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High Saturation Superparamagnetic Properties of Low-Temperature Sintering of Nickel Oxide

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Abstract. Application of nickel oxide as magnetic nanofluid become more interesting. This research aims to synthesized nickel oxide as magnetic material and analyzed its behaviour under low sintering temperature. This research used synthesized nickel oxide with sol-gel method heated at two temperature variations. The first sample heated at 220˚ C until self-combustion process occurred; and the second sample heated at 80˚ C until it turned into a gel, similar to the general sol-gel process. After that, all samples received sintering treatment at 400˚ C. This research used XRD test resulted in peaks appearances in all samples, indicating all samples were single-phase samples. The first sample showed a crystallite structure size of 72.66 nm before sintering and 39.64 after sintering, whereas the second sample displayed a crystallite structure size of 48.46 nm before sintering and 62.28 nm after sintering. The SEM test result exhibited agglomerated particles and sphere morphology. The FTIR test results presented differences as-prepared sample of solgel method. The functional groups and bonds show broadening peak at O-H bond. Lastly, the VSM test results showed a soft magnetic material with superparamagnetic properties and has high saturation magnetic in the second sample.

INTRODUCTION

Metal transition, such as nickel oxide, remains the attention of researchers. Nickel oxide is a material with excellent p-type semiconductor antiferromagnetic properties [1], [2]; excellent optical, electrical, and magnetic properties [3], [4]; and excellent usages, as good as lithium battery, such as catalyst material, gas sensor, solid-state sensor, electrochromic devices and many more electrical devices [5]–[10]. NiO classifies as a good ion storage material [11]; hence, it becomes a new field to explore its properties and uniqueness particularly as the result of each synthesis process.

There are many ways to synthesize NiO, e.g., solvothermal [12], hydrothermal [13], coprecipitation [14], chemical coprecipitation [9], [15], and the ever-popular sol-gel process [7], [16]. Each method and process created unique characteristics that facilitate its application in various devices.

Based on the above reasons, researchers studied nickel oxide to obtain information on how to improve and manipulate it using several processes then observed the effects and results. This paper synthesized nickel oxide using sol-gel method then sintered at 400˚ C to get a powdered sample.

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METHODOLOGY

This research used the sol-gel process to obtain the influences of its properties and in turn, achieved the information on how to manipulate the material according to the appliances. The sol-gel process is a process that started from dissolving the material into molecules, then saturated the compound to decompose the dissolvent and formed synthesized elemental compound in the form of nanopowder [17], [18].

The next step was to sinter the nanopowder by heating the material at 400˚ C for one hour in the open air with the aim of providing a regular atom formation process marked by the presence of crystallites. After, the resulted samples received characteristical tests.

Experimental setup

Sigma Aldric supplied the precursor in the form of nickel (II) nitrate hexahydrate powder. The admitted ratio of a dissolvent, here used ethylene glycol, was 1:3 with the precursor. This research then used a magnetic stirrer to stir the sample for 30 minutes. Next step was heating the sample divided into two: first sample at 220˚ C and the second sample at 80˚ C. There was a self-combustion in a sample heated at 220˚ C where it released a small explosion during the heating process. The sample heated at 80˚ C turned into a gel. After, there was the drying and crushing process that turned the samples into powder. The samples received the sol-gel process then sintered at 400˚ C with 1 hour holding time to increase the forming crystallites.

This research used the XRD (X-Ray Diffraction) Panalytical model X'pert Pro type device to find out the properties of samples' crystallinity. Then, the samples underwent SEM test using SEM-EDAX (Scanning Electron Microscopy with EDAX feature, FEI model Inspect-S50 type) to find out the morphologies and the components of the resulted compound. After, this research used FTIR test using Fourier Transform Infrared Shimadzu model IRPrestige-21 type to know the functional groups and bonds on the samples. Lastly, VSM (Vibrating Sample Magnetometer) used the Oxford model VSM 1.2H/CF/HT type to get the coercivity, retentivity, and magnetic saturation of the nano samples.

RESULT AND DISCUSSION

Phase Identification

Identifying the phase and crystallite structure in all four samples used XRD test. Figure 1 shows the XRD graph of the samples of six diffraction peaks: [111], [101], [200], [110], [220], and [311]; throughout different synthesis temperatures and sintering process shaped in face-centered cubic. The peaks show that the sintered sample at 400˚ C caused a higher peak. The lattice parameter did not show any significant differences in each sample, or in other words, the samples had similar lattice parameters.

TABLE 1. XRD Test Results Analysis at the Highest Peak (200)

Sample Name	Temperature [°C]	Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Crystallite Size (nm)
1st Unsintered NiO -SC	220	44.43	372.82	0.1181	2.03883	72.66
1st Sintered NiO - SC	220	44.48	1375.77	0.2165	2.03658	39.64
2nd Unsintered NiO -SG	80	44.50	588.74	0.1771	2.03582	48.46
2nd Sintered NiO - SG	80	44.47	1060.53	0.1378	2.03693	62.28

This research used the Scherrer formula below to obtain the crystallite sizes.

$$\beta \cos \theta = k\lambda/D + 4\varepsilon \sin \theta.$$

The samples heated at 220° C showed that the sintering process made the crystallite smaller. However, the samples heated at 80° C showed that the sintering process made the crystallite larger. Table 1 sums the XRD data.

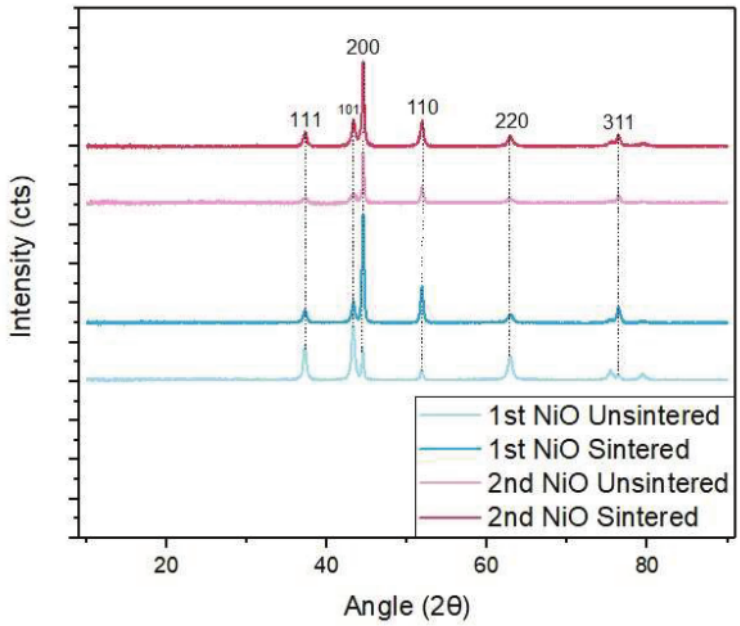

FIGURE 1. Phase Identification of Nickel Oxide by Different Condition

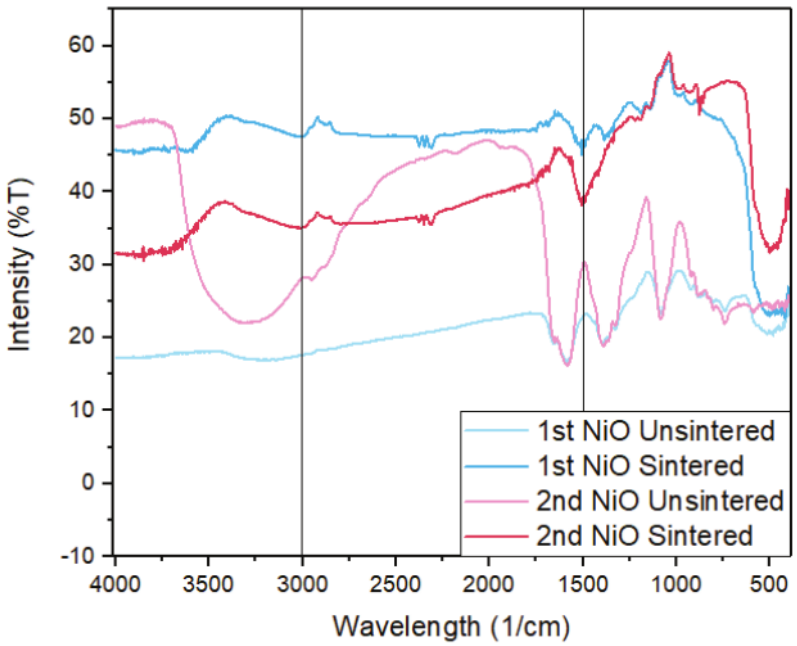

FIGURE 2. Molecular bonding on NiO

This research used the FTIR test to analyze the samples' functional groups. The results show a significant difference between each unsitered sample. However, the results of the sintering process show that the intensity of the 1st sintered sample is higher than the 2nd sintered sample. Figure 2 shows an increment peak from 2nd

sintered NiO which show a stretching bond on O-H peak at 3000-3500 cm-1 and there is an H-O-H bond at position 1630-1500 cm-1 [19] - [21]. This shows that there are bonds of water molecules which have decreased due to the sintering process. 412-500 cm-1 is Ni-O bond which has been increased by showing a sharp peak of the wavelength [19] - [21]. This is because the process is sintered at 400˚ C for 60 minutes.

The morphology results at Figure 3 displays the agglomerated particles due to the absorption of water level from the air that increased the humidity of the sample. The samples also have irregular shape without nucleation and growth reaction.

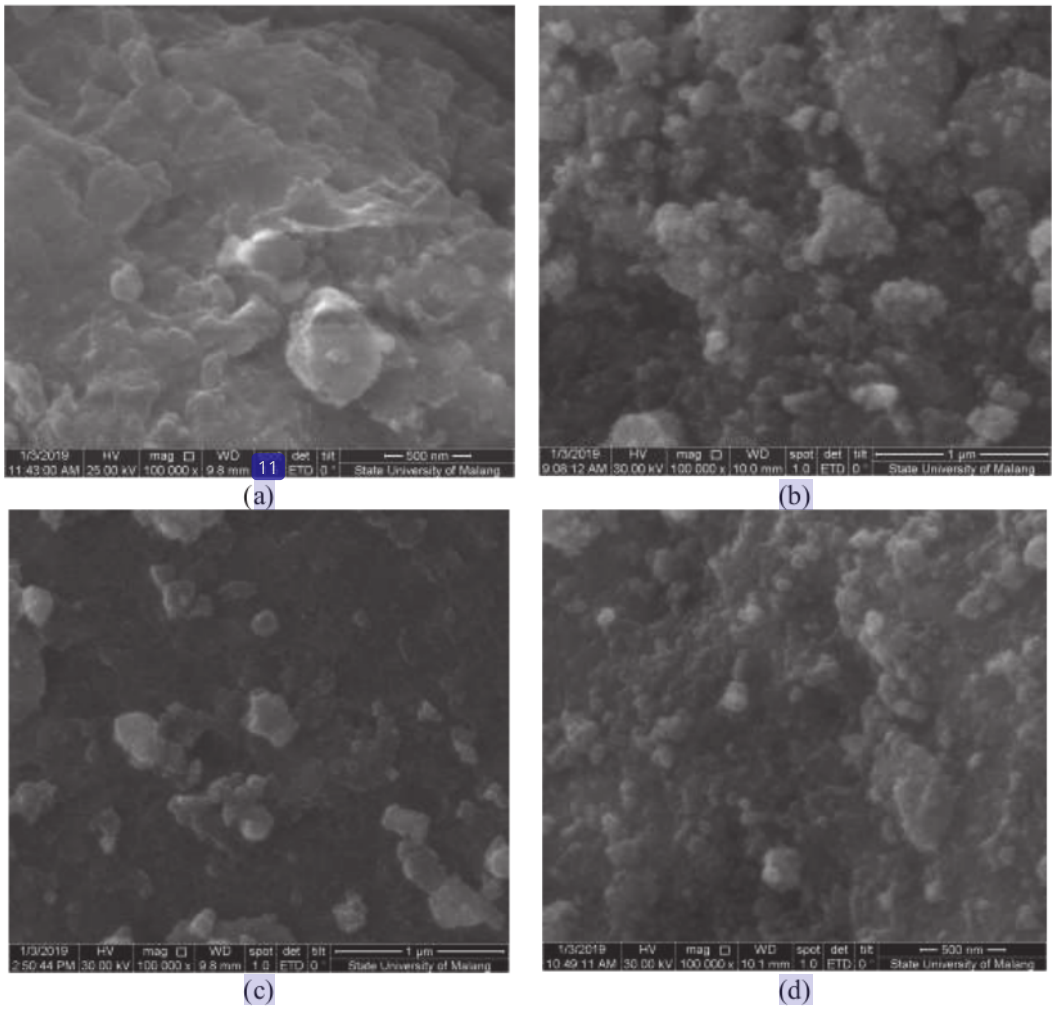

FIGURE 3. Morphology of NiO (a) Unsintered sample, self combustion method, black powder, (b) Sintered sample, self combustion method green powder, (c) Unsintered sample, solgel method, green powder, (d) Sintered sample, solgel method, green powder

TABLE 2. Magnetic properties analysis based on Hysteresis Curve

Sample	Temperature [°C]	Hc [T]	Mr [emu/g]	Ms [emu/g]
1st Unsintered NiO	220	0.039	7.209	18.302
1st Sintered NiO	220	0.054	12.399	23.175
2nd Unsintered NiO	80	0	0	0
2nd Sintered NiO	80	0.052	23.888	57.719

Table 2 displays the magnetic properties analysis. The obtained hysteresis graph through VSM test shows that all samples are soft magnetic with superparamagnetic properties except for the unsintered NiO at 80° C. The 2nd unsitered sample showed no magnetic properties. All samples, including the peaks, were below X-axis line that confirmed the condition as non-magnetic. However, the after-sintering process showed the different result with the saturation reached 57.719 emu/g, and the retentivity increased into 23.888 emu/g, made it the highest in all samples. This case indicated a ripening after sintering.

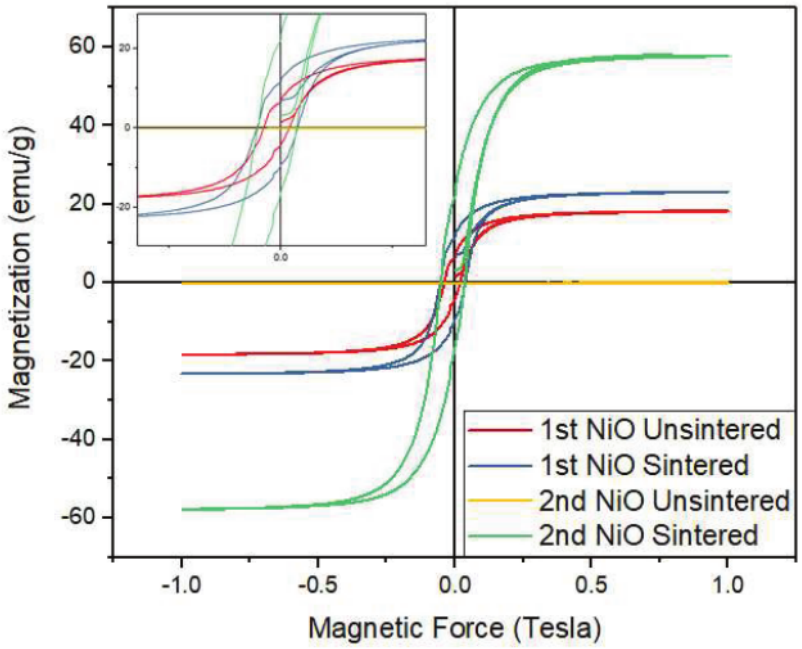

FIGURE 4. Hysteresis Curve from VSM Test Results

CONCLUSION

This research succeeded in studying synthesized NiO using sol-gel method and sintering process. The phase identification, morphologies, functional groups, and magnetic properties testing used XRD, SEM, FTIR, and VSM tests. The XRD test confirmed the single-phase samples with peak shaped as face-centered cubic in the particles. Observed from the peaks, the sintering process at 400° C caused a higher peak with smaller crystallite size. The SEM-EDAX test showed that the samples had agglomerated particles due to the water-bonded interaction in the air that increased the humidity in the sample. The samples also confirmed to have irregular shapes without nucleation and growth reaction. The FTIR test results presented a significant difference between each unsitered sample. However, the sintering process resulted in the higher intensity from the 1st sintered sample compared to the 2nd sintered sample. The VSM test results displayed in the form of the hysteresis graph exhibited the superparamagnetic properties of the samples; the highest saturation and retentivity occurred in the sample heated at 80° C: 57.719 emu/g and 23.888 emu/g. The unsintered sample was non-magnetic and confirmed to be a non-magnetic material.

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PAGE 1

PAGE 2

PAGE 3

PAGE 4

PAGE 5

PAGE 6

PAGE 7