High Remanance of Strontium Hexaferrite from Millscale Derived

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ABSTRACT

Strontium hexaferrite was prepared by using millscale as raw materials. Millscale is an impure iron oxide. The millscale was milled to obtain < 20μm size powder particle in diameter and the oxide powder was separated into magnetic and non-magnetic particle in a 1 tesla external field. Magnetic particles such as magnetite (Fe_3O_4) were separate and wuestite (FeO) was oxidised into hematite (Fe_2O_3) at 500°C for 10 hours in air. The conventional sintering method was used for preparing green powder of strontium ferrite with composition $SrO.6Fe_2O_3$. This process was done by utilising dry method in the mixing of Fe_2O_3 and $SrCO_3$. The final sample was in pallet shape with 10.84 mm diameter width and 11.39mm tickness. The sample was magnetized, followed by measurement of remanance, coercive force and $(BH)_{maks}$ of strontium ferrite permanent magnet. Resistivity, curie temperature and density of sample were also measured. The results showed that mill scale has the potential to be a raw material for a high quality permanent magnet material in the future.

Keywords: *Millscale, Remanance, Hematite*

Introduction

Ferrites are mixed with metal oxides which contain iron oxide as its main component. It is hard, polycrystalline, generally grey or black in colour and classified as ferrimagnetisms (Snelling 1988). There are three important classes of commercial ferrites. They are: soft ferrites with the cubic spinel structure, soft ferrites with garnet structure and hard ferrites with the magnetoplumbite (hexagonal) structure.

Hexagonal ferrite $MFe_{12}O_{19}$ is amongst the most widely used materials for permanent hard magnet applications (Kojima 1982, Cantrell 1992). Commercial ferrites are typically prepared by multi step ceramic synthesis that involves mixing, grinding and firing of the constituent oxides and carbonate at temperature above 1300°C. This hard ferrite product is used in various industries like consumerism and military. They are used in small motors, amplifiers, microphones and telephones.

Strontium ferrite magnets have been the primary ferrite materials in the commercial product since about 1980. It was found that the magnetisation and anisotropic constant of Strontium ferrite was greater than barium ferrite and the Curie temperature was slightly higher (Mitsuo 1999).

Millscale is the waste product thrown away from the final task of fabrication and manufacturing of steel. The main source of this project is millscale from the Southern Steel Sdn Bhd. There are approximately 80 000 tons of millscale thrown away annually in Malaysia (Kittel 1949).

Experimental Procedure

Three major steps were involved in the sample preparation: powder preparation, compact formation and sintering. The millscale were crushed in the steel pot with small steel balls to obtain micro-sized powder particle. The steel pot was then put on rotating rollers for 20 hours. The crushed powder was then separated into magnetic and non-magnetic portion using Curie temperature technique to get wuestite (FeO). The wuestite was then oxidised in the electric furnace at temperature of 500°C in air for the period of 10 hours to form hematite (Fe_2O_3). Related chemical reactions:

$$2FeO + \tfrac{1}{2}O_2 \longrightarrow Fe_2O_3 \qquad [1]$$

Hematites from the millscale were poured with strontium carbonate in the steel pot with steel ball. The milling time was 20 hours at room temperature. The sample was then presintered at the temperature of 1200°C for a period 10 hours in air. Subsequently, the powder was crushed using milling machine for 20 hours and then was sieved to achieve single domain particles. Granulation was carried out by adding of polyvinyl alcohol (PVA) and addition of zinc stearate as the lubricant. The granulated powder was moulded into pallet shaped by pressing it with a force of 60 kN and was sintered in air at 1250C for 10 hours. The pallet shape sample was then magnetised.

Sample density was measured by using Archimedes method:

$$\text{Density} = (W_1 / W_2)\, \rho_{water} \quad [2]$$

Water density (ρ_{water}) equals to 1 g/cm^{-3}.
W_1 is the weight of sample in air
W_2 is the weight sample in water
Hysteresis parameters tested were remanence B_r, coercive force H_C, energy product $(BH)_{max}$, saturation Magnetisation M_s

There are 2 theories that were used in this research:

1. B-H without demagnetised factor. (In infinite size)
   $$B_{net} = B_{app} + 4\pi M_s \quad [3]$$
2. B-H in material with demagnetised factor.
   $$B_{net} = B_{app} + (4\pi - D) M_s; \quad [4]$$
   Where D is demagnetised Factor
   Dimensional Ratio = (thickness / diameter)

Results and Discussion

Figure 1 shows that the sample was identified as a single-phase Hematite, Fe_2O_3. From the comparison made, all the peaks are the same with the industrial standard.

Sample density was measured by using Archimedes method. The density of the sample was 4.73 g/cm^{-3} that is lower than the density of the calculated theory (5.08 g/cm^{-3}). This is because the hematites from millscale have lower purity compared to pure hematite Fe_2O_3 (Eric 1999).

There were pores that resulted in low density sample. Some of the pores were formed due to the existence of impurity such as PVA and Zinc Stearate. These impurities vaporised during sintering leaving behind pores. In addition, the pores may have been caused by improper pressure applied during the pressing.

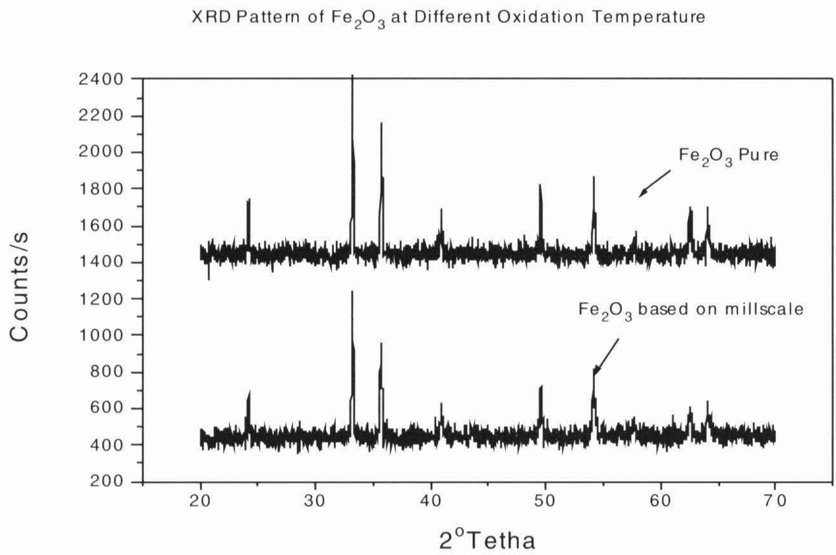

Fig. 1: X-Ray Diffraction (Arbitrary Unit) for Hematite (Fe_2O_3) Oxidized at 500°C.

Remanence of the sample strontium ferrites was higher than that of the one prepared by Eric Tan (ibid) and it showed that parallel domain alignment is not easily affected (Table 1). For the coercive force of the sample, it showed that the sample was not easy to be demagnetised because the coercive force is a measurement of the required field to destroy parallel domain alignment. Figure 2 shows the hysteresis loop for the sample.

Table 1: Comparison of Hysteresis Parameter with Strontium Ferrite Based on Pure Sample

Sample	B_r (kG)	H_C (kOe)	$(BH)_{max}$ (MGOe)	M_S (kOe)
Mill scale	3.5	1.55	1.6	0.2
Sample pure (Eric 1999)	1.41	0.4022	0.17891	0.33

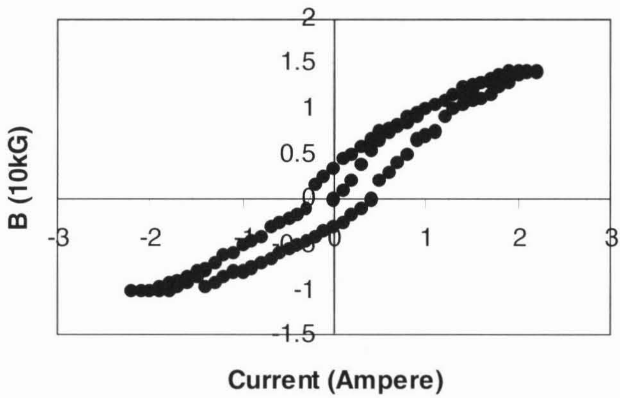

Fig. 2: Graph Bnet versus Current (Ampere)

Conclusion

The millscale is very cheap and easily obtained. It could be concluded that the low cost purification technique yielded highly purified wuestite. The FeO conversion to very pure hematite, confirmed by the XRD phase pattern and SEM observation resulted in high induction remanence. The effect of prolonged milling time, 20 hours, is speculated to be significant contributor to the formation of needle shape crystallinites.

The sample consisted of micro-hexagonal particles of around 1-2μm in diameters and a very narrow size distribution was observed. Magnetic properties of this sample show saturation magnetised of around 20 emu/g (2000 Oe) and a coercivity of 1500 Oe leads to single domain behaviours of the particles.

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