Rev. Adv. Mater. Sci. 28 (2011) 94-97

PREPARATION OF Fe-8%Ni ALLOYED POWDER FABRICATED BY NOVEL RECYCLING PROCESS

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Received: February 17, 2011

Abstract. Fe-8%Ni alloyed powder was fabricated by oxidation and mechanical alloying technique using Fe-36%Ni scrap. Oxidation behavior of Fe-36%Ni alloy was studied in air using thermogravimetric apparatus (TGA), and oxidation rate was shown to be sharply increased after 500 °C. It was found that the alloyed scrap was completely oxidized at 1100 °C for 96 hours. Fe_2O_3 and NiO were observed to be the major phases in oxide scale of the scrap. The scrap completely oxidized was ball-milled to make average particle size of 1.5 μm. The oxide powder was synthesized in wet-ball milling with addition of Fe_2O_3 powder in order to meet the desire composition. Finally, Fe-8%Ni alloyed powder was obtained after reduction in H_2 gas atmosphere at 650 °C for 5 hours, and the powders were examined by X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscopy (SEM).

1. INTRODUCTION

Fe-Ni alloys are of great interest due to their low thermal expansion and remarkable magnetic and mechanical properties. Especially, they have been widely used as structural materials in architecture fields [1]. Fe-Ni alloy powders have been prepared by various methods, including melting, mechanical alloying, chemical reduction, and gas condensation [1-6]. Unfortunately, most processes to fabricate Fe-Ni alloys have used high purity Fe and Ni powders. In this study, Fe-8%Ni alloy powders were synthesized using Fe-36%Ni alloy scraps, which is a novel recycling process newly proposed to utilize wasted materials. The oxidation and mechanical alloying technique were conducted to fabricate Fe-8%Ni, and the oxide morphology, mean powder size, and chemical composition were investigated by X-ray diffraction (XRD), X-ray fluorescence (XRF), and scanning electron microscopy (SEM).

2. EXPERIMENTAL PROCEDURES

Fe-36%Ni scrap obtained from industrial wastes left behind after original materials have been processed was used to fabricate Fe-8%Ni alloy powder, and it has a shape of ribbon with thickness of about 2 mm shown in Fig. 1. The collected scraps were cut into a rod-shape with about 10 cm in length for the oxidation process.

Overall process to fabricate Fe-8%Ni powders from the alloyed scrap is shown in Fig. 1. Fe-based scraps were oxidized in air at 1100 °C for 96 hours. The rod-shape of Fe-36%Ni oxidized scraps was ball-milled for 12 hours to fine powders having about 1~5 μm in size, and then they were uniformly mixed again with Fe_2O_3 powders with a similar size in order to meet the desired composition of Fe-8%Ni powder. The powder mixture was wet milled in acetone at 70 rpm for 24 hours with weight ratio of ball to powder as 3:1. After drying the wet milled pow-

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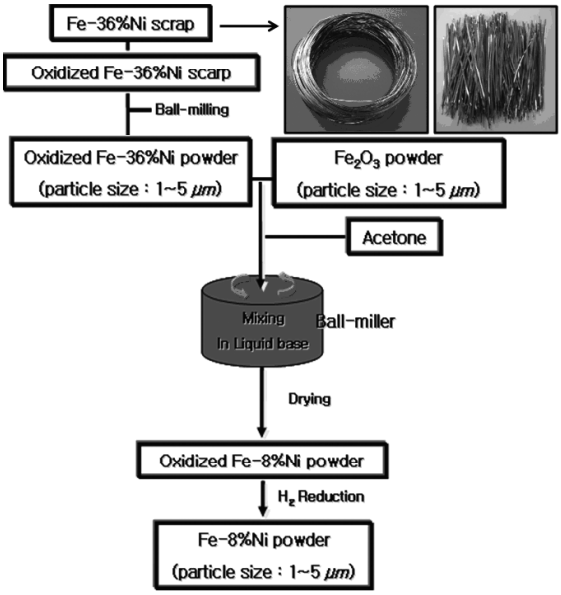

Fig. 1. Flowchart of overall process for fabrication of Fe-8%Ni alloy powders.

ders in a dry oven at 150 °C, they were reduced using H_2 gas at 650 °C for 5 hours. Finally, the fine Fe-8%Ni powder with 1~5 μm in size was obtained after cooled down in UHP argon atmosphere.

3. RESULTS AND DISCUSSION

Thermogravimetric analysis (TGA) was conducted to study oxidation behavior of Fe-36%Ni scrap to find out suitable oxidation temperature and time. Fig. 2 shows oxidation ratio and weight gain of Fe-36%Ni as a function of temperature and time in air. The weight gained was slightly increased up to 1100 °C for 48 hours and then abruptly increased as oxidation time increased from 48 to 96 hours at 1100 °C. Oxidation ratio was shown to be increased with increasing temperature and time and it can be seen that the alloy scrap might be completely oxidized at 1000 °C for 96 hours. From the data obtained, it can be concluded that oxidation should be carried out at least 1100 °C for 96hours in air to transform the specimen to oxide phases.

Fig. 3 shows a schematic diagram of oxidation layer of Fe-36%Ni alloy formed in regard to oxidation temperature and Ni ratio (%) in Fe-Ni Alloy [7]. It is seen that A-type of oxidation layer on the surface can be formed when the oxidation temperature is below 850 °C, whereas oxide layers might expect to be formed as a B-type in experimental conditions of this study at the temperature range of 950 to 1100 °C with 36% Ni content. Based on the diagram of oxide layers stated previously, cross-sectional areas of Fe-36%Ni alloy oxidized at increasing temperature were observed to characterize oxidation behavior and morphology of the layers.

Fig. 4 shows the cross-sectional areas of Fe-36%Ni oxidized scrap using scanning electron microscopy (SEM). From the image of each layer, it was found that oxidation almost did not occur at the inside of Fe-36%Ni alloy at 950 °C for 10 hours, but Fe_3O_4 was only formed on the outer surface. The thickness of oxidation layer was found to be increased as temperature increases, which meant the increase of oxidation. In addition, oxide scale conducted at 1100 °C for 96 hours was found to have similar microstructure of α-Fe_2O_3, FeO+Fe_3O_4, and Fe_3O_4 as expected in Fig. 3 and each oxide was widely spread in most oxide areas. The struc-

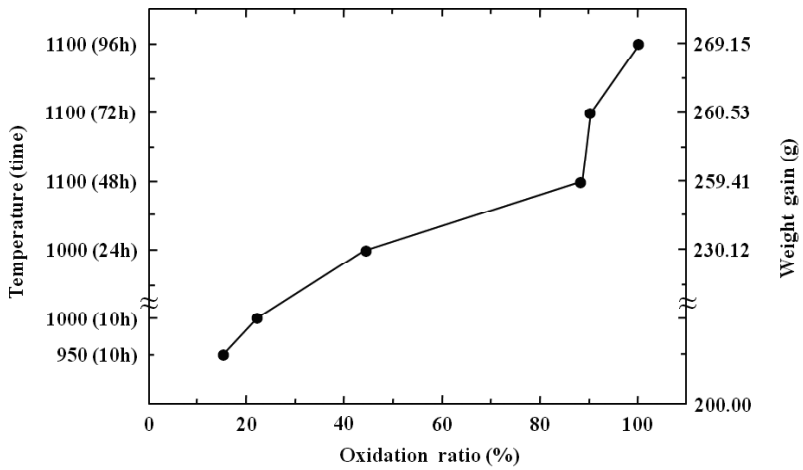

Fig. 2. Weight gain and oxidation ratio of Fe-36%Ni alloy scrap conducted in air as a function of temperature and time.

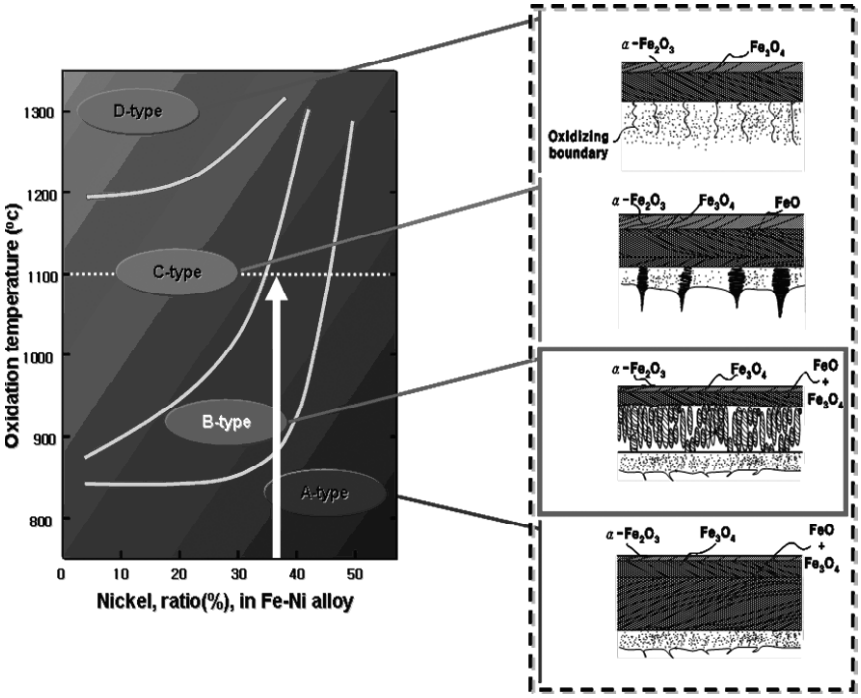

Fig. 3. Schematic diagram of oxide layers formed depending on oxidation temperature and Ni content in Fe-Ni alloy.

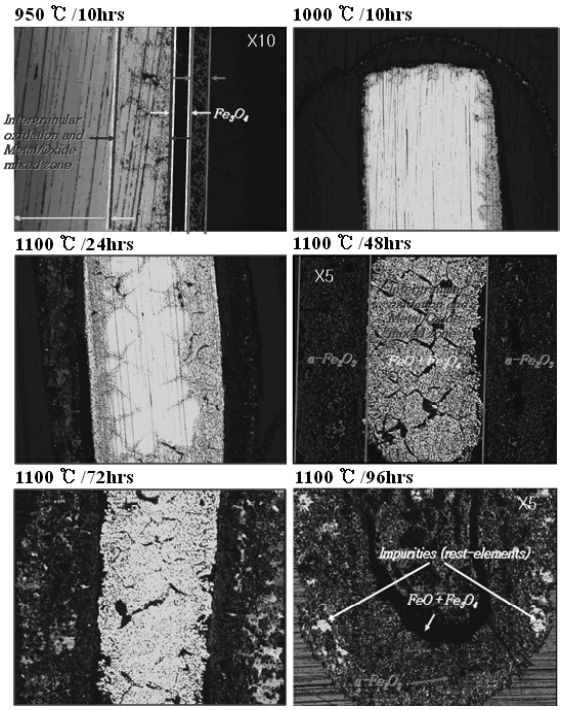

Fig. 4. Cross-section area of Fe-36%Ni alloy scrap oxidized in air as a function of temperature and time.

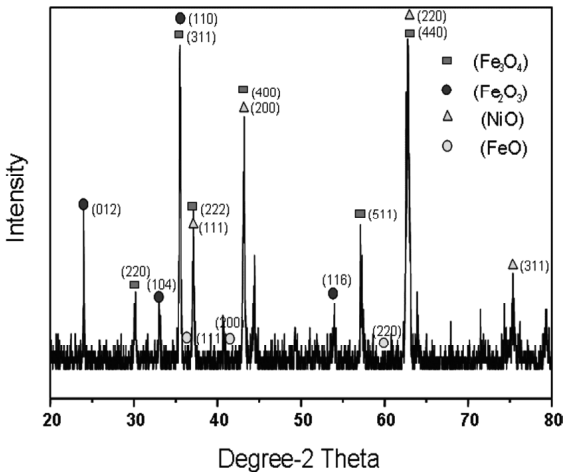

Fig. 5. XRD patterns of Fe-36%Ni alloy scrap oxidized at 1100 °C for 96 hours.

ture of oxide scale of Fe-36%Ni scrap was observed to be composed in the order of oxide surface layer, mixture layer of metal-oxide, and metal substrate, and the oxide surface and the mixture layer were grown as oxidation time increased. It might be explained that O_2 was penetrated through the surface layer into metal substrate leading to form the metal-oxide mixture layer at the beginning stage. Subsequently, Fe ion from the substrate easily diffused out to develop the mixture layer and then continued to move to outer layer, since diffusion coefficient of Fe ion in oxide phases such as FeO, Fe_2O_3, Fe_3O_4, and NiO was known to be much higher than that in metal phases such as Fe and Ni. Fig. 5 illustrates XRD patterns of Fe-36%Ni scrap oxidized at 1100 °C for 96 hours. According to the result of XRD, the alloyed scrap was mainly made up of a mixture of

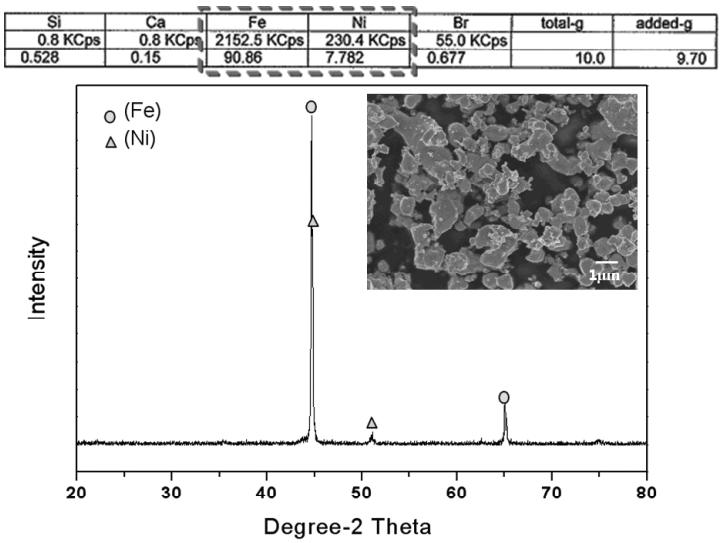

Fig. 6. XRD pattern, chemical composition, and SEM image of Fe-8%Ni powder after reduction of H_2 gas at 650 °C for 5 hours.

Fe_2O_3 and NiO with minor phases of FeO and Fe_3O_4, which were expected in cross-sectional area shown in Fig. 4.

In order to fabricate Fe-8%Ni alloy powder in desired composition, the oxidized scrap in rod-shape must be evenly mixed with pure Fe_2O_3 powder first. To do that, rod shape of the scraps should be ball-milled to be similar size of Fe_2O_3 powder whose mean size was analyzed to be about 1.5 μm. In other words, Fe-36%Ni scrap oxidized in this study must be grinded in the size of under 1.5 μm to mix it uniformly with Fe_2O_3 powder. Finally, about fine powders with an average size of 1.5 μm were obtained after ball-milled for 12 hours. Oxidized Fe-36% Ni alloy powder and Fe_2O_3 powder were wet ball-milled in acetone for 24 hours at 70 rpm, and then dried at 150 °C in dry oven. The oxide sample was continuously reduced by hydrogen gas at 650 °C for 5 hours and then cooled down to room temperature under UHP Ar gas atmosphere to prevent oxidation.

Fig. 6 shows the results of XRF analysis confirming that target composition of Fe-8%Ni was successfully accomplished, and the alloy powders synthesized were found to be composed of only Fe and Ni indentified by XRD analysis. As shown in the image taken by SEM, Fe-8%Ni alloy powder with mean size of about 1 μm was finally obtained.

4. CONCLUSION

Fe-8%Ni alloyed powder was fabricated by oxidation and mechanical alloying technique using Fe-36%Ni scrap.

1. Fe-36%Ni alloy scrap was completely oxidized at 1100 °C for 96 hours, and it was mainly composed of Fe_2O_3 and NiO with FeO and Fe_3O_4 as minor phases.
2. Fe-8%Ni alloy powder was successfully obtained from the mixture of Fe-36%Ni oxidized scrap and Fe_2O_3 powder after reduction with H_2 gas.

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