JOURNAL OF MATERIALS SCIENCE 27 (1992) 937–940

Possibility of grain refinement for superplasticity of a Mg–Al–Zn alloy by pre-deformation

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For a commercial Mg–Al–Zn alloy sheet, tensile tests are carried out under various strains, strain rates and temperatures to investigate the possibility of grain refinement by dynamic recrystallization during pre-deformation. It is found from the microstructural observations that relatively fine grains of 10 μm or so, in diameter are attained under the conditions of 250 °C and 8.3×10^{-4} s^{-1}. The specimen pre-strained under this condition also exhibits a fairly good superplasticity of total elongation beyond 300%.

1. Introduction

In recent years the characteristics of superplasticity have been reported for existing commercial alloys, especially for aluminium alloys, such as Al–Cu [1, 2], Al–Zn–Mg–Cu [3–6] and Al–Li alloys [7, 8], and their superplastic formings are expected to be put into practical use.

Magnesium also has some advantages as a structural material. For instance, magnesium is lighter than aluminium and possesses high rigidity. However, the use of magnesium and its alloys has been limited to a narrow range because of the hard formability. If the superplastic formability is attained in magnesium-based alloys, the use of magnesium will be notably extended, since magnesium itself can be unlimitedly supplied from sea water.

It is known, that the grain boundary sliding which governs the superplastic deformation occurs only when grain sizes are less than 10 μm or so [9, 10]. Therefore, like the other alloys, the grain refinement is considered to be indispensable, in order to give the magnesium-based alloys a high ductility. Backofen *et al.* [11] have attained the grain refinement of a magnesium based alloy ZK60A by pellet consolidation, and reported the achievement of elongation of 1700% at 310 °C and 0.04 s^{-1}. However, only few studies have been found for the superplasticity of magnesium-based alloys [12–14].

In this study, in order to examine the possibility of the grain refinement by pre-deformation of a commercially magnesium-based alloy, the tensile tests are carried out under various temperatures and strain rates. The microstructural changes of the material during the deformation are observed, and the optimal conditions for the grain refinement are investigated in view of realizing superplasticity.

2. Experimental procedure

The material used in this study is a commercial Mg–Al–Zn alloy. The chemical composition of the material is indicated in Table I. The tensile specimens were cut from a plate of 2.0 mm in thickness in such a way as to be parallel to the rolling direction, and the gauge length was set to be 10 mm. Before the tests, the specimens were annealed at 300 °C for an hour in order to arrange the microstructural conditions.

The tensile tests were carried out in an Ar atmosphere, in an infrared image furnace equipped to an Instron type machine. The test temperatures were scheduled at 200, 250, 300, 350, 400, 450 and 500 °C. During the tests the temperatures were controlled with an accuracy of ± 2 °C. The specimens were deformed up to 20, 40 and 60% in elongation, and up to fracture, respectively, by a constant crosshead velocity between 0.05 and 50 mm min^{-1}. The initial strain rates of the tensile tests ranged between 8.3×10^{-5} and 8.3×10^{-2} s^{-1}.

The microstructures of the specimens after elongations of 20, 40 and 60% and after fracture were observed by optical microscopy. The grain size was measured by the linear intercept method, and the mean grain diameter, d, can be determined by the following conversion [15]:

$$d = 1.74 \frac{l}{MN}$$

where l is the total line length employed, M is the magnification of the photograph, and N is the number of boundaries intersected by the test line.

The effect of the grain refinement on the ductility was examined by additional tensile tests, and these will be described later in the discussion.

3. Results

Fig. 1 shows the microstructure of the specimen before tensile testing. The grains are equiaxial and the mean grain diameter is approximately 33 μm. Thus, the grain size is considered too large to cause superplasticity.

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TABLE I Chemical composition of the material used (wt %)

Al	Zn	Mn	Fe	Mg
1.86	0.79	0.053	0.001	bal.

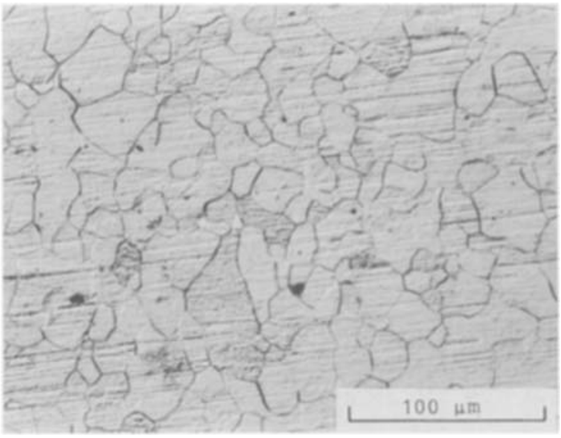

Figure 1 Microstructure of specimen before tensile test.

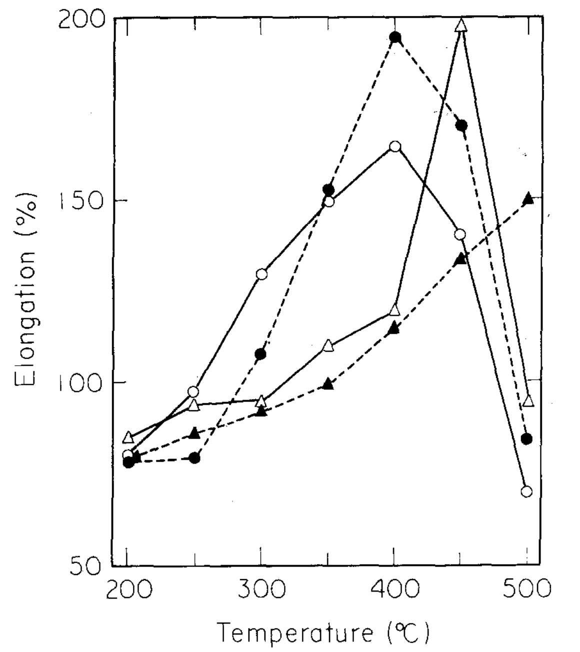

Figure 2 Relations between temperature and fracture elongation for various initial strain rates: (○) 8.3×10^{-5}; (●) 8.3×10^{-4}; (△) 8.3×10^{-3}; (▲) 8.3×10^{-2} s^{-1}.

Fig. 2 shows the relations between the test temperature and the fracture elongation for various initial strain rates. Fairly large elongations, of 200% or so, are obtained at 400 °C and 8.3×10^{-4} s^{-1}, at 450 °C and 8.3×10^{-3} s^{-1}, and at 450 °C and 8.3×10^{-4} s^{-1}. These values are not large enough, but suggest the possibility of superplasticity. Again, there is a tendency to obtain a large elongation at higher temperatures with increasing strain rate. At lower temperatures, below 250 °C, the fracture elongations cannot reach 100%, independently of the strain rate.

The strain rate sensitivity exponent, i.e. m value, was also measured, and it turned out that the m value is always lower than 0.3 in this test range. From the above results it is considered that grain refinement, by an appropriate thermomechanical treatment, must be performed to attain the superplasticity.

Next, the effects of the working conditions on the microstructural change are examined. Fig. 3 shows the mean grain diameters after fracture by the tensile tests at various temperatures and initial strain rates. It is found that, at temperatures higher than 350 °C, the grain size becomes larger by grain growth with the temperature for all strain rates. This tendency is quite remarkable in cases of 8.3×10^{-5} as well as 8.3×10^{-2} s^{-1}. Fig. 4 shows a typical microstructure for the case where the temperature is high. The grains reach approximately 100 μm due to the grain growth. Grain refinement seems to be impossible at a temperature region over 350 °C, especially over 400 °C, due to the rapid grain growth.

At a temperature of 200 °C, the grain refinement is recognized at lower strain rates (see Fig. 3). Fig. 5 shows the microstructure at 8.3×10^{-5} s^{-1}. The fine recrystallized grains are formed along the grain

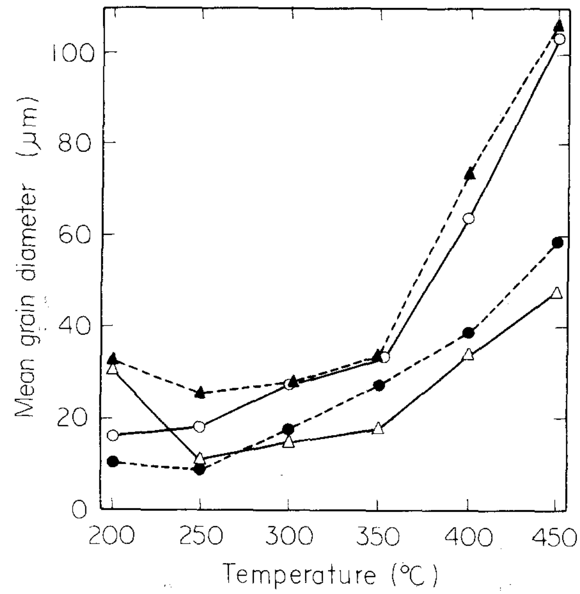

Figure 3 Mean grain diameters of specimens at fracture by the tensile tests for various temperatures and strain rates: (○) 8.3×10^{-5}; (●) 8.3×10^{-4}; (△) 8.3×10^{-3}; (▲) 8.3×10^{-2} s^{-1}.

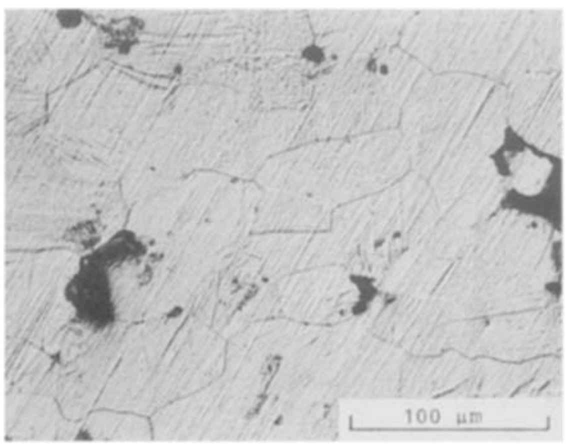

Figure 4 Microstructure of specimen at fracture under the conditions of 450 °C and 8.3×10^{-5} s^{-1} initial strain rate.

boundaries. However, fairly large grains remain, which are stretched in the tensile direction. It is considered that a temperature of 200 °C is a little too low for obtaining complete recrystallization during the deformation.

From the above-mentioned results, it does not seem that temperatures below 200 °C, as well as beyond 350 °C, are suitable for the grain refinement by pre-deformation. Therefore, we examine the microstructural changes during deformation at 250 and 300 °C.

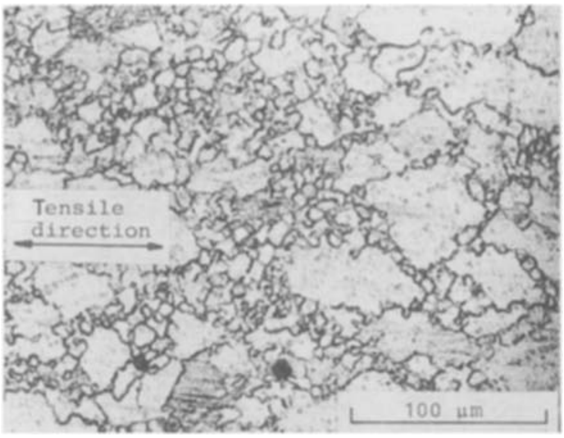

Figure 5 Microstructure of specimen at fracture under the conditions of 200 °C and $8.3 \times 10^{-5}\ s^{-1}$ initial strain rate.

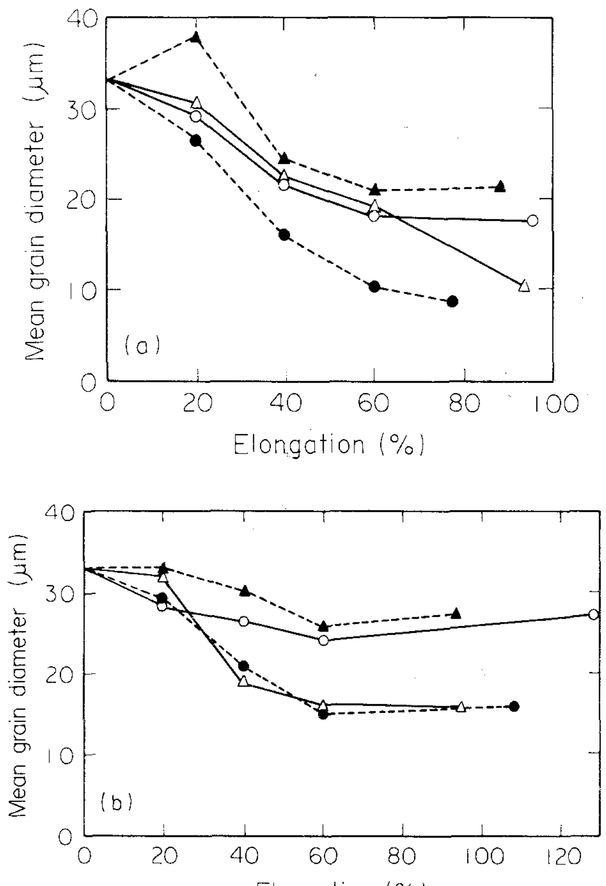

Figure 6 Changes of mean grain diameter during deformation by the tensile tests at: (a) 250 and (b) 300 °C for various initial strain rates (○) 8.3×10^{-5}; (●) 8.3×10^{-4}; (△) 8.3×10^{-3}; (▲) $8.3 \times 10^{-2}\ s^{-1}$.

Fig. 6a and b give the behaviours of the mean grain diameter during deformation at 250 and 300 °C, respectively. In most cases, the grain size tends to become smaller with an increase in strain. The finer grains are obtained at 250 °C, and fairly good grain refinement is attained at an initial strain rate of $8.3 \times 10^{-4}\ s^{-1}$. When the strain rate is higher than that, large grains remain elongated in the tensile direction, similar to the case of Fig. 5.

Fig. 7a–d are the micrographs of the specimens at elongations of 20, 40, 60%, and at fracture, respectively, under the conditions of temperature at 250 °C and the initial strain rate at $8.3 \times 10^{-4}\ s^{-1}$. Judging from these micrographs, at first the recrystallization takes place only at the grain boundaries, and subsequently the grain refinement proceeds over the whole structure with increasing strain. Concretely, the mean grain diameter reaches 16.2 μm at 40%, 10.3 μm at 60% elongation and 8.7 μm at fracture (at about 80% elongation).

4. Discussion

In this study, to examine the possibility of the grain refinement by pre-deformation of the commercial Mg–Al–Zn alloy, the tensile tests were carried out at various temperatures and strain rates. It turned out from the microstructural observations that fine grains, smaller than 10 μm in diameter, are produced under appropriate conditions of strain, strain rate and temperature, as shown in Figs 6 and 7.

It must be confirmed whether the grain refinement in this case causes the high ductility, i.e. the superplasticity. With increase in strain the grains become finer, as shown in Fig. 6. However, in reality it becomes difficult to achieve uniform strain over the whole section of specimens through the tensile deformation due to necking. Therefore, additional tensile tests are carried out using the specimens which were deformed up to the elongation of 40% under the conditions of 250 °C and $8.3 \times 10^{-4}\ s^{-1}$. The specimens are elongated further under the three kinds of tensile conditions such that the large fracture elongations were obtained in the previous tensile tests (see Fig. 2).

The results so obtained, exhibit the high ductility of the fine grained specimens. The total elongations of 345, 335 and 315% are attained at 400 °C and $8.3 \times 10^{-4}\ s^{-1}$, at 450 °C and $8.3 \times 10^{-3}\ s^{-1}$, and at 450 °C and $8.3 \times 10^{-4}\ s^{-1}$, respectively.

It is obvious that grain refinement by pre-deformation has a positive effect on the superplasticity. However, magnesium-based alloys are subject to grain growth at high temperature, which reduces superplasticity. The inhibition of the grain growth may be essentially indispensable to attain the high superplasticity in magnesium-based alloys. The alloy ZK60A, in which Backofen *et al.* [11] have reported high superplasticity, contains 0.5% zirconium. The addition of grain-refining and stabilizing elements, such as zirconium, may be a meritorious means for the high superplasticity of the Mg–Al–Zn alloy.

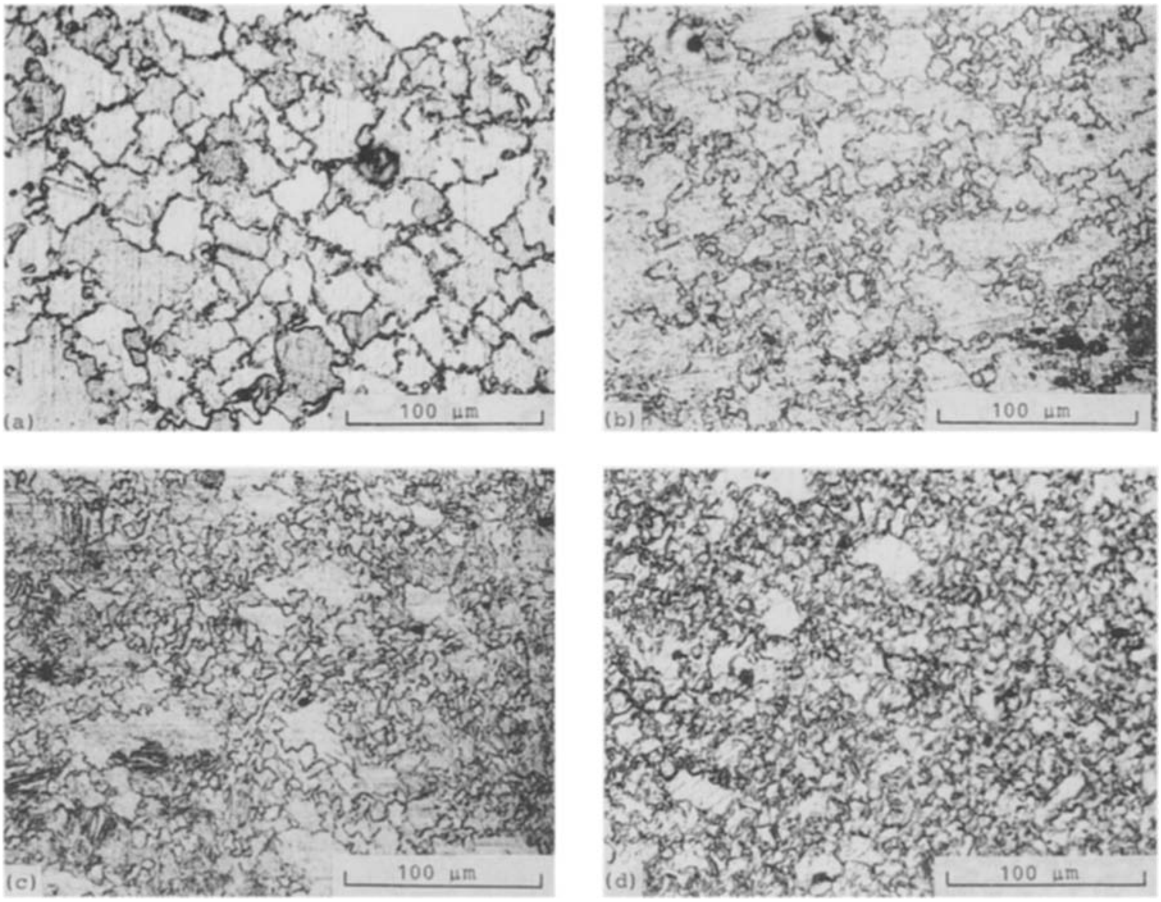

Figure 7 Microstructural change during deformation by the tensile test under the conditions of 250 °C and 8.3×10^{-4} s^{-1} initial strain rate. (a) 20%, (b) 40% and (c) 60% elongation; (d) at fracture.

Acknowledgements

The authors are grateful to Messers S. Murata, T. Fujimura and K. Nishijima for experimental assistance. The first author also acknowledges the financial support from the Japanese Ministry of Education with Grand-in-Aid for Encouragement of Young Scientists. The material used was provided by Ube Industries, Ltd.

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Received 8 October 1990
and accepted 20 March 1991