Corrosion and Mechanical Properties of Recycled 5083 Aluminum Alloy by Solid State Recycling

Yasumasa Chino\textsuperscript{1}, Mamoru Mabuchi\textsuperscript{1}, Satoshi Otsuka\textsuperscript{2,*}, Koji Shimojima\textsuperscript{1}, Hiroyuki Hosokawa\textsuperscript{1}, Yasuo Yamada\textsuperscript{1}, Cui’e Wen\textsuperscript{1} and Hajime Iwasaki\textsuperscript{2}

\textsuperscript{1}Institute for Structural and Engineering Materials, National Institute of Advanced Industrial Science and Technology, Nagoya 463-8560, Japan
\textsuperscript{2}Division of Materials Science and Engineering, Graduate School of Himeji Institute of Technology, Himeji 671-2201, Japan

Corrosion and Mechanical properties of a recycled 5083 aluminum alloy by solid state recycling have been compared with those of a virgin extrusion which was processed from the ingot block. In the solid state recycling, the machined chips were extruded at 723 K with an extrusion ratio of 44 : 1 in air. As a result of the salt immersion tests, the mass loss of the solid recycled specimen was not less than twice of that of the virgin extruded specimen. The deterioration in corrosion properties for the solid recycled specimen was attributed to the excessive contamination of iron which promoted galvanic corrosion. As a result of tensile tests, the solid recycled specimen exhibited a good combination of high strength and high elongation to failure at room temperature. The excellent mechanical properties for the solid recycled specimen were attributed to the refined microstructure. However, the elongation to failure of the solid recycled specimen at elevated temperatures more than 573 K was lower than that of the virgin extruded specimen. The contamination of oxide particles is likely to be responsible for the lower elongation in the solid recycled specimen.

(Received January 14, 2003; Accepted April 8, 2003)

Keywords: aluminum alloy, solid state recycling, hot extrusion, grain refinement, mechanical properties, corrosion properties

1. Introduction

Aluminum alloy has some advantages such as the low density, high corrosion resistance and high thermal conductivity. For low CO$_2$ emission of transportations, application of aluminum alloy to structural components of vehicles is increasingly becoming important due to its high specific strength. Now, aluminum alloy ranks the second in consumption in the world among metals\textsuperscript{11} and recycling of it is one of important technologies for materials circulation.

A large energy of about 2.6 $\times$ 10$^7$ J/t is needed when an aluminum virgin ingot is smelted from bauxite. However, the energy for recycling by remelting of aluminum scraps is about 1.0 $\times$ 10$^4$ J/t, which is only 4% of the smelting energy from bauxite. Hence, aluminum scraps should be recycled to reduce the environmental loads. Actually, recycling of aluminum scraps is being done by the some recycling processes on the basis of remelting.\textsuperscript{2–5} However, it is metallurgically difficult to refine aluminum scraps by the remelting processes. Hence, most of the current recycling processes are down grade recycling\textsuperscript{6} and there are some problems such as contamination of iron, silicon and so on from aluminum scraps,\textsuperscript{3} resulting in a reduction in service properties.

The desirable recycling is recycling from scraps to high performance materials with low energy consumption. Solid state recycling\textsuperscript{7–13} is one of solutions for such low energy recycling. In the solid state recycling, metal scraps are directly recycled by plastic deformation process such as hot extrusion,\textsuperscript{7–10} forging\textsuperscript{11} and BMA (bulk mechanical alloying).\textsuperscript{12,13} It should be noted that remelting is not needed for the solid state recycling. In the previous works,\textsuperscript{7–9} the solid recycled magnesium alloy showed high strength due to microstructural control by the plastic deformation process.

Thus, the solid state recycling leads not only to a reduction in recycling energy consumption, but also to improvement of mechanical properties of recycled materials.

The aim of the present research is to apply the solid state recycling using hot extrusion to aluminum alloy. For magnesium alloys, there are some studies on mechanical properties of recycled specimen by solid state recycling.\textsuperscript{7–10,13} However, there are few studies on the solid state recycling for aluminum alloys.\textsuperscript{12} In the present paper, machined chips of 5083 aluminum alloy are recycled by solid state recycling using hot extrusion and mechanical and corrosion properties of the recycled aluminum alloy have been investigated.

2. Experimental Procedure

Chips were prepared as aluminum alloy scraps by machining an as-received 5083 aluminum alloy\textsuperscript{14} in a lathe without lubricants. The machined chips are shown in Fig. 1.
The average length, width and thickness of the machined chips were 9.3, 1.8 and 0.3 mm, respectively. The machined chips cleaned by acetone were filled into a container with a diameter of 40 mm and extruded at 723 K with an extrusion ratio of 44:1 in air. For comparison, extrusions were processed from an as-received 5083 aluminum alloy ingot block under the same conditions as the extrusions from machined chips. In the present study, the extrusion specimen from machined chips is called the solid recycled specimen and the one from the as-received block is called the virgin extruded specimen.

A metallographic investigation was carried out by optical microscopy (OM) and transmission electron microscopy (TEM). The grain size of the specimens was measured by the linear intercept method. Specimens for the observation were etched using Berker’s reagent. The corrosion properties of the solid recycled specimen and the virgin extruded specimen were evaluated by the salt immersion test in which the mass loss of the specimens after 12 and 24 h in 3 mass% NaCl solution was measured. The dimensions of specimen for the salt immersion test were 30 mm × 12 mm × 3 mm. The chemical composition of the solid recycled specimen and the virgin extruded specimen was measured by the ICP emission spectrochemical analysis and EPMA analysis.

Tensile tests were carried out at a strain rate of 1.7 × 10^{-3} s^{-1} to investigate mechanical properties at room temperature to 673 K. The tested specimens had a gauge length of 10 mm and a gauge diameter of 2.5 mm. 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. Cavities were investigated by scanning electron microscopy (SEM). Cavity size distributions were examined based on quantitative metallographic measurements, assuming a spherical shape. Also, grain boundary sliding was investigated by scanning electron microscopy.

3. Results and Discussion

3.1 Microstructures

Microstructure of the as-received specimen is shown in Fig. 2(a) and that of the solid recycled specimen in the transverse section to the extrusion direction is shown in Fig. 2(b), respectively. In the as-received specimen, coarse grains of about 390 μm were observed by OM observation. However, in the solid recycled specimen, a small equiaxed grain with grain size of 6.2 μm was observed by TEM observation. Dynamic recrystallization occurs during hot extrusion for Al–Mg aluminum alloys. Therefore, it is suggested that a fine-grained microstructure in the solid recycled specimen is attributed to dynamic recrystallization during hot extrusion. Microstructure of the virgin extruded specimen was almost the same as that of the solid recycled specimen. The grain size of the virgin extruded specimen was 4.7 μm.

3.2 Corrosion properties

The salt immersion tests for the solid recycled specimen and the virgin extruded specimen were carried out using 3 mass% NaCl solution at 293 K. Figure 3 shows the mass loss of the specimens after 12, 24 h in the solution. The mass loss of the solid recycled specimen after 24 h was 8.7 × 10^{-4} kg/m² which was not less than twice of that of the virgin extruded specimen (≈ 3.6 × 10^{-4} kg/m²).

Table 1 shows the chemical composition of representative small quantity elements for both the specimens. For reference, the JIS standard specification of 5083 aluminum alloy is also listed in Table 1. It is noted that the contamination level of iron in the solid recycled specimen was larger than that of the virgin extruded specimen and it was not satisfied with Japanese Industrial Standard of 5083 aluminum alloy. The composition of other elements for the
solid recycled specimen was almost the same as that of the virgin extruded specimen and satisfied with the Japanese Industrial Standard.

Figure 4 shows iron particles observed by SEM (a) and the corresponding Fe and O images by EPMA (b) and (c), respectively, for the solid recycled specimen. An oxygen rich region is observed in an iron rich region. An oxygen rich region would be composed of aluminum and iron oxides. The region would be a sintered interface of the machined chips. It is suggested that iron particles shown in Fig. 4 are contaminants introduced by the recycling. The size of detected iron particles was less than 10 μm. It is known that contamination of iron in aluminum alloy promotes galvanic corrosion due to the formation of local cell. 19) Therefore, it is suggested that excessive contamination of iron caused the deterioration of corrosion properties for the solid recycled specimen.

The surface profiles of both the specimens after the salt immersion tests are shown in Fig. 5. Corrosion for the virgin extruded specimen occurred randomly. On the other hand, the corrosion sites were aligned parallel to the extrusion direction for the solid recycled specimen. In Fig. 4, the iron contaminants were distributed on the interface of the machined chips. It is therefore suggested that corrosion occurred preferentially on the interface of the chips where the iron particles were located. In the previous work, 8 we carried out the solid state recycling of Mg alloy scraps from thixo-molding (casting) process. In this case, the chemical composition of the recycled Mg alloy was almost the same as that of the virgin ingot. Therefore, in the present work, the excessive contamination of iron might occur during machining process, but during hot extrusion. Anyway, when the solid state recycling is applied as recycling of fine scraps such as machined scraps without laundering process, contamination is inevitable. Therefore, further researches are needed to develop the laundering process and to deepen understanding of the contamination tolerance for the solid state recycling.

### Table 1 The chemical composition of representative small quantity elements in 5083 aluminum alloy for the solid recycled specimen and the virgin extruded specimen.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Si</th>
<th>Cu</th>
<th>Mn</th>
<th>Fe</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>JIS standard specification (5083 Al alloy)</td>
<td>≤0.40</td>
<td>≤0.10</td>
<td>0.40~1.0</td>
<td>≤0.40</td>
<td>≤0.25</td>
</tr>
<tr>
<td>Solid recycled specimen</td>
<td>0.15</td>
<td>0.03</td>
<td>0.80</td>
<td>0.74</td>
<td>0.01</td>
</tr>
<tr>
<td>Virgin extruded specimen</td>
<td>0.15</td>
<td>0.03</td>
<td>0.83</td>
<td>0.26</td>
<td>0.01</td>
</tr>
</tbody>
</table>

### Table 2 Tensile properties at room temperature of the 5083 aluminum alloy specimens.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Ultimate tensile strength (MPa)</th>
<th>0.2% Proof stress (MPa)</th>
<th>Elongation to failure (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received specimen</td>
<td>277</td>
<td>131</td>
<td>13</td>
</tr>
<tr>
<td>Solid recycled specimen</td>
<td>329</td>
<td>179</td>
<td>16</td>
</tr>
<tr>
<td>Virgin extruded specimen</td>
<td>345</td>
<td>187</td>
<td>17</td>
</tr>
</tbody>
</table>

Fig. 4 Iron particles in the solid recycled specimen observed by SEM (a) and the corresponding Fe and O images by EPMA (b) and (c), respectively.
the solid recycled specimen were almost the same as those of the virgin extruded specimen.

The Hall-Petch relation between yield stress and grain size is given by the eq. (1)

\[
\sigma = \sigma_0 + Kd^{-1/2}
\]

where \(\sigma\) is the yield stress of a polycrystalline metal, \(\sigma_0\) is the yield stress if there is no resistance to slip across the grain boundary, \(K\) is a constant and \(d\) is the grain size. The variation in 0.2% proof stress as a function of (grain size)\(^{-1/2}\) for 5083 alloy is shown in Fig. 6, where \(\sigma_0\) and \(K\) for 5083 alloy are taken to be 150 MPa \(^{20}\) and 63 MPa \(^{-1/2}\) \(\mu m\) \(^{-0.5}\) respectively. Additionally, the data of the as-received specimen, the solid recycled specimen and the virgin extruded specimen are added. It can be observed that the data fits line, approximately. Therefore, it is suggested that the excellent mechanical properties for the solid recycled specimen are mainly attributed to the refined microstructure by hot extrusion.

The variation in ultimate tensile strength and elongation to failure as a function of testing temperature for the solid recycled specimen and the virgin extruded specimen is shown in Fig. 7. The tensile strength of the solid recycled specimen was almost the same as that of the virgin extruded specimen. However, the elongation to failure of the solid recycled specimen at the testing temperatures more than 573 K was lower than that of the virgin extruded specimen.

Figure 8 shows the side surfaces of the solid recycled specimen (a) and the virgin extruded specimen (b) deformed to fracture at 673 K. A measured point of side surface for the solid recycled specimen and the virgin extruded specimen was a point about 3 mm and 6 mm apart from the fractured section, respectively. A bumpy surface was observed for both specimens, indicating that grain boundary sliding occurred for both specimens. Also, the grain size of the solid recycled specimen after the tensile test at 673 K (\(=10\) \(\mu m\)) was almost the same as that of the virgin extruded specimen (\(=13\) \(\mu m\)). Thus, there was no difference in deformation and grain growth behavior between the solid recycled specimen and the virgin extruded specimen.

Cavitation is often observed in a wide range of superplastic materials whose dominant deformation process is grain boundary sliding.\(^{22-25}\) Cavities often grow during grain boundary sliding, resulting in premature fracture. Therefore, it is worthwhile to investigate cavitation for understanding of the low elongation of the solid recycled specimen. The cavity diameter distributions in unit area (\(= 1 \text{mm}^2\)) for the both specimens are shown in Fig. 9, where the specimens were
deformed to the true strain of 0.6 at 1.7 x 10^-3 s^-1 and 673 K. The total number of cavities for the solid recycled specimen was 2,624 which was larger than that of the virgin extruded specimen (= 989). Furthermore, the average cavity size for the solid recycled specimen of 2.5 μm was larger than that of the virgin extruded specimen (= 1.7 μm).

Mahoney and Ghosh suggested that large size reinforcements play the role as barrier of grain boundary sliding in the aluminum alloy matrix composites and also promote cavitation during superplastic flow, resulting in premature fracture. It was shown in the previous work on the solid state recycling of a magnesium alloy that contamination of oxide particles in the solid recycled specimen promotes cavity nucleation, resulting in low elongation of the solid recycled specimen. The inhomogeneous distribution of the oxide particles shown in Fig. 4(c) would promote excessive cavity formation in the solid recycled specimen. Therefore, it is suggested that the low elongation at elevated temperatures for the solid recycled specimen is attributed to cavitation stimulated by the oxide contamination introduced during hot extrusion for the aluminum alloy as well as the magnesium alloy. It has been reported that large elongation is obtained in aluminum alloys containing fine and homogeneously dispersed particles. Hence, further investigations are needed to investigate the process conditions for homogeneous dispersion and refinement of the oxide particles in the solid recycled aluminum alloy.

4. Conclusions

Corrosion and mechanical properties of the recycled 5083 aluminum alloy by solid state recycling have been compared with those of the virgin extrusion which was processed from the ingot block. The results are summarized as follows.

(1) As a result of the salt immersion tests, the mass loss of the solid recycled specimen was not less than twice of that of the virgin extruded specimen. The deterioration in corrosion properties for the solid recycled specimen was attributed to the excessive contamination of iron which promoted galvanic corrosion.

(2) The solid recycled specimens exhibited a good combination of high strength and high elongation to failure at room temperature. The excellent mechanical properties for the solid recycled specimen were attributed to the refined microstructure.

(3) The elongation to failure of the solid recycled specimen was lower than that of the virgin extruded specimen at the testing temperatures more than 573 K. The contamination of oxide particles would be responsible for the lower elongation in the solid recycled specimen.

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.

REFERENCES