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Optical-Acoustic Spectroscopy of Metal Elastic Property Modifications Due To Thermal Treatment and Shock Strengthening.

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Results of optical-acoustic researches of thermal and shock influence on change of elastic properties and durability of metals due to reorganization of their structure are submitted in the paper. To reveal the scales of modifications, we study iron, nickel, copper, and aluminum alloy specimens with various initial structures before and after shock loading. The loading was accomplished by pressure impulses of amplitude ~1.0GPa and duration ~$8*10^{-8}$s. The initial structure of the specimens was modified by vacuum annealing for 1 hour at a given temperature. After the annealing, the specimens were cooled in a furnace. Measurements of elastic waves in two orthogonal directions are carried out by the pulsed optical-acoustic method before and after shock loading. The analysis of velocities allows to determine changes of material elastic properties and to estimate the contribution of different mechanisms of plastic flow to hardening metals with various initial structures. The pulse optical-acoustic method allows to carry out measurements over a wide spectral range. It gives a chance to obtain frequency dependences of the attenuation and acoustic velocity in metal samples after preliminary heat treatment and shock hardening. The analysis of changes of the dependences allows to estimate scales of structural reorganizations which have occurred in the samples due to shock loading. It is shown, that the rotational machine of plastic flow is the basic in samples with the grain size ~ 20μm, and the dislocation and twin mechanisms - in coarse-grained samples. With this method, one can gain information on restructuring on a scale of $\geq 10^{-7}$–10^{-6}m.

1 Introduction

Interrelation between plastic deformation of materials and modifications in their structure is well known [1–4]. However, information on characteristic structure levels responsible for deformation when loading conditions and initial material structures vary is very scarce. This is because the techniques for studying restructuring under deformation are not sufficiently advanced and few complex experimental investigations of this phenomenon (especially after shock tests) have been carried out.

Upon studying elastoplastic deformation in metals due to submicrosecond shock impulses, it was found [5] that the grain size dependence of hardening differs much from the Hall–Petch relation:

$$\sigma_H = \sigma_0 + KD^{-1/2} \qquad (1)$$

were σ_0 is the strength of the matrix, K is a coefficient of proportionality, D is a grain size.

It was assumed that this is associated with a change in the scale levels of restructuring that are responsible for plastic deformation in materials with various initial structures.

2 Objects and experimental methods

To reveal the scales of modifications, we studied 0.3ZhR iron, N1 nickel, and aluminum alloy D16 specimens with various initial structures before and after shock loading. The loading was accomplished by pressure impulses of amplitude ≈1.0GPa and duration ≈$8*10^{-8}$s [5]. The initial structure of the specimens was modified by vacuum annealing for 1 hour at a given temperature. After the annealing, the specimens were cooled in a furnace.

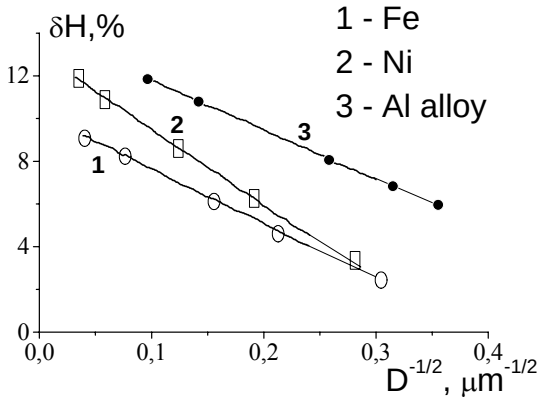

Figure 1: Dependence of the hardening on grain size.

On Figure 1 dependence of the hardening on grain size obtained in our experiments is shown. The Hall–Petch relation (1) does not hold for the plot. We have maximum of ΔH at maximal values of grain size.
These features, apparently, are due to changing of the deformation mechanism that gives the government contribution to the metal mechanical properties after deformation.
The pulsed acousto-optic method was applied [6–8] (using a 1.06μm laser with pulse width of 25ns and pulse energy of ≤30mJ) to study this features. The frequency dependence of the attenuation and acoustic velocity over a wide spectral range was obtained.
Acoustic pulses were recorded with a piezoelectric detector made of a 30μm-thick polyvinyldiphenyl film placed on a surface of acousto-optic cell made of fused quartz. The cell served as an optical path and acoustic waveguide [9]. The pass band of the detector was about 150MHz.

3 Results and discussion

Figure 2 shows the typical waveforms of the (1) first and (2) second acoustic echo pulses in the iron specimen (annealed at 100°C), first and second pulses imposed on each other, as well as the magnitudes and phases of their Fourier transforms.
In the experiments, we measured the velocity c_0 of acoustic pulse propagation, as well as determined (by analyzing the spectra of the pulses) the frequency (ω) dependence of velocity c(ω) and attenuation α(ω) from the relations [8]:

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

where $c(\omega) \approx c_0 + \Delta c(\omega)$, Δx is the specimen thickness, and $A_{1,2}$ and $B_{1,2}$ are the coefficients before real and imaginary parts of the Fourier spectrum for the first and second acoustic pulses.
Figure 3 demonstrates frequency dependences of acoustic velocity and attenuation before and after shock loading of the iron specimens annealed at 1000, 700, and 100°C. From the variation of these curves, one can estimate the scales of restructuring due to shock loading.
For the specimens with a grain size d~220μm and low dislocation density that were annealed at 1000°C, the curves $\Delta c(\omega)/c_0$ before and after the shock are almost the same throughout the frequency range. The acoustic velocity also remains unchanged, c_0= 5948±5m/s. The attenuation coefficients do not differ up to ≈25MHz. However, in the high-frequency part of the spectrum, the attenuation after the shock increases significantly because of a high concentration of linear defects (dislocations and twins) generated by plastic deformation. The characteristic size (scale) of such structure imperfections (modifications) is on the order of ~10^{-7}–10^{-6}m [1–4]. It appears that the strain hardening of the specimens with such an initial structure was the highest just for this reason [5].

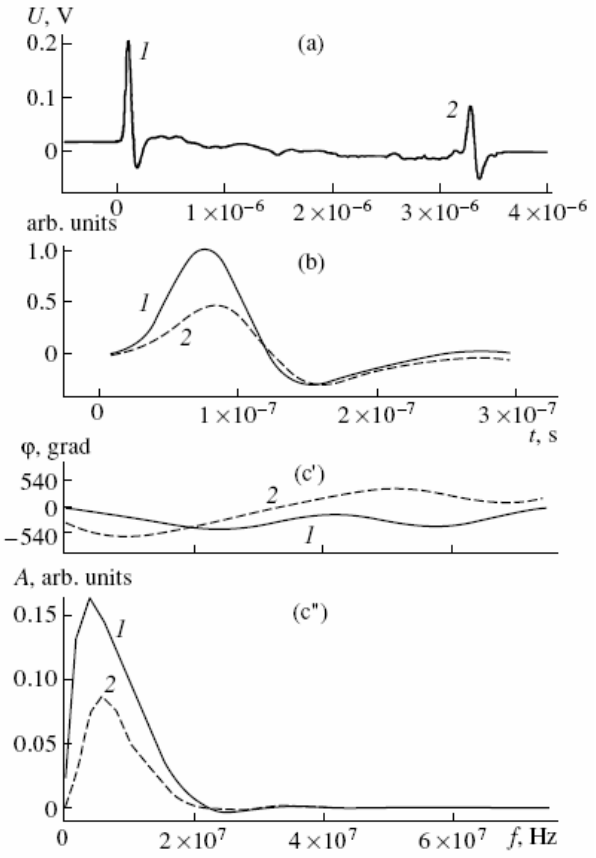

Figure 2: (a) Waveforms of the (1) first and (2) second acoustic pulses in the iron (annealed at 100°C), (b) pulses are imposed on each other, and (c') phase and (c") magnitude of the Fourier spectra of the first and second acoustic pulses.

In the specimens with the structure modified by annealing at 700°C (d≈20μm), the shock loading rises appreciably the spread in the velocity $\Delta c(\omega)/c_0$ and attenuation α(ω) throughout the frequency range. In the shock-loaded specimens, the acoustic velocity grows from 5956 to 5974m/s. This difference exceeds the measurement accuracy thrice and is associated with the change in the crystallographic orientation of entire grains or their fragments that have a characteristic size of 10^{-6}–10^{-5}m. This change is due to the rotational mechanism of plastic deformation [2–4]. Accordingly, the attenuation grows at frequencies ≤25MHz because acoustic waves scatter by fragment or grain boundaries. The increase in the attenuation after the shock in the

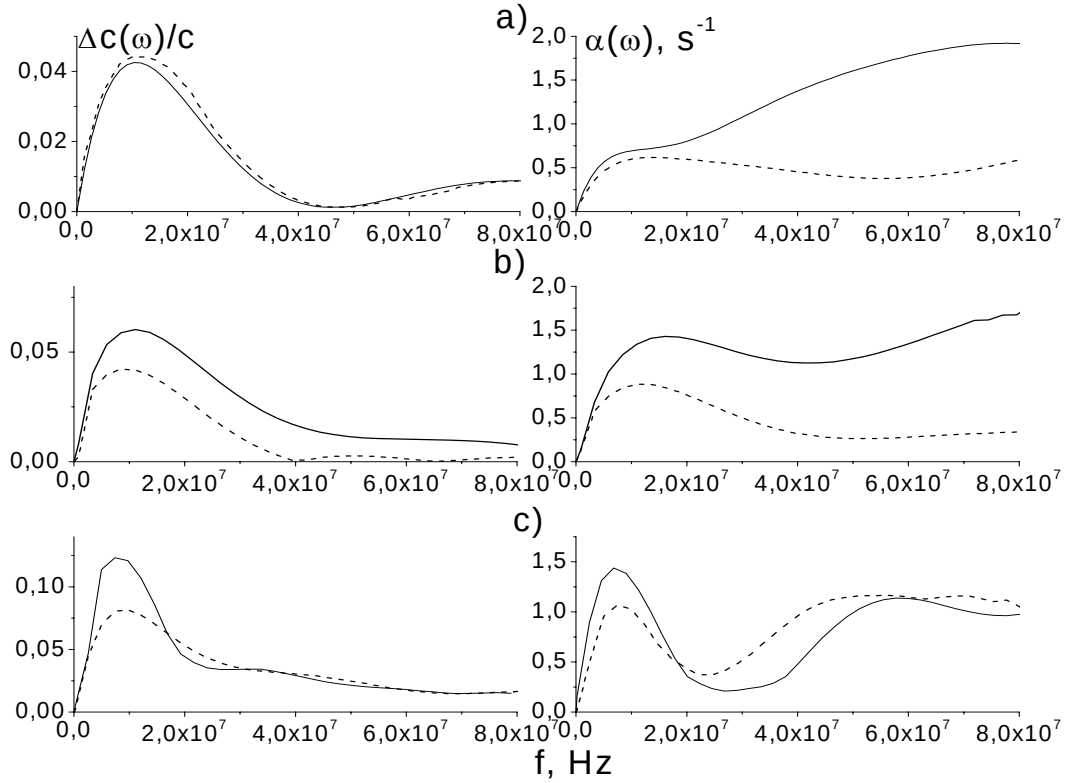

Figure 3: Frequency dependences of the acoustic velocity .$\Delta c(\omega)/c_0$ and attenuation $\alpha(\omega)$. Annealing at (a) 1000°C (d≈220μm), (b) 700°C (d≈20μm), and (c) 100°C (d≈20μm). Dashed line, before the shock; solid line, after the shock.

high-frequency part of the spectrum is due to a higher concentration of the linear defects, as before. Thus, both the rotational mechanism and defect structure modifications are responsible for plastic deformation of the iron specimen whose initial structure was obtained by annealing at 700°C.

In the specimens annealed at 100°C in which the grain size was the same as before (d≈20μm) but the starting defect density was higher, $\Delta c(\omega)/c_0$ and $\alpha(\omega)$ grow markedly after the loading only at frequencies≤25MHz. In the shock-loaded specimens, the acoustic velocity increases by 24 m/s from c_0=5966±5 to 5990±5m/s. In the high-frequency part, the velocity spreads and attenuation coefficients before and after the tests differ only slightly. A possible reason for such behavior of $\Delta c(\omega)/c_0$ and $\alpha(\omega)$ is that the rotational mechanism of plastic yield causing restructuring on the scale level comparable to the grain size becomes prevailing in the specimens with the initial structure described above.

The data for the N1 nickel specimens qualitatively agree with those for the iron specimens shown in Figure 3.

Thus, in the case of a submicrosecond shock, the rotational mechanism governs plastic deformation in polycrystals with a grain size on the order of the shock loading front (≈10^{-5}m). According to [5], the strain hardening in such specimens is the lowest.

Our results combined with those in [5] also indicate that the violation of the Hall–Petch relation (1) (a rise in the hardening with grain size) is explained by the decisive role of the intragranular defect structure modification (an increase in the concentration of dislocations and twins) in the strain hardening. For a submicrosecond shock loading, this effect is the most pronounced in coarse-grained specimens and apparently in single crystals.

To determine the contribution of rotational mechanism, we made the measurements of longitudinal sound velocity in samples with different grain sizes. The velocities in directions along and across the direction of disturbance wave propagation were measured. The inaccuracy of measurement was smaller than 0.1%.

These data together with values of sound velocity in crystal in direction of maximal anisotropy [10] enabled us to estimate the changing of crystallographic orientation of metal structure Δn. (Δn – is the number of grains that changed their crystallographic orientation). The dependence Δn from D is shown on Figure 3. Δn increases substantially if grain size is less then the spatial dimension of the shock loading front (about 40 μm in our case).

$$D \le c_l \tau_f \qquad (3)$$

At this grain size the rotational mechanism of plastic flow becomes the deciding mechanism in the relaxation of pulse stress. It is confirmed by practically the complete absence of shock hardening in samples when D<40 μm.

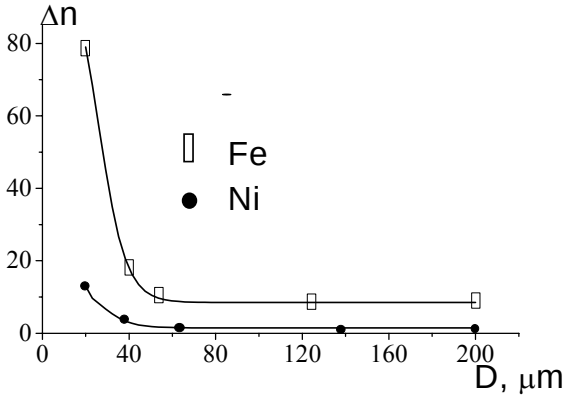

Figure 3: Dependence Δn on D.

To conclude, we note that the method of acousto-optic spectroscopy has fast response and shows promise for studying restructuring in metals. The pulse optical-acoustic method allows to carry out measurements over a wide spectral range. With this method, one can gain information on restructuring on a scale of $\geq 10^{-7}$–10^{-6}m.

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