Materials Transactions, Vol. 43, No. 10 (2002) pp. 2437 to 2442
Special Issue on Superplasticity and Its Applications
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Superplasticity and Cavitation of Recycled AZ31 Magnesium Alloy Fabricated by Solid Recycling Process

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Superplastic behavior and cavitation have been investigated for the solid recycled AZ31 magnesium alloy made of machined chips. The elongation to failure of the solid recycled specimen was lower than that for the extruded specimen from a virgin block (virgin extruded specimen) at elevated temperature, although the strain rate sensitivity for the solid recycled specimen was the same as that for the virgin extruded specimen. The volume fraction of cavities and the total number of cavities for the solid recycled specimen were larger than those for the virgin extruded specimen. Moreover, cavity nucleation occurred not only at grain boundaries, but also in the grains for the solid recycled specimen. It is suggested that contamination of oxide particles at grain boundaries and in grains promotes cavity nucleation in the solid recycled specimen and adversely affects the elongation to failure.

(Received April 19, 2002; Accepted August 7, 2002)

Keywords: *magnesium alloy, superplasticity, cavitation, solid recycling process, hot extrusion, grain refinement, grain boundary sliding*

1. Introduction

Magnesium alloys are currently the lightest alloys used as structural metals and they have the advantages of, for example, specific strength and specific elastic modulus. Magnesium alloy products have been applied to such uses as automobile parts and electric appliance cases.[1,2] In order to increase the applicability of magnesium alloys, it is necessary not only to attain good characteristics (high strength, high ductility, high corrosion resistance, creep resistance, and so on), but also to develop useful recycling processes.[3] Some recycling processes for magnesium alloys, such as remelting,[4,5] electrorefining in molten salt[6] and vacuum distillation,[7] have already been proposed and some processes have been applied in actual recycling.[4,5]

Recently, we proposed the "solid recycling process" as a new method of recycling metals.[8–10] In the solid recycling process, metal scraps are recycled by consolidation using plastic deformation processes such as hot extrusion and forging. It should be noted that remelting is not required in the solid recycling process. Furthermore, the solid recycled magnesium alloy shows high strength due to grain refinement and homogeneous dispersion of the oxide film on the surface of scraps.[8] Thus, the solid recycling process leads not only reduction of the energy required for recycling, but also improvement of the mechanical properties.

In general, magnesium alloys show poor ductility and formability in a solid state due to the HCP; this prevents the practical use of wrought products. However, it has been reported that grain refinement is attained by hot deformation due to dynamic recrystallization in magnesium alloys[8–15] and that grain refinement leads to superplasticity and excellent post-deformation mechanical properties.[8–15] Superplastic forming is expected to be applied to the actual processing for magnesium products. A large number of studies on the superplastic behavior of magnesium alloys have been reported.[8,15–21] However, studies on the superplastic behavior of recycled magnesium alloys are few.[8,16] Mabuchi *et al.*[8] investigated the superplastic behavior of AZ91 magnesium alloy recycled by the solid recycling process, and showed that the solid recycled alloy shows a superplastic behavior, but that the elongation to failure of the solid recycled specimens is lower than that of the extruded specimen processed from an as-cast ingot. They suggested that the lower elongation for the solid recycled alloy may be attributed to cavitation induced by the dispersed oxides. In the present study, we focus our attention on the cavitation during superplastic deformation and relationships between cavitation and superplastic behavior of the solid recycled AZ31 magnesium alloy were investigated.

2. Experimental Procedure

Chips were prepared as magnesium alloy scraps by machining an as-received AZ31 (Mg–2.9 mass%Al–0.85 mass%Zn–0.46 mass%Mn) magnesium alloy in a lathe without lubricants. These machined chips are shown in Fig. 1. The machined chips were filled into a container with a diameter of 40 mm and extruded at 503 K with an extrusion ratio of 44:1 in air, that is, degassing treatments was not done in the process. For comparison, extrusions were processed from an as-received AZ31 magnesium alloy block under the same conditions as the extrusions from machined chips. In the present study, extrusions from machined chips are called solid recycled specimens and those from as-received blocks are called virgin extruded specimens.

Tensile tests at room temperature were carried out at strain rate of $1.7 \times 10^{-3}\,s^{-1}$ for the investigation of mechanical properties. Superplastic behaviors were investigated by tensile tests at elevated temperature of 623 K and at strain rates from $3.3 \times 10^{-4}\,s^{-1}$ to $3.3 \times 10^{-2}\,s^{-1}$. The tested specimens had

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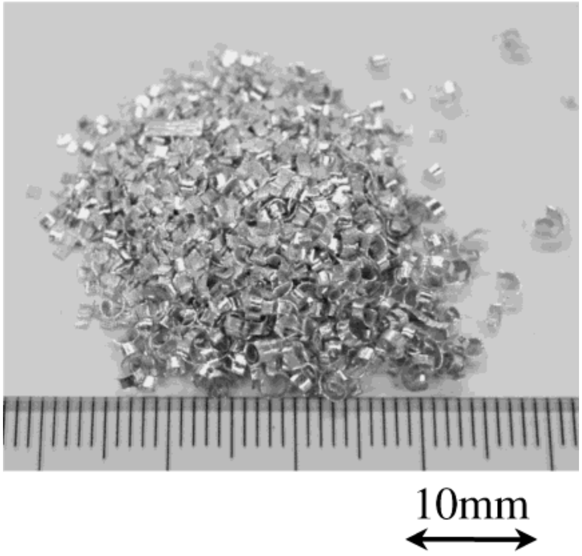

Fig. 1 Photograph of machined chips of AZ31 magnesium alloy.

a gauge length of 10 mm and a gauge diameter of 2.5 mm for both the tests. The tensile axis was parallel to the direction of extrusion. The temperature variation during the tensile tests at elevated temperature was not more than 1 K.

A metallographic investigation was carried out by optical and scanning electron microscopies. The grain size of the specimens was measured by optical microscopy using the linear intercept method. Specimens for the observation were etched using a solution of 20 ml acetic acid, 3 g picric acid, 50 ml ethanol and 30 ml distilled water. Cavities were investigated by optical microscopy. Cavity size distributions were examined based on quantitative metallographic measurements, assuming a spherical shape. The volume fraction of cavities in the gauge length of the deformed specimens was measured by hydrostatic weighing in water, using a corresponding gauge head as a density standard. Also, the grain boundary sliding was investigated by scanning electron microscopy.

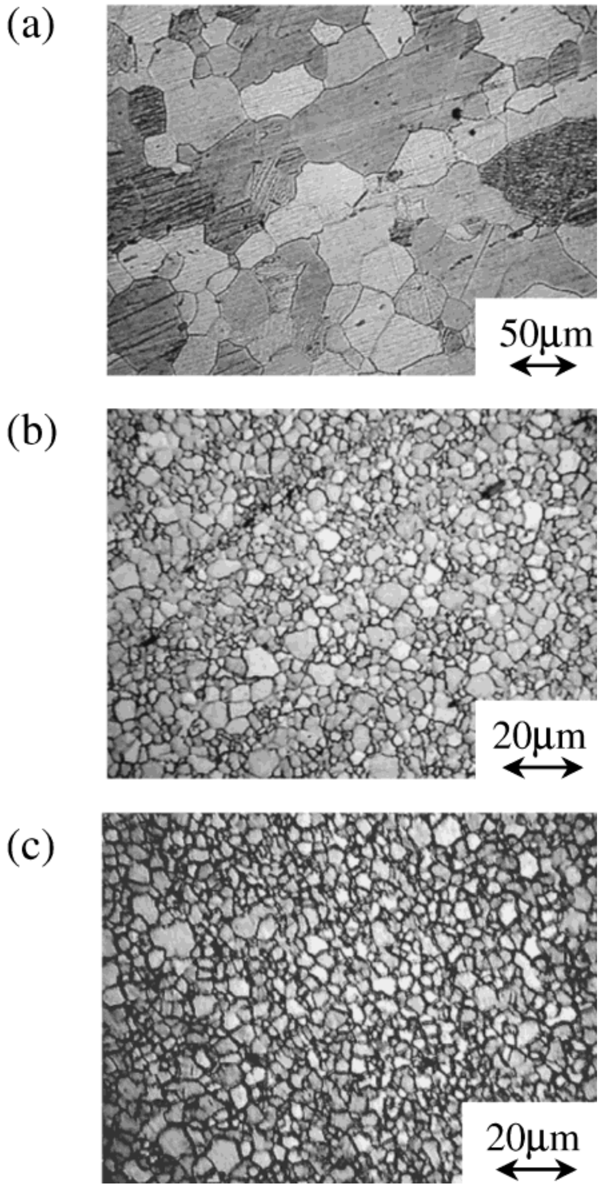

Fig. 2 Microstructure of AZ31 magnesium alloy specimens: (a) the as-received specimen, (b) the solid recycled specimen, (c) the virgin extruded specimen.

3. Results and Discussion

3.1 Microstructure and mechanical properties

Microstructures of the as-received specimen (a), the solid recycled specimen (b) and the virgin extruded specimen (c) are shown in Fig. 2. In the as-received specimen, equiaxed coarse grains were observed. The mean size of the grains was 52 μm. On the other hand, a very small grain size of 3.5 μm was obtained for both the solid recycled specimen and the virgin extruded specimen. Mohri *et al.*[11] investigated microstructural evolution during hot deformation in a AZ91 magnesium alloy, and they revealed that a fine-grained microstructure was obtained due to continuous dynamic recrystallization. Therefore, it is suggested that the grain refinement of the extruded specimens is attributed to dynamic recrystallization during extrusion.

By annealing at 623 K for 1 h, the grain size increased from 3.5 μm to 7.0 μm in both specimens. This indicates that the dispersed oxides for the solid recycled specimen have no influence on grain growth behavior. Detailed investigations for effects of dispersed oxides on the grain growth behavior are still in progress.

Mechanical properties of the as-received specimen, the solid recycled specimen and the virgin extruded specimen are summarized in Table 1, respectively. The solid recycled specimen showed a good combination of high ultimate tensile strength of 312 MPa, 0.2% proof stress of 247 MPa and elongation to failure of 22% compared with those of the as-received specimen. These values are almost the same as those of the virgin extruded specimen. The good mechanical properties for the solid recycled specimen are likely attributed to grain refinement and dispersion of oxide layers by hot extrusion and these results are in agreement with the previous works by AZ91 magnesium alloy.[8]

3.2 Superplasticity

The variations in elongation to failure (top figure) and flow stress (bottom figure) at 623 K for the solid recycled speci-

Table 1 Tensile properties at room temperature of the AZ31 magnesium alloys.

Alloy	Ultimate tensile strength (MPa)	0.2% Proof stress (MPa)	Elongation to failure (%)
As-received specimen	247	182	19
Solid recycled specimen	312	247	22
Virgin extruded specimen	308	249	25

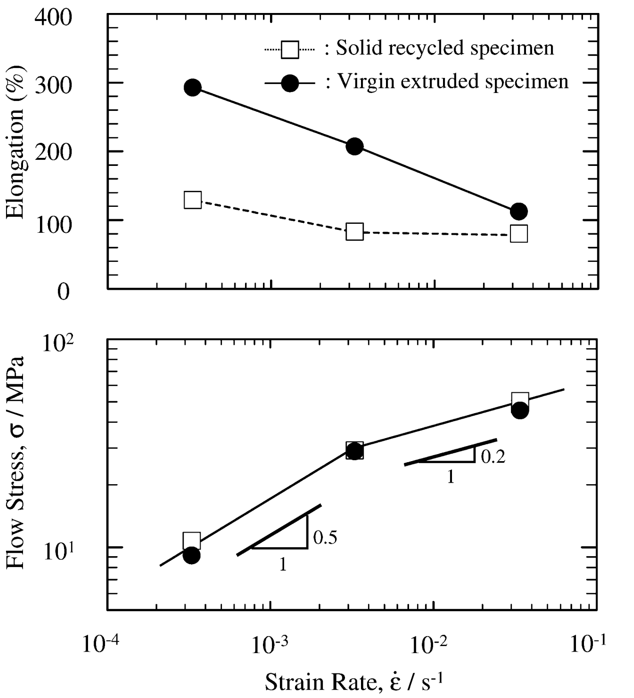

Fig. 3 The variation in elongation to failure (top figure) and flow stress (bottom figure) at 623 K as a function of strain rate for the solid recycled specimen and the virgin extruded specimen.

men and the virgin extruded specimen are shown in Fig. 3 as function of strain rate, where the flow stress was determined at $\varepsilon = 0.1$. The elongations of the virgin extruded specimen were 300% at $3.3 \times 10^{-4}\,s^{-1}$ and 110% at $3.3 \times 10^{-2}\,s^{-1}$, respectively. On the other hand, lower elongations of 130% at $3.3 \times 10^{-4}\,s^{-1}$ and 80% at $3.3 \times 10^{-2}\,s^{-1}$ were obtained for the solid recycled specimen.

The outward appearances of tensile specimens deformed to fracture at $3.3 \times 10^{-4}\,s^{-1}$ are shown in Fig. 4. The fracture surface of the virgin extruded specimen showed diffusional necking. However, the one of the solid recycled specimen showed premature fracture.

The relationship between the flow stress and the strain rate can be given by

$$\sigma = K\dot{\varepsilon}^m \tag{1}$$

where σ is the flow stress, K a constant, $\dot{\varepsilon}$ the strain rate and m the strain rate sensitivity. In general, large elongation is obtained in the strain rate range where a high m value is observed. A high m value of about 0.5 was found in the strain rate range of 10^{-4}–$10^{-3}\,s^{-1}$ for both the solid recycled specimen and the virgin extruded specimen. This suggests that grain boundary sliding[22–24)] is the dominant deformation process at 10^{-4}–$10^{-3}\,s^{-1}$ for both the solid recycled specimen and the virgin extruded specimen.

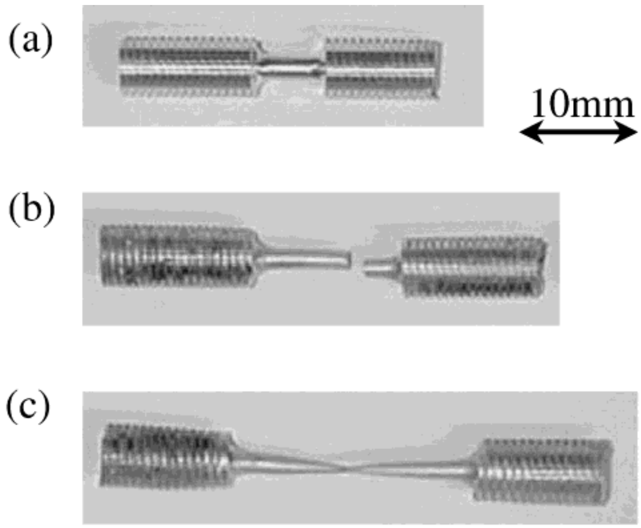

Fig. 4 Outward appearance of the AZ31 magnesium specimens deformed to fracture at $3.3 \times 10^{-4}\,s^{-1}$ and 623 K: (a) the undeformed specimen, (b) the solid recycled specimen, (c) the virgin extruded specimen.

Figure 5 shows the side surfaces of the tensile specimens deformed at $3.3\times10^{-4}\,s^{-1}$. A bumpy surface, which indicates the occurrence of grain boundary sliding, was observed for both the specimens. It is obvious that grain boundary sliding occurred in the strain rate range of 10^{-4}–$10^{-3}\,s^{-1}$ for both the specimens. Thus, there is no difference in deformation mechanism at 10^{-4}–$10^{-3}\,s^{-1}$ between the solid recycled specimen and the virgin extruded specimen. The origin of the large difference in elongation at $10^{-4}\,s^{-1}$ between the solid recycled specimen and the virgin extruded specimen is clearly not the deformation mechanism.

In a high strain rate range of 10^{-3}–$10^{-2}\,s^{-1}$, the low m value of about 0.2 was obtained and little grain boundary sliding was observed in both the solid recycled specimen and the virgin extruded specimen, indicating that there is no difference in deformation mechanism between the solid recycled specimen and the virgin extruded specimen in a strain rate range of 10^{-3}–$10^{-2}\,s^{-1}$ as well as 10^{-4}–$10^{-3}\,s^{-1}$. It is of interest to note that the difference in elongation at a high strain rate of $10^{-2}\,s^{-1}$ between the solid recycled specimen and the virgin extruded specimen is somewhat smaller than that at a low strain rate of $10^{-4}\,s^{-1}$. One reason for the difference in elongation might be suggested that grain boundary sliding can not be accommodated by diffusion processes in a solid state at high strain rate.

3.3 Cavitation

The cavity volume fraction was investigated at a strain rate of $3.3 \times 10^{-4}\,s^{-1}$ where there was a large difference in elongation between the solid recycled specimen and the virgin extruded specimen. The variation in the cavity volume fraction as a function of strain is shown in Fig. 6. The volume fraction of cavities for the solid recycled specimen was higher than that for the virgin extruded specimen. This indicates that significant development of cavities is responsible for the smaller

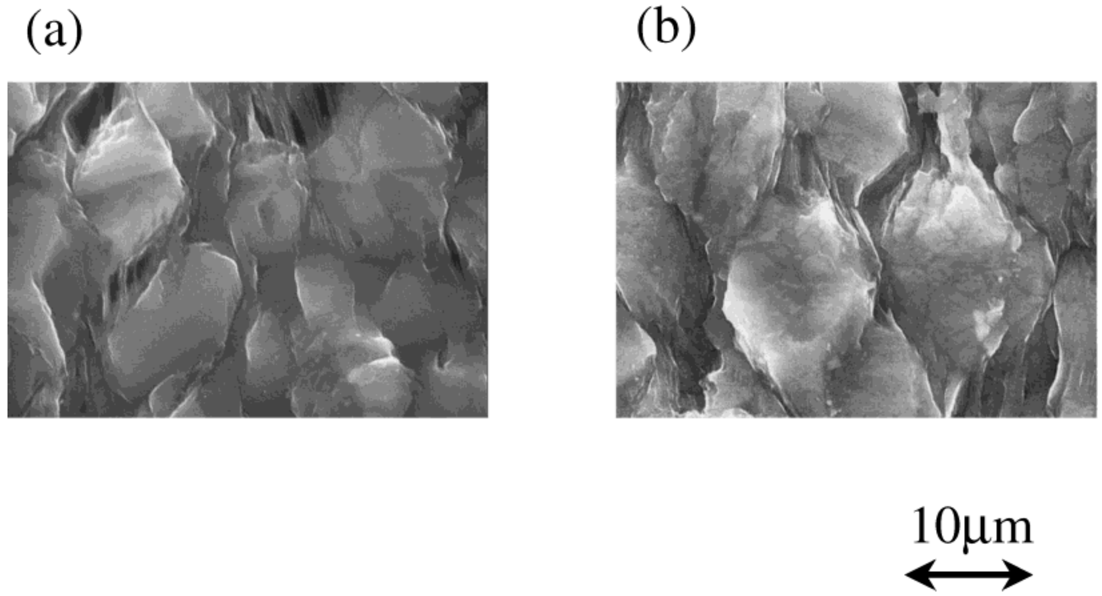

Fig. 5 The side surface of the tensile specimens deformed to fracture at $3.3 \times 10^{-4}\,s^{-1}$ and 623 K: (a) the solid recycled specimen, (b) the virgin extruded specimen.

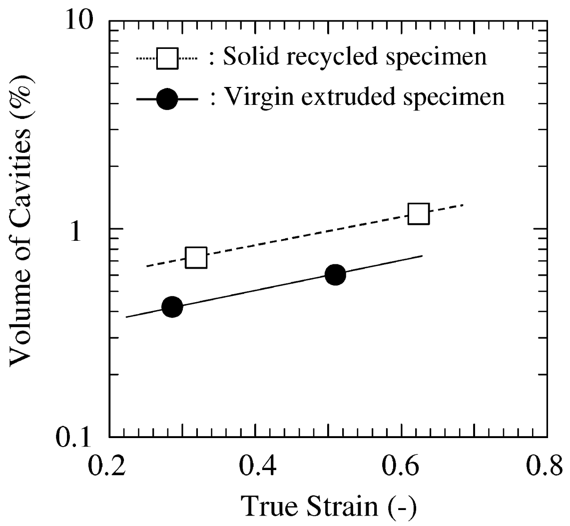

Fig. 6 The variation in cavity volume fraction as a function of true strain for the AZ31 magnesium alloy specimens deformed at $3.3 \times 10^{-4}\,s^{-1}$ and 623 K.

elongation of the solid recycled specimen.

Cavitation is often observed in a wide range of superplastic materials.[25–28] Cavities grow during straining, resulting in premature fracture. When cavity growth is strain-controlled, the volume of cavities increases exponentially with strain. This relationship is represented by

$$C_V = C_{V0}\exp(\eta\varepsilon) \quad (2)$$

where C_V is the volume fraction of cavities, C_{V0} is the volume of cavities at zero strain and η is the cavity growth-rate parameter.[29,30] The data for each specimen fitted the line of logarithmic C_V plotted against ε, as shown in Fig. 6. The value of η for the solid recycled specimen was 1.7. This value is almost the same as that for the virgin extruded specimen. It is of interest to note that the cavity growth rate for the solid recycled specimen is the same as that for the virgin extruded specimen, though the cavity volume fraction of the former is higher than that of the latter.

The value of η often has a value in the range from 2 to 4 for superplastic aluminum alloys though it is dependent on the chemical compositions, strain rate, temperature, and grain size.[29,31–33] However, the values of η for the magnesium alloy measured in the present experiment were lower than those for aluminum alloys. Detailed investigation of the cavity growth behavior in magnesium and aluminum alloys is still in progress.

The cavity diameter distributions for both the specimens are shown in Fig. 7, where the specimens were deformed to $\varepsilon = 0.30$ and 0.55 at $3.3 \times 10^{-4}\,s^{-1}$, respectively. For both the specimens, the number of large cavities increased with strain while the total number of cavities decreased with strain, suggesting that greater cavity nucleation occurred in the initial stage of the deformation process less than $\varepsilon = 0.30$. Mabuchi *et al.*[8] investigated oxide dispersion in the solid recycled AZ91 magnesium alloy and they showed that the oxide film on the machined chips is dispersed finely and homogeneously during hot extrusion. In the present study, the total number of cavities in the solid recycled specimen was larger than that in the virgin extruded specimen, particularly, in the initial stage of deformation. Therefore, it is suggested that the dispersed oxides promoted cavity nucleation in the solid recycled specimen.

Figure 8 shows the microstructures of both the specimens deformed to $\varepsilon = 0.55$ at $3.3 \times 10^{-4}\,s^{-1}$. In general, cavities are formed at grain boundaries in superplastic materials because cavity formation is attributed to stress concentration caused by grain boundary sliding.[25–28] For the solid recycled specimen, however, cavities were observed in the interior of grains as well as at grain boundaries. Watanabe *et al.*[20] noted that dislocation movement in the interior of grains plays an important role in accommodating grain boundary sliding in superplastic magnesium alloys. Therefore, it is suggested that such dislocations are piled at the oxide particles dispersed in

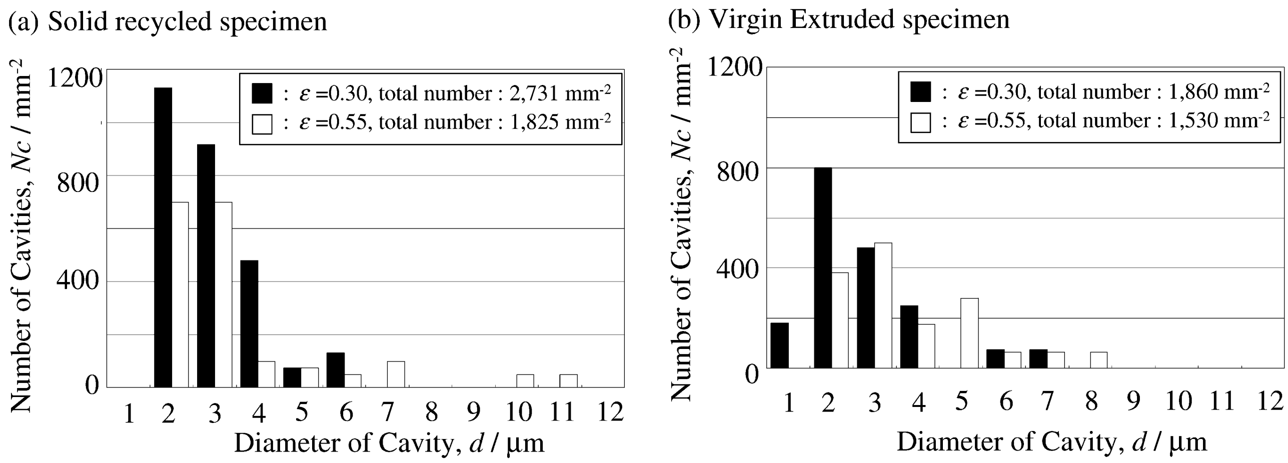

Fig. 7 Cavity size distributions for the AZ31 magnesium alloys specimens deformed at $3.3 \times 10^{-4}\,s^{-1}$ and 623 K: (a) the solid recycled specimen, (b) the virgin extruded specimen.

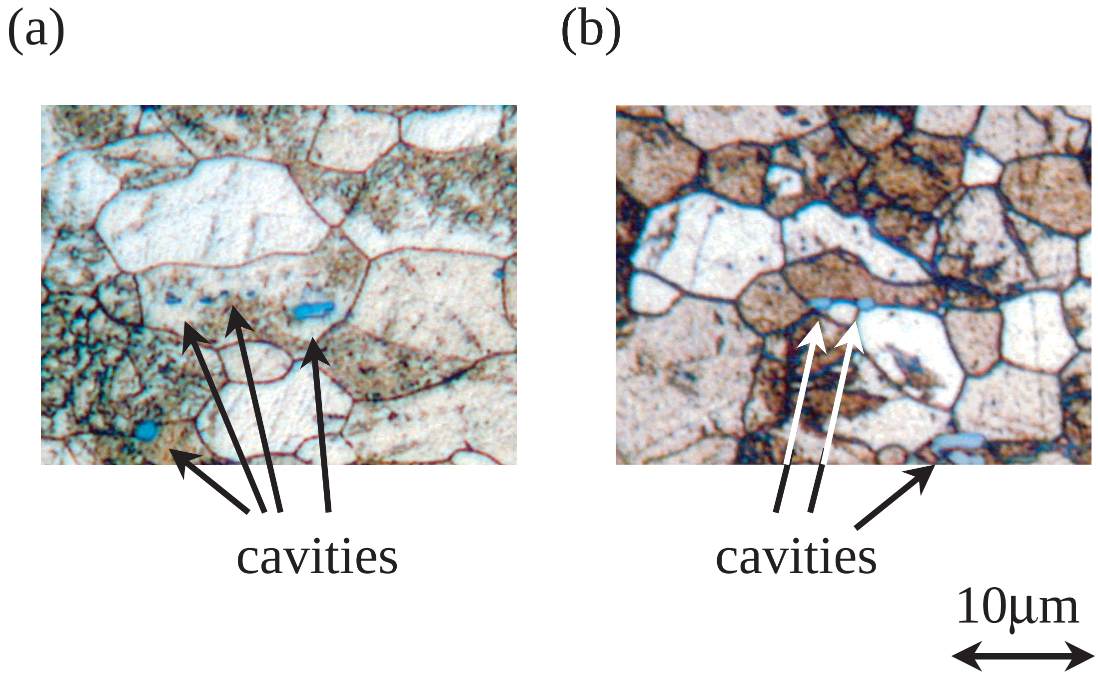

Fig. 8 Microstructures of the specimens deformed to $\varepsilon = 0.55$, where the specimens were deformed at $3.3 \times 10^{-4}\,s^{-1}$ and 623 K: (a) the solid recycled specimen and (b) the virgin extruded specimen.

grains, resulting in cavity formation in the interior of grains in the solid recycled specimen. It is demonstrated that contamination of oxide particles at grain boundaries and in grains promotes cavity nucleation, resulting in lower elongation of the solid recycled specimen.

4. Conclusions

Superplasticity and cavitation of a recycled AZ31 magnesium alloy fabricated by the solid recycling process have been investigated. The results are summarized as follows.

(1) Grains were refined to a mean size of about 3.5 μm by hot extrusion at 503 K for the solid recycled specimen as well as the virgin extruded specimen. The tensile properties of the solid recycled specimens and the virgin extruded specimens have a good combination of ultimate tensile strength, 0.2% proof stress and elongation to failure compared with that of the as-received specimens.

(2) The elongation to failure of the solid recycled specimen was lower than that of the virgin extruded specimen at $3.3 \times 10^{-4}\,s^{-1}$ and 623 K though the strain rate sensitivity was the same for both the specimens.

(3) The volume fraction of cavities and the total number of cavities in the solid recycled specimen were larger than those in the virgin extruded specimen.

(4) Cavity nucleation occurred not only at grain boundaries, but also in the grains in the solid recycled specimen, suggesting that the dislocations for accommodation are piled at the oxide particles which are dispersed in grains.

(5) It is suggested that contamination of oxide particles at grain boundaries and in grains promotes cavity nucleation in the solid recycled specimen and adversely affects the elongation to failure.

Acknowledgements

M. M. gratefully acknowledges the financial support from the project “Barrier-Free Processing of Materials for Life-Cycle Design for Environment” by Ministry of Education, Culture, Sports, Science and Technology of Japan. Also, Y. C. gratefully acknowledges the financial support by Industrial Technology Research Grant Program in 2002 from the New Energy and Industrial Technology Development Organization (NEDO) of Japan.

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