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SUPERPLASTIC DEFORMATION OF AN X7093 Al ALLOY

SUPERPLASTIČNA DEFORMACIJA Al-ZLITINE X7093

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We have investigated the superplastic deformation mechanism of a powder-metallurgy, high-zinc X7093 Al alloy. The objective was to examine the rate-controlling mechanisms that govern its superplastic deformation. The investigations were carried out in the temperature range 490–524 °C and strain rates of 4.17×10^{-5} s^{-1} to 2.1×10^{-2} s^{-1}. The maximum ductility was slightly more than 500 % at 524 °C and 4.2×10^{-4} s^{-1}. The values of the stress exponent (n) and the activation energy (Q) indicated that the deformation is rate-controlled by the climb within the grain-boundary diffusion path. The existence of a temperature-dependent threshold stress was confirmed.

Keywords: superplasticity, 7xxx Al alloys, deformation mechanisms

Izvršena je bila študija mehanizma superplastične deformacije Al-zlitine 7093 s cinkom, izdelane po postopku metalurgije prahov. Cilj je bila preiskava mehanizma kontrole hitrosti, ki ureja superplastično deformacijo zlitine. Preiskave so bile izvršene v temperaturnem območju 490–524 °C in hitrostih obremenjevanja $4{,}17 \times 10^{-5}$ – $2{,}1 \times 10^{-2}$ s^{-1}. Maksimalna duktilnost je bila malo nad 500-odstotna pri 524 °C in $4{,}2 \times 10^{-4}$ s^{-1}. Vrednosti napetostnega eksponenta (n) in aktivacijska energija (Q) so pokazale, da je hitrost deformacije kontrolirana s plezanjem po difuzijskih poteh v mejah zrn. Potrjen je tudi obstoj temperaturno odvisnega praga napetosti.

Ključne besede: superplastičnost, 7xxx Al-zlitine, mehanizmi deformacije

1 INTRODUCTION

The ability of crystalline solids to undergo extremely large tensile deformations at elevated temperatures is commonly referred to as superplasticity. At least two requirements have to be fulfilled for superplastic deformation[1]: (i) a very small grain size (typically less then 10 μm) and (ii) a proper combination of temperature (T) and strain rate ($\dot{\varepsilon}$). Superplastic flow can be described by the creep-derived Dorn[2] equation:

$$\dot{\varepsilon} = \frac{AD_0G}{kT}\left(\frac{b}{d}\right)^{\mathrm{p}}\left(\frac{\sigma-\sigma_0}{E}\right)^{\mathrm{n}}\exp\left(\frac{-Q_{\mathrm{ap}}}{RT}\right) \tag{1}$$

where $\dot{\varepsilon}$ is the strain rate, A is a microstructure-dependent coefficient, D_o is the diffusion coefficient, G and E are, respectively, the shear and elastic modulus, k is Boltzmann's constant, while R and T have their usual meanings. The influence of the grain size is described by $(b/d)^p$, where b is the Burger's vector and d is the average grain size, while p is the 'grain size exponent', σ is the flow stress and σ_o refers to the so-called threshold stress, while n is the 'stress exponent' (the reciprocal integer value of strain-rate-sensitivity, m); Q_{ap} is the apparent thermal activation energy for the diffusion process that controls the superplastic deformation. Both n and Q can be simply determined[3] from experimental results:

$$m = \frac{1}{n} = \left(\frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}}\right)_{\mathrm{T}} \quad Q = -R\left(\frac{\partial \ln \dot{\varepsilon}}{\partial (1/\mathrm{T})}\right)_{\sigma} \tag{2}$$

The basic mechanism of superplastic deformation is grain-boundary sliding (GBS)[4] – the cause of the high strain-rate sensitivity m, which is, in turn, responsible for the delayed necking during the tensile deformation. In general, $m > 0.35$ is regarded[5] as the condition for superplasticity. Regardless of the m value, GBS requires some kind of stress accommodation at the triple points of the grain boundaries.[6] It is stated that the rate of accommodation mechanisms controls the overall superplastic strain rate.[7]

Previous studies[8–11] have shown that 7xxx Al alloys can be readily thermomechanically processed to attain superplastic capabilities. The optimum conditions for superplasticity are mostly achieved at testing temperatures of T = 500–530 °C and slow strain rates of about 10^{-4} s^{-1}.[12,13] It is, however, highly dependent on the grain size. Tensile elongations as high as 2000 % have been reported.

The objective of this paper is to characterize the superplastic deformation behavior of the second-generation, powder-metallurgy x7093 Al alloy. Based on strain-rate change tests and superplastic tensile tests, a thermal activation analysis was applied to identify the governing and rate-controlling deformation mechanisms. The alloy used in this research has an average grain size d = 10.7 µm.

2 EXPERIMENT

The material was supplied by the manufacturer in the form of a hot-worked billet. The nominal chemical composition of the alloy is given as follows (in mass fractions, w/%): Zn - 9, Mg – 2.2, Mg – 1.5, Zr – 0.14, Ni – 0.1 and O – 0. 35. Zinc, magnesium and copper are the main alloying elements for 7xxx [14] alloys. A specific feature of this alloy is the exceptionally high level of zinc. Apart from the Zn, the oxygen level, typically for powder-metallurgy alloys, is rather high. Oxygen, as well as zirconium and nickel, forms a series of insoluble dispersoids.

In order to achieve a fine-grained microstructure, a thermo-mechanical treatment, often used for high-strength 7xxx alloys, is utilized in the present study. It was made in four steps: (i) solid-solution treatment at 490 °C followed by water quenching, (ii) over-aging at 400 °C for 16 h, (iii) warm rolling from h_o = 10 mm down to h_f = 1 mm at 200 °C and, (iv) recrystallization at 480 °C for 30 min. The rolling was conducted on a laboratory two-roll high-rolling mill with the rolls preheated at 70 °C. Between the two successive rolling passes, the material was held in a furnace for 5 min at 200 °C. During every pass the thickness reduction was Δh = –1 mm so the accumulated deformation was progressively increasing as the thickness of the plate was reduced. In the final pass, the thickness reduction was –50 %, while the overall accumulated true strain was ε = –2.3.

Flat tensile samples of 8-mm gauge length, typical for superplastic examinations, were machined from the plate with the axis parallel to the rolling direction. Tensile specimens were tested at temperatures of 490–524 °C and the initial strain rates in the range 4.17×10^{-5} s^{-1} to 2.1×10^{-2} s^{-1}. For each test, the force *vs*. elongation was recorded and converted to true stress (σ) – true strain (ε) and the instantaneous true strain rate ($\dot{\varepsilon}$).[15] The tests were conducted using an Instron testing machine with a constant cross-head displacement. The machine was equipped with a three-zone furnace and the temperature was controlled by three independent chromel-alumel thermocouples. The temperature accuracy was held within ± 2 °C.

Two kinds of tensile tests were carried out: (i) continuous elongation-to-failure tests and (ii) strain-rate-change tests. In the second type of tests, the flow stresses were recorded at a number of different cross-head speeds. Initially, the specimens were deformed to ε = 0.15 at the testing temperature to ensure a homogenous deformation. Then, the tests were carried out over small strain-rate increments and repeated several times.

3 RESULTS

The stress dependence of the strain rate, under isothermal conditions, is shown in the double logarithmic plot of **Figure 1**. An apparent linear dependence can be observed within the range of the applied strain rates and temperatures. The experimental data were fitted with the least-squares procedure using Equation (3):

$$\sigma = K\dot{\varepsilon}^{m} \Rightarrow \log\sigma = \log K + m\log\dot{\varepsilon} \tag{3}$$

where K denotes the strength coefficient (e.g., $K = \sigma$ when $\dot{\varepsilon}$ = 1 s^{-1}), all other symbols are already mentioned. The results of the analysis are inserted into **Figure 1**. Both m and K are reciprocally influenced by the temperature and the applied strain rate. The

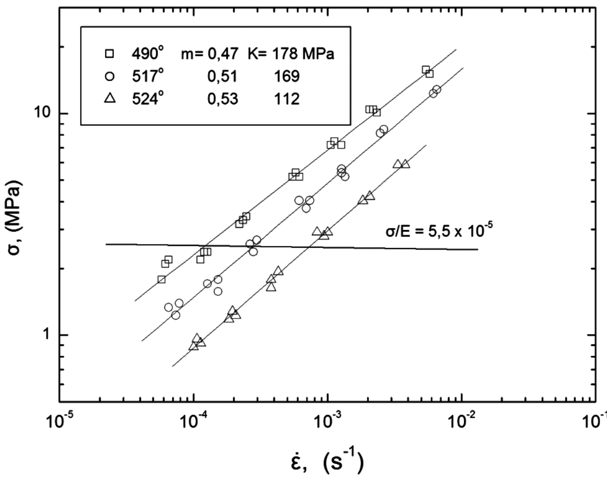

Figure 1: The strain rate as a function of the stress, double logarithm scale. The results of the least-square analysis are inserted into the plot.

Slika 1: Hitrost obremenjevanja kot funkcija napetosti, dvojna logaritemska skala. Rezultati analize najmanjših kvadratov so vstavljeni v diagram.

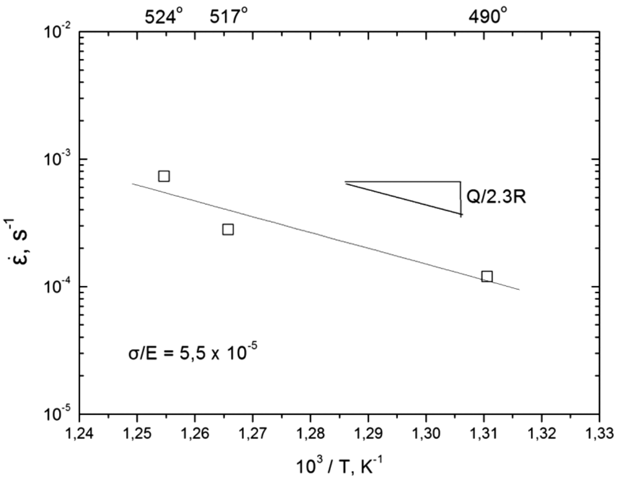

Figure 2: The apparent activation energy vs. the deformation. Indicated stress corresponds to the full line in **Figure 1**.

Slika 2: Navidezna aktivacijska energija proti deformaciji. Prikazana napetost ustreza polni liniji na **sliki 1**.

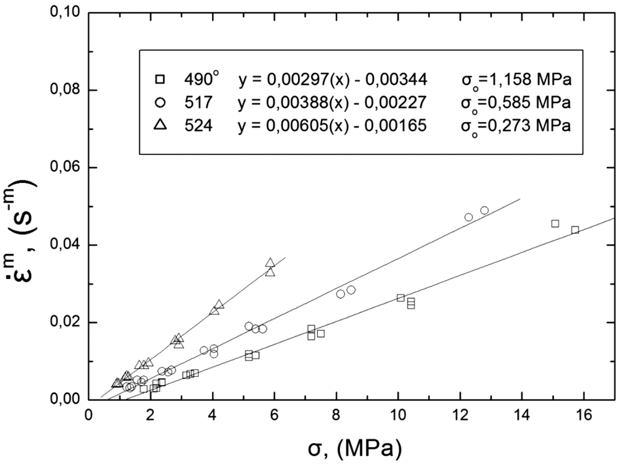

Figure 3: The strain rate as a function of the stress. The least-squares analysis for the threshold stress determination.

Slika 3: Hitrost obremenjevanja kot funkcija napetosti. Analiza najmanjših kvadratov za določitev praga napetosti.

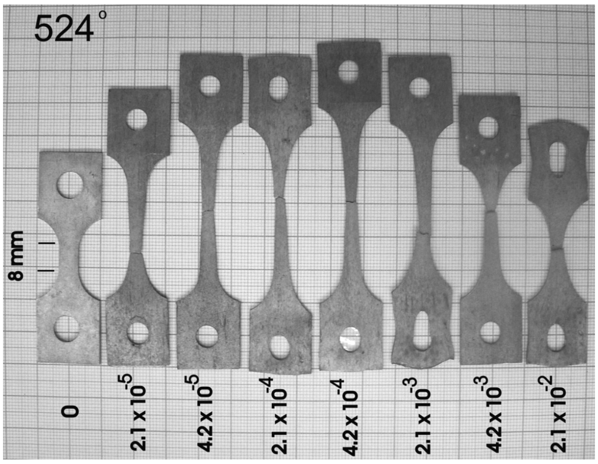

Figure 5: Specimens continuously (stress-strain) tested at 524 °C with various applied strain rates

Slika 5: Vzorci, kontinuirno preizkušeni (napetost-hitrost) na 524 °C z uporabljenimi različnimi hitrostmi obremenjevanja

calculated m-values can be regarded as being typical for the superplastic behavior of Al alloys.

The apparent activation energy for the deformation, Q_{ap}, was determined by plotting the logarithm of strain rate *vs.* 1/T at a constant stress level, i.e., the modulus-normalized stress $\sigma/E = 5.5 \times 10^{-5}$. Such a plot is depicted in **Figure 2**. The straight line, obtained by the least-squares method, reveals a slope equal to $Q/(2.303\mathrm{R})$. The calculated value of Q is 95 kJ/mol. This is close to the value of the activation energy for grain-boundary diffusion in aluminum (84 kJ/mol) and far less than for the lattice diffusion (142 kJ/mol).[16] The same results (with scatter of ± 4 kJ/mol) were calculated for other stress levels (in the range of σ = 2–10 MPa), indicating that Q is not stress dependent.

Among the phenomena governing superplastic deformation, the existence and origins of the threshold stress (σ_o) are the subjects of wide dispute.[17–19] Experimentally, the threshold stress can be determined by plotting $\dot{\varepsilon}^{1/n}$ *vs.* σ (with n being true-stress exponent) and linearly extrapolating values to zero strain rate. Such a plot, as shown in **Figure 3** for $n = 2$, reveals that the threshold stress exists in this alloy and that the values decrease with increasing temperature. The existence of the threshold stress implies that the superplastic deformation is driven by the effective stress $\sigma_{eff} = \sigma - \sigma_o$, rather than by the nominal applied stress.

After rearranging Dorn's equation (Eq.1) into the dimensionless, normalized form, it can be used to further clarify the superplastic deformation behavior. Tensile test data are plotted in **Figure 4** as $[\dot{\varepsilon}kT/(D_{GB}Gb)]$ *vs.* $(\sigma - \sigma_0)/E$. Here, D_{GB} refers to grain-boundary diffusion, $D_{GB} = 5\times10^{-14} \exp(84.000/8.314T)$ (m³/s), $G = 3.0 \times 10^4 - 16T$ MPa, $E = 2G(1 + \nu)$, $b = 2.86 \times 10^{-10}$ m and $k = 1.381 \times 10^{-23}$ J/K. Several features on this plot deserve to be emphasized: all the experimental data merged into a

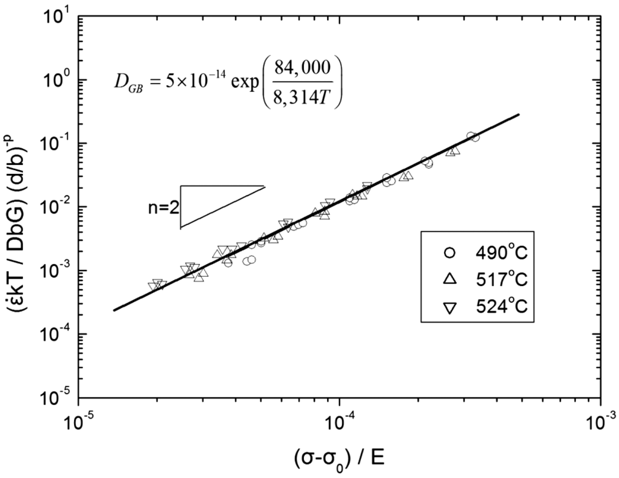

Figure 4: The normalized strain rate vs. the normalized effective stress

Slika 4: Normalizirana hitrost obremenjevanja proti normalizirani efektivni napetosti

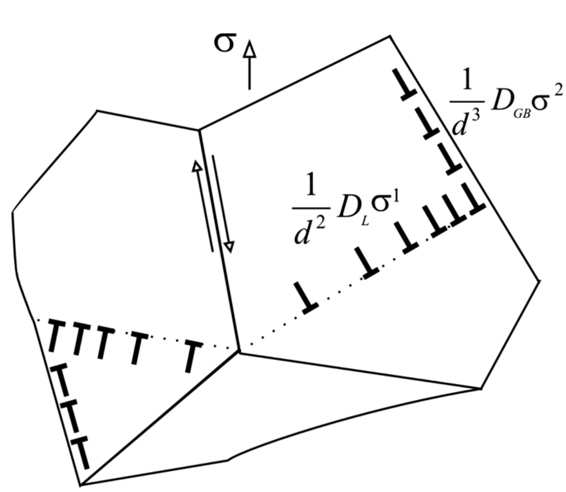

Figure 6: The sketch of grain-boundary sliding accommodated by dislocation glide and climb

Slika 6: Shema drsenja meje zrna zaradi drsenja in plezanja dislokacij single line, thereby proving that the activation energy and the threshold stress are correctly determined[20]; the slope of the line confirms the stress dependence $\dot{\varepsilon} \propto \sigma^2$, i.e., the stress exponent n = 2; the experimental data reveal the best fit with the grain size dependence $\dot{\varepsilon} \propto d^{-3}$.

In accordance with the determined strain-rate sensitivity (m), elongation-to-failure tests have revealed superplastic behavior with moderate elongations in the range 100–500 %. The best superplastic properties were observed at 524 °C. **Figure 5** shows a set of tensile specimens tested at this temperature. The highest elongation, 525 %, was achieved at the strain rate 4.2×10^{-4} s^{-1}. However, rather large elongations were observed across a very broad spectrum of strain rates –4.2×10^{-5} to 4.2×10^{-3} s^{-1} , which is two orders of magnitude. This can perhaps be attributed to the presence of insoluble dispersoids that inhibit grain growth during deformation.[21–23] In addition, it can be observed that all the specimens exhibit no-necking.

4 DISCUSSION

Over past decades, considerable efforts in physical metallurgy have been focused on superplasticity.[24] A number of mechanisms (and speculations) have been proposed to explain the mechanisms and kinetics of the process. Nevertheless, a consensus was attained long ago that superplasticity is essentially a grain-boundary sliding phenomenon.[25] Dispute has been raised about the nature of the accommodation processes that relieve the stress concentrations at the grain boundaries. Stress relief by diffusional flow was proposed as a 'grain switching' mechanism accommodated by diffusion mass transfer.[26] The model predicts m = 1, which is far higher than the results in **Figure 1**. The majority of superplastic aluminum alloys, within the appropriate temperatures and strain rates, exhibit n = 2 ($m \approx 0.5$) behavior, as also confirmed in this paper. Furthermore, it was suggested that at stresses higher than $\sigma > 10^{-5}$E, accommodations by slip are a more probable mechanism.[27] Based on accommodation by dislocation slip, quite a few theoretical models have been proposed.[28–33] Despite the distinctions based on a particular dislocation configuration during deformation, all the models describe a rate-controlling equation in the form of Eq.1. The differences are reflected in the values of n, Q and p. In short, the differences can be summarized as follows. The stress exponent is related to the dislocation movement through the glide and climb. When the glide is rate-controlling (class I solid-solution alloys), the stress exponent n = 1 [34,35] or 3.[36] In this case, the activation energy for the deformation is expected to be close to the lattice self-diffusion D_L or chemical diffusivity D_{chem}. When climb is the rate-controlling mechanism (typically for class II solid-solution alloys), the stress exponent n = 2 [6] and grain-boundary diffusion D_{GB} is prevailing. However, it should be stressed that during the deformation, both glide and climb take place. These are sequential processes,[4] meaning that glide happens before climb. Thus, the slower one will determine the rate of grain-boundary sliding:

$$\frac{1}{\dot{\varepsilon}_{total}} = \frac{1}{\dot{\varepsilon}_{glide}} + \frac{1}{\dot{\varepsilon}_{climb}} \tag{4}$$

Only when $\dot{\varepsilon}_{climb} >> \dot{\varepsilon}_{glide} \Rightarrow \dot{\varepsilon}_{total} \approx \dot{\varepsilon}_{climb}$, as is the case with the presented results.

The climb itself is diffusion driven, both by lattice and grain-boundary diffusion. These are parallel, independent processes and the faster one is rate-controlling. If the lattice diffusion is faster, the grain-size exponent should be p = 2.[1,37] If the rate of grain-boundary diffusion is a controlling process, then p = 3.[38,39] (It is necessary to mention the superplastic analogy to Nabarro-Herring *vs.* Coble diffusional creep[1,4,5]). In symbolic notation, the model can be set up as in Eq.5, which seems to properly explain the deformation behavior of the investigated alloy:

$$\dot{\varepsilon}_{climb} >> \dot{\varepsilon}_{glide} \Rightarrow \dot{D}_{GB} > \dot{D}_{L} \Rightarrow \dot{\varepsilon}_{total} \propto \frac{1}{d^3} \dot{D}_{GB} \sigma^2 \tag{5}$$

Equation 5 envisages sequential strain rates of glide and climb as well as the diffusion rates (D-dotted) of the lattice and grain-boundary path. The sketch of the proposed model, based on ref.[25,27–30], is plotted in **Figure 6**. In brief, grain-boundary sliding is impeded at the triple grain boundaries. It generates local stress concentrations and, at some critical intensity of resolved shear stress, dislocation slip is initiated. The emission of new dislocations relieves stresses at the triple points and the GBS continues. Generated dislocations further glide on favorable slip systems and pile-up at the opposite side of the grain. At some level of dislocation pile-up, the climb kicks off and initiates the dynamic recovery. As a final consequence, the overall superplastic strain rate is controlled by the dynamic recovery, which is, *ipso facto*, dependent on the climb rate along the grain-boundary diffusion path.

5 CONCLUSIONS

The superplastic behavior of a high-zinc aluminum x7093 alloy was investigated. The calculated values of the stress exponent (n = 2), strain-rate sensitivity (m = ≈0.5), thermal activation energy for deformation (Q – close to activation energy for grain-boundary diffusion) and grain size exponent (p = 3) suggest that the superplastic strain rate is controlled by the rates of climb and grain-boundary diffusion. A rate-dependent deformation model was proposed.

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6 REFERENCES

[1] T. G. Nieh, J. Wadsworth, O. D. Sherby, Superplasticity in Metals and Alloys, Cambridge University Press, Cambridge 1997
[2] J. Harper, J. E. Dorn, Acta Metall., 5 (**1957**), 654
[3] J. P. Pilling, N. Ridley, Superplasticity in Crystaline Solids, The Institute of Metals, London 1989
[4] T. H. Courtney, Mechanical Behavior of Materials, McGraw-Hill, London 1990
[5] T. G. Langdon, The interpretation of creep mechanisms in high Temperature Flow, In: E. M. Taleff, C. K. Syn, D. R. Lesuer (Eds.), Deformation, Processing and Properties of Structural Materials, TMS, Warrendale, 2000, 109
[6] O. D. Sherby, E. M. Taleff, Mat. Sci. and Eng., A322 (2002), 89–99
[7] J. C. M. Li, A. K. Mukherjee (Eds.), Rate Processes in Plastic Deformation of Materials, Proceedings of the John E. Dorn Symposium, Metals Park, 1975
[8] F. C. Liu, Z. Y. Ma, Scripta Mater., 58 (**2008**), 667
[9] R. B. Vastava, T. G. Langdon, Acta Metall., 27 (**1979**), 251
[10] P. Shariat, R. B. Vastava, T. G. Langdon, Acta Metall., 30 (**1982**), 285
[11] F. A. Mohamed, S. A. Shei, T. G. Langdon, Acta Metall., 23 (**1975**), 1443
[12] P. K. Chaudhury, F. A. Mohamed, Acta Metall., 36 (**1988**), 1099
[13] P. K. Chaudhury, V. Sivaramakrishnan, F. A. Mohamed, Metall. Trans., 19A (**1988**), 2741
[14] J. A. Wert, N. E. Paton, C. H. Hamilton, M. W. Mahoney, Metall. Trans., 12A (**1981**), 1267
[15] G. E. Dieter, Mechanical Metallurgy, McGraw-Hill, London 1988
[16] H. Frost, M. F. Ashby, The Plasticity and Creep of Metals and Ceramics, Pergamon Press, Oxford 1982
[17] P. K. Chaudhury, F. A. Mohamed, Acta Mater., 36 (**1988**), 1099
[18] G. S. Murty, M. Koczak, Mat. Sci. Eng., 100 (**1988**), 37
[19] Y. Li, T. G. Langdon, Scripta Mater., 36 (**1997**), 1457
[20] A. K. Mukherjee, T. B. Bieler, A. H. Chokshi, Superplasticity in Metals and Ceramics, 10th Riso International Symposium om Metallurgy and Materials Science, Roskilde, Dennmark, 1989
[21] D. W. Kum, W. J. Kim, G. Frommeyer, Scripta Mater., 40 (**1999**), 223
[22] K. Matsuki, G. Stanick, H. Natagawa, M. Tokizawa, Z. Metallkde., 79 (**1988**), 231
[23] W. J. Kim, K. Higashi, J. K. Kim, Mater. Sci. Eng., A260 (**1999**), 170
[24] T. G. Langdon, Seventy five years of superplasticity: historic developments and new opportunitie, J. Mater. Sci., 44 (**2009**), 5998
[25] A. Ball, M. M. Hutchison, Metal. Sci. J., 3 (**1969**), 1
[26] M. F. Ashby, R. A. Verrall, Acta Metall, 21 (**1973**), 149
[27] O. D. Sherby, J. Wadsworth, Mat. Sci. Tech., 1 (**1985**), 925
[28] R. C. Gifkins, Strength of Metals and Alloys, Pergamon Press, Oxford 1982
[29] A. K. Mukherjee, Mater. Sci. Eng., 8 (**1971**), 83
[30] R. C. Gifkins, Metall. Trans., 7A (**1976**), 1225
[31] J. H. Gittus, J. Eng. Mat. Tech., 99 (**1977**), 244
[32] A. Arieli, A. K. Mukherjee, Mat. Sci. Eng., 45 (**1980**), 61
[33] V. Paidar, S. Takeuchi, Acta. Metall., 40 (**1992**), 1773
[34] O. D. Sherby, P. M. Burke, Prog. Mater. Sci., 13 (**1968**), 325
[35] H. Fukuyo, H. C. Tsai, T. Oyama, O. D. Sherby, Iron Steel Inst, 31 (**1991**), 76
[36] J. Weertman, Trans. ASM, 61 (**1968**), 681
[37] G. Krauss (ed.), Deformation Processes and Structure, ASM, Metals Park, 1984, 355
[38] C. H. Hamilton, N. A. Paton (ed.), Superplastic forming of structural materials, The Metallurgical Society of AIME, Warrendale, 1982, 241
[39] G. Frommeyer, W. Kowalski, R. Rablbauer, Met. and Mater. Trans., 37A (**2006**), 3511