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Unique microstructural and mechanical properties of Al-Zn alloys processed by high-pressure torsion

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Abstract: Al-Zn alloys with different Zn concentrations between 2 and 30 wt.% were processed by high-pressure torsion (HPT) to produce ultrafine-grained (UFG) materials. Microstructural and mechanical and properties of these UFG alloys were then investigated using depth-sensing indentations (DSI), focused ion beam (FIB), scanning electron microscopy (SEM) and differential scanning calorimetry (DSC). Emphasis was placed on the decomposition due to the HPT process, as well as on its effects on the mechanical properties of the UFG alloys. 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 wets 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 at room temperature is demonstrated by deforming micro-pillars, illustrating a potential for the effective application of these UFG materials in micro-devices

Keywords: Al-Zn alloys, phase decomposition, grain boundary sliding, indentation, micro-pillars, strain rate sensitivity, ultrafine grains

1. Introduction

It is well-known that ultrafine-grained (UFG) materials can be achieved by applying severe plastic deformation (SPD) techniques such as equal-channel angular pressing (ECAP) or high-pressure torsion (HPT) methods [1-3]. In general, the SPD-processed, UFG materials have reasonably saturated microstructures with steady-state dislocation density [4] so that the role of the grain boundaries is enhanced in the subsequent deformation processes. There are evidences suggesting that grain boundaries sliding (GBS) can occur easily even at room temperature in ultrafine-grained materials [5]. However, most of SPD-processed materials exhibit very limited ductility and low strain rate sensitivity (SRS) [6]. As the low ductility at ambient temperature restricts the use of these kind of materials (possibly having high strength) for structural applications, the enhancement of the ductility of UFG materials become as a topical subject of several investigations [7-10].

In this paper, some recent results are summarized describing the microstructural and the flow characteristics of the HPT-processed Al-Zn alloys, thereby giving insights into the unique microstructural and mechanical properties of these materials, especially into that of Al-30Zn alloy [11-15]. It should be emphasized that the Al-Zn alloys are probably the best known of all aluminum-based alloys. Some typical compositions, such as eutectic Al-95wt%Zn, eutectoid Al-70wt%Zn and solid solutions with Zn contents lower than 31.6%wt, have been extensively studied [11-23].

## 2. Microstructure and mechanical properties of Al-Zn alloys

a) *Unusual super-ductility microstructure-decomposition of HPT-processed Al-30Zn alloy.*
Supersaturated solid solution Al-30 wt.%Zn alloy, which is an important basic material in the aluminum industry, was processed by high-pressure torsion (HPT) [11] so that an ultrafine-grained microstructure developed which can be deformed by tensile for unusually high elongations up to 150% as shown in Fig. 1.
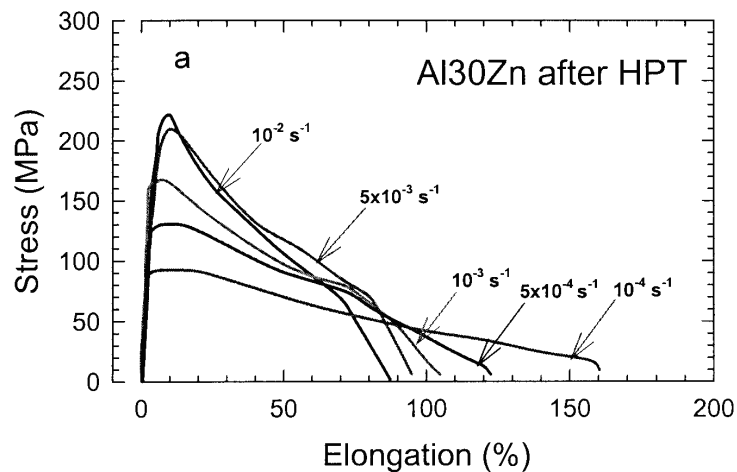

Figure 1 Stress-strain curves obtained at different strain rates on
HPT-processed Al-30%Zn samples deformed by tensile test [11].

Together with the super-ductility of this HPT-processed Al30Zn alloy, microstructural investigations taken by using transmission electron microscopy (TEM) and energy dispersive spectroscopy (EDS) [14,20] have showed a decomposed structure which contained equiaxed ultrafine Al grains having a size of 300-400 nm and smaller Zn grains located at the triple junctions of the aluminium grains. Furthermore, more than 50% of the Al/Al grain boundaries is wetted by Zn-rich layers as shown in Fig. 2.

a)   b)
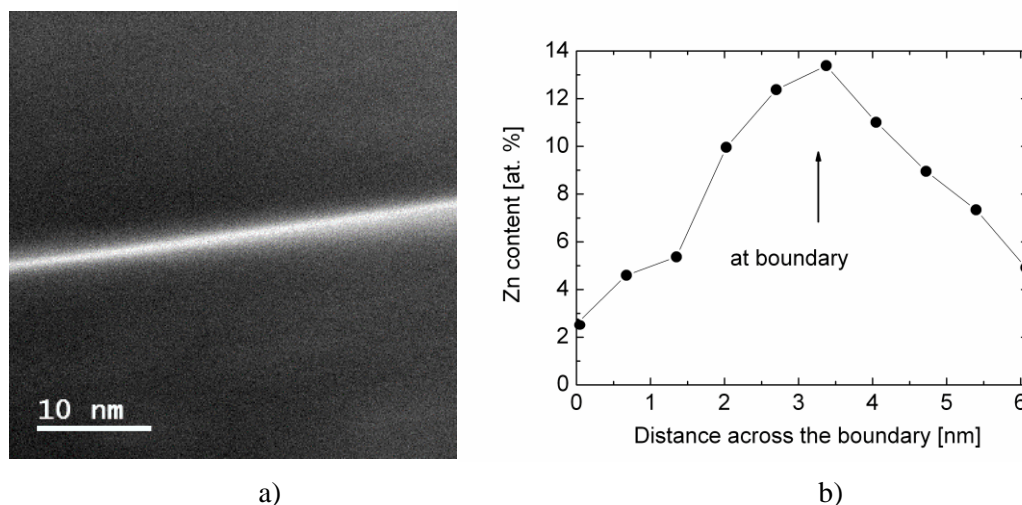
Figure 2 Zn-rich layer at an Al/Al grain boundary in the HPT-processed
Al-30Zn alloy, demonstrated by a) TEM and b) EDS results [14].

**b)** *Effect of Zn content on the mechanical properties of Al-Zn alloys*
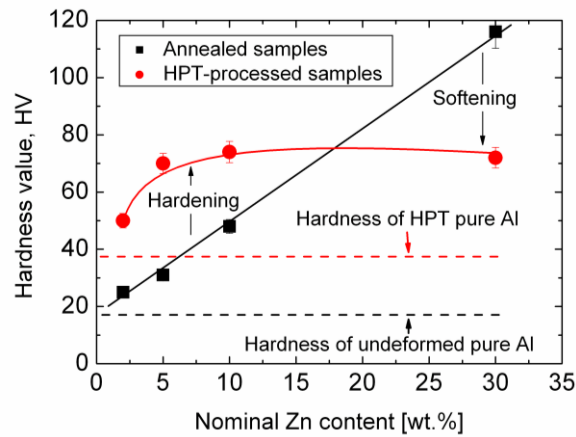
Figure 3 Effect of HPT processing on microhardness
of different Al-Zn alloys [15]

Figure 3 shows the effect of Zn concentration on the hardness of the HPT-processed A-Zn alloys. In order to emphasize the significant changes, the hardness values of the annealed samples are also plotted in Fig.3. For Zn concentrations not larger than 10 wt%, the normal strengthening-effect of SPD can be observed as the hardness of the HPT-processed samples is higher than that of the annealed samples. In the case of the Al-30Zn alloy, however, there is a strong reduction in hardness, indicating an abnormal softening due to HPT. The abnormal softening of the high Zn-concentrated Al-30Zn alloy is a consequence of the mentioned strong decomposition of the annealed microstructure in this alloy [14,19].

c) *The effect of the decomposed microstructures on the differential scanning calorimetry (DSC) thermograms*
Typical DSC thermograms taken on the HPT-processed Al-Zn samples having different Zn content can be seen in Fig. 4a. The thermogram obtained for the HPT-processed Al-30Zn sample is plotted together with that of the annealed state in Fig. 4b. It can be seen that the latter thermogram has well-defined endothermic indicating the dissolution of Guinier-Preston (GP) zones and the solid solution of Zn in Al [24]. However, for the HPT-processed Al-30Zn sample, the first DSC peak was not observed since SPD facilitated the development of Zn grains without the formation of metastable GP zones.
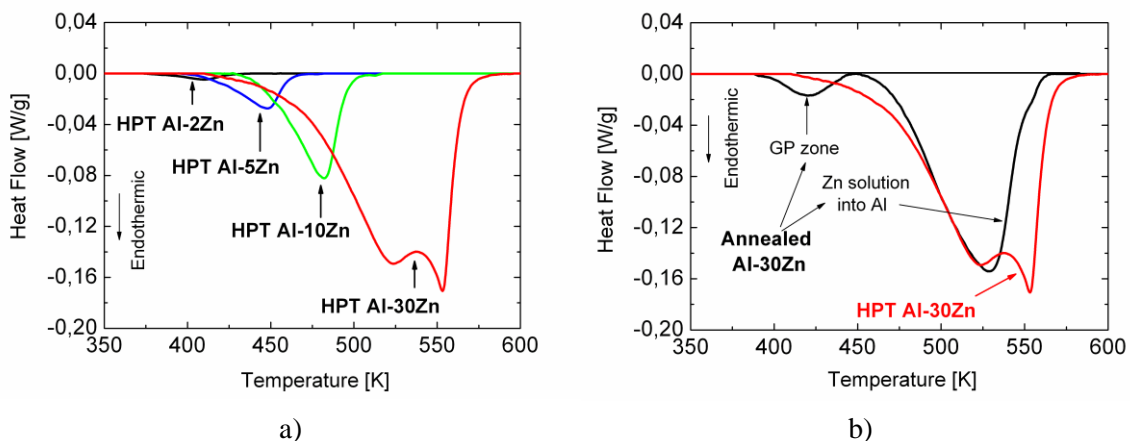
a)   b)
Figure 4 DSC thermograms taken at on a) HPT-processed Al–Zn alloys with different
Zn contents and b) annealed and HPT-processed Al–30 wt.% Zn alloys [15].

Considering the HPT samples with different Zn contents (Fig. 4a), it can be seen that there is a significant difference between the Al-30Zn sample and the other three lower Zn-concentrated alloys, as a double-peak thermogram is observed only for Al-30Zn sample. The two peaks of this curve are located at T=525 K and 555 K. The peak at 525 K corresponds to the dissolution of Zn in Al, which is similar to the annealed state (see Fig. 4b). The other endothermic peak at 555 K most probably indicates the dissolution of the Zn-rich boundary layers. This second peak cannot be observed at lower Zn contents. Therefore, the present of this peak indicates the significant amount of the Zn-rich layers in the Al-30Zn alloy.

d) ***The effect of the Zn-rich layers on the plastic behaviors of the HPT-processed Al-Zn alloys***
In order to study the plastic behavior of the HPT-processed samples, the investigation was focused on the determination of strain rate sensitivity (SRS) because it is well established that this parameter correlates to the ductility [25]. The SRS values were determined by using the indentation creep method developed in [26]. The obtained SRS values 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 ($\pm$ 0.02)	0.08	0.14	0.17	0.25

It can be seen that 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 [6]. In the present case, the relatively high SRS is attributed to the Zn addition. Determining the SRS and activation energy characterizing the process of plastic deformation in HPT-processed Al-30Zn alloy [13] it was shown that the process of plastic deformation is controlled mainly by the Zn diffusion along Al/Al grain boundaries.
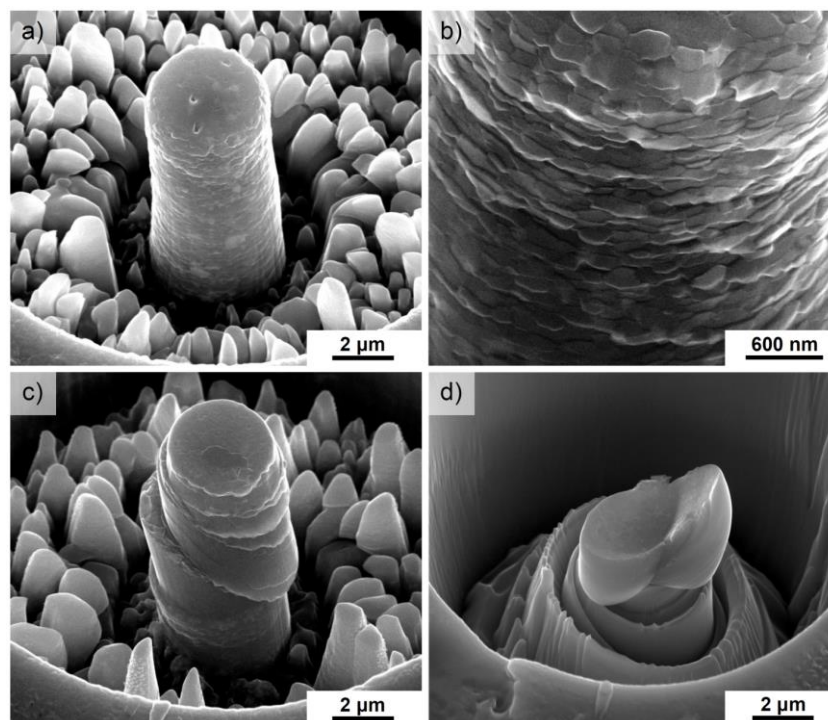
Figure 5 Surface morphologies of compressed micro-pillars for HPT-processed Al–30Zn in low and high magnifications shown in a) and b), respectively, c) HPT-processed Al-10Zn [15] and d) annealed, coarse-grained Al-30Zn [14] samples.

This mechanism is enhancing the role of the grain boundary sliding at room temperature. 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 leads to intensive grain boundary sliding and then to the mentioned super-ductility. In the case of lower Zn-content alloys, where the strain rate sensitivity is much smaller, the maximum elongation was lower the 50% [19].

In order to check the relationship between strain rate sensitivity and plasticity, as well as the effect grain boundary sliding in the investigated samples, micro-pillars were deformed by using nanocompression [14,15].

An examination of the surface morphologies of the compressed pillars by scanning electron microscopy (SEM) revealed significant differences between the basic mechanisms of plastic deformation in the different Al-Zn samples (see Fig. 5). In the case of the HPT-processed Al-30Zn sample (see Figs. 5a and 5b) the surface morphologies demonstrate clearly the occurrence of intensive grain boundary sliding, as individual ultrafine grains emerging from the pillar surface are visible [14]. The relatively high SRS of this sample is certainly a consequence of the high fraction of Al/Al grain boundaries wetted by Zn-rich layers, which lead to intensive grain boundary sliding. For the HPT-processed Al-10Zn sample having lower SRS, only strain localization and individual slip bands can be observed (Fig. 5c) [15]. In order to emphasize the super-ductility of the HPT-processed ultrafine-grained Al-30Zn sample, the surface morphology of the compressed pillar on the annealed Al-30Zn [14] can also be seen in Fig. 5d. In contrast with the UFG counterpart, the ductility of coarse-grained, annealed Al-30Zn sample having very low SRS of only 0.03 [13] seems to be very poor [14]. It can be seen that the deformation process of this sample consists of only few large strain fluctuations indicated by extreme slip bands in Fig. 5d. It has also been shown before that these large strain fluctuations lead to difficulties in plastically forming micrometer-scale single crystals because of the possibility of catastrophic failure. For this reason, therefore, micrometer-sized samples of coarse-grained metals are not suitable for use in the fabrication of micro-devices. On the other hand, the stable deformation by grain boundary sliding of the UFG micro-pillars, without the occurrence of any catastrophic avalanches, emphasizes the advantage of the sliding mechanism, suggesting an important potential for using these UFG materials in the fabrication of micro-devices.

3. Summary

Microstructures and mechanical properties of HPT-processed UFG Al-Zn alloys were studied by using transmission electron microscopy (TEM), tensile and microhardness tests, depth-sensing indentation (DSI), scanning electron microscope and focused ion beam (SEM/FIB), as well as by differential scanning calorimeter (DSC). The main results are the followings:

i)   For low Zn contents normal strengthening was dominant whereas for the highest Zn concentration an abnormal softening was observed due to microstructural decomposition.

ii)   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.

iii)   Occurrence of intensive grain boundary sliding at room temperature in the UFG Al-30wt%Zn alloy, leading to its super-ductility at room temperature.

iv)   As a consequence of the role of the grain boundaries, the deformation process of UFG materials is relatively homogenous and this may have important practical implications for the electronics industry and especially for using these materials in micro-devices.

Acknowledgements

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