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Experimental studies of changes in the volume of crystallizing metal under high pressure

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The influence of pressure on the formation of the structure and properties of metals deserves attention both from a scientific point of view and in a purely applied aspect. A scientific and methodological approach has been proposed to study the crystallization process, which involves measuring the volume of crystallizing metal under high pressure conditions. It was found that to compensate for the decrease in the volume of the metal during crystallization, it is necessary to adjust the pressure value to the metal's resistance to „shear deformations“, which ensures the delivery of individual atoms to crystals in which structural defects occur. The measurement results were used in the development of pressure application schemes, as well as in calculations of parameters of research and industrial equipment.

Keywords: pressure overlap diagrams, compressibility, viscosity, elasticity, structural defects, metal properties.

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In industrial contexts, the use of pressure as an additional parameter of external influence on metal crystallizing in ingots or cast blanks is fraught with both scientific and purely technological problems. At the same time, it should be noted that technologies combining the processes of liquid metal casting and pressure treatment of crystallizing metal are in demand, are considered to be promising, and have high innovative potential [1,2].

Viewed as an object of research, pressure on metal is associated with several complex phenomena simultaneously. From the thermodynamics perspective, pressure is related to an increase in melting temperature by the well-known Clausius–Clapeyron formula.

The pressure acting on liquid metal facilitates an increase in the cooling rate and, according to the Tammann theory, increases the number of crystallization nuclei and the rate of their growth. It affects the variation of composition and the interaction of phases. The fluidity, viscosity, plasticity, strength, and elasticity of metals are also related to pressure. Therefore, the aim of the present study is to establish the patterns of influence of pressure on the behavior of crystallizing metal and on the improvement of its physical, mechanical, and functional properties. To achieve this research goal, one should consider not individual, but interconnected phenomena of different nature and varying degrees of complexity.

One of the reasons for the lack of data on the behavior of liquid metal under pressures up to 500 MPa is the complexity of arrangement and performance of the corresponding experiments. The lack of results of quantitative evaluation of the properties of molten metal under pressure is an obstacle to the development of the theory of liquid state and the reason for insufficiently fast development of metal physics, metallography, and technologies and equipment for metallurgical production.

According to [3], the viscosity of the liquid phase undergoes no significant changes in the process of solidification, although the concentration of dissolved substances increases. In rheological terms, the solid phase in the liquid–solid phase coexistence region behaves more like a liquid than like a solid.

The reliability of results of experimental studies and their relevance to practical applications are largely related to the scale factor. The issue of choosing the shape and size of test samples becomes complex and controversial.

The widely used V95 aviation alloy was chosen as the initial material. The test sample was a cylinder 80 mm in diameter and 70 mm in height. The diagram of application of pressure is shown in Fig. 1.

According to this diagram, mold cavity *7* is filled first, and plungers *8* and *9*, which are driven by individual hydraulic drives generating a force of 0.3 MN, then exert pressure on the examined metal. When plunger *8* covers the pouring hole, plunger *9* is switched on, and liquid metal remains in a state of uniform compression under the pressure of plungers until the start of crystallization. This state has not been studied yet, and the prospects for its practical application remain uncertain.

After a short period of time, crystals start forming due to heat removal at the interfaces of liquid metal with the plungers and walls of the mold. As crystals continue to grow, the resistance to the motion of plungers increases. A physical question regarding the state of metal atoms under pressure and the specifics of their interaction then arises.

It was found that the plungers penetrate deep and press in a significant volume of metal, which contradicts the idea of

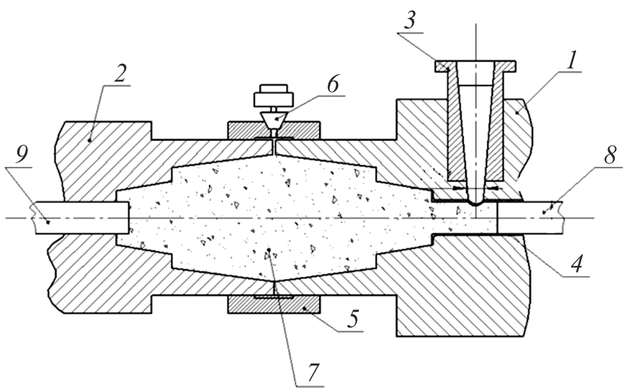

Figure 1. Diagram of the process. *1* — Fixed press plate, *2* — movable press plate, *3* — pouring basin, *4* — pressing chamber, *5* — band, *6* — vacuum pump connection, *7* — mold cavity, *8* — right pressing plunger, and *9* — left pressing plunger.

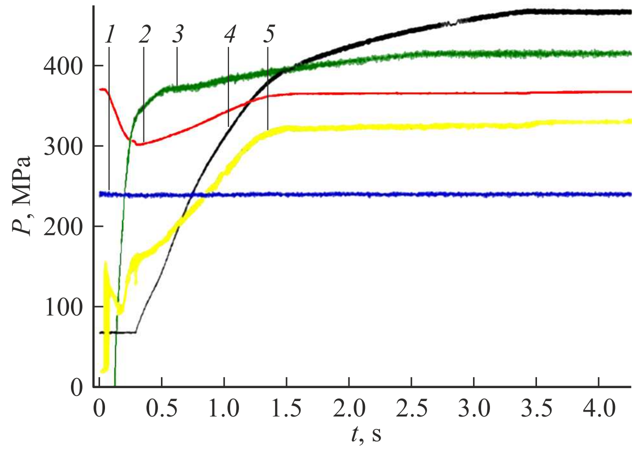

Figure 2. Computer record of the application of pressure and the motion of pressing plungers. *1* — Motion of the movable plate, *2* — mold locking force, *3* — motion of the left pressing plunger, *4* — motion of the right pressing plunger, and *5* — pressure of the working fluid in the hydraulic system.

incompressibility of liquid. A software and hardware measurement complex was designed and constructed in order to study the relationship between the pressure magnitude and the motion of pressing plungers [4].

Following proper processing, the measurement signals from sensors were recorded in the form of computer cyclograms. Notably, it follows from Figs. 2 and 3 that the plungers move in different manners; a dependence on pressure of the working fluid in the hydraulic system may also be traced. It can be seen from Fig. 3, where the data obtained after processing the cyclograms are presented as functions of time, that plunger *8* shifted by 75 mm into the bulk of metal within ~ 2 s, which corresponds to metal compressibility Z_1 of $\sim 4.5\%$. Here,

$$Z_1 = \Delta V/V_0 = S_1 f/V_0,$$

where $S_1 f$ is the volume of pressed-in metal equal to the product of displacement of plunger *8* and its cross-section area and V_0 is the volume of metal poured into the mold. At time $t = 15$ s, the displacement of plunger *8* was $S_1 = 100$ mm.

Plunger *9* was switched on with a delay of 3 s and shifted by $S_2 = 100$ mm; as a result, the relative reduction of the poured metal volume reached $Z = 12\%$. This result exceeds significantly the value of $\sim 6\%$ obtained for the free relative reduction in volume of aluminum alloys within the interval from the crystallization temperature to room temperature [5]. The obtained data suggest that the components of volume reduction are balanced as follows:

$$\Delta V^G = \Delta V_1 + \Delta V_2 = 6V_0/100, \quad (1)$$

$$\Delta V^p = \Delta V_1 + \Delta V_3 + \Delta V_4 = 12V_0/100. \quad (2)$$

Equation (1) characterizes volume change ΔV^G in the process of crystallization and cooling under the influence of gravity, while Eq. (2) corresponds to volume change ΔV^p and cooling to the temperature of the end of crystallization

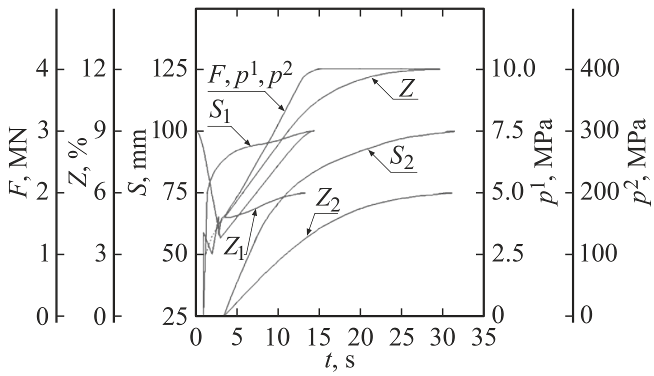

Figure 3. Results of processing the computer record of pressure treatment of liquid and crystallizing metal. F — Mold locking force, p^1 — pressure in the hydraulic system, and p^2 — pressure applied to the examined metal.

under pressure. In formulae (1) and (2), ΔV_1 is the reduction in volume of liquid metal upon cooling to the temperature of the end of crystallization, ΔV_2 is the reduction in volume of metal upon cooling from the temperature of the end of crystallization to room temperature, ΔV_3 is the reduction in volume of liquid metal under pressure, and ΔV_4 is the reduction in volume of metal in transition from the structureless liquid state to the crystalline solid state under pressure. Component ΔV_2 in missing in Eq. (2), since the recording of sensor readings was stopped at the end of crystallization. This component

$$\Delta V_2 = 3\alpha_T \Delta T = 3 \cdot 23 \cdot 10^{-6} \cdot 600\,\text{K} = 4.14V_0/100, \quad (3)$$

where α_T is the thermal coefficient of linear expansion of aluminum alloy and ΔT is the interval from the temperature of the end of crystallization to room temperature.

According to Eq. (1), $\Delta V_1 = 1.86V_0/100$. Then

$$\Delta V_3 + \Delta V_4 = (12 - 1.86)V_0/100. \qquad (4)$$

Equation (4) may be used to quantify the change in volume of crystallizing metal under pressure. With a pressure of 400 MPa applied, the relative compression of liquid and crystallizing metal is

$$Z^* = (\Delta V_3 + \Delta V_4)/V_0 \cdot 100 \sim 10\%. \qquad (5)$$

Of fundamental importance is the experimentally verified fact that the volume of liquid metal decreased by $\sim 4.5\%$ before the onset of crystallization under a pressure of ~ 100 MPa. Apparently, this is induced by convergence of atoms that are completely and (or) partially fixed in the process of Brownian motion. This state of metal was achieved technically and technologically, but its influence on the crystallization process and the properties of metal products has not been studied to date.

The convergence of atoms at temperatures above the temperature of the onset of crystallization is likely to alter the conditions of interatomic interactions and release of latent heat of crystallization. In confirmation of Landau's crystallization theory [6], a quasi-crystalline phase was noted in [7] in alloys of the Al–Mn system subjected to crystallization under pressure. In this context, the process of application of pressure to crystallizing metal opens up new possibilities for industrial production of special-purpose quasi-crystalline alloys. One important task in this regard is to study the force interaction of atoms under pressure with the aim of establishing the dependence of the mutual spatial arrangement of atoms in crystallized metal on the magnitude and the rate of application of pressure. In turn, the degree of perfection of the crystalline metal structure may be associated with the density of dislocations and the efficiency of interatomic interactions in the course of loading and deformation. The lack of these dependencies is the key obstacle to solving various problems relevant to the design and fabrication of materials with specified properties.

Considerable research attention is paid to the study of metal properties both in Russia and abroad. The obtained results have brought impressive gains, and metal products are being used more and more efficiently.

At the same time, state-of-the-art experimental physics techniques still do not allow one to examine the properties of metals in a molten state at high temperatures. This refers to thermophysical, chemical, mechanical, and other properties in their interdependence and interaction or, in other words, a group of properties wherein compressibility under high pressures provides new information necessary for investigating and understanding the nature of interatomic bonds and interactions.

The results of studies into the properties and behavior of liquid metal under pressure are in demand in the development of new technologies, equipment, and automated control systems for the fabrication of metal products with a novel combination of physical and mechanical properties. Volumetric shrinkage compensation does not only eliminate structural defects of crystallizing metal (pores, cavities, looseness), but also establishes a better mutual arrangement of atoms, ensuring integrity of hydraulic and pneumatic parts and products that withstand high internal pressures.

As a proof of high structural integrity, 20 gearbox housings to be operated under a pressure of 40 MPa were manufactured using the new process on order of the „Armatura“ design bureau in Kovrov, which is currently a branch of the Khrunichev State Research and Production Space Center. After mechanical processing, each individual housing was tested under a pneumatic pressure of 50 MPa; in the event of „inleakage“ (pressure drop), fabricated parts were rejected. Each of the 20 manufactured housings passed tests under pressure up to 100 MPa, was mounted for proper operation, and retained its structure and properties after two years of normal operation at the testing ground.

Another example of qualitative improvement of metal properties as a result of high-pressure crystallization is a batch of 500 electric heaters made of an aluminum-based alloy with built-in heating elements, which was manufactured using the new process on order of NIKIMT-Atomstroy JSC. The continuous uptime was increased by a factor of 5 or more.

Research and development works on mastering the production of disks for trucks by casting with crystallization under high pressure are currently being carried out in collaboration with UC Rusal.

Conflict of interest

The authors declare that they have no conflict of interest.

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