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Engineering Magnesium Ferrite-Graphite Composites for Microwave Shielding

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Abstract—*Development of high performance shielding materials demands the need of composite material. Composite of ferromagnetic, highly resistive magnesium ferrite with conducting graphite has been synthesised by solid-state reaction method. Nanoparticles of magnesium ferrite-graphite composite have been synthesized by Solid-State method. Composition of magnesium ferrite and graphite has been taken in 95:05, 85:15 and 75:25 by weight percent . Double phase composite has been confirmed by X-ray diffraction pattern. Crystallite size of ferrite has been determined 3nm to 4nm with increasing graphite weight ratio. Size of grains in the range 100–300nm has been measured by Scanning electron microscope. Magnetization measurement of magnesium ferrite nanoparticles has been analysed by Vibrating sample magnetometer. Magnetization of the composition found to increase from 10emu/g to 20 emu/g with increasing graphite ratio. Morphology of the composite exhibited uniform distribution of both the compounds. Grain size of ferrite decreased with increasing graphite ratio. Shielding effectiveness of the composite has been investigated by Vector Network Analyzer (VNA) in X band (8-12 GHz) region. Absorption dominated shielding effectiveness (SE) of the composite 75:25 has been obtained 14.97dB and Effective absorption ($Aeff$) is 18.323 dB at 12 GHz depicting effective microwave absorption. High permeability and high magnetization properties make effective microwave absorbers of ferrite-graphite composite(75:25) can be used in broadband transformers and stealth materials.*

Keywords*: Magnesium ferrite, Solid State Reaction Method, Magnetization, Effective Absorption.*

1. INTRODUCTION

Nowadays the proliferation of electronics & instrumentation in Industrial, commercial, healthcare and defence sectors has led to a novel kind of pollution known as "Electromagnetic Interference" which is due to interference of current induced by electric field and magnetic field emanating from wide range of electrical circuitry.

Therefore the need for stealth materials evolved to shield the instrument/device from spurious electromagnetic radiation. Since metals tend to reflect, thus need of highly resistive ferrite composites gained demand over last four decades. Several attempts were taken to combine the good electrical properties of graphene with ferrites, but very few attempts were initiated for magnesium ferrite due to its lower magnetic moment. A CNT Polystyrene foam structure composite with an Electromagnetic Interference Shielding effectiveness (SE) of 19dB has been reported [6]. Synthesis of CNT's encapsulated with Fe nanoparticles have shown good absorption behaviour [7]. EMI Shielding effectiveness (SE) up to 21dB at 8.2GHz for solution processable functionalized graphene (SPGF) in to an epoxy containing 15 wt% (8.8 vol%) SPEG has been reported [8].

In present paper we have reported the potential of Magnesium ferrite graphite composite to absorb and dissipate incident electromagnetic waves by converting them to thermal energy. The resulting composite possesses good magnetic permeability and high dielectric properties with moderate conductivity making it feasible stealth material against EM radiation.

2. EXPERIMENTAL

Magnesium ferrite-Graphite composite was synthesized by solid-state reaction method, where $MgCO_3$ and Fe_2O_3 has been taken in 1:1molar ratio followed by wet grinding in acetone using Agate Mortar Pestle for 1 hour. Homogenized ground powder was pre-sintered at 800 degrees for 12 hours.

Pre-sintered magnesium ferrite and graphite powder were taken in weight ratio95:05(Sample A), 85:15 (Sample B) and 75:25 (Sample C). Mixed powder was then grinded for 30 minutes followed by pelletization. Samples A, B and C were pressed in to rectangular pellets of size 22.83 mm x 10.13 mm for X-band frequency range. Pressed pellets were annealed at 650^{o}C for 4 hours in vacuum furnace.

Structural characterization was carried out by taking X-ray diffraction pattern using Bruker Axes diffractometer . Magnetization of the samples was determined by Vibrating Sample Magnetometer, Lakshore 7304. Microstructure of composites was analyzed by taking Scanning electron microscopy, Zeiss. Shielding effectiveness of the composite was investigated for X-Band (8 to 12 GHz) using Vector Network Analyzer (VNA), Agilent.

3. RESULTS AND DISCUSSION

X-ray Diffraction

Crystalline phase formation of composites has been analysed by X-ray diffraction pattern as shown in Fig.1. Diffraction pattern of composite shows existence both phase peaks distinctly. In XRD pattern diffraction peaks at $2\theta=28^0$,33^0,49^0,55^0 corresponds to diffracted from graphite planes (002) (101) (104) respectively. Diffracted peaks at $2\theta=30^0$,45^0,58^0,62^0 signifies (111) (311) (400) (440) planes of spinel ferrite. The observed peaks matched with standard pattern for cubic spinel $MgFe_2O_4$ (ICDD-01-073-1720) and graphite (ICDD -01 2536). The crystallite size of $MgFe_2O_4$ was calculated using Debye-Scherrer formula. It was found to be 3-4 nm range. With increasing graphite ratio, intensity of graphite plane peaks increased.

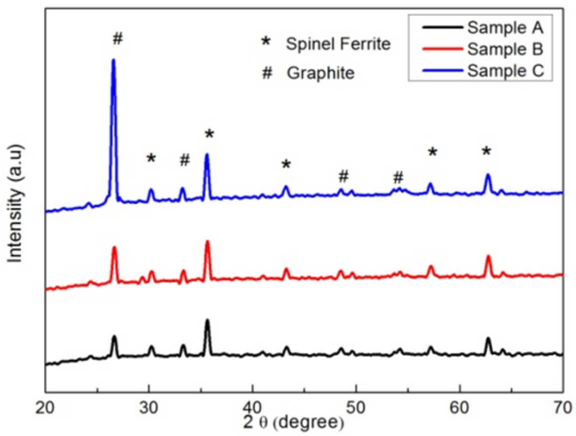


Fig. 1: XRD of $MgFe_3O_4$-Graphite Composite for Sample A, B and C

4. MAGNETIZATION MEASUREMENTS

Magnetic behaviour of synthesized composite pellet has been observed by taking magnetization loop is shown in Fig.2. Shape of the MH loop implies soft magnetic nature of the composite, wherein magnetization decreases from 20.5 emu/g for sample A to 10.1 emu/g for sample C .The magnetization decreases with increase in graphite content in the composite, due to decreasing weight ratio of magnetic ferrite material. Coercivity and remanent behaviour of the composite decreased with increase in graphite content. It suggests that graphite help in easy domain wall motion of ferrite and improves the magnetic dipole switching.

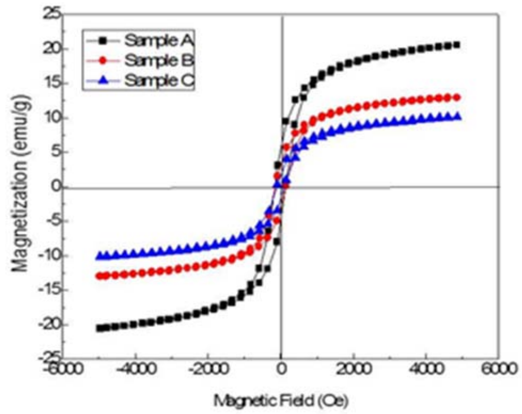


Fig. 2: M-H Curves for $MgFe_3O_4$ –Graphite Composite Sample A, B & C.

5. SCANNING ELECTRON MICROSCOPY

Microstructure of the composite pellets has been observed by taking scanning electron micrograph images as shown in Fig.3 (a-f). SEM image of sample A Fig. 3 (a) depicts homogeneous distribution of two phases. A wide range of grain size distribution from 3nm to 4 nm has been observed. Porous microstructure has been obtained for higher ferrite ratio. Pore size distribution has been observed from 0.01-2 µm range.

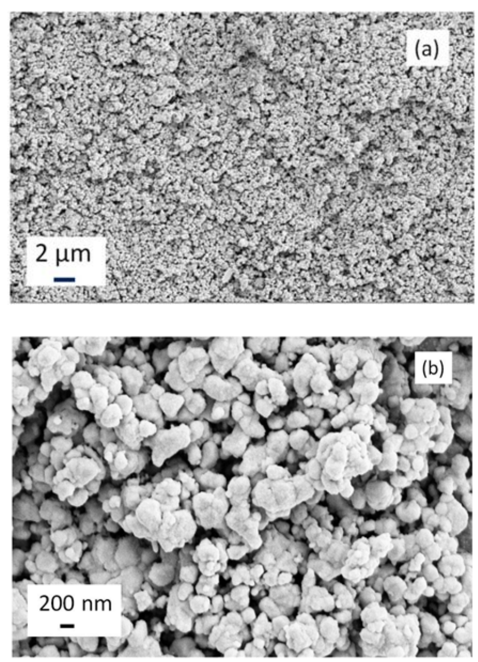


Fig. 3(a, b) SEM images of Sample A at 6K and 49K magnification.

Fig.3 of sample B, exhibited that tiny grains collapsed on large grains. Also small grains coalaesce to form well connected aggregate large grains. It depicted increasing graphite inclusions with less pore size distribution.

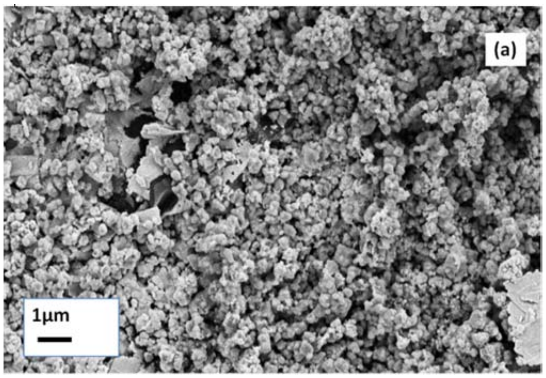


Fig. 3(a) SEM images of Sample B at 10K magnification.

SEM image Fig. 3(e-f) of sample C depicts fused grains of bigger size along with less tiny grains.

Wide pore size distribution with collapsed grains has been observed with increased graphite ratio.

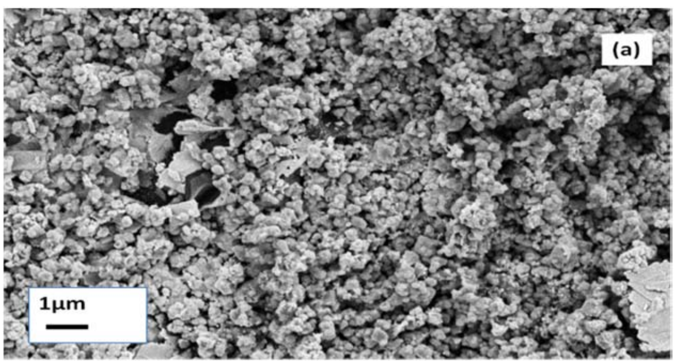


Fig. 4: SEM images of Sample C at 10K and 63 K magnification.

6. MICROWAVE ABSORPTION MEASUREMENTS

Microwave shielding capability of magnesium ferrite-graphite composite has been carried out by taking shielding effectiveness in X-band (8-12 GHz) as shown in Figure 6. The total shielding efficiency (SE_T) relates the incident EM wave to the transmitted one as [5]:

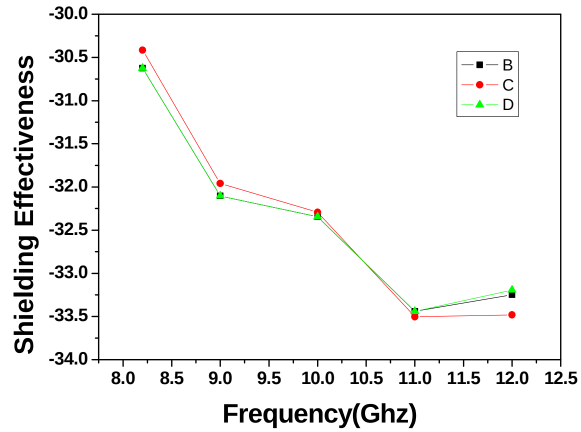
$SE_T(dB) = 10 \log(P_I/P_T) = SE_R + SE_A$ (1)

$SE_R = 10 \log(1 - R)$ (2)

$SE_A = 10 \log(1 - A_{eff}) = 10 \log[T/(1 - R)]$ (3)

where SE_T is a logarithmic ratio in decibels (dB), T, R and A_{eff} represents the transmission, reflection and absorption coefficients. SE_R represents the shielding effectiveness due to reflection and SE_A represents the shielding effectiveness due to absorption.

Magnesium ferrite shows effective shielding due to reflection constant for the X-band (8-12 GHz).



SE due to absorption exhibit sharp increase from sample A to sample C. The maximum value of SE has been observed 14.97dB and A_{eff}= 18.32 dB at 12 GHz for sample C revealing effective microwave absorption by the composition. Phenomena responsible for microwave absorption in magnesium ferrites is due to ferromagnetic resonance where precision frequency of unoccupied d-orbital (responsible for magnetic moment) matches with the incident RF frequency, thus a resonance occurs which is depicted by absorbance peaks of RF frequency [3]. However magnetization value of composite decreases by increasing graphite ratio. Moreover it has been observed that increase in graphite concentration both S11 (reflection coefficient), S22 (transmission coefficient) and return loss increased sharply from -0.312 dB to -0.752 dB with increase in frequency. T he rise in conductivity and grain connectivity by increasing graphite ratio in ferromagnetic, low loss, high resistive magnesium ferrite, resulting in to increase in microwave absorption for the composite sample C. The absorption in X-band frequency range may be due to increasing dielectric loss in the ferrite-graphite composite, where graphite being conducting increases the dielectric loss in composite. Electromagnetic energy concentrates due to high magnetic permeability properties of ferrite inclusions and due to polarization of conducting graphite in dielectric magnesium ferrite base, of sample C shows maximum value. Energy dissipates mainly due to composites interfacial boundaries formation in the samples in X-band (8 to 12 GHz), due to conductivity of graphite.

7. CONCLUSION

Microwave absorbing ferrite-graphite composites were prepared using Solid-State Reaction Method. The optimum absorption value with return loss lower than -10dB was obtained at 12 GHz for 75:25 magnesium ferrite-graphite ratio maximum for (sample C). With increase in graphite content, the minimum reflection loss increases with increase in frequency in X-Band. With excessive increase in graphite content, absorption peak reduces, suggesting that ratio of graphite has optimum value for microwave shielding.

Shielding effectiveness of composite has been explored for X-Band that shows maximum value of 14.97dB for sample C at 12 GHz . Such high value of shielding confirms the composite formed may be used as effective microwave absorber in X-Band**.** Thus energy is absorbed synergistically by composite of magnesium ferrite and graphite is an inexpensive and effective alternative for microwave stealth applications.

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