SHORT
COMMUNICATIONS

Influence of Barocryodeformation on the Hydrogen Concentration and Acoustic Emission in VT1-0 Commercial Titanium

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Abstract—The concentration of hydrogen with different binding energies and acoustic emission initiated by indentation in VT1-0 commercial titanium in the initial state and after barocryodeformation are measured. The acoustic parameters are found to correlate with the hydrogen concentration in the material.

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A key problem of materials science is to improve the physicomechanical properties of metals and alloys. Barocryodeformation (BCD) is viewed as a promising way of solving this problem. BCD means that a metallic material is plastically deformed at cryogenic temperatures by extruding a workpiece through a die using an intermediate solid that is ductile at the temperature of deformation [1]. This process transforms a metal to a highly dispersed state and eventually improves its strength properties [2, 3].

Titanium and its alloys, which offer a high specific strength up to 450–500°C and a good corrosion resistance in many aggressive media [4], are promising materials, which are being progressively more widely used in chemical machine building, aviation industry, and other branches of industry. Therefore, development of new techniques for processing titanium-based materials and estimating their quality is of great significance.

In this work, we study the influence of BCD on the concentration of dissolved hydrogen and acoustic emission (AE) parameters in VT1-0 commercial titanium in the initial state and after BCD by 20, 45, 55, and 65%. The percentage of impurities in the material was 0.18 Fe, 0.10 Si, 0.07 C, 0.01 H, 0.04 B, and 0.12 O [5].

Hydrogen in a metal occupies traps of a different origin, i.e., traps where it has different binding energies. Therefore, it has different diffusion mobilities and can influence the strength and ductility of the material [6] as well as acoustic emission [7, 8]. The sensitivity of the AE method is extremely high: the AE parameters may be affected even by a change in the "intrinsic" hydrogen concentration as small as several

hydrogen atoms per several hundreds of thousands of atoms of a metallic matrix [9, 10].

From extrudates obtained by BCD by 20, 45, and 65% at 77 K, we prepared specimens in the form of 0.8- to 1.0-mm thick pellets 3–4 mm in diameter. The specimens (including those made of as-obtained titanium) were mechanically polished to provide a flat shiny surface. They were first subjected to indentation followed by recording of AE signals, and then the hydrogen concentration in them was measured.

Measurements of the hydrogen concentration and the distribution of hydrogen over binding energies were carried out by high-temperature vacuum extraction using an AV-1 hydrogen analyzer [11]. Successively heating the titanium specimens to 200, 350, 450, 500, 550, 700, and 800°C, we determined the amount of the extracted hydrogen (see the table). The procedure of hydrogen concentration measurement is described in detail in [12]. First, upon heating to 200°C, the hydrogen adsorbed on the surface of the specimen evolves. At 350–550°C, diffusion-mobile hydrogen (with a low binding energy) is liberated, and at temperatures above 550°C strongly bound hydrogen (with high binding energies) is released.

Figure 1 shows extraction curves for all specimens. It follows from the table and Fig. 1 that the extraction curves almost coincide. The largest amount of the hydrogen is liberated at high temperatures, i.e., when it is in the strongly bound state (with binding energies of about 1.4 eV). When the degree of BCD δ grows, so does the amount of hydrogen ($Q_1, Q_2, Q_3 < Q_\Sigma$), reaching a maximum at $\delta = 55\%$. Thus, BCD not only disperses the material but also changes the amount of

Amount of the hydrogen extracted from VT1-0 commercial titanium in the initial state and after barocryodeformation (δ is the degree of deformation)

Extraction temperature, °C	Amount of extracted hydrogen				
	Initial state	δ, %			
		20	45	55	65
200	0.19	0.22	0.47	0.65	0.39
350	0.50	0.28	0.68	0.76	0.24
450	0.88	0.53	1.01	0.85	0.77
500	0.45	0.41	0.82	1.36	0.81
550	0.59	0.50	1.34	0.51	0.38
700	6.67	7.40	6.60	6.77	6.42
850	3.52	3.36	4.04	5.17	4.16
Amount of surface-adsorbed hydrogen Q_1	0.19	0.22	0.47	0.65	0.39
Amount of diffusion-mobile hydrogen Q_2	2.42	1.72	3.85	3.48	2.20
Amount of strongly bound hydrogen Q_3	10.19	10.76	10.64	11.94	10.58
Total amount of hydrogen in specimen Q_Σ	12.80	12.70	14.96	16.04	13.17

hydrogen in it. It was therefore of interest to see how these two effects in combination influence the AC parameters.

Acoustic emission was initiated by indenting a hard-alloy cone using an IM-4A mechanical testing machine (Fig. 1). The maximal indentation load was 1000 N. To record AE signals and convert them to electrical ones, we used an MSAE-L2 transducer and an MSAE-FA010 amplifier. The total amplification was equal to 87 dB. All AE signals were subdivided into groups (by the method described in [13]) based upon the shape of the spectral density curve, and then the spectral portrait (the averaged shape of the power spectral density curve), median frequency (the frequency halving the area under the spectral density curve), and signal energy in each group were analyzed.
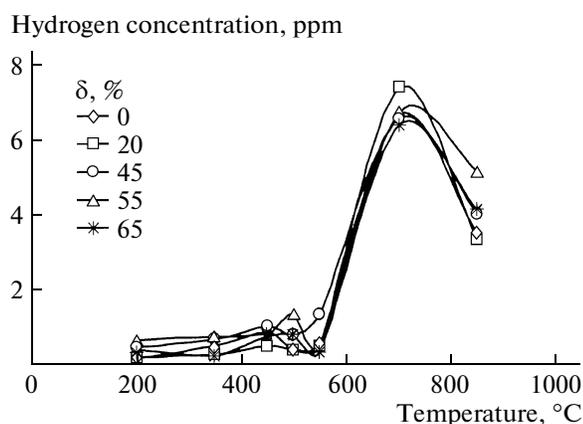
For all the specimens, the AE composition was almost uniform: 90–99% of signals belonged to one group (they seemed to be of a dislocation nature). However, the parameters of these signals varied with degree of deformation. Figure 3 plots total number N of AE signals (averaged over two indentations), signal energy E, and median frequency F versus degree of BCD δ.

As the deformation increases, so does the median frequency, while the signal energy drops. This may be related to the dispersion of the material structure under low-temperature all-round compression [1]. This changes the mobility of dislocations and, accord-
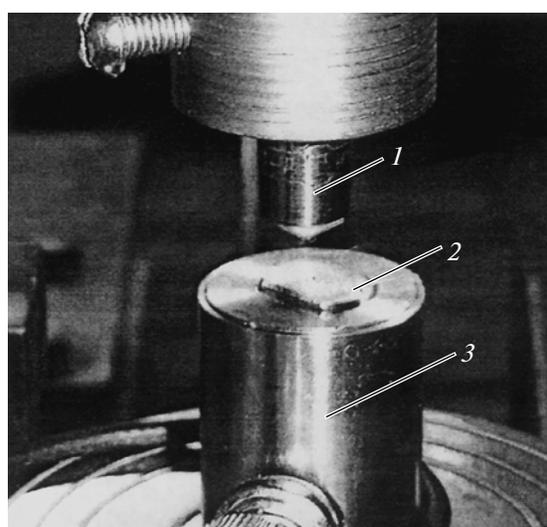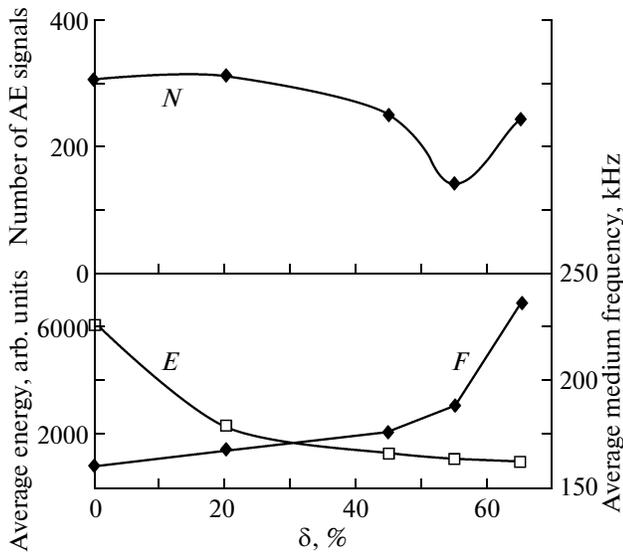

Fig. 1. Temperature variation of the amount of the hydrogen extracted from commercial titanium subjected to different degrees of BCD δ.

Fig. 2. Loading device of the setup: *1*, loading unit with conic indenter; *2*, test specimen; and *3*, MSAE-L2 acoustic emission transducer.

Fig. 3. Total number (N), mean energy (E), and mean median frequency (F) of AE signals vs. degree of BCD δ for indented specimens of the commercial titanium.

Fig. 4. Spectral portraits of AE signals upon indentation of the commercial titanium (*1*) in the initial state and after BCD by (*2*) 20, (*3*) 45, (*4*) 55, and (*5*) 65%.
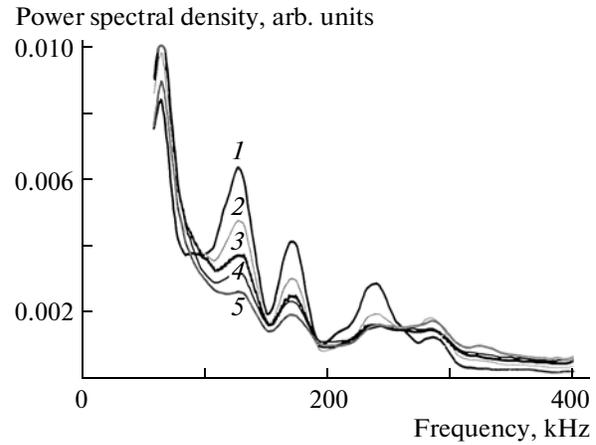
ingly, the AE parameters. Since the structure components of the titanium subjected to BCD become disperse, the dislocation free path decreases, resulting in a monotonic decrease in the AE energy and a rise in the median frequency. However, total number N of recorded AE signals depend on the degree of BCD nonmonotonically (Fig. 3).

AE spectral analysis shows that the spectral density curve levels off with an increase in the degree of BCD, the curve varying most noticeably at low frequencies (100–200 kHz) (Fig. 4).

As the degree of BCD grows, the main peaks in the curve of the AE signal composition spectral density lower.[1] This may be explained by an increase in the number of inner degrees of freedom. On the one hand, nanodimensional grains with different orientations of the crystal lattice dissipate the acoustic signal energy owing to natural oscillations; on the other hand, the energy of the acoustic signal associated with the dislocation growth decreases because grains become finer.

Comparing the acoustic parameters (Fig. 3) with the hydrogen extraction curves (Fig. 5), one easily finds a correlation between number N of AE signals and hydrogen concentration Q_Σ in the specimen (primarily concentration Q_3 of strongly bound hydrogen). However, the curves of the diffusion-mobile hydrogen (Q_2) and of the surface-adsorbed hydrogen (Q_1) also correlate with N.

These findings agree well with the well-known effect of AE suppression when a metal becomes satu-

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[1] The first peak at low frequencies is not taken into consideration, since it is at the boundary of the filter passband and so can be heavily distorted by conditions of spectral density curve normalization (normalization of the area under the curve to unity)

rated with hydrogen (see, e.g., [7, 8]) and confirm a high sensitivity of the AE method, which is capable of sensing a change even in the intrinsic hydrogen concentration (i.e., a concentration without special hydrogenation) in the metal.

Thus, we measured the concentration of hydrogen with different binding energies and indentation-initiated acoustic emission signals in VT1-0 commercial titanium in the initial state and upon barocryodeformation and found a correlation between the acoustic parameters and hydrogen concentration in the material. A high sensitivity of the AE method is supported. It is shown that this method combined with indentation is highly promising for estimating the properties of materials upon BCD.
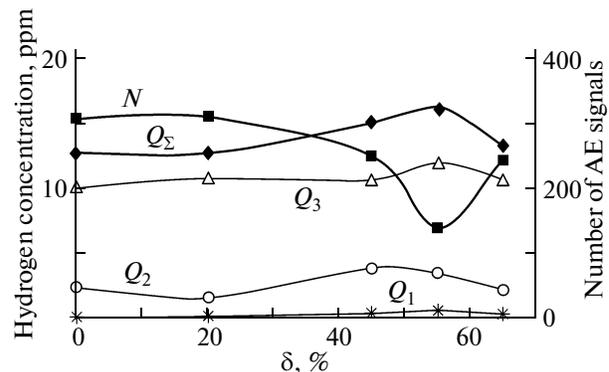
Fig. 5. Amount Q of hydrogen with different binding energies and number N of AE signals upon indentation of VT1-0 commercial titanium specimens subjected to BCD (δ is the degree of deformation). Q_1, Q_2, and Q_3 are the amounts of surface-adsorbed, diffusion-mobile, and strongly bound hydrogen, respectively, and Q_Σ is the total amount of hydrogen in the specimen.

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