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ARTICLE

Uniform Elongation and Yield-Drop Phenomenon in Magnetically Annealed 1050 Aluminum Alloy Prepared by CryoECAP

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Abstract: The ultrafine grained (UFG) 1050 aluminum alloy was prepared by equal channel angular pressing at cryogenic temperature, namely cryoECAP process. The tensile behavior and microstructures of UFG 1050 aluminum alloy after annealing at 90–210 °C for 4 h without and with high magnetic field of 12 T were investigated by tensile tests, transmission electron microscope, and electron backscattered diffraction analyses. After cryoECAP and annealing treatments, the 1050 aluminum alloy has ultrafine grains with 0.7–1.28 μm in size, the ratio of ultimate tensile strength to yield strength is less than 1.24, and the uniform elongation is less than 2.3%. With increasing the annealing temperature from 90 °C to 210 °C, the yield-drop phenomenon becomes more obvious due to the decrease in mobile dislocations to maintain the applied strain rate during tensile deformation. The uniform elongation decreases from 1.55% to 0.55%, the dislocation density reduces from $5.6 \times 10^{14} \text{ m}^{-2}$ to $4.2 \times 10^{13} \text{ m}^{-2}$, and the fraction of high-angle grain boundaries (HABs) increases from 63.8% to 70.8%. These phenomena cause the higher annihilation rate of dislocations, thereby leading to the degradation of strain hardening effect. During annealing under high magnetic field at 90–210 °C, the low fraction of HABs (61.7%–66.2%) can provide a slower annihilation rate of dislocations, therefore resulting in the higher uniform elongation (0.64%–1.60%) and slower decrease in the flow stress after the yield peak.

Key words: magnetic annealing; ultrafine grain; uniform elongation; yield-drop phenomenon; high-angle grain boundaries; dislocations

Ultrafine grained (UFG) materials attract much attention due to their excellent mechanical properties, compared with those of the traditional coarse-grained materials^[1–4]. However, the yield-drop phenomenon and the limited uniform elongation can be observed in the tensile stress-strain curves of UFG materials^[5–9], which are completely different from those in the coarse-grained materials.

The uniform elongation indicates the plastic strain before the occurrence of plastic instability or localized deformation during tensile test, which can reflect the deformation stability. It is reported that the uniform elongation in the nanocrystalline and UFG materials (<3%) is much smaller than that in the coarse-grained materials^[10], which results from the plastic instability (necking) at the early stage of tensile

deformation^[8]. It is also found that the limited uniform elongation in UFG aluminum is influenced by grain size, temperature, and strain rate^[5,11–13]. Tsuji et al^[5] found that the uniform elongation suddenly drops to several percent when the grain size is about 1 μm in the AA1100 aluminum alloy and interstitial-free steel after accumulative roll-bonding processing and annealing treatments. Sun et al^[11] found that the AA1050 aluminum alloy after equal channel angular pressing (ECAP) deformation by route A has the grains with average size of 0.55 μm and high-angle grain boundaries (HABs) accounting for approximately 67%, whereas that deformed by route C has grains with average size of 0.64 μm and HABs accounting for approximately 38%. The alloys after deformation by route A and route C have the limited

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uniform elongation < 2.5% after tensile tests at 25 °C, whereas those after tensile tests at -196 °C have high uniform elongation exceeding 18%. Moreover, the uniform elongation of the alloys deformed by route C is about 23% after tensile tests at strain rate of 1 s⁻¹ and temperature of -196 °C, which is higher than that deformed by route A (about 18%).

The occurrence of yield-drop phenomenon (flow stress drops sharply after yielding) can be observed in the tensile curves of UFG AA1050 aluminum alloy^[7,14], AA1100 aluminum alloy^[5], aluminum alloy with high purity^[15], Al-0.3wt% Mg alloy^[16], and interstitial-free steel^[5]. Huang et al^[14] found that the yield-drop phenomenon does not occur in the ECAPed AA1050 aluminum alloy with grain size of 0.35 μm, but it happens in the alloys with grain size of 0.47–1.03 μm after annealing at 200–250 °C for 1–8 h. Yu et al^[17] reported that the yield-drop phenomenon has a close relationship with tensile temperature for the AA1050 aluminum alloy after ECAP and annealing treatments. The yield-drop phenomenon occurs under the conditions of grain size < 1 μm and tensile temperature = 25 °C, and it also occurs under the conditions of grain size < 0.59 μm and tensile temperature = -196 °C. It is suggested that the occurrence of yield-drop phenomenon in UFG materials may result from the lack of mobile dislocations. However, the evolution of yield-drop phenomenon is rarely reported through the perspective of dislocation variation in UFG materials with different grain sizes. In this research, the evolution of dislocations in UFG 1050 aluminum alloy at the yield peak state during tensile deformation was analyzed.

The magnetic field has great influence on the motions of dislocations and grain boundaries, which are also the fundamental processes of recovery and recrystallization^[18–21]. Bhaumik^[18] and Molodov^[19] et al found that the high magnetic field can enhance the mobility of dislocations, and accelerate the recovery and recrystallization processes. Harada et al^[20] investigated the grain structure of nanocrystalline Ni during annealing at 300 °C from 120 s to 1800 s under a direct-current magnetic field of 1.2 MA/m. It is found that the magnetic annealing enhances the grain growth at the early stage of annealing and finally leads to the homogeneous grain structure. According to Ref.[22], the high magnetic field of 12 T can slightly promote the grain growth of UFG 1050 aluminum alloy during annealing at 150–250 °C for 1 h, and suppress the formation of HABs. In recent years, the effects of annealing with external field on the microstructures and tensile properties of UFG aluminum alloys have been widely researched, including the effect of direct-current annealing on the mechanical properties^[23] and yield strength^[24] of UFG 1050 aluminum alloy and the relationship between dislocation and vacancy cluster with yield strength under magnetic annealing^[25]. However, the unique tensile behavior of UFG aluminum alloys under the magnetic annealing is still obscure. In this research, the UFG 1050 aluminum alloys with different grain structures were obtained by annealing at 90–210 °C for 4 h without and with high magnetic field of 12 T, and the effects of grain structures on the tensile instability were investigated.

1 Experiment

UFG 1050 aluminum alloy was prepared by ECAP for 8 passes through route B_c at cryogenic temperature, namely cryoECAP process. The specimens were soaked in liquid nitrogen at -196 °C for 20 min, and then transferred to equal channel angular extrusion abrasives for extrusion within 15 s. Afterwards, the cryoECAPed specimens were annealed at 90–210 °C for 4 h without and with high magnetic field of 12 T. The detailed cryoECAP procedure and the high magnetic apparatus were based the description in Ref.[22]. Three kinds of specimens were prepared in this research: the initial specimens only after cryoECAP process (cryoECAP specimens), the specimens after cryoECAP and annealing without high magnetic field (normally annealed specimens), and the specimens after cryoECAP and annealing with high magnetic field (magnetically annealed specimens).

The tensile specimens with gauge dimension of 20 mm×6 mm×2 mm were cut from the longitudinal section of the cryoECAP, normally annealed, and magnetically annealed specimens. Tensile tests were conducted on Shimadzu AG-X universal testing machine at room temperature. The initial strain rate of 5×10⁻⁴ s⁻¹ was used for all tests. Each tensile property was tested four times under different conditions. The specimen with coarse grain size of 28 μm was also tested for reference, and it was called as the coarse-grained specimen. The tensile fracture surfaces were observed by SSX-550 scanning electronic microscope (SEM).

The grain size and boundary misorientation angle in the cryoECAP, normally annealed, and magnetically annealed specimens were examined by LEO 1530 FE-SEM with Oxford-INCA electron backscattered diffractometer (EBSD). EBSD specimens were cut from the longitudinal section of the normally annealed and magnetically annealed specimens (plane Z). The specimen structures were characterized by FEI TECNAI G² 20 type transmission electron microscope (TEM). Thin foils were parallel to plane Z. Experiment parameters for preparation of EBSD and TEM specimens were described in Ref.[22]. The microstrains in specimens were examined on plane Z by X-ray diffraction (XRD, PW3040/60X) analysis with Cu Kα radiation. Then, the dislocation density ρ could be calculated by Eq.(1)^[26], as follows:

$$\rho = 14.4 \frac{e^2}{b^2} \quad (1)$$

where e is the microstrain and b is the value of Burgers vector (0.286 nm for aluminum).

2 Results

2.1 Tensile behavior

The engineering stress-engineering strain curves of the coarse-grained, cryoECAP, normally annealed, and magnetically annealed specimens are shown in Fig. 1. It can be seen that the engineering stress-engineering strain curve of the coarse-grained specimen can be divided into four stages (stage A–D in greenish yellow curve). Stage A: a sharp increase in flow stress at the early stage of tensile test ($\epsilon < 0.15\%$); stage

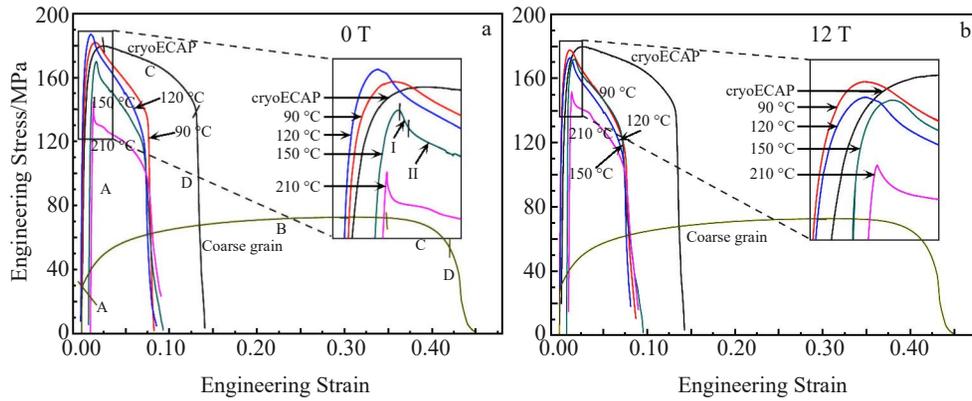


Fig.1 Comparison of engineering stress-engineering strain curves between the specimens after annealing without (a) and with (b) magnetic field of 12 T

B: a long period of strain hardening ($\epsilon=0.15\%–35\%$); stage C: strain softening ($\epsilon=35\%–42\%$); stage D: a sharp decrease in flow stress until fracture ($\epsilon>42\%$). The engineering stress-engineering strain curve of cryoECAP specimen shows distinct characteristics from those of the coarse-grained specimen. Stage A: the first stage is $\epsilon<0.24\%$; stage B: the second stage reduces sharply at $\epsilon=0.24\%–2.3\%$; stage C: the strain softening stage is obvious after the ultimate tensile strength appears at $\epsilon=2.3\%–12.8\%$; stage D: the fourth stage is at $\epsilon>12.8\%$. Compared with that of cryoECAP specimen, the flow stress of normally annealed and magnetically annealed specimens at 90 °C falls faster at the strain softening stage.

It is worth noting that the unexpected yield-drop phenomenon occurs in the specimens after normal and magnetic annealing at 120–210 °C, i.e., the decreasing rate of flow stress for the specimens normally/magnetically annealed at 120–210 °C after the yield peak is much faster than that annealed at 90 °C. According to the enlarged figure in Fig. 1a, it can be seen that the strain softening stage of the specimens after normal and magnetic annealing at 120–210 °C consists of two cases: the fast decrease stage (I) and the slow decrease stage (II) of flow stress. Compared with the cryoECAP specimen, the as-annealed specimens have shorter strain softening stage and present quicker decrease in flow stress after the yield peak. Additionally, the flow stress after yield

peak of the specimens treated by magnetic annealing at 120–210 °C decreases slower than that treated by normal annealing at 120–210 °C does (Fig. 1b).

In this research, the uniform elongation is defined as the engineering strain from initial stage to the yield peak of the tensile engineering stress-engineering strain curve; the total elongation is the engineering strain from initial stage to fracture. The relationships between annealing temperature and uniform elongation as well as total elongation in the normally annealed and magnetically annealed specimens are shown in Fig. 2. The uniform elongation of cryoECAP specimen is 2.3% (Fig. 2a), which is 93.5% lower than that of the coarse-grained specimen (35%). The uniform elongation of the specimen after normal annealing at 90 °C is 1.55%, and it is further decreased with increasing the annealing temperature. The uniform elongation of the specimen after normal annealing at 210 °C is only 0.55%, which is 76% lower than that of the cryoECAP specimen. The uniform elongation of the specimens after magnetic annealing at 90–210 °C is 0.64%–1.60%, which is larger than that of the normally annealed specimen at the same temperature. The total elongation of cryoECAP specimen is 13.4%, and it decreases significantly to 8.3% for the specimen after normal annealing at 90 °C. The total elongation of specimens after normal annealing at 120–210 °C changes slightly (Fig. 2b). The total elongation of the

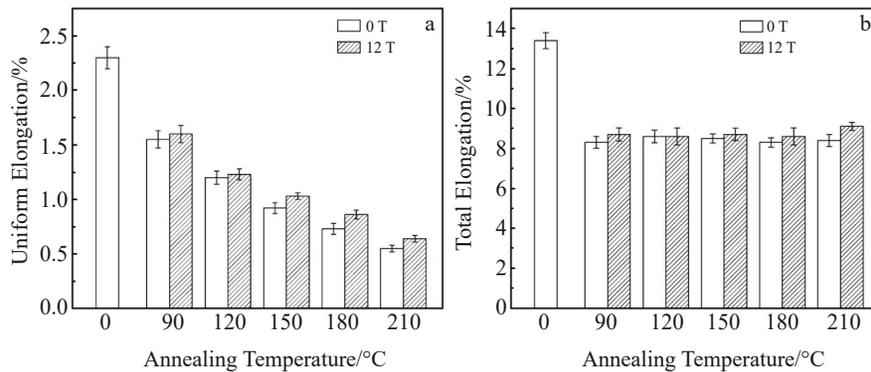


Fig.2 Uniform elongation (a) and total elongation (b) of cryoECAP, normally annealed, and magnetically annealed specimens

magnetically annealed specimens is 8.6%–9.1%, which is slightly larger than that of the normally annealed specimens at the same temperature.

As shown in Fig. 1a, the coarse-grained specimen has the highest ratio of ultimate tensile strength to yield strength (2.37), exhibiting continuous strain hardening behavior after yielding. The cryoECAP specimen shows a limited strain hardening stage, and its ratio of ultimate tensile strength to yield strength is 1.24. The changes in the ratio of ultimate tensile strength to yield strength of specimens after normal and magnetic annealing at different temperatures are displayed in Fig. 3. It can be seen that the ratio of ultimate tensile strength to yield strength of specimen after normal annealing at 90 °C is 1.17, and it is gradually decreased with increasing the temperature. The ratio of ultimate tensile strength to yield strength of specimen after normal annealing at 210 °C is 1.0, indicating that there is no strain hardening effect after yielding in this specimen. Compared with that of the normally annealed specimens, the ratio of ultimate tensile strength to yield strength of specimens after magnetic annealing at 90–210 °C is larger (1.06%–1.18%).

2.2 Microstructure

The grains in cryoECAP specimen are composed of elongated and equiaxed ones. The dislocation tangle phenomenon is obvious and the microstructure is inhomogeneous, as shown in Fig. 4a. With increasing the temperature from 90 °C to 210 °C, the dislocations migrate from grain interior to grain and/or subgrain boundaries. Then, the dislocations pile up at the grain and/or subgrain boundaries and become ordered gradually, thereby decreasing the dislocation density. Moreover, the number of equiaxed grains and the grain size are increased slightly with increasing the temperature. The grain microstructure of the specimen after normal annealing at 210 °C is completely transformed into the equiaxed grain microstructure, so the dislocation density decreases significantly. Most grain boundaries are well-defined, presenting the grain boundary fringes. With the application of high magnetic field, the dislocations pile up at the grain and/or

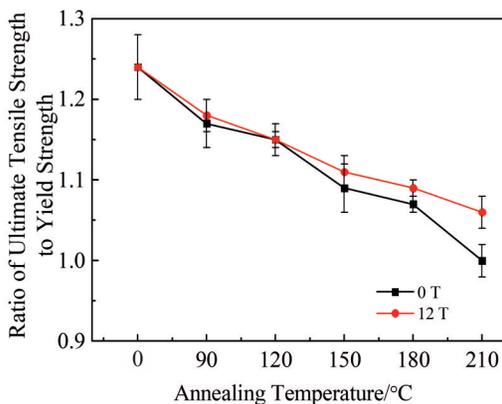


Fig.3 Ratio of ultimate tensile strength to yield strength of cryoECAP, normally annealed, and magnetically annealed specimens

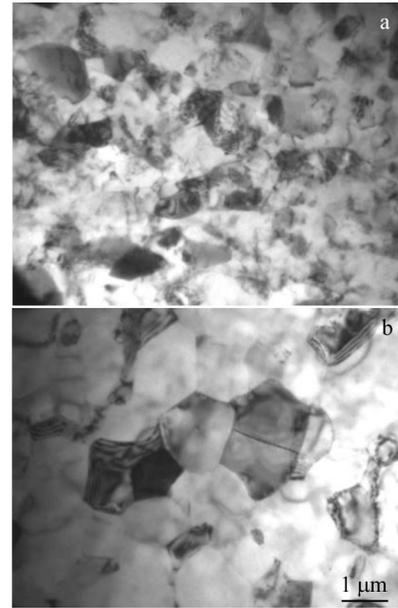


Fig.4 TEM microstructures of cryoECAP specimen (a) and normally annealed specimen at 210 °C (b)

subgrain boundaries with even more ordered structure, and more subgrain boundaries with clear figure are formed^[22].

The structure parameters of the cryoECAP, normally annealed, and magnetically annealed specimens are listed in Table 1. It can be seen that the average grain size is 0.7 μm in the cryoECAP specimen, and it is increased slightly from 0.81 μm to 1.28 μm for the normally annealed specimens with increasing the temperature from 90 °C to 210 °C. The average grain size is slightly larger for the specimens after magnetic annealing at 90–150 °C, and it is similar to that after normal annealing at 210 °C. The grain boundaries with misorientation angle larger than 15° are considered as HABs, and those with misorientation angle smaller than 15° are regarded as low-angle grain boundaries (LABs). The fraction of HABs is 62.3% for the cryoECAP specimen, and it is increased from 63.8% to 70.8% for the normally annealed specimens with increasing the temperature from 90 °C to 210 °C. The fraction of HABs for the magnetically annealed specimens (61.7%–66.2%) is lower than that for the normally annealed specimens at the same temperature. The dislocation density is $7.2 \times 10^{14} \text{ m}^{-2}$ for the cryoECAP specimen, and it is decreased from $5.6 \times 10^{14} \text{ m}^{-2}$ to $4.2 \times 10^{13} \text{ m}^{-2}$ for the normally annealed specimens with increasing the annealing temperature from 90 °C to 210 °C. As for the magnetically annealed specimens, the dislocation density is lower (3.4×10^{13} – $4.3 \times 10^{14} \text{ m}^{-2}$), compared with that of the specimens after normal annealing at the same temperature.

Fig.5 shows SEM fracture surfaces of different specimens after tensile tests. Almost no necking phenomenon occurs in the cryoECAP specimen during the tensile deformation, as shown in Fig.5a. Numerous microvoids with similar sizes and uniform depth distribution exist throughout the whole fracture surface, as shown in Fig.5b. With increasing the annealing

Table 1 Structure parameters of UFG 1050 aluminum alloys after different treatments

Specimen	Condition	Average grain size/ μm	HAB fraction/%	Dislocation density/ m^{-2}
cryoECAP	cryoECAP	0.70	62.3	7.2×10^{14}
	90 °C/0 T	0.81	63.8	5.6×10^{14}
Normally annealed	150 °C/0 T	1.07	68.9	1.8×10^{14}
	210 °C/0 T	1.28	70.8	4.2×10^{13}
Magnetically annealed	90 °C/12 T	1.01	61.7	4.3×10^{14}
	150 °C/12 T	1.18	64.6	8.2×10^{13}
	210 °C/12 T	1.28	66.2	3.4×10^{13}

temperature from 90 °C to 210 °C, the necking region in the fracture surface is decreased gradually. When specimen is normally annealed at 210 °C, an obvious necking phenomenon appears (Fig.5c), and the microvoids have different sizes and depth characteristics (Fig.5d). All cryoECAP, normally annealed, and magnetically annealed specimens exhibit the limited uniform elongation, whereas their total elongation is reasonable, indicating that their fracture surfaces show the ductile characteristics.

3 Discussion

3.1 Effect of highly magnetic annealing on uniform elongation of UFG 1050 aluminum alloy

The grains in the cryoECAP specimen consist of elongated and equiaxed grains, and the average grain size is 0.7 μm . Many tangled dislocations can be clearly observed, as shown in Fig. 4a. With increasing the annealing temperature from 90 °C to 210 °C, the number of the equiaxed grains is increased gradually^[24], and the grain size is slightly increased for the normally annealed and magnetically annealed specimens. The average grain sizes of these two kinds of specimens are similar of 1.28 μm for the specimens normally

annealed and magnetically annealed at 210 °C (Table 1). It can be seen that the grain microstructures in the specimens normally annealed and magnetically annealed at 90–210 °C still remain the ultrafine morphology. During annealing at 90–210 °C, numerous dislocations pile up at the grain and/or subgrain boundaries in the normally annealed and magnetically annealed specimens^[24]. Therefore, the high strength of these UFG specimens is attributed not only to the refinement strengthening (Hall-Petch strengthening) but also to the dislocation strengthening. Thus, the strength of UFG 1050 aluminum alloy is significantly enhanced, compared with that of the coarse-grained specimen, as shown in Fig.1.

The engineering stress-engineering strain curves of UFG 1050 aluminum alloy show extremely different characteristics from those of the coarse-grained 1050 aluminum alloy. The limited uniform elongation can be observed in the cryoECAP, normally annealed, and magnetically annealed 1050 aluminum alloy (Fig. 1). This limited uniform elongation is attributed to the plastic instability at the early stage of deformation, and it is obviously related to the low strain hardening rate^[12]. During tensile deformation, the specimen is mechanically stable until the strain hardening rate is lower

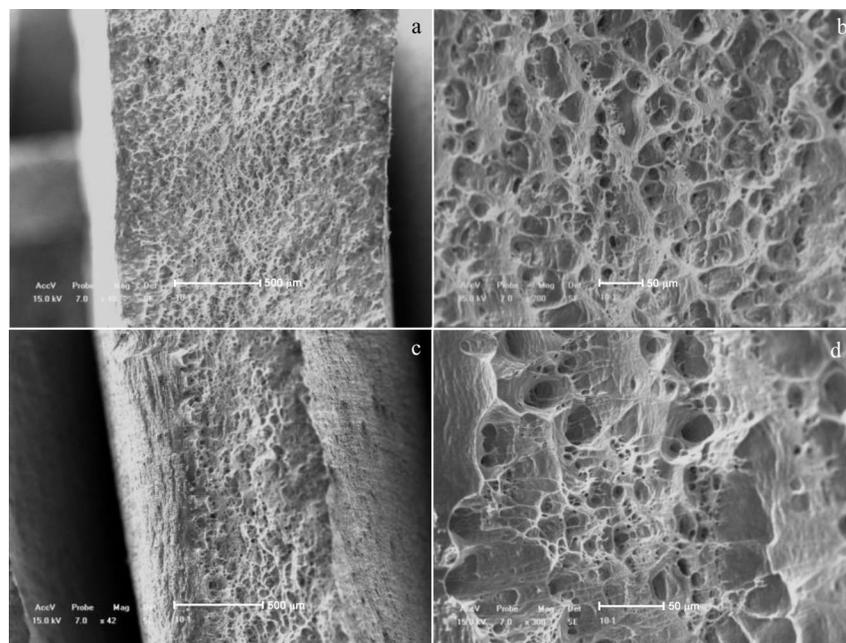


Fig.5 SEM fracture surfaces of cryoECAP specimens (a–b) and normally annealed specimens at 210 °C (c–d)

than the flow stress. Then, the plastic instability occurs, which can be expressed by Considère criteria^[27], as follows:

$$\left(\frac{\partial\sigma}{\partial\varepsilon}\right)_{\dot{\varepsilon}} \leq \sigma \quad (2)$$

where σ and ε are the true stress and true strain, respectively; $\dot{\varepsilon}$ is the strain rate. Therefore, the specimens with high strain hardening capacity tend to be stable, whereas those with low strain hardening capacity are unstable.

As shown in Fig.3, the cryoECAP specimen presents a quite limited strain hardening stage and has relatively low ratio of ultimate tensile strength to yield strength (1.24), which is 47.7% lower than that of the coarse-grained specimen. Thus, it can be deduced that the strain hardening capacity decreases sharply for the UFG 1050 aluminum alloy. With increasing the temperature from 90 °C to 210 °C, the ratio of ultimate tensile strength to yield strength is decreased, indicating the reduction in the strain hardening capacity of annealed specimens. It should be noted that the strain hardening phenomenon disappears after yielding in the specimen normally annealed at 210 °C (the ratio of ultimate strength to yield strength is 1.0).

Strain hardening is caused by the dislocation generation and storage during deformation. It is reported that the strain hardening rate in UFG materials is strongly dependent on the grain size, grain boundary characteristics, and deformation temperature. Hayes et al^[28] reported that the grain boundaries can act as both sources and aggregation area for dislocations, which can contribute to the plastic deformation in UFG aluminum. Therefore, the large grain boundary area in UFG materials can supply plenty of effective dislocation aggregation area, leading to lower strain hardening rate due to the decreasing dislocation storage within grain interiors^[17]. Sun et al^[11] pointed out that in UFG structure, the grain size is smaller than the mean free path of the mobile dislocations. Thus, it can be deduced that once a dislocation is generated from the grain boundary in UFG materials, it may travel across grain interior, which can hardly interact with other dislocations or dislocation clusters, and finally disappears at another grain boundary, resulting in low strain hardening rate. Consequently, due to the refined grains in cryoECAP, normally annealed, and magnetically annealed specimens, their strain hardening rates are significantly low, as indicated by the left side of Eq. (2). Additionally, UFG microstructure greatly increases the flow stress, as indicated by the right side of Eq. (2), especially at the early stage of plastic deformation (Fig.1). As a result, during tensile test, the decreasing rate of strain hardening rate of cryoECAP, normally annealed, and magnetically annealed specimens is no more than the increasing rate of flow stress. Thus, the plastic instability occurs at the early stage and finally causes the limited uniform elongation ($\leq 2.3\%$).

The uniform elongation of cryoECAP specimen is 2.3%. The fraction of HABs in cryoECAP specimen is 62.3%, which suggests that the predominant boundaries are HABs. As for the normally annealed specimens, with increasing the temperature from 90 °C to 210 °C, the uniform elongation is

decreased from 1.55% to 0.55%, as shown in Fig.2a, while the fraction of HABs is gradually increased from 63.8% to 70.8% (Table 1).

The grain boundary characteristics can significantly affect the deformation behavior of UFG materials^[11]. Wyrzykowski et al^[29] found that the dislocations disappear faster at HABs, compared with the case at LABs in aluminum alloys at room temperature, and the dislocation spreading rate can be controlled by the self-diffusion of grain boundaries, which highly depends on the grain boundary structures. Therefore, it can be inferred that the increase in the fraction of HABs due to the increasing temperature can enhance the recovery rate in the normally annealed specimens, and hinders the dislocation accumulation within grain interior, thereby resulting in the decreased strain hardening capacity. Moreover, the decreased dislocation density (from $5.6 \times 10^{14} \text{ m}^{-2}$ to $4.2 \times 10^{13} \text{ m}^{-2}$) also reduces the strain hardening capacity with increasing the temperature. Thus, with increasing the temperature from 90 °C to 210 °C, the plastic instability occurs at the early stage of tensile deformation due to the decreased strain hardening capacity, therefore leading to the decreased uniform elongation.

The magnetically annealed specimen has a higher uniform elongation (0.64% – 1.60%) and a lower fraction of HABs (61.7%–66.2%), compared with those after normal annealing at the same temperature. According to TEM observation results, the number of dislocations inside the grains significantly decreases, the dislocations are more ordered, and they pile up at the subgrain and/or grain boundaries in the specimens after magnetically annealed at 90–210 °C^[24]. Moreover, the grain size is slightly larger in the specimens after magnetical annealing at 90 and 150 °C (Table 1).

It is reported that the magnetic field can enhance the dislocation mobility without external mechanical stress^[21,30–31]. Alshits^[21] and Morgunov^[30] et al found that the magnetic field can weaken the interactions between dislocations and obstacles, which results in the release of dislocations from pinning center and finally promotes the dislocation motion. Molodov et al^[31] revealed that the free energy difference between adjacent grains is caused by the proper anisotropy under a suitable magnetic field. Then, a driving force for boundary displacement is generated, which does not depend on the grain boundary properties and can promote the grain boundary motion from the grains with lower free energy towards the ones with higher free energy. It is also reported that the interactions of lattice and grain boundary dislocations with paramagnetic defects in the crystal structure can enhance the mobility of dislocations and grain boundaries when the nonmagnetic materials are annealed under the magnetic field^[32].

Since the processes of recovery and recrystallization in UFG material are based on the interactions between dislocations and the motion of grain boundaries, it can be deduced that the magnetic annealing can enhance the recovery and recrystallization^[21,30–32]. Bhaumik^[18] and Molodov^[19] et al found that the magnetic field can promote the dislocation

motion and grain boundary mobility in the deformed cold-rolled 3103 aluminum alloy during annealing at 288–330 °C, and then accelerates the recovery and recrystallization. During the recovery and recrystallization, the distortion energy generated during plastic deformation can be rapidly consumed due to the high magnetic field, particularly during the recovery and the early stage of recrystallization. At that time, there is not enough energy to form HABs, leading to the low fraction of HABs in the magnetically annealed specimens (Table 1).

According to Fig.3, the ratio of ultimate tensile strength to yield strength of magnetically annealed specimens is 1.06%–1.18% at 90–210 °C, which is larger than that of normally annealed specimens (1.0%–1.17%). Therefore, it can be concluded that the lower fraction of HABs in the magnetically annealed specimen provides a slower dislocation annihilation rate, thus resulting in the higher strain hardening capacity, which suppresses the plastic instability at the early stage of tensile deformation and then leads to the higher uniform elongation, as shown in Fig.2a.

3.2 Effect of highly magnetic annealing on yield-drop phenomenon in UFG 1050 aluminum alloy

The yield-drop phenomenon can be observed in the specimens after normal and magnetic annealing at 120–210 °C, and it becomes more obvious with increasing the temperature (Fig.1). However, it can be seen that the flow stress after yield peak decreases more slowly in the magnetically annealed specimens, compared with those after normal annealing at the same temperature. The dislocation density is $7.2 \times 10^{14} \text{ m}^{-2}$ in the cryoECAP specimen, and it decreases from $5.6 \times 10^{14} \text{ m}^{-2}$ to $4.2 \times 10^{13} \text{ m}^{-2}$ in the specimens after normal annealing from 90 °C to 210 °C (Table 1). As for the magnetically annealed specimens, the dislocation density is even lower (3.4×10^{13} – $4.3 \times 10^{14} \text{ m}^{-2}$), compared with those after normal annealing at the same temperature.

Shear strain (γ)^[17] can be expressed by Eq.(3), as follows:

$$\gamma = bNA \quad (3)$$

where b is the value of Burgers vector, N is the number of mobile dislocations per unit volume, and A is the area of moving dislocations. The deformation of UFG materials is controlled by the dislocations generated from grain boundary sources instead of intragranular ones^[29,33], and the N value is closely related to the number of grain boundary sources. In UFG materials, the surface area of grain boundaries is increased due to the reduced grain size, which enhances the dislocation annihilation rate and then reduces the A value. In this case, a higher value of N is needed to maintain the applied strain rate. Once the number of the mobile dislocations is lower than the required value of N , a higher dislocation motion velocity is required to maintain the strain rate, therefore leading to high stress and the appearance of yield peak. It should be noted that the lower dislocation density results from the stronger dislocation annihilation effect of the extremely fine grain boundaries, which contributes to the smaller N value and results in the yield-drop phenomenon in

UFG materials.

No yield-drop phenomenon occurs in the specimens after cryoECAP, normal annealing, and magnetic annealing at 90 °C during tensile test. Yu et al.^[17] reported that for the UFG 1050 aluminum alloy, the dislocations introduced by ECAP process cannot be removed completely during annealing treatment at temperatures lower than 150 °C. Thus, these specimens have enough mobile dislocations to maintain the applied strain rate and do not exhibit the yield-drop phenomenon. TEM images of the specimens after cryoECAP and normal annealing at 90–210 °C at the yield peak state are shown in Fig.6. Numerous tangled dislocations exist in grain interior and at subgrain and/or grain boundaries in cryoECAP specimen, as shown in Fig.6a. During normal annealing at 90 °C, nearly no dislocations exist in the grain interior, whereas they can be observed at the subgrain and/or grain boundaries, as shown in Fig.6b. Therefore, it can be concluded that the cryoECAP specimen and the specimen after normal annealing at 90 °C have enough mobile dislocations to maintain the applied strain rate, i. e., the yield-drop phenomenon does not occur in cryoECAP specimen or in the specimen after normal annealing at 90 °C.

During normal annealing at 150 and 210 °C (Fig.6c and 6d), the tangled dislocations piled-up at subgrain and/or grain boundaries are transformed into ordered ones, and the dislocation density decreases. Moreover, the increase in the fraction of HABs with increasing the annealing temperature can enhance the dislocation annihilation during tensile deformation. Consequently, the occurrence of yield-drop phenomenon in the normally annealed and magnetically annealed specimens at 120–210 °C may result from the shortage of the mobile dislocations during tensile deformation. The decrease in dislocation density and the increase in the fraction of HABs jointly enhance the yield-drop phenomenon with increasing the annealing temperature.

According to the true stress-true strain curves of the normally annealed and magnetically annealed specimens at 150 and 210 °C in Fig.7a and 7b, the flow stress of the magnetically annealed specimen decreases slowly after the yield peak, and the yield-drop phenomenon is weakened, compared with those of normally annealed specimens. Fig.7c and 7d show the strain hardening rates of specimens normally annealed and magnetically annealed at 150 and 210 °C in yield-drop stage (stage I), as indicated in Fig.7a and 7b, respectively. It can be seen that the strain hardening rate of the normally annealed specimen at 150 °C is decreased firstly and then increased with increasing the strain. Moreover, the strain hardening rate of this specimen in stage I is negative. As the stress-strain curve reflects the competition result of strain hardening and dynamic recovery, it can be deduced that the dynamic recovery plays the dominant effect in stage I during tensile deformation. Thus, it can be concluded that the true stress-true strain curves of the cryoECAP, normally annealed, and magnetically annealed 1050 aluminum alloy can be divided into three stages: abruptly increased flow stress stage, strain softening stage, and sharply decreased flow stress stage.

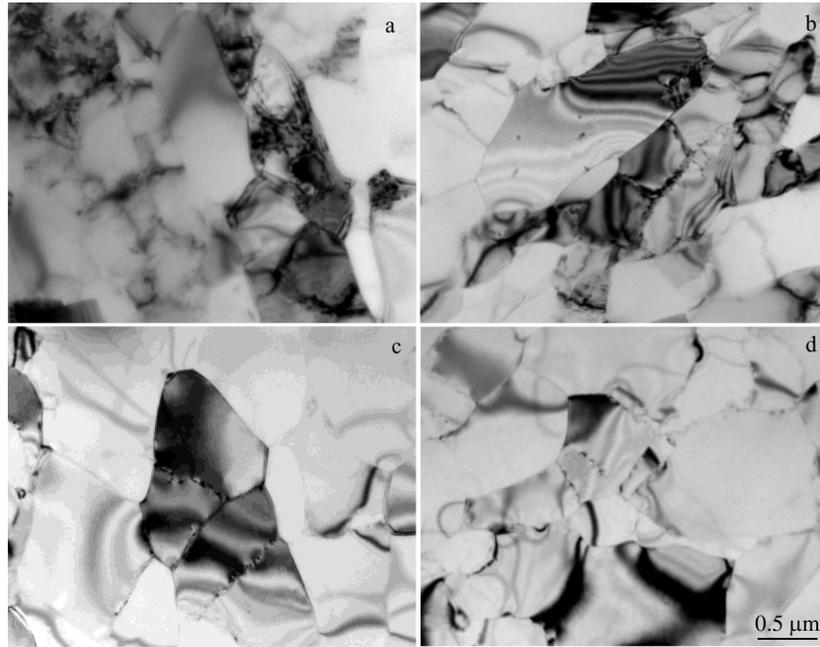


Fig.6 TEM images of cryoECAP specimen (a) and normally annealed specimens at 90–210 °C (b–d) at yield peak state: (a) $\varepsilon=2.3\%$; (b) 90 °C, $\varepsilon=1.55\%$; (c) 150 °C, $\varepsilon=0.92\%$; (d) 210 °C, $\varepsilon=0.55\%$

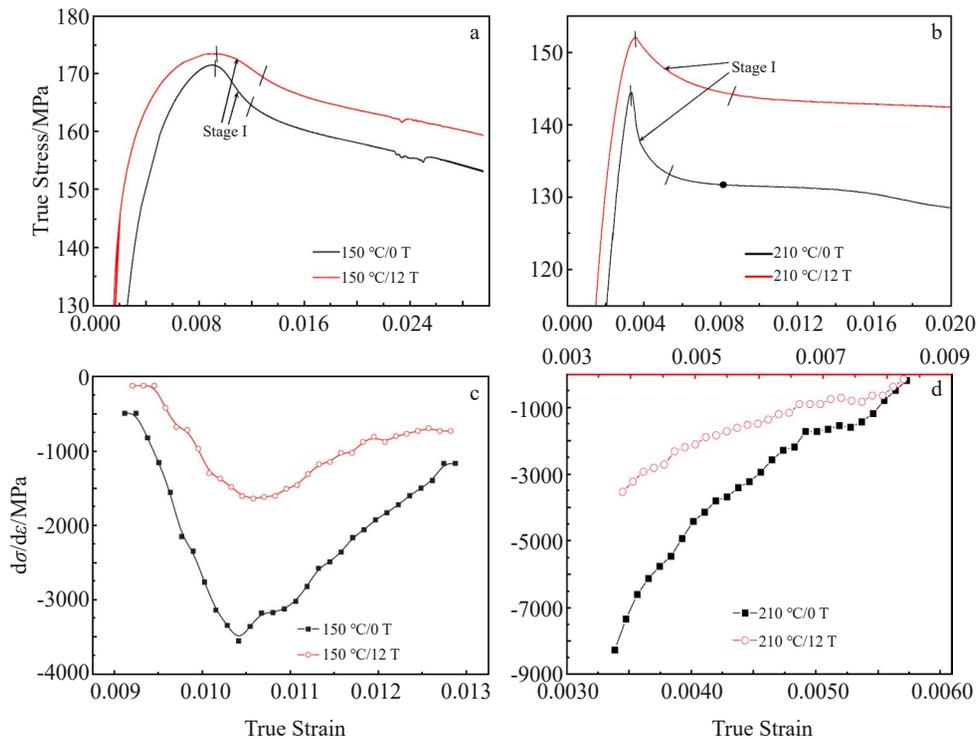


Fig.7 True stress-true strain curves of specimens after normal annealing and magnetic annealing at 150 °C (a) and 210 °C (b); strain hardening rates of specimens after normal annealing and magnetic annealing at 150 °C (c) and 210 °C (d) in yield-drop stage

The yield-drop phenomenon occurs in the specimens after normal annealing and magnetic annealing at 120–210 °C, and the strain softening stage can be divided into a fast decreasing sub-stage and a slow decreasing sub-stage. For the normally annealed specimens at 210 °C, the strain hardening rate is increased with increasing the strain. It is worth noting that the strain hardening rate of magnetically annealed specimens at

150 and 210 °C is higher than those of normally annealed specimens. This result indicates that the magnetically annealed specimens have higher capacity of dislocation generation and storage in stage I during tensile deformation.

Fig.8 illustrates TEM microstructures of normally annealed and magnetically annealed specimens at 210 °C in the yield-drop stage during tensile test. Compared with those in the

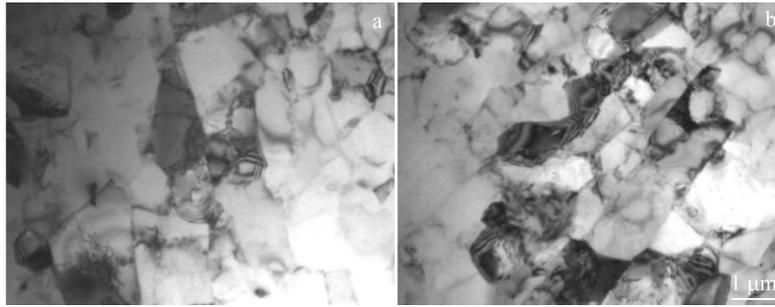


Fig.8 TEM microstructures of specimens after normal annealing (a) and magnetic annealing (b) at 210 °C in yield-drop stage ($\epsilon=0.4\%$ and $\epsilon=0.6\%$ for normally annealed and magnetically annealed specimens, respectively)

specimen before tensile test, many grains are elongated under the tensile stress and the dislocation density increases significantly in the normally annealed specimen in the yield-drop stage, as shown in Fig.8a. It can also be seen that most dislocations accumulate at the grain boundaries. A higher density of dislocations tangled at the subgrain and/or grain boundaries and inside the grains can be found in magnetically annealed specimen in the yield-drop stage during tensile test, as shown in Fig. 8b. The phenomenon proves that the magnetically annealed specimen has higher capacity of the dislocation generation and storage, which enhances the strain hardening rate and finally leads to the decrease in the flow stress after yield peak.

Based on the molecular dynamics simulations^[34], it is found that the grain boundary characteristics have great influence on the dislocation activity during tensile deformation. Additionally, the LABs are the more efficient sites for dislocation nucleation, compared with HABs. Consequently, it can be deduced that the higher fraction of LABs in the magnetically annealed specimens at 150–210 °C can provide more mobile dislocations to maintain the applied strain rate during tensile deformation, improve the capacity of dislocation generation and storage, and finally lead to the slow decrease in the flow stress after yield peak (Fig.1).

4 Conclusions

1) The uniform elongation, dislocation density, the ratio of ultimate tensile strength to yield strength, and the fraction of high-angle grain boundaries (HABs) in the 1050 aluminum alloy after equal channel angular pressing at cryogenic temperature (cryoECAP) are 2.3%, $7.2 \times 10^{14} \text{ m}^{-2}$, 1.24, and 62.3%, respectively. With increasing the annealing temperature from 90 °C to 210 °C, the fraction of HABs is increased from 63.8% to 70.8%; the uniform elongation is decreased from 1.55% to 0.55%; the dislocation density is decreased from $5.6 \times 10^{14} \text{ m}^{-2}$ to $4.2 \times 10^{13} \text{ m}^{-2}$; the ratio of ultimate tensile strength to yield strength is decreased from 1.17 to 1.0 for the normally annealed specimens.

2) With the application of high magnetic field, the number of dislocations inside the grains is significantly decreased, the dislocations are more ordered, and they are piled up at the subgrain and/or grain boundaries. The rapid consumption of

high distortion energy during recovery and the early stage of recrystallization results in the low fraction of HABs (61.7%–66.2%), therefore leading to the higher ratio of ultimate tensile strength to yield strength (1.06–1.18) and the higher uniform elongation (0.64%–1.60%).

3) The true stress-true strain curves of the cryoECAP, normally annealed, and magnetically annealed 1050 aluminum alloy can be divided into three stages: abruptly increased flow stress stage, strain softening stage, and sharply decreased flow stress stage. The yield-drop phenomenon occurs in the specimens after normal annealing and magnetic annealing at 120–210 °C, and the strain softening stage can be divided into a fast decreasing sub-stage and a slow decreasing sub-stage.

4) At the yield peak state during tensile deformation, many tangled dislocations exist within grains and at subgrain and/or grain boundaries. With increasing the annealing temperature from 90 °C to 210 °C, the dislocation density is decreased rapidly, and thus the yield-drop phenomenon occurs. With the application of high magnetic field, the low-angle grain boundaries with high fraction can act as the effective sites for dislocation nucleation, which finally leads to the slower decrease in the flow stress after yield peak.

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低温等径角挤压制备 1050 铝合金在磁退火后的均匀延伸率和屈服下降现象

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摘要: 采用低温等径角挤压 (cryoECAP) 制备了超细晶 (UFG) 1050 铝合金。采用拉伸试验、透射电子显微镜和电子背散射衍射等方法, 研究了 UFG 1050 铝合金在 90~210 °C、无磁场和 12 T 强磁场下退火 4 h 后的拉伸行为和显微组织。1050 铝合金经 cryoECAP 退火后, 晶粒尺寸为 0.70~1.28 μm, 极限抗拉伸强度与屈服强度之比小于 1.24, 均匀延伸率小于 2.3%。随着退火温度从 90 °C 上升到 210 °C, 屈服下降现象变得明显, 这是因为在拉伸变形过程中, 为了维持所施加的应变速率, 可动位错有所减少。均匀延伸率从 1.55% 下降到 0.55%, 位错密度从 $5.6 \times 10^{14} \text{ m}^{-2}$ 下降到 $4.2 \times 10^{13} \text{ m}^{-2}$, 大角度晶界含量从 63.8% 增加到 70.8%, 使得位错湮灭速率提升, 从而导致了应变硬化能力的降低。在 90~210 °C 的强磁场退火条件下, 低含量的大角度晶界 (61.7%~66.2%) 可以提供一个较慢的位错湮灭速率, 从而导致较高的均匀延伸率 (0.64%~1.60%) 和更慢的屈服点后的流变应力下降。

关键词: 磁场退火; 超细晶; 均匀延伸率; 屈服点现象; 大角度晶界; 位错

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