J. Chem. Chem. Eng. 6 (2012) 358-362

Analysis of Acoustic Emission Signals Accompanying Growth of Single Aluminum Crystals: Experimental Results and Theoretical Model of the Cluster

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Received: November 16, 2011 / Accepted: January 05, 2012 / Published: April 25, 2012.

Abstract: The purpose of the work is to identify the acoustic emission (AE) signal in the melt and from the interphase during the crystal growth and to establish the connection between issue parameters: the number of signal events of frequency and the signal power with the growth conditions of temperature gradient and crystallization rate. Experiments on single crystal growth were carried out using hardware and software system which allows to perform spectral Fourier analysis of AE signals and to simultaneously remove the cooling curve for the entire period of crystallization. On the basis of spectral analysis of AE signals, a theoretical model of clusters in the aluminum melt was designed. The experimental results indicate an uneven abrupt advancement of the interface according to the configuration of each individual cluster.

Key words: Growth models, interfaces surface, growth from melt, single crystal growth, Bridgman growth, acoustic emission method, metals, sound conductor.

1. Introduction

Representations of the proximity of the structures of the melt and solid phase were expressed long ago [1-2], but the evidence was obtained by an indirect method and based on the features of the behavior and properties of a variety of liquids.

In Ref. [3], an explanation of the appearance of acoustic emission (AE) signals in the audio field was given. In this paper, continuing the studies in the ultrasound region on the basis of the experimental results, the understanding of the crystal formation process and causes of the AE signals' appearance by the example of growing Al single crystals were presented.

In the authors' work, to obtain information on the structural state of the melt and the crystallization process, the most informative method was proved by spectral analysis of acoustic emission (AE) signals, accompanying the growth of single crystals.

2. Experimental Section

2.1 Experimental Installation

To solve these problems, the installation was developed (Fig. 1), which allowed growing single crystals of metals with a melting point 1200 °C by the method of Bridgman in an atmosphere of spectrally pure argon. The waveguide system with built-in thermocouple placed in the melt filmed thermogram during the crystallization and transferred to the registrar in real-time acoustic background.

As a model material Al with a purity of 99.999% was chosen. Single-crystal growth was carried out with the seed orientation <100> along the growth axis in the crucible of the BN. Single-crystal of Al was put in a soft cup to avoid that signals are not associated with the

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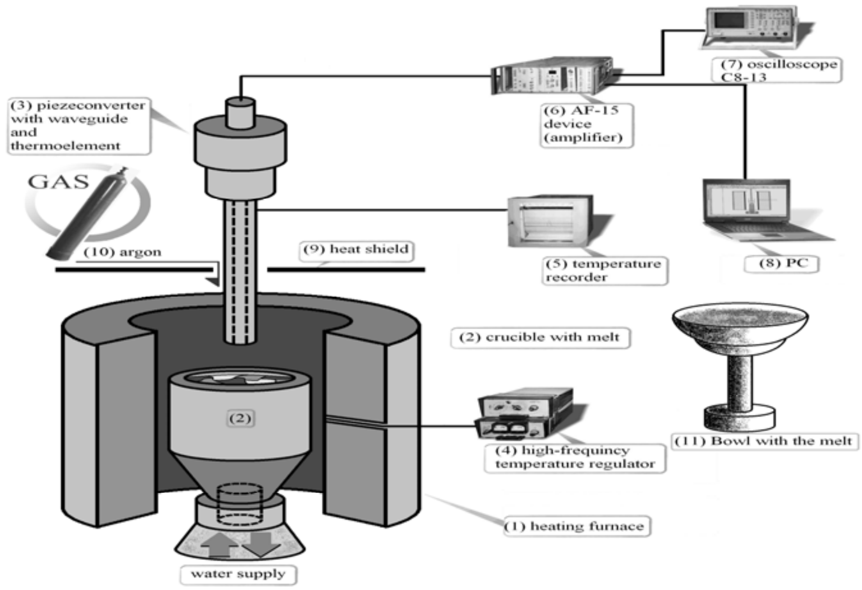


Fig. 1 Experimental installation for AE study at metal crystallization.

process of crystallization. In the experiment with the growing of single crystals, the furnace two was moving up at a stationary crucible and AE signals from the interface which reached the waveguide and transmitted to the sensor three.

Piezoelectric transducer was used with a frequency range of 20-200 kHz. The gain of the electric path was 92 dB (10^4). Hardware-software system made it possible to carry out Fourier analysis of AE signals. The analysis of the signals was made by means of such programs as Mathcad [4] and ISVI (Instrumental Systems Technology) [5].

2.2 The Performance and Processing of the Experiment

The experiment was divided conceptually into two stages:

(1) Analysis of AE signals in the melt in the process of reducing the melt temperature from 780 °C to 680 °C with step of 20 °C;

(2) Analysis of AE signals in the presence of the solid phase:

(a) At a fixed furnace (G = 25 °/cm);

(b) When the furnace driving up at a rate V = 4.8 mm/min (G = 10 °/cm).

While analyzing signals in Mathcad Complex Fourier coefficient (C_s) is a function of frequency v_s (time τ), as shown in Eq. (1) and Eq. (2):

$$C_s = \frac{1}{T_0}\int_0^{T_0} f(\tau)\cdot e^{-iw_s\tau}d\tau \tag{1}$$

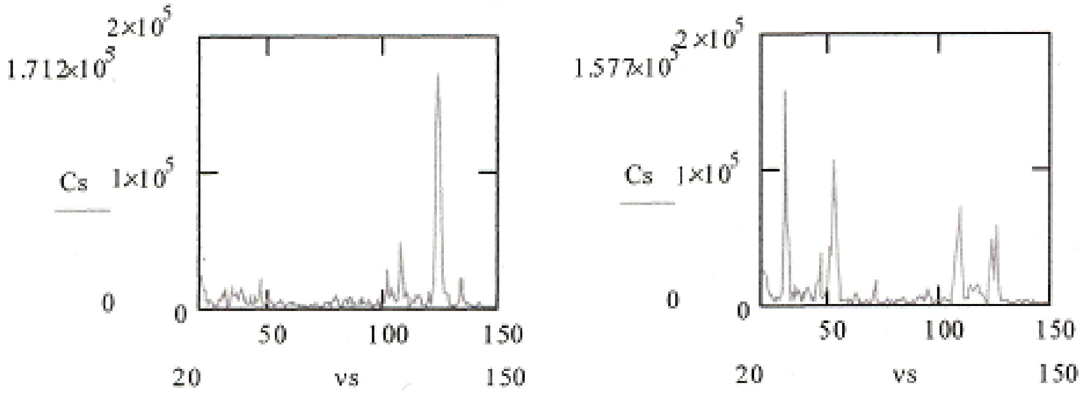
$$w_s = s\cdot w_0 = \frac{2\pi\cdot s}{T_0} \tag{2}$$

where: T_0—the period of the signal taken per unity, τ—time, w_s—equidistant points where complex Fourier coefficients are calculated, s = 0, 1, 2, …, (N-1).

With the introduction of the waveguide in the melt superheated to 120 °C above the crystallization point, the acoustic signals were not observed. Lowering the temperature to 20 °C led to the appearance of single signals (Fig. 2a, Table 1) in the resonance region of the waveguide system.

At the same reducing rate of the temperature from 700 °C to 680 °C (Fig. 2b, Table 2), AE signals were observed in the same frequency range as the growing single crystals, but with very low power (up to 6 times less than in the crystallization).

Fig. 3 shows the amplitude-frequency analysis of five AE signals with fixed furnace (Fig. 3a) and 260 signals in its motion (Fig. 3b). The range of frequencies



(a) (b)

Fig. 2 The amplitude-frequency analysis of signals in the melt. (a) Temperature range 780-760 °C; (b) Temperature range 700-680 °C.

Table 1 AE signal frequency and its complex Fourier coefficient, five AE signals with fixed furnace.

Number	Frequency (kHz)	Complex Fourier coefficient
1	29.399	63652
2	41.753	5.35×10^5
3	51.421	1.43×10^5
4	58.403	2.42×10^5
5	64.85	1.84×10^5
6	101.37	98711
7	113.73	3.7×10^5
8	122.32	4.25×10^5
9	125.54	8.29×10^5
10	133.06	1.11×10^5

Table 2 AE Signal frequency and its Complex Fourier Coefficient, 260 AE Signals with Moving Furnace.

Number	Frequency (kHz)	Complex Fourier coefficient
1	29.259	2.57×10^5
2	39.769	3.36×10^5
3	52.782	1.13×10^5
4	72.3	18765
5	99.828	19246
6	113.34	24350
7	123.35	2.05×10^5
8	125.35	2.73×10^5
9	133.36	27577

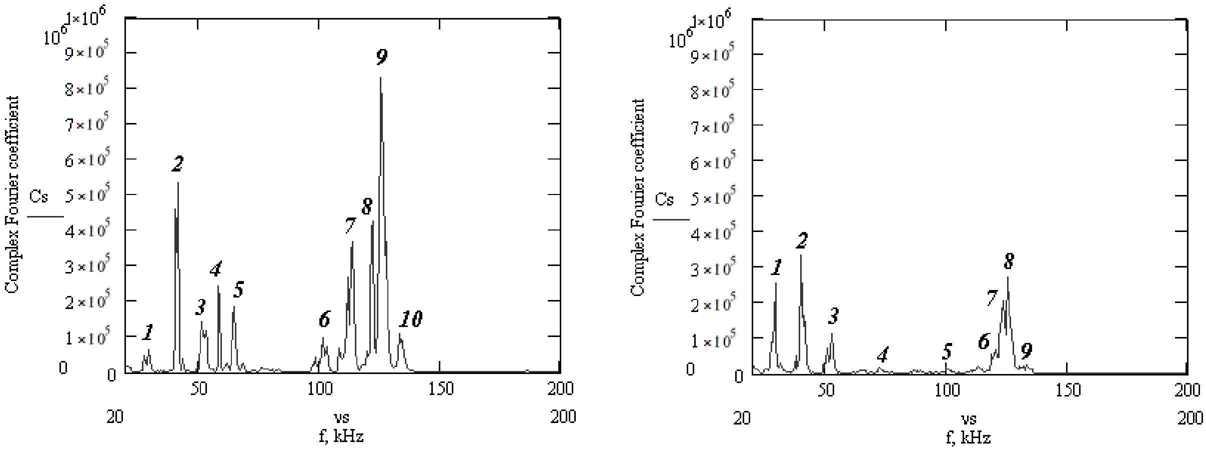
in both cases is similar, but there is difference in distribution of power harmonics.

Fig. 4 shows the distribution of signals on the frequencies for the two rates of growth. This implies that an increase of the crystallization rate by 2 times increases the number of signals in 2 times and removes their display in a high-frequency region. Fig. 5 shows one of the five signals of Table 1 (Fig. 5a) and its Fourier analysis (Fig. 5b). In the present structure of the signal characteristic frequencies in excess of the background in more than 10 times, and the frequency step of adjacent frequencies is approximately 10 kHz.

3. Results and Discussion

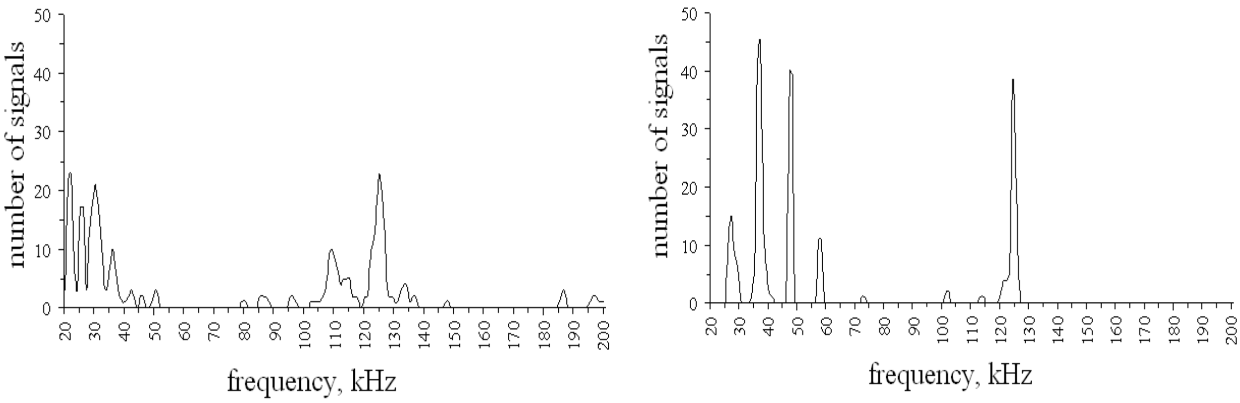
Having analyzed the results of signal processing they are concluded that the frequency spectrum of AE signals does not depend on the crystallization conditions (velocity (V) and gradient (G)) and takes intervals like 20-50 kHz and 110-130 kHz. Since the signals were observed even in the melt with a slight gradient, it can be assumed that in the melt there is a preserved area with a long-range order. In Fig. 6, based on an analysis of the data in Table 1, the cluster was simulated (the region in the melt with long-range order).

In the process of crystallization, individual cores of crystal structures which vary in size and architecture settle on an interface and give the start for the further



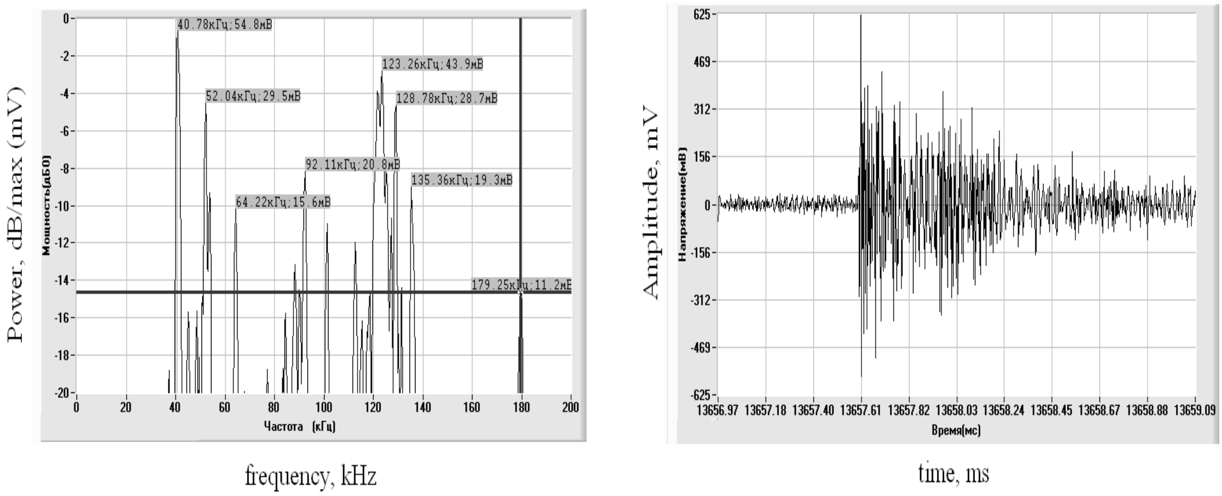
(a) (b)

Fig. 3 Amplitude-frequency analysis: (a) five AE signals with fixed furnace; (b) 260 AE signals with moving furnace.



(a) (b)

Fig. 4 The distribution number of signals on the frequencies: (a) for the rate equal 2.8 mm/min; (b) for the rate equal 4.2 mm/min.



(a) **(b)**

Fig. 5 Acoustic emission signal: (a) analog representation; (b) Fourier analysis.

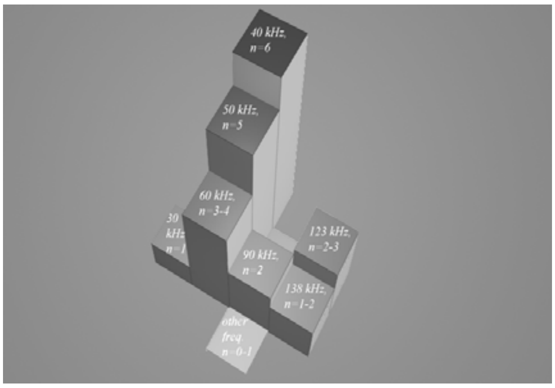


Fig. 6 The model cluster, which preserved the crystal structure in melt (long-range order).

growth of the solid phase. At the periphery of the structural formation, there is a multi-profile, consisting of columns with a base area equal to the base-centered cubic lattice of aluminum and with a height ranging from 1 to 6 of interplanar distances.

Each step of the cluster has its own penetration into the melt and the free volume released by the crystallization, so there is a frequency spectrum of signals.

The acoustic effect is the result of the collapse of redundant hydrostatic pressure. This assumption is well supported by a simple calculation implemented pulse frequency of AE during crystallization, as shown in Eq. (3).

$$f = \frac{V}{L \cdot n} \quad (3)$$

where: f—frequency of moving crystallization front; V—speed of growth of single crystals; L—the period of the Al crystal lattice; n—the number multiple of the interplanar distance.

The calculations were made on the assumption that $V = 4.2$ mm/min, $L = 4.05 \times 10^{-7}$ mm (interplanar distance for the lattice of aluminum), as shown in Table 3.

Table 3 The results of calculating the frequency components of signals.

n—the number multiple of the interpanar distance	the frequency corresponding to the specified number n
1	246
2	123
3	82
4	63
5	49
6	41
7	35
8	30.8
9	27.3
10	24.6

4. Conclusions

If it is assumed that promotion of the crystallization front is normal to the interface with a period which is determined by the number of interplanar distances of the stage, then, for a settled crystalline cluster and for each step of the profile, there is its own number of interplanar distances which are forming a step, and its own implemented frequency.

Acknowledgment

The authors thank Sokolov O. B. and Ugodnikova L.A. of Ural State University of Railway Transport for development of a program that allows to process AE signals.

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