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OPTOACOUSTIC INVESTIGATIONS OF MECHANICAL PROPERTIES OF ULTRAFINE-GRAINED ALUMINUM AD1 AFTER SEVERE PLASTIC DEFORMATION BY TORSION AND THERMAL TREATMENT

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Abstract. The results of investigations of mechanical properties of metals before and after severe plastic deformation and thermal treatment of commercial aluminum AD1 are presented in this paper. The use of optoacoustic technique allowed to measure values of propagation speed of longitudinal elastic waves in a plate and in a thin rod for various regimes of processing and to show the correlation of speed and pulse attenuation with a change of material microstructure. The sound velocity is determined by predominant crystallographic direction of face-centered cubic lattice of aluminum, and attenuation of acoustic pulse is determined by presence and distribution of defects in material structure.

1. INTRODUCTION

One way to obtain materials with ultrafine grained structure is severe plastic deformation by high pressure torsion [1]. Different combinations of regimes of thermal treatment and severe plastic deformation can lead both to significant improvement in physical and mechanical properties of metals and alloys, and to their substantial reduction [1,2].

The results of studies on changes of mechanical properties of aluminum AD1 after severe plastic deformation and thermal treatment using both conventional testing methods and with the use of optoacoustic technique [3] are presented in the paper.

The principal possibility of using the optoacoustic effect for nondestructive materials testing is well known [4,5]. The use of laser methods of acoustic pulses excitation has a number of advantages compared with methods of conventional acoustic defectoscopy and microscopy.

This is primarily due to the fact that duration of acoustic signals excited by pulse laser in Q-switching regime typically is about $(1\div3)\times10^{-8}$ s, while their amplitude can vary over a wide range depending on radiation intensity. The analysis of changes in parameters of such pulses during propagation in sample volume allows providing information in frequency range up to 50÷100 MHz that exceeds the capability of conventional acoustic defectoscopy by an order.

It should also be noted that in case of direct radiation exposure on a sample, the parameters of generated acoustic pulse are defined as by value of optical absorption coefficient due to the defect structure of surface layer of material and thermal conductivity, whose value is largely determined by the volume density of structural defects.

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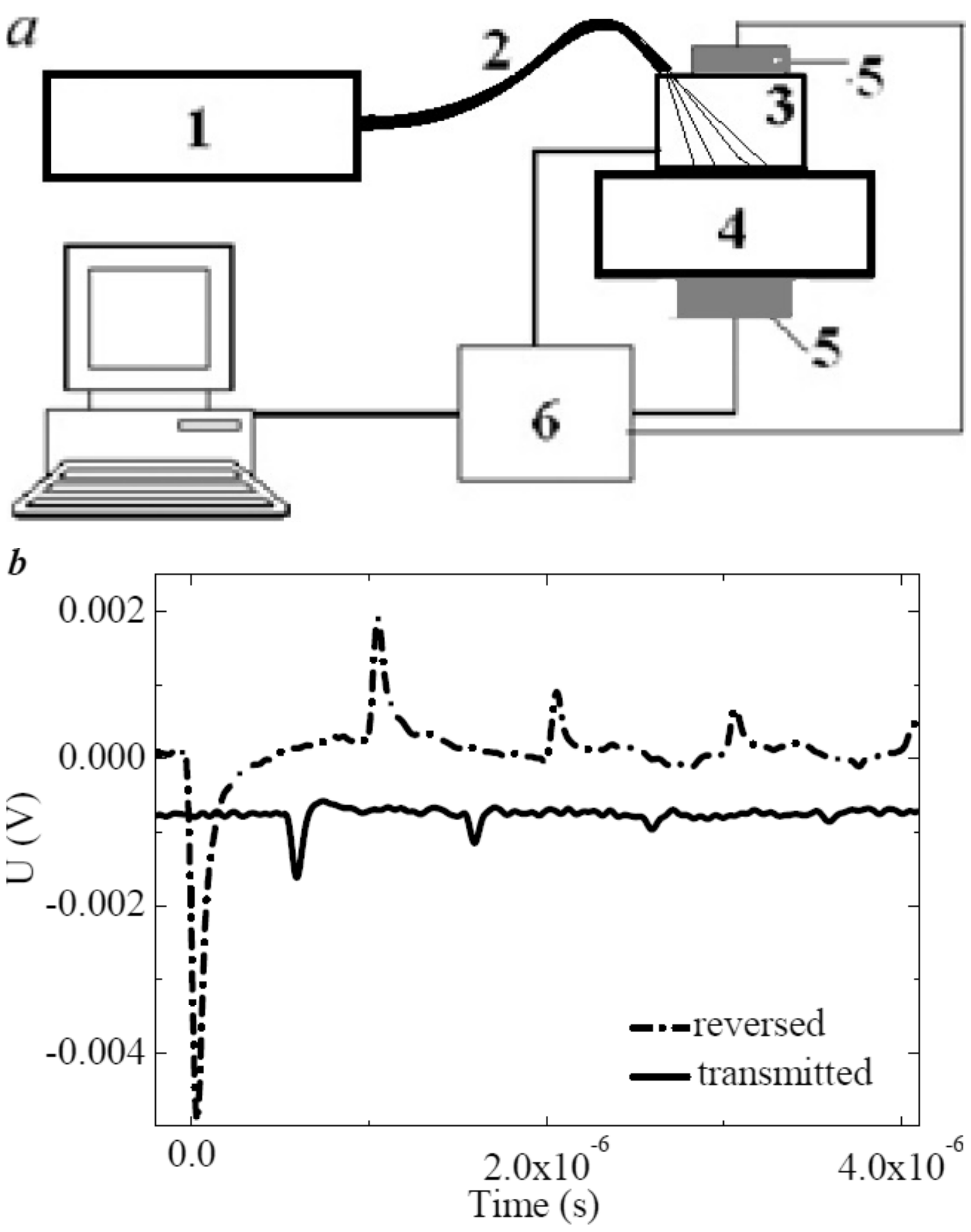


Fig. 1. a) The scheme of optoacoustic defectoscope and optoacoustic cell: 1 – the laser, 2 –the optical fiber, 3 – the waveguide, 4 – the sample, 5 – the piezoelectric receiver, 6 – the oscilloscope; b) the oscillograms of acoustic echo-pulses in glass (h = 2.91 mm).

Thus possibilities of acoustic spectroscopy of mechanical properties of materials provided by the optoacoustic method are very promising for controlling changes of elastic and strength characteristics of materials as a result of structural transformations after thermal treatment and severe plastic deformation.

2. EXPERIMENTAL TECHNIQUE

2.1. Technique of optoacoustic measurements

The developed optoacoustic technique allows controlling simultaneously both the reflected acoustic pulses at one-sided access to the object and the transmitting pulses at double-sided access to samples under study.

The method scheme is shown in Fig. 1a. Radiation pulse is led up through the optical fiber (2) to the measuring cell, which represents a cylinder of transparent material (3) with plane-parallel bases. Thin layer absorbing radiation, in which radiation pulse generates an acoustic pulse, is applied on one of the bases of the cylinder. At the same time it is also an acoustic duct for excitable acoustic pulses recorded by broadband piezoelectric detectors (5). Acoustic contact of a sample under study with the surface of the cylinder-acoustic duct is provided through a thin layer of water. The signal is delivered from piezoelectric receivers (5) to the digital oscilloscope (6) with a bandwidth of 500 MHz and a sampling frequency of 2 GHz.

Several acoustic echo-pulses repeatedly passed through the sample were simultaneously recorded in the experiments. This allowed increasing measurements accuracy of propagation speed and attenuation of sound in materials. Fig. 1b shows oscillograms of acoustic signals in glass sample for the case of simultaneous recording of both the reflected and the transmitted acoustic signals.

Spectral analysis, in particular, the analysis of dependence of speed and attenuation coefficient change on the frequency (of speed dispersion and pulse attenuation) allows obtaining more detailed information about the material structure.

The form of pulse propagating in a medium with attenuation can be represented as:

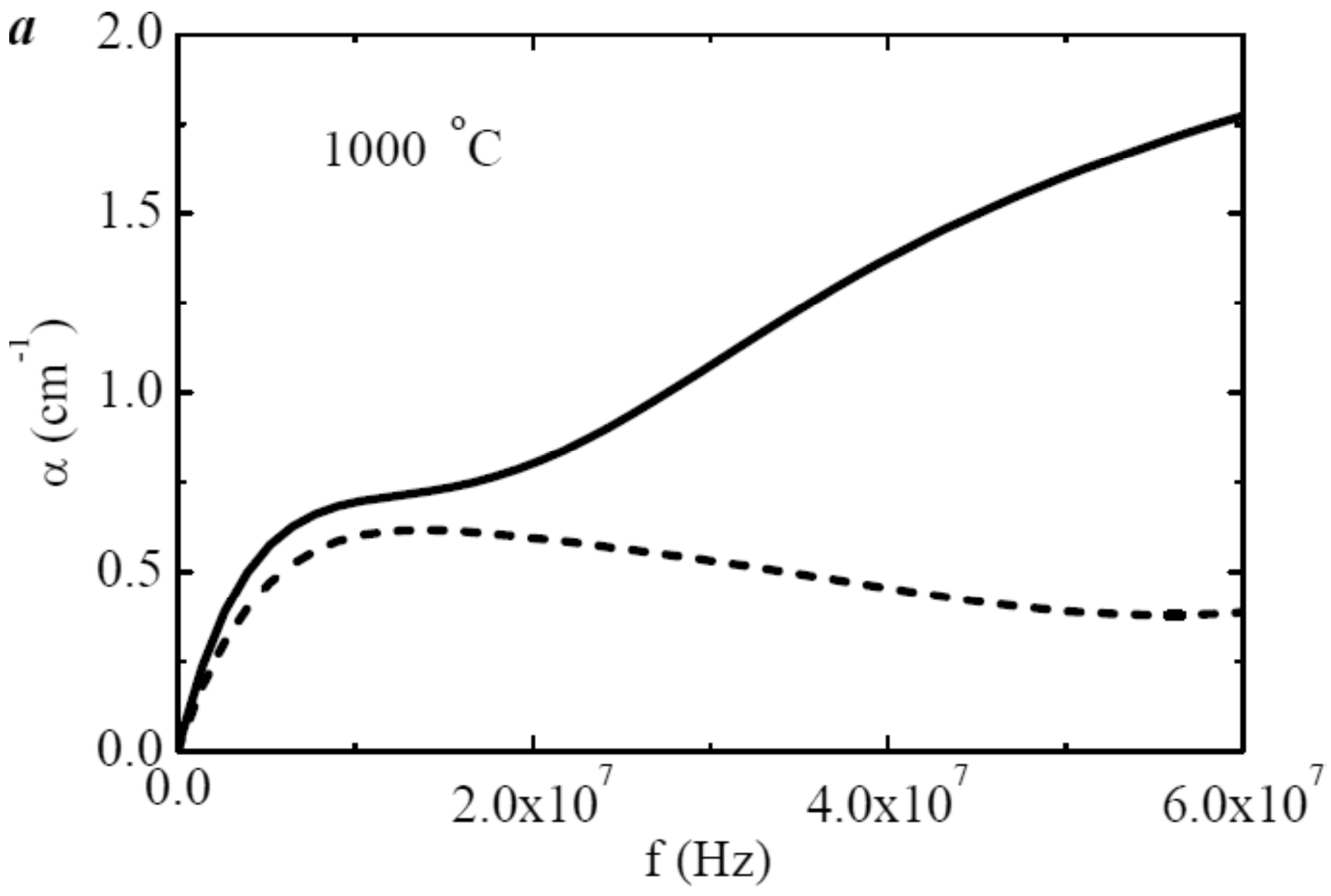
$$u(x,t) = \frac{1}{\sqrt{2\pi}}\int_0^{\infty}\left|F(\omega)\right|\exp\left[-\alpha(\omega)x\right]\times$$
$$\cos\left[\omega t - k(\omega)x - \varphi(\omega)\right]d\omega$$
$$F(\omega) = \frac{1}{\sqrt{2\pi}}\int_{-\infty}^{\infty}u(0,t)e^{-i\omega t}dt, \tag{1}$$
$$k(\omega) = \frac{\omega}{c(\omega)} = \frac{\omega}{c_0} - \frac{\omega\Delta c(\omega)}{c_0^2},$$

where $\varphi(\omega)$ - the argument of function $F(\omega)$; $\alpha(\omega)$ - the attenuation coefficient of ultrasonic oscillations; $k(\omega)$ - the wave number; x, t – the coordinate è the time; c_0 - the speed of longitudinal ultrasonic wave; $\Delta c(\omega)$- the function, describing frequency dependence of speed on frequency, where $c(\omega) \approx c_0 + \Delta c(\omega)$.

Then the dependence of speed $c(\omega)$ and pulse attenuation $\alpha(\omega)$ on frequency ω can be represented by the following relations [4]:

$$\Delta c(\omega) = \frac{c_0}{\omega\Delta x}\left[\operatorname{arctg}\frac{B_1}{A_1} - \operatorname{arctg}\frac{B_2}{A_2}\right],$$
$$\alpha(\omega) = \frac{1}{2\Delta x}\ln\frac{A_1^2 + B_1^2}{A_2^2 + B_2^2}, \tag{2}$$

where Δx – the sample thickness; A_i, B_i – the coefficients at the real and the imaginary components



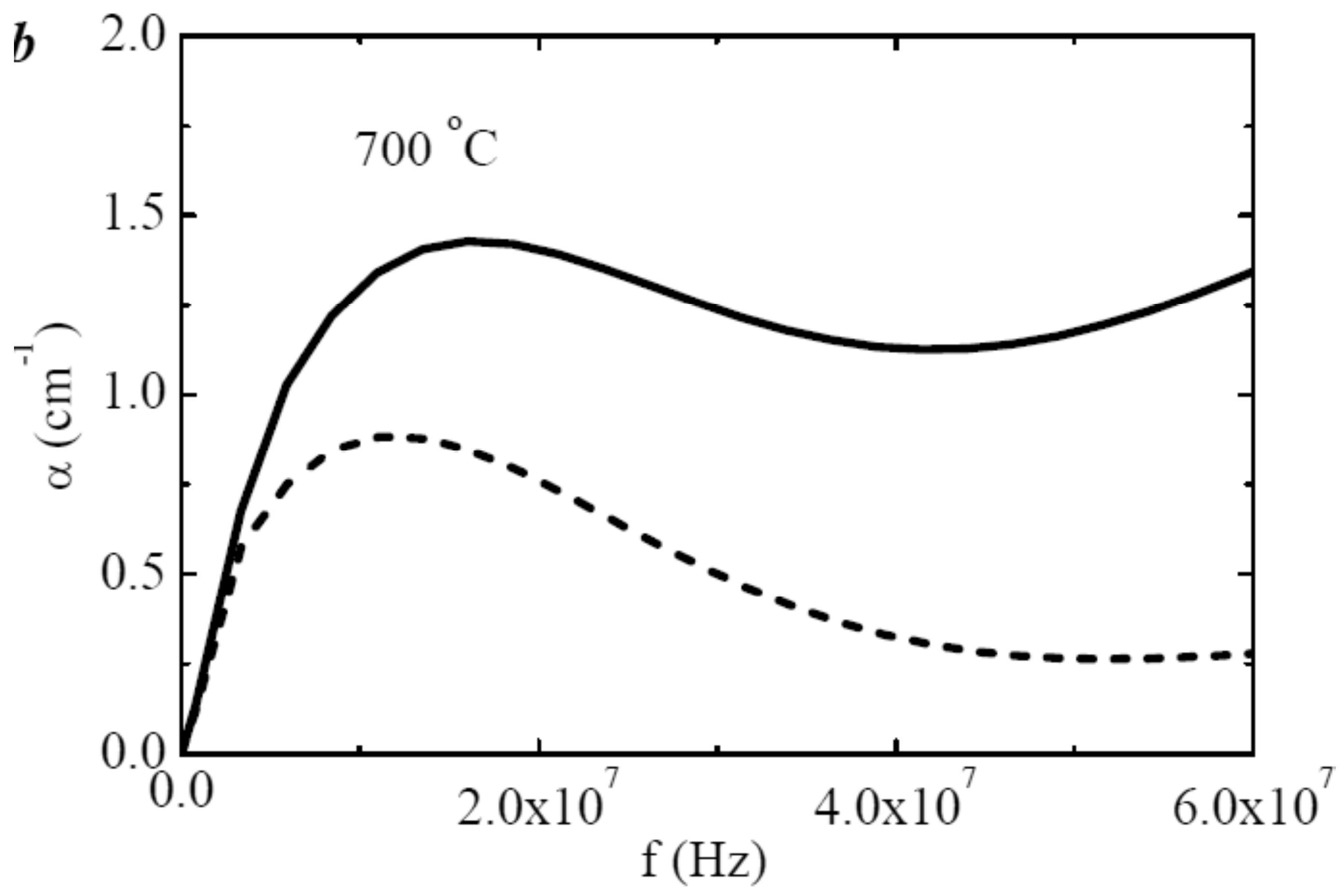


Fig. 2. Frequency dependencies of the attenuation $\alpha(\omega)$: dash – before the shock; continuous –after the shock. a) Annealing at 1000 °C (grain size $d \approx 220$ mm); b) Annealing at 700 °C (grain size $d \approx 20$ mm).

of the Fourier spectrum of the first and the second acoustic pulses.

The frequency dependencies of attenuation before and after shock loading of iron samples preliminary annealed at 1000 °C and 700 °C are given in Fig. 2 as an example. Shock load was carried out by laser pulse with duration of 70 ns and amplitude of 1 GPa. Analysis of changes of frequency characteristics allows estimating the scale of restructurings occurred in the samples as a result of shock loading [6]. Presented results demonstrate high efficiency and perspectivity of the optoacoustic method of spectroscopy for investigations of structural changes in materials, allowing obtaining information on structural changes with the scales $\geq 10^{-7} \div 10^{-6}$ m. The possibility of noncontact control of materials properties using the optoacoustic method should also be noted [7].

Therefore, this technique can be applied both in problems of fundamental and of applied character, particularly in studies of mechanical properties of materials with nano- and ultrafine grained structures.

2.2. Material of investigations

Investigation of changes of mechanical parameters of material with ultrafine grained structure was conducted on commercial aluminum AD1, whose composition is shown in Table 1.

Samples with different grain sizes were produced by means of severe plastic deformation by torsion and thermal treatment. Regimes of deformation and thermal treatment, which were chosen for producing samples with different grain size and mechanical properties [1] are presented in Table 2.

Severe plastic deformation was conducted on a press for high pressure torsion Walter-Klement GmbH HPT-07. The billets with diameter of 20 mm and thickness of 2.5 mm, which were cut from the AD1 plate parallel to the rolling plane, were placed

Table 1. Composition of commercial aluminum AD1.

Fe	Si	Mn	Ti	Al	Cu	Mg	Zn	Impurities
up to 0.3	up to 0.3	up to 0.025	up to 0.15	min 99.3	up to 0.05	up to 0.05	up to 0.1	others, 0.05

Table 2. Regimes of deformation and thermal treatment.

Marking	Regime
1.1	Annealing: 2 hours at 0.7T_m °C,water quenching; HPT: pressure 6 GPa, 10 turns;
1.2	HPT: pressure 6 GPa, 10 turns.
1.3	HPT: pressure 6 GPa, 10 turns. Annealing: 1 hour at 0.2 T_m °C
1.4	HPT: pressure 6 GPa, 10 turns. Annealing: 1 hour at 0.35 T_m °C
2.1	As-received condition
2.2	Annealing: 2 hours at 0.7 T_m °C

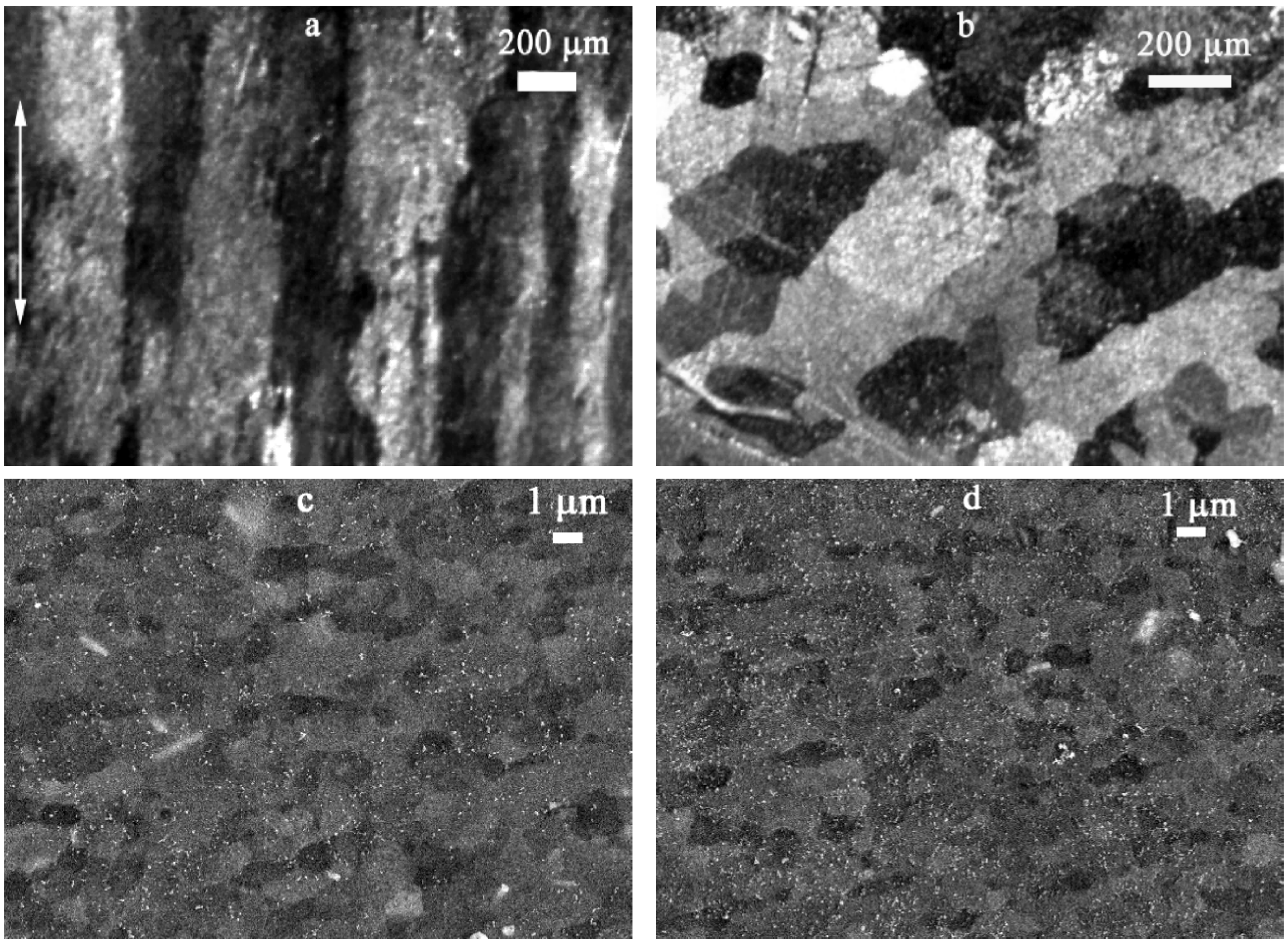


Fig. 3. The microstructure of aluminum alloy AD1 before and after different processing regimes (Table 2): a) the as-received condition (arrow – the rolling direction); b) the regime 2.2; c) the regime 1.2; d) the regime 1.1.

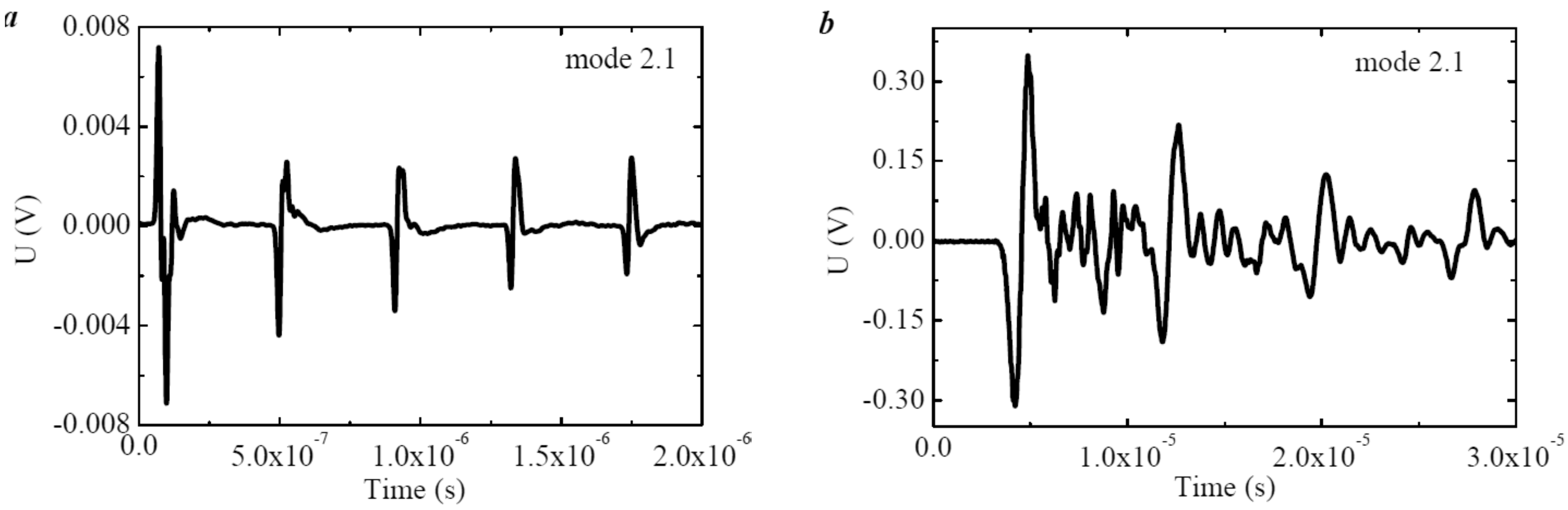


Fig. 4. Acoustic signals in a plate and a rod in the samples of as-received material ($\delta c/c \leq 0.25\%$).

in the press. The billets were compressed to the required pressure, whereafter the torsion with a given number of turns was conducted under conditions of hydrostatic pressure. As a result, the discs with diameter of 20 mm and thickness of 1.5 mm were obtained, and after polishing to roughness of 1 micrometer the disks thickness was about 1.3 mm.

The microstructure of aluminum AD1 before and after processing is shown in Fig. 3. The as-received grains with the width of 160-280 micrometers are elongated in the rolling direction, and their length can reach up to 4 mm or more (Fig. 3a). Annealing at $0.7T_m$ °C for 2 hours leads to structure recrystallization (Fig. 3b). Severe plastic deformation by torsion allowed obtaining samples of aluminum AD1 with a grain size of 500-900 nm (Figs. 3c and 3d).

The microstructure homogeneity in the samples was tested by measuring the Vickers microhardness along the diameter of the sample. Dehomogenization (microhardness reduction) was observed only in the center of the sample in the area of 0.5-1 mm radius.

The material density was measured by the method of hydrostatic weighing of disk billets.

The quasistatic tensile test of samples was conducted on a standard test machine with the constant strain rate of 0.0014 1/s. For this purpose the samples with width and length of the working portion of 2 and 6 mm were cut from disk billets on electrical discharge machine.

The rods 19×2×1.5 mm were cut from disk billets for the measurement of rod speed of sound.

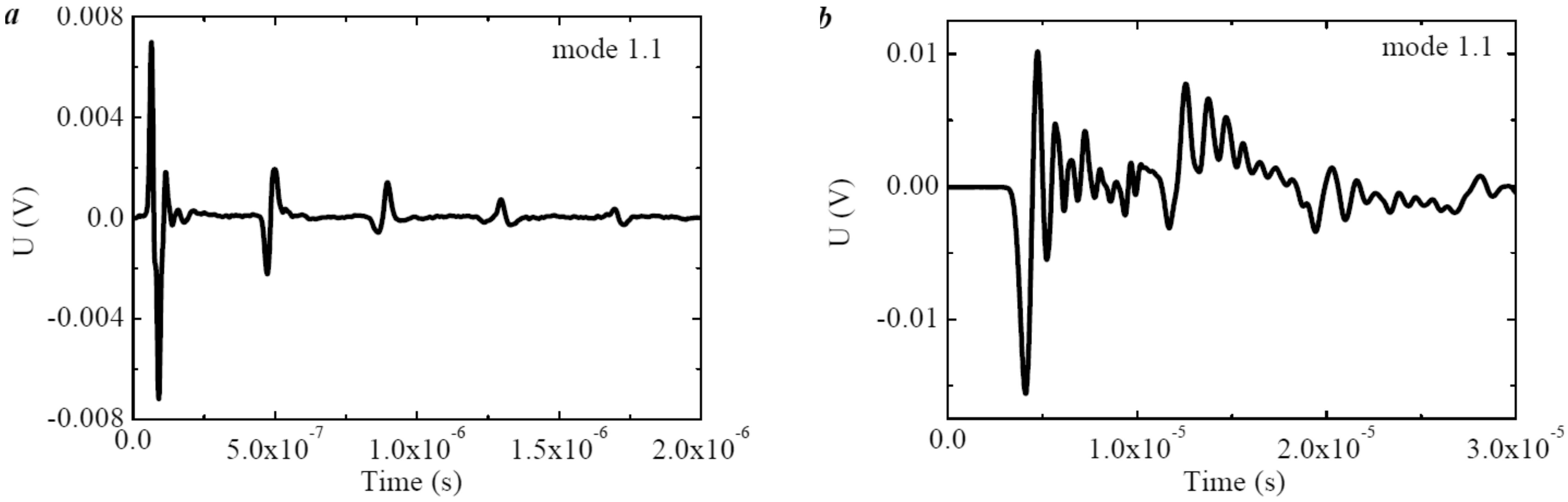


Fig. 5. Acoustic signals in a plate and a rod in the sample after the processing regime 1.1 ($\delta c/c \leq 0.25\%$).

3. RESULTS AND DISCUSSION

3.1. Results of optoacoustic measurements

The results of optoacoustic diagnostics of restructurings in aluminum AD1 as a result of severe plastic deformation and thermal treatment are shown in Figs. 4 and 5.The results of measurements are shown in Table 3. The error of speed measurement did not exceed 0.25%.

Measurements of longitudinal sound speed in plates and rods with the use of the optoacoustic technique allowed determining the elastic moduli and Poisson's ratios. Constants were calculated from the known expressions for speeds of elastic waves in a half-space and a thin rod [9]:

$$c_L = \sqrt{\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}}, \quad c_r = \sqrt{\frac{E_r}{\rho}}, \tag{3}$$

where ρ is the material density, E is the elastic modulus, E_r is the Young's modulus for a rod. Knowing the speeds c_L and c_r and assuming $E \cong E_r$ one can determine the values of the elastic modulus E and the Poisson's ratio ν.

The mechanical parameters of the samples of aluminum AD1, obtained as result of different processing regimes are presented in Table 3.

HV is the Vickers microhardness; ρ is the density; c_L is the longitudinal sound speed in material; c_r is the rod speed of sound; ν is the Poisson's ratio; d is the grain size, σ_B is the tensile strength, $\sigma_{0.2}$ is the yield strength.

3.2. Texture and acoustic properties

Deformation processing of metals can specify the defined texture of material that leads to anisotropy of material properties. The measurements suggest that various processing of material led to a variety of elastic rates (Table 3). It is known that the values of sound velocity are different for different crystallographic directions. Hooke's law for an elastic body can be written as [8,9]:

$$\sigma_i = C_{ij}\varepsilon_j \;\; i, j = 1, 2, \ldots 6, \tag{4}$$

where σ_i and ε_i are the stress and the strain, C_{ij} is the elastic tensor. For a cubic crystal the number of independent elastic moduli is reduced to three: C_{11}, C_{12}, and C_{44}. Such simplification allows obtaining simple expressions for calculation of velocities of longitudinal elastic waves in three crystallographic directions [8]:

$$\begin{aligned} \rho c^2_{L[100]} &= C_{11} \\ 2\rho c^2_{L[110]} &= C_{11} + C_{12} + 2C_{44} \\ 3\rho c^2_{L[111]} &= C_{11} + 2C_{12} + 4C_{44}. \end{aligned} \tag{5}$$

Using the known data for the elastic moduli of aluminum monocrystal [8] C_{11} = 108.2 GPa,

Table 3. The parameters of aluminum AD1 with different processing regimes.

Regime	HV	ρ,kg/m^3	c_L,m/s	c_r,m/s	E,GPa	ν	d,μm	$\sigma_{\hat{a}}$,MPa	$\sigma_{0.2}$,MPa
2.1	29 ± 0.5	2714±6	6275±20	5160±20	72.3	0.321	220±60	92±4	71±4
2.2	26 ± 0.3	2712±4	6440±20	5150±20	72	0.344	160±70	56±9	40±8
1.1	57 ± 0.4	2697±1	6660±20	5100±20	70.1	0.363	0.7±0.1	218±1	90±17
1.2	54 ± 1	2717±6	6460±20	5070±20	70	0.353	0.7±0.2	200±1	80±20
1.3	61 ± 0.3	2709±4	6420±20	5000±20	67.7	0.336	0.7±0.2	226±4	92±20
1.4	33 ± 0.3	2709±4	6400±20	5020±20	68.3	0.353	6±3	102±9	83± 6

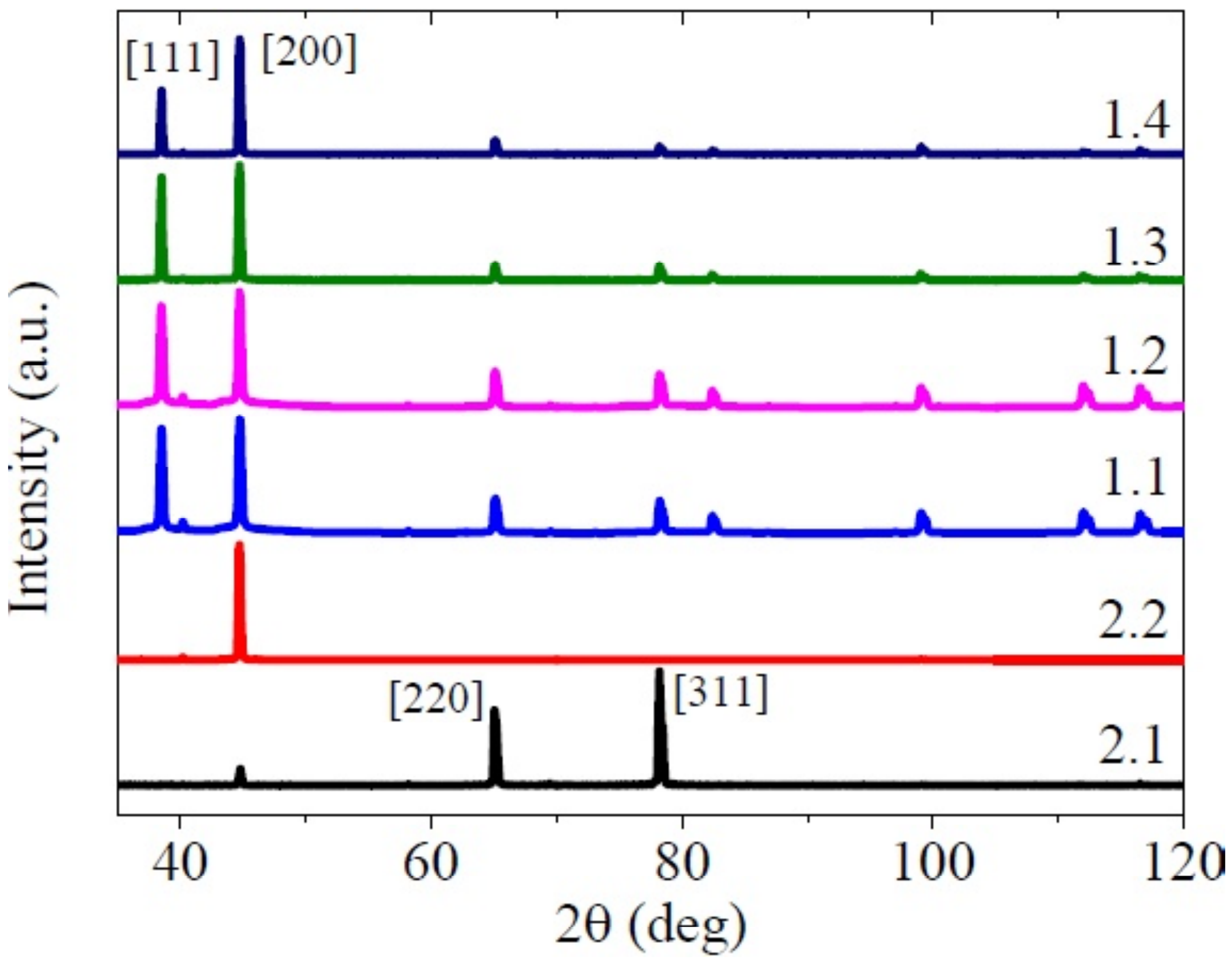


Fig. 6. The X-ray pattern of the samples of commercial aluminum AD1 after various processing regimes (Table 2). The analysis was conducted for the direction orthogonal to the sample plane.

C_{12} = 61.3 GPa, and C_{44} = 28.5 GPa and the value of aluminum density ρ = 2699 kg/m^3 one obtains values $c_{L[100]}$ = 6330 m/s, $c_{L[110]}$ = 6476 m/s, and $c_{L[111]}$ = 6524 m/s, and for the data from work [10] C_{11} = 113.2 GPa, C_{12} = 66.63 GPa, and C_{44} = 27,83 GPa - $c_{L[111]}$ = 6524 m/s.

Therefore, comparing the calculated values of elastic rates for the three main crystallographic directions and the values of elastic rates obtained at optoacoustic measurements, one can estimate the predominant crystallographic directions of ultrafine grained structure after processing. Thus, in the direction perpendicular to the disk plane for the regime 2.1 (as-received) predominates the crystallographic direction [100], for the regimes 2.2, 1.2, 1.3, and 1.4 the [110] direction and for the regime 1.1 - [111].

The obtained estimates correlate with X-ray patterns of samples, see Fig. 6. The texture axis in the [311] direction is most pronounced for the as-received sample (regime 2.1), only the [200] direction is observed for the annealed sample (regime 2.2). The two preferential directions [111] and [200] are observed for the regimes with severe plastic deformation by torsion.

Beside the possibility of determining the elastic moduli, the optoacoustic diagnostics allows analyzing the frequency dependencies of sound velocity and attenuation in a wide frequency band that allows receiving additional information about the materials structure. The frequency dependencies of attenuation in the samples under study are shown in Figs. 7a and 7b.

The considerably larger attenuation in compare with the initial material (2.1) is observed especially

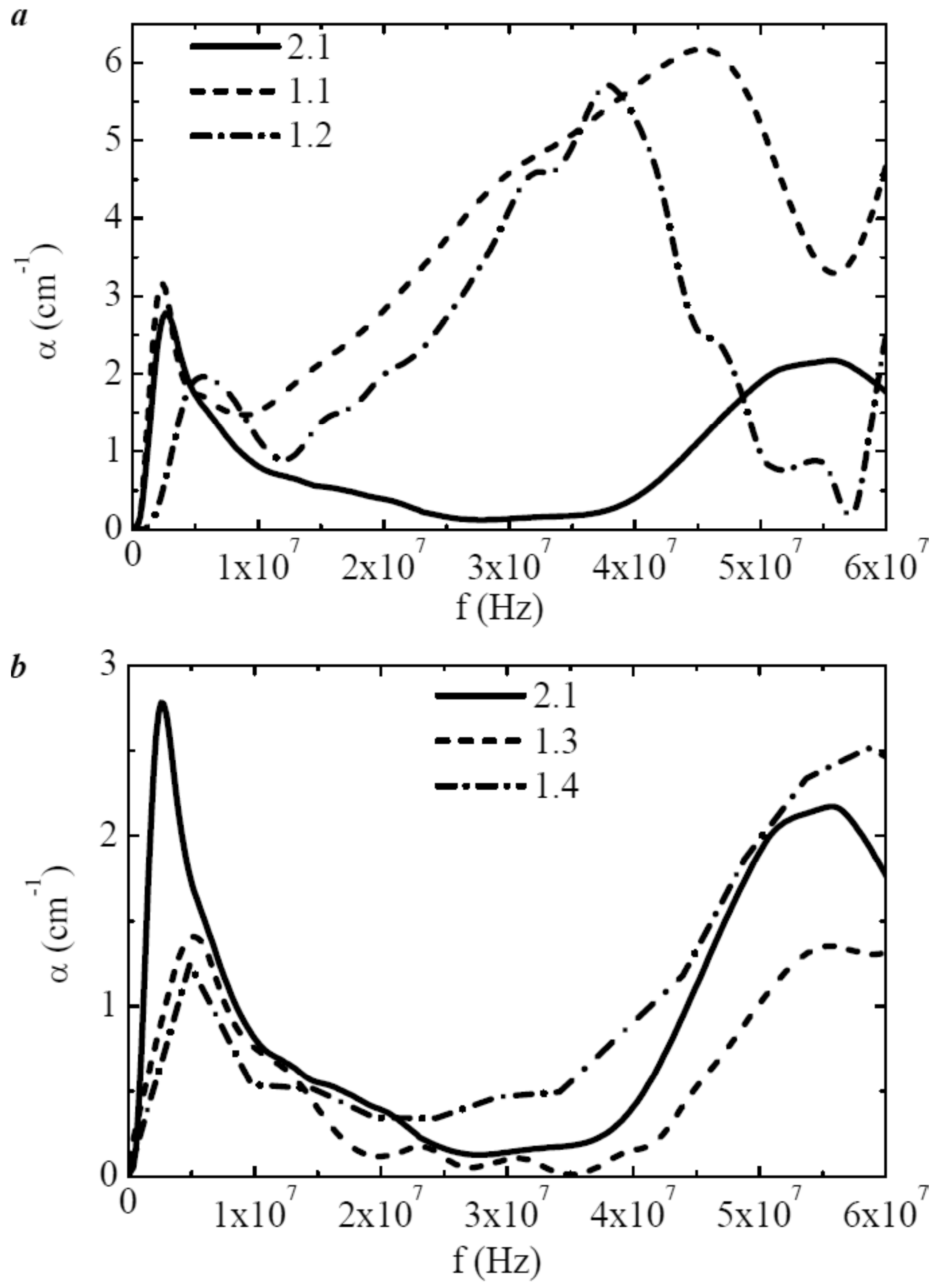


Fig. 7. Frequency dependencies of the elastic waves attenuation in the as-received samples - 2.1 and after various processing regimes 1.1, 1.2, 1.3, and 1.4.

at high frequencies, in a preliminarily annealed sample 1.1 that is to a great extent due to the presence of pores in the sample, which determine its minimal density. Substantially smaller attenuation at frequencies less than 6 MHz and its significant growth at high frequencies >6 MHz (Fig. 7a) is observed in the sample (1.2) obtained from the as-received condition only by means of severe plastic deformation by torsion. The decrease of attenuation at low frequencies is determined by the absence of pores in this sample, as evidenced by the large value of its density. It should be noted that both samples have the same grain size, but the preliminarily annealed sample 1.1 has a somewhat larger micro-hardness, higher yield strength and tensile strength (Table 3).

Attenuation in the frequency range up to 4 MHz in the samples 1.3 and 1.4 annealed after severe plastic deformation (Table 2) is much smaller than that in the sample 2.1 and comparable at higher frequencies. In other words, the annealing after severe plastic deformation reduces the defects density in microstructure of samples. It should be noted that the annealing at the temperature of recrystallization (sample 1.4) leads to an increase in grain size and the associated reduction of micro-hardness and yield stress, in accordance with the ratio of the Hall-Petch relation.

4. CONCLUSIONS

The results of investigation of mechanical parameters of commercial aluminum AD1 before and after severe plastic deformation by torsion and thermal treatment are presented. In short, these results are as follows:

Severe plastic deformation by torsion produces the ultrafine-grained structure of alloy AD1 and leads to an increase in its micro-hardness, yield strength and tensile strength more than two times.

It is shown that the optoacoustic method allows one to measure with high accuracy both the longitudinal and rod speeds of sound and, as a consequence, to determine the elastic moduli and Poisson's ratios for different modes of processing of metals.

Changes in the values of sound velocity are determined by the occurrence of priority crystallographic directions produced in the bulk material at various processing modes.

It is possible to control structural transformations in materials, utilizing the analysis of the frequency dependence of the attenuation of acoustic pulses in a wide spectral range.

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