

Magnetic Characterisation Using Approximation Method (APM) of $\text{BaFe}_{12}\text{O}_{19}$ Derived from Millscale

R. S. Azis
M. Hashim
N. Yahya
H. Azhan
R. Alias
N. M. Saiden
N. A. Aini
A. A. Rejab

ABSTRACT

This paper describes a new method for measuring the magnetic properties of hexagonal Barium ferrite using approximation method (APM) theory. The sample was prepared by recycling the waste steel product from Malaysian steel factories. Using a Curie temperature separation technique, the wustite, FeO contained in the millscale was separated by this new technique using de-ionized water at 90°C/100°C in the presence of 1T external field. The wustite was then oxidized in the air at 400°C/500°C/600°C for 10 hours. An XRD phase analysis showed that a very high percentage of Fe_2O_3 was present in the final powder preparation. A conventional processing method was then done to prepare hexagonal $\text{BaFe}_{12}\text{O}_{19}$ pallet shaped samples. Analysis of samples was done on grain size, saturation magnetization, coercive force and remanence. The magnetic properties were measured using approximation method (APM) theory. The high remanence 2000Oe, 2000Oe saturation magnetisation and 1500Oe coercive force of sample derived from millscale shows that this technique used has a high percentage for magnetic properties measurement in the future.

Keywords: *Magnetic, Approximation Method, Millscale*

Introduction

The millscale was a steel waste product from steel factories. It is estimated that there are approximately 80 000 tons of millscale thrown away annually in Malaysia (Eric 1999). Millscale is an impure ferum oxide, a low cost material that can be obtained at steel factories in Malaysia. This immense amount of millscale per annum present to us a challenge to purify the polycrystalline wustite, FeO; it contains to a conversion stoichiometric hematite, Fe₂O₃. Hematite (Fe₂O₃), an important ore of iron, is the main raw material needed for fabrication of ferrites. The purified raw materials almost affects decidedly in the final quality of a processed material (Bando et. al. 1971).

Hexagonal barium ferrite is widely used as permanent magnet due to its excellent magnetic properties such as high Curie temperatures, magnetic anisotropy and coercivity (Kojima 1982). These materials offer many advantages in terms of high-density recording and chemical stability (Benito G. et. al 2001). The typical method to obtain ferromagnetic hexagonal oxide particles in general is the solid-state reaction. This method consists in heating the mixtures of precursors at temperatures as high as 1000°C giving rise to particles larger than 1mm. In order to reduce the particle size, other methods have been investigated such as aerosol pyrolysis (Gonzalez 2000), crystal-glass (Kubu et. al 1982), sol-gel (Shrik et. al. 1970), chemical co-precipitation (Fujiwara 1985), etc. However, these methods are more complex and more expensive than the ceramic methodology. Therefore, the aim of this paper is to develop an improved method to synthesize barium hexaferrite micro-particles based on the solid state reaction with prolonged milling time conventional method derived Fe₂O₃ from millscale.

The approximation method (APM) was introduced by the demagnetization factor observation. The macroscopic field of the polarization are contributed by the equation:

$$E = E_o + E_1$$
$$E = (E_{app} - NM_s) + 4p M_s$$

Assuming that $E_{eff} = E_a$ as the approximation method technique approached, where E^{eff} is the field effective, E_a is the field in air, E_o is the applied field, E_1 is the field due to the uniform depolarization, N is demagnetization factor and M_s is saturation magnetization. The field E_1

is called the depolarization field, within the body it tends to oppose the applied field E_0 as in the Figure 1.

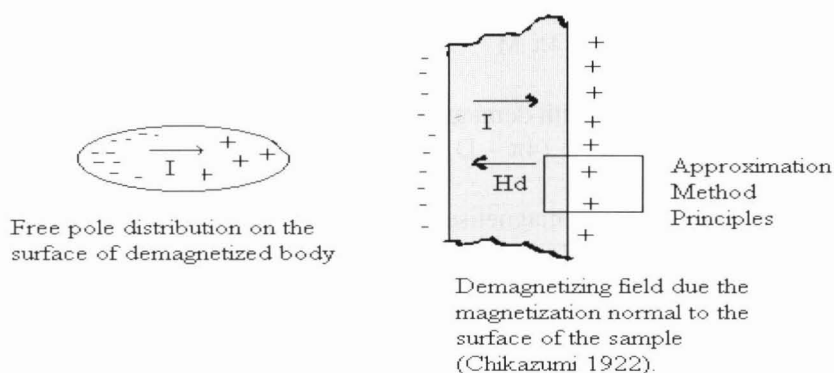


Fig. 1: The figure shows the physics principles of APM.

Each measurement is based on the Bozorth data for demagnetizing factors for sample-magnetized parallel to the long axis of the sample.

Methodology and Characterization

The APM was designed to measure the magnetic properties of sample. The sample was prepared by the conventional method of preparing hard magnetic material. The millscale was crushed for 20 hours to obtain the prescribed size of powder. The crushed powder was then separated into magnetic and non-magnetic portion using Curie temperature technique. Wustite was then oxidised in the electric furnace at temperature of 400°C/ 500°C/600°C in the air for 10 hours. A conventional processing method was then used to fabricate pallet shaped $BaFe_{12}O_{19}$ samples utilizing from the Fe_2O_3 derived from millscale. X-Ray measurements were carried out in Siemen D5000 machine using Cua radiation, with $\lambda = 1.54056 \text{ \AA}$. The scanning speed of the counter is 2q per min.

The magnetic measurement was carried out after each sample was placed in the approximation method (APM) technique design. The sample was then connected to the probe and Walker Analog Magnetometer as shown in a schematic diagram in Figure 2. A series of applied magnetic field (B_{apply}) from the lowest current to the saturation magnetization was achieved. The relative remanence and saturation magnetization were

calculated by introducing the theory of approximation method shown as below:

B-H without demagnetised factor. (In infinite size)

$$B_{net} = B_{app} + 4\pi M_s \quad [1]$$

B-H in material with demagnetised factor.

$$B_{net} = B_{app} + (4\pi - D) M_s; \quad [2]$$

Where D is demagnetised Factor

Dimensional Ratio = (thickness / diameter)

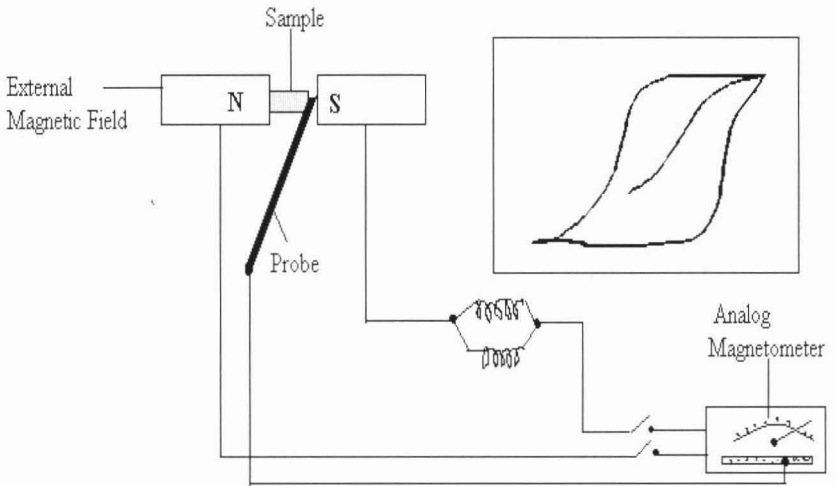


Fig. 2 : A Schematic Diagram of the Approximation Method (APM) Designed for Measuring Magnetic Properties of the Sample

Results and Discussion

Figure 3 shows the XRD phase analysis results for the Southern Steel samples after oxidation in air 400°C/500°C/600°C. Broad and clear diffraction lines begin to appear for powders oxidized at 400°C, which corresponded to progression of crystal growth of the entire particles. Sharp and clear diffraction lines pattern can be observed for powders oxidized at 600°C. The maximum duration to draft a sharp peak and patterns is 10 hours oxidation in air.

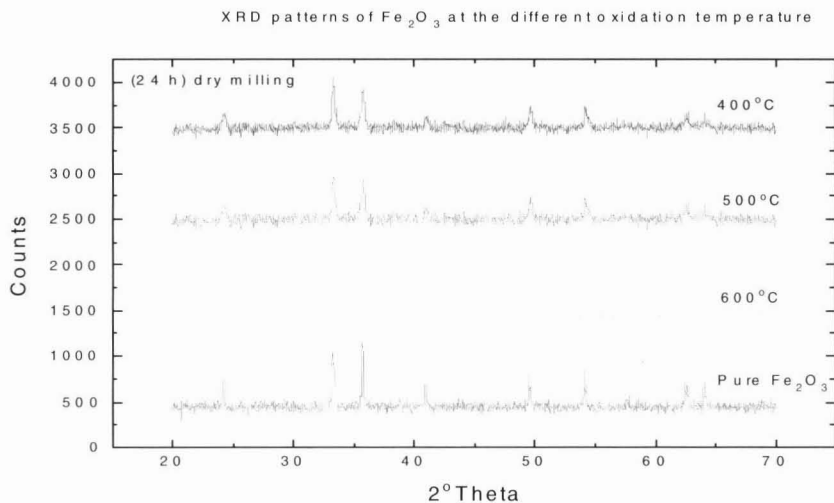


Fig. 3: XRD Phase for Fe_2O_3 derived from Millscale and Pure Fe_2O_3 Powder

Figure 4 also indicates the sharp peak from the x-ray observation of barium hexaferrite at different type of sintering temperatures. X-ray diffraction structural investigations (Fig. 3 and Fig. 4) demonstrate the same phase content of powders, milled at 20 hours. The increasing milling times described peaks was diffractogram and indicated the hexagonal $\text{BaFe}_{12}\text{O}_{19}$ formation.

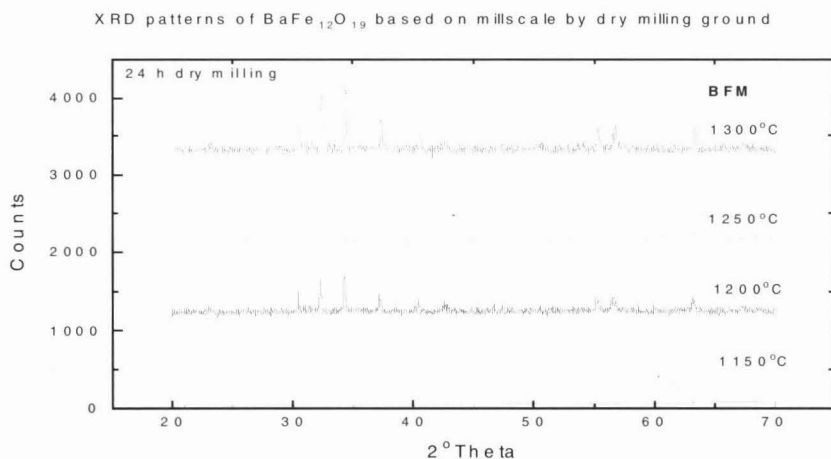


Fig. 4: XRD Phase for $\text{BaFe}_{12}\text{O}_{19}$ Derived from Millscale at Difference Sintering Temperatures

Magnetic behaviour of barium hexaferrite derived from millscale is presented in Figure 5 and Figure 6. The microstructures observed are displayed in Figure 7. Hysterisis loop samples BFM1150, BFM1200 and BFM1250 showed significant differences in the saturation magnetisation (M_s) and coercivity values (H_c). The maximum M_s value is 2000Oe for samples, which is lower than the theoretical one calculated for single crystal of barium hexaferrite particles, i.e. 72 emu/g (5.729 kOe) (Shrik and Buessen 1969). This can be explained partially by the lack of full

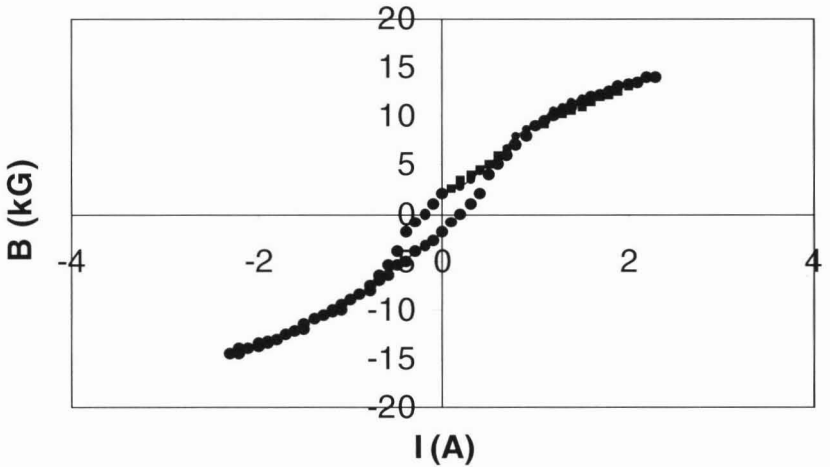


Fig. 5: Hysterisis Loop BFM1150

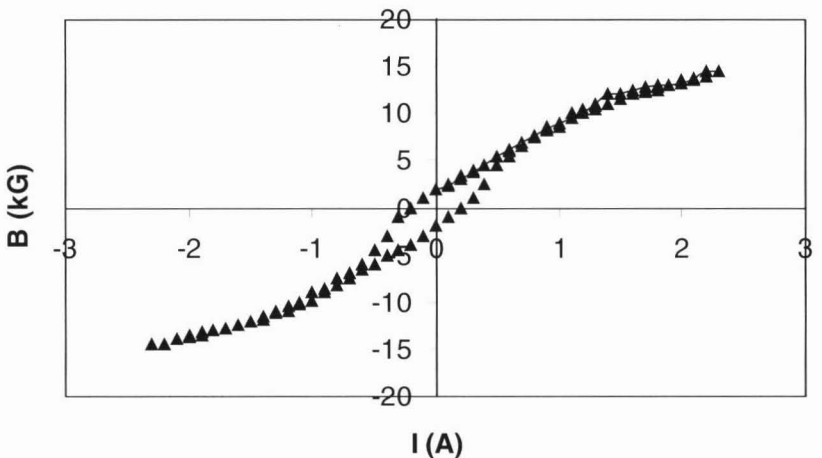
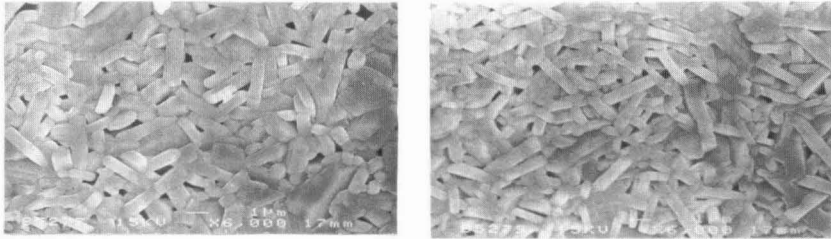


Fig. 6: Hysterisis Loop BFM1250



(A)

(B)

Fig. 7: Microstructure of $\text{BaFe}_{12}\text{O}_{19}$ Derived from Millscale at Different Sintering Temperatures: (A) BFM1150 (B) BFM 1250

saturation reached at the maximum applied field during the APM measurement. Anyway, the smallness in size of the particles can also contribute to that reduced value. Several theories, including the surface effects, spin cating and sample inhomogeneity, have been proposed to account for the relatively low magnetization in fine particles (Shafi et. al. 1997). Thus, particles of similar size prepared by different methods have been reported to give rise to lower or similar M_s value (Haneda et. al. 1974, Rezlescu et. al. 1999), even when higher temperatures have been used in synthesis. Even though, the 1500Oe coercive force, 2000Oe saturation magnetisation and 2000Oe of remanence is quite enough for magnetic properties barium hexaferrite derived from the waste steel product, millscale.

Conclusion

It could be concluded that the low-cost purification technique yielded highly purified Fe_2O_3 successfully to achieve a high saturation magnetization (15 000G), 2000G remanence and 1500Oe coercive force by the APM approach as indicated by XRD peaks phase and hysteresis loop observation. This technique has a high potential for improving the magnetic properties measurement design for the future.

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R. S. AZIS, M. HASHIM, N. YAHYA, R. ALIAS, N. M. SAIDEN, N. A. AINI, & A. A. REJAB, Faculty of Science and Environmental Studies, Universiti Putra Malaysia, Serdang

H. AZHAN, Faculty of Science, Universiti Teknologi MARA, Pahang. azhan@pahang.uitm.edu.