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Blow Forming of Mg Alloy Recycled by Solid-State Recycling*

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Blow forming characteristics of AZ31 Mg alloy recycled by solid-state recycling were investigated. Cylindrical scraps and machined chips were recycled by hot extrusion and hot rolling in air. Oxide layers were observed in the recycled specimens by oxygen mapping with EPMA (Electron Probe Micro Analyser). The interval of the oxygen layers for the specimen from machined chips was much shorter than that for the specimen from cylindrical scraps. As a result of tensile tests, the mechanical properties of the specimen from cylindrical scraps were found to be almost the same as those of a rolled specimen from a virgin ingot. On the other hand, at elevated temperatures, the elongation of the specimens from machined chips was low, compared with those of the rolled specimens from a virgin ingot. The large amount of oxide contamination is likely to be responsible for the lower elongation of the specimens from machined chips. In blow-forming tests, the specimen from cylindrical scraps exhibited excellent formability similar to the rolled specimen from a virgin ingot. However, the specimen from machined chips showed poor formability. Thus, oxide contamination adversely affected the formability of recycled Mg alloy.

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1. Introduction

Mg alloys are currently the lightest alloys used as structural materials, and Mg products have been applied for structural uses such as automobile parts and electric appliance cases.[1,2] In order to achieve greater applicability of Mg alloys, it is necessary not only to attain good characteristics (high strength, high corrosion resistance, *etc.*), but also to develop useful recycling processes. Some recycling processes such as remelting,[3,4] electrorefining[5] and vacuum distillation[6] have been proposed.

Recently, "solid-state recycling" by hot extrusion has been proposed as a new method for recycling Mg alloy scraps.[7–11] In the recycling process, metal scraps are directly recycled by hot extrusion. The solid recycled Mg alloy from machined chips shows high strength due to grain refinement and dispersion of the oxide layer at the scrap surface.[7] However, the elongation to failure of the solid recycled specimens was lower than that of the extruded specimen processed from an as-cast ingot, particularly, at elevated temperatures.[10] Therefore, it is important to investigate the formability of the solid recycled Mg alloy.

In general, Mg alloys show poor ductility and formability due to the HCP structure. Therefore, superplastic forming is expected to be applied in the actual processing of Mg products. Solid-state recycling of Mg alloy is promising for superplastic forming because the solid recycled Mg alloy possesses fine-grained microstructure. In the present study, we have carried out blow-forming tests[12] on solid recycled AZ31 Mg alloy to investigate its superplastic formability.

2. Experimental Procedure

Machined chips with the average dimensions of $12\,\text{mm} \times 1.9\,\text{mm} \times 80\,\mu\text{m}$ and cylindrical scraps with the dimensions of $\phi 8\,\text{mm} \times 10\,\text{mm}$ of AZ31 (Mg-3 mass%Al-1 mass%Zn-0.5 mass%Mn) Mg alloy were prepared as Mg alloy scraps. The machined chips and cylindrical scraps are shown in Fig. 1. The scraps were filled into a rectangular container with cross-sectional dimensions of $50\,\text{mm} \times 30\,\text{mm}$ and extruded at 673 K with the extrusion ratio of 6:1 in air. For comparison, extrusions were processed from an as-received AZ31 Mg alloy block under the same conditions as used for extrusion from the scraps. The cross-sectional dimensions of the extruded bar was $50\,\text{mm} \times 5\,\text{mm}$. After annealing at 691 K for 2.6×10^5 s, the extruded bars were rolled at 673 K up to the rolling ratio of 80%. The rolling direction was perpendicular to the extrusion direction (see Fig. 2). In the present study, specimens recycled from machined chips and from cylindrical scraps, and specimens from a virgin ingot are called "specimens from machined chips", "specimens from cylindrical scraps" and "virgin specimens", respectively.

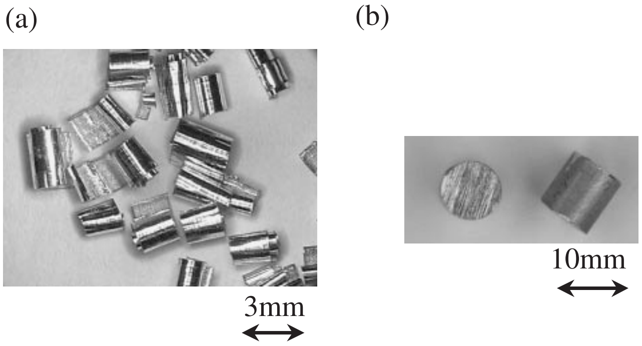

Fig. 1 Photographs of (a) machined chips and (b) cylindrical scraps of AZ31 Mg alloy.

The microstructures of the specimens were observed by optical microscopy. The image of oxygen in the specimens was detected by EPMA. The oxygen concentration in the

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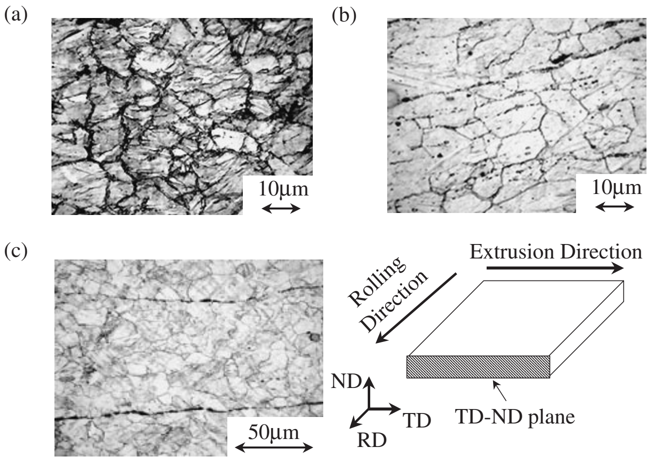

Fig. 2 Microstructures and schematic view of the AZ31 Mg alloy specimens: (a) the virgin specimen, (b) specimen from machined chips and (c) specimen from cylindrical scraps.

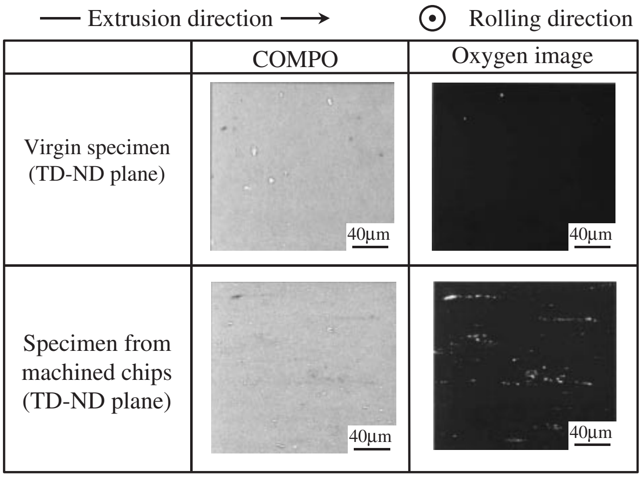

Fig. 3 COMPO images and oxygen images by EPMA analysis of the virgin specimen and the specimen from machined chips.

specimens was measured by the GDMS (Glow Discharge Mass Spectrometry).[13)]

Tensile specimens with 10 mm gage length, 5 mm gage breadth and 1 mm gage thickness were machined. Tensile tests were carried out from room temperature to 723 K with an initial strain rate of $1.7 \times 10^{-3}\,\mathrm{s}^{-1}$ where the angle between the tensile direction and the rolling direction was 0 degrees. Also, step strain rate tests of the virgin specimen were carried out at 673 K at strain rates of 10^{-5}–$10^{-1}\,\mathrm{s}^{-1}$ to investigate superplastic properties.

Discs of 70 mm in diameter were machined from the rolled specimens. The specimens were clamped between two hollow dies and bulged at 673 K using N_2 gas pressure. Domes and cups of height up to 20 mm were formed in the die cavity, which was 40 mm in diameter.

3. Results and Discussion

The microstructures and a schematic view of the rolled specimens are shown in Fig. 2, where the TD-ND plane is observed. The grain sizes of the specimen from machined chips, the specimen from cylindrical scraps and the virgin specimen were 13.3 μm, 14.6 μm and 13.5 μm, respectively. The grain size of the virgin specimen after annealing (prior to rolling) was 294 μm, indicating that the grains were refined by dynamic recrystallization[14)] during hot rolling. It is notable that black (dotted) lines were observed parallel to the TD-ND plane, that is, the extrusion direction in both the specimen from machined chips and the specimen from cylindrical scraps. The black (dotted) lines are related to the oxide surfaces of the scraps, as shown later. The interval of black (dotted) lines was approximately 6 μm for the specimen from machined chips and more than 70 μm for the specimen from cylindrical scraps, respectively.

Figure 3 shows COMPO images and oxygen images obtained by EPMA for the virgin specimen and the specimen from machined chips, where the TD-ND plane is observed. In the specimen from machined chips, local strong peaks of oxygen were observed parallel to the TD-ND plane. The interval of the strong oxygen peaks was approximately 5 to 20 μm, which is in rough agreement with the interval of the black (dotted) lines shown in Fig. 2. Therefore, it is likely that the black (dotted) lines shown in Fig. 2 correspond to the oxide film contaminated during the recycling process.

As a result of the measurements of oxygen concentration by GDMS, the oxygen concentrations in the specimen from machined chips, the specimen from cylindrical scraps and the virgin specimen were found to be 0.3 (mass%), 5.2×10^{-3} (mass%) and 1.4×10^{-3} (mass%), respectively. It is noted that the oxygen concentration in the specimen from machined chips is approximately two hundred times higher than that in the virgin specimen. On the other hand, the specimen from cylindrical scraps showed almost the same value as the virgin specimen, indicating that oxide contamination is very low in the specimen from cylindrical scraps.

The mechanical properties of the rolled specimens at room temperature are summarized in Table 1. The specimen from machined chips showed a high ultimate tensile strength, a high proof stress and a low elongation to failure, compared with the virgin specimen. The contamination of the oxide layers is likely to be responsible for the high strength and low elongation of the specimen from machined chips.[7)] On the other hand, the mechanical properties of the specimen from cylindrical scraps were almost the same as those of the virgin specimen.

The variations in ultimate tensile strength and elongation to failure as a function of the testing temperature in the specimens are shown in Fig. 4. The elongations to failure of

Table 1 Tensile properties at room temperature of the rolled AZ31 Mg alloy specimens.

Raw materials	Ultimate tensile strength (MPa)	0.2% Proof stress (MPa)	Elongation to failure (%)
Virgin specimen	274	178	10
Machined chips	299	192	5.9
Cylindrical scraps	278	181	11

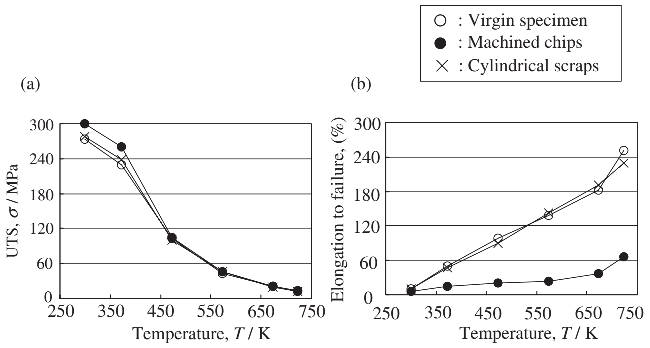

Fig. 4 The variation in (a) ultimate tensile strength and (b) elongation to failure of the rolled specimens of AZ31 Mg alloy as a function of test temperature.

the specimens from machined chips at elevated temperatures of more than 373 K were much lower than those of the virgin specimen. In a previous study,[10] it was shown that the total number of cavities in the specimen recycled from machined chips after tensile tests at 623 K was much larger than that in the virgin specimen. It is suggested that the large amount of contamination of the oxide layers, as shown in Fig. 2, causes excessive cavity nucleation and has an adverse effect on the elongation to failure, resulting in the smaller elongation of the specimen from machined chips. On the other hand, the elongations of the specimens from cylindrical scraps were almost the same as those of the virgin specimen at all testing temperatures.

The variation in stress at 673 K as a function of strain rate for the virgin specimen is shown in Fig. 5. In the previous study,[10] there was no difference in the deformation mechanism between the specimen recycled from machined chips and the virgin specimen. It can be seen in Fig. 5 that a high strain rate sensitivity of approximately 0.5 was obtained in a low strain rate range less than $10^{-3}\,\mathrm{s}^{-1}$. It is suggested that superplastic behavior occurred in the strain rate range below $10^{-3}\,\mathrm{s}^{-1}$.

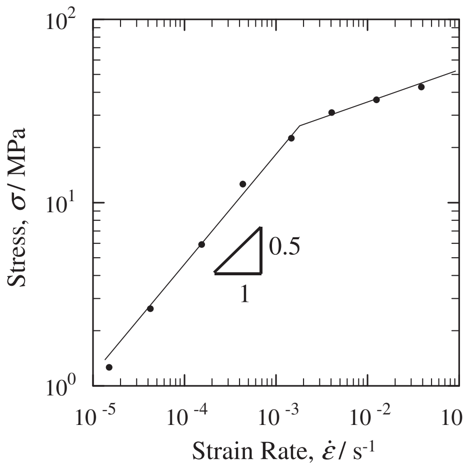

Fig. 5 The variation in stress at 673 K as a function of strain rate for the virgin specimen of AZ31 Mg alloy.

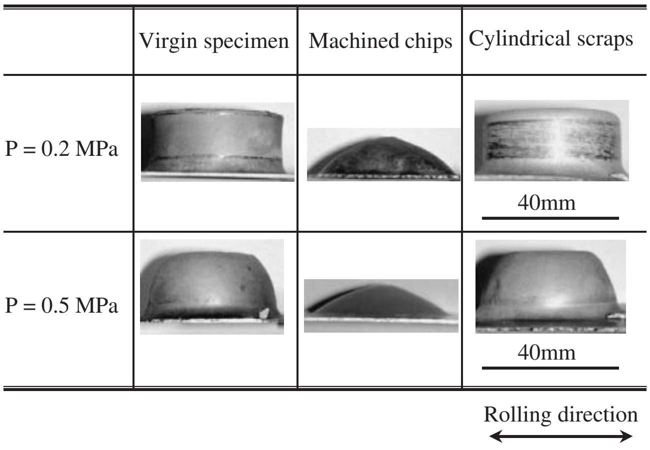

Fig. 6 The results of the blow forming tests at 673 K and at 0.2 MPa and 0.5 MPa.

Blow forming tests were carried out at 673 K with pressures of 0.2 MPa and 0.5 MPa. The stress at the dome apex can be given by[15]

$$\sigma_c = \sigma_t = \frac{PB}{4S}\left(H + \frac{1}{H}\right), \tag{1}$$

where σ_c is the stress in the circumferential direction, σ_t is the stress in the thickness direction, P is the forming pressure, B is the die radius, S is the thickness and H is the relative dome height. The strain rates at 0.2 MPa and 0.5 MPa are of the order of $10^{-5}\,\mathrm{s}^{-1}$ and $10^{-4}\,\mathrm{s}^{-1}$, respectively, from eq. (1) and the results in Fig. 5. The results of blow forming tests are shown in Fig. 6. The cup shape and dome shape were successfully formed with the virgin specimen. The high formability of the virgin specimen is a result of superplastic deformation. The specimens from machined chips were fractured before the dome shape was formed. The low formability of the specimen from machined chips is likely to be attributed to the large amount of contamination by oxides. Thus, oxide contamination adversely affected the formability of recycled Mg alloy.

On the other hand, the specimen from cylindrical scraps exhibited high formability that was of almost the same degree as that of the virgin specimen. It is suggested that there is a critical condition of the permissible contamination concentration for high formability, below which adverse effects of contamination are negligible and high formability is attained, but above which formability is reduced, for recycled Mg alloy. The critical condition of contamination for high formability may depend not only on the concentration of contaminations, but also on the shape, the distribution and the composition of contamination. Further research is needed to elucidate the critical condition of contaminations for achieving high formability of the recycled Mg alloy.

4. Conclusions

Blow forming of AZ31 Mg alloy recycled by solid-state recycling was conducted, and the mechanical properties and formability of the recycled specimens were investigated. The results are summarized as follows.

(1) As a result of tensile tests from room temperature to 723 K, the mechanical properties of the specimen from cylindrical scraps were found to be almost the same as those of the rolled specimen from a virgin ingot.

Moreover, as a result of blow forming tests, the specimen from cylindrical scraps was found to exhibit high formability, which was of almost the same degree as that of the virgin specimen. This is because the oxide contamination in the specimen from cylindrical scraps was law.

(2) However, the elongation to failure of the specimen from machined chips was low, compared with that of the virgin specimen. In addition, the specimen from machined chips exhibited poor formability. Oxide layers with a short interval of approximately 10 µm were observed in the specimen from machined chips. A large amount of oxide contamination is likely to be responsible for the low elongation and poor formability of the specimen from machined chips.

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