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Utilization of Dross for Fabrication of Al_2O_3/A6061 MMCs by the PLF Method

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We have developed the powder liquid forging process(PLF) for producing metal matrix composites(MMCs). This unique technique has been applied to new MMCs utilized aluminum alloy dross. Since the dross is composed of aluminum alloy and aluminum oxides such as colundum and spinel as usual, a trial was made to fabricate dross/aluminum alloy MMCs. The properties of the produced MMCs were examined and compared with those of Al_2O_3*/A6061 and* Al_2O_3*/AC8A MMCs. The PLF technique was found to be applicable to the production of dross bearing MMCs. To improve the properties of the dross/Al alloy MMCs, it is necessary to investigate an appropriate pre-treatment of dross.*

1. Introduction

In order to exchange the commercial materials to ecomaterials, the following three check points are important for the investigation of materials design:

(1) to improve the recyclability.
(2) to keep the properties comparable with those of virgin materials.
(3) to reduce the environmental load in a whole material flow; production, usage, recycling, and re-production and to save the natural resources including the energy needed for the material flow.

The point (3) is particularly important among them from a view point of ecomaterial, which indicates the desirable direction of future materials development.

In a case of material flow of aluminum alloy, one of the difficulties to be overcome is the treatment of dross which has not been used effectively so far. The dross does not contain any harmful alloy element for recycling. Then, if the separation of Al alloy and Al oxides in the dross was accomplished easily, the alloy could be used again with no serious problem. However, since it is difficult to separate them from each other in actual, we consider the use of dross as fillers of metal matrix composites(MMCs) before sending it back to smelting. In general, composite materials are not favorable for recycling, but Al-oxides/Al alloy MMCs are considered to be recyclable and less harmful for the recycling of Al alloys scrap even when MMCs and Al alloys were mixed. Although this idea belongs to a kind of *cascade* use, it must be useful to improve the whole Al material flow at present situation.

To utilize the dross for MMCs, conventional MMC producing methods are not suitable. Therefore, we have employed the PLF method which has been studied first by Watanabe and Saitoh[1][2] and then by Tomota et al[3]-[5]. The trial of dross/Al alloy MMCs is to be reported shortly in reference[6]. In this paper, therefore, the detailed experimental procedures and results are given and their properties are discussed in comparison with those of Al_2O_3/A6061 or

Al_2O_3/AC8A MMCs.

2. Experimental Procedure

2.1. PLF Procedure

The outline of PLF process that we used was shown in Fig. 1. The dross was taken from a holding furnace of molten A5182 alloy which were used for top-plate of beverage can. The chemical compositions of air atomized A6061 and AC8A powders are;

1.12Mg-0.64Si-0.15Fe-0.26Cu-0.15Mn - 0.17Cr-0.01Zn-0.01Ni-bal.Al and **11.25Si-0.19Fe-1.22Cu-1.22Mg-0.02Mn-0.02Cr-0.03Zn-1.48Ni-bal.Al** (in mass%) respectively. The dies shown in Fig.2 was commonly used for compacting powders mixture and for PLF. Since Al alloy powders are covered by oxide films, it is quite difficult to sinter by conventional procedure. Then, when the mixed powders are heated at a temperature a little above the melting point, molten Al alloy would be held within oxide film for a while. Therefore, a billet was compressed to break the oxide film for promoting the sintering. The shape and dimensions of MMCs produced were cylinder in shape with 42 mm in diameter and about 20 to 30 mm in length. Some of the specimens were subsequently hot-forged or hot-extruded at 673K.

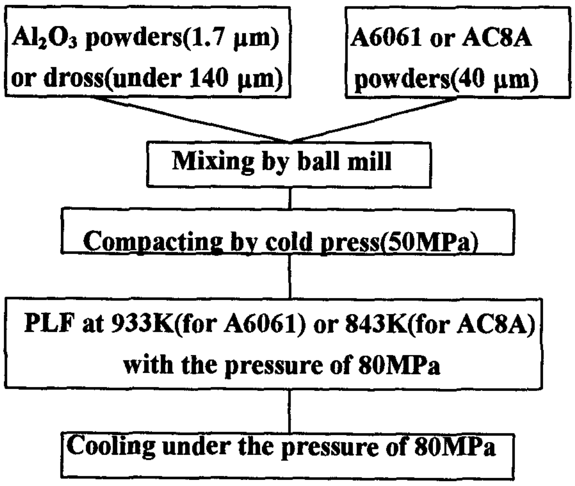

Fig.1 Schematic illustration of PLF process

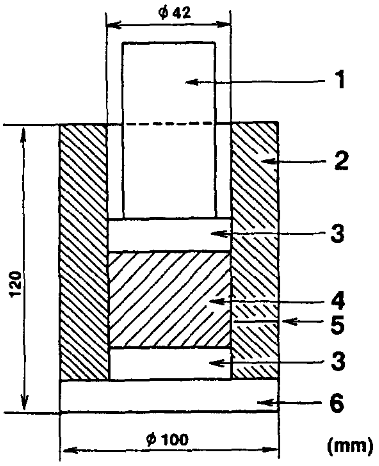

Fig.2 Die assembly for PLF: 1: stem, 2: container, 3:dummy block, 4: billet (sample), 5: thermocouple, and 6: bottom plate.

2.2 Measurements of Physical Properties

The microstructure of the specimens was observed by light microscopy or SEM, where the samples were polished and etched by DAS etchant. The density was measured by conventional method based on Archimedes principle(JIS Z2501, Z2505). Rockwell and Vickers hardness test and tension tests were carried out. Thermal diffusivity was examined by means of a laser flash method.

3. Experimental Results and Discussion

3.1 Dross/A6061 MMCs

Figure 3 shows examples of X-ray analyses of the dross, by which it was found that the dross used was composed of Al alloy, Al_2O_3, $MgAl_2O_4$ (spinel) and other oxides(presumably MgO etc.). The diffraction peak height was different from sample to sample : Figure 3(a) is an example in larger particles while (b) is that in smaller ones. The volume fraction *vs.* X-ray diffraction peak intensity relationship was constructed by using pre-mixed Al_2O_3 and Al alloy powders with the volume fraction from 0 to 100%. The volume fraction of Al_2O_3 was then estimated to be 56 volume percents by using this calibration curve, taking the average of five samples, and neglecting the other oxides. Hereafter, the Al_2O_3 volume fraction in dross/Al alloy MMCs was determined by this way. The chemical compositions of the dross was also examined by EPMA and it was found that the dross contained a considerable amount of Mg and Cl.

First, we made a 30% Al_2O_3 dross/A6061 MMC. Before mixing by a ball mill, the dross particles were broken to smaller pieces and filtered to be under 149μm in diameter. MMCs were made according to the procedure shown in Fig. 1. The microstructure of the MMCs produced was observed, so that large voids were detected by optical microscopy.

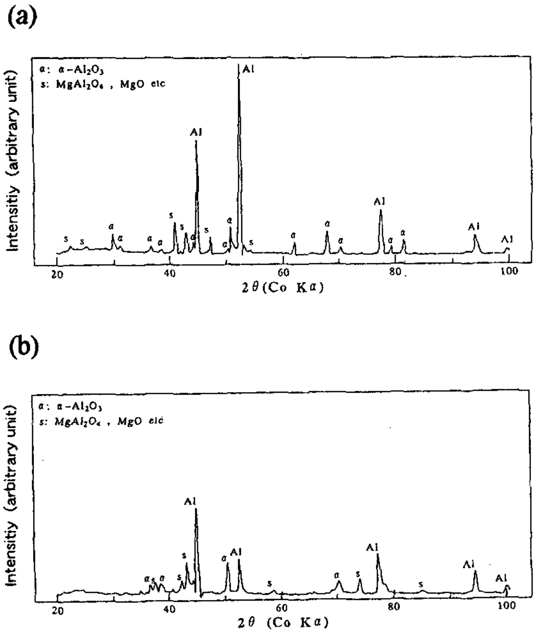

Fig.3 Results of X-ray diffraction of dross: (a) larger particles and (b)smaller particles(under 149μm)

Hardness (H_RF) was measured on the longitudinal section of the as-PLF billet from the top to the bottom. The results are presented in Fig. 4. The scattering in hardness was relatively small within a whole billet. The average hardness was however smaller by about 7% compared with that of the 30% Al_2O_3/A6061 MMC that was previously studied[4].

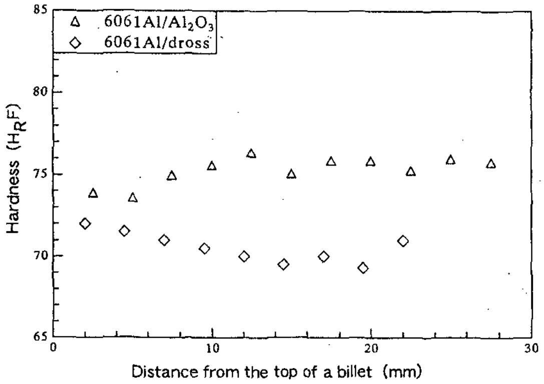

Fig.4 Results of hardness tests at the longitudinal section of as-PLF billets.

One of the origins of the voids were considered due to Cl_2 gas generation during the PLF. Chloride or Cl_2 is usually added to the molten alloy to remove hydrogen, so that Cl must remain in the dross. Then, we heated the dross at 673K for 1.8ks aiming the degassing before PLF. And the volume fraction of Al_2O_3 was reduced down to 10 or 20%. The optical microstructure of a new MMC was shown in Fig. 5 where voids could not be detected but large white regions corresponding to Al alloy phase were still observed. The densities became 2.63g/cm^3 for 10% Al_2O_3 MMC and 2.71-2.74 g/cm^3 for 20% one, respectively, which were lower than the theoretically predicted values(2.82 and 2.94). From these differences, the volume fraction of void was calculated to be about 7%.

Thermal conductivity was determined by using thermal diffusivity measured by the

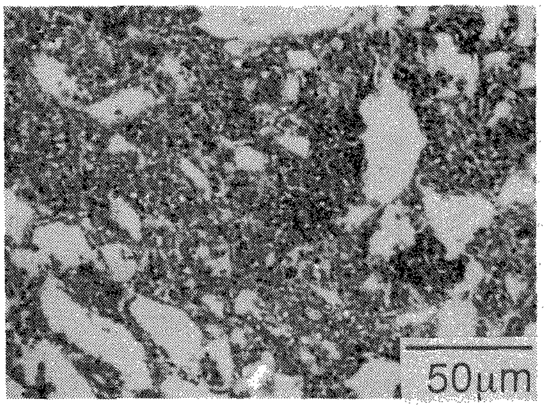

Fig.5 Optical micrograph of a 10% Al_2O_3 dross/A6061 MMC.

laser flash method and measured density, and estimated specific heat. The thermal conductivities obtained for 10% and 20% Al_2O_3/A6061 MMCs were 124 and 92 W/mK while theoretically predicted values were 143 and 132 W/mK, respectively. Here the predictions were performed by using the equivalent inclusion method[7].

It was also found that the hardness of the MMCs increased only a little by aging. This seems to be ascribed to the loss of Mg concentration in the matrix due to the formation of spinel($MgAl_2O_4$). The detail will be explained in the following section(see Fig. 7).

3.2 Properties of Al_2O_3/A6061 and Al_2O_3 /AC8A MMCs

The data on Al_2O_3 (1.7μm)/A6061 MMC were reported in reference[4]. In order to seek the improvement of the dross bearing MMCs, the previous work is briefly reviewed and original data on Al_2O_3 (1.7μm)/AC8A MMCs are presented here, for comparison.

(a) Al_2O_3 (1.7μm)/A6061 MMC

Figure 6 shows a typical optical micrograph. The Al alloy and the reinforcement were mixed well and voids could not be detected. Since A6061 alloy is usually used after aging(T6 treatment), age-hardening behaviour was examined. As shown in Fig.7, the amount of age-hardening in MMCs was very small in contrast with large amount of age-hardening in A6061 alloy(indicated by 0% in the figure). Similar results were obtained in dross/ A6061 MMCs as described above. The reason of this loss in age hardening was studied by X-ray analyses: the concentration of Mg in the matrix was reduced by the formation of spinel, $MgAl_2O_4$ at the interface of Al_2O_3 and the matrix[4].

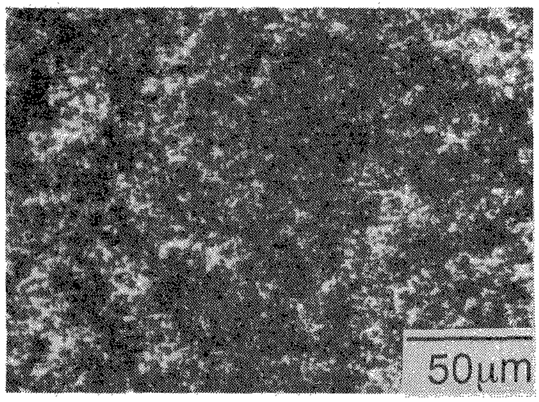

Fig.6 Optical micrograph of a 30% Al_2O_3/A6061 MMC.

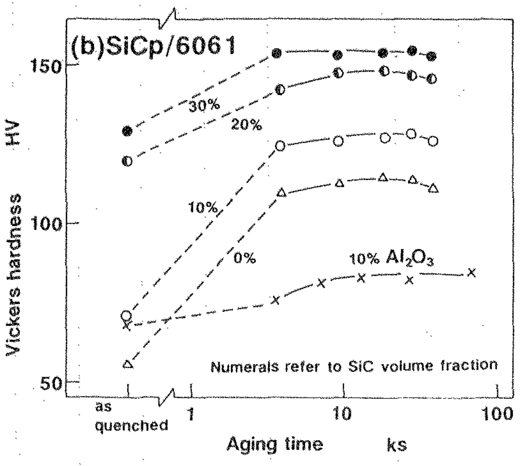

Fig.7 Hardness of MMCs as a function of aging temperature.

Although the age-hardening was deteriorated, the formation of the sipinel at the interface is believed to be favorable for bonding of Al alloy and ceramic powders. The tensile strengths of Al_2O_3 and SiC/A6061 MMCs are listed in Table 1. Despite the strength at room temperature in the former was inferior to the latter due to the loss of age-hardening, the strength at 573K was comparable in both MMCs. The formation of spinel is postulated to enhance the interface strength.

Table 1 Tensile properties of MMCs

	Tensile Strength(MPa)		Elongation(%)	
20%MMC	300K	473K	300K	473K
Al_2O_3/A6061	**381**	**213**	**4.0**	**6.5**
SiC/A6061	**417**	**214**	**1.9**	**7.6**

The flow curves, i.e., work hardening of MMCs have been predicted successfully by means of micromechanics[8]. The application of Eshelby transformation problem, Mori-Tanaka mean field concept and the secant method have enabled us to predict the overall flow curves of MMCs[8]. If we adopt the flow curve of A6061 T-6 heat-treated alloy for the deformation of the matrix, we could obtain the flow curves drawn in Fig.8 which are higher than the experimental ones. Since the agreement between calculations and measurements is quite excellent in SiC/A6061 MMCs, this discrepancy is believed to stem from the change in aging behaviour of the matrix in the MMCs. It should be noticed that the properties of the components are sometimes altered in MMCs. To evaluate the properties theoretically, these changes must be taken into consideration.

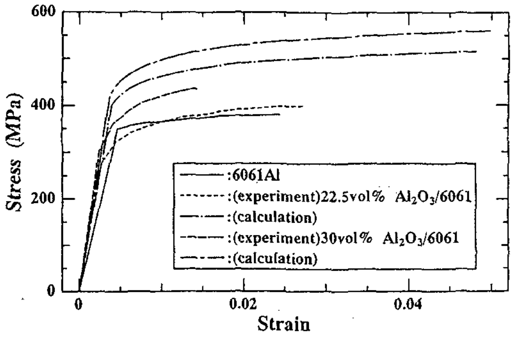

Fig.8 Theoretical prediction of flow curves.

(b) Al_2O_3/AC8A MMCs

In order to make a sound MMC with a higher volume fraction of Al_2O_3, an alloy for casting must be better to use. Thus, AC8A was chosen for the matrix. SiC powders with 3.0 μm in diameter were also employed as fillers for comparison. The densities of the products were presented in Fig.9 as a function of volume fraction. It is found that the Al_2O_3 volume fraction can be increased more easily in comparison with SiC. This must be caused from the spinel formation described above.

Vickers hardness in both MMCs was shown in

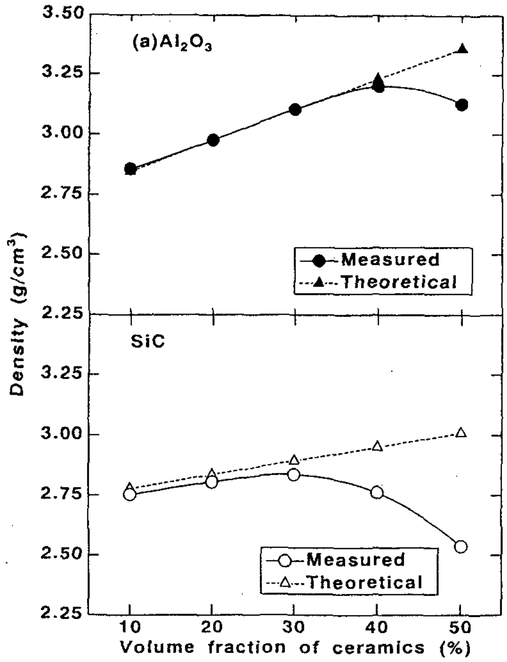

Fig.9 Densities of Al_2O_3/AC8A MMCs as a function of ceramics volume fraction

Fig. 10. In case of Al_2O_3/AC8A MMCs, the volume fraction of Al_2O_3 could be increased up to 40% without serious problem.

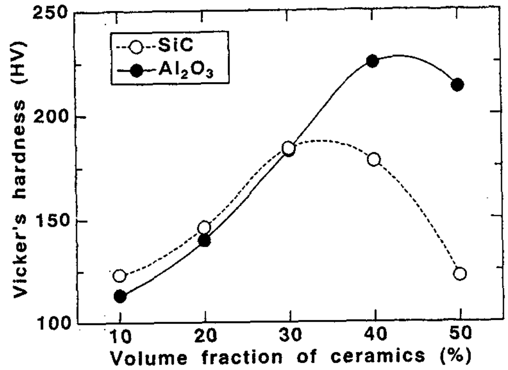

Fig.10 Vickers hardness as a function of volume fraction of filler.

3.3 Improvement of dross /Al alloy MMCs

The PLF process was found to be applicable to the production of dross bearing MMCs. Their properties are however not satisfactory. Based on the experimental results described above, the following proposals would be made for further improvement of dross utilized MMCs.

(a) Development of pre-treatment for degassing from the dross.
(b) Adjusting the dross particle to smaller size.
(c) Changing the matrix from A6061 to alloys for casting, for instance, AC8A.
(d) Application of hot-forging for microstructure control.

4. Concluding Remarks

In order to improve the current Al material flow, the trial of effective utilization of the dross was investigated, that is, the MMC containing the dross was made by employing the unique PLF process. As MMCs have been called tailor made materials, the properties can be predicted theoretically to some extent. Through the comparison of the properties in produced dross bearing MMCs with the predictions and those of usual powders bearing MMCs, the ideas to improve dross bearing MMCs were discussed.

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