International Journal Of Engineering Research And Development
e- ISSN: 2278-067X, p-ISSN: 2278-800X, www.ijerd.com
Volume 14, Issue 5 (May Ver. I 2018), PP.40-44

Nanocrystalline Barium Ferrites for Microwave Absorber Applications

P.Phanidhar[1] And S.R. Murthy[2]
[1] Research Scholar, Department of Physics, JNTUH, Hyderabad.
[2] Department of Physics, Osmania University, Hyderabad..
Corresponding Author: P.Phanidhar

ABSTRACT:- Nanocrystalline Barium Ferrite ($BaFe_{12}$ O_{19}) have been prepared by microwave sintering method at different sintering temperatures. The prepared samples were characterized by X-ray diffraction. The crystalline size of these samples has been varies from 40 nm to 60nm. The lattice constant and bulk density of these samples increase with an increase of sintering temperature. The temperature variation of electrical conductivity (σ) of these samples was measured by two probe method. The electrical conductivity in these ferrites has been explained on the basis of hoping mechanism. The plots of log σ T versus $10^3/T$ are show a transition near the Curie temperature. The activation energy in the ferromagnetic region is in general less than that in the paramagnetic region. The dielectric properties such as dielectric constant (ε′), dielectric loss tangent (tan δ). The dielectric constant and loss tangent are found to be decreasing with the increase in frequency. The dielectric properties have been explained on the basis of Maxwell-Wagner's two-layer model and hopping of the charge. The magnetic properties such as initial permeability (μ_i) and relative loss factor (RLF) have been investigated as a function of frequency in the range 20 KHz to 30 MHz

KEYWORDS:- Barium ferrite, Microwave-hydrothermal method, Microwave Sintering, Electrical conductivity, Dielectric properties

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Date of Submission: 02-05-2018 Date of acceptance: 17-05-2018

I INTRODUCTION

It is a well-known fact that the dielectric properties of ferrites strongly depend on frequency. As the quality of ferrite powder has a strong influence on the performance of the final device hence, the study of dielectric properties and ac electrical conductivity at different frequencies will give valuable information about the conduction phenomenon in ferrites. Since $BaFe_{12}O_{19}$ is quite versatile because of its microwave device applications and the dc resistivity of a ferrite is an important property, since it determines devices performance at high frequencies, where eddy currents losses may be high, resulting in a significant loss of energy. Therefore we are reporting the synthesis and study of electric and dielectric properties of nanocrystallne M-type Barium hexa-ferrite in this paper.

II EXPERIMENTAL

The pure chemicals used for the synthesis of Barium ferrite samples by microwave -hydrothermal are Ba $(NO_3)_2.6H_2O$ and Fe $(NO_3)_3{\cdot}9H_2$. The salts were dissolved in de-ionized water. The solution of both of these precursors with molar ratio (Fe/Ba = 12) was prepared. Then the samples were microwave treated at 170°C/1hr. After the microwave hydrothermal treatment, the solid and solution phases were separated by centrifugation and washed with de-ionized water and ethanol for several times to remove any soluble salts. Then they obtained wet powder was dried at 60˚C overnight. The synthesized nano powders of ferrite were fine granulated by adding 2 % poly vinyl alcohol (PVA) as a binder and followed by grinding for 4h. The granules were compacted into disks (dia- 8mm, thickness-2 mm) and to roids (Dout-8 mm, Din-5mm, thickness-3 mm) using a hydraulic press at a pressure of 150 MPa for 10 min. Finally, the nano powder was microwave sintered at different sintering temperature 700°(MS1), 800°(MS2), 875°(MS3), 950˚C(MS4)/40min). The sintered powder was characterized by XRD ((Philips Model no: PW-1730) and structural morphology was studied by FESEM.

The electrical conductivity of nanocrystalline Barium ferrite samples have been measured over a temperature range of 303 to 823 K using the two probe method. The values of See beck coefficients were also measured on the ferrite samples to confirm whether the conductivity was p-type of n-type and it was found that the all samples used in the present investigation were n-type. The frequency dependence of dielectric constant (ε΄) and dielectric loss tangent (Tan δ) on the nanocrystalline barium ferrites has been measured in the frequency range of 20Hz to 1MHz by using LCR meter at room temperature. The magnetic properties such as initial permeability (μ_i) and relative loss factor (RLF) have been investigated as a function of frequency in the range 75 kHz to 30 MHz

III RESULTS&DISCUSSIONS

Fig 1 .Shows the XRD patterns of the sintered samples at different sintering temperatures. It can be seen from the figure that the sample sintered at 700°C shows the minute trace of α- Fe_2O_3 and $BaFe_2O_4$ phase with hexa-ferrite phase. The samples with sintering temperature more than or equal 800°C shows the complete hexa-ferrite phase without showing any residual phases and this temperature is less than that of early reported [5] . This was achieved mainly due to the applying of higher initial microwave reaction time and sintering time used for preparation of the powders. With increasing sintering temperature more than 800°C, a single phase hexa-ferrite has been observed. With further an increasing of sintering temperature all peaks of $BaFe_{12}O_{19}$ phase were becomes narrower and sharper, manifesting particle size increases and with an improvement of crystalline and match with the standard, JCPDS file no: 27–1029.

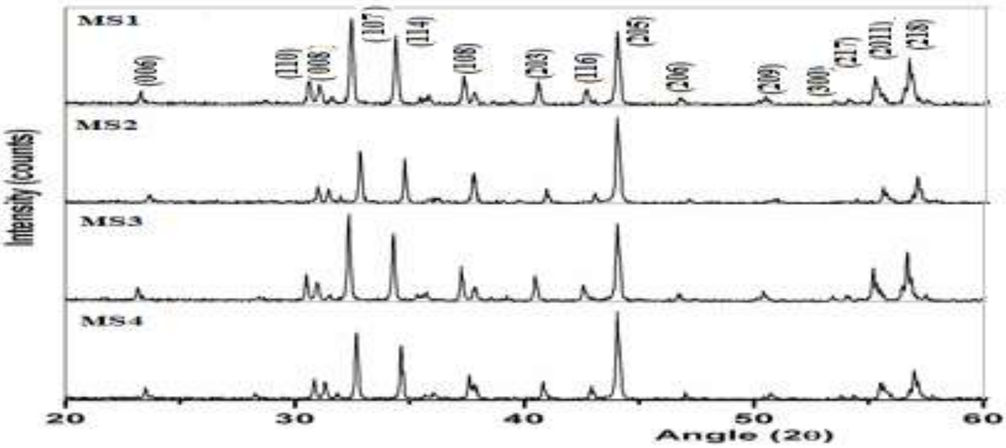

Fig.1 XRD pattern for sintered Barium ferrite samples

The average crystallite size was determined from the position of the strongest (1 0 7) diffraction peak using the well-known Scherrer equation and average crystallite size (D) and presented in the Table 1. It can be seen from the table that with an increasing sintering temperature from 800 to 900°C the relative intensity of the peak (107) increases and other peaks becoming equally sharper indicating an increase in particle size.

Fig. 2 shows FESEM pictures for all the sintered samples under investigation. It can be seen from the figures that when the samples sintered at 700 °C, grains were found to be a uniform in both shape and size where the majority are of acicular form and the size ranged from 0.5 to 2 mm. In case samples sintered at 800 °C, it is clear that the particles are hexagonal platelet crystals and the average grain ranged from 3.5 to 10 mm with the largest grains in the range of 20–25 mm. There is clear evidence that some of these grains have coalesced to form larger grains within the material formed at high temperature as a consequence of thermodynamically driven mass diffusion mechanisms.

The bulk density(d_{bulk}) of the presently investigated samples was accurately measured using the Archimedes' principle by taking an average of eight trials and the calculated d_{bulk} value is presented in Table 1.It can be seen from the table that the densification rate has been significantly increased in the microwave sintering process. In the microwave sintering process needs only 20 min to reach 700°C and to obtain a sample with density as high as 90% of TD. The density of microwave sintered sample has increased to 96% of TD with an increase of sintering temperature from 800°C to 900°C. Thus, higher densification was achieved in a shorter period by using the microwave sintering process.

TABLE:1 PREPARATION DATA ON BARIUM FERRITES

Sintering temperature (°C)	Crystallite size (nm)	Grain size (nm)	Lattice constant (Å) a	c	Bulk density (g/cc)	σ $(\Omega^{1}.cm^{-1})$	Q (μV/K)	Activation energy (eV) E_f	E_p
MH1	40	50	5.865	22.891	4.592	$1.49x10^{-8}$	-730	0.53	0.76
MH2	45	58	5.874	23.121	4.664	$1.35x10^{-8}$	-740	0.41	0.48
MH3	59	65	5.887	23.165	4.746	$1.29x10^{-8}$	-755	0.29	0.35
MH4	66	73	5.892	23.182	4.832	$1.25x10^{-8}$	-765	0.24	0.32

The lattice constant for all the present samples has been calculated with the help of XRD pattern. Lattice parameters (a) and (c) for all samples under investigation have been estimated and the average values of lattice constant for nanocrystalline barium ferrite samples are presented in the Table 1.

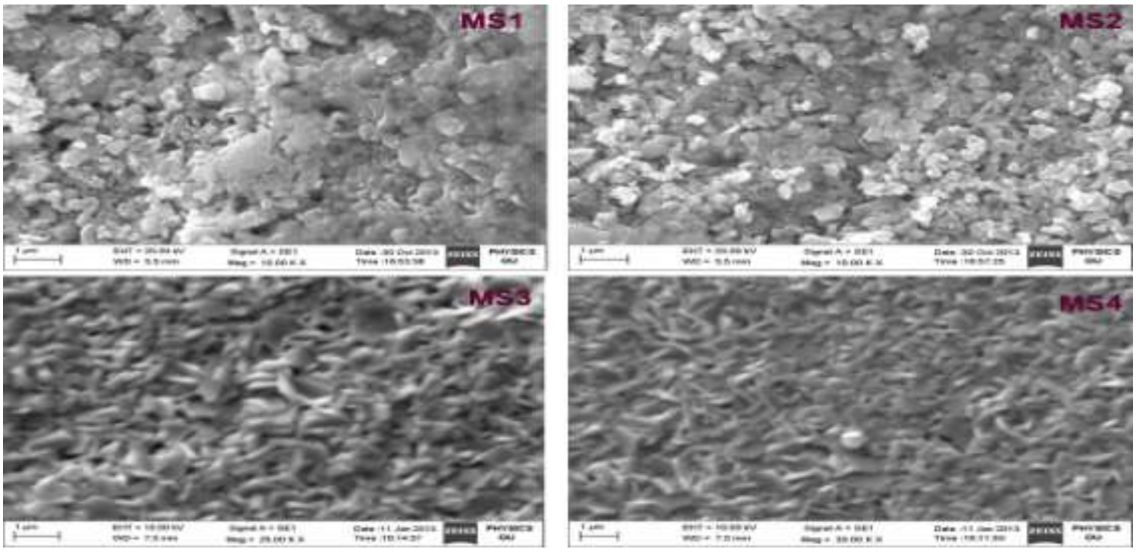

Fig. 2. FESEM pictures on sintered Barium ferrite.

The observed values of electrical conductivity (σ) and Seebeck coefficient (Q) for all the samples at 300 K are given in Table 1. It can be seen from the table that for present ferrites, as the microwaves treatment temperature increases from 700 to 900˚C that the value of σ varies from 1.49 x10^{-8} to 1.25x10^{-8} Ω-cm. It is evident from the table that the conductivity continuously increases with an increase of sintering temperature. It can also be observed from FESEM figures that the surface of the samples shows few pores formed by the escaping gases during the microwave reaction. This type of porous network is typical of microwave sintered powders. These porous powders are highly friable, which facilitate easy grinding to obtain finer particles. In the microwave sintering, the local temperature differences in pores and interfaces in the earlier stage of sintering are considered to be the origin of the microwave effect. The microwave radiation effect on grain growth is expected to be smaller than the effect on densification in the case of densified sample as these interfaces disappear in the later stage of sintering. As a consequence, enhanced grain growth is not observed with isothermal microwave sintering. Due to this reason microwave sintered samples possesses small and uniform grain structure. This is may be reason for an increase of σ in these ferrites with an increase of sintering temperature. The sign of the See beck coefficient for all samples is observed to be negative and therefore, all of them are expected to behave as n-type semiconductors.

Fig. 3 give the plots of temperature (T) variation of log σ T for all the microwave sintered nanocrystalline ferrites. It can be seen from the figures that the value of σ increases with an increase of temperature and show a change in slope at a certain temperature T_1 (K). This temperature T_1 value is in good agreement with the Curie temperature (Tc) of the corresponding sample showing that the kink in each case has occurred at the Curie point. The Curie temperature of the presently investigated sample has been obtained from a study of μ_i – T and the Curie temperatures obtained for σdc vs temperature (T) studies on microwave sintered nanocrystalline are nearly equal and it is 712 K. It can be seen from the figure that the value of σ increases with an increase of temperature by about four orders of magnitude. This sharp decrease of σ with temperature is mainly due to the thermally activated mobility of the charge carriers but not to the thermally activated creation of these carriers. For the present ferrites the activation energy required for charge hopping process was calculated using Arrhenius relation, where is activation energy k Boltzmann constant and ρ resistivity at temperature T is and ρo a constant. For all the samples was found to be >0.2 eV indicating that conduction is of electron hopping type [8].The activation energies in the ferrimagnetic (EF) and paramagnetic region (EP) are calculated by drawing the slopes at linear portion of the plots of log σ T verses 103/T and it was found to be that the activation energy in paramagnetic region is higher than that in ferrimagnetic region.

The dielectric properties such as dielectric constant (ε′) and dielectric loss (tan δ) are important for practical application point of view for ferrites used at high frequency range. Therefore, the dielectric constant measurements were carried out in the wide frequency range of 10 Hz to 1MHz. Figure 4 shows the frequency dependence of dielectric constant up to 1 MHz at room temperature. The dielectric constant in low frequency region decreases continuously with the frequency. The dielectric dispersion shown in the figure is consistent with the Koop's two layer model and Maxwell – Wagner polarization theory [8-10]. As the frequency of applied electric field increases, an assembly of space charge carriers in the composite medium requires finite time to

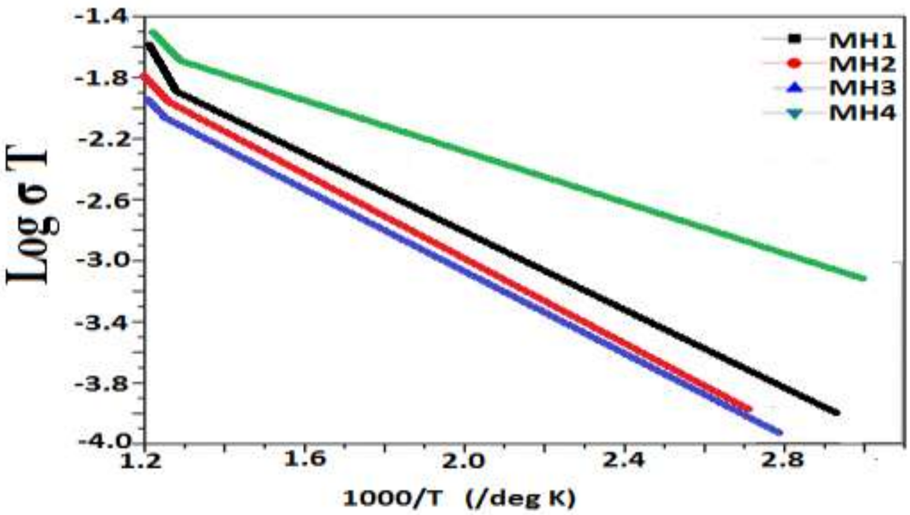

Fig. 3. Electrical conductivity plots versus temperature for Barium Ferrites

align their axis of dipoles parallel to the direction of applied field as a consequence, the dielectric constant decreases. The high value of dielectric constant at low frequencies can be associated with the space charge polarization. At higher frequencies the dielectric constant is independent of frequency due to inability of dipoles to follow the frequency of applied field. As the ferrite content in the composites increases the dielectric constant decreases. The electron exchange between the Fe2+ and Fe3+ ions cannot follow the frequency of applied field at high frequencies, as a result the dielectric constant decreases. Figure 2 also shows the variation of dissipation with frequency for all the ferrites under investigation. From the figure it can be observed that all the samples show the dispersion at lower frequencies. The plots are similar in nature as that of dielectric constant with frequency.

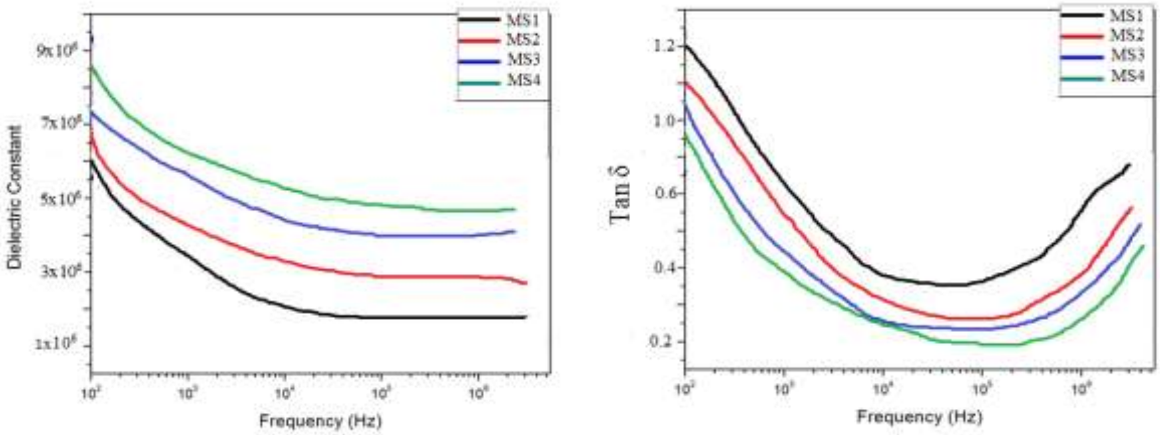

Fig. 4 Frequency dependence of dielectric constant and Tan δ on Barium ferrites

Fig. 5 shows the variation of initial permeability (μ_i) and relative loss factor (RLF) as a function of frequency in the range 75 kHz to 30 MHz, respectively. The initial permeability in ferrite is because of domain wall displacement and remains almost constant with frequency as long as there is no phase leg between the applied field and the domain wall displacement [10]. The variation of initial permeability with frequency shows that μi has fairly constant values of over a large frequency range which is a desirable characteristic for broadband pulse transformers and wide band read-write heads for video recording. It was not however possible to observe the complete resonance peak as it seems to appear at frequencies beyond 30 MHz, which is the upper limit of the frequency used in our studies. As the resonance frequency represents the high frequency limit up to which the material can be used in a device and in this study the resonance is occurring at higher frequencies this leads to the extended zone of utility for present nano hexa-ferrite [11]. The variations in magnetic loss in the form of relative loss factor (tanδ/μi) as a function of frequency shows that RLF decrease initially with frequency, reaching a minimum value, and the starts to rise thereafter. The frequency at which RLF is minimum, called threshold frequency. The loss is due to the leg of domain walls with respect to the applied alternating field and is attributed to imperfections in the lattice. The low value of RLF is required for high frequency magnetic applications. The RLF value observed in this work is of the order of 10^{-4} in a wide frequency range, 75 kHz to 30 MHz, which means that the investigated nano hex-ferrite will be more useful in this regards.

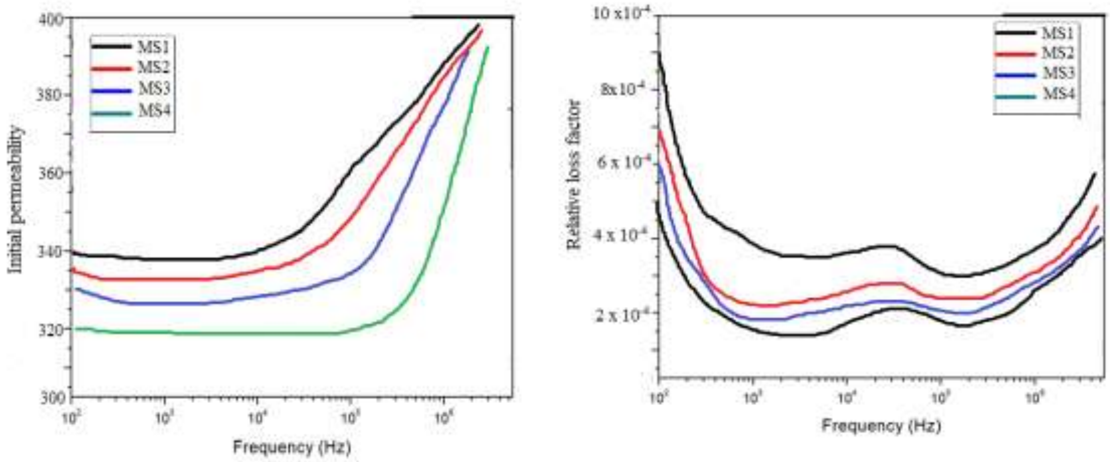

Fig. 5 Frequency dependence of initial permeability and Relative loss factor on Barium Ferrites.

IV CONCLUSIONS

The nanocrystalline $BaFe_{12}O_{19}$ hexa-ferrite has been successfully prepared by the microwave-hydrothermal technique. The sample shows a high value of dc resistivity of the order of 10^8 Ω-cm and very low values of the dielectric loss tangent. Frequency dependence of dielectric constant showed normal dielectric behaviour and is in good agreement with Koop's phenomenological theory of dielectric dispersion. The improved value of dc resistivity makes present $BaFe_{12}O_{19}$ very suitable for microwave applications.

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P.Phanidhar. "Nanocrystalline Barium Ferrites for Microwave Absorber Applications." International Journal Of Engineering Research And Development , vol. 14, no. 05, 2018, pp. 40–44