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The Oxidation and Magnetic Properties of MP Recording Media Particles

J. A. Hutchings and K. O'Grady

Abstract—MP recording media pigments have been subjected to elevated temperatures of 180°C in air at various rates of heating from room temperature. It has been found that the rate of heating of these particles determines the final oxide which the particles form. Analysis of the switching field distribution (SFD) of such particles before and after oxidation shows a broadening of the SFD to lower switching fields. A comparative analysis with X-ray spectra performed on the oxidized particles indicates that particles with lower values of coercivity have been oxidized preferentially.

Index Terms—Metal particles, oxidation, recording media.

I. INTRODUCTION

ACCICULAR MP recording media particles consist of a core of iron cobalt alloy of up to 30% cobalt surrounded by an oxide layer. Some systems are known to exhibit a wide SFD which has been attributed to crystallographic defects [1]. The aim of this work is to examine the correlation between the wide SFD and the oxidative stability of these particles. In particulate recording media, such a wide SFD causes problems of over-write arising from those particles having the largest switching fields in the distribution and low switching fields can lead to thermal loss of data and print through [2]. A wide SFD also leads intrinsically to the broadening of transitions. If the width of the SFD is characterized by the coercive squareness, S^* , then the transition width, a , is given by (1) in which H_C is the coercivity, M_r is the remanence [3]:

$$a = 2M_r \frac{\delta F(S^*)}{H_C}. \quad (1)$$

With the use of fundamental metal particles there is also the potential problem of oxidation which is minimized via the passivation of the particle surface by the use of coatings e.g. Si, Al and Y, and subsequent controlled oxidation [4], [5]. However, some susceptibility to oxidation remains and we associate this behavior with surface or bulk defects. Hence, in this paper we have attempted to correlate reversal effects and chemical stability.

II. EXPERIMENTAL

MP particles were analyzed to investigate their structural and magnetic properties. The morphology and particle size were analyzed by transmission electron microscopy with the particles being dispersed in an ultra-sonic bath in a mixture of ethanol and

TABLE I
CHEMICAL COMPOSITION AND PHYSICAL SIZE OF THE INVESTIGATED PARTICLES

(%wt of total Particle elements by ICP (± 0.1))			TEM Size (nm) L=Length W=Width
Co	Al	Y	
14.2	0.6	≈ 0.1 (trace amounts)	L=87 \pm 25 W=16 \pm 5

distilled water using oleic acid as a dispersant. The average particle size was determined by measuring the lengths and widths of 50 particles from a TEM micrograph to obtain an estimate of particle size. The physical parameters for these particles are given in Table I. Plasma spectroscopy (ICP) was used to obtain a chemical analysis of the particles.

Magnetic measurements on the particles were performed on using a PAR 2000 vibrating sample magnetometer in which the samples were packed in tubes of 5:1 axial ratio in order to reduce the demagnetizing factor to a value close to zero.

The particles were progressively oxidized by exposure to air at elevated temperatures of 180°C. In common with the work of Ohno *et al.* [6], we have found that the heating rate from ambient temperature affects the oxidation process. Accordingly, although the particles were being oxidized in a furnace, the actual temperature of the particles was monitored via a chromel-alumel thermocouple with a reference junction at 0°C.

III. RESULTS AND DISCUSSION

The chemical analysis results given in Table I show the particles to contain cobalt, aluminum and trace amounts of yttrium. The work of Ohno *et al.* [6] describes how a rapid rate of heating of a passivated particle may crack the oxide layer surrounding it and expose a fresh reactive iron surface to the air. This will result in a highly exothermic, self-sustaining reaction. A gentler rate of heating does not result in such a reaction and allows for a greater control over the process. The data in Fig. 1 shows that particles oxidized at 0.5°C/min show a spike in the temperature/time graph after 3.5 hours, ($T \approx 110^\circ\text{C}$) indicating a rapid oxidation or pyrophoric reaction. Those particles oxidized at 0.1°C/min show a rise to 180°C without rapid oxidation.

Fig. 2 shows the SFD for the original sample and those heated at the 0.5°C/min and 0.1°C/min. These SFDs were obtained from the differential of the isothermal remanent magnetization curves as described in the work of O'Grady [7] and show the irreversible magnetic fraction of the particles as a function of applied field. In the cases of both heated samples, it can be seen that the high field end of the SFD remains unaffected as compared with the original SFD, but with oxidation, the lower field

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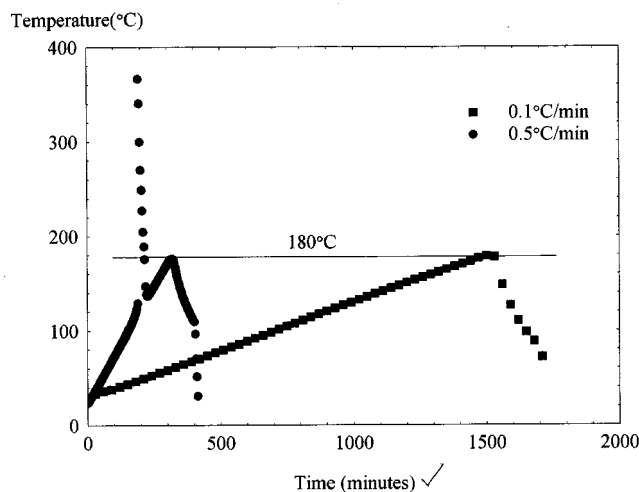


Fig. 1. Particle temperature with oxidation time.

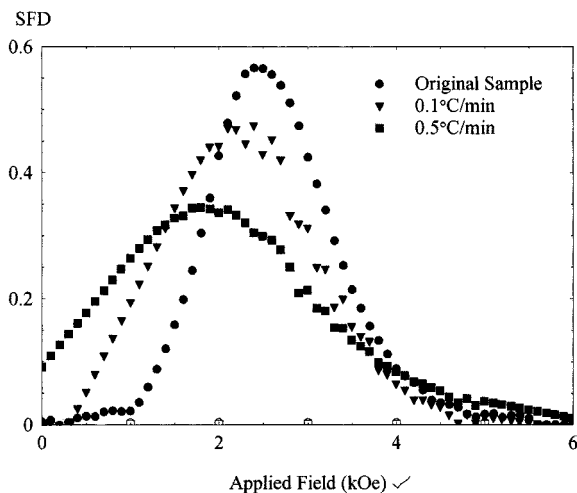


Fig. 2. SFD's of oxidized particles.

TABLE II
BULK MAGNETIC PROPERTIES OF OXIDIZED MP PARTICLES

Sample	M_s (emu/g)	Coercivity (kOe)	SFD FWHM (kOe)	M_r (emu/g)
Original Sample	100	1.96	1.58	50
0.1°C/min	43	1.58	2.06	21
0.5°C/min	30	0.85	2.75	11

end of the SFD moves to still lower fields. A summary of these measurements is shown in Table II.

From the results given in Table II it should be noted that the FWHM of the SFD increases with the rate of oxidation. The 0.1°C/min SFD width is much closer to the original SFD than the rapidly oxidized sample which also shows a paramagnetic fraction. Also, looking at the values for bulk magnetic measurements, it can be seen that the values for saturation magnetization and coercivity for the 0.5°C/min sample are in broad agreement with some of the samples of Chin [8] and Zhang [9] who performed magnetic measurements on various cobalt modified

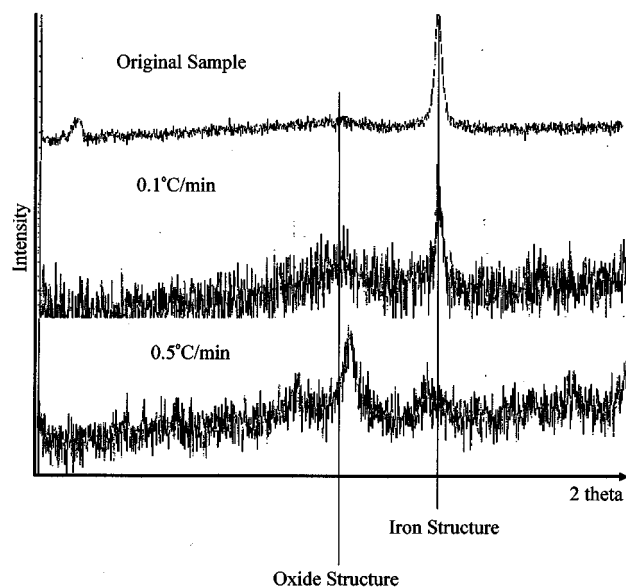


Fig. 3. X-ray diffraction spectra of oxidized particles.

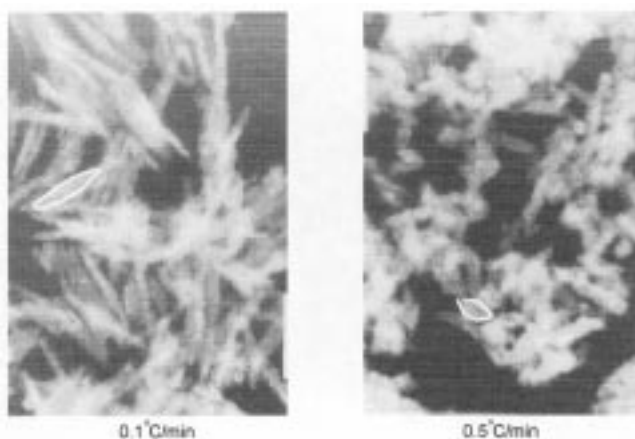


Fig. 4. Electron micrographs of oxidized particles.

magnetite particles, although the true nature of the oxide is not known.

In Fig. 3, it can be seen that for the original unheated samples the X-ray spectra show a strong peak corresponding to iron, with a small feature at around $2\theta = 40^\circ$ that corresponds to the passivating oxide layer. For the 0.1°C/min heated sample, the X-ray spectrum shows this feature to be more pronounced indicating an increase of iron oxide presumably on the particles surface, although the iron feature is still dominant. For the 0.5°C/min sample the oxide line is dominant although some evidence of elemental iron remains. Also, the oxide line is now indicative of a magnetite like structure.

TEM was performed on the oxidized particles and the micrographs can be seen in Fig. 4. The 0.1°C/min samples have the same acicular shape as the original, whilst the 0.5°C/min samples are vastly different in shape. A particle for each sample has been highlighted on the micrograph to illustrate this point.

Hence it is clear that the SFD for both oxidized samples is broader due to changes in those particles having lower coercivities.

Comparing the results for the 2 heated samples, they would suggest that as the high coercive force end of the SFD remains anchored, some particles are more resistant to oxidation than others. For the case of the slowly oxidized particles, the physical analysis shows that the particles are still iron cored, thus the high coercive force end of the distribution is made up of unoxidized particles, whilst the lower end of the SFD is composed of particles that have experienced oxidation. A possible explanation for this may be found in the work of Morales [1] reporting on the work of Okamoto [5]. A particle with a smooth surface will have a higher coercivity as it has less surface/bulk defect sites that will lead to magnetization reversal by such mechanisms as weakening of the exchange coupling. Hence, lower coercive force particles may find an easier route to oxidation than smoother surfaced higher coercive force particles, hence the form of the SFD's. For the case of the rapidly oxidized particles, it is interesting to note that a high coercivity end of the distribution does survive. This may be the case that during the pyrophoric reaction, not all of the sample experienced the reaction and ignited, although at such temperatures generated in the sample, this result would again suggest some particles more resistant to oxidation.

It is hoped to be able to use this phenomenon of oxidation of lower coercive force particles to be able to separate out the various fractions within a distribution.

If such particles oxidize to a magnetic iron oxide, then for a given particle size, an oxidized particle will have a saturation

magnetization roughly half that of an un-oxidized particle. Hence, future work hopes to use a magnetic filter system to separate out such oxidized particles while separated in a dilute dispersion.

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