Influence of ultrasound on the structure and properties of nickel processed by equal-channel angular pressing

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Abstract. Commercially pure nickel was processed by equal-channel angular pressing (ECAP) and subjected to ultrasonic treatment (UST) with a stress amplitude of 100 MPa. The microstructure, microhardness and mechanical properties of the deformed and sonicated samples were studied. It was shown that the UST affected the microstructure of ECAP nickel. A refinement of grains in the longitudinal section, changes in the texture, and an increase in the dislocation density and microstrain were observed. Tensile tests have shown an increase in the elongation to failure without any noticeable changes in the tensile strength. The possible mechanisms of the action of ultrasound on the structure and properties of the deformed nickel are discussed.

1. Introduction

Technologies for the production and processing of metallic materials based on the ultrasonic effects are now widely used. Depending on the parameters of the ultrasonic action and the material used, it is possible to obtain its certain structural states and properties. For example, the use of ultrasound for the crystallization of melts leads to a more uniform and fine-grained structure of cast metals and alloys [1]. The impact of high intensity ultrasound is used for surface hardening of materials [2]. At present, ultrasonic welding of various materials finds its application and provides the basis for the development of a new technology of ultrasonic additive manufacturing [3].

At the same time, much attention is now paid to the study and application of materials subjected to severe plastic deformation, which demonstrate high strength, fatigue, and other improved properties but suffer from structural features that result in a low ductility, impact toughness and thermal stability of the microstructure [4]. Annealing used for the structure relaxation of deformed materials often leads to an undesirable grain growth that decreases their strength properties.

Earlier it was shown that the moderate-intensity ultrasonic treatment (UST) reduced the microstrain and dislocation density and increased the thermal stability of ultrafine-grained pure nickel processed by high-pressure torsion [5,6]. UST of commercially pure nickel subjected to ECAP can lead both to relaxation and hardening of the structure, depending on the stress amplitude that affects the mechanical properties [7-9], and can increase the impact toughness of the material [10].
In this paper, the effect of UST with a stress amplitude of 100 MPa on the structure and properties of commercially pure nickel processed by ECAP is investigated. This amplitude was chosen on the basis of the results of our previous studies [7-9].

2. Experimental materials and procedure

The rods of commercially pure nickel (grade NP2 according to the Russian classification) with a length of 100 mm and a diameter of 20 mm were subjected to 8 passes of ECAP following route Be at a temperature of 350 °C on a tool with an intersection angle of channels 120°. Then, the resulting samples were extruded at a temperature of 300 °C to a length of 115 mm and a cross-sectional diameter of 18 mm. The total deformation degree was equal approximately to 8.

Two samples after deformation were processed by UST according to the scheme described in [8]. A standing wave with a sinusoidal stress distribution was excited in the sample. The antinode of compression-tension stresses was located in the center of the rod, their amplitude was approximately equal to 100 MPa.

Cylinders with a length of 32 mm were cut from the central part of the samples after ECAP and after ECAP + UST. The change in the amplitude of the UST at a small distance from the center of the sample is negligible, and it can be assumed that over this length the amplitude of the UST was equal to 100 MPa. From these cylinders, flat samples for tensile tests in the longitudinal direction and specimens for studying the microstructure and measuring the microhardness were cut.

Tensile tests were carried out on an Instron testing machine at room temperature. The specimens for these tests had a gauge length of 10 mm, width of 3 mm and thickness of 1.5 mm. The Vickers microhardness of the samples was measured using an Axiovert 100A equipment with an MNT_10 attachment with a load on the indenter of 100 g and an exposure time of 10 s.

EBSD analysis was performed using a TESCAN MIRA 3 LMH FEG microscope. Scanning was carried out with steps 100 and 50 nm from square grids with areas of 400 and 100 µm² respectively. The surface of the samples for scanning was prepared by mechanical and subsequent electrolytic polishing.

X-ray diffraction (XRD) analysis of the investigated cylindrical samples was carried out on a DRON-4 apparatus in planes perpendicular and parallel to the axis of the cylinder. The CuKα line was used with Bragg-Brentano focusing. A Soller collimator for primary beam and a graphite monochromator for the diffracted beam were used. The experimental data were processed by means of the software «Maud» (Materials Analysis Using Diffraction).

3. Experimental results

Figure 1 shows the microstructure of nickel after ECAP and after ECAP + UST in longitudinal and cross sections, obtained by EBSD analysis. It is noticeable that after the UST the grains are visually finer. In the cross-section, twins are observed in some grains (shown by arrows in figures 1c and d).

The quantitative results of X-Ray diffraction and EBSD analyses for longitudinal and cross sections are presented in table 1. It can be seen that the grain sizes in both states are rather large (2-4 microns). UST facilitated a reduction in the average grain size and a slight increase in the microstrain in the longitudinal direction, but the microstrain in the cross direction practically did not change. The dislocation density also increased after the UST.

The UST affected the distribution of grain boundaries by the misorientation angle. Table 1 presents the fractions of three types of grain boundaries: low-angle boundaries (LABs, misorientation angle from 2° to 15°), high-angle boundaries (HABs, >15°), and boundaries close to special ones (CSL). The volume fraction of LABs did not change, but the fraction of CSL significantly decreased (especially in the longitudinal section), a part of them apparently were transformed into the HABs.

Figure 2 shows the inverse pole figures of nickel after ECAP and after ECAP + UST in longitudinal and cross sections, composed according to X-Ray diffraction data. In the initial deformed state, one can see a multicomponent texture with preferential orientations <100> and <110>, in addition, there
are components <311> and <210>. After the UST, the intensity of some of them decreased, and a
tendency to form a one-component texture with a preferential orientation <100> is observed.

Table 1. XRD and EBSD data for longitudinal (l.s.) and cross sections (c.s.) of samples after ECAP
and after ECAP+UST with the amplitude of 100 MPa.

| State           | X-Ray | EBSD                  |
|-----------------|-------|-----------------------|
|                 | Domain size, nm | Microstrain, ×10⁻⁴ | Grain size d, µm | Dislocation density, m²² | LAB, % | HAB, % | CSL, % |
| ECAP, l.s.      | 332.6±5.3 | 4.78±0.14 | 4.4 | 1.8 | 13.21×10⁻¹³ | 52 | 29 | 19 |
| ECAP+UST, l.s.  | 354.6±7.1 | 6.02±0.32 | 2.4 | 1.2 | 18.80×10⁻¹³ | 54 | 38 | 8  |
| ECAP, c.s.      | 240.1±3.8 | 6.49±0.21 | 3.3 | 13.58×10⁻¹³ | 43 | 21 | 36 |
| ECAP+UST, c.s.  | 311.1±4.1 | 6.37±0.57 | 2.9 | 16.55×10⁻¹³ | 50 | 17 | 33 |

Figure 1. Microstructure of nickel after ECAP (a, c) and after ECAP+UST with the amplitude of
100 MPa (b, d). a, b - longitudinal section (the deformation axis is horizontal), c, d – cross-section.
Arrows show the twins in the structure.

Figure 2. Inverse pole figures of nickel in longitudinal (a, b) and cross (c, d) sections: a, b - after
ECAP, b, d - after ECAP+UST with amplitude of 100 MPa.
The results of tensile tests and measurements of the microhardness of nickel after the treatments are given in table 2. It can be seen that the elongation to failure increases by 8-9%, which is more than a quarter increase in the elongation as compared to the initial ECAP-deformed state. At the same time, there are no noticeable changes in the tensile strength and the microhardness.

Table 2. Mechanical properties and microhardness of nickel after ECAP and ECAP+UST with the amplitude of 100 MPa.

| State      | Ultimate strength, MPa | Elongation to failure, % | Microhardness Hv, МПа |
|------------|------------------------|--------------------------|------------------------|
| ECAP       | 439±18                 | 32,5±2,5                 | 1574±27                |
| ECAP+UST   | 440±21                 | 41,3±2,0                 | 1554±59                |

In our earlier paper [7], it was also shown that the UST resulted in texture changes of nickel subjected to ECAP. Moreover, after the UST a slight increase in the ductility of nickel was obtained during the tensile tests without noticeable changes in the tensile strength. However, in the present study, the high-amplitude UST obviously led not to a relaxation of the internal stresses, as it was in [7], but, on the contrary, to their increase. This is probably due to additional grain refinement and an increase in the density of dislocations. But the UST process is different from the deformation by its mechanisms. Both the generation and the accumulation of dislocations, as well as their reorganization, annihilation inside the grains take place during the action of oscillating stresses. The contribution of each of these mechanisms, as was shown in [5-8], depends on the amplitude of the UST. Therefore, a decrease of the grain size does not lead to a significant increase in the internal stresses and other characteristics, and even contributes to some increase in the ductility of the material.

Thus, our recent and present studies show that the effect of UST on the structure and mechanical properties of UFG materials can be very diverse. This can lead to an increase in the ductility and a further increase in the ultimate strength, or to an increase in the ductility without a changing the strength. The effect obviously depends on the amplitude of the ultrasound and on the initial state of the UFG material. In order to establish all regularities of the structure and property changes in UFG metals under ultrasound, further systematic studies are needed.

4. Conclusions
The results of microstructure studies, tensile tests and microhardness measurements show that the UST with an amplitude of oscillating stresses equal to 100 MPa of nickel processed by ECAP with a moderate deformation degree results in texture changes (the tendency to form one-component texture), a reduction of the average grain size, a slight increase of dislocation density and a noticeable increase in ductility without changes in tensile strength and microhardness. This effect is related to specific mechanisms of changes in the microstructure operating during the UST process.

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References
[1] Abramov O V 1999 High-Intensity Ultrasonics: Theory and Industrial Applications (CRC Press, Boca Raton) pp 515-522
[2] Mordyuk B N and Prokopenko G I 2007 J. Sound Vibr. 308 855
[3] Gallego-Juarez J A and Graff K 2014 Power Ultrasonics. Applications of High-Intensity Ultrasound (Woodhead Publishing) pp 313-332
[4] Valiev R Z, Zhilyaev A P and Langdon T G 2014 Bulk Nanostructured Materials:
Fundamentals and Applications (New Jersey: Wiley) p 338

[5] Samigullina A A, Mukhametgalina A A, Sergeyev S N, Zhilyaev A P, Nazarov A A, Zagidullina Yu R, Parkhimovich N Yu, Rubanik V V and Tsarenko Yu V 2018 Ultrasonics 82 313

[6] Nazarova A A, Mulyukov R R, Rubanik V V, Tsarenko Yu V and Nazarov A A 2010 Phys. Metals Metallogr. 110 574

[7] Samigullina A A, Tsarenko Yu V, Rubanik V V, Popov V A, Danilenko V N and Mulyukov R R 2012 Letters on Materials 2 214

[8] Samigullina A A, Nazarov A A, Mulyukov R R, Tsarenko Yu V and Rubanik V V 2014 Rev Adv. Mater. Sci. 39 48

[9] Zhilyaev A P, Samigullina A A, Medvedeva A E, Sergeev S N, Cabrera J M and Nazarov A A 2017 Mater. Sci. Eng. A 698 136

[10] Samigullina A A, Mulyukov R R, Nazarov A A, Mukhametgalina A A, Tsarenko Yu V and Rubanik V V 2014 Letters on Materials 4 52