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Kinetics of Solidification and Crystallization of Liquid Axial Zone of Fe-C Alloys in Cylindrical Molds

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Abstract

According to the results of digitization of the experimental studies carried out in the past concerning Fe-C alloys solidification in cylindrical molds of castings with a carbon content of 0.04%, 0.1%, 0.4%, 0.93%, 1.42%; 2.44%, 3.28%, 4.45%, 4.83% and their subsequent interpolation in the range of 0.04 \div 4.83%C there were obtained the curves of the advancement of the pour point, liquidus and solidus in the coordinates of the relative thickness of the solidified metal layer x/R and the parametric criterion t/R^2 . Their usage is proposed for the development of modes of physical and chemical influence on the liquid metal in the axial zone of the casting after solidification of its calculated layer. Calculation of the mass of modifiers or deoxidizers for introduction into the axial zone was performed in relation to the total mass of metal in the liquid and liquid-solid zones of the casting. The technique for calculating the mass and time of introduction a graphitizing modifier into the axial zone of rolling rolls made of hypereutectoid steel with 1.7%C is proposed to reduce the negative impact of cementite, chromium and molybdenum carbides on the structure of the axial zone of the rolls. The obtained curves can also be used to assess the accuracy of computer modeling of the processes of Fe-C alloys solidification and further adaptation of mathematical models by the correction of thermophysical coefficients, the values of which are not always known in the liquidus-solidus temperature range.

Keywords: Fe-C alloys, Solidification, Kinetics, Pour point, Modification, Axial zone, Liquid-solid

1. Introduction

Unlike alloying, the effect of the modifying is limited in time. This determines the introduction of the modifier into the liquid metal in a ladle or in the feeding system. There are known methods of obtaining castings with different macro- and microstructures over their thickness [1, 2] when modifying alloys in the feeding system at the in-mold process with placing the ligature in a special reaction chamber with a centrifugal slag collector [3, 4]. The metal

flow melts the ligature while filling the mould. Slag and nonmetallic inclusions float into the upper part of the reaction chamber and the modified metal enters the casting through the feeder in the lower part of the reaction chamber [5]. The disadvantage of this technology is an increase in the consumption of metal in the reaction chamber, the need to use components of the charge with a minimum sulphur content, and the impossibility to influence to the process of crystallization of the liquid axial zone of the casting.

Therefore, the introduction of a modifier into the liquid core of the casting after filling a mold and solidification of the working



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layer of the given chemical composition is one of the effective methods of influencing the structure of the metal. The solubility of gases decreases, bubbles, shells and other defects are formed with the beginning of crystallization of the alloy. However, it is necessary to know the kinetics of solidification of the casting in order to deoxidize the metal, refine the microstructure, modify and alloy the liquid metal in foundry.

Unfortunately, it is not always possible to calculate the kinetics of moving liquid metal front, i.e. pour point inside the forming casting by mathematical modeling [6-8].

According to the known scheme of crystallization (Fig. 1) there are three zones coexisting in the castings in the range of liquidus-solidus temperature: a liquid phase in the center, a liquid-solid zone that provide fluidity, and a solid-liquid zone. Filtration of the melt between dendrite branches is realized in the last zone, where shrinkage porosity is formed (1, Fig. 1) [9,10].

Liquidus and solidus temperatures have been determined for a range of Fe-C alloys. However, the kinetics of changes in the liquid-solid and solid-liquid zones along the thickness of the casting, the position of the pour point between these zones are known only for certain alloys that are cooled in molds and castings.

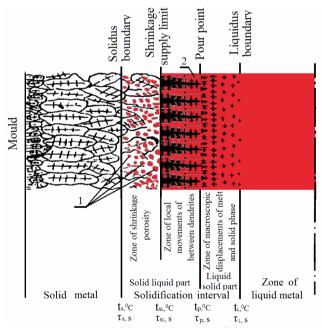


Fig. 1. Scheme of the crystal structure formation and advancement of the solidification area [5] with additions: t_s and τ_s , t_s L and τ_s , t_p and τ_p , t_L and τ_L are the temperature and time of solidus, feeding, pour point and liquidus boundaries in the casting, respectively; 1- volumetric solidification of residual melt and formation of shrinkage porosity; 2- direction of movement of liquid phase for shrinkage feeding

Carrying out such studies requires combination the temperature measurement throughout the thickness of the casting and the pour point of the final-solid phase at various stages of solidification for establishing the position of the pour point fronts. The lack of data on the duration of Fe-C alloys in the liquid-solid and solid-liquid state (see Fig. 1) does not allow to regulate the solidification

processes of castings, to develop methods of intervention in the process of crystallization of the central zones of castings to improve their physical and mechanical properties.

The experimental studies of castings solidification in cylindrical iron moulds were presented in [9, 10]. The temperature measurements by thermocouples were combined with the determination of pour point of the liquid phase at various stages of solidification and with the introduction of radioactive isotopes W¹⁸⁵, Ag¹¹⁰, Ca⁴⁵, Co⁶⁰, Zn⁶⁵, Zr⁹⁴ in the form of ligatures to clarify the position of the pour point boundary. The ligature was prepared based on the chemical composition of the studied Fe-C alloy, with small additives of the radioactive isotope. When performing the experimental studies, the ligature was melted in a separate furnace and introduced into the liquid core of the castings at different stages of their crystallization to establish isotope filtration between the dendrites in solid phase.

Solidification of Fe-C alloys was studied for carbon content of 0.04%, 0.10%, 0.40%, 0.93%, 1.42%, 2.44%, 3.28%, 4.45%, 4.83% and with a minimum amount of impurities of other chemical elements. For example, the kinetics of solidification of cylindrical castings with 0.10% and 0.40% carbon is shown in Fig 2.

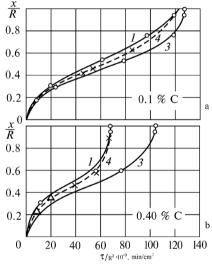


Fig. 2. Experimental curves of solidification kinetics in the mold of cylindrical castings with a carbon content of 0.10% (a) and 0.40% (b) [9,10]: abscissa axis $-\tau/R^2\cdot 10^{-3}$, min/cm²; x/R—ordinate axis is a relative thickness of solidified metal; 1 – kinetics of the liquidus boundary movement; 3 – kinetics of boundary movement at the end of "standing" solidus; 4 – kinetics of movement of the pour point boundary; o – temperature measurement; x – pour point; Δ – introduction of radioactive isotopes

The results of the experiments are presented by the curves of the kinetics of solidification of the liquidus (1 in Fig. 2), pour point of the liquid metal residue (4) and solidus (3) in the coordinates of the relative thickness of the solidified layer x/R and τ/R^2 – the parametric criterion of B.B. Gulyaev, where τ is the solidification time; R – casting radius; x is the thickness of a solidified metal layer [9]. However, the results of nine experimental studies [9,10] were not extended for all Fe-C alloys composition in the range of $0.04 \div 4.83\%$ C.

A comparison of the introduction of radioactive isotopes with the method of pour point a liquid core at different stages of solidification of castings with 0.40%C, 0.93%C, 1.42%C, 2.42%C showed that the isotope spreads in the liquid area and in the liquid solid part (see Fig. 1). Movement beyond the pourability limit was not observed, with the exception of alloys that crystallize in a wide temperature range [9, 10].

Gulyaev B.B. showed for the first time [11] that the determination of the hardening coefficient based on the experimental crystallization temperatures of castings combined with the method of casting unhardened metal can be represented as a dependence between values in which the Fourier criterion $(Fo=at/R^2)$ is replaced by the parametric criterion t/R^2 . With a constant material of castings, molds, metal overheating, the calculation of the solidification time can be performed according to Eq. 1:

$$\tau = Fo \frac{c\rho}{\lambda} R^2 = Gu \cdot R^2, \tag{1}$$

where

$$a = \frac{\lambda}{c\rho}$$
; $\frac{\tau}{R^2} = Fo \frac{c\rho}{\lambda}$; $Gu = Fo \frac{c\rho}{\lambda}$, a, c, ρ — thermal conductivity,

specific heat capacity and density of the alloy, respectively; τ – solidification time; x is the thickness of the solidified metal layer; R is the determining size of the casting.

The criterion τ/R^2 allows for a quantitative assessment of the solidification process. It relates the solidification time (τ) to the geometric dimensions of the casting (R), providing a normalized measure that can be used to compare solidification behaviours across different casting sizes and shapes. By using τ/R^2 , the study effectively characterizes the movement of the solidus and liquidus boundaries during the solidification process. This helps in understanding how the solidification front advances and how it is influenced by factors such as carbon content and temperature. Also

Gulyaev coefficient Gu summarizes the thermophysical properties of the alloy, which change during the solidification of the metal layer x/R from the surface (x/R=0) to the center (x/R=1) of the casting, and in the known equation of the square root $(\tau=k^2/x^2)$ [12] coefficient k is constant during the solidification of the alloy from the surface to the center. Therefore, the calculation using the square root equation does not include the accelerated advancement of the crystallization front in the axial zone of the casting.

The value of the latent heat of crystallization, the irregularity of its release at liquidus and solidus temperatures, and the coefficients λ , c, ρ are not always known for Fe-C alloys in the range of crystallization temperatures. Equation (1) is a first approximation in technical calculations and such a processing scheme for experimental studies with a constant material of castings, foundry molds, and overheating was successfully used by different scientists [13-18]. For example in the study [14] the parametric criterion τ/R^2 was characterized as tangent to each point on the curve of the kinetics of the advancement of crystallization fronts. Calculations according to this method allow to establish technological regimes of influence to the crystallization process of the casting in molds.

Therefore, the purpose of the work is to digitize carried in the past the experimental thermographic measurements of solidification in cylindrical molds of Fe-C alloys castings with extended carbon content of 0.04÷4.83%, i.e. for 0.04%, 0.10%, 0.40%, 0.93%, 1.42%, 2.44%, 3.28%, 4.45%, 4.83%, establishing the thickness of two-phase liquid-solid and solid-liquid zones in ingots and castings and development of modes of influence on the crystallization of the axial zone after solidification of a given metal layer.

2. Experimental methods

The experimental data are of the original studies [9,10] in which Fe-C alloys were smelted in the 500 kg induction furnace using charge with armco iron and carbon to exclude the influence of alloying elements. Prior pouring to the mold the melt was overheated of 6-7% above the liquidus temperature. Six Pt-Rh (30% Rh) – Pt-Rh (6% Rh) thermocouples were placed at the middle height of cylindrical castings with a diameter of 250 mm (Fig. 3). The data were recorded with the EPP-09 potentiometer with an accuracy of 2–3°C and an interval 5 and 3 seconds between points. Castings were cooled in a cylindrical cast iron mold with a wall thickness of 75 mm. The kinetics of pouring was studied by the introduction of a radioactive isotope at different time intervals and pouring of the remaining melt from the mold by its turning out. Every alloy was poured out 3-5 times and then cooled with molds in a caisson.

Digitization of the experimental curves of solidification kinetics was carried out using the AutoCAD 2010 software after scanning and enlarging the curves [19]. The relative thickness of the solidified metal layer x/R was measured in increments of $0.05x/R \pm 0.001$, the parametric criterion τ/R^2 with an accuracy of $\pm 1\%$, and an example of the curves for calculating τ/R^2 of the kinetics of advancing the solidus boundary for an alloy with 0.40%C is given in Fig. 4.

The obtained array of 1134 data points was entered into the Microsoft Excel program. Graphs were built to establish the influence of the carbon content on the parametric criterion τ/R^2 of the solidification of the pour point fronts, liquidus and solidus in the range of chemical compositions of $0.04 \div 4.83\%C$.

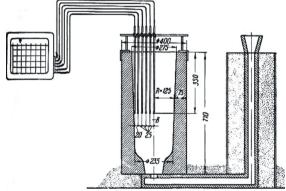


Fig. 3. The scheme of the casting mold and the placement of thermocouples in a cylindrical mold [10]

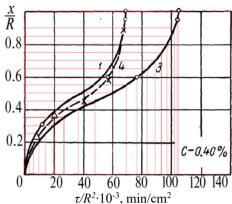


Fig. 4. Example curves of digitization of Gulyaev parametric criterion (τ/R^2)

3. Results and discussion

It is necessary to know the mass of liquid metal remaining after the solidification of the metal layer on the casting to establish the modes of influence on the process of crystallization of the axial zone of the casting. In addition, there is a need to set the start time of introducing the modifier or graphitizing elements into the axial zone. However, knowledge of the kinetics of advancing the solidus boundary (100% solid phase) along the thickness of the casting cannot be the basis for an accurate calculation. Thus, the liquid phase from the core of the casting does not provide melt for shrinkage in the zone of microscopic displacements (see Fig. 1), and in the zone of local displacements only minimal filtration of the melt between the branches of the dendrites is possible, as established by the distribution of radioactive isotopes in [5, 6]. Only the determination of the kinetics of the advancement of the pour point boundary allows for a more accurate calculation of the volume and mass of liquid metal in the liquid-solid and liquid sections (see Fig. 1), and then to calculate the mass of chemical elements to be injected into the axial zone of casting.

According to nine experimental thermographic studies [5,6], the kinetics of the solidification of the pour point front in Fe-C alloys was calculated in the coordinates τ/R^2 and x/R, their interpolation in the range of $0.04 \div 4.83\%$ C and with 20 curves of the relative thickness of the solidified metal layer with a step of $0.05 \ x/R$ – from the smallest one to the center of the cylindrical casting x/R=1 (Fig. 5).

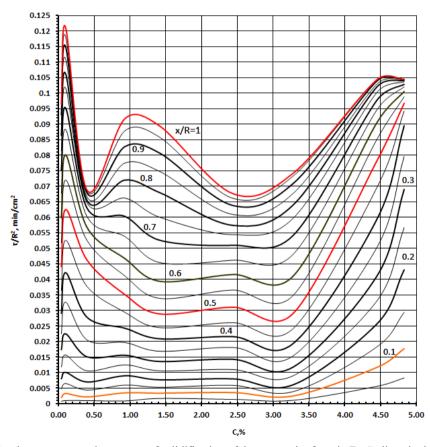


Fig. 5. The influence of carbon content on the process of solidification of the pour point front in Fe-C alloys in the range of 0.04÷4.83%C during cooling in the mold

The maximum and minimum values of the duration of the formation of the pour point boundary can be explained by the calculations of the amount of solid phase (m) at a temperature 5^{0} C below the liquidus, temperature (Δt) and concentration (ΔC_{uhm}) intervals of solidification (Fig. 6) [20].

Thus, at 0.1% carbon, the amount of the solid phase is m $\approx 65\%$, at 0.45%C m $\approx 6\%$, which coincides with the first maximum and minimum in Fig. 5. As the carbon content increases to 0.5%, the amount of the solid phase increases, and then gradually decreases to zero at point C with a carbon content of $\approx 4.3\%$. The solidification concentration interval increases to a maximum at point C, and the solidification temperature interval is maximum at $\approx 2\%$ carbon and decreases to zero at point C.

To find the influence of all three indicators on the kinetics of casting solidification pour point (Fig. 5), liquidus (Fig. 7) and solidus (Fig. 8) requires a separate analysis and further research.

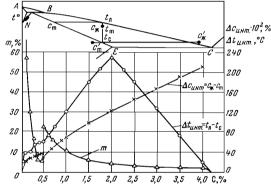


Fig. 6. Change in the amount of the solid phase (m) at a temperature 5°C below the liquidus, temperature (Δt_{umm}) and concentration (ΔC_{umm}) of the solidification intervals depending on the carbon content in Fe-C alloys [20]

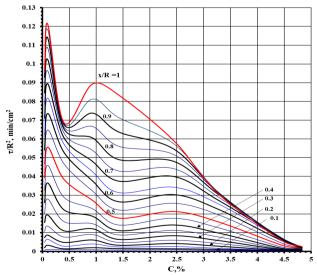


Fig. 7. The influence of carbon content on the process of solidification of the liquidus boundary in Fe-C alloys in the range of 0.04÷4.83%C during cooling in the mold [19]

The duration of solidification at liquidus (τ_L) is maximum at a minimum carbon content. Thus at C=0.1%

$$\tau_{0.1L} = 0.1211 \cdot R^2 \text{ (min)},$$
 (2)

and when carbon increases to 4.45%

$$\tau_{4.45L} = 0.0059 \cdot R^2 \text{ (min)}.$$
 (3)

The obtained results can be explained by the fact that in steel castings (C=0.1%) thermocouples record a long temperature stop at the liquidus temperature, and at the solidus temperature only a bend is visible on the cooling curve.

This indicates a greater share of crystallization of the solid phase and release of latent heat of solidification at the liquidus temperature, which increases the duration of cooling at t_L . On the contrary, during the solidification of pre-eutectic cast iron, the thermocouples register a long temperature stop at the solidus temperature and the release of a larger share of the latent heat of crystallization.

A comparison of the effect of carbon content on the total duration of solidification of the solidus boundary (τ s) showed (Fig. 8) that at C=0.1%:

$$\tau_{0.1S} = 0.12575 \cdot R^2 \text{ (min)},$$
 (4)

and with an increase in carbon to 4.45%

$$\tau_{4.45S} = 0.1800 \cdot R^2 \text{ (min)},$$
 (5)

which is 30.5 times greater than that of liquidus.

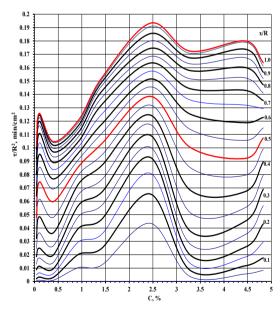


Fig. 8. The effect of carbon content on the process of solidification of the solidus boundary in Fe-C alloys in the range of 0.04÷4.83%C during cooling in the mold [21]

Thus, according to the data, cast irons with a carbon equivalent greater than 3.5 contain only 2-3% of the solid phase at a temperature 5°C below the liquidus (see Fig. 6) and have a higher fluidity compared to steel [20].

The above curves of the solidification kinetics of Fe-C alloys can be used to calculate the solidification of alloys with impurities of other chemical elements up to $\approx \!\! 1\%$ using known formulas for the carbon equivalent (C_E), which establishes the position of the chemical composition in relation to the eutectic point [23]. For example, in the first approximation for steels:

$$C_E = C + Mn/6 + (Cr + V + Mo)/5 + (Ni + Cu)/15$$
,
and for a cast iron

$$C_E = C + 0.3 \text{ Si} + 0.33 \text{ P} - 0.027 \text{ Mn} + 0.4 \text{ S}$$

As it is noted in [22], no commercial computer software can accurately simulate the solidification process and take into account the influence of all thermophysical coefficients varying in the range of solidification temperatures. Therefore, it is reasonable to use the calculations of the kinetics of the advance of the solidification fronts of liquidus, solidus and pour point of liquid metal obtained experimentally to assess the accuracy of computer modeling of the processes of solidification of Fe-C alloys with carbon content of 0.04÷4.83%. It is necessary for the adaptation of mathematical models by correction the thermophysical coefficients for castings and mold material.

The obtained standards of the Fe-C alloy solidification process can be used for manufacturing real castings of different configurations, pour point temperatures, etc.

Calculations of the duration of solidification of the casting fronts (Fig. 5), liquidus (Fig. 7) and solidus (Fig. 8), which are based on the relative thickness of the solidified metal layer x/R, allow us to establish the kinetics of solidification of castings with a certain chemical composition. Thus, the constructed curves of solidification kinetics in molds of cylindrical castings made of Fe-1.7%C alloy are presented in Figure 9.

They are used in the production of rolled rolls from hypereutectoid steel [24]. The rolls of eutectoid steel with 1.5÷ 1.7% carbonare of high wear resistance and cheaper than forged ones, with carbon content less then 0.85÷0.95%. However, the disadvantage of cast rolls is a decrease in temporary resistance, relative elongation and impact toughness due to the formation of a continuous carbide network around the austenite grains [25].

According to the obtained data (3 in Fig. 9), the duration of solidification of 100% of the solid phase (τ_8) in the center of the casting corresponds the value of the parametric criterion τ/R^2 for solidus:

$$\tau/R^2 = 16.50 \cdot 10^{-2} \text{ min/cm}^2, \tag{6}$$

and for casting Ø400 mm (R=20 cm), the duration of solidification at x/R = 1.0 is equal to:

$$\tau_{1.7S(1.0)} = 16.50 \cdot 10^{-2} \cdot (20)^2 = 66 \text{ min}$$
 (7)

Thus, the minimum duration of Ø400 mm ingot existence in the mold is 66 minutes, which allows then to move or disassemble the mold without liquid metal pour point from it. According to [13,14] the ratio of the duration of solidification of castings in the form of a plate (τ_P) , a cylinder (τ_C) , and a sphere (τ_{SP}) is in the first approximation $\tau_P : \tau_C : \tau_{SP} = 1 : 0.25 : 0.11$, which allows to estimate the duration of solidification of individual parts of the shaped casting.

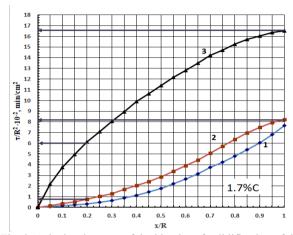


Fig. 9. Calculated curves of the kinetics of solidification of the liquidus (1), pour point (2) and solidus (3) fronts in the casting of Fe-1.7%C alloy cooled in a cylindrical cast iron mold

The square root law (one of the solutions to Stefan's problem [12]) establishes the thickness of the solidified metal layer for a flat semi-infinite casting. Such calculation of a steel flat casting ($\tau = x^2/k^2$) at x=20 cm and the coefficient k=0.30...0.35 cm/s^{0.5} [26] gives the solidification time of 74 ... 54 min. The calculation using the ratio in [22] shows that spherical casting with a radius of 20 cm solidifies during 8.14 ... 5.9 minutes, and a cylindrical casting – during 18.5 ... 13.5 minutes, which is 3.6 - 4.9 times less compared to the experimental and calculated data according to formula (7).

While alloys solidification in the mold the solubility of gases decreases with bubbles formation. In addition non-metallic inclusions move to the liquid axial zone. So, it is reasonable to suggest introducing a deoxidizer or modifier into the central part of the casting, which has not yet solidified, to reduce their negative impact on the metal, after solidification the necessary working layer on the casting. The gradual melting of these additives will ensure a smooth change in the chemical composition, micro- and macrostructure between the working layer and the axial zone of the casting.

For example, to produce the Ø400 mm cylindrical ingot of the Fe-1.7%C alloy (Fig. 10), it is reasonable to assume the working layer thickness x = 40 mm. Then the ratio x/R = 0.2, and the time of penetration of the pour point boundary is $\tau_{1.7V(0.2)}$ it is possible to determine according to the graph (2 in Fig. 9):

$$\tau/R^2 \cdot 10^{-2} = 0.75425 \text{ min/cm}^2,$$
 (8) or $\tau_{1.77(0.2)} = 0.75425 \cdot R^2 \cdot 10^{-2} = 0.75425 \cdot 20^2 \cdot 10^{-2} \approx 3 \text{ min } 1 \text{ s}$

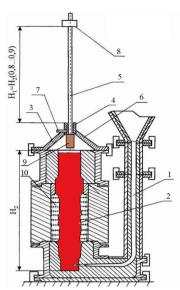


Fig. 10. Casting mold for the production of two-layer sheet rolling rolls with a device for modification, immersed in the lower neck [27]: 1 – gating system; 2 – liquid metal remaining after solidifying of the working layer; 3 – device for modifier introducing; 4 – guide pipe; 5 – a bar with a modifier on the lower part; 6 – support; 7 – bar movement lock; 8 – height limiter for the movement of the modifier in liquid metal; 9 – aluminum; 10 – a layer of metal solidified on the mold before aluminum was immersed

Next, it is necessary to calculate the mass of liquid metal (2 on Fig. 10) in the axial zone of the ingot with a diameter of 320 mm to introduce a deoxidizer or modifier. When the height of the ingot is 2000 mm and the density of the liquid alloy is $\approx\!7000~\text{kg/m}^3$, its mass is of 1125.4 kg. For example, with the introduction of aluminum in the amount of 0.1%, its mass is of 1.125 kg. One of the well-known methods of immersion of aluminum (9 on Fig. 10) after solidification of the steel layer is by the steel armature (5 in Fig. 10) in the lower part of the axial zone of the mold [27]. Melting of aluminum and its floating will ensure the Fe-1.7%C alloy deoxidation, graphitization and reduction of cementite in the axial zone of the casting.

As it was experimentally shown the steel armature is not melted, but only loses its strength and is bended. Therefore, the time of its removal from the casting was determined upon penetration of the casting border into the axial zone (2 in Fig. 9):

$$\tau/R^2 \cdot 10^{-2} = 8.2097 \text{ min/cm}^2,$$
 (9) or $\tau_{1.7V(1.0)} = 8.2097 \cdot R^2 \cdot 10^{-2} = 8.2097 \cdot 20^2 \cdot 10^{-2} \approx 32 \text{ min 50 s}$

However, it is necessary to reduce the obtained value of 32 min 50 s, taking into account the armature dimensions of and prevention its solidification in the casting. When calculating based on the solidus temperature (see 3 in Fig. 9), the duration of solidification of the axial zone of the $\emptyset 400 \text{ mm}$ casting is 66 minutes (see calculations 6 and 7), which is significantly longer than calculation (9).

The application of the method for influencing to the liquid axial zone of Fe-C alloys proposed in this study results in improving physical, mechanical and service properties of the casting.

4. Conclusions

- The digitization of numerous experimental studies carried out in the past by different authors for solidification of Fe-C alloys as cylindrical castings containing carbon of 0.04%, 0.1%, 0.4%, 0.93%, 1.42%, 2.44%, 3.28%, 4.45%, 4.83% in the chill-mold was performed in this study. The resulting curves of promotion of the pour point, liquidus and solidus in the coordinates of the relative thickness of the solidified layer of metal x/R and parametric criterion τ/R² in the range of 0.04 ÷4.83%C were obtained.
- 2. A change in the size of two-phase liquid-solid and solid-liquid zones in ingots and castings was established. It is proposed to calculate the required amount of the modifier or deoxidizer for the introduction into the central liquid and the liquid-solid zone of the ingot at the solidification of the specified metal layer. The gradual melting of these chemical compounds provides a smooth change in the micro- and macrostructure between the solidified layer and the axial zone.
- 3. The calculation of the mass of the modifier or the deoxidizer for introduction into the axial area is reasonable to perform on the residues of metal in the liquid and liquid-solid parts of the casting. As an example, the method of calculating the mass and time of introduction of the graphitizing modifier into the axial area of rolling rolls made of eutectoid steel with 1.7%C to reduce the negative effects of cementite, Cr and Mo carbides on the structure of the axial zone of the rolls.
- 4. The results obtained by the experimental studies of the kinetics of promotion of fronts of pour point, liquidus and solidus are reasonable to use to evaluate the accuracy of computer modeling of solidifying Fe-C alloys and subsequent adaptation of the mathematical models by adjusting the thermophysical coefficients for metal.

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