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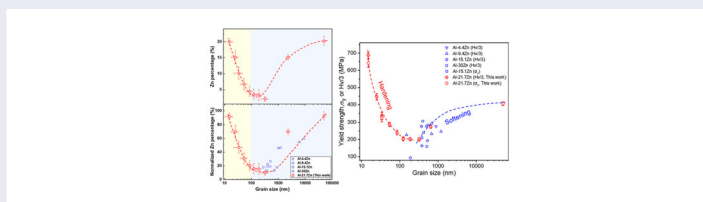
Giant hardening and formation of nanograined supersaturated solid solution in Al–Zn system

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ABSTRACT

Grain refinement via severe plastic deformation (SPD) can induce the decomposition of Al–Zn supersaturated solid solution, resulting in strain softening rather than hardening. So far, it is very challenging to improve the strength of binary Al–Zn alloy by refining grains through SPD. Herein, a single-phase supersaturated solid solution nanostructure with relaxed grain boundaries has been successfully generated in Al–21.7 at% Zn alloy by cryogenic high-pressure torsion. Instead of softening, giant hardening is achieved. The nanocrystalline Al–Zn alloy with grain size of 15 nm has an ultrahigh yield strength of about 642 MPa.



IMPACT STATEMENT

Giant hardening has been achieved in Al–Zn alloy with extremely fine nanograins and single-phased supersaturated solid solution under extremely high shear strain via cryogenic high-pressure torsion.

ARTICLE HISTORY

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KEYWORDS

Al–Zn alloy; high strength; supersaturated solid solution; grain boundary relaxation; phase decomposition

1. Introduction

Metallic materials with excellent mechanical properties are desirable for structural applications. Severe plastic deformation (SPD) has been demonstrated to increase the mechanical properties by introducing a large number of defects in the metallic materials, such as vacancies, dislocations and grain boundaries (GBs) [1–3]. According to the Hall–Petch relationship [4, 5], the strength can be significantly improved when grains are refined into nanoscale [6–8]. Further increase of strength can be achieved by solid solution hardening and precipitation hardening, etc. Due to the intensive interactions between defects and second-phase particles, the solid solubility was found to increase by facilitating the fragmentation and dissolution of the particles to form supersaturated solid solutions [9], increasing the hardening effect of the solid solution. It is not surprising, therefore, that

the coupling effect of GBs hardening and solid solution hardening has been frequently reported in the literature to endow the nanograined (NG) alloys with much high strength in many alloys, such as Al–Zr [10], Al–Ca [11] and Nb–Ti alloy [12].

However, it has been shown that SPD can also lead to softening under large strain [13], especially in alloys with relatively low melting points even if the solute content is high, such as Pb–Sn [14] and Al–Zn [15, 16] alloys. Al–Zn alloy is a typical binary system with low solubility in the equilibrium state at room temperature [17]. Extensive studies [15, 16, 18–21] have shown that the prepared supersaturated Al–Zn solid solution can be decomposed into an ultrafine grained (UFG) two-phase mixture of Zn-rich phase and ‘purified’ Al matrix after SPD processing at room temperature. The complete phase decomposition is generally believed to be attributed to the

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accelerated diffusion of Zn atoms along GBs by vacancies fluxes, as well as enhanced lattice diffusion by dislocations and vacancies [16, 20]. After plastic deformation of Al–Zn alloy, the grain size of Al matrix does decrease with increasing strain and finally reaches a steady level of 300–500 nm, however, the strength is found to decrease with increasing strain [15, 16, 22]. Alhamidi et al. [15] point out that softening by high-pressure torsion (HPT) is mainly due to the destruction of nanosized spinodal structure in the coarse-grained Al–Zn solid solution and the formation of equiaxed submicrometer grains. The periodic structural modulation by spinodal decomposition yields a strong coherent internal stress field, thereby hinders dislocation motion, which is more effective to increase the strength than solid solution hardening in Al–Zn system [15]. Meanwhile, the grain coarsening can occur at room temperature as a result of poor thermal stability and enhanced atomic diffusivity of Al–Zn alloy, which further decreases the strength. Thus, it is very challenging to strengthen the Al–Zn alloy by the traditional SPD methods.

Introduction of GBs with low energy is practically feasible in enhancing the thermal and mechanical stabilities of nanograins and can facilitate grain refinement into nanoscale [23–25]. Our previous studies [26] indicated that deformation-induced autonomous GB relaxation can occur when grains are refined into nanoscale, which makes GBs transform into low energy state as observed in pure Al [27, 28] and Al–Mg alloy [29]. In principle, the relaxed GBs can suppress precipitation at GBs due to enlarged kinetic barriers for nucleation and the retarded diffusion along the GBs [30]. Thus, one may anticipate that decomposition may also be inhibited in Al–Zn alloy by the introduction of low-energy GBs into the supersaturated solid solution nanostructure.

In this work, by using cryogenic HPT at large strain, we successfully refined the grain size into nanoscale and produced considerable amounts of low energy GBs in the alloy, in which hardening rather than softening was found with decreasing grain size in nanograins.

2. Experimental

In brief, disc-shaped Al–21.7Zn (in at%) alloy was deformed by high-pressure torsion (HPT) at liquid nitrogen temperature. Microstructures were characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD), orientation imaging microscopy (OIM) and high-angle annular dark-field scanning transmission electron microscope (HAADF-STEM). Mechanical properties were determined using micropillar compressive test and microhardness test.

More information on experimental can be found in the Supplemental material.

3. Results and discussion

3.1. Microstructure evolution under plastic strain

The initial microstructure of the CG Al–Zn alloy contains equiaxed grains of average size of about 50 μm with random orientation (Figure S1a). TEM observations and EDS results (Figure S1b,c) indicated the formation of supersaturated solid solution of matrix, with an actual Zn concentration of 20.3 at% (Figure S1d). Notably, Zn atoms were found to be unevenly distributed inside the α -Al FCC lattice in the form of structural modulations with the size of 1–2 nm (Figure S1e,f), a typical feature for spinodal decomposition at the very early stage [31, 32].

Typical two-phase UFG structure (UFG-320, Figure 1(a,b)) was introduced after HPT deformation for 0.5 revolutions (corresponds to an equivalent strain of 3.8). It exhibited α -Al grains of 320 nm, and Zn-rich phases located both at GBs and triple junctions of α -Al grains (mean size of 180 nm) and in the grain interior (diameters below 30 nm, Figure 1(c)). Elemental mapping (Figure 1(c)) revealed the depletion of Zn in the α -Al grain interior, with Zn concentration of ~ 2 at% (Figure 1(d)), much lower than the average Zn content in the CG and fully consistent with those reported in the literature [16, 20].

With further deformation to the equivalent strain of 100 (total revolution of 28), an equiaxed nanograin structure with random orientations and a grain size of 15 nm is formed (sample NG-15), as shown in Figure 1(e,f). The extreme grain refinement is mainly induced by the combination of low temperature and large strain. The low deformation temperature can effectively suppress the annihilation of dislocations and increase the accumulated dislocation density, thereby facilitates grain refinement [33]. It can be seen that, compared with conventional SPD methods, cryogenic HPT deformation employed in this work is more efficient in accelerating grain refinement with comparable equivalent strain (Figure S2).

Interestingly, selected area electron diffraction (SAED) pattern (insert of Figure 1(e)) identified only a single-phase FCC α -Al structure. EDS analysis in Figure 1(g) revealed that Zn atoms are homogeneously distributed in the α -Al grains with an average Zn concentration of ~ 20 at% (Figure 1(h)), identical to the initial CG. The compositional analysis and SAED pattern verified the formation of a supersaturated solid solution in the extremely fine nanograin structure. A relatively high fraction of Σ CSL GBs ($\sim 26\%$) were detected in the NG-15

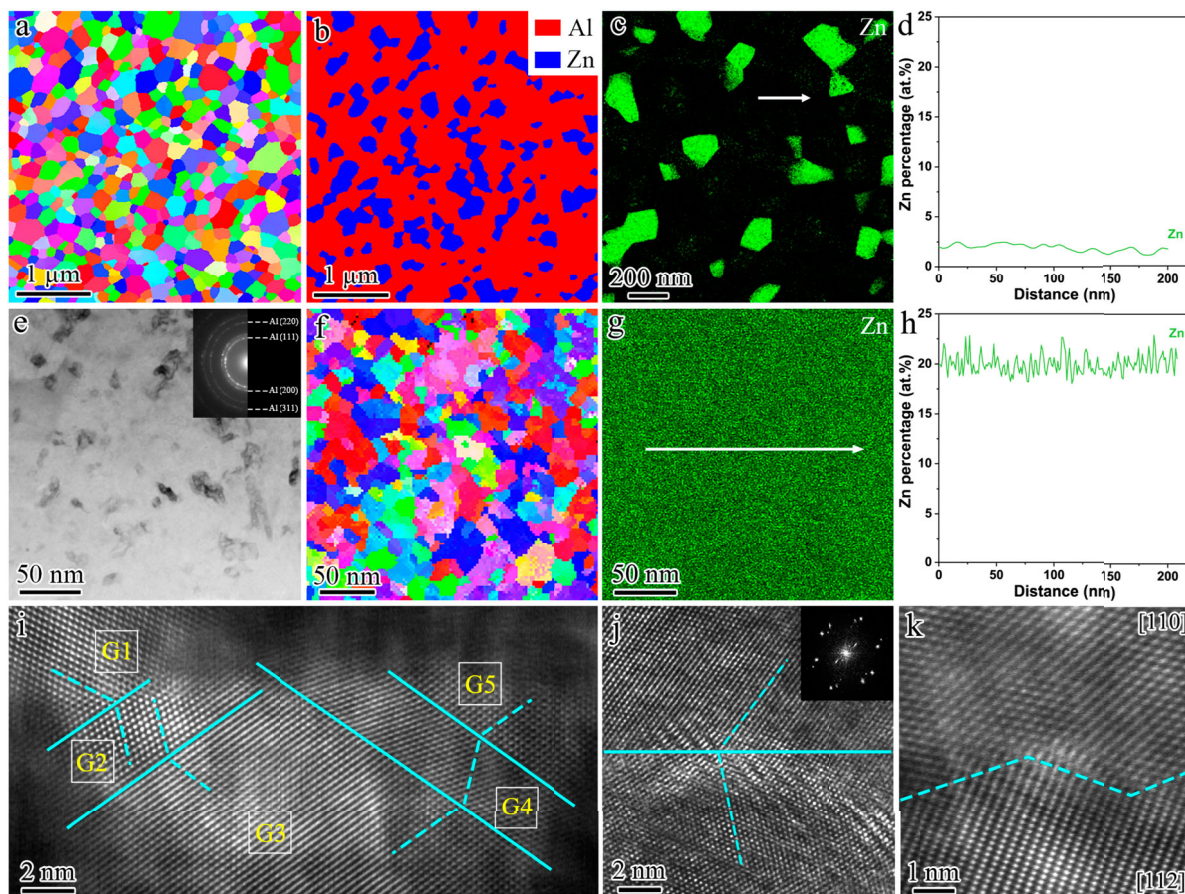


Figure 1. Characterizations of LNT-HPT processed UFG-320 (a–d) and NG-15 (e–k) samples: (a) IPF map and (b) the corresponding phase map, (c) The Zn elemental mapping and (d) line-scan of Zn concentration along the white arrow in (c). (e) A bright field TEM image and corresponding SAED pattern (inserted). (f) IPF map. (g) The Zn elemental mapping and (h) line-scan of Zn concentration along the white arrow in (g). (i–k) High resolution TEM image of GB structures in NG-15 sample.

sample by statistical PED analysis of GB characteristics, among which the fraction of $\Sigma 3$ boundaries was found to be about 7.5%. Under high-resolution TEM investigations, GBs with ordered configurations can be frequently observed. A typical image is shown in Figure 1(i), where multiple $\Sigma 3$ boundaries are observed. In addition, a CSL $\Sigma 11$ boundary (Figure 1(j)) and a faceted GB between [110] and [112] zone axis (Figure 1(k)) is clearly seen. Such an ordered GB structure signifies that the GBs are relaxed into a low excess energy state under heavy deformation [26–29]. As grain size refined below a critical value, the dominated deformation mechanism would shift from multiplication of full dislocations to activation of partial dislocations. GB relaxation is then induced by the dissociation of GBs through the emission of partial dislocations and stacking faults from GBs, leading to the atom rearrangement in the vicinity of GB region and a reduction in GB energy [26]. As a result, GB relaxation is stimulated at large equivalent strain in cryogenic HPT processing, with the formation of equiaxed nanograins and GBs with low energy.

3.2. Formation of supersaturated solid solution with extremely grain refinement

To further verify the formation of supersaturated solid solution with nanograin structure, we also performed quantitative XRD analysis (Figure 2(a)). For comparison, we additionally prepared another nanograin structure with the grain size of 34 nm under an equivalent strain of 69 (NG-34, Figure S3), in which atomic-level faceted interface and partial dissolution of Zn-rich phases (~ 10 at% Zn in α -Al) were detected. The diffraction peaks for Zn can hardly be detected in the CG sample, clearly shows that Zn atoms are fully dissolved in Al lattice thus a supersaturated state has been attained. Zn peaks with high intensity appeared in the UFG structure upon deformation while their relative intensities continuously decreased with increasing strain until completely vanished in the NG-15 structure. In Figure S4, the peak position of α -Al (111) shifted to lower angle in the UFG structure with respect to CG. However, it gradually shifted back to higher angle in the nanograin samples. The peak positions of α -Al in the NG-15 are the same as that

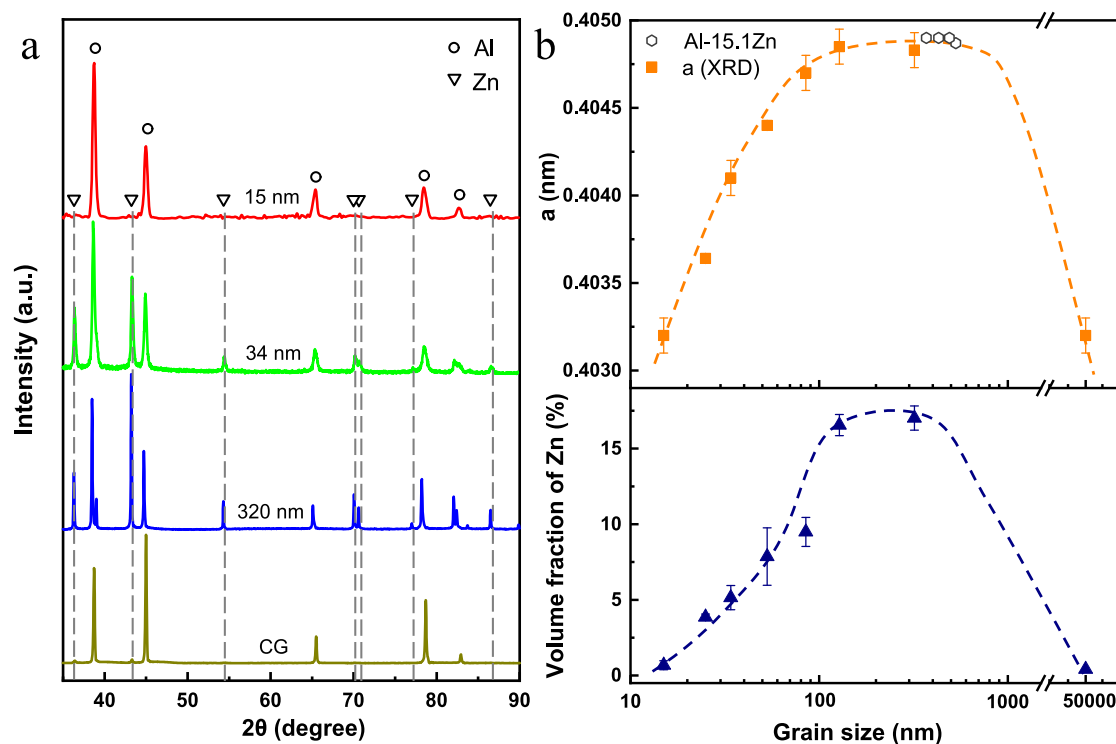


Figure 2. (a) XRD patterns of the CG and as-deformed Al–21.7Zn alloy. (b) Variation of lattice parameter (upper stack) and volume fraction of Zn-rich phase (lower stack) with grain size in the as-deformed Al–21.7Zn alloy. Data collected from Al–15.1Zn alloy processed by means of severe plastic deformation are included for comparison [20].

of the original CG sample, indicating that NG-15 alloy is a single-phase supersaturated solid solution.

We systematically calculated the lattice parameter of α -Al matrix and the volume fraction of Zn-rich phase from XRD patterns. For the grain size of α -Al at sub-micron, the lattice parameter *a* increases rapidly from 0.4032 nm to 0.4048 nm (Figure 2(b)), approaches to that of pure Al (0.4049 nm). With the further decrease of grain size, lattice parameter begins to decrease and finally to a value of 0.4032 nm at grain size of 15 nm, which identical to that of the original CG (0.4032 nm). The volume fraction of Zn-rich phase initially increases to 17 vol % and then decreases with the decrease of grain size, in accordance with that of lattice parameter. Summarizing the Zn concentration inside α -Al grains as a function of the grain size and further compared with other Al–Zn alloys with different Zn concentrations (converted into at%) processed by means of traditional SPD [16, 20, 34–38] after normalization (Figure 3). We found the Zn concentration inside α -Al grains distributed collectively within 2–4 at% in the grain size span of 100–1000 nm. Noting that this value is higher than the equilibrium solubility of Zn in Al at room temperature (below 1 at%), indicating that little amounts of Zn-rich phases have dissolved into the α -Al grains at larger strain after their initial precipitation upon SPD.

In principle, the dissolution of second-phase particles after deformation is mainly attributed to their intensive interaction with defects [12, 39–42]. The multiple dislocation glide events across interfaces would transport tiny and high-energy particles (which eventually dissolve through the Gibbs–Thomson effect) [42] and/or atoms of these particles into the matrix phase [43]. In the present work, the low enthalpy of mixing in Al–Zn system (7.8 kJ/mol [44]) does make it possible to form single-phase supersaturated structure under large plastic deformation, as suggested by simulation [45] and experiments [46]. However, due to the low recovery and recrystallization temperature of Al–Zn alloy, significant structural recovery would take place after deformation and consequently causing a reduction of defects, which in turn makes it hard to induce full dissolution of Zn-rich phases. This idea is verified in previous reports that two-phase UFG structure is still retained even for equivalent strain exceeds 100 in Al–Zn alloys [15, 20].

The formation of single-phase nanograined supersaturated solid solution can be mainly attributed to two factors, namely high accumulated dislocation density by cryogenic deformation under large strain and the strain-induced autonomous GB relaxation. The employed low deformation temperature and large strain can remarkably increase the net dislocation density as evidenced in NG-34 and NG-15 samples (Table 1). In this case,

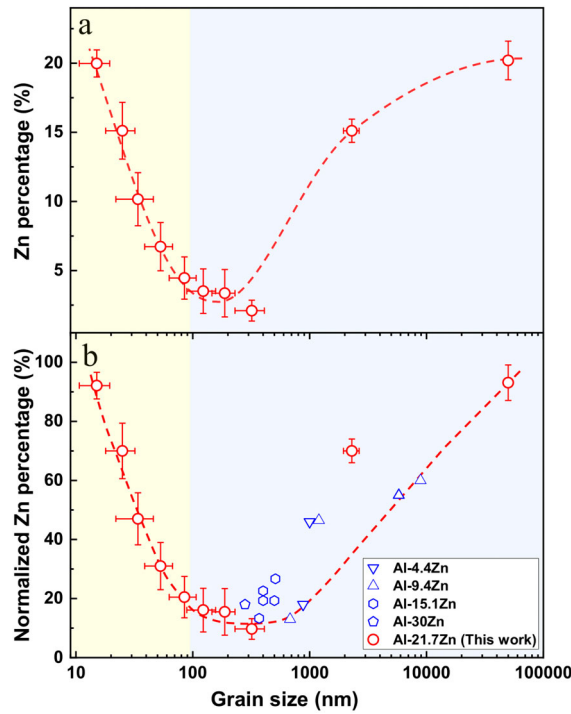


Figure 3. Variation of (a) measured and (b) normalized Zn percentage inside α -Al grains as a function of grain size of the as-deformed Al-21.7 Zn alloy. Data collected from Al-Zn alloy with different Zn concentrations processed by means of severe plastic deformation are included for comparison (Al-4.4 Zn [20, 37], Al-9.4 Zn [20, 36], Al-15.1 Zn [20, 34, 37, 38, 48] and Al-30 Zn [35]).

Table 1. Parameters of microstructure in the cryogenic HPT deformed Al-Zn samples obtained by XRD spectrum analysis using TOPAS software.

Samples	d_{TEM} (nm)	d_{XRD} (nm)	Microstrain (%)	Dislocation density (m^{-2})
UFG-320	320	223	0.0626	3.4×10^{13}
NG-34	34	41	0.4668	1.38×10^{15}
NG-15	15	20	0.5057	3.06×10^{15}

the interaction between dislocations and Zn-rich phase would be significantly intensified (Figure S5), which promotes the dissolution of Zn-rich phase.

More importantly, the formation of low-energy GBs after relaxation plays a crucial role. The lowered GB energy significantly elevates the energy barrier for precipitate nucleation, thus the precipitation at those relaxed boundaries is thermodynamically unfavorable or even suppressed. Telang et al. [47] reported that low Σ CSL boundaries were more resistant to the precipitation of carbides in 600 alloy owing to their low boundary energy and low boundary curvature. With the aid of relaxed GBs, the supersaturation of Zn atoms in the α -Al matrix from dissolved Zn-rich phase can be stabilized from precipitation. In the case where the precipitation is completely

inhibited, a supersaturated single-phase solid solution is eventually formed (Figure 1).

3.3. Softening in UFG and giant hardening of NG

Figure 4(a) shows the compressive engineering stress-strain curves. Evidently, the mechanical property of four types of samples is highly dependent on the initial grain size and the microstructure. Specifically, CG pillar has a high yield strength of ~ 406 MPa. However, the UFG-320 sample yields at only 204 MPa followed by plateau, indicates the occurrence of GB-mediated deformation process [34]. In contrast, the yield strength of the NG-34 sample increases to 324 MPa and further reaches 642 MPa in the NG-15 sample.

SEM images of the micropillars before and after compression are represented in Figure 4(b–e). After compression, slip traces can be clearly seen on the surface of CG pillar (Figure 4(b2)) indicating dislocation slipping-dominated deformation. As for UFG-320 sample, individual grains were observed to protrude from the pillar surface (Figure 4(c2)), suggesting the occurrence of GB sliding and in concert with literature results [34, 35, 48–51]. For both NG-34 and NG-15 samples, non-uniform deformations were observed (Figure 4(d2 and e2)), indicating that dislocation-mediated deformation exists in NG samples.

The yield strength of deformed Al-Zn alloy with respect to grain size was plotted in Figure 5. At the early stage of HPT deformation, the strength decreases significantly from 406 MPa to about 204 MPa, which is consistent with the reported strain softening in literature [15, 16]. The structure modulation in CG (Figure S1e,f) could significantly hinders dislocation motion thus offers better hardening effects [31] than solid solution hardening as its effect is weak in Al-Zn alloy [15]. The observed strain softening can be attributed to the disappearance of modulated structure after deformation and formation of equiaxed Al and Zn grains [15]. Although grain refinement can make some contribution to hardening following the Hall-Petch effect, strain-induced grain refinement provides limited hardening due to the low Hall-Petch slope [52] and the relatively large grain size (300–500 nm) of α -Al matrix (Figure 1(a,b)). The overall effect is softening.

With further decreasing of grain size, instead of displaying the softening effect, the strength begins to increase and finally reaches 642 MPa at 15 nm. The hardening of NG is quantitatively estimated based on the following general equation [10, 53]:

$$\sigma_y = \sigma_0 + \sigma_{ss} + \sigma_{GB}, \quad (1)$$

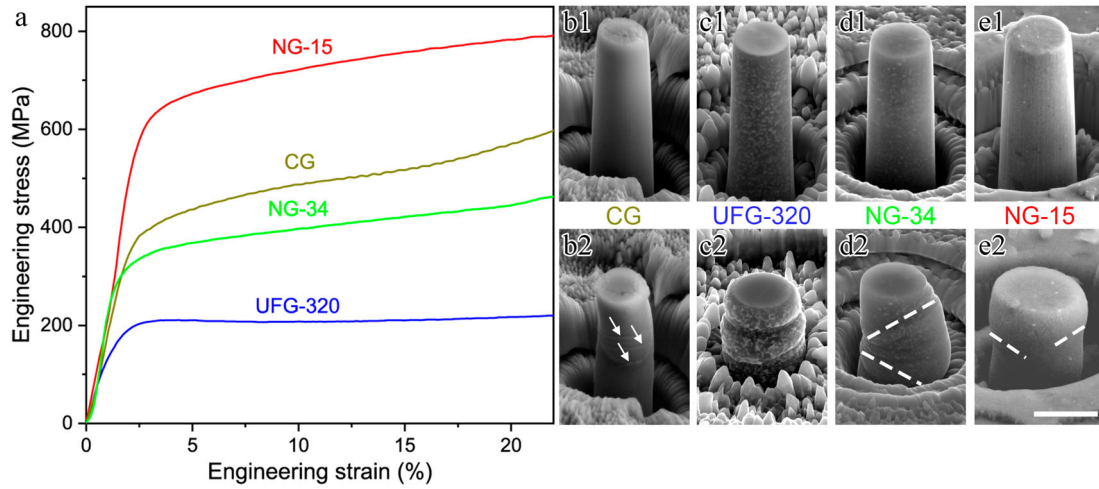


Figure 4. (a) Engineering stress-strain curve of CG and as-deformed Al-21.7Zn alloy (as indicated). SEM image of CG (b1–b2), UFG-320 (c1–c2), NG-34 (d1–d2) and NG-15 (e1–e2) micropillar before and after compression, respectively (image tilt angle 52°). Scale bar, 5 μm .

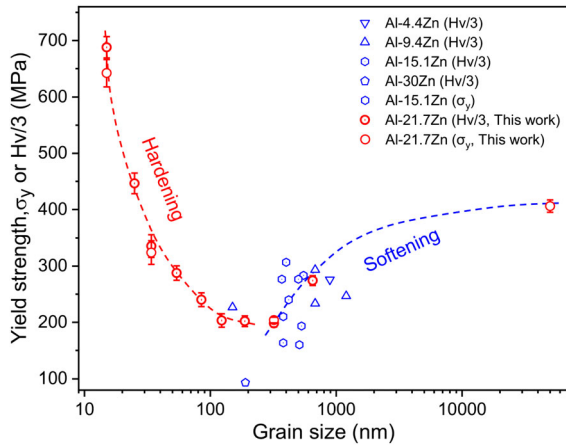


Figure 5. Plot of yield strength as a function of the grain size of the nanostructures Al-21.7Zn alloy. Data collected from Al-Zn alloy with different Zn concentrations processed by means of severe plastic deformation are included for comparison (Al-4.4Zn [16], Al-9.4Zn [16, 36], Al-15.1Zn [16, 18, 22, 48, 51] and Al-30Zn [15]).

where σ_y is the yield strength obtained from micropillar compression, σ_0 is the Peierls–Nabarro stress in pure Al (taken as 5.5 MPa [54]). σ_{ss} and σ_{GB} are the hardening contribution from solid solution and GBs, respectively.

The solid solution hardening effect can be expressed as [55]:

$$\sigma_{ss} = AC_0^{2/3}, \quad (2)$$

where A is a constant (3.085 for Zn in Al [55]) and C_0 is the concentration of solute in weight percent. Using the detected Zn concentration in the α -Al grains of 20 at % (37.6 wt%) for NG-15 sample, the solid solution hardening (σ_{ss}) is estimated to be 35 MPa according to Eq.(2).

The strength contribution arising only from GBs (σ_{GB}) can be therefore calculated through the total strength subtracts hardening from other mechanisms that calculated above, $\sigma_{GB} = \sigma_y - \sigma_0 - \sigma_{ss}$. The calculated σ_{GB} for NG-15 sample is about 601.5 MPa. Based on the classical Hall–Petch relation ($\sigma_{GB} = k_y d^{-1/2}$) [4, 5], the corresponding Hall–Petch slope (k_y) can be estimated as 0.074 MPa $\text{m}^{1/2}$. Apparently, the Hall–Petch slope in NG-15 sample is much higher than that of pure Al (0.04 MPa $\text{m}^{1/2}$ [52]), suggesting a stronger resistance to dislocation glide for relaxed GBs.

4. Conclusion

In summary, a single-phase Zn-rich supersaturated solid solution with extremely fine nanograin was formed in Al-21.7 at% Zn alloy utilizing cryogenic HPT deformation. Deformation-induced GB relaxation as well as significant increase in dislocation density were responsible for the formation of single-phased nanograined structure. In this way, giant hardening is achieved with the yield strength of 642 MPa, about 438 MPa higher than that of the ultrafine-grained counterpart and overcoming traditional strain softening of the Al–Zn alloy. Our results indicate that the application of extreme large strain at cryogenic condition is an effective strategy for stabilizing supersaturated solid solution nanostructures in Al–Zn alloy and other phase decomposition alloys in order to obtain ultrahigh strength.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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