

Bulk Ultrasonic Treatment of Crystalline Materials

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Abstract: Ultrasound is widely used in the treatment of materials. Its applications in melt processing, surface hardening or finishing, metal forming, welding, etc., are well known and have been reviewed in numerous review articles and books. Among ultrasound-assisted processes, the direct action of ultrasonic waves on the structure and properties of bulk materials is of special interest. Ultrasonic waves induce oscillating shear stresses in materials which exert mechanical forces on crystal lattice defects, primarily on dislocations, which can yield a number of interesting effects on the structure and properties of crystals. The present paper aims to review studies on the effects of ultrasonic treatment (UST) on crystalline materials. First, the methods for the excitation of standing ultrasonic waves in bulk samples of materials are analyzed. Then, early studies on the effect of UST on the dislocation structure and phase composition, hardness and strength of materials with different initial structures are analyzed. An emphasis is then made on the influence of UST on the structure and mechanical properties of advanced ultrafine-grained (UFG) metals and alloys processed by severe plastic deformation (SPD). The results of simulations of ultrasound's effect on the dislocation and atomic structures of materials by dislocation and molecular dynamics methods are also reviewed.

Keywords: ultrasonic treatment (UST); dislocation structure; hardening; softening; microstructure; dislocation dynamics; molecular dynamics; atomic structure



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1. Introduction

Ultrasonic action is one of the most versatile methods used for the treatment of materials. For instance, the ultrasonic irradiation of crystallizing melts of metals or alloys is known to result in a more homogeneous and fine-grained structure of ingots and correspondingly in their higher strength and ductility as compared to ingots obtained by ordinary casting [1,2]. The application of ultrasound in the treatment of liquid metals has been reviewed in a number of books and reviews and is a branch of modern metal processing technology. Another direction of the application of ultrasonics in the processing of metals is related to the acoustoplastic effect, i.e., to an enhancement in the strain rate or a reduction in the flow stress of metals upon a superposition of ultrasonic stresses to the quasistatic deformation stress, which was first discovered by Blaha and Langenecker [3]. This effect gave rise to various efficient methods of metal forming, such as forging, drawing, rolling, etc., with ultrasound [4–6]. A third wide area for applications of ultrasound in materials technology pertains to surface treatment (ultrasonic peening, ultrasonic surface hardening, ultrasonic impact treatment, ultrasonic finishing, etc.) [7–10]. Another application of ultrasonic vibrations, ultrasonic metal welding, has grown into a mature technology applied in many industries nowadays [11] and given rise to an important branch of additive manufacturing referred to as ultrasonic consolidation, or ultrasonic additive manufacturing [12,13]. Additionally, ultrasound is widely used in many other metal processing technologies, such as cutting, machining, etc. [14].

The above-mentioned applications utilize different processes induced by ultrasonic vibrations and waves. The ultrasonic treatment (UST) of metal melts is based on processes occurring in sonicated liquids, such as cavitation, degassing, etc. [1,2]. An acoustoplastic effect is caused by a superposition of oscillating stresses induced by ultrasonic waves with

an applied quasistatic stress, which in combination result in an increase in the mobility of dislocations [15,16]. Ultrasonic peening is carried out by an impact deformation of metal surfaces by a hard tool, the high-frequency vibrations of which are driven by ultrasonic equipment [17]. Ultrasonic welding occurs due to friction and shear deformation, resulting in the bonding of metal sheets or wires under the influence of a vibrating welding tip clamping them to each other and to a die [11].

Among all these processes, the direct action of ultrasonic waves on the structure and properties of bulk solids is of special interest. Ultrasonic waves induce oscillating shear stresses in materials which exert mechanical forces on crystal lattice defects, primarily on dislocations. Depending on the characteristics of the waves, the state of materials and other factors, this can yield a number of interesting effects on the structure and properties of metals. Ultrasound itself, without any static stresses, can significantly influence the dislocation structure and properties of materials. Experimental studies and simulations have shown that these effects can span a wide range of effects from the accumulation of dislocations to their rearrangement or annihilation and from work hardening to work softening.

The present paper aims to review studies on ultrasound's effects on materials. Since impact surface treatment using ultrasound and melt processing by ultrasound are often called "ultrasonic treatment", the direct treatment of solid materials by alternating stresses induced by bulk ultrasonic waves is referred to as the bulk ultrasonic treatment (bulk UST) of crystalline materials. Since this paper is completely devoted to the bulk UST, we will further omit the word "bulk" and simply say "UST", but one should keep this difference in mind. Additionally, only the treatment of crystalline materials will be considered. Thus, the ultrasonic treatment of melts and surface treatments are out of the scope of the present review.

2. Mechanisms of the Effect of Ultrasound on the Structure of Materials

First of all, let us consider the basic mechanisms of the effect of ultrasonic waves on the microstructure of materials.

Ultrasonic waves used in the treatment of materials normally have frequencies near $f \approx 20$ kHz. The sound speed in metallic materials, $c \sim \sqrt{E/\rho_m}$, where E is the Young's modulus and ρ_m is the density of material, is about 5×10^3 ms⁻¹. Then, the wavelength of the ultrasound, $\lambda = c/f$, has values of a couple hundred millimeters. All the microstructural parameters of materials (grain or subgrain sizes, average distances between dislocations, etc.) are much less as compared to this length of scale. Therefore, the origin of the effect of ultrasonic waves cannot be related to any size effect. The fact that size effects do not play a significant role in the influence of UST on the structure of metals is confirmed indirectly by the experiments carried out in [18]. In the cited work, the dislocation structure of Ni was studied after cyclic straining with essentially differing frequencies of 50 Hz and 20 kHz up to 10^6 cycles at temperatures of $T = 770$ and 300 K. The dislocation structures formed in all cases were similar, which indicated that elastic waves with different wavelengths exerted the same effects on dislocations.

On the other hand, ultrasonic waves induce oscillating elastic stresses in materials. Usually, UST processes are carried out in a resonant regime in which a standing ultrasonic wave is excited in materials. In this case, at any point of a material, a stationary stress field of the form $\hat{\sigma}(\vec{r}, t) = \hat{\sigma}_0(\vec{r}) \sin(2\pi ft)$ is induced with the amplitudes of the vibrations depending only on the coordinates of the point. It is this stress field which causes the forces acting on crystal lattice defects.

The main defects that are generated and accumulated, modifying the microstructure of crystalline materials, are dislocations [19,20]. Dislocations are generated by different sources and move under the effect of an applied stress due to a mechanical force given by the Peach–Koehler formula, according to which the force acting on a segment $d\vec{l}(\vec{r})$ of a dislocation with the Burgers vector $\vec{b}$ in an effective stress field $\hat{\sigma}_{ef}(\vec{r})$ is given by the

equation $d\vec{f} = \left(\hat{\sigma}_{ef} \cdot \vec{b} \right) \times d\vec{l}$ [20]. In general, the effective stress field consists of an internal field, $\hat{\sigma}_{in}$, created by other crystal lattice defects and an external one, which can have static and time-dependent components: $\hat{\sigma}_{ex}(\vec{r}, t) = \hat{\sigma}_{ex0}(\vec{r}) + \hat{\sigma}_{ex}(\vec{r}, t)$. If the time-dependent component is induced by a standing ultrasonic wave, then the effective stress takes the form $\hat{\sigma}_{ef}(\vec{r}, t) = \hat{\sigma}_{in}(\vec{r}) + \hat{\sigma}_{ex0}(\vec{r}) + \hat{\sigma}(\vec{r}) \sin(2\pi ft)$, where $\hat{\sigma}$ is the amplitude of the oscillating stress.

A variety of effects can occur for different combinations of the three components of this stress. When ultrasonic stress acts simultaneously with quasistatic stress, dislocations can overcome barriers, which are caused, for instance, by internal stresses, and one observes acoustic softening, i.e., an increase in the strain rate. When only an ultrasonic stress of a low amplitude is applied, dislocations vibrate without overcoming barriers, and this regime is used in mechanical spectroscopy [21]. An intensive ultrasound (with high values of $\hat{\sigma}$) applied to a non-deformed metal ($\hat{\sigma}_{in} = 0$) results in the generation of dislocations and ultrasonic hardening. If a material is preliminarily deformed so that a high level non-uniform internal stress field $\hat{\sigma}_{in}$ exists, the combined action of this field and the applied ultrasound, $\hat{\sigma}_{in} + \hat{\sigma} \sin(2\pi ft)$, has an effect which depends on the amplitude of the ultrasound. It is expected that in some range of moderate amplitudes, which allow for the breaking out of dislocations from local barriers but are not enough for their intensive generation, oscillatory stresses induce a drift movement of existing dislocations and the subsequent transformation of a dislocation structure, the overall trend of which is a full or partial cancellation of the internal stresses $\hat{\sigma}_{in}$. This kind of interaction of ultrasonic waves with a material will result in a recovery of its structure similar to the effect of annealing.

In this review, we will focus on the effects caused by ultrasonic treatment in an absence of static applied stresses, i.e., $\hat{\sigma}_{ex0} = 0$. First, we will consider the cases, when $\hat{\sigma}_{in} = 0$, i.e., in an initial state before UST, a material does not contain defects with long-range stress fields. In a real experiment, of course, this condition can be fulfilled only approximately, since even well-annealed materials contain a certain density of defects. Then particular attention will be given to the effect of ultrasound on materials subjected to heavy deformation, which contain a high density of defects with long-range stress fields and thus are in a high-energy nonequilibrium state. These considerations will be preceded by an analysis of the methods of the excitation of ultrasonic waves in bulk materials using ultrasonic vibration systems.

3. Methods and Instruments of Bulk Ultrasonic Treatment of Metals

In order to induce alternating stresses in a bulk material, one should excite ultrasonic waves, which is only possible if there is an acoustic contact between a source of vibrations and a sample of the material. This can be done either by the direct attachment of a sample to a vibrating surface or through an immersion liquid. The first studies on the effect of ultrasound on deforming metals were based on the use of an immersion liquid. For example, in their pioneering work, Blaha and Langenecker [3] placed a tensile specimen of zinc in a liquid bath in which ultrasonic oscillations were generated. This method allows for transmitting a sonic energy with intensities lower than 10 Wm^{-2} only, since the deterioration of sample surfaces due to cavitation can occur at higher intensities [22]. Ultrasonic waves of higher intensities can be induced only in a sample directly attached to a concentrator of an ultrasonic vibrating system.

A typical ultrasonic vibration system is schematically depicted in Figure 1. Ultrasonic generator 1 generates electric oscillations, which are transmitted to ultrasonic transducer 2. The latter can be piezoceramic or magnetostrictive and transforms electric oscillations to mechanical vibrations, i.e., oscillating displacements at the end face of the transducer. Vibrations are in a resonance when the frequency of the electric signal is equal to the natural frequency of the longitudinal vibrations of the transducer. Normally, transducers vibrate with an amplitude up to 10–15 μm . In order to obtain vibrations with higher amplitudes, concentrator 3 is used, which has the same natural frequency as the transducer and amplifies and transfers vibrations to an instrument or to sample 4 attached to it.

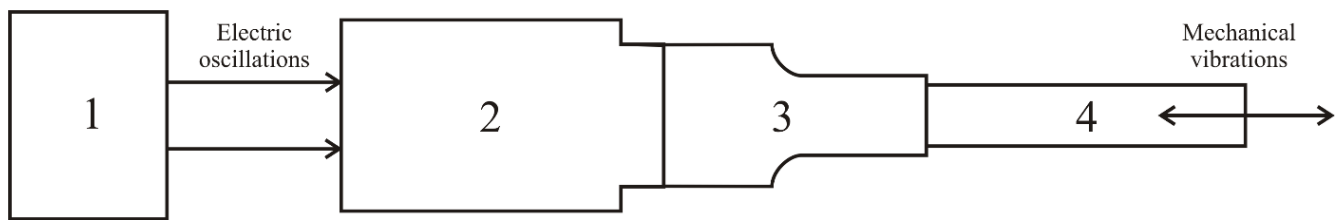


Figure 1. Schematic representation of a typical ultrasonic vibration system: 1—ultrasonic generator; 2—ultrasonic transducer; 3—concentrator; 4—an instrument (sample).

The simplest and most direct method of bulk UST consists of the use of a half-wave sample of the study material (Figure 2). Indeed, if one attaches a bar of metal, the length of which is equal to the half-wavelength of ultrasound in this metal, i.e., $l = \lambda/2 = c/2f$, a standing wave is excited, in which the amplitude of the displacements along the sample changes according to the following relation:

$$\xi = \xi_0 \cos kx, \quad (1a)$$

where ξ_0 is the maximum amplitude at which the sample ends vibrate, $k = 2\pi/\lambda$ is the wavenumber and x is the distance from the end of the bar attached to the concentrator (Figure 2). Then the amplitudes of the normal elastic strain and stress are calculated, respectively, as $\varepsilon(x) = |d\xi(x)/dx|$ and $\sigma(x) = E\varepsilon(x)$, where E is the Young's modulus of the metal, i.e.:

$$\varepsilon(x) = \varepsilon_0 \sin kx, \quad \varepsilon_0 = k\xi_0, \quad (1b)$$

$$\sigma(x) = \sigma_0 \sin kx, \quad \sigma_0 = Ek\xi_0. \quad (1c)$$

The graphs of the dependencies of displacement and strain/stress amplitudes on a position along the bar are shown in Figure 2. One can see that the displacements are zero in the middle of the bar, $x = l/2$, while the stresses are at their maximum.

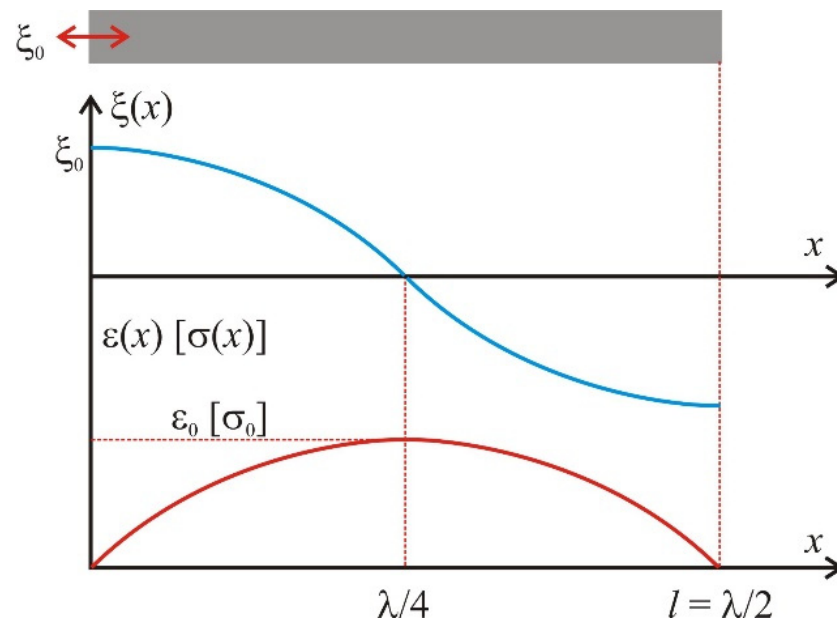


Figure 2. Schematic representation of a half-wave sample and the distribution of displacement, strain and stress amplitudes along it.

As one can see from the above consideration, the use of half-wave samples provides a very convenient way to study the effect of ultrasound with different amplitudes on the structure of materials on a single sample. Indeed, different cross-sections of the samples experience oscillating normal stresses with amplitudes in the range from 0 to σ_0 , and

based on Equation (1c) one can easily calculate the stresses measuring the displacement amplitude at the end of the bar. Due to its convenience, this method has been widely used in experiments. For instance, Langenecker [23] used a half-wave specimen with a length of 60 mm corresponding to a frequency of about 40 kHz clamped in special equipment which made it possible to carry out in situ observations of its dislocation behavior under the action of ultrasound in a column of a microscope. Based on this method, Belostotskiy et al. [24] and Samigullina et al. [25] studied the effect of UST on the microstructure and properties of nickel in a wide range of amplitudes of oscillating stresses up to 150 MPa and 100 MPa, respectively. If a sample of a resonance length has a dog-bone shape, i.e., a thinner central part, the stress amplitude in this part can be enhanced and the upper limit of the UST intensity can be increased [26].

In some early studies, the samples themselves did not have a resonance length corresponding to a frequency of the ultrasonic transducer used. For example, in [27], copper samples with a length of 10 mm were sonicated in a vibrating system with a frequency of 20 kHz. Unfortunately, the authors did not report details on the measurements of the stress amplitudes in their experiments. In general, there are two main disadvantages of using samples with lengths significantly different from those of the resonance ones. First, the vibrations of the whole system occur at a frequency far from the nominal frequency of a transducer, which significantly impairs its operation and may result in its failure. Second, the calculation of stress amplitudes at points of interest becomes more complicated in this case.

Nevertheless, in many cases, it is impossible to make resonance-length samples of a material. For instance, beginning in the 1980s, nanocrystalline materials have been intensively studied [28]. In many cases, these materials are produced in very small quantities; the specimens normally are discs with a diameter about 10 mm and a thickness of the order of a millimeter. In such cases, special instruments can be made in which small metal samples are tightly clamped so that a good acoustic contact is provided. Such instruments, to the authors' best knowledge, were first used in studies on the effect of ultrasound on the structure of ultrafine-grained materials [29–31]. These instruments and the corresponding schemes of UST are presented in Figure 3.

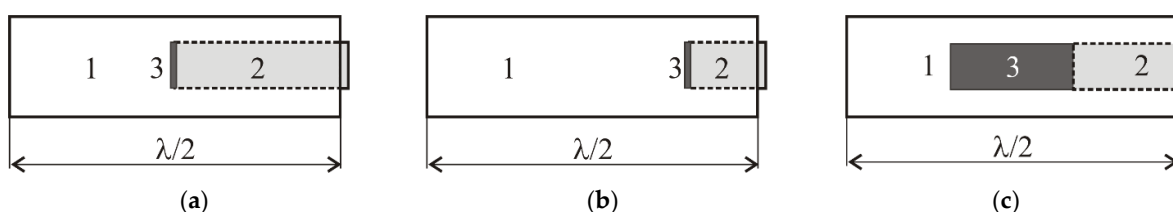


Figure 3. Three types of waveguides for ultrasonic treatment of samples with non-resonant dimensions are as follows: for disc samples, clamping them in a stress antinode (a); or alternatively, on an intermediate position with non-zero stresses (b); for cylindric samples with lengths significantly less than a half-wavelength of ultrasound (c). 1—half-wave cylinder; 2—clamping cylinder; 3—sample.

In the first case (Figure 3a), a disc-shaped sample is clamped into a cylindric waveguide exactly at its middle plane. The waveguide can be made of any sufficiently hard, suitable material that has a high sound conductivity (some steels, titanium and its alloys). The surfaces of samples, the bottom of the hole of the cylinder and the end of the clamping cylinder should be well polished in order to provide a good mechanical contact for all surfaces. In this case, the sample will experience oscillating normal stresses, which can be easily calculated by the same formula (Equation (1c)) as for that of a half-wave sample. Independently of the material of a sample, the Young's modulus of the waveguide material should be used in this case. This scheme was used in [30]. Placing a sample in an antinode of stresses puts fairly strict demands on the quality of surfaces and is associated with significant losses in the energy of waves. A compromise can be achieved by placing the sample at some distance from the middle plane, as shown in Figure 3b [29]. Figure 3c

represents the case of a cylindric sample with a length significantly less than the half-wave length. Processing in such an instrument allows one to sonicate samples suitable for further tensile tests to determine their mechanical properties [31].

As an example of the real equipment used for UST, a photo of a vibration system with the waveguide depicted in Figure 3c is presented in Figure 4 [32].

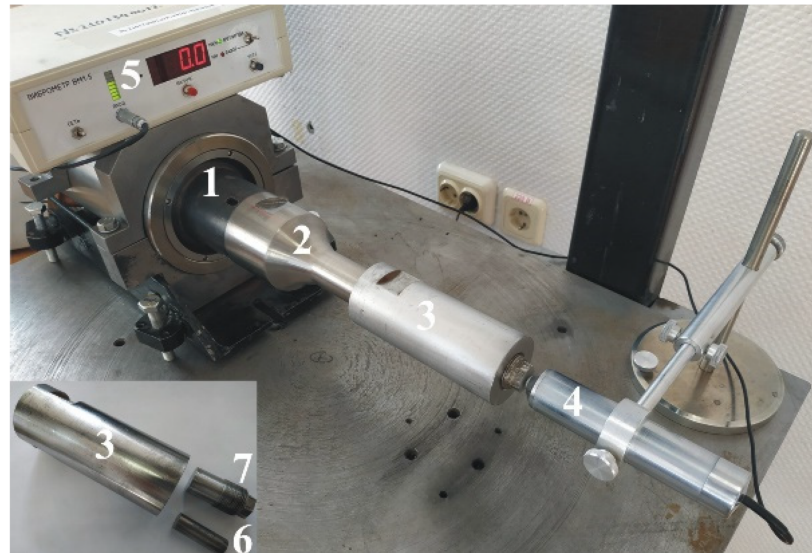


Figure 4. An example of equipment for ultrasonic treatment: ultrasonic transducer (1), concentrator (2), waveguide with a sample (3), capacity sensor (4), amplitude-detecting device (5), sample (6) and clamping cylinder (7) [32].

4. Structural Changes in Metals and Alloys under Ultrasonic Irradiation

4.1. Zinc Crystals

The first studies on the effect of ultrasound on the dislocation structure of metals were carried out by Langenecker and co-workers shortly after their discovery of the acousto-plastic effect [23,33]. In [33], ultrasound was applied during room-temperature tensile tests of zinc crystals. At low amplitudes of vibrations, their repeated application did not result in work hardening after the elimination of the ultrasound. When the sound pressure amplitude became higher, above approximately 1.2 MPa, after each ultrasonic action the flow stress increased and levelled up after several actions.

Oelschlagel [34] attached zinc samples to a vibrating system and irradiated them for 10 min at intensities above 15 W/cm². The yield stress of the samples significantly increased after the treatment as compared to that of unsonicated samples; moreover, the greater the energy of the ultrasound, the larger was the enhancement. At the highest amplitude, a yield point in the deformation curve was observed, which indicated the lack of mobile dislocations.

4.2. Aluminum and Its Alloys

In [35], specimens of high-purity Al with a 2.3 mm diameter and 50 mm length were annealed at 500 °C for 5 min prior to irradiation. Then they were screwed on the tip of an ultrasonic transducer horn. Irradiation was performed for 3 min at about a 25 W/cm² intensity in a pulse regime. The authors observed a significant macroscopic bending of the samples by the end of the treatment; however, as they assumed, the temperature increase was not more than 40 °C. Unfortunately, this effect was not discussed or given any explanation. Probably, this happened due to an accumulation of a high density of dislocations of the same sign, which resulted in a curvature of the material. Transmission electron microscopy (TEM) studies of central zones of the sample cross sections were carried out for both the sonicated and non-sonicated samples. Seven sections of the irradiated

specimen differently located with respect to the plane of attachment to the horn were examined and, interestingly, the microstructure was found not to depend on their position. Given the fact that the stress amplitudes were different at these sections, as discussed above in Section 2, one should expect differences in the dislocation density. As the authors noted, all areas contained a well-formed dislocation cell structure.

Langenecker [23] developed special equipment for in situ observations of processes occurring during the ultrasonic irradiation of samples in the column of an electron microscope. A specimen tightly attached to a horn with one end and free on the other end was introduced into the column of a microscope so that it could be tilted, rotated and moved. The specimen had a length of about 60 mm which is equal to the half-wavelength for a frequency of 40 kHz. Using the developed equipment, the author directly observed the generation of dislocations and formation of subboundaries consisting of dislocation networks during the in situ UST of pure aluminum. Unfortunately, to the authors' best knowledge, there was no continuation of this very interesting work.

Vildanova et al. [36] studied the effect of ultrasound on the structure and mechanical properties of aluminum and aluminum alloy AL27-1. An UST was carried out in an oil bath at temperatures of 70–120 °C and holding times of 0.5–1 h. While small ingots of AL27-1 alloy with a diameter of 14 mm did not show any considerable effect from the ultrasound at 80 and 95 °C, larger ingots with a diameter of 55 mm, which initially had worse mechanical properties, exhibited a two to three-fold increase in their elongation to failure after the UST. In the microstructure of sonicated alloys, regions with a dislocation density of the order of 10^{14} m^{-2} were observed as well as a large number of dislocation loops and dispersion particles, which was explained by an acceleration of ageing processes due to the generation of vacancies. Sole ageing at the same temperature did not result in these effects. In Al single crystals after UST, an increased dislocation density near grain boundaries and a cell structure were observed.

During the UST of commercially pure Al and Al-4% Cu alloy with a strain amplitude of $\varepsilon_0 = 2.2 \times 10^{-4}$ for up to 40 min at a temperature of -30°C , an eight-fold increase in dislocation density was observed and the dislocations were distributed uniformly [37]. In contrast, after treatment at a high temperature (280 °C), the dislocation density first increased but then dropped due to the formation of dislocation subboundaries.

4.3. Copper Crystals

Gindin et al. [27] studied the mechanical properties of 99.97% pure copper single crystals subjected to vibrations with stress amplitudes up to 8 MPa. Three tension experiments were carried out: one without ultrasound, one with the simultaneous application of ultrasound during tension, and one with tension after a preliminary UST. The application of ultrasound during deformation resulted in a significant reduction in the yield stress and a longer easy glide stage, which is consistent with the acoustoplastic effect. In contrast, the preliminary UST resulted in a significant increase in the yield stress (from 7.5 MPa without the UST to 22 MPa after the UST with an 8 MPa stress amplitude). The easy glide stage was longer than it was without the UST. The significant increase in the yield point of the preirradiated samples was associated with an increase in the density of lattice defects and a redistribution of dislocation pinning sites under ultrasonic irradiation.

Different results were obtained by the same authors on samples of oxygen-free copper [38]. The samples with a length of 20 mm were subjected to UST with amplitudes of 1 to 15 μm at temperatures $T = 77$ and 300 K for 1 s and immediately after this were tested in tension. The authors did not report the values of the stress amplitude and related the effects only to the displacement amplitude. They found that after UST with amplitudes below about 4 μm , the yield stress of the material decreased. This effect was significantly more pronounced at $T = 77$ K. For instance, the yield stress decreased from 15.5 MPa in the initial state down to 12 MPa after the UST with $\xi_0 = 1.3 \mu\text{m}$ at 300 K and nearly three times after the UST with $\xi_0 = 2.5 \mu\text{m}$ at 77 K. With an increase in the vibration amplitude above 4 μm , the yield stress and length of the easy glide stage increased. The latter behavior was

explained by the generation of dislocations during the UST predominantly in the “weak” regions of the materials, due to which their microstructure becomes more homogeneous. Due to an increase in the local flow stress in the initially weak regions, the yield stress of the material increases. On achieving this stress, dislocations in all regions become mobile and make a larger contribution to the plastic strain than those in a non-irradiated sample without a considerable increase in the flow stress. At low acoustic amplitudes, oscillating stresses result in a redistribution of pinning points. For these cases, the authors analyzed the forces acting on the pinning points from segments of a vibrating dislocation line. Two segments of such a dislocation are represented in Figure 5.

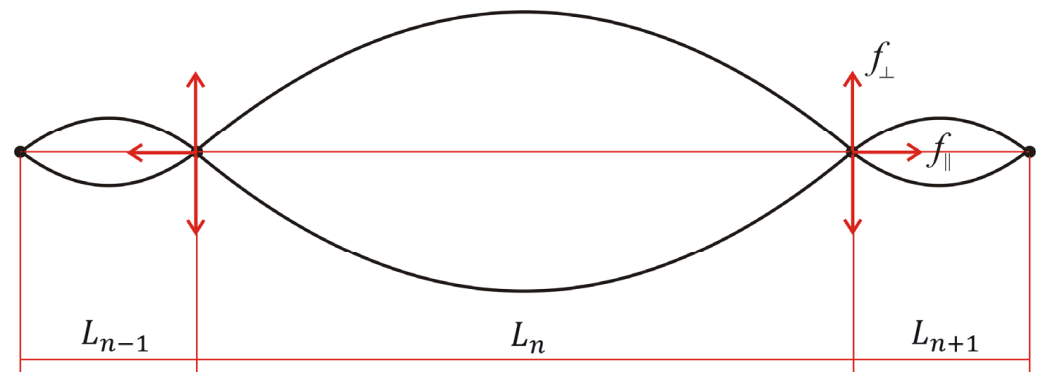


Figure 5. Scheme representing the forces acting on pinning points from segments of an oscillating dislocation [38].

The force acting on a pinning point (as an example, consider one that delimits segments n and $n + 1$) can be decomposed into components normal and parallel to the dislocation line in the equilibrium position: $f_{\perp}$ and $f_{\parallel}$, respectively. The normal component has an average value equal to zero, while the parallel one is always directed towards the shorter segment. Under this force, the pinning point migrates to further shorten the shorter segment. Therefore, ultrasonic irradiation results in a non-uniform distribution of the pinning points and the formation of long dislocation segments. During subsequent tensile deformation, the easy depinning of these segments and unzipping of the dislocation occur. As a result, the yield stress is lower than that of the unirradiated sample. After the UST, pinning points gradually return to their initial positions and a recovery of the yield stress occurs. The higher the temperature, the shorter is the time necessary for this recovery.

Unfortunately, the authors did not describe the initial state of the material they used in [38]. Comparing the values of the yield stress of the two types of copper samples presented above (7.5 MPa for 99.97% Cu and 15.5 MPa for oxygen-free Cu), it could be suggested that in the latter case, the material was in a preliminarily deformed state, which is why it had a higher dislocation density. In this case, an UST with a moderate intensity might result in the softening of the material.

Hayes and Shyne obtained indirect evidence for the generation of nonequilibrium vacancies during the ultrasonic irradiation of copper [39]. Half-wavelength samples of oxygen-free high-thermal-conductivity copper were irradiated with a maximum acoustic stress of 32 MPa. The treatment caused a great increase (30–100 times) in the recrystallization rate. The degree of the enhancement of grain growth kinetics was proportional to the stress amplitude. The same time dependencies of the grain growth were observed for ultrasonically treated and non-treated samples, but the activation energy dropped from 132 kJ/mol down to 71–105 kJ/mol due to the UST. This value is close to the activation energy for vacancy migration, and the generation of nonequilibrium vacancies due to UST was suggested to be a possible reason for the observed effect.

4.4. Nickel and Its Alloys

Changes in the substructure misorientations formed during UST were found to differ from those observed during quasistatic cold deformation [40]. In the cited paper, the

microhardness of a Ni-1.18Al alloy was found to increase by 30–45% during several minutes of an UST with amplitudes of 10–20 μm , while the same change occurred after 5–8% cold deformation. Meanwhile, cold deformation resulted in an increase in the average disorientation by about 30–50 mrad vs. 5 mrad after the UST.

In the earliest works on UST, the structure evolution and property changes were studied without relating them to the intensities of the ultrasound action, i.e., to amplitudes of alternating stresses. Belostotskiy et al. were probably the first to study the dependence of the structure and microhardness of sonicated nickel on the amplitude of ultrasound [24]. They used half-wave samples of 99.99% pure Ni with a length of about 140 mm and diameter of 3–4 mm soldered to a concentrator. In an initial, well-annealed state, the samples consisted of grains with sizes of 0.2–0.4 mm free of dislocations. An UST was carried out with stress amplitudes in the middle of samples up to 130 MPa with water cooling and without cooling. In the first case, the temperature increase during the treatment was not higher than 20–40 °C, while without cooling, it increased up to about 600–800 °C in the middle sections of samples during the UST for 2 min. In the latter case, the temperature was non-uniformly distributed along the samples, having maximum values in the middle sections and minimum ones at the ends of samples.

Figure 6 represents the distributions of the stress amplitude during the UST and microhardness values after the UST with cooling and without cooling for a time interval of $t = 2$ min. The stress amplitude at the sample center was $\sigma = 110$ MPa. The graphs show that the UST at room temperature resulted in a significant hardening of the nickel. The authors also analyzed the dependence of the microhardness on the stress amplitude. Up to $\sigma = 80$ –90 MPa, the strengthening was proportional to the amplitude; in the range of $\sigma = 90$ –120 MPa, it grew proportionally to the square of the amplitude; and above $\sigma = 120$ MPa, it levelled up and the failure of the samples occurred. Microstructure studies showed that in all stages, a multiplication of dislocations occurred. In the first stage, no surface relief related to this multiplication was observed, but in the second stage, slip bands were observed, the density of which increased with the stress amplitude. The dependence of the microhardness on a cross-section position along the sample without cooling indicates that in the central part there is competition between the accumulation of dislocations and recovery. Due to this, the maximum values for the microhardness were observed at two points located some distance from the specimen center. At the highest amplitudes of the UST, a fracture of the samples occurred. At the ambient temperature, it had a predominantly interfacial character, although there were some intragranular fractures. At high temperatures, it had a completely intergranular character and chains of intergranular pores were observed. The authors concluded that these pores were formed due to the sink of nonequilibrium vacancies which formed due to the effect of the ultrasound.

In general, the formation of nonequilibrium vacancies is a typical process, which occurs during plastic deformation due to the emission from jogs on moving dislocations or due to a dislocation annihilation [20]. Several indirect proofs were obtained indicating the generation of a high concentration of vacancies during the UST of Ni. For instance, an analysis of volume changes during post-UST annealing showed that an 8 min action of ultrasound with a stress amplitude of 100 MPa and a 1 min action with an amplitude of 130 MPa resulted in a vacancy concentration of approximately 1.5×10^{-4} which was approximately two times higher after a 20% quasistatic deformation [41]. The estimated dislocation density after the UST was of the same order as after the quasistatic deformation and amounted about 10^{15} m^{-2} . Similar studies were carried out based on measurements of changes in the electrical resistivity during annealing after UST [42]. Therein, the authors observed two distinct temperature intervals for the recovery of electrical resistivity: a lower-temperature one at 150–300 °C and a high-temperature one at 400–600 °C. The low-temperature interval corresponded to a sink of vacancies and the high-temperature one to a recovery of dislocations. From these data, a nonequilibrium vacancy concentration of 1×10^{-4} and a dislocation density of $3 \times 10^{14} \text{ m}^{-2}$ were calculated. Later, Belostotskiy estimated vacancy concentrations in copper and nickel after USTs in a range of stress amplitudes up to 140

and 200 MPa, respectively, at the temperature of liquid helium (4.2 K), the temperature of nitrogen (77 K) and in water at room temperature (approximately 295 K) [43]. In the Ni at room temperature, the electrical resistance attained a saturation value at stresses of about 120–140 MPa, while at low temperatures it increased monotonously in all the intervals of the amplitudes studied. At the temperature of the helium, the vacancy concentration and dislocation density were estimated to be about $C_v \approx 4 \times 10^{-5}$, $\rho_d \approx 1.8 \times 10^{14} \text{ m}^{-2}$ for Cu and $C_v \approx 8.5 \times 10^{-5}$, $\rho_d \approx 1.3 \times 10^{14} \text{ m}^{-2}$ for Ni. The author concluded from this study that the ratio of the concentration of point defects to the density of dislocations formed during the UST was two to three times higher than that for quasistatic plastic deformation.

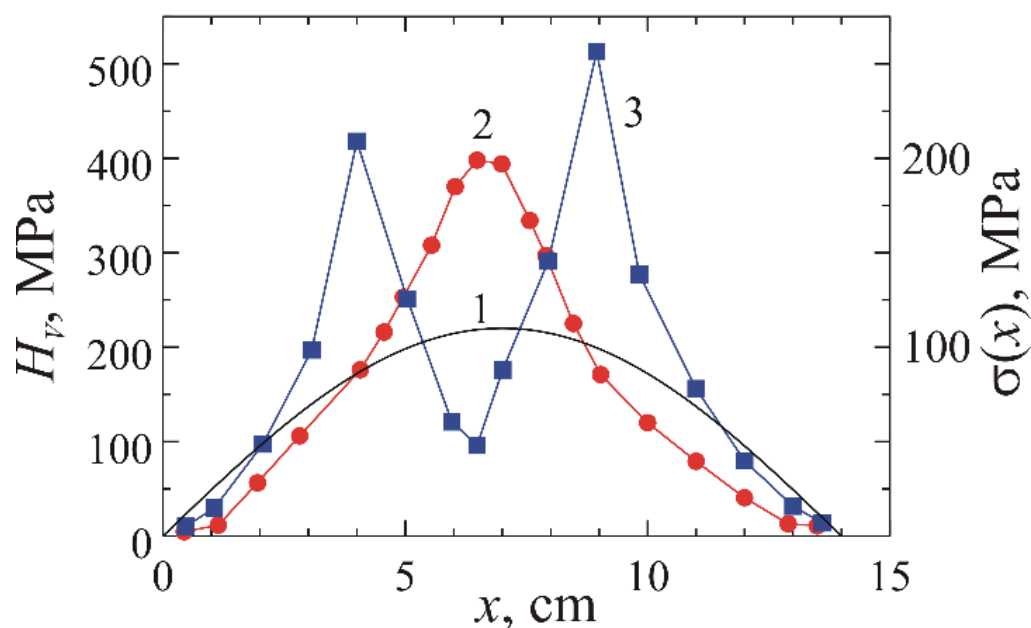


Figure 6. Distributions of the stress amplitude during UST (1) and microhardness values after UST with cooling (2) and without cooling (3) for time interval of $t = 2$ min. The graphs were plotted using the data presented in [24].

A modern scanning electron microscopy technique, electron backscatter diffraction (EBSD), allows us to carry out detailed comparative studies of selected areas of materials before and after ultrasonic treatments. Such a study was carried out recently in [44,45]. Commercially pure Ni was annealed at a temperature of 1300 °C for 1 h in order to produce a coarse-grained microstructure with a low density of lattice dislocations. A disk-shaped specimen was cut from the annealed sample, and a $2 \times 2 \text{ mm}^2$ sized area was marked on its polished surface by means of a special indenter. After an examination of this area by EBSD, X-ray diffraction analyses and microhardness measurements, the specimen was subjected to an UST with a stress amplitude of 20 MPa. After the treatment, the specimen surface was polished again and the marked area was re-examined by the same methods used before the UST. Comparing the results of the EBSD analysis, conclusions on the dislocation activity and structural evolution during the UST were made. The microstructures after UST with higher amplitudes (40 and 80 MPa) were also studied in detail.

Figure 7 presents the inverse pole figures (IPFs), kernel average misorientation (KAM) maps and pole figures obtained by the EBSD analyses of the selected area of the nickel specimen before (a, c, e) and after (b, d, f) the UST. A comparison of the microstructures allows one to distinguish several consequences of the dislocation activity inside the grains. Some twins inside the grains and formerly the smooth grain boundaries attain a curlier shape, which is a result of an interaction of lattice dislocations with grain boundaries (the generation or trapping of lattice dislocations). An increase in the dislocation density after the UST is clearly seen from the KAM-maps as well. Not only groups of statistically distributed dislocations are observed in the grains, but also changes in the substructure

occur. These changes consist in an appearance of new, misoriented cells inside some grains (examples are indicated by letters A and B in Figure 7b) and the disappearance or transformation of existing boundaries (for example, C and D in Figure 7a). Additionally, a migration of the low-angle boundaries is observed (E and F in Figure 7b).

The quantitative results of the EBSD and x-ray diffraction (XRD) analyses and the microhardness values for both the initial and post-UST states with different stress amplitudes are given in Table 1. One can see that, due to ultrasonic irradiation with the amplitude of 20 MPa, the total dislocation density measured by XRD increased by two orders of magnitude, while the density of geometrically necessary dislocations increased by one order of magnitude. There is also a significant increase in the fraction of low-angle boundaries (LABs) and a decrease in the fraction of boundaries close to special ones. Evidently, this is due to the disappearance of twin boundaries and the formation of new LABs. All these structural changes result in an increase in the microhardness by more than 300 MPa. At the same time, the average sizes of coherent scattering domains and grains do not change significantly. Additionally, the IPF and KAM images show that high-angle boundaries (HABs) do not move during the UST process and the grains retain their shapes. On the contrary, LABs can migrate, providing substructure changes.

Obviously, the stress amplitude of 20 MPa is not enough for an intensive development of the dislocation substructure and the dislocations mainly accumulate as statistically stored ones. This is consistent with the results of discrete dislocation dynamics simulations, which show that dislocations of different signs gliding on different slip planes can form stable clusters, some of which are immobile under oscillating stresses and others can slowly drift [46]. The clusters can meet other ones to make larger clusters and a slow process of substructure formation occurs. Then, the density of statistically stored dislocations will be significantly higher than that of geometrically necessary dislocations. With an increase in the stress amplitude, the fraction of immobile dislocation clusters decreases, since the stress becomes sufficiently high to break dislocation dipole structures with smaller separations. Additionally, under higher stresses, dislocations can glide through denser dislocation forests. As a result, the formation of low-angle dislocation boundaries becomes possible. This results in a decrease in the density of statistically stored dislocations and simultaneously an increase in the density of geometrically necessary dislocations. A rearrangement of the dislocations into cell boundaries leads to a screening of their long-range stress fields and this causes a decrease in the root-mean-square strain measured by XRD, as compared to the state obtained by UST at 20 MPa. With a further increase in the stress amplitude, the cell structure develops towards a subgrain structure with decreasing sizes and increasing misorientations of the subgrains, and the fraction of HABs increases back. Both the accumulation of dislocations at lower amplitudes of UST and the development of cell and subgrain structures result in a steady increase in the microhardness of the material.

Table 1. The data of X-ray diffraction, EBSD analyses and microhardness measurements of coarse-grained nickel in the initial and post-UST states with stress amplitudes of 40, 60 and 80 MPa [44].

Specimen	X-ray			EBSD					Hv, MPa
	<d> (nm)	Microstrain	Dislocation Density (m^{-2})	<d> (μm) Intercept	<d> (μm) Diameter	LAB (%)	CSL (%)	Dislocation Density (m^{-2})	
CG	782 ± 85	7.19×10^{-5}	1.28×10^{12}	156	180	7.8	50.7	2.44×10^{11}	1060 ± 50
UST 20	681 ± 24	6.57×10^{-4}	1.34×10^{13}	164	173	19.4	40.7	3.16×10^{12}	1375 ± 47
UST 40	354 ± 44	2.10×10^{-4}	0.8×10^{13}	212	147	71.9	7.3	7.6×10^{12}	1570 ± 45
UST 80	377 ± 18	3.78×10^{-4}	1.37×10^{13}	103	80	59.4	17.4	10.2×10^{12}	1645 ± 43

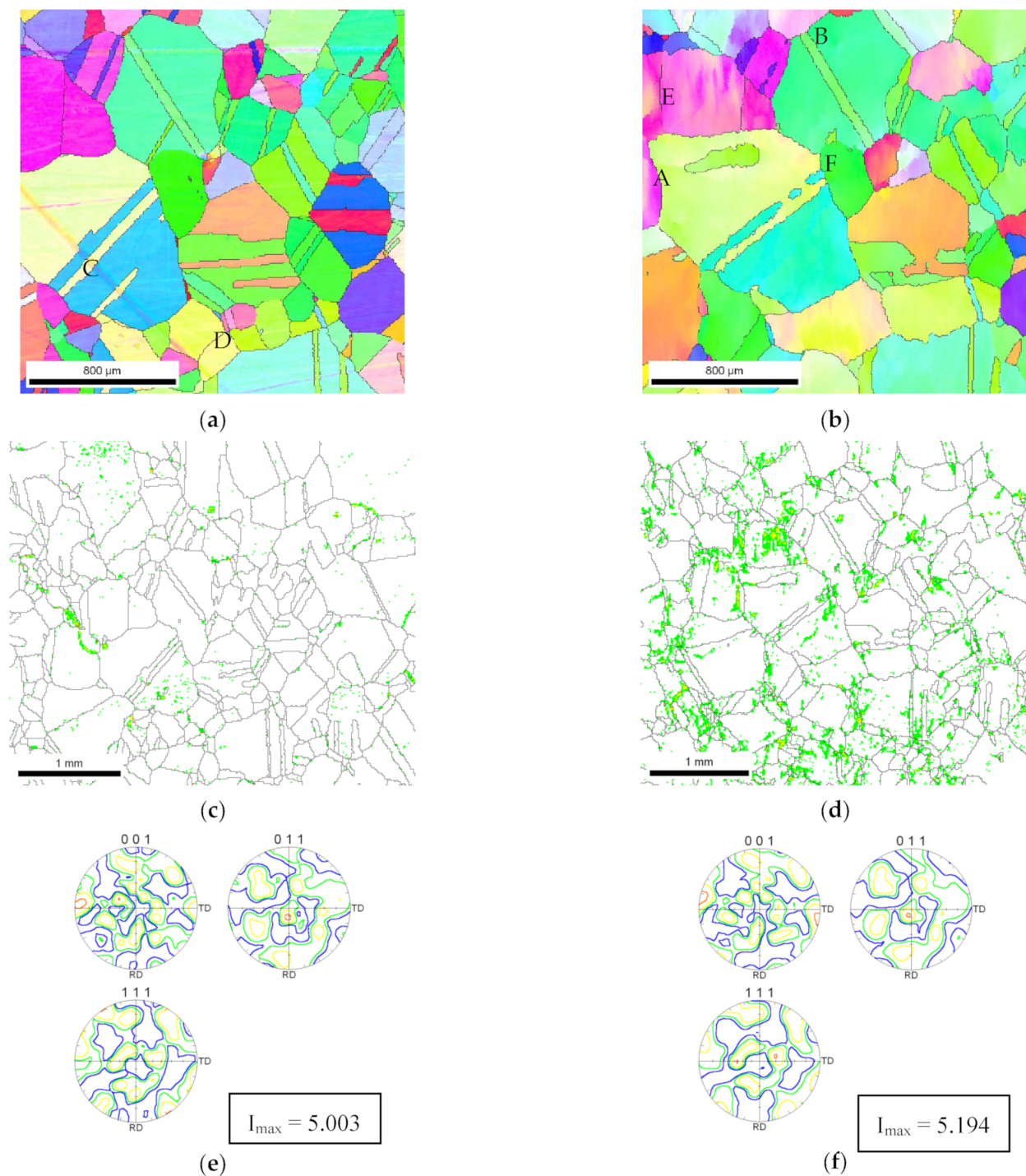


Figure 7. The IPF maps (a,b), KAM-maps (c,d) and pole figures of coarse-grained nickel: (a,c,e)—before ultrasound treatment; and (b,d,f)—after ultrasound treatment with an oscillating stress amplitude of 20 MPa [44].

4.5. Steels

Different subboundaries ranging from highly irregular tangles to regular hexagonal arrays were also observed after UST of half-wave samples of Kh18N10T steel with displacement amplitudes of 15–40 μm [47]. Single dislocation loops and dipoles were also typical of the microstructure. These studies verified the dislocation multiplication was due to the effect of high-intensity ultrasound.

Interesting studies of the effect of ultrasonic stress amplitude and preliminary cold deformation on the mechanical properties of mild steel were carried out in [48]. Ultrasound with stress amplitudes in the interval of 70–350 MPa was applied to the samples of material in three different initial states: as-annealed (A); and subjected to 5% (B) and 10% (C) cold drawing. In all cases, the yield and ultimate strengths increased and the ductility decreased with the stress amplitude, but in the deformed state, these changes commenced at lower amplitudes. In annealed steel, the strength and ductility did not change significantly up to about 280 MPa, while after the UST of steel in state B, the strength started to increase at 210 MPa and in state C at 140 MPa. The maximum elongation started to decrease at 140 MPa in state B and at 70 MPa in state C. The authors related this behavior to an operation of dislocation sources. In the deformed state, due to a higher density of dislocations, the density of their free segments capable of acting as dislocation sources is higher. Therefore, during UST, these sources will generate more dislocations, and work hardening will occur at lower amplitudes of ultrasound.

4.6. Refractory Metals

In [49], the effect of ultrasound with an intensity of 150 W/cm² on the structure and temperature dependence of dislocation damping in molybdenum was studied. The treatment resulted in a significant increase in the dislocation damping, which was an indication of an increase in the dislocation density. Indeed, electron microscopic studies showed that the dislocation density increased from 10¹¹ to 10¹² m^{−2} in the initial annealed state to 2.7×10^{13} m^{−2} after 15 min irradiation and 7×10^{13} m^{−2} after 1 h irradiation, which was similar to the dislocation density in molybdenum single crystals after 10% upsetting deformation. Studies of the effect of UST on molybdenum mono- and polycrystals at different temperatures showed that up to $T = 250$ °C dislocations were distributed uniformly, while at temperatures in between 400 and 700 °C, they were arranged in cell boundaries [50]. At room temperature, the microhardness increased only near the middle parts of the half-wave samples where the amplitude of stresses was higher than 210 MPa. The microhardness increased mainly up to 30 s of irradiation and then levelled up. The ductility of the material decreased significantly after 10 s of irradiation. This was explained by the generation of point defects and microcracks.

4.7. Ionic Crystals

Extensive studies of the dislocation structure of ionic crystals subjected to the action of ultrasonic waves were carried out by Tyapunina and her co-workers [51–54]. The results of these studies were summarized in their monograph [55]. These authors widely used half-wave samples glued to the concentrator of a vibration system. The method allowed studies of the effect of UST with strain amplitudes ranging from 10^{−7} to 10^{−3}, measurements of the temperature during UST, etc. In the following paragraphs, a short summary of the results obtained by this group are presented.

The authors introduced the notion of dynamic yield stress, which was defined as the stress amplitude during UST at which a massive multiplication of dislocations starts. This characteristic was found to consist of two terms, one of which was proportional to the static yield stress and the second one depended on the frequency of ultrasound. For instance, the threshold amplitude below which there were no dislocation displacements in KCl amounted to about 5 MPa. The displacements of dislocations were studied by the etch pit method. It was shown that the number of dislocations displaced under the action of ultrasound depended on its amplitude and correlated with the distribution of stress amplitudes along a sample. Dislocation path lengths were much larger than the calculated amplitude of dislocation oscillations, which showed that a progressive motion of the dislocations occurred under a symmetrically oscillating applied stress.

At a given value of the frequency and at amplitudes exceeding the dynamic yield strength, the dislocation density increases with time and attains a saturation value. The saturation occurs either with time at a constant amplitude, or with an increase in the

ultrasound amplitude at a given time of sonication. The initial dislocation structure has little effect on the dislocation density upon a saturation. Like the dynamic yield strength, the stress at which a saturation occurs was found to be frequency-dependent.

The experiments showed that under the action of ultrasound, an intense deformation polygonization occurred. The distribution of polygonization boundaries in the samples correlated with the distribution of stresses in a standing ultrasonic wave. Their density was the highest near the middle sections of the half-wave samples. The polygonization process was due to both the dislocations that previously existed in the crystal and those newly formed during irradiation.

An excess concentration of vacancies, along with the dislocation density, was also measured in alkali halide crystals strained by ultrasound in a frequency range from 20 to 200 kHz [56]. The experiments were carried out on as-grown crystals of KBr, KCl and NaCl using half-wave parallelepiped-shaped samples. To find the concentration of point defects during the ultrasound exposure, the method of detecting F-centers was used. This method is based on the phenomenon which consists in a formation of coloring centers during ionizing irradiation in ionic crystals due to an interaction of excited states with point defects [57]. In [56], γ -rays from a ^{60}Co source were used. The initial concentration of point defects was found to be about 10^{23} m^{-3} in KBr and KCl and $3 \times 10^{23} \text{ m}^{-3}$ in NaCl. At the stress amplitudes between 6 and 10 MPa, a sharp, exponential increase in the concentration of point defects with the amplitude was revealed, but at higher amplitudes the excess concentration levelled off at $(1-2) \times 10^{23} \text{ m}^{-3}$. The authors suggested that the main mechanism for the formation of nonequilibrium vacancies under ultrasound was the annihilation of dislocation dipoles.

5. Ultrasound-Induced Dispersion Strengthening of Alloys

Oscillating stresses with normal and shear components and changes in local temperature during ultrasonic irradiation can cause changes in the diffusion coefficient and conditions for phase equilibrium and consequently result in phase transformations [22,58]. In this relation, the effect of UST on the processes of precipitation hardening is of particular interest. Precipitation hardening, or age hardening, is a process which is used to increase the yield stress and hardness of alloys by the precipitation of hard particles in an oversaturated solid solution during ageing [59].

One of the first studies of the effect of UST on the ageing of alloys was carried out by Gorsky and Efremov [60]. The authors used ultrasound with φ frequency of $f = 30 \text{ kHz}$ and an intensity of $I = 10 \text{ Wcm}^{-2}$ to process duralumin with 4.5% Cu, 0.8% Mg and 0.5% Si at room temperature and found that the natural ageing of the alloy was enhanced by a factor of about 63.

Pogodina-Alekseeva and Eskin studied the effect of ultrasound with φ frequency of 1.0 MHz and an intensity of 1.6 Wcm^{-2} on natural and artificial ageing processes in a series of aluminum alloys (Russian grades D1, D16, V95 and AL4) [61]. In D1 and D16 alloys, a 20- to 25-fold increase in the rate of ageing was observed and the final hardness was higher in the sonicated samples than in the unsonicated one. In the other two alloys, a significant enhancement of the ageing rate was found too.

The effect of ultrasound on the dispersion hardening of duralumin was also studied in [62]. The authors used vibrations in a wide range of frequencies, 300 to 1500 kHz, and also found that the ageing rate was significantly enhanced under irradiation. No dependence of the effect on the frequency was observed, but with an increase in the intensity of ultrasound, the strengthening rate became higher.

Al'ftan and Ermakov stated that, due to the absence of measuring techniques for vibration amplitudes, only the specific power of an ultrasonic transducer was estimated in the first works on UST, but the amplitudes of the oscillating strains of the samples were not measured and the real amount of ultrasonic energy imparted into the samples might be too low [63]. The authors studied the ageing process in Russian-grade KhN80T nickel–chrome–titanium alloy subjected to UST with frequencies in an interval from 20 to 26 kHz using

105 mm long cylindric samples. The displacement amplitudes were measured at the ends of the samples and the stress amplitudes in their middle sections were calculated according to Equation (1c). The dispersion strengthening curves in terms of the time-dependencies of Rockwell hardness values obtained from the experiments with and without the sonication of samples are given in Figure 8. The grey bar on this graph shows the interval of hardness values which is achieved in this alloy by standard ageing (16 h at 700 °C). Under the effect of ultrasound, this zone is reached in a time interval of 20–25 min, which is 40–50 times less than that of standard ageing.

The authors carried out a phase analysis of the material after each treatment and found that the volume fraction of the intermetallics of types $(\text{Ni, Cr})_3(\text{Al, Ti})$ amounted to nearly 9%, which was the same as after standard ageing. One can also see from the figure that the effect of UST greatly depends on its intensity. The higher the intensity, the more pronounced is the enhancement of the ageing rate.

A considerable enhancement in ageing kinetics was also found during the UST of KhN77TYuR alloy in [64]. In this work, the stress amplitude at displacement nodes of 120 mm long half-wave samples was 24 MPa at 700 °C and 23 MPa at 750 °C (the vibration amplitude at the sample end was equal to 5 μm). It was demonstrated that the dispersion hardening was not the same along the samples: it was maximum was near the middles of the samples and nearly zero at their ends, which is consistent with the distribution of stress amplitudes.

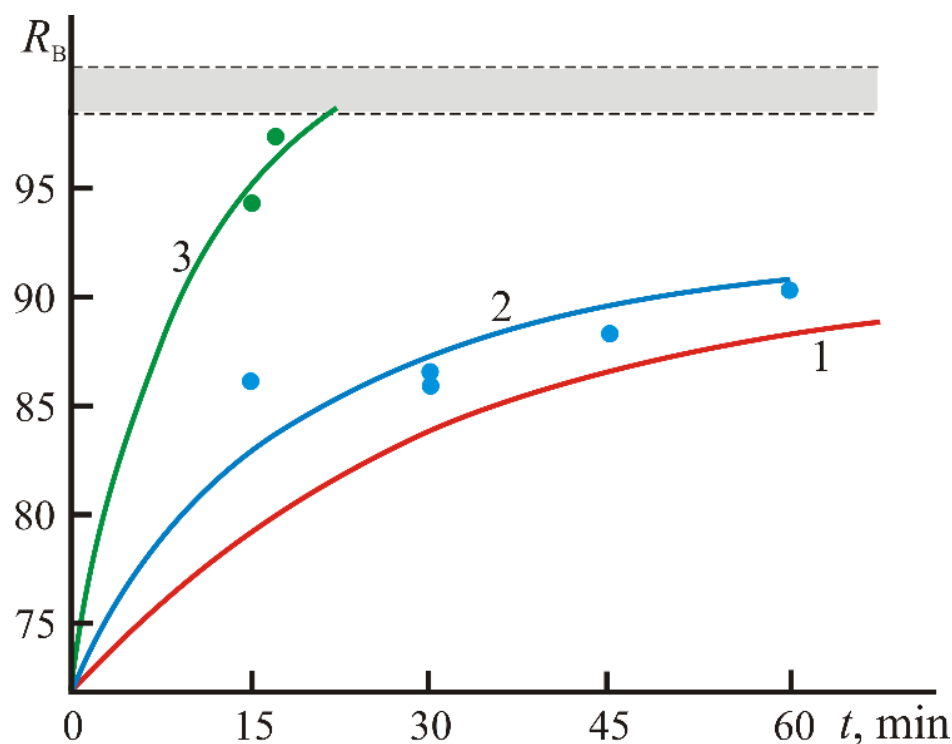


Figure 8. Dispersion strengthening curves for KhN80T alloy for different regimes of ageing: standard (static) ageing at 700 °C (1), 700 °C with UST at $\sigma_0 = 25$ MPa (2), 700 °C with UST at $\sigma_0 = 35$ MPa (3); the grey bar represents the interval of hardness which is reached by standard ageing at 700° for 16 h (adapted from [63]).

A different result was obtained during ultrasound-assisted ageing experiments on Tomas steel in [65]. Ultrasound with a frequency of 430 kHz and intensity of 6.5 Wcm^{-2} was used in the ageing of quenched Tomas steel with 0.06% carbon at 20 °C. No considerable effect of the ultrasound on the ageing process was found. The authors suggested that at room temperature, the energy input from the vibrations was not enough for the straining

the crystal lattice to a necessary level at which the diffusion of strengthening atoms would be sufficiently enhanced.

Hayes and Shyne investigated the kinetics of precipitation hardening in a copper–beryllium alloy [66]. Half-wavelength specimens were subjected to UST with a maximum acoustic stress amplitude of 35 MPa. The authors found only a small acceleration of the precipitation rate due to the effect of the ultrasound. They showed that the increase in the ageing kinetics was caused by an acoustic enhancement of the grain–boundary precipitation reaction. The ultrasonic energy did not significantly change the apparent activation energy for precipitation or alter the time-dependence of the precipitate nucleation and growth rates. In the cited work, a clear dependence of the hardness on a position along the half-wave sample was also observed.

In his review, Mordyuk [22] analyzed different data on the effect of ultrasonic vibrations on the intensity of allotropic transformations, processes of the breakdown of solid solutions, diffusion processes, etc. He assumed that the results of a few works in which no effect was observed could be explained by possible disadvantages due to the experimental techniques and low intensities of ultrasound used.

Changes in the microhardness of Al–Cu–Mg alloy AK4-1 (analogue of alloys 2618 and A92618) under the influence of the ultrasound at different temperatures was studied in [67]. Preliminarily, a supersaturated solid solution was obtained by quenching the alloy from a temperature of 530 °C. The UST was carried out in time intervals from 0.5 to 10 min at temperatures of 20 °C, 80 °C, 130 °C and 185 °C, and an increase in the microhardness with the time of treatment was demonstrated. Interestingly, the effect of the preliminary UST on the microhardness change during subsequent artificial aging also was investigated in the cited work. The samples after quenching were sonicated in time intervals of 3 and 10 min and then annealed at 185 °C for 16 h along with the non-sonicated samples. After 4 h of ageing the samples subjected to the preliminary UST, the microhardness attained a saturation value of about 1400–1500 MPa, while in the non-irradiated samples, the maximum microhardness of 1350 MPa was reached only after 20 h ageing. So, not only the UST during ageing affected the kinetics of dispersion hardening but also the preliminary UST allowed the authors to obtain a higher strength by subsequent ageing. Unfortunately, they did not describe the regimes of UST, so it is not clear what was the intensity of the ultrasound in their experiments.

The conclusions of most of the above considered studies were based solely on hardness or microhardness measurements, assuming that the precipitation of strengthening particles was the main source of the observed hardening. Exceptions are [39,63], in which structural studies were carried out to confirm the results of the hardness measurements.

Hardness and tensile stress measurements also were accompanied by electron microscopic observations of microstructures in a study of the effect of UST on the properties of Al alloys containing 3.8%, 4.1% and 5.2% of the main alloying component, Cu [8]. In this work, the samples were subjected to UST at different temperatures with strain amplitudes equal to 0.8, 1.0 and 1.2 times the fatigue limit at a corresponding temperature. It was found that the UST resulted in the shortening of the time necessary to achieve the maximum hardness by a factor of about 30 to 100 as compared to the normally required ageing time. Structural observations showed that the UST resulted in a substructure and higher density of prismatic dislocation loops and Al₂Cu precipitates, and the sizes of precipitates were smaller than after the ordinary ageing. The tensile strength increased with the strain amplitude.

So, most of the studies, in particular, those involving structural analyses along with hardness measurements, clearly demonstrated that UST has a considerable effect on the kinetics of the precipitation of hardening phases in alloys, resulting in an increase in the rates of precipitate formation, decrease in their sizes and an enhancement in the strength and hardness.

6. Recovery Processes under Ultrasonic Treatment

As discussed in Section 2, in some circumstances, cyclic straining can result in a relief of internal stresses and the softening of materials. This assumption has been confirmed by a number of experimental studies.

Hempel [68] studied the behavior of residual stresses in samples of a chrome-molybdenum steel preliminarily subjected to shot-peening during subsequent fatigue tests. They found that the application of alternative stresses below the fatigue limit resulted in a relaxation of residual stresses. At the stress amplitude of about 700 MPa, 100 cycles of straining were enough to reduce the residual stress down to a level of about 1/7th of the initial value. Sinclair [69] also showed that materials preliminarily subjected to cyclic stresses below their fatigue limit resisted failure for a longer time in subsequent tests at stress amplitudes above the fatigue limit.

Samples of KhGV steel with dimensions $4 \times 7 \times 90 \text{ mm}^3$ were tempered in oil with and without the application of vibrations in [70]. When the samples were tempered with ultrasound, a sharp decrease in residual stresses was observed. Treatment for 0.5 h with ultrasonic vibrations resulted in a 50% reduction in the stresses, which is equivalent to tempering for 2 h at 180 °C. In other words, a four-fold enhancement of the stress relief rate was caused by ultrasound.

Remarkable results on the interplay between the hardening and softening effects of ultrasound were reported by Kulemin [58]. Polycrystalline aluminum was annealed at $0.5T_m$, then deformed by cold rolling or tension up to 5% strain. Half-wave samples in both the annealed and deformed states were subjected to UST at different amplitudes for time intervals up to about 200 min. The microstructures after these treatments were studied by XRD, and the dislocation density and microhardness were measured. The changes in the microhardness with time for the different values of the stress amplitude obtained during these experiments are schematically represented in Figure 9.

When an initially annealed sample is subjected to UST, its microhardness increases due to the multiplication of dislocations, as we have discussed above. This increase saturates with time at a certain level (curve *D* in Figure 9). In contrast, UST of preliminarily deformed sample results in a decrease in the microhardness. This occurs if the stress amplitude exceeds some threshold value σ_{m1} , above which the dislocations generated by pre-strain become mobile. The higher the stress amplitude, the more intensive is the softening (curves *A*, *B*, *C*, $\sigma_{mA} < \sigma_{mB} < \sigma_{mC}$). However, at amplitudes exceeding some critical value (denoted as σ_{m2}), the microhardness attains a minimum and starts to increase again. During very long USTs, it can further attain a maximum and then decrease. This behavior was explained based on the dependencies of the microhardness during UST of the annealed and deformed samples on the amplitude, which are schematically represented in Figure 10. Here, curve 1 for the annealed sample plots the values of microhardness after the UST up to saturation. For the deformed samples (curve 2), the microhardness values are taken at the minima points. Here, σ_{m1} stands for a threshold value of the amplitude when dislocations in the deformed material become mobile and its softening begins. The second threshold stress, σ_{m2} , corresponds to the point at which the two graphs intersect. At this point, the structure of the deformed material relaxes to the same level of nonequilibrium which is created by the UST of the annealed material. At this point, the softening of the deformed metal changes to hardening. When the stress amplitude is less than σ_{m2} , the point of intersection is never reached, and during very long USTs, the microhardness will attain an asymptotic value and never increase (curves *A* and *B* in Figure 9).

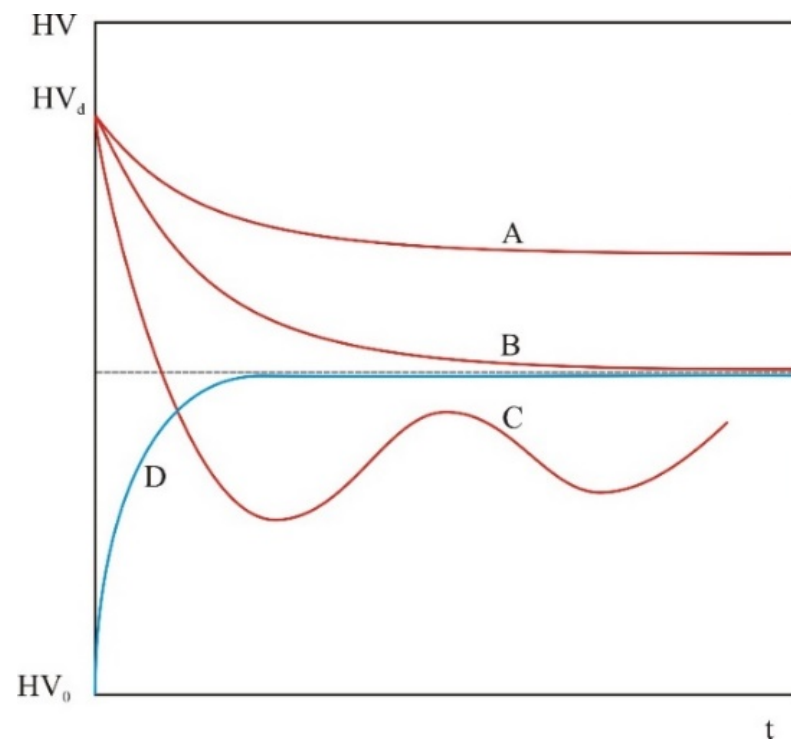


Figure 9. Time dependencies of microhardness of annealed and deformed samples during UST with different amplitudes of the stress: A, B, C—initially deformed state; D—initially annealed state; $\sigma_{m1} < \sigma_{mA} < \sigma_{mB} < \sigma_{m2} < \sigma_{mC}$; the meanings of designations are explained in the text [58].

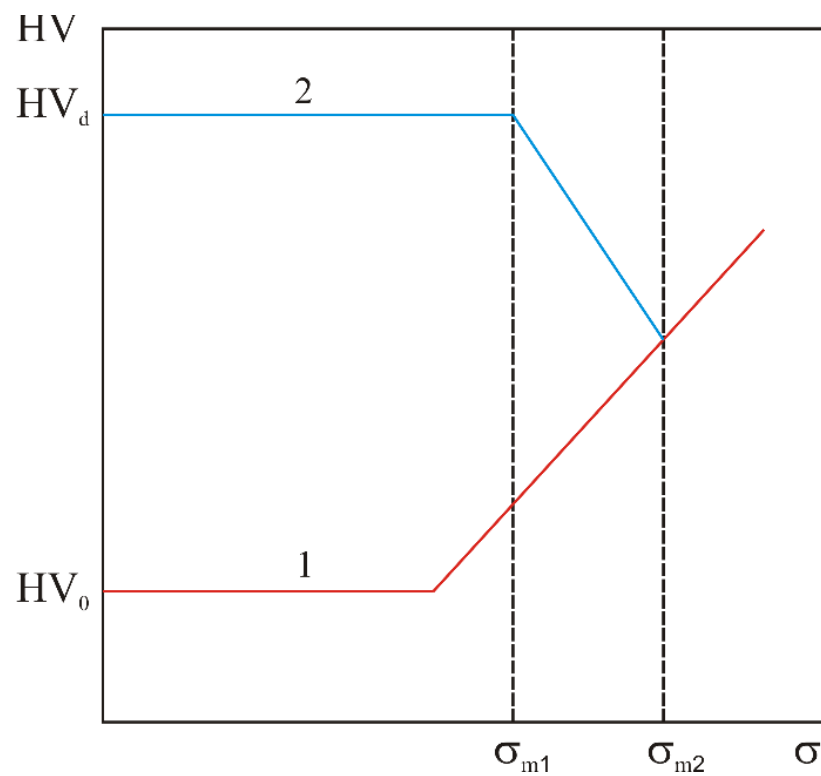


Figure 10. Amplitude dependencies of the microhardness during UST. Curve 1—annealed sample, UST up to the stage of saturation; Curve 2—deformed sample, after time interval corresponding to the minimum of microhardness [58].

A similar interplay of acoustic strengthening and softening was revealed by Mats et al. [71,72]. In [71], polycrystalline hafnium was cold rolled up to reduction ratios of 18–70% and then subjected to an UST, the scheme and intensity of which were not reported in the paper. The UST of samples made of sheets rolled to a reduction up to 20% resulted in an increase in the microhardness and yield stress. This increase diminished and disappeared with an increase in the strain. With a further increase in the strain above 20%, the strengthening effect of the UST changed to softening, which was the maximum for the strain of 70% in which the yield stress reduction was about 200 MPa. The observed behavior was explained by a generation of dislocations in the non-deformed and slightly deformed metal and a relaxation in the internal stresses in the highly strained samples. In [72], the microhardness and impact toughness of specimens of 15Kh2NMFA steel subjected to USTs with varying durations was studied. The dependence of microhardness on the time of ultrasound action was non-monotonic. During the first 5 min, the hardness increased from a value of 2200 MPa in the initial state up to 3000 MPa after 5 min of the UST, then it decreased and attained a minimum of 2300 MPa after the UST for 15 min and then it increased again to 2500 MPa after 25 min of the UST. After 5 min of the UST, the samples exhibited a considerably enhanced impact toughness and a shift in the ductile–brittle transition by about 15 °C to the left. This behavior was also explained by a non-monotonic character of the evolution of a dislocation structure during the UST.

Ultrasonic vibrations were shown to accelerate the processes of the relaxation of internal stresses in preliminarily deformed rods of tungsten and molybdenum too [73].

Zohrevand et al. carried out extensive studies of the effect of UST on the microstructure, microhardness and tensile properties of three steels preliminarily deformed by 10% and 20% tension: 316 austenitic stainless steel [74], austeno-ferritic 2205 duplex stainless steel [75] and 304 austenitic stainless steel [76]. In these works, half-wave cylindric samples designed for tensile tests were mounted in a testing machine with an incorporated vibration system. These samples were first subjected to a specified value of tensile strain and then to UST while keeping them mounted in the machine. The vibration system was designed to ensure the location of stress antinodes in the central parts of the samples. Unfortunately, the authors did not measure or calculate the stress amplitudes, but reported only the values of ultrasound power, 300 and 500 W.

The microstructure of the 20% tensile deformed sample of 316 steel consisted mainly of deformation twin bands, i.e., at this level of strain, the main deformation mechanism was twinning. After UST, the density of twins decreased, i.e., de-twinning occurred due to the ultrasound action. At the same time, in grains in which no twins were formed by pre-strain, subgrains were formed during the subsequent UST. The formation of subgrains in low-stacking-fault-energy 316 stainless steel confirms that ultrasonic vibrations stimulated the dislocation climb and cross-slip processes.

In the case of 2005 steel, the observed decrease in the microhardness, dislocation density and yield point, in addition to the XRD peak shifting and texture evolution, indicate the residual stress reduction imposed by the UST process. At the same time, no intermetallic phases which are commonly formed in thermal annealing of duplex stainless steels were produced under UST. The fracture surface analysis of ultrasonically treated samples showed a more ductile fracture than the untreated sample. Similar to the annealed structure, the lamellar morphology of the deformed duplex structure transformed into an equiaxial structure following the UST.

The results obtained for two similar austenitic stainless steels 304 and 316 show that the stacking fault energy has a significant influence on the microstructure evolution. The higher dislocation activity of 316 steel with a greater stacking fault energy indicates that dislocation cross-slip is the primary mechanism of microstructural variation during the UST process. The decrease in the twin fraction shows that detwinning is an active mechanism during UST. The formation of low-strain regions on triple junctions and near grain boundaries was observed which also implies the static recrystallization in 316SS alloy during UST.

Voyevodin et al. studied the effect of ultrasonic action on the relaxation processes and radiation embrittlement of reactor vessel steels 15Kh2MFA and 15Kh2MNFA [77]. Due to effects accumulated during deformation processing, these materials are prone to low-temperature radiation embrittlement. After standard treatments, these steels have a cell structure of ferrite with a dislocation density of about $3 \times 10^{13} \text{ m}^{-2}$. Their ductility and impact toughness decrease significantly during irradiation. The UST of samples of the steels was carried out at amplitudes of 12 to 19 μm at a temperature of 227 °C for 2 h. After the UST and irradiation, 15Kh2MFA steel had a considerably higher elongation to failure than the samples subjected to irradiation without UST during the tensile tests at temperatures from -70 °C to 227 °C. Both steels exhibited a significant enhancement in impact toughness. For example, 15Kh2MFA steel had the following values of this characteristic in different states at a temperature of -53 °C: 550 kJm^{-2} in the as-received state, 330 kJm^{-2} after electron irradiation, 680 kJm^{-2} after the UST and 650 kJm^{-2} after the UST followed by irradiation. These effects of the UST were explained by the cleaning of the cells in the ferrite grains from the dislocation pile-ups which resulted in a relief of peak internal stresses and the formation of highly dispersed carbides.

So, UST can lead to a relaxation of the internal stresses in deformed materials, which is related to rearrangements in their dislocation structure. The higher the preliminary strain, i.e., the farther a metallic system is from equilibrium, the more pronounced is the relaxation effect of UST. As such, UST seems to be a useful tool for controlling the microstructures of heavily deformed metals. In the last few decades, such heavy or severe plastic deformations have been applied to fabricate ultrafine-grained (UFG) materials, which exhibit many advanced mechanical and physical properties [78–84]. It was demonstrated that these materials were characterized by a nonequilibrium, high-energy structure of grain boundaries, which was primarily caused by extrinsic grain boundary dislocations (EGBDs) formed due to the interaction of the boundaries with lattice dislocations during deformation processing [78,85]. Therefore, studies of the effect of UST on the microstructure and mechanical properties of these materials are of interest.

7. Effect of Bulk Ultrasound Treatment on the Structure and Mechanical Properties of UFG Metals

7.1. Ultrafine-Grained Nickel

The first experimental studies of the effect of UST on the structure and thermal stability of UFG materials were carried out using the example of an UFG nickel processed by severe plastic deformation (SPD) using the high pressure torsion (HPT) method [29,86]. Disc-shaped samples of Ni were subjected to HPT under a pressure of 6 GPa, which resulted in an UFG structure with an average grain size of 180 nm. Some of the samples were annealed for 2 h at temperatures from 130 to 500 °C. Other were irradiated by ultrasound for 30 s with stress amplitudes of 40, 80, and 140 MPa to study them in the as-irradiated state and after subsequent annealing using the same regimes as for the non-irradiated samples. The microstructures, internal strains and microhardness were studied for all the states.

Figure 11 presents the results of TEM studies of the structure of the HPT-processed and annealed Ni. The as-processed material had an UFG structure with the mean grain size of about 180 nm. The grain boundaries had a nonequilibrium structure, which is evidenced by extinction contours (Figure 11a). After annealing at 130 °C, a slight grain growth occurred so the mean grain size was about 240 nm (Figure 11b). In this structure, the grain boundaries acquired a more regular shape; some of them exhibited a band contrast typical of equilibrium boundaries in well-annealed polycrystals. After annealing at 150 °C, a significant grain growth was observed, and some individual grains had sizes of about 1.5 μm , while the main UFG matrix had a mean grain size of 300 nm (Figure 11c). After annealing at 175 °C, a uniform coarse-grained structure with a mean grain size of 2.2 μm formed (Figure 11d). A further increase in the annealing temperature led to grain growth that was typical for the recrystallization process.

After the UST, the microstructure of UFG nickel consisted of equiaxed grains with a mean size slightly larger than that in the as-HPT processed state (Figure 12a). In this structure, fewer defects were observed; the grain boundaries became straight and acquired a banded contrast. This indicated a relaxation of the nonequilibrium structure in the material. After subsequent annealing at 150 °C, still no grain growth was observed (Figure 12b), while the as-prepared UFG Ni exhibited some grain growth at this temperature (Figure 11c). The grain growth that occurred in the as-HPT deformed nickel at 150 °C in this case was observed at 200 °C. The structure at this temperature is a bimodal one; the mean grain size of the matrix is 350 nm and the individual coarse grains is 3 µm. A similar structure is observed at the annealing temperature of 250 °C (Figure 12c). After annealing at the temperature of 300 °C, one observes an equiaxed coarse-grained structure with a mean grain size of 3–4 µm (Figure 12d).

The values of the microstrain calculated from the XRD studies were approximately equal to 0.065%, 0.058%, 0.052% and 0.034% after the SPD and SPD followed by the UST with stress amplitudes of 40, 80 and 140 MPa, respectively.

So, the first studies demonstrated that the UST of UFG nickel processed by SPD methods could result in a considerable recovery of the microstructure of this material towards an equilibrium and subsequently to a relaxation of internal stresses and increase in the thermal stability of the microstructure.

Extensive studies of the effect of UST on the structure and properties of HPT-processed nickel were carried out in [30,87]. Samples of UFG Ni processed by HPT were subjected to UST with stress amplitudes of 15, 30, 45, 60 and 90 MPa. X-ray, TEM and EBSD studies of the microstructure, measurements of the microhardness and excess energy released during differential scanning calorimetric scans were carried out for each state. These studies showed that the dependence of the effect of ultrasound on its amplitude was non-monotonic. At the amplitudes below 60 MPa, no signs of the relaxation of the structure were observed. Instead, the microstrain, microhardness and stored energy increased, attained maxima at about 15 MPa, then decreased up to the amplitude of 60 MPa and then increased again (Figure 13a,b). The TEM and EBSD studies showed an increase in the density of dislocations inside the grains at 15 MPa and the relaxation of nonequilibrium grain boundaries at 60 MPa.

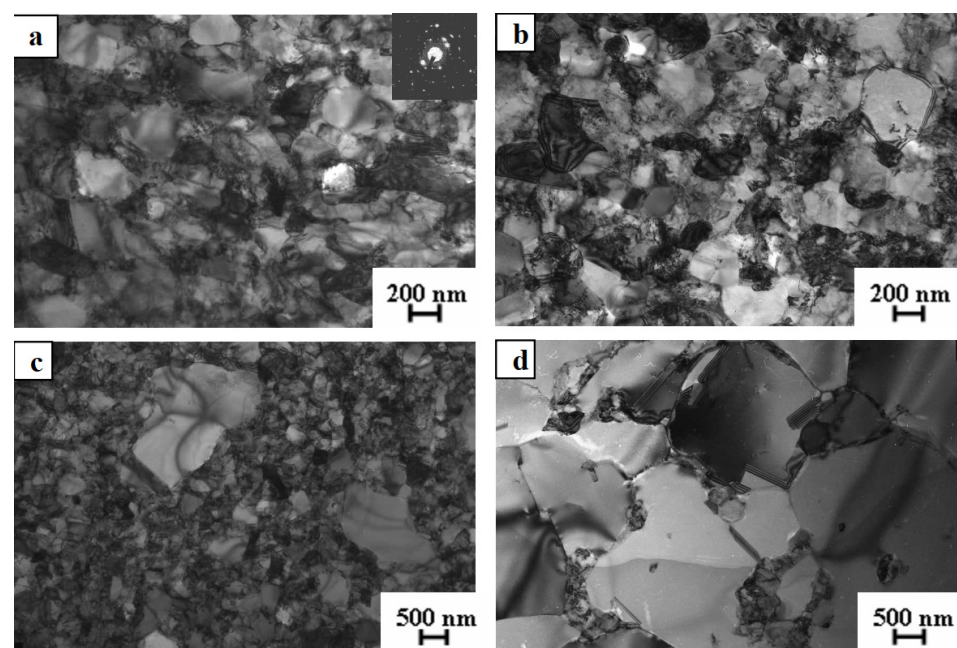


Figure 11. Microstructure of nickel after HPT (a) and subsequent annealing at the temperatures of 130 (b), 150 (c) and 175 °C (d) [86].

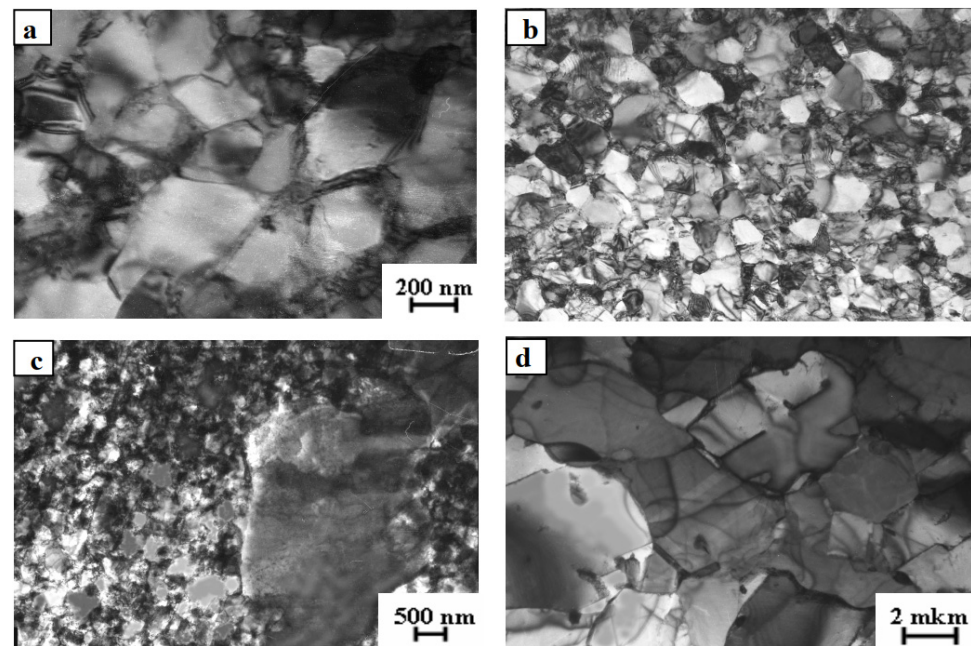


Figure 12. Microstructure of NC nickel after ultrasonic treatment (a) and subsequent annealing at the temperatures of 150 (b), 250 (c) and 300 °C (d) [86].

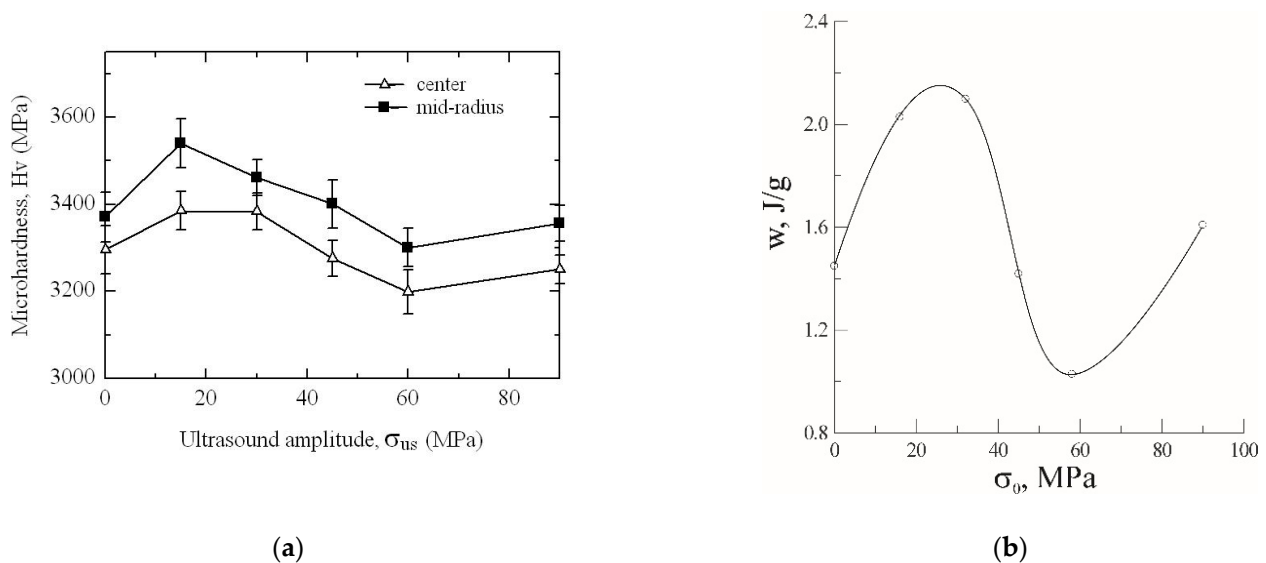


Figure 13. The dependence of microhardness, microstrain (a) and excess energy released during DCS scans (b) of UFG nickel processed by HPT on the amplitude of UST [30].

From the data of the TEM studies, it was concluded that the UST with the amplitude of 15 MPa activated a generation of new dislocations and their accumulation inside grains, which caused an increase in the microhardness, internal stresses and dislocation density, but this stress was not enough for an incorporation of the dislocations into the grain boundaries and the transmission to neighboring grains. The amplitude of 60 MPa was enough for the transmission of dislocations through grain boundaries, their incorporation into grain boundaries, and other processes of rearrangements. This could have led to a change in the misorientations of the boundaries, grain rotation, more effective screening of long-range stress fields of dislocations and corresponding stress relaxation. With a further increase in the stress amplitude, the rate of dislocation generation increases. An UST with an amplitude of 90 MPa is sufficiently intensive to increase the overall density of defects. The accumulation of the latter dominates over the processes of the annihilation

and rearrangement of dislocations. So, it was demonstrated that the ultrasonic treatment of severely deformed materials could result both in structure relaxation and hardening effects; which of these effects was realized depended on the amplitude of the ultrasound.

The effect of UST on the mechanical properties of UFG nickel was studied in [25]. Cylindrical samples with a diameter of 20 mm and length of 100 mm were processed by 12 passes of equal-channel angular pressing (ECAP) at a temperature of $T = 350\text{ }^{\circ}\text{C}$ according to the route B_C , i.e., rotating the samples 90° around their axes in the same direction between subsequent passes [79]. After the ECAP, the samples were subjected to a slight extrusion in order to make their length suitable for the excitation of a standing ultrasonic wave for the frequency of 22 kHz. As a result, two cylindrical samples with a diameter of 18 mm and length 104 mm were fabricated. One of the samples was taken as a reference, while the other was subjected to UST by the method of a half-wave sample with a maximum stress amplitude of $\sigma_0 = 100\text{ MPa}$. The irradiated sample was sliced into 42 discs with a 2.5 mm thickness, and two small samples for tensile tests were cut from each disc. Tensile tests of these samples allowed the authors to plot the dependencies of the characteristics of the strength and ductility on the stress amplitude.

The results of the tensile tests are collected in Figure 14 as the dependencies of the ultimate strength σ_u and elongation to failure δ on the amplitude of oscillating stresses. Two wings of the graphs correspond to the two halves of the sonicated sample. As references, the data for the sample after ECAP are given by the open circles. These data were obtained for the cross sections of the sample which are located different distances from the center and are plotted against the UST amplitudes corresponding to the same locations on the sonicated sample.

The distributions of the ultimate strength and elongation to failure after UST are fairly symmetric with respect to the center of the sample. This means that the attenuation of the wave was not significant and a symmetric standing wave was established in the sample under irradiation. The yield stress σ_y varies in the range from about 800 MPa up to about 1050 MPa in a way similar to the behavior of the ultimate strength.

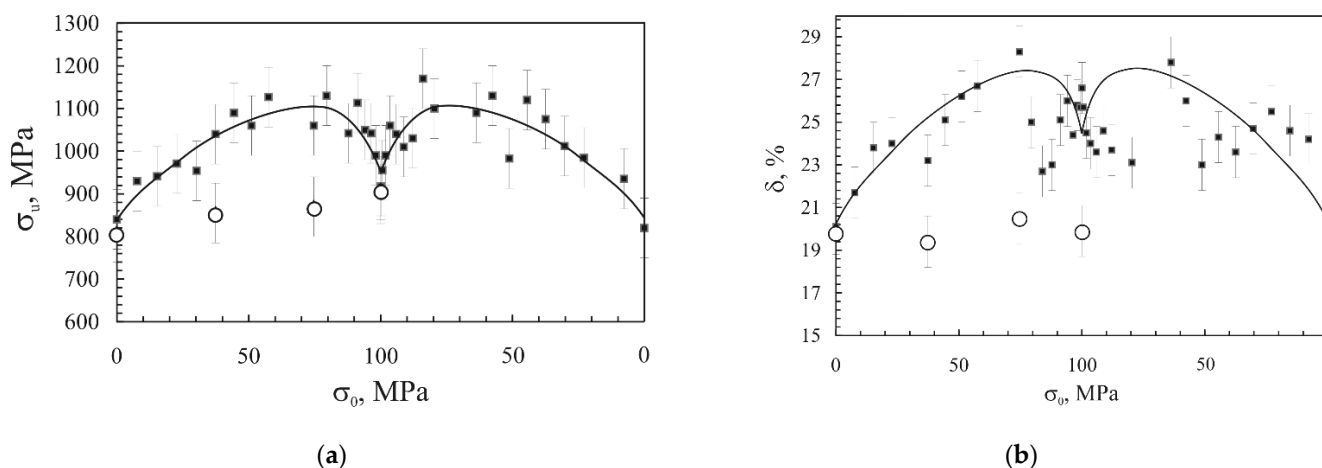


Figure 14. Dependencies of the ultimate strength (a) and elongation to failure (b) of ultrasonically irradiated nanostructured Ni on the amplitude of oscillating stresses. The solid lines give the trends of the changes in characteristics. Open circles are datum points for the non-sonicated ECAP Ni at the sample positions corresponding to four values of the stress amplitude in the sonicated sample [25].

Thus, the results show that the UST leads to a considerable increase in both the strength characteristics and elongation to failure. This increase is not monotonic and exhibits a maximum near the value of $\sigma_0 = 75\text{ MPa}$, after which the effect diminishes with a further increase in the stress amplitude. Thus, there exists an optimal value of the stress amplitude at which the UST can result in an improvement of the mechanical properties of UFG nickel.

It is noteworthy that the changes in ductility and strength of UFG Ni with the UST intensity have the same trends; i.e., they increase simultaneously up to the optimal stress

amplitude. As was noted above, this behavior is not typical, since normally an increase in the ductility is followed by a loss in strength and vice versa.

Later, a detailed analysis of the microstructure and properties of the same samples cut from the cylinder subjected to UST in [25] was carried out [88], and it was shown that the values of the microstrain and microhardness behaved the same way as the strength and ductility; i.e., they increased up to a stress amplitude of about 80 MPa and decreased at 100 MPa at the middle of the sample. The decrease in these characteristics at the middle of the sample was explained by the temperature effect due to the heating of the sample during the UST, which is the maximum at the position of the highest stresses.

UST also significantly affects the impact toughness of UFG nickel. In [89], impact toughness measurements were carried out for samples of ECAP-processed Ni in the as-deformed state and the states after additional annealing at 100 °C and UST with the stress amplitudes of 20, 50 and 100 MPa. The impact fracture energy of the ECAP-processed sample was equal to 116 J, which resulted in the value of impact toughness: $KCV = 2.9 \text{ Jm}^{-2}$. The samples after annealing and UST did not fail even at the maximum value of the impact energy, 706 J, so it was not possible to calculate the impact toughness.

7.2. Hexagonal Close-Packed Metals and Alloys

Zirconium is one of the most widely used materials with an hcp lattice. Applications of this metal and its alloys include various fields, such as structures in nuclear power engineering, biomedical implants, shape memory materials, etc. [90]. The mechanical properties of zirconium alloys can also be enhanced by SPD-processing, but their ductility becomes significantly lower than that in coarse- or fine-grained states [91]. The effect of ultrasound on the structure and mechanical properties of Zr and Zr-alloys were studied in a series of papers by Sokolenko et al. [92,93] and Mats and Netesov [94].

In [92], pure Zr was rolled at a temperature of $-173 \text{ }^{\circ}\text{C}$ up to a strain of 3.9 to obtain a nanosized structure with a grain size of about 80 nm and dislocation density in grains of about $3.4 \times 10^{14} \text{ m}^{-2}$. An UST with a stress amplitude of 70 MPa and duration of 10 min did not noticeably change the grain size, but fragment boundaries became clearer and the dislocation density became lower. In contrast, 1 h of annealing at 750 °C resulted in a recrystallized structure with an average grain size of 1 μm . Creep experiments at room temperature and at 427 °C showed that annealing resulted in a significant drop in the strength of the samples and an increase in ductility. In the case of post-rolling UST, the strength drop did not exceed 15% and the ductility changed considerably. These effects of ultrasound were explained by a redistribution of dislocations, modification in the structure and formation of more perfect and ordered grain boundaries, i.e., by the self-organization of the dislocation subsystem under cyclic stresses. Similar results were obtained on Zr-1Nb alloy subjected to post-rolling UST with an amplitude of 80 MPa [93].

Nanostructured Zr-2.5%Nb processed by rolling was irradiated by ultrasound with stress amplitudes of 85 and 200 MPa in [94]. The changes in the microhardness of the samples greatly depended on the amplitudes of the ultrasound. At an amplitude of 85 MPa, a monotonic decrease in the microhardness occurred from the initial value of approximately 3200 MPa down to about 2500 MPa in 6 min. In contrast, at an amplitude of 200 MPa, the microhardness increased up to about 4500 MPa in the first 1.5 s, then decreased to a value near 3000 MPa and leveled off after 3 min of irradiation. Obviously, the former behavior is related to a relaxation of nonequilibrium structures created by deformation, while the latter resembles the behavior discussed above in relation to Figure 9. The very intensive cyclic straining causes a massive dislocation generation and the additional strengthening of the material. However, when a new, more nonequilibrium structure is created, it becomes unstable under the same ultrasound and its relaxation occurs with a related decrease in microhardness.

The next important hcp metal, Ti, and its alloys are widely used in aviation, aerospace, metallurgy, shipbuilding and automobile industries [95]. Their main advantage over other metals and alloys is their combination of properties, such as lightness, strength, high

corrosion resistance, low thermal expansion coefficient and the ability to work in a wide range of temperatures. Another important property of titanium is its biocompatibility, which allows its use for the production of implants for medical uses [96]. SPD methods, such as HPT and ECAP, have been successfully applied to process UFG structures in pure titanium and its alloys and extraordinary strength and superplasticity characteristics were obtained in the resulting materials [79,80]. Nevertheless, for these materials, it is also important to further enhance their strength, fatigue and superplasticity characteristics and increase their ductility and impact toughness. Therefore, attempts have also been made to modify their structure and properties by means of post-SPD ultrasonic treatment.

In [32,97], the effect of UST on the structure and mechanical properties of α -titanium alloy Ti-5Al-0.5V was investigated. The UFG structure in the rods of this alloy was formed by means of six passes of ECAP in a die with an angle between channels 120° by the Bc root at a temperature of 600°C followed by an extrusion at a temperature of 400°C to a cross-sectional diameter of 16 mm. Then the samples were cut to cylinders with a length of 40 mm. Some of these cylinders were treated by ultrasound and the amplitude of oscillating tension–compression stresses was 100 MPa according to the scheme of Figure 3c.

TEM studies of the sonicated and non-sonicated samples showed that no considerable recovery processes occurred due to the UST. The TEM indicated indirectly that the dislocation density was higher than 10^{13} m^{-2} . The dislocation density was determined by the XRD method; additionally, the dislocation density and concentration of vacancy clusters were characterized by the positron lifetime spectroscopy in the cross and longitudinal sections of the cylindrical samples. The data of the XRD and positron lifetime spectroscopy studies on the dislocation density agree with each other fairly well and yield the values of about $(3\text{--}3.5) \times 10^{14}\text{ m}^{-2}$ in the ECAP-processed sample and $(5\text{--}6.5) \times 10^{14}\text{ m}^{-2}$ in the state after UST. Similarly, the vacancy concentration increased from 2 to 3 ppm up to about 6 to 8 ppm. It was concluded that, although the samples processed by the post-ECAP UST exhibited a higher concentration of vacancy clusters and density of dislocations compared to those of the samples without UST processing, the differences were not great and can be considered to be within the range for experimental errors.

Consistently with the results of structural studies, the microhardness and tensile strength values showed a small increase after the UST: $H_v \approx 3090\text{ MPa}$ and $\sigma_u \approx 1400\text{ MPa}$ in the ECAP-ed sample; and $H_v \approx 3170\text{ MPa}$ and $\sigma_u \approx 1433\text{ MPa}$ in the UST-processed one, respectively.

In [98], disk-shaped samples of commercially pure titanium VT1-0 (Grade 2) processed by HPT under a pressure of 7 GPa up to 10 revolutions, having a mean size of fragments of about 250 nm, was subjected to UST. The UST was carried out at the frequency of 20 kHz in a time interval of 60 s, with stress amplitudes of 40, 60 and 80 MPa. The results obtained showed that the oscillating stresses with an amplitude up to 80 MPa also did not result in noticeable changes in the microstructure and microhardness of severely deformed Ti.

In [99], the ultrafine-grained microstructure in titanium VT1-0 was formed by room-temperature rolling up to a strain of 3.0. Part of the samples were subjected to the UST for a time interval of 30 min with an amplitude of shear stresses of 65 MPa. The rolling resulted in an UFG structure with grain sizes of 55–140 nm; the grains were nearly free of lattice dislocations. The UST did not change the grain size but the structure was more homogeneous and stress-free. Creep tests at temperatures of 20°C and 350°C showed an increase in the tensile strength while maintaining the yield stress and ductility. The strength increase amounted to approximately 8% at 20°C and 20% at 350°C . This is in contrast to the above considered behavior of Zr and Zr alloys, which exhibited softening due to UST. The authors related this difference to a difference in the values of the stacking fault energy, which is equal to 20 mJm^{-2} in titanium and 220 mJm^{-2} in zirconium. The low stacking fault energy in Ti made the recovery processes in this metal more difficult, which is why the action of the ultrasound resulted in an additional strengthening.

Magnesium alloys with an hcp lattice also have been widely applied as structural and functional materials [100]. For these alloys, strength and corrosion resistance are important

issues. In [101], the effects of SPD and post-SPD UST on the strength and corrosion resistance of Mg-5.1 Al-2.5 Zn wt.% were studied. SPD processing was carried out using ECAP followed by a reduction up to an accumulated strain of about 2.0, which resulted in the formation of a bimodal structure with grain sizes of 1–2 and 10–20 μm . The UST was carried out for 120 s with the strain amplitude of $\varepsilon_0 \approx 3.7 \times 10^{-4}$. This treatment resulted in a decrease in the lattice parameter and an increase in the dislocation density. While the deformation processing resulted in a decrease in the volume fraction of second phases due to their dissolution, the UST led to a slight increase in this fraction, indicating some decomposition of the solid solution. Table 2 summarizes the mechanical properties of the alloy in SPD-processed and ultrasonically treated states in comparison to the initial state.

Table 2. Mechanical properties of Mg–Al–Zn alloy in different structural states [101].

State	Ultimate Strength, MPa	Yield Stress, MPa	Elongation, %
Initial	190 $\pm$ 10	120 $\pm$ 5	8 $\pm$ 1
SPD-processed	280 $\pm$ 10	185 $\pm$ 10	14 $\pm$ 1
SPD + UST	310 $\pm$ 5	215 $\pm$ 10	14 $\pm$ 1

As one can see from the table, the UST led to strengthening, in addition to that achieved by SPD processing, while maintaining ductility on the same level. In addition, it was found that SPD and the UST had significant opposite effects on the corrosion resistance of the alloy. The corrosion depth indicator equal to 52 mm/year for the initial state increased up to 145 mm/year after the ECAP followed by a reduction, while the subsequent UST decreased to 58 mm/year, i.e., nearly restored the initial corrosion resistance, simultaneously improving the mechanical properties.

Thus, different effects of UST on the structure and properties of SPD-processed materials with an hcp crystal structure have been obtained. In UFG Zr and its alloys, an UST with amplitudes near 80 MPa results in considerable softening and increase in ductility. In a Mg alloy, in contrast, additional strengthening occurs without a change in the ductility. Only a slight increase in the dislocation density and vacancy concentration is observed during the UST of UFG Ti and its alloys. On the other hand, a relaxation of internal stresses and considerable increase in the strength can occur at longer expositions to ultrasonic irradiation. Therefore, more systematic studies including wide intervals of stress amplitudes and irradiation times are needed to obtain the optimal conditions for providing a significant effect on the structure and properties of titanium materials. Additionally, the structural responses of UFG materials on UST depend not only on the crystal lattice, but also on the stacking fault energy of a particular metal.

A possible explanation of the difference in the behaviors of fcc Ni and hcp Ti under ultrasound was given based on the results of computer simulations and is discussed in Section 9.

8. Simulations of the Effect of Ultrasound on Crystal Lattice Defects

As shown in Section 2, ultrasonic waves influence the structure of materials through oscillating stresses, which act on crystal lattice defects, mainly dislocations. Therefore, this influence can be studied by using models which allow for a description of the effect of oscillating stresses on dislocations. There are two such modeling methods. One is the discrete dislocation dynamics method (often abbreviated as the DDD method), which is based on solving equations of the motion of discrete dislocations or their segments under the action of stresses [102]. The second one is the molecular dynamics (MD) method, which is based on solving equations of the motion of atoms [103]. Both methods are now very successfully and widely used in studies of different processes in crystalline solids. Since the 1980s, the DDD and MD methods have also been applied to studies on the effect of ultrasound on the defect structure of crystals.

8.1. Discrete Dislocation Simulations

To the authors' best knowledge, Fokin and Khannanov were the first to study theoretically and simulate numerically the behavior of a dislocation system in a spatially uniform field of oscillating shear stresses [104]. In this work, the authors considered a dislocation tripole (Figure 15). When a sinusoidal shear stress $\tau = \tau_0 \sin 2\pi f t$ is applied to this system, each dislocation makes an oscillatory movement. An isolated dislocation will vibrate quite symmetrically around its initial position. In a tripole, however, the dislocations interact with each other, and, due to the asymmetry of the system, their movement can be more complicated.

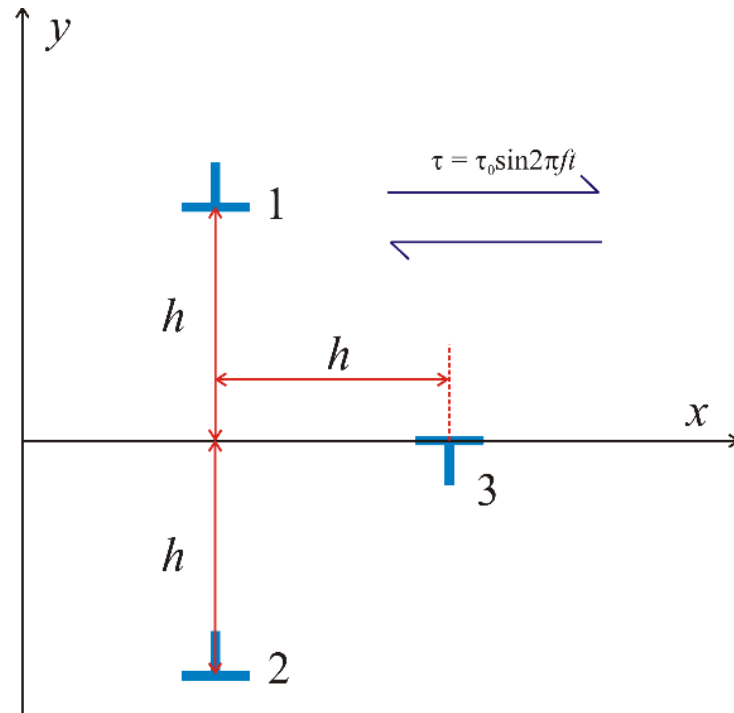


Figure 15. Dislocation tripole in the field of an applied sinusoidal shear stress. 1, 2 and 3 denote the numbers of dislocations.

The dependence of the velocity of lattice dislocations, v , on the shear stress, τ , obeys a power law:

$$v = B|\tau|^m \text{sign } \tau, \quad (2)$$

where B and m are material-dependent constants [105]. In general, exponent m has values ranging from several units to a couple of dozens; i.e., the dependence is strongly non-linear. Qualitatively, however, two types of dependence can be distinguished: a linear one ($m = 1$) and non-linear one ($m > 1$). Fokin and Khannanov [104] showed that in the case of linear dependence, the dislocations vibrate symmetrically around their initial positions and there is no drift movement of the tripole. However, in a non-linear case, a drift motion of the dipole as a whole occurs.

The authors solved the motion equations for dislocations analytically for the case when the displacements of dislocations are smaller than the characteristic tripole size, h , and the dislocation interaction forces are smaller than the forces originating from the applied stress, $b\tau_0$, and calculated the drift velocity $V = (\dot{x}_1 + \dot{x}_2 + \dot{x}_3)/3$, where x_1 , x_2 and x_3 are the coordinates of dislocations 1, 2 and 3 (Figure 15), for the case of $m = 3$:

$$V = CB\tau_0^3 \left[\frac{Gb}{2\pi(1-\nu)\tau_0 h} \right]^3 \left(\frac{L}{h} \right)^3, \quad (3)$$

where $C = 0.132$, $L = B\tau_0^3/2\pi f$, and G and ν the shear modulus and Poisson ratio, respectively. For the general case, the solutions were analyzed by numerically solving the motion equations.

Murzaev et al. analyzed the dynamics of all possible configurations of dislocation tripoles and found that not all of them could be mobile under ultrasound [106,107]. There are three initially stable configurations; each of which have stationary solutions with a non-zero drift velocity. Other configurations are non-mobile or transform to one of the three mobile tripoles in a transient stage of vibrations. The authors generalized their results and put forward a hypothesis that if a dislocation cluster had any asymmetry in the location of the centers of mass of positive and negative dislocations, then under the influence of standing sound waves, these structures could drift.

The first discrete dislocation simulations of polygonization under the influence of ultrasound were carried out in [108]. During low-temperature deformation, due to the existence of dry friction forces acting on dislocations, the latter form non-ideal, loose dislocation boundaries which have a non-screened long-range stress field and an excess of elastic energy (Figure 16a). Shear stresses induced by an ultrasonic wave allow the dislocations to overcome the friction stresses and make them mobile. Therefore, the dislocations will move to their new positions to form fine, flat walls or networks with fully compensated long-range stresses (Figure 16b). In the cited work, a simple example of a system composed of straight edge dislocations was considered and it was found that, indeed, an initially loose dislocation system quickly transformed into a tilt dislocation wall. This effect was called the “ultrasonic stimulation of the polygonization process” [108].

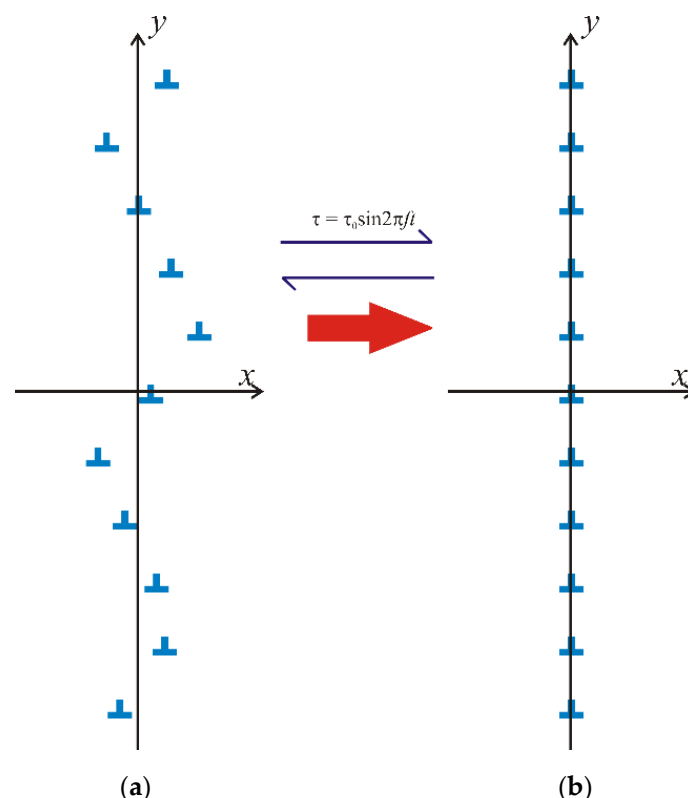


Figure 16. A schematic representation of a loose dislocation wall (a) and ideal, flat wall formed due to ultrasonic treatment (b).

Tyapunina and co-workers carried out computer simulations of an operation of Frank–Read sources and the initial stages of slip band formation under the action of ultrasound [55]. For the Frank–Read sources, dislocation configurations corresponding to the different phases of an external oscillating shear stress were calculated sequentially. The calculations showed that for sources with a length of the order of 1–10 μm , the time required for the

formation of a closed dislocation loop was of the same order of magnitude as the period of ultrasound in the kilohertz frequency range. Inhomogeneous internal stress fields can strongly influence the evolution of a dislocation segment under the action of an ultrasound. This is caused by the fact that they introduce an asymmetry into an effective stress acting on dislocation segments. Simulations of the initial stage of slip band formation show that a source which creates a loop in one plane cannot work effectively under the action of an ultrasound. All dislocations ejected in one half-cycle annihilate in the next half-cycle, when the source generates dislocations of the opposite sign. For the efficient operation of the source under cyclic loading, it is necessary to fully account for the possibility of cross-slip so that dislocations of opposite signs can move on parallel planes. Under cyclic loading, the operation of one source leads to the formation of a cluster of dislocations of opposite signs, i.e., a dipole cluster. As a result, a linear relationship is established between the cluster length and source operating time. After the removal of ultrasonic excitations, a process of stress relaxation can occur due to the redistribution of dislocations in the slip band and the formation of new dislocations. The tensor density of dislocations in a relaxed cluster is close to zero. This conclusion is confirmed by the absence of long-range stresses and features of dislocation structures observed in crystals deformed by ultrasound. In real situations, there is a finite probability of the cross-slip at different distances, so that the operation of a source under the action of an ultrasound should be accompanied by dipole annihilation and the formation of point defects, which is consistent with the experimentally observed distribution of anion vacancies in the bulk of the crystal.

Tyapunia and co-workers [109] also studied the dynamic behavior of a dislocation dipole under oscillating stresses based on the linear version of Equation (2), i.e., at $m = 1$. They found that, at small values of the stress amplitude, each dislocation vibrated around its stable equilibrium position, but above a certain value of the amplitude, they vibrated around a common position, which coincided with the unstable equilibrium position of the static dipole.

Bushueva et al. simulated the transformation of a system of randomly distributed edge dislocations of a single slip system in a NaCl-type crystal [110]. The system consisted of equal numbers of dislocations of two different signs. The simulation showed that the ultrasound led to a redistribution of dislocations, and as a result, dynamically ordered dislocation structures were formed. The type of structures formed depends on the ultrasound amplitude, frequency and action time. The formed ordered dislocation structures are not broken up after stopping the irradiation. At smaller stress amplitudes, single-layer walls of similar dislocations consisting of three to five dislocations are formed and grow further with time. At higher amplitudes, the walls become of a multiple-layer character and contain dipole and multipole configurations of dislocations. The authors demonstrated experimentally that such type structures were formed in MgO and Zn crystals.

Simulations carried out in [110] concerned dislocations distributed in an infinite solid without other defects. Since, in general, crystalline materials are polycrystals, the role of grain boundaries should be considered indispensably. Moreover, of particular interest is the effect of ultrasound on dislocation structures in deformed polycrystals having nonequilibrium grain boundaries, as discussed above. The behavior of such systems at different conditions has been extensively studied recently in [46,111–114].

In [111,112], a model square-shaped columnar grain was considered (Figure 17a). At four junctions of the grain wedge, disclinations composing a disclination quadrupole were placed and equal numbers of dislocations of opposite signs belonging to a slip system were randomly distributed in the grain. If the grain boundaries are high-angle ones, as is most general, they are non-transparent for dislocations, while low-angle boundaries allow them to penetrate into neighboring grains. The motion of dislocations in these two cases were simulated in [112]. Figure 17b–d shows an intermediate stage of the motion of the dislocations, as well as the final structures with non-transparent and transparent grain boundaries, respectively. From these figures, one can see that the dislocation system is polarized under the effect of the UST: defects of one sign are collected on the left boundary

and those of the opposite sign on the right boundary; this occurs to compensate the stress field of the disclination quadrupole.

The evolution of dislocation structures at different numbers of dislocations and amplitudes of ultrasound was studied in more detail in [111]. Figure 18 shows the maps of shear stress-field component σ_{xy} of a disclination quadrupole and a quadrupole with randomly distributed dislocations in the initial state and after different times of ultrasound action. One can see that, indeed, the entrance of dislocations into grain boundaries compensates for the long-range stress fields of the disclinations. This effect largely depends on the amplitude of the oscillating stress. Figure 19 demonstrates stationary structures which are formed after 1200 oscillation periods for several values of the dimensionless parameter K , which determines the stress amplitude: $K = 2\pi(1 - \nu)B\tau_0^{m+1}/Gb\omega$, where B , G , b , ν , τ_0 have the same meanings as in Equation (3), and ω is the strength of the disclinations. At low amplitudes, the number of dislocations entering grain boundaries is relatively small. This results only in a partial relaxation of the stress fields. In this case, some dislocations form dipole and multipole configurations inside the grain. Such clusters can be mobile or immobile. When the stress amplitude is high enough, nearly all dislocations enter the grain boundaries and a more complete compensation of the internal stresses occurs.

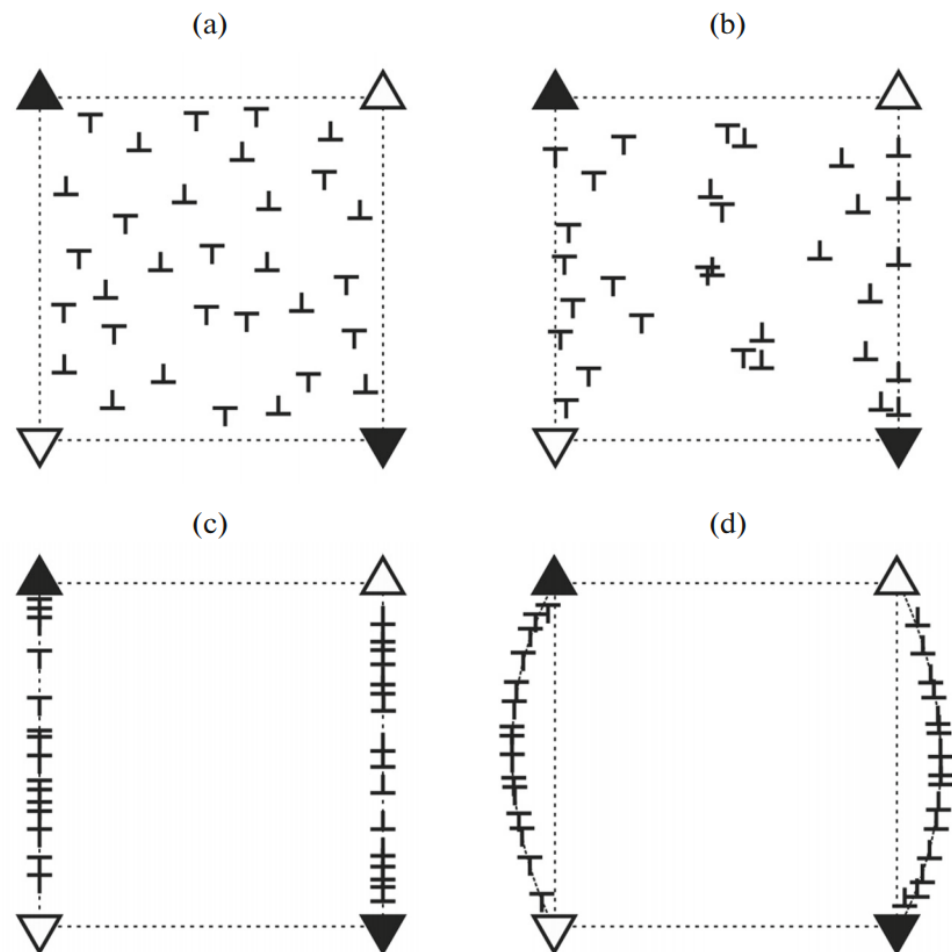


Figure 17. Configurations of a dislocation system in a model square grain with junction disclinations: (a) initial configuration; (b) intermediate stage of UST; (c) final state after UST in the case of impenetrable boundaries; (d) the same in case of transparent boundaries [112].

A similar model, but with dislocations gliding on three slip systems, was considered in [46,113] and the most general case of a multicrystal consisting of nine grains, also with three slip systems, was investigated in [114]. In general, the results obtained using these more generalized models are similar to those considered above; i.e., dislocations enter

grain boundaries and compensate for the internal stresses induced by junction disclinations. However, the presence of multiple slips results in a higher probability of short-range interactions between the dislocations of different slip systems, which leads to the formation of immobile locks in the grains followed by the buildup of a substructure. At low amplitudes, a large number of dislocation locks are formed inside the grains. At high amplitudes, these dislocation locks are destroyed and a more thorough “cleaning” of the grain from the dislocations takes place.

Presented in Figure 20 are the dependencies of the relative elastic energy E/E_0 , where E is the current elastic energy and E_0 the initial one, of the whole system consisting of disclinations and dislocations on time for different ultrasound amplitudes and grain sizes calculated for the parameters of α -iron [114].

An important conclusion from Figure 20a is that the dependence of the final energy reduction due to UST changes non-monotonically with the amplitude, and there is an optimal amplitude at which it is the maximum. This conclusion agrees with the experimental data considered above [87].

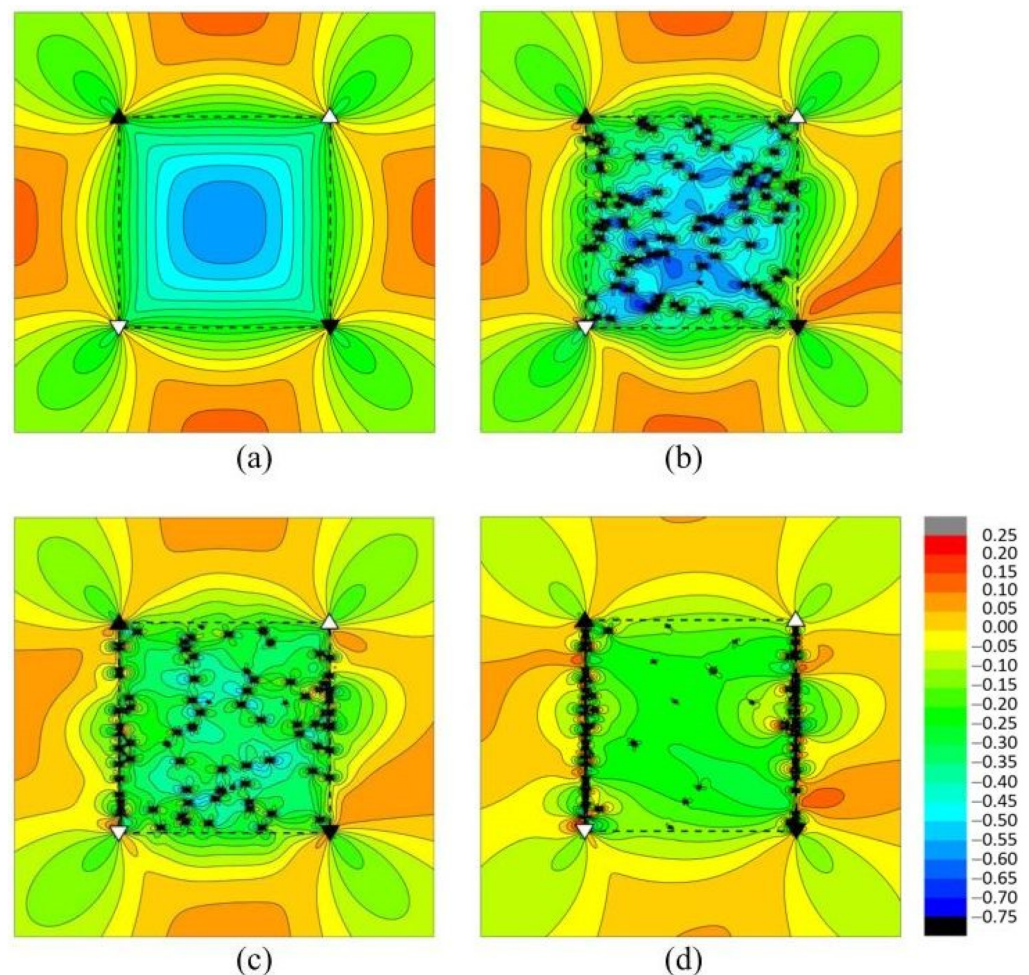


Figure 18. Maps of the shear stress-field component σ_{xy} inside and outside the model grain: (a) disclination quadrupole without lattice dislocations; (b) initial disordered system of 100 dislocations and disclination quadrupole before the ultrasonic action; (c) the same system after 100 and (d) 600 oscillation periods. The dashed line shows the square-shaped cross section of the grain. Black triangles indicate positive wedge disclinations, white ones negative wedge disclinations. Stresses are expressed in dimensionless units [111].

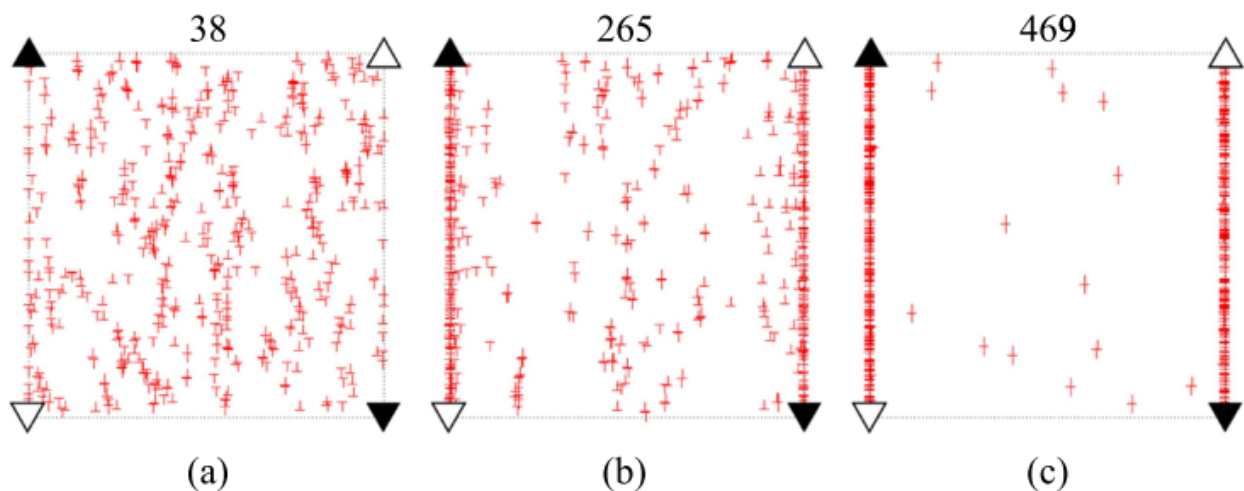


Figure 19. Dislocation positions after ultrasonic treatment during 1200 oscillation periods for different values of parameter $K = 2\pi(1 - \nu)B\tau_0^{m+1}/Gb\omega$: (a) $K = 0.05$; (b) $K = 0.5$; (c) $K = 2$. Black triangle schematically indicates positive wedge disclination, white triangle indicates negative wedge disclination. The dotted lines display the square-shaped grain. The corresponding values of the total number of dislocations entered into the vertical grain boundaries are indicated above the figures [111].

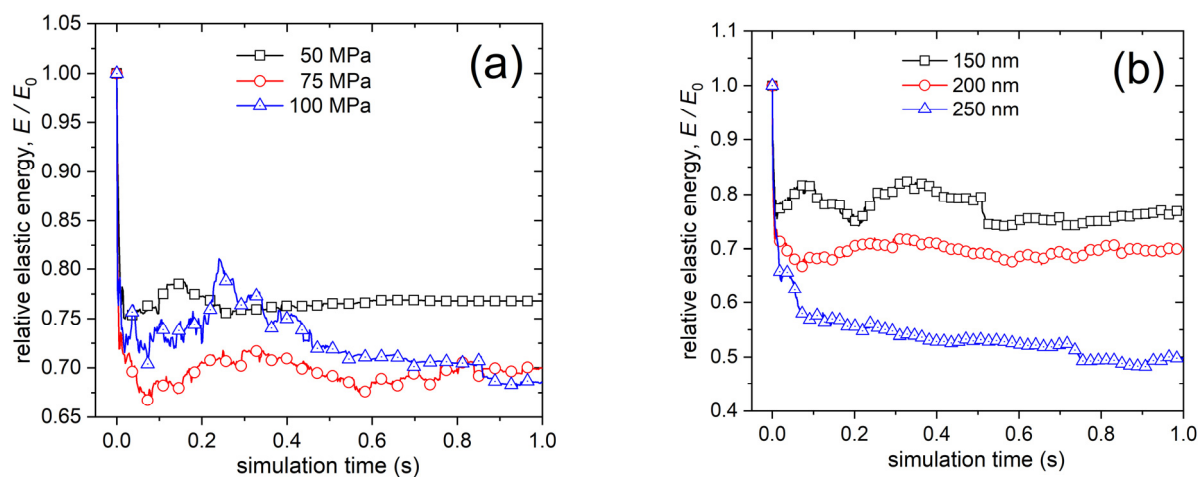


Figure 20. The relative (normalized to the initial value) elastic interaction energy as a function of simulation time calculated for different ultrasonic amplitudes (a) and different grain sizes (b). The grain size is 200 nm in (a) and ultrasonic amplitude is 75 MPa in (b) [114].

8.2. Molecular Dynamics Simulations

The first MD studies of the response of GB atomic structure, energy and mechanical properties of nanocrystals to cyclic straining were carried out by Rupert and Schuh [115]. They constructed nickel nanocrystals with grain sizes from 3 to 10 nm by the Voronoi tessellation method and simulated their behavior under the effect of annealing at 600 K and cyclic strains of loading and unloading. In the as-constructed nanocrystal model, the grain boundaries are in a nonequilibrium, high-energy state. This was also shown earlier in the studies of nanocrystals by Hasnaoui et al. [116]. Rupert and Schuh demonstrated that both the thermal effect and cyclic straining resulted in a significant decrease in the average atomic energy, while the fraction of grain boundary atoms did not change. This meant that grain boundary structure relaxation occurred without a change in the grain size. This relaxation resulted in an increase in the yield stress of the simulated nanocrystals.

The nonequilibrium grain boundary structure considered by Rupert and Schuh and Hasnaoui et al. is essentially different from that caused by EGBDs which form during SPD processing. The grain boundaries in nanocrystals constructed by a Voronoi tessellation

induce only short-range internal stresses, which can relax by the local reconstruction of the grain boundary atomic structure involving the local diffusion or athermal displacements of atoms to distances comparable to interatomic distances. Meanwhile, EGBDs result in long-range stresses, which can relax only by the elimination of EGBDs from the boundaries which requires diffusion to distances comparable to the grain size [117] or athermal processes of dislocation emission. Therefore, of particular interest is the behavior of mesodefects existing in the grain boundaries of UFG metals, for example, junction disclinations, under oscillating stresses. Simulations of this behavior using MD were carried out in [118–122].

In [118,119], the behavior of a dipole of wedge junction disclinations in a [112] tilt grain boundary in Ni under oscillating stresses was considered. The atomic systems for the simulations were constructed by joining bicrystals having different misorientation angles. For instance, in [118], two bicrystals with [112] tilt GBs of $\Sigma = 11/\theta_1 = 62.96^\circ$ and one with GB of $\Sigma = 31/\theta_2 = 52.20^\circ$ with cuts along {111} crystallographic planes were prepared (Figure 21). Then, by applying appropriate shears to the atoms in order to “sew together” the {111} planes, the initial model was obtained. This model allowed for using periodic boundary conditions along the x and z axes and had open surfaces normal to the y axis. Minimizing the energy of this structure at a temperature of absolute zero, a bicrystal with a high-energy nonequilibrium tilt boundary containing a disclination dipole with a strength equal to a difference between the misorientation angles of joined grain boundaries, $\omega = (\theta_1 - \theta_2)$, was obtained.

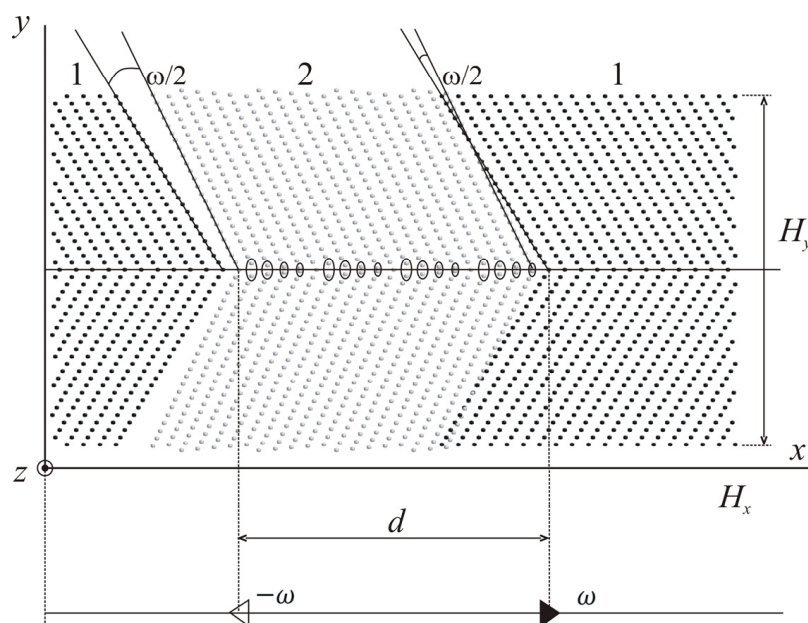


Figure 21. Schematics of the construction of a computational cell [118] by using two bicrystals ($\Sigma = 11/\theta_1 = 62.96^\circ$ (1) and $\Sigma = 31/\theta_2 = 52.20^\circ$ (2)).

The nonequilibrium character of this boundary is demonstrated by the atomic energy map presented in Figure 22a. One can see that near the cores of the disclinations, there are high-energy atoms and the stress field is a long-range one; i.e., it extends to distances comparable to the distance between the disclinations.

This system was subjected to further equilibration at a temperature of 300 K and then subjected to a sinusoidal stress along the x axis, $p_x = p_{x0} \sin[(2\pi/\tau)t]$, with a period of $\tau = 160$ ps. A study of the evolution of the atomic structure with time under this stress allowed the authors to reveal the mechanism of relaxation of the nonequilibrium grain boundary. The atomic energy map for the final structure after five cycles of cyclic straining presented in Figure 22b shows that the grain boundary acquired an equilibrium structure free from long-range stresses.

The mechanism by which such a recovery of the grain boundary occurs was shown to be the emission of lattice dislocations. Figure 23 demonstrates this for the case of another grain boundary with the same tilt axis, but with different misorientation angles [119]. It clearly shows that an edge dislocation with a Burgers vector of $(a/2)\langle 110 \rangle$ type was emitted from the boundary. An analysis shows that the sign of this dislocation is such that it carries away the dislocation charge associated with the disclination dipole. Eventually, the emission of a certain number of dislocations cancels the entire dislocation charge of the disclination dipole.

In the bicrystal model, lattice dislocations emitted from a grain boundary are absorbed by free surfaces which immediately eliminate their stress fields. In a real polycrystal, they are trapped by opposite-grain boundaries so their long-range stresses are not readily cancelled. This can make the above considered mechanism work differently in polycrystals. Therefore, a study of the effect of ultrasound on the nonequilibrium structure of grain boundaries in a polycrystalline material is of interest.

Introducing dislocations into grain boundaries, i.e., creating EGBDs, in computer models of polycrystals is not a trivial task. A method for doing this for the case of columnar, quasi-two-dimensional polycrystals was proposed in [120] and then used for extensive studies of the effect in [121]. The idea behind the method of construction of an atomistic model of a columnar nanocrystal with nonequilibrium grain boundaries is illustrated in Figure 24.

Consider a computational cell of an fcc nanocrystal consisting of infinitely elongated columnar grains (Figure 24). The cell consists of four grains, which have a common crystallographic axis [112], the direction of which coincides with that of the z -axis. The grains have a regular hexagon-shaped cross section with an edge a . The maximum size of the hexagons, $d = 2a$, is taken as the grain size.

The grains of the system can be given arbitrary orientations by rotating them around the tilt axis [112]. In Figure 24, grains 1 and 2 have orientations of the slip planes parallel to xOz and yOz planes, respectively, and grains 3 and 4 are rotated to angles $\pm 20^\circ$ from the orientation of grain 1 to form a symmetric high-angle-tilt GB between each other.

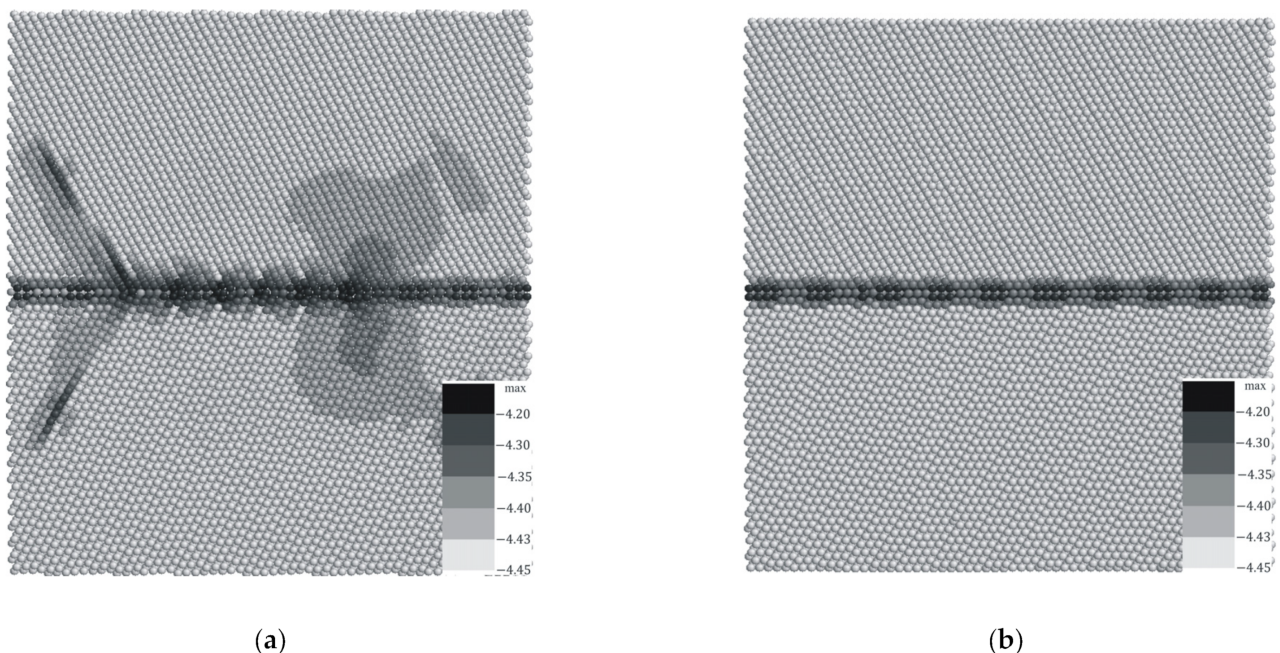


Figure 22. Stress maps of a bicrystal containing a disclination dipole (a) and with a relaxed structure after five cycles of loading with a stress amplitude of 500 MPa (b) [118].

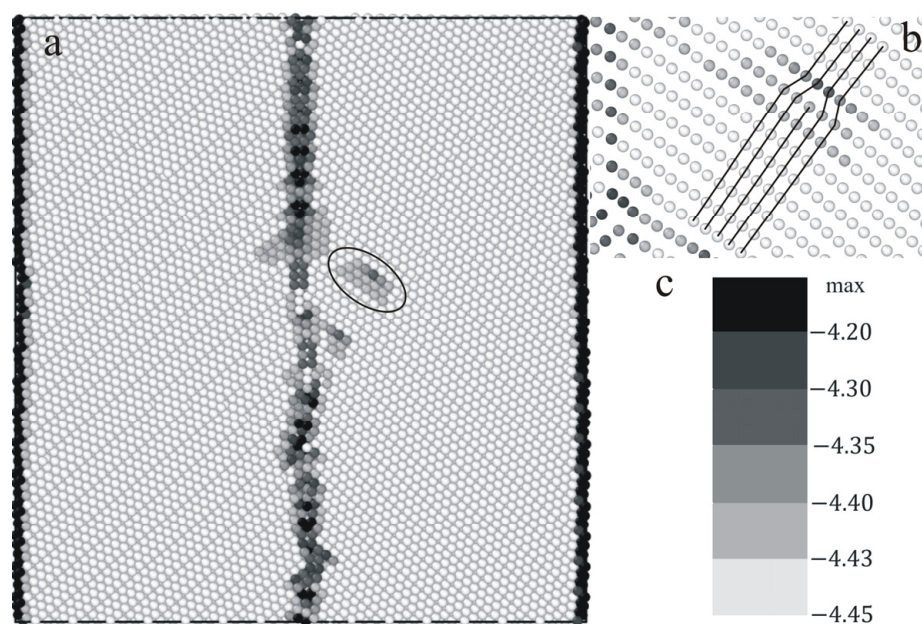


Figure 23. Atomic energy map of nonequilibrium $\Sigma = 15$ GB with a disclination dipole after relaxation at 300 K (a), a magnified image of lattice dislocation located near the GB in the region indicated by an ellipse (b) and energy levels (c) [119].

In order to construct a nanocrystal with equilibrium GBs, the grains are directly filled in by atoms belonging to the crystal lattices of corresponding orientations. The construction of initial structures with nonequilibrium GBs is carried out as follows. Grains 3 and 4, provided that they are isolated, will be distorted into the regions highlighted by a grey color in Figure 24, if some simple shear strain γ by the slip of the dislocations in the (111) slip planes is applied to each of them. In order to obtain corresponding grains with EGBDs, one simply has to distort crystallites with perfect lattices occupying the gray regions into the ones provided for grains 3 and 4, respectively. In fact, there is no need in an explicit definition of the distorted grain regions. Instead, one can fill in larger regions by the atoms belonging to lattices of appropriate orientations, then apply shear distortions $-\gamma$ with respect to the centers of grains 3 and 4 and select only the atoms that belong to these grains. Finally, one will obtain grains 3 and 4, which contain the precursors of the EGBDs schematically depicted in Figure 24. By varying the values of γ , nanocrystals with different degrees of grain boundary nonequilibrium can be created.

Minimizing the initial structures with nonequilibrium grain boundaries by using two different protocols and allowing the EGBDs to stay at their positions, the starting structures for the simulations were constructed with $\gamma = 0.05, 0.10$ and 0.15 . Correspondingly, the systems were denoted as 0.05_1, 0.05_2, 0.10_1, 0.10_2, 0.15_1 and 0.15_2. These structures were subjected to an oscillating stress $p = p_0 \sin(2\pi k/40)$ ($k = 1, 2, \dots, 40$) and for each value of the stress, 2000 steps of MD run were performed. Thus, the period of oscillations was equal to $\tau = 160$ ps. Up to ten cycles of the stress oscillations were simulated. The amplitudes of the applied stress amounted to $p_0 = 2, 3$ and 4 GPa [121].

Figure 25 presents Moiré patterns formed due to a superposition of the visualized atomic structures of nanocrystals with two different levels of nonequilibrium with a periodic pixel structure of a computer monitor. These patterns allow for the visualization of scaled-up distortions of the crystal lattices of the grains in the nanocrystal. One can see that the lattice planes are highly distorted and the distortions increase with increasing values of γ .

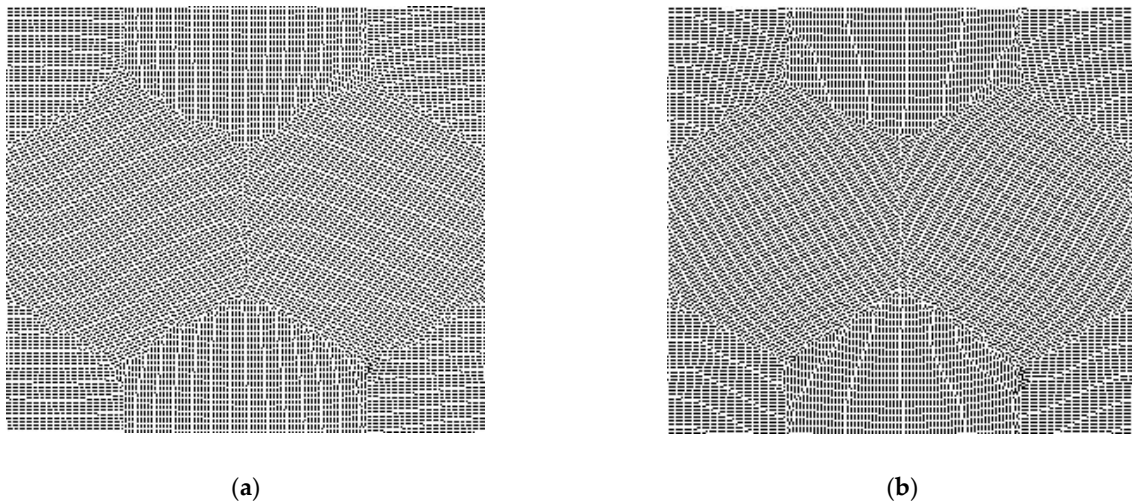


Figure 25. Moiré patterns from the atomic structures of nanocrystals with EGBDs for initial states 0.05_1 (a) and 0.15_1 (b) [121].

Table 3. Energies of grain boundaries in equilibrium and nonequilibrium nanocrystals at zero and room temperatures [121].

State No.	Equilibrium	0.05_1	0.05_2	0.10_1	0.10_2	0.15_1	0.15_2
$T = 0$ K							
E_{def}, Jm^{-2}	1.27	1.41	1.39	1.56	1.54	1.79	1.74
E_{ex}, Jm^{-2}	0	0.14	0.12	0.29	0.27	0.52	0.47
$T = 300$ K							
E_{def}, Jm^{-2}	1.26	1.36	1.34	1.52	1.50	1.75	1.64

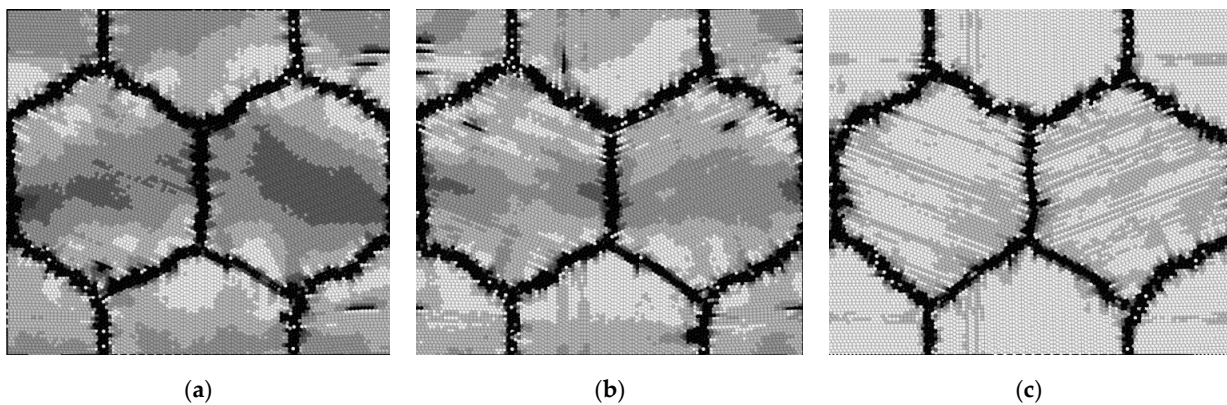


Figure 26. Atomic energy maps of the system 0.15_1 after 10 cycles of loading with stress amplitudes $p_0 = 200$ (a), 300 (b) and 400 MPa (c) at temperature of $T = 300$ K [121].

Table 4. Defect energies per unit GB area in nanocrystal 0.15_1 after 10 cycles of oscillating loading with different amplitudes of the stress [121].

p_0, GPa	E_{def}, eV	E_{def}, Jm^{-2}
0	0.0183	1.75
2	0.0174	1.66
3	0.0158	1.50
4	0.0127	1.21

Thus, the computations show that the process of lattice dislocation generation by nonequilibrium GBs is a mechanism of the effect of ultrasonic treatment on the structure and properties of ultrafine-grained materials processed by deformation methods.

9. Problems and Prospects

The control of the performance of materials through a modification of their microstructures is one of the most important directions of materials science. Modern technology enables a great number of material treatment methods based on actions on microstructures by means of mechanical forces and/or thermal, electrical, magnetic and other fields. Among these, the use of ultrasonic fields occupies a worthy position. As the present review has shown, the action of ultrasound itself is capable of causing significant changes in the structure of materials, resulting in a wide range of effects on their mechanical properties, and, as such, the bulk UST of crystalline materials can be considered as a separate field of technology. Despite the large number of studies carried out up to date and reviewed here, there are problems which should be solved in order to obtain more complete knowledge of the nature and regularities of ultrasound's effect on the microstructure and properties of materials.

The first problem is related to the development of methods for exciting acoustic stresses in bulk solids. Theoretically, the most efficient method should induce a homogeneous oscillating stress field in materials and exclude the loss of acoustic energy when transferring it from an instrument to a material. In the authors' opinion, future studies in this direction should be largely based on finite element simulations of vibrations in systems, including vibration sources and samples taking into account the non-ideal acoustic contacts between them.

The second important direction in this field includes the fundamentals of the ultrasound action on the microstructure and mechanical properties of advanced materials with different crystal structures. It is expected that more extensive experimental studies involving modern techniques, such as EBSD and computer simulations based on dislocation or molecular dynamics and multiscale models, will help to establish the potential of UTS in stabilizing nonequilibrium materials, enhancing their strength, recovering ductility, etc.

The enhancement of ageing kinetics in alloys by ultrasonic action seems to be a promising phenomenon which may find applications in material treatment technologies. Therefore, a study of the ultrasound-assisted ageing of modern commercial alloys and steels might be a fruitful direction for future research in this field too.

Finally, we have seen that UST can influence not only the mechanical performance but also some of the physico-chemical properties of materials. For example, the corrosion resistance of UFG alloys which is significantly degraded after SPD can be recovered to its initial values by UST. Therefore, the effects of UST on corrosion resistance, electric conductivity, shape-memory characteristics and other properties of alloys deserve extensive study in the future.

10. Summary

The present paper aimed to review the studies on the effect of bulk UST on the structure and mechanical properties of crystalline materials from the earliest to the most recent works. The following most significant conclusions can be formed from this review.

1. Ultrasonic irradiation is a versatile method of the treatment of crystalline materials, the effect of which spans from the intensive generation of dislocations and work hardening to the rearrangement and annihilation of dislocations with subsequent softening. In materials with a low density of dislocations, i.e., well-annealed materials, USTs result in a dislocation accumulation and work hardening. In materials with a high density of dislocation, i.e., deformed ones, USTs lead to the relaxation of the dislocation structure and in the most cases to softening.
2. USTs not only change the dislocation structure of materials but greatly affect the ageing in alloys. UST-assisted ageing can result in an order-of-magnitude increase in

the rate of the precipitation of hardening particles which enables us to carry out the ageing processes at lower temperatures than those of ordinary ageing.

3. The post-SPD treatment of ultrafine-grained materials by ultrasound can result in a relaxation of their nonequilibrium structure and internal stresses, thereby enhancing their thermal stability and ductility without compromising the strength achieved due to the SPD. This effect is particularly pronounced in fcc Ni, but for hcp metals and alloys, the result may depend on their stacking fault energy.
4. Discrete dislocation dynamics and molecular dynamics simulations allow for in-depth studies of the effect of cyclic stresses on the dislocation and atomic structures of deformed materials and reveal two basic mechanisms of the relaxation under the effect of UST: the absorption and emission of lattice dislocations by nonequilibrium grain boundaries, which compensates for the EGBD systems accumulated in the grain boundaries during the SPD.
5. Developing methods for exciting ultrasonic stresses in bulk materials, studying the fundamentals of the effect of UST on the structure and properties of nonequilibrium materials with different crystal structures, the ageing of alloys and the physico-mechanical properties of commercial alloys can be considered as key directions for future research in this field.

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Abbreviations

The following abbreviations are used in this manuscript:

CSL	Coincidence site lattice
DDD	Discrete dislocation dynamics
EBSD	Electron back scatter diffraction
ECAP	Equal channel angular pressing
HPT	High pressure torsion
IPF	Inverse pole figure
KAM	Kernel average misorientation
LAB	Low-angle boundary
HAB	High-angle boundary
MD	Molecular dynamics
SPD	Severe plastic deformation
UST	Ultrasonic treatment
UFG	Ultrafine-grained
XRD	X-ray diffraction

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