Effect of Grain Boundary Phase Transitions on the Superplasticity in the Al–Zn system

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1 Introduction

Most important properties of modern materials in high-technology applications are strongly influenced by the occurrence of interfaces such as grain boundaries (GBs) [1]. In the last decade the study of nanocrystalline solids has increased considerably. The inherently high concentration of GBs in these materials makes the understanding of interface properties of great importance. Processes that can modify the properties of the GBs affect significantly the bulk behavior of polycrystalline materials. Among these processes the GB phase transitions can be mentioned as important examples [2–7]. In recent times, GB wetting phase transitions have been included in the traditional equilibrium diagrams of several systems [7–13].

The GB energy, $_{\mathrm{GB}}$, plays a critical role for the occurrence of wetting. Figure 1 shows the contact angle Θ formed between a bicrystal and a liquid phase. When σ_{GB} is lower than $2\sigma_{\mathrm{SL}}$, where σ_{SL} is the energy of the solid/liquid interface, the GB is nonwetted and $\Theta > 0°$ (Fig. 1a). However, the GB is wetted and the contact angle $\Theta = 0°$ if σ_{GB} $2\sigma_{\mathrm{SL}}$ (Fig. 1b). Taking into account the temperature dependences of σ_{GB} and $2\sigma_{\mathrm{SL}}$, where the two curves intersect the GB wetting phase transition will take place upon heating (Fig. 1c). The temperature of the intersection is identified as the wetting temperature, T_{W}, and for every temperature higher than T_{W} the contact angle is $\Theta = 0°$.

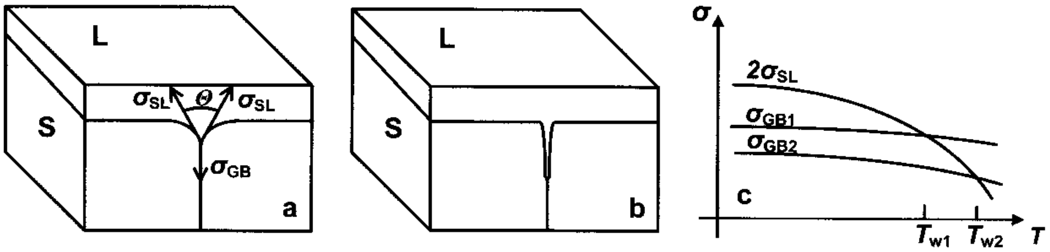

Figure 1: A nonwetted GB in contact with a liquid phase at $T < T_{\mathrm{W}}$, $\Theta > 0°$ (a). A completely wetted GB, $\Theta = 0°$, $T \geq T_{\mathrm{W}}$ (b). Schematic dependences of $\sigma_{\mathrm{GB}}(T)$ and $2\sigma_{\mathrm{SL}}(T)$ for two different GBs. They intersect at the T_{W1} and T_{W2} of the GB wetting phase transition (c). L = liquid; S = solid

The superplasticity has drawn much interest in recent years. Usually this property was observed at relatively low strain rates, typically about 10^{-4} to 10^{-3} s^{-1}. Sometimes it occurs at extremely high strain rates (up to 10^{2} s^{-1}) and in this case it is referred as high-strain rate superplasticity (HSRS) [14–19]. There is agreement that a small grain size is important for the occurrence of HSRS. Additionally, this phenomenon has often been observed at temperatures close to the matrix solidus temperature [20,21]. The phenomenon of HSRS is most pronounced in the Al–Mg–Zn ternary alloys. The reason for HSRS could be the GB phase transitions. Therefore, we decided to study the GB phase transitions in the Al–Mg and Al–Zn binary systems. Recently, the grain boundary wetting phase transition in the two-phase (S+L) region of the Al–Mg system has been investigated [22]. In this work, the occurrence of *prewetting* or *premelting* was given as the reason for the HSRS. In the present contribution, an analogous study has been performed for the Al–Zn system.

2 Experimental

Cylindrical samples (diameter 7 mm) of seven Al–Zn alloys with Zn contents of 10, 20, 30, 40, 60, 75 and 85 wt.% were produced from Al (99.999 wt.%) and Zn (99.995 wt.%). Slices (2 mm thick) of the different alloys were cut and sealed into evacuated silica ampoules with a residual pressure of approximately $4 \cdot 10^{-4}$ Pa at room temperature. Then, several samples were annealed in furnaces for three days at temperatures between 390 and 630 °C, in steps of 20 °C, and subsequently quenched in water. The accuracy in the annealing temperature was ±1 °C. After quenching, the specimens were embedded in resin and then mechanically ground and polished, using 1 μm diamond paste in the last polishing step, for the metallographic study. The samples were then etched and investigated by means of light microscopy.

A quantitative analysis of the wetting transition was performed adopting the following criterion: every GB was considered to be wetted only when a liquid layer had covered the whole GB; if such a layer appeared to be interrupted, the GB was regarded as a nonwetted GB. Accordingly, the percentage of wetted GBs was determined on the basis of light microscopy analysis. At least 100 GBs were analysed at each temperature.

3 Results and Discussion

Figure 2 shows optical micrographs of samples annealed at four different temperatures. As can be seen almost all the GBs have been covered by a liquid layer after annealing at 620 °C (dark layers at the original GBs in Fig. 2a). Upon annealing at 560 °C the number of wetted GBs is considerably lower (Fig. 2b), reaching 66.7 % of the total, whereas in the sample annealed at 480°C this fraction was just 35 % (Fig. 2c). In the samples treated at 440 °C and lower temperatures no GBs were wetted (Fig. 2d). Light gray in the interior of the grain indicates that partial melting has occurred (Fig. 2b and c); however, such a liquid in the solid remains isolated. Pores (see black spots), probably produced by the rapid quenching, can also be observed. The fraction of wetted GBs is shown as a function of temperature in Fig. 3a. A gradual increase of this fraction can be observed between 440 and 620 °C from 0 to 100 %. Therefore, the GB wetting phase transition proceeds in the Al–Zn system between 440 and 620 °C. All GB wetting phase transition tie-lines corresponding to each individual GB lie between these two temperatures. In other words, the minimal (T_{wmin}) and maximal (T_{wmax}) temperatures of GB wetting phase transition in the Al–Zn system are 440 and 620 °C, respectively. Assuming that the bulk solidus and bulk liquidus do also hold for the GB phases at their interface, the respective tie-lines at T_{wmin} and T_{wmax} have been drawn in the two-phase (S+L) region area of the Al–Zn bulk phase diagram (Fig. 3b).

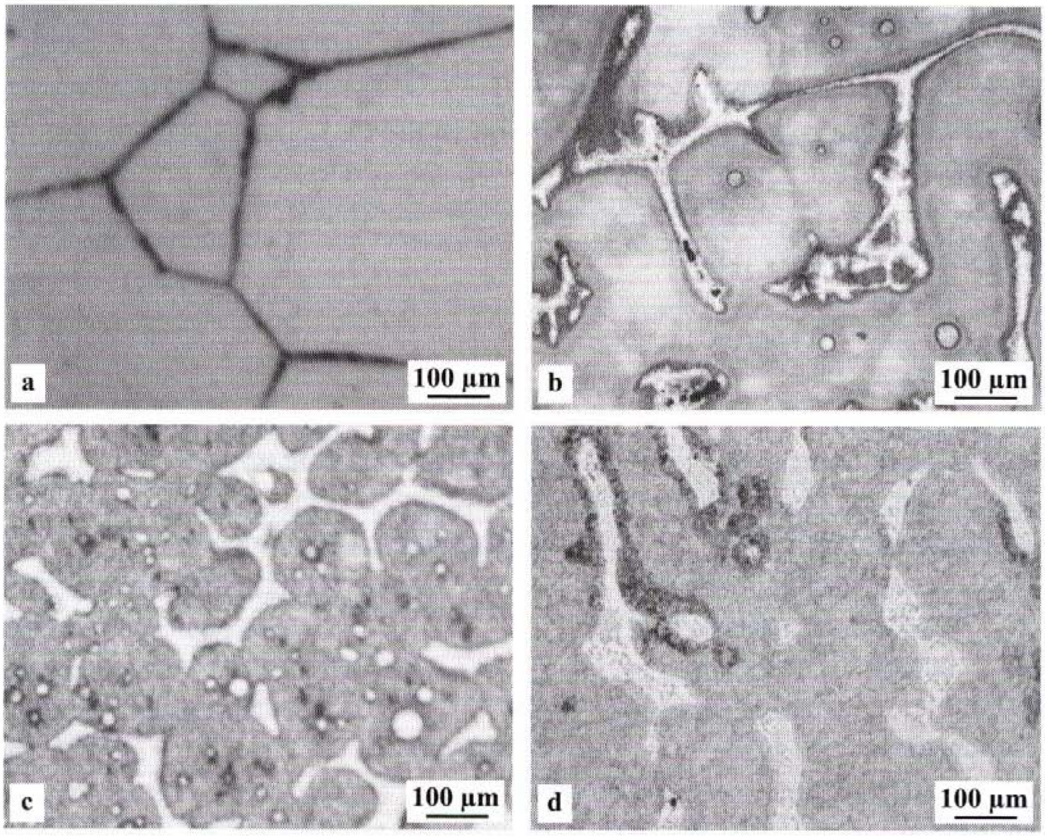

Figure 2: Optical micrographs of samples annealed at 620 °C (a), 560 °C (b), 480 °C (c) and 440 °C (d)

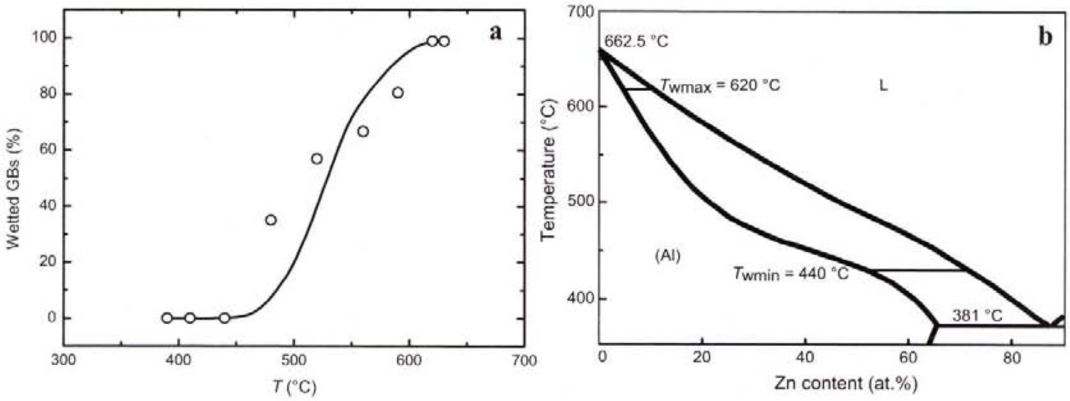

Figure 3: Temperature dependence of the fraction of wetted GBs in the Al–Zn system (a). Al–Zn equilibrium phase diagram with the lines of bulk phase transitions [23] and the tie-lines of GB wetting phase transitions (thin lines) (b)

Taking into account that wetting of GBs has been observed in the bulk (S+L) two-phase region of the Al–Zn system, it can be suggested that a *premelting* or *prewetting* of the GBs may also take place in the S single-phase region near the bulk solidus line. This kind of GB phase transitions has extensively been observed for several systems [2–6, 11, 24, 25] and is interpreted according to the theory developed by Cahn [26, 27]. Due to this phenomenon a thin liquid-like layer can be formed at the GBs.

The presence of a liquid layer at the original positions of the GBs will critically affect the mechanical properties of an alloy, as was observed by Iwasaki et al. in the study of pure shear of a commercial Al–Mg alloy [28].

In several nanostructured Al ternary alloys and nanostructured Al metal-matrix composites, containing Zn and Mg, high-strain rate superplasticity has been observed [16–21, 28–32]. The maximal elongation-to-failure increases drastically from 200–300 % up to 2000–2500 % in a very narrow temperature interval of about 10 °C just below the respective solidus temperature. Until now no satisfactory explanation has been offered for this phenomenon.

Due to the fact that the Al–Zn and Al–Mg systems are the basis of multicomponent alloys which present HSRS, and having observed wetting of GBs for these systems, it is suggested that GB premelting or prewetting is responsible for the HSRS. In that case, a liquid-like thin layer would cover the GBs, leading to an enhanced plasticity of the materials. Considering this hypothesis and using results published on HSRS for Al–Zn–Mg alloys, it could be observed that GB wetting proceeds in multicomponent alloys as well as in binary systems [20,21, 29–32]. From the micrographs published in [20, 21, 29, 30] it was estimated that $T_{wmax} = 535$ °C for the 7xxx Al–Mg–Zn alloys (Fig. 4). At 475 °C about 50 % of GBs are still wetted (see $T_{W50\%}$ in Fig. 4). Unfortunately, the micrographs of the Al–Mg–Zn alloys published in [20, 21, 29, 30] do not permit us to estimate $T_{wmin} < T_{w50\%}$. Comparing the T_w values of multicomponent alloys with those of binary alloys, it seems to be that the presence of third or fourth elements lowers the wetting temperature. But further investigations must be performed to clarify this tendency.

Finally, most of the HSRS tests were performed at temperatures slightly below the bulk solidus and the temperature of maximal elongation-to-fracture is also close to the bulk solidus temperature. Therefore, we conclude that *premelting* or *prewetting* can be the reason for the HSRS in these alloys.

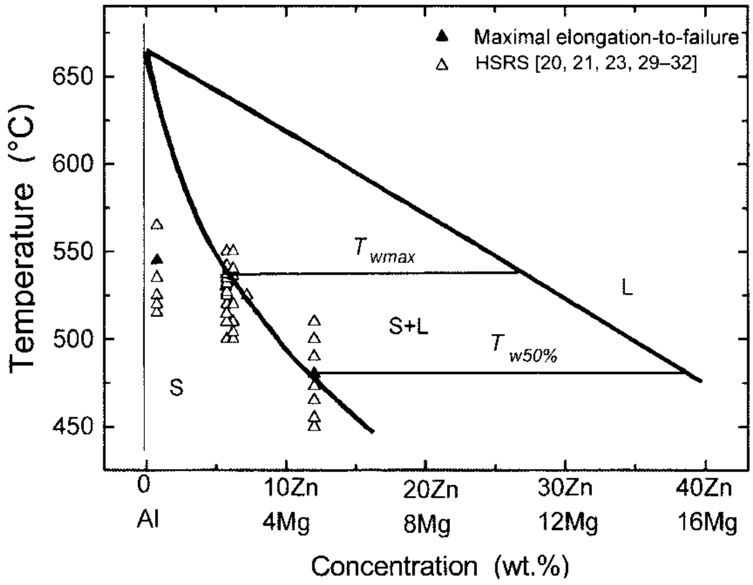

Figure 4: Pseudobinary phase diagram for 7xxx Al–Zn–Mg alloys (thick lines), containing GB wetting phase transition lines (thin lines)

4 Conclusions

Grain boundary wetting has been observed in Al–Zn alloys. On the basis of grain boundary wetting data the maximal and minimal temperatures, T_{wmax} and T_{wmin}, of grain boundary wetting phase transitions have been indicated and the corresponding tie-lines in the two-phase (S + L) region of the respective bulk phase diagram have been drawn. The occurrence of grain boundary prewetting or premelting is considered as the origin of high-strain rate superplasticity.

5 Acknowledgements

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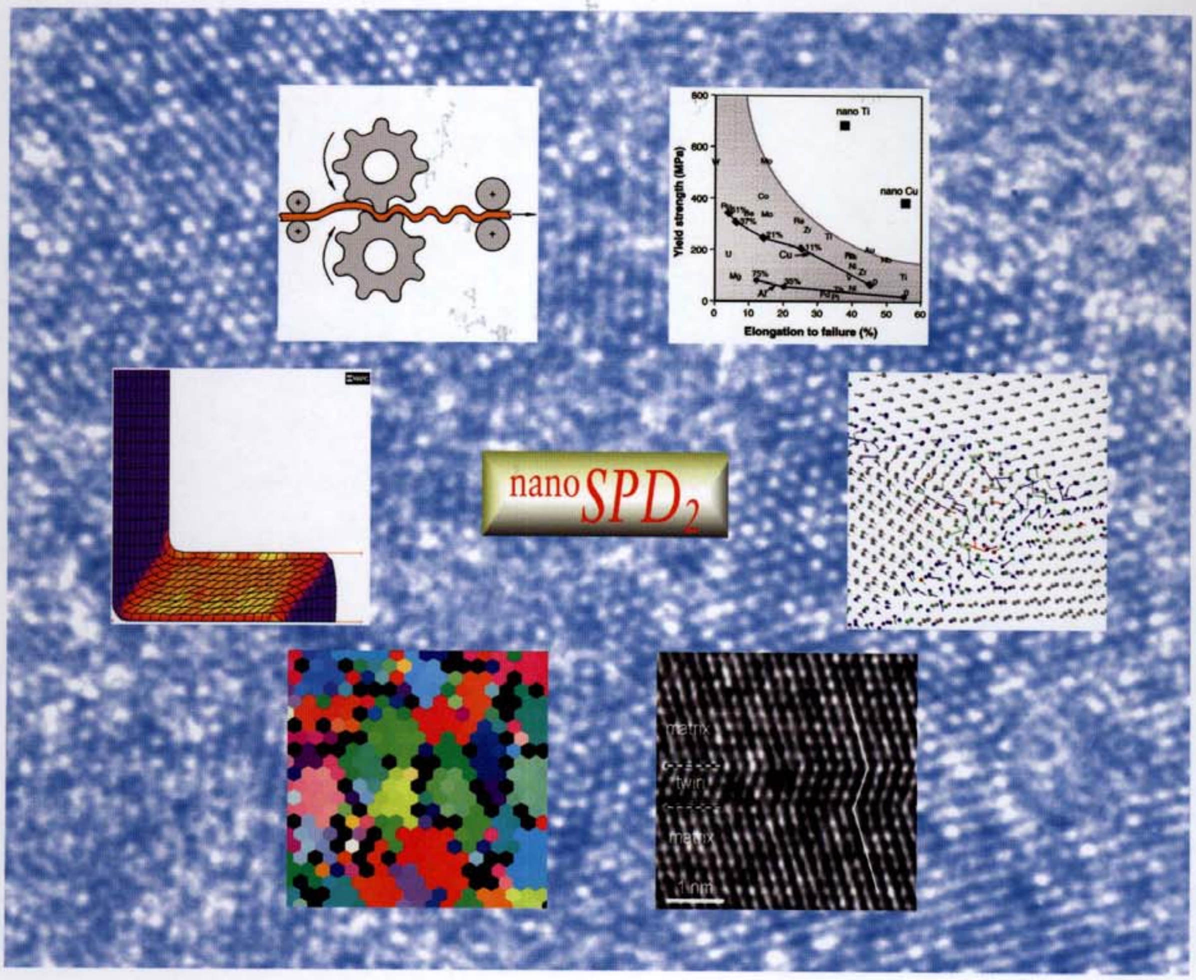
Nanomaterials by Severe Plastic Deformation



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The cover picture symbolizes the various aspects of current research in the field of severe plastic deformation (SPD). All figures have been taken from papers of the present Proceedings of "NANOSPD2".

Bottom left position

Structure of ultrafine and nanograins developed during High Pressure Torsion of Cu as investigated by Electron Back Scatter Patterning (EBSP). With a hydrostatic pressure of 8 GPa, an von Mises equivalent strain of $e = 145$ has been reached. The side length of image is 800 nm. (paper by T.Hebesberger, A.Vorhauer, H.P. Stüwe, R.Pippan, p. 447)

Bottom right position

HRTEM micrograph of HPT deformed Ni3Al (Cr,Zr)+B at room temperature up to a shear strain ~ 800, showing a deformation twin with atomic resolution which is typical of this deformation. The white bar represents a distance of 1nm (paper by Ch.Rentenberger, H.P.Karnthaler, R.Z.Valiev, p. 80)

Right side top

Cold rolling of Cu and Al increases their yield strength but decreases their elongation to failure (ductility). The extraordinary combination of both high strength and high ductility in nanostructured Cu and Ti processed by SPD clearly sets them apart from coarse-grained metals (paper by R. Z. Valiev, p. 109)

Right side middle

Triple junction between 3 grains, which slide significantly relative to each other because of a macroscopic deformation of 1.3 %. Molecular Dynamics Simulation of a full 3D grain boundary network yields atomic displacement vectors with the colour determined by the magnitude of the displacement (paper by H. Van Swygenhoven, P.M. Derlet, A. Hasnaoui, p. 599)

Left side top

Repetitive Corrugation and Straightening (RCS), a new mode of Severe Plastic Deformation to achieve bulk nanomaterials (paper by N.Tsuji, Y.Saito, S.H. Lee, Y. Minamino, p. 479)

Left side middle

Strain intensity and distribution for Equal Channel Angular Pressing (ECAP) as calculated by FEM for Aluminium alloy 5083 using the MARC™ code (paper by P.A.Gonzales, C.J.Luis, p. 251)

Background

HRTEM image of nanocrystalline and amorphous phase in Ni-50.3%-Ti after HPT processing with pressure of 6 GPa (paper by T.Waitz, V.Kazykhanov, R.Z.Valiev, H.P.Karnthaler, p. 351)

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