

INFRARED AND MILLIMETER WAVES

**VOLUME 12 ELECTROMAGNETIC WAVES
IN MATTER, PART II**

Edited by **KENNETH J. BUTTON**



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NATIONAL MAGNET LABORATORY
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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 magneto-optical 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.

CONTENTS OF OTHER VOLUMES

Volume 1: Sources of Radiation

- J. L. Hirshfield*, Gyrotrons
H. J. Kuno, IMPATT Devices for Generation of Millimeter Waves
Thomas A. DeTemple, Pulsed Optically Pumped Far Infrared Lasers
G. Kantorowicz and P. Palluel, Backward Wave Oscillators
K. Mizuno and S. Ono, The Ledatron
F. K. Kneubühl and E. Affolter, Infrared and Submillimeter-Wave Waveguides
P. Sprangle, Robert A. Smith, and V. L. Granatstein, Free Electron Lasers and Stimulated Scattering from Relativistic Electron Beams

Volume 2: Instrumentation

- N. C. Luhmann, Jr.*, Instrumentation and Techniques for Plasma Diagnostics: An Overview
D. Véron, Submillimeter Interferometry of High-Density Plasmas
J. R. Birch and T. J. Parker, Dispersive Fourier Transform Spectroscopy
B. L. Bean and S. Perkowitz, Far Infrared Submillimeter Spectroscopy with an Optically Pumped Laser
Wallace M. Manheimer, Electron Cyclotron Heating of Tokamaks

Volume 3: Submillimeter Techniques

- T. G. Blaney*, Detection Techniques at Short Millimeter and Submillimeter Wavelengths: An Overview
W. M. Kelley and G. T. Wrixon, Optimization of Schottky-Barrier Diodes for Low-Noise, Low-Conversion Loss Operation at Near-Millimeter Wavelengths
A. Hadni, Pyroelectricity and Pyroelectric Detectors
A. F. Gibson and M. F. Kimmitt, Photon Drag Detection
F. W. Kneubühl and Ch. Sturzenegger, Electrically Excited Submillimeter-Wave Lasers
Michael von Ortenberg, Submillimeter Magnetospectroscopy of Charge Carriers in Semiconductors by Use of the Strip-Line Technique
Eizo Otsuka, Cyclotron Resonance and Related Studies of Semiconductors in Off-Thermal Equilibrium

Volume 4: Millimeter Systems

James C. Wiltse, Introduction and Overview of Millimeter Waves

Edward K. Reedy and George W. Ewell, Millimeter Radar

Charles R. Seashore, Missile Guidance

N. Bruce Kramer, Sources of Millimeter-Wave Radiation: Traveling-Wave Tube and Solid-State Sources

Tatsuo Itoh, Dielectric Waveguide-Type Millimeter-Wave Integrated Circuits

M. Tsuji, H. Shigesawa, and K. Takiyama, Submillimeter Guided Wave Experiments with Dielectric Waveguides

Gary A. Gordon, Richard L. Hartman, and Paul W. Kruse, Imaging-Mode Operation of Active NMMW Systems

Volume 5: Coherent Sources and Applications, Part I

Benjamin Lax, Coherent Sources and Scientific Applications

J. O. Henningsen, Molecular Spectroscopy by Far-Infrared Laser Emission

F. Strumia and M. Inguscio, Stark Spectroscopy and Frequency Tuning in Optically Pumped Far-Infrared Molecular Lasers

Jun-ichi Nishizawa, The GaAs TUNNETT Diodes

V. L. Granatstein, M. E. Read, and L. R. Barnett, Measured Performance of Gyrotron Oscillators and Amplifiers

F. K. Kneubühl and E. Affolter, Distributed-Feedback Gas Lasers

Volume 6: Systems and Components

J. E. Harries, Infrared and Submillimeter Spectroscopy of the Atmosphere

D. H. Martin, Polarizing (Martin-Puplett) Interferometric Spectrometers for the Near- and Submillimeter Spectra

P. L. Richards and L. T. Greenberg, Infrared Detectors for Low-Background Astronomy: Incoherent and Coherent Devices from One Micrometer to One Millimeter

M. V. Schneider, Metal-Semiconductor Junctions as Frequency Converters

Paul F. Goldsmith, Quasi-Optical Techniques at Millimeter and Submillimeter Wavelengths

G. D. Holah, Far-Infrared and Submillimeter-Wavelength Filters

Volume 7: Coherent Sources and Applications, Part II

Thomas A. DeTemple and Edward J. Danielewicz, Continuous-Wave Optically Pumped Lasers

R. G. Harrison and P. K. Gupta, Optically Pumped Mid-Infrared Molecular Gas Lasers

Konrad Walzer, On the Optimization of Optically Pumped Far-Infrared Lasers

- J. P. Pichamuthu*, Submillimeter Lasers with Electrical, Chemical, and Incoherent Optical Excitation
- D. D. Bičanić*, Generation of Tunable Laser Sidebands in the THz Region by Frequency Mixing of the HCN Laser and a Microwave Source in a Metal-Semiconductor Diode
- J. Nishizawa and K. Suto*, Semiconductor Raman and Brillouin Lasers for Far-Infrared Generation
- Donald E. Wortman and Richard P. Leavitt*, The Orotron
- K. E. Kreischer and R. J. Temkin*, High-Frequency Gyrotrons and Their Application to Tokamak Plasma Heating
- John L. Vomvoridis, P. Sprangle, and W. M. Manheimer*, Theoretical Investigation of the Application of Quasi-Optical Open Resonators to the Electron Cyclotron Maser
- D. Dialetis and K. R. Chu*, Mode Competition and Stability Analysis of the Gyrotron Oscillator

Volume 8: Electromagnetic Waves in Matter, Part I

- G. W. Chantry*, Properties of Dielectric Materials
- W. F. X. Frank and U. Leute*, Far-Infrared Spectroscopy of High Polymers
- S. Perkowitz*, Submillimeter Solid-State Physics
- B. Jensen*, Review of the Theory of Infrared and Far-Infrared Free-Carrier Behavior in Semiconductors
- A. Hadni*, Review of Recent Improvements in Pyroelectric Detectors
- Tyuzi Ohyama and Eizo Otsuka*, Cyclotron and Zeeman Transitions in Photoexcited Semiconductors at Far Infrared
- F. Gervais*, High-Temperature Infrared Reflectivity Spectroscopy by Scanning Interferometry
- P. Goy*, Millimeter and Submillimeter Waves Interacting with the Giant Atoms (Rydberg States)
- J. C. Maan*, Far-Infrared Spectroscopy of InAs-GaSb Layered Structures

Volume 9: Millimeter Components and Techniques, Part I

- K. Miyauchi*, Millimeter-Wave Communications
- T. Itoh and J. Rivera*, A Comparative Study of Millimeter-Wave Transmission Lines
- Marvin B. Klein*, Dielectric Waveguide Electrooptic Devices
- Edward E. Altshuler*, Millimeter-Wave Propagation and Remote Sensing of the Atmosphere
- J. W. M. Baars*, Technology of Large Radio Telescopes for Millimeter and Submillimeter Wavelengths

G. Boucher, Ph. Boulanger, P. Charbit, G. Faillon, A. Herscovici, E. Kammerer, and G. Mourier, A Gyrotron Study Program
Anders Bondeson, Wallace M. Manheimer, and Edward Ott, Multimode Analysis of Quasi-Optical Gyrotrons and Gyroklystrons

Volume 10: Millimeter Components and Techniques, Part II

David B. Rutledge, Dean P. Neikirk, and Dayalan P. Kasilingham, Integrated-Circuit Antennas
K. Sigfrid Yngvesson, Near-Millimeter Imaging with Integrated Planar Receptors: General Requirements and Constraints
R. K. Mains and G. I. Haddad, Properties and Capabilities of Millimeter-Wave IMPATT Diodes
A. L. Cullen, Millimeter-Wave Open Resonator Techniques
G. Chanin and J. P. Torre, ³He Refrigerators and Bolometers for Infrared and Millimeter-Wave Observations
Cheng-he Xu and Le-zhu Zhou, Microwave Open Resonators in Gyrotrons
C. W. Roberson, J. Pasour, F. Mako, Robert F. Lucy, Jr., and P. Sprangle, A Free-Electron Laser Driven by a Long-Pulse Induction Linac

Volume 11: Millimeter Components and Techniques, Part III

I. G. Eddison, Indium Phosphide and Gallium Arsenide Transferred-Electron Devices at Millimeter Wavelengths
Tsukasa Yoneyama, Nonradiative Dielectric Waveguide
Yat Man Choi and Douglas J. Harris, Groove Guide for Short Millimetric Waveguide Systems
F. Kremer, A. Poglitsch, D. Böhme, and L. Genzel, The Application of Over-sized Cavities to Millimeter-Wave Spectroscopy
V. A. Flyagin, A. L. Gol'denberg, and G. S. Nusinovich, Powerful Gyrotrons
G. S. Nusinovich, Some Perspectives on Operating Frequency Increase in Gyrotrons
Algie L. Lance, Wendell D. Seal, and Frederick Labaar, Phase Noise and AM Noise Measurements in the Frequency Domain
A. Gover, H. Freund, V. L. Granatstein, J. H. McAdoo, and Chai-mei Tang, Basic Design Considerations for Free-Electron Lasers Driven by Electron Beams from rf Accelerators

Volume 13: Millimeter Components and Techniques, Part IV
(In Press)

- V. A. Flyagin and G. S. Nusinovich*, Powerful Gyrotrons for Thermonuclear Research
- P. Sprangle and T. Coffey*, New High-Power Coherent Radiation Sources
- K. R. Chu and D. Dialetis*, Kinetic Theory of Harmonic Gyrotron Oscillator with Slotted Resonant Structure
- Wolfgang Menzel*, Integrated Fin-Line Components for Communication, Radar, and Radiometer Applications
- J. L. Douane*, Propagation and Mode Coupling in Corrugated and Smooth-Wall Circular Waveguides
- G. L. Carr and S. Perkowitz*, Far-Infrared Properties of Inhomogeneous Materials
- M. Tacke*, Solid-State Spectroscopy with Far-Infrared Continuous-Wave Lasers

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

- Charles R. Seashore*, Millimeter-Wave Integrated-Circuit Transducers
- Kai Chang*, Millimeter-Wave Planar Integrated Circuits and Subsystems
- James R. James and Ann Henderson*, Planar Millimeter-Wave Antenna Arrays
- Aileen M. Yurek, Charles D. Striffler, and Chi H. Lee*, Optoelectronic Devices for Millimeter Waves
- Daniel Massé, Michael G. Adlerstein, and Lowell H. Holway, Jr.*, Millimeter-Wave GaAs IMPATT Diodes
- G. F. Brand*, Tunable Gyrotrons

CONTENTS

LIST OF CONTRIBUTORS	vii
PREFACE	ix
CONTENTS OF OTHER VOLUMES	xi

Chapter 1 Millimeter-Wave Dielectric Properties of Materials

Mohammed Nurul Afsar and Kenneth J. Button

I. Introduction	1
II. Electromagnetic Quantities	5
III. Definition of a Low-Loss Material	6
IV. Measurement Methods	6
V. Discussion	17
Appendix: Compendium of Data	20
References	41

Chapter 2 Low-Frequency Vibrations in Long-Chain Molecules and Polymers by Far-Infrared Spectroscopy

John F. Rabolt

I. Introduction	43
II. Homopolymers	45
III. Polypeptides	58
IV. Copolypeptides	63
V. Proteins	64
VI. Polynucleotides	66
VII. Conducting Polymers	66
VIII. Other Polymers of Interest	67
IX. Conclusion	67
References	68

Chapter 3 Infrared Magneto-optical Spectroscopy in Semiconductors and Magnetic Materials in High Pulsed Magnetic Fields

Noboru Miura

I. Introduction	73
II. Experimental Techniques	76

CONTENTS

III.	Cyclotron Resonance and Electron Spin Resonance in Ultrahigh Magnetic Fields	87
IV.	Far-Infrared Magneto-optical Spectroscopy in Semimetals	109
V.	Faraday Rotation and Spin-Flip Transition in Magnetic Substances	127
VI.	Summary	138
	References	140
Chapter 4	Spectral Thermal Infrared Emission of the Terrestrial Atmosphere <i>Gert Finger and Fritz K. Kneubühl</i>	
I.	Introduction	145
II.	The LOWTRAN Model	148
III.	Water-Vapor Continuum Absorption	154
IV.	Experimental Setup	158
V.	Experimental Results	165
VI.	Interpretation of the Water-Vapor Continuum	180
VII.	Conclusion	188
	References	190
Chapter 5	Frequency Tuning and Efficiency Enhancement of High-Power Far-Infrared Lasers <i>B. G. Danly, S. G. Evangelides, R. J. Temkin, and B. Lax</i>	
I.	Introduction	195
II.	Tunable Raman Laser Theory	198
III.	Tunable Raman Laser Experiment	236
IV.	Efficiency Enhancement of Far-Infrared Lasers	257
V.	Summary	274
	References	275
Chapter 6	Far-Infrared Laser Scanner for High-Voltage Cable Inspection <i>P. K. Chco</i>	
I.	Introduction	279
II.	System Requirements and Performance Estimates	281
III.	Component Development	287
IV.	Conclusion	300
	Appendix: Mie Scattering from Voids	301
	References	313
	INDEX	315

CHAPTER 1

Millimeter-Wave Dielectric Properties of Materials*

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I. INTRODUCTION	1
II. ELECTROMAGNETIC QUANTITIES	5
III. DEFINITION OF A LOW-LOSS MATERIAL	6
IV. MEASUREMENT METHODS	6
A. Dispersive Fourier Transform Spectroscopy	7
B. Mach-Zehnder-IMPATT Spectrometer	12
C. Open-Resonator Method	14
V. DISCUSSION	17
A. Absorption Effects	18
B. Dispersion Effects	19
APPENDIX: COMPENDIUM OF DATA	20
A. Fused-Silica Glass (SiO_2)	20
B. Alumina (Al_2O_3)	20
C. Beryllia (BeO)	20
D. Corning Macor	21
E. Corning 9616 Green Glass	21
F. Silicon	21
G. Gallium Arsenide (GaAs)	22
H. Zinc Sulphide (ZnS)	22
I. Zinc Selenide (ZnSe)	22
J. Fluorocarbon Electronic Cooling Fluids	22
K. Dow Corning Dimethyl Polysiloxane	23
REFERENCES	41

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 ($\tan \delta$) 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, $\pm 10\%$ 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

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

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 “ $\tan \delta$,” 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, $\tan \delta$, which is sometimes unreliable at millimeter wavelengths.