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Electromagnetic interference shielding effectiveness of short carbon fibre-filled polychloroprene vulcanized by barium ferrite

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Short carbon fibre-filled polychloroprene rubber composites vulcanized by barium ferrite were used for the measurement of electromagnetic interference (EMI) shielding effectiveness, at varying carbon fibre concentrations, aspect ratios and sample thicknesses, in the frequency range of 100 to 2000 MHz. The return loss values of the composites have also been studied at different carbon fibre concentrations and aspect ratios in the frequency range of 1100 to 2000 MHz. It was observed that at a particular frequency with increasing carbon fibre concentration the shielding effectiveness of the composite increases and return loss decreases. It was also observed that composites prepared by a cement mixed method with a high fibre aspect ratio ($L/D \simeq 100$) show a higher shielding effectiveness and lower return loss than composites prepared by a mill mixed method with a low fibre aspect ratio ($L/D \simeq 25$). This indicates that loss due to absorption increases with increase in carbon fibre concentration and aspect ratio. Correlation between shielding effectiveness and volume resistivity of the composites indicates that the shielding effectiveness depends not only upon the volume resistivity of the composite, but also on the aspect ratio and sample thickness.

1. Introduction

In recent years electrically conductive polymer composites have received attention from the electronics and electrical industries, due to their potential applications in the areas of electromagnetic interference (EMI) shielding and electrostatic discharge (ESD) dissipation, and as semiconducting material in hybrid electronic circuits. Polymers as electrical insulators are transparent to electromagnetic radiation and thus do not provide EMI shielding. Electrical conductivity is a prerequisite for EMI shielding effectiveness. Both conductivity and EMI shielding effectiveness can be improved by the incorporation of conductive fillers in a polymer matrix [1–3]. Recently, EMI shielding effectiveness of polypropylene composites filled with aluminium-coated glass fibre and nickel-coated carbon fibre in the X-band frequency range of 8–12 GHz has been reported [4–5]. Electromagnetic-compatible (EMC) plastics and rubber gaskets of low volume resistivity (0.001 to 0.01 Ω m), containing silver balls or silvered brass balls, have been reported to be used as EMI shielding materials and hermetic seals [6, 7].

In our earlier communication [8], we have reported the EMI shielding effectiveness of carbon fibre-filled polychloroprene in the X-band frequency range [8–12 GHz]. It was observed that the shielding effectiveness depends not only upon electrical conductivity of the composite, but also upon the fibre distribution, fibre aspect ratio and sample thickness. In the present investigation, we report the results of our studies on shielding effectiveness (SE) and reflection loss (RL) of carbon fibre-filled polychloroprene composites vulcanized by barium ferrite. Barium ferrite has been found to act as a vulcanizing agent for polychloroprene [9]. The effects of carbon fibre concentration, fibre aspect ratio (L/D) and sample thickness on SE have been studied in the frequency range of 100 to 2000 MHz. The RL values of the composites at varying carbon fibre concentrations and aspect ratios have also been studied in the frequency range of 1100 to 2000 MHz.

2. Experimental procedure

2.1. Materials

Polychloroprene rubber (CR) of grade Neoprene WRT, manufactured by Dupont Ltd, USA was used. Short carbon fibre (CF) used was of grade RK 30/12K, obtained from R.K. Carbon Fibre Ltd, UK. Barium ferrite used was of commercial grade. The characteristics of barium ferrite and CF are given in Tables I and II, respectively.

2.2. Preparation of composites

The rubber–fibre mixes were prepared by two mixing methods, namely the "mill mixed method" and "cement mixed method". Details have been discussed elsewhere [10].

2.2.1. *Mill mixed method*

Compounded CR containing barium ferrite was prepared by a conventional two-roll rubber mill (0.36 m × 0.15 m). Short CF of aspect ratio 800 was added to the compounded CR on the two-roll mill and blended for about 7–10 min in order to achieve good dispersion.

2.2.2. *Cement mixed method*

Compounded CR containing barium ferrite was put in chloroform (solvent) in order to form a paste. Short CF ($L/D = 800$) was dispersed into the paste by mechanical stirring. The fibre-filled paste was dried at room temperature to constant weight. The dried rubber–fibre mix was passed through the two-roll mill about 10 to 12 times for uniform dispersion.

Formulations, fibre aspect ratios and volume resistivity of the composites for both mill-mixed and cement-mixed methods are given in Tables III and IV, respectively. The rubber–fibre mixes were moulded at 150 °C to their respective optimum cure times determined by a Monsanto rheometer R-100, as discussed elsewhere [10]. Samples of two different thicknesses, namely 1.7 and 3.5 mm, were moulded.

TABLE I Characteristics of barium ferrite

Chemical composition	$BaO \cdot 6Fe_2O_3$
Density	5.2×10^3 kg m^{-3}
Surface area	0.42×10^3 m^2 kg^{-1}
Electrical resistivity at 25 °C	10^6 Ωm

TABLE II Characteristics of RK 30 (12K) carbon fibre

Filament length, L	6×10^{-3} m
Filament diameter, D	7.5×10^{-6} m
Filament shape	Round
Density	1.78×10^3 kg m^{-3}
Electrical resistivity	1.5×10^{-5} Ω m
Typical carbon content	95 wt %

2.3. Measurement of EMI shielding effectiveness and return loss

An EMI shield is a barrier made of conductive material that attenuates radiated electromagnetic energy. The shielding effectiveness (SE) is defined as the logarithm of the ratio of transmitted power (p_T) to the incident power (p_I) and expressed in decibels, i.e.

$$\mathrm{SE} = 10 \log_{10}(P_T/P_I) \text{ dB} \qquad (1)$$

The mechanism of SE is shown in Fig. 1, where the shielding material attenuates the radiated electromagnetic energy through direct reflection (R), multiple reflection (B) and absorption (A). SE can therefore be expressed as the sum of the above three components:

$$\mathrm{SE} = R + A + B \qquad (2)$$

A schematic diagram of the experimental set-up for the measurement of SE is shown in Fig. 2a. A sweep oscillator (HP 8620A) in the frequency range of 100 to 2000 MHz (HP 8621B) is connected with a standing wave ratio (SWR) meter (HP 415E) through an attenuator, a coaxial air line (GR 874-LIO) of 0.1 m length as a sample holder and a crystal detector (HP 423B).

The return loss (RL) of the composite can be obtained from the SWR value S by the relation

$$\mathrm{RL} = -20 \log_{10}\left(\frac{S-1}{S+1}\right) \qquad (3)$$

The S value of the composite was measured by the

TABLE III Formulations, fibre aspect ratio and volume resistivity of composites prepared by mill mixed method

	Composite No.					
	G	5m	10m	15m	20m	30m
Composition (phr)						
Neoprene WRT	100	100	100	100	100	100
Barium ferrite	10	10	10	10	10	10
Carbon fibre	–	5	10	15	20	30
Fibre aspect ratio, L/D	–	30	30	30	25	25
Volume resistivity (Ω m)	1.1×10^7	9.8×10^6	3.5×10^6	1.4	1.54×10^{-1}	3.5×10^{-2}

TABLE IV Formulations, fibre aspect ratio and volume resistivity of composites prepared by cement mixed method

	Composite No.					
	2.5c	5c	10c	15c	20c	30c
Composition (phr)						
Neoprene WRT	100	100	100	100	100	100
Barium ferrite	10	10	10	10	10	10
Carbon fibre	2.5	5	10	15	20	30
Fibre aspect ratio, L/D	110	110	110	100	100	100
Volume resistivity (Ω m)	7.5×10^6	14.5×10^{-2}	9.2×10^{-3}	5.4×10^{-3}	3.6×10^{-3}	2.3×10^{-3}

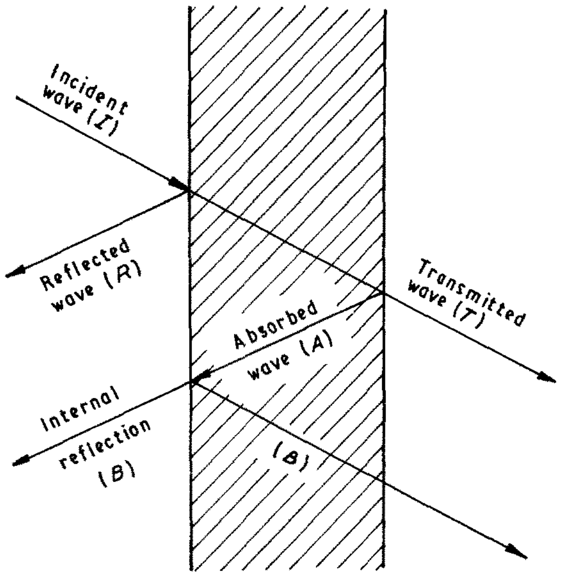

Figure 1 Mechanism of EMI shielding effectiveness.

experimental set-up shown in Fig. 2b. In this system, a sweep oscillator (HP 8620A) with plug-in r.f. section (HP 8621B) is connected with a coaxial terminator (HP 909A) through a slotted section containing a tunable detector mount (HP 440A) and coaxial air line as a sample holder. An SWR meter (HP 415E) was connected directly with the tunable detector mount.

A sectional view of the 0.1 m coaxial air line along with the inserted sample is shown in Fig. 3a. Fig. 3b depicts the corresponding sample dimensions. Two different sample thicknesses, namely 1.7 and 3.5 mm, were used for SE measurement. For SWR measurement the sample thickness was 3.5 mm.

The method of EMI shielding effectiveness measurement is basically an insertion loss measurement by substitution. Initially calibration was made with the empty sample holder (coaxial air line), and then the measurement was made with the sample inserted in the holder (Fig. 3).

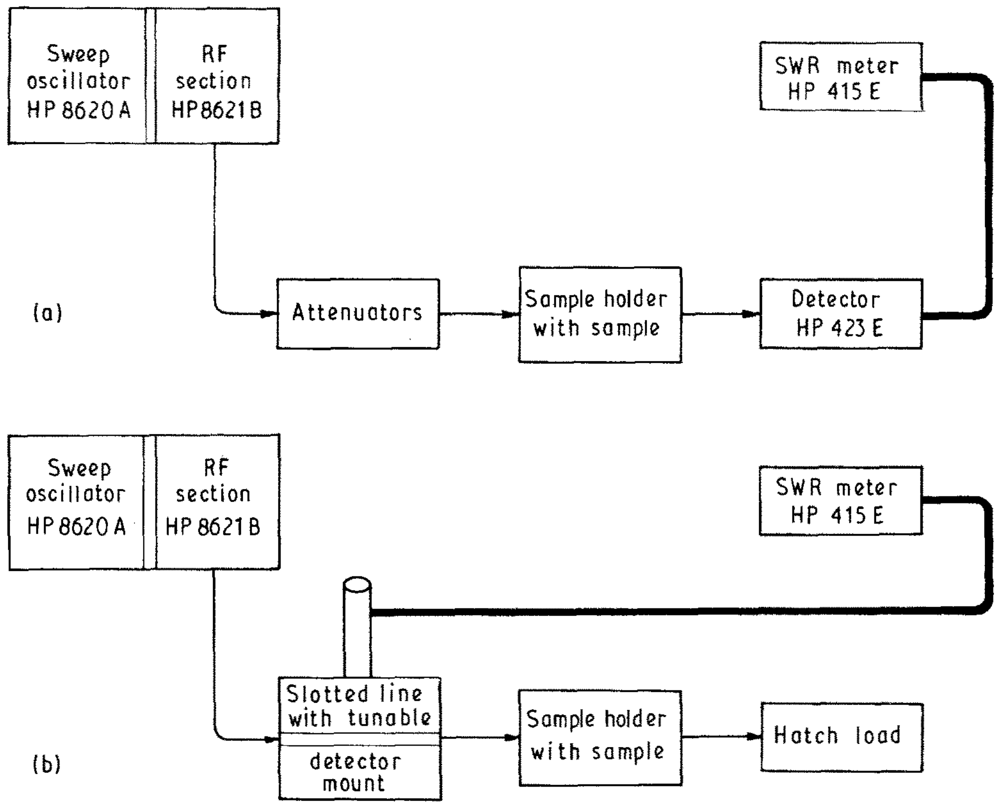

Figure 2 Schematic diagram of the experimental set-up for the measurment of (a) EMI shielding effectiveness and (b) SWR value.

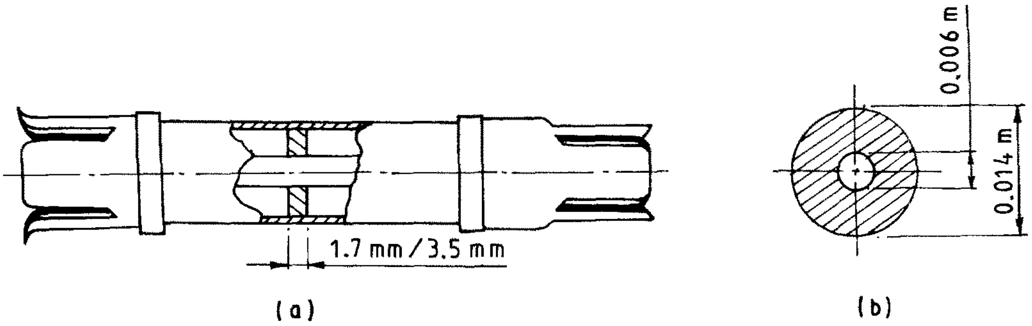

Figure 3 (a) Sectional view of 0.1 m coaxial air line sample holder along with an inserted sample; (b) sample with dimensions used in coaxial air line.

The value of SWR was obtained by measuring the level difference of the maximum and minimum points of the standing wave pattern when the sample was kept in the holder. Due to the limitations of the instrument, only the frequency range of 1100 to 2000 MHz could be used for SWR measurement.

3. Results and discussion

The spatial distribution of fibres in the composites was examined by SEM studies. Figs 4a and b show SEM photomicrographs of tensile fracture surfaces of the composites 20m (Table III and 20c (Table IV) prepared by mill-mixed and cement-mixed methods, respectively. The fracture surfaces of the composites indicate that the fibre distribution in both composites was random and the CF length was higher in 20c than in 20m. It was observed that the fibre aspect ratio (L/D) which was initially 800 got reduced to 25 in the mill mixed method and to 100 in the cement mixed method after processing (Tables III and IV).

3.1. Effects of carbon fibre concentration, aspect ratio and sample thickness

The variations of EMI shielding effectiveness (SE) as a function of frequency (100 to 2000 MHz) of CF-filled polychloroprene rubber composites, prepared by mill-mixed and cement-mixed methods, are shown in Figs 5 to 8. Barring a few composites of low CF concentration, the SE exhibits frequency dependency in all cases. The composites of low CF content show weak frequency dependency and have low SE values which are due to the high volume resistivity of the composites. However, at any particular frequency it was observed that the SE of the composite increased with increasing CF concentration. It was also observed that the SE of the unfilled composite (composite G of Table III) was negligible within the frequency range.

Figs 5 and 6 depict the SE of composites of different fibre aspect ratios, prepared by mill-mixed and cement-mixed methods, respectively, of the same thickness (i.e. 1.7 mm). It is apparent that the composites from the cement mixed method (Fig. 6) with a high fibre aspect ratio ($L/D \simeq 100$) show a higher SE than the composites from the mill mixed method (Fig. 5) with low fibre aspect ratio ($L/D \simeq 25$), although in both cases the CF concentration and sample thickness were the same. For instance, the composites 30c and 30m prepared by cement ($L/D \simeq 100$) and mill ($L/D \simeq 25$) mixed methods show SE values of 30 and 17.5 dB, respectively, at the frequency of 2000 MHz. The observed difference in SE values is mainly due to the effect of fibre aspect ratio, because in both com-

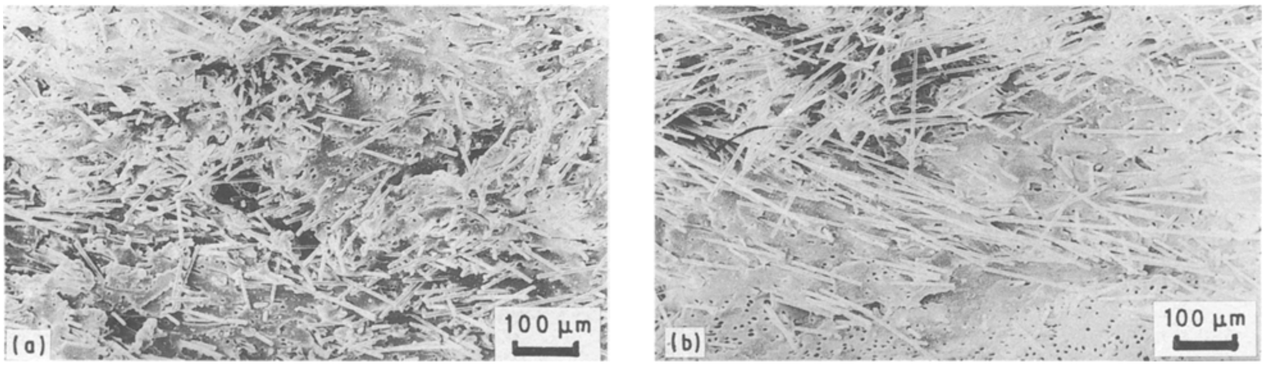

Figure 4 SEM photomicrographs of tensile fracture surfaces of polychloroprene–carbon fibre composites: (a) composite 20m and (b) composite 20c.

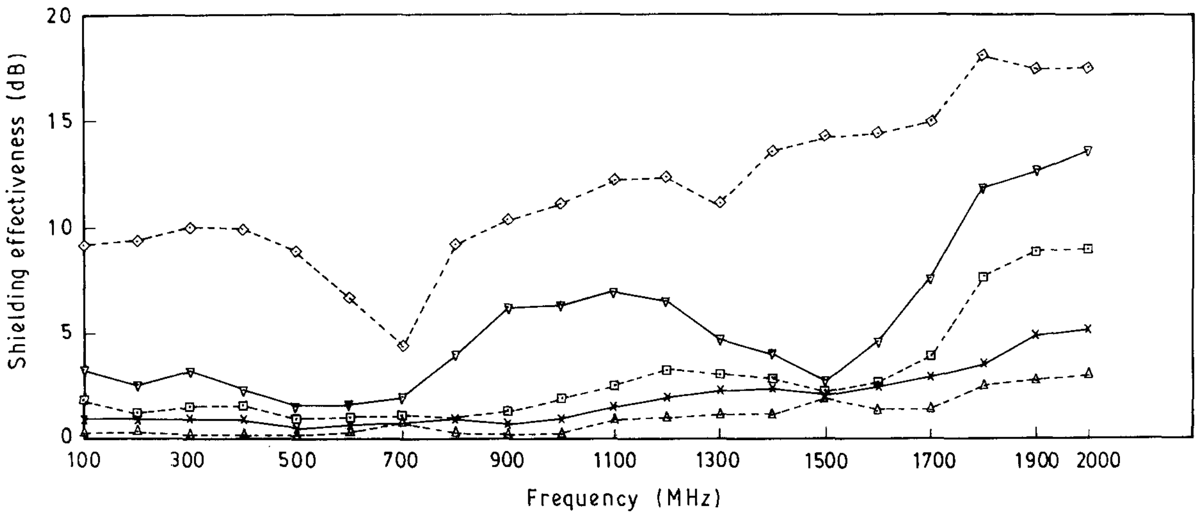

Figure 5 Shielding effectiveness as a function of frequency for composites of 1.7 mm thickness and fibre $L/D \simeq 25$, prepared by mill mixed method: (△) 5m, (×) 10m, (⊡) 15m, (▽) 20m, (◇) 30m.

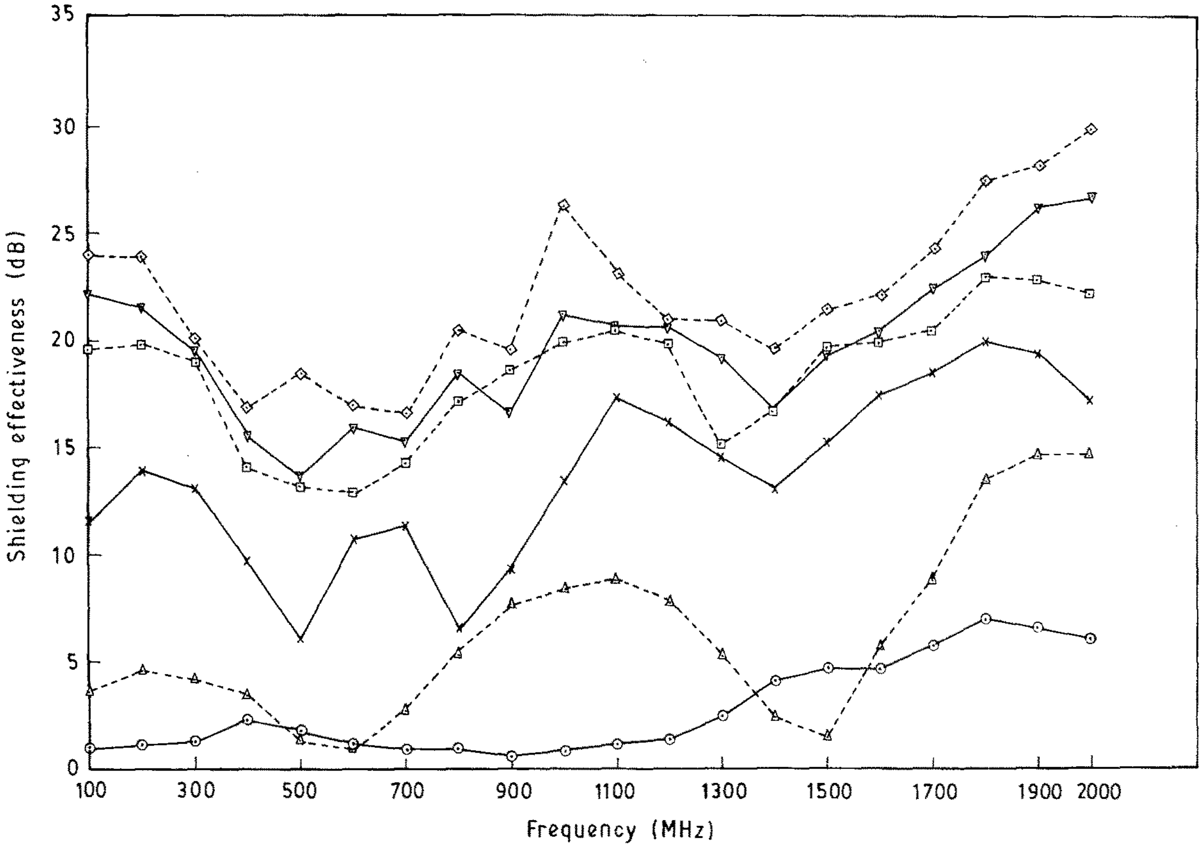

Figure 6 Shielding effectiveness as a function of frequency for composites of 1.7 mm thickness and fibre $L/D \simeq 100$, prepared by cement mixed method: (○) 2.5c, (△) 5c, (×) 10c, (⊡) 15c, (▽) 20c, (◇) 30c.

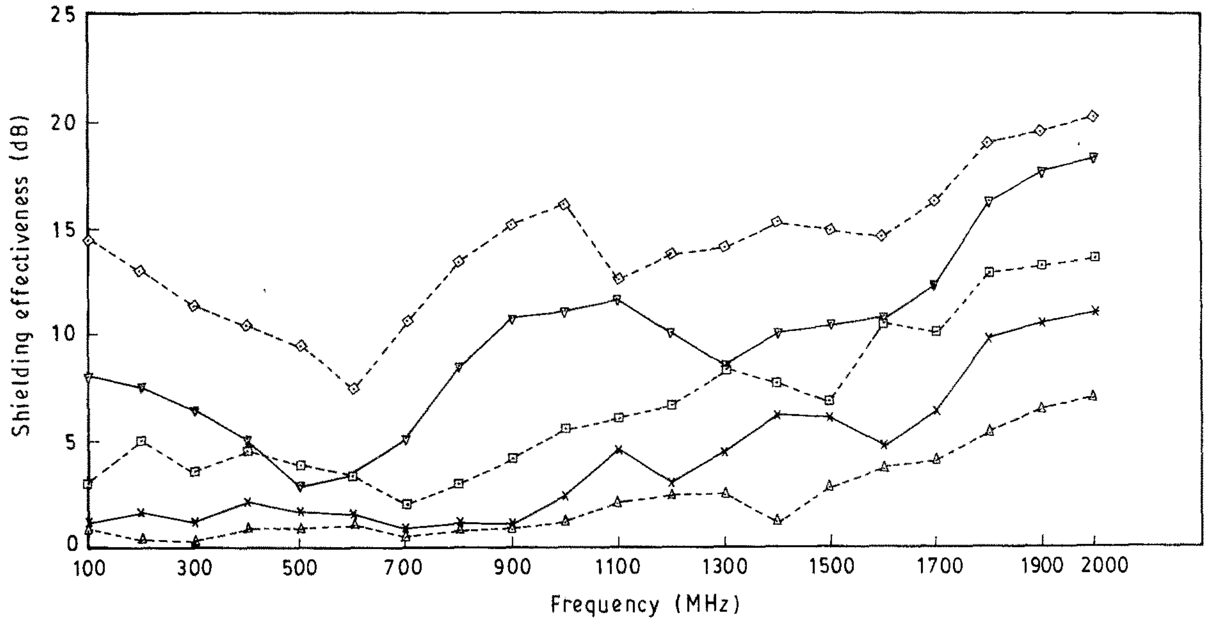

Figure 7 Shielding effectiveness as a function of frequency for composites of 3.5 mm thickness and fibre $L/D \simeq 25$, prepared by mill mixed method: (△) 5m, (×) 10m, (⊡) 15m, (▽) 20m, (◇) 30m.

posites the CF concentration was 30 phr (parts per hundred parts of rubber) and the sample thickness was 1.7 mm. Since the fibre randomness in the composites and electrical conductivity largely depend on the fibre aspect ratio, it is obvious from this instance that a very high fibre aspect ratio favours increased electrical conductivity and effective EMI shielding. Similar observations were made with the composites of 3.5 mm thickness (Figs 7 and 8).

The effect of composite thickness on SE was also studied when the CF concentration and aspect ratio were kept the same. The measured values of SE of composites prepared by the mill mixed method having two different thicknesses of 1.7 and 3.5 mm are shown in Figs 5 and 7, respectively. Similar observations were made in case of cement mixed composites, as shown in Figs 6 and 8. It is evident that with increase in composite thickness, the SE of the composite in-

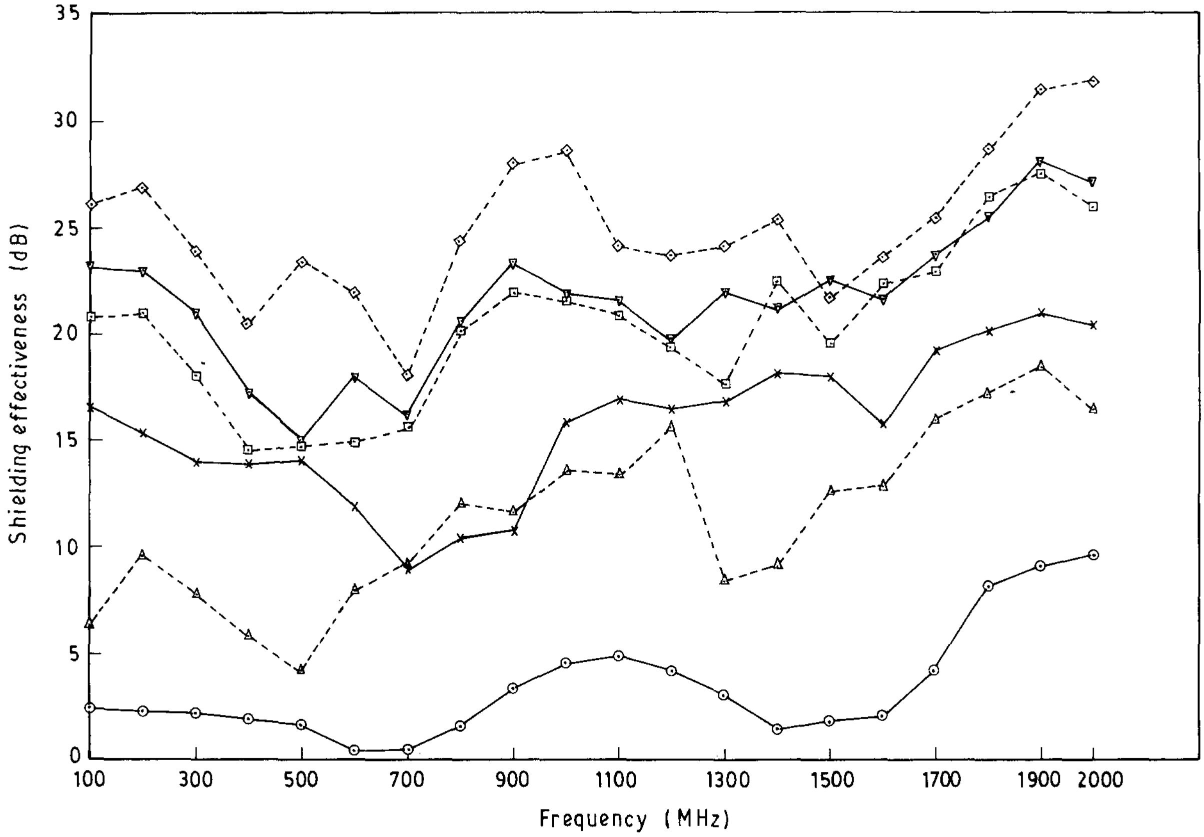

Figure 8 Shielding effectiveness as a function of frequency for composites of 3.5 mm thickness and fibre $L/D \simeq 100$, prepared by cement mixed method: (⊙) 2.5c, (△) 5c, (×) 10c, (⊡) 15c, (▽) 20c, (◇) 30c.

creases. For example, the composite 30c (Figs 6 and 8) at the frequency of 2000 MHz shows an SE of 30 and 32 dB for thickness of 1.7 and 3.5 mm, respectively, when the CF concentration (30 phr) and aspect ratio ($L/D \simeq 100$) were kept the same.

3.2. Effects of CF concentration and fibre aspect ratio on return loss

The values of SWR (S), needed for RL measurement using Equation 3, were obtained with the experimental set-up of Fig. 2b. Figs 9 and 10 show the variation in RL values with frequency (1100 to 2000 MHz) of composites of 3.5 mm thickness prepared by mill and cement mixed methods, respectively. It was observed that with increase in CF concentration the RL values of composites decreased. However, some composites of low CF concentration (5m and 10m of Table III and 2.5c of Table IV) show irregular variation of RL values with frequency. This is due to the high volume resistivity of the composites at low CF concentration.

The effects of CF aspect ratio on RL values are also shown in Figs 9 and 10. For any particular concentration of CF, a composite with high fibre aspect ratio ($L/D \simeq 100$, Fig. 10) shows a lower RL value compared to a composite of low fibre aspect ratio ($L/D \simeq 25$, Fig. 9). It is apparent from the preceding discussion that with increasing CF concentration and fibre aspect ratio in composites, the RL value decreases while the SE increases. This indicates that the loss due to the absorption (A) increases.

3.3. Relationship between EMI shielding effectiveness and volume resistivity of composites

The relationship between SE and volume resistivity of the composites (summarized in Tables III and IV), prepared by mill-mixed and cement-mixed methods, at a particular frequency of 2000 MHz are shown in Fig. 11a and b, respectively. It was observed that with increasing CF concentration the volume resistivity of the composite decreased and the SE gradually increased. A sharp decrease in volume resistivity was observed at a critical CF concentration which is shown in Fig. 11a and b. Beyond the critical point, further increase in CF concentration causes a slow decrease in volume resistivity but a rather moderate increase in SE. Furthermore, the increase of SE is more pronounced in composites of the cement mixed method (Fig. 11b) with a high fibre aspect ratio ($L/D \simeq 100$) as compared to the composites of the mill mixed method (Fig. 11a) with a low fibre aspect ratio ($L/D \simeq 25$). From Fig. 11a and b it is also evident that although volume resistivity is independent of composite thickness, the SE value increases with increase in thickness at a particular frequency.

4. Conclusions

1. With increase in carbon fibre concentration in the composites of barium ferrite-vulcanized polychloroprene, the EMI shielding effectiveness increases and the return loss decreases. Composites from the cement mixed method with a high fibre aspect ratio (L/D

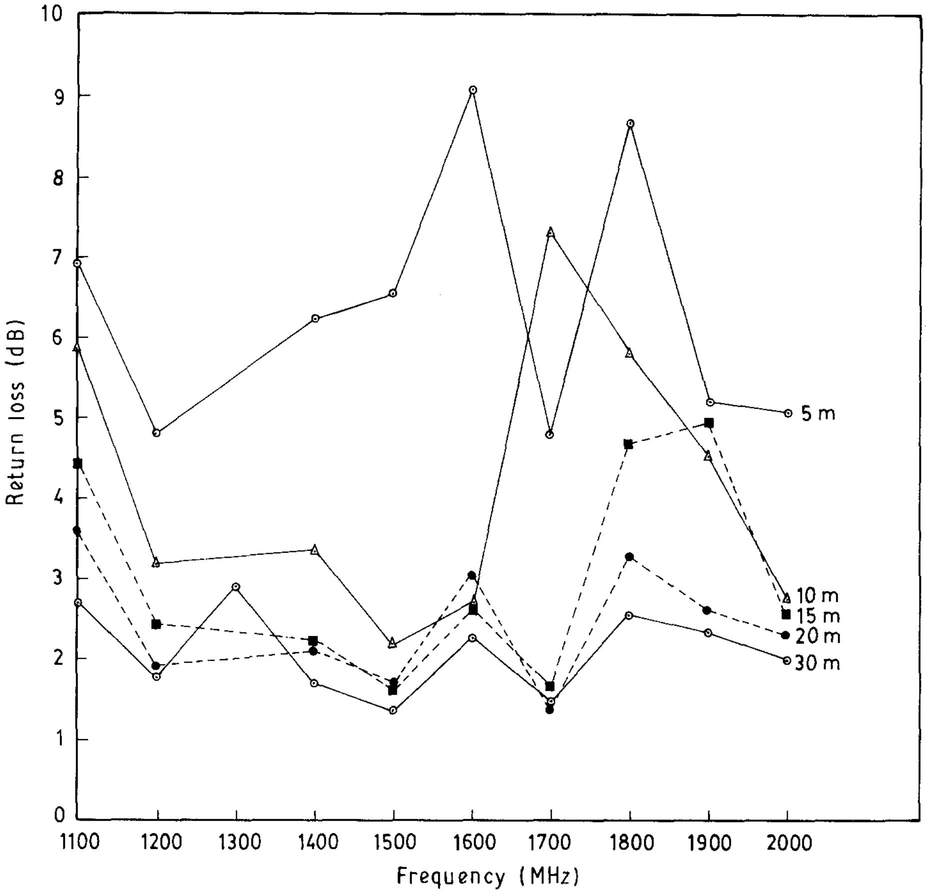

Figure 9 Return loss as a function of frequency for composites of 3.5 mm thickness and fibre $L/D \simeq 25$, prepared by mill mixed method.

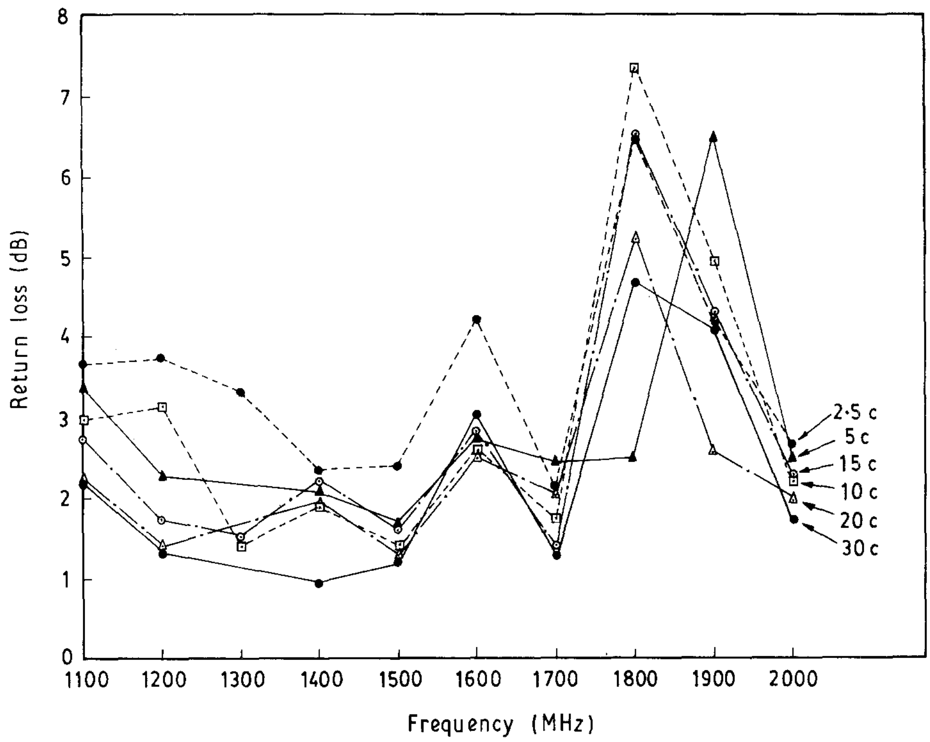

Figure 10 Return loss as a function of frequency for composites of 3.5 mm thickness and fibre $L/D \simeq 100$, prepared by cement mixed method.

$\simeq 100$) show a higher shielding effectiveness and lower return loss values than composites from the mill mixed method with a low fibre aspect ratio ($L/D \simeq 25$).

2. At any particular frequency, with increase in composite thickness the shielding effectiveness of the composite increases.

3. The composites with high fibre concentration, high fibre aspect ratio and high composite thickness show maximum shielding effectiveness and minimum return loss, which indicates that the loss is due to increased absorption.

4. From the relationship between volume resistivity and shielding effectiveness it is evident that at a par-

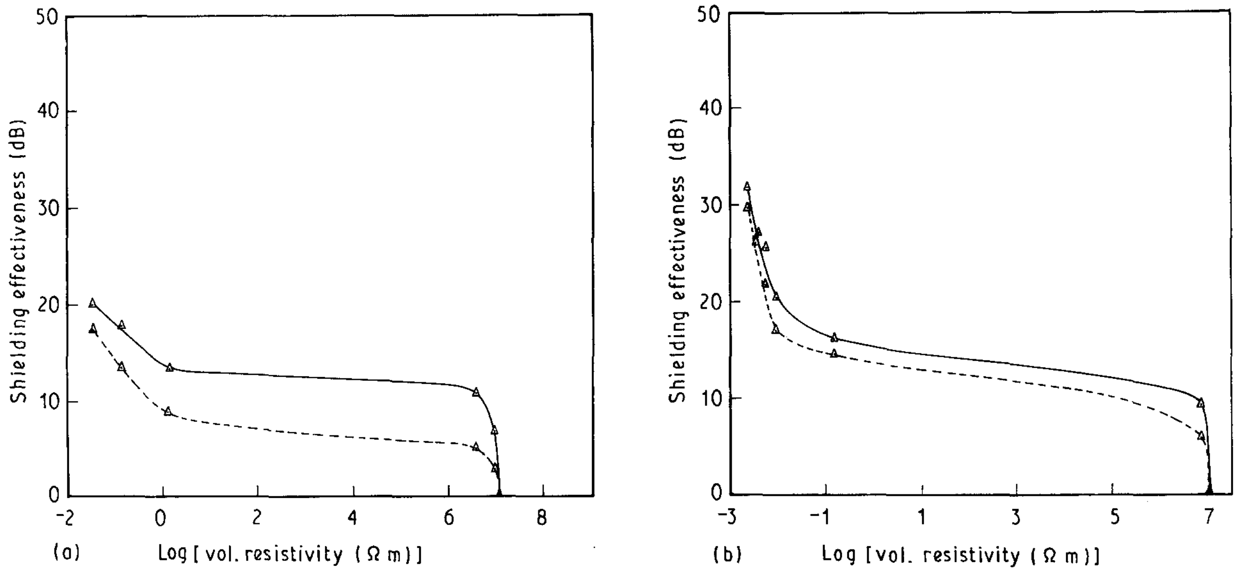

Figure 11 Relationship between EMI shielding effectiveness and volume resistivity of composites prepared by (a) mill mixed method ($L/D \simeq$ 25) and (b) cement mixed method ($L/D \simeq$ 100). Frequency = 2 GHz; thickness (--) 1.7 mm, (—) 3.5 mm.

ticular frequency, the shielding effectiveness depends not only upon the conductivity of the composite, but also upon the fibre aspect ratio and composite thickness.

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