

**SEM AND MAGNETIC CHARACTERIZATION OF HEXAGONAL PLATE-LIKE IRON OXIDE PARTICLES.** J. R. Correa(1), D. Canetti(1), J. C Llopiz(1), J. Dufour(2). (1) Facultad de Química, Universidad de La Habana, Zapata y G. CP 10400, Havana, Cuba; (2) Escuela Superior de Ciencias Experimentales y Tecnología, Universidad Rey Juan Carlos, 28933 Madrid, Spain. Email: correa@fq.uh.cu.

Magnetite ( $\text{Fe}_3\text{O}_4$ ) and maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) are the more commonly iron oxides used as magnetic recording materials [1]. Wet methods are used to synthesize an oxyhydroxide precursor for the purpose of yielding magnetite and maghemite particles with desirable form. Knowledge of synthesis particular conditions is of great importance since the properties of iron oxides basically depend on the accuracy of chemical composition, crystallite size, morphology, and in general these factors determine the preparation method [2]. Control of crystallinity, shape homogeneity, phase purity and presence of additives enables the change in electric and magnetic properties of iron oxides [3, 4]. Several works were done concerning the influence of the synthesis conditions on the physical properties of magnetite and maghemite [5, 6]. The purposes of present investigation are: i) to synthesize a cobalt-substituted iron oxide precursor with plate-like morphology (hexagonal), ii) to study the decomposition, reduction/oxidation and characterization of the final products (magnetite and/or maghemite) derived from the iron oxide (P0-900; P90/120-900) and from the cobalt substituted (P90/120-900C2) by means of solid state analytical techniques like Scanning Electron Microscopy, X-ray Diffraction, IR Spectroscopy and Magnetometry, and iii) to correlate the crystallinity of the iron oxide precursor and additive presence with the physical properties of the final product. For pure iron and cobalt modified oxides with plate-like morphology, the alkaline treatment result in an increase in crystallinity of the former oxyhydroxide and also induces phase transformations.

At selected temperatures the favoured phase was hematite instead of goethite. This indicates that internal rearrangement mechanism takes place instead of redissolution/precipitation [7]. The micrographs obtained by Scanning Electron Microscopy technique of selected samples are shown in figures 1-3, thus for untreated sample in alkaline medium, particles are smaller than when samples are kept in KOH during 120 h. Particle size, measured by this technique, is 104 nm to P0-900 and 195 nm to P90/120-900. Small particles linked to the big ones are observed in specimen P90/120-900C2 and particle size analysis shows a bimodal system in which the mean geometrical correspond to 85 and 339 nm respectively. In all cases distribution is log-normal and the particles shape reveals more or less hexagonal plates. Magnetic measure of untreated oxidized sample, without additive is shown in figure 4. Coercivity increases in the following order: untreated oxide (498 Oe) - untreated oxide plus 5% of Co + B (779 Oe) - treated oxide in KOH 30 h plus 5% of Co + B (1490 Oe). Upon increasing alkaline treatment time and calcination temperature of iron oxide precursor with plate-like morphology, increases the coercivity value of the final product, which appears between the expected limits for its application in magnetic recording materials. Although the presence of cobalt at the particle surface yields in increase of the coercivity of the magnetite, it delays as well the reduction time to magnetite.

## References

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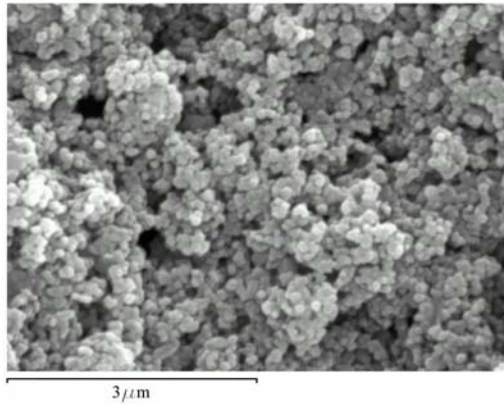


Fig. 1.- SEM micrograph of oxide precursor P0-900 (untreated in KOH and calcined to 900 °C).

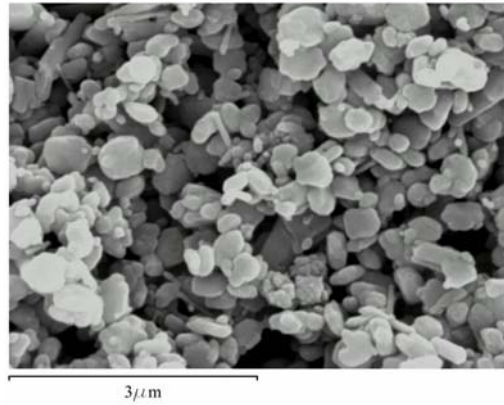


Fig. 2.- SEM micrograph of oxide precursor P90/120-900 (treated 120 h in KOH at 90°C and calcined to 900 °C).

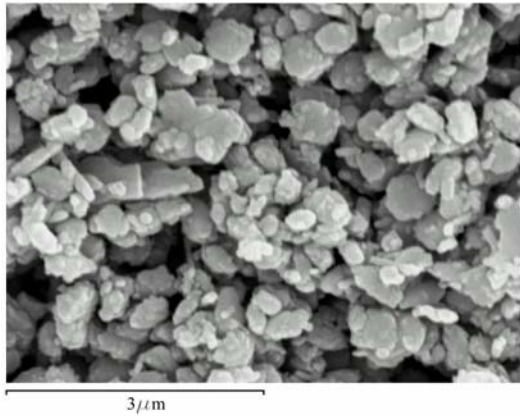


Fig. 3.- SEM micrograph of oxide precursor P90/120-900 C2. (treated 120 h in KOH at 90°C and calcined to 900 °C with 5 % Co/Fe).

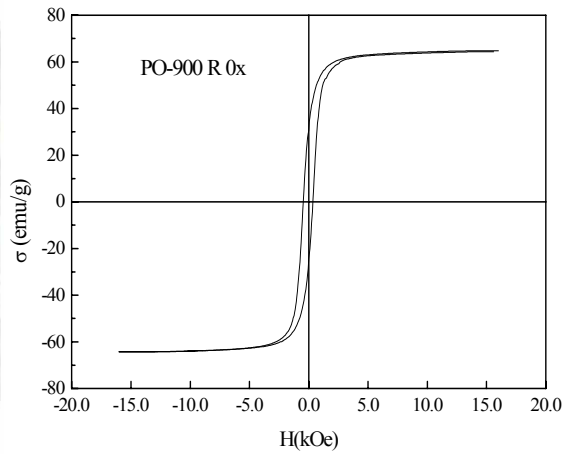


Fig. 4.- Hysteresis loop of untreated precursor calcined to 900 °C, reduced and re-oxidized.