

Formation of the Ice Casting Pattern Structure and Methods of Its Modeling

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Abstract: The composition and structure of the material of the single-use ice casting pattern in many respects determine the quality both of the pattern and of the casting, obtained with its help, as far as later on that material is used in the production of the casting sand mold. Therefore, it is very important to understand the mechanism and kinetics of ice solidification in the process of obtaining the pattern as well as distribution of impurities in the ice. The modern crystallization patterns, taking into account the theory of clusters and fractals, are considered, and the examples of the patterns are given.

Keywords – Ice casting patterns, aqueous composition, dendrites, clusters, fractals.

I. INTRODUCTION

Development of new cryo-technology types for foundry allows increasing its environmental cleanliness with the help of replacement of the traditional polymer pattern molding materials by frozen water. Casting for ice patterns (CIP) allows noticing some crystallization phenomena laws, which are important for creation of qualitative casting patterns with a variety of technological properties. Ice transparency is long used to facilitate visual observation of dendrites structure formation for physical modeling of crystallization processes for metals, polymers, etc. The subject of this paper is peculiarities of ice crystallization during cooling of the water to the temperatures of $-20\dots-15^{\circ}\text{C}$, modern variants of its modeling and the use of research results for production of ice casting patterns [1].

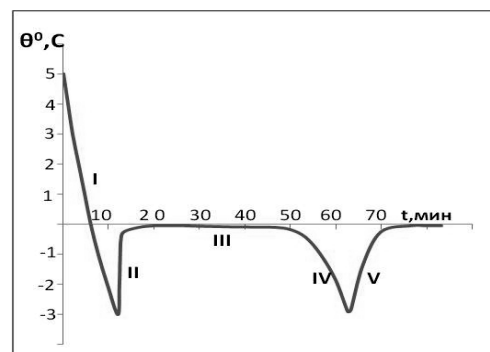
For the production of ice pattern samples, there were used pure tap water and the aqueous composition with binding additions up to 25% for the purpose of the further partial impregnation of the sand of the mold, formed around the ice pattern during its melting. In some cases, fine bentonite or refractory filler - disthene-sillimanite - were injected into the pattern material or pattern coating in order to create antiburning properties of the sand mold surface. The molding details are described in the paper [1]. Most often single-use ice casting patterns after placing them into the mold sand are exposed to melting with the removal of fluid excess from the mold cavity. Therefore, the temperatures $-20\dots-15^{\circ}\text{C}$ are sufficient for cooling.

II. ANALYSIS OF THE LATEST INVESTIGATIONS

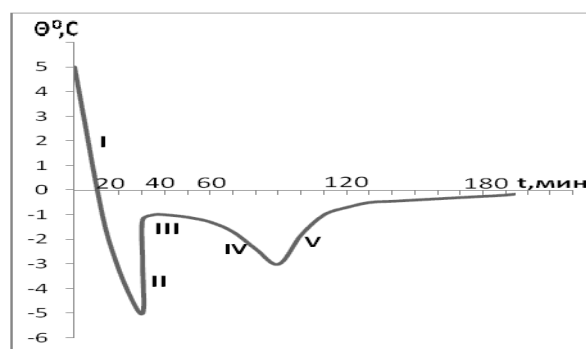
Analysis of the latest investigations revealed the absence of the studies of the structure of industrial constructions from ice. However, similar phenomena are considered in structural cryology [2] and permafrostology [3], where ice crystals are

studied mainly as the material basis of our planet's cryosphere. In this connection, we note that circulation of ice patterns material with its melting and partial evaporation in the foundry area is in some way similar to water circulation in the nature of our planet. Such circulation is a global system of transfer that supports energy and ecological balance of the Earth. Probably, with the exception of water, there are no other natural materials, which can exist in solid, liquid and gaseous state in the narrow temperature range $-20 \dots +20^{\circ}\text{C}$.

In [3], which is considered a classic paper in this area, experiments on the freezing for water-saturated samples of typical types of soils, in particular, of sand and of bentonite, are presented. The following method was used: the soil was frozen in a test tube, immersed into a freezing mixture in the Dewar flask with the temperature set at -10°C . The temperature θ of cooling and freezing of the soil was recorded on the photographic paper with the help of the mirror galvanometer (Fig. 1).



a)



b)

Fig. 1. The curves of cooling for: a) the sand with humidity $W=19,6\%$; b) the dispersed bentonite clay with humidity $W=80,5\%$.

In Fig. 1a the curve of freezing of water-saturated Luberets quartz sand consists of several portions. Portion (I) corresponds only to the decrease in the sample temperature during cooling and overcooling with a minimal temperature $\theta = -3^{\circ}\text{C}$. Water starts to freeze in the sample and such quantity of latent heat of the ice formation is released that the soil temperature rises sharply to $\sim 0^{\circ}\text{C}$ (portion II, Fig. 1a). At this temperature, freezing of all free water occurs in the water-saturated sample, and the time of freezing depends on the composition humidity, the cooling rate and the sample sizes. Then follows the portion, which is characterized by a stabilization of the temperature at 0°C , that corresponds to the temperature of the soil freezing. If we continue to freeze the sample, then after a certain time the temperature starts to decrease first by the curvilinear law. Concavity of the curve shows that in the area a latent heat of the ice formation still continues to release and loosely bound water, i.e. water with a variable phase composition, freezes. However, at temperatures below -1°C cooling follows the linear law (portion IV, Fig. 1a) and the sand can be considered to be frozen, i.e. practically all the water in it has turned into ice. Then the temperature rises (portion V, Fig. 1a): first linearly and later curvilinearly as far as latent heat of ice formation starts to be absorbed before reaching the melting temperature of the sample.

The same also applies to clay samples (Fig. 1b). The main difference is that after the temperature jump a lower temperature becomes steady ($-2,5 \dots -0,1^{\circ}\text{C}$) and the curve of the temperature stabilization portion looks sloping. This is caused by the phenomenon of swelling, or micelle formation, of the clay particles, that leads to a partial water binding and that may also be a consequence of some increase in the concentration of salts in the water.

For foundry cryotechnologies there is an analogy here: freezing of the sand sample is comparable with processes of frozen sand mold obtaining, and freezing of the water-saturated clay sample – with ice pattern obtaining on freezing an aqueous composition in the mold.

III. RESULTS AND THEIR DISCUSSION

To study the process of aqueous composition freezing under conditions typical for an experimental production of casting patterns, we measured the temperature during cooling of the samples in the air inside the freezer at the temperatures $-17 \dots -15^{\circ}\text{C}$. In a cylindrical vessel from flexible thin-walled plastic with a diameter of 100 mm three types of samples were frozen, weighing 1 kg each: one from tap water and two aqueous compositions with binders, 25% solution of liquid sodium glass (the density of 1.08 g/cm^3) and 25% solution of dextrin (the density of 0.98 g/cm^3) respectively. The temperature was measured in the sample center; the initial temperature was $+3^{\circ}\text{C}$.

Analysis of the curves shows that all three samples started to cool simultaneously in 5 minutes after having been placed in the freezer. Cooling of the liquid glass composition was a little faster than cooling of the other two compositions due to higher thermal conductivity. The graphs show that a gradual decrease in temperature is followed by small horizontal portions. The appearance of the first horizontal portions on the

left, close to 0°C , is connected with the transition of the water from liquid to solid state, when a release of latent heat of the ice formation occurs. Since in the sample center the liquid was frozen in the last turn, its temperature before freezing was fixed at fractions of a degree higher than 0°C . Some synchronous violation of the curves smoothness may be explained by a periodicity of the camera compressor turning on-off, by processes inside the solutions or by the measurement error.

The obtained results showed that the composition with the addition of liquid glass was crystallized in 30-32 min., faster than two others, which solidified approximately within the same time limit (40-45 min.). Power inputs of the freezer Nord-155 with 200 litres volume for maintaining the inside temperature below -18°C upon cooling of 1 kg of ice to the measured temperature $-12 \dots -11^{\circ}\text{C}$ during 3 hours are not more than 0.18 kWh. This can be used for calculating the productivity of freezers during obtaining patterns in the experimental production.

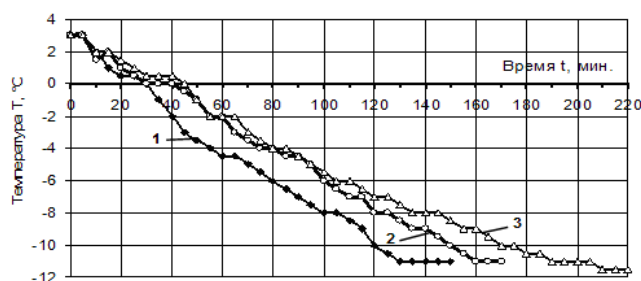


Fig. 2. Graphs of freezing of water samples with different components: 1 – 25% liquid glass, 2 – tap water, 3 – 25 % dextrin

For modeling of the process of aqueous solution crystallization in the process of obtaining the ice patterns, we briefly recall some well-known regulations, considered in more details in the recent publications [2, 4, 5]. This process starts with a release of crystals nuclei from the supercooled solution and the subsequent crystallization occurs upon further cooling of the solution on the formed nuclei. Obtaining large crystals in the course of crystallization indicates that the number of nuclei, formed at first, is relatively small because the distribution of newly released crystals on cooling the solution on a large number of nuclei leads to the formation of small crystals. The higher is the cooling rate, the more such crystal nuclei are formed in the solution and the smaller is the size of the formed crystals. I. Andreev proposed to determine the crystal growth rate V (in g/sec) at the formed centers of crystallization from the equation [5]:

$$V = \frac{dx}{dt} = kS(x - x'),$$

where $\frac{dx}{dt}$ is the amount of substance, crystallized in a unit of time; k is the coefficient depending on the diffusion of molecules, which reached the solidification temperature d ,

and on the average length of the diffusion path δ ; $k = \frac{d}{\delta}$; S is the surface of the released solid phase; x is the supercooled solution concentration; x' is the solubility of crystals nuclei at a given degree of their dispersibility.

The mechanism of growth of ice crystals in water is most adequately reflected in the cluster model of H. Frank and V. Wan [2]. According to this model, water is a conglomerate of large associates of molecules H_2O (clusters), appearing and then dissociating. X-ray and IR investigations of water showed that molecules arrangement inside the clusters was similar to their arrangement in the crystal lattice of ice. A decrease in temperature leads to an increase in the number of molecules in the cluster. The average number of molecules in the cluster is 15 ... 50 units at the temperature $65^\circ C$, 90 ... 120 units at $0^\circ C$ and 150 ... 180 units at $-20^\circ C$. Thus, according to this model, even before the formation of the first stable ice crystals (Fig. 3a), there are ice-like formations in the water [2].

The growth of ice crystals occurs not constantly as a result of deposition of separate molecules on the edges, but by jumps due to addition of separate blocks of approximately 10^{-7} - 10^{-3} cm. This causes a change of crystallographic axes in some micro-blocks for a few angular minutes and even for a few degrees. In crystals, formed in the conditions of supercooling down to $-10^\circ C$, a disordered orientation of separate blocks reaches 1-3 angular degrees, and in the case of small supercooling such disordered orientation is not more than fractions of a degree. However, the mechanism of cluster growth does not exclude the possibility of joining of separate molecules to the growing crystal face, although such process is obviously a secondary one, especially in the case of significantly supercooled water. Crystal growth can occur not only in a solution but also in gaseous medium, then water molecules from the vapor are precipitated on ice, for example, forming snowflakes in nature (Fig. 3b).

In the paper [4] the Deryagin's expression for calculation of the growing ice mass flow is given. The rate of the ice crystal growth depends on the substance inflow rate and the rate of the crystallization heat removal. Based on the consideration of the kinetics of phase interaction in the area of the boundary of phase transitions, S. Grechishchev obtained the following expression [2]:

$$v_w d(Cq_w) = -Q \frac{dT}{T} - v_{ice} dG^{sk} - (v_{ice} - v_w) dP,$$

where v_w and v_{ice} are specific volumes, respectively, of the water film and of the ice, q_w is the moisture flux, C is the coefficient, G^{sk} and P are the pressures, respectively, in the ice and in the water film. It is easy to see the similarity of these two expressions.

In recent works on modeling, clusters are described as fractals [4, 5] and clustering is considered as one of the ways of fractals appearance in irreversible processes. Here we should mention the Sander's model with limited diffusion of aggregation (LDA) [6]. Unordered, chaotic growth of the crystal, identified with LDA, brought to a finite limit, is a

consequence of motion of randomly wandering particles to the cluster that generates fractal structures.

One of the experimental examples, confirming the adequacy of LDA, is electrolytic deposition of metals on a small electrode. For the purpose of clarity, usually first a two-dimensional model of the crystallization of the type "particle-cluster" is built and after a more complicated model "cluster-cluster" is used [5]. Here, it is assumed that the cooling rate is high enough to neglect the mentioned solubility x' of the formed crystal nuclei at a given degree of their dispersibility. The same assumption is used in constructing a three-dimensional model, which is already compared with experiments. Modeling "particle-cluster" on the square lattice of the size $L \times L$, $L = 10 \dots 1000$ units is carried out being subject to three following rules: 1) initially there is a single point nucleus, which is a central cell of the lattice; 2) a particle is defined as a square cell with a unit side; the particle starts from a random cell on the edge of the lattice and performs a random walk on the lattice as long as it touches a cluster particle or a particle from a group of neighboring particles; 3) the process is terminated as soon as the growing cluster reaches the edge of the lattice. Rule 1 models the growth of only one cluster, rule 2 allows only dendritic clusters without appearance of loops, the 3 stops numerical modeling.

The dendritic growth is shown with the help of the LDA model with one seed particle in the center of the square lattice (Fig. 3c). Here, connection of the particle with the cluster can be weak (only one bond with the cluster), medium (2 bonds) and strong (3 bonds). The number of bonds is defined as the number of the particle vertices, touching the cluster. It is easy to prove that the number of bonds in this model can not exceed 3.

Fractal dimension D_0 of the set X is numerically defined as follows. We cover X with a square lattice with the cell side ε . Then we calculate the number $N(\varepsilon)$ of cells, covering the set X . Now, by definition:

$$\dim_M X = D_0 = \lim_{\varepsilon \rightarrow 0} \frac{\ln N(\varepsilon)}{\ln \varepsilon^{-1}}.$$

In the two-dimensional case the fractal dimension of dendrites is close to 1.7. This means that the aggregate mass increases as $L^{1.7}$, where L is the linear dimension. Therefore, the average density changes as $\frac{L^{1.7}}{L^2} = L^{-0.3}$, i.e. decreases, in

the full accordance with appearance of these forms of growth. In the three-dimensional LDA the fractal dimension is usually close to 2.5.

At LDA separate particles settle on the aggregate after some random walks, generating fractals, which model the growth of ice dendrites in water. They are characterized by a tree-like structure with numerous "fjords" on many size scales that is also comparable with the snowflake construction (Fig. 3 b,c). The cause of the formation of such structures at LDA is that a walking particle usually settles near the fractal ledge, but not in the depth of the fjord, as shown in detail below.

To calculate the probability of the particle joining to the cluster, we note that the particle, being a square, can have one,

two or three common vertices with the cluster. In Fig. 3d the cluster, consisting of three particles (the dark colored cells), is shown. The walking particle can join the cluster, when it comes into one of the 12 white cells. The number of one-step ways, by which a particle can get to the white cell, is recorded inside the cell. Among the 12 white cells there are 5 corner cells, 6 middle cells and 1 fjord.

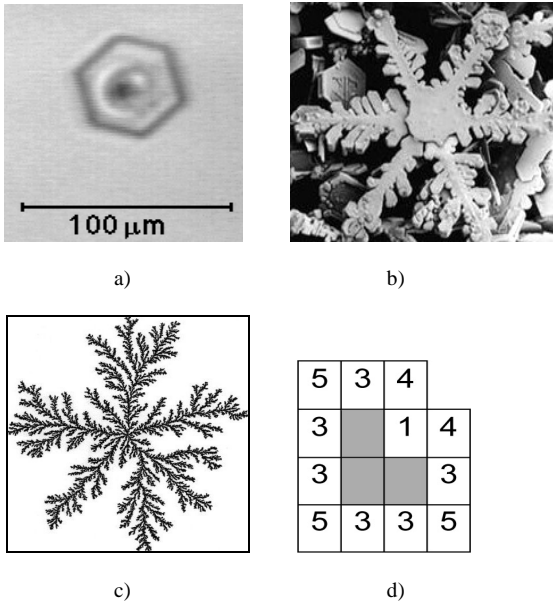


Fig. 3. Crystal structures and their models: a) the photomicrograph of the hexagonal ice crystal, grown from a nucleus [2]; b) a snowflake under a microscope [2]; c) a cluster in the LDA model [5]; d) the scheme of the cluster from 3 particles with probabilities of the particle joining to the cluster [5]

The corner cells have one bond with the cluster, the middle cells - 2 bonds, the fjord - 3 bonds. There are 42 one-step routes, leading to the white cells from the outside, therefore the first three probabilities p_k that the particle gets into the white cell with k bonds are:

$$p_1 = \frac{23}{42} \approx 0.5476, \quad p_2 = \frac{12}{42} \approx 0.4286, \quad p_3 = \frac{1}{42} \approx 0.0238.$$

It is clear that the particle has small chances to join the cluster inside the fjord. This means that the concentration of ice particles is less for the crystallization inside the fjord, since many particles have already joined the cluster. On the contrary, the concentration is high in the neighborhood of the ledges, as far as few particles from this neighborhood joined the dendrite so far.

The similarity between the constructions of ice dendrites and LDA forms is explained by the fact that both processes are described by the Laplace equation from potential theory where the potential gradient corresponds to the field of diffusion in LDA and the cluster surface of LDA corresponds to the equipotential surface. With this approach to LDA it follows that particles will "stick" with bigger probability to the parts of the cluster, where the potential gradient is high, i.e. near the ledges. Ice dendrite growth usually occurs in the direction of maximum potential gradient. On the contrary, deep fjords (Fig. 3 b,c) are well shielded and therefore they either grow very slowly, or do not grow at all. Such correspondence

between potential theory and the described mechanism of fractal growth of the crystals was fully justified by accurate measurements and numerical solutions of the potential equation.

Forms of ice crystals, growing in solutions on a solid basis, which is here the wall of the mold, are peculiar. With a wall thickness of ice models about 10 mm, the cooling rates are quite high for freezing inside Nord-155 freezer at the above indicated temperatures. Therefore, the dendritic structure shown in [4] is rarely possible in the center of the model wall. Usually axes of crystals of the second and third order stop to form and needle (banded) forms of ice crystals (Fig. 4a) appear. The last ones grow on the base mainly by the principal axis along the normal to the base, according to the so-called Bertin's law [2], in conditions of heat transfer in this direction when supercooling into the stage of protocrystallization is limited by more or less thin layer of water, adjacent to the base. The thinner is the supercooled layer at the time of crystal nucleation, the more monotonous is the orientation.

The same banded structure of ice crystals can be seen as a result of crystallization from water with impurities of charcoal particles, located with more or less equal intervals in the thin layers between the crystal planes (Fig. 4b).

Sometimes dissolved or emulsified technological additions are injected into the ice pattern composition, for example, a binder for obtaining a shell mold by impregnation of the sand contact layer at the pattern melting. Fig. 4c,d illustrate the distribution of such impurities. In Fig. 4c the ice of sea-water is shown [2].

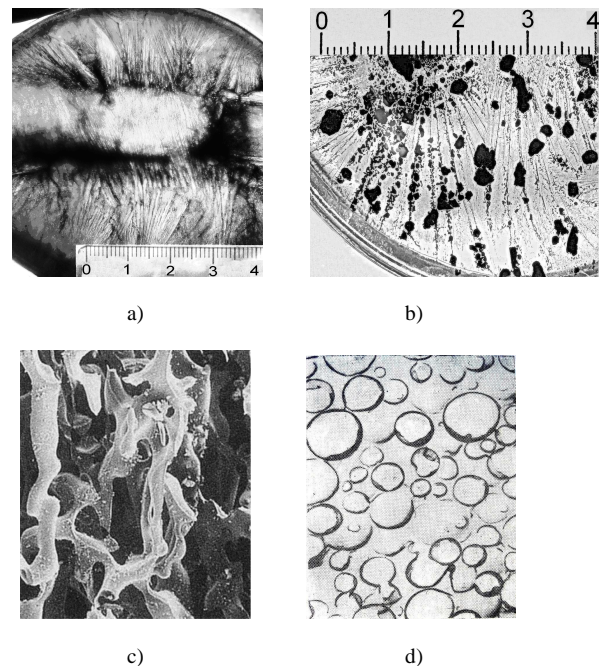


Fig. 4. Ice samples: a) ice with additions of ink; b) ice with additions of charcoal grains; c) ice of sea-water; d) electronic-micrograph of the frozen emulsion chip

During the cooling of the solution, separate formation of ice lattices occurs for each of the salts. Here, the solution-melt delamination occurs, and salts are displaced to the periphery of the area of nucleus growth and then to the periphery of the

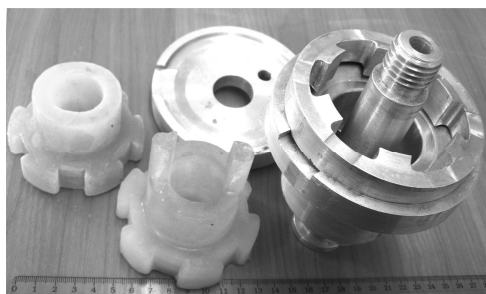
area of the ice crystal growth. "Squares" and "channels" are formed around the crystals from the cells of the solution with the increased concentration. Mineral impurities and dissolved salts in the process of growth are distributed among elementary plates inside the crystals and between them.

In salty ices, the brine forms layers in basal planes of crystals that separate the crystals on a number of plates. The faster the crystallization goes and the higher the concentration of salts is, the thicker are the layers of brine (up to several millimeters at the temperature close to 0°C) and the thinner are the separated elementary plates of ice. The greater is the concentration of impurities in the solution, the smaller is the distance between adjacent layers of inclusions in the crystal.

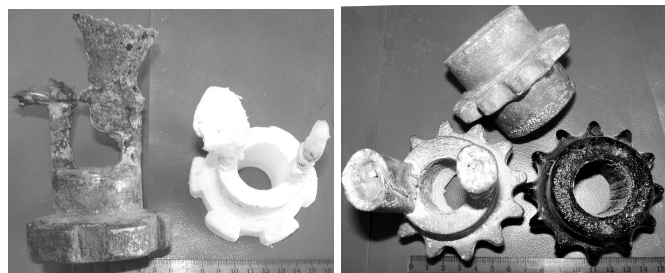
In Fig 4d the electronic-micrograph of the chip of the frozen emulsion of the type "water in oil", formed by the introduction of water into the melt of the polyurethane monomer, is presented [7]. In this process, the particle size of the dispersed polyurethane latex is $0.03 \dots 500 \mu\text{m}$.

Advances in technology of water-dispersion varnishes and resins, based on the progress in the emulsion polymerization field as well as on investigations of colloid-chemical properties of the polymer dispersions and film-forming mechanism, allowed obtaining a number of new water-dispersion binder materials, suitable for freezing in ice casting patterns. Scientists-casters have yet to work out technologies of application of these binders for obtaining of shell molds around such patterns, including in pairs with fast-acting hardeners. Several examples of such ice patterns production are shown in Figure 5.

In Fig. 5a the ice patterns from the service water are shown; in Figure 5b – the pattern from the above mentioned 25% solution of liquid glass as well as the casting, obtained from cast iron with lamellar graphite of ultimate tensile strength 20 kgf/mm; in Fig. 5c – the ice pattern from an aqueous solution of technical lignosulphonates (TLS) and two castings from non-ferrous alloys.



a)



b)

c)

Fig. 5. Ice patterns: a) of the half-coupling with an open mold; b) of the half-coupling with the casting; c) of the pinion with two castings

TLS are cheap and not scarce organic aqueous binders that provide good peeling from the molds, including the molds on the basis of cold-hardening mixture (CHM). In further studies it is planned to develop the technology of obtaining of shell molds, which are hardened, similarly to CHM, by chromic anhydride Cr_2O_3 or by other compounds with hexavalent chromium in the amount of 0.3-0.7% of the mixture weight. TLS are used in the molding mixtures in the amount of 3-5% (less often up to 8%), in combination with clay or natural molding clay sands in the amount of 2-3%. When introducing TLS into the pattern composition, it is diluted with water to a density of not more than $1.17\text{-}1.20 \text{ g/cm}^3$, the pattern temperature is reduced to $-20 \dots -17^{\circ}\text{C}$, and the powder hardener is placed into the coating sand layer.

When we inject organic binders into the material of ice patterns, it is appropriate to take into account the possibility of occurrence of the biological cryoprotectors action. In medical and laboratory practice about a dozen of these compounds are used, which act similar to proteins, preventing ice crystallization in the cell solution. It is considered that they are adsorbed on the surface of the ice crystal, and thus prevent it from growing. This has been observed on the obtained samples with dextrin in the form of fine-grained structure. For some samples, obtained under identical conditions with the sample from Figure 4a, where the grains were visible with the naked eye, the mesh of intergranular boundaries was so fine that it was not visible without increasing.

IV. CONCLUSIONS

In this paper a range of issues related to the mechanism and kinetics of ice solidification in the process of obtaining a single-use casting pattern as well as distribution of impurities in ice have been considered. Analysing the example the application of advanced mathematical methods of crystallization visual modeling in view of theory of clusters and fractals has been shown. Examples of casting patterns and their structures are given. The physical component of the modeling allows describing the growth of ice formations during the pattern forming. The mathematical component makes it possible to build and study the numerical model, explaining mechanisms and causes of crystals growth. For the technological use of the results, aspects of their adequate interpretation in terms of the physical model are important, as well as some obtained numerical characteristics and proposed research areas to bring the CIP technology to experimental and industrial applications.

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