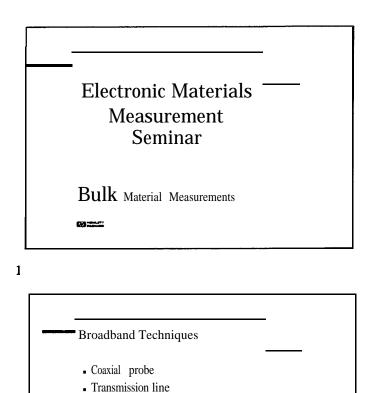
Hewlett-Packard

Electronic Materials Measurement Seminar

Bulk Material Measurements

Kobe Instrument Division



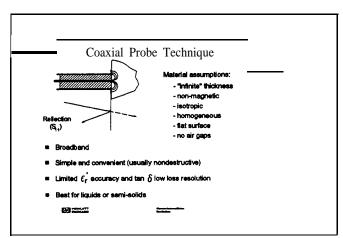


. Free space

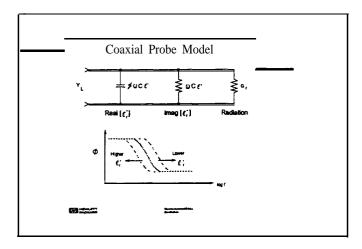
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We will be focussing on three broadband techniques.

2

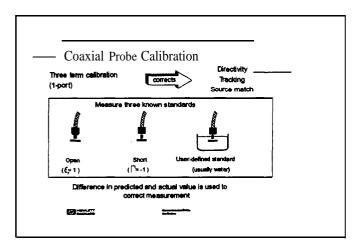


The coaxial probe is a convenient and broadband technique for lossy liquids and semisolids. The MUT is assumed to be "infinite" in thickness, uniform throughout, nonmagnetic and to have uniform orientation. The probe can be used for pliable solids if no air gap is present between the probe and sample. A coaxial probe fixture connects to a vector network analyzer that measures the reflection from the MUT which is then converted to permittivity.



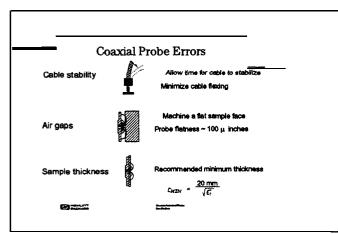
The coaxial probe geometry can be modeled with a simple circuit, where the total admittance, Y, is composed of a pure capacitance (phase change) that can be related to $_{r}$ and a conductance (magnitude change) that can be related to $_{r}$. An additional conductance represents radiation losses which limits the probe sensitivity especially at higher frequencies.

4

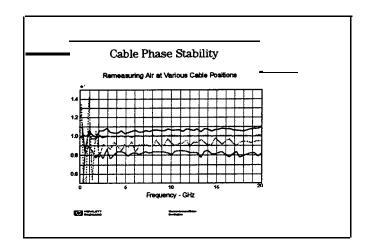


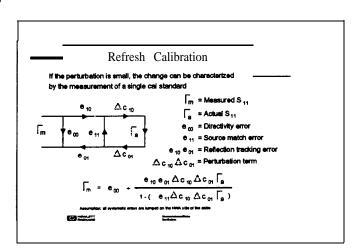
A three term calibration corrects for the direct.ivity, tracking and source match errors that can be present in a reflection measurement. In order to solve for these three *error* terms, three well known standards are measured and the difference between the predicted and actual values are used to remove the systematic (repeatable) errors from the measurement. The three known standards are air, a short circuit and water.

5

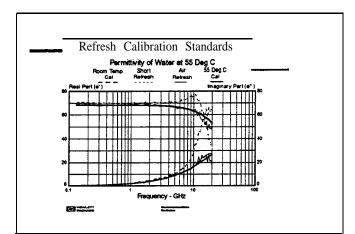


Even after calibrating the probe, there are additional sources of error that can affect the accuracy of a measurement. It is important to allow enough time for the cable (that connects the probe to the network analyzer) to stabilize before making a measurement and to be sure that the cable is not flexed between calibration and measurement. For solid materials, air gap between the probe and sample can be a significant source of error unless the sample face is machined to be at least as flat as the probe face. The sample must also be thick enough to appear "infinite" to the probe.





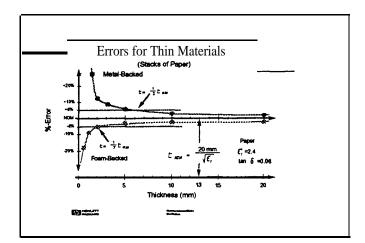
8



If the cable is moved or flexed after a calibration, the resulting measurement can vary **significantly**, causing measurement repeatability to be sacrificed. Instead of moving the probe to the sample, it is best to fix the probe and cable in one position while bringing the sample to the probe. The same result would be seen if a change in temperature **occurred** after a calibration.

If the change in the measurement (due to cable movement or temperature change) is small enough, it may be possible to characterize that small change by measuring a single calibration standard, rather than the three standards required for a normal calibration. A refresh calibration modifies an existing calibration by remeasuring just one standard. This simplifies measurements that must be made at several different temperatures or measurements where the cable must be moved.

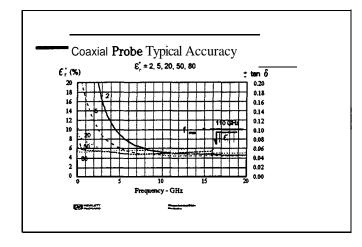
A measurement of 55" C water shows that a room temperature calibration introduces errors into the measurement. But when a short circuit or air is used to refresh the calibration it accomplishes **almost the** same results as a full calibration made **at 55°C**.



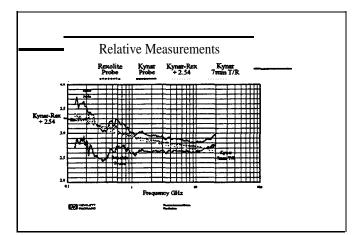
The sample size must be chosen such that any reflections from boundaries are not detected by the probe. The effects of sample thickness are shown by the measurement of paper that has been stacked to various thicknesses. In one case the paper is backed by metal and in the other case by foam. At the recommended minimum sample thickness (t_{min}) the error from nominal is less than 5%.

The typical accuracy of a measurement made with a coaxial probe is dependent on both frequency and dielectric constant. In general, the probe is more accurate at higher frequencies, although there is a maximum frequency limit. The probe is also more accurate for higher permittivities. At best the accuracy **in**,' is 5% and tan is 0.05.

10

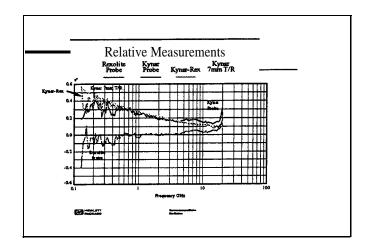


11



measurement may be limited, it does exhibit good measurement repeatability. Therefore, it can make very good relative measurements provided a known reference can be found. In this example kynar is measured with the probe and with a more accurate transmission line (T/R) technique. Rexolite, which is known to have a permittivity of 2.54, is then measured with the probe. A more accurate measurement of kynar is produced by making a measurement relative to rexolite and then adding a value of 2.54 to the difference.

Although the absolute accuracy of a probe

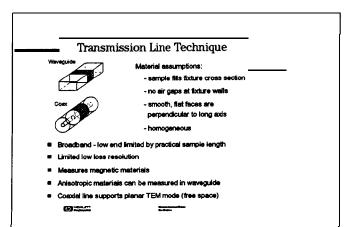


Coaxial Pr	robe Fixtures	
 HP 850708 	dielectric probe kit	
- 200 MHz t	o 20 GHz	
-Temperatur	e range of -40 to +200 degrees C	
-includes PC software	C-AT or HP 9000 series 300 compatible	
	tion and interest Birlin	

A similar measurement of the loss factor $(\mathcal{E},")$ of kynar relative to rexolite can be made.

The HP 85070B dielectric probe covers a broad 200 MHz to 20 GHz frequency range and withstands high temperatures and corrosive chemicals with its hermetic glass to metal seal. It is compatible with a wide range of HP vector network analyzers. The software to control the network analyzer and convert the measured S-parameter to permittivity is included with the probe kit.

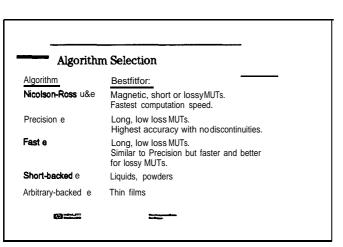
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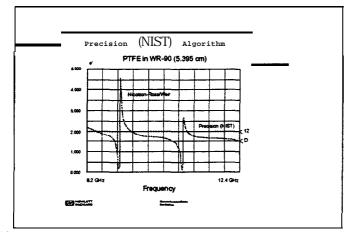
15

The transmission line is a broadband technique for machineable solids. The MUT is assumed to completely fill the cross section of the fixture with no air gaps, have smooth flat faces and to be uniform throughout. Coaxial airline fixtures are broadband, but the samples are more difficult to machine. Waveguide fixtures extend to the mm-wave frequencies and the samples are simpler to machine, but their frequency coverage is banded. A transmission line fixture connects to a vector network analyzer that measures the reflection and transmission from the MUT which are then converted to permittivity and permeability.

	•	ithms	
Algorithm	Measured	Optimum Length	Results
Nicolson-Ross u&e (PN 8510-3)	S ₁₊ S ₂₊ S ₁₂ S ₂₂ (or S ₁₊ S ₂₁)	λg/4	Er & µr
Precision e (NIST)	S ₁₁ , S ₂₁ , S ₁₂ , S ₂₂	$n\lambda_g/2$	εŗ
Fast e	S ₁₊ S ₂₁ , S ₁₂ , S ₂₂ (or S ₁₊ S ₂₁)	$n\lambda_g/2$	εŗ
Short-back e	S 11	$\lambda_g/2$	εŗ
Arbitrary-back e	S 11	$\lambda_g/2$	¢,



17

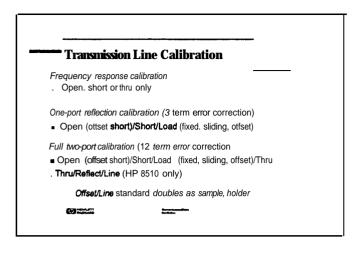


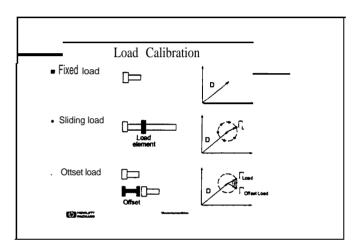
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These are examples of algorithms that may be used to convert the measured S-parameters to permittivity or permeability. The first three require a two-port fixture. The last two require a one-port fixture which may be better for liquids or powders where a shorted waveguide section can be turned on end and filled. One-port fixtures may also be better for measurements at high temperatures where one end of the waveguide can be heated while cooling mechanisms keep the network analyzer cool.

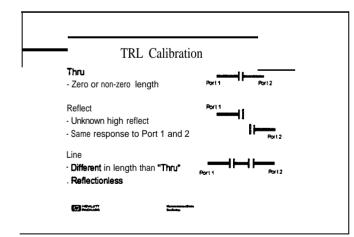
The Nicolson-Ross algorithm is best for magnetic materials. The Precision algorithm is best for the highest accuracy. The Fast algorithm is similar to the Precision, but it is faster. The Short-backed algorithm is best for liquids or powders. The Arbitrary-backed algorithm is best for thin films.

The Nicolson-Ross algorithm suffers from periodic dropouts at every half wavelength when the reflection from the front face of the sample cancels out the reflection from the back face. The Precision and Fast algorithms do not suffer from these discontinuities.





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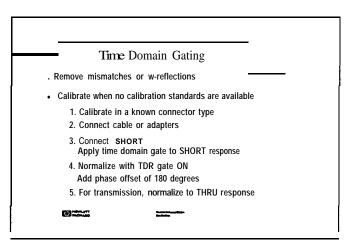
21

The network analyzer must be calibrated before making a measurement to remove the systematic errors from the system. The simplest calibration is a frequency response calibration because it requires only one calibration standard. One-port fixtures require a one-port calibration to compensate for all three error terms in a reflection measurement. Two-port fixtures require a more time-consuming two-port calibration for the greatest accuracy in removing all twelve error terms in a transmission and reflection measurement.

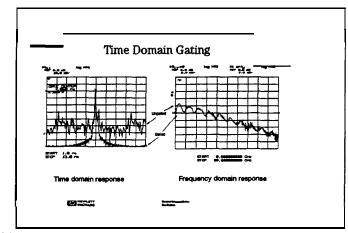
There are three types of loads that can be used in a calibration to solve for the directivity error term. The simplest is a fixed load where the directivity error is directly related to the measurement of the load. A sliding load traces out a circle around the point of the directivity vector as the load element is moved along the airline and the center of the circle is calculated. An offset load requires two measurements, one with a fixed load and the second with the same fixed load offset by a specific length. By knowing two points and the angle between them, the center of the directivity circle can be calculated.

A TRL or Thru-Reflect-Line calibration is the most accurate type of two-port calibration. The Thru can be a zero length or non-zero length Thru that connects port 1 to port 2. The Reflect can be any unknown high reflection device as long as it presents the same high reflection to port 1 and port 2. The Line must be different in length from the Thru and is assumed to be reflectionless.

TRL C	alibratio	n Residual	Errors	
Fewer know	n standard	s required		
Simple star	ndards (esp	ecially for no r	n-coaxial me	dia)
Highest pre	ecision			
Residual Errors	Fixed Load	Sliding Load	offset Load	TRL
Directivity	-40 dB	-52 dB	-60 dB	-60 dB
Match	-35 dB	-41 dB	-42 dB	-60 dB
Tracking	0.1 dB	0.047 dB	0.035 dB	0 dB



23

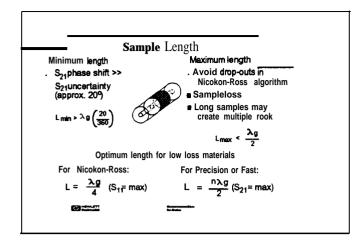


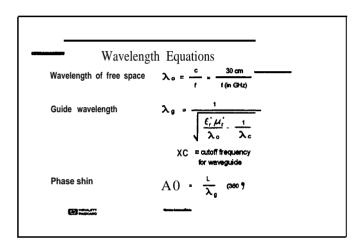
24

A TRL calibration requires fewer standards which do not have to be well known. It offers the highest precision of any network analyzer calibration. The HP 85 10 is the only network analyzer that has a true TRL calibration. The HP 8720 has a modified version of TRL called TRL* that is not recommended for the transmission line technique since it requires a well matched device.

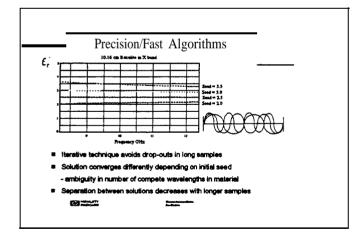
Time domain is available as Option 010 on HP vector network analyzers. Rather than viewing data in the frequency domain it can be converted to the time domain using an inverse fast Fourier transform. Time domain gating can be equated to a bandpass filter in time that removes mismatches or re-reflections outside of the gate. This can also be a valuable tool when calibration standards are not available.

When no calibration standards are available, first calibrate in a known connector type then connect the needed cables or adapters. **Connect** a short circuit at the reference plane and place the time domain gate around it. In the frequency domain normalize to the gated response and offset the phase by 180 degrees.





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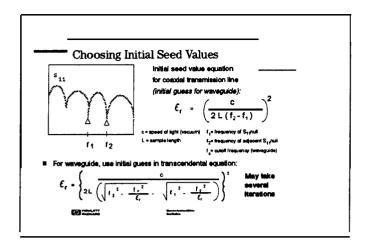


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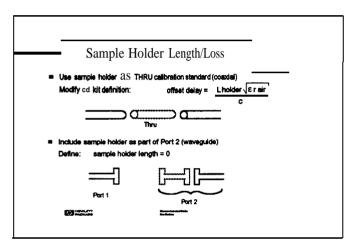
Choosing the optimum sample length will improve the accuracy of a measurement. The minimum length of the sample is limited by the phase uncertainty of the network analyzer. The maximum sample length is limited by the 1/2 wavelength dropouts with the Nicolson-Ross technique, the sample loss and by the fact that long samples may lead to multiple roots. The optimum sample length depends on the chosen algorithm.

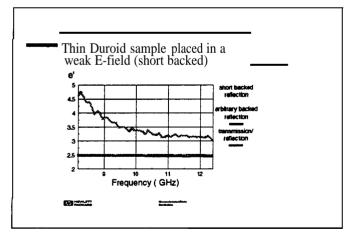
The wavelength in free space is dependent on the speed of light divided by the frequency. The wavelength in the transmission line media is dependent on the permittivity and permeability of the sample, the wavelength in free space and the cutoff wavelength (neglect for coaxial). The phase shift through the sample is dependent on the length of the sample divided by the guide wavelength.

The Precision and Fast algorithms are iterative techniques that do not have the l/2 wavelength dropout problem of the Nicolson-Ross model. However, it may be possible for the solution to these algorithms to converge differently if there is an ambiguity in the number of complete wavelengths that might be present in the sample length.







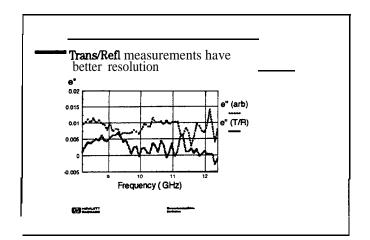


30

Iterative algorithms require an initial seed value. The equation to compute an initial seed value for coaxial fixtures can be determined by locating the nulls in the S_u frequency response. This same equation can be used as an initial guess for waveguide fixtures. That initial guess can then be used in the transcendental equation (which may take several iterations to converge).

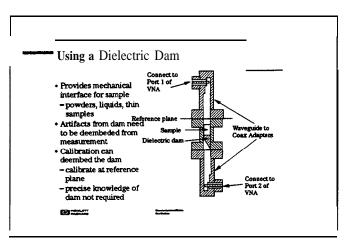
The accuracy of a measurement can be improved by accounting for the loss of the sample holder which is assumed negligible. For coaxial fixtures, the sample holder can be defined as part of the Thru standard by modifying the offset delay in the calibration kit definition. For waveguide fixtures, the sample holder can be included as part of port 2 during the calibration, such that the calibration standards are inserted between port 1 and the sample holder. In this case the sample holder length must be defined to have zero length.

Measurements of 59.3 mil Duroid: In a short-backed l-port transmission line, the E-field must approach zero near the short. Therefore, thin samples have a small impact on the reflection coefficient, causing large errors in permittivity. More flexible algorithms permit the sample to be positioned against an arbitrary impedance, allowing the user to maximize the E-field in the sample. Measurement of the same sample offset from the short by a Teflon spacer (arbitrary backed reflection) closely matches that of a transmission/reflection measurement.

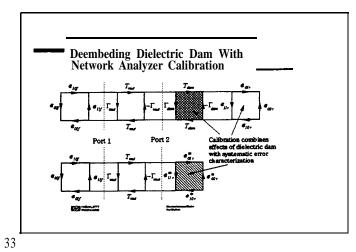


Transmission/Reflection measurements provide better resolution than reflection measurements alone. This graph shows the loss factor measurement of the 59.3 mil Duroid sample. Better resolution is obtained with a transmission/reflection measurement. The dynamic range of a reflection measurement is limited by the directivity (about 40-50 dB in this case) while the dynamic range of a transmission measurement is limited by the crosstalk (about 100 dB in this case).

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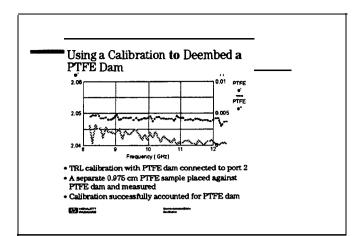


32



Short backed measurement fixtures are often used for the measurement of liquids and powders because their structure is ideally suited for sample containment. A dielectric dam can be used with transmission/reflection measurements to provide the physical containment of the sample. The reflections and transmission loss and delay of the dielectric dam must be deembeded or removed from the transmission and reflection measurement before the sample permittivity and permeability can be calculated.

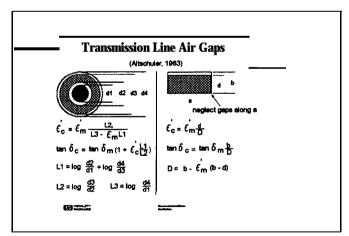
If the characteristics of the dam (permittivity and dimensions) are accurately known they can be mathematically removed or deembeded. It is also possible to deembed the effects of the dam without knowing the permittivity or dimensions of the dam. This can be done by having the dam present during the calibration. The s-parameters of the dam required for deembeding will be combined with the systematic error terms during the vector error correction resulting in the appropriate deembeding for subsequent measurements.



The effectiveness of using a dielectric dam is demonstrated with this measurement. A PTFE dam is present during the network analyzer calibration. After calibration another piece of PTPE is placed against the dam and measured. Physically there will not be a reflection from the sample/dam interface because they are the same material but mathematically there is a reflection at the sample/dam interface which enables the data reduction algorithm to correctly determine the permittivity of the measurement sample.

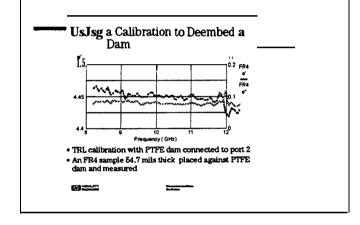
A thin sample was placed against the Teflon dam and measured. The dam insured that the sample was perpendicular to the longitudinal axis. This technique has also been useful in measuring powders and semi-solids.

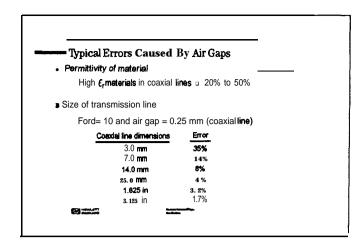
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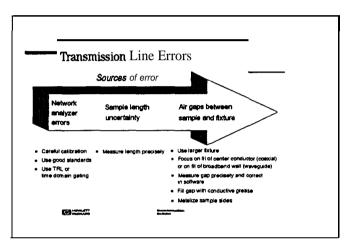


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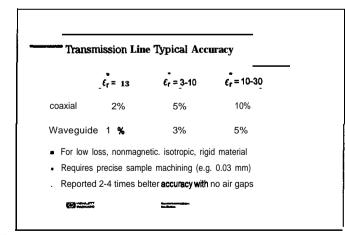
Air gap between the fixture walls and the sample can be one of the largest sources of error in a transmission line measurement. For coaxial fixtures, the air gap along the center conductor wall has a much bigger effect than the air gap along the outer conductor wall. Likewise, for waveguide fixtures, the air gap along the long wall has a much bigger effect than the air gap along the short wall. Air gap correction algorithms can improve the accuracy of a measurement **if** the air gap is uniform and can be precisely measured.







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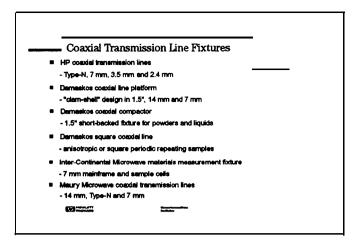


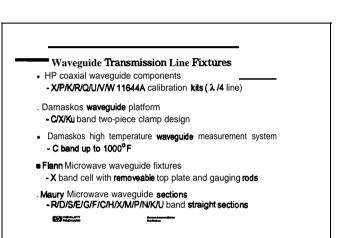
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Materials with higher permittivities and smaller diameter transmission lines will be more susceptible to error from air gap.

Even after calibrating the network analyzer there is additional measurement uncertainty introduced by sample length uncertainty and air gap between the sample and fixture walls. These effects can be minimized but never completely removed.

The typical accuracy of a transmission line measurement ranges from 1% to 10% or higher depending on the MUT and how well it is machined.

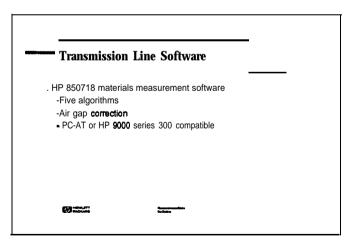




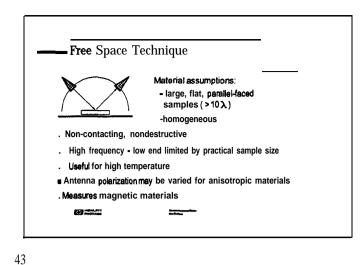
The HP 8505X-series coaxial verification kits contain airlines that can also be used as sample holders. There are several third party suppliers of coaxial transmission lines that provide fixtures for a wide variety of applications.

The HP 11644A-series waveguide calibration kits contain a l/4 wavelength line and a straight section that can also be used as sample holders. There are several third party suppliers of waveguide transmission lines that provide fixtures for a wide variety of applications.

41**L**



The HP 85071B materials measurement software controls the network analyzer and converts the measured S-parameters to permittivity and permeability. It has five different algorithms to choose from (one-port and two-port) and has an air gap correction algorithm. The software is compatible with a wide variety of HP vector network analyzers.



Free Space Methods

S-parameter (reflection/transmission)

Open (Fabry-Perot) resonator

• RCS (Radar Cross Section)

Reflection

. NRL arch Transmission . Tunnel

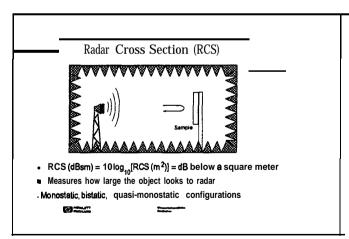
Cavity

0

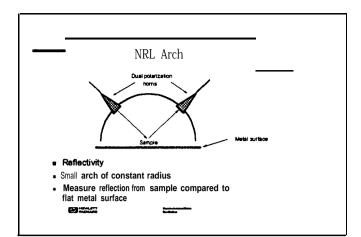
Free space is best for high temperature measurements since the sample is not enclosed in any kind of fixture. The MUT is assumed to be large, flat and uniform throughout. The free space antennas are connected to a vector network analyzer that measures the reflection and transmission from the MUT which are then converted to permittivity and permeability.

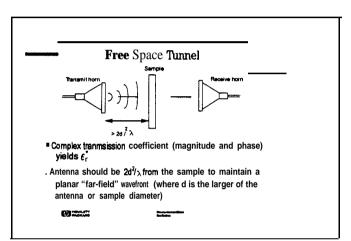
There are many free space measurement methods available to choose from.

44



Radar cross section measurements determine how large an object looks to a radar.

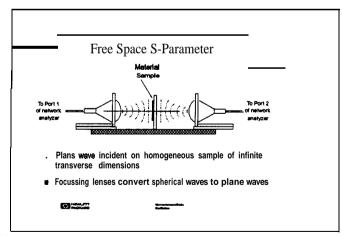




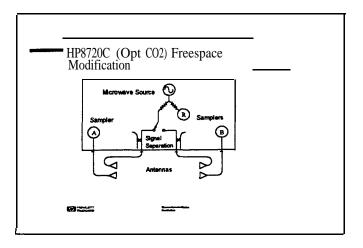
An NRL arch measures the reflectivity of a sample.

For a free space tunnel, the sample is placed between the transmit horn and the receive horn. The received signal is measured and converted to permittivity. The antenna should be placed at least **2d**/ away from the sample to ensure a planar far-field wavefront at the sample.

47



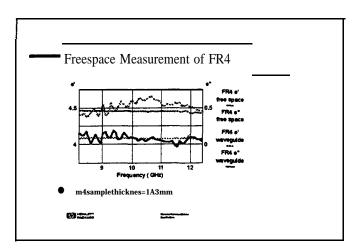
An S-parameter configuration offers several advantages in the area of calibration to provide a more accurate measurement. By measuring all four S-parameters, a TRL (Thru-Reflect-Line) or TRM (Thru-Reflect-Match) calibration can be used. Some systems incorporate focusing lenses into the antenna that convert spherical waves to plane waves. This allows the antenna spacing to be closer and the sample size to be smaller.



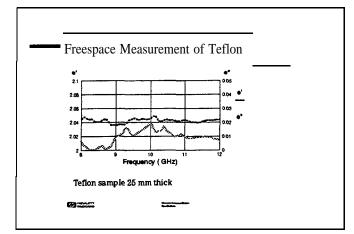
Using the same antenna for transmit and receive degrades directivity, particularly if non-focused antennas are used. The internal reflections can easily dominate the desired signal if the same antenna is used for transmission and reception. Using separate transmit and receive antennas overcomes this problem yielding better directivity. The HP872OC option CO2 provides front panel access to the samplers allowing afull s-parameter test set to be constructed in free space. This allows the transmission/reflection algorithms in the HP8507 1B to be used directly with the free space setup.

This plot shows measured **results** on FR4 (1.43 mm thick) using both the free space and waveguide techniques. The residual systematic errors after calibration are still lower in the waveguide measurement.

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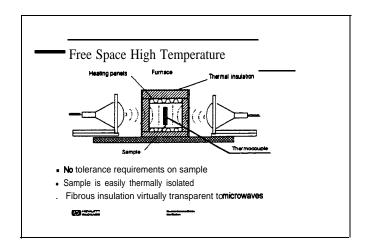


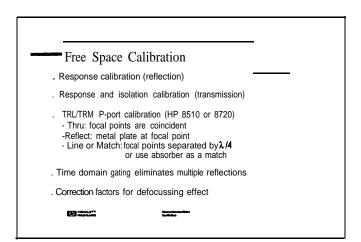
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The effects of systematic errors are reduced for thicker materials. The results for 25mm thick Teflon are more consistent than the freespace results of the 1.43 mm thick FR4. Measurement of thicker samples "distributes" the systematic errors because the systematic error becomes a lower percentage of the sample response.

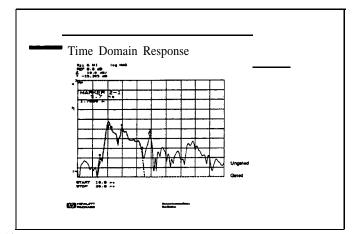




High temperature measurements are not a problem in free space since the sample is never touched or contacted. The sample can be heated by placing it within a furnace that has "windows" of insulation material that are transparent to microwaves.

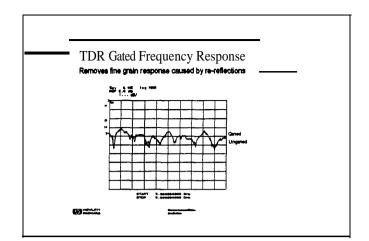
Free space calibration standards present special problems since they are "connectorless". A calibration can be as simple as a response calibration to a full two-port calibration depending on the convenience and accuracy desired. A TRL (Thru-Reflect-Line) or TRM (Thru-Reflect-Match) calibration may actually be easier than other calibration techniques in free space. Time domain gating is often used to take the place of or supplement an existing calibration.

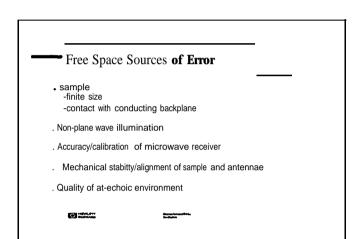
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A time domain response shows the mismatch effects which may be degrading the quality of a free space measurement. If a time domain gate is applied around the signal of interest, many of the unwanted re-reflections can be eliminated.

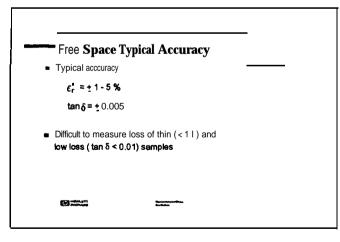




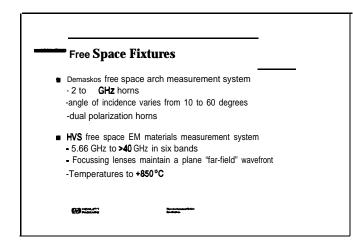
Comparing the gated versus nngated response in the frequency domain shows how mismatch effects can add unwanted fine grain ripple to a free space measurement.

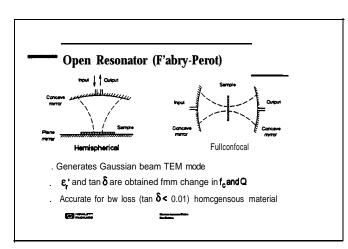
Free space measurements are susceptible to errors due to finite sample size and non-plane wave illumination. Care should be taken to minimize these effects.

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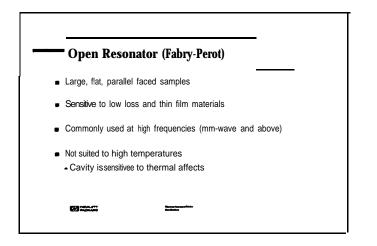


The typical accuracy that can be achieved with a free space measurement is under 5%. It is still difficult to measure very thin and low loss samples.





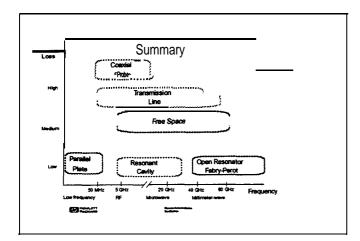
59

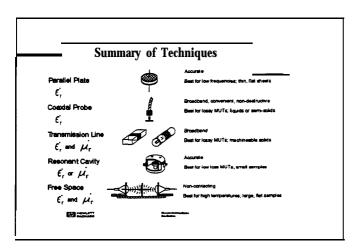


There are several third party suppliers that provide free space antennas and hardware that can be used with HP **network analyzers**.

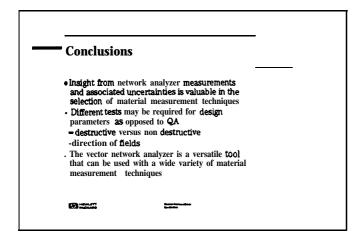
An open resonator (often called the Fabry-Perot technique) is best for measurements at very high frequencies and is very accurate especially for low loss materials.

The open-resonator is a combination of a free space and cavity technique. It measures large, flat samples with greater accuracy at higher frequencies.





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Insight gamed *from* and understanding of network analyzer and associated uncertainties is valuable in the design and selection of material measurement techniques. The technique chosen depends on a

The different measurement techniques are mapped out according to frequency range of operation and suitability to the MUT. lossiness.

Other factors such as accuracy, convenience and the material shape and form are also factors in selecting a measurement technique.

analyzer and associated uncertainties is valuable in the design and selection of material measurement techniques. The technique chosen depends on a variety of factors and may differ depending on the intended use of information. For example, it would be important to insure that the field directions matched the final application when measuring anisotripic materials with the intent of obtaining design parameters. If the intent is to check for consistency field direction may be less important. The wide variety of techniques presented can all be implemented using a vector network analyzer.

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Cavity Resonator	Kent ASTM D 2520, Dube Hill Nishikawa (MuRata)	Split, Non-Destructive		
Coax Probe	HP 85070 Fan, Misra	Metal-Backed		
Dispersion	Gonzalez York	Evaluation, Review		
Full Sheet Resonance	Woolaver York Howell Napoli Ladbrooke	Closed Edges Original Method (1971) Error Analysis & Correction		
Lumped Capacitance	Iskander			
Resonant Probe	Wang	Non-Destructive, Localized		
Review of Methods	York 01yphant			
Standard Method Strip Lines	ASTM D3380, IPC 650 Das Peterson Riedell			
Strip Resonators	ASTM D3380, IPC 650 Bogatin Mayercik Gipprich Tanaka	2-Port Sttipline 1-Port Microstrip, Time Domain Ring Ring, 2-Port Line Stripline, Loss Measurements		
Waveguide Slab	York Bahl			
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Professional Societies

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