Preparation And EM Absorption Properties of Gamma (γ) Nano-Ferrite In Epoxy Based Nano-Composites

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Abstract- *Gamma (γ) Nano-ferrite in epoxy based Nano-composites have been successfully prepared in toroidal shape and the microwave absorbing properties for different thicknesses (t) have been studied using simulation code for metal backed single layer absorber. The vector network analyser (Model PNA E8364B, software module 85071E) attached with co-axial measurement set up has been utilized to investigate the electromagnetic and microwave absorbing properties of the samples in the frequency range of 2 to 12 GHz. The fabrication of the samples have been carried out using Gamma (γ) Nano-ferrite in different ratios (20, 40 and 60 %) as filler in two component epoxy matrix. The complex permittivity and complex permeability of the composites have been found to be frequency dependent. The results also showed that with increasing Gamma (γ) Nano-ferrite filler content, the maximum reflection loss increases sharply. The maximum reflection loss is found to be thickness (t) dependent and increases with increasing thickness (t) of the sample with minimum peak shifting towards lower frequency. The minimum reflection loss peak of – 46.4 dB is achieved at matching frequency (f_m) of 4.56 GHz with more than 10 dB (i.e. 90 % loss) in frequency band 2.0 GHz -7.1 GHz for sample thickness (t) of 8.0 mm. It is also evident that minimum peak shifts towards lower frequency with increasing thickness. The morphology and the thermal behavior of the Nano-composite samples have also been investigated through scanning electron microscope (SEM) and thermo gravimetric analysis (TGA) techniques.*

Keywords- Microwave, RCS, Reflection Loss, Gamma Nano-ferrite, Nano-composite, Stealth materials

I. INTRODUCTION

The development of RADAR during the Second World War leads to the intensive investigations about interaction of electromagnetic radiation with different types of materials so that an efficient counter measure against RADAR detection could be achieved [1,2]. Radiation absorbent materials (RAM) are those materials which have been specially designed and shaped to absorb incident electromagnetic radiation (EMR), generally in microwave (MW) region of interest, as effectively as possible, from as many incident directions as possible [3]. Microwave is a narrow region of electromagnetic spectrum lying between infrared and radio-wave that have a frequency range from around 1 GHz to 300 GHz with corresponding wavelengths ranging from around 30 cm to 1 mm. The quantum energy of microwave photons ranges from 10^{-6} to 10^{-3} eV which is in the range of energies separating the quantum states of molecular rotation and torsion [4]. The interaction of microwaves with matter other than metallic conductors will be to rotate molecules and produce heat as result of that molecular motion [4]. The effectiveness of RAM is determined by its ability to absorb the electromagnetic radiation and lower the resulting level of reflected EMR. The coating of such materials on the exterior surfaces of military vehicles such as aircrafts, ships, aerial vehicles, tanks etc. help them to conceal from detection by radar. RAM can be prepared by a variety of electrically conducting and magnetic materials in fine powder form and loaded in various kinds of polymeric binders [4, 5]. There are various mechanisms by which microwaves interact to a material medium and there are several ways that the microwave energy is subsequently lost to the system. The main loss mechanisms are electric, hysteresis and resonance (domain wall), conduction (eddy current), and electron spin resonance (ESR) [4-7]. It is often difficult to determine which loss mechanism or combination of mechanisms is happening for a particular sample in the given situation. However, the Permittivity (ε) and permeability (μ) are the two important electromagnetic design parameters for the stealth material and EMI shielding [4-5]. In the case of a lossy medium, ε and μ are complex numbers. For absorbent materials, most of the electromagnetic energy must be attenuated [4]. Energy absorption into materials from microwave fields occurs by three main methods: dipolar, magnetic and conductive losses [4-5]. Electromagnetic absorption is also of high importance in aerospace engineering where the development of a thin, lightweight absorbing material in a broad range of frequencies is an essential part of the stealth technology [4]. An absorber soaks up the incident electromagnetic energy, thereby reducing the net energy available for reflection back to radar [1-4]. In other words, the more absorptive the material is the more invisible for radar an aircraft can be. Though the current state of the stealth technology is highly classified, to the best of our knowledge today's Radar Absorbent Materials (RAM) still suffer a trade-off between the broadband effectiveness and the absorber weight that can significantly reduce aircraft's payload [1-2, 10-12].

Research in the development of electromagnetic/microwave absorbing Nano-composites/ lossy materials is attracting much more attention from many years due to extensive growth in electronic devices for various applications in different GHz frequencies. These microwaves absorbing Nano-composites are in great demand due to their applications in defense sector as well as in civil sectors [1, 12-15].

In defense sector, microwave absorbing Nano-composite materials can be employed to stealth application i.e. reduction of radar cross-section and protection from microwave irradiation. Stealth technology is a sub-discipline of EWCM (electronic warfare counter measures) which covers a range of techniques to make weapon systems such as aircrafts, tanks, warships, and missiles low visible or ideally invisible to the enemy's radar. For the stealth performance, the RCS (radar cross section) of weapon systems should be minimized because the distance detected by radar is proportional to the fourth root of the RCS [4-5]. Radar cross section (RCS) is the measure of a target's ability to reflect radar signals in the direction of the radar receiver i.e. RCS is a measurement of the strength of the radar signal backscattered from a target object for a given incident EM wave power to describe the extent to which an object reflects an incident EM wave [2,4-5]. The RCS of an object can be reduced basically by shaping the external features of the target in such a way that reduce the EM waves backscattered to the direction of radar source or by employing the coating of radar absorbing materials on the target surface [7,9,13-15].

In civil sectors, microwave absorbing Nano-composites are attracting more attention recently due to the extensive growth in the application of electronic devices such as computer local area networks, mobiles phones, laptops, microwave oven, palmtops, medical equipments, Wi-Fi systems, satellite communications, bank ATM etc. The Electromagnetic (EM) noise generated from these devices is increasing day by day. These unwanted EM noise/ increasing environmental pollution of microwave irradiation are sources of common problems for malfunctioning of various electronic devices, formation of false / ghost images, inconsistent signals etc. due to cross-talk. To eliminate these problems suppression of electromagnetic interference (EMI) and electromagnetic compatibility (EMC) is needed [1-5].

A typical absorber consists of an absorptive layer that is backed by a metal sheet or foil, as shown in figure 1 [4].

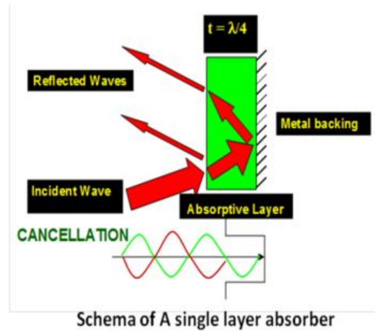


Figure 1: Metal backed Single layer absorber

When microwave impinges on the absorptive layer, electrons present in the materials move under the microwave-induced magnetic field, which then induces eddy current loss due to resistive heating. These eddy currents create secondary small magnetic fields opposite to the excitation field causing reflection of the incident microwave. In this process, a significant part of the incident wave is reflected at the air-absorber interface, some get absorbed in the material due to the dielectric and magnetic losses (ε_r", μ_r") and rest is reflected back from metal backing. These two reflected waves are out of phase by 180^0 and cancel each other at air-absorber interface, if the complex permittivity, permeability, and thickness of the absorbing layer are adjusted to the quarter wave thickness (t) criteria [4]:

$$t = \frac{\lambda_0}{4\sqrt{(|\mu_r|.|\varepsilon_r|)}} \quad (1)$$

Where, λ_0 is the free space wavelength of the incident wave and $|\mu_r|$ and $|\varepsilon_r|$ are the moduli of μ_r and ε_r respectively.

In the case of metal backed single layer absorber, the normalized input impedance (Z_{in}) with respect to its impedance in free space and the reflection loss (R_L) in decibel (dB) with respect to the normal incident plane wave are given by [4,5]:

$$Z_{in} = \sqrt{(\mu_r/\varepsilon_r)}\ \tanh[(-j2\pi/c)\sqrt{(\mu_r\varepsilon_r)}\ f.t] \quad (2)$$

$$R_L\,(dB) = -20\ \log_{10}[|(Z_{in}-1)/(Z_{in}+1)|] \quad (3)$$

Where, $\mu_r = \mu_r' - j\ \mu_r''$ and $\varepsilon_r = \varepsilon_r' - j\ \varepsilon_r''$ are the relative complex permeability and permittivity of the absorber medium respectively, t is the sample thickness, c is the velocity of light and f is the frequency of microwave.

A lot of researches are going on to study the various types of ferrites such as Alpha ferrite, Gamma Ferrite, Zn ferrite, Mn ferrite, Ni-Zn ferrite and Mn-Zn ferrite for absorption of Microwaves. In the present paper, we have studied and reported the electromagnetic and reflection loss properties of Gamma Nano-ferrites (wt. % 20, 40 and 60) in epoxy matrix based Nano-composites in 2-12 GHz frequency range.

II. EXPERIMENTAL

2.1 MATERIALS: The following materials were used for study

(i) Araldite LY 5052 which is a low viscosity epoxy resin.
(ii) Aradur HY 5052 which is a mixture of polyamines.
(iii) Gamma (γ) Nano-ferrite

2.2 Preparation of the microwave absorbers of Gamma (γ) Nano-ferrite filler in Epoxy based matrix

Epoxy consists of two components (i) Araldite LY 5052 and (ii) Aradur HY5052. These components were thoroughly mixed in 100:38 ratio (by weight) to prepare epoxy matrix. The ingredients for the synthesis of γ Nano-ferrite filler in epoxy based Nano-composite absorbers are given in table 1. The fabrication of the Nano-composite sample has been carried out using wt% 20, 40 & 60 of γ Nano-ferrite filler in epoxy matrix. The γ Nano-ferrite filler was thoroughly mixed in two component epoxy matrix. The mixture was homogenized in mortar and pestle. Then the material was loaded in a specially designed mould to produce torroidal shaped Nano-composite absorber samples. The mould was then kept in a hydraulic press moulding machine. The machine was then switched on for a certain period of time at pre-selected pressure & temperature (not disclosed). Hydraulic press was then switched off and allowed to cool naturally to ambient temperature. The material was then taken out from the mould and finally the torroidal shaped samples with ID: 3mm, OD: 7mm were obtained.

Table 1: Sample compositions for γ Nano-ferrite filler in epoxy matrix

Sample No.	Wt % of γ Nano-ferrite filler	Wt % Epoxy Matrix
1.	0	100
2.	20	80
3.	40	60
4.	60	40

2.3 Characterization of samples:

(a) Complex Permittivity (ε) and complex permeability (μ) of the samples have been measured by employing the two port Vector Network analyzer (Model PNA E8364B, Software module 85071E) technique in 2-18 GHz frequency range.
(b) Surface morphological and microstructure of Nano-composite samples were studied using scanning electron microscope (SEM) (Carl Zeiss SEM EVO 50XVP)
(c) Thermo gravimetric analysis (TGA) of samples was performed to study the thermal stability and thermal degradation behaviour on a thermal analyzer (Mettler Toledo, USA) in the temperature range between 20 and 800 °C for samples at a heating rate of 5°C/min under air atmosphere at a flow rate of 20 ml/min.

III. RESULTS AND DISCUSSION

3.1 Microwave parameters (complex permittivity and permeability):

Figure 2 shows the complex permittivity of virgin epoxy matrix in 2-18 GHz frequency range. The permittivity of virgin epoxy matrix is varying very marginally with the applied frequency from 2-12 GHz. It shows that the electrical polarisibility (interfacial) of the material has changed very small value. Imaginary part of the spectra shows that there is negligible loss with the applied frequency.

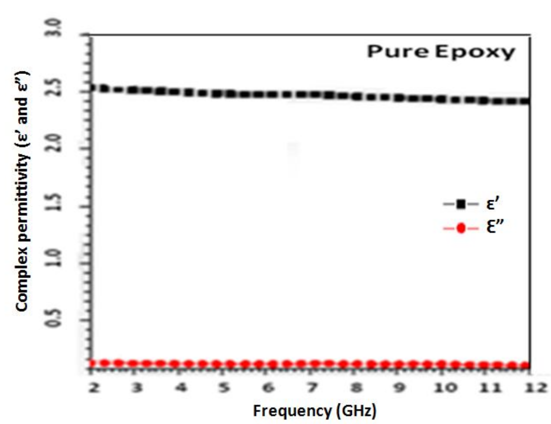


Figure 2: Complex Permittivity of Virgin Epoxy Matrix

Figure 3 shows the complex permeability of virgin epoxy matrix in 2-18 GHz frequency range. The flat frequency independent response of permeability spectra shows that virgin epoxy matrix is unaffected /unperturbed because it is un-doped.

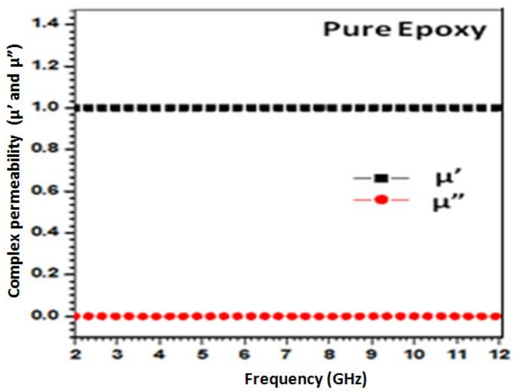
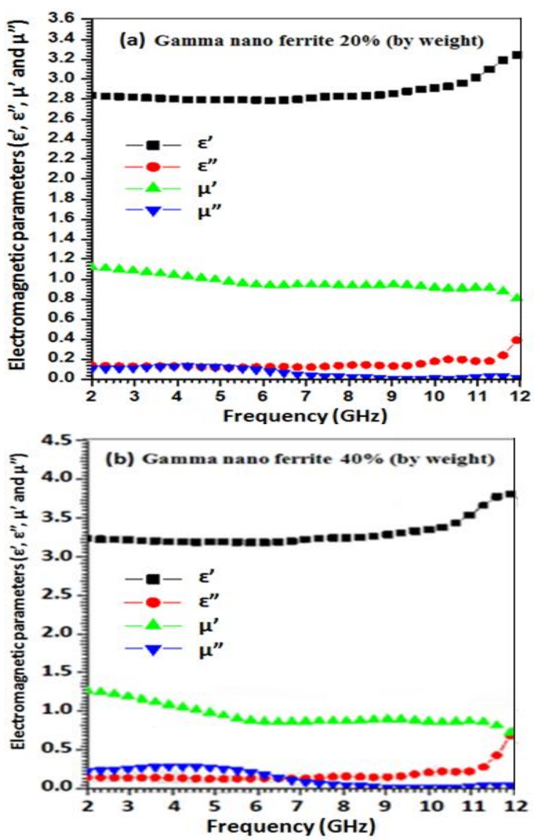


Figure 3: Complex Permeability of Virgin Epoxy Matrix

Figure 4 (a), (b) and (c) show the variation of complex permittivity (ε' and ε") and complex permeability (μ' and μ") of various compositions (wt % 20, 40 and 60) of gamma Nano-ferrite fillers in epoxy matrix.



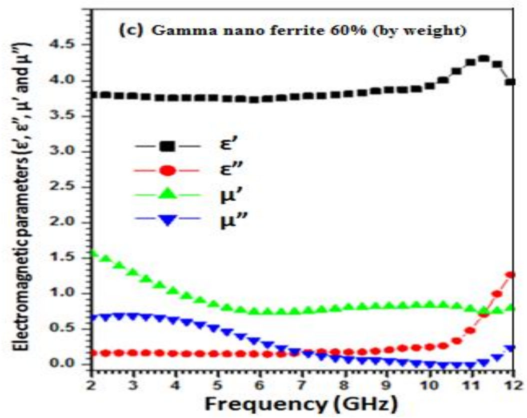


Figure 4: Complex Permittivity and Complex permeability spectra of Gamma (γ) Nano-ferrite (a) wt% 20, (b) wt% 40 and (c) wt% 60 in epoxy based Nano-composites

From figure 4 (a), (b) and (c), it is observed that similar frequency response of the electromagnetic parameters (ε' ε", μ' and μ") are shown. Further, it is also evident from figure 4 (a), (b) and (c), with increasing γ-ferrite content, di-electric constant (ε') also increases, where as di-electric loss (ε") is frequency dependent. Complex permeability (μ' and μ") shows similar pattern with its value fluctuating around μ'=1

3.2 Reflection loss (R_L) properties:

The reflection loss R_L (in dB) of the various compositions (wt .% 20, 40, 60) of Gamma (γ) Nano-ferrite filler in epoxy based Nano-composites have been calculated by employing equations (2) and (3) for various thicknesses (t) using experimentally obtained values of ε_r and μ_r for metal backed single layer condition as shown in figure 5 (a), (b) and (c). In figure 5 (a), it is evident that the reflection loss R_L (dB) for 20% Gamma (γ) Nano-ferrite/epoxy Nano-composite increases with increasing thickness from t=4.0 mm to 9.0 mm and shifted towards lower frequency side. The reflection loss R_L (dB) at matching frequency (f_m) for different thicknesses (t) is summarized in table 2.

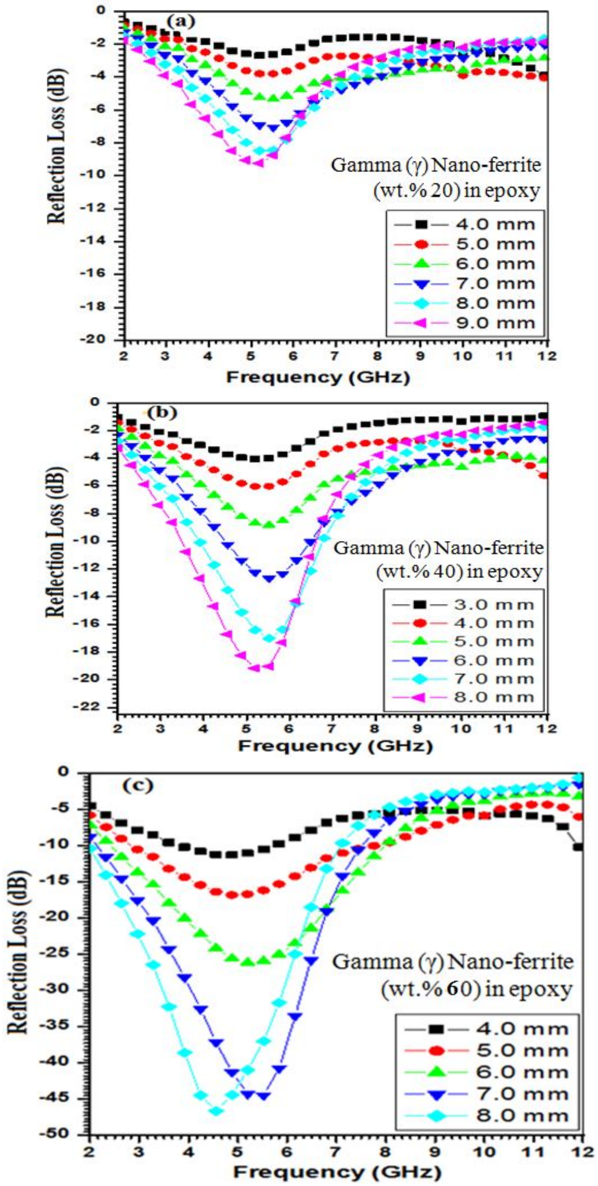


Figure 5: Reflection Loss (dB) Vs. Frequency (GHz) response at various thicknesses (t) for different compositions

Table 2: Reflection loss R_L (dB) at matching frequency (f_m) for 20% Gamma (γ) Nano-ferrite/epoxy Nano-composite different thicknesses (t)

$R_{L,min}$ (dB)	Matching frequency (f_m) (GHz)	Thickness (mm)	Radar Band
-2.6486	5.52	4	C
-3.8086	5.52	5	C
-5.3191	5.52	6	C
-7.0524	5.52	7	C
-8.4853	5.2	8	C
-9.2333	5.2	9	C

In figure 5 (b), it is evident that the reflection loss R_L (dB) for 40% Gamma (γ) Nano-ferrite/epoxy Nano-composite increases with increases thickness from t=3.0mm to 8.0 mm. The reflection loss R_L (dB) at matching frequency (f_m) for different thicknesses (t) is summarized in table 3.

Table 3: Reflection loss R_L (dB) at matching frequency (f_m) for 40% Gamma (γ) Nano-ferrite/epoxy Nano-composite different thicknesses (t)

$R_{L,min}$ (dB)	Matching frequency (f_m) (GHz)	Thickness (mm)	Radar Band
-4.0543	5.2	3	C
-6.0385	5.52	4	C
-8.8391	5.52	5	C
-12.6723	5.52	6	C
-17.0209	5.52	7	C
-19.1695	5.52	8	C

In figure 5 (c), it is evident that the reflection loss R_L (dB) for 60% Gamma (γ) Nano-ferrite/epoxy Nano-composite increases with increases thickness from t=4.0 mm to 8.0 mm. The reflection loss R_L (dB) at matching frequency (f_m) for different thicknesses (t) is summarized in table 4.

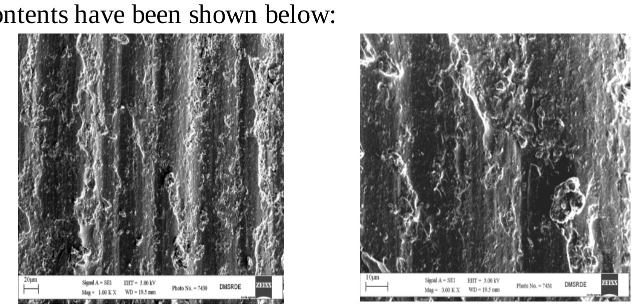
Table 4: Reflection loss R_L (dB) at matching frequency (f_m) for 60% Gamma (γ) Nano-ferrite/epoxy Nano-composite different thicknesses (t)

$R_{L,min}$ (dB)	Matching frequency (f_m) (GHz)	Thickness (mm)	Radar Band
-11.3163	4.88	4	C
-16.8141	4.88	5	C
-26.1573	5.2	6	C
-44.5030	5.52	7	C
-46.4380	4.56	8	C

From figure 5 (a), (b) and (c), it is also observed that with increasing Gamma (γ) Nano ferrite content from 20% to 60%, the reflection loss also increases drastically.

3.3 Scanning Electron Microscopy of γ Nano- ferrite filler/Epoxy Matrix absorber

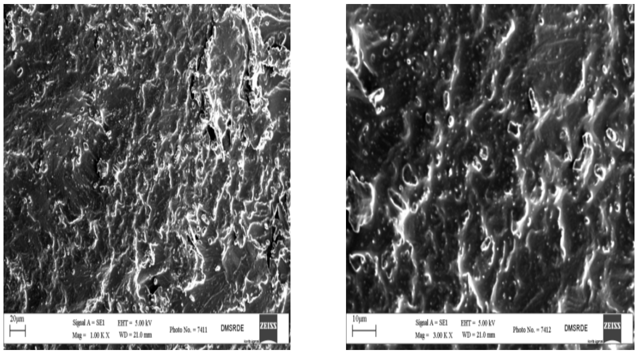
The Morphological properties of the virgin epoxy matrix & Nano-composite samples have been analyzed by scanning electron microscopy (SEM) (Carl Zeiss EVO-50). The SEM micrographs of the samples having different filler contents have been shown below:



(a) 1 KX magnification **(b) 3 KX magnification**

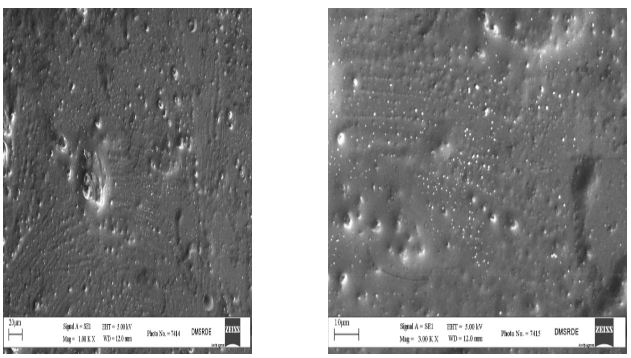
Figure 6: SEM of Pure Epoxy Matrix

Figure 6 shows the micro-structure of virgin epoxy matrix at 1K and 3 K magnifications. Figure 7, 8 and 9 shows the micro-structures of Nano-composites with increasing Gamma (γ) Nano-ferrite fillers (wt. % 20, 40 and 60) at 1K and 3 K magnifications.



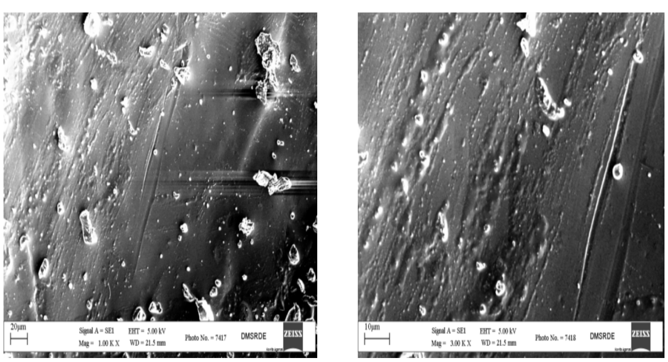
(a) 1 KX magnification **(b) 3 KX magnification**

Figure 7: SEM of 20% γ Ferrite in Epoxy Matrix



(a) 1 KX magnification **(b) 3 KX magnification**

Figure 8: SEM of 40% γ Ferrite in Epoxy Matrix



(a) 1 KX magnification **(b) 3 KX magnification**

Figure 9: SEM of 60% γ Ferrite in Epoxy Matrix

It is evident that with increasing filler content, the filler becomes more prominent in the micro structure as obvious.

3.4 Thermo-gravimetric analysis (TGA) of Gamma (γ) Nano-ferrite / Epoxy Nano-composites

Thermo-gravimetric analysis (TGA) has been done to study the thermal stability and degradation behavior of the prepared Nano-composites. The thermographs are shows in figure 10 (a) and (b) for two different compositions of Gamma (γ) Nano-ferrites i.e. 20% and 40% respectively in epoxy matrix. TGA shows that the two step decomposition of the Nano-composite. The first decomposition starts around 350 ^{0}C due to Araldite and second decomposition starts at 710 ^{0}C due to Aradur. From TGA it has been confirm that the Nano-composite is thermally stable upto 350 ^{0}C.

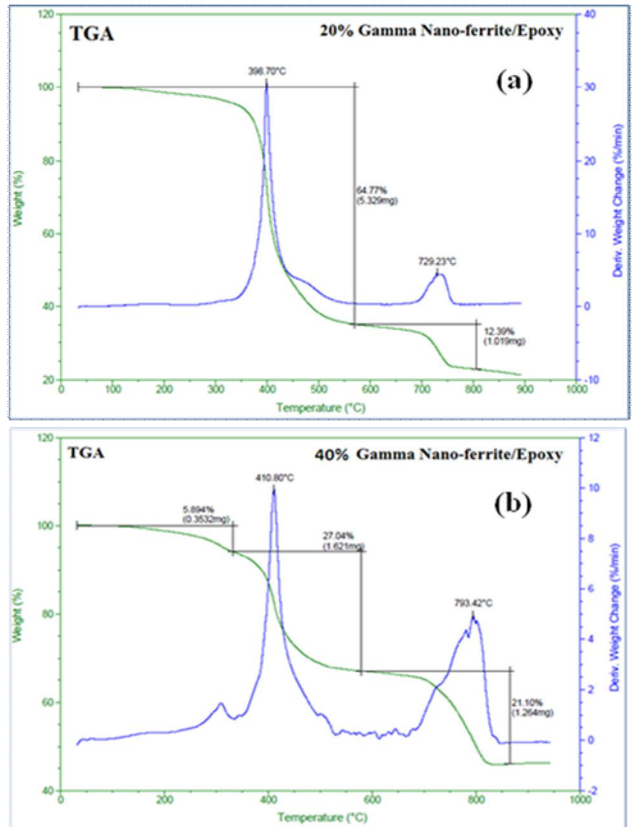


Figure 10: TGA thermographs of γ Nano-ferrite (a) 20% and (b) 40% in epoxy based Nano-composites

IV. CONCLUSIONS

With increasing γ ferrite content, the reflection loss drastically increases with thickness (t) in 2-12 GHz frequency for various compositions of γ ferrite Nano-composite. The surface morphology of the samples has been studied with the help of SEM. The SEM micrographs of various compositions are evolved with increasing γ ferrite content. SEM confirms micro structure of the Nano-composite. The thermal behaviours of the Nano-composites have been studied with TGA. Different samples were found to have different thermal degradation behaviour with thermal stability up to 350 ^{0}C. We have studied the microwave absorption properties in the S, C and X (8-12 GHz) bands. The maximum reflection losses have been found to increase with increasing thickness of the Nano-composite. The electromagnetic parameters (ε, μ) are found to vary with applied frequency of microwave. The Nano-composite has potential application as a Radar absorbing material, electromagnetic shielding screens, as coatings or jackets and stealth in S (2-4 GHz), C (4-8 GHz) and X band (8-12 GHz).

V. ACKNOWLEDGEMENT

Authors are thankful to Dr. N Eswara Prasad, Outstanding Scientist & Director DMSRDE, Kanpur for permitting and providing facility this work. Authors are thankful to Vice chancellor Dr. S Pal, DIAT, Pune and Dr. Sangeeta Kale, Dean and Head of Department (Applied Physics) DIAT, Pune for their support, motivation and guidance. Authors are also thankful to Dr. TC Shami and Shri Alok Dixit for their support the measurement of electromagnetic properties and Dr. Kavita Agarwal for Scanning electron microscope (SEM) of the samples.

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