ARCHIVES OF METALLURGY

Volume 47 2002 Issue 1

JANUSZ KRÓL*, MARTA TAŁACH-DUMAŃSKA*

THE SUPERPLASTIC STATE IN A7475 ALLOY RELATED TO THE STRUCTURE AND MECHANICAL PROPIERTIES

STRUKTURA I WŁASNOŚCI MECHANICZNE STOPU A7475 W STANIE NADPLASTYCZNYM

The superplastic deformation process is interesting not only from the scientific point of view but also because of benefits it offers when applied in industry. The generation of superplastic structures in aluminium alloys of the 7XXX type depends on the formation of precipitates in suitable amount, size and dispersion which may be obtained by thermomechanical treatment. The A7475 alloy was continuously cast, extruded and thermomechanically treated. The investigations on creating precipitates and microstructure of the AlZnCuMgCr alloy were performed using optical, scanning and transmission electron microscopy and X-ray phase analysis. The morphology of the alloy after a full thermomechanical treatment showed fine, equiaxial average grain about 12 μm in size. The strain rate sensitivity coefficient m determined from tensile tests was estimated between 0.56–0.70. About 495 pct elongation was obtained in a tension test at the flow stress of 3 MPa, strain rate $8 \times 10^{-3}s^{-1}$ and temperature 790 K. It demonstrates that material of good superplastic properties was elaborated.

Wytworzenie struktury superplastycznej w stopach aluminium typu 7XXX jest zależne od powstania wydzieleń o odpowiedniej wielkości, dyspersji i ilości, co może zostać uzyskane na drodze obróbki termomechanicznej.

Stop A7475 odlewany metodą ciągłą i wyciskany, był następnie obrabiany w procesach termomechanicznych. Badania tworzących się wydzieleń i mikrostruktury w stopie AlZnCuMgCr przeprowadzono metodami mikroskopii optycznej, skanningowej i elektronowej oraz rtg. analizy fazowej. Morfologia stopu po pełnej obróbce termomechanicznej charakteryzowała się drobnymi, równoosiowymi ziarnami o wielkości około 12 μm. Współczynnik czułości na szybkość odkształcenia określony w teście rozciągania osiągnął wartość $m = 0{,}56-0{,}70$. Maksymalne wydłużenie uzyskane w teście rozciągania przy naprężeniu płynięcia ok. 3 MPa, szybkości odkształcenia $8 \times 10^{-3}s^{-1}$ i w temperaturze 790°K osiągnęło wartość 495%. Świadczy to o uzyskaniu materiału o dobrych własnościach superplastycznych.

Keywords: superplasticity, thermomechanical treatment, elongation, strain rate, flow stress.

* INSTYTUT METALURGII I INŻYNIERII MATERIAŁOWEJ IM. A. KRUPKOWSKIEGO PAN, 30-059 KRAKÓW, UL. REYMONTA 25.

1. Introduction

Designers of new materials share deep interest in alloys which reveal superplastic properties. The problems of the formation of superplastic state in the alloys are very interesting considering not only the scientific but also the industrial application aspects. The superplastic deformation allows to produce elements of complicated shapes in only one operation and at much lower stresses than in the conventional deformation processes. Superplasticity can be defined as the ability of alloys to become deformed to a very high degree at low flow stress. The principal mode of superplastic deformation is the grain boundary sliding (gbs) which can occur continuously only in accomodation processes depending on the deformation rate and temperature [1–4]. According to the main deformation mechanism (gbs) a significant improvement in the superplastic properties of the alloy can be achieved through the refinement of the grain size and the nearly equiaxial shape of the grains of the microstructure which should be stable at high temperatures [5]. This type of structure can be obtained in high strength aluminium alloys by different types of thermomechanical treatment (TMT) [6–10]. Changes of grain size achieved in the TMT processes have been attributed to differences in solute elements Zn, Mg and Cu [11] and to the amount and distribution of the fine Cr – bearings dispersoids [12]. Generally, thermomechanical treatment comprises three stages: heat treatment, plastic deformation and recrystallization. The important factors of the first stage are temperature and duration of the supersaturation and overageing (depending strongly on the history of the alloy). At the second stage the degree and temperature of plastic deformation are very important. The degree of deformation should be high (80–90 pct) and the temperature near 293 K or below zero [13]. Recrystallization (the third stage), should be performed at high temperature and within a relatively short time (less than 30 min.). The very important factor at this stage is the heating rate which should be as high as possible [14].

In the alloy of the 7XXX type, large, binary (Al–Zn, Al–Cu, Mg–Zn), ternary (Al–Zn–Mg, Al–Mg–Cu) precipitates and very fine chromium dispersoids are formed in the as cast state [6, 10]. According to Nes [15] the small particles decrease the nucleation rate with increasing f/r ratio (f – volume fraction and r – radius of the particles) while the large particles increase the density of nucleation sites at the recrystallisation stage [16]. During the supersaturation process, at the homogenization temperature, the large precipitates dissolve but the dispersoids remain in the alloy. In the overaged state the "large" precipitates [$MgZn_2$; M (a mixture of $MgZn_2+Al_2CuMg$) – and T (a mixture of $Al_2Mg_3Zn_3+Al_6CuMg$) – phases] are formed in the alloy [17]. During plastic deformation, the deformation zones and the shear bands (at high degree of deformation), appear [9, 13] around the "large" (above 2 μm) precipitates. The density of these shear bands depends on the amount of solute left in the solid solution after overageing [9]. During recrystallization, high-rate nuclei formation in these zones and bands takes place, producing very fine structure [16]. The dispersoids limit the grain growth by hindering the migration of grain boundaries and decrease the recrystallization rate by exerting the drag force on the migrating boundaries [6, 13]. The dispersoids, present in the grain boundaries, hinder the grain growth and stabilize the fine-grain structure during superplastic deformation at high temperature. Following the thermomechanical treatments (with modifications) stable, fine and nearly equiaxial, grained structure in the A7475 alloy was obtained.

2. Experimental procedure

2.1. Thermomechanical treatments of the investigated alloy

The superplastic structure was obtained in the thermomechanical treatments:

– type "*A*" – supersaturation (763 K/5 hours) and water quench + overageing (673 K/8 hours) and water quench + plastic deformation (rolling up to 85 pct. at room temperature) + recrystallization (753 K/0.5 hours),

– type "*B*" – homogenization (753 K/8 hours) + cooling at the rate of 22 K/min to 688 K + 688 K/5 hours + cooling at the rate of 14 K/hour to 523 K + 523 K/4 hours and water quench + plastic deformation (rolling up to 85–90 pct. at room temperature) + recrystallization at 753 K for 0.5 hour,

The recrystallization was performed in a salt furnace to obtain high enough heating rates (about $10^2 Ks^{-1}$).

2.2. Material and methods of investigation

The A7475 ingot (continuously cast) of the composition: 5.85 Zn, 1.65 Cu, 2.40 Mg, 0.20 Cr, 0.02 Ti, (Si+Fe)<0.1 and Al – balance (all in wt. pct.), was homogenized and extruded. This material was then thermomechanically treated in *A* and *B* routes.

Structure studies were carried out using:

– PHILIPS CM 20 transmission electron microscope. Thin foils for transmission electron microscope (TEM) studies were produced by jet electropolishing in methyl alcohol/nitric acid solution and ion beam thinning using Gatan 660 ion mill. PHILIPS XL – 30 scanning electron microscope for the micrographs and quantitative analysis of the precipitates and dispersoids with the energy dispersive spectrometry (EDS) method. Light microscope NEOPHOT, on the samples prepared by etching of the mechanically polished surface with the Wilcox solution. *X*-ray PHILIPS PW 1710 diffractometer for the *X*-ray phase analysis of the precipitates and matrix on the samples after different stages of the thermomechanical treatment. The CoK_α filtered radiation was used. INSTRON 6025 testing machine was used to determine the superplastic characteristics of the samples in a tensile test. The tensile tests were performed at the temperature 790 K at strain rates between $8.3 \times 10^{-4} s^{-1}$ to $1.67 \times 10^{-1} s^{-1}$. Tensile specimens, 16 mm in gauge length and 3.5 mm in width, cut from 2 mm thick sheets after the whole TMT process, were used. The m coefficient was obtained from the stress vs strain rate dependence.

3. Results and discussion

3.1. Light microscopy analysis

The structure of the investigated alloy in the as cast state, after extrusion and homogenization and after the A and B of TMT was examined by the light microscopy method. Large grains (150–250 μm) with high amount of precipitates on their boundaries were observed in the alloy after casting. Much smaller grains with distinctly lower amount of precipitates were found after the extrusion and homogenization. The superplastic structure with fine (about 15 μm) equiaxial grains was observed in Figures 1 (after *A* treatment) and 2 (after *B* treatment). The differences between structure after *A* and *B* treatments were small, the grain size the same, although the shape of the B grains was slightly elongated. It was clearly seen on the microstructures of higher magnification that the morphology was composed of grains of two kinds – the large ones about 15 μm and the small ones, below 7 μm in size, as it is also seen on the microstructures given in the papers [5, 7, 13]. The average grain size in the both samples mwas estimated at 12 μm.

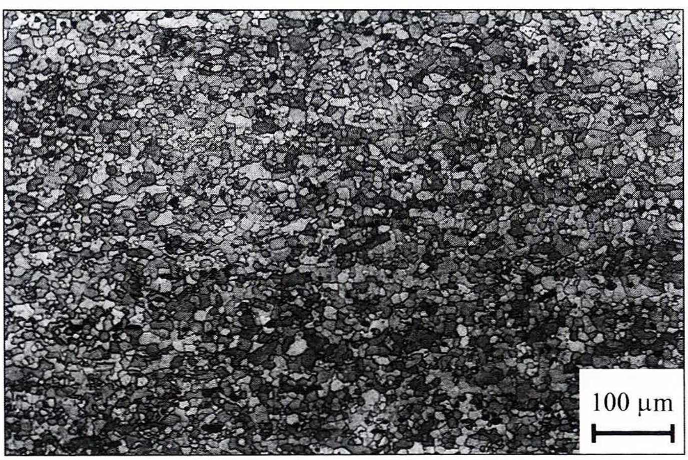

Fig. 1. Microstructure of the A7475 alloy after TMT "*A*" route

3.2. Scanning microscopy analysis

The fine, nearly equiaxial structure obtained after full *A* and *B* thermomechanical treatments can be seen in Figures 3 and 4. Large, elongated grains, refined by deformation and recrystallization processes were observed on Figure 4. Large precipitates (about 3 μm) and dispersoids (less than 1 μm) were found on grain boundaries and inside the grains. According to EDS measurements, in the selected fields of the sample the amounts of Mg, Zn and Cu up to 3.38 pct, 18 pct and 11 pct respectively, were found, which suggested the formation of the $MgZn_2$, Al–Zn–Mg or Al–Mg–Cu precipitates, however in the neighbourhood of dispersoids the increase of the Cr amount (up to 3.3 pct) was observed. It suggested the formation of the Al–Mg–Cr dispersoids.

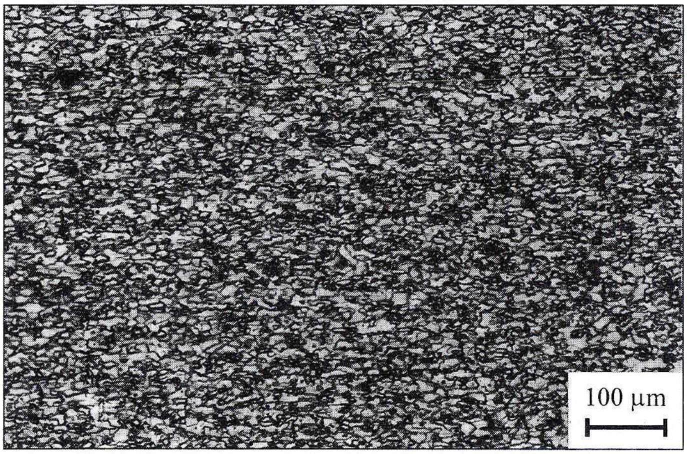

Fig. 2. Microstructure of the A7475 alloy after TMT "*B*" route

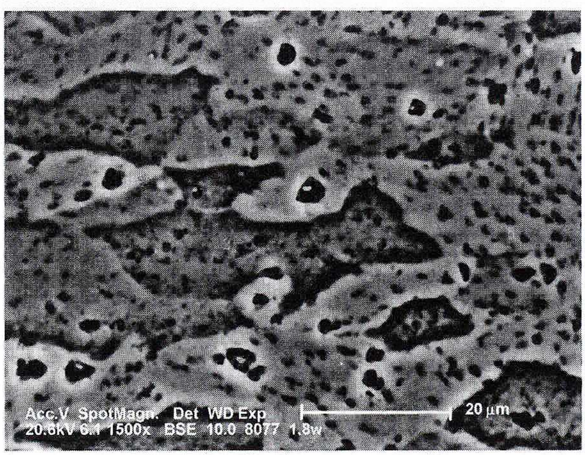

Fig. 3. Scanning electron micrograph showing fine, nearly equiaxial structure of the A7475 alloy after TMT "*A*" route

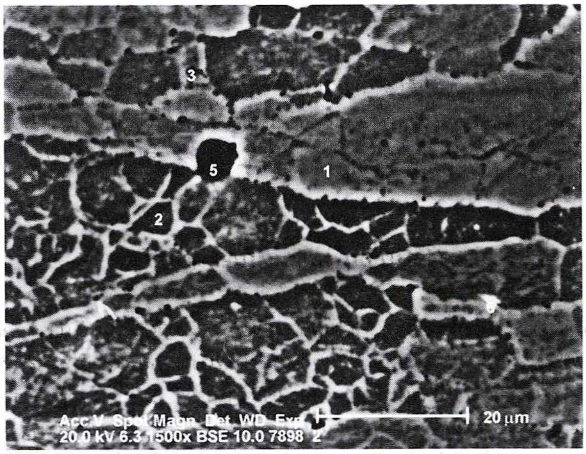

Fig. 4. Scanning electron micrograph showing elongated morphology with subgrains of the A7475 alloy after TMT "*B*" route

3.3. Transmission electron microscopy

The type and morphology of the dispersoids were investigated using transmission electron microscopy and selected area diffraction pattern methods. According to the SADP obtained at the foil zone axis orientation [112], a plate-like dispersoid of the size of about 300 nm corresponding to the $Al_{18}Mg_3Cr_2$ intermetallic compound was observed (Fig. 5). Dispersoids of the same type as that in Figure 5 were seen in another

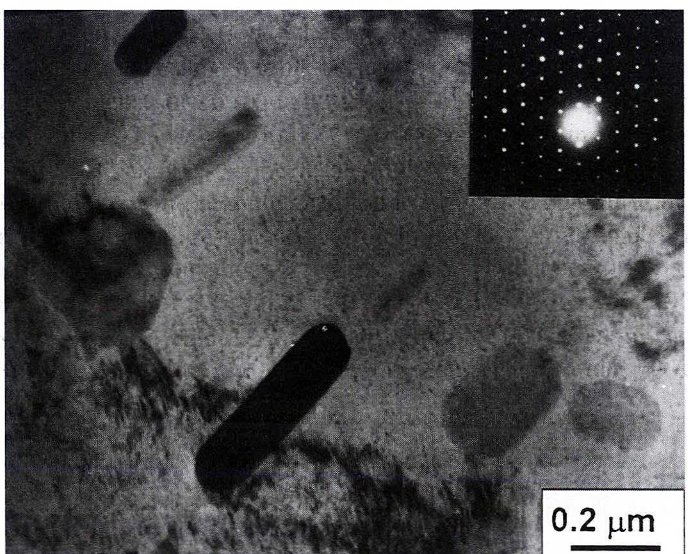

Fig. 5. Transmission electron micrograph and SADP of plate-like shaped dispersoid of the A7475 alloy after TMT "*A*"

microstructure (Fig. 6). It has elongated shape and lay within the grain boundary. Dispersoids of various shapes and size, between 100–500 nm, were observed in the investigated alloy.

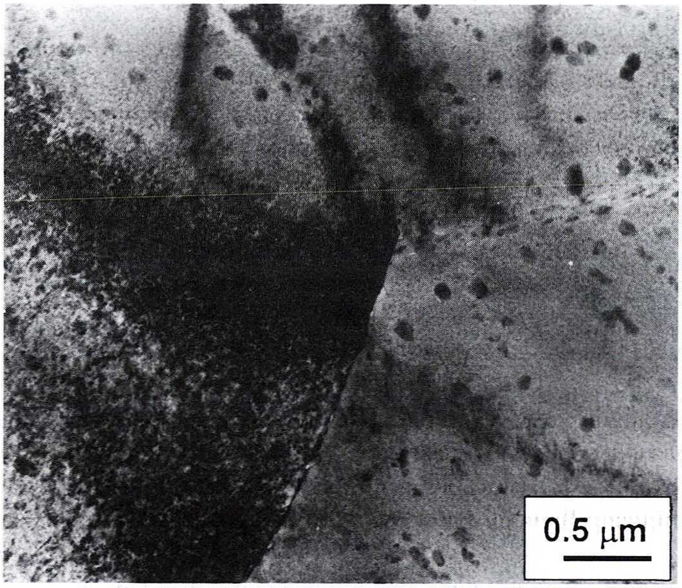

Fig. 6. Transmission electron micrograph showing elongated dispersoids in the grain boundary of the A7475 alloy after TMT "*A*"

3.4. *X*-ray phase analysis

The EDS results were confirmed by the *X*-ray phase analysis. The stable Cr – phase ($Al_{18}Mg_3Cr_2$) which formed as dispersoids was found in all the samples throughout all stages of TMT. Large amount of $MgZn_2$, Al_2Cu and Al_2CuMg precipitates was observed in the as cast state. After supersaturation only a very small amount of Al_2CuMg was found, but after overageing the $MgZn_2$ and Al_2CuMg – phases were again observed.

3.5. Tensile test

Examinations of mechanical properties verified the superplastic state quality of the investigated alloy after both *A* and *B* thermomechanical treatments. After the *B*-type of TMT the highest elongations at a rather high deformation rate were obtained. The dependence of the elongation on strain rate is shown in Figure 7. At the rate of $8\times10^{-3}s^{-1}$ the elongation of about 495% was reached. Curve 1 refers to a sample after rolling to 88% with small drafts per pass. Curve 2 refers to a sample which was rolled only up to 80% with higher drafts and it shows slightly lower results. The microstructure of sample 1 revealed finer grains than the other sample. The dependence of flow stress on the strain rate for the two samples of the alloy after B TMT process is shown in Figure 8. The flow stress changed strongly with the deformation rate and at the lowest rate ($8.3\times10^{-4}s^{-1}$) reached 0.81 MPa but at rate $1.67\times10^{-1}s^{-1}$ it increased to 12.61 MPa. The difference in the slopes of the curves is due to slightly different grain size obtained by TMT in which the rolling was performed with different drafts per pass.

Publications on superplasticity show that the strain rate sensitivity coefficient is directly related to the tension ductility of the metallic alloys [18, 19]. The strain rate sensitivity factor "*m*" calculated from the dependence given in Figure 8, reached the values 0.56 and 0.70. The differences in these *m* values are due to the diversification of the draft height per pass and degree of deformation at the plastic deformation stage of the thermomechanical treatment process. The highest elongation for the alloy after "*B*" TMT reached only 495 pct. at 790 K and $8\times10^{-3}s^{-1}$ strain rate. These results are in good agreement with the data given in literature. D.H. Shin et al. [10] obtained in the 7475 alloy with the average grain size of 8 μm the strain rate sensitivity coefficient slightly lower than 0.67, and the elongation of 730 pct at the strain rate of $2.8\times10^{-3}s^{-1}$ and temperature 783 K. Jiang Xinggang et al. [6] attained in the 7475 alloy with the average grain size of 10 μm, deformed at 783 K and $8\times10^{-3}s^{-1}$, the *m* value 0.55 and the elongation slightly above 500 pct. In the presented paper, at the relatively high *m* values lower elongations were obtained. It can be due to the rather disadvantageous shape of the grains and to the formation of dispersoids elongated in shape in the grain boundaries, as it is seen on the Figure 6, which can block, to some extent, the grain boundary sliding during superplastic deformation, as it was stated also in [18].

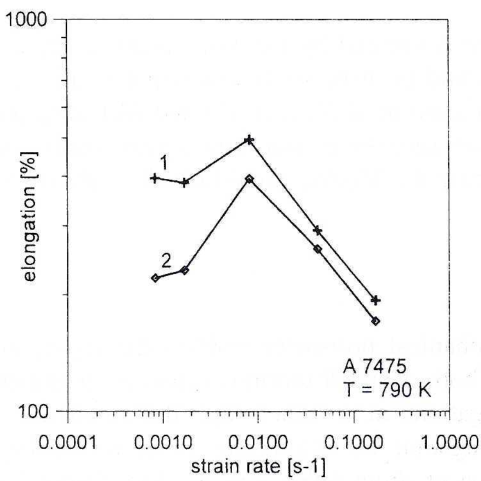

Fig. 7. Dependence of the elongation on strain rate of the A7475 alloy after TMT of the "*B*" type

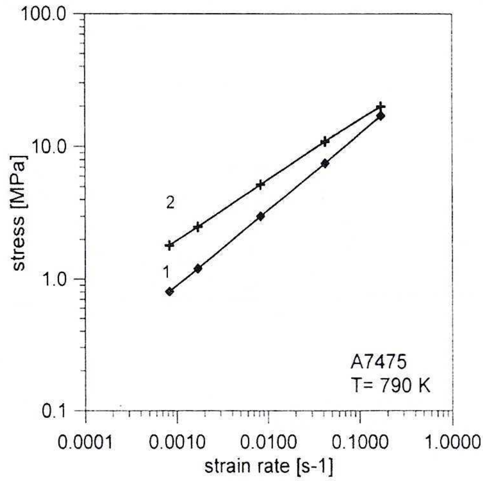

Fig. 8. Dependence of the flow stress on strain rate of the A7475 alloy after TMT of the "*B*" type

3.6. Mechanical properties of the alloy after superplastic deformation and heat treatment

After superplastic deformation realized in a compression die test the alloy was heat treated in the two – stage, T 76 treatment (supersaturated at 753 K and aged at 393 K/6 hours + 438 K/15 hours). The results obtained on this alloy, after heat treatment, in a tension test performed at room temperature reached the values of the ultimate tensile strength between 537 to 564 MPa and the elongations 15.1 % and 10.4 %, respectively, which are in good agreement with those given by T.G. Nieh et.al [19].

4. Conclusions

1. The high values of elongation at relatively high deformation rates and low flow stress confirmed the good quality of the superplastic state of the alloy after both the types of the thermomechanical treatment.
2. The alloy after superplastic deformation and heat treatment achieved higher ultimate tensile strength and much higher elongation than the alloy after extrusion.
3. The formation of the fine grain structure depends on the presence of the „large" precipitates and dispersoids during plastic deformation and recrystallization processes, respectively.

REFERENCES

[1] M.F. Ashby, R.A. Verall, Acta Metall. **21**, 149 (1973).
[2] R.C. Gifkins, Metall.Trans. **7A**, 1225 (1976).
[3] O.D. Sherby, J. Wadsworth, J. Sci.Technol. **1**, 925 (1985).

[4] C.M. Packer, R.H. Johnson, O.D. Sherby, Trans.Met.Soc.AIME **242**, 2485 (1968).
[5] D.H. Shin, Ch.S. Lee, W.J. Kim, Acta Metall. Mater. **456**, 5195 (1997).
[6] J.A. Wert, N.E. Paton, C.H. Hamilton, M.W. Mahoney, Metal. Trans. A **12A**, 1267 (1981).
[7] Jiang Xinggang, Cui Jianzhong, Ma Longxing, Z. Metallkd. **84**, 216 (1993).
[8] Alcoa route, http://www.mm.mtu.edu/drjohn/data – al/7475.html
[9] G.H. Mahon, D. Warrington, R.G. Butler, R. Grimes, Mat. Sci. Forum **170–172**, 187 (1994).
[10] D.H. Shin, S.H. Meng, J. Mat. Sci. Letters **8**, 1380 (1989).
[11] J. Waldman, H. Sulinski, H. Markus, Metal. Trans. **5A**, 473 (1974).
[12] E. Di Russo, M. Conserva, M. Buratti, F. Gatto, Mater.Sci. Eng. **14**, 23 (1974).
[13] H.S. Yang, A.K. Mukherjee, W.T. Roberts, J. Mat. Sci. **27**, 2515 (1992).
[14] C.C. Bampton, J.A. Wert, M.W. Mahoney, Metall. Trans. A **13A**, 193 (1982).
[15] E. Nes, Scripta Metall. **10**, 1025 (1976).
[16] F.J. Humphreys, Acta Metall. **15**, 1323 (1977).
[17] L. Ceschini, G.P. Cammarota, G.L. Garagnani, E. Landi, Prakt. Metallogr. **32**, 546 (1995).
[18] R. Monzen, M. Futakuchi, K. Kitagawa, T. Mori, Acta Metall. Mater. **41**, 1643 (1993).
[19] T.G. Nieh, J. Wadsworth, O.D. Sherby in "Superplasticity in metals and ceramics" Cambridge University Press, 1996. p. 65.

REVIEVED BY: JAN DUTKIEWICZ

Received: 10 September 2001.