

Enhanced Ductility of High-Strength Ultrafine-Grained Aluminum Alloys at Ambient Temperature (Review)

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Article history

Received June 02, 2022
Accepted June 10, 2022
Available online June 18, 2022

Abstract

Bulk nanostructured, or ultrafine-grained metals and alloys structured by severe plastic deformation (SPD) methods usually demonstrate high strength and reduced ductility. The poor ductility is a critical issue which limits their practical applications. Significant efforts were made to improve tensile ductility of the SPD-processed metallic materials while keeping sufficiently high strength. In this paper we present a short overview of the developed approaches for simultaneous improvement of the strength and ductility of Al-based alloys with an emphasis on the recent finding and physical reasons of the plasticity enhancement. The main attention is paid to achieving increased ductility of high strength aluminum alloy at room temperature.

Keywords: Aluminum alloys; Ultrafine-grained structure; Ductility; Strength

1. INTRODUCTION

Aluminum and Al-based alloys are very attractive for wide application in various industries such as aerospace, shipbuilding, automotive and electrical engineering. A wide demand for these materials is primarily due to the successful combination of such physical characteristics as high electrical conductivity, corrosion resistance and low specific weight. The main disadvantage of Al and its alloys is their relatively low strength, which, for example, does not allow the use of Al in its pure form to create wires for overhead power lines, so aluminum wires have to be reinforced with a steel core [1]. The latter leads to an increase in power losses during the electric current transmission, a complication of technology and an increase in the cost of the wire production. In order to increase the strength of Al alloys, in addition to suitable alloying, an approach associated with the formation of ultrafine-grained (UFG) structure with a grain size of 100 to 1000 nm or nanocrystalline (NC) structure with a grain size <100 nm by methods of severe plastic deformation (SPD) is becoming increasingly attractive, since, in accordance with the Hall-Petch relation [2], the refinement of the grain structure generally leads to a significant increase in strength. There are many researches where this approach was successfully demonstrated for a number

of commercial Al alloys, such as AA1350 [3,4], AA1570 [5], alloys of the 6xxx [6,7,8], 2xxx series [9,10] and others. However, in addition to achieving high strength, in all these works, after the formation of UFG or NC structures, a significant drop in the ductility was observed, in some cases almost to a brittle state, which greatly limits the possibility of introducing these materials in industry.

It is known that in UFG and NC metals and alloys, plastic deformation can occur due to motion of lattice dislocations, if the grain size is larger than a certain critical value, and due to action of mechanisms associated with grain boundaries (GBs), such as grain boundary sliding (GBS), twinning, emission of dislocations from GBs, if the grain size is smaller than a certain critical value [11]. With a decrease in the grain size and, accordingly, with an increase in the volume density of GBs, the processes of plastic deformation associated with GBs will dominate. The activation energy of these processes is comparable to the energy of crack generation; therefore, the implementation of these processes will be difficult [12].

In general, the trade-off between strength and ductility is a traditional problem in materials science and engineering. These two properties are generally mutually exclusive. Therefore, for practical applications, one usually has to choose between high strength or high ductility, not both

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as desired. Hence it is necessary to search for effective methods for increasing the ductility in UFG and NC Al-based alloys while maintaining high strength. Several research groups reported on metallic NC and UFG materials with good ductility and high-strength at ambient temperatures and superplasticity at high temperatures; see, for example, reviews [13–17]. Meanwhile, however, significant advances have been made in this field in the last few years. This paper provides a brief overview of the main currently known approaches to increase the ductility of UFG and NC Al alloys while maintaining a high level of strength with an emphasis on the recent findings and physical reasons of the ductility enhancement. Particular attention is paid to the recently proposed new approach to increase ductility while maintaining a high level of strength, based on recently discovered two fundamentally new phenomena in UFG Al: hardening by annealing, or annealing-induced hardening (AIH) [3,18,19], and an increase in ductility as a result of subsequent deformation after annealing, deformation-induced softening (DIS) [3,19], which are not typical for coarse-grained (CG) polycrystalline metals.

2. MAIN APPROACH FOR IMPROVING TENSILE DUCTILITY OF ULTRAFINE-GRAINED AND NANOCRYSTALLINE ALUMINUM-BASED ALLOYS

Traditionally, approaches for improving tensile ductility of UFG and NC materials are divided into two groups. The first group implies a variation of testing parameters, such as rate, temperature, type of deformation, etc., in other words, the determination of testing conditions in which increased plasticity is realized. In this review, we will not consider this approach in detail, since it is implemented mainly at elevated temperatures and only in some aluminum alloys. We will only briefly characterize this group of approaches and present the currently available results of their application to achieve improved tensile ductility (and even superplasticity) in UFG and NC alloys based on aluminum. We will focus on the second group of approaches.

The second group of approaches is based on the control of microstructural parameters by forming the desired microstructural elements that provide ductility enhancement in UFG and NC materials. Usually, such approaches are briefly called as microstructural design or microstructural engineering.

2.1. Variation of testing parameters

In accordance with the Hart's criterion [20], uniform deformation turns to the stage of deformation localization under the condition:

$$\frac{d\sigma}{d\varepsilon} \leq (1-m) \cdot \sigma, \quad (1)$$

where $\frac{d\sigma}{d\varepsilon}$ is strain hardening, σ is flow stress, ε is strain,

$m = \frac{d \ln \sigma}{d \ln \dot{\varepsilon}}$ is strain rate sensitivity coefficient, $\dot{\varepsilon}$ is strain rate.

With a high value of the strain rate sensitivity coefficient (m), the material can effectively resist inhomogeneous deformation even in the absence of significant strain hardening. In metals with face-centered cubic crystal lattice, when the grain structure is refined to a nanosize [21], an increase in the sensitivity to the strain rate is observed, especially at low strain rates [13,14].

It is known, that at high homologous temperatures ($T > 0.5T_m$, where T_m is the melt temperature), strain rate sensitivity coefficient in UFG and NC alloy could become high ($m = 0.2-0.4$), which is typical for diffusion-controlled GBS [12]. The latter could significantly improve the stability of uniform deformation, leading to superplasticity. A similar result was demonstrated in Refs. [22,23], where it was shown that UFG alloys of the 7xxx series manifested superplasticity.

In Ref. [22], during mechanical tests in the strain rate range of $10^{-2}-10^{-4} \text{ s}^{-1}$ at elevated temperatures (120–200 °C) samples of UFG industrial AA7475 (Al–Zn–Mg–Cu) alloy demonstrated the ability to superplasticity at high values of the strain rate sensitivity coefficient (Fig. 1). The elongation to failure at a temperature of 200 °C and a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ reached a value of $\sim 700\%$, a strain rate sensitivity coefficient was equal to $m = 0.73$ in the strain rate range $5 \times 10^{-4}-10^{-3} \text{ s}^{-1}$ [22].

In Refs. [23,24], similar results were obtained for the UFG Al–4.8Zn–1.2Mg–0.14Zr (wt. %) alloy. During mechanical tensile tests at elevated temperatures (120–170 °C) in the strain rate range of $10^{-4}-10^{-2} \text{ s}^{-1}$, the value of elongation to failure reached 500%, the strain rate sensitivity coefficient being equal to $m = 0.4$.

Separately, it is worth noting highly alloyed UFG Al alloys of the Al–Zn system [25,26,27], which having a high strain rate sensitivity coefficient m , demonstrated not only high ductility, but even superplasticity at room temperature ($T < 0.5T_m$). According to Refs. [25,26], such high ductility is explained by the intensification of GBS processes, which are characterized by high values of strain rate sensitivity coefficient m .

For example, in Ref. [27], the Al–30Zn alloy (wt. %) with UFG structure demonstrated ductility of $\sim 105-160\%$ at room temperature (RT) in the strain rate range of $10^{-3}-10^{-4} \text{ s}^{-1}$, the strain rate sensitivity coefficient varied within 0.24–0.29. Such superplastic flow in high-alloyed Al–Zn system at RT is associated with extremely

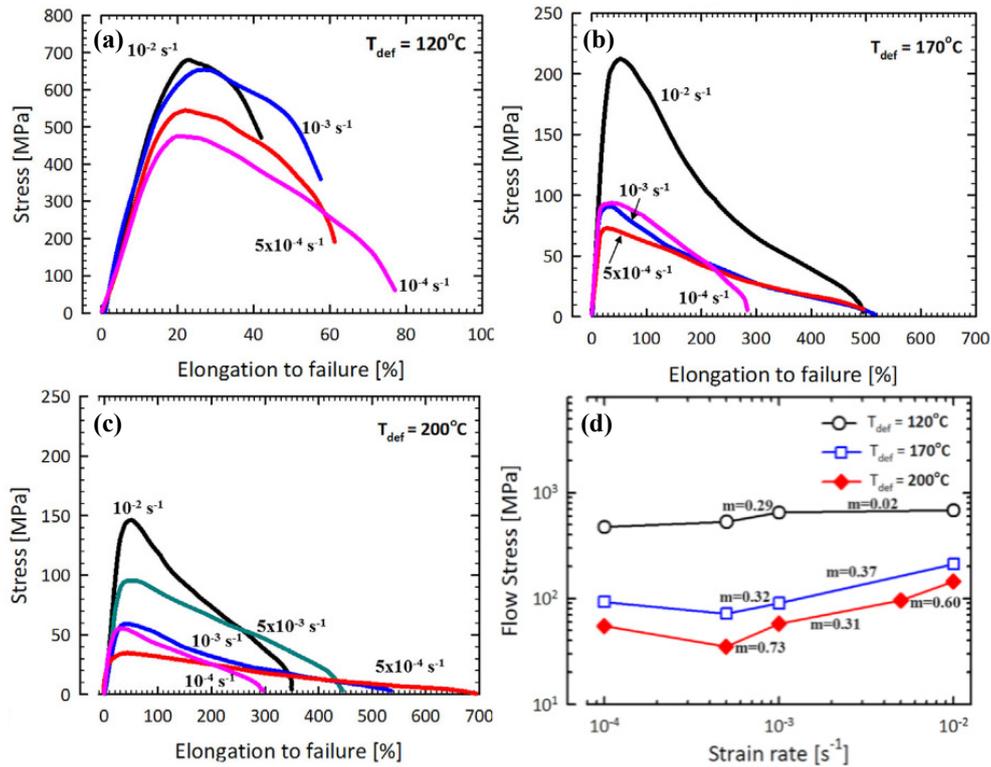


Fig. 1. Stress-strain curves for samples of UFG alloy AA7475 obtained at different strain rates at elevated temperatures: (a) 120 °C, (b) 170 °C, (c) 200 °C, (d) Flow stress-strain rate curve with determined strain-rate sensitivity coefficient for various temperature. Reproduced from Bobruk et al. [22], with permission, © 2018 Wiley-VCH Verlag.

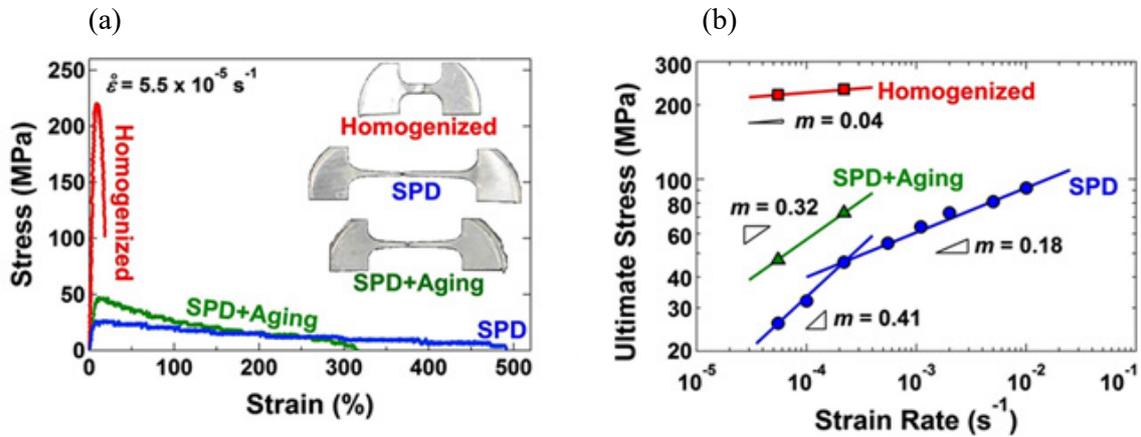


Fig. 2. (a) Stress-strain curves at the strain rate $5.5 \times 10^{-5} \text{ s}^{-1}$ and RT, (b) change in ultimate stress and the values of the strain rate sensitivity coefficient m for the Al-30Zn (at. %) alloy processed by HPT. Reproduced from Edalati et al. [25], with permission (Open Access), © 2018 Nature Publishing Group.

high level of Zn segregation (even wetting) at GBs [26,27]. In Ref. [25], UFG Al-30Zn (at. %) alloys processed by high pressure torsion (HPT) (SPD state in Fig. 2) demonstrated superplasticity at RT in a wide strain rate range of $5.5 \times 10^{-5} - 10^{-2} \text{ s}^{-1}$, strain rate sensitivity coefficient varied within 0.18–0.41. Maximum elongation of 480% was achieved, when tested at a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$.

High ductility $> 80\%$ at RT has been also achieved in HPT-processed Al-7Si (wt. %) alloy, which had a strain rate sensitivity coefficient of ~ 0.14 (Fig. 3) [28].

Thus, the results shown above indicate that the superplasticity of aluminum-based UFG alloys can be obtained not only at elevated temperatures, but at RT, with GBS playing a key role in the plastic deformation process, as evidenced by the high values of the strain rate sensitivity coefficients.

However, it should be noted that such outstanding values of ductility (and even superplasticity) were manifested either when tested at elevated temperatures at certain strain rates and, as a rule, were accompanied by a significant decrease in strength, or required high alloying, which

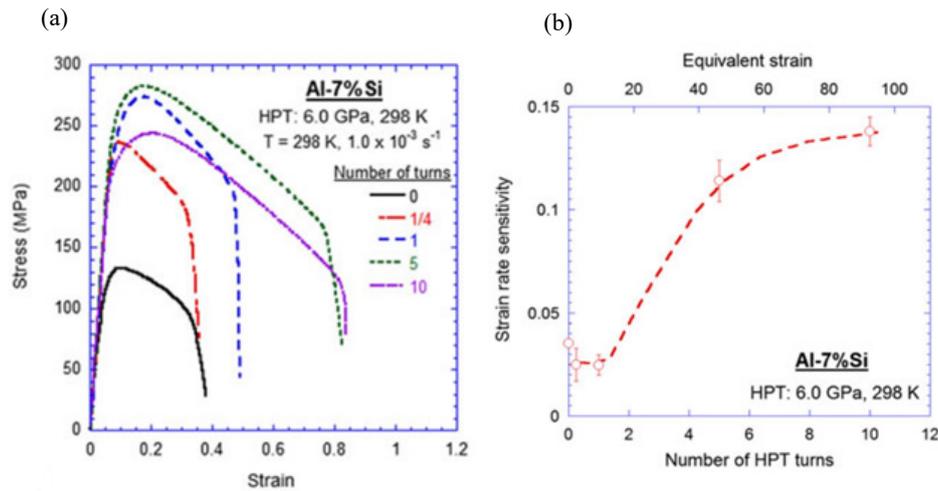


Fig. 3. (a) Stress-strain curves for the samples of Al-7Si (wt. %) alloy at RT, (b) strain-rate sensitivity and strain changing depending on the number of HPT turns. Adapted from Mungole et al. [28].

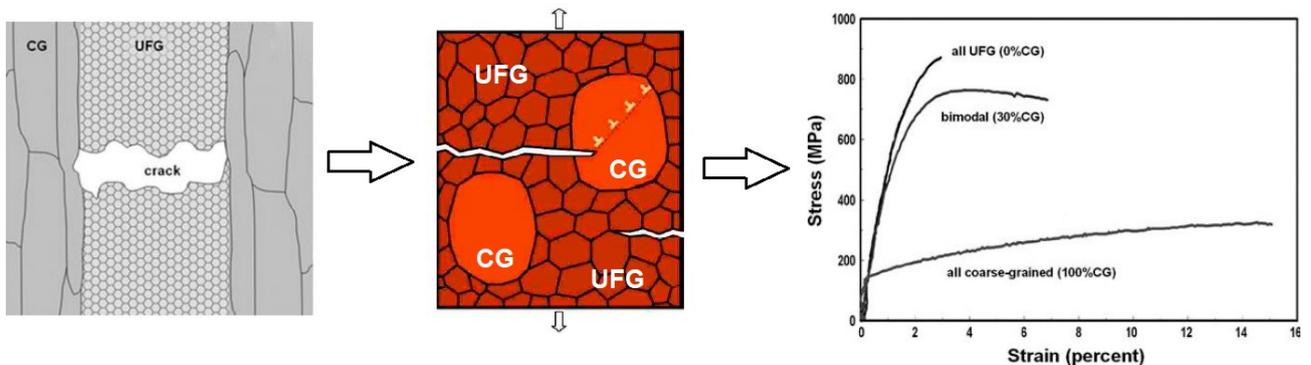


Fig. 4. Concept of a bimodal grain distribution, leading to an increase in plasticity, in the exemplary case of the Al-7.5Mg (wt. %) alloy (CG – coarse grain, UFG – ultrafine grain). Reproduced from Lee et al. [30], with permission (Open Access), © 2010 Springer.

provided wetting of GBs and promoted implementation of GBS mechanisms under RT conditions [25]. Thus, it can be concluded that this group of approaches is quite effective for finding the optimal operating modes for some individual aluminum alloys, but is not universal for solving the problem of combining high strength and ductility in a wide range of UFG Al alloys.

2.2. Microstructural engineering

As noted above, to increase plasticity, of greatest interest are approaches based on the design of the microstructure — the creation of specific regulated structures that increase the ability of UFG or NC materials in a high-strength and even superstrong state to plastic deformation at ambient temperatures.

2.2.1. Formation of bimodal structure

First of all, such approaches include the formation of a bimodal structure by various methods [12,29]. This approach involves the introduction of coarse grains (up to

several microns in size) into the UFG (or NC) matrix material. The concept of this method is that the UFG/NC matrix provides high strength, while coarse grains suppress the growth of cracks by blunting their tips due to the emission of lattice dislocations. In this case, the accumulation of lattice dislocations inside the volume of coarse grains provides strain hardening, which leads to an increase in the magnitude of plastic deformation before failure. This approach is shown schematically in Fig. 4.

In order to obtain such types of structures in Al alloys, powder metallurgy methods are generally used, in particular, mixing of nanosized powders obtained by cryomilling with conventional (micron-sized) powders, followed by consolidation of the resulting mixture by various methods [29–35]. As a result of the formation of a bimodal structure, an increase in ductility was observed while maintaining a high level of strength in a number of UFG Al alloys. For example, in Ref. [29], a bimodal grain structure was formed in the AA5083 alloy by cryomilling and subsequent consolidation at elevated temperatures, which demonstrated acceptable plasticity (8.4% elongation to failure) and, at the same time, a high level of ultimate

tensile strength (462 MPa). A similar approach was also applied for the Al–7.5Mg alloy [30]. By increasing the proportion of coarse-grained powder to 30%, it was possible to increase plasticity by more than 2 times, while the yield strength decreased only by 15% compared to that in a sample with a completely homogeneous UFG structure [30]. As a result, an outstanding combination of reasonable ductility ($\delta \sim 7\%$) and ultrahigh strength $\sigma_{UTS}^{UFG+CG} \sim 760$ MPa was achieved.

In addition to powder metallurgy methods, a combination of SPD and annealing can be used to form a bimodal structure. In Ref. [36], Al–2.5Cu (wt. %) and Al–2.5Cu–0.3Sc (wt. %) alloys processed by equal channel angular pressing (ECAP) at room and cryogenic temperatures were studied. During ECAP at cryogenic temperature, a bimodal grain structure was formed in the alloys. The formation of such a structure is due to the effect of low temperature on the process of activation of dislocation slip and, as a consequence, the difficulty of forming a subgrain structure at cryogenic temperature. After the deformation processing, the samples were subjected to aging at 125 °C for up to 40 hours. It is shown that samples after ECAP at cryogenic temperature demonstrate increased strength and plasticity compared to samples after ECAP at RT, and subsequent aging leads to an additional increase in strength and plasticity while maintaining a bimodal grain size distribution.

It was shown in Ref. [37], that after 7 cycles of accumulative roll bonding (ARB) at RT and annealing at a temperature of 300 °C for 30 min, a bimodal structure is formed in samples of the AA5052 alloy. As a result, the ductility after annealing significantly increased ($\delta \sim 16\%$), however the strength, although it exceeded the values in the state before ARB processing, decreased by more than two times ($\sigma_{UTS} \sim 260$ MPa).

In a number of special multicomponent alloys, a bimodal structure can be formed directly during SPD at elevated temperatures. As shown in Ref. [38], after ECAP at elevated temperatures ($T = 325$ °C) in samples of the 1570C alloy (Al–5Mg–0.18Mn–0.2Sc–0.08Zr–0.002Be (wt. %)), a bimodal grain size distribution was formed.

In recent years, significant progress in obtaining bimodal structures from initial powders has been achieved through the use of additive technologies (AT) [39]. It was shown in Ref. [39] that the samples of the Scalmalloy (Al–Mg–Sc–Zr) alloy obtained by additive manufacturing methods (in particular, by the method of selective laser melting (SLM)) has a bimodal structure (Fig. 5) and demonstrates very impressive strength ($\sigma_{UTS} \sim 415$ MPa) and ductility ($\delta \sim 14$ –17%). The Sc-/Zr-modified Al-alloys (Scalmalloy) is well processable with SLM, reaching densities $> 99\%$. The reasons for the bimodal grain size distribution are not yet fully understood. As assumed in

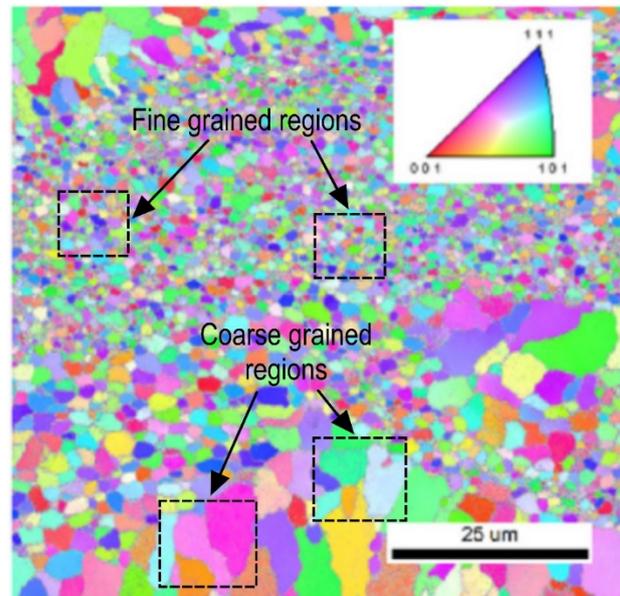


Fig. 5. EBSD map of a sample (the Scalmalloy Al–Mg–Sc–Zr alloy) manufactured with SLM, showing a bimodal grain size distribution. EBSD – electron back scattering diffraction.

Refs. [40,41], a high number of Al–Sc seed crystals are formed during consolidation, favoring the formation of fine-grained material, whereas the CG-regions are formed in areas, where the density of such seed crystals is reduced. In such areas, grains grow along the cooling gradient, leading to the formation of coarser elongated grains, as typical for additive manufacturing (AM) processed materials. As a result, areas with a fine-grained microstructure alternate with areas of coarser elongated grains. Due to the relatively high cost of scandium and manufacturing technology, such alloys are not considered promising for commercial applications, although their properties are quite excellent. In addition, the number of conventional aluminum alloys that do not have inherent and substantial processing difficulties by AT is limited.

Thus, there is no doubt that the formation of a bimodal structure can provide an increase in plasticity in UFG materials, while maintaining an increased level of strength compared to the CG state, as shown above. However, it should be noted that the strength of materials with this type of structure will be lower compared to UFG materials with totally uniform UFG structure [42]. In addition, the formation of a bimodal structure in Al-based alloys is usually carried out using cryogenic temperatures or expensive additive technologies, the latter being applicable only to a narrow range of systems.

2.2.2. Introduction of nanosized precipitates

Another approach to create a microstructural design that provides a simultaneous increase in the yield strength and ductility is the introduction of nanosized precipitates

of secondary phases into the structure [43]. The concept of this approach is that when nanosized precipitates are introduced into the volume of UFG (NC) grains, un-sheared nanosized precipitates contribute to the accumulation of dislocations inside the grains, which in turn leads to increased strain hardening and an increase in plasticity. In addition, nanosized precipitates lead to the suppression of dislocation glide, increasing the stress required to move dislocations through such particles, which should lead to an increase in the yield strength [42].

The most common method for introducing nanoscale precipitates into the grain structure is the aging (artificial or natural) of UFG (NC) alloys in the state of a supersaturated solid solution [44–52]. There are also known cases of the formation of nanoprecipitates during SPD at elevated temperatures [53,54].

This approach has been successfully realized on various systems of precipitation-hardened aluminum alloys, such as Al–Zn–Mg [46], Al–Mg–Si [47,48,55], Al–Cu–Mg [56], Al–Zn–Mg–Cu [57] alloys and Al–10.8Ag (wt. %) alloy [49]. For example, the authors of Ref. [46] studied the AA7075 alloy with nanostructure formed during rolling at a cryogenic temperature. In order to form particles of the secondary phase, annealing was carried out at low temperatures of 50 °C and 80 °C for 5 and 9 hours, respectively. As a result of the formation of a large number of particles of η and η' phases after annealing, the yield strength 615 MPa and plasticity > 10% were achieved. Thus, the yield strength increased by ~ 12%, and the plasticity increased by about 2 times compared with the NC state without precipitates. The AA7075 alloy was also studied in Ref. [54]. However, in this case, nanoscale precipitates (CrMn, metastable phases η_p and η') were revealed after ECAP performed at a temperature of 250 °C. As a result, the yield strength of this alloy was ~ 350 MPa and ductility ~ 19–20%. Similar results were also obtained for AA6060 [58], AA6061 [55,59], AA6063 [47], and Al–10.8Ag [49] alloys.

In Ref. [44], a rather interesting method was shown to modernize the described approach via the precipitation optimization and texture design. In this work, the Al2024 (Al–4.3Cu–1.5Mg–0.6Mn–0.5Fe–0.5Si–0.3Zn (wt. %)) alloy was studied. In order to optimize the distribution of precipitations, formation of its embryo (nanoclusters) of precipitates was used. Initially, in order to obtain precipitates, a series of annealing was carried out at a temperature of 433 K for 10, 23, and 43 hours. Then the samples were subjected to the cold rolling (CR) method, followed by repeated annealing at temperatures of 373 and 433 K for 100 and 150 hours. As a result, the alloy samples annealed for 10 hours at $T = 433$ K, after CR [44] and repeated annealing at $T = 373$ K for 150 hours, demonstrated high ductility (~ 10%) and very high strength (~ 675 MPa). The authors explain the achievement of such high values of strength and plasticity simultaneously by the following processes (Fig. 6). An appropriate fraction of deformable precipitations was introduced into the CG matrix by preliminary aging, which facilitated fast reduction of (sub)grain size during subsequent CR and formation of nanolaminated structure with strong texture. At high SPD by CR, the strain-induced dissolution of primary (initial) precipitates in the matrix occurs, accompanied by the formation of high-density clusters of alloying atoms at dislocations inside nanosized grains. It is assumed that during subsequent annealing, these clusters serve as nuclei for the formation of nanoscale precipitates, which prevent the movement of dislocations, and also promote accommodation of plastic deformation under loading, leading to an increase in both strength and plasticity. The layered structure, in view of the high textural hardening (~ 110 MPa), is also involved in providing high strength (its relative contribution to the total strength was 20–25%).

It should be noted that this approach to achieve a combination of high strength and high ductility by introduction of nanoscale precipitates can only be applied to age-hardenable alloys [60].

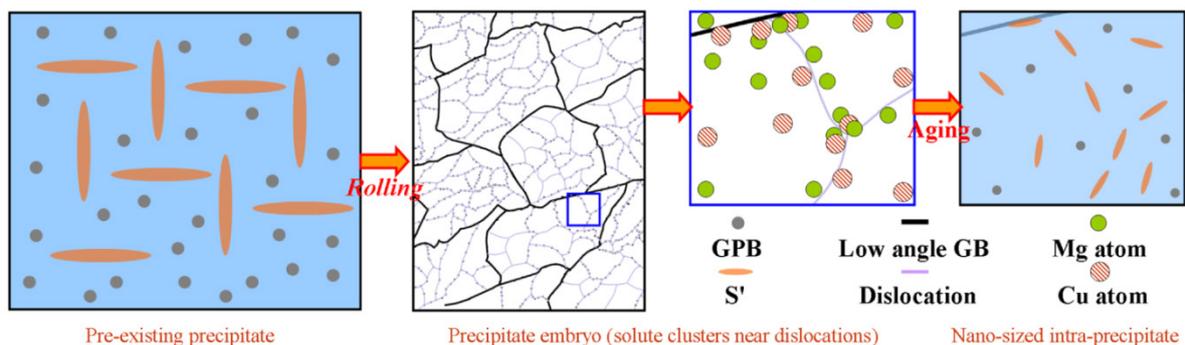


Fig. 6. A schematic showing the precipitation optimization in NS Al2024 alloy. Adapted from Xu and Luo [44].

2.2.3. Introduction of nanotwins

The concept of this approach is to introduce nanosized twins into grain interior. Since twin boundaries have significantly lower energy compared to high-angle grain boundaries (HAGBs), which are typical for UFG and NC materials, they are much more resistant to the process of migration and serve as barriers for moving dislocations, resulting in dislocation accumulation in grain interior. The latter provides an increase in strain hardening and, as a consequence, an increase in plasticity. Twins also well contribute to strengthening.

This approach is mentioned in this review solely to create the integrity of the review. Despite the fact that this approach has been successfully used to increase plasticity in NC Cu [61] and steel [62], it cannot be applied to UFG and NC aluminum and its alloys due to the high stacking fault energy in aluminum lattice [63]. However, it should be noted that there are a few studies where the authors claim to have observed twin structures in some UFG and NC Al-alloys of the Al–Mg system [64,65,66]. Just recently, a study [67] was published, which showed that twinning deformation can be used to overcome the limitation of work hardening, which leads to a decrease in ductility in UFG Al alloys. However, in Ref. [67], a special, high-entropy UFG $\text{Al}_{0.3}\text{CoCrFeNi}$ alloy has been studied. Despite the fact that the impressive mechanical characteristics (the yield strength 900 MPa, the ultimate tensile strength 1074 MPa, plasticity ~ 25%) were achieved as a result of the formation of a large number of deformation nanotwins, which contribute to the accumulation of dislocations in grains [67], similar high-entropy alloys are beyond the scope of this review and mentioned only to complement the modern state of research in this direction.

2.2.4. Creating a defect-free structure

This approach was demonstrated in Ref. [68], where a bulk NC Al–5Mg (at. %) alloy was produced using in situ consolidation through mechanical alloying during milling at liquid nitrogen [68]. As a result, the microstructure with the average grain size of 26 nm with a relatively narrow grain size distribution was obtained, a supersaturated solid solution of Mg in aluminum matrix was formed. This alloy demonstrated the ultimate tensile strength of 742 MPa and yield stress of 620 MPa, elongation to failure reached 8.5%. The authors believe that the observed strain hardening is associated with the accumulation of dislocations during plastic deformation. The authors also explain such good plasticity by the fact that the proposed novel technique of in situ consolidation during milling permitted them to consolidate the material without porosity and microcracks.

2.2.5. Other approaches

In Ref. [69], the Al–1Mg (wt. %) alloy was studied after HPT treatment. It is known that at such low concentrations, Mg in the Al matrix exists as a solid solution [70]. After HPT, the samples showed brittle fracture. Subsequent annealing for 10 minutes at 150 °C led to a significant increase in ductility up to 20%, while the ultimate strength decreased by only 25%. However, the authors do not explain the physical reasons for this effect.

2.2.6. Grain boundary engineering

Alternative approach to enhance ductility of the UFG Al-alloys can be grain boundary engineering. There are a number of works that show that one of the ways to increase the ductility of UFG aluminum is to change the structure and state of GBs. Thus, in Ref. [71], an UFG structure was formed in commercially pure aluminum by the method of accumulative roll bonding (ARB), which provided a simultaneous increase in both strength and ductility with an increase in the number of ARB passes. This effect was explained by formation of a large number of HAGBs during ARB processing. The authors believe that GBs in the UFG structure are effective sources and sinks for dislocations, and the probability of interaction between dislocations and GBs in such a structure increases with the number of ARB passes, therefore, the thermally activated process of dislocation annihilation in GBs plays a decisive role in increasing plasticity [71].

Recently, a new approach (also based on grain boundary engineering) has been proposed to achieve high strength and high plasticity in UFG Al and some UFG Al-based alloys structured by SPD, which consists in a special deformation-heat treatment (DHT) of the material after SPD processing. Such deformation-heat treatment includes low-temperature short-term annealing and subsequent small additional HPT deformation of UFG or NC alloys structured by HPT method [3,18,19,72–74]. It has been demonstrated that the ductility of commercially pure (CP) aluminum processed by HPT can be significantly increased by short-term annealing at 150 °C and subsequent additional deformation by HPT to 0.25 turns at RT [3]. In UFG CP Al, very high ductility (41%), exceeding the ductility of the CG material, was achieved, while maintaining a high level of strength (Fig. 7) [3].

Such an increase in plasticity due to additional deformation is not typical for CG structures. It was shown that the effect of an increase in plasticity after additional HPT deformation (plastification effect) was caused by the introduction of an additional dislocation density into the structure of HAGBs relaxed after annealing.

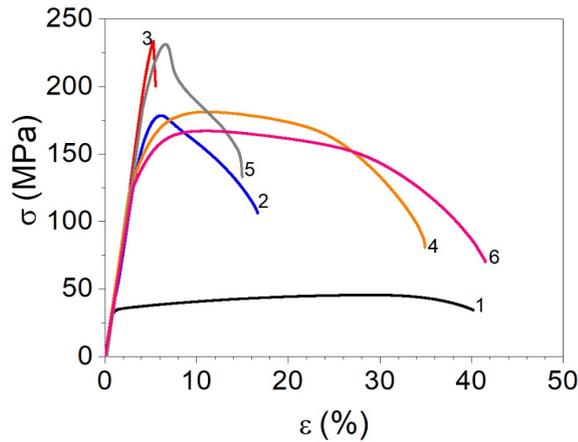


Fig. 7. Stress–strain diagram of CP Al specimens: (1) initial CG state, (2) after 10-revolution HPT at room temperature, (3) after HPT and annealing at 150 °C; (4) after the treatment similar to the treatment for (3) plus 0.25-revolution HPT at room temperature; (5) the same treatment as that for (4) plus annealing at 150°C for 1 h; (6) the same treatment as that for (5) plus 0.25-revolution HPT at room temperature. Reproduced from Orlova et al. [19], with permission, © 2018 Walter de Gruyter GmbH.

To explain the plastification effect, a theoretical model was proposed [19,72]. According to the model, the plastic deformation in UFG Al occurs through emission of lattice dislocations (LDs) from triple junctions of GBs containing pile-ups of GBDs, glide of the LDs across neighboring grains, their accumulation at and climb along the opposite GBs (Fig. 8). The increase in plasticity after DHT is due to the introduction of an additional density of GBDs during additional deformation into the HAGB structure relaxed after annealing. The GBD pile-ups become relatively stronger and can emit a large number of LDs under a relatively lower external stress, which results in a relatively lower strength and higher ductility of the UFG material. Thus, according to Refs. [19,72], the nature of the increase in plasticity due to deformation is associated with a change in the dislocation structure of the GBs (the degree of nonequilibrium of the GBs), which facilitates the emission of dislocations from triple junctions.

It is noteworthy that the very short-term low-temperature annealing of HPT-processed Al leads to an increase in strength, accompanied by a drastic drop in ductility almost to the brittle state, an effect that is absolutely not typical for the CG state [3,19].

The effect of annealing-induced hardening (AIH) was first discovered by Huang et al. [18] for Al structured by the ARB method. An increase of the yield stress by about 8% was found after annealing of ARB-processed Al at 150 °C for 30 min. Later, a great increase in ultimate tensile strength (50%) and yield strength (30%) was

achieved (Fig. 9) for HPT-processed Al due to similar annealing at 150 °C for 1 hour [3,19].

In Ref. [18], the authors explain the effect of AIH in ARB Al by a decrease in the dislocation density in grain interior during annealing, which leads to a decrease in the sources of dislocations in the grains and, consequently, to an increase in the yield strength. Within the framework of the model [19,72], the AIH effect and a dramatic decrease in plasticity after annealing in HPT-processed Al are due to relaxation of non-equilibrium HAGBs during annealing, which is accompanied by annihilation of extrinsic dislocations at GBs, which leads to depletion of the dislocation pile-ups, which are formed at triple junctions under external stresses (Fig. 8). Hence, an increase of external stress is required for dislocation emission and fewer dislocations may be emitted. This explanation is in full agreement with the experimental findings [3,19] for CP Al, including the effect of deformation temperature on the AIH effect [75]. The colossal effect of AIH was also observed for the Al–0.4Zr (wt. %) alloy [76,77]. The authors convincingly showed that this effect significantly exceeds the maximum possible dispersion strengthening in this system [76, 77] and therefore cannot be caused by it. The authors believe that, similarly to the case of CP Al, the AIH effect in UFG Al–0.4Zr (wt. %) alloy is also associated with the relaxation of nonequilibrium HAGBs [76,77,78]. An additional reason for the increase in the strength of the UFG Al–Zr alloy upon annealing is the formation of nanosized Al₃Zr precipitates at grain boundaries, and most likely these two processes are interrelated [76,77]. Relaxation of GBs during annealing was directly observed in situ annealing experiment in a scanning transmission electron microscope [78].

In subsequent studies, a similar plastification effect due to DHT ‘low temperature annealing (AN) + small deformation (0.25 HPT)’ was found in the pre-aged (AG) HPT-processed Al–1.5Cu (wt. %) [73] and Al–1.47Cu–0.34Zr (wt. %) alloys [74]. An example of a significant increase in ductility while maintaining high strength of HPT-processed Al–1.47Cu–0.34Zr (wt. %) alloy is shown in Fig. 10 [74].

It is known that some UFG Al-based alloys demonstrate high ductility and even superplasticity due to the high intensity of GBS processes and, as a result, a high strain rate sensitivity coefficient (Section 2.1). It was shown in Ref. [79] that the strain rate sensitivity coefficient in HPT-processed Al–1.47Cu–0.34Zr (wt. %) alloy before and after DHT remains almost unchanged, which indicates that the GBS does not play a key role in the observed plastification effect (Fig. 11).

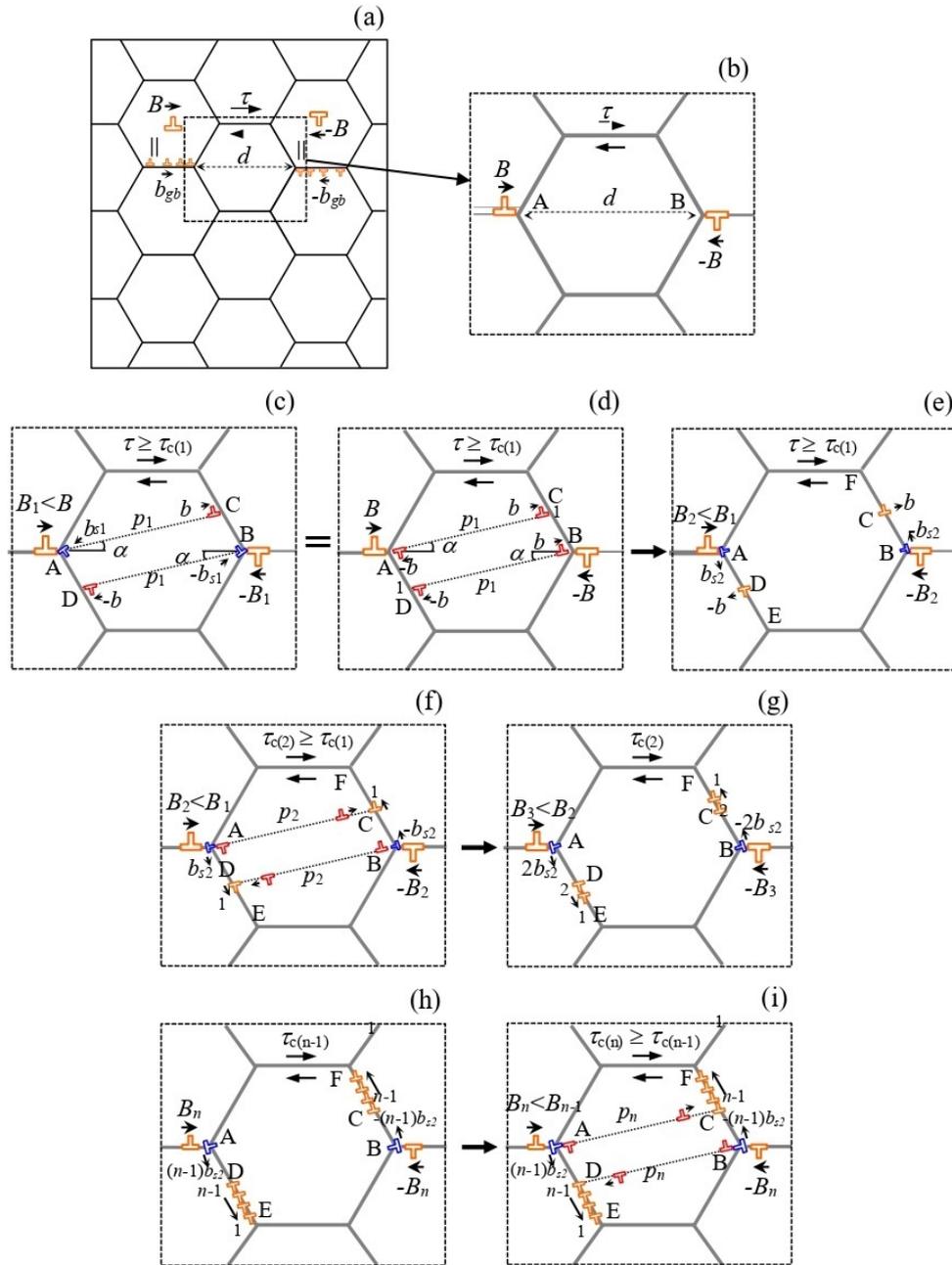


Fig. 8. Model of a micromechanism of the process of a defect structure transformation in HPT-processed UFG material after annealing and subsequent small HPT deformation (schematically): (a) UFG material with two pile-ups of GBDs that are modeled by $\pm B$ -superdislocations (general view), (b) Magnified inset illustrating the initial defect structure, (c–g) Successive emission of pairs of LDs from triple junctions A and B, their capture by the GBs AE and BF, and their climb along these GBs, (h) Defect configuration after the $(n-1)$ th event of the LD emission, (i) Emission of the n th pair of the LDs. Reproduced from Skiba et al. [72], with permission, © 2020 Springer.

On the other hand, an increase in ductility while maintaining a high level of strength is well explained by the above model, developed for the HPT-processed Al [19,74]. According to this model, the increase of plasticity after annealing of the HPT-processed Al–1.47Cu–0.34Zr (wt. %) alloy and additional 0.25 HPT deformation is associated with introducing new dislocations into the relaxed structure of HAGBs. On the other hand, it can be related to more homogeneous distribution of internal

stresses thanks to the different ‘dislocation–segregation/precipitation’ configurations at/near GBs after the additional 0.25 HPT [74]. It should be noted that in both Al–1.5Cu and Al–1.47Cu–0.34Zr (wt. %) alloys structured by the HPT method, although a drastic drop in plasticity occurred after low-temperature annealing, but the AIH effect was not observed [73,74]. The authors of Ref. [74] explain this by counter microstructural changes, such as a slight increase in the average grain size, a decrease in the

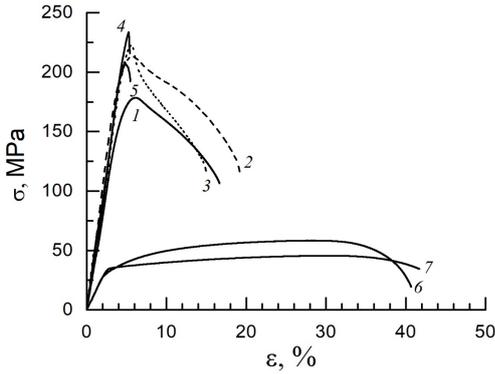


Fig. 9. Stress–strain diagrams of CP Al samples after HPT treatment for 10 revolutions at: (1) RT, after subsequent annealing at temperatures of (2) 363, (3) 403, (4) 423, (5) 473, and (6) 673 K, and also in the initial CG state (7). Reproduced from Mavlyutov et al. [3], with permission, © 2017 Springer.

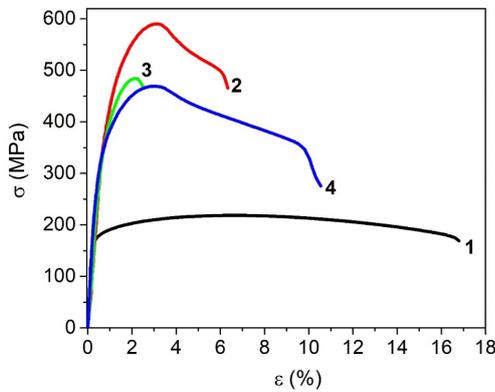


Fig. 10. Stress–strain curves of Al–Cu–Zr alloy in the (1) AG, (2) AG + HPT, (3) AG + HPT + AN, (4) AG + HPT + AN + 0.25HPT states. Reproduced from Orlova et al. [74], with permission, © 2021 Elsevier.

dislocation density, and a change in the parameters (size and amount) of precipitates at GBs, which result in a strength decrease.

3. CONCLUDING REMARKS

The UFG Al-based alloys typically exhibit high mechanical strength and low tensile ductility, which severely limits their practical application. Many efforts of researches were directed on searching the way to improve the ductility of UFG and NC metals and alloys. The approaches to improve tensile ductility are based either on manipulation with testing parameters (temperature, strain rate and others), or on idea of intelligent microstructural design in the UFG materials. This paper overviewed the main approaches to increase ductility while retaining high level of strength in UFG and NC Al-based alloys, with an emphasis on the recent finding and physical reasons of the plasticity improvement.

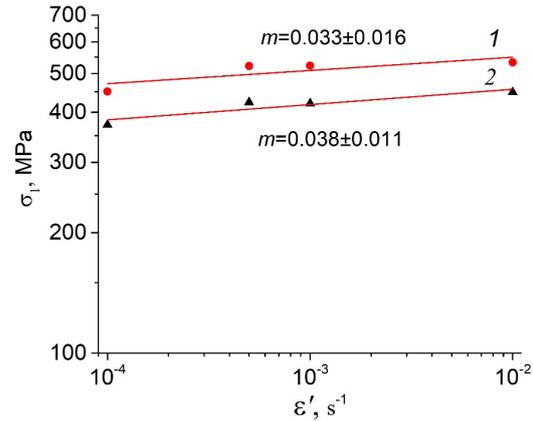


Fig. 11. Dependences of the flow stress at $\varepsilon = 1\%$ on the strain rate in logarithmic coordinates for the Al–1.47Cu–0.34Zr (wt. %) alloy in the states: (1) AG + HPT, (2) AG + HPT + AN + 0.25HPT. Reproduced from Sadykov et al. [79], with permission, © 2022 Ioffe Institute.

We paid much attention to the recently suggested approach of plastification of UFG Al-based alloys, while keeping high strength, through additional deformation-heat treatment ‘low-temperature annealing + small additional deformation’ after SPD processing. Substantial increase of ductility with keeping high strength was achieved for UFG CP Al and Al–0.4Zr (wt. %), as well as for UFG high-strength Al–1.5Cu and Al–1.47Cu–0.34Zr (wt. %) alloys, structured by HPT processing. This most probably points to the versatility of this approach for many other alloys. However, such suggestion requires further experimental research. According to Refs. [19,72], this approach is based on modification of the GB network structure. However, a number of questions remain unanswered:

- influence of the parameters of UFG structure, such as the shape and size of grains, distribution of GBs on misorientation angle, specific structure of GBs (presence of segregation and/or precipitation etc.) on the effect of deformation-induced plastification and the associated AIH effect;
- influence of type and magnitude of additional deformation on the plastification effect;
- temperature range and strain-rate range of manifestation of the plastification effect and key microstructural parameters affecting these ranges.

Despite the developed theoretical model [19,72] explaining quite well the plastification effect in HPT-processed Al [3,19], the physical nature of the ductility enhancement in aluminum alloys after the DHT ‘annealing + deformation’ requires a deeper understanding, and theoretical description, including of such points as influence of specific structural features of grain boundaries (segregations and/or nanoprecipitates) on the effect of

plastification in UFG or NC structures. Such knowledge would contribute to the physical basis of developing alloys with outstanding combination of high strength and high ductility through grain-boundary engineering.

ACKNOWLEDGEMENTS

The work was supported by the Russian Science Foundation (project № 22-19-00292).

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УДК 691.771:539.214:539.4:620.186

Повышенная пластичность высокопрочных ультрамелкозернистых алюминиевых сплавов при комнатной температуре (обзор)

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Аннотация. Дан краткий обзор теоретических моделей, описывающих различные зернограничные преобразования, происходящие в пластически деформированных нанокристаллических материалах. Показана важная роль границ зерен и их преобразований в процессе пластической деформации нанокристаллических материалов. Теоретические результаты обсуждаются и сравниваются с имеющимися данными экспериментальных исследований и компьютерного моделирования.

Ключевые слова: алюминиевые сплавы; ультрамелкозернистая структура; пластичность; прочность