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Photometric analysis of the structure evolution on the Pb-19.4%Sn melt surface in the S-L temperature range

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Abstract. The structure evolution of alloys in solidification range is considered as the first-order phase transformation from the solid state to the liquid one, which occurs by the mechanism of nucleation and growth of more symmetrical phase to less symmetrical crystalline phase. The kinetic regularities of this transformation are studied by the method of the photometric analysis of structure images (PHASI), which makes it possible to establish the temperature dependence of the relationship between the solid and liquid phases and their distribution on the melt surface. The PHASI method is based on the combined analysis of the brightness spectra of the visible light reflections from the sample surface and of the distribution of its scattering centers in different intensity intervals. The data on the structure evolution of the Sn+19.4%Pb alloy upon melting and solidification were considered in parallel with the measured spectra of sound signals. It was revealed that a distinct maximum is observed in the temperature dependence of radiation energy in the temperature range of phase transformation from the liquid into the solid state and hot crack formation occurs near the transition zone in the region of the contact of the ingot with the crucible.

1. Introduction

Melting and solidification of metals and alloys are the most important processes of metallurgical technologies. They begin the technological cycle resulting in the formation of the required service properties of materials. However, their study meets great difficulties during the use of both theoretical and experimental methods. These difficulties are generally caused by the no equilibrium character of the factors, which play the leading role in the occurrence of these processes and include the temperature and gravitation fields and the distribution of impurity elements. Additional difficulties are introduced by long-term contacts with the lining materials of furnace and crucibles and the gas environment. The melts of metals and alloys are characterized by a great variety of heat transfer processes such as the thermal conductivity in the layers of solid metal and in the transition layers in the zone of contact with the heat insulation of furnaces. These processes are complicated by the effect of radiation from the melt surface and by the convection flows in the volume of melt. The description of the structural state of the melt upon crystallization requires many physical variables, which are interrelated and cannot be obtained from speculative estimations. The metrological characteristics of contact sensors upon experiments change because of the high temperatures of melts, and this causes additional difficulties. Melting and solidification in their physical essence are the transient processes of metallurgical production, and, therefore, the entire combination of the characteristics describing the chemical and phase compositions of material and its structural state are unknown functions of time. Their knowledge is the necessary, but insufficient condition of the manufacture of high-quality metallurgical products.

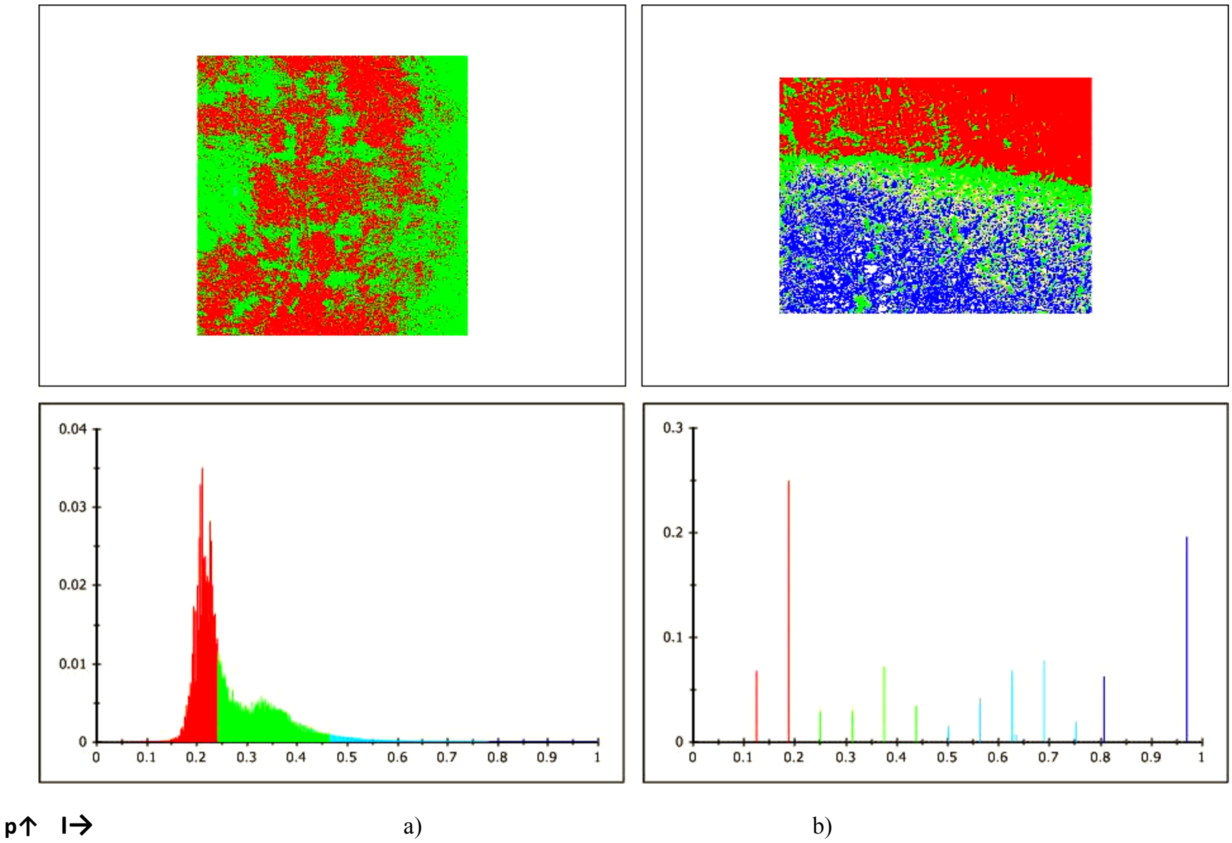
2. Experimental

The purpose of this work was a complex study of the aggregate and structural states of the Sn+19.4%Pb alloy upon melting and solidification by the measurements of its temperature and acoustic emission signals and the surface photometry. The selection of the alloy was caused by its sufficient low melting temperature and by its relatively low chemical activity in the molten state. The alloy ingot of 59g in weight was put into an alundum crucible and then melted in a vertical furnace. In the cooled state, the ingot was 34 mm in diameter and about 10-13 mm in height. A chromel-alumel thermocouple was connected to the ingot through a channel along the furnace, and a wide-band sensor of acoustic signals was connected through a waveguide. Temperature, time, and acoustic emission signals were recorded in continuous regime. The melt surface was periodically photographed on top through the channel.

The error of temperature measurements did not exceed 1°C in the entire range. The surface of the alloy was studied by the method of the photometric analysis of structural images (PHASI), which was developed by the authors [1]. The physical principles of the method and the software program of its realization are considered in detail in our works [2, 3]. Here we describe it only briefly. PHASI is a program-analytical complex using a personal computer. Its work is based on the combined analysis of the body surface images and the brightness spectra of the visible light reflection from it before and after the external action of known nature. In the present work, the surface images were photographed by a digital camera directly during the experiment. Figure 1 shows the typical results of the image processing by PHASI. The reflection of visible light from the source of lighting from the mirror surface was used as the reference reflection. The reflection brightness spectrum is built in the "spectral density of the reflection brightness - intensity of the reflection brightness" coordinates. Different intervals of the reflection intensity are shown in different colors, for example: maximum brightness – in dark-blue, minimum - in red. In the present work, the PHASI method was used to study the structure evolution of the alloy upon its heating and cooling as a function of the temperature field distribution character. For this aim, it was necessary to establish the relationship between the spectral distribution characteristics of the intensity of the reflection from the alloy surface and the temperature measurements by thermocouple.

3. Results and discussion

Since the thermocouple records a local temperature, we tried to establish the relationship between temperature and the energy of the difference spectrum of the reflection intensity, E_r (in conventional units), taken from a local ingot surface section of 30-35 mm^2 in area, chosen near the contact of thermocouple with the ingot for constructing the calibration graph, which transforms the measured spectrum characteristic into temperature in degrees. It was found that the obtained data cannot be plotted at the same curve. There are two separate dependences for the stages of heating and cooling. They are represented in Figure 2. The upper curve is related to the heating stage, and the lower one is related to the cooling stage. Both curves exhibit distinct maxima, which virtually coincide with the melting point of this alloy [4]. It was impossible to use the obtained dependences for studying the distribution of temperature field along the ingot radius because of their no monotonic nature and the absence of any correlation between the variables. Upon heating, the alloy underwent significant structural changes. A transition zone 1.5-1.7mm wide, which is distinctly outlined because of the color contrast, was formed in it at the perimeter of the contact with the crucible.



a) b)

Fig. 1. Surface fragment (x12) of the Sn-19.4% Pb alloy ingot at T = 29.4°C: (a) reflection from the mirror, (b) reflection from the surface fragment.

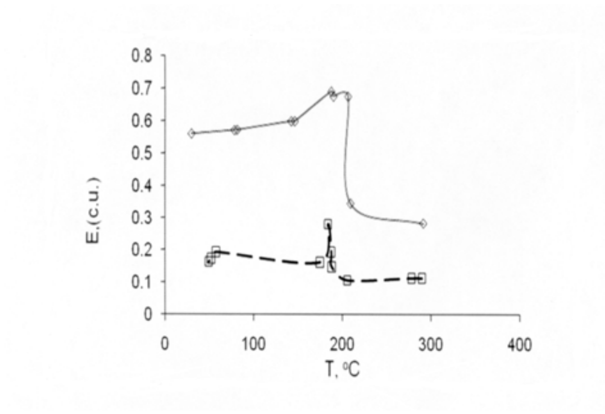
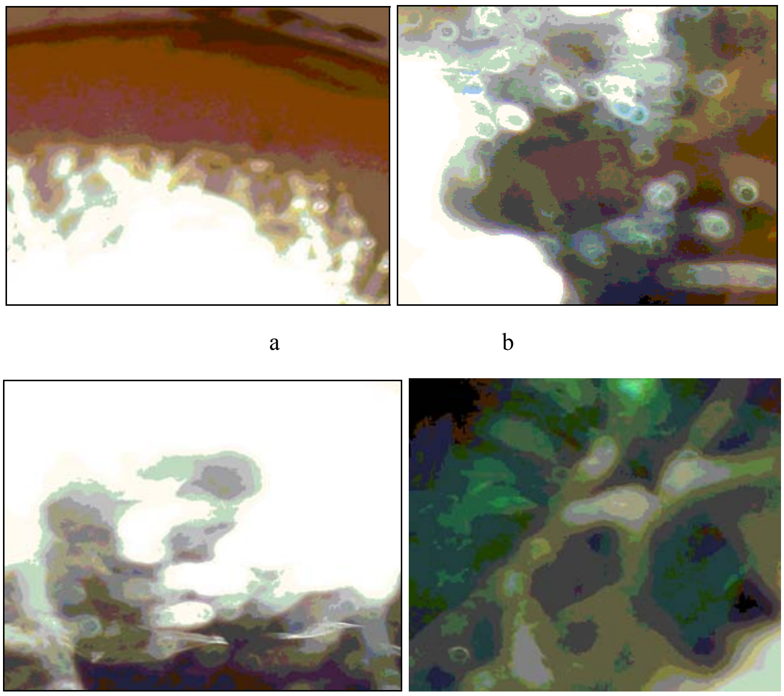


Fig. 2. Temperature dependences of conditional energy of the radiated spectrum (E_r) from an alloy surface area.

Upon heating, the temperature field remains no uniform in the radial and tangential directions up to the end of solidification. The pattern appearing on the surface of the alloy in the zones of increased temperature resembles the accumulation of bubbles. An important role in the surface color changes can belong to the alloy oxidation processes. The noted specific features of the structure evolution of the alloy upon heating can be seen in Figure 3. The photographed surface images are given without any changes in their color range. Other attempts to obtain information about the radial distribution of temperature field of the ingot at different cooling stages were undertaken, in particular, by using complete integrals of the reflection brightness spectrum (E_p, in conventional units) related to the corresponding temperatures. The results of such attempt are illustrated in Figure 4. The shown dependence $E_p = f(T)$ was also used as a calibration curve for the estimation of local temperatures from the results of the treatment of the reflection brightness spectra. This curve also has a maximum corresponding to the temperature of the maxima in the dependences shown in Figure 2. Its existence is confirmed by multiple repetitions of the measurements. In spite of obvious disadvantages of the dependence, for such aims they seem to be sufficiently plausible for all three moments of cooling time. Figure 5 shows the radial distributions of temperature fields for different cooling times. It is evident from Figure 5 that temperatures over the ingot cross section are completely equalized during half-hour. Upon cooling, the acoustic sensor repeatedly fixed the signals of acoustic emission. Figure 6 shows the cooling temperature curve, in which the vertical lines indicate the pulses of acoustic signals so that the length of the lines reflects the energy of acoustic signals in a scale of 1 mV^2 s in 14 mm. In view of the fact that the temperature interval, in which the acoustic signals appear, lies in the region of relatively low temperatures (150-50°C), their origin should be related to the propagation of the solidification cracks nucleating at the boundary of transition zone of the ingot and propagating in the radial direction.



a b

c d

Fig. 3. Structural specific features (x12) of the Sn-19.4% Pb melt surface (a) transition zone at T = 264°C, (b) accumulation of bubbles at T = 177°C, (c), (d) surface fragments at the opposite ends of the ingot diameter at T = 291°C.

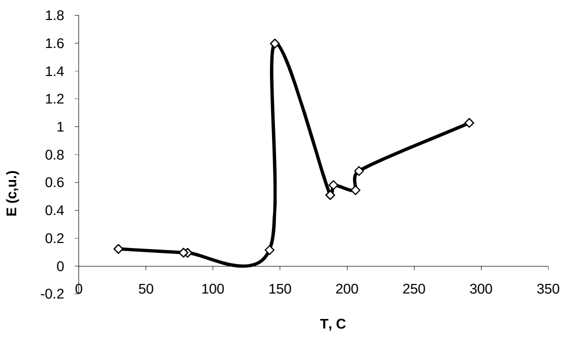


Fig. 4. Temperature dependence of spectral radiation energy (E_p) of the ingot surface.

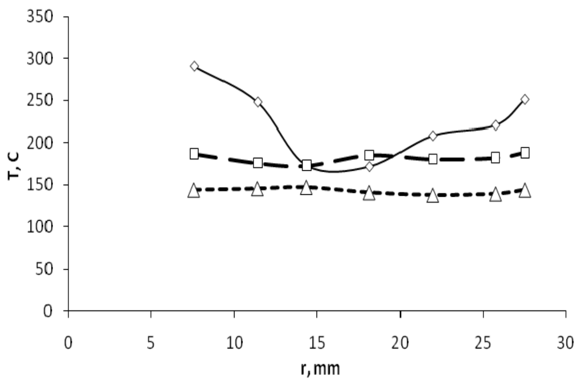


Fig. 5. Distribution of ingot temperature field constructed by the data of the dependence shown in Fig. 3: immediately after furnace switch-off (◊); and upon cooling in furnace for 29 min (□); and for 35 min (Δ).

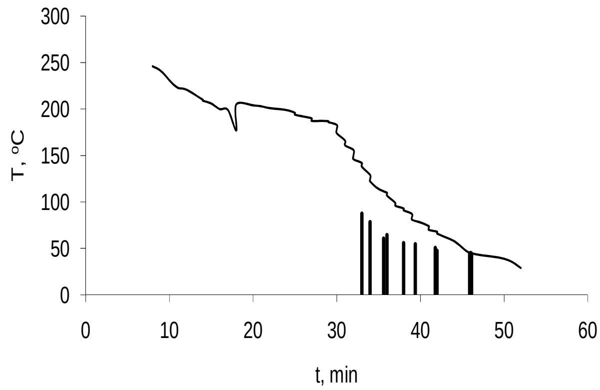


Fig. 6. Thermo gram of alloy cooling with the indications of time and energy of acoustic signals.

The mechanism of the formation of such cracks was determined by N.N. Prokhorov [5], who established the existence of the brittleness temperature range between the half-sum of liquidus and solidus temperatures and the liquidus temperature. The high rate of deformations and the low plasticity of alloys in this range promote the formation of hot cracks during welding and solidification of ingots. Actually the presence of such cracks was observed in the photographs at the moment of furnace switch-off (Figure 7). The direct measurements made it possible to reveal the growth of these cracks by 0.25-0.42 mm upon cooling.

Conclusions

It was revealed by the photometric method of the analysis of structural images on the ingot surface:

1. The surface structural no uniformity is caused by the presence of local temperature gradients and by chemical interaction with the air medium.
2. A distinct maximum is observed in the temperature dependence of radiation energy in the temperature range of phase transformation from the liquid into the solid state.
3. With increasing temperature, the reflection brightness maximum is shifted to higher intensities.
4. The no uniformity of temperature field in the solid ingot occurs at the stage of heating and the initial stage of solidification. The equalization of temperature/
5. Crack formation occurs near the transition zone in the region of the contact of the ingot with the crucible. Their growth is observed in a temperature range of 150-50°C.

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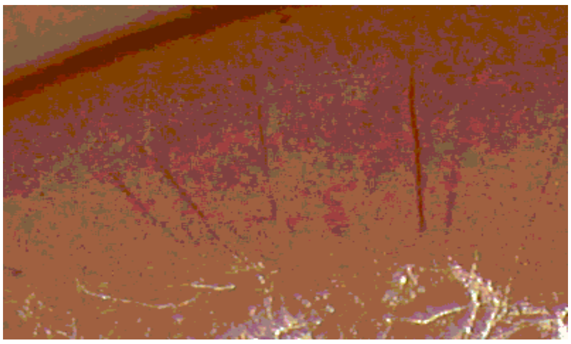

Fig. 7. Cracks at the boundary of the transition zone of the ingot. (x12)

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