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Sintering Effects on Structural and Magnetic Behaviours of NdFeB Magnets

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Abstract—Hard magnetic material '$Nd_2Fe_{14}B_1$' was synthesized via powder metallurgy technique and studied for its structural and magnetic properties. The prepared sample was sintered at different temperatures and characterized by XRD, Optical Microscope and vibrating sample magnetometer (VSM). The crystal structure, phase composition and crystallite size were determined with the help of data obtained from XRD. The tetragonal crystal structure of $Nd_2Fe_{14}B_1$ was confirmed with lattice parameters '*a*' = 8.795 Å and '*c*' = 12.225 Å. The crystallite size of the samples was decreased from 58.3 nm to 43.7 nm with increase in sintering temperature from 700°C to 720°C. The micro structural observations showed that the grains were smoothly and uniformly distributed, resulting in an enhancement of valuable mechanical properties of the materials. Saturation magnetization (M_s) and coercivity (H_c) of $Nd_2Fe_{14}B_1$ were increased with increase in sintering temperature, due to decrease in crystallite size, making the materials more suitable candidate for various industrial uses including in voice coil motors and electric and hybrid electric vehicles.

Keywords: $Nd_2Fe_{14}B_1$ magnets, powder metallurgy technique, sintering effect, sturctural and magnetic proporties

1. Introduction

The demand of NdFeB magnets has been increasing rapidly in various fields of life since their development in 1984, due to their excellent magnetic performance and low cost. The coercivity mechanism of Nd–Fe–B based permanent magnets has recently been invigorated due to increasing needs of materials having high coercivity and large energy product (BH) for Voice Coil Motor, electric vehicles, hybrid electric vehicles, etc. Microstructure of NdFeB is a combination of magnetically hard and soft phases. α-Fe exists as hard phase in these magnetics, resulting in an enhancement of remanence [1]. Y. M. Rabinovich et al. [2] has reported the improvement in the coercivity and maximum energy product $(BH)_{max}$ with the substitution of Pr in NdFeB

sintered permanent magnets. Previous works have proved that additions of alloying elements such as Co, Al, Cu, Cr, Ti, Zr, Dy, Nb, etc. can improve the corrosion resistance and structural and magnetic properties of NdFeB [3]. NdFeB magnets can be categorized as microcrystalline and nanocrystalline. There are different techniques for the fabrication of these materials. Nanocrystalline NdFeB ribbons can be synthesized by melt spinning technique [4], whereas nano grain materials can be produced via mechanically alloying or Hydrogen Disproportionation Desorption Recombination (HDDR) techniques [5]. In this paper, we are reporting the sintering effects on the structural and magnetic properties of NdFeB magnets.

2. Experimentation

Powder metallurgy technique was used for the preparation of NdFeB permanent magnets. The precursor materials Neodymium (Nd), Iron (Fe) and Boron (B) were in powder form, all with purity level 99.99 % (4N). Digital Balance was used to measure the precise masses of these elements according to the calculated values. The measured quantities were mixed together and pressed under 50 kN pressure in hydraulic press. One large sample with dimensions 8 cm ×4 cm × 1 cm was prepared and divided into halves. One sample (4 cm × 4 cm × 1 cm) was sintered at 700°C whereas other (4 cm × 4 cm × 1 cm) was sintered at 720°C for 10 hours in a tube furnace. The crystal structure of the magnets was determined by X-ray diffraction (XRD) with Cu-kα radiation (λ = 1.5405 Å) at room temperature. Microstructure analysis was carried out using optical Microscopy whereas magnetic properties were studied by vibrating sample magnetometer.

3. Results and Discussion

3.1 X-Ray Diffractometery

X-ray diffraction analysis was performed for the confirmation of expected fabrication and determination of crystal structure of the prepared samples. XRD patterns shown in Fig. 1 were plotted using data obtained from X-ray diffractometer and prominent peaks were matched with JCPDS (card number 01-086-0273) and indexed accordingly. For each peak, interplaner spacing‘*d*’ was calculated using Bragg’s law ($n\lambda = 2d\ sin\theta$) and was compared with interplanar spacing‘*d*’ in JCPDS database. The calculations of ‘*d*’ for particular peaks, corresponding to the reflections from the (210), (301), (222), (116), (424) and (433) planes for both samples, verify the crystal structure of $Nd_2Fe_{14}B_1$ materials as tetragonal.

The lattice parameters for both samples were determined using XRD Software (Cell). The *hkl* values and corresponding 2θ values of all prominent peaks were inserted in the Software and lattice parameters were known for the samples which have been tabulated in Table 1. These values of lattice parameters are in good agreement with the standard values for $Nd_2Fe_{14}B_1$ magnets.

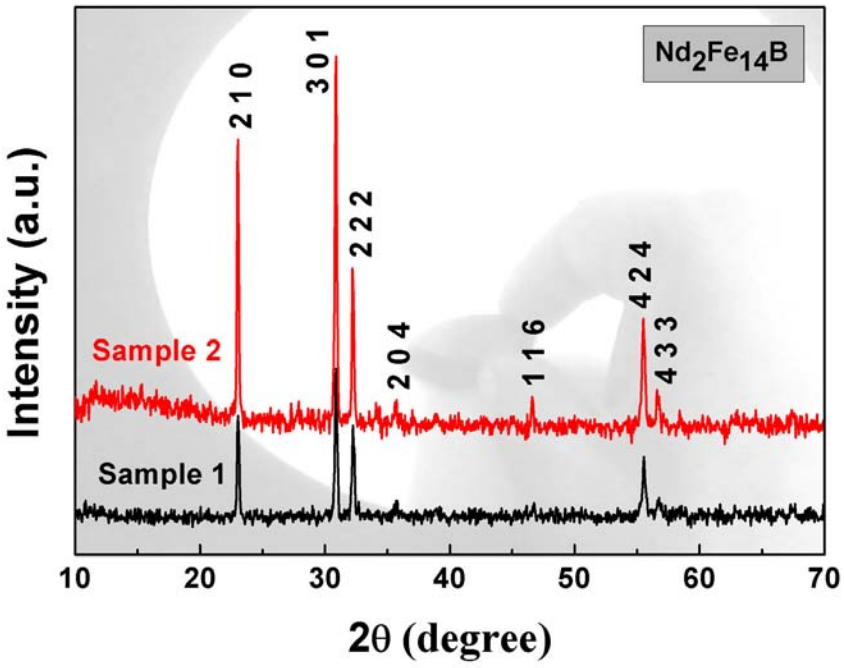

Fig. 1: XRD patterns of $Nd_2Fe_{14}B_1$ magnets sintered at 700°C (*Sample 1*) and 720°C (*Sample 2*)

The crystallite sizes of both samples were calculated from most intense peaks (3 0 1) in the XRD patterns using Scherrer formula [6,7]:

$$\text{Crystallite Size } (t) = \frac{k\lambda}{BCos\theta_B} \quad (1)$$

Here, '*k*' is Scherrer constant and its value is '0.94' for our material, '*B*' is full width at half maximum (FWHM) of respective peaks, 'θ_B' is Bragg's angle and 'λ' is wavelength of Cu-kα radiation (1.5405 Å) used during the XRD analysis of the samples [6-7]. The calculated values of crystallite size for both samples are given in Table 1 which shows that the crystallite size of $Nd_2Fe_{14}B_1$ magnets was decreases from 58.3 nm to 43.7 nm, with increasing sintering temperature from 700°C to 720°C.

Table 1: Structural and magnetic parameters of NdFeB magnets

Sample	Lattice Parameters		Unit Cell Volume (nm^3)	Crystallite size (nm)	ρ_x (gcm^{-3})	M_s (kA/m)	H_c (kA/m)	$(BH)_{max}$ (kJ/m^3)
	$a = b$ (Å)	c (Å)						
1	8.795	12.224	0.945	58.3	7.59	34.46	169.07	416
2	8.795	12.224	0.945	43.7	7.59	41.25	172.05	423

Unit cell of tetragonal Neodymium Iron Boron ($Nd_2Fe_{14}B_1$) magnet contains four formula units. So, the X-ray Density (ρ_x) of the material was determined using the following relation [7]:

$$\rho_x = \frac{n\mathrm{M}}{\mathrm{N_A}V} \quad (2)$$

In this relation, 'M' is molar weight of one formula unit, 'N_A' is Avogadro number, '*n*' is number of formula unit (in our case; $n = 4$) and *V* is the Volume of Unit cell. These results imply X-ray Density (ρ_x) 7.60 g/cm^3 which agrees well with the theoretically calculated value 7.59 g/cm^3 [8].

The present investigation showed that unit cell of NdFeB magnets have a tetragonal structure with lattice parameters $a = b = 8.795$Å and $c = 12.225$Å [9], and consists of four formula units which have total number of 68 atoms. In this unit cell, there are six distinctive Iron (Fe) sites, two dissimilar Neodymium (Nd) sites and one Boron (B) site. The present investigation of the crystal structure of Neodymium Iron Boron ($Nd_2Fe_{14}B_1$) illustrates that at room temperature, the crystal constants agree well with those found earlier [10-11].

3.2 Optical Microscopy

Optical microscopy is a suitable technique to study the microstructure of the materials which tells us about the thermal and mechanical behaviors of that materials. To observe the grains clearly in the optical microscope, etching of samples in dilute solution of HCl was performed for 5 minutes. These micrographs illustrate the high crystallinity of NdFeB permanent magnets, as shown in Fig. 2. The grains are uniformly and smoothly distributed throughout the surface and properly connected with one another. These types of grain distributions illustrate the good mechanical properties of the material.

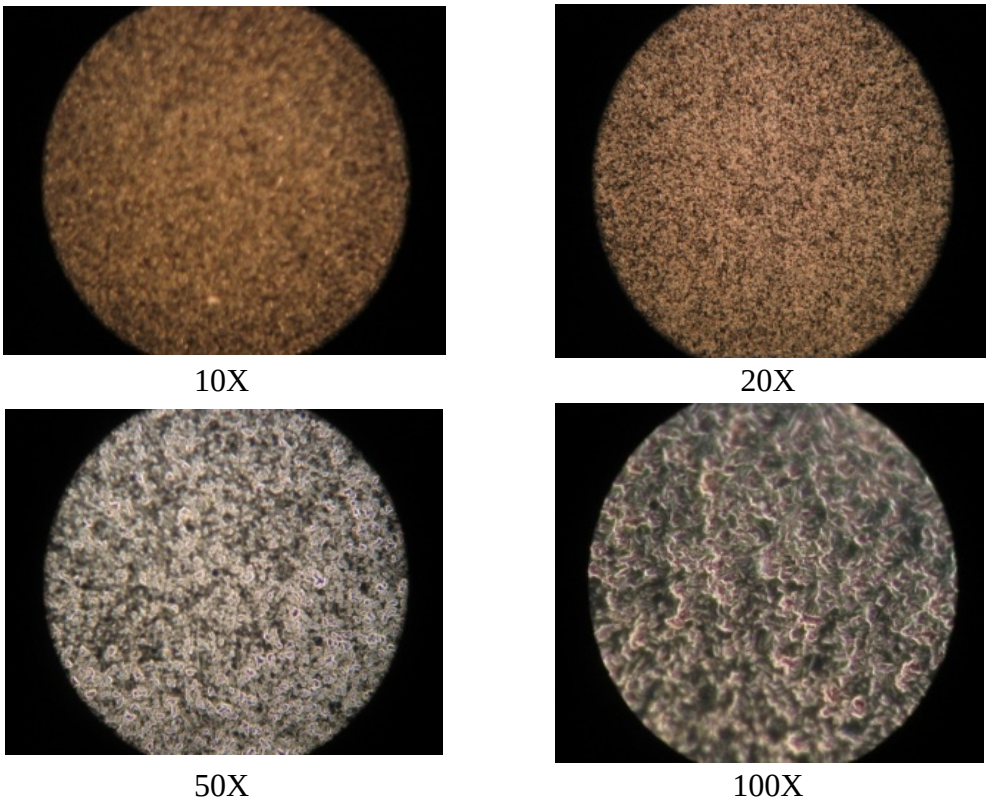

Fig. 2: Optical micrographs of NdFeB magnets after etching in dilute HCl

3.3 Magnetic Properties

M-H loops of the samples are shown in the Fig. 3 which are illustration of the data obtained from vibrating samples magnetometer, at room temperature. The applied magnetic field '*H*' was varied from -8000 Oe to +8000 Oe. Magnetic properties such as saturation magnetization (M_s), coercivity (H_c) and maximum energy product $(BH)_{max}$ were calculated from these plots and their values are given Table1. Magnetic flux density *B* for the energy product $(BH)_{max}$ was calculated with the help of following equation:

$$B = \mu_o (H+M) \quad (3)$$

Here, μ_o is constant of permeability.

It was observed that saturation magnetization (M_s) and coercivity (H_c) of the materials were increased due to the decrease in crystallite size. Hence, magnetic behavior of the samples was straightforwardly affected by alteration in crystallite size of the material [12]. Maximum energy product $(BH)_{max}$ of 423 kJ/m^3 was achieved which shows good magnetic properties of NdFeB permanent magnets. Moreover, the values for M_s, H_c, and $(BH)_{max}$ are much on a better side which shows that by appropriate sintering, good magnetic properties can be achieved in NdFeB magnets.

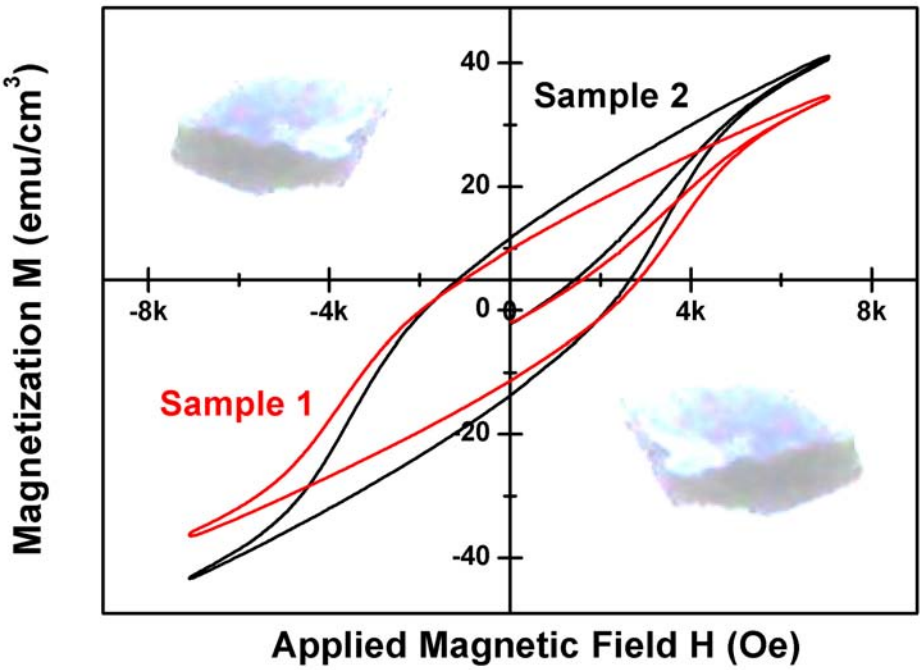

Fig. 3: M-H loops for NdFeB magnets sintered at 700^{o}C (*Sample 1*) and 720^{o}C (*Sample 2*)

4. Conclusion

NdFeB permanent magnets were prepared via powder metallurgy technique and sintered at 700°C and 720°C. Tetragonal structure was the main phase of NdFeB magnets with lattice parameter $a = b = 8.795$ Å and $c = 12.225$ Å. Microstructural

analysis of $Nd_2Fe_{14}B_1$ shows that there are uniform connectivity and homogenous distribution of grains which were developed in the single phase structure of $Nd_2Fe_{14}B_1$. M-H loops of the samples showed an increase in coercivity (H_c) from 169.09 kA/m to 172.03 kA/m and maximum energy product $(BH)_{max}$ from 416 kJ/m^3 to 423 kJ/m^3. It is observed that saturation magnetization (M_s), Coercivity (H_c) and energy product $(BH)_{max}$ are directly affected by changes in crystallite size of the materials. Moreover, our values for M_s, H_c, and $(BH)_{max}$ are much better than previous reports which shows that by appropriate sintering, good magnetic properties can be obtained in NdFeB magnets for required applications.

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