Wave Absorption and Thermal Stability of Nickel Coated Al_2O_3 Core-shell Composite Powder

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Abstract. As a composite cermet material, the nickel coating on the surface of alumina particles has important application value because of its good electromagnetic wave absorption performance. In this study, nickel was deposited on the surface of alumina nanopowders by hydrothermal method and sintering method. The results show that the XRD results of nickel alumina show that Ni-Al_2O_3 nanoparticles has FCC face-centered cubic structure. The grain size is about 50 nm. The absorption characteristics of electromagnetic waves were measured at 2-18 GHz by HP 8722 es microwave network analyzer. The results show that Ni-Al_2O_3 nanoparticles has certain wave-absorbing properties.

1 Introduction

The surface effect, the volume effect and the quantum size effect of nano-magnetic metal powder make it produce many physical and chemical effects different from the bulk material, and have both free electron absorption and magnetic loss, and have excellent absorption properties for high frequency electromagnetic wave[1, 2]. This is because, compared with the traditional particles, nano-particles are easy to generate interfacial polarization and multiple scattering or reflection of electromagnetic waves, because of their small particle size, large specific surface area, high surface atomic ratio and increased dangling bonds, which make them as an important wave absorption mechanism. At the same time, the quantum size effect of nano-materials causes split of the electron energy level of nano-particles, and the split energy level interval is just in the energy range of microwave, which leads to the formation of new wave absorption channels [3-5]. Various metallic magnetic material systems with nanoflake morphology have been studied to enhance high-frequency permeability [6 - 8].

However, the high permittivity and the easy oxidation and corrosion of metallic magnetic nanoflakes inhibit the application in practical microwave absorption. In particular, the high permittivity damages impedance match that is one of the keys in microwave absorption [9]. In this work, considering that Ni-Al_20_3 has high resistivity and long-term resistance to oxidation and corrosion, the Ni-Al_20_3 nanoflakes were coated with thin Ni-Al_20_3 by a simple process to improve the microwave absorption performance of Ni-Al_20_3 nanoflakes. The morphology, micro- structure, phase structure, and static magnetic properties were also investigated in detail. Among them, alumina is used as a group because of its good thermal stability and mechanical properties, and the powder coating obtained by hydrothermal method is simple and easy to obtain, which can be used as a coating in many fields of electromagnetic wave absorption.

2 Experimental part

2.1 Preparation

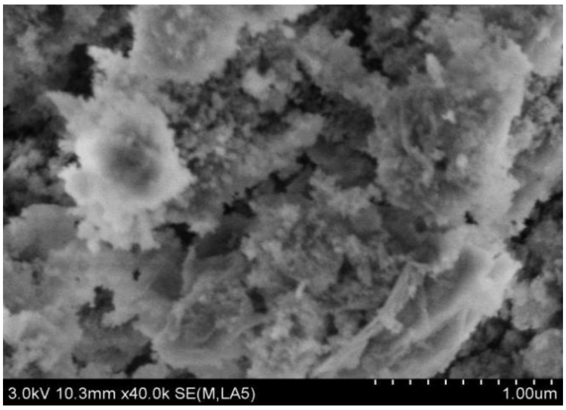
The Ni plating was repeated hydrothermal mixture to increase the Ni content after the Ni-initially Ni-coated Al_2O_3 particles were heat-treated at 1200℃ in N_2. The Al_2O_3 particles having an average particle size of 20 nm are also coated with Co particles. The Ni and Co coated Al_2O_3 moiety was heat treated at 1200℃ in N_2 and coated with Ni and Co particles. A hydrothermal mixture of Al_2O_3 and Ni and Co particles was prepared using a rotary inclined cylinder including a stone basin to compare the micro-structure of the sintered body with the micro-structure of the Ni and Co coated Al_2O_3 particles.

2.2 Characterization

The nano-Al_20_3 powders used in this experiment was about 20 nm with 99.9 % purity, other reagents also had 99.9 % purity. Take 10 ml Ni_2SO_4 (0.2 mol /l) and 2 g alumina powders hydrothermal reaction for 12 hours, and take 10 ml $CoCl_2$ (0.2 mol / l) and 2 g alumina powders hydrothermal reaction for 12 hours as a contrast group. The appearance of powders was studied by field emission scanning electron microscopy (FESEM); X-ray diffraction (XRD) pattern was used to analyze the phases of the powders, and the composition was determined by energy dispersive X-ray spectroscopy (EDS) by S-570 scanning electron microscopy. DSC-Differential Scanning calorimeters and TG-Thermogravimetric Analyzers are test by SDT Q600 Thermogravimetric Analyzer (U.S.A Ta instrument co., ltd) the test was carried out in air at a heating rate of 10 K/ min. Dielectric properties was measured by HP 8722ES microwave net work analyzer in the band of 2-18 GHz [10, 11]. For measurement of the microwave properties, the samples were dispersed in paraffin homogeneously with a sample-to-paraffin volume ratio of 1:4, and then the mixture was pressed into a toroidal shape with an inner diameter of 3.04 mm and an outer diameter of 7.00 mm. The relative complex permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and permeability ($\mu_r = \mu' - j\mu''$) were extracted from the measured scattering parameters. The reflection loss was calculated by using the complex permittivity and permeability.

3 Results and discussion

3.1 SEM and XRD analysis



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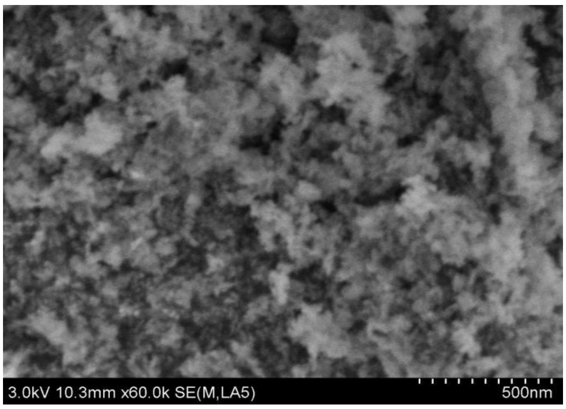


b

Fig. 1 a Low- and b high-magnification FESEM images of the as-prepared Ni·Al_2O_3 and Co·Al_2O_3 products. Ni:Co (2:0, 2:1)

Figure 1 shows morphology photographed by scanning electron microscope (SEM), we can see the surface of Ni / Co-alumina grains, Figure 1 (a) indicated the diameter of the powder is smaller than 30 nm. When the time of hydrothermal reaction prolonged to 12 h, the morphology of nickel layer just consisted of small irregular polyhedral particles, the nickel deposition piled up like small mound (Figure 1 (b)). All Ni-alumina grains are looks regular an fine, The average diameter of those grains probably were about 30 nm, the nickel is evenly distributed on the outer surface of the alumina.

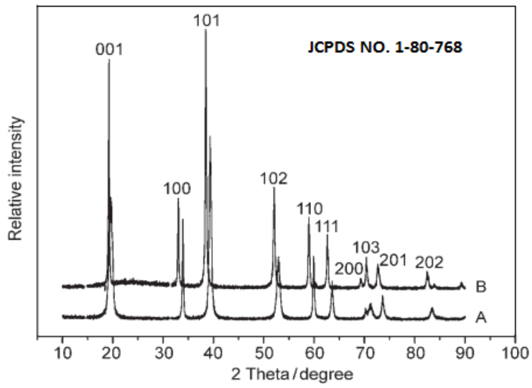
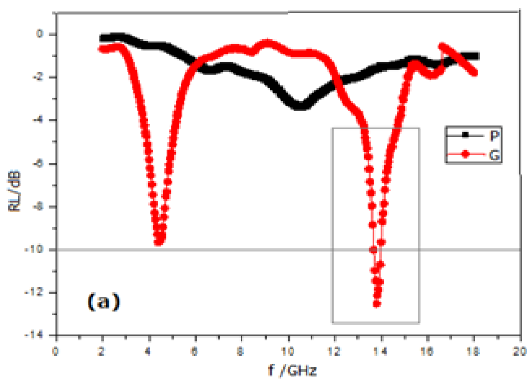


Figure 2. XRD patterns of as-prepared Ni-Al_2O_3 products a nano-particles.

X-ray diffraction (XRD) spectrum (analyzed by Jade 5.0 software) of micro-structured nickel flower-like structure film shows that crystals of sedimentary layer face-centered cubic structure (fcc) have two characteristic peaks at 44.49 ° and 51.85 ° , the lattice plane index marked (210). Since the shape of the crystal mainly depends on the relative growth rate in different directions [12], the peak is sharp, indicating that the prepared product has good crystallinity. No other impurity peaks can be detected, such as nickel oxide or nickel hydroxide, which means the purity of the synthesized nickel crystal.

3.2 Analysis of Electromagnetic Wave Absorbing and dielectric factor loss



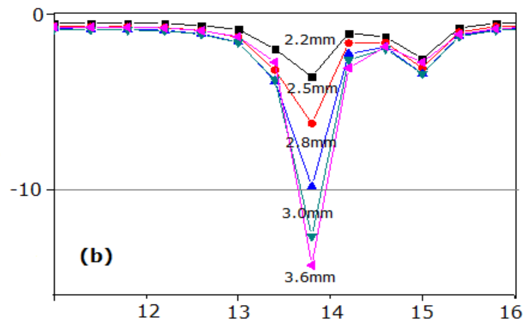


Figure 3. The electromagnetic wave absorption of 99.99% 175 mesh ultrafine aluminum powder (P) as group A and 175 mesh Ni-Al_2O_3 powder(G) as group B were measured by the reflection transmission network method at the test frequency ranged from 8 to 20 GHz. The reflection loss (RL) curves of Al_2O_3, which Sample A and Sample B have the same thicknesses. (b) is the partial magnification of (G) ranged from 11 to 16 GHz.

Finally, the microwave absorption performance should be analyzed to discuss the effect on the incorporation of nano aluminum embedded with Ni nanoparticles. The calculated reflection loss (RL) performance from the electromagnetic parameters (permittivity and permea-bility) of the as-prepared samples is shown in Figure 3. For this sample, the RL maximum is -13 dB at 13.8 GHz when thickness is 3.6 mm with an effective absorption bandwidth (RL≤10 dB) of 1.5 GHz. Additionally, the sample exhibits more excellent absorption performance than ultrafine aluminum powder in GHz frequency band.

However, the maximum RL can reach -13 dB at 13.8 GHz when thickness is 3.6 mm, which limits its application in lightweight microwave absorbing materials. In addition, when the thickness is 2.8 mm, the absorption bandwidth (RL≤-10 dB) ranges from 13.32 to 13.84 GHz. The increasing temperature can cause the skeleton collapse of frame work without the organic linkers, resulting in the attenuation of microwave absorption. Overall, among composite absorbers, Ni based composites have exhibited efficient microwave absorption [13]. It is indicated that the as-prepared materials can obtain good microwave absorption performance with thinner thickness, which corresponds to a promising potential for efficient and lightweight microwave absorbing materials.

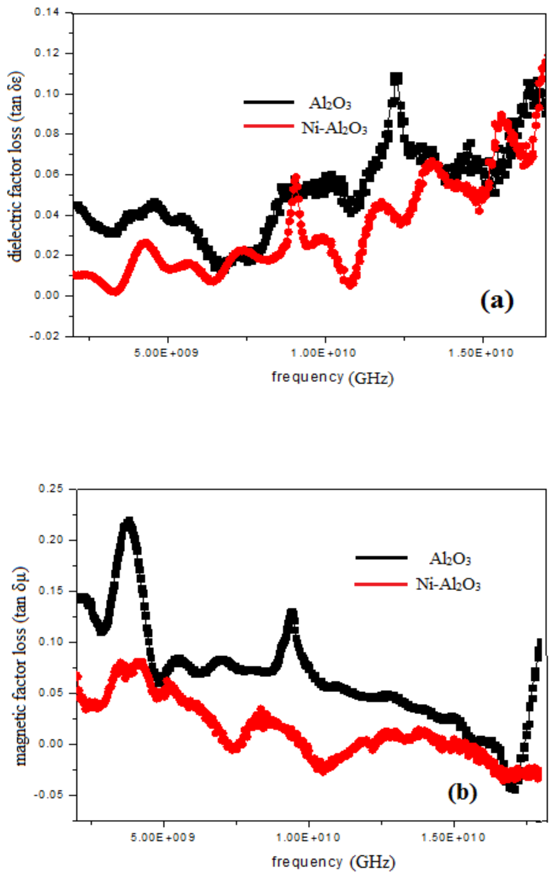


Figure 4. The dielectric factor loss (tan δε = ε''/ε') (a) and the magnetic factor loss (tan δμ = μ''/μ') (b) of Ni-Al2O3 nano-particles and solid paraffin. Attenuation constant of Al_2O_3–paraffin composites versus frequency.

The complex permittivity and complex permeability of Ni-Al_2O_3 nano-particles and solid paraffin prepared by mass ratio of 1: 1 were measured [14]. Solid paraffin is an insulating, non-magnetic material, it is a transparent material for electromagnetic waves. Solid paraffin in the complex permittivity of the imaginary part and the complex permeability of the imaginary parts are 0, solid paraffin in the composite material only play a role in the matrix and binder [15]. It can be concluded that the electrical loss and magnetic loss of the composites are due to the interaction between the Ni nanowires and the nanoparticles.

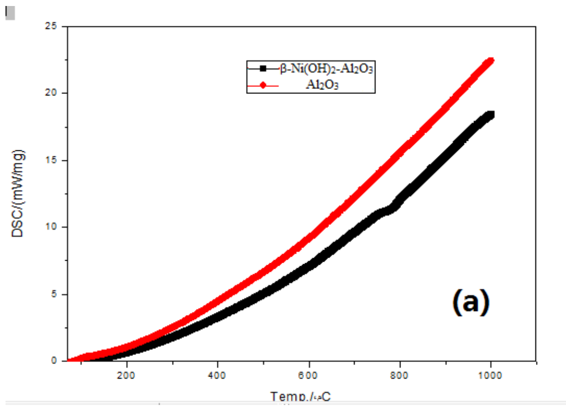
the attenuation constant can be calculated using the following equation:

$$\alpha=\left(\sqrt{2}\pi f/c\right)\times\sqrt{\left(\mu''\varepsilon''-\mu'\varepsilon'\right)+\sqrt{\left(\mu''\varepsilon''-\mu'\varepsilon'\right)^2+\left(\mu''\varepsilon''+\mu'\varepsilon'\right)^2}} \quad (1)$$

The relationship between the real and imaginary parts of the complex permittivity of Ni-Al_2O_3 nanopowder composites in the frequency range of 2-18 GHz is shown in Figure 4. However, visible from Figure 4, the real part of the dielectric constant of Nickel nanowire paraffin composites is much larger than that of Ni-Al_2O_3 / nano powder / solid paraffin composites. From the -Al_2O_3 nanoparticles / paraffin composite complex dielectric factor loss, it can be seen from dielectric factor loss part at 8 GHz and 16 GHz on the frequency range of 2-12

GHz, respectively, there were several peaks. The cause of the these peaks may be due to the quantum size effect of nano-particles, who makes the electronic energy level separation, In addition, they may due to electromagnetic scattering from the thermal motion of the electric field, impurities, defects as well as electronic and electrons interaction, all may be the cause of appearing multiple absorption peaks in the microwave band.

3.3 Thermogravimetric analysis



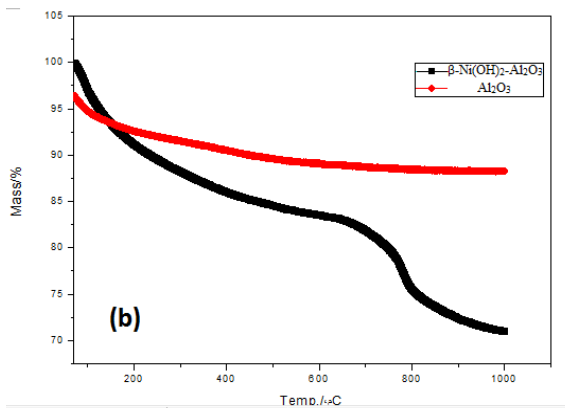


Figure 5. The DSC (a) and TGA (b) curve are related to that conversion process of β-$Ni(OH)_2$ nanocrystals to Ni-Al_2O_3 nanoparticles.

The thermal decomposition process DSC and TGA curve of β-$Ni(OH)_2$-Al_2O_3 was as seen in Figure 5. For Al_2O_3 powder, that onset of mass los in the TG trace occurs at 1000°C, which stand for the peak position in the DSC trace, as seen in Figure 5(a). The total mass loss is slightly less than the theoretical value of 7.4 %, indicating that the thermal decomposition of Al_2O_3 is incomplete. The value of the nanoparticles was 30.2 % corresponding to the formation of pure β-$Ni(OH)_2$-Al_2O_3, but the amount of residual β-$Ni(OH)_2$-Al_2O_3 was lower than the XRD detection limit for Ni-Al_2O_3, as seen in Figure 5(b). The total water loss of Al_2O_3 powder is lower than the total water loss of the Ni-Al_2O_3 nanoparticles, it should be noted that, the specific surface area is become larger because of the composition of smaller fine holes.

3 Conclusions

The structure and morphology of Ni-Al_2O_3 powder were analyzed. the results show that the Ni-Al_2O_3 nano-powder prepared by hydrothermal method has a face-centered cubic single crystal structure and the grain size is about 30 nm. The real part of that dielectric constant of nickel nanowire / solid paraffin composite is much larger than the real part of the dielectric constant of Ni-Al_2O_3 nano-powder / solid paraffin composite, and the imaginary part ε " of the dielectric constant peaks in the range of 3 - 6 GHz and 15 - 17 GHz. The real part of the permeability of the two composites decreases first and then increases with the increase of frequency, and the minimum value is obtained in the range of 8 - 14 GHz. for Ni-Al_2O_3, the electromagnetic wave absorptivity appears two peaks at 5 - 6g Hz, 17 - 18 GHz, 4 - 5 GHz and 16 - 17 GHz, respectively.

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