INVESTIGATION OF OPTIMIZED PROCESS PARAMETERS ON DENSIFICATION OF SAMARIUM COBALT 1:5 SERIES ($SmCo_5$) MAGNETS

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ABSTRACT

The aim of this study is to investigate the effect of different process parameters on densification of Samarium Cobalt 1:5 series ($SmCo_5$) magnet by powder metallurgy technique. Different parameters for sintering and heat treatment process such as particle size, load of press, sintering time and temperature, furnace atmosphere and heating rate were tested in order to achieve the highest density. To analyze and evaluate the microstructure and particle size of manufactured magnets scanning electron microscopy (SEM) and X-ray diffraction (XRD) tests were conducted. Results of different tests showed that sintering temperature as well as furnace atmosphere is among the most important parameters affecting on final density of the samples. Investigations illustrated that highest density could be obtained with the sintering of green bodies which made from initial particles with the size of 3 to 6 μm in vacuum condition at 1135 ℃ for 30 min by rapid heating at the shortest time.

KEYWORDS

Samarium Cobalt, rare earth permanent magnet, powder metallurgy, heat treatment.

1. INTRODUCTION

Magnetic behavior of materials mainly depends on their electronic structure which can provide the magnetic dipoles [1-4]. Samarium Cobalt magnets are usually characterized due to their appropriate magnetic properties such as high magnetic remanence and high coercive force which makes $BH)_{max}$ up to 30 MGOe, appropriate thermal stability or small variation coefficient of magnetic properties with temperature therefore are the most suitable option for application at temperature above 300℃ among hard ferromagnetic materials. The development of $SmCo_5$ based magnets was result of the research on the anisotropy of YCo_5 by Hoffer. G and et al. In order to develop a high dense and stable $SmCo_5$ magnet, liquid phase sintering is needed. This magnet can make $BH)_{max}$ equals to 22 MGOe at its extreme energy level [5-10]. The magnetic properties of materials can be divided into two general categories: structure sensitive and structure insensitive. The first category refers to properties which are not affected by change in materials processing (heat treatment or mechanical deformation) or by small changes in composition, including small amount of certain impurities. Structure insensitive properties include the saturation magnetization and resistivity. These properties are largely dependent on the composition of the particular alloy and do not change substantially in the process of manufacturing from the alloy [11-14]. Fine magnetic properties of Samarium Cobalt magnets are due to elements exist in the composition. These properties inherently exist in molecular orbitals of rare earth elements. Usually 3d sub-orbitals of transition metals make magnetic anisotropy and help to high magnetization and fine properties at high temperature. Interstitial metal compounds which form by combination of rare earth elements and transition metals elements make interactions between the orbitals of layer 4f and 3d that enhance the coupling of orbital-spin and eventually increase the system anisotropy [15-19]. Samarium Cobalt 1:5 series magnets have high magnetocrystalline anisotropy that form based on orbital-spin coupling and interaction of crystal-field under the influence of electrostatic force [14]. Magnetic properties of Samarium Cobalt magnets are controlled by pinning walls mechanism [19-21]. Therefore magnetic properties are greatly influenced by microstructure. An appropriate microstructure obtains after a long and complicated heat treatment process [11-12]. The most important factors on microstructure are density of sample and grain size [13]. To improve magnetic properties, both higher density and smaller grain size are required. Microstructure of Samarium Cobalt 1:5 series contains of $SmCo_5$ matrix phase with Sm_2Co_7 magnetic phase and also Sm_2O_3 oxide which exist in composition in correlation of cavities and porosities made by powder metallurgy process [16-19].

Critical single domain size for $SmCo_5$ estimates from equation below:

$D_c = 18\gamma / \mu M_s^2 = 2\ \mu m$

Knowing single domain size is very important since residual magnetic field largely depends on grain size and magnetic domain size [21-22].

Manufacturing process of Samarium Cobalt by sintering and powder metallurgy technique is as follows: preparing initial alloy, powder milling, mixing powder, pressing and magnetic orienting, sintering and homogenization, heat treatment, machining operation and finally magnetization [23-31].

In this paper the effective parameters such as size and distribution of initial powder, press force, furnace atmosphere, heating rate, sintering time and temperature and heat treatments on developing Samarium Cobalt 1:5 series magnet by powder metallurgy technique were investigated and finally by comparing the results, the best option for developing this magnet has been presented.

2. EXPERIMENTAL

2.1. Analyzing the initial powder and compound

The amounts of elements existing in primary material were analyzed chemically by X-ray fluorescence (XRF) (BRUKER, S4PIONEER, Germany) using Rhodium anode. Results are shown in table 1.

Table1. Result of element analysis of initial compound 1:5 series.

Element	Concentration (%W/W)
Co	62.74
Sm	20.56
Pr	13.50
La	1.14
Fe	0.991
Rh	0.248
Ni	0.208
Cu	0.146
Eu	0.126
Zr	0.041
Cr	0.032
Br	0.018
Rb	0.014
Total	99.764

X- ray diffraction (XRD) patterns of samples were taken with Bruker (D8 advance, Germany) X-ray diffractometer over a 2θ range from 10° to 80° using Cu K_α radiation (λ=1.5406 Å) in the step size of 0.02, equipped with X'Pert HighScore for data analyzing.

The average size and shape of particles of various samples were determined from their scanning electron microscopy images (SEM; stereo SCAN, S360 version V03.03, England). Due to the electrostatic charge of powders and hence diverting the electrons, the powders were gold sputtering in order to improve the quality of SEM micrographs.

2.2. Development of the sample

To make a single domain Samarium Cobalt magnet by powder metallurgy technique, initial size of powders should be at least less than 10 µm [10,16 and 33]. Milling process was performed in a planetary ball mill using a Φ 10 and 20 mm steel balls and under the argon atmosphere with purity of 99.999% and milling speed of 350-450 rpm for 0.5-1 hours. The powder/ball weight ratio was determined 1 to 20. Then, milled powders were pressed into a toroidal shape with an outer diameter of 15 mm and inner diameter of 8 mm and a thickness of 3 mm by a hydraulic uniaxial press with the pressure of 40 to 60 kg/cm^2 in the presence of magnetic field with intensity of 1.5 to 2 Tesla was used to align the magnetic particles [10, 16 and 33]. Afterward, samples were encapsulated in vacuumed cleaned quartz tubes for sintering and heat treatments process. After each step density of samples were measured by the water boiling method.

2.3. Sintering and heat treatments processes

he Heat treatments process of Samarium Cobalt 1:5 series magnets were performed through the following steps: sintering at about 1130-1150˚C for 0.5-1 hour. Solid solution, isothermal aging and finally slow cooling and then soaking took placed in order to make the magnetic phase of matrix and secondary phase. Quenching in cold water was performed to prevent the decomposition of formed phases immediately after heat treatment process [27-28].

3. RESULTS and discussion

3.1. Effect of particle size on final density

In general, magnetic properties of a Samarium Cobalt magnet depends on it microstructure predetermined by processing step such as jet milling, compaction, sintering and post sintering. Each processing step is important and should be controlled carefully to improve the hard magnet. Since the particles with average size of 5 µm have single domain behavior, milling was done to reach single domain size particles; in addition, this has significant effects on magnetic and mechanical properties and improves the final density of sample [26-36]. According to SEM micrographs Fig. 1, 2 and 3, analyzing the figures by Image Analyzer software revealed that most of the green powder size was about 10 to 18 µm. Milling process was continued to obtain single domain grains with size of 3 to 6 µm [20,37]. Afterward, pressing in the presence of a magnetic field aligned all the particles that their easy axis had same direction which caused a high magnetic remanence in a specific direction. Table 2 shows the effects of particle size on final density of samples. Then, sintering was done at 1135˚C with 15ºC/min heating rate and 30 minutes soaking time. The results show that the density of a sintered 1:5 series body was higher when the magnet made with the fine particles powders. As a result, milling for 1 hour at 400 rpm has the best conditions. SEM micrographs also show that the shape of particles were changed to spherical which is more suitable for pressing and has less porosity, namely by milling the green powder, better density would be obtained because of smaller pores.

Table2. Effect of particle size on final density of 1:5 series.

Sample	Particle size(µm)	Milling rate(RPM)	Milling time (min)	Final density(gr/cm^3)
1	15-25	400	0	7.75
2	8-12	400	30	7.88
3	3-6	400	60	7.95

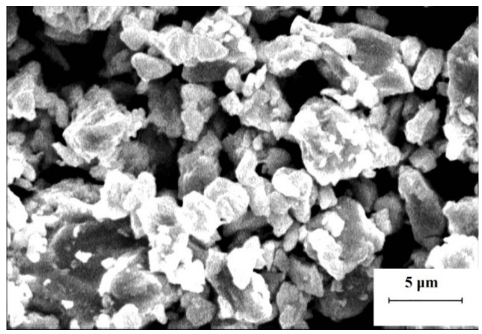

Fig1. SEM micrographs of green powder of 1:5 series before milling (magnification: 1000).

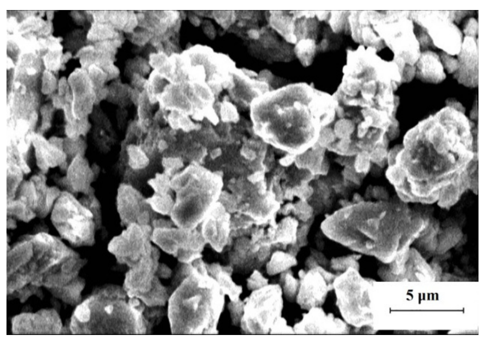

Fig2. SEM micrographs of green powder of 1:5 series after 30 minutes milling (magnification: 1000).

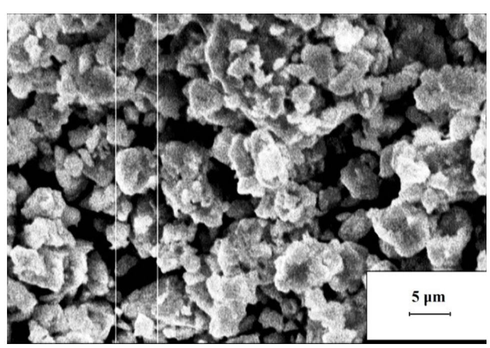

Fig3. SEM micrographs of green powder of 1:5 series after 1 hours milling (magnification:1000).

3.2. Effect of pressing pressure

Pressing pressure is one of the important parameters affecting the green and final density of the sample. Low pressing pressure decreases the density and high pressing pressure makes problems such as rapid degradation of the mold, misaligning of the powder and cracking the sample. Therefore, choosing the optimum pressure seems necessary. In this way, different pressures were tested to determine the optimum pressure and results are showed in table 3 versus final density of samples. Results show that the appropriate pressure is 50 kg/cm^2. Pressures above 50 kg/cm^2 would make crack in the sample or decreases the final density and pressures less than 50 kg/cm^2 may prevent densification in particles and reduce the final density of samples.

Table3. Relation of pressure with final density of 1:5 series.

Sample	Pressure Kg/cm^2	Final density gr/cm^3
1	40	7.85
2	50	7.93
3	60	7.8

3.3. Effect of furnace atmosphere on final density

Diffusion in solids at high temperature significantly increases; therefore, unwanted phases may form or the formation of desired phases may be prevented. This phenomenon is very important for Samarium Cobalt magnets due to the high chemical activity of elements in their composition [12, 38-39]. For this purpose, samples were sintered at 1135˚C for 30 minutes under three different conditions. In the first case, sintering process took place in a furnace under air atmosphere. Sample was sintered and quenched in cold water. In this case, the density was measured 5.91 g/cm^3. In the next case sample was placed in quartz tube and sintered under argon atmosphere with purity of 99.999%. In this case, the density was measured 7.54 g/cm^3. In the last case, sample was placed in quartz tube with vacuum conditions. Consequently, the density was increased to 7.91 g/cm^3. Because of high reactivity of rare earth elements in the composition especially Samarium, in the first case the surface was completely oxidized. Surface oxidation can be considered as important cause for the coercive force loss of powders. Duo to the presence of open pores or microcracks, the oxidation rate of massive bodies can be increased [40-41]. For the second case sublimation of slight percentage of alloying elements such as Samarium and accumulation of water vapor in the powders as well as argon gas at high temperature increases vapor pressure in quartz tube so that high pressure gases penetrated into the pores of sample and prevent fully densification. Therefore, the vacuum is the best choice for high temperature process.

3.4. Effect of heating rate on final density

Thermal shock, thermal expansion and outgassing process mainly discussed when studying heating rate of Samarium Cobalt magnet [28]. Apart from these parameters, grain growth with increasing time and temperature are two other important issues. In permanent magnets based on rare earth elements which magnetic properties are based on domain walls pinning mechanism, grain growth should be prevented through reducing time and temperature of sintering process. In other words, sintering time should be as short as possible and also sintering temperature should be low [16]. In order to obtain appropriate heating rate, heating process of Samarium Cobalt 1:5 series conducted in three different conditions. The samples A, B and C has been raised from room temperature to 1135˚C for 180, 120 and 60 minutes respectively, and kept at this temperature for half an hour. Figure 4 showed that the sample can be reached to appropriate density by rapid heating. If heating rate would be too slow, desired density could not be obtained.

Higher heating rate would prevent Samarium sublimation and decreases inner pressure of the tube so that makes it possible to reach higher density.

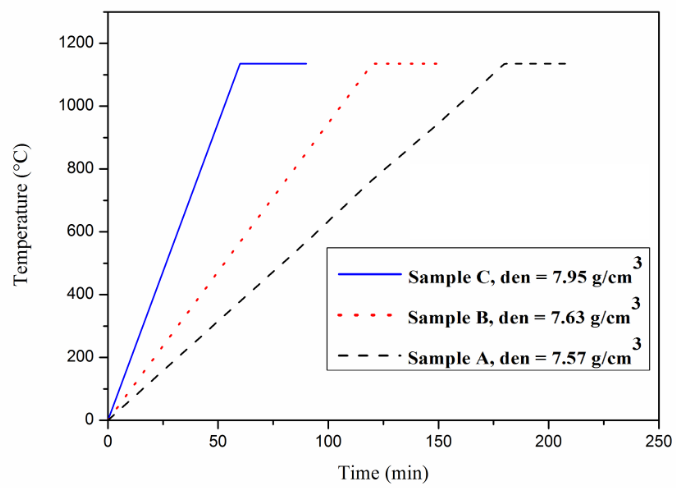

Fig4. Effect of heating rate on final density of Samarium Cobalt 1:5 series, sample A, B and C.

3.5. Effect of sintering temperature on final density

Sintering helps to homogenize the microstructure and increase the densification of samples. Sintering time and temperature have many effects on grain size [16]. In order to analyze effects of sintering temperature on final density and also choosing the most appropriate temperature, samples were sintered at 5 different temperatures between 1130 to 1150˚C for 30 minutes at same conditions.

In the first case, sintering process took place by heating the sample to 1150˚C. The sample lost its shape totally and the density was 7.74 g/cm^3. In the second case, sintering took place by heating the sample to 1145˚C. The sample deformed from its original shape and the density was 7.81 g/cm^3. In the next case, sintering took place by heating the sample to 1140˚C. In addition, only the edge of sample lost their original shape and the density increased to 7.98 g/cm^3. Afterward, for the next one, sintering took place by heating the sample to 1135˚C. The sample retained its original shape and the density measured 7.95 g/cm^3. Finally, for the last case, sintering took place by heating the sample to 1130˚ C. In this sense, the sample also retained its original shape but the density decreased to 7.83 g/cm^3. One can see from Fig. 5 that in low sintering temperatures, high density, appropriate magnetic saturation and magnetic remanence are obtained. In addition to the above mentioned, time and temperature affect the grain size, too. Since by increasing sintering time and temperature, grains are grown and reach to the size which is bigger than the scope of single domain. As a result the most appropriate sintering temperature for Samarium Cobalt 1:5 series is 1135˚C.

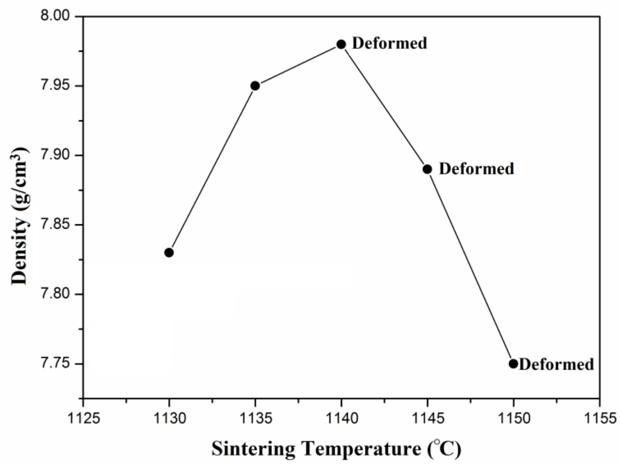

Fig5. Final density of Samarium Cobalt 1:5 series based on sintering temperature.

3.6. Heat treatments

The coercivity of $SmCo_5$ magnets is strongly related to the temperature of heat treatment. In the as-sintered condition (i.e. sample only heat treated at 1100–1150˚C), low coercivity (0.1–0.5 T) is often observed. High coercivity (>3.0 T) only develops after a heat treatment performed at 850–900˚C. If the samples are submitted to heat treatment at 700–750˚C, the resulting coercivity is very low (<0.5 T), a fact that has been attributed to the eutectoid decomposition of $SmCo_5$ phase. Important microstructural aspect for the properties of permanent magnets are the matrix grain size, crystal texture, nature of the grain boundaries and distribution of any secondary phases. In order to obtain these conditions, samples heat treated precisely.

Immediately after sintering, the coercivity is poor. Annealing above the eutectoid temperature, followed by rapid cooling through the critical range where decomposition occurs fairly quickly will develop the best permanent magnet properties [41]. According to Fig. 6 in heat treatment process of the sintered samples, temperature was reduced to 800-900˚C for 30 minutes to 5 hours and finally cooled to room temperature by different rates [27,32]. Normalizing or quenching in water were two ways in order to cooling the samples. In the first and second cases, temperature of sintered samples for ageing treatments decreased to 850˚C with the rate of 2˚C/min and soaked for 90 minutes. In the following, sample A quenched at cold water and sample B normalized to room temperature slowly. Density of Sample A and B were 7.83 g/cm^3 and 7.79 g/cm^3 respectively. In the last case, temperature of sample C decreased to 850˚C with the rate of 5˚C/min and kept at this temperature for 90 minutes. Eventually, sample C quenched in water. In this case density increased to 7.85 g/cm^3.

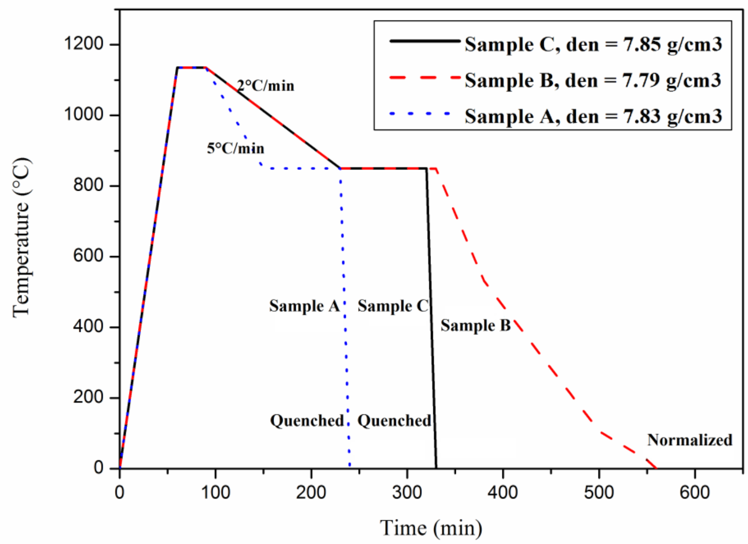

Fig6. Heat treatment process of Samarium Cobalt 1:5 series, sample A, B and C.

3.7. Phase analysis of Samarium Cobalt 1:5 series

The ideal microstructure of $SmCo_5$ sintered magnets consists of aligned single-domain grains with an ideal $SmCo_5$ structure. It is well known that sintered magnets are demagnetized by the domain wall motion, thus the coercive force is determined by the nucleation field of reversed domains. Nucleation of reversed domains takes place in regions with low magnetocrystalline anisotropy which are concentrated near grain boundaries [39-41]. Figure 7 represented the result of XRD patterns of Samarium Cobalt 1:5 series initial composition. It was obvious that $SmCo_5$ phases exist.

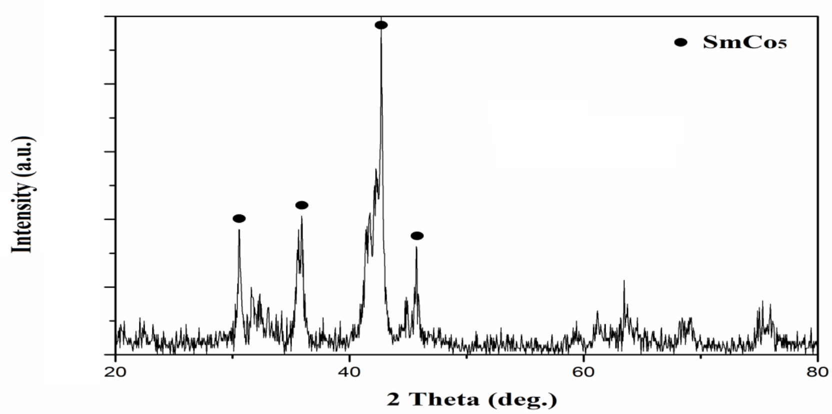

Fig7. XRD analysis of initial composition of Samarium Cobalt 1:5 series.

As can be seen in Fig. 8, the microstructure of Samarium Cobalt 1:5 series after heat treatment consist of $SmCo_5$ phase along with Sm_2Co_7 as the secondary phase. XRD analysis was taken from the sample in order to make sure of forming Sm_2Co_7 phase after heat treatments. The remanence of a permanent magnet depends fundamentally on the volume fraction and the saturation magnetization of the phases in the micro structure, as well as on the alignment of the ferromagnetic phases. In $SmCo_5$ magnets, the microstructural constituents are the $SmCo_5$ matrix phase, Sm_2Co_7 secondary phase and another ferromagnetic phase and Sm_2O_3 oxide. In sintering process, Sm_2Co_7 transforms into $SmCo_5$ due to oxidation or evaporation, the resulting $SmCo_5$ will follow the orientation of the parent Sm_2Co_7 phase [39-41].

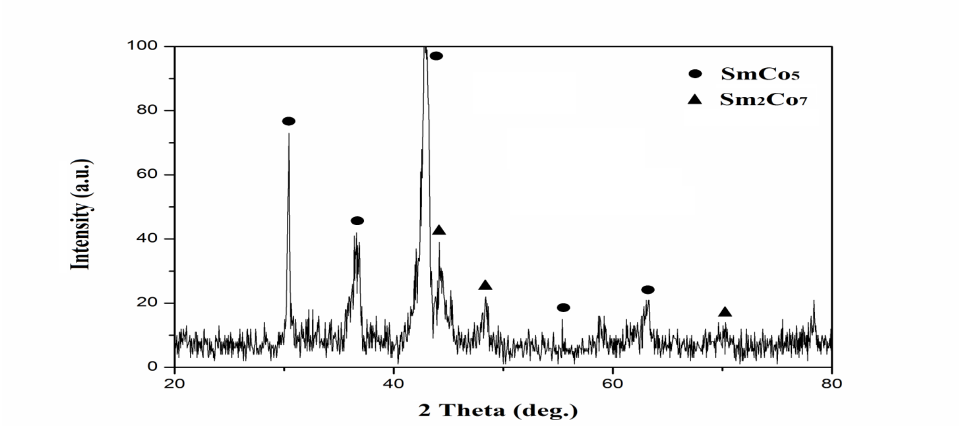

Fig8. XRD analysis of Samarium Cobalt 1:5 series after 30 minutes sintering at 1135˚C and aging for 2 hours at 850˚C.

Finally for magnetizing the samples, an external magnetic field which is 2 to 3 times stronger than the residual field with a magnitude of 2.5 to 3.5 Tesla was applied throughout the ring. Hystograph model MESSTECHIK BROCKAUS was used to measure the magnetic properties of sintered samples. Fig 9 is hysteresis curve for a Samarium Cobalt 1:5 series which is first sintered at 1190˚C and homogenization at 1175˚ C and then heat treated at 850˚C for 8 hours and finally its temperature is reduced to 400˚ C for 11 hours.

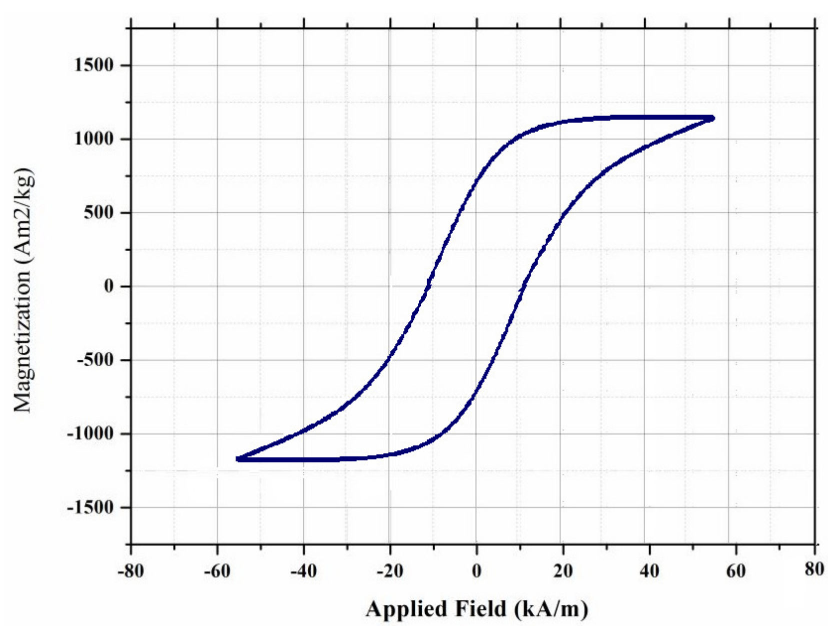

Fig9. Hysteresis curve for a sintered Samarium Cobalt 1:5 series after heat treatments at 850˚C for 8 hours and reducing to 400˚ C for 11 hours.

4. CONCLUSIONS

If the highest possible remanence and energy product in addition to good property stability at elevated temperature for a permanent magnet are desired, the magnet must have a density near 100% and a high degree of grain orientation. The density of $SmCo_5$ compound was varied by controlling the grain size and the highest density could be obtained by using a 3-6 µm green powder. SEM micrographs also showed that the shape of particles were changed to spherical which was found to be more suitable for obtaining high density. In addition, the density of constituent phases in a sintered $SmCo_5$ magnet were depend on difference parameters such as sintering, heat treatment and slow cooling. The highest density of sample without shape deformation achieved by sintering at 1135˚C under vacuum condition. Meanwhile, the sample can be reached to appropriate density by rapid heating at the shortest time. Consequently, heat treatment process was done to increase coercivity. In this way the sample cooled to 850˚C and soaked for 90 minutes. XRD analysis showed that microstructure of Samarium Cobalt 1:5 series after heat treatment consist of $SmCo_5$ phase along with Sm_2Co_7 as the secondary phase. Presence of this phase in grain boundaries increases the magnetic coercivity of system which acts by domain boundaries pinning mechanism.

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