# A microstructure-based model for describing strain softening during compression of Al-30%wt Zn alloy

M. Borodachenkova<sup>1,\*</sup>, J. Gracio<sup>1</sup>, R.C. Picu<sup>2</sup> and F. Barlat<sup>3,1</sup>
<sup>1</sup>Center for Mechanical Technology and Automation, Department of Mechanical Engineering, University of Aveiro, 3810-193 Aveiro, Portugal
<sup>2</sup>Department of Mechanical, Aerospace and Nuclear Engineering, Rensselaer Polytechnic Institute, Troy, NY 12180, USA
<sup>3</sup>Materials Mechanics Laboratory, Graduate Institute of Ferrous Technology, Pohang University of Science and Technology, San31, Hyoja-Dong, Nam-Ku, Pohang, 790-784, South Korea

#### **ABSTRACT**

A microstructural-based model, describing the plastic behavior of Al-30wt% Zn alloy, is proposed and the effect of solid solution decomposition, Orowan looping, twinning and grain refinement is analyzed. It is assumed that the plastic deformation process is dominated by strain-induced solute diffusion and dislocation motion. To capture the essential physics, a law describing the evolution of the mean free path of dislocations with plastic strain is proposed which reproduces the experimentally observed strain softening.

# 1. STATE OF THE ART

Aluminum zinc alloys have been studied intensively in the past few years [1–16]. The main studies were dedicated to the improvement of strength and formability by refining the microstructure. Namely, previous work has shown that it is possible to obtain nanoscale two phase fcc/hcp crystals with very low dislocation density. Such microstructure is obtained via decomposition of the supersaturated solid solution [1, 9]. Simultaneously, the decomposition process strongly decreases the residual stresses. Al-Zn alloys are composed from two phases: Al and Zn. Al is a fcc phase where the mechanical behavior is essentially related to the motion and the interactions of dislocations. In the case of monotonic loading, the Taylor equation, which relates the stress to a mean dislocation density, is adequate to represent the flow stress of the alloy. For the assessment of ductility, additional information about the evolution of the dislocation density is required. This was emphasized and analyzed in the work of Kocks and Mecking [17]. Dislocations form three-dimensional arrays whose structure depends on the strain, strain rate, temperature etc. Pure aluminum and solute-strengthened alloys such as Al-Zn and Al-Mg accumulate dislocations differently. Pure aluminum is a dislocation cell-forming material [18-20] whereas, Al-Zn and Al-Mg are non-cell-forming materials [1, 21, 22]. In aluminum alloys such as Al-Cu, Al-Zn, or Al-Mg-Si, the substructure is more difficult to characterize than in pure aluminum because alloying elements, either in solid solution, precipitates or intermetallic particles, interact with dislocations. In such materials, only complex interlacing of dislocations without apparent organization could be observed [23–24]. Zn segregates in Al-Zn alloys as pure metal with the usual hcp structure. In this phase the main

Fax: +351 234 370953

<sup>\*</sup>Corresponding author E-mail: mborodachenkova@ua.pt, Phone: +351 234 370 827

deformation mechanisms are slip on the basal  $(0001) < 11\overline{2}0 >$  and pyramidal systems  $(11\overline{2}2) < 11\overline{2}3 >$  and twinning on the  $(10\overline{1}2) < 10\overline{1}1 >$  system [25].

The understanding of precipitation mechanisms is critical for achieving optimal properties [22, 26, 27]. A number of experimental studies on the aging behaviour of Al–Zn alloys are available [5, 9]. It was also shown that the attainment of a fine scale two phase microstructure in the aluminum zinc system can be obtained by severe plastic deformation [1–3]. For instance the structure, phase composition and thermal evolution of binary Al–Zn alloys before and after high-pressure torsion was studied in detail [11]. It was shown that heating HPT-deformed samples from room temperature to 300°C, leads to alloy decomposition, and formation of a relatively fine-grained (Al) equilibrium solid solution. In another work [28] it was shown that torsion straining produces an essentially equiaxed grain structure with some mixing of the two separate phases. After pressing to a strain of 0.8 at a temperature of 373 K, submicrometer grains are obtained but with agglomerates of ultrafine Al-rich and Zn-rich grains formed by the division of the original grains into smaller grains with minimal mixing of the two phases.

In the present work we propose a microstructural-based model to describe the softening process occurring during compression of super saturated solid solution Al-30wt% Zn alloy. The model is based on strain induced diffusivity of Zn and evolution of the mean free path of dislocations during plastic deformation.

## 2. EXPERIMENTAL DETAILS AND RESULTS

In this work a super saturated solid solution (SSSS) of Al-30wt%Zn was subjected to severe plastic deformation in compression, until a plastic strain of 0.25 and a strain rate of  $8.3 \times 10^{-4} \, \mathrm{s^{-1}}$ . The compression was applied to a small disk-shaped metal work piece. The sample was 10 mm in diameter and 0.38 mm in thickness. Since the samples are very thin, interrupted tests were carried out at different stress levels to accurate measure the corresponding strains.

In addition, hardness measurements were performed using a nanoindenter with load and displacement resolutions of 50 nN and 0.01 nm. Transmission electron microscopy (TEM) was carried out using a JEM-4000 FX microscope operating at 400 kV. The indentations and TEM observations were performed in the same area. A typical stress-strain curve for this alloy obtained in compression is shown in Figure 1. The experimental stress values have

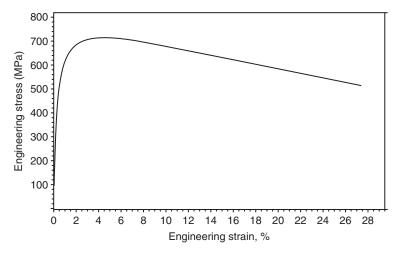


Figure 1 Stress-strain curve for the Al-30wt% Zn alloy subjected to uniaxial compression

been compared with hardness measurements carried out at different strain levels. The material strain hardens up to an engineering strain of about 5%, after which strain softening is observed.

# 3. MICROSTRUCTURAL ANALYSIS

The microstructure of the super saturated solid solution is shown in Figure 2. It contains Al and Zn grains with grain sizes respectively of 15 and 1 micron. The Zn grains are decorating the grain boundaries and present an ellipsoid shape.

The microstructure of the material after compression up to 0.05 strain is shown in Figure 3. The most prominent feature is precipitation of Zn particles in the bulk and at the grain boundaries of Al grains. Moreover, tangles of dislocations were observed between precipitates.

With increasing strain the dislocations become pinned at Zn particles, and start forming regular arrays in the grains as shown in Figure 4.

Interestingly, in different grains/precipitates of Zn different deformation mechanisms operate. Specifically, it is observed that while dislocation activity dominates in some grains,



Figure 2 Microstructure of the supersaturated solid solution before compression. Zn particles are present at grain boundaries.

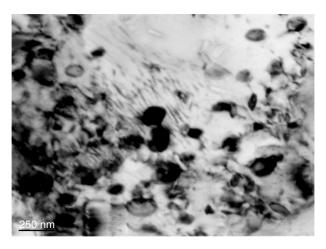


Figure 3 Microstructure Al-30% wt Zn alloy after a plastic strain of 0.05.

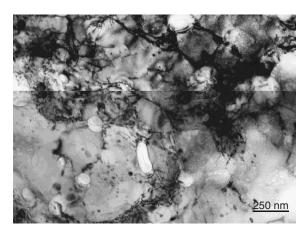


Figure 4 Pinned dislocations and formation of regular arrays in the grains observed during compression of Al-30%wt Zn after a plastic strain of 0.1.

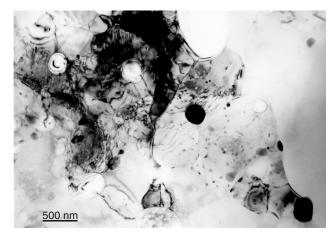


Figure 5 Microstructure Al-30% wt Zn alloy after a plastic strain of 0.25.

twinning takes place predominantly in other Zn grains. This is expected, and in agreement with previous observations [25].

After compression up to 0.25 the grain sizes of Al and Zn decrease, respectively to 4 micron and 252 nm. Other important characteristics of the microstructure after compression are: (i) increase of the average size of Zn particles in the bulk of Al grains (from 20 to 60 nm); decrease of the concentration of Zn particles in the material bulk and increase of Zn precipitates near or at grain boundaries (Figure 5).

# 4. DISCUSSION AND MODELING

The physical mechanism by which decomposition of supersaturated solid solution occurs in Al-Zn alloys was described previously [1]. In the present analysis we consider that microstructure evolves during compression according to the schematic view represented in Figure 6.

The dislocations in the bulk of Al grains serve as nucleation sites for Zn precipitates. Zn also precipitates at grain boundaries. With increasing deformation the dislocations

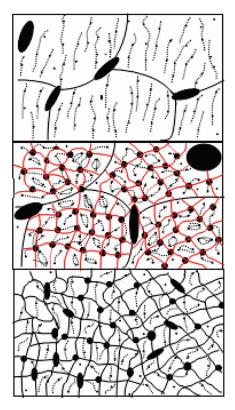


Figure 6 Schematic view of microstructure evolution during compression. In this sketch the large black ellipses indicate Zn particles/grains located at the grain boundaries, while the smaller black circles indicate Zn precipitates within the grain. The black and red continuous lines indicate dislocations pinned by precipitates and forming regular arrays in the grains, while the dotted lines indicate free dislocations which act as nucleation sites for new Zn precipitates.

rearrange and self-organize in new grain boundaries which become heavily enriched in Zn. The pinned dislocations mediate Zn redistribution between the pinning particles by pipe diffusion. In addition, the intense pinning produces an effective division of the grains in sub-grains. These sub-grain boundaries contribute to speeding up solute diffusion towards growing precipitates. The grain boundary and pipe diffusion is much faster than the bulk diffusion of solute.

The plastic deformation of Al is strongly controlled by slip system activity. An effective critical shear  $\tau$  is necessary for activating dislocations, which increases as a function of the accumulated shear  $\gamma$  in the grain, and is the same for every system of every grain.  $\tau$  and  $\gamma$  are related to the macroscopic flow stress and the strain increment through the average Taylor factor M, as:

$$\sigma = M\tau$$
 (1)

$$d\gamma = Md\varepsilon \tag{2}$$

The shear stress is assumed to depend on the average dislocation density through the usual Taylor law

$$\tau = \tau_0 + \alpha \mu b \sqrt{\rho} \tag{3}$$

where  $\tau_0$  is the friction stress, b is the length of the Burgers vector of dislocations,  $\alpha$  is a constant describing dislocation-dislocation interaction,  $\mu$  is a shear modulus,  $\rho$  is dislocation density (approximately  $10^{12}$  m<sup>-2</sup>).

By considering a homogeneous dislocation distribution, the evolution of the average dislocation density  $(\rho)$  with strain  $(\gamma)$  can be described by:

$$\frac{d\rho}{d\gamma} = \frac{d\rho^+}{d\gamma} - \frac{d\rho^-}{d\gamma} \tag{4}$$

where  $\frac{d\rho^+}{d\gamma}$  and  $\frac{d\rho^-}{d\gamma}$  are the dislocation storage and annihilation terms, respectively.

In the approach by Kocks and Mecking [17], the annihilation process is thermally activated and governed by the glide of dislocations at low and medium temperatures. They obtain the following equation for the dislocation density evolution:

$$\frac{d\rho}{d\gamma} = \frac{1}{bL} - f\rho \tag{5}$$

where L is the dislocation mean free path and f is the dynamic recovery term. The microstructural analysis reveals that during the first steps of compression, Al grains are covered by high population of small precipitated Zn particles which impede the movement of dislocations. Under these circumstances a new term should be added to Eqn. (1) accounting for the Orowan mechanism which in turn should be dependent on both the distance between Zn particles ( $\omega$ ) and the size of Zn particles (d)

$$\sigma = M(\tau + \tau_{orowan}) \tag{6}$$

where

$$\tau_{orowan} = \frac{0.85\mu b \ln\left(\frac{d}{b}\right)}{2\pi(\omega - d)} \tag{7}$$

With increasing strain several concomitant phenomena should be considered: the size of Al grains decrease from 15 to 4 micron and the Zn particles in the bulk of Al grains increase in size and move to grain boundaries (Figure 6). Therefore, after a certain amount of plastic deformation, it appears necessary to use both the dislocation density and a size parameter, the mean free path of dislocations, to model the mechanical behavior. At the very beginning of plastic deformation when intensive precipitation of Zn particles occurs, it is reasonable to

assume the mean free path (L) to be approximately proportional to the mean distance between precipitates  $(\omega)$ . However, we speculate here that after a few percent of deformation (0.05) the mean free path increases with increasing strain and saturates towards the value of the grain size. These two effects can be considered simultaneously by modifying the previous equation proposed by Gracio [29] to:

$$L = \frac{\lambda \omega}{\lambda - \varepsilon} \tag{8}$$

where  $\lambda$  is a material constant equal to 0.5.

In eqn (8) L is not anymore a variable dependent on the square root of the dislocation density, but rather an independent state variable that increases as a function of strain, as observed experimentally. Such evolution leads to a double effect of L on the softening process (see eqns (3), (6) and (7)). On one hand, when Zn particles move to grain boundaries, the movement of most of the dislocations is facilitated leading to enhanced recovery. On the other hand, when L increases the Orowan strengthening is strongly attenuated according to the right term of eqn (6). The TEM analysis has shown that uniaxial compression produces a strong grain refinement of Zn grains. Namely, after a plastic deformation of 0.25, the Zn grains size decreases to 252 nm. Our microstructural analysis indicates that the occurrence of slip and twinning is drastically reduced as the grain size decreases. This is in agreement with previous works which indicated that the Hall-Petch slope for pure Zn with a grain size between  $10^{-8} - 10^{-6}$  m is much smaller than that for Zn with a grain size in the range  $3 \times 10^{-5} - 4 \times 10^{-4}$  m [30]. In our model we consider, for simplicity, the contribution of the Zn grains to the flow stress to be identical to the yield stress of pure zinc (150 MPa).

The global stress is computed as the sum of the stress associated with the deformation of Al and Zn grains, weighted by the respective volume fractions:

$$\sigma_{total} = (1 - f)\sigma_{Al} + f\sigma_{Zn} \tag{9}$$

The parameters of the model are listed in Table 1.

Figure 7 compares the experimental and calculated results. At the first steps of deformation the model fits well the experimental curve. The theoretical curve predicts a smaller flow stress in the strain softening range. This discrepancy should be due to the omission of the Hall-Petch effect in Zn grains.

The above model should be considered only as a simple approach to describe the softening process during compression of supersaturated Al-30wt% Zn. With this approach it

	Al-Zn
$\mu$ (elastic shear modulus)	26 [GPa]
b (Burgers vector)	0.287 [nm]
$\alpha$ (dislocation-dislocation interaction)	0.33
$\tau_0$ (initial critical resolved shear stress)	30 [Mpa]
$\omega$ (initial distance between Zn precipitates)	300 [nm]
d (initial size of Zn precipitations)	20 [nm]
D (initial grain size of Al grains)	15[ <i>um</i> ]

Table 1 Parameters of the model.

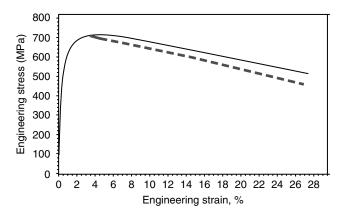


Figure 7 Comparison of the experimental and calculated stress-strain curves.

is not possible to evaluate the separate effects of the different strengthening mechanisms namely solid solution, Hall-Petch, Orowan and dislocation density. Such analysis will be published in a future work.

## 5. CONCLUSIONS

In the present work we present a new microstructure-based model for describing the softening process occurring during compression of supersaturated Al-30% wt Zn alloy. The model describes well the above-mentioned phenomenon based on a new evolution law relating the mean free path of dislocations (L) with plastic strain. Namely L plays a double role on the softening process: on one hand promotes dislocation annihilation and decreasing dislocation density and on the other hand promotes stress relaxation in the bulk of the grains with increasing plastic deformation.

## **ACKNOWLEDGMENTS**

MB, JG and FB acknowledge the financial support of Portuguese Foundation of Science and Technology projects PTDC/EME-TME/105688 / 2008 and PEST-C/EME/UI0481/2011.

#### REFERENCES

- [1] Mazilkin A.A., Straumal B.B., Borodachenkova M.V., Valiev R.Z., Kogtenkova O.A., Baretzky B., Gradual softening of Al–Zn alloys during high-pressure torsion, *Materials Letters*, 2012, 84, 63–65.
- [2] Mukhopadhyay A.K., Kumar A., Raveendra S., Samajdar I., Development of grain structure during superplastic deformation of an Al–Zn–Mg–Cu–Zr alloy containing Sc, Scripta Materialia, 2011, 64, 386–389.
- [3] Cepeda-Jiménez C.M., García-Infanta J.M., Ruano O.A., Carreño F., Mechanical properties at room temperature of an Al–Zn–Mg–Cu alloy processed by equal channel angular pressing, *Journal of Alloys and Compounds*, 2011, 509, 8649–8656.
- [4] Ng H.P., Bettles C.J., Muddle B.C., Some observations on deformation-related discontinuous precipitation in an Al-14.6at%Zn alloy, *Journal of Alloys and Compounds*, 2011, 509, 1582–1589.
- [5] Deschamps A., Texier G., Ringeval S., Delfaut-Durut L., Influence of cooling rate on the precipitation microstructure in a medium strength Al–Zn–Mg alloy, *Materials Science and Engineering*, 2009, 501, 133–139.
- [6] Pucun Bai, Xiaohu Hou, Xiuyun Zhang, Chunwang Zhao, Yongming Xing, Microstructure and mechanical properties of a large billet of spray formed Al–Zn–Mg–Cu alloy with high Zn content, *Materials Science and Engineering*, 2009, 508, 23–27.

- [7] García-Infanta J.M., Zhilyaev A.P, Sharafutdinov A., Ruano O.A., Carreño F., An evidence of high strain rate superplasticity at intermediate homologous temperatures in an Al–Zn–Mg–Cu alloy processed by highpressure torsion, *Journal of Alloys and Compounds*, 2009, 473, 163–166.
- [8] Saray O., Purcek G., Microstructural evolution and mechanical properties of Al-40 wt.%Zn alloy processed by equal-channel angular extrusion, *Journal of Materials Processing Technology*, 2009, 209, 2488–2498.
- [9] Straumal B., Valiev R., Kogtenkova O., Zieba P., Czeppe T., Bielanska E., Faryna M., Thermal evolution and grain boundary phase transformations in severely deformed nanograined Al–Zn alloys, *Acta Materialia*, 2008, 56, 6123–6131.
- [10] Gubicza J., Schiller I., Chinh N.Q., Illy J., Horita Z., Langdon T.G., The effect of severe plastic deformation on precipitation in supersaturated Al–Zn–Mg alloys, *Materials Science and Engineering*, 2007, 460–461,77–85.
- [11] Mazilkin A.A., Straumal B.B., Rabkin E., Baretzky B., Enders S., Protasova S.G, Kogtenkova O.A., Valiev R.Z, Softening of nanostructured Al–Zn and Al–Mg alloys after severe plastic deformation, *Acta Materialia*, 2006, 54, 3933–3939.
- [12] Málek P., Cieslar M., The influence of processing route on the plastic deformation of Al–Zn–Mg–Cu alloys, Materials Science and Engineering, 2002, 324, 90–95
- [13] Wu Y.L, Froes F.H, Alvarez A, Li C.G, Liu J, Microstructure and properties of a new super-high-strength Al–Zn–Mg–Cu alloy C912, *Materials & Design*, 1997, 18, 211–215.
- [14] Deschamps A., Le Sinq L., Bréchet Y., Embury J.D., Niewczas M., Anomalous strain hardening behaviour of a supersaturated Al-Zn-Mg alloy, *Materials Science and Engineering*, 1997, 234–236, 477–480.
- [15] Stolyarov V.V., Latysh V.V., Shundalov V.A., Salimonenko D.A., Islamgaliev R.K., Valiev R.Z., Influence of severe plastic deformation on aging effect of Al-Zn-Mg-Cu-Zr alloy, *Materials Science and Engineering*, 1997, 234–236,339–342.
- [16] Wood J., McCormick P.G., Plastic flow instability in a precipitation hardened Al-Zn-Mg alloy, Acta Metallurgica, 1987, 35, 247–251.
- [17] Kocks U.F., Mecking H., The Physics and Phenomenology of Strain Hardening, *Prog Mater Sci*, 2003, 48, 171–273.
- [18] Lopes A.B., Barlat F., Gracio J.J., Ferreira Duarte J.F., Rauch E.F., Effect of texture and microstructure on strain hardening anisotropy for aluminum deformed in uniaxial tension and simple shear, *International Journal of Plasticity*, 2003, 19, 1–22.
- [19] Barlat F., Ferreira Duarte J.M., Gracio J.J., Lopes A.B., Rauch E.F., Plastic flow for non-monotonic loading conditions of an aluminum alloy sheet sample, *International Journal of Plasticity*, 2003, 19, 1215–1244.
- [20] Hansen N., Distributions of glide systems in cell forming metals. In: Lowe T.C., Rollett A.D., Follansbee P.S., Daehn G.S. (Eds.), Modeling the Deformation of Crystalline Solids, TMS, Warrendale, PA, 1991, 37–49.
- [21] Hansen N., Microstructure and flow stress of cell forming metals, Scripta Metallurgica Materialia, 1992, 27, 947–950.
- [22] Gracio J.J., Barlat F., Rauch E.F., Jones P.T., Neto V.F., Lopes A.B., Artificial aging and shear deformation behaviour of 6022 aluminium alloy, *International Journal of Plasticity*, 2004, 20, 427–445.
- [23] Baudelet B., Deguen M., Felgères L., Parnière P., Rondé-Oustau F., Sanz G., Analyze microstructurelle de l'influence des trajectoires de déformation, Mém. Scient. Revue Metall, 1978, 75, 409–422.
- [24] Rauch E.F., Gracio J.J., Barlat F., Lopes A.B., Ferreira Duarte J., Hardening behavior and structural evolution upon strain reversal of aluminum alloys, *Scripta Materialia*, 2002, 46, 881–886.
- [25] Risebrough N. R., The Deformation Characteristics of Zinc and Cadium, Department of Metallurgy, UBC, 1965.
- [26] Picu R.C., Vincze G., Ozturk F., Gracio J.J., Barlat F., Maniatty A.M., Strain rate sensitivity of the commercial aluminum alloy AA5182-O, *Materials Science and Engineering*, 2005, 390, 334–343.
- [27] Picu R.C., Vincze G., Gracio J.J., Barlat F., Effect of solute distribution on the strain rate sensitivity of solid solutions, *Scripta Materialia*, 2006, 54, 71–75.

- [28] Furukawa M., Horita Z., Nemoto M., Valiev R. Z., Langdon T.G., Fabrication of submicrometer-grained Zn–22% Al by torsion straining, *Journal of Materials Research*, 1996, 11, 2128–2130.
- [29] Gracio J.J., Fernandes J.V., Schmitt J.H., Effect of grain size on substructural evolution and plastic behaviour of copper, *Materials Science and Engineering*, 1989, 118, 97–105.
- [30] Conrad H., Narayan J., Mechanisms for grain size hardening and softening in Zn, Acta Materialia, 2002, 50, 5067–5078.