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EFFECT OF SYNTHESIS CONDITIONS ON YTTRIUM IRON GARNET (YIG) NANOCRYSTALLINE POWDER VIA SOL GEL METHOD

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Abstract: Nanocrystalline powder of pure Yttrium Iron Garnet ($Y_3Fe_5O_{12}$) was synthesized using citric acid as chelating agent. Garnet samples were obtained after calcinations in air at different temperatures for 3 hrs. The X-ray Diffraction showed garnet phases of nano ferrites only at high temperatures. The morphological study and elemental composition of the YIG sample was analyzed using High Resolution Scanning Electron Microscope (HR-SEM) and Energy Dispersive X-ray spectrometry (EDX). Dielectric constant and dielectric loss of the nanomaterial at different temperatures were studied with respect to frequency.

KEY WORDS: garnet, ferrites, HR- SEM, dielectric studies.

1. INTRODUCTION

Magnetic ferrites in nano form are extensively used in wide spectra of applications. Among these nanostructured materials of different shapes and sizes, transition metal ferrite nanoparticles have attracted current research due to their technological applications. Yttrium iron garnet ($Y_3Fe_5O_{12}$) is a versatile ceramic material possessing high melting point, large resistivity, better electromagnetic properties, high thermal stability, low thermal expansion, better chemical stability. Also, it has a large Faraday rotation, high initial permeability, high saturation magnetization and strong coercivity. It has attracted considerable attention due to its technological importance in various applications such as microwave devices, acoustic, optical, isolators, circulators, high quality filters, phase shifters, electronics industry and magnetic optical devices.(Majid et al).

Majid et al reports that conventional ceramic methods were customarily used for the preparation of garnets which involves high temperatures resulting in loss of fine particle. Nevertheless, in recent years, wet-chemical routes have been developed to address the synthetic difficulties, to prepare fine particles of pure garnet powders. Variety of chemical approaches including sol-gel, co-precipitation and glycothermal synthesis has been reported to prepare submicron to nanocrystalline powders of YIG.

Wang Minqiang et al suggest sol-gel method as a widely used technique involving atomic scale mixing that leads to final nanocrystals with narrow size distribution. The objective of the present study is to understand the transformations from amorphous to crystalline phase during the formation of YIG powders by cost effective sol gel method. The effects of synthesis parameters on surface morphology and dielectric measurement of YIG are investigated successfully.

2. EXPERIMENTAL PROCEDURE

Yttrium nitrate ($Y(NO_3)_3.6H_2O$, 99.99%) and iron nitrate ($Fe(NO_3)_3.9H_2O$, 99.99%) for the synthesis of yttrium iron garnet ($Y_3Fe_5O_{12}$) were dissolved in the aqueous solution of citric acid ($C_6H_8O_7.H_2O$) the chelating agent. The molar ratio of metal nitrates to citric acid was 1:1. The solution was stirred at 310 rpm for 1 day. The temperature was increased to 80°C with continuous stirring until the formation of gel. The samples in gel form were dried at 110°C for 34hours. The dried powder was ground for 5 hours and finally sintered at 1150°C for 4hours in air furnace. For sample sintered at 950°C the precursors were stirred only for one day without changing other conditions for research purpose.

The phase identification and crystalline structure of the prepared samples of $Y_3Fe_5O_{12}$ by sol gel method at different temperatures were investigated using X-ray diffractometer (Bruker D8 advance) operated at 40 kV and at 30 mA with CuKα radiation (λ =1.5406 Å). FEI Quanta FEG 200 - High Resolution Scanning Electron Microscope coupled with Energy Dispersive X-ray spectrometry (EDX) was used to examine the morphology and elemental composition. HIOKI LCR meter was utilized for dielectric measurements.

It is significant to note that the proposed sol–gel preparation gave reproducible results.

3. RESULTS AND DISCUSSION

3.1. Powder X-ray diffraction:

The X-ray diffraction pattern of YIG nanoparticles synthesized by sol–gel method at different temperatures is shown in Fig.1. From the result, it is observed that $Y_3Fe_5O_{12}$ samples showed single phase garnet peaks only at high temperatures. No reaction between the starting powders

occurred prior to sintering as well as after sintering at 550°C. This result is in line with the reports of Rodziaha et al that crystalline YIG could not be formed at too low sintering temperatures, as low sintering temperature provides insufficient energy to stimulate a reaction. From XRD report the pulverized sample shows no peaks but at 550°C there is indication of rising peaks which confirms the importance of high temperature for the preparation of garnet. Well defined peaks occurred only at higher temperature. The temperature can be further raised to obtain a well defined garnet phase. Crystallite size of YIG at 950°C is calculated to be 51nm using Scherer formulae. The average crystallite sizes of the synthesized powders were determined using the X-ray broadening of the (4 2 0) diffraction peak by the well-known Scherrer equation,

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

where D is crystallite size in nm, λ the radiation wave length (0.15405 nm for Cu Kα), β is the corrected full width at half maximum and θ is the diffraction angle as reported by Hosseini et al.

Table1.Calculation of different parameters from XRD

YIG	2θ	θ	FWHM	d-spacing	D nm
950°C	33.2	16.6	0.17	2.16	51

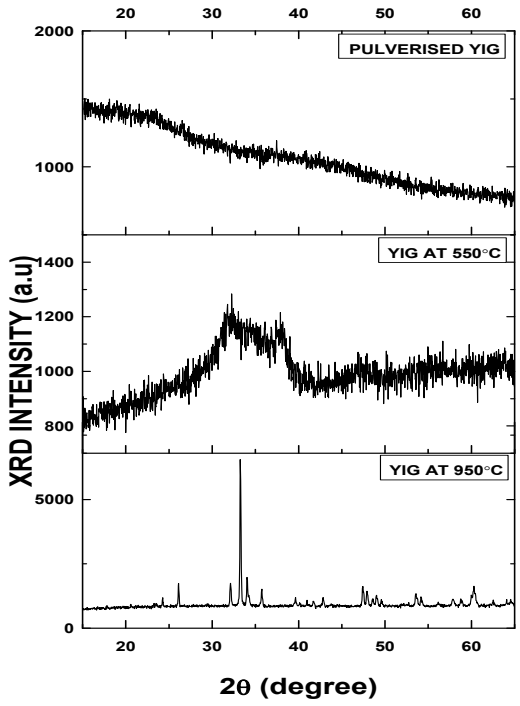


Fig.1 XRD pattern of YIG at 110°C, 550°C, 950°C

The porosity **(P)** of the sample was determined using the formula,

$$P = 1 - d_m/d_x,$$

where d_m and d_x are the measured density and theoretical density, respectively. The measured density was calculated using the relation,

$$d_m = m/\pi r^2 h$$

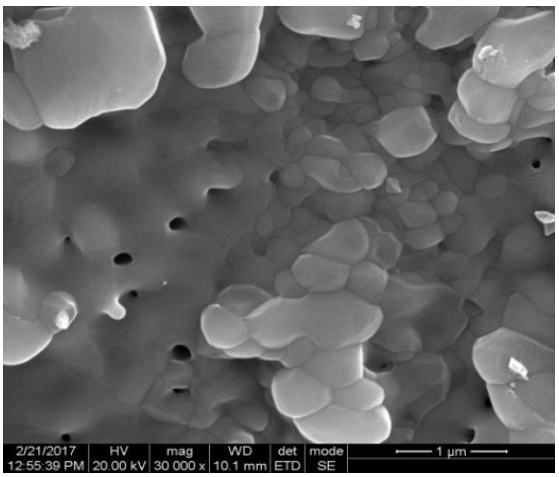
where h is the height, r is the radius and m is the mass of a cylindrical pellet of the sample. The theoretical density was calculated by using the formula

$$d_x = 8M/NV$$

Here,8 is the no. of formula units in a unit cell, N is Avogadro's number,M is the molecular weight of one formula unit and V is the volume of the unit cell (Nasir et al). Porosity is calculated as, P (fraction) = 0.49 for sample sintered 950°C.

3.2. The structure and morphology of YIG nanoparticles

The morphological study and the dimensions of the grain size of the YIG samples were analyzed using High Resolution Scanning Electron Microscope (HR-SEM). A scanning electron microscope (SEM) is a type of electron microscope that produces images of sample by scanning the surface with focused beam of electrons. The SEM image at 1 μ m in fig.2 is indicative of developing particles along with agglomerated particles. The EDX spectrum confirms the presence of elemental composition without impurities. Overall images show large number of pores which indicates easy breaking of agglomeration at higher temperatures.



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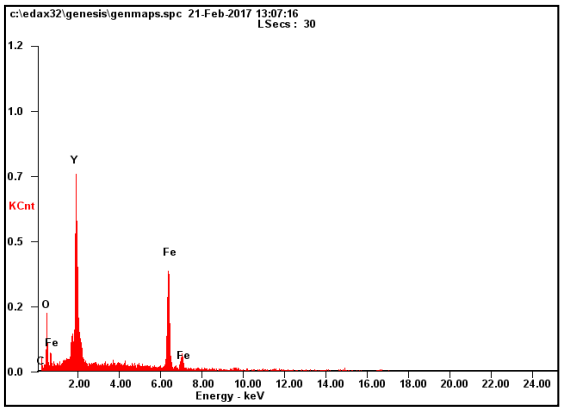
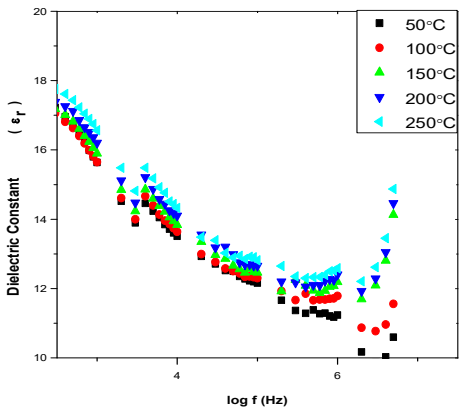


Fig.2 Scanning electron microscopy image for sample $Y_3Fe_5O_{12}$ at 950°C

3.3. The dielectric properties of the YIG

The dielectric constant and the dielectric loss of the YIG nanoparticles were studied at different temperatures in the frequency region 50 Hz–5 M Hz. With increase in temperature, electrical conductivity increases due to the increase in thermally activated drift mobility of electric charge carriers according to the hopping conduction mechanism. Therefore, the dielectric polarization increases causing an increase in ε and tan δ with increase in temperature (Ayhan Mergen et al). For high-frequency applications, low dielectric constant materials are preferred because of lower dielectric loss and skin effect. At high frequency, the dielectric loss becomes dominant and hence lower the dielectric constant, lower is the dielectric loss (Nasir et al). From fig.3a) and b) it is evident that YIG sample sintered at 950°C exhibits low dielectric constant and low dielectric loss and thereby is a suitable candidate for high frequency applications.



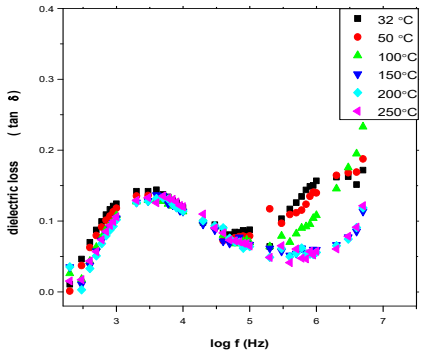


Fig.3 a) Frequency dependence of dielectric constant of YIG sample at 950 °C.
b) Frequency dependence of dielectric loss of YIG sample at 950 °C.

4. CONCLUSION

The sol–gel method of preparation of YIG in aqueous media is inexpensive and thus appropriate for the large scale production of YIG nanocrystalline powder. Single phase $Y_3Fe_5O_{12}$ (YIG) garnet ferrites can be obtained only at high temperature which was confirmed by XRD and SEM analysis. To conclude, at low sintering temperatures the reactions are incomplete and only at high temperatures sufficient energy is provided to stimulate the reaction leading to the formation of nanocrystalline garnet phases. Therefore, synthesis conditions greatly influences the structure of garnet phases. Results from dielectric studies reveal that YIG nanomaterial sintered at high temperature will find its use in high frequency applications because of the lesser dielectric losses at such frequencies.

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