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Influence of Zn content on the microstructure and mechanical performance of ultrafine-grained Al-Zn alloys processed by high-pressure torsion

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Abstract: Al-Zn alloys were processed by high-pressure torsion (HPT) to produce ultrafine-grained (UFG) materials. For low Zn contents, HPT gave strengthening due to grain refinement while for the highest Zn concentration the decomposition of the microstructure yielded an abnormal softening at room temperature. The microstructure decomposition led also to the formation of a Zn-rich phase which wet the Al/Al grain boundaries and enhanced the role of grain boundary sliding with unusually high strain rate sensitivity. The occurrence of intensive sliding in these UFG alloys is demonstrated by deforming micro-pillars.

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1. Introduction

The best-known Al-Zn alloys, such as eutectic Al-95 wt.% Zn, eutectoid Al-78 wt.% Zn and solid solutions with Zn contents lower than 31.6 wt%, have been extensively studied [1-9]. Recently, an Al-30 wt.% Zn alloy was processed by severe plastic deformation (SPD) using the high-pressure torsion (HPT) technique [10]. As a consequence of SPD, an ultrafine-grained microstructure developed which exhibited unusually high elongations up to 150% and relatively high strain rate sensitivities at room temperature [2]. In addition, the HPT-processed material showed an abnormal softening despite the UFG microstructure. This abnormal softening, as well as the unusually high ductility of this UFG material, was explained by the simultaneous effect of grain refinement and the decomposition of the microstructure during HPT. The objective of the present study was to reveal the influence of Zn concentration on the microstructure and mechanical properties of Al-Zn alloys processed by HPT, thereby giving further insight into the unique properties of UFG Al-Zn alloys.

2. Experimental materials and procedures

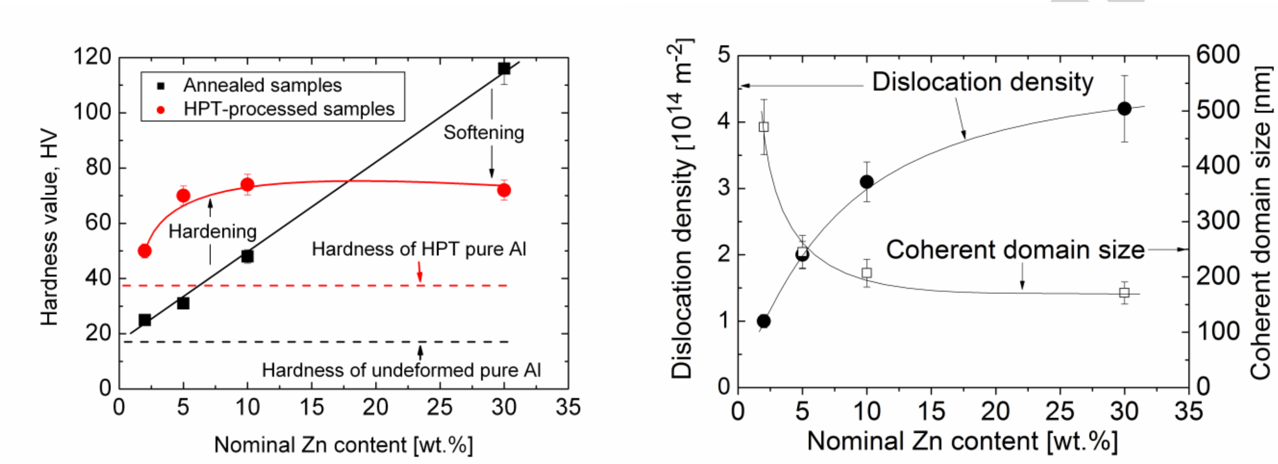
High purity (4N) Al and Al-Zn alloys with Zn contents of 2, 5, 10 and 30 wt.% were prepared by vacuum induction melting. Disks with diameters of 20 mm and thicknesses of 0.8 mm were homogenized at 500 ^{o}C for 1 h, then quenched into room temperature water and processed by HPT as described in [4-6]. Samples were cut from the half-radius of the HPT-processed disks. The microstructure of these specimens consisted of equiaxed ultrafine grains with an average size of ~200-500 nm.

Microhardness measurements were performed using a Zwick Roell ZHμ hardness tester with a Vickers indenter at load of 1 N. Nano-compression tests were conducted on micro-pillars having diameters of ~3 μm and heights of ~10 μm at room temperature using a UMIS device with a flat-ended conical diamond indenter. Details of the pillar preparation were given earlier [3].

The coherent domain size and the dislocation density in the HPT-processed disks were determined from analyses of X-ray diffraction line profiles measured by a high-resolution rotating anode diffractometer (type: RA-MultiMax9, manufacturer: Rigaku) using CuKα_1 (wavelength λ = 0.15406 nm) radiation.

The microstructures were also examined by differential scanning calorimetry (DSC) using a Perkin-Elmer DSC2 calorimeter in the temperature range between 300 and 650 K at a heating rate of 20 K min^{-1}.

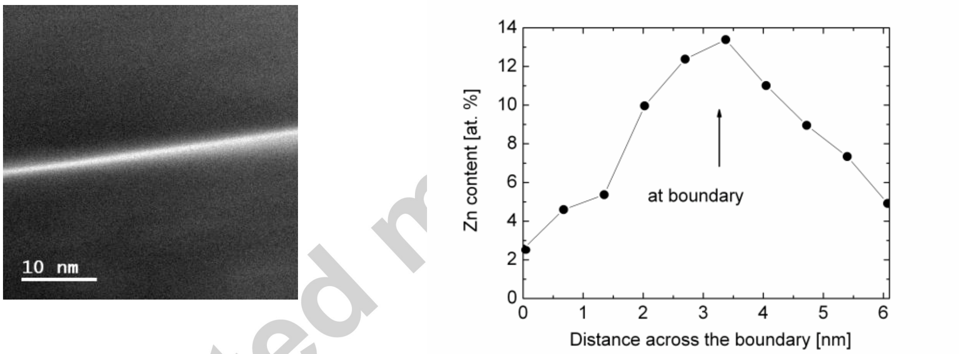
3. Experimental Results and Discussion



a) b)

***Figure 1**: Effect of HPT processing on a) microhardness and b) coherent domain size and dislocation density as a function of the nominal Zn content.*

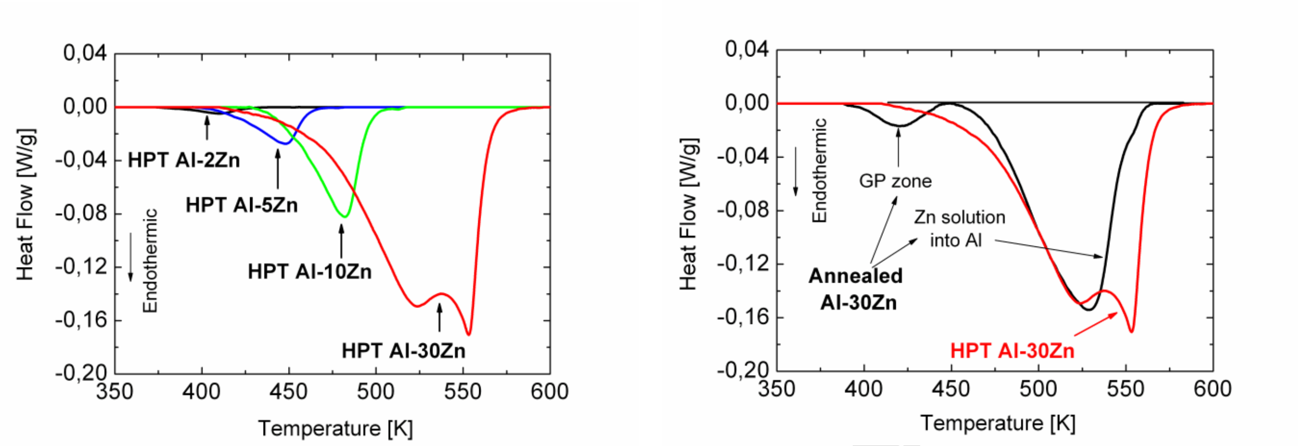
Figure 1 shows the effect of Zn concentration on the hardness (Fig. 1a) as well as on the coherent domain size and the dislocation density in the Al matrix (Fig. 1b). The dislocation density increases while the coherent domain size decreases with increasing Zn concentration due to the pinning effect of Zn on dislocations. The coherent domain size corresponds to the subgrain size, therefore it is smaller than the grain size determined by microscopy. In order to emphasize the significant changes, the hardness values of the annealed samples are also plotted in Fig. 1a. It can be seen that for Zn concentrations not larger than 10 wt%, the hardness of the HPT-processed samples is higher than for the annealed samples. In the case of the Al-30Zn alloy, however, there is a strong reduction in hardness from HV=118 to 72 due to HPT. Furthermore, the hardness of the HPT-processed Al-30Zn sample is almost the same as for the lower Zn-concentrated Al-5Zn and Al-10Zn alloys even though the average dislocation density of this sample is much higher than observed in the lower Zn-concentrated alloys (Fig. 1b). The abnormal softening of the high Zn-concentrated Al-30Zn alloy is a consequence of the strong decomposition of the annealed microstructure in this alloy as shown earlier [4,5]. Recent transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS) results on grain boundaries (GBs) [4,6] showed that more than 50% of the Al/Al grain boundaries are wetted by Zn-rich layers (see Fig. 2). In addition to the Al grains and Zn particles, these Zn-rich layers are certainly new components in the decomposed microstructure.



a) b)

Figure 2: *Zn-rich layer at an Al/Al grain boundary in the HPT-processed Al-30Zn alloy, demonstrated by a) TEM and b) EDS results [4].*

Figures 3 shows typical DSC thermograms taken on the HPT-processed samples having 2, 5, 10 and 30 wt.% Zn (Fig. 3a). The curve obtained for the HPT-processed Al-30Zn sample is compared with the conventional state in Fig. 3b. The latter thermogram has well defined endothermic peaks corresponding to the dissolution of Guinier-Preston (GP) zones and the solid solution of Zn in Al [11]. For the HPT-processed samples, the first DSC peak was not observed since SPD facilitated the development of Zn grains without the formation of metastable GP zones. Considering the HPT samples with different Zn contents (Fig. 3a), it can be seen that there is a significant difference between the Al-30Zn sample and the other three lower Zn-concentrated alloys. A double-peak thermogram is observed only for Al-30Zn, which consists of two peaks located at T=525 K and 555 K, respectively. The peak at 525 K corresponds to the dissolution of Zn in Al which is similar to the annealed state (see Fig. 3b). The other endothermic peak at 555 K most probably indicates the dissolution of the Zn-rich boundary layers.



a) b)

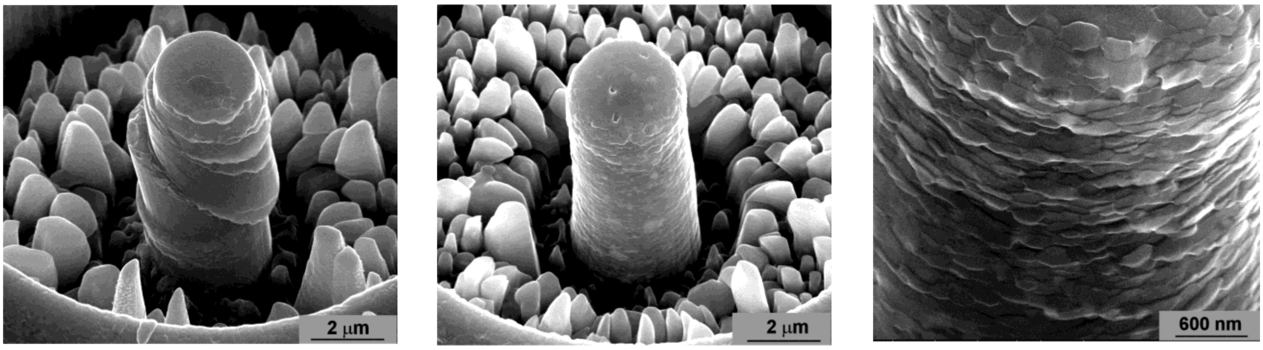
Figure 3: *DSC thermograms taken at heating rate of 20K/min on a) HPT-processed Al–Zn alloys with different Zn contents and b) conventional and HPT-processed Al–30 wt.% Zn alloys.*

In order to study the plastic behavior of the samples, the investigation focused on strain rate sensitivity (SRS) because it is well established that this parameter correlates to the ductility [12]. The SRS values determined by the indentation creep method [13] are listed in Table 1.

Table 1: *The strain rate sensitivity of the HPT-processed Al-Zn alloys.*

Sample	Al-2Zn	Al-5Zn	Al-10Zn	Al-30Zn
SRS value (± 0.02)	0.08	0.14	0.17	0.25

The SRS increases with Zn content and an unusually high value (0.25) was obtained for the Al-30Zn alloy. It is well known that UFG materials produced by SPD generally exhibit extremely low SRS of about 0.01-0.03 [14]. In the present case, the relatively high SRS is attributed to the Zn addition. Thus, the unexpectedly high SRS obtained for the HPT-processed Al-30Zn sample is a consequence of the high fraction of Al/Al grain boundaries wetted by Zn-rich layers which lead to intensive grain boundary sliding and then to a super-ductility [2].



a) b) c)

Figure 4: *Surface morphologies of compressed micro-pillars for a) the HPT Al-10Zn sample and b), c) the HPT Al–30Zn sample in low and high magnifications, respectively.*

In order to check the relationship between strain rate sensitivity and plasticity of these samples, micro-pillars were deformed by nano-compression. An examination of the surface morphologies of the compressed pillars by SEM revealed significant differences between the basic mechanisms of plastic deformation in the Al-10Zn and Al-30Zn samples (see Fig. 4). For the Al-10Zn sample having lower SRS (~0.17) strain localization and individual slip bands are visible (Fig. 4a), whereas in the Al-30Zn sample with higher SRS (~0.25) no strain localization and slip bands can be observed (Fig. 4b). Furthermore, the surface morphologies demonstrate clearly the occurrence of intensive grain boundary sliding in the plastic deformation of the latter sample. Indeed, individual ultrafine grains emerging from the pillar surface are visible in Fig. 4c as rings of very fine grains around the sample.

4. Conclusions

The microstructures and mechanical properties were studied for different UFG Al-Zn alloys processed by HPT. For low Zn contents normal strengthening was dominant whereas for the highest Zn concentration an abnormal softening was observed due to microstructural decomposition. It was shown that the strong microstructure decomposition in high Zn-concentrated alloy leads also to the formation of Zn-rich grain boundary layers which wet the Al/Al grain boundaries and enhance the role of grain boundary sliding in plasticity with an unusually high strain rate sensitivity.

Acknowledgements

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Highlights

1) Normal strengthening and abnormal softening in Al-Zn with different Zn concentrations.

2) Strong decomposition of the annealed microstructure in Al-30Zn alloy.

3) Enhanced role of grain boundary sliding with unusually high strain rate sensitivity.