# DIELECTRIC MATERIALS MEASUREMENTS

Network Measurements Division 1400 Fountaingrove Parkway Santa Rosa, California 95403

> AUTHORS: David Blackham Frank David David Engelder

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## ABSTRACT

The electro-magnetic properties of materials must be accurately measured before these materials can be skillfully applied. This paper covers the basics of permittivity (dielectric constant) and permeability at high frequencies, including terminology and data formats used in the field

Next, it surveys a variety of measurement methods based on RF/microwave network analyzers, and discusses the strengths and limitations of each. This includes recent enhancements to the popular "S-parameter method" (HP Product Note 8510-3), plus a new coaxial dielectric probe for making permittivity measurements easier and more convenient.

Finally, the presentation addresses some applications, which require accurate dielectric measurements.

## AUTHORS

David Blackham is an R&D Development Engineer at HP's Network Measurements Division in Santa Rosa, California. He received his BSEE from Brigham Young University and MSEM from Stanford University. David joined HP in 1979 and worked on the HP 8340A Synthesized Sweeper, scalar detectors and bridges, and microwave vector network analyzers. He is currently working on material characterization.

Frank David is an R&D Project Manager at HP's Network Measurements Division in Santa Rosa, California. He earned his BSEE from the University of California at Berkeley, and his MSEE from Oregon State University. After joining HP in 1969, Frank designed microwave components for the HP 8755 Frequency Response Test Set and HP 8566A Spectrum Analyzer. He later managed the millimeter-wave mixer and HP 8720 Network Analyzer projects.

David Engelder received a BSEE from the University of California, Santa Barbara, in 1979. He joined HP's Network Measurements Division in Santa Rosa, California, in 1980 — working first as an engineer and later as a manager in the Product Support area of Marketing. Dave is currently in Product Marketing, promoting the HP 8720 and applying microwave network analyzers to dielectric materials measurements.



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This presentation concerns the electro-magnetic properties of materials at high frequencies; specifically:

- Permittivity (or dielectric constant); and
- Permeability

It is important to understand and measure these properties to skillfully apply materials in a given application. In the traditional electronic industries, this information is obviously needed for solid design and quality control. In addition, this paper will also relate these properties to industrial high-power microwave heating and drying.





The presentation has three parts:

- Basics
- Measurement Methods
- Applications

Let's begin by reviewing the basic principles and terms used in this field.





The laws of "electro-magnetics" describe the behavior of electric fields and magnetic fields. In the time-varying case (e.g. a sinusoid), both kinds of fields appear together. This radiation can propogate through free space, or through materials. As frequency changes across the spectrum, the electro-magnetic radiation appears in many different forms. However, the same basic laws apply.

Today, we are concerned with the RF and microwave part of the spectrum.

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When electric and magnetic fields pass through a material, each can interact with that material in two ways:

• Storage: Energy may be exchanged between the field and the material, in a bi-directional (lossless) manner.

• Loss: Energy may be permanently lost from the field, and absorbed in the material (usually as heat).

The electric interactions are quantified by permittivity  $(\epsilon_r^*)$ , also called dielectric constant  $(\kappa^*)$ . The magnetic properties are described by permeability  $(\mu_r^*)$ . These are complex numbers with two parts:

- Real Part: Represents storage term; denoted with '.
- Imaginary Part: Represents loss term; denoted with ".

This paper focuses on permittivity, since many common materials are completely non-magnetic.

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Complex permittivity can be drawn as a simple vector diagram. The real and imaginary parts form a 90-degree angle to each other. The vector sum forms an angle  $\delta$  with the j-axis.

The "lossiness" of a material is the ratio of energy lost to energy stored, or  $\epsilon''/\epsilon'$ . This ratio is the tangent of the angle  $\delta$  — so people call it:

- tan δ ("tan delta")
- loss tangent
- tangent loss
- s tangent loss

Tan  $\delta$  is directly related to D (dissipation factor) and Q (quality factor).

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This graph has  $\epsilon$ ' (storage) along the vertical axis, and tan  $\delta$  (loss) on the horizontal axis (both logarithmic). The values for several common materials are shown as dots (at a single frequency and temperature).

"Low-loss" materials, such as Teflon, have small values of tan  $\delta$ . They are commonly used in electronic applications such as: insulators (e.g. for cables), substrates, and dielectric resonators.

"High-loss" materials include water, food, and many natural materials. These materials quickly absorb microwave energy, and so are not used for electronic components. However, they are important to:

- Understanding microwave radiation in the "real world"
- Material analysis (e.g. moisture content)
- High-power microwave processing (heating and drying)







At the microscopic level, several dielectric mechanisms can contribute to dielectric behavior:

Dipole orientation (and ionic conduction) interact strongly at microwave frequencies. Water molecules, for example, are permanent dipoles, which rotate to follow an alternating electric field. These mechanisms are quite lossy — which explains why food heats in a microwave oven.

Atomic and electronic mechanisms are relatively weak, and usually constant over the microwave region.

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Each dielectric mechanism has a characteristic "cutoff frequency." As frequency increases, the slow mechanisms drop out in turn, leaving the faster ones to contribute to  $\epsilon$ '. The magnitude and "cutoff frequency" of each mechanism is unique for different materials.

Water has a strong dipolar effect at low frequencies — but its dielectric constant rolls off dramatically around 20 GHz. Teflon, on the other hand, has no dipolar mechanisms — so its permittivity is remarkably constant well into the millimeter-wave region.



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For dipolar dielectrics (such as water), a "relaxation constant"  $\tau$  describes the time required for dipoles to become oriented in an electric field. (Or the time needed for thermal agitation to disorient the dipoles after the electric field is removed.) At low frequencies, the dipoles can "follow" the field and  $\varepsilon$  will be high.

At high frequencies, the dipoles can't follow the rapidly changing field — and  $\epsilon'$  falls off.

The loss factor  $\varepsilon$ " peaks at the frequency  $1/\tau$ . Here, energy is transferred into the material at the fastest possible rate.



For high-loss materials, both  $\varepsilon'$  and  $\varepsilon''$  change dramatically with frequency. A Cole-Cole plot (similar to a Smith chart) is often used to plot the "frequency response" of materials. Simple lossy materials (e.g. water) scribe a semi-circle on a Cole-Cole plot. More complex materials may form an ellipse, or an arc with bumps on it.

The two traces demonstrate that  $\varepsilon^*$  (for water) changes dramatically with temperature, as well as frequency.



Consider a plane boundary formed by two materials (e.g. a dielectric slab in air). The electro-magnetic impedance of a medium depends on  $\varepsilon^*$  and  $\mu^*$ , so the boundary has an impedance mismatch. Incident radiation will be partly reflected, and partly transmitted into the dielectric.

 $\varepsilon$ =2.04 for Teflon, which is fairly close to air ( $\varepsilon$ =1). The reflection coefficient at this boundary is roughly 0.17 (a return loss of 15 dB).

For water,  $\varepsilon$  =80. The large mismatch reflects about 80% of the field back into air (return loss of only 2 dB). For heating/drying applications, boundary reflections are an important part of the overall efficiency.





Once inside a material, energy is "lost" (absorbed as heat) at a rate dependent on  $\varepsilon^*$ . The field strength follows an exponential decay, related to distance d by an attenuation factor  $\alpha$ . One can also define a "penetration depth" at which the field strength or power has dropped by 1/2 or 1/e.

Teflon is very low loss — just 0.0006 dB/cm. The fields decay very slowly over a large distance.

Water, on the other hand, loses 3.9 dB/cm (at 3 GHz). Energy is transferred into the water over a very short distance.

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These are the key points — the properties that describe the interaction between electro-magnetic fields and materials. Of course, these properties are the foundation for applying materials. But they also form the basis for the various methods to measure these properties.





The presentation now turns to three methods used to measure  $\varepsilon^*$  and/or  $\mu^*$  over frequency.

(Note: All three approaches are based on RF or microwave network analyzers. Other methods exist, but are not addressed in this paper.)



HP's network analyzers make a good foundation for all swept high-frequency stimulus-response measurements. To measure dielectric materials, two additional elements are needed:

• Fixture — contains material; applies electro-magnetic fields in a predictable way; allows connection to network analyzer.

• Software — converts S-parameter data from network analyzer into  $\varepsilon^*$  and/or  $\mu^*$  (depends on fixture).

Each method (and fixture) has strengths and weaknesses.

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Cavities are high-Q resonant structures. A sample of the material-under-test (MUT), inside the cavity, affects its center frequency and Q (quality factor). From these two parameters, the complex permittivity of the sample — both  $\epsilon$ ' and  $\epsilon$ " — can be calculated.

Two approaches can be used:

• Perturbation: measure  $f_c$  and Q of empty cavity, then find shifts caused by small sample in cavity.

- Absolute: directly calculate relation from  $f_{\rm c}$  plus Q to  $\epsilon^*$ (requires precise understanding of fields in cavity).



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Cavity methods are very accurate, and can measure tan  $\delta$  of very low-loss materials with high precision.

However, most cavities resonate (and yield  $\varepsilon^*$ ) at discrete frequencies. The analysis can be very complex, especially for the absolute approach. The MUT must be destructively sampled, and formed into a precise shape. And few fixtures are commercially available — most users design and manufacture their own.

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Transmission-line methods involve putting the MUT inside a portion of an enclosed transmission line. The line is usually rectangular waveguide or a coaxial airline.



The measurement can be based on the reflection coefficient  $(S_{11})$ , transmission coefficient  $(S_{21})$ , or both.

The popular "S-parameter" approach (Nicolson-Ross or Weir) uses both  $S_{11}$  and  $S_{21}$  to calculate both  $\varepsilon^*$  and  $\mu^*$ . HP described this technique, adapted for the HP 8510, in Product Note 8510-3 (Aug 85).

Note that  $S_{11} \mbox{ and } S_{21}$  are composed of multiple-reflections from both boundaries.

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These traces are  $\epsilon'$  versus frequency for Teflon, measured in a broadband coaxial transmission line with an HP 8510. (Results courtesy of NIST.)

The blue trace uses the traditional Nicolson-Ross algorithm. The periodic drop-outs occur at frequencies where the sample is  $n\lambda/2$  wavelengths thick. There, the reflections from the two boundaries exactly cancel each other, and S<sub>11</sub> drops to zero. Researchers usually avoid this problem by keeping the sample thickness below  $\lambda/2$  at the highest frequency.

The red trace is actual data, using the same equipment and S-parameter data as before. However, new algorithms developed by Baker-Jarvis at NIST — have solved the  $n\lambda/2$ dropout problem. This method now works well with samples much longer than  $\lambda/2$  — yielding better results for tan  $\delta$  on low-loss materials. (Note: New NIST algorithms apply to non-magnetic materials.)



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Transmission-line methods have advantages over cavity methods: They yield swept-frequency results over a wide range of frequencies. The Nicolson-Ross algorithms provide both  $\varepsilon^*$  and  $\mu^*$ . And transmission-line fixtures are easier to build and analyze.

However, the transmission-line methods are not as accurate or sensitive to low tan  $\delta$ . Sample preparation is just as difficult, and is usually destructive.

(Note: As of July 1990, HP offers the software to perform these calculations as the HP 85071A Materials Measurement Software. HP also supplies waveguide and coax accessories to serve as fixtures.)

#### Slide 8769



The third method is simply an open-ended coaxial line. The MUT is measured by simply touching the probe to a flat face of a solid, or immersing its end into a liquid. The fields at the probe end "fringe" into the material, causing a reflection  $(S_{11})$  that can be related to  $\varepsilon^*$ .



The geometry can be modeled with a simple circuit, where the total admittance Y is composed of:

• A pure capacitance (phase change), related to  $\epsilon^\prime$  (storage in MUT).

- A conductance (magnitude change), related to  $\epsilon^{\prime\prime}$  (losses in MUT).

• An additional conductance, represented radiation losses.

This model yields a single-pole frequency response. The probe's sensitivity in measuring  $\varepsilon'$  is related to the slope of the phase response. Hence, the accuracy in  $\varepsilon'$  of this method is optimum over a 2-decade frequency range, depending on the MUT. Radiation losses vary with  $\varepsilon'$ ,  $\varepsilon''$ , and frequency — the model must be quite complex to accurately account for them. These losses limit the measurement sensitivity in  $\varepsilon''$  (or tan  $\delta$ ), especially at higher frequencies. This is because  $G_{T}$  is in parallel with  $G_{\varepsilon}''$  — as  $G_{T}$  grows more conductive, it becomes more difficult to determine  $G_{\varepsilon}''$  from Y.

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Ultimately, the model relates  $S_{11}$  of the probe-plus-MUT (measured by a network analyzer) to  $\varepsilon^*$  for the material. Here, a grid of  $\varepsilon$  and tan  $\delta$  is mapped over a polar chart of  $S_{11}$  for a single frequency.

The network analyzer's uncertainty in measuring  $S_{11}$  is a small uniform circle on the polar chart. This map, then, also indicates the resolution in measuring  $\epsilon^*$  due to the  $S_{11}$  uncertainty. Where the  $\epsilon^*$  lines are widely spaced, the small  $S_{11}$  uncertainty causes little error in measuring  $\epsilon^*$ . Where closely spaced, the  $\epsilon^*$  resolution is not as good.



These plots show how the map of  $\varepsilon'$  and tan  $\delta$  can change with frequency. At this low frequency, the  $\varepsilon^*$  lines are closely spaced for virtually all values.

#### Slide 8983B



These plots map  $\varepsilon'$  and tan  $\delta$  at a high frequency. The network analyzer's uncertainty in S<sub>11</sub> would be negligible when  $\varepsilon'$  is low, and would become more significant as  $\varepsilon'$  increased.



These plots demonstrate the probe's resolution versus frequency, for various values of  $\varepsilon$ ' and tan  $\delta$ . The vertical axis represents both %-uncertainty in  $\varepsilon$ ' and absolute uncertainty in tan  $\delta$ . Obviously, the probe's accuracy depends on:

- $\epsilon$  of the MUT
- $\epsilon^{\prime\prime}$  or tan  $\delta$  of the MUT
- Frequency

These graphs assume that  $S_{11}$  uncertainty is 0.05 at all frequencies, and plot the corresponding maximum error in  $\varepsilon^*$ . In fact, the specified  $S_{11}$  uncertainty is often smaller, and the actual  $S_{11}$  accuracy is typically much smaller still. However, there may be other sources of error — such as calibration quality or contact gaps between the probe tip and a solid MUT.

Slide 8985 PROBE CALIBRATION Similar to 1-port (3-term) cal for vector network analyzer (Directivity, Tracking, Source Match) 1. Use model to predict S<sub>11</sub> of 3 standards: • Air RC = 1 • Shert RC = -10 • User-Defined Standard (ea. Weet) 2. Measure standards 3. Calculate difference arrays and apply "correction" to later measurements Partner

For best results, the probe (with network analyzer) should be "calibrated" by measuring known standards. The model is used to predict  $S_{11}$  for various standard materials, such as air. The difference between this predicted value and the actual measured  $S_{11}$  is used to correct subsequent measurements.

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Here are some actual measurement results, using the probe:

For Teflon (not shown), the values of  $\varepsilon'$  are consistent with the literature across the entire frequency range. The previous slides predicted the poor resolution at low frequencies, since  $\varepsilon'$  is so low. (The  $\varepsilon''$  data is not useful for such a low-loss material.)

For water,  $\varepsilon'$  again agrees with published values. Since  $\varepsilon'$  is high, there is no noise at low frequencies — including 2.45 GHz.  $\varepsilon''$  also measures as expected.

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The key advantage of the probe method is its convenience: for most materials, no sample preparation is required and the method is non-destructive. The probe works well for liquids and semi-solids — making it popular for food and biological researchers.

While the probe provides reasonable results over a 2-decade bandwidth, it is not as accurate as other methods and cannot resolve  $\varepsilon$ " or tan  $\delta$  for low-loss materials.

(Note: As of July 1990, a probe — with its dedicated software and accessories — are offered as HP 85070A Dielectric Probe.)

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Let's summarize the three methods, while comparing their key strengths and weakness. This chart plots accuracy versus convenience.

Cavity methods offer the highest accuracy, especially for low tan  $\delta$ . But they are narrowband, complicated, and require destructive sample preparation.

Transmission-line methods provide the widest possible frequency range — and can determine both  $\varepsilon^*$  and  $\mu^*$ . However, they are not as accurate as cavities, and still require destructive sample preparation.

The probe method is by far the most convenient: it yields wide-band results, and requires no sample preparation (non-destructive). The probe is especially convenient for liquids and semi-solids (e.g. food and biological materials). Generally, however, the probe offers less precision than other methods.

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Let's see how these measurements can help in applying materials and microwaves in practice. We'll touch briefly on their use in electronic components — then explore world of commercial microwave industrial processing.



Here again is the map of materials.

Low-loss materials are commonly used in electronics (along with lossy absorbers or shielding). When used in cables,  $\varepsilon'$ affects the impedance and  $\varepsilon''$  the loss. In substrates,  $\varepsilon'$  sets the impedance/capacitance of lines. Used in dielectric resonators,  $\varepsilon'$  sets the frequency for a given geometry, while  $\varepsilon''$  determines the Q and spectral purity. Materials are also used in ferrites, antenna lenses, windows, radomes, and waveguides —  $\varepsilon^*$  is always the critical design parameter.

High-loss materials are rarely used in electronics. However, the same microwave expertise and technology (historically developed for defense applications) is required to apply high-power microwave energy to (commercial) industrial processing. (The following topics will reinforce the theory just presented, regardless of application.)

#### Slide 8988

EXAMPLES		ADVANTAGES			
Drying:	Factive     Factive     Factor     Fact				
Feed Precessing:	<ul> <li>Most tempering</li> <li>Bacen cesking</li> <li>Freeze dehydratien</li> <li>Blanching</li> </ul>	Greater penetration depth     Lawer material temp.     Cantinuous processing     Faster processing			
Rubber/ Plastic Processing:	Rubber vulcenization     Plastic curing	e Greeler penstralion depth e Lewer auroce lemp. e Faster processing			
Ceramic Processing:	Greater penetration depth				

This is a partial list of applications where industrial microwave processing has been used successfully. Because of its unique advantages, microwave processing has displaced conventional heating in many applications even though microwave energy is more expensive on a costper-Joule basis.

We'll see now that a solid understanding of dielectric behavior, and the ability to measure  $\varepsilon^*$  over frequency and temperature, enable us to confidently and analytically design microwave processing systems.

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		("ISM") FRE	QUENCE	:5	
6.780	<u>+</u>	0.015 MHz	2.450	<u>+</u>	0.050 GHz
13.560	<u>+</u>	0.007 MHz	5.800	<u>+</u>	0.075 GHz
27.120	±	0.163 MHz	24.125	<u>+</u>	0.125 GHz
40.680	<u>+</u>	0.020 MHz	61.250	+	0.250 GHz
133.920	÷	0.870 MHz)	122.500	+	0.500 GHz
915.000	<u>+</u>	13,000 MHz	245.000	+	1.000 GHz
					PAT 8989

The "ISM" frequencies are those allocated for industrial processing in the US. (Most other nations allocate a similar list).

However, the vast majority of today's industrial processing (and home microwave ovens) use only the 2.45 GHz band. Might other approved frequencies be better choices?

Yes! Let's see why . . .

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One reason microwave processing can be more "effective" is differential heating.

Consider a mixture of two materials, where the loss factors  $\epsilon_1$ " and  $\epsilon_2$ " are different. Microwave fields will couple more strongly with the higher-loss material, and heat it selectively.

In making paper, this effect is used to "level" the moisture (make it uniform across the web). In another example, selective heating removed high-loss contaminants (sulfer compounds) from low-loss coal.

Obviously, measuring how  $\epsilon_1$ " and  $\epsilon_2$ " vary with frequency helps to choose the frequency that optimizes differential heating.



Another reason microwave heating can be more effective is its ability to penetrate materials and uniformly heat the entire volume of an object. (Conventional approaches, by contrast, heat the outside surface and rely on conduction to transmit heat to the core.)

A good example is vulcanizing or molding rubber (e.g. tires). Since rubber has high thermal insulation, the heat conduction from surface to core is slow and time-consuming. Higher surface temperatures can't be used, since the surface layer of the product would be degraded. Instead, microwave heating raises the temperature quickly and uniformly, thoughout the object's volume. For best results, the penetration depth must be optmized for the  $\varepsilon^*$  of the material and the physical dimensions of the object. A knowledge of  $\varepsilon^*$  versus frequency is crucial. In many cases, the operating frequency is the only parameter in which one has any choice.





For a given electric field strength, the power dissipated in a material is proportional to  $\varepsilon$ " and f. In turn,  $\varepsilon$ " is a function of frequency (with a typical shape, as shown). The top curve is power dissipation — the product of  $\varepsilon$ " and f. If we assume a fixed field strength, the efficiency of power transfer into the material can be maximized by choosing the right frequency.



In practice, other concerns — such as penetration depth — may be more important than simple efficiency. But consider the problem of drying thin sheets of material (paper, fabric, wood veneer). In this case, maximum power absorption is essential. The left plot shows  $\varepsilon$ " for water across frequency, at several temperatures.

Taken at two ISM frequencies — 2.45 GHz and 24.125 GHz — this same data can be plotted versus temperature. Note that  $\varepsilon$ " is much higher at 24.125 GHz, for all temperatures between freezing and boiling.





This table compares the power absorption factors for heating water at 2.45 and 24.125 GHz.

 $\epsilon$ " at 24.125 GHz is 3, 6, or even 9 times higher than at 2.45 GHz. The frequency itself, of course, is about 10 times greater. The overall efficiency of power dissipation, then, is  $\epsilon$ " times f — which is up to 90 times higher at 24.125 GHz than at 2.45 GHz! (Ironically, this frequency is very rarely used.)

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There is one final reason to characterize  $\epsilon^{*}$  before processing with microwaves: safety.

The top equation describes the energy balance for microwave heating. The microwave energy absorbed in a material ( $\epsilon$ "P) must equal the energy lost by convection (H term) plus the energy raising the material's temperature (mC term).

But we know that  $\varepsilon$ " is a function of temperature. A polynomial can approximate this relation, and be used in the top equation. Solving the differential equation gives curves of temperature versus time. The three curves are for three levels of incident microwave power. Two power levels stabilize over time at a certain temperature. But the third demonstrates "thermal runaway" — causing the material to melt, burn, or even explode.

## Slide 8994 SUMMARY & CONCLUSIONS • BASICS EM Fields Interact with Materials Real (Storage) and Imaginary (Loss) Reflection, Absorption, Penetration • MEASUREMENT METHODS Network Analyzer + Fixture + Software Cavity - Transmission-Line - Probe • APPLICATIONS Quantitative Knowledge Gives Better Results Need to Know (; versus Frequency

In summary, we laid the foundation for a good understanding of dielectric behavior, and described several methods for measuring complex permittivity  $\varepsilon^*$ .

These properties must be known before materials can be used in electronic components. Microwave designers are very familiar with the impact of  $\varepsilon^*$  in their designs.

But we have also demonstrated the need to characterize  $\varepsilon^*$  over frequency to optimally apply microwave energy in commercial processing.

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