



Research Article

In Search of Magnetic Properties of Samarium Cobalt ($\text{Sm}_2\text{Co}_{17}$) within a Low-Temperature Sintering Process

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Abstract

Samarium cobalt is known as super high density magnetic material with large magnetic anisotropy energy. Samarium-cobalt exhibits manipulative magnetic properties as a rare-earth material which has different properties in a low sintering temperature. It is therefore of paramount importance to investigate samarium cobalt ($\text{Sm}_2\text{Co}_{17}$) magnetic properties in the low temperature sintering condition. $\text{Sm}_2\text{Co}_{17}$, which is utilized in this research, is synthesized via the sol-gel process at sintering temperatures of 400, 500, and 600 °C. Subsequently, the crystallites indicate the formation of a single-phase $\text{Sm}_2\text{Co}_{17}$ on all the samples in all temperature variations. Moreover, the peaks in the X-ray diffraction analysis of crystallite sizes calculated using the Scherrer equation are 17.730, 15.197, and 13.296 nm at 400, 500, and 600 °C. Through scanning electron microscopy, the particles are found to be relatively large and agglomerated, with average sizes of 143.65, 168.78, and 237.26 nm. The functional groups are also analyzed via Fourier-transform infrared spectroscopy, which results in the appearance of several bonds in the samples, for example, alkyl halides, alkanes, and esters with aromatic functional groups on the fingerprint area and alkynes, alkyl halides, and alcohol functional groups at a wavelength of above 1500 cm. The test results of the magnetic properties using vibrating-sample magnetometer (VSM) revealed high coercivity and retentivity in the samples sintered at 400 °C. However, the highest saturation occurs in the samples sintered at 600 °C. At a low sintering temperature (below 1000 °C), samarium cobalt shows as the soft magnetic material.

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1. Introduction

Generally, synthesis of rare-earth materials is carried out using complex methods and at extremely high sintering temperatures (over 1000

°C). Research on samarium cobalt as a rare earth metal has been a prevalent issue in the academia, particularly the investigations of the changes in its magnetic and physical properties at low sintering temperatures (below 1000 °C). Samarium-cobalt has very high magnetic properties, especially in terms of coercivity and magnetization saturation, and is greatly dependent

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on microscopic conditions, such as: the microstructure of the material, magnetic moments, and magnetic anisotropy. The magnetic properties can be controlled by adjusting the amount of cobalt in samarium. The development of nanotechnology has become prevalent and is one of the reasons for the increasing technological development. Researchers have attempted to determine and manipulate the nanomaterial properties according to their fields, one example being the magnetic nanoparticles. Magnetic materials have been widely used, *e.g.* for storage memory, sensor, and drug delivery [1–10]. Moreover, each material element can be developed and manipulated via nanotechnology.

Due to the limited number of rare-earth materials, they are difficult to find. However, they have high values as their properties are better compared with common material properties. Furthermore [11], these materials exhibit a manipulative property, which makes them easier to develop. In short, their properties are easier to change.

The magnetic properties of rare-earth materials largely influence the current technology and can be applied in mechanical, electrical, and medical fields. The examples of rare-earth materials exhibiting good magnetic properties are samarium and cobalt [12,13]. Theoretically speaking, Samarium-cobalt was developed as early as around 1960 based on the works carried out by Karl Strnat and Alden Ray at Wright-Patterson Air Force Base and University of Dayton. In particular, Strnat and Ray developed the first formulation of SmCo_5 . They generally do not only have the same rating in strength of neodymium magnets, but also a higher temperature rating and a higher coer-

civity. However, it is brittle, easily cracked, and broken. Samarium-cobalt magnets have a maximum energy product (BH_{max}) that ranges from 16 Megagauss-Oersteds (MG.Oe) to 33 MG.Oe, which is approx. 128 kJ/m³ to 264 kJ/m³; and their theoretical limit is 34 MG.Oe, about 272 kJ/m³. Sintered magnets on the Samarium-cobalt material exhibit magnetic anisotropy. Consequently, they can only be magnetized within their axis of magnetic orientation. This is done by aligning the crystal structure of the material during the manufacturing process. Samarium exhibits good sensitivity toward the magnetic field and is also temperature-resistant, whereas cobalt has a good magnetic energy storage.

Based on the above reasons, the researchers aimed to discover significant information to increase and manipulate samarium-cobalt via numerous processes. To obtain powdered samples with sintering temperatures of 400, 500, and 600 °C, this research employed the sol-gel and sintering methods. The sol-gel method in the synthesis of samarium-cobalt has several advantages over other methods. In the sol-gel method, there is a phase change from the colloid suspension (sol) to form a continuous liquid phase (gel) in the vaporization process [14]. The two processes in the sol-gel method can increase the magnetic saturation value up to 80–120 emu/g [11,15,16]. Then, the sintering process is added at a certain temperature and in a certain holding time at the peak temperature it is able to produce a material with a large magnetic coercivity reaching 3.43 kOe [17,18].

2. Method and Materials

This research employed the sol-gel method to obtain results and factors influencing the physical and magnetic properties. This paper described how to manipulate and apply samarium-cobalt in numerous fields. In the sol-gel process, the materials were dissolved into molecules. Then, the compound was saturated to decompose the used solution and to form synthesized compounds from molecules to nanopowders [19,20].

After forming the nanopowders, sintering was conducted by heating the materials at 400, 500, and 600 °C under an open-air condition for sample atomization. The formation of crystallites indicated atomization. Then, posttreatment of powder underwent several material characterization tests.

Precursors in the form of cobalt(II) nitrate hexahydrate and samarium(III) nitrate hexahydrate powder were obtained from Sigma-

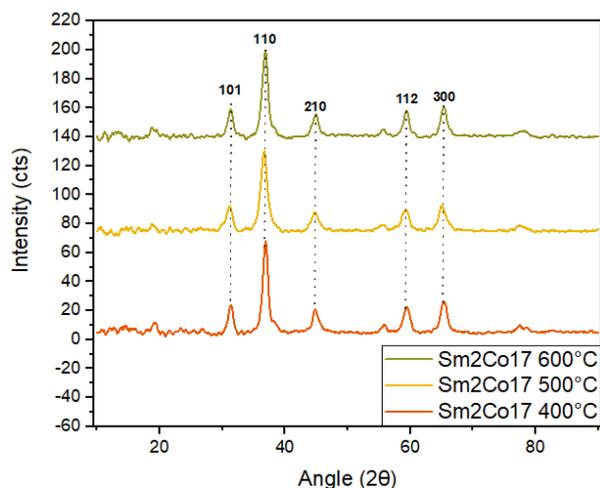
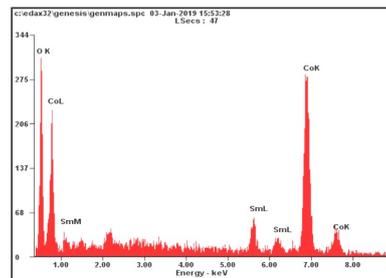
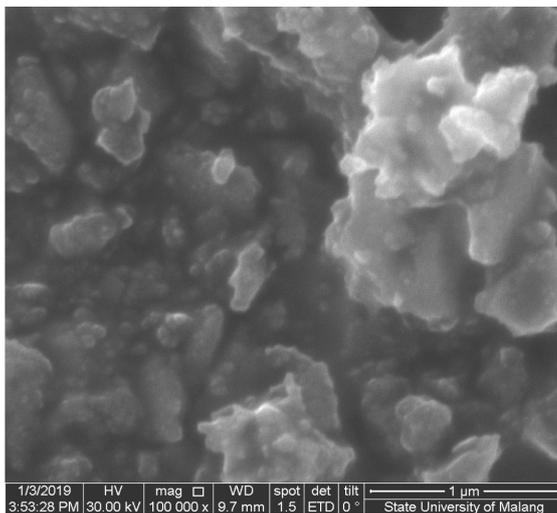
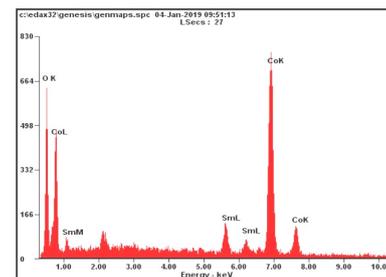
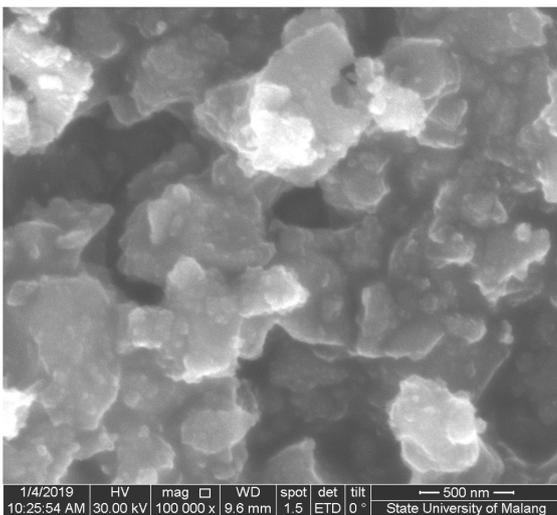


Figure 1. X-ray diffraction analysis result at various sintering temperatures.



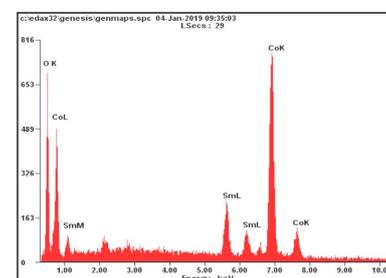
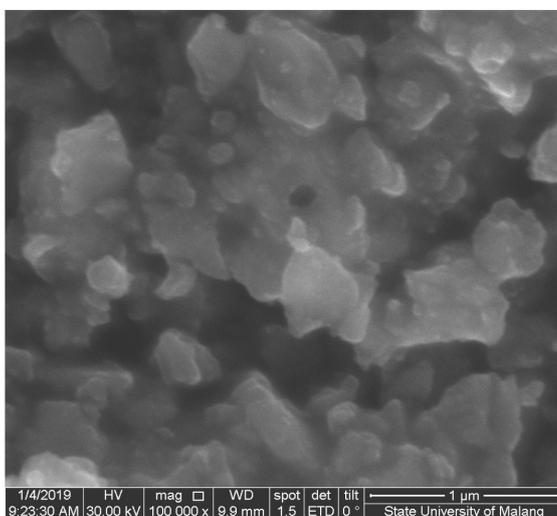
Element	Wt%	At%
OK	23.02	55.43
SmL	14.45	03.70
CoK	62.52	40.87
Matrix	Correction	ZAF

(a)



Element	Wt%	At%
OK	20.10	51.09
SmL	14.81	04.00
CoK	65.09	44.91
Matrix	Correction	ZAF

(b)



Element	Wt%	At%
OK	18.38	50.14
SmL	23.48	06.82
CoK	58.13	43.04
Matrix	Correction	ZAF

(c)

Figure 2 Morphological analysis of $\text{Sm}_2\text{Co}_{17}$ from the results of the SEM/EDX at various sintering temperature: (a) 400 °C, (b) 500 °C, and (c) 600 °C.

Aldrich (99% purity). To obtain $\text{Sm}_2\text{Co}_{17}$, the ratio of the samarium and cobalt precursors was set to 2:19. Ethylene glycol was added in the solution with a 1:3 ratio to the total amount of precursor. The samples were stirred using a magnetic stirrer for 2 h to dissolve the precursors. Subsequently, heat treatment was performed up to temperatures of 70–80 °C until the samples turned into gel. Then, the drying and crushing processes were initiated until the powder samples were obtained.

After the samples were synthesized via the sol–gel method, they were sintered at 400, 500, and 600 °C for 1 h to increase crystal formation. The samples were then tested via the following techniques: X-ray diffraction (XRD) microscopy (PanAnalytical Xpert Pro) with a long-range angle of 10°–90° 2 θ to determine the crystallinity; scanning electron microscopy (SEM)/EDX (SEM with EDAX feature Model FEI, Inspect-S50) at a magnification of 100.000 \times to obtain the morphologies and nano-elements produced; Fourier-transform infrared spectroscopy (FTIR) (FTIR model, IRPrestige-21, Shimadzu) at a wavelength of 500–4000 cm to find the functional groups and bonds; and vibrating-sample magnetometry (VSM) (vibrating-sample magnetometer model, OXFORD, VSM 1.2H/CF/HT) up to 1 tesla of magnetic force to determine the magnetic coercivity, retentivity, or saturation in all the samples.

3. Results and Discussions

The XRD graphics presented below demonstrates that all the samples have five peaks: (101), (110), (210), (112), and (300). In other words, they formed a single-phase $\text{Sm}_2\text{Co}_{17}$ with crystallites. In summary, the temperature

increases during heat treatment which is associated with the crystallite size of the nanoparticles is trivial. Heat treatment can improve crystallization, which, in turn, causes the grain size to increase [21]. The increment of grain size occurs as the influence of cobalt phase characteristics and the similar results also appeared in previous studies of cobalt crystallites [22,23]. Moreover, the lattice parameters of each sample have similar values and do not significantly change the temperature variations in the sintering process.

Figure 1 shows that the cubical structure was observed at the highest peak (110). To measure the size of the crystallites, the Scherrer equation [24–26] was utilized as shown as Table 1. The samples heated at 400, 500, and 600 °C had crystallite sizes of 17.730, 15.197, and 13.296 nm, respectively. The decrease of crystallite size happened since there is a diminish grain in special condition, whereas the sintering of the powders with nanoparticles dimensions is performed. When sintering is performed with different stress, the inducement evokes different strain responding mechanism.

From the results of the SEM/EDX, Figure 2(a) presents the samples sintered at 400 °C; Figure 2(b), the samples sintered at 500 °C; and Figure 2(c), the samples sintered at 600 °C. Generally, all the samples had similar morphologies. The nanopowders were found to be agglomerate and forming clusters that contain nanobulks. Such occurrence indicated that the internal magnetic force of $\text{Sm}_2\text{Co}_{17}$ is sufficient to enable bonding among the particles. Moreover, the emergence of some large-sized particles was due to particle unification during sintering [21]. The average sizes of the particles were 143.65, 168.78, and 237.26 nm at 400, 500, and 600°C, respectively, which were calculated using the ASTM standard method.

The results of the FTIR presented in Figure 3 reveal several bonds in the samples. The discovery of alkyl halides, alkanes, esters, and aromatic (C–Br), (C–H), (C–O), and (C–C) functional groups in the fingerprint area found at a wavelength of 0–1500 cm. Moreover, (C=C), (=C–H), and (O–H) bonds, which are alkynes, alkyl halides, and alcohol functional groups, were observed above the wavelength of 1500 cm. The appearance of these bonds was due to the reduction process from the imperfect synthesis during the sol–gel process, thus leaving several impurities from the used solution [27]. No varieties in the peaks of the samples were observed, only declining intensity and increasing

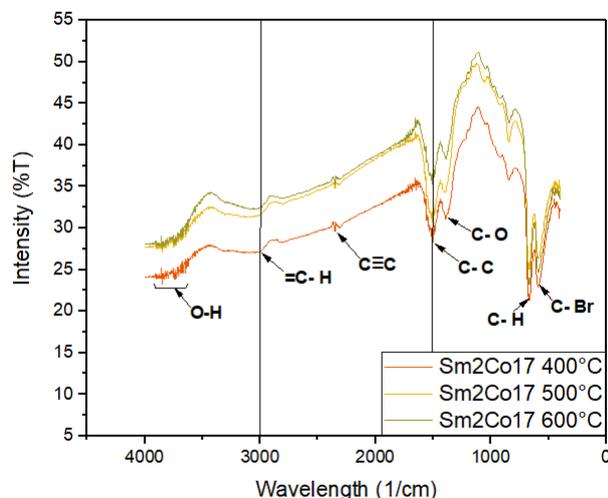


Figure 3. FTIR analysis of $\text{Sm}_2\text{Co}_{17}$ at various sintering temperatures.

The VSM test was conducted to obtain three magnetic properties: magnetic saturation, the ability to generate magnetic force as long as the material obtains the external magnetic field; magnetic retentivity, the ability to store magnetic energy that is converted to internal magnetic force after obtaining the external magnetic field; and coercivity, the value that determines the amount of magnetic field required to diminish the internal magnetic force from a material.

Figure 4 demonstrates the highest saturation value from the samples sintered at 600 °C, namely, 0.457 emu/g, and classified as superparamagnetic. However, retentivity and coercivity only appeared in the samples sintered at 600 °C, with 0.003 emu/g and 0.015 T, as presented in Table 2. This occurrence was induced by the imperfect formation of crystallites at 500 °C and 400 °C. The results also called by non-hysteresis condition with zero coercivity and retentivity, which indicated that the sample ex-

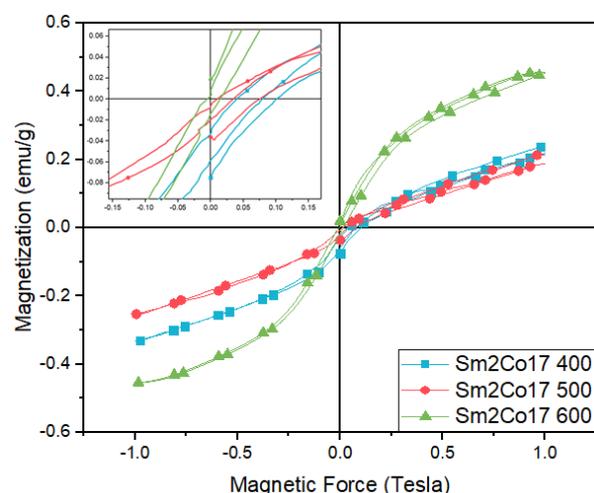


Figure 4. Hysteresis curve comparison of $\text{Sm}_2\text{Co}_{17}$ at different sintering temperatures.

hibits an isotropic characteristic without a preferential axis [27]. Moreover, it indicates that under a normal sintering temperature, the material temperatures of 500 °C and 400 °C exhibit ferromagnetic properties with a magnetic saturation of 0.219 and 0.239 emu/g [27–29]. In the magnetic nanoparticles, the particle size significantly influences the magnetic strength [18]. The particle size also greatly influences the magnetic force in the magnetic nanoparticles [18]. Even though the sintering process causes crystallization, the size of the particles will increase [21] and cause lower magnetic properties compared with bulk alloy materials.

There are 3 theories about the phenomena of changing magnetic behavior that take place. First, there is super-exchange interaction which is a change in the magnetic moment caused by a change in the direction of the electron spin because it is affected by atomic elements which have a more dominant spin moment when the formulas of the two elements are combined with the composition different. This causes the material to be ferromagnetic and paramagnetic when it is not energized by an external magnetic field. The mechanism is shown in Figure 5.

Second, there is hyperfine coupling [30,31] which occurs because during the heating process the atoms are reactive with random movements causing some of the electron spins of the outer shell of the atom to have their own magnetic moment direction and affect the magnetic moment of the atomic nucleus. This causes the material to be able to turn superparamagnetic and antiferromagnetic when it is not energized by an external magnetic field.

The third is the Zeeman Effect [32,33] which has 2 types, the first is the effect of an external magnetic moment which only affects the atomic nucleus, causing the magnetic mo-

Table 1. Crystallinity analysis of $\text{Sm}_2\text{Co}_{17}$ from the XRD result in (110).

Temperature (°C)	Pos. [°2θ]	Height (cts)	FWHM [°2θ]	d-spacing (Å)	Crystallite Size (nm)
400	36.86	49.38	0.5510	2.4381	17.730
500	36.88	68.01	0.4723	2.4371	15.197
600	36.88	55.46	0.6298	2.4370	13.296

Table 2. Analysis of the magnetic properties of $\text{Sm}_2\text{Co}_{17}$ based on the VSM graphics.

Temperature (°C)	H_c (T)	M_r (emu/g)	M_s (emu/g)
400	0	0	0.239
500	0	0	0.219
600	0.015	0.003	0.457

ment to be zero. The second type is when an external magnetic moment is applied which causes a change in the spectral line/electron trajectory caused by an external magnetic field so that it can change the direction of the electron spin rotation so that the magnetic moment changes to follow or reject the direction of the external magnetic field.

4. Conclusions

The XRD analysis revealed the formation of a single-phase $\text{Sm}_2\text{Co}_{17}$ indicated by the crystallite peaks. The analysis also concluded that the increase in temperatures led to the formation of smaller-sized crystallites. Furthermore, the SEM analysis portrayed the particle morphologies of uniting and agglomerate particles that form clusters in all the samples with average sizes of 143.65, 168.78, and 237.26 at 400, 500, and 600 °C, respectively. In addition to the XRD and SEM results, the FTIR analysis discovered several bonds in the samples, for example, alkyl halides, alkanes, esters, and an aromatic functional group at the fingerprint and then alkynes, alkyl halides, and alcohol functional groups above the wavelength of 1500 cm. Lastly, the VSM analysis exhibited the highest saturation was observed in the samples sintered at 600 °C. Through the low-temperature sintering process, samarium-cobalt was found to exhibit the properties of a soft magnetic material.

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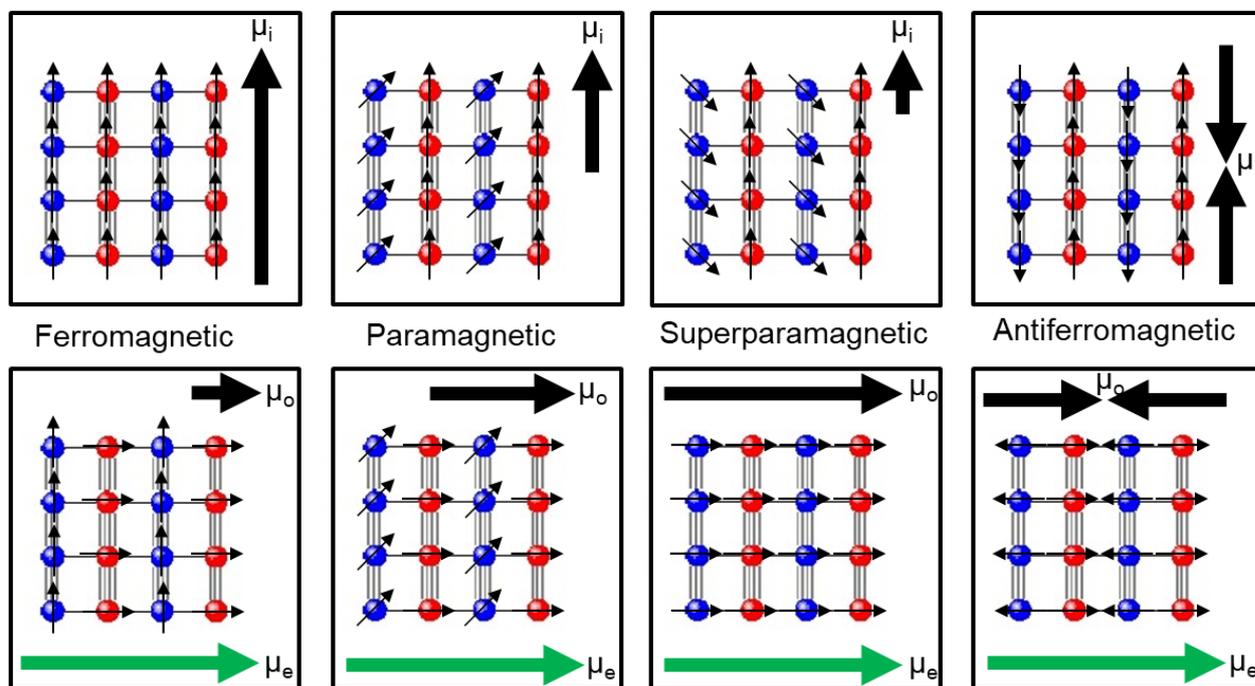


Figure 5. The mechanism of superexchange interaction

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