

Microwave absorption property of hydrothermal synthesized RGO/PbFe₁₂O₁₉ nanocomposite

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Abstract- In this work, we have studied the microwave absorption properties of nanocomposite of Hexagonal shaped lead hexaferrite (PFO) with reduced graphene oxide (RGO). The hard magnetic lead hexaferrite powder is synthesized using solution combustion method and characterized using powder XRD. Nanocomposite of PFO-RGO is prepared using hydrothermal method. The as prepared nanocomposite is characterized using powder XRD and FESEM. The microwave absorption study of PFO-RGO composite is studied in 2-18 GHz. The simulated RL loss plot suggests that the PFO-RGO composite can achieve a minimum RL of -45 dB at 12 GHz. Therefore, this composite system can be used as promising microwave shield material.

Keywords—nanocomposite; lead hexaferrite; RGO; Microwave absorption.

I. INTRODUCTION

Ever since the WWII, the researches in the radar absorbing materials have been increased, in order to enhance the invisibility of war aircraft. These signal generally lies in the microwave frequency band of X and Ku bands, therefore the absorption of these frequency is essential for achieving a better stealth aircraft. Various oxide materials and dielectric material have been used in past for achieving better absorption of radar signal.[1] Apart from research point of view different engineered absorber have been designed for better attenuation of microwave, such as single layer Jaumann, Salisbury absorber, double layered absorber, iron ball paints[2, 3]. But this structure has been very costly and the fabrication needs a thick layer of absorber which makes the planes efficiency very low. Thus the layered of paint or composite with dielectric or magnetic lossy embedded particle have been attracted for this purpose.[4]

In the past, to attempting the microwave absorption various dielectric materials has been synthesized..[5] For this purpose the composite material has shown great advantage over the single media absorber. Apart from magnetic and dielectric media, graphene and CNT has been used for microwave absorption[6]. These carbon nanomaterials have shown excellent properties like high conductivity, high aspect ratio, high mechanical strength, and light weight. Graphene has shown better microwave absorption because of its 2D layered structure with the defect of the layered plane and the defect of

its surface because of C=O and C-H group which enhance the polarizability.[7]

Thus the composites of graphene and dielectric/ magnetic material have been shown a great potential for microwave absorption[8]. Apart from dielectric loss, magnetic loss of EM wave is further synchronized the absorption of microwave through various magnetic loss phenomenon. For that purpose composite of ferrites, garnets, hexaferrite[9] and magnetic material has been explored for microwave absorption over the past year. Hexaferrite has been advantageous over ferrite material due to its merit such as high magnetic anisotropy field, high coercivity, and high chemical stability. In this work the composite of lead hexaferrite with graphene has been studied for microwave absorption in the X and Ku band. The reflection loss of composite material has been calculated for the composite.

II. EXPERIMENTAL PROCEDURE

Synthesis of lead hexaferrite; Lead hexaferrite has been synthesized by sol-gel solution combustion route. Pb(NO₃)₂, Fe(NO₃)₃·9H₂O and urea were taken in 100 ml borosilicate beaker with 15 ml Deionized water. The stoichiometric composition nitrates and fuel were calculated based on propellant chemistry calculation for combustion. The prepared solution was then heated at 70 °C with constant stirring under magnetic hot plate to dissolve the solute in the solution. The prepared solutions were having an acidic pH value of 1-2. Ammonia was added drop wise with constant stirring until the final pH reached to the value of 8-9. When the pH of solution reaches to the value of 8-9, we observe the formation of gel. These homogeneous gels were kept in a muffle furnace which was preheated to 500 °C for auto combustion. The as burnt black colored were crushed in a mortar pestle and then calcined at 1000 °C for 1 hour in muffle furnace.

Synthesis of RGO-PFO composite; RGO-PFO composite has been synthesized by reducing the graphene oxide powder hydrothermally. For this purpose GO was synthesized by Hummers method.[10] This GO powder and PFO (1:1 ratio) were added to 25 ml DI water with 2 ml of Hydrazine hydrate. The solution was sealed in 50 ml Teflon autoclave and kept at 160 °C for 18 hours. After the completion of hydrothermal process, the obtained solution was washed with DI water and

dried in oven at 80 °C for 12 hours. For the comparison RGO powder without PFO were also prepared with same procedure as above without added PFO powder in it.

Characterization: The structural characterization of samples were carried out by using “PANalytical” X-ray diffractometer (Cu-K_α radiation, Ni filter) in the θ -2 θ geometry in 2 θ range of 10-90°. The phases were identified by comparing their respective Bragg positions with the standard ICSD database. Surface morphology of calcined samples was obtained using Field emission scanning electron microscope (FESEM). Microwave absorption in the X and K_u band frequency has been performed using a vector network analyzer (VNA), (Anritsu, MS4642A). The composites were compression molded to toroidal shaped donuts to fit in the coax measurement setup. The prepared RGO-PFO composite were mixed with paraffin wax and pressed into toroidal shaped samples (\varnothing_{out} = 7 mm; \varnothing_{in} = 3 mm) with 5 mm length. The complex permittivity and permeability has been calculated from the Nicholson Ross algorithm. [11]

III. RESULTS AND DISCUSSION

Figure 1a shows the powder XRD pattern of RGO-PFO composite. The peak at 25° confirms the formations of RGO as well as the PFO peaks are well matched with the ICSD database of standard lead hexaferrite. Figure 2 shows the FESEM image of RGO-PFO composite. The hexagonal shaped lead hexaferrite and planer graphene sheet are observed in SEM image. The lead hexaferrite particles are agglomerated due to its magnetic anisotropy.

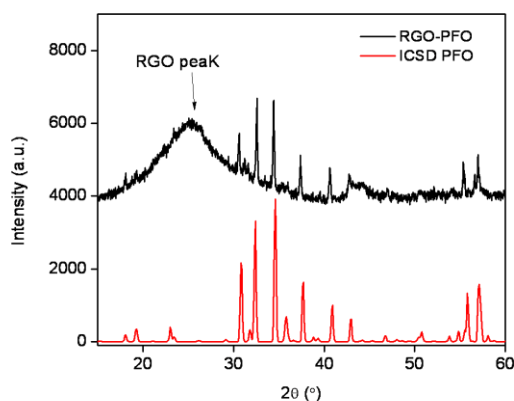


Fig. 1 XRD pattern of RGO-PFO powder

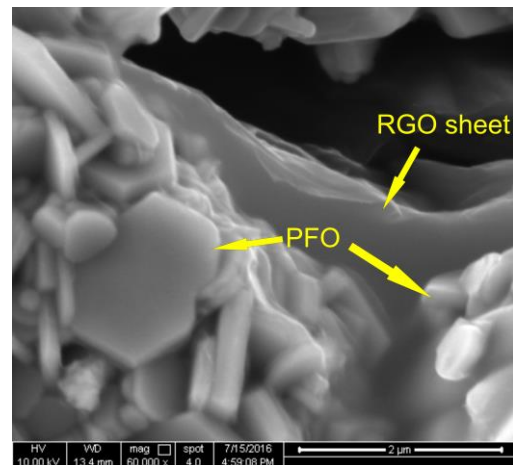


Fig. 2 FESEM image of RGO-PFO composite

Microwave absorption study: according to EM theory the dielectric and magnetic loss both contribute the microwave absorption. to investigate the microwave absorption mechnism we have obtained the complex permittib=vityan d permeability in the 2-18 GHz. the complex permittivity and permeability are given as the follwoiung equations

$$\epsilon = \epsilon' - \quad (1)$$

$$\mu = \mu' - \quad (2)$$

The real part of permittivity and permeability represent the storage of electric and magnetic energy, whereas the imaginary parts represent the electrical and magnetic loss in the samples, respectively.

Figure 3 shows the complex permittivity and complex permeability of the composites in the 2-18 GHz. there are two relaxations peaks observed at 13 and 17 GHz. These relaxation peaks could be attributed to the Debye relaxation in the samples. The complex permeability plot also shows two peaks at 11 and 17 GHz. The presence of magnetic PFO particles leads to the magnetic resonance in the composite which result in the magnetic loss in the specimen.

The reflection loss can be calculated using the permittivity and permeability dat using the following equation s

$$RL (dB) = 10 \log \frac{|Z_{in}|}{|Z_{in}|} \quad (3)$$

$$Z_{in} = \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left(j \frac{2\pi f d}{c} \sqrt{\epsilon_r} \right) \quad (4)$$

The thickness dependent reflection loss plot is showing in figure 3c. The RL loss values are simulated form the permittivity and permeability data. A minimum reflection loss of -40 dB is observed at 7.5cm thick specimen at 13 GHz. there are multiple peaks also observed at different thickness and with varying frequency. These losses in the composite can be attributed to the magnetic losses from the PFO and dielectric loss can be attributed to the RGO. Therefore, composite of RGO-PFO makes better microwave absorber.

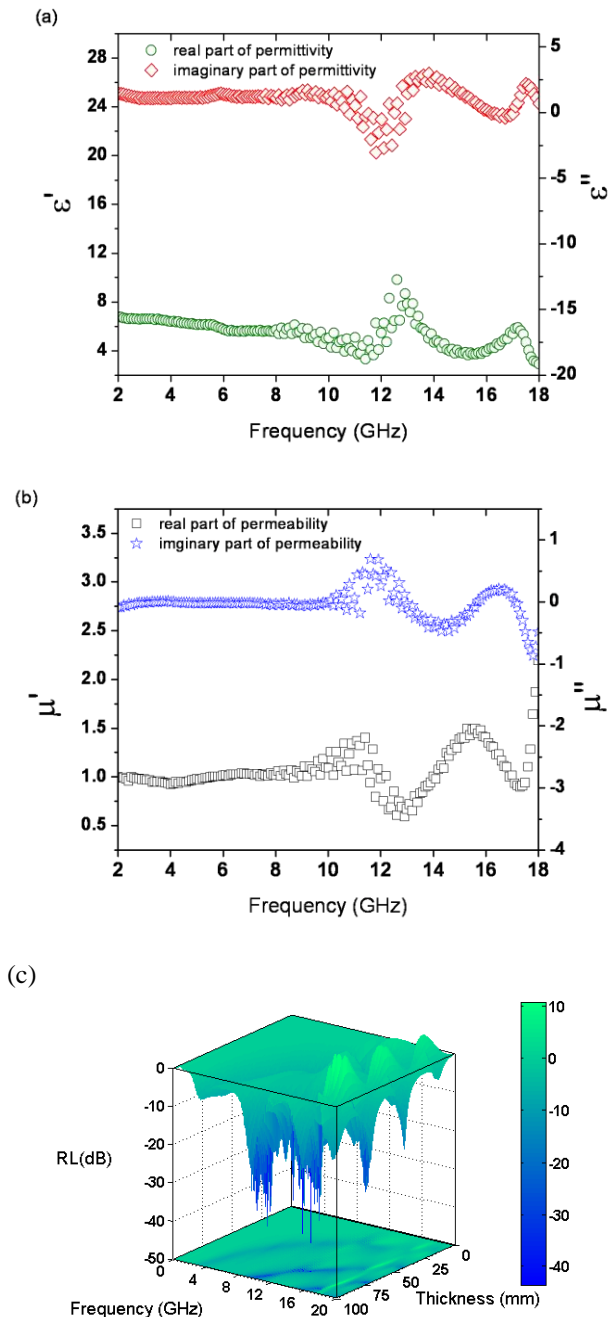


Fig.3 (a) complex permittivity, (b) complex permeability and (c) Simulated 3D reflection loss plot of RGO- PFO composite.

IV. CONCLUSION

The magnetic PFO sample is synthesized using sol-gel combustion method. The as prepared PFO powder mixed with

GO powder and the RGO-PFO composite is prepared using hydrothermal method. The as prepared composite is characterized using power XRD and its surface morphology is imaged using FESEM. The microwave absorption study of the composite in 2-18 GHz is studied using VNA. The simulated reflection plot shows a minimum reflection loss of -40 dB at 7.5 cm thickness. These primary results shows the RGO- PFO composite can be used as radar shield layer.

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