EQUIPMENT FOR INJECTION MOULDING OF THERMOSETTING MATERIALS BASED ON CERAMIC AND METAL-CERAMIC POWDERS

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Results of design and process investigations of the construction of technological equipment for injection moulding of articles with complex shapes from thermosetting materials based on ceramic and metalceramic powders are presented. Numerical simulation of the proces of heat transfer is conducted. The parameters of a commercial plant (GShP-2) and of a newly designed plant for injection moulding are determined. The newly designed plant produces a uniform distribution of temperature in the functional loop and creates a thermosetting material and injectible material that are uniform in terms of properties.

Keywords: injection moulding, pressure, temperature, moulding, sintering, packing factor, hardness, fabrication plant, modeling, finite-element method, thermal conductivity

The advantage of the technology of hot slip casting lies in the possibility of fabricating articles having complex shapes from all the different types of materials that are produced by methods of powder metallurgy. The essence of the technology consists in the making of thermosetting moulding systems of powders of solid materials in a mixture with organic substances and subsequent pouring of these systems at a required temperature into metallic moulds. The casting systems are cooled in the mould and harden, ingots being formed possessing the configuration of the working part of the mould. To obtain finished articles, the ingots are subjected to heating to the sintering temperatures of the powders. The basic laws governing the production of ceramic and metal-ceramic materials by means of hot slip casting together with subsequent sintering have been established [1, 2].

The technology of hot slip casting has been successfully applied at the Institute for Problems of Materials Science (IPMS) of the National Academy of Sciences of Ukraine for the fabrication of ceramic articles having complex shapes (nozzle vanes and blades of gas turbines, nozzle apparatuses and impellers of gase-turbine engines). The flow chart of a laboratory-scale plant for hot slip casting of the design created by the IPMS is presented in Fig. 1. The plant consists of a frame in the form of a sectional construction containing a lower plate (10), crossbeam (2), and two braces (3). A heated beaker 6 is fixed to the lower plate of the frame. A casting mould (4) which is held against the beaker by the screw (1) is mounted on the cap (5) of the beaker. Filling of the casting mould with slip is achieved by means of compressed air fed through a connecting pipe situated in the cap of the beaker from a tank provided with a reductor. If necessary, the work space may be preliminarily evacuated by means of a vacuum pump.

In recent years there has been great interest expressed in an analog of hot slip casting, referred to as the technology of injection moulding of articles of complex shape. The technology differs substantially from the technology of hot slip casting. In hot slip casting low compressed-gas pressures (up to 0.5 MPa) are employed, whereas the injection moulding technology employs presures up to 10-50 MPa created by ram- or screw-type injection casting machines. The injection moulding method has been successfully applied in the production of articles from metallic powders for several decades. The use of higher pressures in the injection moulding method improves the quality of the material of the ingots due to the decrease in the quantity of pit- and pore-type defects and, thereby, increases the yield of

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Fig. 1. Diagram of a plant for casting of ceramic. (1) Screw; (2) crossbeam; (3) brace; (4) casting mould; (5) cap; (6) beaker; (7) heater; (8) insulator; (9) pouring gate; (10) plate.

finished articles at different stages of the technological treatment. The physico-mechanical properties of the sintered article are also substantially improved. The development of high-strength technical-grade ceramics, the outlook for its use in high-temperature technology, in electronic industry, and in medicine all require the use of the method of injection moulding for the fabrication of articles of complex shape out of ceramic and metal-ceramic materials [3]. However, there is only limited information available on the parameters and design features of equipment used for injection moulding of thermosetting materials based on ceramic powders.

The objective of the present study is to conduct a set of design and technological investigations to learn how to create process equipment for injection moulding of thermosetting materials based on ceramic and metal-ceramic powders.

The GShP-2 extruder manufactured by the Odessa Nonstandard Equipment Plant was selected as the base plant. The extruder is a ram-type machine with vertically arranged working cylinder intended for injection moulding of articles from plastic. A modernized design of an extruder for injection of ceramic and metal-ceramic thermosetting materials is shown in Fig. 2. The plant has a framework design consisting of a pedestal (1) in the center of which a regulating table (2) is mounted. A hydraulic cylinder (11) is mounted at the center of the upper plate (10) of the pedestal, with a ram (9) attached to the cylinder rod. To eliminate the gap between the ram (9) and the walls of the tank (5) used to store the material, the ram is equipped with sealing rings made of fluoroplastic. The tank (5) is attached to the spring-loaded cross-beam (6) which is mounted on braces (the braces and springs are not shown). A nozzle (3) is attached to the lower part of the tank. Heating of the tank is accomplished by means of a heating coil (4). The design of the nozzle (3) (with inner cavity conically shaped) creates favorable conditions for injection of highly viscous thermosetting materials into the metal die pressure cast mold (13). To make it easier to load the material into the tank and reduce the temperature gradient within the tank, a heated feeder (12) that ensures preheating of the thermosetting material is used.

Fig. 3. Structural model (a) and finite-element grid (b, c) for the power unit of an injection moulding plant. (1) Functional loop filled with thermosetting material; (2, 3, 4, 5) steel nozzle, cylinder, hydraulic cylinder rod with ram, and structural element of cross-beam, respectively.

Analysis of the physico-mechanical properties of specimens of the material obtained by means of injection moulding on this plant showed [4] that the pressure exerts a substantial influence on the packing factor k_{pack} of the material, which in turn determines the mechanical properties of the compacts; thus, if the pressure is increased from 0.5 MPa to 10-50 MPa, the hardness grows by 50-70% and the bending strength by 20-25%. The influence of injection pressure on the physico-mechanical properties is also observed following sintering of the materials; if the pressure is increased from 0.5 MPa to 40-50 MPa, the packing factor in the two metal-ceramic materials WC − Co and WC − Ni grows from 0.95-0.96 to 0.990-0.998 and hardness by 11-14%, while in AlN based materials the hardness increases by 5-7%. However, it was established that there exists a significant scatter of these characteristics particularly in ceramic specimens. It was established by means of metallography and fractography that the degree of nonuniformity of the materials with respect to porosity and the presence of pitting are the basic reasons for instability in the physicomechanical properties.

According to the results of technological and computational investigations, the causes for nonuniformity in the material of the compacts can be found in stratification of the thermosetting material in the working cylinder upon injection moulding due to the lack of any mixing of the material and the vertical placement of the cylinder, a circumstance that is especially typical of powders of hard alloys; the fact that no evacuation of the thermosetting material is performed in the functional loop prior to injection; and nonuniform heating of the thermosetting material throughout the entire functional loop.

The heat transfer process was modeled in order to estimate the temperature gradient in the working zone. It is known [1] that the working medium must be heated to a temperature between the temperature of solidification of wax ($T \approx 59^{\circ}$ C) and the temperature at which thermosetting material becomes distended ($T = 85{\cdot}90^{\circ}$ C). To ensure minimal viscosity of the material, maximal casting capacity, and uniformity of properties within the volume of thermosetting material, the temperature must be varied slightly ($T \approx 80^{\circ}$ C).

Fig. 4. Distribution of temperature $({}^{\circ}C)$ in injection casting plant (a) and in the lower portion of the plant at the exit from the nozzle (b).

The distribution of the temperature in the working zone was determined by numerical solution of the problem of thermal conductivity for the entire injection casting plant on the basis of the finite-element method [5]. In view of the axial symmetry of the plant and the boundary conditions on its surface, half of its axial section was considered (Fig. 3a). The construction was quantized by means of 7101 triangular elements connected at 3748 nodes (Fig. 3b). The finiteelement grid was made denser in the region of the nozzle (Fig. 3c). The boundary conditions for the thermal conductivity problem were specified as follows. On surfaces *cd* and *ef*, both of which are sufficiently distant from the functional loop, temperature was set to $T = 45^{\circ}\text{C}$; on the lateral surface of the cylinder *ab* heat-exchange conditions with the heating element were selected so that they would determine the design temperature in the functional loop (heatexchange coefficient $\alpha = 50 \text{ W/m}^2\text{·}^{\circ}\text{C}$) and ambient temperature $\Theta = 100^{\circ}\text{C}$); a condition of convective heat exchange with the air was adopted on the remaining portion of the surface (α = 50 W/m²⋅°C, Θ = 45°C).

The thermal conductivity* of the structural elements of the injection casting plant, λ (W/m⋅°C) was determined thus: steel, 39 [6]; AlN-based material, 1.63; Si_3N_4 , 0.72; and material based on WC – Co and WC – Ni, 1.33 and 0.96, respectively. It was also established that the temperature distribution is a steady-state distribution in the entire volume being considered (Fig. 4a). In the functional loop temperature is varied in the range 64-80°C, with the greatest gradients observed in the upper part of the loop and the principal outflow of heat occurring through the piston and rod of the hydraulic cylinder. In the region of the nozzle the temperature drop does not exceed 3 deg (Fig. 4b). Thus, the maximal temperature drop in the thermosetting material along the axis of symmetry amounts to ≈16 deg, which does not ensure that the properties of the material will be uniform throughout the functional loop.

In light of the foregoing discussion, a design of a plant for injection moulding of thermosetting materials based on ceramic and metal-ceramic powders was developed. A flow chart of the plant is presented in Fig. 5.

The horizontal position of the working cylinder (5) which is mounted on the frame (1) produces a simpler configuration and more convenient maintenance. The reciprocating motion of the ram (4) is supplied by the hydraulic

^{*}The experimental investigations of the thermal conductivity of thermosetting materials was performed by A. P. Podoba.

Fig. 5. Plant for injection casting of thermosetting materials based on ceramic and metalceramic powders with evacuation of the functional loop. (1) Frame; (2) bracket; (3) hydraulic cylinder; (4) ram; (5) cylinder; (6) support; (7) feeder; (8) adapter; (9) nozzle; (10) pressure casting mold; (11) screw; (12) support.

cylinder (3) which is mounted coaxially with the working cylinder on the frame of the plant. The hydraulic cylinder is powered from an independently situated hydrostation. The previously conditioned thermosetting material is loaded into the feeder (7) from which it fed through the internal channel of the adapter (8) into the working cylinder. A planetarymotion mixer in the cap of the feeder is provided in order to prevent any stratification of the material associated with the different specific weights of the initial powder and the binder. Rotation of the mixer is realized by the drive on the crossbeam, which is attached to the brace. Evacuation of both the feeder and the cavity of the working cylinder by means of a rotary pump are incorporated into the present design. Evacuation of the cavity of the working cylinder may be accomplished simultaneously with evacuation of the feeder or separately. To make servicing operations, that is, cleaning of the inner surfaces of the feeder, replacement of the mixer, and loading of material, the design allows for removal of the cross-beam from the feeder. Feeding of the material into the metal die pressure cast mold is accomplished through the nozzle (9) which is situated at the exit from the working cylinder. The internal cavity of the nozzle is conically shaped, which produces an increase in the rate of injection of the cast system into the metal die pressure cast mold. A slide valve is mounted at the exit from the nozzle in order to hermetically seal the working cylinder in the course of evacuation and injection. A valve of analogous design (Fig. 6) is mounted between the feeder and the adapter (8).

Fig. 6. Slide valve. (1) Flange; (2) valve; (3) half-ring; (4) flange; (5) handle.

Fig. 7. Structural model (a) and finite-element grid (b, c) for power assembly of newly designed injection casting plant. (1) Functional loop filled with thermosetting material; (2, 3, 4, 5) steel nozzle, cylinder, hydraulic cylinder rod with ram, and structural element, respectively.

The slide valve consists of two flanges (1) and (4) with central openings the diameter of which is equal to the diameter of the inner channels of the feeder and the adapter. The flanges are adapter flanges and serve for preventing abrasive wear of the connecting surfaces of the feeder and the adapter. A half-ring (3) is mounted between the two flanges, with a valve (2) being located within the half-ring. The valve opens and shuts off the lock channel that connects the feeder to the adapter. The valve is in the form of a disk with eccentrically realized opening the diameter of which is equal to the diameter of the central opening of the two flanges (1) and (4). Rotation of the valve is realized by the handle (5) the displacment of which is limited by the sector recess angle of the half-ring (3). To ensure that the lock channel is hermetically sealed, all the faces are sealed with circularly shaped rubber rings

To ensure uniform heating of the material throughout its entire volume, all the surfaces in contact with the material are equipped with water jackets into which enters water from a thermostat that has been heated to a required temperature. Such a design prevents overheating, swelling, and boiling over of the thermosetting material. A calculation of the temperature field in the newly designed plant has shown that if the thermosetting material is warmed up to a temperature of about 80° C, its maximum temperature drop in the functional loop is to 2-6 deg and depends on the type of material.

The temperature distribution in the functional loop was determined by analogy with the above problem. A structural model of the plant (Fig. 7a) was quantized by means of 2202 triangular elements connected together at 1242 nodes (Fig. 7b). In the region of the nozzle, where the temperature gradients are maximal, the finite-element grid was concentrated (Fig. 7c). A temperature $T = 20^{\circ}$ C was set on the surface *cd*, which is quite distant from the working zone; a condition of convective heat exchange with the liquid ($\alpha = 7600 \text{ W/m}^2\text{·}^{\circ}\text{C}$, $\Theta = 80^{\circ}\text{C}$) was specified on the lateral surface of the cylinder, surface *ab*, with the temperature of the liquid determining the design temperature in the working zone; a condition of convective heat exchange with the air ($\alpha = 50 \text{ W/m}^2$ ⋅°C, $\Theta = 20$ °C) was adopted on the remaining portion of the bounding surface.

Fig. 8. Stational temperature field (°C) in newly designed injection casting plant in the case of moulding articles from AlN.

Fig. 9. Stationary temperature fields in the region of exit of thermosetting material from nozzle in injection casting of materials based on: AlN (a), $Si₃N₄$ (b), VK15R (c), VN20Gr2 (d). $T_{min} = 76.2°C$ (a), 73.9°C (b), 75.8°C (c), and 74.9 $^{\circ}$ C (d).

As a result of a calculation, the stationary distribution of the temperature throughout the entire volume of the plant was determined (Fig. 8). The temperature is distributed in the functional loop practically uniformly and for the most part is equal to 80° C with slight temperature gradients observed in the lower part of the loop in the region near the nozzle (Fig. 9). Here the minimal temperature depends on the thermal conductivity of the injected material and the maximal design value of the temperature drop ($T \approx 6$ °C) is observed for Si₃N₄ based material which has the lowest thermal conductivity.

From a simulation of the process of heating of thermosetting material it was established that in most of the functional loop the temperature is constant and equal to the temperature of the liquid in the heater. Consequently, heating conditions are optimal in the newly designed injection casting plant and make it possible to mould a thermosetting mass of injected material that is practically uniform in terms of properties. If necessary (with the use of polymer binders) glycerine and oil may serve as the working liquids, thus making it possible to heat the thermosetting material to temperatures of 130-180°C. The injection casting plant has been manufactured and is being used in the manufacture of articles.

Thus, by numerical modeling of the heat transfer process and technological investigations it has been shown that the temperature drop in the working volume of a type GShP-2 industrial plant amounts to only 16° C, which does not ensure uniformity of thermosetting material and attainment of the optimal properties of the material of ceramic and metal-ceramic articles.

As a result of design and technological ivnestigations a plant has been developed for injection casting of thermosetting compounds formed from ceramic and metal-ceramic powder under pressures up to 10 MPa with evacuation of the working volume, thus ensuring moulding of ingots of complexs shapes and attainment of optimal properties of the material forming the ingots.

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