A short review on toughened epoxy based nanocomposites for EMI shields

Ravi Kant¹ and M. Fahim2*

¹ Department of Chemistry, Zakir Husain Delhi College, University of Delhi, New Delhi 110002 INDIA ² Department of Physics, Zakir Husain Delhi College, University of Delhi, New Delhi 110002 INDIA

> *Volume 1, Issue 3, May 2024 Received:18 January, 2024; Accepted: 9 April, 2024 DOI: https://doi.org/10.63015/5N-2416.1.3*

**Corresponding Author Email: fahim99@hotmail.com*

Abstract: Modern era devices and appliances require shielding from electromagnetic interference to ensure stable and efficient functioning. This is all the more pertinent in view of the safety and protection of electronic gadgets used in defence security equipment. It has been observed that the most prominent radiations of EMI fall in the range of microwave (GHz) and radio waves (MHz). Those materials which protect equipment from electromagnetic interference are called EMI shields, since they shield the equipment by either reflecting, absorbing or transmitting the electromagnetic radiation. However, not all materials can be used as EMI shields. The basic material requirement to be used as EMI shields include good magnetic permeability and dielectric properties as well as strong mechanical properties. Currently EMI shields made up of metals or silicones are used in the form of solid enclosures, cables, gaskets, O-rings, films, coatings, fabrics, tapes etc. With time and research new advanced lightweight materials have been developed which have shown potential for use as EMI shields. These include carbonaceous and ferrite nanofillers based polymer nanocomposites. These materials are unique because they provide shielding for a wide range of microwave frequency 2-18 GHz by a judicious choice of conductive, dielectric and magnetic nanofillers. The present review article provides an overview of the research work carried out on toughened epoxy (epoxy modified with other polymers) filled with multifunctional nanoparticles and their characterization for EMI shielding efficiency. Various factors and features have been critically analysed in this article. The entire focus of the article is aimed at Toughened epoxy as a base matrix which can support EMI shielding in the complete frequency range of 2-18 GHz and overcome inherent disadvantages of non-toughened epoxy.

Keywords: Electromagnetic interference, Toughened epoxy, EMI shielding, composites

1. Introduction and Literature Review: Electromagnetic interference (EMI) shielding assumes paramount significance since EMI is one of the most undesirable by-products of telecommunication devices and high frequency electronics. Any device or technology which deduces, process, reflect and transmit or utilizes electrical energy of any form may emit radiations[1]. The performance and shell life of electronic gadgets is adversely affected due to EMI and is a major concern for defence security equipment [2].It has been observed that the most prominent radiations of EMI waste stand in the range of microwave and radio waves. Hence, most of the research

work is focussed on developing EMI shields for this frequency range. Electromagnetic interference (EMI) shields are conventionally based on metals, silicones, ceramics and cements. They are used in the form of metallic enclosures, wire mesh and screens, gaskets, Orings, cable shields and coatings. With time and research, however, the focus has shifted to composite materials which provide a spectrum of EMI shielding properties. These are achieved by adding desired fillers in the base matrix and making a judicious choice of processing

Scheme 1: Mode of electromagnetic Interference (EMI)

techniques. For instance, a composite material formed with polymer as base matrix(continuous phase) and ceramic/ferrites/conducting fillers as discontinuous phase, can be used to absorb a particular range of microwave frequency band. These frequency bands, identified as S (2 GHz to 4 GHz), C (4 GHz to 8 GHz), X (8 GHz to 12 GHz), Ku (12 GHz to 18 GHz), K (18 GHz to 26.5 GHz), and Ka (26.5 GHz to 40 GHz), still pose a challenge. However, same material cannot absorb all the frequency bands. In order to fabricate a material which can absorb all range of microwave frequency, multi layered/ mixed filler composites show a lot of promise. Hence, the main objective of this review is to critically analyse the current research and recent advancements in polymer composites materials which can absorb microwave frequency from 2 GHz to 18 GHz for applications in fields like telecommunication, microwave heating and health care sectors. The aim is to achieve a shielding effectiveness (SE_T) of greater than 60 dB. The subsequent section gives a review of the research work done in this field in the last five years.Electromagnetic interference (EMI) or radio frequency interference (RFI) shielding is a means by which leakage of strong electromagnetic fields is prevented using a shield or barrier, which if not prevented can interfere with electronic devices and signals. Since electronic devices and circuits deal with small voltage and current, strong electromagnetic fields can either distort their performance or eventually damage them. EMI is basically the coupling of signals from one source to another (receiver) through a path as shown schematically in **scheme 1**. The source is a system that creates disturbance, receiver is the sensitive signal or device whose output signal is distorted by interference and path is the system where signal coupling occurs. EMI is of two types as shown in **scheme 2**. EMIs can also be categorised based on the bandwidth of incident disturbance. A narrowband disturbance has a bandwidth \leq receiver while for a broadband disturbance, bandwidth \geq receiver.

Basic Mechanisms of EMI shielding: An electromagnetic wave consists of oscillating electric (**E**) and magnetic fields (**B or H**) which are perpendicular to each other and to

Scheme 2: Types of Electromagnetic interference (EMI)

the direction of propagation of wave. Both the fields travel with the same frequency. When electromagnetic wave travels through a conductive medium, the electric component is blocked. Similarly, if the medium has high magnetic permeability, the magnetic component is blocked. Well known Maxwell's equations of electromagnetic theory are used to find the response of any material to the incident electromagnetic wave [3]. These equations have been derived for free space as well as the medium through which it is passing. Based on the dielectric, magnetic and conductive properties of the medium, these equations are changed. For dielectric or low conductivity materials, their dielectric permittivity (ε) and magnetic permeability (μ) are expressed as complex numbers viz. $(\epsilon = \epsilon')$ is") and $(\mu = \mu'$ -ju"). These parameters govern the interaction of any material with the electric and magnetic field vectors of electromagnetic wave. The real part denotes stored energy while the imaginary part denotes loss or dissipation of energy as heat when em eave is absorbed within the material. The ratio of magnetic permeability and dielectric permittivity, defined as impedance matching $(\sqrt{\mu/\epsilon})$, and reciprocal of their product $[1/\sqrt{(\mu \varepsilon)}]$ which gives the velocity of em wave. eventually determine how the em wave will propagate in that medium. In free space, this impedance is equal to 377 ohms and velocity $=$ 3 x 10^8 cm/s using the universal constants μ_0 and ε_0 for free space. An impedance mismatching occurs at the interface of media with different impedances and the wave is reflected. If the impedance mismatch is higher more reflection takes place at the interface[4].

From these equations it is imperative that when any base polymer matrix is dispersed with dielectric, conductive or magnetic fillers of nanosize, its interactions significantly change. EMI shielding mechanism is largely based on this concept wherein either of the electric or magnetic component is filtered outby shields using reflection, absorption (due to multiple internal reflection) or transmission mechanisms. In the reflection mode, the electric field vector is attenuated since the mobile charge carriers of shield interact with incoming EM wave and are redistributed along the conductor which eventually creates an opposing electromagnetic field. The two opposing EM fields cancel out each other. The higher the conductivity, the better is the shielding efficiency. However, for high frequency electromagnetic wave (low wavelength) there is a limitation due to the size of the holes of shielding enclosure and skin effect which lowers the conductivity in the inner section as charges accumulate on the top surface of the conductor. In such a case either the surface area of conductor can be increased, or the surface is coated with highly conductive material. Contrary to reflection, absorption of EMI acts on the magnetic field vector of electromagnetic wave. Due to the external magnetic field of incident electromagnetic wave, the magnetic lines of force travelling through the material are intercepted and eventually absorbed. Thus, a shield material absorbs the magnetic and electric lines of force by creating a path within itself. However, in such a material conductivity is very low which do not provide protection from electric component of incident electromagnetic wave. Secondly, the oscillating em wave at higher frequencies generate eddy currents which possess their own magnetic field opposite to the external magnetic field. Thus, if a material is chosen whose electrical conductivity is high it creates stronger eddy currents. Absorption due to multiple reflections occurs by either scattering of electromagnetic waves or having multiple reflection boundaries. Thus, the desired properties of EMI shielding material are (i) electrical conductivity and (ii) magnetic permeability. Metals are the preferred choice

because they have higher electric conductivity, magnetic permeability, strength and ductility. However, they are expensive. Carbon steel alloys, mild carbon steel and ferrite stainless steel and Fe-Ni alloys such as Mu-metal are used for EMI shielding because they have high relative permeability of 10^4 at 1 kHz. Carbon allotropes such as exfoliated graphite, graphene, carbon fibres and carbon nanotubes are used as fillers in composites that can be used for EMI shield. All the fillers can be dispersed in a polymer matrix, metal, ceramic or cement. They have high conductivity; high aspect ratio and high porosity and they can operate through multiple reflection mechanism. For high frequency shielding applications, graphene and CNTs are mostly used because the dimension of the materials is less than the skin depth., which makes them better conductors than metals in GHz region. Conducting polymers (PANI, Polypyrole etc) conduct electricity between atoms due to the conjugated bonds. This enables the delocalization of π -electrons which act as mobile charges. However, their use is still being explored. Silicone embedded with Nickel or graphite is effective at shielding radio frequency between 20 Hz and 10^4 Hz without any metal component. Foil and fabric (nylon, polyester interwoven with metals also provide protection as EMI shields.

Common materials used for EMI shielding:

Metal based advanced materials have always been the choice for EMI absorption. However, polymer based materials have shown lot of promise to replace metals for EMI shielding applications. Largely two types of polymer based EMI shielding materials have been identified 1) Intrinsic and 2) compound type. Intrinsic polymers have poor mechanical and processing aspect which limit their utilization in EMI absorption. While compound type material provides ease of modification, designing and fabrication as per desired range of EMI absorption. Polymer-based composites are being designed so as to achieve high dielectric constants, good electric conductivity and magnetic permeability which are required for low EMI permeation. Many polymers are used today for their property of absorbing radiations including polyurethane, epoxy, polyaniline etc. Both biodegradable and nonbiodegradable polymers have been explored for such application.

Material requirement for EMI shielding: Different criteria have been used to synthesize materials for EMI shielding applications. These include versatile surface chemistry, high aspect ratios, addition of electromagnetically charged particles, excellent mechanical and electrical properties, conductivity, reflection losses and multiple internal reflections which play a key role in EMI shielding. In general, two regions of EMI shielding are identified. If the distance between the radiations source and the shield is $>\lambda/2\pi$, it is known as far field shielding region while it is known as near field shielding region if the distance is $\langle \sqrt{2\pi} \vert 1 \vert$. EMI plane wave theory is applied in the far field shielding region while theory based on contribution of electric and magnetic dipoles is applied for near emi shielding[2].The two major pre-requisites for EMI absorption are dielectric and magnetic losses. Secondly, impedance matching (i.e. matching of complex permittivity and permeability), interfacial polarization, attenuation capability and multiple reflection are equally desirable. The ratio of E to H is called wave impedance and for free space intrinsic impedance is equal to 377 ohms. In order to design materials possessing these properties, multifunctional polymer nanocomposites (PNCs) filled with ferrite and carbonaceous nanofillers have been developed and characterized for their ferromagnetic resonance and hysteresis loss, reflection loss (RL), shielding effectiveness (SE) and absorption bandwidth. It has been observed that single layer absorbers have their own limitation such as narrow absorption bandwidth. Hence double-layer absorbers have been suggested for achieving a wider bandwidth.

In effective absorbing material, firstly the intrinsic impedance value of the material is made to approach impedance of the free space in a matching layer and, $E_A = KVsin^2\theta$

secondly, the incident EM wave is attenuated rapidly in the second absorbing layer [5].These layers are made up of material having better dielectric and magnetic permeability and their functions can be interchanged[6].In absorbing layer maximum absorption can occur when normalised impedance (Zo) and input impedance is equal to one. In such an ideal situation, EM wave can penetrate and get absorbed completely within the material [7,8]. In real situation, attempts are being made to achieve over 90% absorption by making input impedance almost equal to normalised impedance. A term Reflection loss (RL) can indicate absorbing capacity of the shield material and is evaluated as [9],

$$
RL(dB) = 20\log\left|\frac{z_{in-z_0}}{z_{in}-z_0}\right| \text{ (for single layer}
$$

microwave absorber) (1)

where z_{in} and z_0 are the input impedance and vacuum impedance respectively,

$$
\mathbf{z}_{in} = \mathbf{z}_0 \sqrt{\frac{u_r}{\varepsilon_r}} \tan h \left[j \frac{2\pi^f t}{c} \sqrt{u_r} \varepsilon_r \right] \dots (2)
$$

 $(t = thickness of specimen, f = frequency of the$ incident EM wave, $c = EM$ wave velocity in vacuum). For double layer

:

$$
Z_{in} = \frac{\sqrt{\frac{\mu_2}{\varepsilon_2}(\sqrt{\frac{\mu_1}{\varepsilon_1}}tanh[j(\frac{2\pi fd_1}{c})\sqrt{\mu_1\varepsilon_1}]+\sqrt{\frac{\mu_2}{\varepsilon_2}tanh[j(\frac{2\pi fd_2}{c})\sqrt{\mu_2\varepsilon_2}]})}}{\sqrt{\frac{\mu_2}{\varepsilon_2}+\sqrt{\frac{\mu_1}{\varepsilon_1}}tanh[j(\frac{2\pi fd_1}{c})\sqrt{\mu_1\varepsilon_1}]}tanh[j(\frac{2\pi fd_2}{c})\sqrt{\mu_2\varepsilon_2}]}}
$$
...(3)

Absorption of EM wave can be quantified in terms of percentage as:

Absorption (%) =
$$
100 - [10^{(RL/10)} \times 100]
$$

Ferromagnetic properties namely coercive field (H_c) , saturation (M_s) and remanent magnetization obtained using B-H hysteresis loop can be used to find anisotropy constant (K) and energy (E_A) as

(for a single domain nanocrystalline material $)$ (4)

where K, V and θ are the anisotropy constant, volume of nanocrystal and orientation of applied field induced magnetization with respect to easy axis, respectively

$$
K = \frac{\mu_0 M_s H_c}{2} \qquad \qquad \dots(5)
$$

where $\mu_0 = 4\pi \times 10^7$ H/m is the permeability in vacuum. The dissipation or tangent loss is obtained using following equations,

$$
\mu^* = \mu' - j\mu'' \text{ and } \varepsilon^* = \varepsilon' - j\varepsilon'' \qquad \dots (6a,b)
$$

tan δ_e = ε''/ ε' (dielectric loss tangent)...(7)

 $\tan \delta_m = \mu''/\mu'$ (magnetic loss tangent) ..(8)

tan δ = tan δ_e + tan δ_m (total loss tangent) ..(9)

The complex permeability (µ***) and permittivity (ε***) values of the samples can be calculated from the reflected (S_{11}) and transmitted (S_{21}) signals measured using a waveguide technique^[10].Using these shielding parameters, shielding effectiveness can be measured as,

$$
SE_{Total} = SE_A + SE_R + SE_M \qquad \dots (10)
$$

which is the sum of absorption (SE_A) , Reflection(SE_R), and multiple reflections(SE_M) and is obtained using following equations discussed below.

$$
SE_{\text{Total}} = 10\log(\text{Pi/Pt}) = 20\log(\text{Ei/Et}) = 20\log(\text{Hi/Ht})
$$

....(11)

$$
SE_R = -10\log_{10}(1 - S^2_{11}) = 10\log_{10}(1 - R)...(12)
$$

where $R = S^2_{11}$

 $SE_A = -10log_{10}(S^2_{21}/1-S^2_{11}) = 10log_{10}(T/1-R)$ where $T = S^2$ \dots (13)

where subscript i and t represent properties of incident and transmitted waves, respectively. S_{11} and S_{21} denote the scattering parameters of the forward reflection (R) and backward transmission coefficients (T) respectively. The multiple reflection scattering parameter SEM is calculated as

$$
SE_{M} = 20log_{10}(1 - e^{-2t/\delta}) = 20log_{10}(1 - 10^{SEA/10})
$$
...(14)

where t is the thickness of shielding material, δ is the skin depth. However, SE_M can be neglected for $SE_A > 10dB$ as amplitude of EM wave dies down with thickness. The attenuation constant $(α)$ is related to the skin depth (δ) as $\alpha = \frac{1}{s}$ $\frac{1}{\delta}$ and $\delta = 1/\sqrt{(\pi f \mu \sigma)}$ where μ is the permittivity and σ is the conductivity of the shield material. It is obtained using following relation,

$$
\alpha = \frac{\sqrt{2\pi}f}{c} \{ \mu^{\prime\prime} \equiv \varepsilon^{\prime\prime} \equiv - \mu' \varepsilon' + \left[\left(\mu''^{2} + \mu'^{2} \right) \left(\varepsilon''^{2} + \mu'^{2} \right) \right] \right] 1/2 \}^{1/2}
$$

....(15)

Common methods by which shielding effectiveness is calculated/measured include (i) measurement through a waveguide and (2) through antenna and receiver. A free and enclosed environment is required to attenuate the external noise interference.

Toughened epoxy based nanocomposites:

Epoxy resin is a versatile thermoset polymer having numerous industrial applications. However, its intrinsic brittleness limits its engineering applications. To mitigate this disadvantage and increase its range of application, it is toughened using elastomers such as rubber with no compromise of its original properties. The, so called, rubber toughened epoxy resins when filled with nanofillers show further improvement in elastic properties as well as glass transition temperature T_g of base matrix[11,12]. The toughening may be attributed to shearing, debonding and cavitation of epoxy-rubber molecular chains[13].For developing rubber toughened epoxy-based nanocomposites, generally butadiene-acrylonitrile based rubbers such as CTBN, ETBN, ATBN and VTBN have been used. The mechanism for the formation of carboxyl terminated butadiene acrylonitrile copolymer and triphenylphosphine toughened epoxy resin can be find elsewhere [14, 15].In a research paper

published almost a decade back, it was reported that for C8 ether linked bismaleimide toughened epoxy filled with carbon black, a higher electrical conductivity and impact strength can be achieved with just 5 wt% of CB[16].This was attributed to the covalent bonds between CB and C8 e-BMI/epoxy matrix. Ten years later, the same group reported on tuning the characteristics related to microwave absorption capability of rGO filled toughened epoxy composites via SiC- induced phase separation [17]. Using vector network analyzer (VNA) measurements it was observed that simple RGO-based composites show poor reflection loss (RL) characteristics. However, with SiC loading the same material gains excellent absorption with RL of -46.5dB (effective absorption bandwidth of over 2GHz) for a thickness upto 10 nm. Earlier the same authors had reported RL −51.6 dB (99.999% microwave absorption) at 9.51 GHz and an effective absorption bandwidth of 2.48 GHz for m-caproamine/imidazole toughened epoxy composites with3 wt% r-GO composites compared to unfilled m-CA/ER composite [18].Toughening of epoxy using rubber and filled with multifunctional nanoparticles has also been reported extensively in the literature. For instance, the treatment of epoxy with 10% recycled rubber increased its toughness that was confirmed through scanning electron microscopy and nanoindentation, static (3PB). Addition of Nano-magnetic iron oxide, $Fe₃O₄$, Nickel and aluminium particles enhanced the magnetic permeability and dielectric properties of the resultant composites [19]. However, the material has not been characterized for their microwave absorption properties. An exhaustive literature on this topic can be found in a review published in 2020 [20]. Recently, EMI shielding properties of thermosetting epoxy foams modified with rubber (CTBN) and functionalised MWCNTs using supercritical carbon dioxide were reported [21]. It was observed that the alignment of oriented fMWCNTs (5 wt%) in foams may have caused an increase in electrical conductivity (0.43 S/m), EMI SE (22.90 dB) and specific EMI SE (37.54 $dB/(g \cdot cm^3)$ with better EMI shielding performance rendered possible due to multiple reflections of microwaves and their eventual attenuation by conductive filler [20].Wang et.al**.**[22] has toughened one epoxy system using CTBN and filled it with MWCNT-COOH claiming that accelerated curing occurred because of bonding developed between COOH-functionalised MWCNT and epoxy resin. The same composite was made electrically conductive with the addition of CNT [23]having enhanced fracture toughness and thermal stability [24]. Jyotish kumar et al.[25] toughened DGEBA epoxy with ABSand MWCNT and reported enhanced stability of the material. It has been reported that nanocomposites based on toughened epoxy using carboxy-terminated butadiene acrylonitrile copolymer and filled with 5 wt% functionalised MWCNT can produce an EMI shielding effectiveness (22.90–37.94 dB) since EM waves are absorbed due to multiple reflection [26]. With 1 wt% graphene nanofiller, rubber toughened epoxy also produces an EMI shielding effectiveness in the same range (22–48 dB)[27].

Numerous studies are available in the literature wherein new polymer materials have been explored for microwave absorption of different ranges of frequency. However, other polymer systems are beyond the scope of this review. Recent studies for these polymer systems can be found in ref [28-30].In one instance, Saini et al. [31] produced a nanocomposite based on polyaniline and MWCNT for microwave absorption in the range of 12.4 – 18 GHz. Dielectric characteristics and microwave absorption of graphene composite materials have been studied by Kevin et al [32] and reported the microwave absorption in different ranges of frequency. Sun et al. [33] developed aFe3O4/CNFs material synthesized by chemical co-precipitation and investigated its microwave absorption properties. Hong-Wen [34] studied the microwave absorption of materials over 9 GHz and reported the reflection loss more than -25 dB for specific frequencies. Microwave absorption properties of highly filled polymer composites with amorphous Fe-B Particles was studied by Kiyotaka et al. [35]. Yichao et al. [36] prepared

anisotropic Fe3O⁴ nanoparticle and a series of Fe3O4/RGO nanocomposites and found that the materials exhibit high-performance microwave absorption properties over 2.0– 18.0 GHz. The optimal reflection loss of the pure Fe₃O₄ composite was reported as -38.1 dB at 14.8 GHz while it reaches to -65.1 dB at 15.2 GHz with 3 wt.% of RGO of same thickness. Microwave absorption of ferriterubber composite at X-band frequencies has been reported by Kim et al [37]. Microwave absorption properties of honeycomb core structures coated with composite absorber was studied by Wang et al.[38] have reported the reflection loss in the range of 12 GHz to 18 GHz.. Young et al [39] have reported the microwave absorber properties of magnetic and dielectric composite materials in the range of 5.8 GHz. These studies show that work is still needed to develop such novel materials which are light weight, flexible and economical and that can be used to absorb microwave of frequency of all ranges from 2 GHz to 18 GHz. A comparison of the EMI shielding performance of Toughened epoxy based composites and Epoxy based composites is given in Table 1.

Concluding remarks: Nanocomposites based on rubber toughened epoxy and filled with carbonaceous nanofillers show enhanced mechanical, thermal, electrical and morphological characteristics due to synergistic effect of matrix and fillers. While nanofillers improve conducting and magnetic

properties, toughened epoxy matrix provides high strength, ductility, and fracture toughness. This synergistic effect results in better EMI shielding effectiveness. However, the current research advocates the use of rubber toughened epoxy foams filled with nanofillers for still better EMI shielding effectiveness. For

instance, EMI SE of 22.90 dB and specific EMI SE of 37.54 $dB/(g \cdot cm^3)$ has been obtained in the epoxy composite foam with 5.0 wt% conductive filler loading which not only increased electrical conductivity but also rendered attenuation of em waves due to multiple reflections. These preliminary results appear to be promising and sets a direction for future research on Toughened epoxy based composites for EMI shielding [61].

Acknowledgement: Authors wish to acknowledge the financial grant sanctioned by DRDO India for carrying out this work.

Conflict of Interest: Authors declare No conflicts of interest

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