JOURNAL OF MATERIALS SCIENCE 36 (2001) 5007–5011

Consolidation of machined magnesium alloy chips by hot extrusion utilizing superplastic flow

HIROYUKI WATANABE
Osaka Municipal Technical Research Institute, 1–6–50 Morinomiya, Joto-ku, Osaka 536–8553, Japan
E-mail: hwata@omtri.city.osaka.jp

KOJI MORIWAKI
Department of Metallurgy and Materials Science, College of Engineering, Osaka Prefecture University, 1–1 Gakuen-cho, Sakai 599–8531, Japan

TOSHIJI MUKAI, KOICHI ISHIKAWA
Osaka Municipal Technical Research Institute, 1–6–50 Morinomiya, Joto-ku, Osaka 536–8553, Japan

MASAHIDE KOHZU, KENJI HIGASHI
Department of Metallurgy and Materials Science, College of Engineering, Osaka Prefecture University, 1–1 Gakuen-cho, Sakai 599–8531, Japan

An examination of consolidation conditions by hot extrusion of AZ31 magnesium alloy machined chips was conducted to enhance the bonding of individual chips, in order to improve the mechanical properties. Hot extrusions were carried out in the superplastic and non-superplastic region. Microstructural observations revealed that grain refinement was attained by extruding machined chips, and the grain sizes of the chip-extruded materials were smaller than 5 μm. The interfaces of individual chips of extruded materials were not identified when the chips were extruded in the superplastic region. The ultimate tensile strength was about 300 MPa and elongation-to-failure was about 10% for chip-extruded materials that were extruded in the superplastic region. These materials were comparable with the as-received alloy with respect to the room temperature strength, although the ductility was reduced to half. It was confirmed that chip consolidation utilizing superplastic flow is useful to enhance the bonding of individual grains.

1. Introduction

Research of magnesium has increased in order to reduce the weight of components from the economical and ecological point of view. There are many potential opportunities for the use of magnesium alloys in motor vehicle components. This is not only a result of magnesium's relatively low density, which can directly and substantially reduce vehicle weight, but is also a result of its good damping characteristics, dimensional stability, low casting cost, and machinability. Because of magnesium's excellent machinability, a large amount of magnesium machined chips will be produced with an increase in demand. Therefore, recycling machined chips is important for the application of magnesium. Magnesium machined chips are currently remelted or burned with sands. However, these treatments are expensive since magnesium is susceptible to oxidation. It has been demonstrated that hot extrusion is an effective process for consolidation of magnesium machined chips [1–7]. Therefore, this would be beneficial as a recycling process. The attraction of this recycling process is that the treatment is conducted in solid state. Experimental evidence revealed that the extrusions processed from machined chips shows not only good combination of high strength and moderate elongations-to-failure of 5–12% at room temperature but also superplasticity at elevated temperatures [5]. However, no detailed investigations have been made into the optimization for consolidation of magnesium machined chips by extrusion.

Mechanical properties in magnesium alloys strongly depend on the grain size. Especially, the effect of grain refinement on room temperature strength is significant, because the h.c.p. structure has a large Taylor factor [8]. Not only high strength but also high ductility at room temperature is attained by grain refinement [8]. Furthermore, fine-grained magnesium alloys exhibit superplasticity at elevated temperature [9]. In this way, grain refinement is desireble to enhance the mechanical properties of magnesium alloys. On the other hand, in order to attain excellent mechanical properties in chip-extruded material, it is important to improve the bonding of individual machined chips. It is required to combine the fine grain size and good bonding of individual chips for attaining excellent mechanical properties in chip-extruded material. As for the grain refinement, Mabuchi *et al*. [5] showed that lower extrusion temperature made resulting grain size smaller. On the other hand, for the further improvement of the bonding between individual chips, it is necessary to break and disperse the oxide layer, that exists on the surface of the chips [3]. To date, it has been demonstrated that powder consolidation by hot pressing utilizing superplastic flow is useful to enhance the densification [10, 11]. Therefore, one possibility to enhance the bonding would be to utilize the superplastic flow for the consolidation. Because the dominant deformation process of superplasticity is grain boundary sliding, it is expected that grain boundary sliding process will break the surface oxide layer of chips.

It is possible to vary the dominant deformation process during extrusion by selecting the extrusion conditions; i.e., temperature, extrusion ratio, die angle and ram velocity. In the present study, extrusions were performed in the non-superplastic and superplastic region. The experimental results suggest that chip consolidation by hot extrusion utilizing superplastic flow is useful to enhance the bonding of individual grains.

2. Extrusion conditions

In the present study, extrusions were performed with three conditions. Extrusion conditions are listed in Table I. The extrusion strain rate, $\dot{\varepsilon}_e$, was determined using following equation [12].

$$\dot{\varepsilon}_e = \frac{6\nu d_0^2}{\left(d_0^3 - d_1^3\right)} \ln\left(\frac{d_0^2}{d_1^2}\right) \tan\theta \qquad (1)$$

where ν is the ram velocity, d_0 the container diameter, d_1 the extrusion diameter, and θ the semi-angle of conical die. The calculated extrusion strain rate is listed in Table I.

Extrusion Conditions were determined to impose an appropriate deformation mode. To predict the deformation mechanism during extrusion, phenomenological constitutive equations proposed for high temperature deformation were used. Three modes of deformation process were taken into consideration; i.e., (a) slip accommodated grain boundary sliding, (b) dislocation glide process with solute atmosphere and (c) dislocation glide process without solute atmosphere.

The constitutive equation to describe high temperature deformation is expressed as [13]

$$\dot{\varepsilon} = A\left(\frac{Gb}{kT}\right)\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n D_0 \exp\left(-\frac{Q}{RT}\right) \qquad (2)$$

where $\dot{\varepsilon}$ is the strain rate, A a constant, k the Boltzmann's constant, T the temperature, G the shear modulus, b the Burgers vector, d the grain size, σ the flow stress, R the gas constant, D_o the pre-exponential factor for diffusion, Q the activation energy, n the stress exponent and p the grain size exponent.

For slip accommodated grain boundary sliding process ($n = 2$), which is accepted as the dominant deformation mechanism for superplastic flow, the equation proposed by Sherby and Wadsworth [14] was used. It has been confirmed that the equation can be applied to the behavior in fine-grained magnesium alloys [9]. The phenomenological relations for dislocation glide process without solute atmosphere in magnesium ($n = 5$) was constructed by Frost and Ashby [15], and that for dislocation glide process with solute atmosphere in magnesium-alumium solid solution alloys ($n = 3$) has been proposed by Watanabe *et al*. [9]. A set of parameters in Equation 2 to express each deformation process is listed in Table II.

The relationship between flow stress and strain rate for slip accommodated grain boundary sliding process, dislocation glide process with solute atmosphere and dislocation glide process without solute atmosphere is shown in Fig. 1. The grain size of each chip-extruded material, which will be shown in Section 4, is listed in Table I. Since three deformation processes are considered to be independent, the fastest one is rate-controlling. Therefore, the predicted dominant deformation mechanism at a given strain rate is given by the shaded curves. Although we considered the condition for $n = 3$ deformation, this model was not relevant for the present study. The extrusion strain rate for

TABLE I Extrusion conditions and expected deformation mechanism during extrusion

	Extrusion condition						Grain size of extrusion	Predicted deformation mechanism
	T (K)	v (mm/sec)	d_0 (mm)	d_1 (mm)	θ, °	$\dot{\varepsilon}_e$ (s^{-1})	d, μm	
A	493	0.2	40	10	45	8.4×10^{-2}	2	Non-superplasticity
B	623	0.2	40	4	45	1.4×10^{-1}	3	Superplasticity
C	703	0.2	40	4	45	1.4×10^{-1}	4	Superplasticity

TABLE II Parameters in constitutive equation for various deformation mechanisms

Deformation mechanism	A	p	n	D_0 ($m^2 s^{-1}$)	Q (kJ/mol)	Ref.
Slip accommodated grain boundary sliding	2.0×10^5	3	2	$5.0 \times 10^{-12}/\delta^a$	92	[14]
Dislocation glides with solute atmosphere	3.0×10^{-2}	0	3	1.2×10^{-3}	143	[9]
Dislocation glides without solute atmosphere	1.2×10^6	0	5	1.0×10^{-4}	135	[15]

[a] δ is the grain boundary width ($\delta = 2b$ in the present analysis).

each extrusion temperature is indicated by the arrow in Fig. 1. It is estimated that conditions B and C are both at the high end of the superplastic region, and condition A is at the transition conditions from superplastic to non-superplastic deformation.

3. Experimental

The material used in the present study was a commercial AZ31 magnesium alloy. Chips were prepared by machining an extruded rod in a lathe. The machined chips were cold pressed, and the billets were then hot extruded in air at the conditions given as listed in Table I.

Tensile specimens, machined directly from the extruded bars, had tensile axes parallel to the extruded direction. The tensile specimens had a gauge length of 5 mm and a gauge diameter of 2.5 mm. Tensile tests were carried out at an initial strain rate of 1×10^{-3} s^{-1} at room temperature.

Microstructures were examined using optical microscopy and transmission electron microscopy. Specimens for the observation of optical microstructures were etched using a solution of 14.3 ml acetic acid, 10 g picric acid, 100 ml ethanol and 14.3 ml distilled water to reveal the grain boundaries. Fracture surfaces were inspected by a scanning electron microscopy.

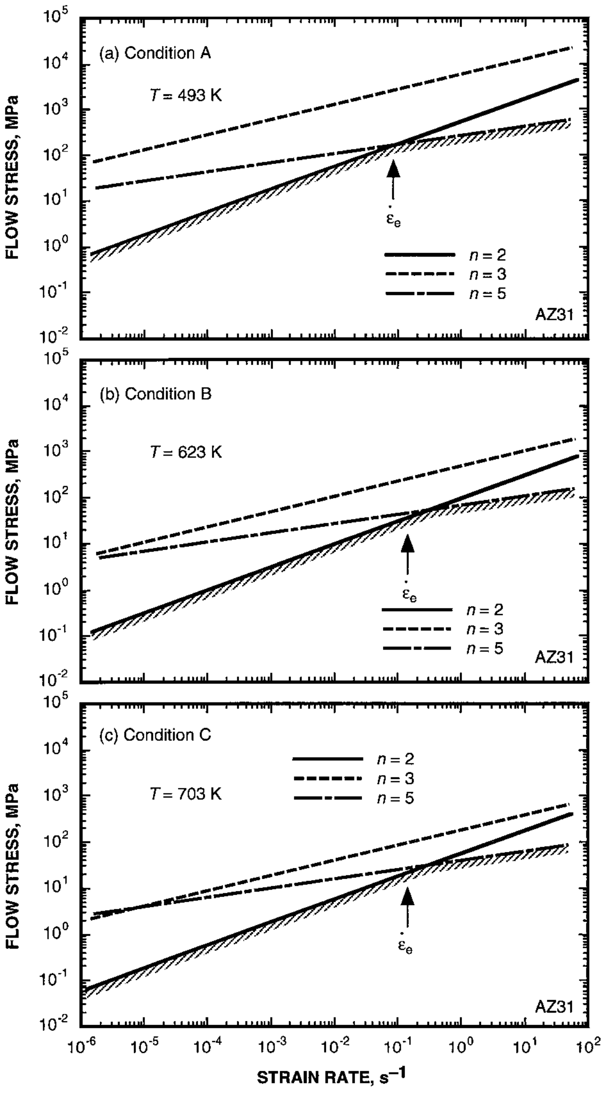

Figure 1 The relationship between flow stress and strain rate for slip accommodated grain boundary sliding process ($n = 2$), dislocation glide process with solute atmosphere ($n = 3$) and dislocation glide process without solute atmosphere ($n = 5$). The predicted dominant deformation mechanism at a given strain rate is indicated by the shaded area. (a) $T = 493$ K (Condition A) (b) $T = 623$ K (Condition B) (c) $T = 703$ K (Condition C). Arrows show the extrusion strain rate, $\dot{\varepsilon}_e$.

4. Results and discussion

4.1. Microstructures of extruded materials

Optical microstructure of the as-received material is shown in Fig. 2. The initial microstructure was

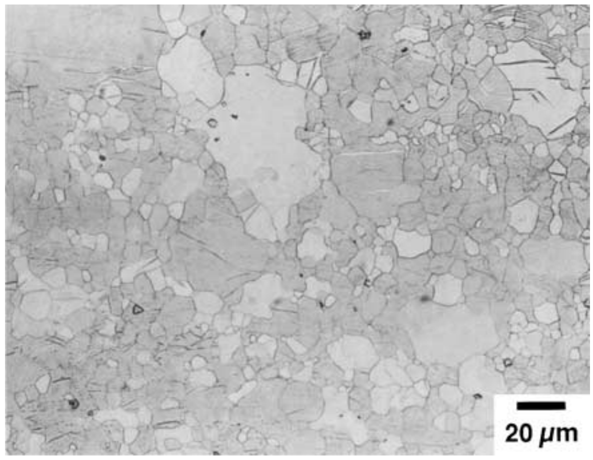

Figure 2 Optical microstructure of the as-received material in the longitudinal section. Extrusion direction is horizontal.

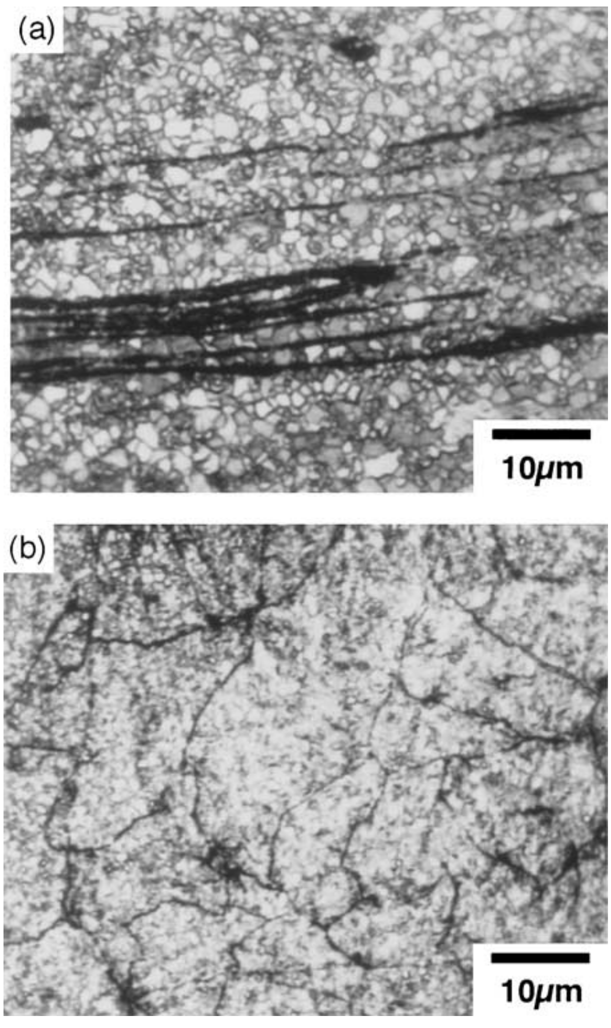

Figure 3 Optical microstructures of the 493 K chip-extruded material in the (a) longitudinal section and (b) transverse section.

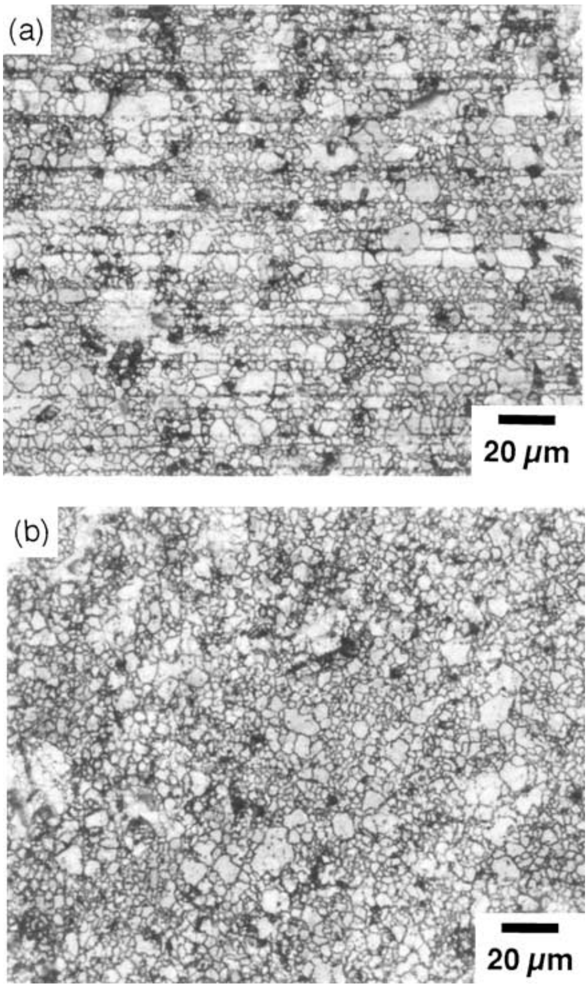

Figure 4 Optical microstructures of the 623 K chip-extruded material in the (a) longitudinal section and (b) transverse section.

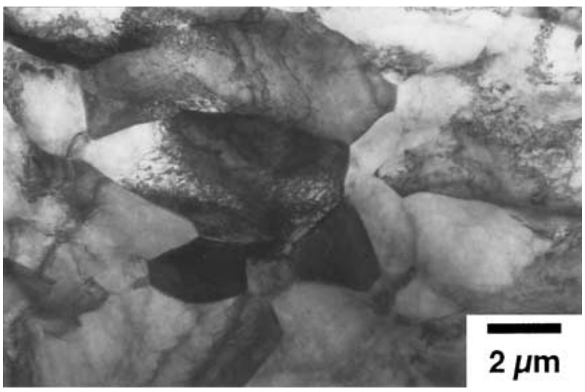

Figure 5 A transmission electron micrograph of the 623 K chip-extruded material in the longitudinal section.

consisted of both relatively fine and coarse grains. Optical microstructures for chip-extruded materials are shown in Figs 3 and 4 for condition A and B, respectively. A transmission electron micrograph for chip-extruded material for Condition B is also shown in Fig. 5. Obviously, grain refinement was attained by extruding machined chips. The grain sizes of chip-extruded material were about 2, 3 and 4 μm for extrusion condition A, B and C, respectively. It is evident that the grain structure of the extruded material is refined with decreasing extrusion temperature.

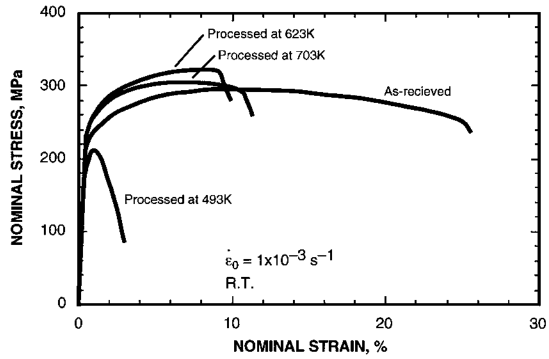

Figure 6 Nominal stress – nominal strain curves of chip-extruded materials and as-received material.

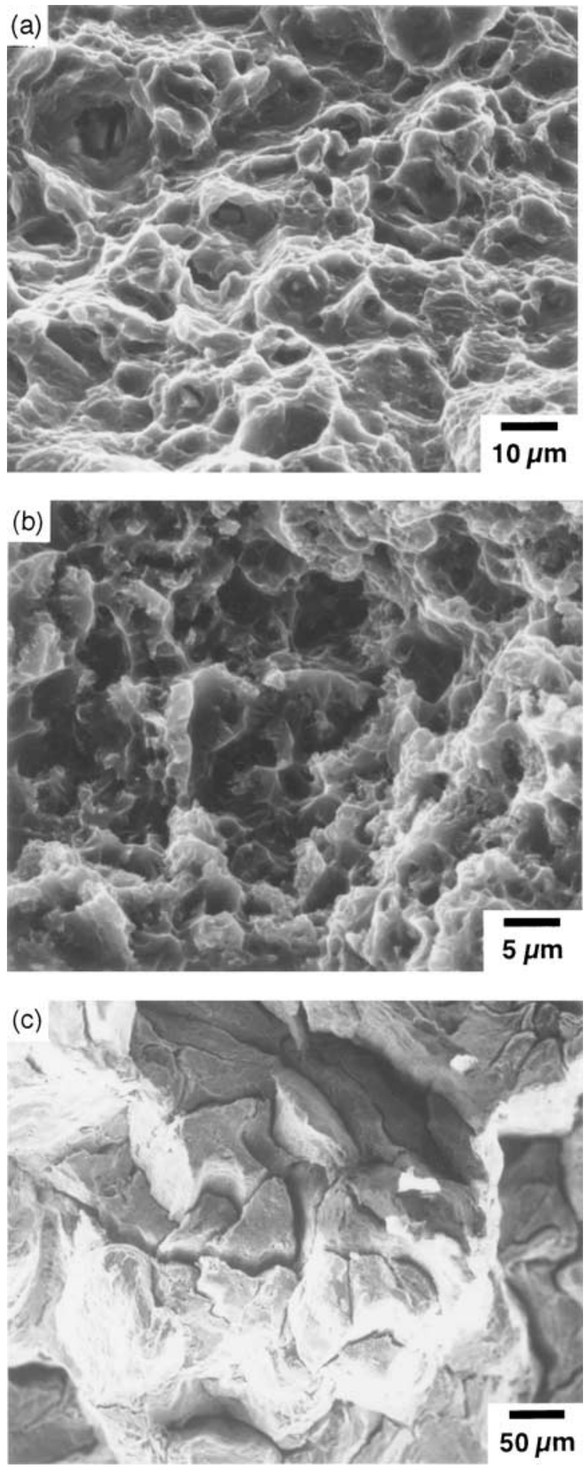

Figure 7 Fracture surfaces of (a) as-received material, (b) 623 K chip-extruded material and (c) 493 K chip-extruded material.

The bonding state is also observed from Figs 3 and 4. The bonding of individual machined chips is likely to be dependent on the deformation mechanisms during extrusion. In the non-superplastic condition of A (Fig. 3), interfaces between chips are clearly observed, indicating that the bonding between chips is weak. On the other hand, in the superplastic conditions of B (Fig. 4) and C, interfaces between chips were not identified and there were no cracks or voids. This result strongly suggests that the extrusion in the superplastic condition is effective to consolidate the machined chips.

4.2. Mechanical properties of extruded materials

The nominal stress – nominal strain curves for chip-extruded materials are shown in Fig. 6. The curve of the as-received material is also included in the figure. It is obvious that a high ultimate tensile strength of about 300 MPa is obtained in chip-extruded materials in the superplastic region. The strength was comparable to the as-received material. In addition, moderate elongation values of about 10% were obtained. On the other hand, chip-extruded material in the non-superplastic region exhibited lower strength and limited ductility. The fracture surfaces of the as-received material, chip-extruded material in the superplastic region (Condition B) and chip-extruded material in the non-superplastic region (Condition A) are shown in Fig. 7. Fracture surfaces for as-received and chip-extruded material in the superplastic region consist of ductile dimples. On the other hand, chip-extruded material in the non-superplastic region is fractured at the interfaces of original machined chips. The results of tensile tests support the good bonding of interfaces between original machined chips for the chip-extruded material in the superplastic region.

5. Summary

1. Utilization of superplastic flow for the consolidation of machined chips by hot extrusion in AZ31 magnesium alloy was investigated for the purpose of enhancing the bonding between individual chips in order to improve the mechanical properties.

2. Grain refinement was attained by extruding machined chips. The grain size decreased with decreasing extrusion temperature. The grain sizes of the chip-extruded materials were smaller than 5 μm.

3. The chip-extruded material in the non-superplastic region was not consolidated successfully. The interfaces of original chips were clearly identified, and this resulted in the limited ductility of extruded material.

4. The chip-extruded materials in the superplastic region exhibited ultimate tensile strength of about 300 MPa and moderate elongations-to-failure of 10%. The interfaces of individual chips were not identified. It was confirmed that chip consolidation utilizing superplastic flow is useful to enhance the bonding of individual chips, and thus to obtain high strength and good ductility.

Acknowledgment

The authors like to express their gratitude to Mr. D. Nakazato in YKK corporation who performed TEM observations.

References

1. R. S. BUSK and T. E. LEONTIS, *Trans. AIME* **188** (1950) 297.
2. M. MABUCHI, K. KUBOTA and K. HIGASHI, *J. Mater. Sci. Lett.* **12** (1993) 1831.
3. *Idem., J. Japan Soc. Powder and Powder Metall.* **40** (1993) 397.
4. M. NAKANISHI, M. MABUCHI, K. KUBOTA and K. HIGASHI, *ibid.* **42** (1995) 373.
5. M. MABUCHI, K. KUBOTA and K. HIGASHI, *Mater. Trans., JIM* **36** (1995) 1249.
6. D-M. LEE, J-S. LEE and C-H. LEE, *J. Japan Inst. Light Metals* **45** (1995) 391.
7. U. DRAUGELATES, A. SCHRAM and C.-C. KENDENBURG, in Magnesium Alloys and their Applications, Wolfsburg, April 1998, edited by B. L. Mordike and K. U. Kainer (Werkstoff - informaitonsgesellschaft, Frankfurt, 1998) p. 381.
8. K. KUBOTA, M. MABUCHI and K. HIGASHI, *J. Mater. Sci.* **34** (1999) 2255.
9. H. WATANABE, H. HOSOKAWA, T. MUKAI and T. AIZAWA, *Materia Japan* **39** (2000) 347.
10. O. A. RUANO, J. WADSWORTH and O. D. SHERBY, *Metall. Trans. A* **13A** (1982) 355.
11. K. ISONISHI and M. TOKIZANE, *J. Japan Inst. Metals* **49** (1985) 149.
12. W. A. WONG and J. J. JONAS, *Trans. Metall. Soc. AIME* **242** (1968) 2271.
13. R. S. MISHRA, T. R. BIELER and A. K. MUKHERJEE, *Acta Metall. Mater.* **43** (1995) 877.
14. O. D. SHERBY and J. WADSWORH, *Prog. Mater. Sci.* **33** (1989) 169.
15. H. J. FROST and M. F. ASHBY, "Deformation-mechanism Maps" (Pergamon Press, Oxford, 1982) p. 44.

Received 26 May 2000
and accepted 9 July 2001