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A microwave absorber based on strontium ferrite–carbon black–nitrile rubber for S and X-band applications

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ABSTRACT

Flexible and thin single layer microwave absorbers based on strontium ferrite–carbon black–nitrile rubber composites have been fabricated employing a specific recipe and their reflection loss characteristics were studied in the S (2–4 GHz) and X-bands (8–12 GHz). The incorporation of carbon black not only reinforces the rubber by improving the mechanical properties of the composite but also modifies the dielectric permittivity of the composite. Strontium ferrite when impregnated into a rubber matrix imparts the required magnetic permeability to the composite. The combination of strontium ferrite and carbon black can then be employed to tune the microwave absorption characteristics of the resulting composite. The complex dielectric permittivity and permeability were measured by employing a cavity perturbation technique. The microwave absorption characteristics of composites were modelled in that an electromagnetic wave incident normally on the metal terminated single layer absorber. The influence of filler volume fraction, frequency, absorber thickness on the bandwidth of absorption are discussed and correlated.

1. Introduction

Microwave absorbing materials are of vital importance in solving the increasing menace of electromagnetic interference problems caused by the proliferation of wireless communication system and other high-frequency devices in the S and X-band frequencies. They are in demand for reducing radiation reflection as well as providing interference shielding (EMI electromagnetic interference shielding) and protection [1–5]. Flexibility, mouldability and environmental resistance are the main factors to be considered in designing an absorber along with a large absorption capability and wide band width. Rubber Ferrite Composites (RFCs) in the form of sheets are flexible, lightweight and possesses good environmental resistance and has the required mechanical strength. Furthermore, absorptivity and bandwidth of absorption can be tailored by a judicious choice of fillers. The dielectric permittivity and magnetic permeability of the composite is greatly influenced by the volume fraction of fillers in the matrix [6]. This multi-functionality of RFCs makes it a potential candidate for microwave absorbing applications.

Spinel ferrites do not perform well in the Giga Hertz range of spectrum due to a drop in the complex permeability as given by Snoek's limit [7–9]. Compared to spinel and garnets, hexagonal ferrites in the pristine form have been found to be good microwave absorbers in the frequency range of 1–100 GHz because of their high saturation magnetization, coercivity, chemical stability, corrosion resistance and adjustable anisotropy through appropriate ion substitution [9]. Hence incorporation of M-type strontium hexaferrite (SrF) in the rubber matrix enhances the magnetic permeability of the composite and in turn modifies the microwave absorption properties. Carbon and different forms of carbon are being used along with other fillers for enhancing the microwave absorption characteristics. Different forms of carbon, namely, Carbon Nano Tube, Carbon Nano Fibre, Carbon Nano Onion, graphene, reduced graphene oxide, carbonyl iron, etc. are increasingly finding its way as a filler in an appropriate matrix [5,10–14]. Carbon black (CB) is a perfect choice because it not only reinforces the rubber matrix but also modifies the microwave absorption characteristics.

The incorporation of CB in the matrix along with SrF is aimed at achieving two objectives. Primarily, CB is a reinforcing agent and imparts the necessary mechanical strength. The secondary objective is to employ CB as a lossy dielectric wherein it contributes

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to the enhancement of microwave absorption by virtue of imparting a large permittivity to the composite. The strategy has been to choose a composite containing an optimum amount of magnetic filler and having the appropriate magnetic characteristics. Since we intend to incorporate CB further into this optimally synthesized composite, we thought it fit to choose the maximum loading of magnetic filler at around 80 phr. It has been assumed that further addition of CB into this will not go beyond the percolation threshold. Moreover incorporation of 30, 40, 50 phr (parts per hundred weights of rubber) of CB into this will not result in inhomogeneity. This is based on past experience. CB is less expensive when compared to CNT, graphene and other carbonaceous materials.

Excellent microwave absorbing materials in the desired frequency range with wide band width must satisfy two fundamental conditions [15,16], (i) the incident electromagnetic wave penetrates the absorber by the greatest extent (impedance match) and (ii) the wave entering into the materials is entirely attenuated and absorbed within the finite thickness of the absorber. The level of absorptivity of an absorber is measured quantitatively using reflection loss in decibels. The reflection loss of −20dB is adopted to be a standard for typical electromagnetic wave absorbers [17]. It means that 99% of the total energy of electromagnetic wave incident on it is absorbed. Percentage of absorption is calculated using the following equation:

$$P = (1 - P_r/P_0) \times 100 \quad (1)$$

where P_r and P_0 are the reflected and incident radiation powers respectively.

Composite based on SrF and rubber samples containing various loadings of CB were prepared and the microwave absorption of these composite samples was studied using a network analyser in the S and X bands. The results were then modelled based on reflected power of a plane wave from a metal terminated one layer absorber system.

2. Experimental

2.1. Materials

2.1.1. Acrylonitrile–butadiene rubber (Nitrile rubber) (NBR)

Nitrile rubber used in this study was Aparene – N 553 supplied by Apar Polymers Ltd., Gujarat, India. This NBR contains 34% acrylo nitrile content by weight.

2.1.2. Carbon black (CB)

High abrasion furnace black (HAF N-330) used in this investigation was supplied by M/S Philips Carbon Ltd., Cochin, India.

2.1.3. Strontium ferrite (SrF)

M-type strontium hexaferrite were prepared by a ceramic technique. Ferrous oxalate dehydrate (FOD) precursors were used for the synthesis. α-Fe_2O_3 was prepared by the decomposition of freshly prepared FOD. Appropriate amounts of strontium carbonate and α-Fe_2O_3 were mixed well in a wet medium and the slurry was subjected to pre-sintering and then fired at 1200 °C for 24 h in a muffle furnace.

2.2. Compounding

Pre characterised SrF powder was incorporated in a nitrile butadiene rubber (NBR) matrix along with CB (High Abrasion Furnace-N-330) according to a specific recipe for the preparation of RFCs. The recipe includes sulphur, reinforcing fillers, accelerator, plasticiser and antioxidants. The compounding ingredients used are given in phr (parts per hundred weights of rubber). Composites were prepared for various loadings of SrF and CB. The mixing was first carried out in a Brabender Plasticorder model PL 3S and then homogenized in a two roll mixing mill [18]. They were then moulded into thin sheets and subjected to various studies. The samples were coded for the ease of identification and the optimised samples are listed in Table 1.1.

Table 1.1
Sample codes and their description.

Sample codes	Description
80SrF	80 phr SrF in NBR
30CBSrF	30 phr CB and 80 phr SrF in NBR
40CBSrF	40 phr CB and 80 phr SrF in NBR
50CBSrF	50 phr CB and 80 phr SrF in NBR
Blank	100 phr NBR

Table 1.2
Effect of CB on the mechanical properties of composites.

Filler loading	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Blank NBR	2	2.15	469
80SrF	3.1	2.12	425
10CBSrF	6.5	3	422
20CBSrF	11.8	5.9	420
30CBSrF	15.9	9	416
40CBSrF	18.2	11.6	395
50CBSrF	18.3	16	360

2.3. Measurements in the S and X-band

The resonant cavity used here is a rectangular waveguide with dimensions of 30.8 × 7.2 × 3.4 cm for S-band and 14.1 × 2.3 × 1 cm for X-band. The length, breadth and height of the cavity are chosen so as to excite the cavity to a fixed TE mode. A schematic diagram of the measurement is shown in Fig. 1. For S-band, cavity is connected to a two port vector network analyser (ZVB4, Rohde & Schwaz) and for X-band measurements the cavity is connected to a four port Agilent network analyser through coaxial cables. The five resonant frequencies of the S-band cavity are 2.3 GHz, 2.55 GHz, 2.86 GHz, 3.22 GHz and 3.6 GHz corresponding to TE_{102} to TE_{106} modes. The X-band cavity excited from TE_{103} to TE_{1010} modes correspond to the frequencies 7.2224 GHz, 7.6764 GHz, 8.239 GHz, 8.8621 GHz, 9.559 GHz, 10.283 GHz, 11.067 GHz and 11.86 GHz. The resonance frequency for different modes (TE_{mnp}) are also calculated from the undermentioned equation 2: [25].

$$f_{mnp} = \frac{1}{2\sqrt{\mu_0\varepsilon_0}}\sqrt{\left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2 + \left(\frac{p}{l}\right)^2} \quad (2)$$

where a, b and l are the dimensions of the cavity. Permittivity and permeability were calculated employing cavity perturbation technique equations cited in Refs. [19–23]. The instrument was calibrated using a standard dielectric material with known properties (FR4 glass epoxy).

The reflection loss characteristics were illustrated by exploiting surface impedance modelling in terms of reflected power of a plane wave from a metal terminated one layer absorber system. The power reflection or reflectivity of the material, generally produced for vertical incidence, is commonly expressed as below [24–26]:

$$R = 20\mathrm{Log}_{10}|\Gamma| dB \quad (3)$$

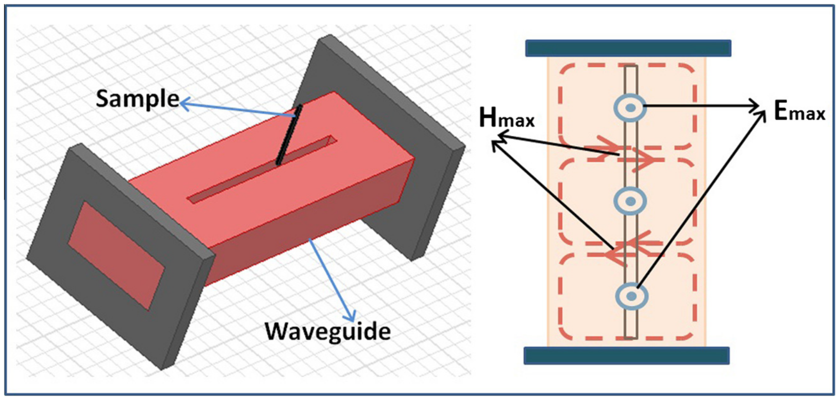

Fig. 1. Schematic diagram of cavity resonator (rectangular wave guide) with field patterns of a typical TE_{103} mode standing wave.

where R is the reflection loss in decibels (dB) and Γ is the reflection coefficient. The reflection coefficient is considered to be a function of surface impedance and impedance can be calculated from complex permittivity and permeability. The impedance matching between the absorber and free space is very important for microwave absorbers. For ideal matching the ratio ε'/μ' should be equal to unity and hence the reflection loss should be theoretically negative infinity. The frequency and the thickness that make $Z_{in} = Z_0$ are defined as the matching frequency f_m and the matching thickness t_m. These were obtained from the method proposed by Kim et al. [6].

At the air material interface total cancellation of reflected wave can occur by interference between the incident and reflected waves. The thickness for which the complete cancellation (incident and reflected waves are in 180° out of phase) of waves takes place is called the optimum thickness and is given by following equation [24].

$$t = \frac{c}{4f}\frac{1}{\sqrt{\mu'\varepsilon'}}\left(1 + \frac{1}{8}\tan^2\delta_u\right)^{-1} \quad (4)$$

Here $\tan\delta_u = \frac{\mu''}{\mu'}$ is the magnetic loss tangent.

3. Results and discussions

3.1. Structure and morphology

X-ray diffraction (XRD) patterns of composite samples were recorded using Rigaku Dmax-C and the structural parameters were evaluated. The structure and planes (h, k, l) of the composites were determined by comparing the measured XRD pattern with standard powder diffraction files (ICDD) [27]. Fig. 2 depicts the XRD patterns of representative samples. XRD reveals that SrF do not undergo any structural change during the different stages of processing of rubber. In Fig. 2, A and Z corresponds to the amorphous peak and peak for Zinc oxide (ZnO) respectively. The presence of ZnO is due to unreacted accelerator used for the curing process. The XRD of pristine SrF powder sample is shown in Fig. 3. An average grain size of 44 nm was obtained from Scherrer's formula.

The fractured surfaces of the samples were examined using a Scanning Electron Microscope (SEM) to study the filler distribution and the micro defect of the samples. Fig. 4 depicts the scanning electron micrographs of pure and CB loaded SrF. The CB incorporated samples display voids or detachment of filler from rubber matrix indicating the reinforcing effect of CB [28]. This is due to the poor interaction between rubber and filler with the increase of CB loading. It is also revealed that the filler remained as agglomerates in the matrix. Along with the agglomerates some rod like particles are also seen. These were further analysed by energy dispersive spectroscopy and were identified as SrF particles.

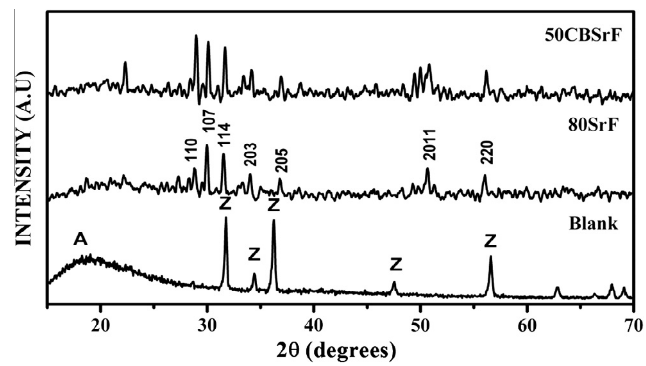

Fig. 2. XRD pattern of various loading of strontium ferrite in NBR.

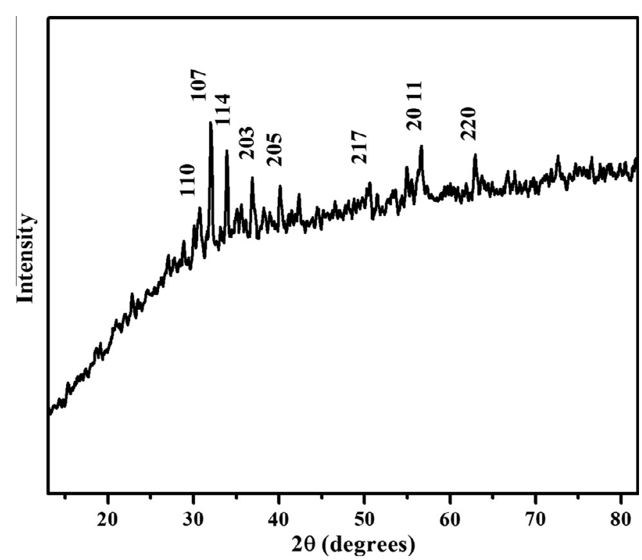

Fig. 3. XRD pattern of pristine strontium ferrite powder.

3.2. Mechanical properties

For many applications even the vulcanised rubber does not exhibit satisfactory tensile strength, modulus, stiffness, etc. These properties can be enhanced by the addition of certain fillers to the rubber during curing. The fillers for rubber can be classified

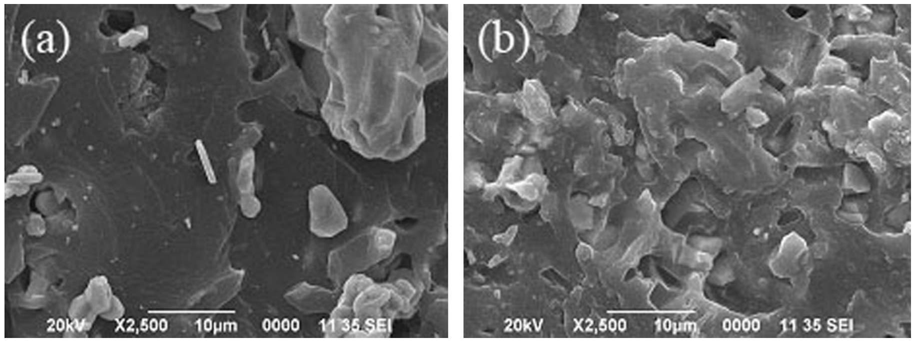

Fig. 4. SEM image of a fracture surface of the representative SrF composites in 10 μm resolution. (a)- 80SrF, (b)- 50CBSrF.

into two, inert fillers and reinforcing fillers. Inert fillers like clay and talc make the rubber mixture easier to handle before vulcanisation but have little effect on its physical properties. Reinforcing fillers, however, improve the physical properties of the vulcanised rubber. Carbon black is a potential reinforcing filler for both natural and synthetic rubbers. In the present study we have exploited the multiple properties of CB like reinforcement of rubber matrix, enhancement of dielectric properties and tuning of bandwidth of microwave absorption.

Mechanical properties of the composite samples were determined as per ASTM 0 412 (1980) with an Instron Universal Testing Machine (UTM), Model 4411 Test System. All tests are carried out at 28 ± 2 °C. The sample is held between the two grips on the UTM, the upper grip of which is fixed. The rate of separation of the power actuated lower grip is fixed at 500 mm/min. The tensile strength, elongation at break and modulus at different elongations are recorded and evaluated. The calculated values are shown in Table 1.2.

Even though the tensile strength of the carbon black is low when compared to other carbonaceous materials, the elastic properties are much superior to that of these materials. The elongation at break is more than 350% for all the samples and this value is close to the blank rubber value. So the composites of present investigation containing CB possess strength of carbon black and elasticity of nitrile rubber.

3.3. Complex dielectric permittivity and magnetic permeability

The incorporation of SrF into the NBR matrix is found to have only a little effect on the absorption properties but the inclusion of CB shows enhanced microwave absorption. The SrF powder is added into the CB–NBR matrix for better impedance match which arises from both dielectric and magnetic contributions. Figs. 5 and 6 illustrate the complex dielectric permittivity and magnetic permeability for the five samples in the S and X-band. It can be observed that all the samples display an almost constant value of ε' throughout the given frequency range (S and X-band). The polarization mechanisms responsible for this frequency range are orientational and space charge polarization. Orientational polarization of ferrites in this frequency range is a consequence of the process of electron hoping between ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions. The SrF shows a ε' value of 4.5 whereas the CB added composites exhibit values in the range of 8–15 for different loading of CB. The sharp increase in real part of dielectric permittivity (ε') values

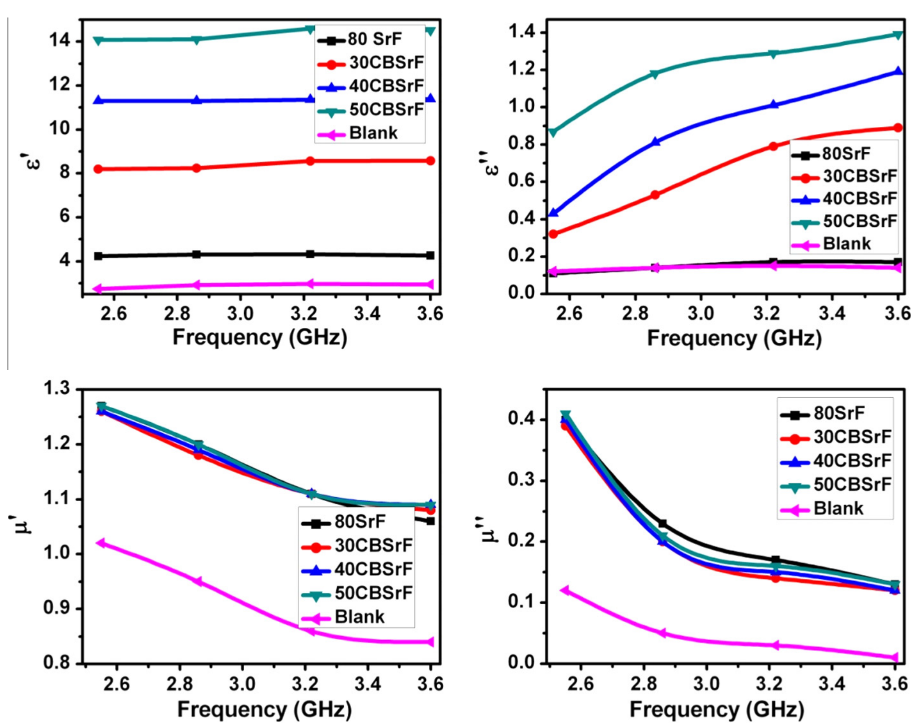

Fig. 5. Variation of complex dielectric permittivity and magnetic permeability of SrF composites with frequency in the S-band.

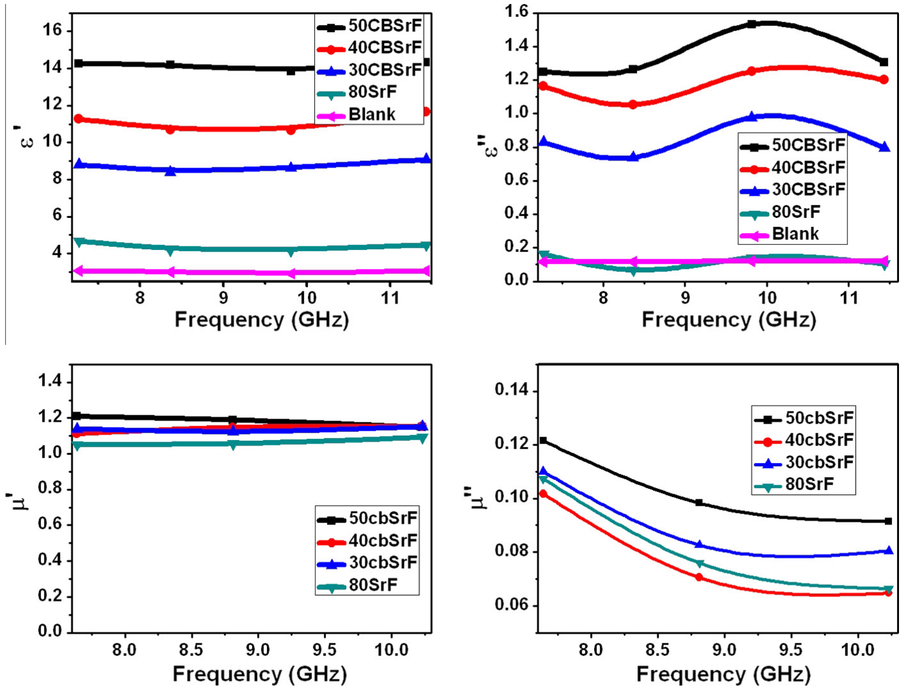

Fig. 6. Variation of complex dielectric permittivity and magnetic permeability of SrF composites with frequency in the X-band.

is because of the accumulation of space charges due to the heterogeneity of the material and the overall effect of individual components as per the effective medium theory. CB is dispersed in the rubber matrix in such a way that it forms isolated aggregate inside the rubber matrix as one can see from the SEM images. Charges accumulate in the interfacial boundaries between the CB aggregates and rubber and a dipole moment is imparted to this cluster. The network of these aggregates inside the matrix contributes to the monotonous increase of ε'. The imaginary part ε'' however shows an increasing trend. These values increase towards the high frequency side and all the samples have shown similar behaviour with the variation of frequency. This is due to the various loss processes as frequency increases. The dielectric loss in the microwave frequency range is a combination of relaxation polarization loss and electric conductance loss. The complex permeability and permittivity of the composites increase as the SrF and CB volume fraction increases. The permeability due to the loading of 80 phr SrF in all the samples was found to be very less. The real part of magnetic permeability μ' values decrease with frequency. The imaginary part μ'' values are very small and do not come close to zero under

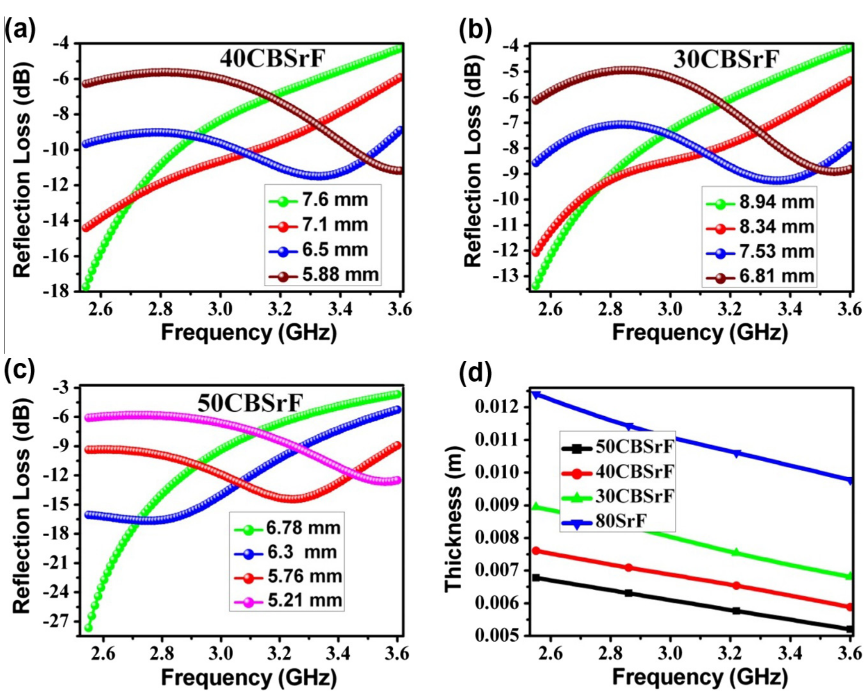

Fig. 7. Variation of reflection loss and the optimum thickness of SrF composites with frequency in the S-band. (a)- 40CBSrF, (b)- 30CBSrF, (c)- 50CBSrF and (d)- thickness versus frequency plot.

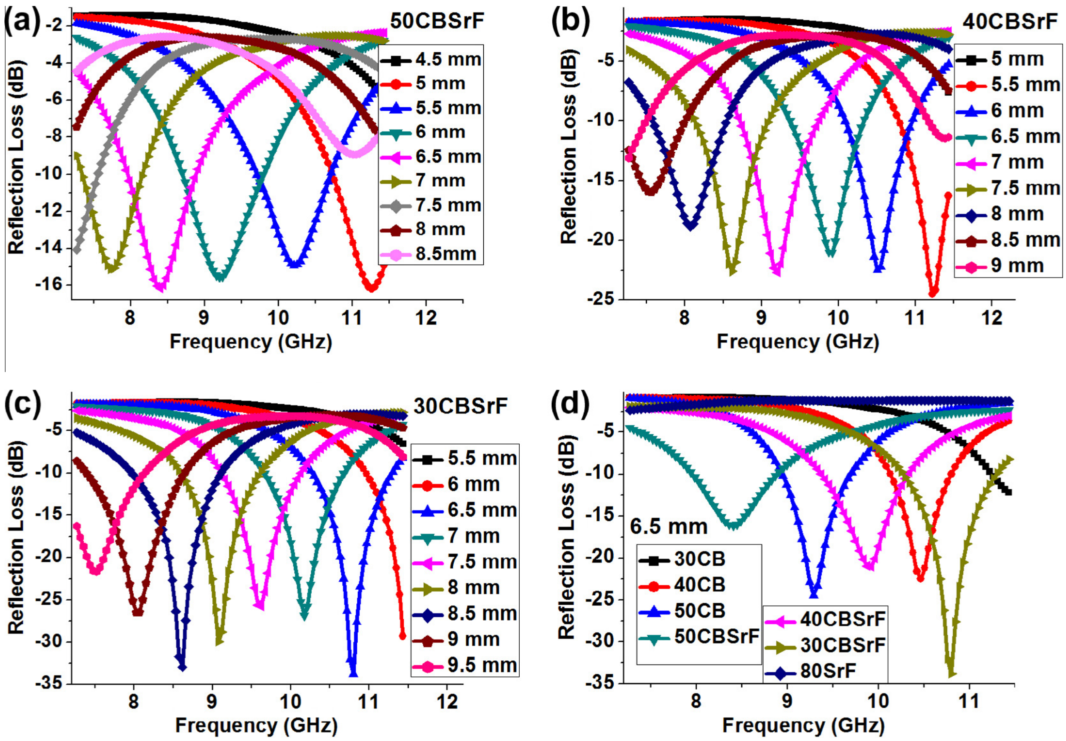

Fig. 8. Variation of reflection loss of SrF composites with frequency in the X-band for different thicknesses.

the given frequency of operation. There are no resonance peaks observed for dielectric loss and magnetic loss. The relative volume fraction of SrF could decrease with the increase in loading of CB, which resulted in a reduced permeability of CB added samples, illustrated in Fig. 5.

3.4. Reflection loss characteristics: Modelling based on single layer absorber backed by reflector

The variation of reflection loss with frequency of SrF based composites in the S-band is shown in Fig. 7 and that in X-band are depicted in Fig. 8. It can be observed from the graphs that the frequency corresponding to the maximum reflection loss shifts towards the low frequency regime with increase in concentration of CB while with decrease in the thickness of absorber; it shifts towards the higher frequency side. This behaviour is consistent with previous results [5,24,29]. The minimum thickness required for the cancellation of the reflected wave is calculated and the results are shown in Fig. 7d. There is a steady decrease in thickness corresponding to the destructive interference of incident and reflected waves. The matching thickness and matching frequency of SrF composite is 7 mm and 2.55 GHz in the S-band. An optimum microwave absorption of −28 dB (99.84%) at 2.55 GHz was recorded in the S-band. The reflection loss characteristics obtained in the X-band is rather interesting in that one can find more than 95% absorption for different thicknesses and at different frequencies. The dependence of bandwidth with optimum thickness of sample 50CBSrF over an absorptivity of 90% (−10 dB reflection loss) is plotted in Fig. 9. A bandwidth of 1.5 GHz was registered for 50CBSrF with an optimum thickness of 5.5–6.5 mm for X-band and 700 MHz with an optimum thickness of 5.7–6.5 mm for S-band. The CB added composites renders enhanced and broad band microwave absorbing properties than pure SrF rubber composite. Maximum reflection loss lies in between −14 and −35 dB. That means the absorption is in between 96% and 99.968%.

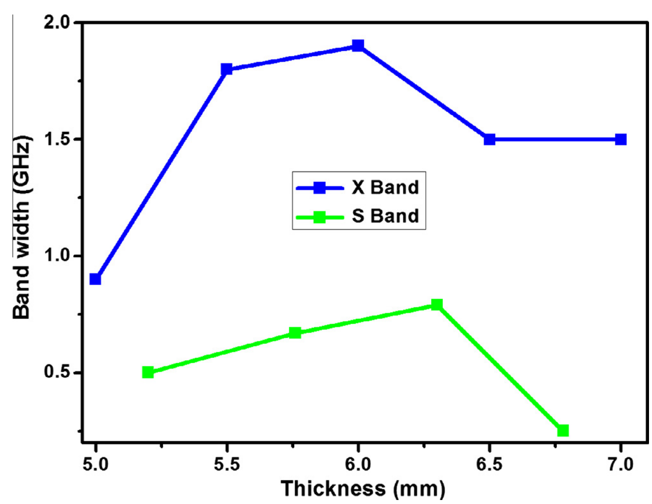

Fig. 9. Variation of bandwidth with absorber optimum thickness of 50CBSrF sample.

4. Conclusions

Hybrid type, S and X-band flexible and broadband microwave absorbing materials were synthesized and the influence of these magnetic–dielectric materials on the microwave absorbing properties have been investigated in detail. CB added composites exhibit enhanced microwave absorption and wider band widths than those for pure SrF rubber composites at its minimum thicknesses. Moreover, by the combination of two fillers (both CB and SrF) in the rubber matrix sample coded 50CBSrF is identified as the best with microwave absorptions greater than 99% (reflection loss ⩾ −28 dB) at 2.55 GHz with 7 mm thickness in the S-band. For the X-band the best performance of 50CBSrF is 97% absorption (reflection loss = −16 dB) at 11.2 GHz obtained at a thickness of 5 mm. The performance with respect to dielectric and magnetic properties in the measured frequency ranges points to the electromagnetic compatibility of the composites, implying improved microwave absorption properties resulting from the cooperative outcomes of a lossy dielectric matrix, a magnetic component and a reinforcing as well as absorbing filler like CB.

Acknowledgments

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