ARTICLE IN PRESS

Journal of Crystal Growth xx (xxxx) xxxx–xxxx

Contents lists available at ScienceDirect

Journal of Crystal Growth

journal homepage: www.elsevier.com/locate/jcrysgro

Experimental research of phase transitions in a melt of high-purity aluminum

V.B. Vorontsov*, V.K. Pershin

Physics and Chemistry Department, Ural State University of Railway Transport (USURT), Yekaterinburg 62034, Russia

A R T I C L E I N F O

Communicated by Giuseppe Falini

Keywords:
A1.Crystal structure
A1.Growth models
A2. Gradient
B1. Alloys
B1. Metals

A B S T R A C T

This scientific work is devoted to the studying of the genetic connection structures of solid and liquid phases.

In this paper Fourier analysis of acoustic emission (AE) signals accompanying heating of high purity aluminum from the melting point up to 860 °C was performed.

The experimental data allowed to follow the dynamics of disorder zones in the melt with increasing melt temperature up to their complete destruction.

The presented results of spectral analysis of the signals were analyzed from the standpoint of the theory of cluster melting metals.

1. Introduction

Compared to the study of metals and alloys in the solid state, structure and properties of the liquid metal are much less studied. This is due to the limitations of the techniques that make it difficult to unambiguously judge the structural changes taking place in the melt when overheated above the liquid line. The lack of systematic study of the structural connection of solid and liquid state is a serious obstacle to the development of the technological foundations of metals with desired properties.

In this regard, the proposed method of obtaining information on the state of the melt from the analysis of the AE signals generated by the melt is new and should help to answer all questions about the metallurgy structural changes in the melt temperature, which lead to a change in the physical properties of the metal.

Study of the reaction of the melt on the structural changes in case of overheating in the form of changes in the nature and distribution of AE signals extends our knowledge about the phase transition.

To obtain information on the structural state of the melt for the first time the spectral analysis of AE signals accompanying the melting and cooling of metals was used.

Information that the AE is accompanied by crystallization and dissolution of the majority of substances having a crystal structure has been experimentally proved by numerous examples in [1–5].

Analysis was selected as a main technique to process AE signals accompanying the melting and cooling of high-purity aluminum. It was complemented by thermal analysis of the melt during the experiment.

2. Experiment

To solve the problem on the installation (Fig. 1), a series of experiments with the melting of Al with scale of purity 99,999 in the crucible of BN boron nitride in the spectrally pure argon atmosphere was carried out. Al samples weighing 26 g were prepared by size of the crucible.

After complete melting of the contents of the melt injected in the form of a waveguide rod of Al_2O_3, with an internal thermocouple at half height of the crucible. With cold face waveguide was in contact with the piezoelectric transducer with the frequency range of 20–200 kHz.

Precision temperature control VRT was used for maintaining a constant melt temperature during the experiment on the installation. Registration of the melt temperature was performed by the temperature recorder.

AE signals along the waveguide reached (piezoelectric transducer), and after amplification by the AF arrived on the PC.

The gain was 92 dB acoustic path (k=4 • 104).

In the experiments the temperature of the melt was increased from 680 to 840 °C at intervals of 20 °C. In each temperature melt was maintained for 30 min. Then, the melt temperature is decreased at the same rate to 680°C.

PC recorded information from the melt in real time. The melt temperature was maintained to within ± 4 °C. For the analysis of AE signals the program ISVI [6] and Math Cad, adapted for the set task by [7] were used.

Experimental data were analyzed initially in analog form on the PC and then:

* Corresponding author.

http://dx.doi.org/10.1016/j.jcrysgro.2016.10.067

Available online xxxx
0022-0248/

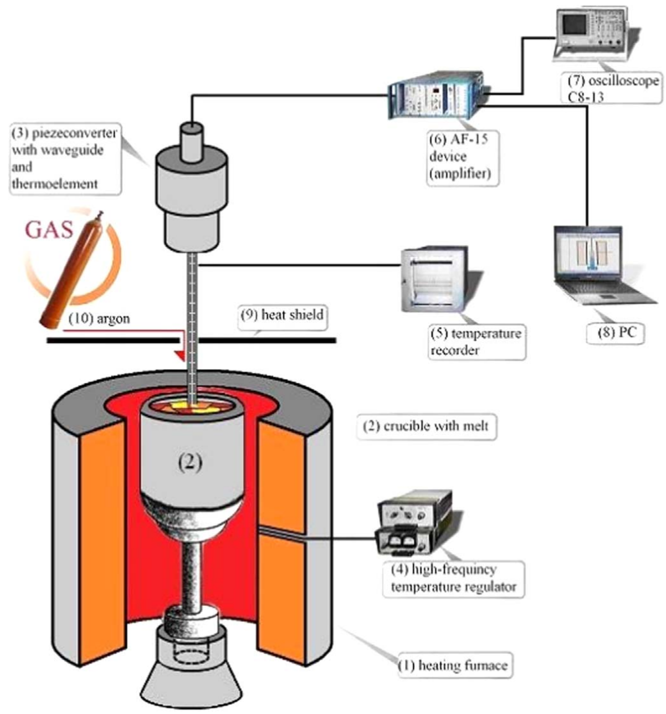

Fig. 1. Experimental installation for investigating of acoustic emission at the process of melting and crystallization of metals.

1. For each temperature the range of the AE signals above background was set; Fig. 2 shows a typical AE signal that accompanies the melting and its Fourier analysis;
2. Fourier analysis of all the AE signals for each temperature was conducted. According to the results the index Cs was obtained.

Cs includes an integral characteristic of the signal amplitude (A) at a certain frequency f.

$$C_s = \frac{1}{To} \cdot \int_0^{\tau_0} f(\tau) \cdot e^{-iw_s\tau} d\tau \tag{1}$$

$$w_s = S \cdot w_0 = \frac{2\pi S}{T_0} \tag{2}$$

Where: T_0 – the period of signal taken per unit

τ – time,

w_s – equidistant points where complex Fourier coefficients are calculated,

S=0, 1, 2,..., (N−1).

Fourier indexes Cs depending on the frequency for the 740 °C and 800 °C temperatures of the melt are shown in Fig. 3(a, b).

3. For each of the melt temperature the total energy in (relative units) Er.u. all AE signals during the analyzed period Cs integrating over all frequency was determined. The results are presented in Fig. 4(red and black line).

4. In Fig. 5 shows the dependence of the number N of signals on the melt temperature.

5. By amplitude-frequency analysis results the dependence of the number of analyzable frequencies signals for each melt temperature is constructed (Fig. 6).

6. In Figs. 7 and 8 there are dependences of number of AE signals on time for the period of an isothermal exposure of a melt when heating and cooling a melt. We see that in 8 min from the beginning signals generation stops both while heating up and cooling a melt. According to the experiment AE signals arose under nonequilibrium conditions in the melt during the transition from one state to another isothermal.

Based on paper [11] AE signals accompany with Al melt overheating to the temperature to 840 °C.

Spectrum analysis showed that the number of AE signals and character of ranges significantly change while melt temperature increasing. These changes are connected with restructuring in a melt which according to [8] up to the certain temperature is not uniform, and consists both of the free atoms and clusters (the micro-areas which saved signs of a solid phase), i.e. a short-range order. The similar pattern was watched when cooling a melt from temperature of 840–680 °C At the temperature 840 °C AE signals accompany with appear-

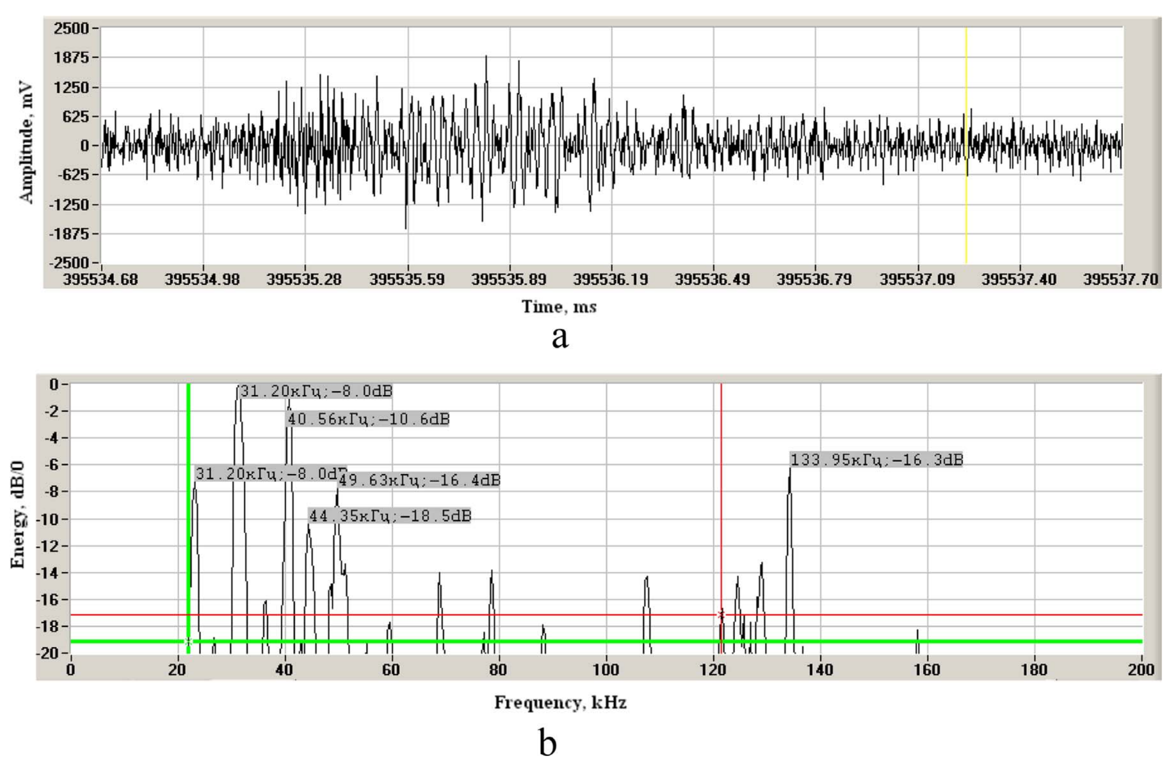

Fig. 2. Acoustic emission signal at temperature of 840 °C. a. The analog signal b. Fourier analysis of signal.