_1=80°C fed to the upper (hot) contour of the crystallizer. Water with the temperature t_2=15-20°C fed to the bottom (cold) contour. The maximum through put of the crystallizer contours in volume of water is 1500 l/hour.

Conical input of the bushing is connected with the working tank of casting installation where beryllia slurry is kept. BeO slurry is a high concentrated structured medium. Slurry flows from

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the tank to the conical inlet of the annular cavity with an initial temperature t_0=80°C. During its flow in result of heat exchange with the walls of mandrel and bushing slurry changes its aggregate state and solidifies.

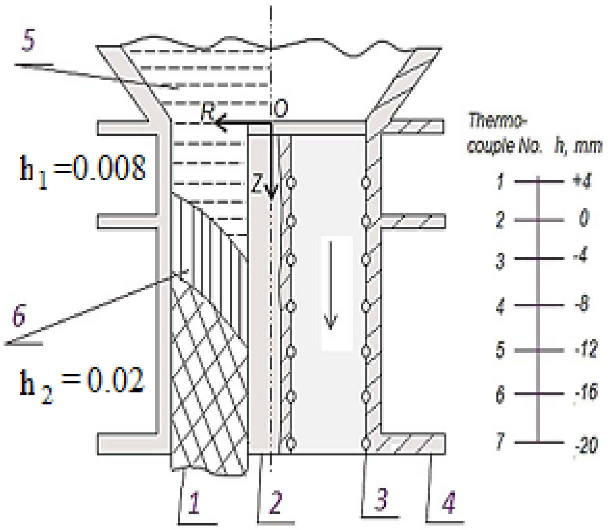

Fig.1. Scheme of molding solidification process and installation of thermocouple in the bushing: 1 – molding, 2 – mandrel, 3 – thermocouple, 4 – bushing, 5 – liquid slurry, 6 – zone of solidification

According to the obtained data, dependence of position of the boundary of solidification slurry from different parameters of casting was built. The shape of the curve of surface solidification, which is dependent on the casting parameter, is defined taking into account that the temperature changes linearly by the height and radius of crystallizer on the short segments.

The influence of molding velocity on the thermal regime of the casting was determined in the first series of experiments. The flow rate and temperature values of hot and cold water in the cooling contours are presented in Table 1. In the experiments molding velocity increases from 20 to 100 mm/min. Fig.2 shows positions of solidification zones depending on the molding velocity. Isotherm of the AB "solidus" corresponds to the temperature 54°C and isotherm of the CD "solidus" to 40°C (Fig.2).

As we see in Fig.2, the increase of the molding velocity leads to expansion of zone of solidification and its movement to the area of heat extraction of cold contour. It explains that with increasing of molding speed heat extraction on the walls of the annular cavity does not have time to cool the slurry, and zone of solidification extends, and it moves down towards the molding velocity.

Table 1. Regimes of experiments as a function of the molding velocity

Refer to fig.2	1	2	3	4	5
Hot water flow rate, l/hour	500	500	500	500	500
Cold water flow rate, l/hour	1500	1500	1500	1500	1500
Molding velocity, mm/min	20	40	60	80	100
Hot water temperature, °C	80	80	80	80	80
Cold water temperature °C	20	20	20	20	20

As we see in Fig.2, the increase of the molding velocity leads to expansion of zone of solidification and its movement to the area of heat extraction of cold contour. It explains that with increasing of molding speed heat extraction on the walls of the annular cavity does not have time to cool the slurry, and zone of solidification extends, and it moves down towards the molding velocity.

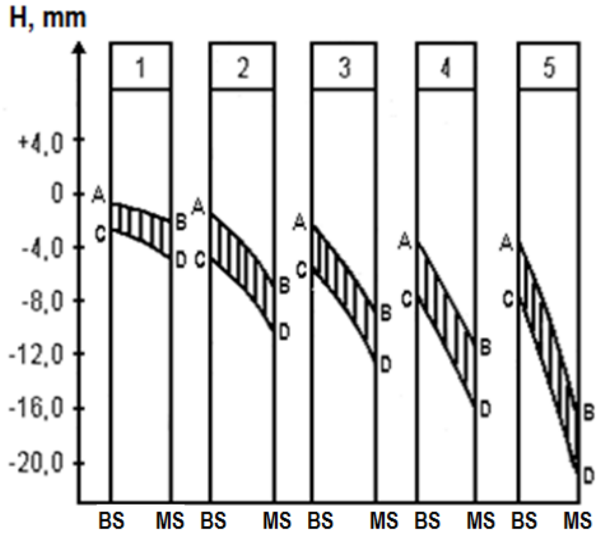

Fig.2. The position of solidification zone depending on the molding velocity: AB – isotherm "solidus" (54°C), CD – isotherm "solidus" (40°C), BS – the surface of the bushing, MS – the surface of the mandrel

In the second series of experiments the influence of cold water temperature and flow rate on the thermal regime of molding solidification was investigated (Table 2). In the experiments the molding velocity is 20 mm/min. Flow rate and temperature of hot water do not change, 500 l/hour and 80°C. Flow rate of cold water changes from 250 to 1500 l/hour, and the temperature from 15 to 20°C.

The results of the second series of experiments are shown in Fig.3. In the first three regimes cold water temperature is 15°C, and the flow rates are

1000, 500 and 250 l/hour, respectively. Solidification zones of molding are located in the area of cold contour, and move downstream with reducing cold water flow rate. Reducing of cold water flow rate leads to reduction of the heat extraction on the wall of bushing (Fig.3). Therefore, solidification zone of molding is moving down the length of the cold contour.

Table 2. Regimes of experiments as a function of the cold water flow rate and temperature

Refer to fig.3	**1**	**2**	**3**	**4**	**5**
Hot water flow rate, l/hour	500	500	500	500	500
Cold water flow rate, l/hour	1000	500	250	1500	250
Molding velocity, mm/min	20	20	20	20	20
Hot water temperature, °C	80	80	80	80	80
Cold water temperature °C	15	15	15	20	20

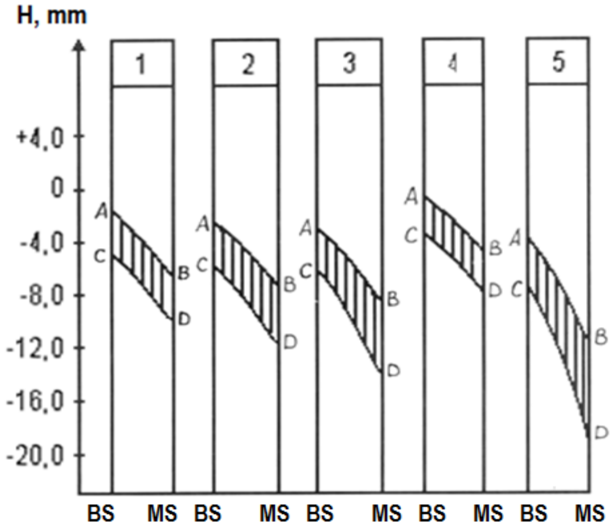

Fig.3. The position of solidification zone depending on the flow rate and the temperature of cold water

In the last two experiments of this series cold water temperature was 20°C, and its flow rate was changing from 1500 to 250 l/hour, respectively. In the fourth experiment, the beginning of solidification zone is located nearer to the hot contour area. Increase of cold water temperature from 15 to 20°C leads to reduction of temperature difference of the hot slurry and cold water. In the conditions of the fifth experiment except increase of cold water temperature takes place reduction of its flow rate from 1500 to 250 l/hour. Therefore, heat extraction reduces and solidification zone of molding moves down towards motion of the thermoplastic slurry (Fig.3).

Accordingly, the experimental data show that the thermal regime of molding of the beryllium oxide thermoplastic slurry is sensitive to change of the molding velocity, and to conditions of heat extraction on the walls of the annular cavity.

MATHEMATICAL MODEL OF THE SOLIDIFICATION PROCESS

Motion and heat exchange of the BeO thermoplastic slurry in the annular cavity are considered. The slurry flows into cavity with initial temperature of 80°C (Fig.1). As it moves the slurry is cooled and solidified, and on the output from the cavity it acquires structural form of the tube. The movement of slurry occurs in the laminar regime. Special feature of the slurry beryllium oxide is its high thermal conductivity; however due to high viscosity of thermoplastic slurry Prandtl number is much higher than one. Density of the slurry mass is function of temperature and increases as solidification.

The problem is studied in Cartesian coordinate system with axis z and r. OZ axis coincides with the cavity axis direction, and OR axis is radially directed to it. Molding velocity is directed vertically downward along the OZ axis. Radial component of the velocity originates due to the heat exchange of the slurry with the walls of annular cavity.

Rheological properties of the slurry change with temperature. The heat of phase transition is released during the change of state. Cooling of the slurry may lead to the irregularity of the temperature profile and rheological properties of the pressing molding. Solidification begins at the walls of the annular cavity, while in the central part slurry may be in liquid state. As a result, in-feeding of slurry for the compensation of internal shrinkage of volume in the cooling zone of the cavity may occur.

According to the experimental data the slurry solidification occurs at the temperature range from 54 to 40°C. Binder of the slurry is in the amorphous state and passes from the liquid amorphous state to the solid-plastic amorphous state in the zone of solidification [5, 7, 8]. The total amount of heat released per unit mass of the slurry mass is determined by the change of enthalpy ΔH at the phase transition zone.

Heat capacity of the slurry changes in the transition zone. Increase of the enthalpy during the phase transition can be determined by the apparent heat capacity method [9-14]. In this method, the latent heat is taken into account by increasing the heat capacity in the phase transition zone. Changing of heat capacity can be represented as [16-18]:

$$c_p = \begin{cases} c_s, & t < t_s \quad \textit{solid phase} \\ c_{in}, & t_s \le t \le t_l \quad \textit{transition zone} \\ c_l, & t > t_l \quad \textit{liquid phase} \end{cases} \tag{1}$$

where $c_{in} = \dfrac{\left\{ \int_{t_s}^{t_l} c(t)dt + H_{1\to2} \right\}}{(t_l - t_s)}$, $H_{1\to2}$ – the phase transition specific enthalpy of beryllia slurry is determined by experimental data and is equal to $H_{1\to2}$=7800 J/kg [15].

In [14] phase transition function $\alpha(\bar{t})$ is introduced to the transition zone to consider the latent heat, and changing of the slurry heat capacity is expressed by:

$$c_p = c_s \cdot (1 - \alpha(\bar{t})) + c_l \cdot \alpha(\bar{t}) + H_{1\to2} \frac{d\alpha}{dt} \tag{2}$$

where c_s – specific heat of the slurry in the solid state, c_l – specific heat of the slurry in the liquid state, $\alpha(\bar{t})$= 0 for the pure solid slurry and $\alpha(\bar{t})$= 1 for the pure liquid slurry, $\bar{t}$ – dimensionless temperature of slurry ($\bar{t}$ =t/t_0, t_0-initial temperature of the slurry at the inlet of the cavity).

According to the experimental data of beryllium oxide slurry with binder mass fraction of $\omega = 0{,}117$ function $\alpha(\bar{t})$ takes a form:

$$\alpha(\bar{t}) = 5.712 \cdot \bar{t} - 2.8544 \tag{3}$$

The equations (1)-(2) of the method of apparent heat capacity include the latent heat of the phase transition, and are convenient for calculations. For convenience position of the transition zone is not known in advance and is determined as a result of the calculations.

The rheological properties of the slurry for binder mass content $\omega = 0{,}117$ depend on temperature, and expressed by empirical formulas [16]:

$$\mu(t) = 293.6259\exp(-0.05816t), \text{(Pa·s)} \tag{4}$$

$$\tau_0(t) = 11.4 + 11.41\exp\left(-\frac{t - 70.05}{5.47}\right), \text{(Pa)} \tag{5}$$

Density of the thermoplastic slurry is defined by the concentrations of the beryllium oxide powder and the binder:

$$\rho = \frac{\rho_{BeO}\rho_{bin}}{(1-\omega)\rho_{bin} + \omega\rho_{BeO}}, \text{(g/cm}^3\text{)} \tag{6}$$

where ρ_{BeO} is the density of the beryllium oxide powder, ρ_{bin} is the density of the binder, ω is relative mass content of the binder in the fractions.

The density of the binder where $\omega = 0{,}117$ is determined by Eq. (7):

$$\rho_{\mathrm{bin}}(t) = 0.852 + 0.0725\cos\beta(t), \text{(g/cm}^3\text{)} \tag{7}$$

where $\beta(t) = 0.0561(\mathrm{t} + 273.15) \text{-} 16.7361$.

The density of the beryllium oxide is $\rho_{BeO} = 3.02\, g/cm^3$. The density of the binder ρ_{bin} is in the range of temperature from $t = 80 - 40^0 C$ changed within 0.7797 to 0.9010 g/cm^3 and the density of the thermoplastic slurry during solidification increases from 2.2457 to 2.3553 g/cm^3 for this fraction $\omega = 0{,}117$.

Thermal conductivity of the slurry depends on the temperature, and for $\omega = 0{,}117$ it has the following form [15]:

$$\lambda(t) = 1.6 + 4.8\exp(-0.017t), \text{(W/m·}^0\text{C)} \tag{8}$$

In the experiments [5, 8] beryllium oxide slurry shows thixotropic properties of non-Newtonian fluid, and is described by Shvedov-Bingham rheological model [17]. The motion of the slurry in annulus is considered to be steady-state and the system of equations in the narrow channel is used for its study [16, 18]:

$$\rho u\frac{\partial u}{\partial z} + \rho v\frac{\partial u}{\partial r} = -\frac{dp}{dz} + \frac{1}{r}\frac{\partial}{\partial r}\left(r\mu\frac{\partial u}{\partial r}\right) - \frac{1}{r}\frac{\partial}{\partial r}(r\tau_0) \tag{9}$$

$$\frac{\partial \rho u}{\partial z} + \frac{1}{r}\frac{\partial r\rho v}{\partial r} = 0 \tag{10}$$

In the limit of solid-plastic state of the slurry the motion equation (9) expresses the squeezing-out of the molding from the cavity and takes the form:

$$-\frac{dp}{dz} = \frac{1}{r}\frac{\partial}{\partial r}(r\tau_0)$$

In contrast to the motion equation (9) conduction heat transfer along the OZ axis is substantially due to solidification of the slurry, and heat of phase transition is determined by the apparent heat capacity method (2). In the steady-state solidification process of slurry energy equation can be written as [12-14]:

$$\rho u c_p \frac{\partial t}{\partial z} + \rho v c_p \frac{\partial t}{\partial r} = \frac{\partial}{\partial z}\left(\lambda \frac{\partial t}{\partial z}\right) + \frac{1}{r}\frac{\partial}{\partial r}\left(r\lambda \frac{\partial t}{\partial r}\right) \quad (11)$$

The following notes are used in the Eq. (9)-(11): *z, r* – axial and radial coordinates; *u, v* – components of the velocity vector; $p, \rho, T, \tau_0, c_p, \mu, \lambda$ – pressure, density, temperature, shear stress, coefficients of thermal capacity, viscosity and thermal conductivity of the slurry, respectively.

In the Eq. (11) temperature is calculated on the Celsius scale for convenience in comparison with experiment.

Condition of the mass flow rate conservation determines the pressure gradient for thermoplastic slurry extrude from the annular cavity [18]:

$$2\pi \int_{r_1}^{r_2} \rho u r dr = \pi (r_2^2 - r_1^2) \rho_0 u_0 \quad (12)$$

where r_1, r_2 – the radius of the mandrel and bushing, respectively.

Distributions of the velocity and the temperature at the inlet of the cylindrical portion are constant along the cross section of the annular cavity; respectively, all the thermo-physical properties of the slurry are constant:

$$u = u_0,\ v = 0,\ t = t_0 \text{ for } z = 0. \quad (13)$$

On the cavity walls in the area of the liquid slurry state for velocity are put conditions of sticking:

$$u_i = v_1 = 0 \text{ for } z > 0,\ r = r_i,\ i = 1, 2. \quad (14)$$

In solid-plastic state they are no flow and slip conditions:

$$v_1 = 0, \left(\frac{\partial u}{\partial r}\right)_{r_i} = 0 \text{ for } z > 0,\ r = r_i,\ i = 1, 2. \quad (15)$$

The assumption is that the heat from the hot slurry is transferred to the walls of bushing and mandrel. Then, condition of heat exchange can be applied to the surface of the mandrel [26]:

$$\lambda \frac{\partial t}{\partial r} = \alpha_d (t_c - t_d) \text{ for } z > 0,\ r = r_1, \quad (16)$$

where α_d – coefficient of heat exchange between the slurry and the wall of the mandrel, t_d – the temperature of the wall of mandrel, t_c – the average temperature of slurry in cross-section of the annular cavity.

If we mark temperature of the water in the hot and cold contours as t_1, t_2, we can put the boundary conditions on the wall of bushing as:

$$-\lambda \frac{\partial t}{\partial r} = k(t - t_i),\ i = 1,2 \text{ for } z > 0,\ r = r_2, \quad (17)$$

where k is the coefficient of heat transfer on the wall of *bushing*.

At the outlet section of the cavity for temperature are put the following condition:

$$\frac{\partial t}{\partial z} = 0 \text{ for } z = l. \quad (18)$$

Eq. (9)-(12) and boundary conditions (13)-(18) are presented in the dimensionless form for the convenience. Coordinates *z, r* are divided by r_1, velocity components *u* and *v* – by u_0, pressure *p* – by the value of dynamic head $\rho_0 u_0^2$, temperature *t* – by t_0, density, yield point, coefficients of thermal capacity, viscosity, and thermal conductivity – by their values at the temperature t_0.The equations in the dimensionless form include Reynolds number *Re*, Prandtl number *Pr*, and Eckert number *Ec*.

Set of Eq. (2)-(12) is solved numerically at boundary conditions of Eq (13)-(18) [18]. The considered zone is divided into elementary cells with sides $\Delta z_i, \Delta r_j$. Different analogues of the motion equation (9) and energy (11) were obtained by the Crank-Nicolson method of the second order precision, but different analogue of Eq. (10) was obtained by two layer scheme of the second order precision [18]. Pressure gradient is defined by the splitting method [18] from the condition of conservation of mass flow rate (12).

The coefficients of heat exchange and heat transfer on the walls of the annular cavity were determined by comparison of experimental and calculation data.

RESULTS OF CALCULATIONS AND COMPARISON WITH EXPERIMENTAL DATA

The calculation is performed under the same regime parameters and conditions as of the experiments. Calculated data by distribution of temperature in an annular cavity according to the conditions of the first series of experiments are demonstrated in Fig.4.

At the inlet of the cylindrical part of the annular cavity the temperature of the slurry is constant and equal to $t_0 = 80^0 C$. In the area of hot contour isolines (isotherms) of the temperature show the zones of the constant values of the temperature and parameters of the slurry mass is in liquid state. In this part the temperature of the slurry and the hot water is the same, heat transfer practically does not occur on the bushing wall.

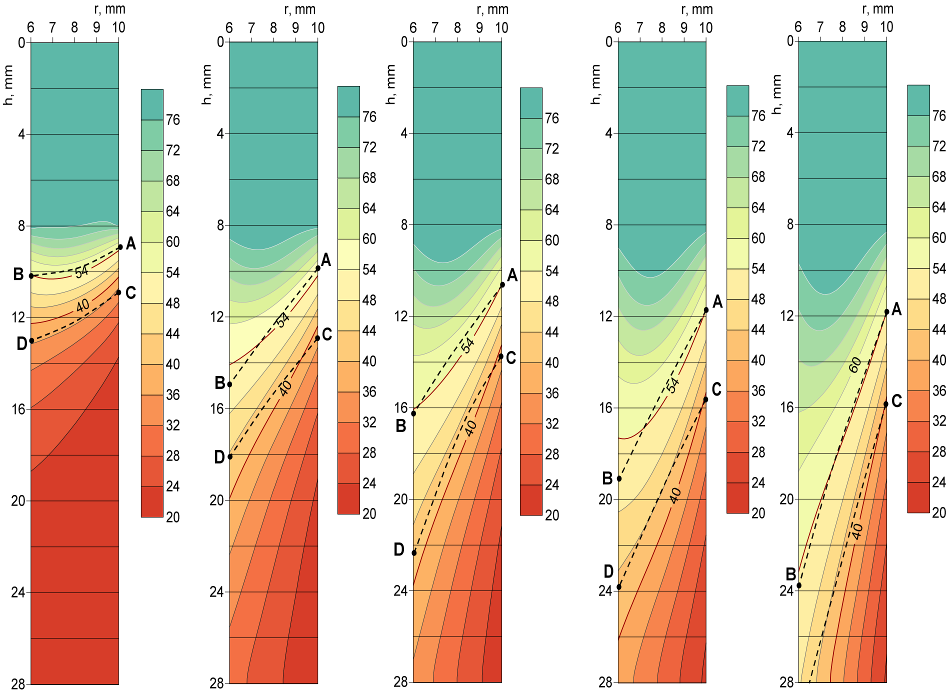

Fig.4. Comparison of the calculated and experimental data of distribution of the temperature depending on the molding velocity (the dashed line is the experimental, and the red line is the calculated)

Cooling of the slurry mass starts in the area of cold contour. Difference of the temperature of the slurry mass and cold water leads to an intensive heat transfer in the second cooling contour, and to reduction of the temperature and to change of rheological properties of the slurry.

The slurry temperature field is variable and changes from 80 to 54^{0}C in the beginning of the second contour.

Isotherm with the temperature 54^{0}C expresses upper bound of solidification zone of the slurry mass, and isotherm 40^{0}C expresses the lower bound of the solidification zone. In the area of solidification the slurry passes from the liquid (viscous-plastic) state to solid-plastic state. The experimental data of isotherms "solidus" AB and "solidus" CD are shown in Fig.2 and 3. It may be noticed an agreement between the calculated data and experiments of positions of isotherms *AB* and *CD*.

At the value of the molding velocity of 20 mm/min position of the transition zone of the slurry from the liquid (viscous-plastic) state to solid-plastic state is located closer to the beginning of the cold contour of cooling. With increasing molding velocity the position of the transition zone begins to pull down towards movement of the molding and takes extensive areas. It explains that with increasing of molding rate convective component of heat flow of the slurry mass increases. The position of the transition zone increases and it covers all length of the mold cavity (Fig.4).

The effect of flow rate and temperature of cold water on the position of the transition zone of the molding by conditions of the experimental researches were determined in the second series of calculations (Table 2). Calculation data of the temperature distribution and the position of the transition zone of molding, limiting by isotherms *AB* and *CD* are shown in Fig.6. In the first three cases, the temperature of cold water is 15^{0}C, and its flow rate reduces from 1000 to 250 l/hour, respectively. Reducing the temperature of cold water increases the intensity of heat extraction and reducing of its flow rate and vice versa, it slows down the process of heat extraction. Increasing the temperature of cold water till 20^{0}C, as well as reducing of its flow rate leads to a reduction of heat extraction.

The calculated temperature data are in agreement with the experiment results (Fig.5).

The experimental and calculated data show that beryllia thermoplastic slurry solidification process can be controlled by adjusting the flow rate and temperature of the hot and cold water.

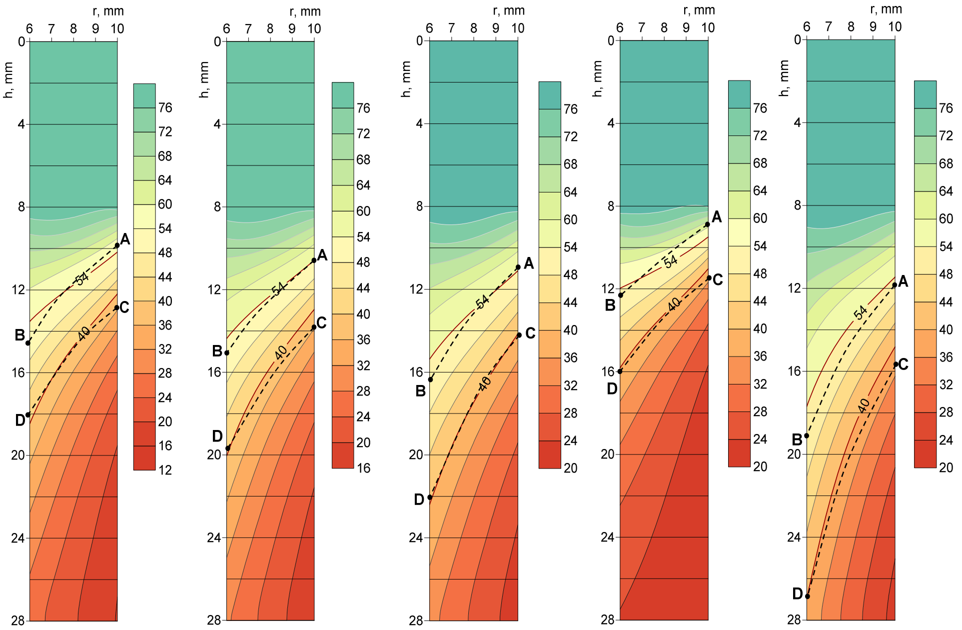

Fig.5. Comparison of the calculated and experimental data of distribution of the temperature depending on the flow rate and the temperature of cold water (the dashed line is the experimental, and the red line is the calculated)

One of the important parameters characterizing the solidification process of thermoplastic slurry is the change of its density in a formative cavity during cooling. The dynamics of changes of slurry density averaged over the cross section, and slurry density on the inside (mandrel) and outer (bushing) walls of the formative cavity for first mode of the experiment (Table 1) is shown in Fig 6. Significant sealing of thermoplastic slurry occurs in the second contour, where the temperature of cooling liquid is 20^0C. The distribution of densities confirms that the cooling extends from the outer wall, since the slurry on the wall is solidified earlier than in other parts (Fig.6).

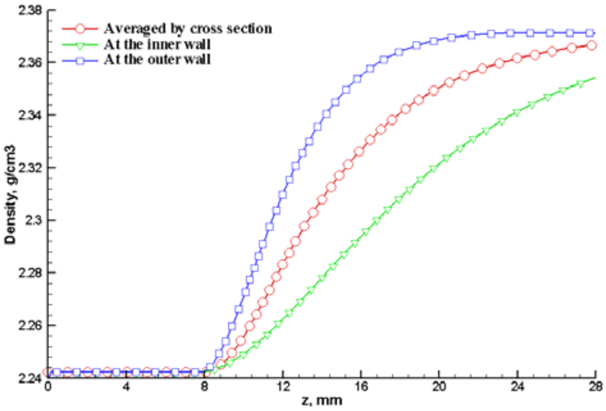

Fig.6. Change of the density of the slurry along the length of the cavity

The intensity of the hot slurry solidification by cooling is estimated by the heat amount transferred by slurry to the cooling liquid through the wall. The distribution of the dimensionless heat flux through the outer wall of the cavity along its length is shown in Fig.7. The heat flow characterizes heat exchange between the hot slurry and the cooling liquid and it is defined as the gradient of the heat amount through the wall.

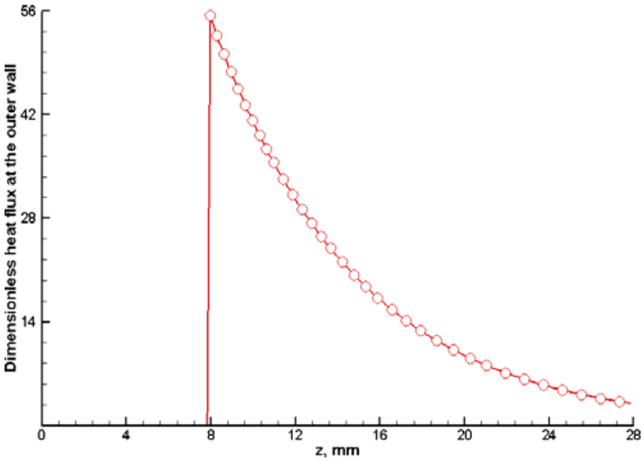

Fig.7. Change of the heat flux at the outer wall along the length of the cavity

As seen from Fig.7 the greatest heat flow comes to the beginning of the cold contour, where the temperature decreases sharply. Then, the heat flow is reduced to a minimum at the end of formative cavity. This is because the slurry temperature until the end of the cold contour approaches the temperature of cooling liquid.

CONCLUSION

During the experiments of the research on the effect of casting regimes on the temperature field in the solidification zone of the molding were identified the followings:

- the position of solidification zone of the slurry mass when molding velocity changes from 20 to 100 mm/min;
- the position of solidification zone of the slurry mass in the form-building cavity depending on the water flow rate and the temperature in the cold contour of cooling.

The upper bound of the solidification zone was estimated by isotherm "solidus" 54^0C, and the lower bound – by isotherm "solidus" 40^0C.

Temperature distribution, estimated during the experiments, in the form-building cavity of bushing depending on the molding velocity and heat extraction conditions on the walls of form-building of annular cavity lets us determine the transition from liquid (viscous-plastic) state to solid-plastic one.

The experiment results were analysed and generalized using mathematical model of the thermoplastic slurry molding process. The latent heat of the phase change has been accounted by the apparent heat capacity.

The mathematical model includes the equations of the law of conservation of mass, momentum and energy of non-Newtonian fluid with the Shvedov-Bingham's rheological model. Rheological and thermo-physical properties of the slurry were found on the basis of experimental data and express dependence on the temperature. The temperature field of the slurry in liquid (viscous-plastic) and solid-plastic states were obtained in the calculations. The positions of isotherm "solidus" (54^0C) and "solidus" (40^0C), expressing the upper and lower boundaries of the solidification zone position were determined.

The results of calculation show physical validity of the proposed mathematical model of the molding process of the thermoplastic slurry beryllium oxide. The model of the process of forming ceramic fabrication is a flexible system, and it includes the rheological and thermal properties of other original materials.

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