Effects of SiO_2 on Magnetic Properties of Al_2O_3-Coated Acicular α-Fe Particles

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Abstract—(SiO_2, Al_2O_3)-coated acicular α-Fe particles were obtained by first coprecipitating Si and Al ions on the surface of α-FeOOH particles and next the α-FeOOH particles were dehydrated and reduced by hydrogen.

The effects of Si ion on Al_2O_3-coated α-Fe particles are as follows: (1) it prevented the acicular α-Fe particles from particle growth and grain growth during reduction; (2) a suitable amount of Si ion (0.3 wt%) in the particles only slightly decreased the σ_s value but significantly increased the $_iH_c$ value of the particles; (3) 0.3 wt% of Si ion effectively prevented the σ_s value of the Al_2O_3-coated α-Fe particles from deteriorating with time, higher Si ion content was required to prevent the $_iH_c$ from deteriorating; and (4) Si ion could not prevent the magnetics of the Al_2O_3-coated α-Fe particle from deteriorating under severe surroundings.

INTRODUCTION

The surface of α-Fe particles used in recording media must be coated with a thin layer of SiO_2 [1], Al_2O_3 [2], Fe_3O_4 [3], borates [4], and phosphates not only for maintaining the particles in their acicular shape during dehydration and reduction, but also in preventing corrosion. The addition of those compounds, although invariably decreasing the σ value of α-Fe particles, increases the $_iH_c$ value, stabilizes $_iH_c$ and σ values and prevent these two values from deteriorating with time. The amount of coating materials needed depends on the surface area of the α-Fe particles. The present authors[5] have demonstrated that the addition of 1.5 wt % Al ion, which maximizes the $_iH_c$ value of α-Fe particles but does not significantly affect the σ value of the particles, is a suitable amount to coat on the surface of α-Fe particles. In this study, various amounts of si ion are added together with 1.5 wt% of Al ion to investigate whether it can further improve or stabilize the magnetic properties of α-Fe particles.

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EXPERIMENTAL

The coating agents Si and Al ion were added in the form of $Al_2(SO_4)_3$ and Na_2SiO_3 solutions into a alkali α-FeOOH suspension solution, which was obtained by chemical precipitation from $FeCl_2$ solution by a NaOH solution and subsequent air oxidation [5]. The concentration of Al ions on the Fe particles was maintained at 1.5 wt%, and the concentration of Si ion was varied from 0 to 1.5 wt%. The acicular α-Fe particles were then produced from (Al_2O_3, SiO_2)-coated acicular FeOOH through air dehydration of FeOOH to α-Fe_2O_3 at 700°C for 1 hr, hydrogen reduction of α-Fe_2O_3 to α-Fe at 500°C for 2 hrs.

The environmental stability of (SiO_2, Al_2O_3)-coated α-Fe particles was tested by placing the particles in the following different environments: (1) in open air at room temperature for up to 6 weeks, (2) at elevated temperatures ranging from 25°C to 360°C for 1 hr, and (3) in a humid environments, with 90% relative humidity at 60°C for up to 6 weeks.

The contents of Si and Al ions in the α-Fe particle were analysed by ICP. The shape, structure, and magnetic properties of the α-Fe particles were measured by TEM, x-ray diffractometry, VSM, respectively.

RESULTS AND DISCUSSION

Electron photomicrographs of the acicular Al_2O_3-coated α-Fe particles (Fig. 1a and 1b), which contain 0.3 wt% and 1.5 wt% of Si respectively, reveal that the higher concentration of Si ion is, the smaller the particle size and the higher the aspect ratio of the α-Fe particles are. The addition of Si ion in the Al_2O_3-coated α-Fe particles does effectively prevent the particle from further growth. The grain size of Al_2O_3-coated α-Fe particles in Fig. 2, as calcalculated by the Scherrer formula from the x-ray diffraction pattern (110 plane) of the α-Fe particles, decreased with the concentration of Si ion. The excessive growth of α-Fe particles and the growth of grain size in the α-Fe particle during hydrogen reduction process are undesirable. Consequently, the addition of Si ion effectively prevented the grains in the α-Fe particle and the particle itself from growth. In particular the Si ion concentration in the range of up to 0.4 wt% is more criti-

cal in blocking the grain from growth.

The addition of Si ion to Al_2O_3-coated α-Fe particles from 0 to 0.3 wt% (Fig. 3) only slightly decreases the σ_s value of the α-Fe particles; however, it dramatically increases the $_iH_c$ value to 1360 Oe. If the addition of Si ion in the α-Fe particles is higher than 0.3 wt%, the $_iH_c$ value decreases and levels off, but the σ_s value sharply decreases. This is due to the fact that a higher reduction temperature is needed for a higher content of Si ion. In the previous report [5], 1.5 wt% Al ion maximizes the $_iH_c$ of the α-Fe particles. An addition of 0.3 wt% of Si ion together with 1.5 wt% Al ion further maximizes the $_iH_c$ of the α-Fe particles to a higher peak (1360 Oe).

After the (SiO_2, Al_2O_3)-coated α-Fe particles were placed in open air (about 75% humidity) at room temperature for up to 6 weeks, both the σ_s and $_iH_c$ values of the particle decreased because of the oxidation of the particles. After 6 weeks of air exposure, the $\sigma_s(t)/\sigma_s(0)$ value in Fig. 4, i.e., the new σ_s value relative to original σ_s value versus time, revealed that 0.3 wt% of Si ion is the most effective concentration in preventing the decrease of σ_s. A higher concentration of Si ion, e.g., 1.5 wt%, could accelerate the decrease of σ_s value. The $_iHc(t)/_iHc(0)$ value in Fig. 5 reveals that Al_2O_3-coated α-Fe particles with 1.5 wt% of Si ion has the highest $_iHc(t)/_iHc(0)$ value. The addition of Si ion improves the $_iH_c$ deterioration resistance of the α-particles to the surrounding environment and is always proportional to the amount of Si ion added.

If the α-Fe particles are placed under more severe surroundings, e.g., at 60°C and 90% humidity for 6 weeks, the addition of Si ion in Fig. 6 and 7 does not increase σ_s or $_iH_c$ deterioration resistance. A higher concentration of Si ion worsens the $_iH_c$ deterioration resistance.

The values of $\sigma_s(T)/\sigma_s(25°C)$ and $_iH_c(T)/_iH_c(25°C)$ in Fig. 8 and 9, respectively, reveal that the magnetic properties of α-Fe particles, as measured at elevated temperatures up to 350°C under inert atmosphere, decrease with an increasing temperature because of the thermal instability and slightly partial oxidation of the materials. Both $\sigma_s(T)/\sigma_s(25°C)$ and $_iH_c(T)/_iH_c(25°C)$ values reduced to around 0.8 when the temperature was heated to 350°C. The $\sigma_s(T)/\sigma_s(25°C)$ and $_iH_c(T)/_iH_c(25°C)$ values increase with the amount of Si ion added because both $\sigma_s(25°C)$ and $_iH_c$ (25°C) values are originally smaller and the reduction is not so obvious.

CONCLUSIONS

The addition of Si ion in Al_2O_3-coated α-Fe particles prevents the acicular α-Fe particles from particle growth and grain growth in the particles. A suitable amount of Si ion (0.3 wt%) in Al_2O_3-coated α-Fe particles only slightly decreases the σ_s value but significantly increases the $_iH_c$ value of the particles. This suitable amount of Si ion also assists the stability toward deterioration with time. Under severe humidity (90%), elavated temperature (60°C), and long exposure time (6 weeks), the addition of Si ion does not prevent deterioration from occuring. The addition of Si ion prohibits both $\sigma_s(T)/\sigma_s$ (25°C) and $_iH_c(T)/_iH_c(25°C)$ values from decreasing. A combination of Si and Al ions addition can be better than either one alone.

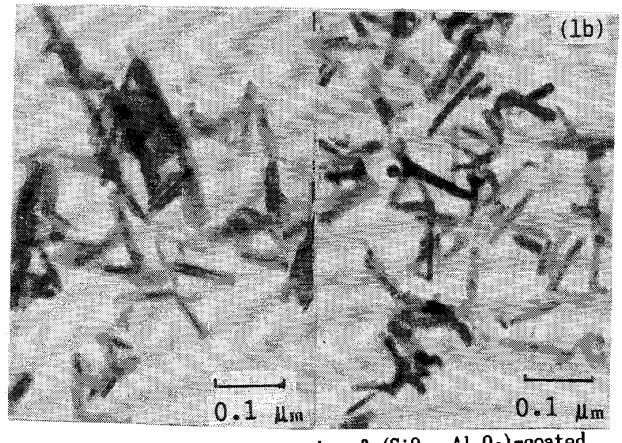

Fig. 1 Electron micrographs of (SiO_2, Al_2O_3)-coated acicular α-Fe particle

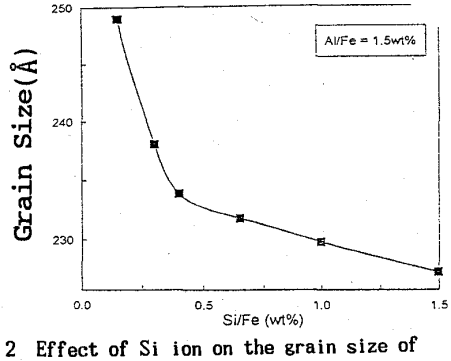

Fig. 2 Effect of Si ion on the grain size of (SiO_2, Al_2O_3)-coated acicular α-Fe particle

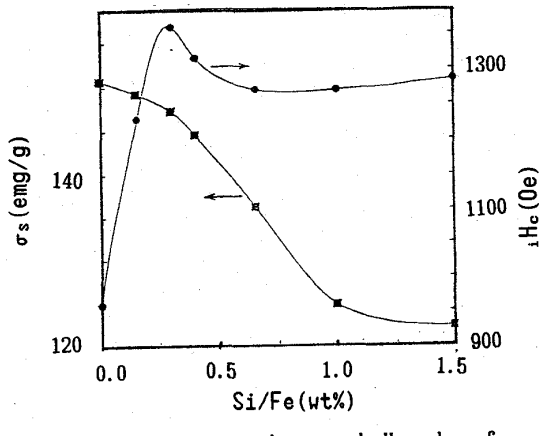

Fig. 3 Effect of Si ion on the σ_s and $_iH_c$ value of (SiO_2, Al_2O_3)-coated acicular α-Fe particle

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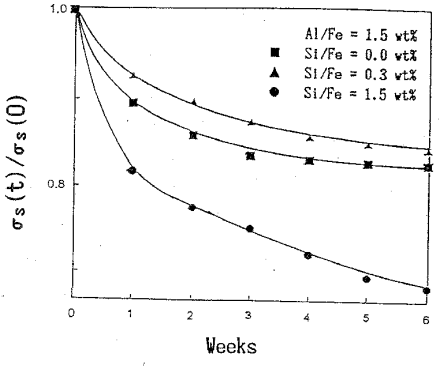

Fig. 4 Change of σ_s with time of (SiO_2, Al_2O_3)-coated acicular α-Fe particle

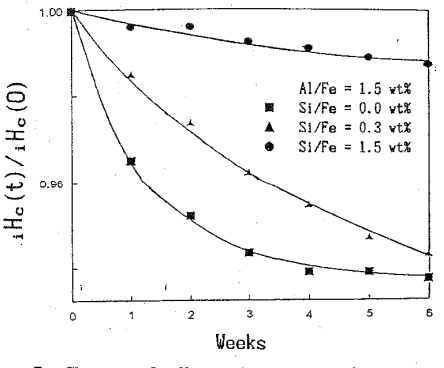

Fig. 5 Change of $_iH_c$ with time of (SiO_2, Al_2O_3)-coated acicular α-Fe particle

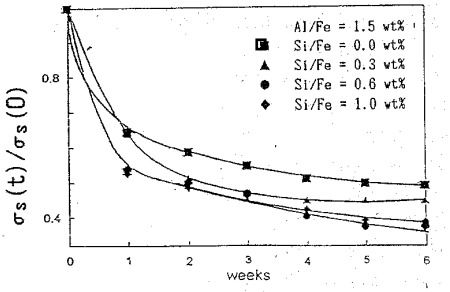

Fig. 6 Change of σ_s with time of (SiO_2, Al_2O_3)-coated α-Fe particle at 90% humidity and 60°C

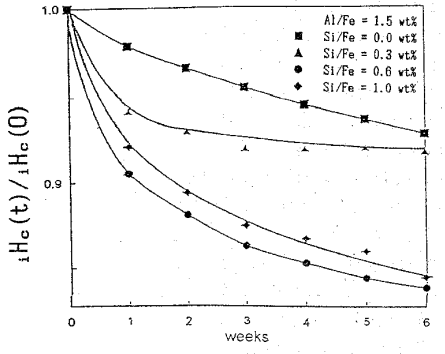

Fig. 7 Change of $_iH_c$ with time of (SiO_2, Al_2O_3)-coated α-Fe particle at 90% humidity and 60°C

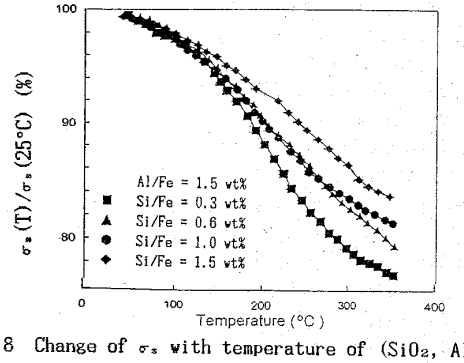

Fig. 8 Change of σ_s with temperature of (SiO_2, Al_2O_3)-coated α-Fe particle at elevated temperature

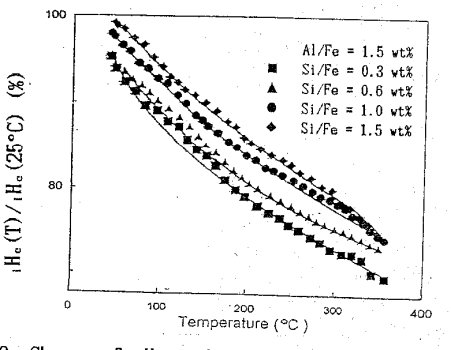

Fig. 9 Change of $_iH_c$ with temperature of (SiO_2, Al_2O_3)-coated α-Fe particle at elevated temperature