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On the Influence of Moisture on Dielectric Properties of Polyetheretherketone (PEEK) Carbon-Fiber Composites

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Abstract

An analysis of local and global mechanisms of heat generation and distribution in carbon-fiber-based composites subjected to an alternating magnetic field has shown that heating is dependent upon the dielectric properties of the polymer matrix. These properties were investigated as functions of temperature, frequency, and moisture content. The results indicate little dependence of the dielectric constant on temperature or frequency, while the loss tangent exhibited a substantial dependence on both frequency and temperature. A substantial dependence of loss tangent on moisture content in polyetheretherketone (PEEK) was found.

Table of Contents

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List of Figures

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List of Tables

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1. Introduction

An investigation of induction heating in carbon-fiber-based composites [1] revealed that the heating resulting from an applied alternating magnetic field is due to dielectric losses in the polymer material that separates crossing fibers in a cross-ply or angle-ply laminate, as long as dielectric breakdown does not occur. The analysis included therein assumed that constant values of the fundamental dielectric properties were known. Heating in a dielectric material subjected to an alternating electric field was shown to be linearly dependent upon these material properties—dielectric constant and dissipation factor (or loss tangent). A global model has been developed [2] that established the electromagnetic response of cross-ply laminated composites to alternating magnetic fields. Three fundamentally separate submodels that consider the in-plane response, the through-thickness response, and the global generation of heat and its quantification as the surface temperature profile were developed. One of the necessary inputs to these models is the complex dielectric constant of the polyetheretherketone (PEEK) polymer. PEEK is the matrix material used in the composite prepreg product APC-2 used in the experimental work and theoretical analysis described by Fink et al. [1].

In a preliminary study, a carbon-fiber-reinforced PEEK cross-ply ($[0/90]_S$) laminate was characterized at various drying times. Ten thermocouples were used to measure the transient temperature profile on the surface of the flat laminated plate, which was heated by magnetic induction using a 10.2-cm-diameter (4 in) Helmholtz coil centered on a 10.2-cm-square (8 in) specimen. The magnetic field was applied to the specimen in five different states of drying: (1) "as-is" (ambient moisture content), (2) 24 hr, (3) 48 hr, (4) 72 hr, and (5) 96 hr at 120 °C and 28-inHg vacuum. Since the same specimen was used for all tests, the drying times were started over after each previous test. Despite PEEK's low reported moisture content of 0.2 to 0.5% [3–5], the average equilibrium temperature exhibited significant decreases with increased drying time, as shown in Figure 1. This apparent dependence of heat production on small changes in moisture content motivated a closer examination of the effects of moisture content on the properties of the material that affect heating under an applied alternating magnetic field.

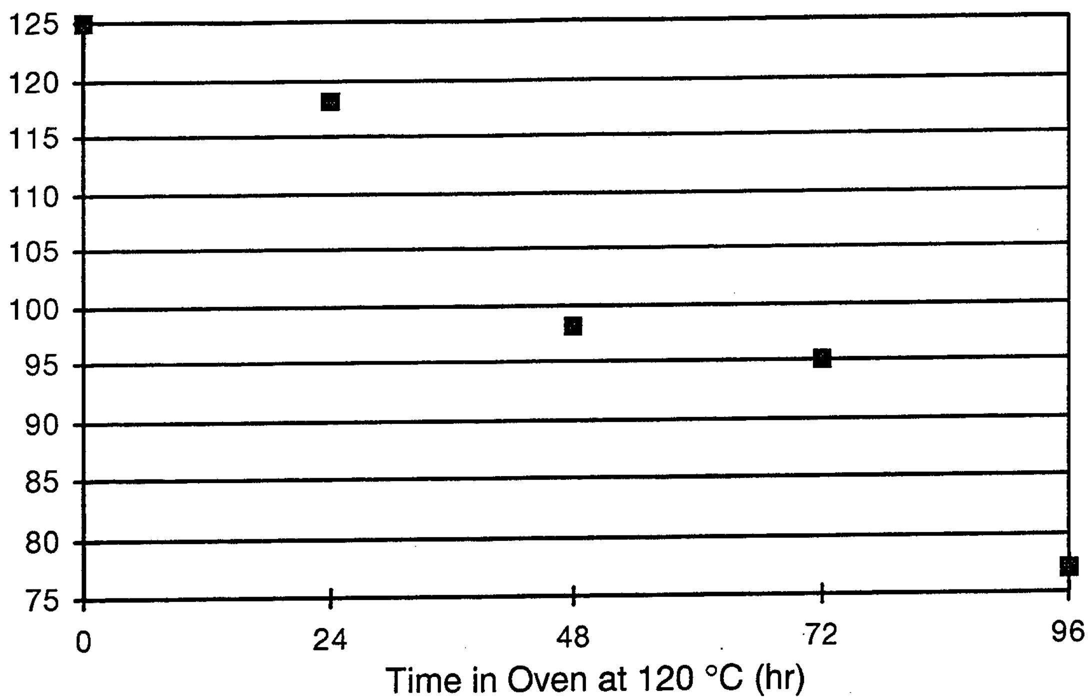

Figure 1. Results of Dried-Laminate Induction-Heating Test.

An equivalent form of an equation for heat generation in our induction heating model [1, 2] can be written as

$$W_j = \frac{\beta_j V_j^2}{h}, \tag{1}$$

where W_j is the heat generation at some node j in the plane of the laminate; V_j is the potential difference between the fibers at node j; h is the distance through which the electric field created by V acts; and β_j is a function of several material, environmental and microstructural properties at node j.

$$\beta_j = \omega \varepsilon_0 \kappa \tan\delta, \tag{2}$$

where ω is the angular frequency, ε_0 is the permittivity of vacuum, κ is the relative dielectric constant of the polymer, and $\tan\delta$ represents the imaginary part of the complex dielectric constant of the polymer. Of the components of equations (1) and (2), only the real and imaginary parts of the complex dielectric constant, κ and $\tan\delta$, are potentially functions of moisture content in the composite's matrix material.

In general, the complex dielectric constant can be significantly affected by the frequency, temperature, and moisture content, as well as by the presence of mobile ions in polymeric systems [6]. Values for these properties are typically reported in the literature at room temperature and line frequency without mention of moisture content or the presence of other ionic species. Reported values are not always applicable at the frequencies often used in induction heating of PEEK-based composites (10^3 to 10^6 Hz range) and at temperatures ranging from room temperature to beyond the melting temperature of PEEK (>340 °C).

Compilations of dielectric constants and dissipation factors [7, 8] have been published for various polymers but are difficult to keep current. Manufacturers often supply data for specific materials at specific temperatures and frequencies; however, it is often necessary to characterize these properties for the particular process parameters of interest. This paper discusses the relation of structure to dielectric properties, describes the methods of experimental measurement performed, and reports the results with respect to temperature, frequency, and moisture content for PEEK.

2. Review of Dielectric Properties

Polymeric materials can be divided into two groups: (1) nonpolar and (2) polar. Nonpolar materials exhibit high resistivity, a low dielectric constant, and a negligible loss tangent; they are, for the most part, unaffected by temperature, frequency, and humidity. Polar polymers generally have lower resistivities and higher dielectric constants and loss tangents.

The dielectric constant (unit capacitance, permittivity) and dissipation factor are very different for nonpolar and polar materials as functions of temperature and frequency. The values of both properties for nonpolar materials are nearly constant over a wide frequency range and for temperatures below the softening point. For polar materials (often called lossy materials), the dielectric constant decreases with an increase in frequency, whereas the dissipation factor increases and decreases cyclically.

Under the influence of an alternating electric field, electric dipoles are oriented in the material such that the magnitude of the induced charges increases as the polarization of the dielectric increases. There are several possible mechanisms of polarization in a dielectric material:

(1) Electric Polarization—a shift in the center of charge of the electron cloud relative to the positive center of charge on the nucleus when an electric field is applied.

(2) Ionic Polarization—displacement of positive and negative ions in relation with one another in respect to their equilibrium positions.

(3) Orientation Polarization—the alignment of permanent dipoles that exist in a material in the absence of an applied field.

(4) Space Charge Polarization—the alignment of mobile charges in the material whose movements are restricted by interfaces or other barriers in the material.

The total polarization can be expressed as the sum of these four mechanisms; however, the contribution of each source is dependent on frequency, temperature, and applied voltage. Of the four mechanisms of polarization, orientation polarization has the greatest effect on the total dissipation of energy at the frequencies considered in this work (at least for a "clean" polymer that is free of excessive foreign ionic species).

Frequency effects on polarization are related to the time dependency of the polarization mechanisms with respect to their response to an applied field. For example, electronic polarization would be expected to occur rapidly, while permanent dipole orientation would be slow in comparison. This is usually explained by the difference in the mass of the electrons vs. the molecules and also on the number of conformations and free volume available for motion in polymers. In thermoplastics, orientation polarization relates to the conformational motion of polymeric chains and ionic polarization to the existence of ionic species or other groups either left over from the processing of the polymer or, possibly, deposited by treated fibers during resin impregnation in composite materials.

Molecular movements take a finite time, and complete orientation as induced by an alternating current may or may not be possible, depending on the frequency of the alternating electric field. Thus, at zero frequency, the dielectric constant will be at a maximum and will remain approximately constant until the dipole orientation time is of the same order as the reciprocal of the frequency. Dipole movement will now be limited, and the dipole polarization effect and the dielectric constant will be reduced. As the frequency further increases, the dipole polarization effect will tend to zero and the dielectric constant will tend to be dependent only on the electronic polarization. When there are two dipole species differing in ease of orientation, there will be two points of inflection in the dielectric constant vs. frequency curve. The dielectric constant of unsymmetrical molecules containing dipoles (polar molecules) will be dependent on the internal viscosity of the dielectric.

Besides a dielectric constant dependence on temperature and frequency, polar molecules exhibit relatively high dielectric power losses at certain frequencies; the maximum power losses corresponding to the point of inflection in the dielectric constant vs. frequency curve (Figure 2). At very low frequencies, the dipole movements are able to keep in phase with changes in the electric field and power losses are low. As the frequency is increased, the point is reached when the dipole orientation cannot be completed in the time available and the dipole becomes out of phase. Measures of the fraction of energy absorbed per cycle by the dielectric from the field are the "power factor" and "dissipation factor." The delay between the changes in the field and the change in polarization leads to a current in a capacitor leading the voltage across it when a dielectric is present. The angle of lead is known as the phase angle and given the symbol θ. The value $90-\theta$ is known as the loss angle and is given the symbol δ. The power factor is defined as

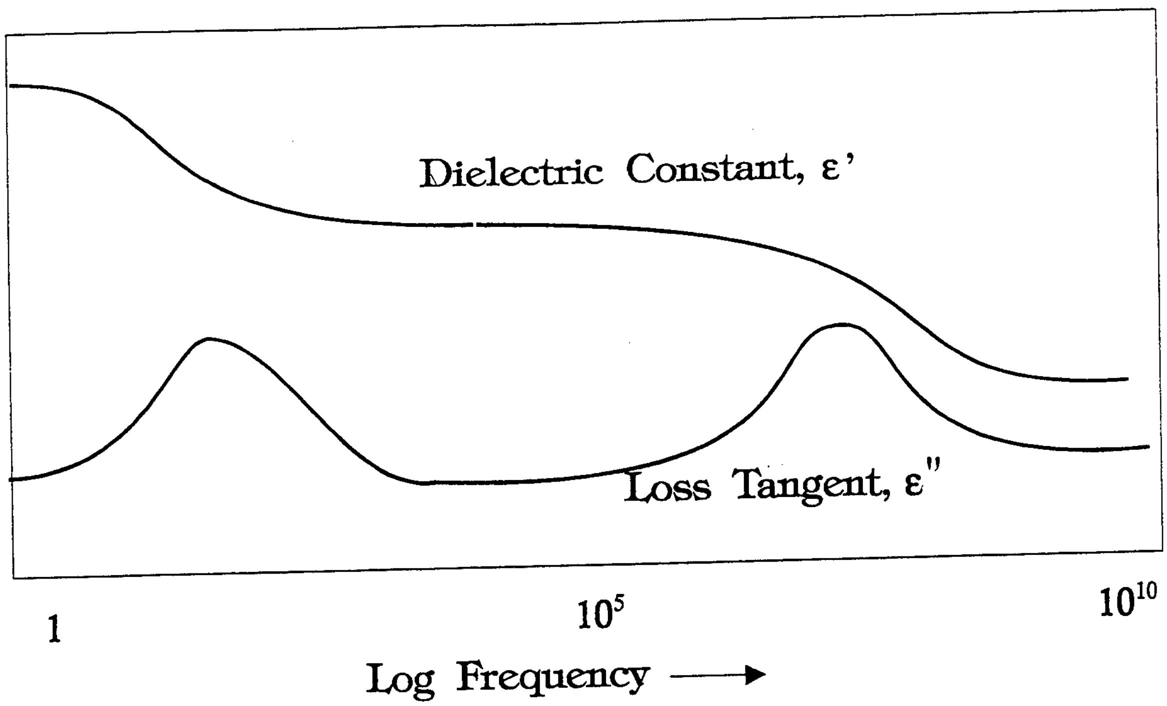

Figure 2. General Relationship Between Real and Imaginary Parts of Complex Dielectric Constant in Dielectric Materials.

cos(θ) and the dissipation factor as tan(δ). When δ is small, the two are equivalent. The "loss factor" is the product of the dissipation factor and the dielectric constant.

At low frequencies, when power losses are low, these values are also low, but they increase when such frequencies are reached that the dipoles cannot keep in phase. After passing through a peak at some characteristic frequency, they fall in value as the frequency further increases. This is because, at such high frequencies, there is no time for substantial dipole movement and the power losses are reduced. Because of the dependence of the dipole movement on the internal viscosity, the power factor, like the dielectric constant, is strongly dependent on temperature.

In polar polymers, the situation is more complex since there are many dipoles attached to one chain. Either these dipoles may be attached to the main chain, or the polar groups may not be directly attached to the main chain and the dipoles may, to some extent, rotate independently.

In the case with dipoles integral with the main chain, in the absence of an electric field, the dipoles will be randomly disposed but will be fixed by the disposition of the main chain atoms. On application of an electric field, complete dipole orientation is not possible because of spatial requirements imposed by the chain structure. Furthermore, in the polymeric system, the different molecules are coiled in different ways and the time for orientation will be dependent on the particular disposition. Thus, whereas simple polar molecules have a sharply defined power loss maxima, the power loss vs. frequency curve of polar polymers is broad, due to the dispersion of orientation times.

When dipoles are directly attached to the chain, their movement will obviously depend on the ability of chain segments to move. Thus, the dipole polarization effect will be less significant below the glass transition temperature than above it. PEEK is such a polymer; the offset oxygen on the ketone segment provides the polarizability of the polymer. It is expected that the loss tangent will have a significant increase directly after the glass transition temperature, which is about 143 °C.

Ionic polarizability is the inducement of dipoles by relative measurement of positive and negative ions in a partial ionic solid. Ionic polarization generally has a predominate influence on the loss tangent at higher frequencies than for dipole polarization. Since the imaginary part of the complex dielectric constant (or dielectric loss factor) is large whenever the frequency reaches the upper limit for any dielectric response mechanism, such as ionic polarization, a second peak in the loss tangent vs. frequency curve is expected at some frequency higher than the dipole polarization peak.

These are generalizations that may not be the case with thermoplastic polymers with embedded fiber reinforcement as in the PEEK/AS4 carbon-fiber system being investigated. The peaks for dipole and ionic polarization may fall within the same elevated frequency range and be difficult to distinguish. Therefore, dielectric properties may be very different for pure polymers and for polymers with fibrous reinforcement. Processing reagents and surface treatment sizings used on fibers to enhance fiber-polymer bonding can contribute ionic functional groups that would serve to increase the ionic polarizability of the polymeric regions.

Takahagi and Ishitani [9] attempted to characterize carbon-fiber surface chemistry. Their research showed a predominance of –OH and C=O functional groups, even without surface treatment. If the loss tangents of any associated molecules caused peaks at or near the same frequency as the dipole polarization inherent in the PEEK polymer, an effective increase in the loss tangent at that frequency would be seen.

Measuring this expected difference in dielectric properties between the neat PEEK polymer and the PEEK polymer with fiber reinforcement is not possible without first removing the fibers from the polymer due to the extreme difference in electrical properties between the polymer and the conductive carbon fibers. Our study of induction heating includes only the dielectric data obtained for virgin PEEK.

Both the dielectric constant and the dissipation factor are related to the presence of moisture. Moisture, absorbed in a filler at interfaces between different components or in laminations, can often be detected by changes in dissipation factor and capacitance [6]. Water molecules are highly polar and highly mobile. At frequencies of 10^5–10^6 Hz, the losses in water are ionic and increase with increases in dissolved ionizable salts. Hartshorn et al. [10] provide loss tangent vs. frequency data for several polymers in wet and dry states. The wet specimens, as a rule, gave higher loss tangent data at all frequencies. Potthoff [11] showed that increasing the temperature also increased the total effect of the presence of moisture in the system.

Moisture content studies on 150G PEEK [3] and PEEK and polyetherimide (PEI)-based composites [4, 5] indicate relatively low moisture contents in these advanced thermoplastic systems; saturated moisture absorption in PEEK-based APC-2 averaged about 0.2 weight-percent at room temperature and 100% relative humidity. Comparatively, the amorphous PES-based RADEL-C/T300 absorbed about 2.5% at equilibrium. The lower moisture absorption of PEEK is due to its semi-crystalline nature while the other resins (PEI and PES) are amorphous.

Buchman and Isayev [5] reported an equilibrium moisture content of about 0.6% for PEI-based CYPAC 7005/G30-500, while Demonet [4] reported 0.56%. The increase in moisture content from PEEK to PEI to PES may correspond to increases in loss tangent.

The next two sections describe the methods used to determine dielectric properties of PEEK over applicable ranges of frequency, temperature, and moisture pertinent to the present study and the results of those tests.

3. Characterization

The real and imaginary parts of the complex dielectric constant were measured at frequencies of 10^3, 10^4, 10^5, and 10^6 Hz using a cell built for similar characterization of filled polymers by Sturman [12, 13]. The physics of the cell and the specimen preparation is described by Sturman [12].

Specimens were cut from a semi-crystalline PEEK plate; the diameter of the machined disks was 4.01 cm and the thickness was 0.32 cm. A metal sleeve connected to a variac power supply surrounded the specimen. The power was incrementally increased and held for 15 min as current caused joule heating in the sleeve which conductively transmitted the energy to the polymer specimen. A thermocouple placed on the inside of the sleeve recorded the equilibrium temperature. Fifteen minutes between cycles allowed the aluminum sleeve, the cell electrodes, and the specimen to reach thermal equilibrium with the temperature increasing in increments of 25 °C from ambient to 250 °C.

The first set of measurements was performed at room temperature and at full moisture content (no drying). Results from three measurements were averaged, tabulated in Table 1 and shown in Figures 3 through 5 for the dielectric constant, loss tangent and resistivity versus frequency respectively. Figure 3 shows that the dielectric constant drops off rapidly between 100 kHz and 1 MHz. Figure 4 shows the expected increase in loss tangent with frequency in this

Table 1. Averaged Data From Three Electrical Property Measurements on PEEK at Room Temperature

Frequency (kHz)	Capacitance (pF)	Dielectric Constant	Loss Tangent	Conductance (1/ohm)	Resistance (ohm)	Resistivity (ohm-cm)
1	13.31	3.744	0.0014	1.00E − 10	1.00E + 10	4.02E + 11
10	13.282	3.736	0.0034	2.90E − 09	3.45E + 08	1.39E + 10
100	13.209	3.716	0.0160	1.33E − 07	7.52E + 06	3.02E + 08
1,000	12.392	3.486	0.0421	7.12E − 06	1.40E + 05	5.63E + 06

Note: The capacitance in vacuo is 3.555 pF for a surface area to diameter ratio of 0.402.

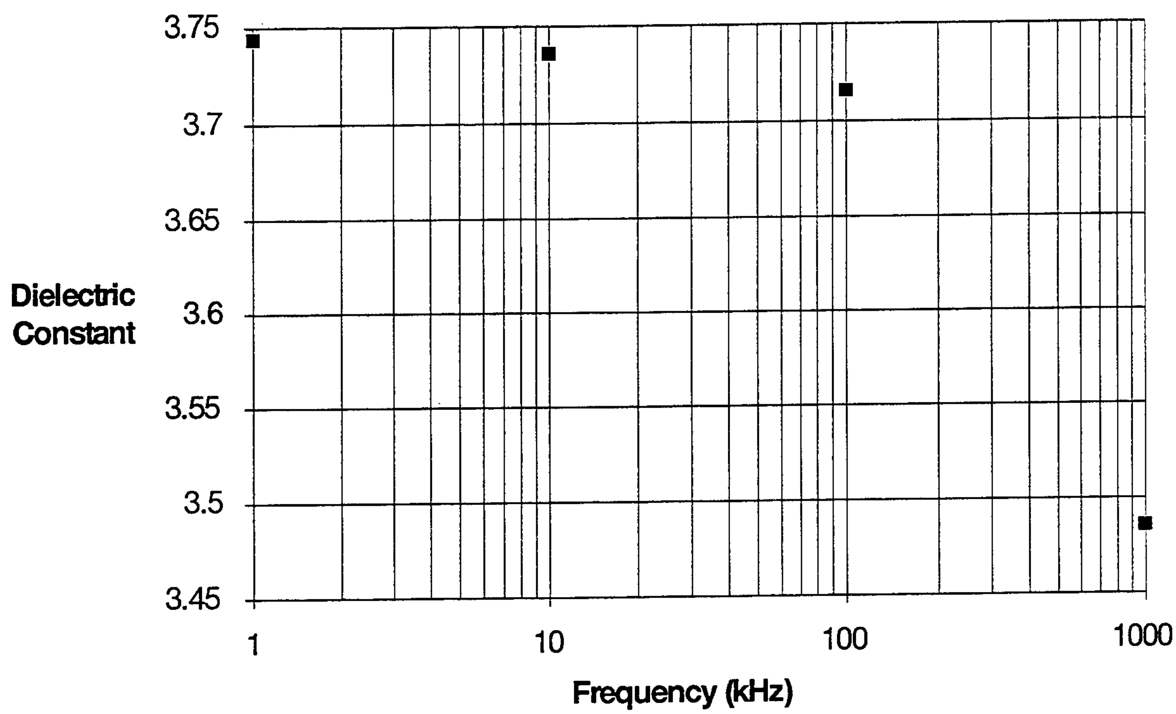

Figure 3. Room Temperature Dielectric Constant Results for PEEK.

range. The resistivity drops off linearly on the log-log plot of Figure 5 but is still of significant value at 1-MHz frequency.

These data are significantly higher in value in the higher frequency range than reported by PEEK's manufacturer, ICI [14]; ICI's data for 450G PEEK indicates loss tangents below 0.004

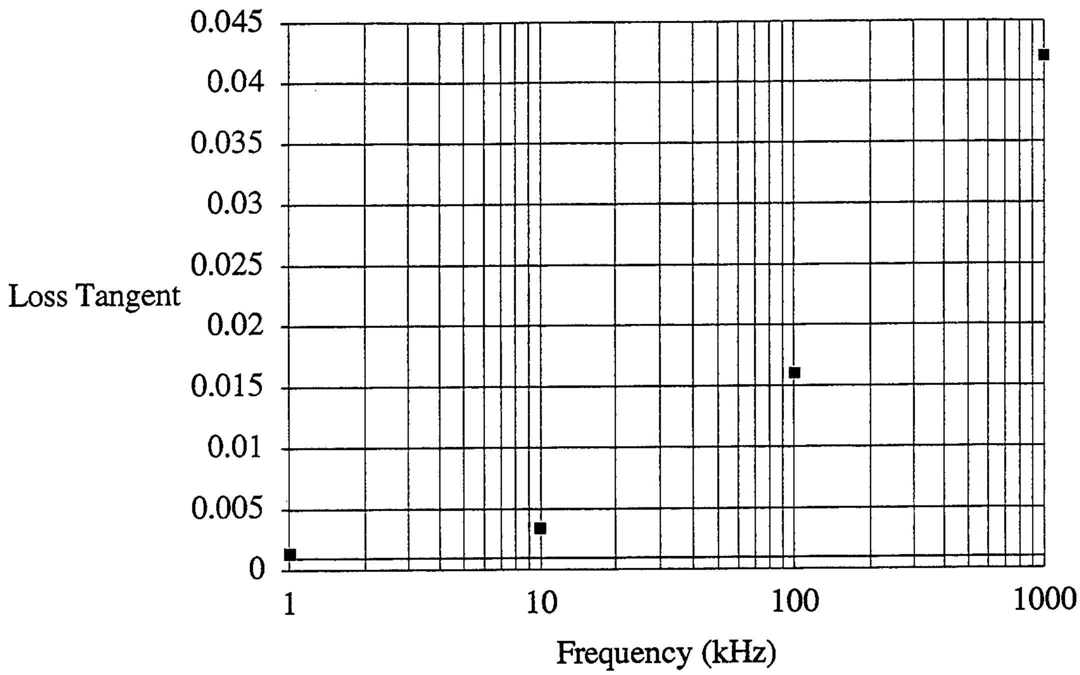

Figure 4. Room Temperature Dielectric Loss Tangent Results for PEEK.

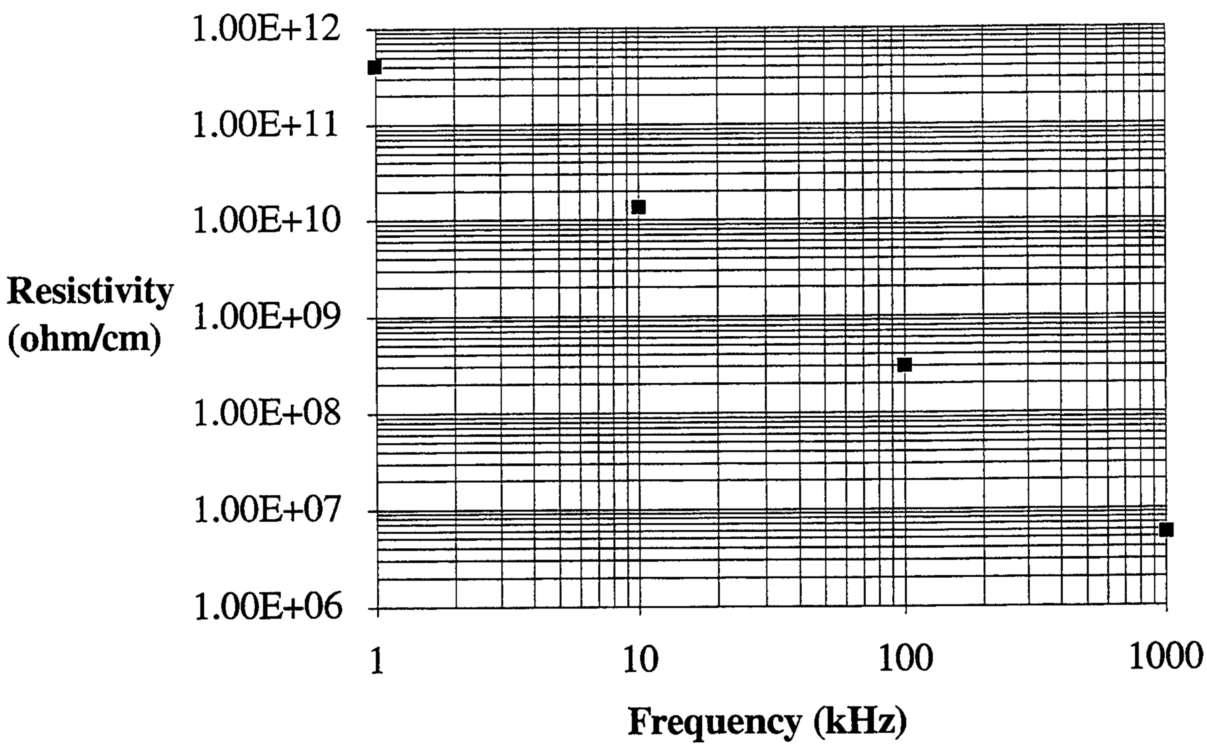

Figure 5. Room Temperature Results for Resistivity of PEEK.

at all frequencies between line frequency and 10^{10} Hz at room temperature. This discrepancy between our results and published data further precipitated an investigation of the effects of moisture content on dielectric loss tangent of PEEK.

Twelve specimens were cut from the original PEEK plate. Nine of the specimens were placed in an oven at 120 °C and 28-inHg vacuum in sets of three for periods of 24, 48, and 72 hr. The first three specimens were tested at temperatures ranging from 25 °C to 250 °C in 25 °C increments (nominally). They were assumed to have full equilibrium moisture content. The other specimens were tested as they were removed from the oven. Since each test took approximately 3 hr to run, some specimens were in the oven for 3–6 hr longer. Table 2 and Figure 6 show the dimensional and weight change data for all specimens. The solid squares represent the test data, while the line represents a spline fit to the average of the three data points at each nominal time plus 3 hr (which is the average time for each test set). From this plot, the maximum moisture content is taken to be 0.28 weight-percent; this value corresponds well to data reported in the literature.

Table 2. Weight Change Data for PEEK's Dielectric Loss Tangent vs. Moisture Content Testing

Time in Oven (hr)	Initial Weight (g)	Test Weight (g)	% Change	Average Change (%)
0	4.764	4.764	0	
0	4.812	4.812	0	0
0	4.693	4.693	0	
24	4.827	4.818	0.18	
27	4.71	4.701	0.19	0.1767
30	4.796	4.788	0.16	
48	4.774	4.762	0.25	
51	4.813	4.801	0.24	0.2533
54	4.682	4.669	0.27	
72	4.792	4.777	0.31	
75	4.845	4.832	0.27	0.28
78	4.613	4.601	0.26	

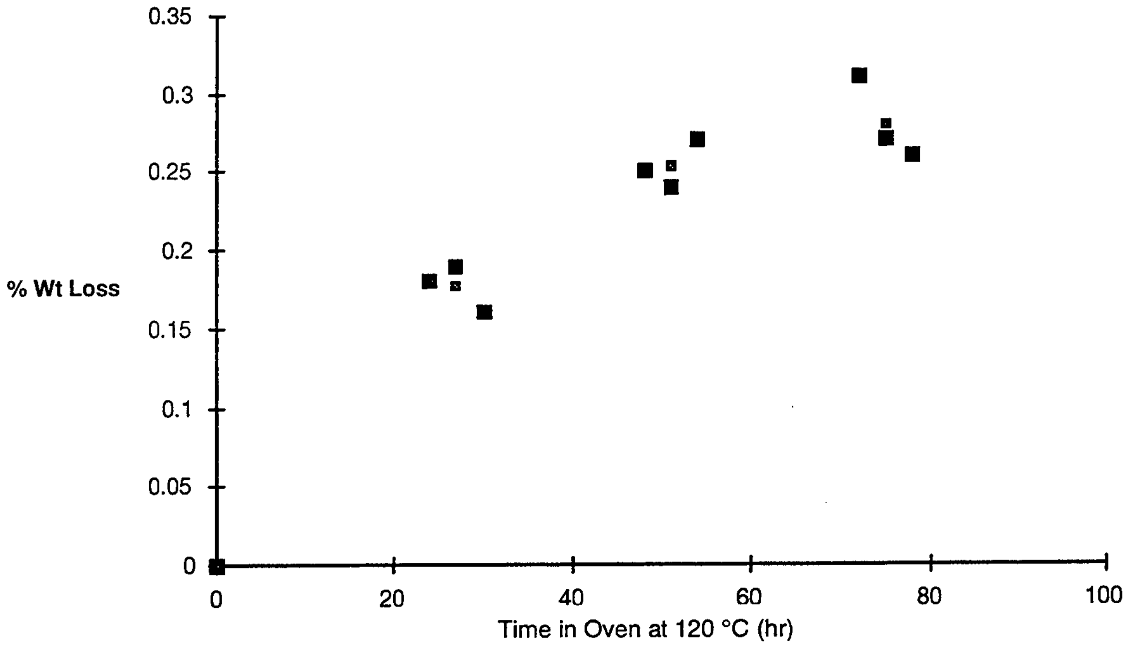

Figure 6. Percent Weight Loss of PEEK Specimens vs. Drying Time. Note That the Large Squares Represent the Three Specimens at Each Nominal Time. The Small Squares Represent the Average of the Three Data Points at Each Nominal Time.

Since maintaining the exact nominal temperatures was not possible, the results for the testing of each specimen have data points at different temperatures. However, the temperatures were generally maintained to within 5 °C of the nominal test temperature, so data has been averaged and plotted, assuming that all tests took place at the nominal test temperature. Figures 7 through 10 show the averaged loss tangent results for the 0-, 24-, 48-, and 72-hr specimens, respectively.

The influence of moisture content on the range of possible values of loss tangent at 100 kHz is shown in Figure 11. Here the 72-hr test is taken to be nearly zero moisture content and the 0-hr test is taken to be 0.28 weight-percent moisture. Also shown on Figure 11 is the value for loss tangent reported by ICI [14]. Although it was not reported as a function of moisture content, it might be concluded that their test specimens were indeed sufficiently dried prior to testing.

No attempt was made to quantify the percent moisture loss in fiber-reinforced PEEK. Furthermore, no attempt was made to increase the moisture content of the PEEK or APC-2

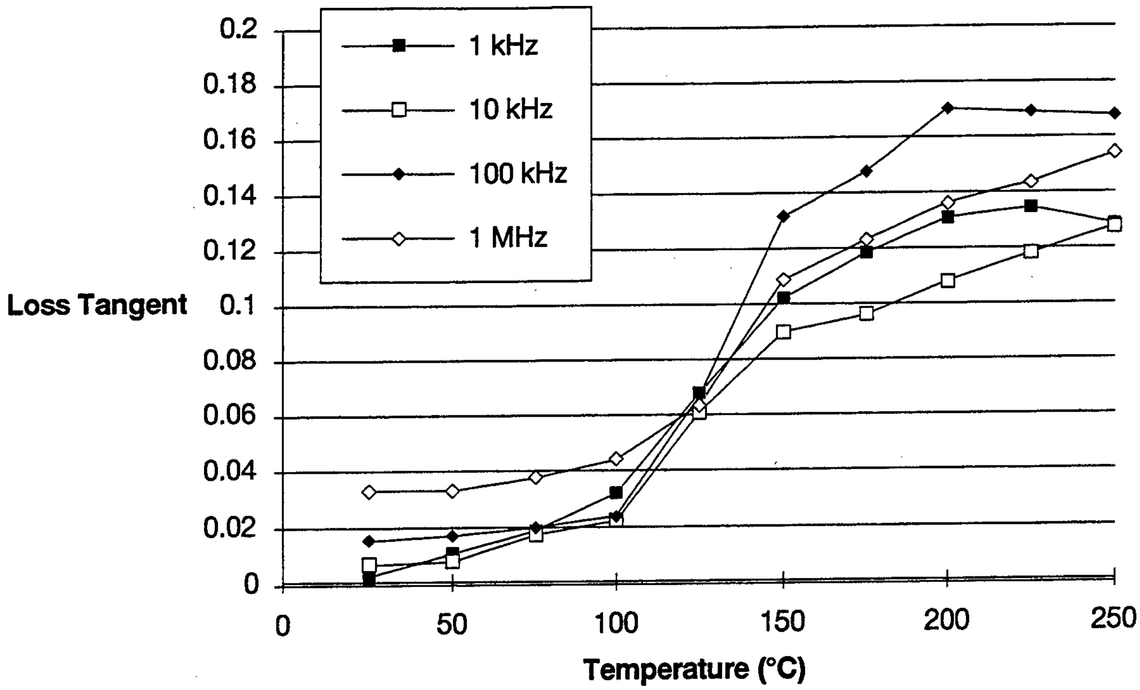

Figure 7. Average Experimental Loss Tangent Data for Three Undried PEEK Specimens.

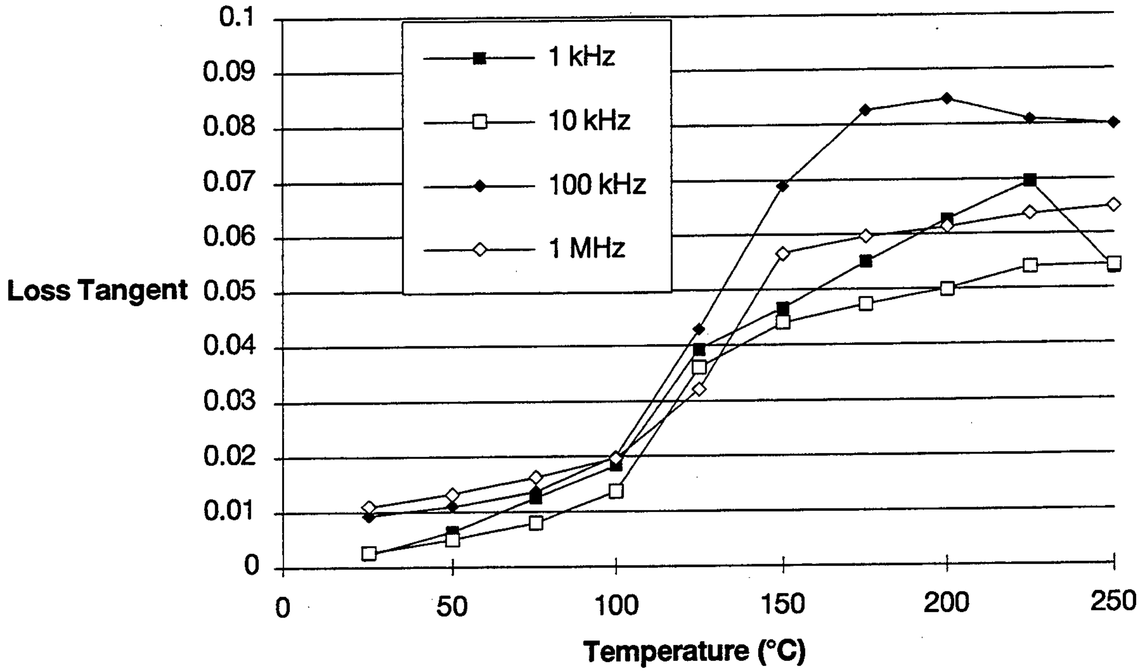

Figure 8. Average Experimental Loss Tangent Data for Three PEEK Specimens Dried at 120 °C for 24 hr.

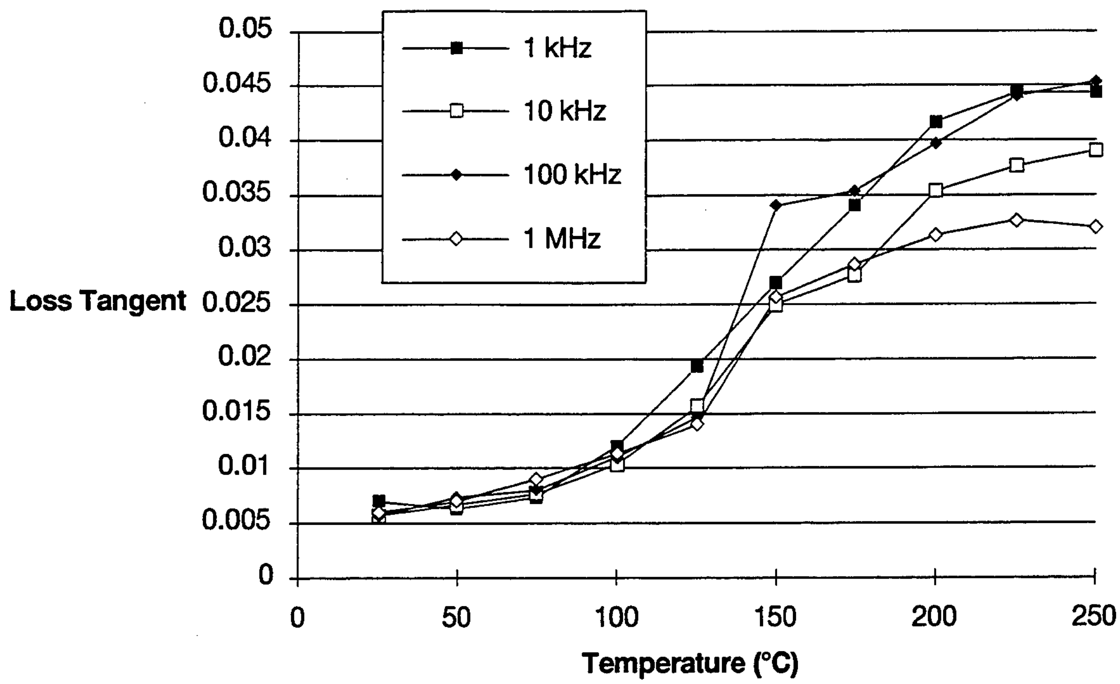

Figure 9. Average Experimental Loss Tangent Data for Three PEEK Specimens Dried at 120 °C for 48 hr.

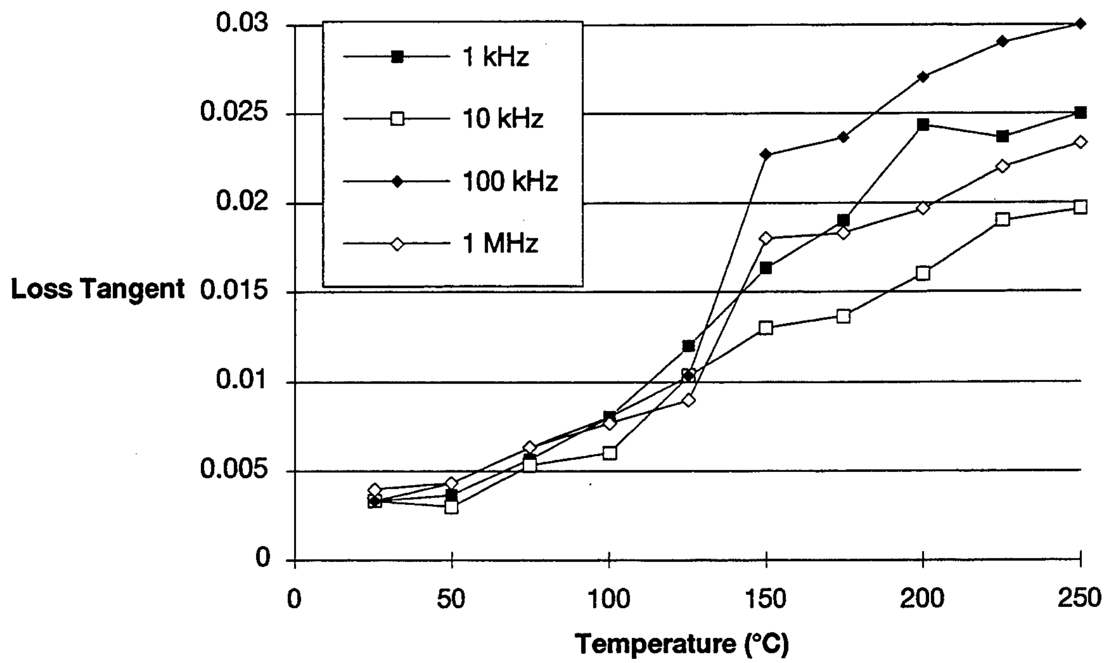

Figure 10. Average Experimental Loss Tangent Data for Three PEEK Specimens Dried at 120 °C for 72 hr.

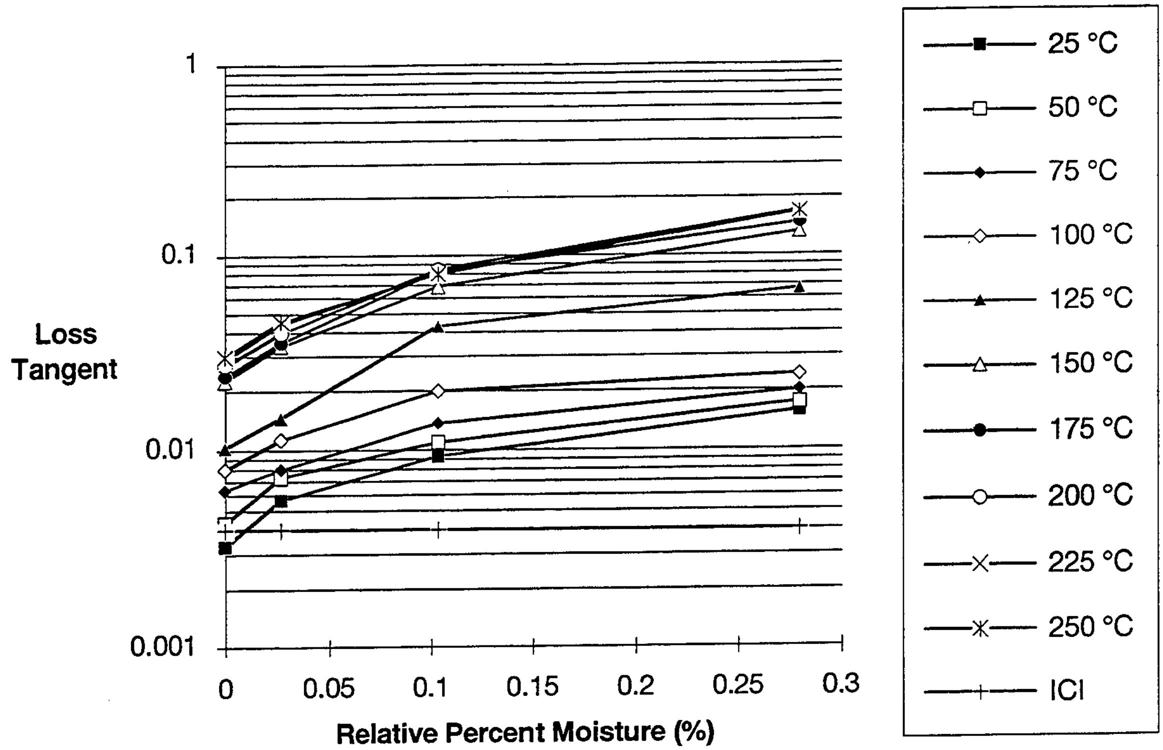

Figure 11. Experimental and ICI-Reported Loss Tangent Data for PEEK at 100 kHz. Note That the Experimental Data Approaches the ICI-Reported Data for Low Moisture Contents.

specimens, which, from the moisture dependence of loss tangent in Figure 10, might further increase heating. From this study, it appears possible to optimally localize heating at a bond interface in fusion bonding applications by increasing the moisture content of that local region; however, adding moisture in the presence of thermal gradients may nucleate undesireable voids in the composite [15].

4. Summary

Heating of continuous carbon-fiber-reinforced polymers (CFRP's) by the application of an alternating magnetic field has been shown to be due to dielectric losses in the polymer. A fundamental analysis indicated that heating in dielectric materials, such as PEEK, is linearly dependent upon the dielectric constant and the dissipation factor. Although not explicitly characterized, induction heating in PEEK-based composites was shown to increase significantly with increases in moisture content and initial as-is loss tangent measurements showed relatively high values compared to those reported by the manufacturer. This precipitated a study of the effect of moisture content on dielectric properties. These properties were theoretically investigated as functions of temperature, frequency, ionic conductivity, and moisture content.

The imaginary part of the complex dielectric constant is an integral parameter in dielectric heating, and induction-heating thermal calculations are complicated by the loss tangent's dependence on frequency, temperature, moisture, and ionic conductivity. Fundamental tests on PEEK were performed to determine these relationships. A significant result is the extreme moisture dependence of the lossiness of PEEK. Under dry conditions, the loss data were near that reported by the manufacturer. Our immediate application of this study is to interpolate the expected loss tangent data for the frequency and temperature range expected in induction-heating experiments. This study has also established the importance of documenting the dependence of dielectric properties on frequency, temperature, and moisture.

From this study, it appears possible to optimally localize heating at a bond interface in fusion-bonding applications by increasing the moisture content of that local region; however, adding moisture in the presence of thermal gradients may nucleate undesireable voids in the composite [15].

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An analysis of local and global mechanisms of heat generation and distribution in carbon-fiber-based composites subjected to an alternating magnetic field has shown that heating is dependent upon the dielectric properties of the polymer matrix. These properties were investigated as functions of temperature, frequency, and moisture content. The results indicate little dependence of the dielectric constant on temperature or frequency, while the loss tangent exhibited a substantial dependence on both frequency and temperature. A substantial dependence of loss tangent on moisture content in polyetheretherketone (PEEK) was found.

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