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Structure and Magnetic Properties of a $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ Alloy Nanopowder Fabricated by a Chemical Etching Method and Milling Procedure

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The magnetic and structural properties of FINEMET (the Hitachi product name of the Fe-Si-B-Nb-Cu alloy) nanopowder with a composition of $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ atomic percent were investigated after annealing, chemical etching, and mechanical milling. The primary and secondary crystallization temperatures were 523 and 550 °C, respectively. The grain size of the particles was adjusted by annealing time. Optimally annealed particles exhibited a homogenous microstructure composed of nanometer-sized crystalline grains. The grain boundary of the annealed particles was etched preferentially by chemical etching. Chemically etched particles were broken at the grain boundary by high-energy ball milling. As a result, a nanometer-sized FINEMET powder with a uniform size of crystalline grains was fabricated.

Keywords : $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy, chemical etching, ball milling

1. Introduction

Magnetic devices are used in electric transforming devices and serve as the core component of electro-electric instruments. The representative materials of these devices are soft ferrite materials. With the recent development of the IT (Information Technology) industry, high-performance at high-frequency, high-storage abilities, and small size have become prerequisites for the electro-electric products. Therefore, soft ferrite materials are faced with the weak points to solve the industrial needs because soft ferrite can't decrease their critical volume due to their low saturation magnetization. [ED HIGHLIGHT-Unsure of inteded meaning; unable to edit with confidence] Thus, amorphous materials have become the new materials of focus to overcome the weak points of soft ferrite materials. Amorphous materials have high saturation magnetization and permeability but also suffer from sudden decreases in permeability and increases in core loss under high frequencies. In order to solve these problems, nanostructured amorphous materials have been considered. Typical alloys of nanostructured amorphous materials are Fe-Si-B-Nb-Cu and Fe-M-B (M = Zr, Hf, Nb, Si, Al). The required properties of these materials are high permeability, saturation magnetization, low coercivity, and core loss. A $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy, known as FINEMET, is an attractive soft magnetic material that can be used in electric power applications such as transformer cores and other inductive devices. It exhibits excellent permeability (~10^5 at 1 kHz), a low saturation magnetostriction (~2×10^{-6}), and a relatively high saturation magnetization (~1.2 T) [1-3].

In order to develop nanostructured amorphous materials, $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ nanopowders were fabricated by chemical etching and mechanical milling in this study. Microstructures of this powder were investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD). Magnetic properties were investigated by vibrating sample magnetometry (VSM).

2. Experiments

A micrometer-sized $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy powder fabricated by a high-pressure water spray method was used as the starting material. The shape of the raw powder was spherical with a particle size of approximately 10 μm. First, the powder was controlled from the spherical shape to a flake shape by attrition milling. Fig. 1 shows

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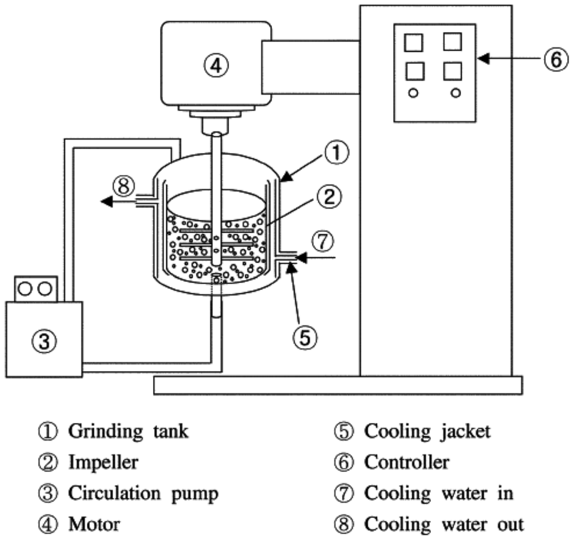


Fig. 1. Schematic diagram of the attrition milling equipment.

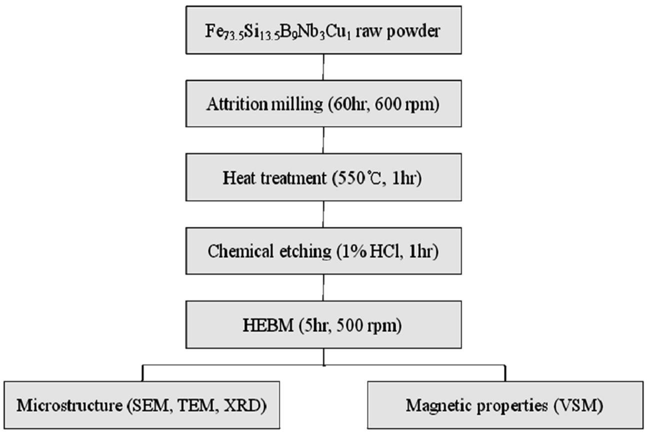


Fig. 2. Flow chart of the experimental procedure.

the attrition milling equipment. Attrition milling was carried out for 60 h at 600 rpm. The rate of ball and powder was 1:1 and that of the powder and ethanol was

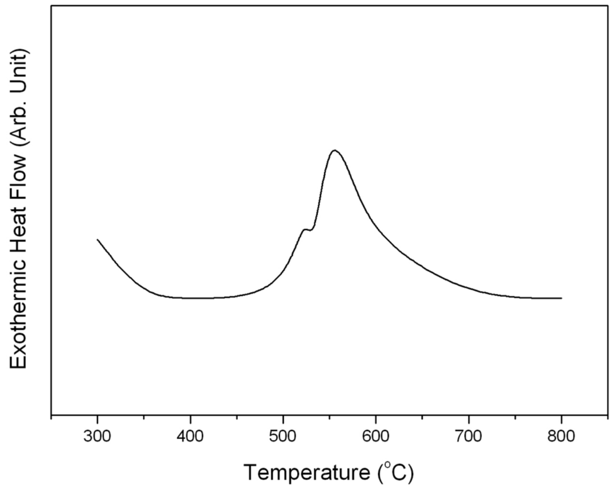


Fig. 3. Crystallization temperature of iron in the $Fe_{73.5}Si_{13.5}$-$B_9Nb_3Cu_1$ powders by DSC analysis.

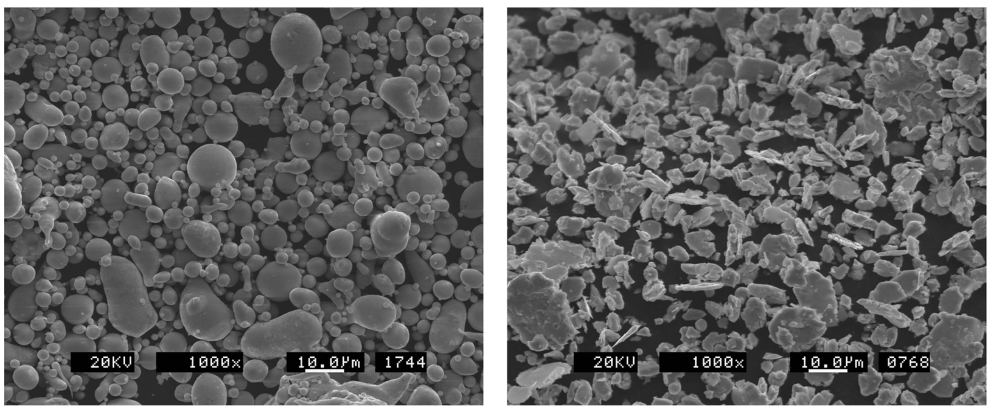
1:5 [4].

After attrition milling, the powder was dried at 70 °C, followed by heat treatment for 1 h at 550 °C. This crystallization temperature of the Fe was obtained by differential scanning calorimetry (DSC) analysis. Chemical etching was performed with 1% HCl for 1 h. After chemical etching, the powder was milled by high-energy ball milling for 5 h at 500 rpm. The procedure of the nanostructured powder fabrication is shown in Fig. 2.

The microstructure of the nanostructured $Fe_{73.5}Si_{13.5}$-$B_9Nb_3Cu_1$ powder was investigated by SEM, TEM, and XRD techniques. Magnetic properties of the powder were obtained by VSM.

3. Results and Discussion

Fig. 3 shows the DSC curve of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder. Primary and secondary peaks appeared at 523



(a) (b)

Fig. 4. Shape variations in the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder by SEM: (a) raw powder; (b) attrition milled powder.

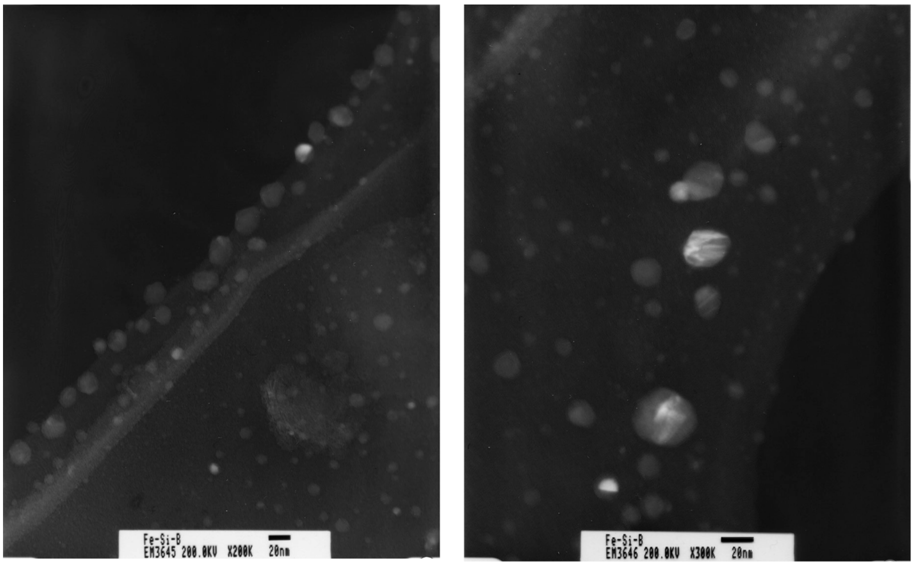


Fig. 5. TEM images denoting particle size and shape of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder after high-energy ball milling.

and 550 °C, respectively. The peaks indicate the crystallization temperatures of iron. As a result, the heat treatment temperature was determined as 550 °C. The shape variation in the powder by attrition milling is shown in Fig. 4. After attrition milling, the particle shape changed to a flake shape, with a 300 nm thickness.

Fig. 5 indicates the particle size and shape of the powder after high-energy ball milling. The particle shape is generally spherical, about 10~25 nm. The variation in the microstructure depends on each procedure, as shown in Fig. 6 with the XRD pattern, which indicates an amorphous structure, Fig. 6(a). The microstructure of the powder has a partial crystal phase during attrition milling, Fig. 6(b), due to the simultaneous re-alloying process. After heat treatment, the microstructure of the powder is changed into the crystal phase completely, Fig. 6(c).

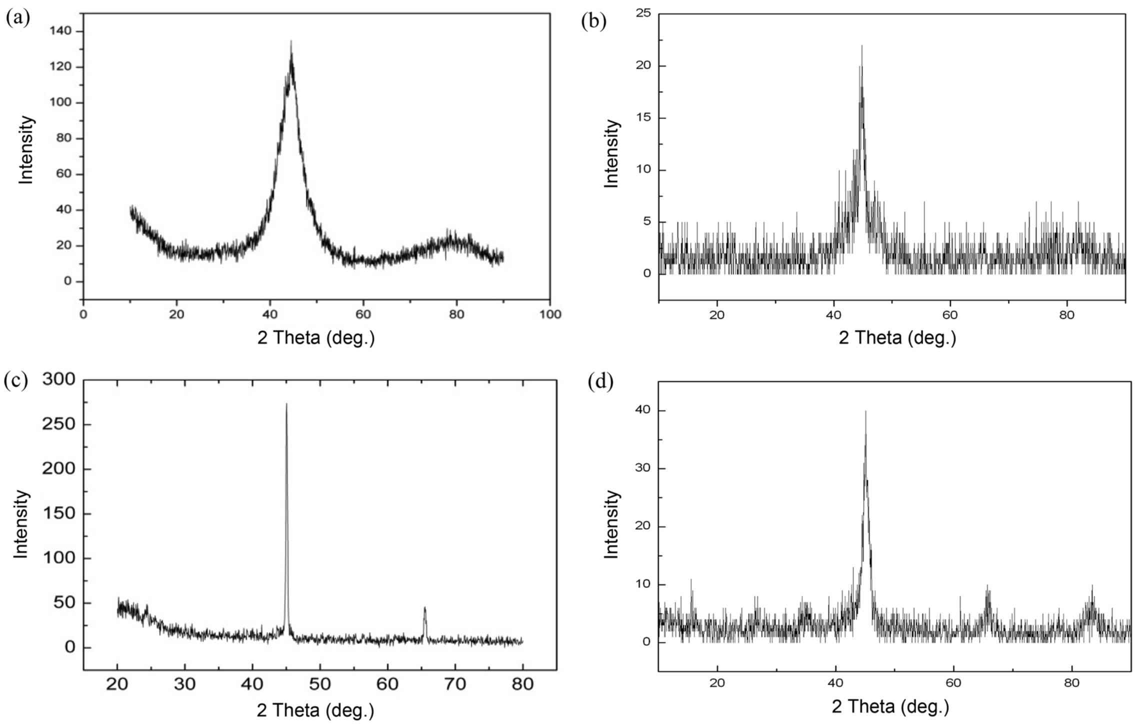


Fig. 6. XRD patterns of the powder according to each procedure: (a) raw powder; (b) after attrition milling; (c) after heat treatment; (d) after high-energy ball milling.

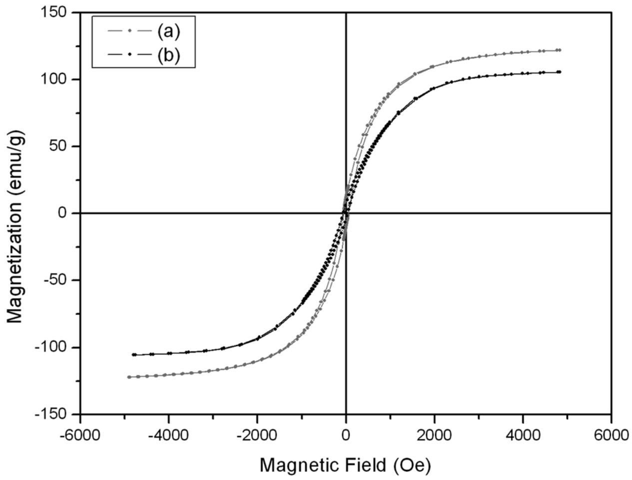


Fig. 7. Hysteresis curves of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powders: (a) before chemical etching; (b) after chemical etching.

Although particle size was rendered nanosized by high-energy ball milling, the crystal phase still remained in the particle, Fig. 6(d).

Magnetic properties of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder are shown in Fig. 7. The saturation magnetization was reduced after chemical etching and high-energy ball milling. The reason for the decrease was the inclusion of impurities that occurred during the chemical etching process. The coercivity of the powder was not changed nearly to that extent.

4. Conclusions

The $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ nanopowder was fabricated by annealing, chemical etching, and ball milling procedures. The spherical shape of the particle was changed into a flake shape by attrition milling. After attrition milling, the particle size was approximately 10 μm with a thickness of nearly 300 nm. The $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder was annealed at 550 °C, the crystallization temperature of iron. The grain boundary of the annealed particles was etched preferentially by chemical etching. Chemically-etched particles were broken at the grain boundary by high-energy ball milling. The particle size was 10~25 nm after high-energy ball milling. The saturation magnetization value of 120 emu/g was reduced to 100 emu/g because of the inclusion of impurities likely introduced during the chemical etching process.

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