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Use of Recycled Steel Machining Chips and Aluminum Can Shreds for Synthesizing Iron Aluminide Intermetallic Alloys

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Some iron aluminide intermetallic alloys based on Fe_3Al or FeAl have been synthesized from recycled raw materials of steel machining chips and aluminum can shreds. The former was an industrial waste made at a bearing case maker, and it had a spiral shape and a length less than 1 mm and contained 0.87 wt% C, 0.21 wt% Si, 1.53 wt% Cr with some other minor elements. The latter, on the other hand, was made by shredding used aluminum cans, and it had a flaky shape and a edge length of 2 to 3 mm and contained 2.3 wt% Mg, 0.36 wt% Fe, 0.88 wt% Mn with some other minor elements. When compacts of the steel and aluminum mixture were heated in a crucible in an argon atmosphere, they exothermically reacted and produced iron aluminide intermetallic alloys. For comparison, similar alloys were produced from pure iron and aluminum by melting them in the same heating apparatus. The bending strength, fracture toughness, Vickers hardness and wear resistance of the alloys synthesized from the recycled raw materials were more excellent than or similar to those of the alloys produced from pure iron and aluminum.

KEY WORDS: recycling; casting; combustion synthesis; SHS (Self-propagating High-temperature Synthesis); intermetallic compound; FeAl; Fe_3Al.

1. Introduction

Iron aluminide intermetallic alloys such as Fe_3Al and FeAl are of interest for structural applications in hostile environments, because they can form an impervious alumina layer that can provide excellent corrosion resistance in a wide range of corrosive environments.[1–3)] They also have relatively low densities (6.7 and 5.6 g/cm^3 for Fe_3Al and FeAl, respectively) and low raw material cost as compared with Ni-based superalloys.

In previous studies, Fe_3Al has been produced by arc melting[4–6)] or induction melting.[7)] On the other hand, FeAl has mainly been produced by powder metallurgical methods such as mechanical alloying,[8)] reactive sintering,[9,10)] combustion synthesis (Self-propagating High-temperature Synthesis, SHS)[11)] and squeeze casting,[10)] while arc melting[12)] seems not to be a major method to produce FeAl. It will be appropriate to produce these iron aluminide intermetallic alloys by a melting and casting process from a viewpoint of cost reduction, because powder is much expensive as the raw material as compared with ingots. Moreover, the cost will be dramatically reduced if recycled raw materials are used in the melting and casting process. The aims of the present study are to produce iron aluminide intermetallic alloys from recycled raw materials and to investigate some mechanical properties of the alloys.

2. Procedure

The recycled raw materials used in the present study were machining chips of carbon steel and shreds of the lids of used aluminum cans. Their chemical compositions are given in **Table 1**. The major impurities are chromium and carbon for the steel chips, while they are magnesium and manganese for the aluminum shreds. **Figure 1** shows the appearances of (a) the steel chips and (b) the aluminum shreds. The steel chips have a spiral shape while the aluminum shreds have a flaky shape. The steel chip is an industrial waste made at a Japanese bearing case maker and they pay a lot of expense on discarding the machining chips. The aluminum shred, on the other hand, is a test product of a Japanese machine maker who designs and sells shredders for used aluminum cans. In aluminum can recycling, the used cans are collected and are usually crashed to

Table 1. Chemical cmpositions of the recycled raw materals (wt%).

Material	Al	C	Cr	Cu	Fe	Mg	Mn	Ni	Si	Ti	Zn
Steel Chips	-	0.87	1.53	0.02	Bal.	-	0.36	0.08	0.21	-	-
Al Shreds	Bal.	-	0.02	0.19	0.36	2.32	0.88	-	0.37	0.06	0.11

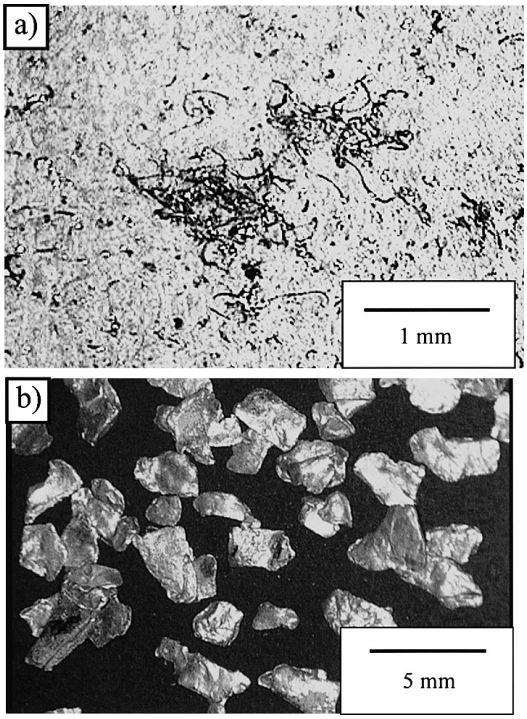

Fig. 1. Appearance of (a) the steel machining chips and (b) the aluminum can shreds.

reduce the volume for effective transportation. However, it seems that shredded cans are better than crashed ones as a recycled raw material because in case of the crashing process, harmful garbage such as used batteries that may be contained in the used cans will not be easily removed.

The two recycled raw materials were mixed and uniaxially pressed at a pressure of 430 MPa to produce a cylindrical compact of a 24-mm diameter and a 30-mm height. The nominal composition of the raw material mixture was varied from Fe–30mol%Al to Fe–50mol%Al by varying the mixing ratio of the two recycled raw materials. The compacts were placed in an alumina crucible and heated in a furnace to approximately 1 670 K at a rate of 10 K/s in an argon atmosphere to melt them. The liquid was kept for 1 h at that temperature, and then furnace-cooled in the crucible to room temperature at an initial cooling rate of approximately 9 K/s. The ingot having a 30-mm diameter and an 80-mm height was heat-treated for 17 h at 1 273 K in air for homogenization, and then naturally cooled outside the furnace.

The ingot was longitudinally sectioned into slices of a 4-mm thickness. Some of the slices were used for metallographic investigations and hardness and Young's modulus measurements, while some were cut into 4 mm–3 mm–30 mm rectangular bars for measurements of bending strength, fracture toughness K_{IC} and wear resistance. A Vickers hardness tester with an applied load of 5 kg was used for the hardness measurement, and a resonance method was used for the Young's modulus measurement. A four-point-bending tester with the distances between the loading points and supporting points of 4 mm and 15 mm was used for the bending strength measurement. The wear testing was performed using the Okoshi-type wear machine[13,14] with a counter material of SKD11 steel (1.5C, 12Cr, 1.2Mo wt%, HRA=82), a relative sliding speed of 1.5 m/s, a sliding distance of 600 m, and an applied stress of 2 MPa. The density of the alloy was measured using Archimedes method. Ideal density of Fe–Al binary alloys was also calculated using the chemical composition, the atomic weights of iron and aluminum, and the lattice parameters of Fe_3Al and FeAl. For comparison, Fe–Al binary alloys were produced from virgin metals. Pure iron and aluminum blocks of 5 to 10 mm in volume-equivalent diameter were similarly heated and melted in the similar crucible. The iron and aluminum had 99.9 and 99.99 wt% purity, respectively. The similar measurements were performed using the ingot produced from the virgin metals.

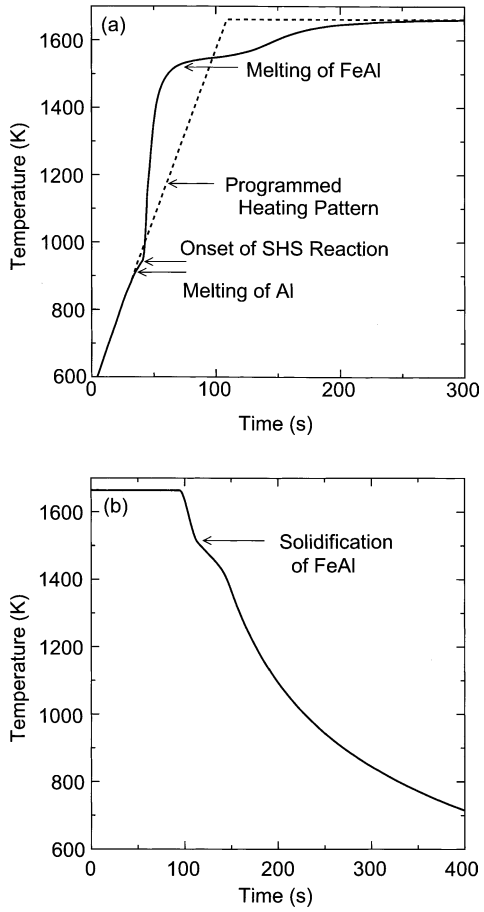

Fig. 2. (a) Heating curve of the compact made of the steel machining chips and aluminum can shreds. (b) Cooling curve after the heating.

3. Results and Discussion

3.1. Synthesis of Iron Aluminide Intermetallic Alloys

Figure 2 shows (a) a heating curve of the compact made of the recycled materials and (b) a cooling curve after heating. The nominal composition of the compact was Fe–47mol%Al. As shown in Fig. 2(a), the temperature of the compact increased at a constant rate in accordance with the programmed heating pattern at the beginning of the heating. However, when the temperature reached approximately 900 K, the increase rate slightly slowed down, and then at approximately 940 K, the temperature dramatically increased to approximately 1 500 K. It is considered that the slight slowdown at approximately 900 K was due to the melting of the aluminum shreds, while the dramatic rise

Table 2. Nominal and measured aluminum concentrations of the alloy samples (mol%).

Raw Materials	Nominal	Measured
Recycled	35	28.66
	41	34.85
	47	40.98
Virgin	35	31.21
	38	35.44
	41	38.06
	47	42.40

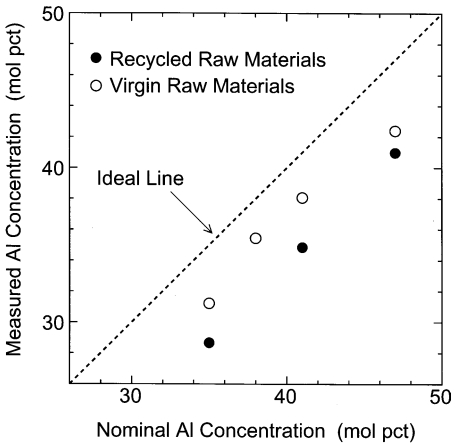

Fig. 3. Relationship between nominal and measured aluminum concentrations of the alloys.

after that was due to the onset of exothermic reactions between the molten aluminum and the heated steel chips such as $3Fe+Al \rightarrow Fe_3Al$ and $Fe+Al \rightarrow FeAl$. It is likely that melting of aluminum brought about a rapid increase in contact area between the aluminum and steel chips, which ignited the exothermic reactions. The temperature rise was very quick, but it stopped at approximately 1 500 K due to melting of FeAl. As shown in Fig. 2(b), the temperature decrease stopped at the similar temperature due to solidification of FeAl. The exothermic reactions seem to be useful for the energy saving in the production process of the iron aluminide intermetallic alloys, but their contribution may not be dramatic because the formation enthalpies of Fe_3Al and FeAl are not very large; they are 24 and 36 kJ/mol,[15] respectively.

3.2. Chemical Composition and Microstructure

The nominal and measured aluminum concentrations of the synthesized iron aluminide alloys are listed in **Table 2**. The measured aluminum concentrations were all lower than the nominal ones for both the alloys synthesized from recycled raw materials and the alloys from virgin raw materials. However, the difference between the nominal and measured aluminum concentrations was slightly larger for the alloys synthesized from the recycled raw materials, as shown in **Fig. 3**. In Fig. 3, all circles distribute along the ideal line that indicates that the measured aluminum concentrations are equal to the nominal ones, but all the circles are beneath the ideal line. Moreover, the solid circles are all beneath the open circles, indicating that the measured aluminum concentration of the alloy synthesized from the recycled raw materials is lower than that of the alloy synthesized from the virgin raw materials. It is likely that oxidation of aluminum during heating of the raw materials reduced the yield rate of the aluminum concentration of the alloys synthesized from the recycled materials, because the aluminum shreds of the used cans had much larger surface area than the aluminum blocks used as the virgin raw material.

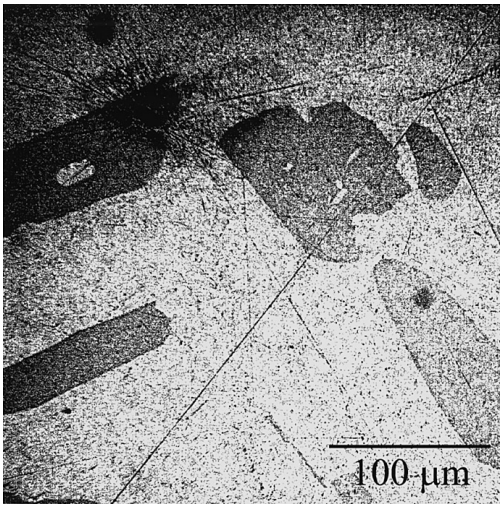

Fig. 4. Microstructure of an alloy synthesized from the recycled raw materials. Aluminum concentration is 34.85 mol%.

Table 3. Chemical compositions corresponding to the microstructure (mol%).

Phase	Al	Fe	Cr	Mn	Si
Bright	38.58	59.63	0.89	0.38	0.53
Dark	29.87	66.39	2.86	0.77	0.10

Figure 4 shows the microstructure of an alloy synthesized from the recycled raw materials. The measured aluminum concentration of this alloy was 34.85 mol%. The microstructure of the alloy seems to consist of two phases of bright matrix and dark islands. An electron probe microanalysis (EPMA) revealed that the dark islands had lower aluminum and silicon concentrations and higher iron, chromium and manganese concentrations than the bright matrix, as shown in **Table 3**. Other two alloys synthesized from the recycled raw materials also had the similar microstructure, although the volume fraction of the dark islands decreased as aluminum concentration increased. We suspected that the dark islands might be multi-component precipitates, although in an X-ray diffraction (XRD) analysis only FeAl was detected for all alloys. However, an Al–Cr–Fe ternary phase diagram[16] indicates that both the compositions shown in Table 3 are within the solubility range of FeAl phase at 873 K. Because FeAl having aluminum concentration of 22.5 to 36.5 mol% transforms into Fe_3Al below 825 K,[16] we considered that the dark islands were Fe_3Al precipitates in FeAl matrix and that segregation during solidification brought about Fe_3Al precipitation even in an alloy having an aluminum concentration higher than 36.5 mol%.

Figure 5 shows the effect of aluminum concentration on the density of the alloy. The density decreased with increasing aluminum concentration for both calculated and measured results. The density of the present alloys is in a range between 5.5 and 6.5 g/cm^3. The densities of the alloys synthesized from the recycled law materials were all lower than the ideal density, while those of the alloys synthesized from the virgin raw materials were all very close to the ideal den-

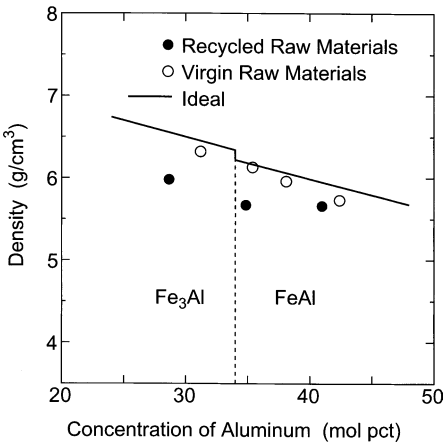

Fig. 5. Relationship between the aluminum concentration and the density of the alloy.

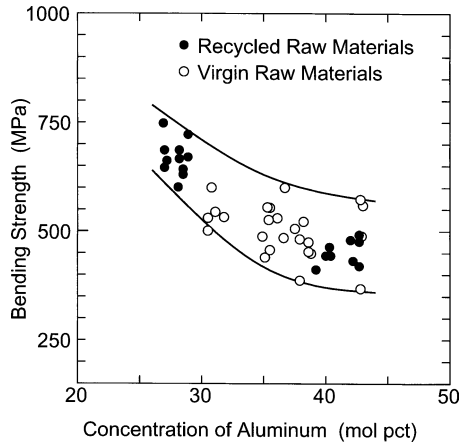

Fig. 7. Relationship between the aluminum concentration and the bending strength.

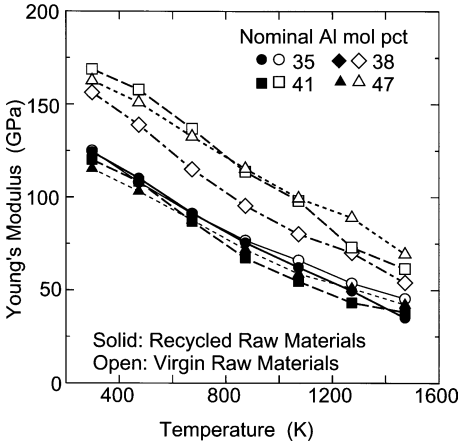

Fig. 6. Young's modulus at various temperatures.

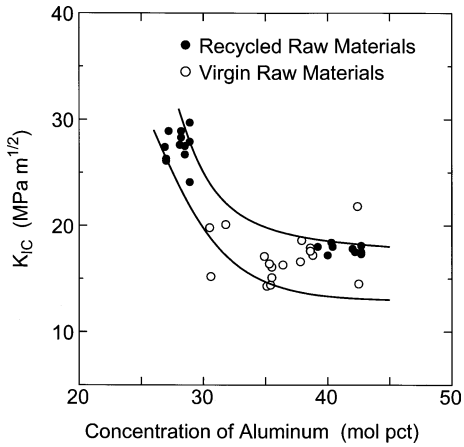

Fig. 8. Relationship between the aluminum concentration and the fracture toughness.

sity. It can be considered that the alloying elements listed in Table 1 reduced the density of the alloys synthesized from the recycled raw materials. However, the alloys synthesized from the recycled raw materials involved a small amount of fine pores, which may be a major reason why the density of these alloys is lower. The dotted line in Fig. 5 indicates the solubility limit of FeAl at 673 K based on an Fe–Al binary phase diagram,[17] which was taken into account in the calculation of the ideal density. However, it should be noted that the solubility limit shown in the figure is not rigorously valid for the alloys synthesized from the recycled raw materials because they contain some alloying elements as shown in Table 1.

3.3. Mechanical Properties

Figure 6 shows the Young's modulus measured at various temperatures between room temperature and 1 473 K. The Young's modulus decreased with increasing temperature for both the alloys, ranging from approximately 170 to 120 GPa at room temperature and 70 to 35 GPa at 1 473 K. The alloys synthesized from the virgin raw materials exhibited higher Young's modulus than the alloys synthesized from the recycled raw materials. Increase in aluminum concentration brought about an increase in Young's modulus of the alloys synthesized from the virgin raw materials, but the effect of aluminum concentration was little for the alloys synthesized from the recycled raw materials. It was considered that the existence of small amount of fine pores in the alloys synthesized from the recycled raw materials might have reduced the Young's modulus of these alloys and have put the effect of the aluminum concentration on the Young's modulus into disorder.

Increase in aluminum concentration reduced both the bending strength and fracture toughness of the alloys regardless of the raw materials. As shown in **Fig. 7**, the bending strength decreased from approximately 700 to 500 MPa as the aluminum concentration increased from 27 to 43 mol%, but the decrease in bending strength was very slight at higher aluminum concentrations. Similarly, as shown in **Fig. 8**, the fracture toughness decreased from approximately 30 to 17 MPa m$^{1/2}$ as the aluminum concentration increased from 27 to 43 mol%, but the decrease in fracture toughness was very slight at higher aluminum concentrations. It was reported that FeAl has lower ultimate tensile strength and poorer ductility compared with Fe_3Al,[18] which agrees with the results shown in Figs. 7 and 8. It may be seen that the alloys synthesized from the recycled raw materials exhibit a little higher values than the alloys synthesized from the virgin raw materials particularly in Fig. 7, but the effects of the raw materials seems to be negligible.

Vickers hardness of the alloy increased with the aluminum concentration, as shown in **Fig. 9**. The increase was very slight when the aluminum concentration was low, but

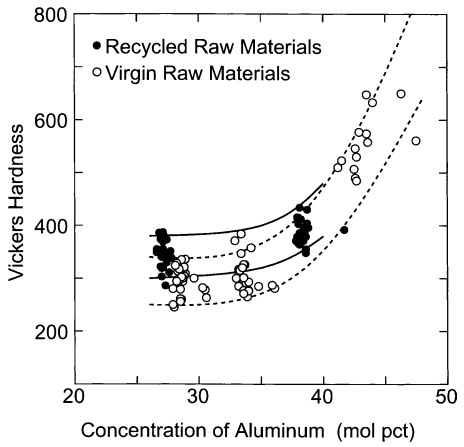

Fig. 9. Relationship between the aluminum concentration and the Vickers hardness.

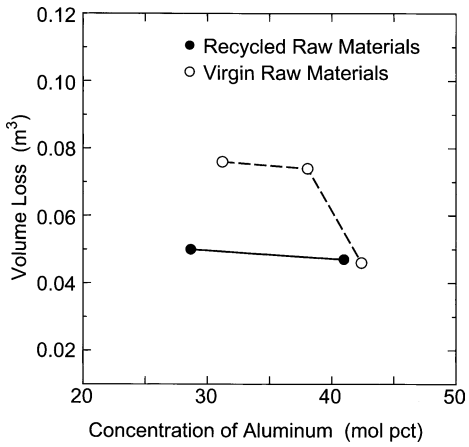

Fig. 10. Relationship between the aluminum concentration and the wear property.

it was remarkable when the aluminum concentration exceeded approximately 35 mol%. Chang *et al.*[19] reported that the Vickers hardness of FeAl increased with the aluminum concentration: it was approximately 250 to 420 at 40 mol% of aluminum concentration while 450 to 630 at 51 mol%. For lower aluminum concentrations, Ikeda *et al.*[20] investigated the effect of aluminum concentration on the Vickers hardness of Fe_3Al and found that the hardness varied in a range between approximately 280 to 320 when the aluminum concentration varied from 24 to 30 mol%. Those values of Vickers hardness are all similar to the present values measured for the alloys synthesized from the virgin raw materials. However, the alloys from the recycled raw materials exhibited higher values, as shown in Fig. 9. The solid solution hardening by the alloying elements was considered as the reason for this.

Figure 10 shows the effect of the aluminum concentration on the wear loss of the alloys after sliding on an SKD11 steel disk for a sliding distance of 600 m under a pressure of 2 MPa. As the aluminum concentration increased, the wear loss of the alloy synthesized from the virgin raw materials remarkably decreased particularly in the high aluminum region, while that of the alloy from the recycled raw materials was smaller over a wide range of the aluminum concentration. The results shown in Fig. 10 seem to be a reflection of the relationship between the hardness and the aluminum concentration shown in Fig. 9. The iron aluminide intermetallic alloys synthesized from the recycled raw materials exhibited higher hardness and more excellent wear resistance than the alloys synthesized from the virgin raw materials.

4. Conclusions

Iron aluminide intermetallic alloys were synthesized from recycled raw materials of steel machining chips made at a bearing case maker and aluminum shreds made of used cans, and some mechanical properties of the alloys were investigated. The results are summarized as follows.

(1) The steel chips and aluminum shreds exothermically react and produce iron aluminide intermetallic alloys, when compacts of the steel and aluminum mixture are heated to a temperature above the melting point of aluminum.

(2) The bending strength, fracture toughness, Vickers hardness and wear resistance of the alloys are more excellent than or similar to those of the alloys synthesized from pure iron and aluminum.

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