0.4 μm Diameter Nickel-Filament Silicone-Matrix Resilient Composites for Electromagnetic Interference Shielding

Xiaoping Shui

D. D. L. Chung

Composite Materials Research Laboratory,
State University of New York at Buffalo,
Buffalo, NY 14260-4400

Resilient silicone-matrix composites containing 7–13 volume percent nickel filaments (0.4 μm diameter) exhibited 74–93 dB electromagnetic interference (EMI) shielding effectiveness at 1–2 GHz and $1 \times 10^{-1} - 2 \times 10^{-2}$ Ω cm DC volume electrical resistivity. The high shielding effectiveness is due to the small diameter of the nickel filaments. The composites are useful for EMI shielding gaskets and cable jackets.

Introduction

Resilient materials that are capable of electromagnetic interference (EMI) shielding are needed for serving as gaskets for sealing enclosures for EMI shielding and as electrical cable jackets for EMI shielding. EMI shielding is increasingly important due to the sensitivity of digital electronics and the increasing use of RF wireless devices, which tend to interfere with the digital devices. Resilient materials for EMI shielding are in the form of polymer-matrix composites in which the polymer is an elastomer, such as silicone. These composites are capable of EMI shielding because they contain a filler which is electrically conducting, such as metal particles, metal flakes, metal fibers, and carbon fibers. For a given filler type, the shielding effectiveness increases with increasing filler volume fraction. Thus, in order to attain a high shielding effectiveness, a high filler volume fraction is needed. On the other hand, the stiffness of the elastomer-matrix composite also increases with increasing filler volume fraction. As a result, the attaining of both high shielding effectiveness and good resilience has been a challenge. Nickel fibers of 20 μm diameter had been previously used as a filler in silicone to produce resilient composites with EMI shielding effectiveness > 50 dB at 1–2 GHz (Zhu and Chung, 1991). Due to the skin depth effect, a smaller diameter of the nickel fibers is preferred for EMI shielding. In this paper, by using nickel filaments of 0.4 μm diameter (Shui and Chung, 1995) in silicone, a shielding effectiveness of 90 dB (at 1–2 GHz) was achieved in the resulting resilient composite. This shielding effectiveness is higher than that of all previously reported resilient polymer-matrix composites (Zhu and Chung, 1991; Radhakrishnan and Saini, 1991; Kost et al., 1983; Jana, 1993).

Experimental Methods

The silicone used was Silicone Rubber RTV615 (two parts) of General Electric Company. The specific gravity was 0.99. The nickel filaments used were the same as those in Shui and Chung (1995). Their diameter was 0.4 μm; their length was >100 μm. They were not straight, but resembled cotton wool. Each nickel filament contained a carbon core of diameter 0.1 μm, so that the filament contained 94 volume percent nickel and 6 volume percent carbon.

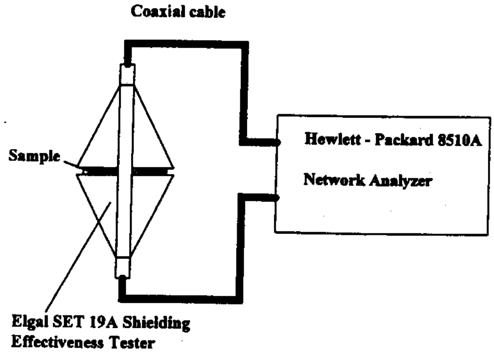

Fig. 1 EMI shielding effectiveness measurement set-up

Composite fabrication was carried out by (1) mixing Part A (resin) and Part B (curing agent) of silicone rubber at a weight ratio of 10:1, (2) adding nickel filaments and mixing, (3) pressing in a mold at 400 psi (2.8 MPa) and room temperature for 12 h, and (4) while keeping the pressure at 400 psi, heating at 60–70°C for 2 h.

The EMI shielding effectiveness was measured in transmission using the coaxial cable method (ASTM-D4935) (Fig. 1). The sample was in the form of an annular ring of outer diameter 3.8 in. (97 mm) and inner diam 1.25 in. (32 mm). The sample thickness was 2.85 mm for all the composites, 3.1 mm for solid copper, and 4.0 mm for solid stainless steel. The frequency was scanned from 1 to 2 GHz such that 101 shielding effectiveness data points were taken within this frequency range. The error of each data point was better than ±1 dB at < 10 dB, and ±5 dB at >70 dB; the error increased with increasing attenuation (dB).

The volume electrical resistivity of the composites was measured by the four-probe method, using silver paint as electrical contacts. The samples were of size 100 × 5 × 3 mm, such that the current was along the longest dimension and the inner (voltage) probes were 50 mm apart in this direction.

The resilience of the composites was tested by compressing cylindrical specimens using a Sintech/2D screw action mechanical testing system at a constant strain of 10 percent for 24 h (during which the stress was measured), and subsequently releasing the load and measuring the permanent set. The speci-

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Contributed by the Electrical and Electronic Packaging Division for publication in the JOURNAL OF ELECTRONIC PACKAGING. Manuscript received by the EEPD*, 1996; revision received November 1996. Associate Technical Editor: R. R. Schmidt.

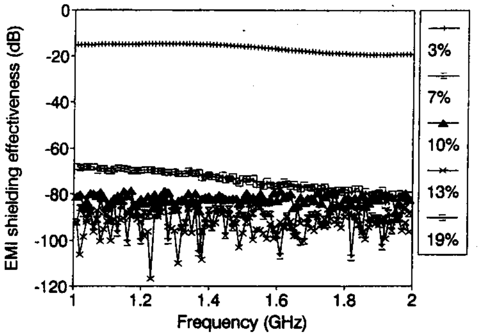

Fig. 2 EMI shielding effectiveness as a function of frequency for silicone-matrix composites with 3–19 percent by volume of nickel filaments

mens were 0.5 in. (13 mm) in diameter and 0.5 in. (13 mm) in height.

Results and Discussion

Figure 2 gives the EMI shielding effectiveness as a function of frequency from 1 to 2 GHz for each composite. Table 1 gives the average shielding effectiveness (averaged over 101 data points from 1 to 2 GHz) for each composite. The shielding effectiveness was over 70 dB for all nickel filament volume fractions from 7 to 19 percent. The composites at ≤ 13 volume percent filaments were flexible, whereas that at 19 volume percent filaments was hard. Thus, both high shielding effectiveness and flexibility were attained at 7–13 volume percent filaments.

The silicone-matrix composites of this work and the polyether sulfone (PES) -matrix composites of Shui and Chung (1995) gave comparable shielding effectiveness at the same filler volume fraction (Table 1), except that silicone gave lower shielding effectiveness at 7 volume percent nickel filaments than PES. This is attributed to the liquid form of the silicone resin and the solid powder form of the PES (Shui and Chung, 1995). The difference in processing probably caused a higher contact resistivity between the adjacent filament agglomerates in the silicone case.

Figure 3 gives the DC volume electrical resistivity as a function of filler volume fraction. The resistivity was much lower for composites with 20 μm diameter nickel fibers (Zhu and Chung, 1991) than composites with 0.4 μm diameter nickel filaments at the same filler volume fraction. The higher resistivity of the latter is attributed to the larger concentration of contact points between adjacent filler units at the same filler volume fraction and to the presence of a polymer matrix film between contacting filaments. Zhu and Chung (1991) fabricated the nickel fiber composites not by using a slurry, but by infiltration of the silicone resin into a nickel fiber compact, so the polymer film between contacting filaments was avoided. Although the infiltration method provides lower resistivity, it can be used to make composites with simple shapes only. On the other hand, slurry methods are more versatile in forming composites of various shapes, whether in bulk or thick film forms.

Table 1 EMI shielding effectiveness (dB) of composites with nickel filaments. The standard deviations are shown in parentheses.

Filler volume percent	Silicone matrix	PES matrix
7	74.2 (4.3)	86.6 (5.1)*
10	82.2 (2.1)	/
13	93.4 (5.3)	83.7 (5.3)*
19	90.5 (5.5)	91.7 (6.6)*

* from Shui and Chung (1995)

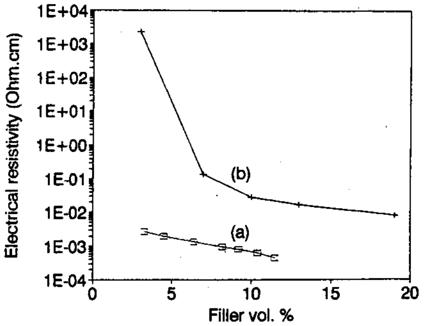

Fig. 3 DC volume electrical resistivity as a function of filler volume fraction for silicone-matrix composites with (*a*) 20 μm diameter nickel fibers (Zhu and Chung, 1991), and (*b*) 0.4 μm diameter nickel filaments (this work)

Table 2 Permanent set after compression at a constant strain of 10 percent for 24 h

Volume percent nickel filaments	Permanent set (percent)
0	0
3	0
7	0.88
10	1.3
13	1.3
19	1.3

Although the resistivities of the nickel filament composites are higher than those of the nickel fiber composites (Zhu and Chung, 1991), they are lower than previously reported elastomer-matrix composites with metal particles (Radhakrishnan and Saini, 1991), carbon black (Kost et al. 1983), or carbon fibers (Jana, 1993) at the same volume fraction.

Table 2 gives the permanent set after compression at a constant strain of 10 percent for 24 h. The permanent set was low for all the composites, although it increased with increasing filament volume fraction. Figure 4 shows that the stress decreased during the 24 h compression (shown for 12 h) for all composites, such that the extent of stress relaxation increased with increasing filament volume fraction. For the neat polymer, stress relaxation was negligible.

Conclusion

Silicone-matrix composites containing 3–19 volume percent nickel filaments (0.4 μm diameter) were fabricated and tested in terms of the EMI shielding effectiveness, DC electrical resistivity, compressive strain reversibility, and stress relaxation. The shielding effectiveness and stress relaxation increased with

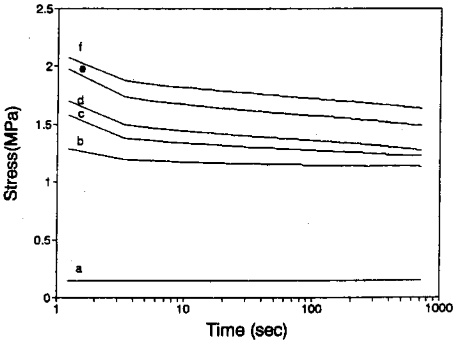

Fig. 4 Variation of stress with time during compression at a constant strain of 10 percent. The nickel filament volume fraction was (*a*) 0 percent, (*b*) 3 percent, (*c*) 7 percent, (*d*) 10 percent, (*e*) 13 percent and (*f*) 19 percent

increasing filament volume fraction, whereas the resistivity and compressive strain reversibility decreased with increasing filament volume fraction. For electrical applications that require resilience, nickel filament volume fractions ranging from 3 percent to 13 percent are recommended, as such composites exhibited EMI shielding effectiveness at 1–2 GHz of 74–93 dB, electrical resistivity of 0.02–0.1 Ω.cm, and permanent compression set of 0–1.3 percent.

Acknowledgment

This work was supported by Department of Navy (SBIR Program) via Applied Sciences, Inc. and by the Center for Electronic and Electro-Optic Materials of the State University of New York at Buffalo. Technical assistance in shielding effectiveness testing by Professor J. J. Whalen of the State University of New York at Buffalo is greatly appreciated.

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