INFRARED AND MILLIMETER WAVES

VOLUME 12 ELECTROMAGNETIC WAVES IN MATTER, PART II

Edited by *KENNETH J. BUTTON*

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PREFACE

This is the second volume in this treatise to deal exclusively with the millimeter and submillimeter properties of materials and the methods of measuring and interpreting these properties. A third volume on this topic is now in preparation, and plans for a fourth are in progress.

The contents of Volumes 8 and 12 speak eloquently for the theme of this subseries on electromagnetic waves in matter. Each book opens with a chapter on dielectric materials studies. G. W. Chantry introduced the topic in Volume 8, and Mohammed Nurul Afsar has now provided us with his extensive dielectric measurements at millimeter wavelengths in Volume 12. In both volumes the second chapter deals with polymers. W. F. X. Frank and U. Leute described their far-infrared spectroscopy of high polymers in Volume 8. John F. Rabolt deals with low-frequency (far-infrared) vibrations of long-chain molecules and polymers in Volume 12. These chapters are followed by semiconductor topics. In Volume 8 S. Perkowitz gave a general treatment of the spectroscopy of semiconductors and B. Jensen gave a thorough treatment of free-carrier behavior in semiconductors (which is most important in the far infrared). In Volume 12 we have a review of the celebrated work of the Miura group, which uses extraordinarily high-intensity pulsed magnetic fields for magnetooptical spectroscopy of semiconductor phenomena.

Following the pathfinding work by Gert Finger and Fritz K. Kneubühl on spectral thermal infrared emission of the terrestrial atmosphere, we have two chapters on far-infrared lasers because lasers are of such great importance to the far-infrared spectroscopy of materials.

A third volume devoted to electromagnetic wave interactions in matter is being readied for press. Volumes 9, 10, 11, and 13 begin a subseries on millimeter-wave components and techniques; only the last of these four volumes is yet to be published.

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Volume 14: Millimeter Components and Techniques, Part V
(In Press)

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CHAPTER 1

Millimeter-Wave Dielectric Properties of Materials*

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I. Introduction

Almost no reliable data have been available in the millimeter and near-millimeter wavelength (60–600 GHz) range because measurements of the dielectric properties of materials at these wavelengths are extremely difficult

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to carry out accurately. The millimeter-wave region lies beyond conventional microwave techniques and forms a "bridge" to the optical techniques. In the past, one could rarely trust the millimeter-wave dielectric data for use in precision engineering design, because any extrapolated microwave method or extrapolated optical method that was used to make the measurements had many serious limitations and uncertainties. Until recently, engineers have been satisfied to know whether a material was "opaque" or "transparent" at millimeter wavelengths. More recently, a measurement good to 10% accuracy was considered to be better than nothing; after all, it is inconvenient and expensive to acquire and use precision-measurement facilities and sophisticated instrumentation. The real danger lies in the literature that is actually misleading. Most frequently the misleading data get into the literature when someone uses a familiar *microwave* instrument, such as waveguide interferometer or a cavity resonator or a Fabry–Perot open resonator, beyond the limit of its classical capabilities. For example, the millimeter wavelengths are too short for the practical use of a microwave single-mode resonant cavity. The millimeter wavelengths are too long at this extreme end of the *optical* spectrum for a familiar blackbody source such as a mercury-vapor lamp to be used; normally, it provides too little energy for millimeter-wave measurements with a Fourier spectrometer. Indeed, the use of a *conventional* plane-wave interference technique employing a mercury lamp to obtain millimeter-wave dielectric data is almost impossible. Nevertheless, the Fourier method has now been improved by Afsar to provide data from 5 mm (60 GHz) into the submillimeter range (Afsar and Button, 1983). New theories were also developed by Afsar giving a full treatment of all beams and interface effects (Afsar, 1977, 1984a; Afsar and Chantry, 1977; Afsar *et al.*, 1976b,c), and great care was taken to increase the efficiency of energy throughput and detection (Afsar and Button, 1981, 1983; Afsar, 1982). In such a special spectrometer, the phase determination, in particular, can be made very accurately when used in the asymmetric mode (dispersive Fourier transform spectroscopy), leading to the determination of the real part of the dielectric constant to five or six significant figures (Afsar and Button, 1983; Afsar, 1982). Because we employ a quasi-optical technique, we measure directly the optical parameters, namely, the absorption coefficient α and the refractive index n, simultaneously. Dielectric parameters ϵ', ϵ'' and loss tangent (tan δ) are easily calculated, as will be demonstrated. The present-day dispersive Fourier transform spectroscopic (DFTS) technique developed by Afsar, measures the refractive index spectrum and, simultaneously, the absorption coefficient spectrum from the analysis of the amplitude and phase information that the specimen has contributed to the output signal (Afsar, 1977, 1982, 1984a; Afsar and Button, 1981, 1983; Afsar and Chantry, 1977; Afsar and Hasted, 1977; Afsar *et al.*, 1976b,c). Although the phase information can be

carried through to a determination of the refractive index (and the real part of the dielectric permittivity) to an accuracy of five or six significant figures for a low-loss material, the absorption coefficient (and loss tangent) can be determined only to about 1%, because the commercially available electronic amplifying equipment cannot ordinarily carry through amplitude information with higher precision and reproducibility (Afsar and Button, 1983; Afsar, 1982).

Several other classical methods are being improved in efforts to provide some kind of data, if not the best, to this barren region of the spectrum. The Fabry–Perot open-resonator method provides about an order-of-magnitude less accuracy in the measurement of loss tangent and only three significant figures in the dielectric constant, but in some ways it is more convenient to use (Cullen and Yu, 1971; 1979; Cook and Jones, 1976; Jones, 1976). Today, the most significant improvement in the Fabry–Perot system would be the use of a superheterodyne receiver with highly stabilized, phase-locked Gunn oscillators (Matsui *et al.*, 1984). The Mach–Zehnder type of spectrometer used with Gunn or IMPATT sources also produces dielectric data at the typical IMPATT frequencies (Birch, 1980, 1981). Precision data in this case again is obtainable only by the use of a specially constructed, highly stable spectrometer system with a high degree of statistical fitting (Afsar, 1984b). Various other techniques such as rotation of a parallel-slab specimen with input and output devices (Shimabukuro *et al.*, 1984), waveguide reflectometer (Vanloon and Finsy, 1973, 1974; Finsy and Vanloon, 1972), oversize cavity resonator (Stumper, 1972, 1973; Stumper and Frentrup, 1976), and oversize waveguide interferometer (Goulon *et al.*, 1968, 1973; Butterweck, 1968) also produce dielectric data in the range 10–2 mm, but the accuracy is again limited to about 10% in most of these techniques. Among all of these methods, the DFTS is the best for the millimeter and submillimeter range. Other methods, such as the six mentioned previously, have their particular applications such as other wavelength ranges, odd specimen sizes, and different physical properties such as liquids and gases (Kolbe and Leskovar, 1982).

Why should we go to all of this trouble and expense just to get another order-of-magnitude, or even a factor of three, higher accuracy, reproducibility, and reliability? Why would a quick measurement providing "engineering values" be unsuitable for the purpose of exercising the trade-off process for the selection of materials for particular applications? The simple answer is that there are wide variations in the parameters of nominally identical specimens at millimeter wavelengths that microwave engineers rarely see at lower frequencies. When we are trying to determine the reasons for these variations so as to choose a "standard material" for our application, ±10% in reproducibility of measurement is just not good enough.

It is very important to have highly reproducible data, so that one would be

able to distinguish the different dielectric properties among nominally identical specimens — dielectric properties that vary among specimens from different suppliers, among specimens prepared by somewhat different methods, or among specimens having physical properties that are not precisely controlled during preparation. In our recent dispersive Fourier transform spectroscopic dielectric measurement work, we have found significant variations in the dielectric properties of such common materials as SiO_2, fused silica glass (Afsar and Button, 1983; Afsar, 1982). There are notable differences in absorption coefficients in Al_2O_3, ceramic alumina, depending on the source of the alumina specimens. For example, hot-pressed ceramic beryllia, BeO, has much lower losses than cold-pressed beryllia.

We would expect to find differences in absorption among high-resistivity semiconductors such as semi-insulating GaAs, and large differences were found (Afsar and Button, 1983). Polymers (plastic) are well known to be very much in need of characterization before they can be used in engineering applications. Their degree of crystallinity must be controlled (Chantry *et al.*, 1971; Davies and Haigh, 1974; Konwerska-Hrabowska *et al.*, 1981). Therefore, it is now essential that a full description of a material be available along with accurate, reproducible measurements of its dielectric properties. Thus, it has been shown that not only is a microwave measurement of loss tangent untrustworthy at millimeter wavelengths but also traditional microwave methods used at millimeter wavelengths can be inaccurate and irreproducible.

The important differences in nominally identical specimens can be detected, verified, and understood only by using the most sophisticated, highly sensitive, and highly stable equipment backed by a most detailed evaluation of the theory of the technique. Therefore, it is important to rely on a "center of excellence" as a source of practical data.

This chapter will include a treatment of the relationship of measured and derived (calculated) quantities as well as brief descriptions and comparisons of some modern dielectric measurement techniques. Appendix A is a compendium of data, where illustrations will show a comparison of some common materials in terms of their spectra of absorption coefficient, refractive index, real and imaginary parts of the dielectric permittivity, and loss tangent. A discussion of differences among nominally identical specimens will also be given. These discussions will provide examples of the importance of "characterization of materials." Sometime in the near future, the community of millimeter-wave engineers should be provided with a "digest of millimeter-wave materials information and measurement." As more data can be collected and added to the illustrations in the appendix, the nucleus of the digest will be created. Up-to-date copies of this fledgling digest will be available from the Millimeter and Submillimeter-Wave Materials Information and Measurement Center at the MIT National Magnet Laboratory.

II. Electromagnetic Quantities

The complex refractive index $\hat{n}$ is derived from the complex dielectric permittivity $\hat{\epsilon}$ of Maxwell's equations so that

$$\hat{\epsilon} = (\hat{n})^2.$$

The real and imaginary parts of n are, by definition,

$$\hat{n} = n - ik = n - i(\alpha/4\pi\tilde{\nu}) = n - i(c\alpha/4\pi\nu),$$

where k is the absorption index $\alpha/4\pi\tilde{\nu} = c\alpha/4\pi\nu$, α the absorption coefficient (cm^{-1}), ν the frequency in hertz, $\tilde{\nu}$ the wave number in cm^{-1}, c the velocity of light in vacuum; and it is convenient to note that 1 wave number (cm^{-1}) = 30 GHz.

The complex dielectric permittivity $\hat{\epsilon}$ has real (ϵ') and imaginary (ϵ'') parts defined as

$$\hat{\epsilon} = \epsilon' - j\epsilon'', \qquad i = j = \sqrt{-1}.$$

Then, our definitions provide us with the simple relationships between the fundamental optical quantities, α and n, and the dielectric quantities ϵ' and ϵ'', as follows:

$$\epsilon' = n^2 - k^2 = n^2 - (\alpha/4\pi\tilde{\nu})^2 = n^2 - (c\alpha/4\pi\nu)^2;$$

$$\epsilon'' = 2nk = (\alpha n)/2\pi\tilde{\nu} = (\alpha cn)/2\pi\nu.$$

The term loss tangent, or the popularly known "tan δ," is the ratio of the imaginary part (ϵ'') to the real part (ϵ') of the dielectric permittivity,

$$\tan\delta = \epsilon''/\epsilon'.$$

In the millimeter-wave region of the spectrum, we need all of these relationships, because we cannot simply extrapolate all of our microwave techniques into this nether region from the long-wavelength side, nor can we extrapolate all of our optical techniques from the high-frequency side. Both the microwave engineers and the optical engineers consider this nether world of millimeter waves by its strict Webster's definition as "world of the dead or of future punishment." The microwave engineer finds his millimeter wavelengths to be too small for fundamental-mode techniques for measurement of complex dielectric permittivity and loss tangent. The optical engineer, who typically uses free-space, plane-wave Michelson interference, finds his source of blackbody radiation (mercury-vapor lamp) to be too feeble.

This anticipation of future punishment can be ameliorated somewhat by the development of some "figures of merit" or means for evaluating trade-off selections of materials. One of these is the definition of a "millimeter-wave low-loss material" to eliminate the use of the loss tangent, tan δ, which is sometimes unreliable at millimeter wavelengths.