ECMS 2018

Preparation and Identification of $CuCr_2O_4$ Nanoparticles and Investigation of its Microwave Absorption Characteristics at x-band Frequency using Silicone Rubber Polymeric Matrix

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Received: 09 April 2018; Accepted: 16 April 2018; Published: 17 April 2018

Abstract: Lately, silicone rubber because of the desirable permittivity on the one hand and various applications in the implants, membranes, solar cells, sensors, semiconductor devices, high frequency devices, photothermal therapy methods, acoustic metamaterials, and insulator materials on the other hand has attracted considerable attentions. In this research, $CuCr_2O_4$ nanoparticles were prepared according to the sol-gel method and then were identified by Fourier transform infrared (FT-IR), X-ray powder diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and vibrating sample magnetometer (VSM). Results showed that monophase crystal structure with identical morphology of $CuCr_2O_4$ nanoparticles has been synthesized. Finally, $CuCr_2O_4$ nanoparticles and silicone rubber were composited and then microwave absorbtion properties of the $CuCr_2O_4$/silicone rubber nanocomposite were investigated by vector network analyzer (VNA) exhibiting 48.56 dB microwave attenuation for the $CuCr_2O_4$/silicone rubber nanocomposite with 2.6 mm thickness at 10.9 GHz frequency while did have more than 92.99% microwave absorption along the x-band frequency.

1. Introduction

In recent years, merged metal oxides nanoparticles with spinel structure (AB_2O_4) have attracted considerable attention because of its wide variety of applications [1-5]. Copper chromite spinel structure ($CuCr_2O_4$) has attracted large attention due to thermal and chemical stability and various applications such as: photocatalytic degradation, photocatalytic H_2 production, oxidation of carbon monoxide, and water treatment. The thermal decomposition, ball milling, co-precipitation, and hydrothermal are methods used to prepare of $CuCr_2O_4$ nanoparticles [6, 7].

The conventional solid state synthetic reaction is the most common methods used to prepare of $CuCr_2O_4$ nanoparticles. In this method, metal oxides of nanoparticles were used as precursors and then were ball milled and sintered in high temperature. The $CuCr_2O_4$ nanoparticles obtained by this method have a large phase impurities due to lack of fine merging precursors. The size, shape, purity, and cristallinity are the most significant factors that have effect on the properties of nanoparticles [8].

In this research, due to the widespread application of the $CuCr_2O_4$ nanoparticles in recent years, the heat treatment effect on the purity of the crystal structure of spinel nanoparticles has been explored by the Sol-gel method. Moreover, microwave absorption properties of the prepared nanoparticles were studied using silicone rubber as polymeric matrix.

2. Materials and Instruments

All the chemicals $Cu(NO_3)_2.3H_2O$, $Cr(NO_3)_2.9H_2O$, citric acid, and ammonia solution were purchased from MERCK and silicone rubber was parovided from ELASTOSIL® M4503. Wacker RTV-2.

The crystal phases of nanoparticles were studied using Philips X'Pert MPD instrument. The SEM micrographs were obtained by Tescan Mira2. The magnetic properties were investigated by IRI Kashan VSM. The chemical functional groups of samples were explored by the Shimadzu 8400 S FT-IR. Agilent technologies, E8364A revealed microwave absorption properties of the samples.

2.1 Synthesis of CuCr2O4 by Sol Gel Method

Cu and Cr nitrates with stoichiometric ratio of 0.01:0.02 were dissolved in deionized water by magnetic stirrer at room temperature and then citric acid was added in the solution. Afterward, alkali pH was adjusted. The solution was heated and maintained at 90°C until a transparent and viscous gel was obtained. The as-obtained gel was transferred into a furnace and annealed at 750°C and 850°C for 4 h to compare the results.

2.2 Preparation of the Microwave Absorbing Sample

The CuCr2O4 nanoparticles were dispersed in the silicone resin and then hardener were added with 20 Wt. %. Subsequently, the nanocomposite was molded to investigate of microwave absorption properties.

1. Results and Discussion

3.1. X-ray Diffraction Analysis

The XRD patterns of CuCr2O4 nanoparticles at 750 and 850 ºC are shown in figure 1. The results indicated that FWHM of (012), (104), (110), and (116) the crystal planes related to the CuCrO2 structure decreased or disappeared by increasing the temperature. All the crystal phases of CuCr2O4 nanoparticles at 850 ºC corresponded to the [34-0424] standard cart and confirmed that the nanoparticles were formed. The size of nanoparticles was calculated 15.5 nm according to the Scherrer equation.

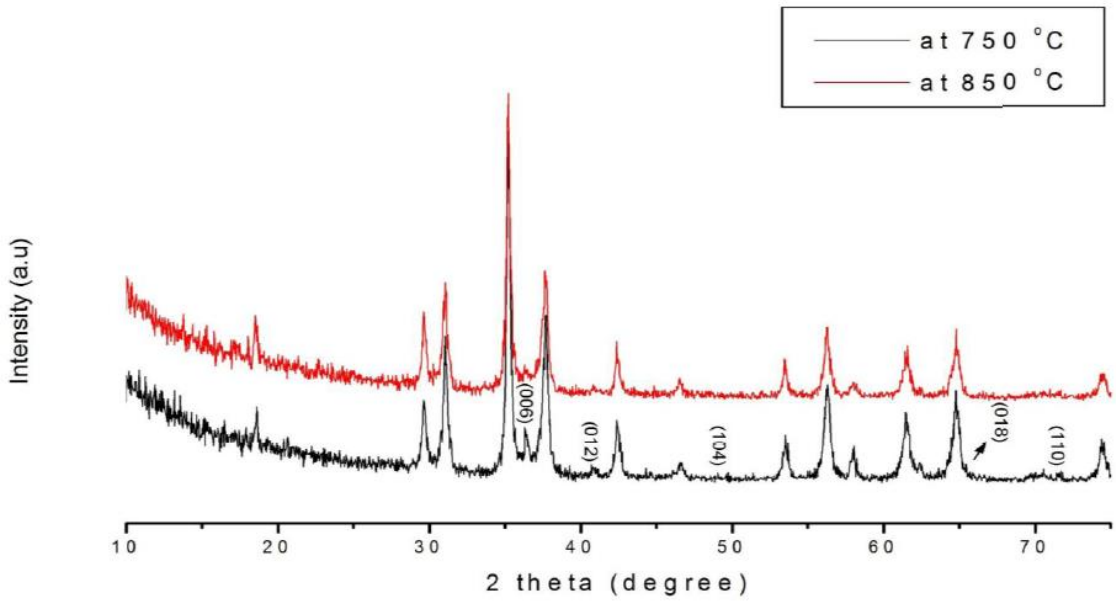

Figure 1. XRD patterns of CuCr2O4 nanoparticles at 750 and 850 ºC

3.2. SEM Images

As shown in figure 2. $CuCr_2O_4$ nanoparticles annealed at 850°C have uniform structure. According to the results, average particle size of nanoparticles was below 100 nm.

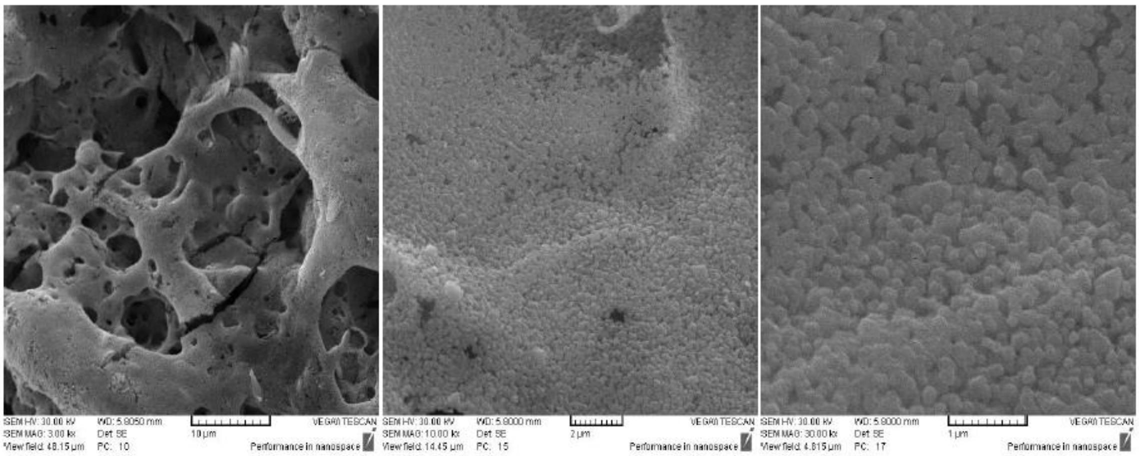

Figure 2. SEM images of $CuCr_2O_4$ nanoparticles at 850 °C

3.3. FTIR Analysis

Chemical structures of $CuCr_2O_4$ nanoparticles were investigated by FT-IR analysis. FT-IR spectrum of $CuCr_2O_4$ annealed at 850°C has been shown in figure 3. The absorption bands at 503.90 cm^{-1} and 612.85 cm^{-1} are related to the Cu-O and Cr-O metal-oxide bond in the finger print region. The peaks about 1600 cm^{-1} and at 3445.86 cm^{-1} are attributed to the bending and stretching vibration of H_2O adsorbed on the $CuCr_2O_4$ nanoparticles surface. The peak at the 2356.24 cm^{-1} is ascribed to the CO_2 that the copper chromite structure adsorbed.

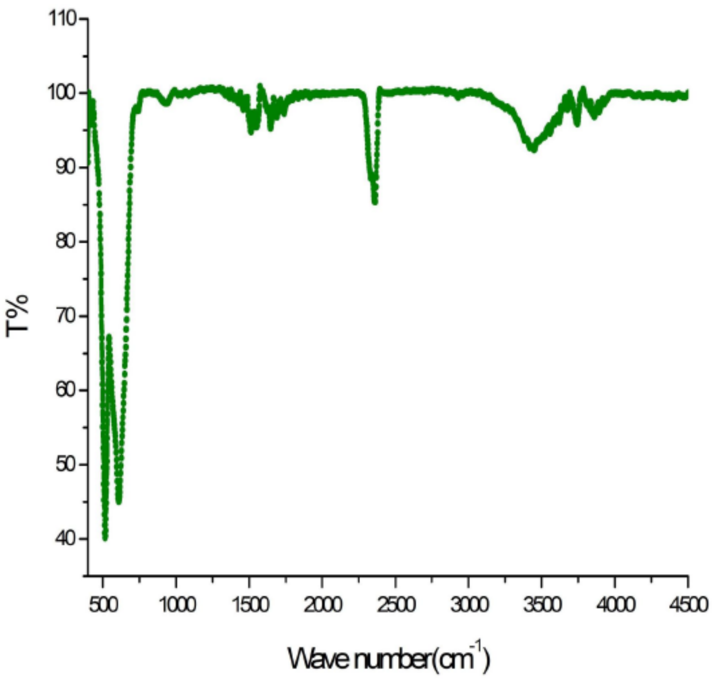

Figure 3. FT-IR spectrum of $CuCr_2O_4$ nanoparticles at 850 °C

3.4. Magnetic Properties

Figure 4. presents magnetic hysteresis loop of $CuCr2O4$ nanoparticles prepared at 850 °C. The magnetic parameters of nanoparticles have been reveled using applied Oersted (Oe) fields from -8.5 kOe to +8.5 kOe. Based on the result, saturation magnetization (Ms), coercivity (Hc), and remanent magnetization (Mr) were 0.21 emu/g, 0.0004 emu/g, and 16.92 Oe, respectively.

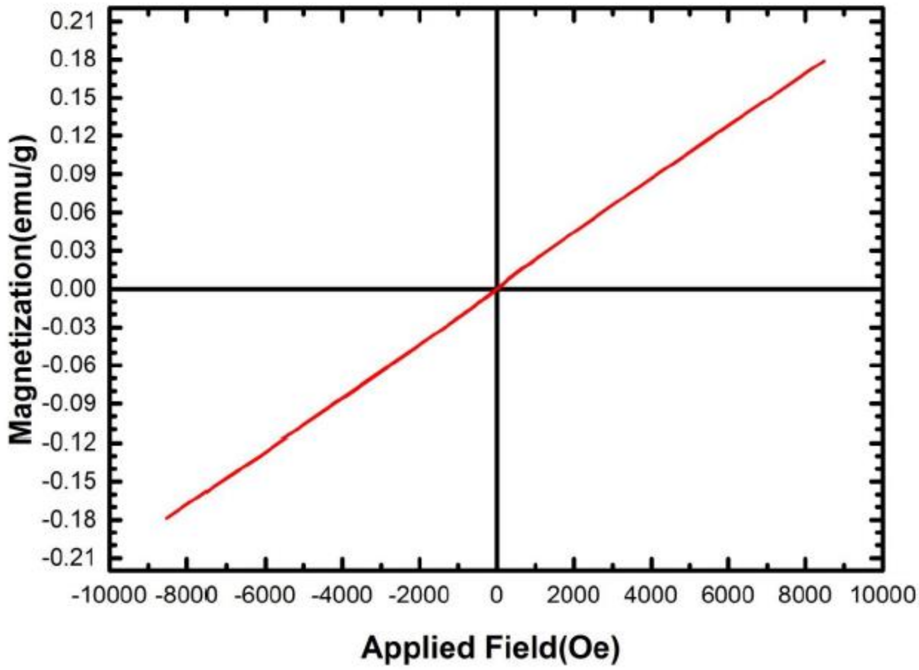

Figure 4. Hysteresis loop of CuCr2O4 nanoparticles at 850 °C

3.5. Microwave Absorption

According to the transmission line theory, permittivity, permeability, as well as diameter of the absorbers are the remarkable factors effecting on the microwave absorption properties[9-13]. Figure 5. Shows microwave reflection losses of the CuCr2O4/silicone rubber nanocomposite with various thicknesses. Results indicated that CuCr2O4/silicone rubber nanocomposite absorbed 48.56 dB with 2.6 mm thickness at 10.9 GHz frequency while did have more than 92.99% microwave absorption along the x-band frequency. The significant bandwidth and microwave absorption properties of the CuCr2O4/silicone rubber nanocomposite were related to proper impedance matching, electron migrating, eddy current loss, magnetic resonance, conductive loss, and interfacial polarization in the absorber medium[14].

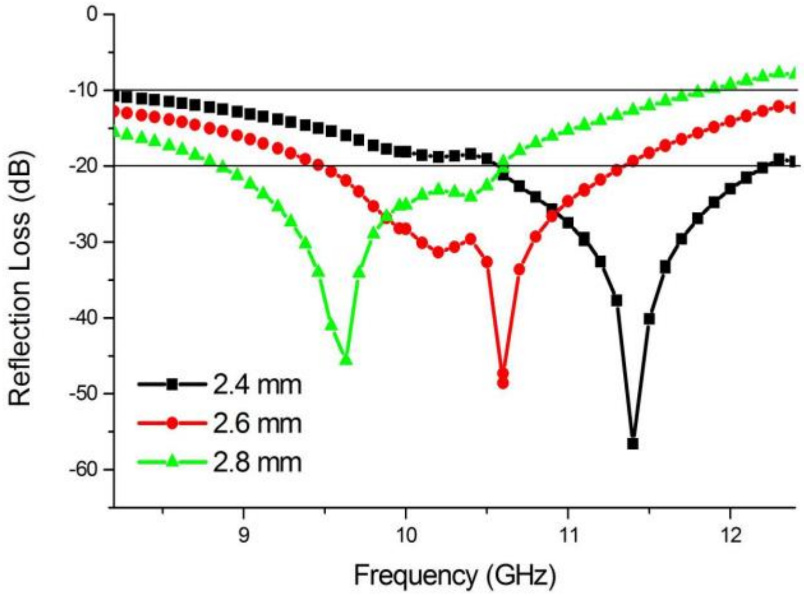

Figure 5. Microwave reflection losses of the CuCr2O4/silicone rubber nanocomposites with various thicknesses

4. Conclusions

Results showed that copper chromite spinel structure was successfully synthesized by the conventional sol–gel method. SEM images showed that uniform structure of nanoparticles was synthesized by this method also the FT-IR spectrum confirmed that nanoparticles were prepared without any organic impurities. Decreased or eliminated FWHM of impurity crystal planes in the XRD patterns demonstrated that annealing temperature did have an important role in crystal purity of CuCr2O4 nanoparticles. According to the results, heat treatment has a large effect on the purity of the CuCr2O4 nanoparticles. Finally, microwave absorption properties of the CuCr2O4/silicone rubber nanocomposite exhibited 48.56 dB microwave absorption with 2.6 mm thickness at 10.9 GHz frequency while did have more than 92.99% microwave absorption along the x-band frequency introduced CuCr2O4 nanoparticles as a promising filler in the microwave absorber.

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