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A COMPARISON ON RADAR ABSORBING PROPERTIES OF NANO AND MICRO-SCALE BARIUM HEXAFERRITE POWDERS REINFORCED POLYMERIC COMPOSITES

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Abstract

It has been highlighted the usage of barium hexaferrite as a radar absorbing material in the polymer composites in our previous works. With this respect in this study, finer barium hexaferrite powders were obtained, and a comparison has been done on the radar absorbing properties by means of usage of micro and Nano-size powders reinforced composite coatings. Barium hexaferrite powders were synthesized by Sol-Gel method in Nano-size with the hexagonal molecular structure obtained powders were added to a polyurethane resin to interpolate radar absorbing property with different loading levels. Later on, metal substrates were coated with those polymeric composites. Besides characterization tests, such as particle size analysis, X-Ray Diffraction, Scanning Electron Microscopy (SEM), scratch test, radar absorbing test were performed with a Network Analyzer to indicate electromagnetic properties of barium hexaferrite reinforced composites. It was concluded that increasing barium hexaferrite powder amount in the composites increased radar absorbing performance. Additionally, by decreasing BaFe12O19 powder size to Nano scale, improved radar absorbing properties were obtained.
Keywords: Radar absorbing materials; Barium hexaferrite; Nanotechnology; Polymer composites; Coatings.

MIKRO VE NANO-BOYUTLU BARYUM HEKZAFERRIT TOZLARI ILE TAKVIYE EDILMIŞ POLIMER KOMPOZITLERIN RADAR ABZORBLAYICI ÖZELLIKLERI ÜZERINE BIR KARŞILAŞTIRMA

Özet

Önceki çalışmalarımızda baryum hekzaferritin polimer kompozit malzemelerde radar emici olarak kullanımı belirtilmiştir. Bu bağlamda bu çalışmada daha ince baryum hekzaferrit tozlar elde edilmiş ve mikro ve nano-boyutlu tozlar ile takviye edilmiş kompozit kaplamaların radar emici özelliklerine dair bir karşılaştırma yapılmıştır. Baryum hekzaferrit tozları Sol-Gel yöntemi ile nano-boyutta ve altıgen bir moleküler yapıya sahip şekilde sentezlenmiş, elde edilen tozlar radar emici özelliği elde etmek için farklı oranlarda bir poliüretan reçinesine ilave edilmiştir. Daha sonra metal altlıklar bu polimerik kompozitler ile kaplanmıştır. Boyut analizi, XRD, SEM, kazıma testi gibi karakterizasyon tekniklerinin yanında barium hekzaferrit ile güçlendirilmiş kompozitlerin elektromanyezit özelliğinin bir Netwotk-Analiz cihazı ile ölçümü gerçekleştirilmiştir. Kompozitlerde baryum hekzaferrit toz miktarının artırılmasıyla radar emici performansın da arttığı sonucuna varılmıştır. Ayrıca, BaFe12O19 partiküllerin boyutunun Nano seviyelerine indirilmesiyle daha iyi radar absorblayıcı özellik elde edilmiştir.
Anahtar Kelimeler: Radar abzaoblayan malzemeler; Baryum hekzaferrit; Nanoteknoloji; Polimer kompozitler; Kaplamalar.

1 Introduction

Understanding the physical and theoretical underpinnings of radar systems is essential to understanding fundamental operation of radar waves which is transmission and reception. When radio waves strike an object, some portion is reflected, and some of this returned to the radar set. Many properties and phenomena of radio waves are crucial to the operation of the radar system. The Earth's atmosphere plays a central role in radar operation, as it is the medium of propagation for the radio waveforms. The Doppler Effect also plays a vital role in practical radar systems, which is a wave phenomenon where a detected wavelength is different from the source wavelength due to a relative radial motion between the source and observer [1, 2].
The amount of reflection and refraction depends on the properties of the surface and the properties of the matter which the wave was originally traveling through. The ability of various substances to reflect radar pulses depends on the intrinsic electrical properties of those substances. Thus, metal and water are good reflectors. Ice is a fair reflector, depending on the aspect. Land areas vary in their reflection qualities depending on the amount and type of vegetation and the rock and mineral content. Wood and fiber glass boats are poor reflectors. It must be remembered that all of the characteristics interact with each other to determine the strength of the radar echo, and no factor can be singled out without considering the effects of the others [3].
Although scientists are trying to improve radar systems, they also study on developing ways to avoid the radar. This relates to the more stealth technology. For reducing the radar cross section there are some methods used. Shaping is the primary method; if a radar signal hits a surface that is perfectly flat then the signal gets reflected in a single direction. If the surface is not perfectly flat then it gets reflected in all directions, only a very small fraction of the original signal is transmitted back in the direction of the receiver [4]. Hence this real, military aircrafts have sharp edges to distribute the incoming radar waves [Figure 1].

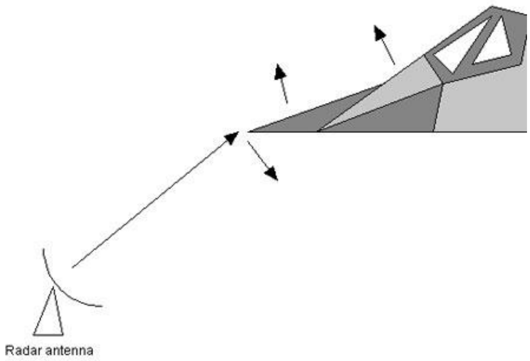
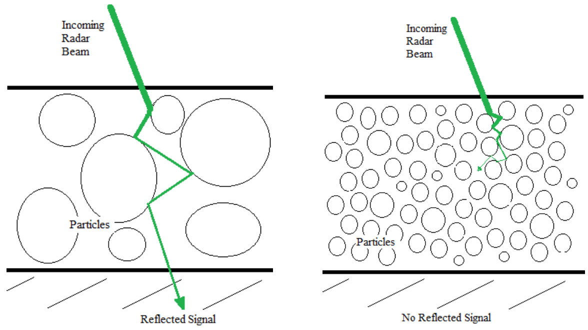


Figure 1. Aircraft with the sharp edges.

The other method is covering the surface with a radar absorbing material which involves distributed loading. Those radar absorbing materials (RAM) have been widely used to prevent or minimize electromagnetic reflections from large structures such as aircraft, ships and tanks and to cover the walls of anechoic chambers [5]. They have imaginary components of permittivity and/or permeability. They are developed from the coatings whose electrical and/or magnetic properties have been altered to allow absorption of radar energy at discrete or broadband frequencies. The coating's absorbency at a given frequency of radar wave depends upon its composition [6]. Research into electromagnetic wave absorbers started in the 1930's, [7, 8] with the first patent appearing in 1936 in the Netherlands. This absorber was a quarter-wave resonant type using carbon black as a lossy resistive material and titanium dioxide for its high permittivity [9-11]. Generally speaking, common dielectric materials used for absorbers such as foams, plastics and elastomers, have no magnetic properties. Magnetic materials exampling ferrites, iron and cobalt-nickel alloys, are used to alter the permeability of the base materials. High dielectric materials, such as carbon, graphite and metal flakes, are used to modify the dielectric properties [12, 13].

In our previous work [14] we studied barium hexaferrit reinforced polymeric composites in micro size in order to be employed as a radar absorbing coating. Barium ferrite with hexagonal molecular structure has fairly large magneto crystalline anisotropy, high Curie temperature and relatively large magnetization as well as chemical stability and corrosion stability [15]. Considering its structure, the numbers of unpaired electrons are occurred in their 4s and 3d shells, due to the spin orientations of the Fe^{3+} ions. Those shells are not fully filled and they lead to magnetic moment. On the other hand, the magnetic moments of barium hexaferrite particles are related with not only the unpaired electrons but even though angular momentum. This momentum represents the product of the body's rotational inertia and rotational velocity. These unique properties of barium hexaferrite reinforced composites significantly increase the microwave absorption performance [16, 17].

In our previous study, we reached 12.13 % as a maximum absorbing value with 20% additive loading level. In the present work, a series of composites were prepared by using a polyurethane matrix with different concentrations of barium hexaferrite powders in Nano size. It was investigated whether any improving obtained in the radar absorbing performance. Thus, it was foreseen that the possibility of the changing the direction of the radar signals would be more likely after hitting the small grains as described in Figure 2.



(a) (b)

Figure 2. Radar absorbing coatings a) with grain size, and b) fine size.

Before starting the coating process, the metal surface was made rough with grinding. It was aimed to obtain the surface intended. By this way, coating will be performed better, and with the sharp edges dissipating the radar signals could be possible as mentioned above. Therefore, in this study, shaping and coating methods were applied together for reducing the radar cross section (or backscattered signal). Synthesized barium ferrite powders were added to the polyurethane resin to interpolate radar absorbing property with different loading levels to see the concentration dependence. Radar absorbing test were applied with a network analyzer.

2 Experimental Procedures

Barium hexaferrite powders were synthesized by Sol-Gel method [14,15], briefly summarized in Figure 3. The used precursors are barium nitrate ($Ba(NO_3)_2$, Aldrich) and ferric citrate mono hydrate ($C_6H_5FeO_7.H_2O$, Fluka), chelating agent is citric acid monohydrate ($C_6H_8O_7.H_2O$) and pH regulator is ammonium hydroxide (NH_4OH). Ferric citrate and barium nitrate were separately dissolved of in citric acid. The solutions were vigorously mixed by magnetic stirrer until the transparent solution was obtained. Ammonium hydroxide was added until reaching of the pH value of the solution to 7 at room temperature and then mixed by magnetic stirrer. Thus, it was aimed to provide homogenous suspension and stable pH condition in the solution after whole solution preparation. The solution was kept in water bath at 80oC for 15 hours in air. Therefore the water in the solution was gradually removed and wet gel with high viscosity was obtained. The wet gel was treated at 180oC for 15 hours in Memmert oven to prepare dry gel. The dry gel was exposed to pre-sintering process at 550oC for 6 hours to evaporate impurities and then was sintered at 1000oC for 5 hours in air in a Ankatest-1 tube furnace (Figure 3).

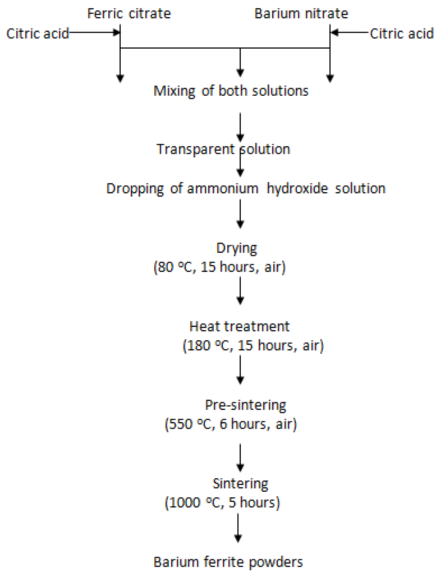


Figure 3. The flow chart for producing barium ferrite powders.

Produced barium hexaferrite powders were added to polyurethane matrix with different loading levels such as 0%, 1%, 5% and 10%. Then metal and glass substrates were coated with those polymer composites and subsequently dried at room temperature in air. The phase analysis of powders was performed with the help of a Rigaku SmartLab X-ray diffractometer. Microstructural cross-sectional areas of composites were examined by using a JEOL JSM-7600F. Electromagnetic parameters of the composites were measured with transmission/reflection method in the region of 8–12 GHz. with a Network Analyzer HP8720D.

3 Results And Discussions

Particle size distribution of the produced BaFe12O19 nanoparticles was represented in Figure 4. As can be seen in the Figure, averagely particle size was found to be about 72.46 nm.

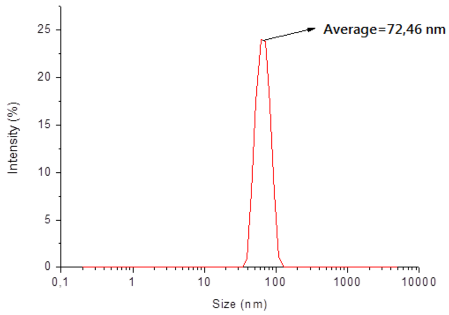


Figure 4. Size analysis of barium hexaferrite powders

Figure 5 clarifies XRD pattern of barium hexaferrite (BaFe12O19) powders produced by Sol-Gel process. A fragment, barium ferrite phase as a major phase and minor phase at the same time as iron oxide (Fe2O3) powder is observed. Reaction metal oxides, it was enough to complete the transformation of the metal compounds. It was indicated that 1000 °C sufficiently high sintering temperature. Heat leads to increased particle growth and on the other hand, it is necessary to consider this while deciding an appropriate sintering temperature [18,19].

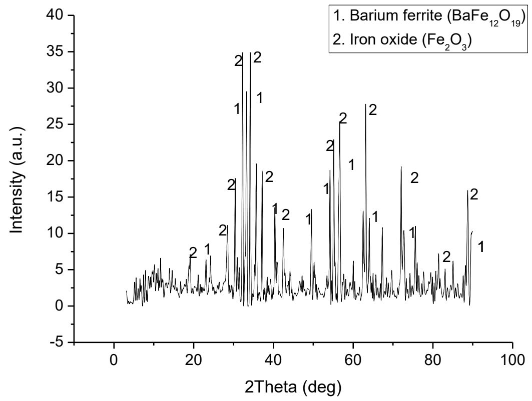


Figure 5. XRD pattern of BaFe12O19 powders produced by Sol-Gel process.

Figure 6 depicts one of the SEM micrographs of BaFe12O19 powder reinforced polymer composite coatings (10% reinforced). Smooth and rough areas on the surface of the coating were indicated, also together with small pores in the structure. A light agglomeration is observed may be due to high surface energy and magnetic interaction of the nanoparticles [20].

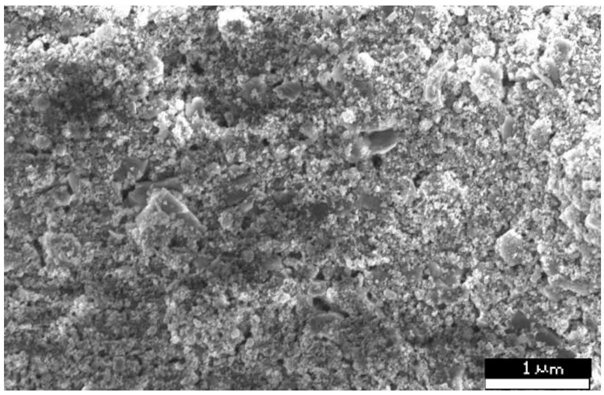


Figure 6. SEM micrographs of BaFe12O19 powders reinforced polymer composite

To estimate adhesion properties of the composites, scratch test has been performed to the composite coatings. With an optical microscope and critical force (Wc) value at which the coating is removed from substrate was determined. After micro hardness value of metal substrate was converted to Brinell Hardness (H) from Vickers Micro hardness by using Standard Hardness Conversion Tables for Metal, the adhesion strength (F) of the coatings was calculated as MPa unit by using Equation 1 [21].

$$F = \frac{H}{\left[\frac{\pi R^2 H - W_c}{W_c}\right]^{1/2}} \qquad (1)$$

It was found that average critical forces of 0%, 1%, 5% and 10% BaFe12O19 added composite coatings are 24.14 mN, 19.12 mN, 19.53 mN and 18.09 mN respectively. It can be seen that the critical forces are small values comparing with the metal coating which may have 120 N critical forces [22]. Moreover, corresponding adhesion strengths are calculated as following; 81.28 MPa, 70.20 MPa, 71.11 MPa and 67.87 MPa, respectively.

For the microwave absorption, network analyzer and coaxial line fixture have been calibrated. The incident power, the reflected and transmitted power were measured. Figure 7 depicts microwave absorption characteristics of the pure polyurethane coating and 1 wt.%, 5 wt.%, 10 wt.% BaFe12O19 reinforced composite coatings. It is clear to see in Figure that there was no any radar absorbing activity in pure polyurethane coating. Whereas, BaFe12O19 reinforced composite coating has a significant absorbing activities. The radar wave absorbing properties of composites were substantially improved after the addition of barium hexaferrite particles; absorbing percentage increases with increasing additive content in the composite coatings [24]. The highest absorbing peak reaches 10.26% at 11.68 GHz with the of 10 wt.% BaFe12O19 loaded sample. In our previous work, for the same loading level (10 wt.%.) max absorption value was 7.32%. As can be seen, in the present experiment, the higher absorption values were reached with same amount BaFe12O19 powders.

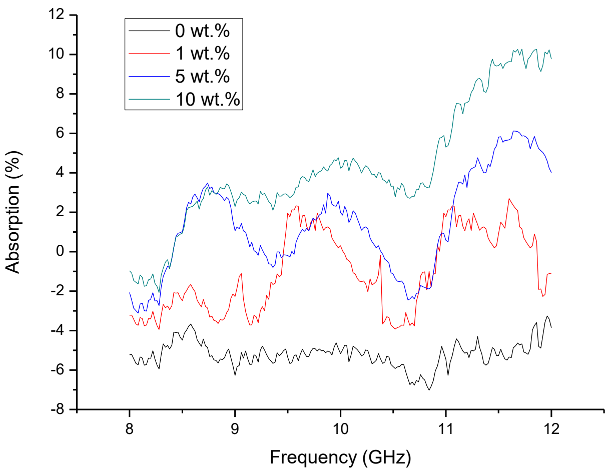


Figure 7. The absorption values (%) versus the frequency range of 8–12 GHz of the radar absorbing composite coated samples.

Taking into consideration above findings, two results can be summarized: first increasing additive content level increasing radar absorbing property. This is the same with our previous works [14]. The second result is that decreasing particle size improves absorbing property comparing with our previous results. The same absorption value has been obtained by getting smaller size of the particles with lower additive quantity. So it is important that usage lower amount of additive will be possible to get the same absorption.

In nanocomposites, by directly influencing the specific surface area of the filler, decreasing particle size increases surface area, and this increases the contact points with the radar signals [25]. Therefore, reducing particle size simply leads to a greater influence of filler-radar signal interactions. As mentioned above, the possibility of the changing the direction of the radar signals would be more likely after hitting the small grains.

To obtain an optimum particle size of the additive, mentioned conditions needs to be taken into consideration. It is obviously seen that to get better properties, particles need to be smaller. Top-down techniques might be expensive for manufacturing Nano-size particles as grinding is an expensive process. However, bottom-up refers to methods where devices create themselves by self-assembly. Sol-Gel is one of the technique bottom-up approaches, and the cost will not be very different for Nano or micro scale synthesizing. On the other hand, mixing is also another important step to get good dispersion, thus agglomeration should not be allowed in the composite.

4 Conclusions

In summary, BaFe12O19 powders were successfully prepared by using Sol-Gel process. Composite coatings were prepared by loading obtained powders and they were characterized after deposition process. In this scope, experimental results were given from structural, microstructural, adhesion and radar absorbing properties of the composite coatings. The results showed that higher magnetic saturation values were obtained from barium ferrite powders reinforced coatings. It was not seen any absorbing activity in % 0 additive content coating. However, in the BaFe12O19 powders reinforced coatings there were significant absorbing activity. Absorption values were increased by increasing additive content. Decreasing particle size to Nano-scale, the same absorption values were obtained with lower amount of additive.

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