INVESTIGATION OF OPTIMIZED PROCESS PARAMETERS ON DENSIFICATION OF SAMARIUM COBALT 2:17 SERIES (SM_2CO_{17}) MAGNETS

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ABSTRACT

Samarium Cobalt 2:17 series (Sm_2Co_{17}) magnet is prepared by powder metallurgy technique. Different parameters for sintering and heat treatment process such as sintering time, temperature, furnace atmosphere and heating rate were tested in order to achieve the highest density for Samarium Cobalt 2:17 series that could be obtained. To analyze and evaluate the microstructure and particle size of fabricated magnets, scanning electron microscopy (SEM) and X-ray diffraction (XRD) tests were used. Results show that sintering temperatures and furnace atmosphere are among the most important parameters that affecting on the density of the samples and consequently the magnetic properties. It is showed that the highest density of 7.98 g/cm^3 (%95 of theoretical density) has been obtained from initial particles with the size of 3 to 6 µm and sintering temperature of 1195^{0}C with a rate of 17˚C/min for 1 hr in vacuum condition.

KEYWORDS

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

1. INTRODUCTION

Samarium Cobalt (Sm_2Co_{17}) magnets are ideal materials for applications such as microwave tubes, sensors and servo motors because of their high magnetic properties, thermal stability and appropriate corrosion resistance. Among all the rare earth permanent magnets, Samarium Cobalt 2:17 series have the highest Curie temperature and the maximum value of saturation magnetization at high temperatures [1-8]. Samarium Cobalt magnets are usually manufactured by powder metallurgy technique. They are brittle and show a high thermal and corrosion resistance [10-13]. They are also relatively expensive because of the rarity of the elements in their composition. Manufacturing process of Samarium Cobalt by powder metallurgy is as following: Preparing initial alloy, powder milling, mixing powder, pressing and magnetic orientation, sintering and homogenization process, heat treatment, machining operation and finally magnetization [9,10,14,15]. Adding Iron to a limit extent to this composition by replacing Cobalt atoms increases the maximum energy $BH)_{max}$, magnetic saturation (M_s) and magnetic remanence (B_r), and it is because of Iron atoms have higher magnetic moments than the Cobalt atoms. However, if the limit is exceeded, magnetic coercivity would be decreased. The Curie temperature reduction is one of the problems that happen when Iron is added to the main composition [18-23]. Copper in the composition makes it more difficult for domain walls to move thus increases the magnetic saturation. Copper also decreases the eutectic temperature of the composition [10,24,25]. Adding Zirconium increases anisotropy and magnetic coercivity, however adding too much will also reduce the magnetic properties [26,27].

Grain size and dislocation density are two important microstructural factors which affect the magnetic properties. It has been proved experimentally that magnetic coercivity has a direct relation with $\square^{0.5}$ (where $\square$ is dislocation density) [28,29]. However, when the grain size is small, grain boundaries increase and it greatly improves coercivity due to the fact that grain boundaries lock magnetic domain walls. Therefore by reducing the size of particles, single domains are produced and consequently the magnetic coercivity decreases to zero. In this case a superparamagnetic material is made [30,31].

In Samarium Cobalt 2:17 series the main matrix is the $Sm_2(CoFe)_{17}$, a phase with rhombohedral unit cell. The matrix increases the magnetic saturation of the system which is surrounded by $Sm(CoFe)_5$, a Cu-rich phase with hexagonal unit cell. This grain boundary phase increases the magnetic coercivity of the system which works by domain boundaries pinning mechanism. Nevertheless, both cellular and lamellar phases with hexagonal unit cell also exist at phase boundaries. A Zr-rich phase is precipitated on cellular phase. This phase also makes an easy diffusion path for Copper atoms motion [32-39].

It has been reported that density of a sample has direct proportion to magnetic properties. Achieving a high dense body requires some factors such as apparent density, particle size, particle size distribution, particle shape, powder purity and etc [40]. In this paper the effective parameters such as size of the powders, pressing load, furnace atmosphere, heating rate, sintering time and temperature and heat treatment cycles on developing a full dense Samarium Cobalt 2:17 series magnet were investigated. The theoretical density of SmCo magnets have been reported 8.2-8.4 gr/cm^3 [42]. In each step of fabrication, the density was measured by Archimedes method precisely.

2. EXPERIMENTAL

2.1. Analyzing the powders

X-ray fluorescence (XRF) analysis (BRUKER, S4PIONEER, Germany) using Rhodium anode was used to determine the elements and the weight percent of them which are existing in primary material. Results are shown in table 1.

Table1. Result of element analysis of initial compound 2:17 series

Element	Concentration (%W/W)
Co	47.93
Sm	25.68
Fe	15.88
Cu	6.38
Zr	3.193
Rh	0.315
Ni	0.285
Si	0.100
Al	0.071
Zn	0.040
Ca	0.024
S	0.019
Total	99.92

Phases available in the powders and samples were determined by X- ray diffraction (XRD) analysis (Bruker, D8 advance, Germany) using Cu K_α radiation (λ=1.5406 Å), equipped with X'Pert HighScore for data analyzing.

Size and shape of particles were examined by scanning electron microscopy (SEM; stereo SCAN, S360 version V03.03, England). The powders were sputtered by gold in order to improve the quality of SEM micrographs.

2.2. Development of the samples

To make a single domain Samarium Cobalt magnet by powder metallurgy technique, initial size of powders should be at least less than 10 μm. Ball milling was performed in a planetary mill having steel balls with diameter 10 and 20 mm. This process was done under the Argon atmosphere with purity of 99.9% and velocity of 350-450 rpm for 1-2 hours. The powder/ball weight ratio was set from 1 to 20. Then, samples were formed with 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 to align the magnetic particles [18,27,32,42-44]. Afterward, samples were encapsulated in the vacuumed high purity quartz tubes for sintering and homogenization process.

2.3 Sintering, homogenization and heat treatment processes of the samples

Sintering process of Samarium Cobalt 2:17 series magnet took place at a temperature range of 1180-1250 ˚C for different time intervals. After sintering, homogenization process took placed in order to form the secondary phases with composition of $SmCo_5$ throughout the matrix phase of the Sm_2Co_{17}. The temperature of this process was about 15-25˚C lower than sintering temperature. Immediately after homogenization process, quenching was performed in a cold water to prevent the decomposition of secondary phase. Next, Samples were heated up to 850˚C and kept at this temperature for 8 hours, finally the temperature reduced to 400˚C during 11 hours. These heat treatments were done for precipitating the Cu-rich phase on matrix grain boundaries enhancing the magnetic remanence [45-47].

3. RESULTS AND DISCUSSION

3.1. Effect of particle size on final density

Size of initial powder and size distributions are two effective factors on densification. The coarse particles increase porosity size; therefore, density would be decreased. Achieving a single domain particle is desirable. Since the particles with average size of 5 μm have single domain behavior [44,50], milling was done on the powders to reach to this particle size. Image Analyzer software was used to obtain the particle size distribution of the initial powders. The results from SEM micrographs Fig. 1 and 2 show that most of the green powder size were between 20 to 40 μm. Milling process was continued to obtain single domain grains with size of 3 to 6 μm [18]. Afterward, pressing was performed in the presence of a magnetic field. It aligned all the particles that their easy axis had the same direction of applied magnetic field in a specific direction. Then, sintering was done at 1200˚C with 15ºC/min heating rate and 1 hr soaking time. After pressing and sintering process density of the samples were measured. Table 2 shows the effects of particle size on final density of the samples. The results show that the 2:17 series powders, milling for 2 hours at 400 rpm have the best result. SEM micrographs show that the shapes of particles were changed to spherical during milling process. Spherical shape is more suitable for pressing and has less and smaller porosities, hence better density would be obtained.

Table2. Effect of particle size on final density of 2:17 series

sample	Milling time (min)	Milling speed(RPM)	Particle size(µm)	Final density(g/cm3)
1	0	-	20-40	7.26
2	120	400	2-6	7.84
3	240	400	undetermined	Powder oxided immediately

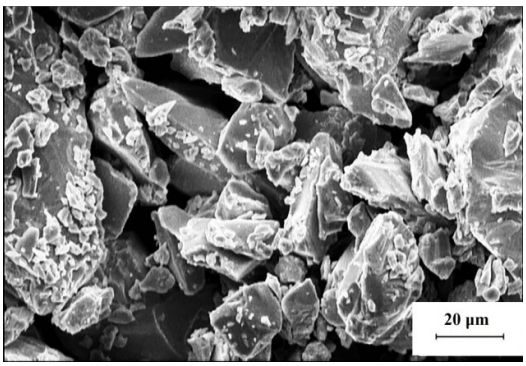

Fig1**.** SEM micrographs of green powder of 2:17 series before milling (magnification:1000)

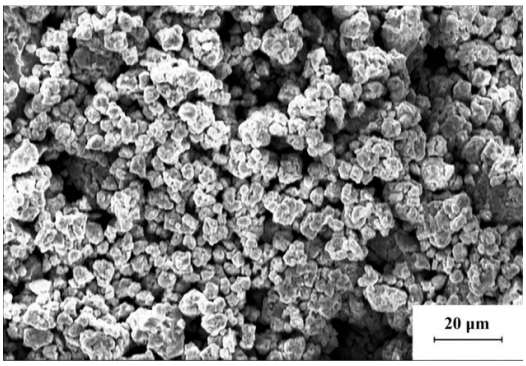

Fig 2. SEM micrographs of green powder of 2:17 series after two hours milling (magnification:1000)

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 is necessary. In this way, different pressures were tested to determine the optimum pressure and results are showed in table 3 versus final density of the 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 the samples.

Table3. Relation of pressing pressure versus final density of 2:17 series

Sample	Pressure Kg/cm^2	Final density g/cm3
1	40	7.34
2	50	7.86
3	60	Cracked

3.3. Effect of furnace atmosphere on final density

Diffusion in solids significantly increases at high temperature; therefore, unwanted phases may be formed 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 [9,10]. For this purpose, samples were sintered at 1200˚C for 60 minutes under four different atmospheric conditions. In the first case, sintering process took place under air atmosphere and then quenched in cold water. In this case, the measured density was 5.20 g/cm^3. In the next case, sample was sintered under hydrogen atmosphere. Since there was no possibility of quenching owing to hydrogen atmosphere, sample was cooled in the furnace. The sample appearance was good at first but within a few days sample was fully destroyed because of the formation of cracks. The final density was decreased to 4.63 g/cm^3. In the third case, sample was placed in quartz tube and sintered under argon atmosphere with purity of 99.999%. In this case, the measured density was 7.67 g/cm^3. In the last case, sample was placed in quartz tube with vacuum conditions. The density was increased to 7.95 g/cm^3. Because of the high reactivity of elements in the composition such as Samarium and Zirconium, in the first case the surface was completely oxidized. Surface oxidation can be considered as important cause for the coercive force loss of powders. Due to the presence of open pores or microcracks, the oxidation rate of massive bodies can be increased. In the second case because of high tendency of rare earth elements to absorb hydrogen at high temperature and releasing hydrogen from pores during cooling [45], sample was bloated and eventually destroyed. For the third 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 have been discussed elsewhere, when studying heating rate of Samarium Cobalt magnet [44]. 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 [51]. In order to obtain appropriate heating rate, the thermal process of Samarium Cobalt 2:17 series was conducted in three different conditions. The temperature of the samples A, B and C was raised from room temperature to 1200˚C in 190, 130 and 70 minutes respectively. They were kept at this temperature for 1 hour. Figure 3 showed that the samples 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.

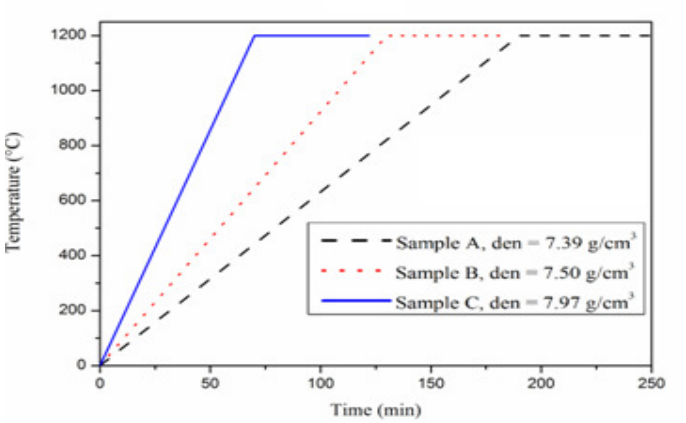

Fig3. Effect of heating rate on final density of Samarium Cobalt 2:17 series, sample A, B and C

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 [51]. 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 1180 to 1250˚C for 1 hour at same conditions.

In the first case, sintering process took place by heating the sample to 1250˚C. The sample lost its shape totally and the density was 7.05 g/cm^3. In the second case, sintering took place by heating the sample to 1220˚C. The sample deformed from its original shape and the density was 7.75 g/cm^3. In the next case, sintering took place by heating the sample to 1205˚C. In addition, only the edge of sample lost their original shape and the density measured 7.90 g/cm^3. Afterward, for the next one, sintering took place by heating the sample to 1195˚C. The sample retained its original shape and the density increased to 7.97 g/cm^3. Finally, for the last case, sintering took place by heating the sample to 1180˚ C. In this sense, the sample also retained its original shape but the density decreased to 7.91 g/cm^3. One can see from fig. 4 that in low sintering temperatures, high density has been obtained. By increasing sintering time and temperature, grain size increases more than the single domain size. The sample which sintered at 1195˚C had higher density than all samples, so it can be concluded that the best sintering temperature for Samarium Cobalt 2:17 series is 1195˚C.

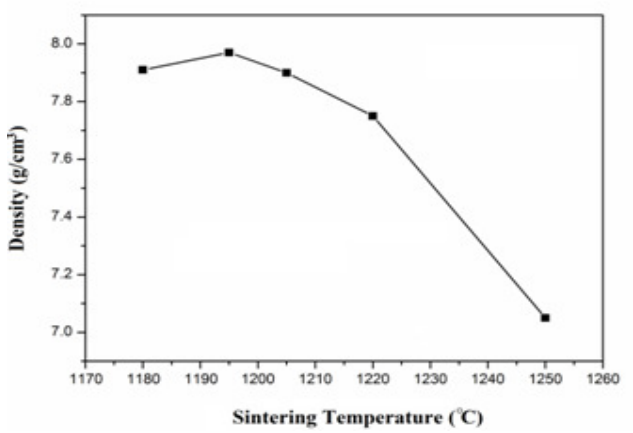

Fig4. Final density of Samarium Cobalt 2:17 series based on sintering temperature

3.6. Effect of homogenization temperature on final density

The purpose of homogenization is to produce highly supersaturated single phase alloy. The solution temperature has a main effect on coercivity of Samarium Cobalt magnets. The relationship between diffusion coefficient D and temperature T can be described as in Eq.(1):

$$D=D_0e^{-Q/RT} \quad (1)$$

Where, D_0 is a constant, Q is activation energy and R is gas constant. With the same solution time, higher T leads to higher D, which is beneficial for the solution of different phases and is easier to obtain a highly supersaturated single phase.

Homogenization process took place in order to make a fully $Sm_2(CoFe)_{17}$ matrix in the sample. Homogenization temperature is usually 15 to 25˚C lower than sintering temperature [45]. To study the effect of homogenization temperature on final density and choosing the best temperature, samples were sintered at 1195˚C for 1hour and kept at five different homogenizing temperatures between 1165 to 1180˚C for another 1 hour.

First sample, homogenized at 1185˚C. In this case density was 7.80 g/cm^3. For the second one, sample homogenized at 1180˚C and the density reached to 7.80 g/cm^3. In the next case, sample was homogenized at 1175˚C and the density increased to 7.93 g/cm^3. In the fourth case, sample homogenized at 1170˚C and the density decreased to 7.83 g/cm^3. Finally, the last sample was homogenized at 1165˚C and the density reached to its minimum at 7.76 g/cm^3. Consequently, a temperature of 1175˚C was found to be the best homogenizing temperature.

3.7. Heat treatments

Sintering and heat treatment are the most important steps in the production of sintered Samarium Cobalt magnets. The aim of sintering is, on one side, achievement of a high density without opened connecting pores whereas, on the other hand, the growth of crystal grains should be avoided. Open pores cause intrinsic oxidation and, therefore, reduce the magnetic remanence. The growth of crystal grains directly reduces the coercivity, making the nucleation of reversed domains easier. The heat treatment after sintering must be chosen to correspond to the chemical composition of the alloy, and its purpose is to obtain a microstructure that improves coercivity.
Magnetic remanence at the end of homogenization process reaches to its required value. In addition, heat treatments should be done in order to increase the coercivity of the samples. In fact before quenching the solid is composed of single 2:17 phase which has low value of coercivity and energy product, so that a heat treatment of 850˚C for 8 hours was employed on the quenched sample to form a multiphase solid with cellular microstructure around the grain boundaries [46]. Afterward, the temperature reduced to 400˚C for 11 hours. Slow cooling may form some deposits around domain walls [47]. Figure 5 represents the whole procedure for heat treatment of Samarium Cobalt 2:17 series magnets.

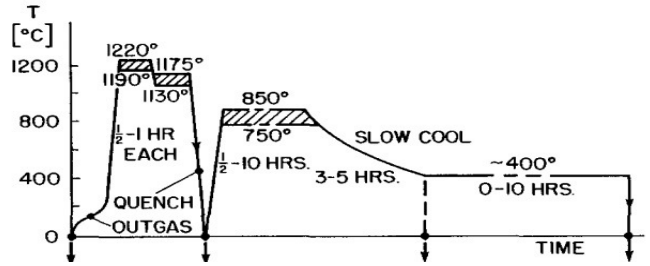

Fig5. Typical temperature profile for sintering and heat treatment for Sm_2Co_{17} [27].

3.8. Phase analysis of Samarium Cobalt 2:17 series

Figure 6 represented the result of XRD patterns of Samarium Cobalt 2:17 series initial composition. It shows that $SmCo_5$ and Sm_2Co_{17} phases exist in the powders.

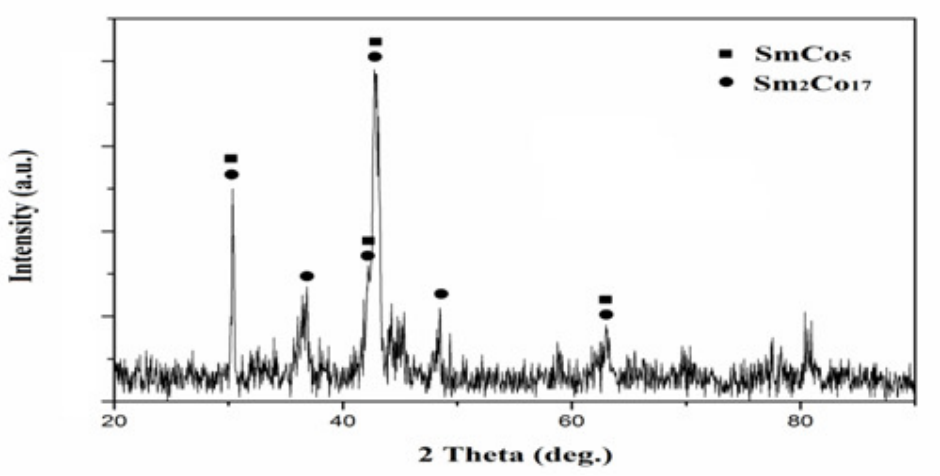

Fig6. XRD analysis of initial composition of Samarium Cobalt 2:17 series magnet

Sintering of Samarium Cobalt permanent magnets was applied for integrating and densification of green bodies. After sintering, homogenization is needed to uniform the microstructure. As can be seen in fig. 7, after sintering and homogenization process represents that Sm_2Co_{17} phase has formed in samples [45]. Fig 8 represents that after heat treating in addition to Sm_2Co_{17} phase, $SmCo_5$ phase also has been formed. The combination of these two phases causes the pinning mechanism of magnetic domain walls and increases the magnetic properties [52].

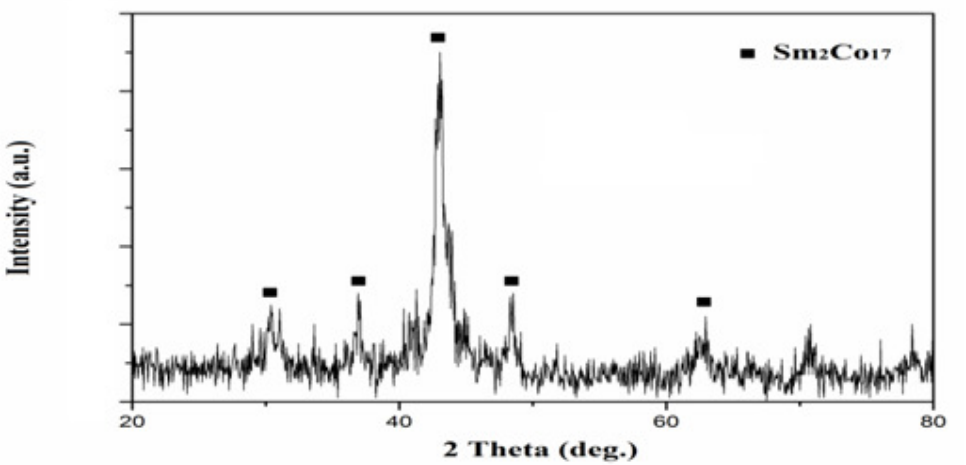

Fig7. X-ray diffraction analysis of Samarium Cobalt 2:17 series after sintering at 1190˚C and homogenization at 1175˚ C before heat treatment

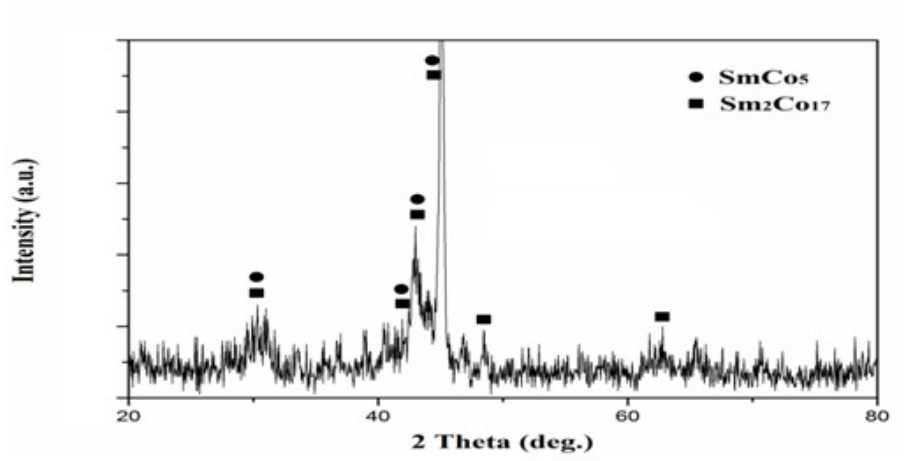

Fig8. XRD analysis of Samarium Cobalt 2:17 series after heat treatments at 850˚C for 8 hours and reducing to 400˚ C for 11 hours. $SmCo_5$ phase has been formed in Sm_2Co_{17} phase

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 2:17 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.

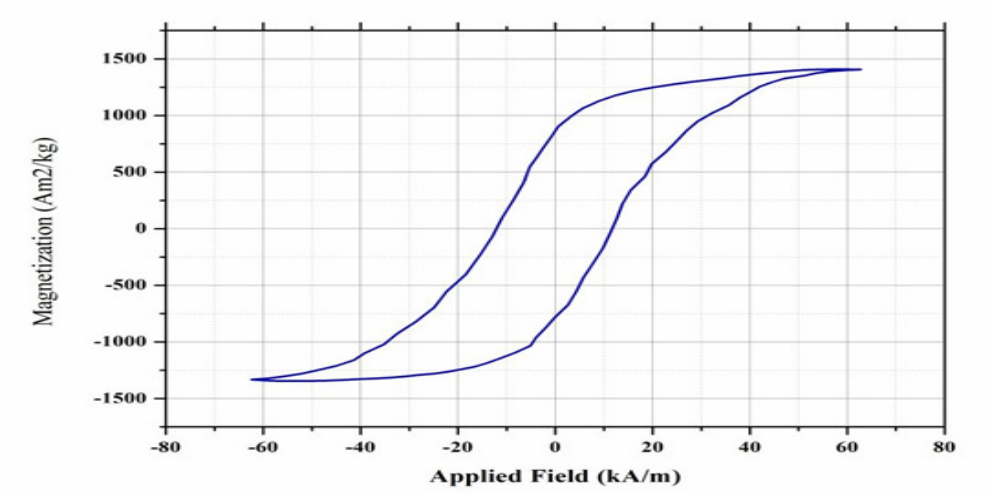

Fig9. Hysteresis curve for a sintered Samarium Cobalt 2:17 series after heat treatments at 850˚C for 8 hours and reducing to 400˚ C for 11 hours.

4. CONCLUSIONS

The density of $Sm_2(Co_{0.67}Fe_{0.23}Cu_{0.07}Zr_{0.03})_{17}$ compound varied by controlling the grain size and the highest density was obtained by using a 3-6 μm green powder. SEM micrographs showed that the shapes of particles were changed to spherical which was found to be more suitable for obtaining high density. The highest density achieved by sintering at 1195˚C with a rate of 17˚C/min under the vacuum conditions. Meanwhile, the sample reached to the appropriate density

by rapid heating at the shortest time. Consequently, a temperature of 1175˚C was found to be the best homogenizing temperature. Heat treatment process in 850˚C kept for 8 hours to form 1:5 phase at grain boundaries and then reduced to 400˚C in the course of 11 hours was done to increase the coercivity. Deposits around magnetic domain walls were formed by applying slow cooling rates.

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