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A Study on the Effect of Temperature in Synthesis and Magnetic Properties of W-Type Hexaferrites Barium Nano composites with Radar Wave Absorbing Properties

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Abstract

Absorption and attenuation of electromagnetic waves spread in air by radar or other equipments is considered as a main problem in military and industrial issues. With increasing development of radars and the raise of detection to solve the issue of camouflage, various methods have been offered including active removal, inactive removal, and absorbing materials and shaping deviate waves. To prepare absorbing materials used in aerospace industry, selecting a wide range of magnetic materials, conducting flow components and dielectric materials applied as matrix are regarded as deterministic factors. The present study attempts to investigate the effect of temperature in synthesis and magnetic properties of w-type hexaferrites barium Nano composites with radar wave absorbing properties. For this purpose, some salts such as cobalt, iron, and barium are employed to produce w-type hexaferrites barium Nano powder (Ba $(Co_xZn_{1+x})_2$ $Fe_{16}O_{27}$) using sol-gel auto-combustion via microwave. Compared to the other methods, the applied method has the advantages of more affordability, shorter synthesis time, pure specimen, lack of pollution, homogenized final phase, and lack of the need to high temperatures. The produced Nano powders are then analyzed through X-ray diffraction (XRD) in different temperatures revealing a close phase to W-type. A scanning electron microscope (SEM) is also used to determine the particles` size at Nano scale. Moreover, the vibrating magnetometer device is applied in order to depict the hysteresis loop of produced powders so that the rings are longer at the temperatures of 800 ° C and 950 ° C indicating higher magnetic saturation property.

Keywords: Hexaferrites barium, Hysteresis loop, Nano powder, Cobalt, Radar- absorbent materials

Introduction

With the defensive need of coping with radars and camouflage planes and ground targets, stealth technology could progress greatly in absorbent material field and make it possible to use these materials significantly in a range of wave lengths, low weights, and low reflection coefficient. Stealth technology could present a top secrete plant by simultaneous use of absorbent materials and forming body at different angles. Each company presented various absorbent materials with different applications; for example, in Germany, Dornier Gmbh Company presented a multi-layer absorbent material including various quantities of iron powder, magnetic ferrite powder, graphite, and carbon (Belyaev Y. P.1988). Finally, in America, Lockheed Corp applied absorbent materials across wing and body to camouflage planes involving elements with the dimensions of 0/025-0/05 of encountering wavelengths (Figure 1). Electromagnetic waves have two magnetic and electronic

fields, so the magnetic and electronic characteristics of absorbent materials are expressed through magnetic permeability parameters and dielectric permittivity parameters (Jung-Hoon Oh, Kyung-Sub Oh , Chang-Gon Kim , Chang-Sun Hong and Dong-Min Lee, 2002). To optimize the absorption rate, these parameters should be evaluated. To magnetize these materials, ferrite, iron, cobalt, and zinc should be used; therefore, the present study is aimed to investigate the magnetic properties of materials as the parameter affecting the absorption amount as well as the effect of temperature in this parameter.

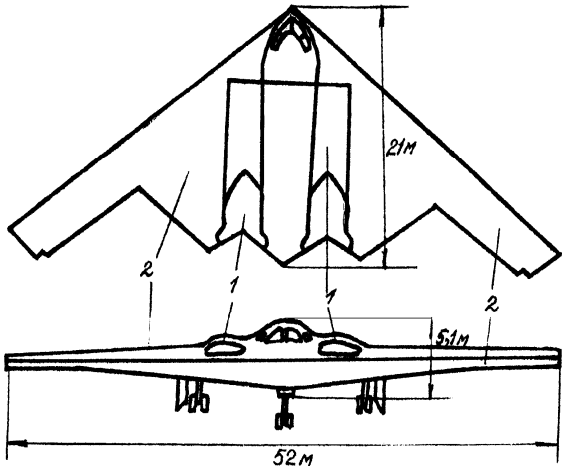

Figure 1- composite absorbent materials in the plane`s structure B-2: 1- carbon-polyimide composite; 2-carbon-epoxy and collar-epoxy

Theory

Attenuation of electromagnetic radiation based on absorption mechanism

The mechanism of attenuation for absorbing materials is carried out in two circumstances including the electromagnetic radiation reflection from the external surface of the object and the energy penetration into the surface of the material and converting to heat in ideal state. To achieve such mechanism, it is needed to use set of numbers with electric and magnetic properties. So magnetic and dielectric permeability of materials is stated in a mixed way:

(1) $\varepsilon = \varepsilon' + i\varepsilon''$

(2) $\tan\delta_{\varepsilon} = \frac{\varepsilon''}{\varepsilon'} = \frac{\sigma}{\omega\varepsilon'} = \frac{1}{\omega\rho\varepsilon'}$

(3) $\mu = \mu' + i\mu''$

Where:

$\varepsilon, \varepsilon'$: Dielectric permeability

$\tan\delta_{\varepsilon}$: Dielectric loss tangent

μ : Magnetic penetration

σ : Angular frequency

ρ : Resistance

Now, the electromagnetic energy reflection from the surface of the absorbing materials is investigated with respect to dielectric and magnetic parameters of ε and μ for an infinite surface of the materials in free space (Figure 2). For this plate, wave's fields on the surface and internal fields of the materials are expressed through Maxwell's equations.

(4) $$\left.\begin{aligned} E_x &= E_m(e^{jkz} + Re^{-jkz}) \\ H_y &= \frac{E_m}{12\pi}(e^{jkz} - Re^{-jkz}) \end{aligned}\right\} z > 0$$

(5) $$\left.\begin{aligned} E'_x &= E'_m e^{jk'z} \\ H'_y &= E_m\sqrt{\frac{\varepsilon}{\mu}}e^{jk'z} \end{aligned}\right\} z < 0$$

E: electric field intensity

H: magnetic field intensity

$k = \frac{2\pi}{\lambda}$ and $k' = kn$ is a multiple from the wave number for the absorbing material.

If the boundary conditions accepted in the surface of the material are applied, a phrase for the reflective energy coefficient will be obtained in the space boundary by equalizing two sides of the equity of the electric and magnetic fields:

(6) $$P_{ref} = \frac{\sqrt{\varepsilon/\mu} - 1}{\sqrt{\varepsilon/\mu} + 1}$$

If $\sqrt{\varepsilon/\mu} = k$, then $P_{ref} = \frac{k-1}{k+1}$

And if $k = z_2/z_1$ while:

Z_2: Impedance of the absorbent material

Z_1: Impedance of the absorbent material space, then:

(7) $$P_{ref} = \frac{z_2/z_1 - 1}{z_2/z_1 + 1} = \frac{z_2 - z_1}{z_2 + z_1} \quad \text{if } z_2 = z_1$$

Thus: $P_{ref} = 0$

The reflection coefficient would be decreased several times obtained from the following relation:

(8) $$R = 10\log[P_{ref}]$$

Where, R is the reflection coefficient from the covering surface.

Considering the relation (7), to receive the minimum reflection, the impedance of the absorbing material should be near to the impedance of the related spaces. So, one method of synthesizing these Ferrite magnets is presented in the present paper.

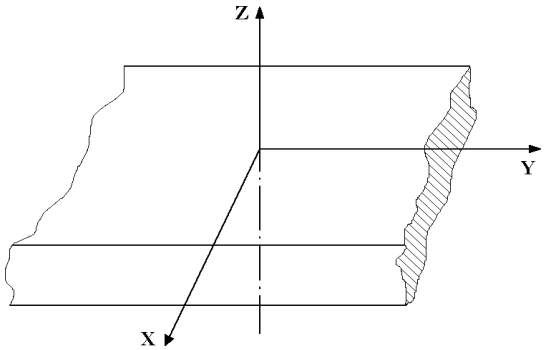

Figure 2- hypothetical infinite plate

Materials and methods

Producing Nano composites specimens

Material selection

To produce W-type Hexaferrite barium Nano composites with the combinations of X= 1, 0/75. 0/25, at Ba $(Co_xZn_{1+x})_2\,Fe_{16}O_{27}$, the following materials are needed:

1- Nitrate barium 99%, %20+0/1632 gr, m= 261/35 gr/mol Ba $(No_3)_2$

2- Cobalt nitrate 99%, 0/1817 gr, m= 129/04 gr/mol Co (No3)2, 6H2O

3- Citric acid 99%, m=192/13 gr.mol $C_6H_8O_7$, 4/12 gr, 1/542 of the relative density, 153c of the melt point

4- Ethylene glycol %99/5 3/35 gm, m=62/7, gr/mol $(CH_2)\,(OH)_2$

5- Benzoic acid %99/5-%100/5 to accelerate the process 0/33 gr, 1/2659 of the relative density, 121/25c of melt point

6- Iron(II) citrate 3/3503 gr, m= 335/03 gr/mol, $feC_6H_5O_{70}$ 5H2 O

7- Zinc chloride 1/3625 gr, m=136/25, gr/mol $ZnCl_2$

8- Acetic acid 4/12 gr

9- K_2CrO_7, to clean the containers

10- Epoxy-resin and hardener have been used as much as 45% of the total powder.

The method of producing specimens

Ba $(Co_xZn_{1+x})_2\,Fe_{16}O_{27}$ synthesis was done using sol-gel auto-combustion via microwave and acetic acid. The amount of Ba $(No_3)_2$ was considered 10-20% more than the normal amount to maintain some residual after evaporation due to low melting point. The amount of salt to produce 335/03g was 0/01 mol (due to limited capacity of laboratory containers) and 0/1817g cobalt, 0/1632g zinc and 0/8453g barium salts were used. Nickel, barium and zinc were used as much as one-sixteen mols. First, barium nitrate and acetic acid were mixed with same quantity and then barium acetate was obtained and then it was mixed with iron citrate. Another combination of salts was synthesized and two liquids were mixed and then some ammoniac was added to the mixture to stabilize PH at 6.5. The liquid then was heated at 70c to obtain sol substance. The obtained sol was shacked for 2 hours and was heated at 110c for 24 hours to dry the gel. The dried gel was ignited (with a red-brown flame) for 15s in 450V power microwave. The burning powder can be calcinated in various temperatures of 600 to 1000c for 2 hours. The hexagonal phase was obtained in the temperature of 800 to 1000c. Then, 55% of the produced powder was mixed with epoxy-resin and hardener. The final powder was distributed on the surface with 1mm thickness and then was baked at room temperature.

Figure 3- a view of the powder ignition

The analysis of XRD

To obtain XRD characteristic, XRD device is used. XRD test reveals the influence of different synthesis temperatures in the curve formation. This method determined the crystal phase. Data are characterized through radiation of Cuk^{α} in 40KV and 20Ma in the 2ɵ=20-70 region and the scan speed of 4º/min. the Fe_2O_3 peaks in 35/685 angel and the peak continues in non calcinated temperature 950c. The phase without calcinations lacks the crystal phase of Ba $(Co_{0.5}Zn_{0.5})_2Fe_{16}O_{27}$, and instead, $Fe_2O_3Fe_2O_4$ is appeared. $Fe_2O_3Fe_2O_4$ has the counter mode. When the calcination temperature reaches 800c we achieve hexagonal. The temperature is very important for hexaferrite Nano composites Ba $(Co_{0.5}Zn_{0.5})_2Fe_{16}O_{27}$. Additional lines and peaks in the diagram may be because of other impurities in synthesized material including the phases of Ba (No_3), Fe_2O_3, γ, and α. M-type phase is also observed in peaks investigation. Therefore, M-type and W-type are exists in a combinational form at 95C by increasing the temperature of the process.

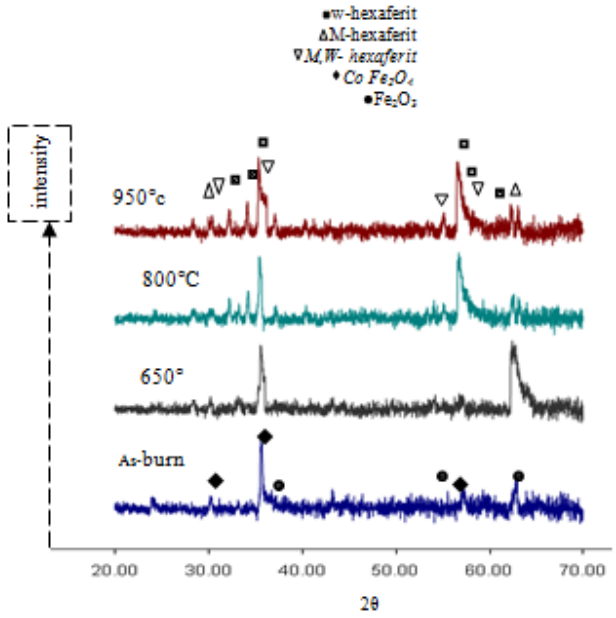

Figure 4- the diagram of XRD hexaferrite barium Nano powder

Results and Discussion

Computation of magnetic parameters affecting the absorption rate

One of the most basic quantities in magnetic is magnetic intensity (H). Applying magnetic field with H intensity is resulted in magnetic induction B. in some cases, B is a linear function of H and the equation (9) is established in the vacuum between these parameters.

(9) B=μH

Where μ_0 is absolute magnetic permeability of the substance and it is not necessarily constant.

Substance becomes magnetic when it is placed in a magnetic field with H intensity and the magnetic induction intensity equals with M^5 .

According to the definition of magnetization, magnetic torque is stated based on volume unit as follow:

(10) $M = m/V$

Where, m is magnetic torque and V id the substance volume.

The relation of three basic magnetic parameters (M, B and H) is stated through the following general equation:

(11) $B = \mu_0(H + M)$

Magnetic permeability is defined as follow:

(12) $X_m = \frac{M}{H} = \frac{B}{\mu_0 H} - 1$

Where

(13) $X_m = \mu - 1$

The value of X_m and its change towards H and temperature determine magnetic characteristic f the substance and forms the substance classification; and when a substance is placed under a magnetic field, the torques of the substance are placed in the direction of the field. When all torques of the substance are placed in the direction of the field, the magnetization is saturated.

Magnetic hysteresis (B_r) indicates magnetic induction which is still remained after applying the reverse field. The necessary negative field to omit magnetic induction is coercivity magnetic force (H_c). Coercivity magnetic force is a magnetic characteristic which is not an inherent characteristic of substance. This parameter highly depends on microstructure of the substance and can be improved by controlling the microstructure. Coercivity magnetic force and saturation magnetization are associated through the following relation (S. I. Park, S. Wlee and C. S. Kim, 1997):

(14) $H_c = 2K / M_s$

Where K is anisotropy constant and M_s is saturation magnetization.

Considering Figure 3, the more the distance between magnetic saturation and coercivity force is in hysteresis loop, the more the magnetic permeability will be. These parameters highly depend on microstructure of the substance; so, the effect of synthesis temperature is investigated in magnetic characteristics and finally in absorption rate of waves.

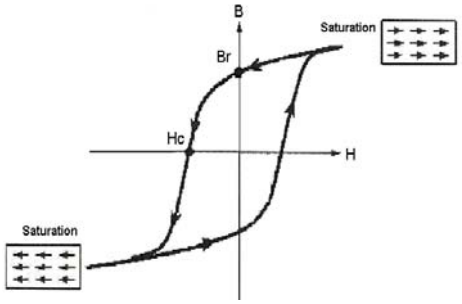

Figure 5- hysteresis loop of a magnetic substance

Measuring the particles` size using SEM

The SEM picture shows the calcinated sample at 950c for 2 hours in the presence of agglomerate. The powder has compressed structure and agglomerate has steady distribution of particles. The average size of the particles is estimated below 100nm. The temperature of the calcination process influences the size of the powder`s particles. Any increase of temperature will increase the growth rate of crystals and atomic diffusion. Therefore, the increase of calcination temperature leads to the formation of powders with larger size particles.

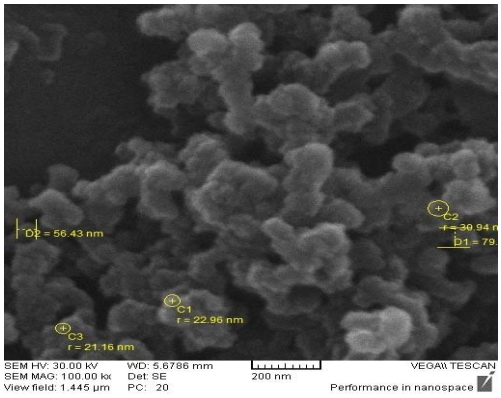

Figure 6- SEM image of synthesized powder with dimensions

Depicting hysteresis loop of hexaferrite barium Nano powder

To determine magnetic hysteresis loop, vibration sample magnetometer device AXS-D VSM50050 was used. Magnetic characteristic was measured at room temperature and with external field of 10 koe. As shown in Figure 7, the powder calicinated at 650° C with 38/86 emu/g,Ms and Hc, 644/30 Oe indicates soft ferrites modes which are consistent with XRD analysis. Ms of the resulted powder shows a uniform increase when the temperature is increased from 800 to 1200 and reaches to a maximum number of 72/30 emu/g. Hc is also increased by the increase of calcinations temperature and then is increased after 900 ° C. thus, magnetic behavior greatly depends on calcinations temperature.

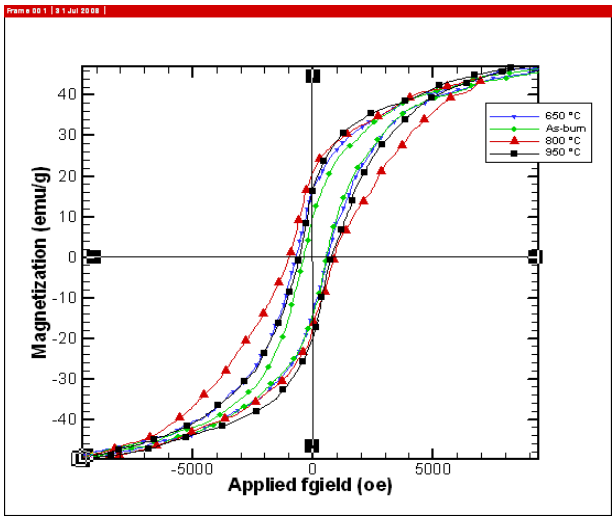

Figure 7- the diagram of hysteresis loop of hexaferrite barium Nano powder

Measuring absorption characteristics

Network analyzer device was used to measure absorbing at the frequency band of 8 to 12/5 GHz. To provide ideal conditions, there is a need to echo free space with a low loss; so, the absorption rate of specimens produced at antenna room in considered number at X frequency indicating the specimens' behavior across the band was measured based on dB.

Figure 7 shows magnetic hysteresis loops of Nano powders produced at the temperatures of 650, 800 and 950 °C and without calcinations. At the temperatures of 800 and 950 °C, hysteresis loop is greater and accordingly, the magnetic characteristic is better. In other words, the distance between magnetic saturation and coercivity force is high at this temperature due to the increase of magnetic permeability. Therefore, calcinations temperature is a key factor to determine magnetic characteristic of the substance which was determine as much 800°C in this experiment.

Figure 8 presents a wide spectrum of magnetic characteristics of a substance in X band by hexaferrite barium Nano composite epoxy with the diameter of 1 mm. although the thickness of the specimen is 1 mm, it shows a good absorption rate; and it will be observed a peak of – 4/12 dB and camouflage is successful in this one radar frequency. Figure 8 also shows absorption rate of the substance with the same thickness on the metal surface with the absorption rate of -9/5527 dB.

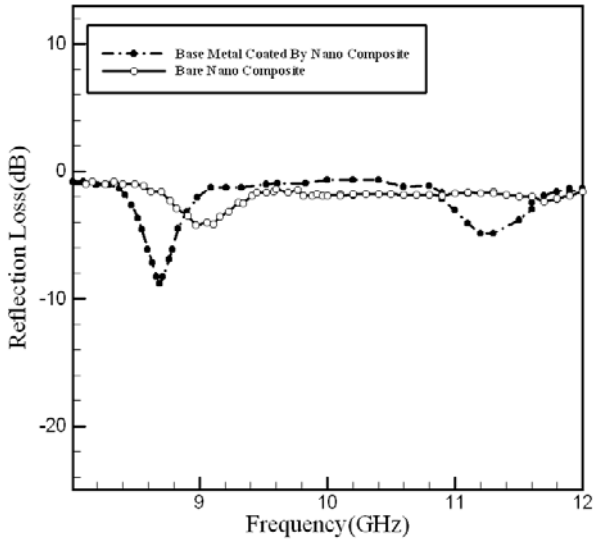

Figure 8- The diagram of absorption waves of Nano composite at the range of 8 to 12/5 GHz with the thickness of 1 mm and on a metal plate

Conclusion

After producing and testing the specimens, it was observed that calcinations temperature affects the structure of hexaferrite barium Nano composite directly. Also, the increase of calcinations temperature up to 950 °C caused to increase the magnetic characteristics leading to the more loss of radar waves` energy. Applying these Nano composites on the metal surfaces leads to more absorption due to the closeness of the absorbent substance's impedance to the impedance of the metal.

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