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FREE-SPACE MICROWAVE MEASUREMENT TECHNIQUE FOR COMPOSITE MINERALS

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11.1 Introduction

In aeronautics and aerospace industries, greatest progress in material sciences has been realized with the use of composite materials.

Generally, the composite materials are made of spherical particles or non spherical particles embedded in a resin matrix, fiber reinforced thermosetting resin, or dielectric, conducting woven fabrics. Various shaped and sized, metallic, carbon, dielectric, conducting polymer, magnetic and chiral products are used as fillers. Various structures have been designed and realized using multilayered structures of one or more composite layers described above.

In the microwave range, the applications extent from the transparent materials such as radomes or electromagnetic windows to absorbing media.

For all applications, the complex propagation constants and the complex permittivity and permeability of the heterogeneous media are needed. The knowledge of absorption, reflection and transmission coefficients versus the frequency, polarization and incident angle gives important information on the properties of the materials and permit classification of the media as transparent, lossy or absorbing.

During the last few years intensive research has been performed in the area of modeling and characterization of heterogeneous structures in order to predict their electromagnetic properties.

Composite materials can be considered as a discrete random media consisting of a random distribution of scatterers embedded in a homogeneous background (or resin matrix medium). Size and shape and volume fraction of each constituent have major effects on complex effective permittivity and permeability of the mixture. As the frequency and size are increased, higher order scattering and scattering effects become important and must be taken into account.

During the last two years, many conferences have been devoted to the modeling of composite and complex structures. PIERS 89, in Boston, [1-9] included a session on electromagnetic wave propagation in dielectric, magnetic or chiral composite materials. The Orlando 1990 SPIE Meeting had a session on modeling of composite materials [10-15], during the Electro-Optical Materials for Switches, Coatings, Sensor-Optics and Detectors Symposium (SPIE 1307). During the San Francisco 1990 MRS meeting, a special symposium was held on the physical phenomena in granular materials. The authors have attempted to analyze the electrical transport, structural phenomena and the effective medium theories in various mixtures [16-25]. All these studies are oriented towards deriving mixture laws to permit deduction of the effective permittivity and permeability of heterogeneous composite media.

On the other hand intensive effort was also directed to characterize the main properties of composite materials. During the Microwave Processing of Materials Symposium at 1990 MRS Spring Meeting, a session on Dielectric Properties and Measurements was held (session L10, references 26-33; 35-37). Two interesting overviews on the various methods for characterization of composite materials were made by Moore [38], at the International Conference on Coatings and Sensors, PennState May 9-11, 1989 and by Afsar [9], during the PIER Symposium, MIT, July 25-29, 1990. Previous publications of Afsar [73] for

millimeter wave dielectric properties, Chantry [74] for microscopic and macroscopic theories of dielectric materials and Moore [83–91] for various measurement techniques are, also, very useful papers that need to be consulted.

With regard to homogeneous media, numerous measurement methods suitable for measurements of complex permittivities and permeabilities have been given in the books edited by Von Hippel [39–41]. These remain the reference works in measuring dielectric and magnetic constants. The widely used methods include transmission line and waveguide methods, cavity measurements, cavity perturbation techniques, interferometric techniques and time domain reflectometry techniques [42]. They are limited frequency band measurements, permitting the determination of the complex permittivity and permeability at fixed frequencies and sometimes versus temperature variations.

Concerning heterogeneous media, the measurements techniques are extensions of the methods derived for homogeneous materials. Despite the first presentation of the network analyzer in 1965, we had to wait until 1974 to see the large band measurement techniques appear. After ameliorations on the accuracy and the development of Von Hippel's methods, the first data treatments have been proposed. Weir [43] used the reflection and transmission coefficients resulting when a test sample was inserted into a waveguide or a TEM transmission line. From measurements, complex permittivity and permeability values have been derived in the range from 100 Mhz to 18 Ghz. A generalization of the Von Hippel method in the waveguide has been made by Ghanen, Roussy and Thiebault [44]. By continuous measurements of reflection coefficients as a function of the position of a short circuit located behind the sample and a suitable numerical treatment of the experimental data, the authors improved the accuracy of the determination of the complex permittivity, reducing random experimental errors. They also extended the methods for measurements of complex permittivity and permeability. Other automatic wideband measurements of anisotropic fluids have been done by Parneix, Legrand and Toutain [45]. In 1985 Hewlett Packard published a product note related to measuring dielectric constant with HP 8510 network analyzer [46]. In 1986, Barry [47] used the S-parameters of a waveguide or stripline cell loaded with the test material to calculate the complex permittivity and permeability. Characterization of ceramics from 0.5 Ghz to 5.5 Ghz has been made.

Coaxial lines terminated by a gap, the gap being filled with an unknown material are also studied. Marcuvitz [34] found an approximate solution by using the small-aperture method treating all higher modes by plane wave approximations. Using the mode-matching method, in which the fields on each side of the coaxial-cylindrical discontinuity are expanded in an infinite series of modes matched accross the boundary to preserve continuity, a general formulation was proposed by Belhadj and Fourier-Lamer in 1986 [48–49]. A wide band cell was built for measuring isotropic materials or liquids from 100 Mhz to 12.4 Ghz. Extension of the mode matching method to define the different modes excited in a discontinuity loaded by a material having complex permittivity and permeability properties allowed Belhadj, Fourier-Lamer and de Chanterac to derive the permittivity and permeability values from S11 and S21 measurements [50].

Among other works, we would like to mention :

- the broad band measurement of dielectric and magnetic complex susceptibilities with TEM stripline cell by Fessant, Gieraltowski, Loaëc and Legall [51].
- the measurements of microwave conductivity and dielectric constant by cavity perturbation method by Chao [52].
- the measurements in the time domain of the transient response to subnanosecond pulses from a dielectric material associated with a fourier transform algorithm to determine the complex permittivity and permeability by Nicholson and Ross [53].
- the transmission line method for complex permittivity measurements of a dielectric sheet sample inserted in a rectangular waveguide through a longitudinal slot (waveguide slab method). The method has been automated fully using a personal computer and an network analyzer by York and Compton and is working in X-band [54].

With waveguide or coaxial transmission line, cavity perturbation, or resonance methods, the dielectric sample must be prepared in a certain shape and the dimensions of the sample must be accurately known and fit perfectly the cross-section of the transmission lines in order to avoid significant errors. Cutting, preparing and measuring samples is not consistent with continuous and real time control of materials.

Three additional main methods are observed in the literature.

The first one is related to the use of the open ended coaxial probe or sensor. First results were reported, in 1976 by Tanabe and Joines

[55]. Open-ended coaxial line sensors, commonly used with network analyzers and other reflectometers for non-invasive measurements of complex permittivity at RF and microwave frequencies, have been developed by various authors who have made improvements in the numerical treatments and improved calibration techniques [56–65]. With proper choice of calibration standards and the use of the formulated admittance model, the complex permittivity of various unknown materials can be determined over a wide range of frequencies (200 Mhz–20 Ghz). In 1990 Hewlett-Packard is proposing an open-ended probe used with the 8720 network analyzer [37].

In the second one, a swept frequency, one-horn interferometer method has been proposed recently by Baker, and Van der Neut [66]. The absorber panel to be tested is placed directly against the aperture of a well matched horn. The measurement procedure has the advantage that small areas of planar absorber panels can be evaluated without cutting the panels. Moreover with proper selection of waveguide components and the aperture matching of an exponential horn, very low levels of reflections can be obtained ($< -40\text{dB}$).

The third one is related to the use of free-space microwave measurement techniques of plane composite materials. This technique was first mentioned in 1948 and after in 1963 [42 a,b] and abandoned for several years due to the lack of accuracy and the difficulty of having large samples for testing. Pentecost and Grace [67] have mentioned this technique for the electrical evaluation of radome materials under high temperature. In 1986, Musil and Zacek [68] published a book related to microwave measurements of complex permittivity by the free-space method. Ho [26] is using this technique in the millimeter range for the characterization of dielectric materials under temperature variations and superconducting materials. In the microwave range, various versions of this method have been used from 4 to 20 Ghz for the characterization of composite material or chiral composites [27], [30], [38], [67–72]. The method is sensitive to thickness variations and the accuracy of the method is limited by the accuracy of the receiver ($\pm 0.05\text{ dB}$ in amplitude and $\pm 1^\circ$ in phase depending on the measurement errors on S-parameters)

Many times, free-space methods are preferred over waveguide, coaxial, cavity, one-horn interferometer or open-ended coaxial probe for the following reasons:

- ceramics and composite materials are inhomogeneous due to their

manufacturing processing. In waveguide, coaxial or cavity techniques, higher-order modes can be excited at the interfaces and must be taken into account

- due to their heterogeneity, small composite samples are not representative of the whole material.
- free-space methods are non destructive and contactless. They are suitable for complex permittivity and permeability measurements under high temperature conditions.
- free-space methods require little or no sample preparation. It is not the case in transmission line or in cavity methods.
- with free-space methods, broadband characterization under various incident angles, polarizations and under temperature conditions can be made on isotropic, anisotropic or bianisotropic materials. This technique is the only technique available for measuring chiral media under co-polarization cross and null-cross polarization.

Measurement techniques mentioned before, deduce by inverse methods the effective complex permittivity and permeability of composite materials or their electromagnetic properties. The data collected from these measurements are very important in deducing the behaviour of the material under electromagnetic exposure. For materials following effective medium theories, the measured data can be used as starting data for a better knowledge of the properties of the embedded particles and will be very helpful in modeling the composite materials. The experimental data constitute proof as to whether or not composite materials modeling makes sense. Modeling of complex composite materials is rather difficult, and new explanations are needed to describe the electromagnetic behavior near percolation zone or with analogical structures. Measurement data can furnish guidelines on the behavior of these complex materials.

In this chapter we shall describe a 10–18 Ghz automatic free-space measurement set-up developed in the microwave laboratory and give results on composite measurements. Part II, will be devoted to the uniform plane wave created by various free-space techniques. In Part III, we will give theoretical calculations of complex permittivities and permeabilities using reflection-transmission and two-configuration reflection methods. In Part IV we shall describe the 10–18 Ghz experimental set-up, and in Part V we shall give some experimental results obtained with composite materials.

11.2 Free-Space Technique

In free-space measurements, the unknown sample is located between the transmitting and receiving antennas. The complex permittivity and permeability are derived from the measurements of the amplitude and phase variations of a plane wave transmitted through or reflected by the sample.

A slab with parallel plane surface and an incident electromagnetic plane wave is the simplest model to be used. In this case, it is possible to relate the phase shift and the attenuation of the incident plane wave to reflection and transmission coefficients for various incident angles and polarizations.

To apply the equations relating attenuation and phase variations to the reflection and transmission coefficients, we have to choose sample dimensions so that the edge diffraction effects are negligible. This means that the minimum transverse dimension of the sample is greater than the E-plane 20-dB beam width of the incident plane wave.

An electromagnetic plane wave can be produced by means of a dielectric lens or by metallic reflectors [68], [75]. Spherical waves are supplied by a microwave feed horn. The plane wave is produced by transformation of these spherical waves by the surface of the lens or reflectors. The lens or metallic reflector profile is defined in such a way that an equiphase field is produced by the antenna aperture. The two systems are different only by the feed horn position. With lens systems the feed horn is located in front of the lens and the generated focused beam emerges behind the lens. With metallic reflectors, both the feed horn and the generated focused beam are situated on the same side of the reflector. The shadow effect of the feed horn could be a disadvantage of these systems and has to be minimized for near field measurements.

Frequently, free-space experimental set-ups use plane waves generated by a dielectric lens. The dielectric lens system operation is completely defined by Musil et al. [68] and applied in many laboratories especially in the USA [26, 30, 38, 70, 72]. Because of the bandwidth limitation of the dielectric lens, we chose the focusing metallic reflectors [centered systems, 75]. This technique was applied at Georgia Tech in 1970 and 1973 to measure the temperature permittivity variations of dielectric radome materials at 2500° F [67], [76].

Focused beams are used to reduce the sample dimensions. In each case the width and the depth of the focused beam, as well as its elec-

tromagnetic properties are directly dependent on the electromagnetic field distribution in front of the focusing aperture. The electromagnetic field configuration of a focused beam produced by a circular aperture antenna has been studied by Musil et al. [68]. In the focal plane and at the vicinity of the focus, the phase is practically constant. Therefore the electromagnetic fields in the neighborhood of the focus plane of the focused antenna has plane wave properties. With centered metallic reflector, Drabovitch [75] derives the same behavior and indicates that a gaussian illumination feed horn creates a radiated field having the gaussian property. This last remark will help us to create radiated fields in the focus plane having very low level sidelobes.

Among various available centered systems, we have chosen the ellipsoidal reflectors using at the first focus a gaussian feed horn. With this configuration the cross-section of the feed horn at the focal plane is the smallest, reducing the shadow effect to the minimum. For transmitting and receiving antennas, the same ellipsoidal reflectors with a gaussian feed horn source have been used. The second focus of each ellipsoidal reflector is common in order to get smaller focusing beam dimensions.

The depth of focus has to be calculated to define the sample dimensions and the maximum incident angle of observation. Making the assumption that the field distribution in the neighborhood of the focus is following gaussian beam law, for the half-aperture beam at $1/e$, we have the relation [75], [77], [78], [79] (Fig. 11.1):

$$W = W_0 \left[1 + \left(\frac{\lambda z}{\pi w_0^2} \right)^2 \right]^{1/2} \quad (1)$$

where: λ : wavelength
 z : propagation axis
 and W_0 : the beam waist defined by:

$$W_0 = \frac{\lambda F}{\pi r} \quad (2)$$

where: F : the focus distance
 r : the beam radius on the focusing optic.

Equation (2) is valid only for:

$$F < \frac{\pi r^2}{\lambda}$$

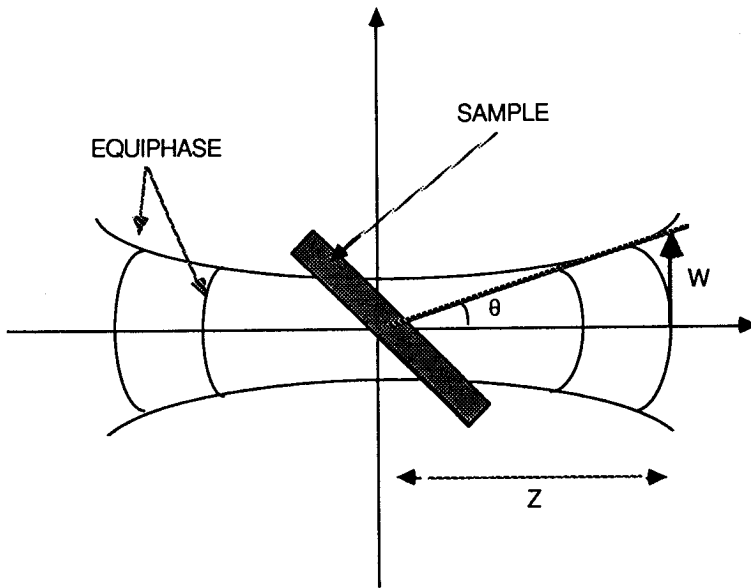


Figure 11.1 Configuration in the focal zone.

The equiphase surface is a plane at the focus and will be distorted as we move away from the focus.

The depth of focus is given by the relation [79]:

$$\Delta Z = \Delta \phi \frac{\pi w_0^2}{\lambda} \quad (3)$$

when $\Delta \phi < 1/2$ radian
 $\Delta \phi$ is the permitted phase shift.

The maximum theoretical incident angle can be calculated from (1-3), and by using Fig. 11.1.

With the geometric dimensions of antennas and focal distance defined in the experimental set-up paragraph, for a 10–18 GHz frequency range working, the 3 dB beamwidth and depth of focus for spot focusing ellipsoidal reflector are 2λ and 5λ respectively. (at 10 dB we have 3λ). From 10–18 GHz, with a $W_0 = 1.43\lambda$, we need sample dimensions of the order of 18×18 cm. The theoretical maximum incident angle will be 65° . In reality the maximum sample rotation will be lower to avoid edge scattering effects.

11.3 Free-Space Method Theory

In Section 11.2 we have seen that gaussian feed horn ellipsoidal reflectors can create quasi-plane waves near the common second focus point of each reflector.

Reflection (R) and transmission (T) complex coefficients are functions of complex permittivities and permeabilities of each layer constituting the whole sample. If one of the layers or the whole sample has unknown properties, the characterization of their properties needs to use inverse procedures based on two experimental measurements of complex R and T coefficients. It is possible to determine these properties from measurements of complex R and T coefficients.

Now, we are interested in the calculation of complex permittivities and permeabilities of homogeneous materials from the measured reflection and transmission coefficients.

Two theoretical methods are available.

a. Two-Configuration Reflection Method

First, let us recall that in a waveguide configuration loaded with an unknown material backed by a short-circuit and open-circuit, Von Hippel [39–40] has derived complex permittivity and permeability values from amplitude and phase reflection measurements. The same technique can be transposed to free-space measurements.

An equivalent technique consists of measuring for some polarization or other the complex reflection coefficient for a material backed by a metal plate and for a material backed by a well known dielectric material, which is in turn backed by a metal plate. The thickness of the dielectric plate is equal to $(\lambda/4) \cos \theta$ (θ being the incident angle). This thickness appears as a limitation, because only measurements for a reduced frequency band and incidence angle range can be made.

A generalization of the Von Hippel method using a known complex permittivity and permeability of the second layer with thickness d_2 has been proposed by Priou et al. [69], [71], [81]. The authors have also shown that another possibility is a composite media backed by a metal plate and the use of two polarizations to measure the reflection coefficient and deduce the complex permittivity and permeability [81]. However this last method is rather difficult to apply. It needs very accurate amplitude and phase reflection coefficient measurements versus frequency and incident angles. A great dispersion in the obtained

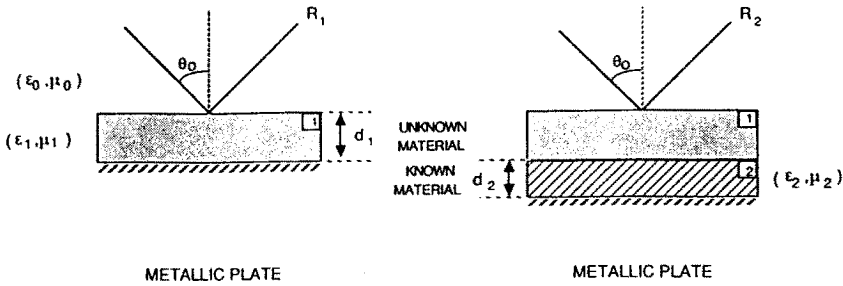


Figure 11.2 Two-configurations reflection method.

results versus incident angle variations is commonly observed.

The approach we use consists of two independent measurements of the complex reflection coefficient (Fig. 11.2) :

- the measurement of an unknown material plate of thickness d_1 backed by a metal plate (R_1).
- the measurement of a multilayered structure (R_2). A material with known permittivity and permeability is interposed between the composite material and the metal plate and has thickness d_2 .

We assume that each material is defined by its respective relative complex permittivity and permeability and its respective thickness. Planes waves with θ incident angle are impinging on the material. Using the matrix formulation developed by Kong [80] for multilayered structures, we get the following expressions:

$$\begin{vmatrix} 1 \\ R_2 \end{vmatrix} = B_{01} B_{12} \begin{vmatrix} A_2 e^{-jk_{z2}d_2} \\ C_2 e^{+jk_{z2}d_2} \end{vmatrix} \quad (4)$$

where:

$$\begin{aligned} \epsilon_i, \mu_i &: \text{electromagnetic properties of } i \text{ layer} \\ k_{z0} &= k_0 \cos \theta_0, \quad k_0 = \text{free space wavenumber} \\ k_{zi} &= k_i \cos \theta_i = k_0 \sqrt{\epsilon_{ri} \mu_{ri} - \sin^2 \theta_i} \end{aligned}$$

- d_1 = thickness of the unknown media
 d_2 = thickness of the known material
 R_2 = reflection coefficient
 A_2, C_2 : constants to be determined

$$B_{01} = \frac{1}{2} \alpha_{01} \begin{vmatrix} e^{jk_{z1}d_1} & R_{01}e^{-jk_{z1}d_1} \\ R_{01}e^{jk_{z1}d_1} & e^{-jk_{z1}d_1} \end{vmatrix} \quad (5)$$

$$B_{12} = \frac{1}{2} \alpha_{12} \begin{vmatrix} e^{jk_{z2}d_2} & R_{121}e^{-jk_{z2}d_2} \\ R_{12}e^{jk_{z2}d_2} & e^{-jk_{z2}d_2} \end{vmatrix} \quad (6)$$

with perpendicular polarization we have:

$$\alpha_{01} = 1 + \frac{\mu_0 k_{z1}}{\mu_1 k_{z0}} \quad (7)$$

$$\alpha_{12} = 1 + \frac{\mu_1 k_{z2}}{\mu_2 k_{z1}} \quad (8)$$

$$R_{01} = \frac{\mu_1 k_{z0} - \mu_0 k_{z1}}{\mu_1 k_{z0} + \mu_0 k_{z1}} \quad (9)$$

$$R_{12} = \frac{\mu_2 k_{z1} - \mu_1 k_{z2}}{\mu_2 k_{z1} + \mu_1 k_{z2}} \quad (10)$$

with parallel polarization:

$$\alpha_{01} = \left(1 + \frac{\epsilon_0 k_{z1}}{\epsilon_1 k_{z0}}\right) \sqrt{\frac{\epsilon_0 \mu_1}{\epsilon_1 \mu_0}} \quad (11)$$

$$\alpha_{12} = \left(1 + \frac{\epsilon_1 k_{z2}}{\epsilon_2 k_{z1}}\right) \sqrt{\frac{\epsilon_1 \mu_2}{\epsilon_2 \mu_1}} \quad (12)$$

$$R_{01} = \frac{\epsilon_0 k_{z1} - \epsilon_1 k_{z0}}{\epsilon_0 k_{z1} + \epsilon_1 k_{z0}} \quad (13)$$

$$R_{12} = \frac{\epsilon_1 k_{z2} - \epsilon_2 k_{z1}}{\epsilon_1 k_{z2} + \epsilon_2 k_{z1}} \quad (14)$$

In the case of the last layer backed by a metal plate, relation (4) changes as:

$$\begin{vmatrix} 1 \\ R_2 \end{vmatrix} = A_2 e^{-jk_{z2}d_2} B_{01} B_{12} \begin{vmatrix} 1 \\ -1 \end{vmatrix} \quad (15)$$

So we get the relation for R_2 :

$$R_2 = \frac{R_{01}e^{2jk_{z1}d_1}(e^{2jk_{z2}d_2} - R_{12}) + R_{12}e^{2jk_{z2}d_2} - 1}{e^{2jk_{z1}d_1}(e^{2jk_{z2}d_2} - R_{12}) + R_{01}(R_{12}e^{2jk_{z2}d_2} - 1)} \quad (16)$$

In order to simplify the equations and by analogy with the formalism used in waveguide methods, we introduce the free-space normalized impedance defined by the relation:

$$z = \frac{1 + R}{1 - R} \quad (17)$$

The same approach was made also by Cullen [82], for the determination of complex permittivity and permeability of ferrite at microwave and millimeter wave frequencies.

For the two configurations under consideration (Fig. 11.2), we get:

– for the first configuration:

$$z_1 = j z_{01} \tan(k_{z1} d_1) \quad (18)$$

– for the multilayered structure (second configuration):

$$z_2 = j z_{01} \frac{\tan(k_{z1} d_1) + z_{12} \tan(k_{z2} d_2)}{1 - z_{12} \tan(k_{z1} d_1) \tan(k_{z2} d_2)} \quad (19)$$

Z_{01} and Z_{12} are wave impedance ratio between two media (Fig. 11.2). Z_1 , Z_2 are known quantities deduced from the measurements of R_1 and R_2 and can be used for the two polarizations.

In the previous equations three terms are unknown: $K z_1$, Z_{01} , Z_{12} .

In fact the last two terms represent the same quantity multiplied by a factor. With the general formulation of the Z_{ij} , for the two polarizations we have the relation:

$$Z_{ij} = Z_{ik} \times Z_{kj}$$

Applied to our problem we derive:

$$Z_{01} = \frac{Z_{02}}{Z_{12}} \quad (20)$$

From (18–20), Z_{12} can be derived:

$$Z_{12} = \sqrt{Z_{02}} \sqrt{\frac{Z_{02} + j(Z_2 - Z_1) \cot g(k_{z2} d_2)}{Z_1 Z_2}} \quad (21)$$

Also Kz_1 can be derived in the same manner:

First we define the quantity Z as:

$$Z = -j \frac{Z_1 Z_{12}}{Z_{02}}$$

So we get:

$$e^{2jk_{z1}d_1} = \frac{j - z}{j + z} = A \quad (22)$$

from which we derive:

$$\alpha = \frac{\log_n |A|}{2d_1} Np/m \quad (22a)$$

$$\beta = \frac{\text{Arg}(A)}{2d_1} rd/m \quad (22b)$$

$$(kz_1 = \beta - j\alpha)$$

From (21-22b), we deduce for each polarization the complex permittivity and permeability of the unknown material. For perpendicular polarization we get the following expressions:

$$\mu_{r1} = \frac{k_{z1}}{k_{z2}} \times \frac{\mu_{r2}}{z_{12 \text{ perp.}}} \quad (23)$$

$$\epsilon_{r1} = \frac{\left(\frac{k_{z1}^2}{k_0^2} + \sin^2 \theta_0 \right)}{\mu_{r1}} \quad (24)$$

For parallel polarization :

$$\epsilon_{r1} = \frac{k_{z1}}{k_{z2}} \times z_{12 \text{ para.}} \times \epsilon_{r2} \quad (25)$$

$$\mu_{r1} = \frac{\left(\frac{k_{z1}^2}{k_0^2} + \sin^2 \theta_0 \right)}{\epsilon_{r1}} \quad (26)$$

An analytical computer program of errors made on the determination of the complex permittivity and permeability versus R_1 , R_2 , d_1 , d_2 and θ parameters is a rather complicated task because of the complexity of the expressions giving complex permittivity and permeability values.

However an analytical calculation of the $d\epsilon$ and $d\mu$ variations versus the experimental values of complex R_1 and R_2 can be performed. We assume that we have $R_1 = \rho_1 \exp j\phi_1$ and $R_2 = \rho_2 \exp j\phi_2$.

The $d\epsilon$ and $d\mu$ expressions are too awkward to be given here [81]. It is sufficient to describe their behaviour:

- in the numerator, $\cot g(kz_2d_2)$ terms are found. These terms go to infinity for some values of d_2 defined by the relation:

$$d_2 = m \frac{\lambda_2}{\cos \theta_2}$$

So we have to choose thickness values for layer 2 at the central frequency equal to:

$$d_2 = (2m + 1) \frac{\lambda_2}{4 \cos \theta_2}$$

- in the denominator we have z_1 and z_2 terms (18–19) which tends to zero when R_1 or R_2 goes to -1 . So we have to avoid experimental conditions where one of the reflection coefficients has value near -1 . That means we have to use samples the thickness of which is far lower than the wavelength.

The numerical simulation of the error must give an evaluation of the influence of all the parameters ($\rho_1, \rho_2, \phi_1, \phi_2, d_1, d_2$ and θ) on the $\Delta\epsilon$ and $\Delta\mu$ values. The numerical calculation uses a development of functions representing the permittivity and permeability of the following form:

$$\begin{aligned} df = & \frac{\delta f}{\delta \rho_1} d\rho_1 + \frac{\delta f}{\delta \phi_1} d\phi_1 + \frac{\delta f}{\delta \rho_2} d\rho_2 + \frac{\delta f}{\delta \phi_2} d\phi_2 \\ & + \frac{\delta f}{\delta d_1} dd_1 + \frac{\delta f}{\delta d_2} dd_2 + \frac{\delta f}{\delta \theta} d\theta \end{aligned} \quad (27)$$

In the worst case, we can assume that errors of each term can be summed to derive the total error in permittivity and permeability values:

$$\Delta f = \left| \frac{\delta f}{\delta \rho_1} \right| + \left| \frac{\delta f}{\delta \rho_2} \right| \Delta \rho_2 + \dots \quad (28)$$

In the numerical calculation we have chosen absolute error values for $\rho_1, \rho_2, \phi_1, \phi_2, d_1, d_2$ and θ parameters:

$$\Delta d = 0.1 \text{ mm}; \quad \Delta \theta = 0.1^\circ; \quad \Delta \rho = 0.1 \text{ dB}; \quad \Delta \phi = 2^\circ$$

In order to define the optimal experimental conditions for some materials, the influence on the $\Delta\epsilon/\epsilon$ and $\Delta\mu/\mu$ accuracy of ρ_1 , ρ_2 , ϕ_1 , ϕ_2 , d_1 , d_2 and θ geometrical parameters have to be evaluated. In fact the d_2 and θ values were previously chosen to verify the following equation at the central frequency:

$$d_2 = (2m + 1) \frac{\lambda_2}{4 \cos \theta_2}$$

The optimisation will be made essentially on d_1 , the thickness of the test material, in the 10–18 GHz frequency range, with θ varying from 10° to 45° , d_1 lower than 1 cm, and with air for the second known layer having 6 mm thickness. In varying the d_1 thickness from 1 to 10 mm, we have studied the $\Delta\epsilon/\epsilon$ and $\Delta\mu/\mu$ variations versus frequency for materials having $\tan \delta$ varying from 10^{-3} to 2,5 [81].

In Fig. 11.3, the results derived for material having $\epsilon' = 4$ and $\epsilon'' = 0,8$ for $d_1 = 1, 4$, and 10 mm are presented. We can deduce:

- for $d_1 = 1$ mm, $\Delta\epsilon/\epsilon$ and $\Delta\mu/\mu$ variations are equal to within 10% in the whole frequency range.
- for $d_1 = 4$ mm the accuracy is increasing for the real part of the permittivity and permeability variations. However the imaginary parts present maximum values when R_1 and R_2 phase pass through 180° .
- for $d_1 = 10$ mm, the accuracy for the real parts is about 5%, and for the imaginary parts is about 15 to 20%. The maxima due to R_1 and R_2 phase variations are more numerous but are well attenuated due to the thickness of the layer.

Other observations can be deduced in computing the average values of $\Delta\epsilon$ and $\Delta\mu$ over the 10–18 GHz frequency band versus thickness d_1 . In Fig. 11.4, ϵ and μ error variations versus thickness over 10–18 GHz for high loss material are presented. For thickness higher than 2 mm errors are about 5%.

Other remarks:

- the sample under test may have a thickness between 1 mm to 8 mm for transparent material and about of 2.5 mm for lossy materials.
- the maxima due to the passing through 180° of the phase of reflection coefficients can be attenuated if R_1 and R_2 amplitudes are lower than -5 dB.
- far from these maxima areas, $\Delta\epsilon'/\epsilon'$ and $\Delta\mu'/\mu'$ are equal to 10%. For materials having $\tan \delta > 0,1$, $\Delta\epsilon''/\epsilon''$ and $\Delta\mu''/\mu''$ can be

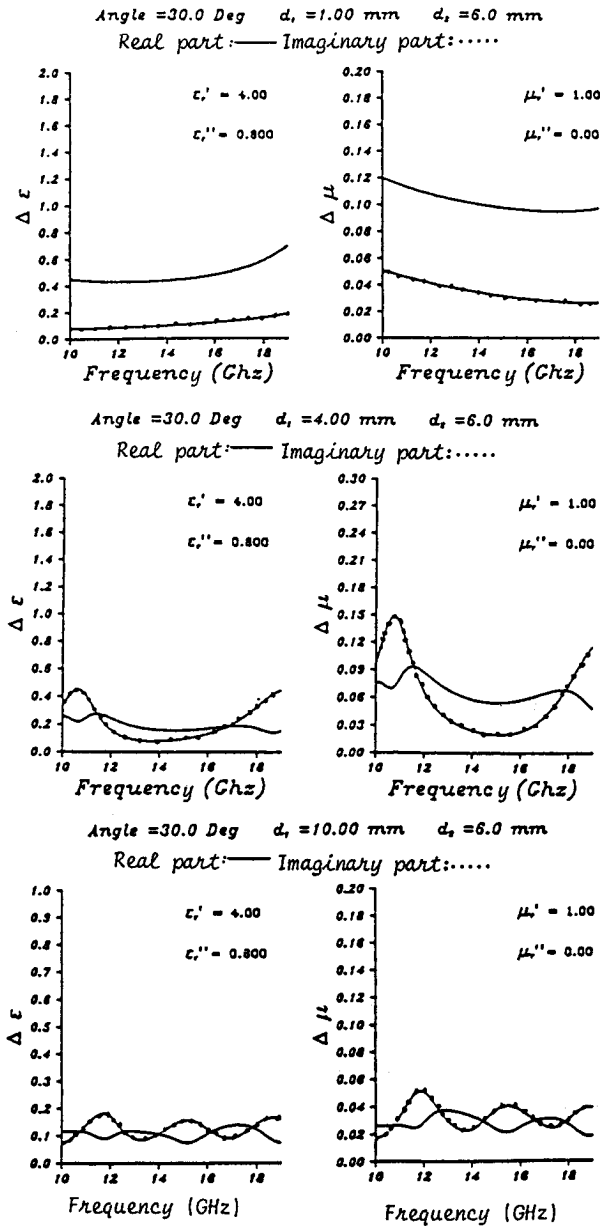


Figure 11.3 Permittivity and permeability error variations versus frequency for 3 D_1 thickness.

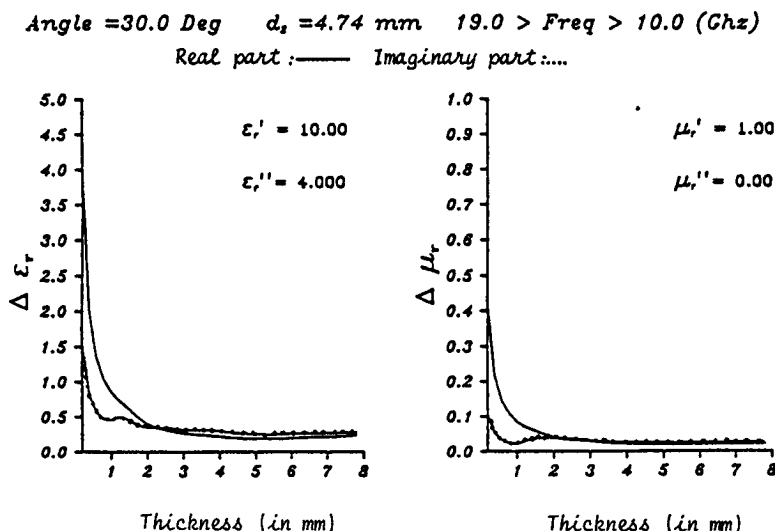


Figure 11.4 Permittivity and permeability error variations versus thickness over 10–18GHz.

better than 10 %. - the characterization accuracy of samples having $\epsilon\mu > 30$ becomes poorer.

b. Reflection-Transmission Method

The technique consists of measuring for some polarization or other the complex reflection and transmission coefficients in frequency and incident angle ranges of a sample having thickness d and permittivity and permeability properties (Fig. 11.5). The same method is used in waveguide techniques. It is a well known method [40]. So we just recall the expressions of the complex permittivity and permeability. Using the same approach developed in the previous chapter and the matrix formulation we get the following equations for transmission and reflection coefficients:

$$T e^{-j d k_{z0}} = \frac{(1 - R_{01}^2) e^{-j d k_{z1}}}{1 - R_{01}^2 e^{-2j d k_{z1}}} \quad (29)$$

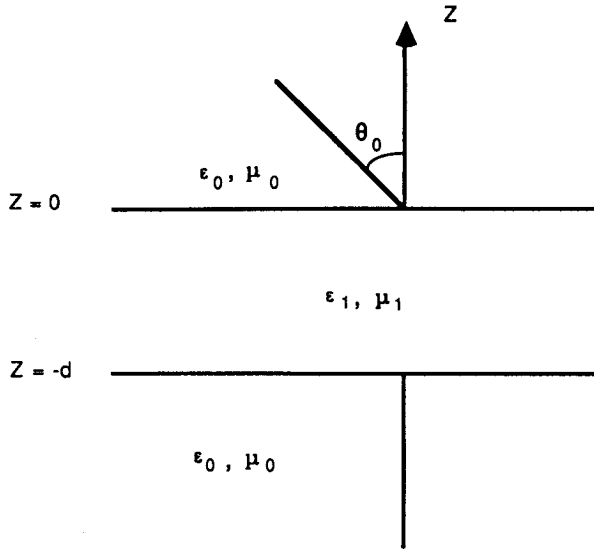


Figure 11.5 R and T configuration.

$$R = \frac{R_{01}(1 - e^{-2jdk_{z1}})}{1 - R_{01}^2 e^{-2jdk_{z1}}} \quad (30)$$

The various parameters are defined in (4) for the wavenumbers and in (8,13) for R_{01} for the two polarizations. From (29–30) we derive :

$$\cos(dk_{z1}) = \frac{1 - R^2 + T^2 e^{-2jdk_{z0}}}{2T e^{-jdk_{z0}}} = B \quad (31)$$

from which we deduce the k_{z1} value:

$$k_{z1} = \pm \frac{AR \cos(B)}{d_1} + 2 \frac{k\pi}{d_1} \quad (31a)$$

$$k_{z1} = \beta - j\alpha$$

R_{01} value can be deduced from (29) or (30) and we get :

$$R_{01} = \frac{R(1 - R_{01}^2 e^{-2jdk_{z1}})}{1 - e^{-2jdk_{z1}}} \quad (32)$$

For perpendicular polarization, from the definition of k_{z1} (4), and (8,31, 31a and b) and (32), we get for permittivity and permeability terms:

$$\mu_{r1} = \frac{k_{z1}(1 + R_{01})}{k_{z0}(1 - R_{01})} \quad (33)$$

$$\epsilon_{r1} = \frac{\left(\frac{k_{z1}^2}{k_{z0}^2} + \sin^2 \theta_0\right)}{\mu_{r1}} \quad (34)$$

And for parallel polarization, with (4, 13, 32) we get :

$$\epsilon_{r1} = \frac{k_{z1}(1 - R_{01})}{k_{z0}(1 + R_{01})} \quad (35)$$

$$\mu_{r1} = \frac{\left(\frac{k_{z1}^2}{k_{z0}^2} + \sin^2 \theta_0\right)}{\epsilon D_{r1}} \quad (36)$$

c. A Computer Algorithm

With (23–26) for the two-configuration reflection method and (33–36) for the reflection and transmission method we get analytical expressions of the complex permittivity and permeability. To solve these expressions, a general computer algorithm described in Fig. 11.6 has been written and implemented on the HP 9000 computer. Figures 11.7 and 11.8 give more information, especially on the resolution of the Kz_1 phase indetermination and on the calculation of the true propagation constant for the two methods.

11.4 Experimental Set-Up

A schematic diagram of the automatic 10–18 GHz free-space measurement set-up is given in Fig. 11.9.

The transmitting and the receiving antennas are spot-focusing metallic reflectors which are mounted on a movable driven metallic carriage as indicated in Fig. 11.10. The spot-focusing antenna consists in an ellipsoidal reflector of 300 mm diameter and 1 m focal distance having a feed horn located at the first focus.

The feed horns are pyramidal horns having an equiphase illumination law over the total frequency band for the two polarizations. The phase centers of these feed horns are practically constant with frequency. These conditions have to be insured to warrant a quasi plane wave at the second focus. The decoupling between co-polarization and cross-polarization is equal or higher than 40 dB. The illumination law of the metallic reflector is a gaussian beam with -15 dB levels on the

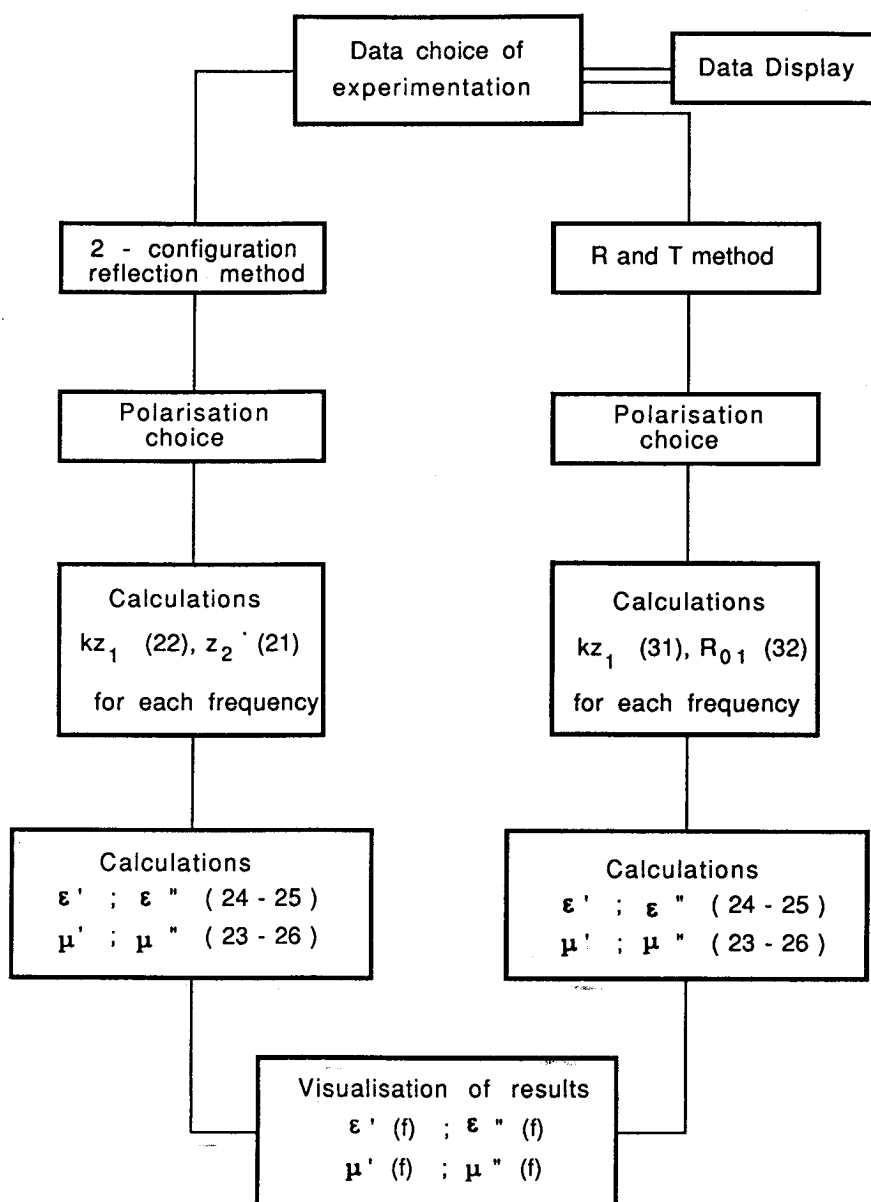


Figure 11.6 General computer program.

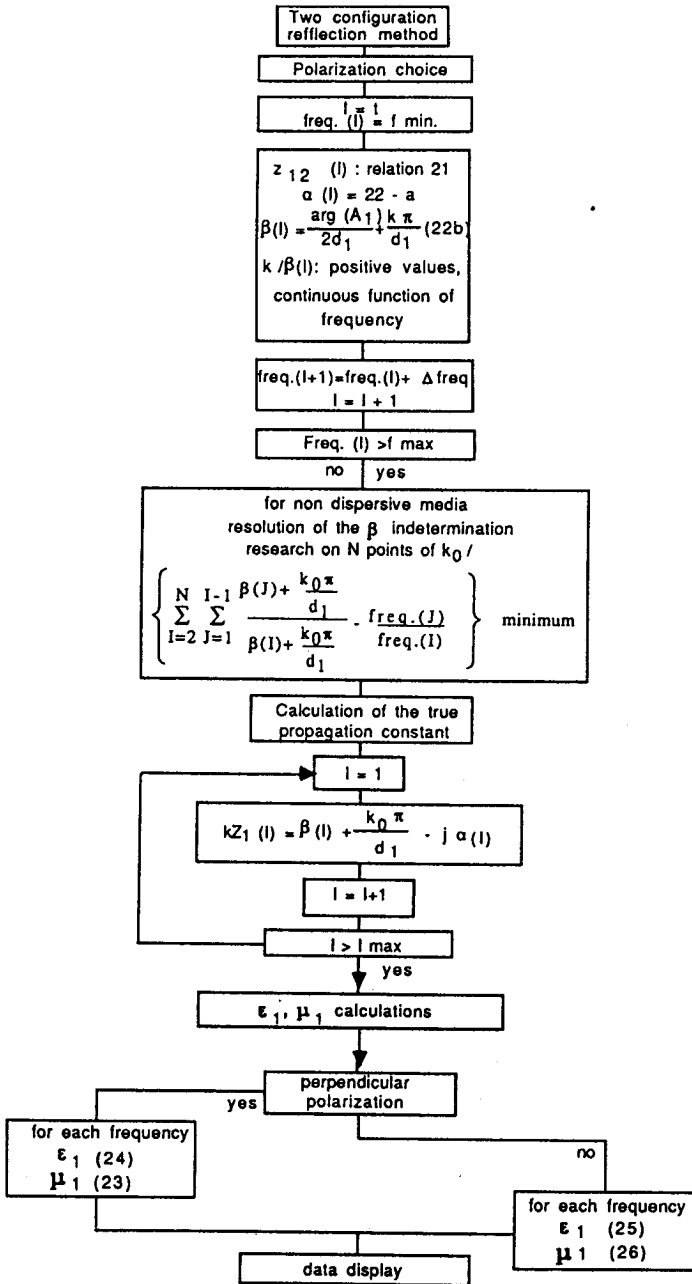


Figure 11.7 Two-configuration reflection method algorithm.

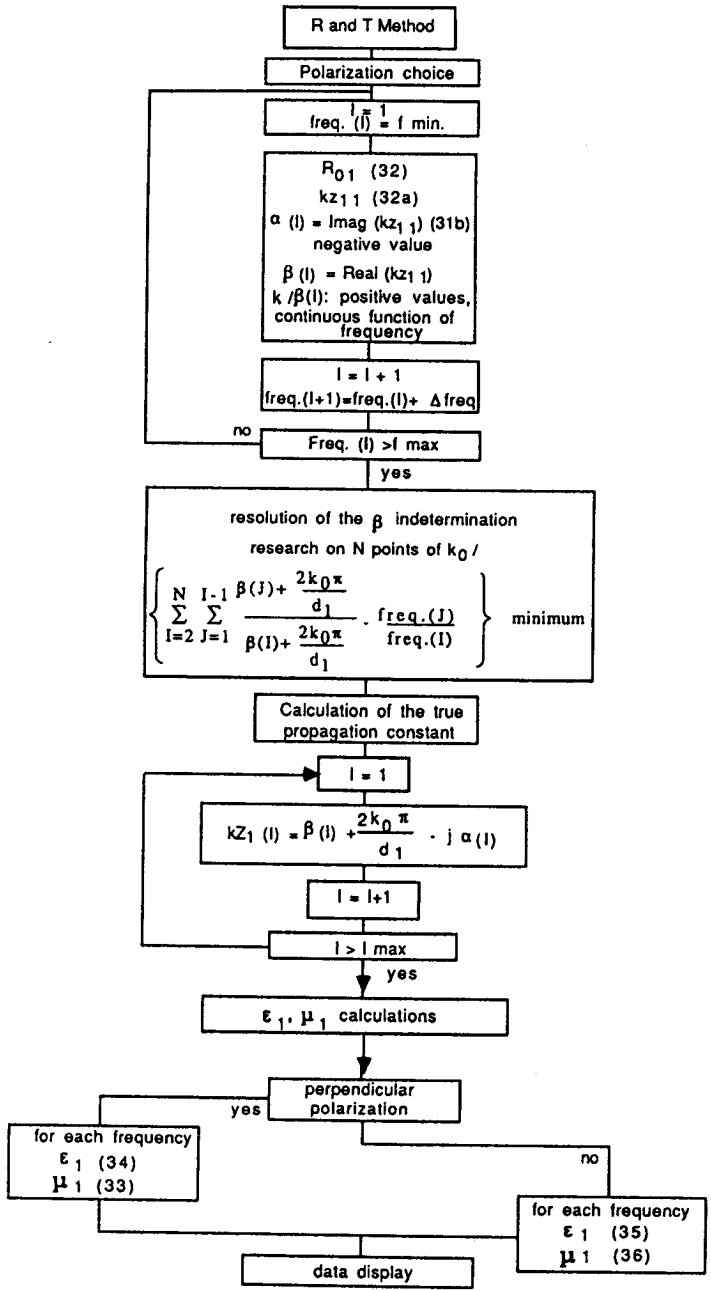


Figure 11.8 R and T method algorithm.

reflector edges with respect to the center part of the reflector. By manually rotating the feed horns, the polarization of the impinging waves can be changed from 0 to 90°. In the future this procedure will be electronically driven from 0 to 180°.

The movable driven metallic carriage has provision for configuring antennas for transmission and bistatic measurements (20° - 70° off normal) and scattering (forward and backward scattering) measurements on a wide angle range. Monostatic measurements will be added in the future by using a network analyser. The angle and position accuracies of the metallic carriage are respectively 0.05° and 0.010 mm and are assured by a very accurate driven positioner (Micro-Controle).

A special fabricated sample holder is placed at the common focal plane for holding planar samples. It is also mounted on a very accurate driven positioner. Samples of transverse cross-section 18 cm × 18 cm can be held ($6\lambda \times 6\lambda$ cross-section) or rotated 360°. The amplitude and phase variations of the beamwidth at the second focus are given in Fig. 11.11 for three frequencies. The 3-dB beamwidth of focus is equal to 2λ , and the first sidelobe levels for the three frequencies are lower than -20 dB. On the phase curve, the phase variation is lower than 10° in the 10-dB range. This fully demonstrates the plane wave like character of the impinging electromagnetic waves. From relation (3) we can calculate the depth of focus. At the middle frequency we have a value of 15 cm (approximately 5λ).

Microwave, RF synthesizers and the superheterodyne receiver were used for measuring complex reflection and transmission coefficients (Fig. 11.9). A comparison of emitted and received signals has been made in harmonic frequency converter unit (HP 8411, 0.11–12.4 GHz), followed by the Hewlett Packard 8410 network analyzer system. A Wiltron 360 MS 20 or Hewlett-Packard 8510B network analyzer system can be used in place of this system for microwave signal generation and reception. The pyramidal feed horns are connected to the measuring system by a rectangular-waveguide-to-coaxial adapter and coaxial cables. A HP 9000 series 300 computer, associated with HP 98785A hard disk, was used to drive all electronic units of positioners and microwave systems. It was also used for data acquisition and treatment, printing and plotting.

For the transmission and reflection cases and for a polarization and an incident angle, first reference measurements are made for each frequency, without a sample in the holder for transmission and with a

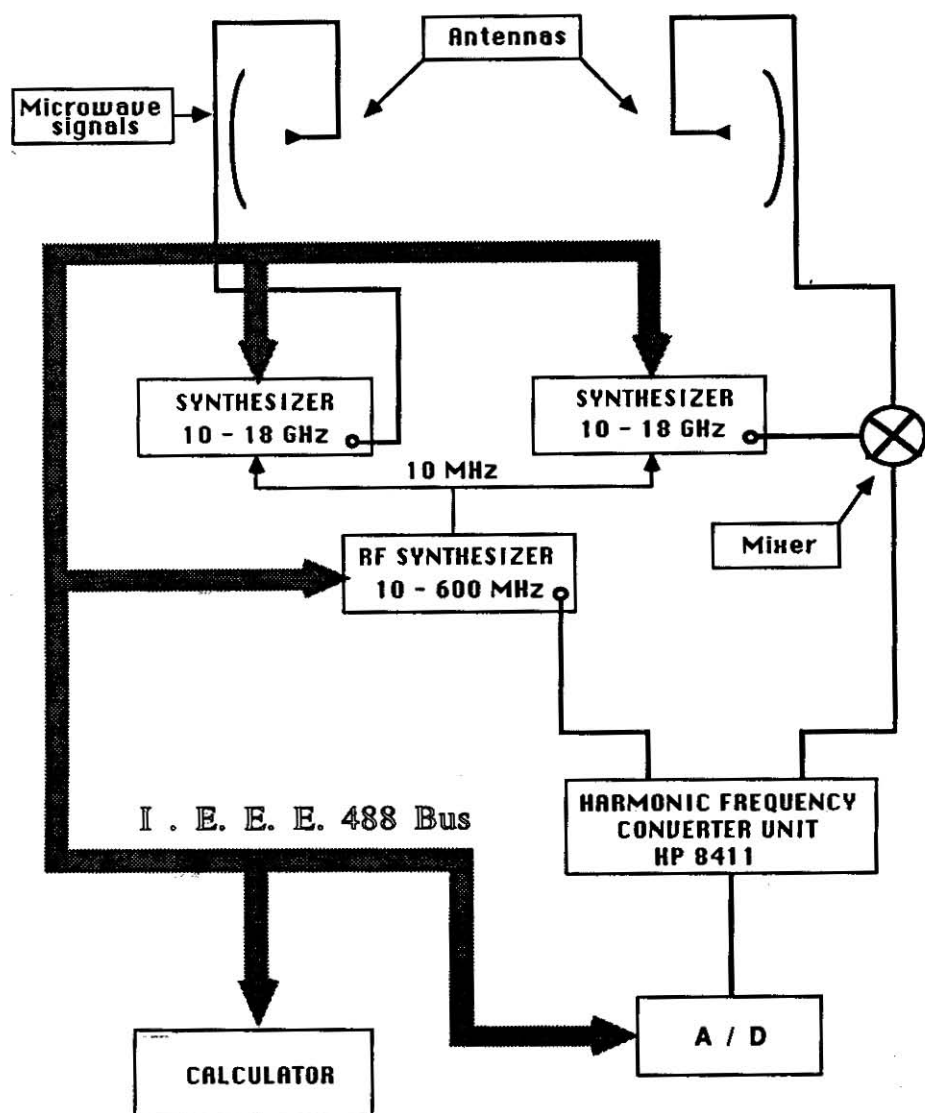


Figure 11.9 A schematic diagram of the 10-18 GHz experimental set up.

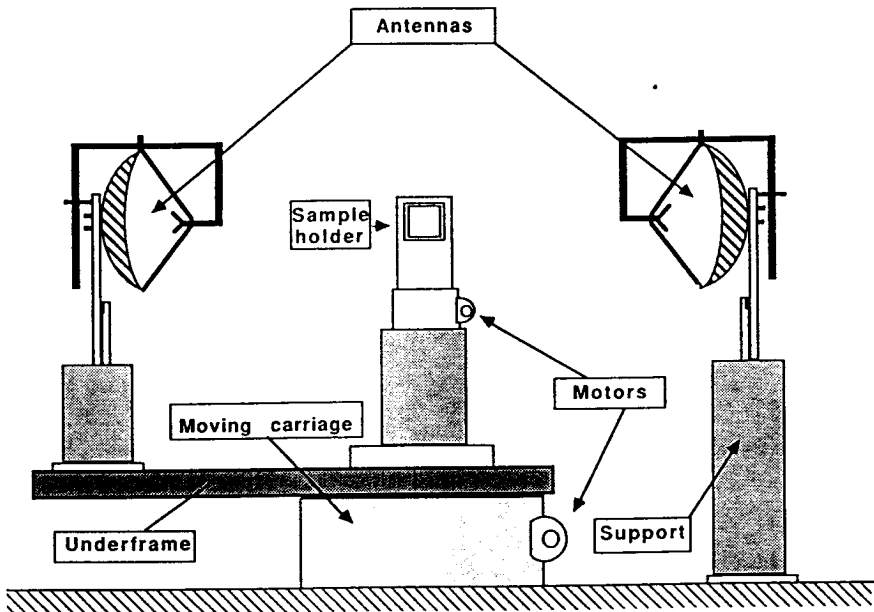


Figure 11.10 A schematic diagram of the movable driven carriage.

metallic plate for reflection. The phase and amplitude reference data are stored in the computer hard disk. Afterward, for the same frequency, polarization and incident angle configuration, we proceed to the measurement of the planar sample held in the metallic cartridge and store the data as amplitude and phase measurements. The double procedure is repeated each time the frequency, the incident angle or the polarization have been changed. A ratio of reference and measurement complex values is then generated (or a subtraction in dB). The result constitutes the raw data used as the entrance data in the algorithm computer programs. Alternatively, Fast Fourier Transform filtered data could be used as entrance data. Independently, these procedures permit the elimination of the major mismatch effects (source and horn VSWR) and all other error sources due to phase variations associated with rotating coaxial cables at each incident angle variation.

An advantage of using an HP or Wiltron network analyzer will be

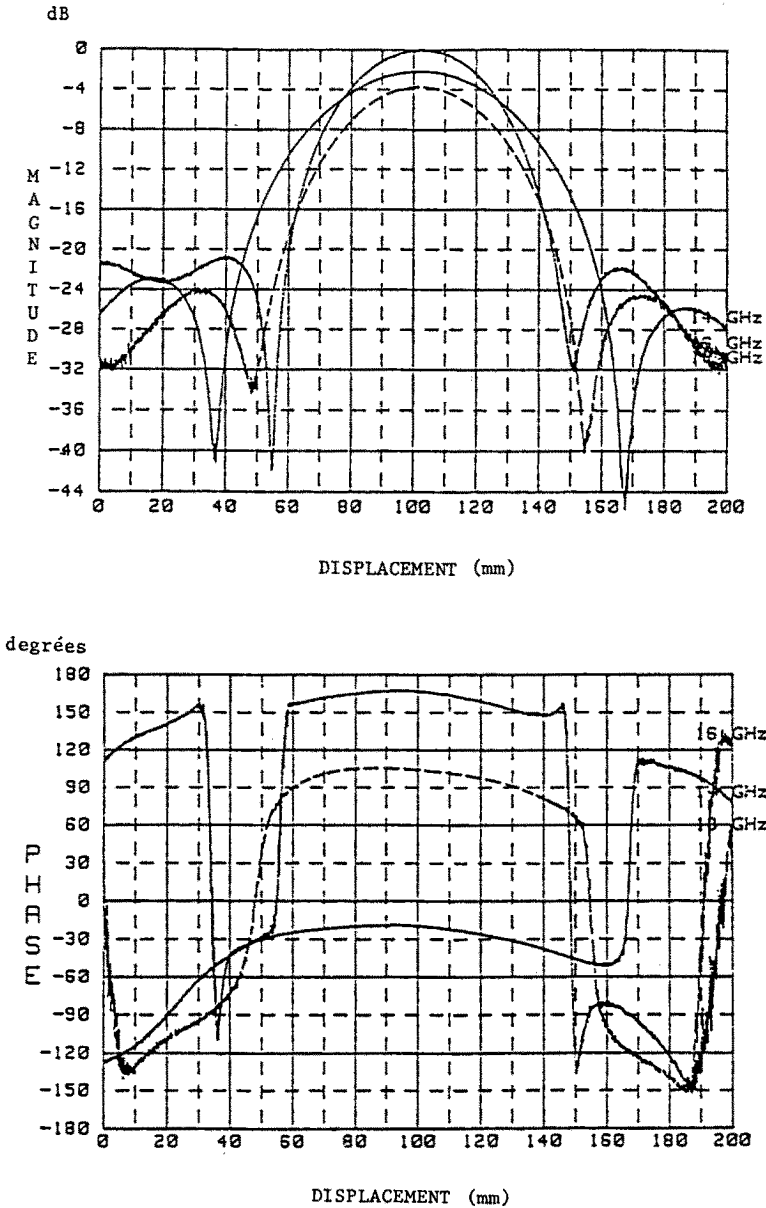


Figure 11.11 Experimental amplitude and phase of the beam in the focal plan.

to implement a two-port TRL calibration technique and time domain gating procedures as done by Varadan et al. [72] on HP 8510B. This contributes to a better correction of VSWR between antennas and VSWR variations between sample and reflectors. A special temperature cell including the metallic holder for temperature measurements and extension in frequency range from 4 to 18 GHz are under consideration.

11.5 Experimental Results

Dielectric constants and loss tangents of composite materials were measured in the frequency range of 10–18 GHz by using the free-space method described above. The samples were rectangular planar plates of 180×180 mm and thickness varying from 0.5 to 4 mm.

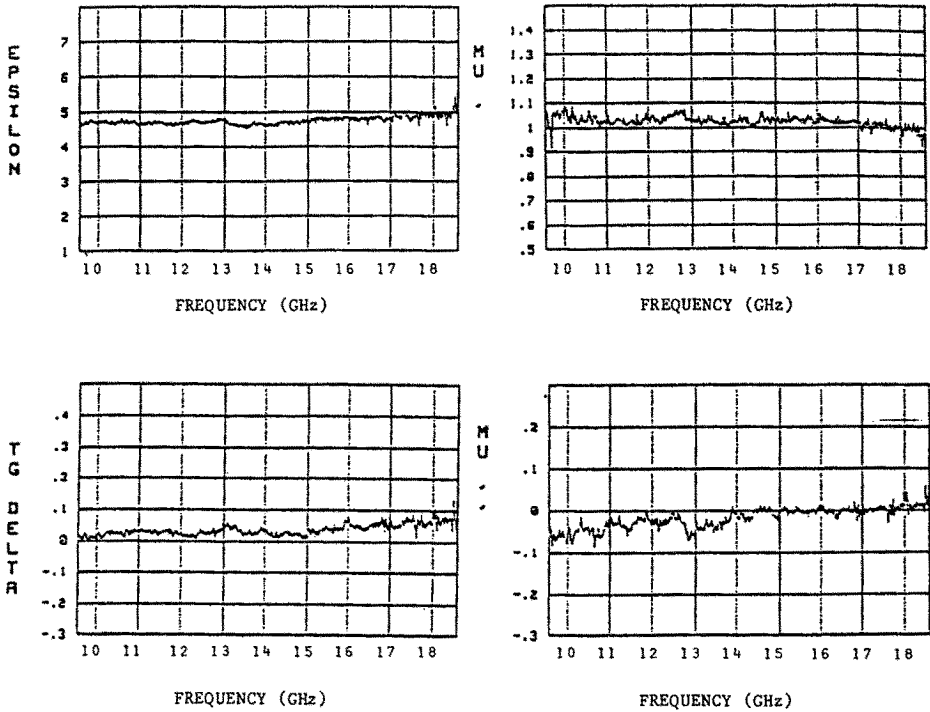
In Fig. 11.12 raw data variations of epoxy laminate dielectric constant and loss tangent are shown in the 10–18 GHz range and for perpendicular polarization by using the two-configuration reflection method. The second layer is constituted by an air layer of 4.74 mm thickness.

In Figs. 11.13 and 11.14, we have the same display for composite materials made of, respectively, 7.5% and 12.5% carbon-resin mixture layers having thickness of 0.84 mm at perpendicular polarization and by using the two-configuration reflection method. The second layer remains an air layer of 4.8 mm thickness. The electromagnetic properties of each layer are respectively:

for a 7.5% carbon-resin mixture layer: $\epsilon' : 6; tg\delta : 0.20; \mu' : 1.15; \mu'' : 0.0$
 for a 12.5% carbon-resin mixture layer: $\epsilon' : 8; tg\delta : 0.35; \mu' : 1.05; \mu'' : 0.0$

It is also possible to use other dielectric material for the second layer without restriction of the method. Some experiments have been made with ceramic materials as the second layer.

The method does not require any assumptions regarding the frequency behavior of each layer, which means that the accuracy could be improved by filtering and smoothing. In Fig. 11.15, frequency variations of complex permittivity and permeability values for a 2.27-mm thick Plexiglas sheet are shown. The R-T method at perpendicular polarization has been used. The dielectric constant and loss tangent are respectively equal to 2.65 and 0.02. These values are generally observed in the literature. As expected the permeability values are $\mu' = 1$ and $\mu'' = 0$.



INCIDENT ANGLE : 30°

PERPENDICULAR POLARIZATION

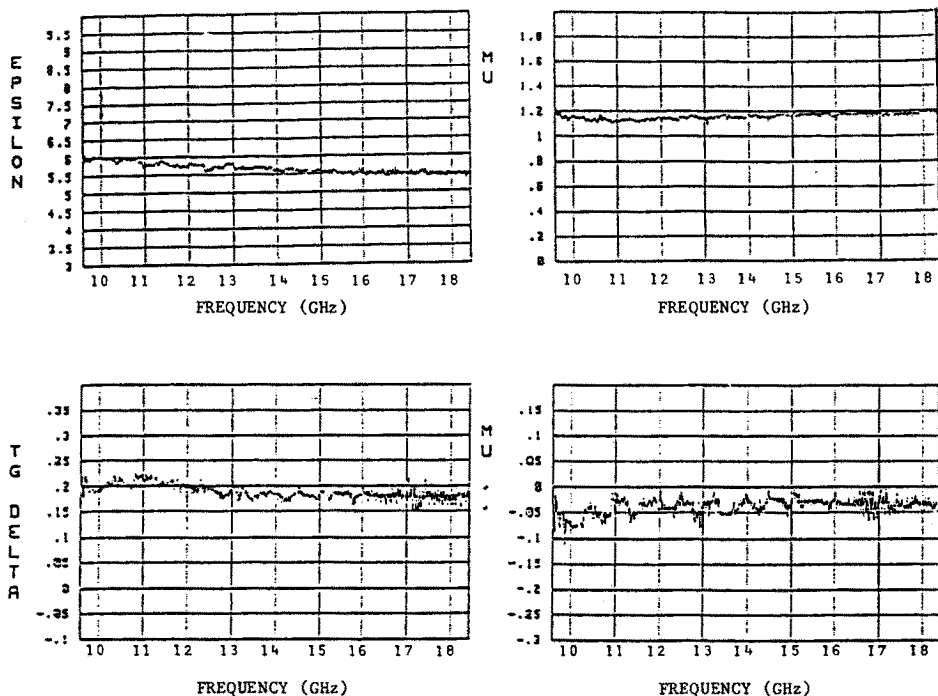
1 TEST MATERIAL

2 LAYER : AIR

D1 = 2.27mm

D2 = 4.74mm

Figure 11.12 Complex permittivity and permeability variations of an epoxy laminate.

INCIDENT ANGLE : 30°

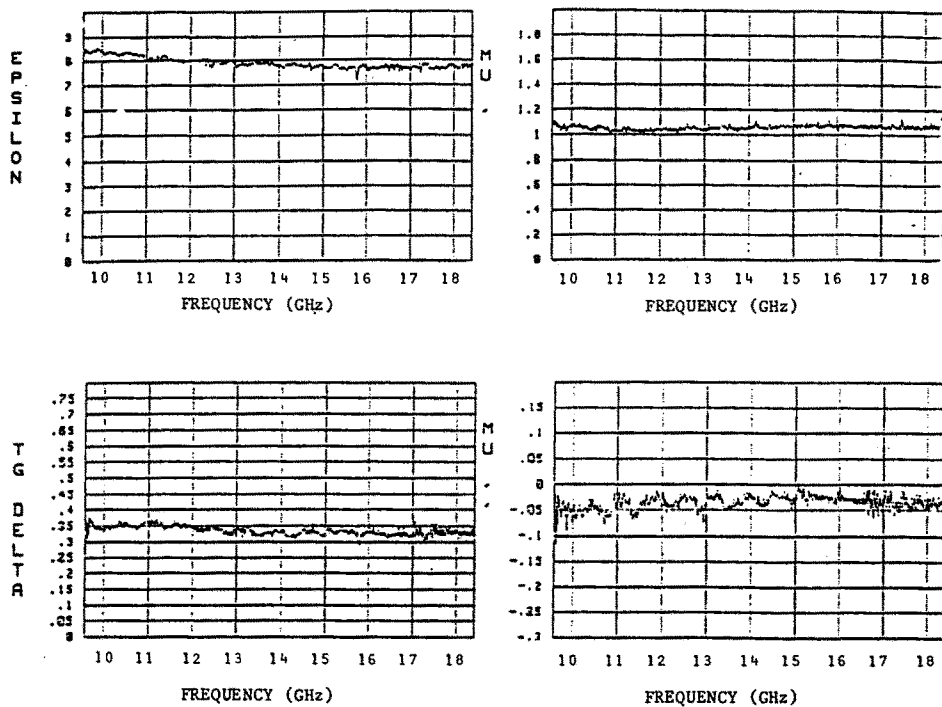
PERPENDICULAR POLARIZATION

1 TEST MATERIAL

2 LAYER : AIR

 $D1 = 0.82\text{mm}$ $D2 = 4.74\text{mm}$

Figure 11.13 Complex permittivity and permeability variations of a 7.5% carbon-resin mixture layer.



INCIDENT ANGLE : 30°

PERPENDICULAR POLARIZATION

1 TEST MATERIAL

2 LAYER : AIR

D1 = 0.84mm

D2 = 4.74mm

Figure 11.14 Complex permittivity and permeability variations of a 12.5% carbon-resin mixture layer.

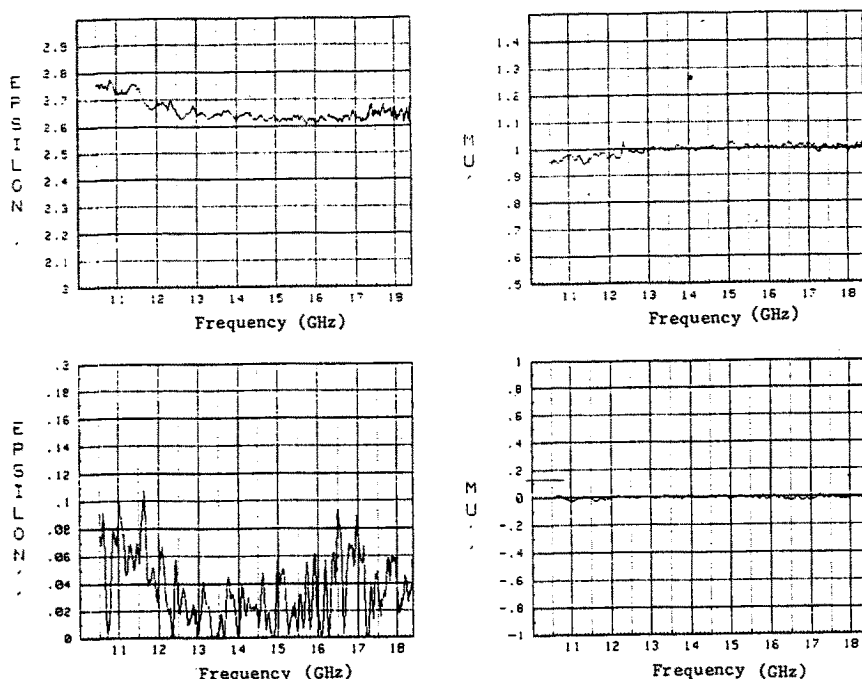


Figure 11.15 Complex permittivity and permeability variations in frequency for a 2.27 mm plexiglass planar sample.

11.6 Conclusions

After an introduction dealing with composite material modeling research and an overview of electromagnetic measurement techniques, we have presented the microwave free-space characterization technique for composite materials. It is an attempt to emphasize the main advantages of this technique compared to waveguide, coaxial, cavity or other methods.

With free-space characterization, two complementary methods have been proposed to derive the complex permittivity and permeability of composite materials: two-configuration reflection and reflection-transmission methods. Experimental set-up using gaussian feed horn metallic reflectors has been built for measurements from 10–18 GHz. Extensions to lower frequencies (4–18 GHz) and for high temperature conditions are in progress.

This non destructive and contactless technique allows the characterization of various kinds of standard, composite or complex materials. The possibility to configure antennas for transmission, monostatic, bistatic ($20^\circ - 70^\circ$ off-normal) and scattering measurements, associated with polarization change is a significant advantage in the characterization of complex media such as chiral materials.

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