Indian J.Sci.Res. 8 (1): 037-040, 2014
ISSN: 0976-2876 (Print)
ISSN: 2250-0138(Online)

PRODUCING W-TYPE HEXAFERRITES BARIUM NANO COMPOSITES WITH RADAR WAVE ABSORBING PROPERTIES VIA A MICROWAVE-ASSISTED SOL-GEL AUTO-COMBUSTION METHOD

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ABSTRACT

Absorption and attenuation of electromagnetic waves spread in air by radar or other equipments is of the main problems 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. The considered Nano powders have been analyzed throughX-ray diffraction (XRD**) in different temperatures revealing a close phase to W-type. A** scanning electron microscope **(**SEM**) has also been used to determine the measure of the particles` size at Nano scale. Moreover, the absorbing properties of the material have been investigated at the limit of X band, specifying that Hexaferrites barium-epoxy Nano composites can act as an absorbent in the frequencies of 8 to 12 GHz, with an absorbing ability of 8db. The main objective of the present study is to produce w-type hexaferrites barium Nano composites with radar wave absorbing properties via a** microwave-**assisted** sol-gel **auto-**combustion method.

KEYWORDS: Hexaferrites barium Nano composites; Nano powders; radar absorbing materials; sol-gel

Hexaferrites barium can be produced using various conventional and non-conventional methods. In the conventional way, solid raw materials and some additives are combined and grind, if necessary, and thenthey are calcinated in air. In this method, impurities due to grinding action may penetrate into the mixture;on the other hand, the Magneticcoercivity force can be accessed only when particles` size is less than 1 micron (Crap et al., 1998). Considering the problems of conventional methods and for some applications, conventional methods have been replaced with non-conventional methods which are referred in brief. Using Co-precipitation method, Chenet al (2002) could produce strontium ferrite Nano particles in presence of acid Polyacrylic. Chemical combination homogeneity, high reactivity of co-precipitation product, high purity, ferrite production in low temperature, and tiny particles are considered as the adavantages of non-conventional methods compared to conventional method. The most important disadvantage of this method is high water consumption. Micharaet al (1997) also applied sol-gel method to produce below 1 micron particles of Hexaferrites barium using iron nitrate, barium nitrate, and citric acid as the primer. The method applied in the present study is very similar to sol-gel auto-combustion method but it has some advantages over it including short synthesis time and the lack of need to high temperatures.

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 penetration 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 penetration

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

μ : Magnetic penetration

σ : Conductivity

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ω : 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 1).For this plate, wave's fields on the surface and internal fields of the materials are expressed through Maxwell's equations.

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

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

E: electric field intensity

H: magnetic field intensity

$k = \frac{2\pi}{\lambda}$, $k' = kn$ 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:

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

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

If the $k = z_2/z_1$ while

Z_2: impedance of the absorbing material

Z_1: impedance of the absorbing material space

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

If $z_2 = z_1$ then $P_{ref} = 0$

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

$$R = 10\log\left|P_{ref}\right| \quad (8)$$

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.

DISCUSSION

Producing Nano composites specimens

To produce W-type Hexaferrite barium Nano composites with the comnonations of X= 0, 0/75.0/5. 0/25, at, 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, 1/542of 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

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

The method

$Ba(Co_xZn_{1+x})_2Fe_{16}O_{27}$ synthesis was done usingsol-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 (figure 2). The burning powder can be calcinated in various temperatures of 600 to 1200c 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.

To measure the particles` sizeusing SEM

The SEM picture shows the calcinated sample at 950c for 2 hours in the presence of agglomerate (figure 3) . The powder has compressed structure and agglomerate has steady distribution of particles. The average size of the particles is estimated below100nm. 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 calcinations temperature leads to the formation of powders with larger size particles.

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 2θ=20-70 region and the scan speed of 4º/min(fuigor4). the Fe_2O_3peaks 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.

Measuring absorbing properties

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 amount of specimen` absorbing was measured based on db.

The particle size mean obtained from SEM indicates that the selected synthesis method is effective to synthesize crystal Nano powders (figore3). The XRD analysis shows close phase to W-type. The thickness wasonly 1 mm but high absorb was achieved as we see the peak -4/12db (figore6). Also, absorbing ability of the material on metal surface with 1mm thickness was -09/5527db (figore5).

CONCLUSION

The Nano powders synthesis was doneusing sol-gel auto-combustionvia microwave. This method has some advantages compared to the previous methods including being economical, short synthesis process, being pure, not making pollution, being homogenized and no need for vacuity and high temperature.

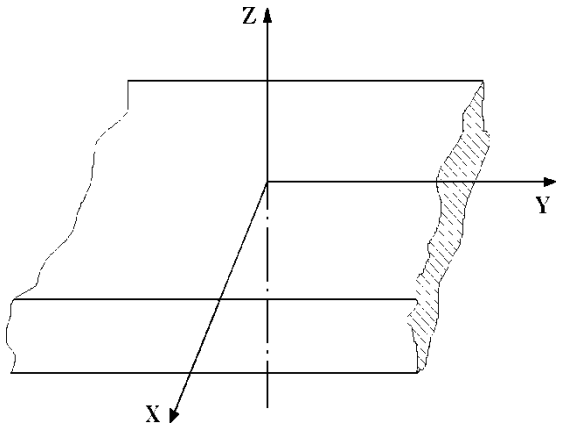

Figure 1: Infinite hypothetical plane of Absorbents

Figure 2: A view of powder being ignited

Figure 3: SEM image of the powders synthesized with particle size

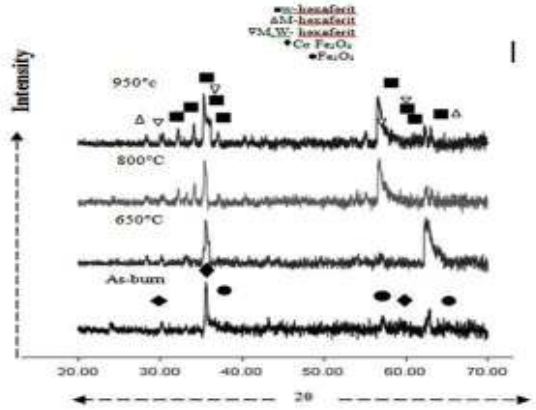

Figure 4: XRD diagram of Barium Ferrite Powder (Ba $(Co_{0.5}Zn_{0.5})_2Fe_{16}O_{27}$)

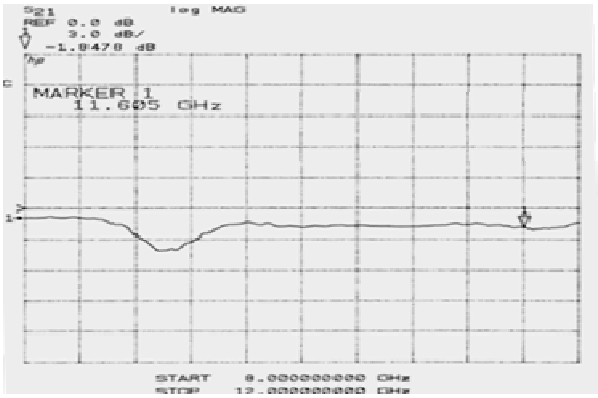

Figure 5: Absorption curve for Nano composites waveforms ranging from 12.5-8 GHz with a thickness of 1 mm

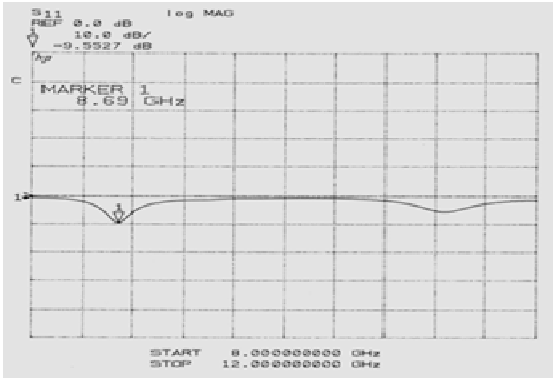

Figure 6: Absorption curve for Nano composites waveforms ranging from 12.5-8 GHz with a thickness of 1 mm on a metal sheet

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