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STRUCTURAL, MORPHOLOGICAL AND MAGNETIC STUDIES OF α-Fe_2O_3 MILLED POWDERS

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Abstract:*Magnetic nanoparticles exhibit unique nanoscale properties and their utilization for various magnetic systems is of significant interest. Hematite (α- Fe_2O_3) is the most stable iron oxide used for catalysts, electrodes, gas sensors, and photo catalytic and anticorrosion protective paints. In the present experimental work α-Fe_2O_3 Nanoparticles were synthesized by ball milling process of α-Fe_2O_3 and milled for different time intervals and were annealed at 1000^o C for 3 hours. The samples were characterised by X-ray diffraction, Scanning Electron Microscopy and Vibrating Sample Magnetometer. The structural analysis revealed rhombohedral structure of the samples. The SEM micrographs of as cast and annealed sample revealed plate like structure of particles having variuos sizes ranging from 19 µm to 146 µm. The magnetic studies of the samples showed a linear relationship between grain size and coercivity indicating a strong ferromagnetic behavior of the samples.*

1. Introduction

Magnetic nanoparticles have attracted many researchers due to their unique nanoscale properties and their utilization for various magnetic systems is of significant interest. Hematite (α- Fe_2O_3) is the most stable iron oxide of low cost, biocompatible and the most environment friendly nature. It finds applications in catalysis, electrodes, gas sensors, anticorrosion protective paints and in optical and electro magnetic devices. α- Fe_2O_3 undergoes a transition from weakly ferromagnetic to anti ferromagnetic below room temperature as the Morin transition. But this transition temperature depends on the particle size. Similarly the other magnetic properties depend on particle morphology, crystallite size. The uniform physical and chemical properties of magnetite nanoparticles greatly depend upon the synthesis route. Researchers have employed different techniques such as sol-gel processes, micro emulsion, combustion, solvothermal, hydrothermal, solvent evaporation etc. to prepare homogenous nano particles of iron oxide. However, these methods usually involve special equipment, high temperatures and the tedious removal of impurities. The synthesis of magnetite nanoparticles with controlled size using ball milling technique is adopted in the present work.

2. Experimental Work:

Commercially purchased α- Fe_2O_3 (Ferric oxide, red) powders of purity 98% was used as starting material for the present experimental work. The milling process of the samples was performed using high energy planetary ball mill of Model type VBCC/PM/15-11/12. Before milling process the grinding medium and the Yttria stabilized Zirconia balls were cleaned with acetone twice. α- Fe_2O_3 powders was taken in the grinding medium and ball to powder ratio was kept as 10:1 and was placed in a high energy ball mill. The sample was milled at the rotation speed of 300 rpm. The sample was milled for 25 hours and a portion of the sample was collected after 5 hours, 15 hours and 25 hours. The milling was stopped and the vial was kept open for every one hour during milling in order to avoid raise of temperature in the vial.

The milled α- Fe_2O_3 samples were annealed to reduce stress and strain in the samples. Before annealing, the time and temperature slots of the muffle furnace were set up using microcontroller of the furnace. The temperature was set as 1000^0C and the time to reach the temperature was set as 3 hours and furnace was maintained at the same temperature for 3 hours.

3. Characterisation techniques:

X-ray diffraction is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of materials. The structure of the samples were analysed by means of X-ray diffraction technique using Cu-Kα radiation of wavelength 1.5418 A^0 with 2θ ranges from 20^0 - 90^0. Crystallite size was determined

using Debye- Scherrer's formula. The Morphological studies were performed using Scanning Electron Microscope(SEM). The magnetic studies of the samples at room temperature were performed using VSM.

4. Results and Discussion:

4.1 Structural Analysis

The X-ray diffraction (XRD) patterns were used to analyse the crystallite nature and to identify the planes present in the milled α-Fe_2O_3 powders and milled annealed samples. Fig 1, Fig 2 and Fig 3 shows the XRD pattern for the samples milled for 0 hrs, 5 hrs and 25 hrs respectively. The predominant peaks observed at (1 0 4), (1 1 0) and (1 1 6) had been indexed to be rhombohedral structure of α-Fe_2O_3 and that are in good agreement with the JCPDS data (89-8103)[1]. Due to milling, no phase change was observed in the XRD patterns. But after milling, the relative intensity of the peaks were found to decrease. The diffraction peaks were sharp for 25 hrs milled sample which indicates that the sample has crystallite nature [2]. The slight broadening of diffraction peaks for milled samples can be attributed to the reduction of crystallite size and the introduction of strain in the sample after milling.

The XRD diffraction patterns of the as cast, 15 hrs and 25 hrs milled and annealed samples were shown in the Fig 4, Fig 5 and Fig 6 respectively. The characteristic diffraction pattern of all the samples show peaks of the planes (1 0 4), (0 2 4), (1 1 0), (1 1 6), (1 2 5), and (3 0 0) that were in good arrangement with the JCPDS data (89-8103) of standard XRD peaks of ferric oxide with rhombohedral structure.

The narrow and sharp peaks of XRD patterns of all annealed samples indicate the crystallite nature of the samples and there is no phase change due to milling or annealing. With increasing milling time, the diffraction peaks become broader and their relative intensity decreases, which may be to the reduction of the particle size[3].

The calculated values of lattice constants, grain size and strain were shown in Table 1. The reduction in the grain size was observed for 15 hrs milled sample that may be due to the fracturing of the material produced due to milling. But increase of grain size was observed after 25 hrs of milling. The increase of the grain size may be to due to agglomeration of fine particles [4].

There is an increase in the lattice parameters with increasing milling time that may be due to the internal strain induced by ball milling process[1] . All of these are in good agreement with the previously reported results. Strain calculated for the (1 0 4) plane shows that a slight decrease in the value of strain which indicates relieve of strain due to annealing.

4.2 Morphological Studies

The SEM micrographs for as cast annealed α-Fe_2O_3 sample was shown in figure 7 and it shows plate like structure. The image shows the presence of particles of various sizes that ranges from 19 µm to 146 µm. The SEM micrographs for 25 hours milled and annealed α-Fe_2O_3 sample shown in figure 8 indicates the presence of particles of various sizes with size ranges from 73 µm to 150 µm.

The SEM images clearly indicates the increase of particle size after milling and the XRD results indicates the increase of grain size after 25 hours of milling that may be to due to aggregate of nano crystalline sub particles at high annealing temperature [4].

4.3 Magnetic Studies

The magnetic analysis of the samples were performed at room temperature using VSM and the magnetic curves of the samples were shown in the fig 9 and fig 10 and the magnetic parameters were shown in table 3. High magnetic coercivity of the samples shows a strong ferromagnetic behavior of the samples[4]. Even after annealing at high temperature the hysteresis loop did not reach magnetic saturation and there was a linear proportionality between the coercivity and grain size of α-Fe_2O_3.

Conclusion

α-Fe_2O_3 powder was milled for different time intervals in the high energy planetary ball mill and the samples were annealed at 1000^0C in the muffle furnace. The micro structure of the samples analysed by XRD technique reveal rhombohedral structure and was confirmed by JCPDS data. No phase change was observed and the strain of the samples was found to increase due to milling. The reduction in the strain was observed due to annealimng. Grain size of the milled and then annealed sample was found to decrease when compared to as cast annealed sample. But the increse of grain size was noted with prolonged milling for 25 hours. The SEM micrograph of as cast and annealed sample showed plate like structure with particles of variuos sizes. The particle size were found to increase for the milled and annealed samples. The magnetic studies of the samples showed a linear relationship between grain size and coercivity. The high coercivity of the milled annealed sample shows the ferro magnetic nature of the samples.

Acknowledgement

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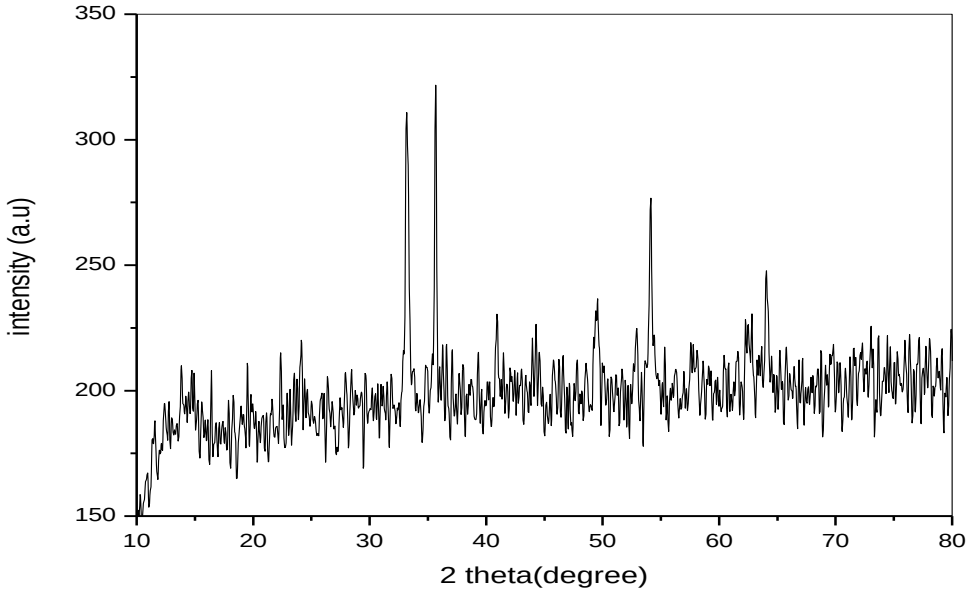

Fig 1. X-Ray Diffractogram of α-Fe_2O_3 Powders

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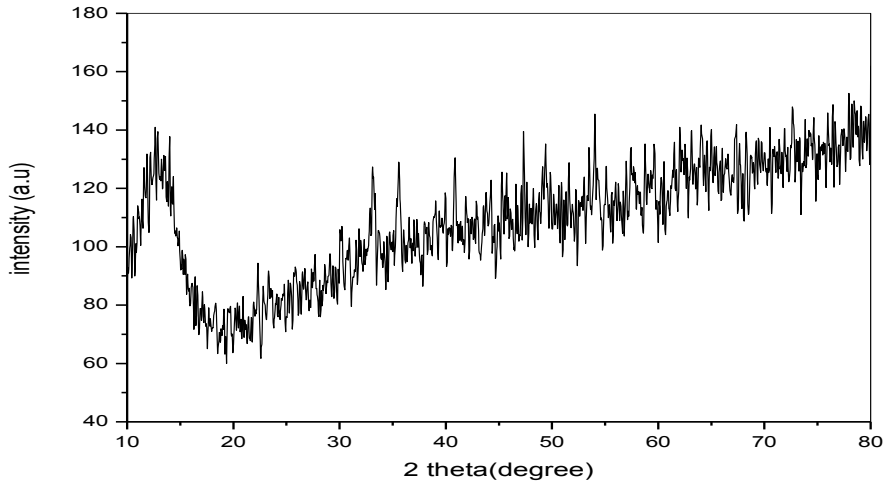

Fig 2 X- Ray Diffractogram of 5 hours milled α-Fe_2O_3 Powders

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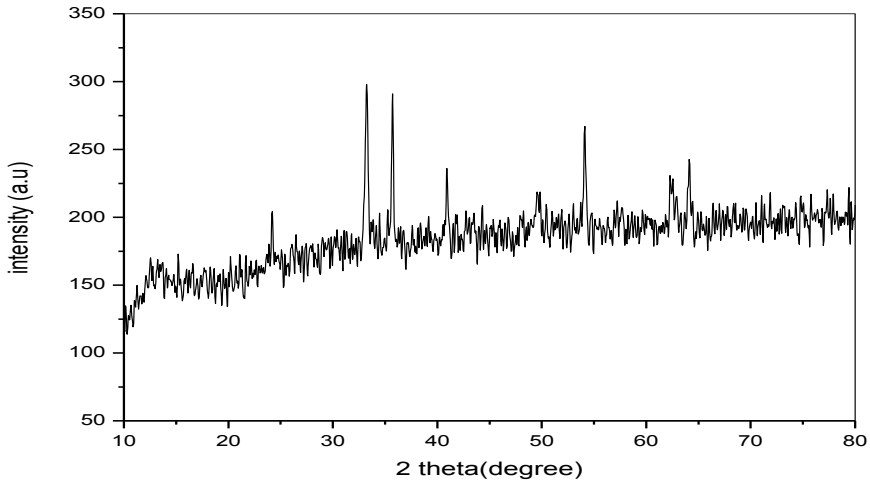

Fig 3 X- Ray Diffractogram of 25 hours milled α-Fe_2O_3 Powders

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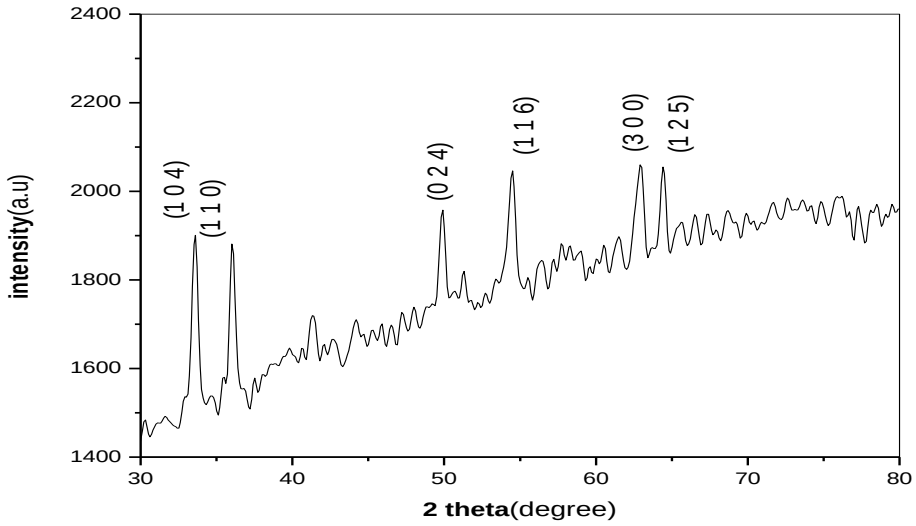

Fig 4 X-Ray Diffractogram of α-Fe_2O_3 annealed powders

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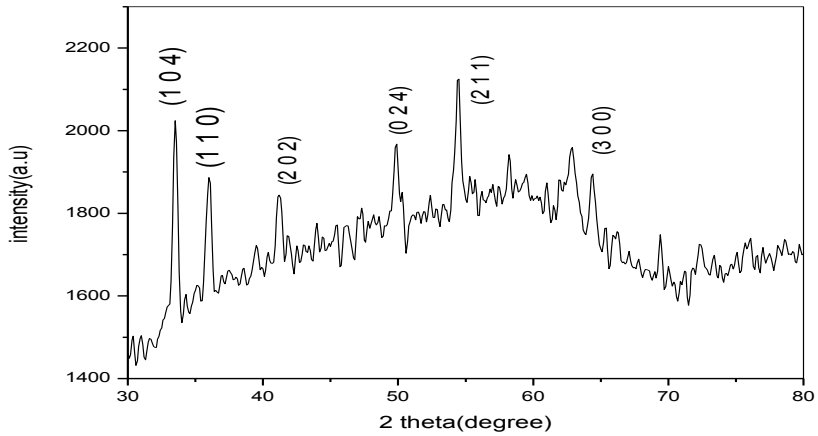

Fig 5 X- ray Diffractogram of 15 hours milled and annealed α-Fe_2O_3 powders

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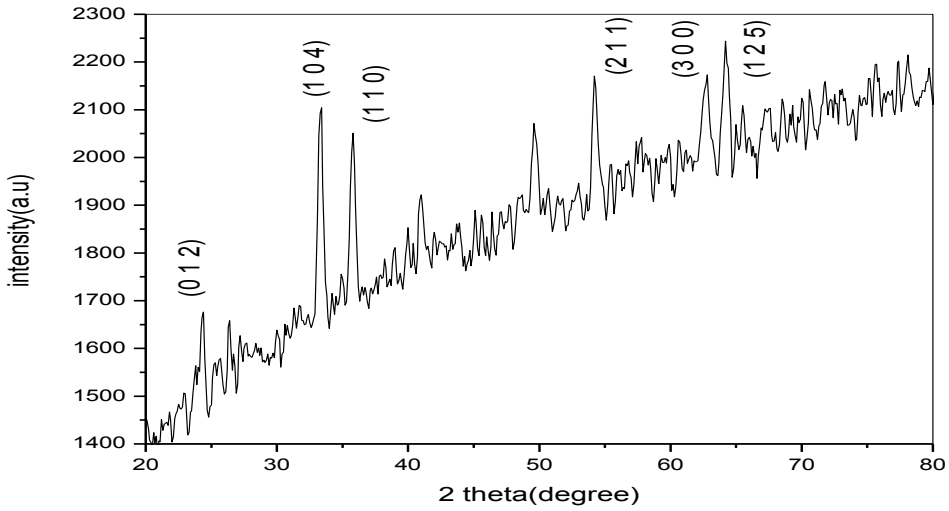

Fig 6 X- Ray Diffractogram of 25 hours milled and annealed α-Fe_2O_3 powders

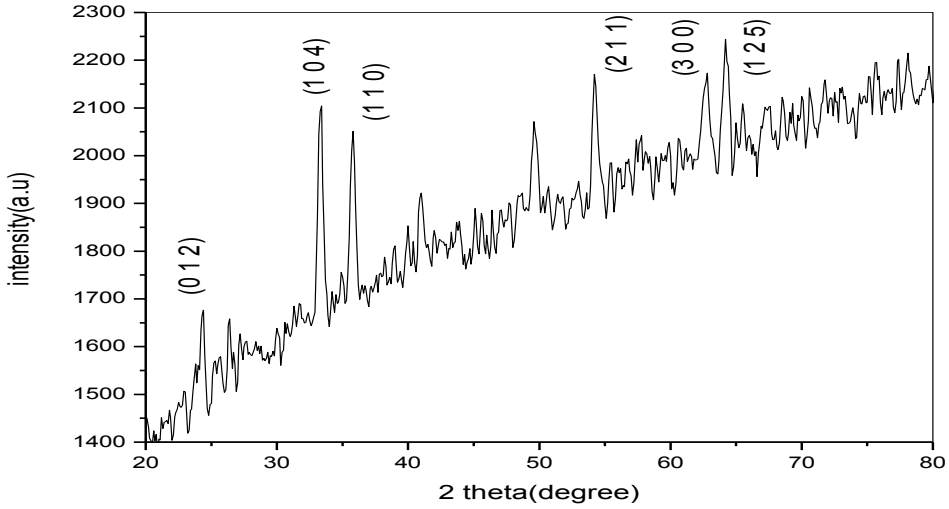

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Table 1. XRD analysis details

Sample	2 Theta (Degree)	(hkl) plane	d value Å	Lattice constant Å	Grain size (nm)	Strain (1 0 4) plane
As cast annealed	33.5361 35.9922 49.8295 54.3966 64.212 66.4312	(104) (110) (024) (116) (125) (300)	2.67004 2.49327 1.82852 1.68529 1.40619 1.44614	a=b=5.6640 c=13.088	12.70 12.83 10.62 13.01 18.05 31.92 _____ **16.523**	0.009454
15hrs milled & annealed	33.4654 35.9558 43.9803 49.8347 56.4000 64.3246	(104) (110) (202) (024) (211) (300)	2.67552 2.49571 2.05717 1.82834 1.63009 1.44706	a=b=5.6678 c=13.119	12.61 12.70 11.96 11.90 23.86 13.98 _____ **14.609**	0.00954
25hrs milled & annealed	24.1968 33.2952 35.7542 56.4000 64.1475	012 104 110 211 300	3.67525 2.68880 2.50931 1.62247 1.45063	a=b=5.681 c=13.179	12.33 13.79 3.327 45.029 39.37 _____ **22.643**	0.00876

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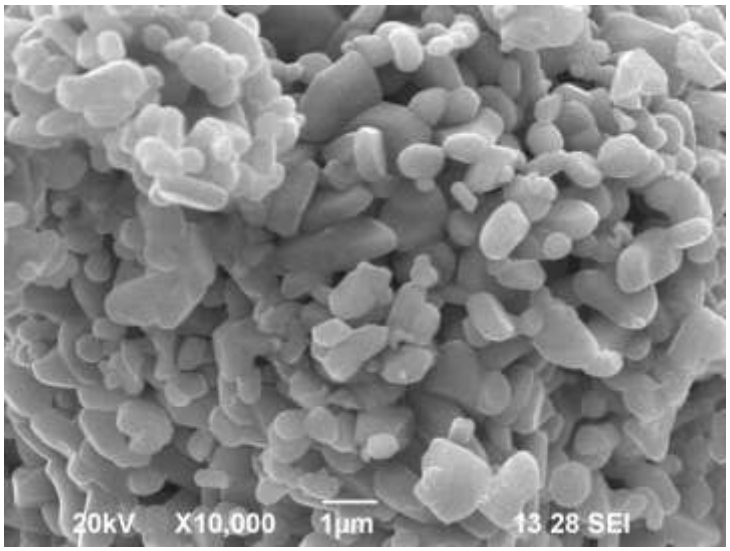

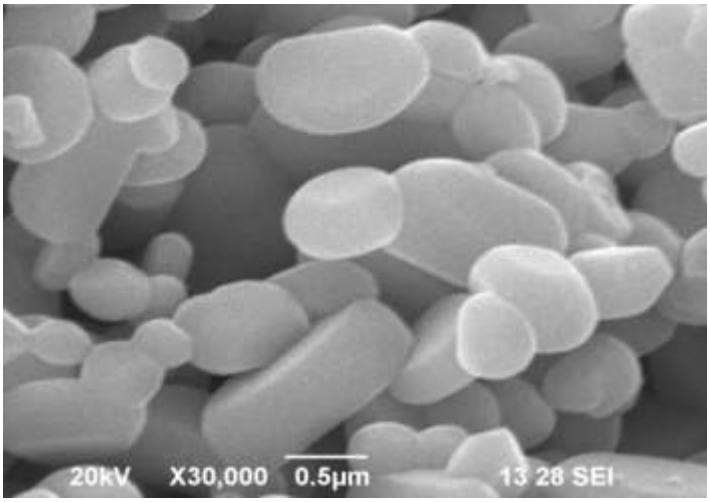

Fig 7 SEM image of as cast α - Fe_2O_3 powder annealed at 1000^0C

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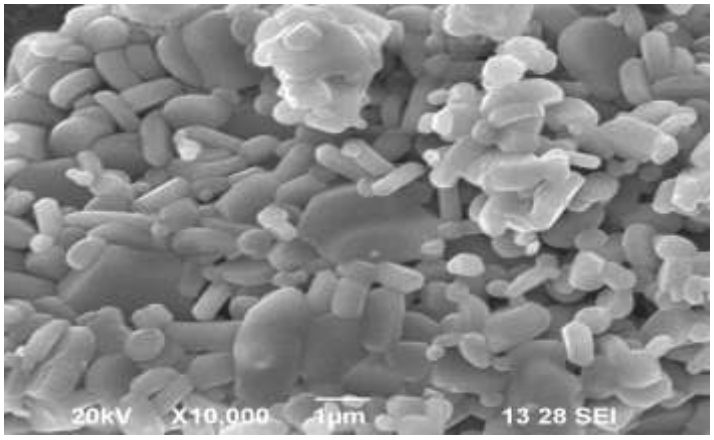

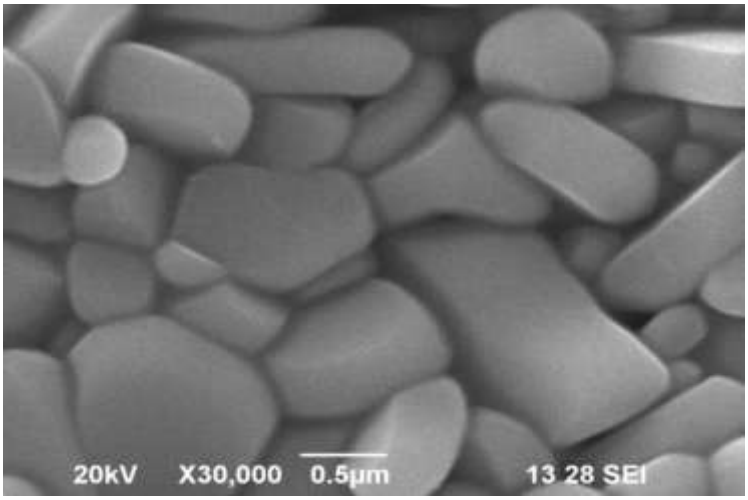

Fig 8 SEM image of 25 hrs milled α-Fe_2O_3 powders annealed at 1000^0C

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Sample	Magnification	Particle size	
		Smaller size	Bigger size
As cast annealed sample	10,000 30,000	19µm 46 µm	89 µm 146 µm
25 hrs milled and annealed sample	10,000 30,000	66.6 µm 73 µm	288.8 µm 150 µm

Table.2. Particle size from SEM images

Material	Grain size (nm)	Retentivity (Mr) (emu)	Coercivity (Hc) (gauss)	Magnetization(Ms) (emu)
as cast sample	16.523	0.0051251	4135.0	0.014932
25milled sample	22.643	0.0050233	4185.0	0.014465

Table.3. VSM data details

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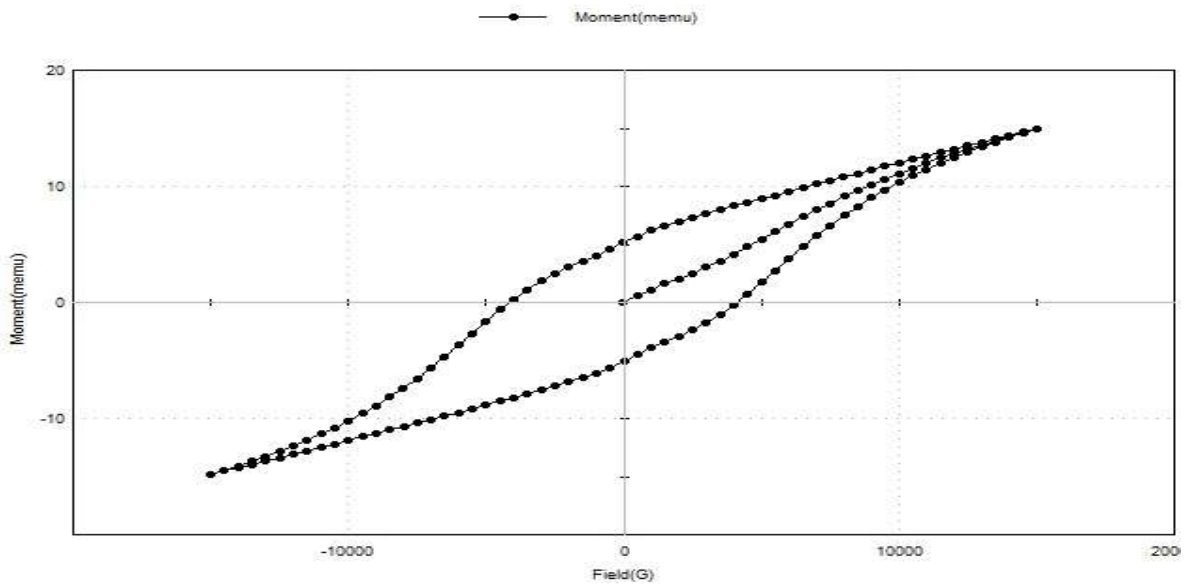

Fig 9 M-H Curve for as cast α-Fe_2O_3 powders annealed at 1000^0C

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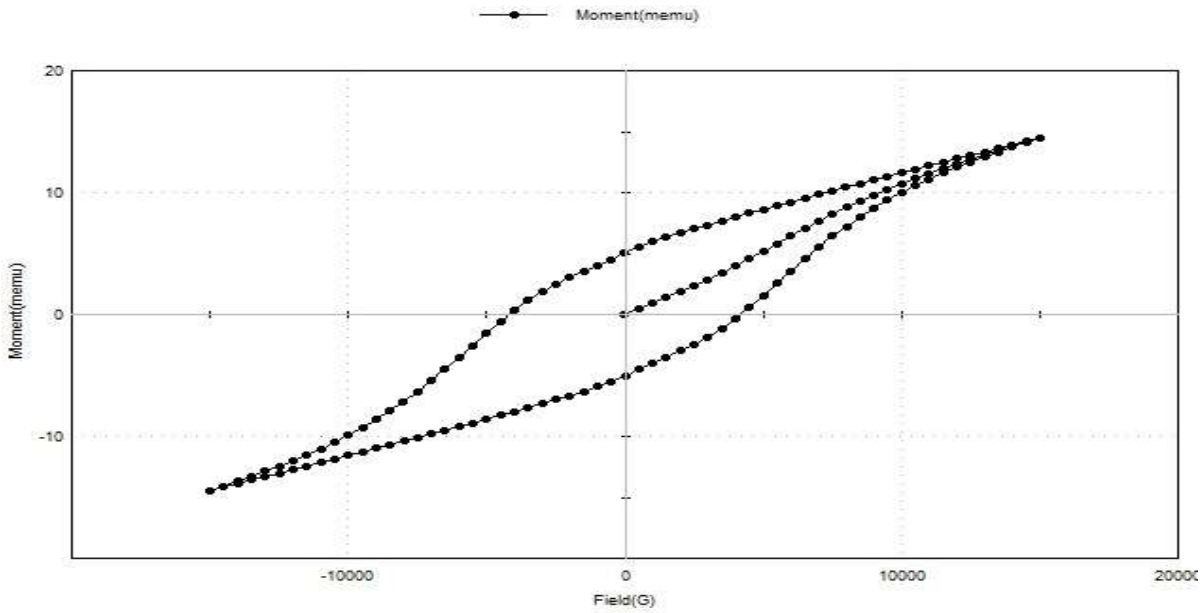

Fig 10 M-H Curve f or 25 hours milled α-Fe_2O_3 powders annealed at 1000^0C

