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Magnetic and Structural Properties of nitrified FINEMET Powders by Using Mechanical Milling Method

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The magnetic and structural properties of FINEMET [$Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ wt %] amorphous powder were investigated after nitrification and mechanical milling. Fe-based amorphous powder was nitrified and crystallized simultaneously at 550 ℃ by using ammonia(NH_3) gas. Nitrified powder exhibits iron nitride phases such as γ′-Fe_4N, Fe_3N and α″-$Fe_{16}N_2$. Nitrified particles were more brittle than raw particles. As a result, nanometer sized nitride powder was fabricated by a high energetic ball milling method. The saturation magnetization (M_s) and coercivity (H_c) of nitrified powder were increased due to nitride phases.

Key words : nanoscale, nitride, FINEMET, amorphous powder, mechanical process

1. INTRODUCTION

The intensive development of material engineering in the last decade caused, among the others, an increase in the interest of the nanocrystalline soft magnetic materials. Such materials may be obtained by various methods, however, the most common method is the thermal nanocrystallization of metallic glasses. The research on nano materials obtained by this method was initiated by Yoshizawa et al. in 1988 [1].

The $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy with a nanocrystalline grain structure, known as FINEMET, is an attractive soft magnetic material, which is found to use in electric power applications such as transformer cores and other inductive devices. It exhibits an excellent permeability (~10^5 at 1 kHz), a low saturation magnetostriction (~2 x 10^{-6}) and a relatively high saturation magnetization (~1.2 T). The premier soft magnetic properties are based on the two-phase microstructure consisting of nanocrysalline ferromagnetic grains surrounded by ferromagnetic amorphous matrix. In order to obtain optimal microstructure and properties, amorphous precursors are generally annealed near 550 ℃, which is above the primary crystallization temperature of ~ 510 ℃. The partitioning of glass formers develops to limit the growth of nanometer-sized crystallines (grains) to typically below 10 nm in the average size. Since the grain size of the nanocrystalline phase(s) is much smaller than the magnetic exchange length, the magnetocrystalline anisotropy and magnetostrictive coefficients are averaged out over many small grains (as explained by a random anisotropy model) [2]. FINEMET and other nanocomposite magnets have been reviewed recently by McHenry et al. [3].

Also the metastable iron-nitrogen compound $Fe_{16}N_2$ has attracted much interest because Kim and Takahashi [4] found that it shows a saturation magnetization higher than that of iron by about 30 %. The investigations of this material have been restricted to thin films mainly because of low solubility of nitrogen in α-iron. Investigations of producing iron-nitride have been made recently applying the ammonia (NH_3) gas.

In this work, we thus report the magnetic and structural properties of nitrified FINEMET powder prepared by mechanical milling method.

2. EXPERIMENT

In the research, the starting materials are FINEMET powder with 99.5 % purity and 10-20 μm diameter prepared by high-speed water spray method. The first milling procedure of the powder was carried out by an attrition milling method, for 10h, 800rpm. The powder was heat treated and nitrified by holding at 550 ℃ for 1h in the ammonia (NH_3) gas atmosphere. After the nitrification process, the second milling procedure was carried out by a high-energetic ball milling method, for 10 h at 500 rpm.

The crystal structure of the powder was analyzed by X-ray diffractometer (XRD) with CuKα radiation source. The scanning electron microscope (SEM) was used for examination of micro structure of powder. The magnetic properties were measured by a vibrating sample magnetometer (VSM).

3. RESULT AND DISCUSSION

X-ray examinations show that the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy powder in the initial as quenched structure is amorphous, which is revealed on the diffraction pattern as a wide-angle peak coming from the amorphous phase (Fig. 1). Nitrification at 550 ℃ causes crystallization of the amorphous matrix and formation of the nitrides, which is testified by peaks coming from the γ′-Fe_4N, Fe_3N, α″-$Fe_{16}N_2$ and α-Fe(Si) phases (Fig. 2)

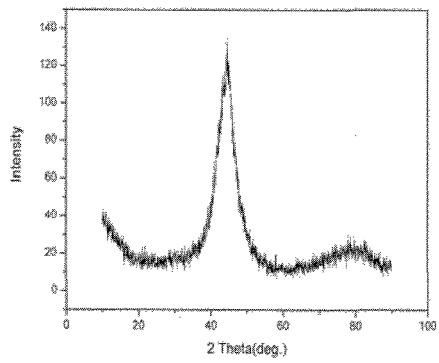

Fig. 1. X-ray diffraction pattern of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy powders in the initial as quenched state.

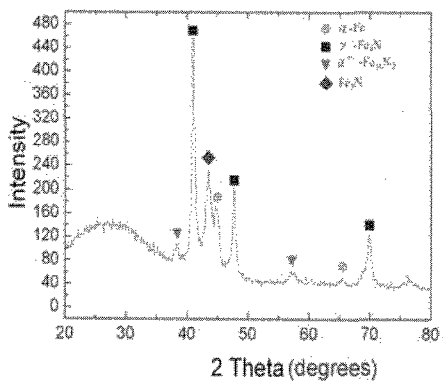

Fig. 2. X-ray diffraction pattern of the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ alloy powders after nitrification at 550 ℃for 1h.

Observation of shapes and dimensions of powder particles, depending on the milling method type, revealed that the particles become flake shapes and that their sizes decrease. The flake shapes were obtained in the process of the attrition milling method, which is a common method to make flake shapes to increase the surface area (Fig. 3 (b)). After the nitrification process, almost all of pure particles changed to nitride particles with γ′-Fe_4N, Fe_3N, α″-$Fe_{16}N_2$ phases (Fig.3 (c)). Since the nitride phases are more brittle than the pure phase, nitride particles are ground more easily by a high-energetic ball mill method (Fig. 3 (d)).

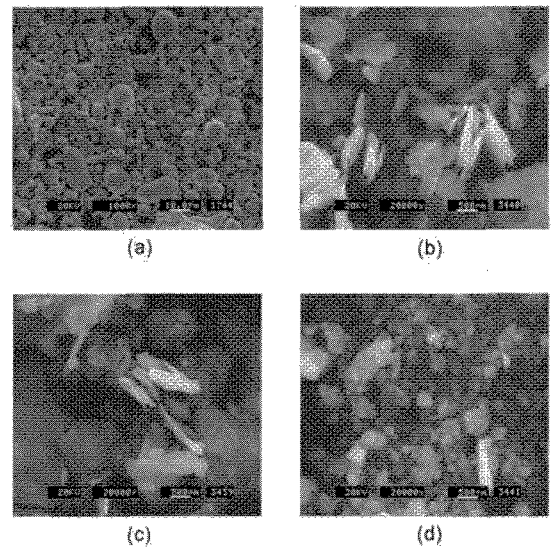

Fig.3. Comparison of shapes and sizes of FINEMET powder particles. (a) Raw powder, (b) After 1st milling process, (c) After nitrification process, (d) After 2nd milling process.

The changes of magnetic properties during the milling and nitrification process are shown in Fig. 4. The saturation magnetization (M_s) after the 1st milling process is similar to that of FINEMET raw powder, though the coercivity (H_c) increases because the crystallization occurs partially during the milling process (Fig. 4 (a)). Both the saturation magnetization and coercivity increase after the nitrification process. The increase of M_s is due to the formation of nitride phases such as γ′-Fe_4N and α″-$Fe_{16}N_2$ phase (Fig. 4 (b)). After the 2nd milling process, the saturation magnetization increases a little as compared with the previous process (Fig. 4 (c)). This increase is caused by eliminating impurities that were formed in the nitrification process during the 2nd milling process.

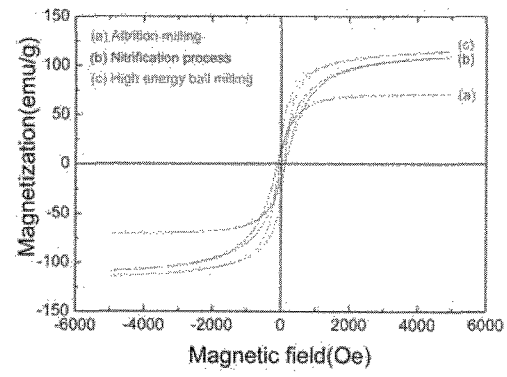

Fig. 4. Hysteresis curves with applied milling and nitrification process.

4. SUMMARY

Fine sized FINEMET powders were obtained by mechanical alloying method. As the milling time increases, the aspect ratio of the powders increases and average granularity of the powders decreases. FINEMET powders are nitrified by flowing NH_3 gas, as a result γ'-Fe_4N, Fe_3N, and α''-$Fe_{16}N_2$ phases are originated. The magnetic properties (M_s and H_c) of the powders increase from nanocrystallization and iron-nitride

5. ACKNOWLEDGEMENTS

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6. REFERENCES

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