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## Gradual softening of Al–Zn alloys during high-pressure torsion

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### ABSTRACT

Evolution of the microstructure and mechanical properties of Al–30 wt.% Zn alloy after the severe plastic deformation (SPD) by the high-pressure torsion has been investigated in detail in dependence on the increasing deformation degree. SPD leads to the gradual grain refinement and decomposition of the Al-based supersaturated solid solution. Measurements of the microhardness reveal a successive softening of the alloy with the increased deformation degree.

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### 1. Introduction

Al–Zn system retains attractiveness for further investigations being a base for numerous superplastic alloys. Recently, it was demonstrated that severe plastic deformation (SPD) is capable of significantly increasing the room temperature ductility of Al–30 wt.% Zn alloy processed by high-pressure torsion (HPT) [1,2]. Previous structural investigation of the HPT-treated Al–30 wt.% Zn alloy demonstrated that grain refinement and decomposition of the supersaturated solid solution simultaneously proceed in the material during the SPD [3]. These processes paradoxically lead to the alloy softening [4]. However, the structural changes took place so fast (within one anvil revolution) that it was practically impossible to investigate them. The objective of the present study is to analyze the process in details in dependence on the deformation degree.

### 2. Experimental

Al–30 wt.% Zn alloy was prepared by induction melting in vacuum from the high purity components (5N Al and 5N5 Zn). After melting they were poured in vacuum into the water-cooled copper crucibles with an internal diameter of 10 mm. The deformation was carried out at the room temperature by the HPT technique. Samples of the alloy were cut by spark machining in the disks with a thickness of about 1 mm and diameter of 10 mm. Each sample was

placed between two anvils; the applied pressure was 6 GPa, the deformation rate was 1 rpm. The degree of the deformation was determined by the number of the anvil rotations  $N$  ( $N=0.1, 0.3, 0.5, 0.7, 1$  and  $5$ ). Additionally, one sample was deformed simply by applying high pressure without any anvil rotation (it is denoted as  $N=0$ ). The state with  $N=0$  corresponds to pressure application, compressive deformation and some out-flow of the material from the die. As a result, the initial thickness of the sample of  $360\text{ }\mu\text{m}$  decreased to  $280\text{ }\mu\text{m}$ . During further rotation of the anvil, the out-flow of the material from the die continues and the sample thickness continues to decrease. It is about  $250\text{ }\mu\text{m}$  for  $N=5$ . The microstructure of the samples was studied by transmission electron microscopy (TEM) by JEM-100CX microscope. The TEM samples were prepared by electrochemical polishing. Dislocation density in TEM investigations was measured using a conventional technique by counting the number of dislocation intersections with a line of a definite length. The X-ray diffraction (XRDA) analysis was used to investigate the phase composition on the SIEMENS D-500 diffractometer ( $\text{CuK}_{\alpha 1}$  radiation). The precision X-ray reflection angles range of  $2\theta > 100^\circ$  is used to determine the lattice parameter of the alloys by Nelson–Riley procedure [5]. Microhardness of the alloy was measured on the PMT-2 device. Cold-worked surface layer of the sample before the measurements was removed by the careful mechanical polishing. Measurements were carried out using the load of 50 mN; 10 measurements were taken for each sample.

### 3. Results and discussion

XRDA of the studied samples showed that the alloy Al–30% Zn in the as-cast state contains two phases, namely (Al) and (Zn)

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solid solutions. HPT did not change the phase composition of the alloy, but the lattice parameter of the (Al) phase. The dependence of the lattice parameter of the Al-based solid solution on the deformation degree is shown in Fig. 1. The detached point at the  $N=0$  (full circle) corresponds to the non-deformed state of the alloy. Other points show the change in lattice parameter with successive increase of the number of revolutions.

The dependence shows that the lattice parameter of (Al) of the studied Al–30% Zn alloy gradually increases with increasing deformation degree. It is known that the atoms of Zn in Al form a substitution solid solution, and the lattice parameter of (Al) solid solution is decreased with increasing the zinc content [6]. Therefore, it follows from the obtained results that the

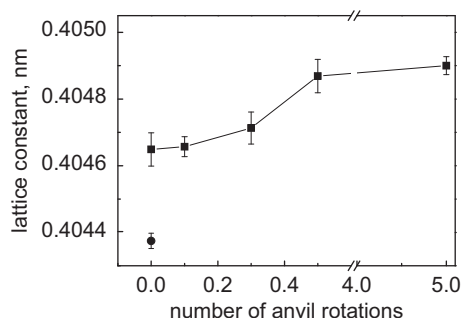


Fig. 1. The dependence of the (Al) solid solution lattice parameter on the deformation degree.

deformation leads to gradual decomposition of the (Al) super-saturated solid solution.

Fig. 2 shows the structure of the Al–30 wt.% Zn alloy in four successive states, i.e. in the initial non-deformed state, and in the state with  $N=0$ , 0.5 and 5. The HPT continuously refines the grain structure. In the state before the deformation (Fig. 2a) the structure consists of rather large (about 15  $\mu\text{m}$ ) (Al) grains. Both (Al) and (Zn) grains are almost dislocation-free with a dislocation density about  $10^{12} \text{ m}^{-2}$ . The dislocations are distributed uniformly. Namely, there are two kinds of grains. The first kind contains no or very little amount of globular Zn precipitates inside. In the second case, the cellular decomposition of the (Al) solid solution took place [7,8], and the grains contain lamellae of (Zn) separated by the areas of (Al) matrix. The observed structural variety is most probably explained by the fact that the grains which solidified at different temperatures contain different amounts of Zn. The Zn particles are also precipitated along the (Al)/(Al) grain boundaries (GBs). This can be explained by the fact that the Zn-rich melt completely wets the (Al)/(Al) GBs above eutectic point [9,10].

Fig. 2b shows the structure of the alloy after HPT with  $N=0$ . The sample was exposed to the pressure for about 5 min in order to equal the time of the deformation to that of the sample with  $N=5$ . The structure refinement begins already under the compressive strain without shear. The Zn lamellae formed in some of (Al) grains started to break into several parts. The morphology of this deformation-driven process is completely different from the conventional diffusional dissolution of lamellar structures in

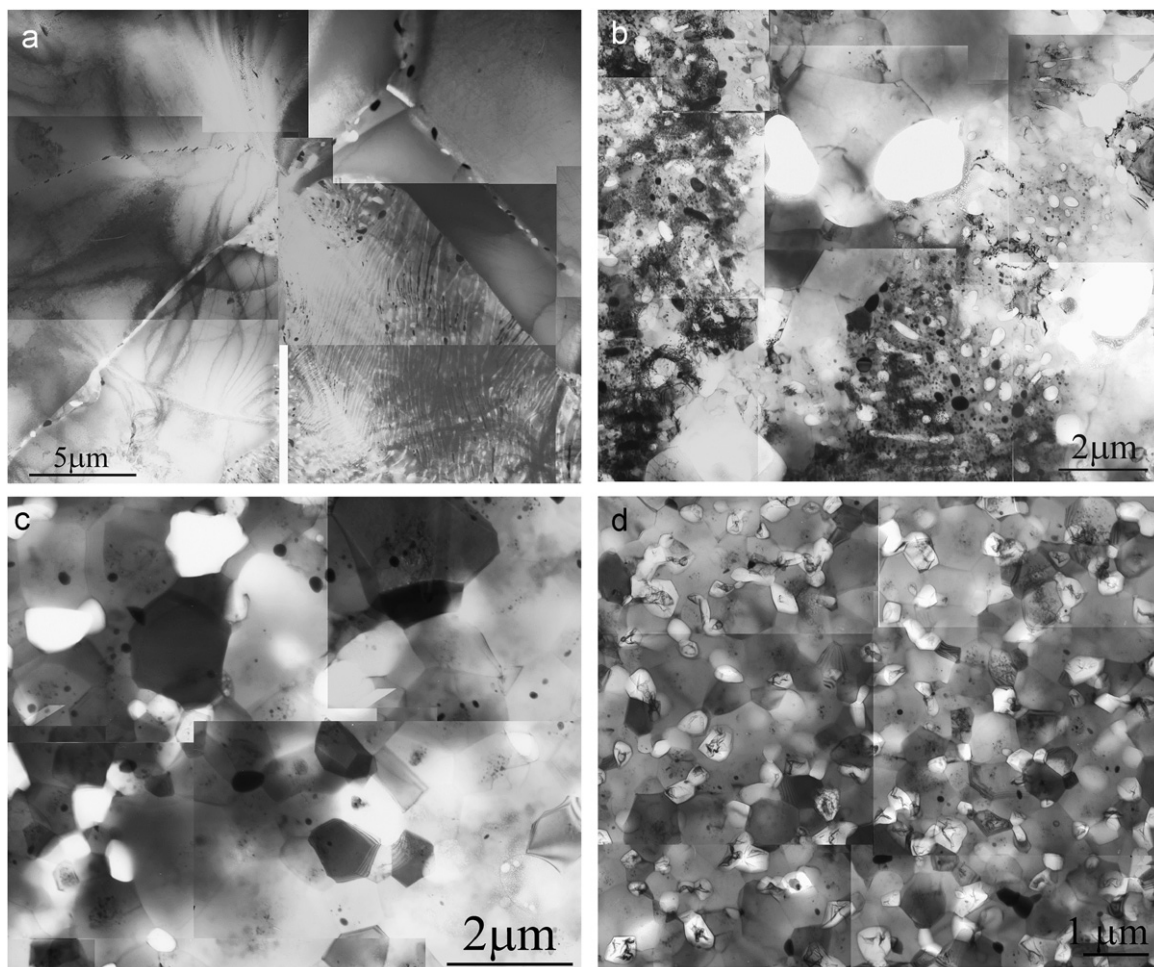
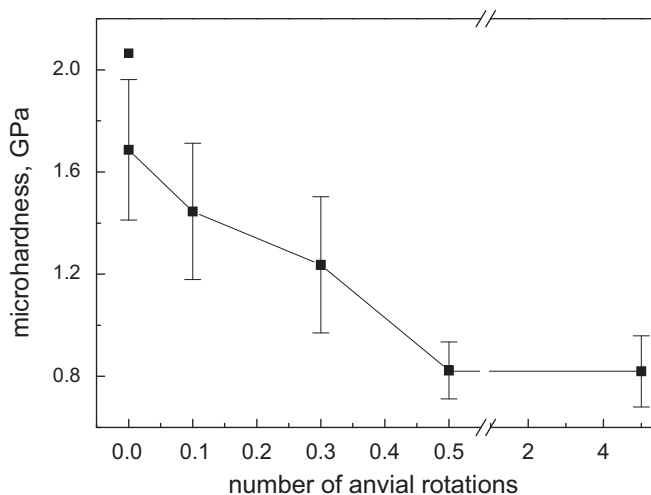


Fig. 2. The structure of the alloy Al–30% Zn in the initial state (a) and after HPT:  $N=0$  (b),  $N=0.5$  (c) and  $N=5$  (d).



**Fig. 3.** The dependence of microhardness of the Al-30% Zn alloy on the deformation degree.

Al–Zn alloys [11]. The size of (Al) grains became about 4  $\mu\text{m}$ . The (Al) grains still contain numerous Zn precipitates. Subsequent HPT-deformation resulted in further grain refinement down to 370 nm at  $N=5$ ; simultaneously Zn particles almost disappear from (Al) grains and form Zn grains in GBs and triple points. Another feature of the alloy structure after the deformation is the low dislocation density around about  $10^{12} \text{ m}^{-2}$ , which is not increased in comparison with the non-deformed sample. The presence of the Zn-rich GB layers which are quite good visible in the starting coarse-grained alloy (Fig. 2a) allows us to suppose that GB sliding plays an important or even predominant role during the SPD processing of the materials as demonstrated in [2]. However, the grain refinement cannot be the result of sliding along existed GBs, it needs the dislocation reactions. The unchanged dislocation density increase can be explained by the dynamic recovery due to the quick diffusional relaxation which proceeds parallel to the deformation. The very fast decomposition of supersaturated solid solution of Zn in Al (Fig. 1 and [3,4]) strongly indicates the intensity of these diffusional processes. The fluxes of non-equilibrium vacancies formed during deformation to their sinks can be responsible for the fast diffusion during HPT of studied Al–Zn alloy.

Microhardness was measured to characterize the changes of mechanical properties of the alloy after SPD. Microhardness was measured in different parts of the sample (both at the edge and in the center), so the value is averaged over the entire area of the sample. The results are shown in Fig. 3. According to the obtained data the hardness decreases with increasing the deformation degree, i.e. the softening of the alloy is observed during its deformation. The paradoxical deformation softening of the alloy can be explained by the concurrence of two opposite processes [11]. In our case the Hall–Petch hardening due to the grain refinement is compensated by the softening due the “purification” of (Al) solid solution [12]. The steady state Zn-content and grain size were rich very quick, already after about  $N=0.5$ .

Usually, the high pressure decreases the diffusivity and grain boundary mobility [13,14]. However, the atom movements caused by strong external forces can drive both accelerated diffusion and phase transformations in the material [15]. Following the idea of Martin proposed for the materials under severe irradiation [16],

we suppose that the forced mixing emulates the increase of entropy and changes the thermodynamic potentials in the alloy. As a result, the phases forming during SPD correspond to the equilibrium at a certain temperature called effective temperature  $T_{\text{eff}}$ . In our case the phases after HPT can be found in the equilibrium phase diagram below 200  $^{\circ}\text{C}$  [17]. It means that  $T_{\text{eff}}$  is close to the ambient temperature. It is because the Zn diffusivity in Al at the temperature of HPT treatment [1–4] is higher than, for example, the diffusivity of Ti in TiNi [15,18], or Nd in Fe–Nd–B alloys [15,19], or Cu in Cu–Ni alloys [20]. In the materials with low diffusivity at the temperature of SPD-treatment,  $T_{\text{eff}}$  can be very high and even exceed the melting temperature [15]. In this case the amorphous phase or phases can be formed [15,18,21].

#### 4. Conclusions

SPD by HPT of the Al–30 wt.% Zn alloy leads simultaneously to the grain refinement, decomposition of supersaturated solid solution of Zn in Al and to the softening of the material. The steady state Zn-content and grain size were riched already after 0.5 revolution of the HPT anvils. The phases formed after HPT can be found in the equilibrium phase diagram below 200  $^{\circ}\text{C}$ . It means that the HPT effective temperature is close to the ambient one.

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#### References

- [1] Valiev RZ, Murashkin MY, Kilmametov A, Straumal BB, Chinh NQ, Langdon TG. *J Mater Sci* 2010;45:4718–24.
- [2] Chinh NQ, Csanádi T, Györi T, Valiev RZ, Straumal BB, Kawasaki M, et al. *Mater Sci Eng A* 2012;543:117–20.
- [3] Straumal BB, Baretzky B, Mazilkin AA, Philipp F, Kogtenkova OA, Volkov MN, et al. *Acta Mater* 2004;52:4469–78.
- [4] Mazilkin AA, Straumal BB, Rabkin E, Baretzky B, Enders S, Protasova SG, et al. *Acta Mater* 2006;54:3933–9.
- [5] Cullity BD. *Elements of X-ray diffraction*. 2nd ed. Boston: Addison–Wesley Publishing; 1967 p. 356.
- [6] Pearson WB. *Handbook of Lattice Spacings and Structures of Metals and Alloys*. New York: Pergamon Press; 1958 p. 1965.
- [7] Pawlowski A, Zieba P. *Mater Sci Eng A* 1989;108:9–17.
- [8] Zieba P, Pawlowski A, Gust W. *Def Diff Forum* 2001;194:1759–65.
- [9] Straumal BB, Gornakova AS, Kogtenkova OA, Protasova SG, Sursaeva VG, Baretzky B. *Phys Rev B* 2008;78:054202.
- [10] Straumal B, Valiev R, Kogtenkova O, Zieba P, Czepe T, Bielanska E, et al. *Acta Mater* 2008;56:6123–31.
- [11] Zieba P, Pawlowski A. *Scripta Metall* 1986;20:1653–6.
- [12] Xu Ch, Furukawa M, Horita Z, Langdon TG. *Acta Mater* 2005;53:749–56.
- [13] Kedves FJ, Erdélyi G. *Def Diff Forum* 1989;66–69:175–88.
- [14] Molodov DA, Straumal BB, Shvindlerman LS. *Scripta metall* 1984;18:207–11.
- [15] Straumal BB, Mazilkin AA, Baretzky B, Rabkin E, Valiev RZ. *Mater Trans* 2012;53:63–71.
- [16] Martin G. *Phys Rev B* 1984;30:1424–36.
- [17] Massalski TB, editor. *Materials Park: ASM International*; 1993.
- [18] Prokoshkin SD, Khmelevskaya IYu, Dobatkin SV, Trubitsyna IB, Tatyaniy EV, Stolyarov VV, et al. *Acta Mater* 2005;53:2703–14.
- [19] Stolyarov VV, Gunderov DV, Popov AG, Gaviko VS, Ermolenko AS. *J Alloys* 1998;281:69–71.
- [20] Straumal BB, Protasova SG, Mazilkin AA, Rabkin E, Goll D, Schütz G, et al. *Mater Sci* 2012;47:360–7.
- [21] Mazilkin AA, Abrosimova GE, Protasova SG, Straumal BB, Schütz G, Dobatkin SV, et al. *Mater Sci* 2011;46:4336–42.