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Properties of Soft Magnetic Material SmCo_5 Synthesized Using Low-Temperature Sol-Gel Method

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Abstract. Samarium as rare-earth materials has excellent magnetic properties when synthesized and combined with cobalt. This paper aims to synthesized SmCo_5 through the sol-gel method with a variation of sintering temperatures at 400, 500, and 600°C to explore some of the properties and possibilities that can be created from these materials. Then, X-ray Diffraction raised the presence single-phased by occurred the crystallites on all samples in all temperature variations with crystallite sizes that calculated by the Scherrer's equation. The results are as follows 17.730; 15.197; and 13.296 nm in 400, 500, and 600°C samples respectively. The morphology showed in SEM results that the particle shapes and sizes were relatively large and agglomerated. The average size of particles of the three sample in 400, 500, and 600 °C sintering temperature are 153.86; 154,81; 193.56 nm. Some bonds on the samples detected on FTIR result. Those are alkyl halides, alkanes, and esters with aromatic functional groups on the fingerprint area; and alkynes, alkyl halides, and alcohol functional groups in above 1500/cm wavelength. The magnetic properties test result using VSM indicated that the material is classified as soft magnet material with high coercivity and retentivity in samples sintered at 500 °C. However, the highest saturation occurred in samples sintered at 400 °C.

INTRODUCTION

Manipulating the properties of the material make humans develop to a more advanced level of technology. It became easier when nanotechnology taking over. Many researchers were still working to find and manipulate the nanomaterial properties in line with their fields, one example being magnetic nanoparticles. Because in the era of electrical devices used in all subject, magnetic material had the main role to improve. Magnetic materials are widely used, e.g., for storage memory, gas sensor, detector, new energy, and drug delivery [1–10].

When discussing material science, earth-rare material is a material that is difficult to obtain with a limited amount on this earth but has a high value called because it is far better in nature than general material elements. Aside from having several better properties [11], earth-rare materials are also manipulative to develop. So, the ability to change its character is much better.

The magnetic properties of the material have a major influence on technology development that can be applied in all fields such as, mechanical, electronic, and medical. Of all material rare earth elements that are good in their magnetic properties, they are samarium and cobalt [12-13]. Samarium is a rare-earth material that is good in terms of its sensitivity to the magnetic field and its resistance to temperature. Whereas cobalt has good properties in the strength and storage of magnetic energy it receives.

Based on the above reasons, research on samarium-cobalt to obtain important information in improving and manipulating it is urgently needed. Various processes are carried out to see the effects and results obtained. In this paper, we will synthesize using the sol-gel method and do the sintering by producing powdered samples which are sintered at temperatures of 400, 500, and 600 °C.

EXPERIMENTAL DETAILS

This research used the sol-gel process to obtain results and factors that influence the properties. The information contains how to manipulate and apply samarium-cobalt in many fields. The sol-gel process is a process to dissolve the materials into molecules. The compound then saturated to decompose the used solution and to form synthesized compounds from molecules to nanopowder [14-15]. After forming nanopowder, sintering process was conducted by heating the materials at 400, 500, and 600 °C with open air condition to atomize the samples. The forming of crystallites indicated atomization. Post-treatment powder then passed several material characterization tests.

Precursor in the form of cobalt(II) nitrate hexahydrate and samarium(III) nitrate hexahydrate powder were obtained from Sigma Aldrich. Samarium and cobalt precursors ratio were 1:5 to get SmCo_5 . Then mixed with ethylene glycol solution with a 1:3 ratio to the precursor. The samples were stirred using magnetic stirrer for 2 hours to dissolve the precursors. After, the heat treatment was conducted up to 70–80 °C until the samples turned into a gel. Then started the drying and crushing process until obtained the powder samples.

Next step was sintered at 400, 500, and 600 °C with 1 h holding. Samples then tested with XRD (X-Ray Diffraction Model PanAnalytical, Type: E'xpert Pro) with long-range angle 10-90° 2 θ to find the crystallinity properties, SEM-EDX (Scanning Electron Microscopy with EDAX feature Model FEI, Type: Inspect-S50) in 100.000 magnification to get the morphologies and nano-elements produced, FTIR (Fourier Transform Infra-Red Model Shimadzu, Type: IRPrestige21) start from 500 until 4000/cm wavelength to find the functional groups and bonds, and VSM (Vibrating Sample Magnetometer Model OXFORD, Type VSM 1.2H/CF/HT) up to 1 Tesla of Magnetic force to find the magnetic coercivity, retentivity, or saturation from all samples.

RESULT AND DISCUSSION

XRD graphic on Fig. 1 shows all samples formed single-phase SmCo_5 with crystallites with all samples have five peaks: [101], [110], [210], [112], and [300]. The highest cubical structure was found at the highest peak [110]. Scherrer formula [16–18] was used to count the crystallite size. Samples heated at 400 °C had 34.937 nm crystallite size, at 500 °C had 29.922 nm crystallite size, and at 600 °C had 13.089 nm crystallite size, as shown in Table 1. In this case, the more heat treatment temperature causing smaller-sized crystallites. The heat treatment increases the chances of crystallites formation, and in turn causing the particle size to increase [19]. This condition happens because the influence of cobalt phase characteristic, for a similar result also appeared in previous studies of cobalt crystallites [20-21]. Besides, the lattice parameters of each sample have similar values and do not give much change with the temperature variations in the sintering process.

TABLE 3. Crystallinity Analysis of SmCo_5 from XRD Test Result at [110]

Temperature [°C]	Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Crystallite Size (nm)
400	37.32	63.981	0.2400	2.4078	34.937
500	37.04	68.005	0.2800	2.4268	29.922
600	36.92	67.985	0.6400	2.4327	13.086

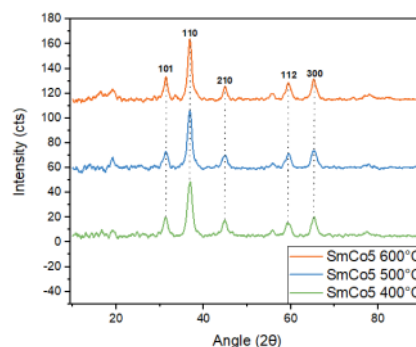


FIGURE 1. X-ray Diffraction result analysis of SmCo_5 over the various temperature of sintering

Based on the results of SEM-EDX in broad outline the three samples have similarities to the morphological form. It can be seen that the morphology of the form of nano powder is close together and attached to form a cluster consisting of bulk. This is due to the ability of the internal magnetic field of SmCo_5 to be sufficient to have a magnetic attraction between the grains so that it is able to bind to each other. In addition, there are several large-size items caused by the union of grains during the sintering process [19]. The average size of particles of the three sample in 400, 500, and 600°C sintering temperature are 153.86; 154,81; 193.56 nm calculated from Fig. 2.

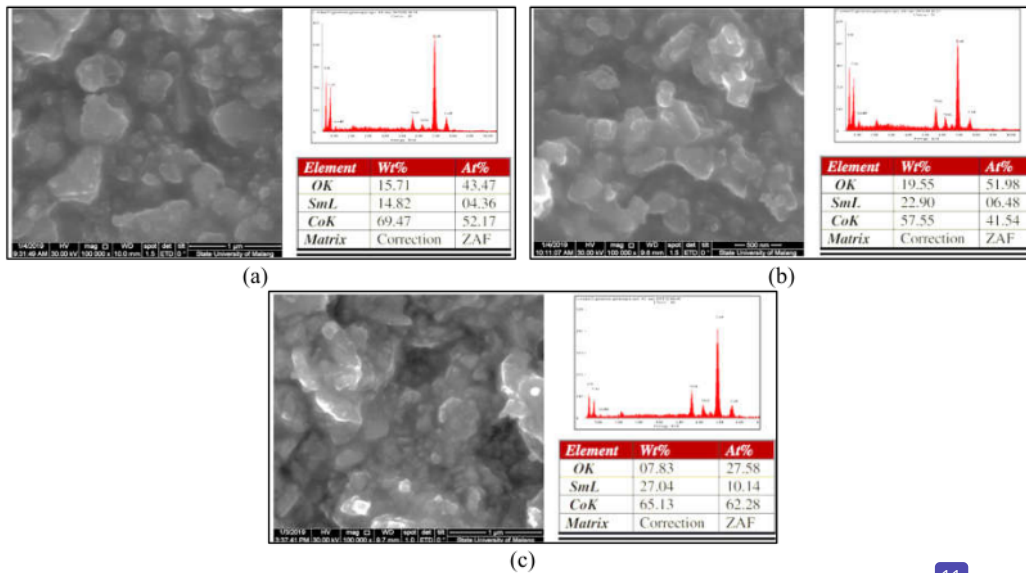


FIGURE 2. Morphological analysis of SmCo_5 From SEM-EDX testing at various sintering temperature namely: a.) 400°C, b.) 500°C, c.) 600°C

FTIR test results in Fig. 3 shows several **110** bands in the samples. Among others, the discovery of alkyl halides, alkanes, esters, and aromatic bonds (C-Br), (C-H), (C-O), (C-C) functional groups on the fingerprint area at 0–1500/cm. Also, (C=C), (=C-H) and (O-H) bonds which are alkynes and alkyl halides functional groups were found above the 1500/cm wavelength. These bonds appeared due to the reduction process from the imperfect synthesis during the sol-gel process, therefore, leaving several impurities from the used solution [22]. There are no varieties in the samples' peaks, only declining intensity along with the increasing temperatures.

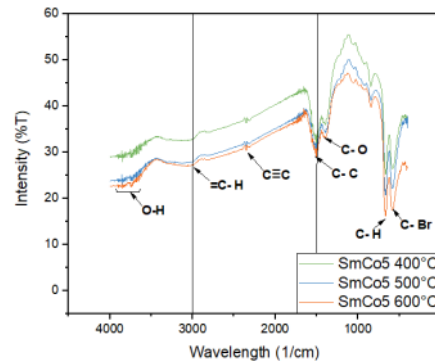


FIGURE 3. The infrared spectrum of SmCo_5 at various sintering temperature analysis

The VSM test result on Fig. 4 has the highest saturation value from the samples with 400°C sintering process that is 0.355 emu/g. However, retentivity and coercivity values only appeared in the samples with 500°C sintering process that is 0.033 emu/g and 0.113 T, as shown in Table 2. Based on the slim S-shaped hysteresis curve, the material has superparamagnetic properties [23-24]. Even though the sintering process causes crystallization, the particle sizes will increase [19] and causing lower magnetic properties compared to bulk materials.

TABLE 2. Magnetic Properties Analysis of SmCo₅ Based on VSM Graphic

Temperature [°C]	H _c [T]	M _r [emu/g]	M _s [emu/g]
400	0.022	0.014	0.355
500	0.113	0.033	0.331
600	0.054	0.010	0.256

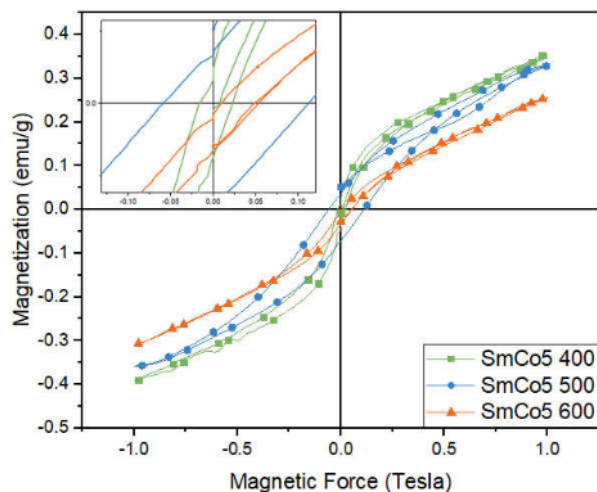


FIGURE 4. Hysteresis Curve comparison of SmCo₅ at different sintering temperature

SUMMARY

The synthesis and characterization SmCo₅ using the sol-gel method has been successfully carried out and producing three samples with heating variations at 400, 500, and 600 °C. X-rays Diffraction results showed a formation of single-phased indicated by crystallite peaks. The analysis concluded that the increasing temperatures caused smaller-sized crystallites. The average size of particles of the three sample in 400, 500, and 600 °C sintering temperature are 153.86; 154,81; 193.56 nm showed by SEM test. Also reveal that the particle morphologies of unified and bonded particles forming clusters in all samples. FTIR test results showed several bonds in the samples, for examples alkyl halides, alkanes, esters, and an aromatic functional group at the fingerprint; and alkynes, alkyl halides, and alcohol functional groups above the 1500/cm wavelength. The soft magnetic properties confirmed by VSM test results follow with the highest coercivity and retentivity in the samples with 500 °C sintering process, although the highest saturation occurred in the samples with 400 °C sintering process.

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