

Mechanical and Dielectric Properties of Polymer Composites Containing Tubules

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ABSTRACT: The thermal, dynamic mechanical, and dielectric properties of copper-coated lipid tubules incorporated into three polyurethane matrices with varying surface tensions have been examined. The tubules did not affect the glass-transition temperature of the polymer matrices, indicating that the tubule-polymer interactions may not be strong enough to restrict the mobility of polymer chains near the filler surface. The composite's elastic modulus can also be adequately modeled using the Nielsen equation. In addition, the real part of the permittivity for the composites increased monotonically over the tubule concentration

range. All samples had a small imaginary part of the permittivity, indicating the tubule concentrations were below the percolation threshold concentration. Also, the three types of matrices had comparable permittivity values at each tubule concentration, suggesting the polymer surface tension did not affect the tubule distribution in the composite. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 88: 3218–3224, 2003

Key words: tubule composite; lipid tubules; surface tension; dielectric properties

INTRODUCTION

Lipid tubules with metal coatings have previously been incorporated into polymer matrices to produce composites with a wide range of dielectric properties.^{1–3} In these studies, the authors have tried to manipulate the dielectric properties of the samples by modifying the tubule or the tubule orientation with respect to an applied electric field. In one study, the tubules were coated with permalloy, a nickel-iron-boron alloy, and added to an epoxy matrix.¹ The tubules were then aligned with a magnetic field before the epoxy gelled to produce an anisotropic material. In another study, different samples with nickel-coated tubules of varying lengths were fabricated using an epoxy matrix.² The authors then compared the dielectric properties of these composites. The tubules have also been coated with copper and incorporated into a vinyl polymer matrix to form isotropically orientated samples.³ As the tubule loading increased, the composite eventually achieved electrical percolation at the percolation threshold concentration. At this point, the tubules form a sample-spanning network in the system.

In those previous studies on tubule composites, the authors did not examine how the polymer matrix may affect the tubule distribution and the resultant

changes in dielectric properties. Dielectric properties have been shown to vary widely and become very sensitive to filler loading^{3–5} near the percolation threshold concentration. Studies involving other composite systems have shown dramatic effects of the polymer matrix on tubule distributions. For systems containing carbon black, the surface tension of the polymer matrix had a large effect on the percolation threshold concentration.^{6,7} Specifically, polymer matrices with higher surface tensions achieved larger percolation threshold concentrations. For example, matrices with surface tensions ranging from 30 to 46 dyn cm⁻¹ percolated at filler loadings ranging from 0.04 to 0.27 volume fraction.⁶ The authors of these studies proposed a model relating the percolation threshold concentration to the surface tension of the polymer, the surface tension of the filler, and the dimension of the filler.⁶ Subsequently, the effect of polymer viscosity was incorporated into the model.^{7,8} Another group also proposed a model relating the percolation threshold concentration to the surface tensions of the polymer and filler.^{8,9}

In addition to the paucity of studies involving polymer matrix effects on tubule systems, there is a lack of studies examining the effects of the tubules on the thermal and mechanical properties of the composite. Fillers have been shown to increase the glass-transition temperature of some composite systems.¹⁰ This is due to the reduced mobility of the polymer chains adsorbed onto the filler surface. In addition, mechanical properties are required to engineer composite systems containing the tubules. These results can be fit to

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a model and perhaps be used to predict other mechanical properties.

In this study, we characterize the thermal, dynamic mechanical, and dielectric properties of three tubule composite systems with varying surface tensions. We examine the effects of tubules on the glass-transition temperature and modulus of the samples. We also study how the surface tension of the polymer matrix affects the tubule distribution and the consequent change in dielectric properties of the composites.

EXPERIMENTAL

Sample preparation

All polyurethane samples were prepared by the one-shot method in which all monomers were mixed at once and allowed to cure. Three types of polyurethane matrices were used in formulating the tubule composites. The first type contained poly(propylene glycol), a diol with a molecular weight of 425, trimethylolpropane propoxylate, a triol with a molecular weight of 308, and poly(propylene glycol), tolylene 2,4-diisocyanate terminated, a diisocyanate monomer with a molecular weight of 1,000. All three monomers were obtained from Sigma-Aldrich (St. Louis, MO) and the samples prepared from them are designated as Ppg. The diol and triol were dried in a vacuum oven at 60°C for 5 and 3 h, respectively, before use. These formulations contained a triol concentration of 25 mol % of the total hydroxyl groups and an isocyanate-to-hydroxyl (NCO:OH) ratio of 1.1. In addition to the monomers, a catalyst, dibutyltin dilaurate (Sigma-Aldrich), and a commercial antifoaming agent, AF-4 (BJB Enterprises, Tustin, CA), were added to the samples. The dibutyltin dilaurate concentration was 0.05 wt % of the monomers and the AF-4 concentration was 0.30 wt % of the monomers. The second type of polyurethane matrix is a fluoropolymer and is designated as Fluor. For these Fluor samples, a commercial fluoropolyol resin (FPWC-10, 21st Century Coatings, Virginia Beach, VA) replaces the poly(propylene glycol) used in the Ppg samples. The fluoropolyol resin contains 70.03 wt % fluoropolyol dissolved in a 50–50 mixture of *n*-butyl acetate and xylene. The fluoropolyol, meanwhile, has an equivalent weight of 375 and is a random copolymer containing epichlorohydrin, 1,3-bis(hexafluor-2-hydroxy-2-propyl) benzene and 2,2,3,3,4,4-hexafluoropentane-1,5-diol. In formulating the fluoropolymer, the FPWC-10 was treated as a diol. The Fluor samples then have a triol concentration of 25 mol % of the total hydroxyl groups and an NCO:OH ratio of 1.1. AF-4 was added at a concentration of 0.50 wt % of the monomers. No dibutyltin dilaurate was added to the formulation. Finally, the third polyurethane matrix contains equimolar amounts of FPWC-10 and poly(propylene glycol) and is desig-

nated as Ppgfluor. This formulation also has a triol concentration of 25 mol % of the total hydroxyl groups and an NCO:OH ratio of 1.1. Both AF-4 and dibutyltin dilaurate were added to the formulation at concentrations of 0.50 and 0.01 wt % of the monomers, respectively.

The lipid tubules and the copper coating process have been described extensively in previous publications.^{11–14} In short, the tubules were formed by first dissolving a phospholipid, 1,2 bis(tricoso-10,12-dinoyl)-sn-glycero-3-phosphocoline, in an ethanol–water solution. The solution is then slowly cooled through the phospholipid's melting temperature of 40°C. Tubules begin to form and can be subsequently coated with a metal plating catalyst. The tubules were then coated with copper by using a commercial electroless plating process. The copper-coated tubules have an average diameter of 1 μm and are 20–30 μm long.

All tubule composites were generally prepared the same way. The monomers, AF-4, and dibutyltin dilaurate were mixed vigorously by hand for 5 min. The tubules were then added to the mixture and mixed for another 10 min. The tubule concentrations in the composites were varied from 6 to 14 vol % of the monomers. The samples were then poured into polystyrene weighing dishes and placed in a vacuum oven for degassing. The Ppg samples were degassed for 1 h, the Ppgfluor samples for 20 min, and the Fluor samples for 35 min. After degassing, the samples were placed in a chamber containing a nitrogen atmosphere and allowed to cure at room temperature for 48 h. The samples were then placed back in the vacuum oven at 60°C and cured under vacuum for an additional 24 h.

Differential scanning calorimetry

A differential scanning calorimeter (DSC 7, Perkin-Elmer, Norwalk, CT) was used to characterize the thermal properties of the samples. Each sample, weighing approximately 15 mg, was scanned at a rate of 10°C min⁻¹ from -60°C to 100°C. The samples remained in a nitrogen atmosphere during each temperature scan. Two scans were performed for each sample, with the second scan used for the data analysis.

Dynamic mechanical analysis

A dynamic mechanical analyzer (DMA 2980, TA Instruments, New Castle, DE) was used to determine the mechanical properties of the samples. The sample, approximately 10 mm wide and 3 mm thick, was placed in a 35 mm long dual-cantilever apparatus. The sample was heated at a rate of 2°C min⁻¹ from -100°C to 100°C in a nitrogen atmosphere. The frequency of oscillation for each experiment was maintained at 1

Hz. The dynamic mechanical properties determined using this setup include E' , the elastic modulus, and E'' , the viscous modulus. The elastic modulus measures the amount of energy stored per oscillation cycle, while the viscous modulus measures the amount of energy dissipated per cycle.

Contact angle measurements and surface tension calculations

Surface tension values of the neat polymers and copper foil were determined by performing contact angle measurements with a video contact angle system (VCA 2500, AST Products, Billerica, MA) using three test liquids. The copper foil (Pirelli Cables, Lexington, SC) was used as a model for the surface of the copper-coated tubules. The test liquids include sessile drops of triple-distilled water (2 μ l), formamide (2 μ l), and methylene iodide (1 μ l). The liquid was initially placed on the sample surface and a picture of the drop was taken after 10 s. The contact angle was then determined by using the product software. Both the formamide and methylene iodide were obtained from Sigma-Aldrich. Once the contact angles were measured, the surface tension of the polymers and copper foil can be determined by using the harmonic mean method. The total surface tension includes dispersion and polar components, which can be calculated by using the following equation¹⁵:

$$\gamma_l(1 + \cos\theta) = 2\left(\frac{\gamma_l^d \gamma_s^d}{(\gamma_l^d + \gamma_s^d)} + \frac{\gamma_l^p \gamma_s^p}{(\gamma_l^p + \gamma_s^p)}\right) \quad (1)$$

where γ_l is the test liquid's total surface tension, θ is the contact angle, γ_l^d is the test liquid's dispersion component of the total surface tension, γ_s^d is the sample's dispersion component of the total surface tension, γ_l^p is the test liquid's polar component of the total surface tension, and γ_s^p is the sample's polar component of the total surface tension. By using two test liquids with known surface tensions, the surface tension components of the sample can be calculated from eq. (1).

Contact angles of the Ppg, Ppgfluor, and Fluor monomers on the copper foil were also measured by using the same video contact angle system. However, a picture of the sessile drop was taken only after waiting 10 min. There was a longer wait because the higher viscous monomers required a longer time to spread over the surface.

Dielectric measurements

A vector network analyzer (HP8510, Hewlett-Packard, Palo Alto, CA) was used to measure the dielectric response of the samples. Each sample, approximately

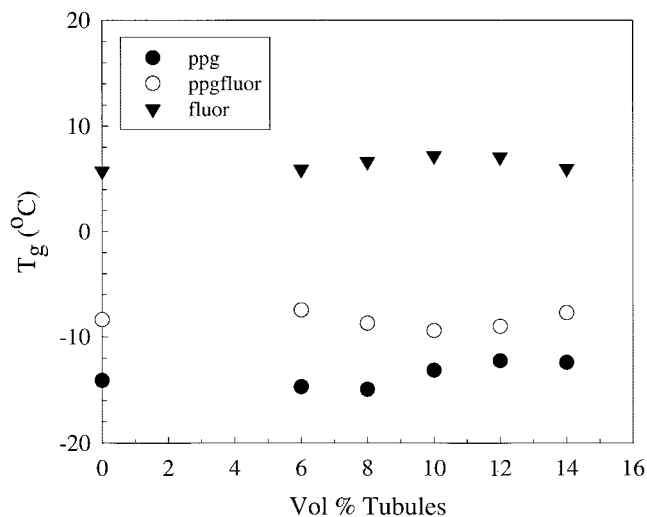


Figure 1 Glass-transition temperature of Ppg, Ppgfluor, and Fluor samples as a function of tubule concentration. The standard deviations are approximately 1°C.

1.3 mm thick and 7 mm in diameter, was placed in a coaxial measurement fixture. Their S-parameters were obtained over a frequency range of 2–18 GHz. The Nicolson and Ross approach was then used to calculate the permittivity values from the S-parameter data.

RESULTS AND DISCUSSION

Effects of tubules on thermal and mechanical properties of composites

The calorimetry results indicate that over the tubule concentration studied, the tubules did not affect the glass-transition temperature of the composite. This is shown in Figure 1, where we plot glass-transition temperature as a function of tubule concentration for all samples. All the Ppg samples had glass-transition temperatures of about -14°C , whereas the Fluor samples had glass-transition temperatures of about 6°C . The Ppgfluor samples had glass-transition temperatures of -8°C , between those of the Ppg and Fluor samples. It should be noted that all samples had one glass-transition temperature and no other peaks associated with melting of a crystalline phase (data not shown). This indicates that all three polymers are amorphous. Previous studies have shown that fillers sometimes increase the glass-transition temperature in polymer composites.^{10,16,17} This is ascribed to polymer chains adsorbing onto the filler surface, thereby restricting their mobility. This reduced mobility results in an increase in the glass-transition temperature. The magnitude of the glass-transition temperature shift can depend on the strength of the polymer–filler interactions, with greater interactions resulting in larger shifts.¹⁷ In some cases, a second glass-transition temperature appears instead of the temperature shift.¹⁸

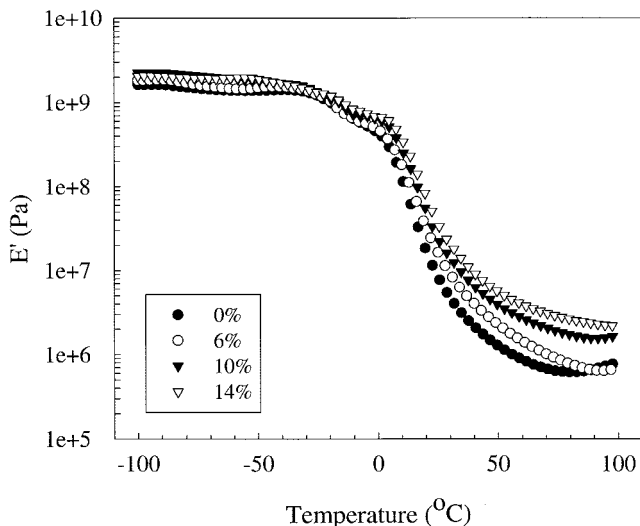


Figure 2 Elastic modulus (E') of the Ppg samples as a function of temperature.

Our data indicate that the tubule–polymer interactions are not strong enough to affect the glass-transition temperature of the composites.

The addition of tubule fillers increases the elastic modulus of the composites over the entire temperature range studied. This is shown in Figure 2, where we plot the elastic modulus of the Ppg samples as a function of temperature. The elastic moduli of the Fluor and Ppgfluor samples exhibit similar behavior so we do not present the data. For temperatures below the glass-transition temperature, the elastic modulus increases only moderately for higher tubule concentrations. As the temperature becomes higher than the glass-transition temperature, the samples containing higher tubule concentrations have much greater moduli than the neat polymer matrix. The main reason for this behavior is that the ratio of the tubule modulus to the neat matrix modulus becomes larger in the rubbery region than in the glassy region.¹⁰ Consequently, the composite should exhibit a larger modulus relative to the neat matrix modulus above the glass-transition region than below it.

The viscous modulus displays the same general behavior as the elastic modulus for increasing tubule concentrations. This is shown in Figure 3, where we plot the viscous modulus of the Ppg samples for various tubule concentrations as a function of temperature. The tubules seem to have a greater effect on the viscous modulus above the glass-transition temperature than below it. The viscous modulus peak has sometimes been used as a measure of the glass-transition temperature. From Figure 3, this peak occurs at the same temperature for all samples, indicating the tubules did not affect the glass-transition temperature of the composites. This supports the results obtained from DSC, which showed that the tubules had no effect on the glass-transition temperature (Fig. 1).

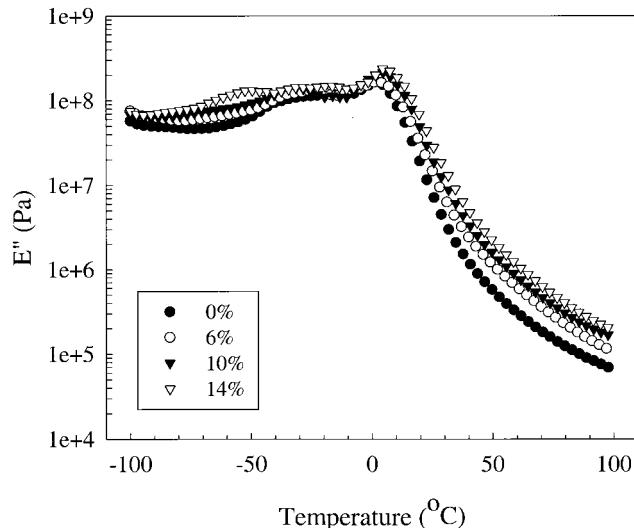


Figure 3 Viscous modulus (E'') of the Ppg samples as a function of temperature.

The $\tan\delta$ results also suggest that the tubules did not affect the glass-transition temperature of the composites over the tubule concentrations studied. Figure 4 shows the $\tan\delta$ results of the Ppg samples for various tubule concentrations as a function of temperature. The $\tan\delta$ peaks, which are commonly used as a measure of the glass-transition temperature, occur at one temperature for all samples. This result corroborates the DSC and viscous modulus data. However, unlike the viscous modulus values, the $\tan\delta$ peaks decrease at higher tubule concentrations. This can be explained by approximating the $\tan\delta$ of the composite as¹⁰

$$\tan\delta = (1 - \phi_f) \tan\delta_m + \phi_f \tan\delta_f \quad (2)$$

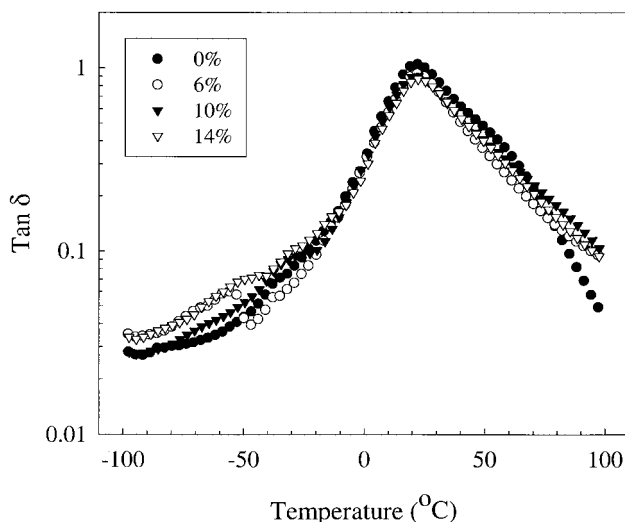


Figure 4 Loss tangent of the Ppg samples as a function of temperature.

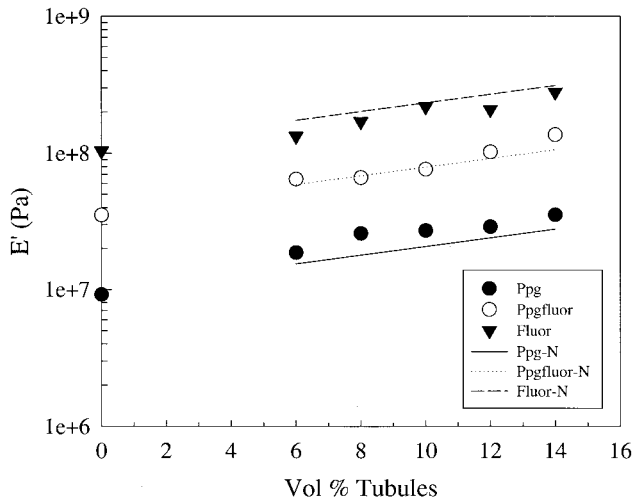


Figure 5 Comparison of the elastic moduli from dynamic mechanical analysis to those predicted from the Nielsen model. Symbols represent the experimental data and lines represent the model predictions.

where $\tan\delta$ is the loss tangent of the composite, ϕ_f is the volume fraction of the filler, $\tan\delta_m$ is the loss tangent of the matrix, and $\tan\delta_f$ is the loss tangent of the filler. Since the $\tan\delta_f$ value is very low compared to that of $\tan\delta_m$, it can be neglected. Consequently, as the tubule loading increases, the composite loss tangent should decrease in value.

The elastic modulus of the composites can be modeled by using equations developed by Nielsen.^{10,19} These general equations are modifications of the empirical equations formulated by Halpin and Tsai. The elastic modulus of the composite, E' , can be calculated from^{10,19}

$$\frac{E'}{E'_m} = \frac{1 + AB\phi_f}{1 - B\psi\phi_f} \quad (3)$$

where E'_m is the elastic modulus of the matrix, A is a parameter that takes into account the geometry of the filler phase and the Poisson's ratio of the matrix, B is a parameter that takes into account the relative moduli of the filler and matrix, ϕ_f is the filler volume fraction, and ψ is a parameter that depends on the maximum packing fraction of the filler. The three parameters A , B , and ψ can be determined from^{10,19}

$$A = K_E - 1 \quad (4)$$

$$B = \frac{\frac{E'_f}{E'_m} - 1}{\frac{E'_f}{E'_m} + A} \quad (5)$$

$$\psi = 1 + \left(\frac{1 + \phi_{max}}{\phi_{max}^2} \right) \phi_f \quad (6)$$

where K_E is the generalized Einstein coefficient, E'_f is the elastic modulus of the filler, and ϕ_{max} is the maximum packing fraction of the filler. By assuming the polymer matrices have a Poisson ratio of 0.5, we obtain a value of $K_E = 10$ for the tubules.¹⁰ If we assume the tubule can be treated as pure copper, its elastic modulus is then 1.3×10^{11} Pa.²⁰ In addition, the maximum packing fraction is approximately 0.52 for the tubules.¹⁰ Substituting these values into eqs. (3)–(6), we can compare the experimentally determined elastic modulus from dynamic mechanical analysis with the elastic modulus predicted from the Nielsen equations. This is shown in Figure 5, where we plot the elastic modulus of all samples at 25°C as a function of tubule concentration. For the three polymer matrices, the fit to experimental data is fairly good.

Dielectric properties of tubule composites

The real (ϵ') and imaginary (ϵ'') parts of the permittivity for all samples remain relatively constant over the frequency range examined in this study. This is shown in Figure 6 for a typical Ppgfluor sample containing 10 vol % tubules. The real part of the permittivity has a value of 12, whereas the imaginary part of the permittivity has a value of 0.6 over the frequency range. Since the permittivity values are essentially constant, we can choose the permittivities at a frequency of 10 GHz as the basis for comparison between different samples.

All the samples have tubule loadings that remained below the percolation threshold concentration. This is indicated by the very small values of the imaginary part of the permittivity for all samples. A previous study involving tubule composites has shown how

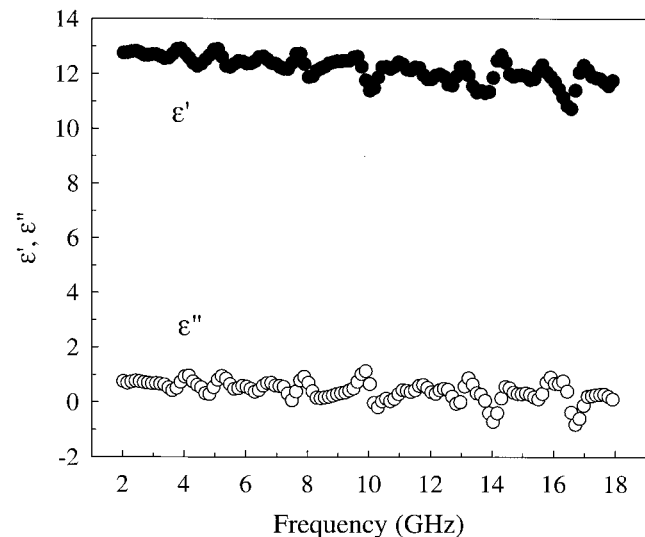


Figure 6 Real and imaginary parts of the permittivity of a representative Ppgfluor sample containing 10 vol % tubule as a function of frequency.

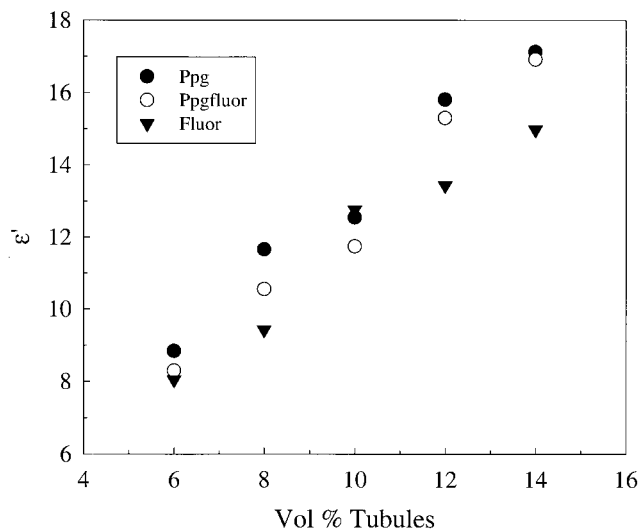


Figure 7 The real part of the permittivity of Ppg, Ppgfluor, and Fluor samples as a function of tubule concentration. The frequency is 10 GHz.

dielectric properties change in percolating systems. Browning et al.³ showed that the real part of the permittivity reached its maximum value and the imaginary part of the permittivity increased rapidly in the vicinity of percolation. This behavior of the permittivities has been predicted by the effective-medium theory for conducting stick composites.⁵ The theory applies to stick-shaped fillers with high aspect ratios (length of stick/diameter of stick), akin to the tubules used in this study.

The real parts of the permittivity for the three different matrices increased monotonically over the tubule concentration range. This is shown in Figure 7, where we plot the real part of the permittivity for all the samples as a function of tubule concentration. The permittivity generally increases by two units for every incremental increase of 2 vol % tubules. The Ppg samples seem to have larger permittivity values, whereas the Fluor samples have the lowest permittivity values. However, the sample standard deviations in these measurements are rather large compared to the differences in permittivity values between different samples. Indeed, the standard deviations range from 10% to 20% of the permittivity values. Consequently, we conclude that at each concentration, the three matrix types have comparable permittivity values.

The three polymer matrices in this study have different surface tension values, but this apparently did not affect their dielectric properties. This can be seen in Figure 7, where the three polymer systems have comparable permittivity values at each tubule concentration. Table I contains the surface tension values of the neat polymer matrices. The Ppg sample has the largest surface tension, whereas the Ppgfluor and Fluor samples have comparable surface tensions. We

TABLE I
Surface Tensions of Polymers and Copper

	Dispersion component (mJ m ⁻²)	Polar component (mJ m ⁻²)	Total (mJ m ⁻²)
Ppg	35.7	5.2	40.9
Ppgfluor	21.5	10.6	32.1
Fluor	17.3	15.6	32.9
Copper	38.5	2.9	41.4

also measured the surface tension of a copper foil, which serves to mimic the properties of the copper-coated tubules. This value is also listed in Table I.

A more relevant parameter that quantifies polymer-filler interactions is the polymer-filler interfacial tension. We can calculate this interfacial tension from surface tension values by using the harmonic mean method¹⁵:

$$\gamma_{12} = \gamma_1 + \gamma_2 - 4 \left[\frac{\gamma_1^d \gamma_2^d}{\gamma_1^d + \gamma_2^d} + \frac{\gamma_1^p \gamma_2^p}{\gamma_1^p + \gamma_2^p} \right] \quad (7)$$

where γ_{12} is the interfacial tension between species 1 and 2, γ_1 is the total surface tension of species 1, γ_2 is the total surface tension of species 2, γ_1^d is the dispersion component of species 1, γ_2^d is the dispersion component of species 2, γ_1^p is the polar component of species 1, and γ_2^p is the polar component of species 2. Table II contains the interfacial tension values between the polymers and copper. The Ppg-Cu interface has the lowest interfacial tension value, whereas the Fluor-Cu interface has the highest value. The surface and interfacial tension values in Tables I and II derive from measurements on fully cured samples. In order to determine how the monomers interact with copper before curing occurs, we determined their contact angles with respect to copper right after mixing the monomers. The results are shown in Table II. The Ppg sample has the lowest contact angle on copper, whereas the Fluor sample has the highest angle. The contact angle data are consistent with the interfacial tension data. A low contact angle or low interfacial tension favors interactions between the monomer or polymer and copper, resulting in more wetting of the copper by the monomer or polymer. Consequently, the tubules become more dispersed and the samples

TABLE II
Interfacial Tensions and Contact Angles of Polymers with Copper

	Interfacial tension (mJ m ⁻²)	Contact angle (°)
Ppg-Cu	0.8	21 ± 3
Ppgfluor-Cu	9.2	25 ± 2
Fluor-Cu	16.8	41 ± 4

have a higher percolation threshold concentration. On the other hand, a high contact angle or high interfacial tension implies a less favorable interaction between monomer or polymer and copper. This results in closer contact between tubules and samples having a lower threshold concentration.

Our dielectric data (Fig. 7) do not seem to be consistent with the interfacial tension data. Based on the interfacial tension values, the Ppg samples should have the lowest permittivity values because the tubules should be most dispersed in the system. This is not the case since all three types of polymer composites have comparable permittivity values at each tubule concentration. This suggests that the interfacial tension does not have an effect on the tubule distribution for our samples. Wu et al.²¹ have also found that interfacial tension between polymer and filler may not be the primary predictor of how fillers distribute in the composite. They studied carbon black in immiscible polymer blends and discovered that the filler preferentially disperses in the polymer with the lower glass-transition temperature rather than the lower interfacial tension. We should note, however, that the maximum tubule concentration examined in this study is 14 vol %. It may well be that the interfacial tension only has an effect at higher concentrations.

CONCLUSIONS

The tubules did not affect the glass-transition temperatures of the three polymer matrices. This may indicate that the tubule-polymer interactions are not strong enough to restrict the mobility of the polymer chains near the filler surface. In addition, the Nielsen model fits the dynamic mechanical data well for all the samples. The real part of the permittivity increased monotonically as tubule concentration increased, whereas the imaginary part of the permittivity remained small. This indicates that all the samples had tubule loadings below the percolation threshold concentration. Although the three polymer matrices have different surface and interfacial tensions, they have

comparable values of the real part of the permittivity at each tubule concentration. This implies the tubules have similar distributions in each matrix and this distribution does not have a clear dependence on the polymer surface tension or polymer-tubule interfacial tension.

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