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Nickel Filament Polymer-Matrix Composites with Low Surface Impedance and High Electromagnetic Interference Shielding Effectiveness

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The processing of nickel filaments of 0.4 μm diameter gives polyethersulfone-matrix composites with high electromagnetic interference shielding effectiveness, high reflection coefficient and low surface impedance at 1–2 GHz. With 7 vol.% nickel filaments, the composite exhibited shielding effectiveness 87 dB (compared to 90 dB for solid copper), surface impedance 1.2 Ω (same as for solid copper), tensile strength 52 MPa, modulus 5 GPa, ductility 1.0%, and density 1.87 g/cm^3.

Key words: Carbon, electromagnetic interference shielding, fibers, filaments, mechanical properties, nickel, polymer-matrix composites, surface impedance

INTRODUCTION

Nickel filaments of submicron diameter have been shown to be exceptionally effective for electromagnetic interference (EMI) shielding at 1–2 GHz, when used as a filler in a polymer.[1] As shown in Table I, nickel filaments of 0.4 μm diameter are more effective than aluminum flakes, steel fibers, carbon fibers (10 μm diameter), carbon filaments (0.1 μm diameter), nickel particles, nickel fibers (2 μm diameter), and nickel fibers (20 μm diameter)[1–3] However, the DC electrical resistance of the nickel filament (0.4 μm diameter) composites is not exceptionally low.[1] The AC surface impedance of these composites have not been previously reported, even though it is relevant to understanding the shielding effectiveness and to the use of the composites for lightweight microwave waveguides (associated with radars in military aircrafts and satellites) and for electrostatic discharge protection. Moreover, the mechanical properties of these composites have not been previously reported, even though they are relevant to the practical use of these composites. The attaining of both high ductility and high shielding effectiveness (low surface impedance) has been a challenge among polymer-matrix composites due to the decrease of the ductility with increasing filler volume fraction and the usual need for a high filler volume fraction in order to attain a high shielding effectiveness (low impedance). This paper is thus focused on the surface impedance and mechanical properties, and their dependence on the filler volume fraction.

EXPERIMENTAL METHODS

The nickel filaments (0.4 μm in diameter, >100 μm in length), composite fabrication (with polyethersulfone or PES as the matrix), and EMI shielding effectiveness measurement were described in Ref. 1. The performance of our nickel filaments was compared with that of three types of fibers and with that of three bulk materials.

The fibers used for comparison with our nickel filaments were:

- Nickel, 2 μm in diameter and 2000 μm in length, as provided by Ribtec (Gahanna, OH)
- Nickel, 20 μm in diameter and 1000 μm in length, as provided as Fibrex by National-Standard Co. (Corbin, KY), and

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Table I. EMI Shielding Effectiveness at 1–2 GHz of Polyethersulfone (PES)-Matrix Composites with Various Fillers

Filler	Vol.%	EMI Shielding Effectiveness (dB)	Ref.
Al flakes (15 × 15 × 0.5 μm)	20	26	2
Steel fibers (1.6 μm dia. × 30 ~ 56 μm)	20	42	2
Carbon fibers (10 μm dia. × 400 μm)	20	19	2
Ni particles (1 ~ 5 μm dia.)	9.4	23	3
Ni fibers (20 μm dia. × 1 mm)	19	5	1
Ni fibers (2 μm dia. × 2 mm)	7	58	1
Carbon filaments (0.1 μm dia. × > 100 μm)	7	32	1
Ni filaments (0.4 μm dia. × > 100 μm)	7	87	1

Note: Sample thickness ~2.8 mm.

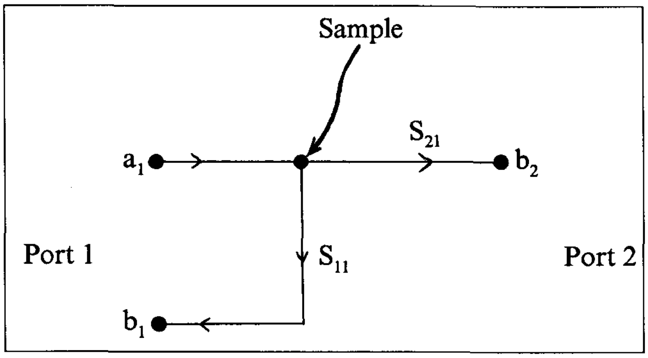

Fig. 1. Method for measuring surface impedance and EMI shielding effectiveness.

- carbon, 0.1 μm in diameter and >100 μm in length, as provided by Applied Sciences Inc. (Cedarville, OH).

Comparison was also made with the performance of bulk copper, nickel, and stainless steel. Due to the large length of the nickel fibers of diameter 2 μm (which resemble cotton wool), the dispersion of these fibers was most difficult.

Composites of each of the four fibrous materials were fabricated by forming a dry mixture of the polymer powder and the filler and subsequent hot pressing in a steel mold at 310°C (processing temperature for PES, as recommended by ICI, Inc.) and 13.4 MPa for ~30 min. For our nickel filaments and the 20 μm diameter Ni fibers, the mixing was carried out dry in a ball mill. For the carbon filaments, mixing was carried out wet—with water in a blender, and then the wet mix was dried at 120°C. For the 2 μm diameter Ni fibers, mixing was performed by hand, as neither the abovementioned dry mixing nor wet mixing was possible. The surface impedance and the EMI shielding effectiveness at 1–2 GHz were measured in the same way, except that the former was obtained by measurement of the radiation reflected from the sample, whereas the latter was obtained by transmission of the radiation through the sample, as illustrated in Fig. 1, where

$$S_{11} = \frac{b_1}{a_1} \tag{1}$$

is the reflection coefficient (related to the surface impedance Z_L of the sample), and

$$S_{21} = \frac{b_2}{a_1} \tag{2}$$

is the transmission coefficient (related to the shielding effectiveness). The instrumental characteristic impedance Z_o is 50 Ω. Because

$$S_{11} = \frac{Z_L - Z_o}{Z_L + Z_o} = \frac{Z_L / Z_o - 1}{Z_L / Z_o + 1}, \tag{3}$$

then

$$\frac{Z_L}{Z_o} = \frac{1 + S_{11}}{1 - S_{11}} = \frac{1 + |S_{11}| e^{i\phi}}{1 - |S_{11}| e^{i\phi}}. \tag{4}$$

Thus, we have

$$\left| \frac{Z_L}{Z_o} \right| = \sqrt{\frac{1 + |S_{11}|^2 + 2|S_{11}| \cos\phi}{1 + |S_{11}|^2 - 2|S_{11}| \cos\phi}} \tag{5}$$

Hence, the surface impedance Z_L can be obtained by measuring S_{11} and the phase angle ϕ. An HP8510A network analyzer and an HP computer (with HP Basic Operation System) were used to obtain both $|S_{11}|$ and ϕ at 1–2 GHz. An HP APC-7 calibration kit was used to calibrate the system. Then, $|Z_L|$ was obtained from Eq. (5). The frequency was scanned from 1 to 2 GHz such that 101 data points were taken within this frequency range.

The sample was in the form of an annular ring of outer diameter 97 mm and inner diameter 32 mm. Silver paint was used to paint the edges of the sample at both the outer and inner diameters. 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.

Tensile testing was conducted using a Sintech 2/D screw-action mechanical testing system. The displacement rate was 1.0 mm/min. Strain was measured using a strain gage (EA-13-120LZ-120, Micro-measurements Group Inc., Raleigh, NC) mounted on each sample. The sample was of size 80 × 8 × 2.85 mm.

Four samples of each type were tested.

RESULTS AND DISCUSSION

Table I shows the EMI shielding effectiveness, reflection coefficient S_{11} and surface impedance, each averaged over the 101 data points in the frequency range 1–2 GHz. The samples were in the form of PES-matrix composites having various volume fractions of four different fillers (Ni filaments, carbon filaments, 2 μm diameter Ni fibers and 20 μm Ni fibers). Measurements are also reported for solid copper, nickel, and stainless steel. Shown in parentheses are the standard deviation of the 101 data points. The effects of the test fixture reflections were not eliminated. The shielding effectiveness results are the same as those in Ref. 1. The S_{11} values show that most of the power incident upon each sample was reflected. The Ni filaments gave composites of very high shielding effectiveness and very low surface impedance even at a low filler volume fraction. For example, at 7 vol.%, the Ni filaments gave a shielding effectiveness of 87 dB and an impedance of 1.2 Ω; both properties are close to those of solid copper. By increasing the volume fraction to 13%, the impedance was even less

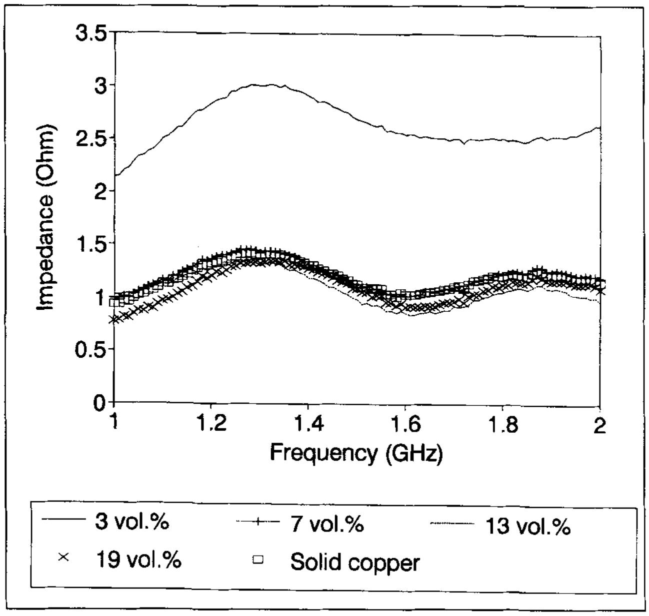

Fig. 2. Variation of surface impedance with frequency of composites with various volume fractions of nickel filaments. Data for solid copper are included as a reference.

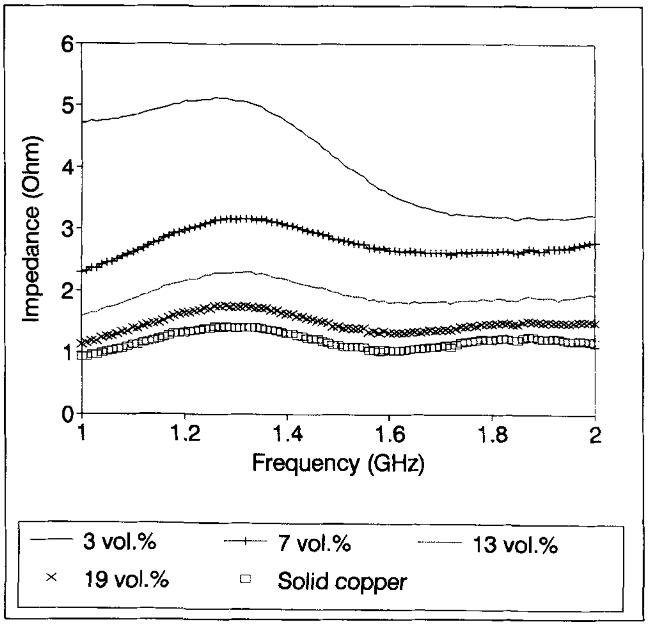

Fig. 3. Variation of surface impedance with frequency of composites with various volume fractions of carbon filaments. Data for solid copper are included as a reference.

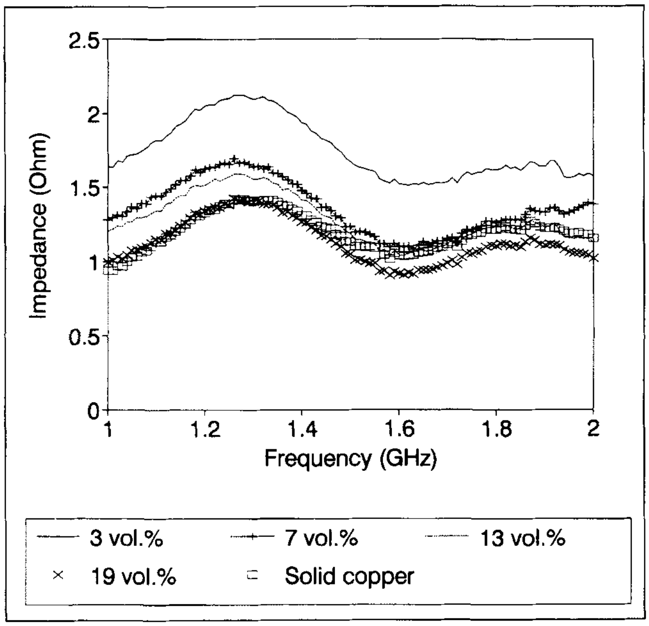

Fig. 4. Variation of surface impedance with frequency of composites with various volume fractions of 2 μm diameter nickel fibers. Data for solid copper are included as a reference.

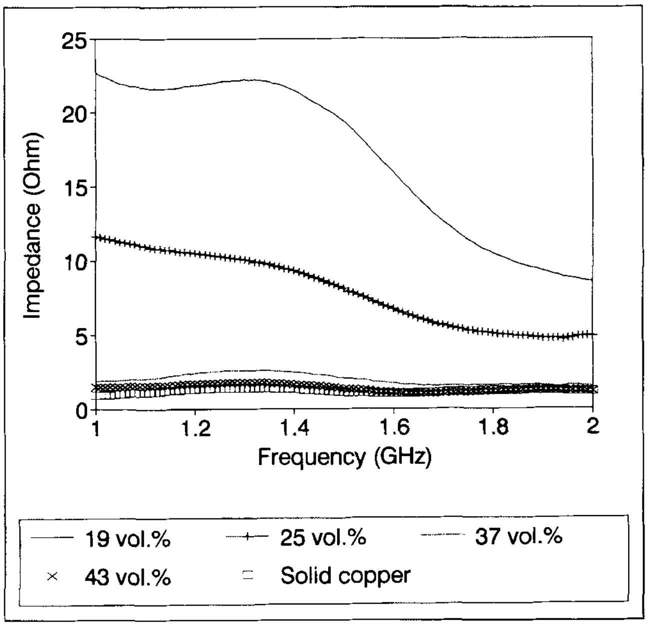

Fig. 5. Variation of surface impedance with frequency of composites with various volume fractions of 20 μm diameter nickel fibers. Data for solid copper are included as a reference.

than that of solid copper. For the carbon filaments, even at 19 vol.%, the impedance was above that of solid copper. For the 2 μm diameter Ni fibers, the volume fraction needed to be 19% in order for the impedance to be below that of solid copper. For the 20 μm diameter Ni fibers, even at 43 vol.%, the impedance was above that of solid copper. Hence, both the impedance and shielding effectiveness results are consistent in indicating the exceptional ability of the Ni filaments. Furthermore, the high values of S_{11} indicate high surface electrical conductance.

Figures 2–5 show the variation of the impedance with frequency for the composites with Ni filaments, carbon filaments, 2 μm diameter Ni fibers, and 20 μm diameter Ni fibers, respectively. In each case, comparison was made with the result of solid copper. For the Ni filaments (Fig. 2), the impedance was close to that of solid copper at volume fractions of 7% or above. For the carbon filaments (Fig. 3), the impedance was above that of solid copper even at a volume fraction of 19%. For the 2 μm diameter Ni fibers (Fig. 4), the impedance was close to that of solid copper at volume fractions of 13% or above. For the 20 μm diameter Ni fibers (Fig. 5), the impedance was above that of copper even at a volume fraction of 43%. Figure 6 shows the comparison among solid copper, solid nickel, and solid stainless steel. Their relative impedance depends on the frequency.

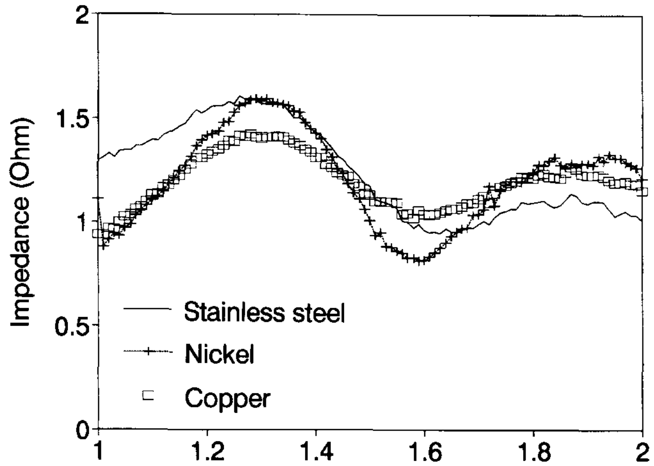

Fig. 6. Variation of surface impedance with frequency of solid copper (□), nickel (+) and stainless steel (—).

Figures 7–10 show the dependence on the filler volume fraction of the tensile strength, modulus, ductility, and density, respectively, of the same materials as Table II. The strength (Fig. 7) decreased monotonically with increasing filler volume fraction for all the fillers except the 2 μm diameter Ni fibers, for which the strength increased with increasing filler volume fraction up to 13% and then decreased with further increase of the volume fraction. This means that the filler-matrix bonding was strongest for the 2 μm diameter Ni fibers, so that only these fibers were able to reinforce the composite. In all cases, the decrease of the strength with increasing filler volume fraction was due to the increase in void content with increasing filler content and the weak filler-matrix bonding. Themodulus (Fig. 8) increased monotonically with increasing filler volume fraction for all the

Table II. EMI Shielding Effectiveness, Reflection Coefficient S_{11} and Surface Impedance at 1–2 GHz for the Nickel Filament Composites, for Three Other Fiber Composites, and for Three Solid Shields

Material	Filler Vol %	EMI Shielding Effectiveness (dB)	S_{11}	Surface Impendance (Ω)	Surface Impedance Relative to Cu
Ni filaments/PES composites	3	42.2(2.4)	0.908(0.007)	2.65(0.21)	2.22(0.13)
	7	86.6(5.1)	0.953(0.005)	1.22(0.13)	1.02(0.02)
	13	83.7(5.3)	0.964(0.005)	1.09(0.15)	0.91(0.07)
	19	91.7(6.6)	0.957(0.005)	1.10(0.15)	0.92(0.04)
Carbon filaments/PES composites	3	20.6(1.3)	0.854(0.022)	4.11(0.78)	3.44(0.64)
	7	31.8(1.7)	0.898(0.008)	2.78(0.22)	2.33(0.13)
	13	53.6(3.5)	0.929(0.007)	1.96(0.18)	1.64(0.07)
	19	73.9(5.1)	0.944(0.006)	1.48(0.15)	1.23(0.02)
2 μm Ni fibers/PES composites	3	45.2(2.5)	0.933(0.007)	1.76(0.20)	1.47(0.13)
	7	58.1(4.2)	0.947(0.006)	1.36(0.18)	1.14(0.11)
	13	60.3(3.2)	0.951(0.006)	1.29(0.17)	1.07(0.09)
	19	71.7(4.6)	0.957(0.005)	1.14(0.15)	0.95(0.08)
20 μm Ni fibers/PES composites	19	4.9(1.9)	0.714(0.084)	17.0(5.2)	14.3(4.5)
	25	10.5(2.3)	0.800(0.039)	7.93(2.41)	6.78(2.17)
	37	38.4(1.9)	0.939(0.012)	1.98(0.39)	1.65(0.25)
	43	73.7(4.4)	0.964(0.005)	1.40(0.25)	1.17(0.17)
Copper	/	90.2(5.0)	0.953(0.005)	1.20(0.13)	1.00
Nickel	/	82.1(6.8)	0.961(0.005)	1.21(0.22)	1.01
Stainless steel	/	88.9(4.0)	0.954(0.007)	1.24(0.22)	1.03

Note: Standard deviations are shown in parentheses.

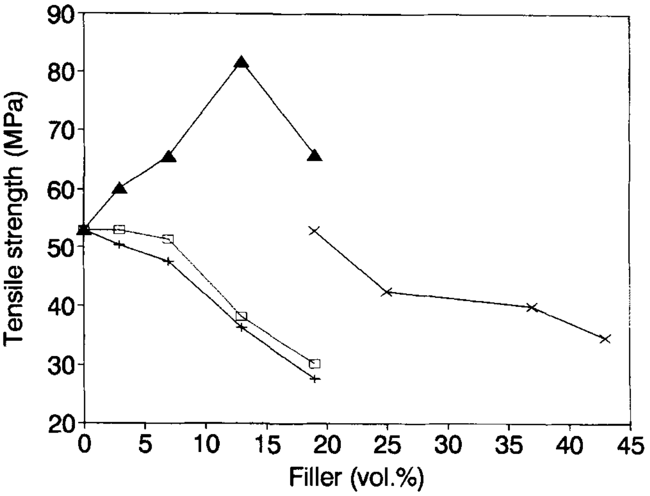

Fig. 7. Variation of tensile strength with filler volume fraction. +: carbon filaments; □: nickel filaments; ▲: 2 μm diameter Ni fibers; x: 20 μm diameter Ni fibers.

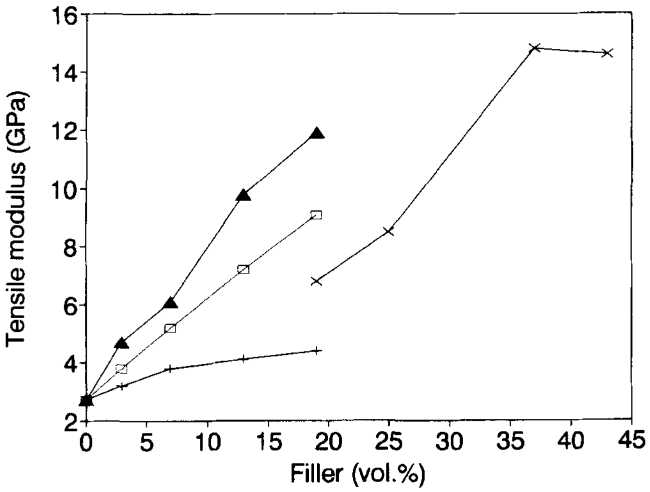

Fig. 8. Variation of tensile modulus with filler volume fraction. +: carbon filaments; □: nickel filaments; ▲: 2 μm diameter Ni fibers; x: 20 μm diameter Ni fibers.

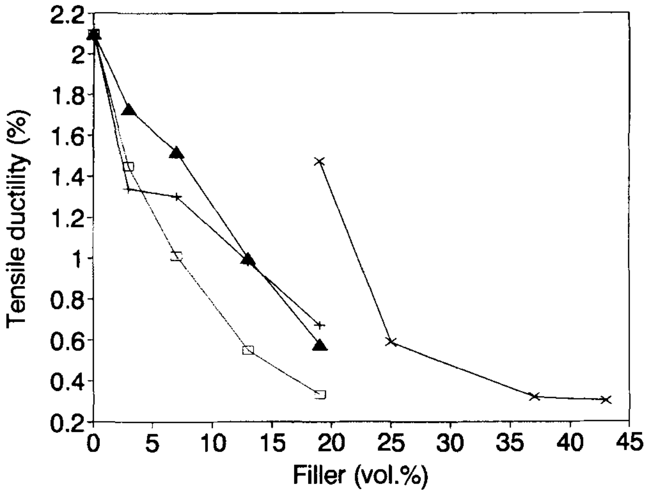

Fig. 9. Variation of tensile ductility with filler volume fraction. +: carbon filaments; □: nickel filaments; ▲: 2 μm diameter Ni fibers; x: 20 μm diameter Ni fibers.

fibers. The ductility (Fig. 9) decreased monotonically with increasing filler volume fraction for all the fibers. The tensile strength was highest for composites with the 2 μm diameter Ni fibers and lowest for composites with the carbon filaments (Fig. 7). The strength (Fig. 7) and modulus (Fig. 8) of the nickel filament composites were higher than those of the carbon filament composites, but lower than those of the 2 μm diameter Ni fiber composites at the same corresponding filler volume fraction. The ductility (Fig. 9) was lower for the nickel filament composites than both the carbon filament composites and the 2 μm diameter Ni fiber composites at the same corresponding filler volume fractions from 7 to 19%. The clinginess (like cotton wool) of the nickel filaments and carbon filaments is believed to cause the ductility of these composites to be lower than those of the nickel fiber composites. The higher ductility (at 7–19 vol.%), lower modulus and lower strength of the carbon filament composites compared to the nickel filament composites are attributed to the smaller diameter and probably weaker filler-matrix bonding of the carbon filaments.

The carbon filament composites are advantageous over all the nickel composites in their low density (Fig. 10). Nevertheless, at a low filler volume fraction of 7%, the nickel filament composite's density was quite low (1.87 g/cm^3).

Although the nickel filament composites are not particularly attractive in ductility, strength or modulus compared to the other composites at the same filler volume fraction (at or above 7%), the nickel filaments are the only filler that gives simultaneously shielding effectiveness exceeding 70 dB, surface impedance $\leq 1.5\ \Omega$, ductility >0.7%, and strength >40 MPa, when all filler volume fractions are considered, as shown in Table III, which lists the composite with the best overall (electrical and mechanical) performance for each filler type.

Due to aerospace applications related to shielding or waveguides, the specific EMI shielding effectiveness (EMI shielding effectiveness divided by the density) and the specific surface conductance (surface conductance divided by the density, where conductance is the reciprocal of impedance) are relevant quantities, which are listed in Table IV for the composite at the filler volume fraction that gives the highest such quantities in each of the four categories in Table II. Both specific quantities are highest for both the composite with 19 vol.% carbon filaments and that with 7 vol.% nickel filaments. Compared to all composites of Table IV, solid copper gave the lowest values for both specific quantities.

In summary, concerning the nickel filaments, 7 vol.% of these filaments provide a composite with shielding effectiveness 87 dB (compared to 90 dB for solid copper), surface impedance 1.2 Ω (same as for solid copper), ductility 1.0%, modulus 5 GPa, strength 52 MPa, density 1.87 g/cm^3, specific shielding effectiveness 47 dB.cm^3/g (compared to 10 dB.cm^{3}g for solid copper) and specific surface conductance 0.44 Ω^{-1}.cm^3/g (compared to 0.09 Ω^{-1}.cm^3/g for solid

Table III. Properties of the Composite with the Best Overall (Electrical and Mechanical) Performance for Each Filler Type

	EMI Shielding Effectiveness (dB)	Surface Impedance (Ω)	Strength (MPa)	Ductility (%)
7 vol.% Nickel filaments	86.6	1.22	51.5	1.01
19 vol.% Carbon filaments	73.9	1.48	27.2	0.67
19 vol.% 2 μm Nickel fibers	71.7	1.14	66.1	0.58
43 vol.% 20 μm Nickel fibers	73.7	1.40	34.6	0.30

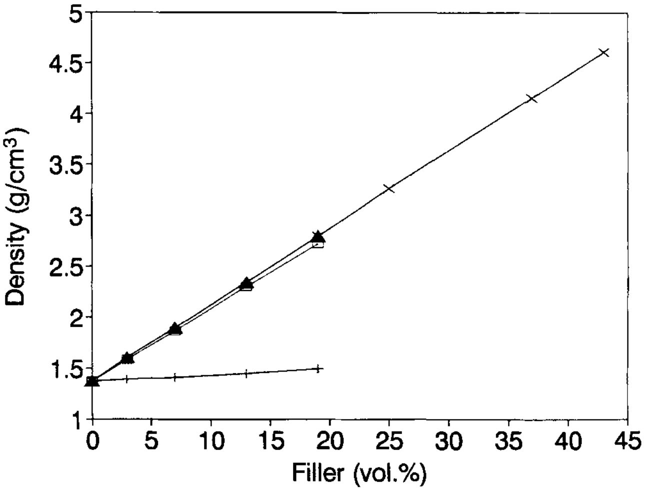

Fig. 10. Variation of density with filler volume fraction. +: carbon filaments; □: nickel filaments; ▲: 2 μm diameter Ni fibers; x: 20 μm diameter Ni fibers.

Table IV. Specific EMI Shielding Effectiveness and Specific Surface Conductance at 1–2 GHz for Competing Composites and Solid Copper

Material	Specific EMI Shielding Effectiveness (dB.cm^3/g)	Specific Surface Conductance (Ω^{-1}.cm^3/g)
7 vol.% Ni filaments	47 (3)	0.44 (0.04)
19 vol.% C filaments	50 (4)	0.45 (0.04)
7 vol.% 2 μm Ni fibers	31 (3)	0.39 (0.05)
43 vol.% 20 μm Ni fibers	16 (5)	0.15 (0.02)
Copper	10 (0.5)	0.09 (0.01)

Note: Standard deviations are shown in parentheses.

copper). In addition, the submicron diameter of the nickel filaments enables the fabrication of small size composites that are uniform in filler distribution. Composites of small size are desired for lightweight waveguides and miniaturized electronics.

The high shielding effectiveness and low surface impedance of the nickel filament composites at 1–2 GHz are due to the small diameter, large aspect ratio and high conductivity of the nickel filaments, and the fact that the interaction of electromagnetic radiation with a conductor weakens with increasing depth from the surface of the conductor.

Although carbon filaments provide composites with the same specific EMI shielding effectiveness and the same specific surface conductance as the nickel filaments, they provide composites with lower shielding effectiveness and higher surface impedance than nickel filaments at the same filler volume fraction.

Calculated values for the reflection loss R are less than the shielding effectiveness SE shown in Table II. For example, R is 15 dB, as calculated for the composite with 7 vol.% nickel filaments, whereas SE is 87 dB for this composite. Calculated values for the absorption loss A are much greater than the measured values. For example, A is 1244 dB, as calculated for the composite with 7 vol.% nickel filaments. These differences between measured and calculated values are attributed to leakage around the test specimen.

The skin depth δ (in meter) for an electromagnetic wave of frequency f in a medium of magnetic permeability μ ($\mu = \mu_r\mu_o$ where $\mu_o = 4\pi \times 10^{-7}$ H/m) and electrical conductivity σ (in S/m) is given by

$$\delta = \frac{1}{\sqrt{\pi f \mu \sigma}} \tag{6}$$

At the same frequency, the higher the electrical conductivity or the higher the magnetic permeability, the smaller the skin depth. For copper, $\mu_r = 1$ and $\sigma = 5.8 \times 10^7$ S/m, so the skin depth is 2.09 μm and 1.48 μm at frequencies of 1 and 2 GHz, respectively.

For nickel, if a standard value of $\mu_r = 100$ is used and $\sigma = 1.15 \times 10^7$ S/m, the skin depth is only 0.47 μm and 0.33 μm at frequencies of 1 and 2 GHz, respectively. These values of the skin depth are much smaller than those of copper due to nickel's ferromagnetic nature. This means that a smaller diameter nickel wire can absorb the same amount of microwave energy as the larger diameter copper wire. It also means that the submicron diameter nickel filaments will absorb microwave more efficiently than nickel fibers of diameter >1 μm. Consistent with the increase of the EMI shielding effectiveness with decreasing nickel filler diameter is the increase of the magnetic hysteresis energy loss with decreasing filler diameter.[4]

CONCLUSIONS

Nickel filaments of submicron diameter were found to give polymer-matrix composites that exhibit exceptionally high EMI shielding effectiveness and exceptionally low surface impedance at 1–2 GHz at filler volume fractions as low as 7%. The shielding effectiveness and surface impedance achieved with 7 vol.%

nickel filaments were respectively 87 dB and 1.2 Ω—both comparable to those of solid copper. The specific shielding effectiveness and specific surface conductance achieved with 7 vol.% nickel filaments were respectively 47 dB.cm^3/g and 0.44 Ω^{-1}.cm^3/g—both much higher than those of solid copper and comparable to those of a composite with 19 vol.% carbon filaments. Due to the low filler volume fraction, the tensile ductility and strength of the composite with 7 vol.% nickel filaments were quite high (1.0% and 52 MPa, respectively); the modulus was 5 GPa.

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