PLASTIC DEFORMATION IN AN Al-Zn-Mg-Cu ALLOY

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The influence of the grain size on the plastic deformation of the Al-Zn-Mg-Cu alloy was investigated in a broad temperature range. The strengthening effect of grain boundaries was observed at temperatures below 473 K and the softening effect at higher temperatures. The increasing ability of grain boundaries to act as lattice dislocation sinks with increasing temperature is probably the reason for this transition. Superplastic properties were observed at 773 K where grain boundary sliding is the most important deformation mechanism. The existence of a grain-size-dependent threshold stress can explain the increase of the apparent grain size exponent p at low stresses.

PLASTICKÁ DEFORMACE SLITINY Al-Zn-Mg-Cu

Vliv velikosti zrna na plastickou deformaci slitiny Al-Zn-Mg-Cu byl studován v širokém intervalu teplot deformace. Zpevňující účinek hranic zrn byl pozorován u teplot nižších než 473 K, odpevňující účinek u teplot vyšších. S rostoucí teplotou vzrůstající schopnost hranic zrn absorbovat mřížkové dislokace je pravděpodobnou příčinou vzniku tohoto přechodu. Superplastické chování bylo pozorováno u 773 K, kde je nejdůležitějším deformačním mechanismem pokluz po hranicích zrn. Existence prahového napětí, které je závislé na velikosti zrna, může vysvětlit vzrůst zdánlivé hodnoty exponentu p u nízkých deformačních napětí.

1. Introduction

Grain boundaries are important microstructural elements in polycrystalline materials which significantly influence their properties. The role of grain boundaries in plastic deformation depends especially on temperature. At low temperatures, the grain boundaries are barriers to the dislocation motion. Their strengthening effect can be simply expressed by the Hall-Petch relation [1, 2]

$$\sigma_{\mathrm{y}} = \sigma_i + kd^{-1/2}, \tag{1}$$

where σ_{y} is the yield stress, d is the mean grain size and σ_i and k are phenomenological constants. An increase in the deformation temperature within the low temperature range reduces usually the slope of the Hall-Petch relation [3].

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The role of grain boundaries is more complicated at elevated temperatures. In some cases, especially in materials with a very coarse grain size, the same relationship between the stress and grain size was observed at low temperatures [3, 4]. Numerous materials exhibit the high temperature deformation which is independent of the grain size and is controlled by the subgrain size [5]. A completely different behaviour was found in fine grained materials where grain boundaries are carriers of a great part of strain through grain boundary sliding and contribute to the softening, so that an increase in strength with increasing grain size was observed [6]. This behaviour is typical of superplastic deformation and may be described by the modified equation for steady state creep [7]

$$\dot{\varepsilon} = K\frac{D_0 G b}{RT}\left(\frac{b}{d}\right)^p \left(\frac{\sigma}{G}\right)^{1/m} \exp\left(-\frac{Q}{RT}\right), \tag{2}$$

where $\dot{\varepsilon}$ is the strain rate, σ applied stress, T temperature, d grain size, D_0 frequency factor, G shear modulus, b Burgers vector, R gas constant, Q activation energy, m strain rate sensitivity parameter, p grain size exponent and K empirical constant.

The main aim of the present paper was to prepare the Al-Zn-Mg-Cu alloy with different initial grain sizes and to study its mechanical properties and deformation structure in a broad temperature and strain rate range. The resulting grain size dependences of mechanical properties and deformation structure are compared with existing models of plastic deformation.

2. Experimental material and procedure

The chemical composition of the Al-Zn-Mg-Cu alloy is given in Table 1. The castings were pressed into plates and then thermomechanically processed into a recrystallized state. Changing the parameters of the treatment, materials with four different mean grain sizes were obtained (Table 2) [8].

Table 1. Chemical composition in wt.%

Zn	Mg	Cu	Cr	Mn	Fe	Si	Al
6.6	2.3	1.7	0.24	0.24	0.12	0.05	balance

Table 2. Initial mean grain sizes

Material	A	B	C	D
d [μm]	13 ± 1	15 ± 1	31 ± 3	49 ± 4

Tensile tests were performed at the strain rate of 7.2×10^{-4} s^{-1} at temperatures between 293 and 773 K. The tests at elevated temperatures were carried out in a furnace with air atmosphere. The test temperature was stabilized within 1 K and the temperature gradient along the gauge length did not exceed 3 K. The strain rate changes were used to determine the strain rate sensitivity parameter m. The scheme of the experiment is described elsewhere [9]. Some specimens were metallographically polished prior to tensile tests. After straining, the deformation structure was studied using light and scanning electron microscopy.

3. Experimental results

Fig. 1 shows the initial parts of the stress-strain curves measured at different temperatures. An increase in temperature results in a gradual suppression of strain hardening. Nearly steady state curves are observed at temperatures above 473 K. The temperature dependence of the yield stress is not monotonous and exhibits a local maximum at 373 K.

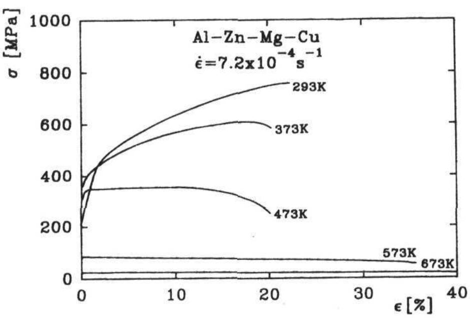

Fig. 1. Initial parts of stress-strain curves in material A.

The ultimate tensile strength R_m and ductility A were chosen as characteristics of the deformation behaviour (Table 3). The grain size dependence of R_m varies with the deformation temperature. As the grain size increases, R_m decreases slightly at temperatures below 473 K, is nearly constant at 473 K, and increases at temperatures above 473 K. Ductility remains nearly constant and grain size independent up to the temperature of 473 K. At higher temperatures, an increase in ductility occurs in finer-grained materials. Superplastic ductilities were observed at 773 K in materials A and B. Similar temperature and grain size dependences were observed for the parameter m.

High temperature deformation is generally strain rate dependent and the grain size effect can also be influenced by the strain rate. To verify this the strain rate change experiments were performed in all materials at 773 K. The resulting log σ vs. log $\dot{\varepsilon}$ curves (Fig. 2) show a great influence of grain size at low and medium strain rates (regions I, II) and a negligible effect at high strain rates (region III). The maximum of the parameter m decreases and shifts to lower strain rates with increasing grain size (Fig. 3). To quantify the grain size effect, the log $\dot{\varepsilon}$ vs. log d curves were plotted for different applied stresses (open symbols in Fig. 4), and the exponent p was determined from their slopes. Fig. 5 (open symbols) shows that p is close to zero at high stresses but increases strongly with decreasing applied stress.

Table 3. Influence of grain size and temperature on the ultimate tensile strength R_m [MPa], ductility A [%] and strain rate sensitivity parameter m

Material T [K]	Properties	A	B	C	D
293	R_m	620	588	580	578
	A	25	21	21	24
373	R_m	519	503	519	478
	A	21	17	21	17
473	R_m	342	342	362	343
	A	21	18	18	22
573	R_m	83	88	91	106
	A	40	45	33	23
	m	0.10	0.10	0.08	0.07
673	R_m	25	26	29	33
	A	133	125	93	67
	m	0.25	0.20	0.16	0.12
773	R_m	2.2	3.4	8.3	14.6
	A	> 300	> 300	93	27
	m	0.65	0.65	0.60	0.34

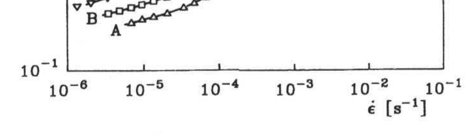

Fig. 2. The $\log \sigma$ vs. $\log \dot{\varepsilon}$ dependences for different grain sizes.

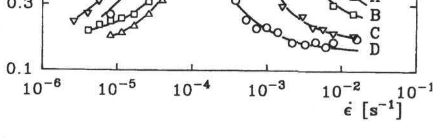

Fig. 3. Strain rate dependence of the parameter m for different grain sizes.

The measured grain size exponent p describes the grain size effect truly only if other parameters appearing in Eq. (2) are grain size independent. Fig. 3 shows that at least in the case of the parameter m this condition is not fulfilled. The p values given in Fig. 5 have to be considered, therefore, as apparent ones. It was

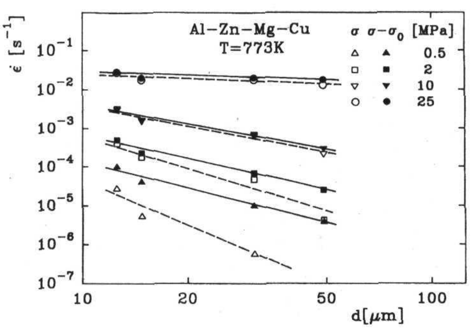

Fig. 4 The $\log \dot{\varepsilon}$ vs. $\log d$ dependences for different applied stress levels σ (open symbols) and different operating stress $\sigma - \sigma_o$ levels (full symbols).

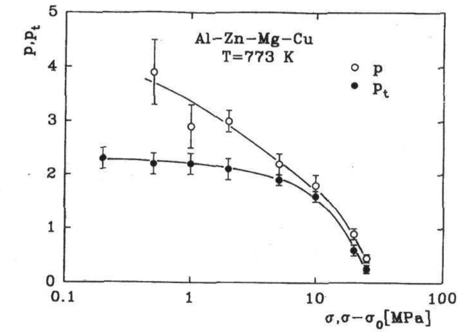

Fig. 5. Stress dependence of the apparent exponent p (open symbols) and true exponent p_t (full symbols).

previously suggested that the unusual behaviour at low strain rates could result from the existence of a threshold stress σ_o [9, 10]. In this case the deformation is not driven by the applied stress but by the stress $\sigma - \sigma_o$ and Eq. (2) can be then rewritten as

$$\dot{\varepsilon} = K\frac{D_0Gb}{RT}\left(\frac{b}{d}\right)^{p_t}\left(\frac{\sigma - \sigma_o}{G}\right)^{1/m_t}\exp\left(-\frac{Q_t}{RT}\right), \quad (3)$$

where m_t, p_t, and Q_t may be considered as the true values of the parameters describing the stress, grain size, and temperature dependence of the strain rate.

The values of σ_o and m_t were determined as those giving the best linear fits of the $\dot{\varepsilon}^m$ vs. σ dependences through regions I and II (Table 4). The quality of these fits is confirmed by a very good linearity of the $\log(\sigma - \sigma_o)$ vs. $\log \dot{\varepsilon}$ dependences in regions I and II without any transients between both regions (Fig. 6). Whereas the threshold stress increases significantly with increasing grain size, the true parameter m_t is influenced weakly. Fig. 7 shows that the main effect is in the shift of the onset of the transient between regions II and III to lower strain rates with increasing grain size.

Table 4. Influence of grain size on the threshold stress σ_o and the true parameter m_t

Material	A	B	C	D
σ_o	0.34	0.42	0.53	1.50
m_t	0.89	0.80	0.80	0.77

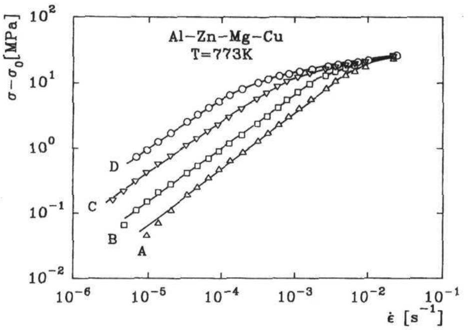

Fig. 6. The $\log(\sigma - \sigma_o)$ vs. $\log \dot{\varepsilon}$ dependences for different grain sizes.

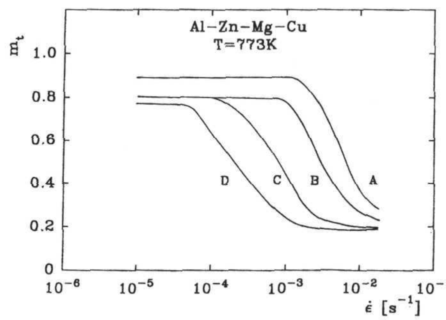

Fig. 7. Strain rate dependence of the true parameter m_t for different grain sizes.

The $\log(\sigma - \sigma_o)$ vs. $\log \dot{\varepsilon}$ curves were intersected at constant $\sigma - \sigma_o$ values. The corresponding $\log \dot{\varepsilon}$ vs. $\log d$ curves are given in Fig. 4 (full symbols) and the resulting true exponent p_t is plotted in Fig. 5 as a function of the operating stress $\sigma - \sigma_o$ (full symbols). Contrary to the apparent exponent p, the true exponent p_t is nearly constant at stresses corresponding to regions I and II and close to the value 2.

4. Discussion

The experimental results given above document clearly that the influence of grain size on mechanical properties, i.e. the role of grain boundaries in plastic deformation, depends significantly on temperature in the Al-Zn-Mg-Cu alloy. Two regions may be distinguished – the "low-temperature" one below 473 K where the grain boundaries contribute to the strengthening and the "high-temperature" one above 473 K where an opposite effect was found. Despite of these differences in the deformation behaviour, both regions can be discussed simultaneously.

The majority of models explaining the low-temperature plastic deformation of polycrystals assume that lattice dislocations (LDs) move on their slip planes within grains and pile up in the vicinity of grain boundaries [11]. The arising stresses can be relaxed by slip in the neighboring grain. Calculating this stress, Eq. (1) can be derived.

The progress in transmission electron microscopy allowed to understand better the processes occurring in the grain boundary. An experimental evidence was obtained that grain boundaries are effective sources of LDs [12]. The mechanism of the LDs generation and emission from the grain boundary is not clear but it is proposed that LDs are formed either by an association of several grain boundary dislocations (GBDs) with small Burgers vectors [13] or by shrinking of the spread core of the trapped lattice dislocation (i.e. of the GBD which was formed by the absorption of an LD in the grain boundary) [12]. Besides being dislocation sources, grain boundaries may also act as LDs sinks. LDs are forced into grain boundaries and form there extrinsic grain boundary dislocations (EGBDs) [14, 15]. Kaibyshev and Valiev [16] measured the densities of both LDs and EGBDs during the cold deformation of an Mg alloy. Whereas a sharp increase in the density of EGBDs and a nearly constant density of LDs was observed at the onset of straining, an opposite result was found at larger strains. This experiment documents clearly that grain boundaries are able to trap LDs even at low temperatures but this ability is limited and, consequently, strain hardening is observed. Such explanation seems to be valid also in our material strained in the "low-temperature" region. The movement of LDs is the main deformation mechanism. This was confirmed by the observation of slip lines in the majority of grains (Fig. 8). The non-monotonous temperature dependence of the yield stress reflects the changes in the phase composition, especially the ageing process above room temperature.

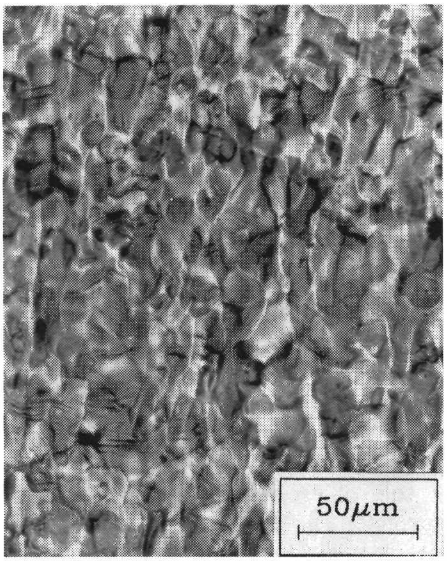

Fig. 8. Surface structure of the material B strained to fracture at 293 K, light microscopy, tensile axis vertical.

The EGBDs may move in grain boundaries by glide or climb, and annihilation processes may take place as deformation temperature increases. Such a recovery mechanism is of great importance especially in fine-grained materials where the formation of a cellular dislocation substructure and, therefore, the occurrence of a recovery inside grains are suppressed. This idea was verified experimentally in the fine-grained 316L austenitic steel [17] where a sharp reduction of the strain hardening coefficient was observed at nearly identical temperature as an increase in the fraction of grain boundaries in which recovery of EGBDs occurred. Varin found that the recovery of EGBDs starts in alloys at temperatures between 0.4 and 0.5 T_m [18]. This agrees well with our results where a change in deformation behaviour was observed at temperatures close to 400 K (0.45 T_m).

The movement of GBDs in the boundary may result in mutual shifts of adjoining grains – grain boundary sliding. The mutual shifts of grains in the direction perpendicular to the rolling plane are documented in Fig. 9a. Fig. 9b reveals that

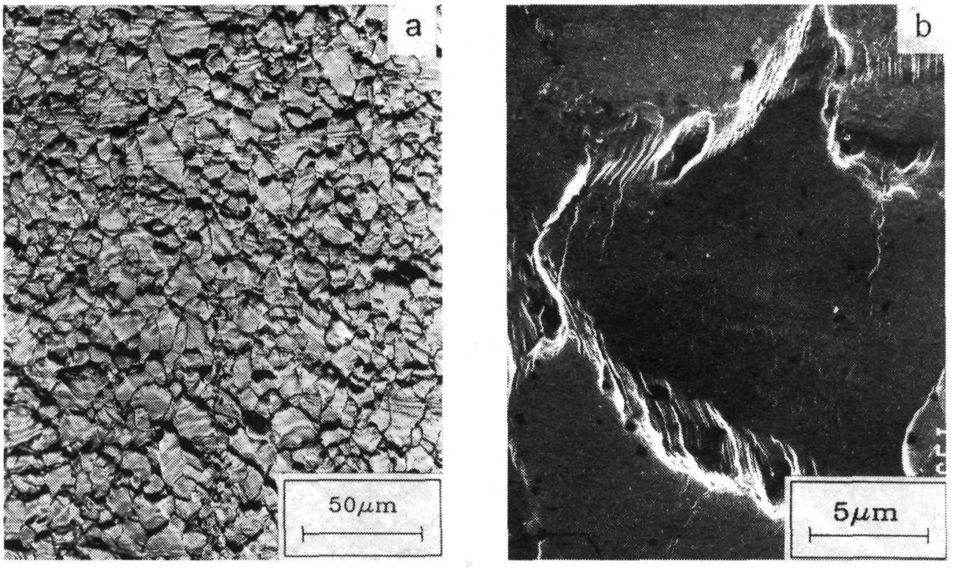

Fig. 9. Surface structure of specimens strained to fracture (tensile axis vertical): a) material B, 573 K, light microscopy, b) material A, 673 K, scanning electron microscopy.

the new surface regions which appear during grain boundary sliding at boundaries perpendicular to the tensile axis exhibit a striated structure. The origin of these "striations" is not yet known. This deformation mechanism was observed in all of our materials strained at temperatures $T > 573$ K. As the grain size decreases the number of grain boundaries increases and an increase in the contribution of grain boundary sliding to the overall strain may be expected. The superplastic behaviour is frequently found under these conditions. Grain boundary sliding needs an accommodation mechanism in order to retain the integrity of the material. A model of superplastic deformation was proposed [19] where lattice dislocation movement serves as this accommodation mechanism. The start of superplastic deformation is considered to be associated with grain boundary sliding, i.e. with the movement of GBDs. The formation of GBD pile-ups facilitates the generation of LDs in grain boundaries. These LDs are emitted from boundaries, cross the adjoining grain, and are absorbed at opposite grain boundaries where they transform into GBDs which in turn cause further sliding.

This model seems to be acceptable at medium strain rates. It predicts the grain size exponent $p = 2$ which is in good agreement with our results obtained at medium strain rates. It fails, however, at high strain rates where the recovery of LDs in grain boundaries is not fast enough. The dynamic recovery inside grains is then more important and the LDs movement brings the largest contribution to the overall strain. This transition results in a lost of superplastic properties, i.e. in a decrease in ductility and parameter m. A decrease of the grain size exponent p to zero is expected. Our results confirm this idea and the deformation is nearly grain size independent at fast strain rates.

As the grain size increases, the number of grain boundaries is reduced. To ensure the strain rate given by the deformation apparatus the rate of grain boundary sliding and recovery of LDs at individual grain boundaries would have to be much larger in the coarser-grained materials than in the finer-grained ones. This is not possible and, therefore, the transition to the non-superplastic region III is expected to shift to lower strain rates with increasing grain size. This expectation was verified in our Al-Zn-Mg-Cu alloy, too (see Fig. 7).

The region of low strain rates is very controversial. A change in the phenomenological parameters m, p, and Q observed at low strain rates may be explained either by the operation of another deformation mechanism (e.g. [20]) or by the presence of a threshold stress (e.g. [10]). The latter approach was chosen in our work. It was shown previously [9] that considering a temperature-dependent threshold stress both the decrease in the apparent parameter m and the increase in the apparent activation energy Q observed in the Al-Zn-Mg-Cu alloy at low strain rates can be eliminated. The resulting true values of m_t and Q_t are nearly identical at both low and medium strain rates which supports the basic idea that the same deformation mechanisms are operating in regions I and II. The same approach was successfully used in the powder metallurgical Al-Zn-Mg-Cu-Zr alloy [21]. Experimental results obtained in the present work enable to test this approach in relation to the influence of grain size. Using Eqs. (2) and (3) a relation between the apparent exponent p and the true exponent p_t can be derived

$$p = p_t + \frac{1}{m_t} \frac{\sigma_o}{\sigma - \sigma_o} \frac{\partial \ln \sigma_o}{\partial \ln d}. \tag{4}$$

Eq. (4) suggests that large differences between p and p_t can arise at low applied stresses if the threshold stress is grain size dependent. Our results revealed a positive slope of the grain size dependence of the threshold stress. Thus, an increase in the apparent exponent p can be expected at very low applied stresses. Such an effect was really observed. The true exponent p_t seems to be nearly stress independent in regions I and II and its value is very close to the value 2 predicted by many models of superplasticity. These results complete our previous results [9] and support the model of a threshold stress for the superplastic flow.

This model is able to describe the superplastic flow phenomenologically but the physical nature of the threshold stress still remains open. It is probable that the threshold stress is connected with processes operating in grain boundaries. However, the existing explanations assuming interactions of GBDs with particles located at grain boundaries [22], with grain boundary ledges [23], or with an atmosphere of impurities segregated at grain boundaries [24] are not able to predict the observed dependences of the threshold stress on temperature and grain size. Further experiments considering the influence of structure on the threshold stress are going on at present.

5. Conclusions

1. An increase in the grain size causes a decrease in the ultimate tensile strength at temperatures below 473 K. Ductilities about 20%, a negligible strain rate sensitivity and moderate strain hardening are typical at these temperatures. The lattice dislocation motion and the limited ability of grain boundaries to act as sinks for lattice dislocations determine the deformation behaviour.

2. A decrease in the ultimate tensile strength with decreasing grain size was observed at temperatures above 473 K. No strain hardening and a gradual increase in ductility and strain rate sensitivity with increasing temperature are typical in this temperature range. The recovery of lattice dislocations in grain boundaries and grain boundary sliding due to the motion of grain boundary dislocations are responsible for this deformation behaviour.

3. The deformation behaviour at 773 K is strongly strain rate dependent. The apparent parameter m reaches its maximum corresponding to superplastic behaviour at medium strain rates and falls both towards high and low strain rates. The apparent grain size exponent p is close to zero at high stresses and increases with a decreasing applied stress.

4. Grain boundary sliding accommodated by the motion of lattice dislocations may be the main deformation mechanism at medium strain rates. The value of the grain size exponent p close to 2 is in accordance with the models of superplastic deformation.

5. The transition to the non-superplastic region at high strain rates reflects probably the limited rate of recovery processes in grain boundaries. The motion of lattice dislocations and dynamic recovery inside grains are probably the controlling mechanisms. A very small grain size effect observed in this region is in accordance with this mechanism.

6. The apparently non-superplastic region I observed at low strain rates can be eliminated if a threshold stress increasing with increasing grain size is considered. The true value of the exponent p_t estimated using this assumption is close to 2 through regions of low and medium strain rates. This is in accordance with the models of superplasticity.

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