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CHARACTERISTICS OF SUPERPLASTIC STATE OF THE 2XXX - TYPE ALLOY AFTER THERMOMECHANICAL TREATMENT

CHARAKTERYSTYKA STANU NADPLASTYCZNEGO STOPU TYPU 2XXX PO OBRÓBCE TERMOMECHANICZNEJ

The suitable structure of an alloy affects its superplastic properties due to a basic mechanisms of superplastic deformation. A fine structure of equiaxial grains appears in the alloy provided there are dispersoids and intermetallic compounds of certain size and distribution.

The composition of the 2XXX – type alloy (with Ni – addition) as well as a special thermomechanical treatment allowed to obtain a suitable structure of 15 μm-grain size and an equiaxial shape. The formation of such microstructure was investigated using optical, scanning and electron microscopy, as well as X-ray phase analysis techniques. The X-ray and electron microscopy with EDAX analyser allowed to determine the type and morphology of the intermetallic compounds, very important to generation the fine-grained structure. The superplastic state regarding quality of the structure was verified in tensile tests performed at the range of deformation rates between 1.4×10^{-4} to 7×10^{-3} s^{-1} and at temperatures from 789 K to 839 K, which allowed to attain the elongation above 300% and strain rate sensitivity coefficient $m = 0.509$.

Podstawowy mechanizm odkształcenia nadplastycznego, poślizg po granicach ziarn, jest ściśle związany ze strukturą i własnościami nadplastycznymi stopu. Wygenerowanie drobnokrystalicznej, równoosiowej struktury w badanym stopie jest związane z obecnością w nim dyspesoidów i związków międzymetalicznym o określonej ilości, wielkości i rozłożeniu. Tak skład stopu jak i określona obróbka termomechaniczna pozwoliły na otrzymanie struktury o średniej wielkości ziarna ok. 15 μm i równoosiowym kształcie. Badania strukturalne stopu prowadzono z zastosowaniem optycznej, skanningowej i elektronowej mikroskopii oraz rtg. analizy fazowej. Metody rtg. analizy fazowej oraz elektronowej mikroskopii z analizatorem EDAX pozwoliły na określenie typu i morfologii związków międzymetalicznych istotnych do wytworzenia drobnoziarnistej struktury stopu. Jakość struktury stanu nadplastycznego, weryfikowana w próbach rozciągania przeprowadzonych w temperaturach od 789 K do 839 K i przy szybkościach odkształcenia od 1.4×10^{-4} s^{-1} do 7×10^{-3} s^{-1}, pozwoliła na uzyskanie wydłużenia powyżej 300% i współczynnika czułości na szybkość odkształcenia $m = 0.509$

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1. Introduction

The aluminium alloys are the structural material widely used in transportation and aerospace industry. To improve their superplastic and strength properties different thermomechanical treatments were applied to obtain a fine-grain structure [1–5]. The superplastic deformation is an advantageous mode of production of elements with complicated shapes in one operation only, due to the ability of these alloys to deform up to a high degrees at low flow stresses.

Grain boundary sliding with accommodation mechanisms is the principal mode of superplastic deformation. According to these, the improvement of superplastic properties can be achieved by the refinement of grain size and transformation of grain morphology into a globular shape. The deformation behaviour of fine grain materials can be explained by two principal modes: grain boundary sliding accommodated by diffusional mass transport [6] or by crystallographic slip [7]. According to these two mechanisms, the deformation should be carried out at high temperatures and low deformation rates.

The fine-grained structure could be achieved by thermomechanical treatment (TMT) routes [1–5] composed, in general, of thermal treatment (supersaturation and ageing), plastic deformation and recrystallisation. During the thermal treatment "large" precipitates of intermetallic compounds are produced, while during the plastic treatment the deformation- and shear bands, are generated in their vicinity. During the recrystallisation stage, these bands form very fine-grain regions due to a high rate of nuclei formation [8]. The growth of grain is limited by dispersoids (in that case, of the Al_3Zr compound) generated in the alloy during casting due to zirconium addition, which hinder the migration of grain boundaries [3, 9]. The dispersoids stabilise the fine structure during superplastic deformation performed at high temperatures for a relatively long time according to low deformation rate.

Numerous investigations have been carried out on superplastic 2XXX alloys. High superplastic properties ($m = 0.49$) were obtained in the 2014 alloy by Li *et al.* [10]. They attained the elongation of 450% at the deformation rate of $8.33\ 10^{-3}\ s^{-1}$ in a tensile test. The fine-grain structure of $d = 13$ μm was accomplished in the AA2014 alloy [11] and 400% of elongation at deformation rate $1 \times 10^{-3}\ s^{-1}$ was achieved. Nearly the same results were obtained in an industrial alloy 2024 [12]. Nieh *et al.* [13] generated a fine structure in the 2024 alloy with high Zr content (0.6 %), which allowed to achieve 500% elongation at 773 K and $3.3 \times 10^{-3}\ s^{-1}$ rate. On the Al-Cu-Mg with Ag addition alloy Dutkiewicz *et al.* [14] obtained superplastic state of good quality due to high cooling rate and formation of Ω phase. Matsuki *et al.* [15] reported to have obtained the elongations up to 400% in the alloy at $m = 0.45 - 0.8$ (at the temperatures $733 - 773$ K).

In the present work, the stable, fine and nearly equiaxial grained structure of high superplastic properties was generated in the investigated alloy having applied the TMT. The verification of the superplastic state quality of the investigated alloy was confirmed in the high temperature tensile tests. The composition of the alloy with Ni addition allowed to generate the very fine intermetallic compounds of the Al-Ni, Al-Cu and Al-Cu-Mg types, very important for the formation of the fine – grained alloy during thermomechanical treatment.

2. Experimental

2.1. Material and methods of investigation

An alloy of 2XXX – type of the composition: 4.2 Cu, 1.8 Mg, 1.2 Ni, 0.3 Zr, Al – bal. (all in wt.%.) was the material for investigation. The Ni and Zr additions were incorporated into the Al alloy as master alloys (Cu-Ni 40 and Al-Zr 10). The alloy was melted in a resistance furnace and cast in a copper mould from temperature 993 K. The ingots were homogenised at 773 K for 2 hours and warm rolled at 653 K up to deformation degree about 30%.

PHILIPS CM-20 transmission electron microscope was used to obtain transmission electron micrographs and EDS analysis (on EDAX analyser). Thin foils for the TEM studies were produced by jet electropolishing in a 2/3 methyl alcohol + 1/3 nitric acid solution, followed by ion beam thinning, using Gatan 660 ion mill.

Light microscope NEOPHOT allowed to observe morphology of the samples prepared by etching of the mechanically polished surfaces with Wilcox solution.

PHILIPS XL-30 scanning electron microscope was used for quantitative analysis and to study the microstructure at a higher magnification.

The X-ray phase analysis of the investigated alloy was carried out on the alloy after different stages of the thermomechanical treatment, using PHILIPS PW 1710 diffractometer.

INSTRON 6025 testing machine was used to determine the superplastic characteristics of the samples in a tensile mode. The tests were performed at temperatures in the range of 789–839 K and at deformation rates 1.4×10^{-4} s^{-1} – 6.94×10^{-3} s^{-1}.

2.2. Thermomechanical treatment

To obtain the fine-grained, equiaxial structure, a special thermomechanical treatment route, elaborated on the basis of literature data [2–4] and own investigations [16, 17], was performed: supersaturation at 763 K for 16 h + ageing at 673 K for 5 h + plastic deformation + recrystallisation at 753 K for 15 or 30 minutes (in a salt furnace).

The deformation process was carried out by cold rolling with the roll diameter of 150 mm and draughts in the range of 15–22% up to 85% of deformation. The recrystallisation stage was performed in salt furnace to ensure relatively high heating rate (about 10^2 Ks^{-1}).

3. Results

3.1. Light microscopy studies

The alloy after casting into a copper mould (Fig. 1) had a fine, dendritic structure with intermetallic precipitates and a small amount of non-metallic inclusions and porosity. Coarse-grains (50–100 μm big) and a small amount of intermetallic precipitates

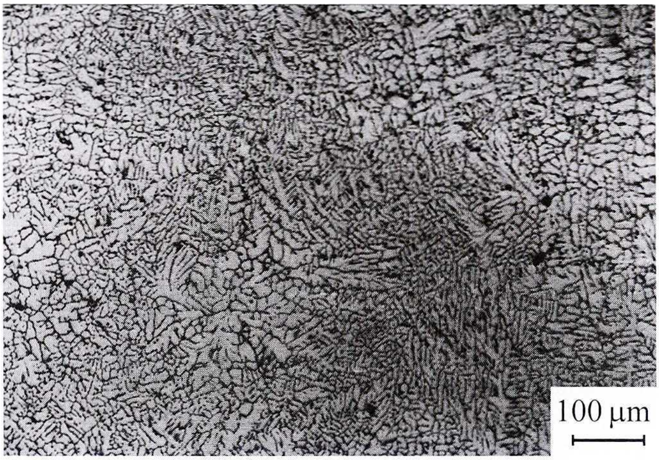


Fig. 1. Optical micrograph of the as cast investigated alloy

were observed after a thermal treatment composed of short homogenisation, warm rolling and long (16 hours) supersaturation (Fig. 2). The grain size after ageing remained almost unchanged, but a quite high amount of intermetallic precipitates formed frequently on

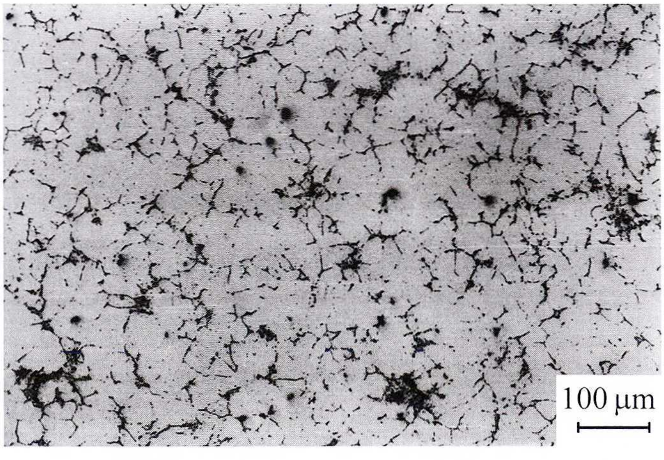


Fig. 2. Optical micrograph of the investigated alloy after supersaturation

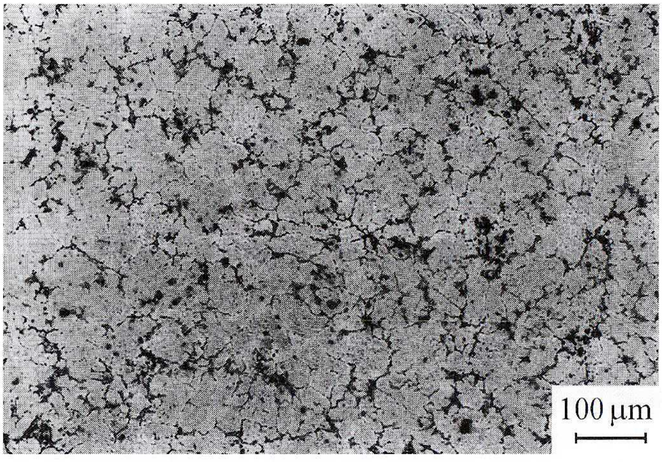


Fig. 3. Optical micrograph of the investigated alloy after ageing

grain boundaries as well as inside the grains (Fig. 3). The precipitate free zones generated in the neighbourhood of grain boundaries. Eventually, a fine-grain, equiaxial structure was observed in the samples after recrystallisation, which was the last stage of the thermomechanical treatment. The grain size was estimated to be 12–20 μm. (Fig. 4), although, very small grains of size of some μm were also observed in the TEM microstructures.

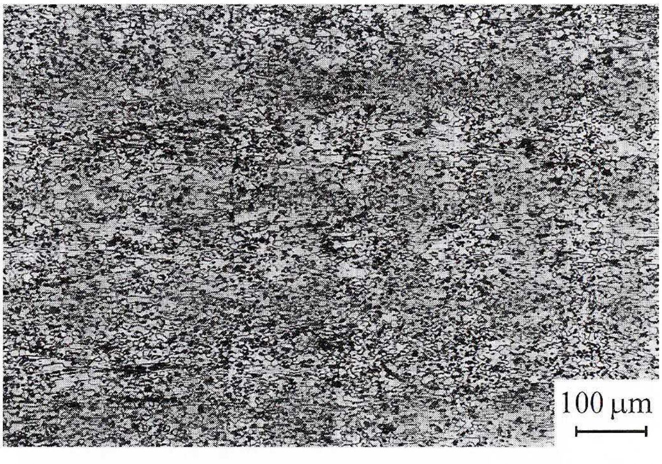


Fig. 4. Optical micrograph of the investigated alloy after recrystallisation

3.2. Transmission electron microscopy

Small (under 100 nm big), globular dispersoids of the type were observed at high magnification (88 000 X) in thin foils of the alloy after supersaturation, (Fig. 5). The field of high density of disperoids with dislocations among them can also be seen

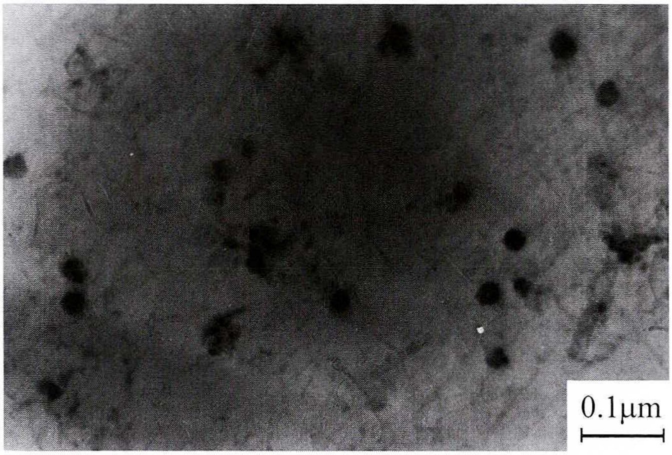


Fig. 5. Transmission electron micrograph of the investigated alloy showing dispersoids after supersaturation

in Fig. 6 in the sample after recrystallisation, cooled at a high rate, which caused high stresses and the formation of a dislocation net. The EDS measurements, performed in the field of high density of dispersoids, showed a high concentration of Zr addition up to 10.7 wt.% (Fig. 7), which confirmed the presence of the Al_3Zr intermetallic compound.

Other intermetallic precipitates in the shape of elongated needles or platelets (Al–Cu or Al–Cu–Mg-type) and irregular (Al_xNi_y-type) of the size of some μm were

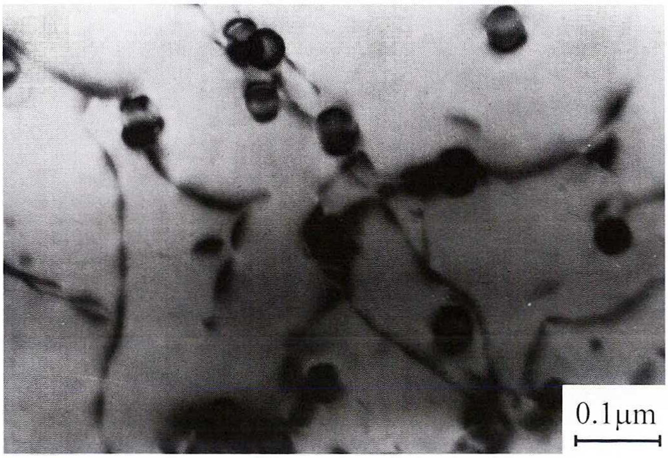
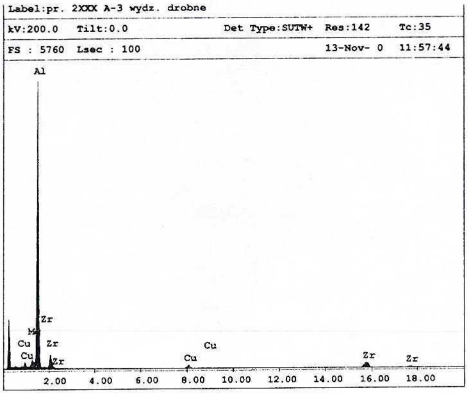


Fig. 6. Transmission electron micrograph showing distribution of dispersoids and dislocation net in the investigated alloy after recrystallisation



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Element	Weight %	Atomic %
MgK	3.9	4.7
AlK	83.5	90.9
CuK	1.9	0.9
ZrK	10.7	3.5
Total	100.0	100.0

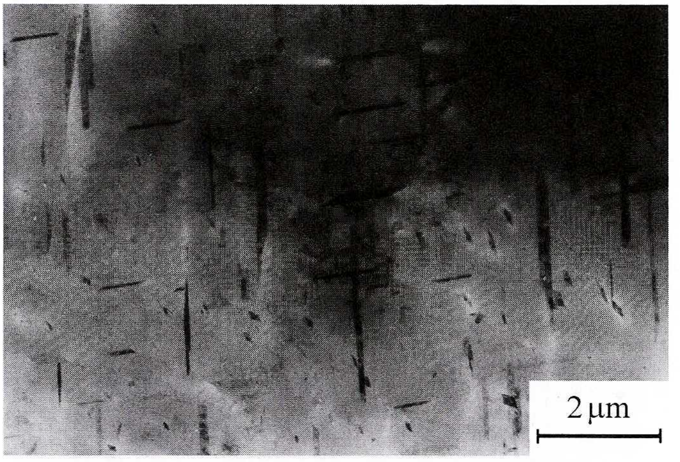
Fig. 7. X-ray chemical analysis (EDAX) from the area showed in Fig. 6

observed in the alloy after ageing (Fig. 8a and b, respectively). In the samples after recrystallisation, small grains of the matrix, were seen (Fig. 9), which confirmed the result from the light microscopy observations, that two kinds of the matrix grains were formed after the thermomechanical treatment: the ''large'' (up to 20 μm) and much smaller ones most probably of low angle grain boundaries. On the basis of the TEM observations, it was possible to determine the size of these ''small'' grains to be 2–5 μm. The mean size of the matrix grains was estimated between 12–15 μm. Also quite large, (above 1 μm-big) intermetallic precipitates of different types were noticed in the grain boundaries (Fig. 10).

3.3. X-ray phase analysis

The presence of high amount of different intermetallic compounds was stated in the investigated alloy after short homogenisation and warm rolling. They were mostly Al_3Ni, and Al_2CuMg precipitates, although smaller numbers of Al_3Ni_5 and AlCuMg ones and random Al_2Cu and AlCu phases were also observable. Much lower amounts and types of these compounds were found in the samples the after long-term supersaturation, whereas higher amounts and variety of the intermetallic precipitates (Al_2CuMg, AlCuMg, Al_3Ni and particularly Al_3Ni_2 and Al_2Cu) were generated in the alloy after ageing. After the 15 min.-recrystallisation, low amounts of Al_3Ni_2 and AlCuMg were found, while after the 30 min.-recrystallisation the precipitates $Al_{15}Cu_6Mg_2$ and Al_2Cu were identified.

a)



b)

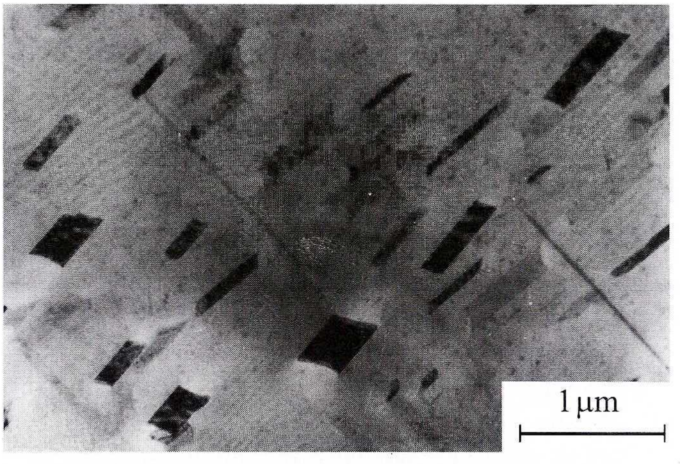


Fig. 8. Transmission electron micrograph of the investigated alloy after recrystallisation showing "large" precipitates of the intermetallic compounds; a) Al_2MgCu – type and b) Al_3Ni-type

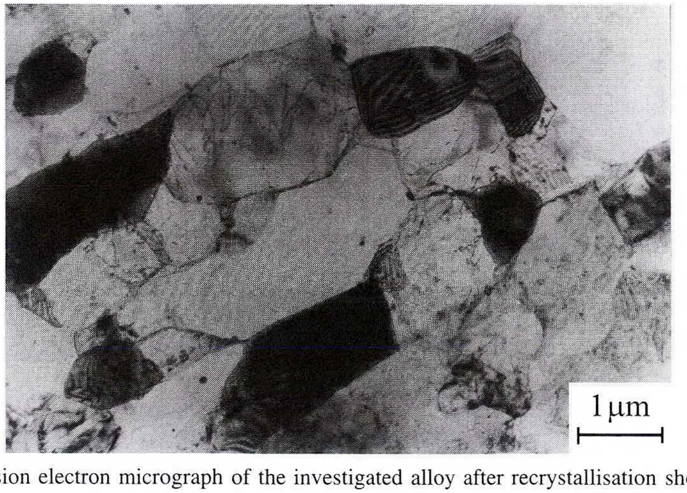


Fig. 9. Transmission electron micrograph of the investigated alloy after recrystallisation showing fine grains of the matrix

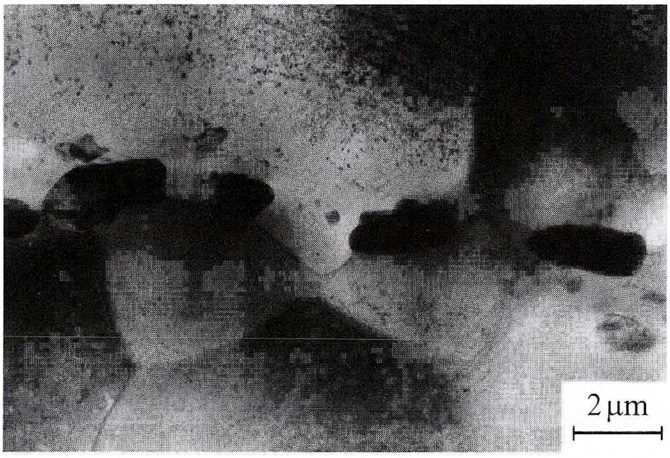


Fig. 10. Transmission electron micrograph showing the intermetallic precipitates in the grain boundaries

3.4. Tensile test

The verification of the superplastic state quality in the investigated alloy was carried out using a high temperature tensile test method. The tests were performed in the temperature range between 789 K to 839 K and at the deformation rates between 1.4×10^{-4} s^{-1} to 6.94×10^{-3} s^{-1}. The best result (highest elongation $\varepsilon = 334\%$) was achieved at 839 K and 2.8×10^{-4} s^{-1} deformation rate, while at about 789 K, only 150% elongation was reached. It was observed that deformation rate affected elongation. At the lowest and highest deformation rates applied, which were 1.4×10^{-4} s^{-1} and 6.94×10^{-3} s^{-1} the elongations of only 145% and 100% (at 839 K) respectively, were attained (Fig. 11 a). The flow stress changed strongly with deformation rate, at the lowest rate reached 1.94 MPa, while at

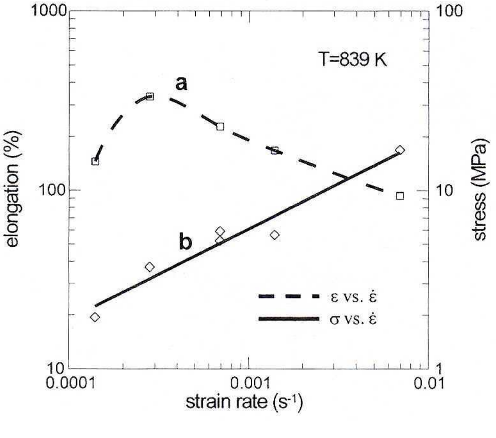


Fig. 11. The dependences of the elongation (a), and flow stress (b) vs. deformation rate

the highest one it increased only to 17 MPa (at 839 K). Papers on superplasticity report the strain rate sensitivity coefficient ‘‘m” to be directly related to the tension ductility of metallic alloys [18, 19]. According to the logarithmic dependence of flow stress on strain rate (Fig. 11 b), the strain rate sensitivity coefficient ‘‘m” reached a relatively high value of 0.509. Taking into consideration the low flow stresses and relatively high value of the ‘‘m” coefficient, it could be stated that the investigated alloy, after the applied thermomechanical treatment is distinguished by a high quality of superplastic state. The both given above results obtained in the presented paper are in good agreement with the data given in the reports of other researchers [10–15].

4. Discussion of results

Casting the alloy into a copper, flat mould allowed to obtain a high cooling rate and a relatively fine-grain structure in the investigated alloy. The formation of the fine – grained structure in the Al-Cu-Mg alloys is, according to W a t a n a b e [20], preferred by the high content of copper. An appropriate sequence of melting of the alloying additions and pouring at an exactly determined temperature allowed to produce the alloy with dispersoids of small size and nearly homogeneous distribution. The fine-grain, equiaxial structure with good superplastic properties was generated due to the thermomechanical process applied. The microstructure of the alloy deformed at superplastic conditions showed fine-grain structure (slightly coarser than after recrystallisation) but the shape of the grains was much more globular compared with the microstructure after recrystallisation (Fig. 12, 13) as it was observed on the microstructures shown by the other scientists [3, 21]. That results reinforces the suggestion that this deformation process has a strong superplastic character with a high contribution of grain boundary sliding and diffusion mass transport mechanism as an accommodation one.

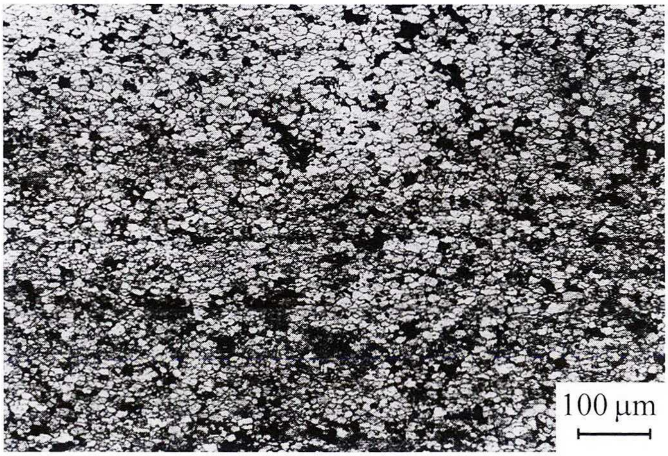


Fig. 12. Optical micrograph of the investigated alloy after superplastic deformation

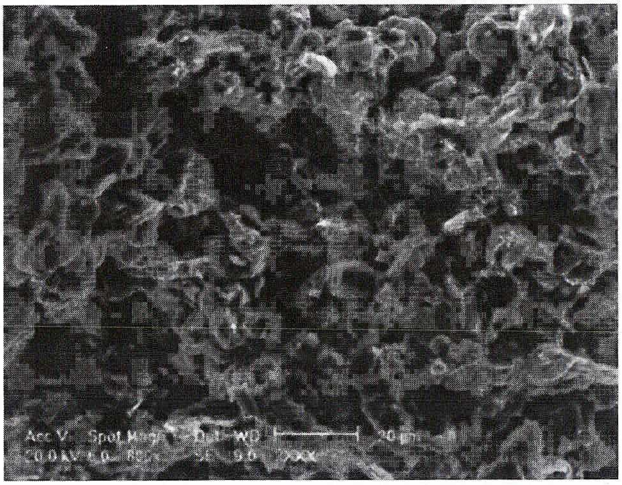


Fig. 13. Scanning electron micrograph of the investigated alloy after superplastic deformation

Deformation was performed at a high (839 K) temperature and a low (2.8×10^{-4} s^{-1}) deformation rate. In that case, the mass transport accommodation mechanism had much more ability to take part in the deformation than crystallographic slip, as it was stressed by A s h b y [6]. The fine-grain structure obtained at recrystallisation stage of thermomechanical treatment confirmed the statement that the distribution, amount and size of the dispersoids were very suitable and stabilised the structure of the alloy even at the superplastic deformation conditions, according to results obtained by S h i n [21] and Y a n g [9]. The value of the highest elongation (334%) obtained in the tension test at 839 K was inadequate for a relatively high value (0.509) of the strain rate sensitivity

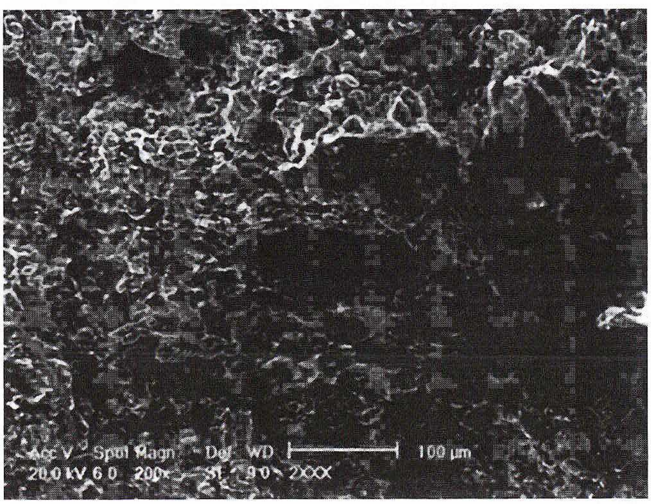


Fig. 14. Scanning electron micrograph of superplastic uniaxial tensile fracture showing cavities

„m" parameter. The best result obtained on the Al-Cu-Mg alloy by Watanabe [20] was $m = 0.7$ and $\varepsilon = 685\%$ at 818 K. According to data of Watanabe [20] and other scientists [10–15], can be suggested that for $m = 0.5$ the elongation in the 2XXX – type alloys should achieve about 450%. The low value of elongation of the investigated alloy might have been caused by an untimely failure of the deformed sample by the presence of the structure imperfections, particularly cavitations. In superplastic deformation conditions intergranular cavitations play a significant role in a final tensile result [22]. Cavitations were observed in the microstructure of the elongated-to-failure sample (Fig. 14). The localisation of flow stress in the vicinity of a cavity, caused the untimely failure of the tensioned sample.

5. Conclusions

1. The applied thermomechanical treatment generated a fine-grain, equiaxial structure defined as high quality superplastic state in the investigated alloy.

2. The appearance of dispersoids of a suitable size, amount and distribution stabilized the structure during the superplastic, high temperature deformation.

REFERENCES

[1]. J.A. Wert, N.E. Paton, C.H. Hamilton, M.W. Mahoney, Met.Trans A **12A**, 12 (1981).
[2]. Jiang Xinggang, Cui Jianzhong, Ma Longxiang, Z. Metallkde **84**, 216 (1993).
[3] Dong Hyunk Shin, Sun Hae Meng, J.Mat. Sci Letters **8**, 512 (1989).
[4] M. Kobayashi, Y. Koima, Report for Research Group For Superplastic Phenomena and Applications in Aluminium Alloys, Osaka, June 1985,
[5] G.J. Mahon, D. Warrington, R.G. Butler, R. Grimes, Mat. Sci. Forum **170–72**, 187 (1994).
[6] M.F. Ashby, R.A. Verall, Acta Metall. **21**, 149 (1973).
[7] O.D. Sherby, J. Wadsworth, Mater. Sci. Technol. **1**, 925 (1985).
[8] F.J. Humphreys, Acta Metall. **15**, 1323 (1977).
[9] H.S. Yang, A.K. Mukherjee, W.T. Roberts, J. Mat. Sci. **27**, 2515 (1992).
[10] X.P. Li, P.L. Antona, Euromat 92, Genova, Italy 24–26 Sept. 92.
[11] P.S. Bate, H. Schofield, Metall. Trans A, **29A**, 1405 (1998).
[12] Ming Gao, C.R. Feng, R.P. Wei, Metall. Trans A, **29A**, 1145 (1998).
[13] T.G. Nieh, J. Wadsworth, Scripta Metall. Mater. **28**, 1119 (1993).
[14] J. Dutkiewicz, P. Malczewski, J. Kuśnierz, T.G. Nieh, Proc. LIMAT – 2001 eds. N.J. Kim, C.S. Lec, D. Eylon. Published by Center for Advanced Aerospace Materials 2001, p. 387.
[15] K. Matsuki, H. Sugahara, T. Aida, Mat. Trans. JIM, **8**, 737 (1999).
[16] J. Król, M. Tałach-Dumańska, Archives of Metall. **47**, 17 (2002).
[17] J. Król, E. Bielańska, M. Dumańska, L. Lityńska, H. Paul, Proc. Conf. "Light Alloys and Composites" Zakopane 13-16.04.99. pp. 221–228.
[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.
[20] H. Watanabe, K. Ohori, Yo Takeuchi, Trans. ISIJ **27**, 730 (1987).
[21] D.H. Shin, Ki S. Kim, D.W. Kum, Soo W. Nam, Met. Trans A, **21A**, 2729 (1990).
[22] C.C. Bampton, W.M. Mahoney, C.H. Hamilton, A.K. Ghosh, R. Raj, Met. Trans A, 14A, 1583 (1983).

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