

Microwave Absorption Study of Nano Synthesized Strontium Ferrite Particles in X Band

Shivani Malhotra¹, Mansi Chitkara² and I S Sandhu²,

¹ *School of Electronics and Electrical Engineering Chitkara University, Rajpura, Punjab (INDIA)*

² *Nanomaterial Research Laboratory, Chitkara University, Rajpura, Punjab (INDIA)*

*shivani.malhotra@chitkara.edu.in, mansi@chitkara.edu.in,
is.sandhu@chitkara.edu.in*

Abstract

Microwave ferrites require high coercivity for their use as microwave absorbers. Strontium hexaferrite is one of the best material due to its magnetic properties, high coercivity, large unilateral magnetic anisotropy and very low cost in comparison to others make it suitable for the use as microwave components specifically microwave absorber. Strontium nano hexa ferrites were successfully synthesized through chemical co-precipitation method. The synthesized nanostructure was characterized by X-Ray powder diffraction (XRD) and fourier transform infrared spectroscopy (FTIR). The XRD result shows the shift from amorphous to crystalline after the calcination of sample at 600°C. The behaviour of ferrite as microwave absorber was also studied for X band of frequencies.

Keywords: *FTIR, Hexaferrites, Microwave absorption, Nanostructure, XRD*

1. Introduction

Recent advancement in microwave absorber technology radar absorbing materials (RAM) has resulted in special materials that can potentially reduce the reflection of electromagnetic signal thus improve the system performance. Ferrites have been attracting attention of microwave researchers for their possible use in miniaturized circuits and as one of the electromagnetic wave absorber. The penetration of em waves is possible in ferrites because of their nonconducting nature. In contrast it is limited in metals because of the skin effect. Due to their low eddy current losses it is treated as best material for a variety of electronic applications in terms of generation, conditioning, and conversion of power [1]. Electromagnetic wave can initiate an inter communication between the wave and the magnetization within ferrite [2-3]. This interaction has been used to produce a plethora of useful devices [4]. The ferrite structure can be garnet, spinel or magnetoplumbite which has a close-packed structure of oxygen anions considered as its backbone [5].

M-type hexaferrites are of interest for the use as recently, Strontium hexaferrite M type (SrFe₁₂O₁₉) have shown excellent properties in the microwave and millimetre wave region of the electromagnetic spectrum [6-7]. M-type hexaferrite (SrFe₁₂O₁₉) is an important ferromagnetic oxide. Due to its high corrosion resistance, high resistivity, high saturation magnetization and excellent chemical stability these materials can be considered for their use as permanent magnets. Their high coercivity, low cost make and large unilateral magnetic anisotropy make them suitable for the use as microwave components. Besides that they also exhibit stable and have high electrical resistivity [8] [9]. Dielectric and magnetic losses of M-type hexagonal nanoferrites at microwave

frequencies make them a special kind of absorbing material. However, the magnetic losses in these materials result from absorption of moving magnetic domains and spin relaxation at high frequency during resonance, alternating electromagnetic field. In lieu of these advances, it gives a clear indication that microwave device technologies is at the turning point for the positive change with the potential to greatly impact a wide range of technologies that involve the transmission ,reception and manipulation of electromagnetic signals.

Many efforts are made in order to achieve large saturation magnetisation and high coercivity but to increase these values we require high temperature which results in large particle size. Many synthesis techniques are used in order to obtain lesser particle size like sol-gel method, ball milling, sonochemical method, citrate gel, chemical co-precipitation etc. In this paper we opted for chemical co-precipitation as it allows the crystallization to occur at low temperature, which results in the formation of nano sized particles due to mixing of starting materials on ionic levels and thus it proves to be low cost method for the production in bulk. The effect on the structural properties with respect to change in calcination temperature and crystallinity has been examined. Particle size is also calculated and behaviour of nanoferrites at microwave frequencies is also studied.

2. Experiment

The polycrystalline $\text{SrFe}_{12}\text{O}_{19}$ was synthesized in aqueous media using chemical co-precipitation method as described by (Gholam *et al.* 2013). The stoichiometric amount of of chloride compounds of sr and Fe with chemical composition $\text{SrCl}_2.6\text{H}_2\text{O}$ by Fisher Scientifics (99% purity) and $\text{FeCl}_3.6\text{H}_2\text{O}$ of (Finer chemical Ltd) were dissolved in deionised water to form uniform solution. Then 10 M alkaline sodium hydroxide (NaOH) was dropwise titrated into the solution and the Ph was adjusted to 12 untill dark brown precipitates appears. The resulted solution was kept for aging for 24hrs.It was then filtered and washing was done for several times with water and lastly with ethanol. The resulted gel was dried in a furnace at 60°C overnight to remove the moisture contents from the product. The dried precipitates were calcined for 4 hrs at a temperature of 600°C in air to give crystalline Sr- hexaferrite. Prepared samples were crushed to get powder form for characterization of samples. The formation of Sr hexaferrite was confirmed by Fourier Transform infrared spectroscopic studies (FTIR). Structural analysis was done using X-Ray diffractometer (XPRT -PRO PW 3050/60) with $\text{CuK}\alpha$ radiations. The absorption properties were studied using microwave bench with VSWR meter.

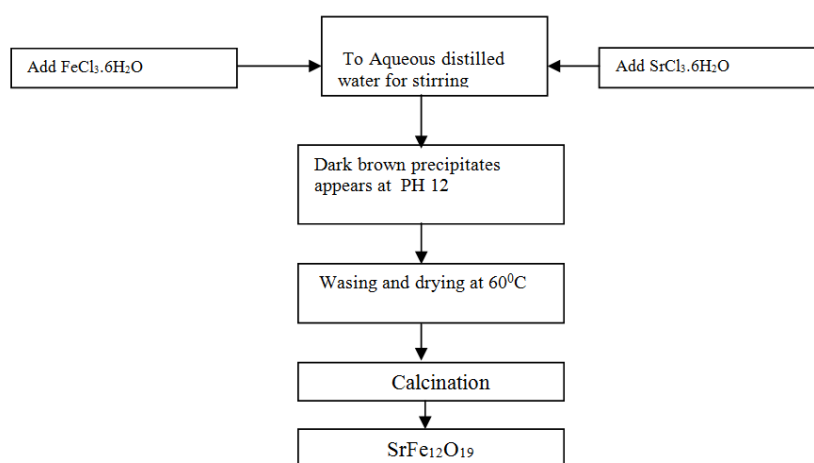


Figure 1. Flow Chart for Method of Preparation

3. Results and Discussion

3.1. FTIR (Fourier Transform Infrared Spectroscopy)

Fourier Transform infrared spectroscopic studies (FTIR) were done in order to determine ferrite formation of samples. FTIR spectra of both samples *i.e.* with and without calcination were accomplished for range 4000 - 400 cm^{-1} (wave number). The powder synthesized was mixed with KBr, pelletized and FTIR spectra were recorded using Perkin Elmer-USA FTIR spectrometer. The transmittance in hexaferrite powder was decreased as fringing fields were absorbed by them. The FTIR spectra of strontium hexaferrite powders show three signature transmittance peaks of hexaferrite at $\sim 408 \text{ cm}^{-1}$, 481 cm^{-1} and 534 cm^{-1} which are same when compared with other published works [10].

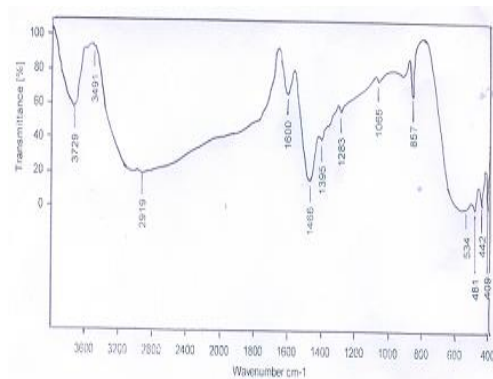


Figure 2. FTIR-spectrum of Calcined Powder

3.2. XRD (X-Ray Diffraction)

Phase identification and structure analysis was done at Punjab university on PAN analytical (Netherlands) X-ray Diffractometer model X-PERT PRO with wavelength (λ) = 1.54 \AA for $\text{CuK}\alpha$ with scattering angle range (2θ) of 20° to 80° with a scan rate of $0.0170^\circ/\text{sec}$ and specimen length of 10 mm .

The particle size d is calculated from the broadening of the X-ray line, using Scherrer's equation (1)

$$d = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

Where λ is the wavelength of X-rays, k is the shape factor and can be taken ~ 0.9 . β is FWHM (full width at half - maximum) expressed in units of 2θ and θ is the Bragg angle. The X-Ray diffraction pattern for calcined and non calcined sample is recorded and analyzed. Fig 3 & 4 represents XRD pattern of strontium hexaferrite both without and with calcinations respectively. All the peaks are identified and matched with JCPDS data. The standard data used for indexing is JCPDS 84-1531.

The pattern shows peak formation and broadening after calcinations of the sample which confirms nano size formation of the sample. The average crystallite size as calculated using the Scherrer equation (1) for major peaks at $2\theta = 33.1786, 35.6684$ was $\leq 50 \text{ nm}$.

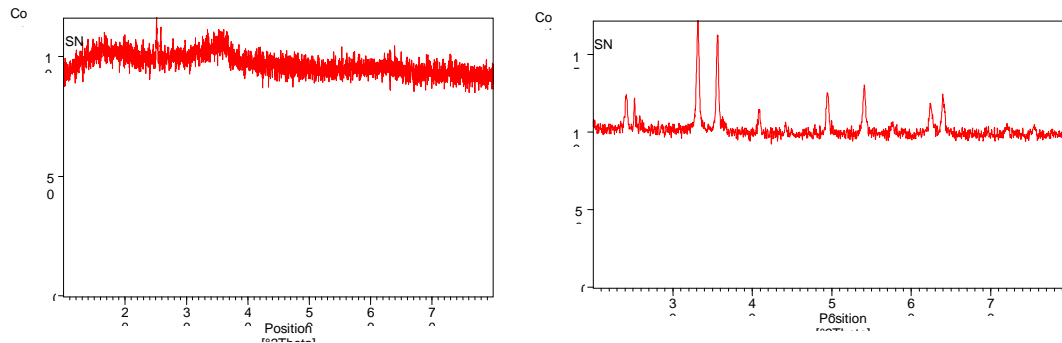


Figure 3. XRD Pattern For Synthesized Sample a) Without Calcinations b) With Calcination At 600°C

3.3. Microwave Absorption of Hexaferrite Nanomaterial

In order to analyze the microwave behavior of strontium hexaferrite as low transmittance and high absorbance was observed using microwave bench and VSWR meter. In X band a good amount of absorbance was recorded due to insertion of strontium hexaferrite powder. The sample was inserted in the microwave bench using cellophane tape. The microwave bench was initially set for minimum loss which was measured using VSWR meter. Then the cellophane tape was used as an obstruction in the path and insertion loss was measured which was negligible. The sample holder was made using cellophane tape in which powdered sample was adjusted and then it was inserted in the waveguide and loss was investigated on VSWR meter. The sample proportion was increased by 100 mg in every step and 10 such reading were noted. Fig 5 shows the arrangement of microwave bench for observing absorption loss in the waveguide due to the presence of powdered hexaferrite sample. From the readings of the VSWR meter a graph was plotted which indicate the increase in absorption level with the increase in quantity of sample as indicated in Figure 6.



Figure 4. Microwave Bench Setup for the Measurement of Microwave Absorption

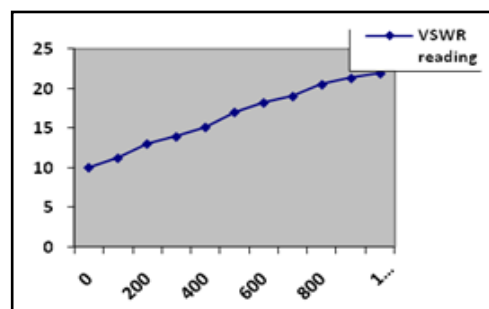


Figure 5. Microwave Absorption Vs wt. of Nanoferrite Powder

4. Conclusion

It has been investigated that the chemical co-precipitation method for synthesis of strontium hexaferrite nanomaterial is an efficient and easier technique. The synthesis process has been first carried out without calcination of sample and then with calcination. The XRD pattern of these samples reveals the transformation from amorphous to crystalline structure. The average particle size of the synthesized strontium nano hexaferrite is 44nm. The findings of the waveguide experiment carried out shows that the strontium hexaferrite nano material is efficient absorbers for electromagnetic waves in X band.

Acknowledgements

The authors are acknowledged to Sophisticated Analytical Instrumentation Facility (CIL XRD wing) Punjab University, Chandigarh for providing XRD patterns for the samples.

References

- [1] C. C. Xiang, Y. Nie, Z. K. Feng, R. ZH. Gong and Z. Yuan, "Low-loss Z-type Hexaferrite for Microwave Antenna Miniaturization Application", Materials Science Forum, vol. 687, (2011), pp. 309-314.
- [2] J. Stefan, "Advantages of Ferromagnetic Nanoparticle Composites in Microwave Absorbers", Published in Magnetics: IEEE Transactions, Inst., Ljubljana Univ, Slovenia, vol. 40, no. 3, (2004).
- [3] B. D. Culity, "Introduction to Magnetic materials", Addison-Wesley Publishing Company, Philippines, (1972).
- [4] J. J. Went, G. W. Rathenau, E. W. Gorter and G. W. Van Oosterhout, "Ferroxdure, a class of new permanent magnetic materials", Philips Tech. Rev., vol. 13, (1952), pp. 194-208.
- [5] N. N. Ghosh, P. Pant, and S. Bhuvanewari, "Chemical Methodologies for Preparation of Micron and Nanometer Scale Ferrites - A Mini Review of Patents", Recent Patents on Nanotechnology, vol. 2, no. 1.
- [6] Y. Wang, T. Li, L. Zhao, Z. Hu and Y. Gu, "Research Progress on Nanostructured Radar Absorbing Materials", Journal on Energy and Power Engineering, vol. 3, (2011), pp580-584.
- [7] A. Ghasemi, A. Paesano Jr., C. Fabiana, C. Machado, S. E. Shirsath, X. Liu, and A. Morisako, "Mössbauer spectroscopy, magnetic characteristics, and reflection loss analysis of nickel-strontium substituted cobalt ferrite nanoparticles", Journal of Applied Physics, vol. 115, (2014), 17A522.
- [8] S. Y. Liao, "Microwave devices and circuits", Prentice Hall of India, (2000).
- [9] V. Kushwah and G S Tomar, "Design of Microwave Patch Antenna using Neural Network" IEEE Intern'l Conference AMS 2009, (2009) May, pp. 25-27.
- [10] J. Y. Shin and J. H. Oh, "The Microwave Absorbing Phenomena of Ferrite Microwave Absorbers," IEEE Transactions on Magnetics, vol. 29, no. 6, (1993), pp. 3437- 3439.
- [11] F. M. M. Pereira, C. A. R. Junior, M. R. P. Santos, R. S. T. M. Sohn, F. N. A. Freire, J. M. Sasaki, J. A. C. de Paiva and A. S. B. Sombra, "Structural and dielectric spectroscopy studies of the M-type barium strontium hexaferrite alloys ($Ba_xSr_{1-x}Fe_{12}O_{19}$)", Journal of Materials Science: Materials in Electronics, vol. 19, no. 7, (2008), pp. 627-638.

Authors



I. S. Sandhu, he obtained his Ph.D. Degree in Solid State Physics from Kurukshetra University, Kurukshetra by doing research work on "Study of Diffusion in Solids & Intermetallic Compounds". He did his M.Sc. in Physics (specialization in Electronics) from Kurukshetra University, Kurukshetra. He has guided 02 Ph.D. thesis and is guiding 04 Ph.D. thesis at present. His research areas include Nanomaterials, Nanoelectronics, and Nanoelectrodynamics. He has published several papers in high impact factor journals, attended and presented papers in national and international conferences. He has

published several book chapters/research articles in international publications, like: Springer, CRC Press, Taylor & Francis Group.



Mansi Chitkara, she obtained her Ph.D. Degree in Mechanical Engineering from Punjab Technical University and the title of her Ph.D. thesis was Synthesis and Characterization of semiconductor nano-materials. She did her Master of Engineering in Industrial Materials & Metallurgy from Punjab Engineering College, Chandigarh. Her research area includes Nano-materials, Nano-Herbal materials synthesis and characterization for Pharmaceutical and Medical applications. She has published more than 35 research papers in reputed International Journals, International/National Conferences. She is currently guiding Ph.D., M.Tech., M.Pharm. students in the field of Nano-science and Technology. She has published several book chapters/research articles in international publications, like: Springer, CRC Press, Taylor & Francis Group.



Shivani Malhotra, she received M.Tech degree in Electronics and Communication Engineering from Punjab University, Punjab (India) in 2008. She is currently pursuing PhD. in Electronics and communication engineering from Chitkara University, Punjab (India). She is currently working in the area of Synthesis and characterization of radar absorbing materials, microwave characterization of ferrites and Gesture recognition and she had presented several papers regarding this in various Conferences.