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PRECISE MEASUREMENT OF REFRACTIVE INDEX AND ABSORPTION COEFFICIENT OF NEAR MILLIMETER WAVE AND FAR INFRARED MATERIALS

FINAL REPORT

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It is no longer necessary to use extrapolated microwave dielectric values when designing millimeter wave components and systems. Our recent highly accurate broadband millimeter wave data on complex refractive index, complex dielectric permittivity and loss tangent are now available to engineers for a variety of materials such as common ceramics, semiconductors, crystalline and glass materials. The fact that dielectric loss increases with frequency in the millimeter, unlike the microwave, is an important feature of our data. Reliable measurements also reveal that the methods of preparation of nominally identical specimens can change the losses by a factor of three.

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Until recently there has been almost no reliable data available in the millimeter and near millimeter wavelength (60 GHz to 600 GHz) range because measurements of the dielectric properties of materials at these wavelengths are extremely difficult 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 waves. More recently, a measurement good to ten percent 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 black body source such as mercury vapor lamp to be used. It normally 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 one of us (Afsar) to provide data from 5 mm (60 GHz) into the submillimeter. [1] New theories were also developed by Afsar giving a full treatment of all beams and interface effects [2,6] and great care was taken to increase the efficiency of energy throughput and detection. [1,7,8] 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. [1,8] Since we employ a quasi-optical tehcnique, 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 via Maxwell's relations. The present day dispersive Fourier transform spectroscopic (DFTS) technique of 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. [1-9] 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 can not ordinarily carry through amplitude information with higher precision and reproducability. [1,8]

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. [1,8] There are notable differences in absorption coefficient in Al_2O_3, ceramic alumina, depending upon 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. [1] Therefore, it is essential now that a full description of a material be available along with accurate, reproducible measurements of its dielectric properties. Thus, it has now been shown that not only is a microwave measurement of loss tangent untrustworthy at millimeter wavelengths also can be inaccurate and irreproducable.

The important differences in nominally-identical specimens can only be detected, verified and understood by using the most sophisticated-highly sensitive-highly stable equipment backed by most detailed evaluation of the theory of the technique. Therefore, it is important to rely upon a "center of excellence" as a source of practical data. Our "Digest of Millimeter Wave Materials Information and Measurement" is now available for distribution.

When reliable and reproducible millimeter wave data become available for a sufficiently large variety of materials as in the case of submillimeter wave data, the origin of the losses can be discussed. In the meantime, we have been able to develop a few clues which, nevertheless, should be helpful in engineering design because all of these materials (GaAs, Si, Al_2O_3, SiO_2, BeO, ZnSe, ZnS, spinel) are widely used in modern electronic systems. For example, in some materials, high purity is not as important as the manufacturer's method or preparation. We also know that, for the semiconductors, the highest resistivity is needed to prevent free carrier absorption and electron plasma reflection.

Absorption Effects:

The good news is that all of these are low-loss materials. The bad news is that the absorption coefficient varies among nominally-identical specimens depending upon different manufacturer's methods of preparation. The fundamental parameter, α, the absorption coefficient, is the reliable figure of merit for comparison of materials. Whenever one tries to use ε'' or $\tan\delta$ as a comparative measure of dielectric losses, they may be misled because ε'' and $\tan\delta$ are frequently dependent

It is surprising that such a common material as SiO_2 (fused silica glass) should be an excellent example of this problem. The absorption coefficient profile of Corning U.V. Grade glass is nearly the same as that of SiO_2 deliberately contaminated with a heavy ion, 7% TiO_2. On the other hand, the lowest loss material on the entire list is water-free SiO_2 from Thermal American Fused Quartz Company. Before we all begin to think that some reliable rules have been developed such as the insensitivity of the absorption to heavy ion contamination. We should note that MACOR, the Corning machineable ceramic, has ten times higher loss than any material in our list.

Hot pressed ceramics have much lower losses than cold pressed ceramics of the same chemical composition. For example, hot pressed BeO containing ¼%lithia flux made by Union Carbide, exhibits 42% less absorption loss at 300 GHz than the cold pressed Ceradyne Cerraloy 418 S 99.5 beryllia.

Somewhat the same effect must occur in the case of ceramic Al_2O_3 because we were surprised again when AL995 had lower losses than AL999 despite the presumption that the latter has higher purity. Crystal sapphire (Al_2O_3) exhibited only half the absorption loss compared to any ceramic alumina, as expected, but was not better than Thermal American water-free fused silica.

The extrapolated microwave data upon which we have been depending in the past for guidance to low-loss millimeter wave materials was the most misleading in the case of magnesium aluminum spinel. Having been led to expect that hot pressed $MgAl_2O_4$ spinel would be an order of magnitude better than alumina, we found only a 17% at 150 GHz.

The semiconductors such as GaAs and silicon are a very special case because of millimeter wave free-carrier absorption and electron plasma reflection. The highest possible room-temperature resistivity must be specified to assure the smallest number of electrons in the plasma. High resistivity, quoted in ohm-cm, is different for different semiconductors having different electron effective masses and different dielectric constant. For example, a single crystal of GaAs having a low resistivity of the order of magnitude of 10^4 ohm-cm was opaque but excellent low-loss measurements were obtained with high resistivity ($\sim 10^8$ ohm-cm) high purity single crystal Gallium Arsenide specimens. In the case of silicon, a high resistivity of 10^4 ohm-cm was satisfactory while 10^2 ohm-cm was a high loss material.

In some cases the parameter $\tan\delta = \varepsilon''/\varepsilon'$ can be misleading in millimeter wave technology. The measurements on low-loss liquids provide us with a convenient illustration of this although the same phenomenon is present in solids to a laser extent. While the absorption coefficient increases with frequency, $\tan\delta$ decreases for low loss liquids such as fluorocarbons and cyclohexane. Increasing absorption is fundamental to all liquids, polar and non-polar, because we are on the tail of a broad submillimeter absorption band. Why, the, should $\tan\delta$ decrease with frequency giving us exactly the opposite impression of the losses? The trouble arises in $\varepsilon'' = \delta n/2\pi\tilde{\nu}$ when the product αn fails to increase with frequency as rapidly as $\tilde{\nu}$. Then the slope of ε'' is negative where the slope of δ is positive.

As standard dielectric reference materials, [10] the liquids surpass all of the solids because of the control over manufacturing processes and microqualitative chemical analysis. We have selected two groups of electronic coolant fluids to be included in our measurement program. The Dow Corning dimethyl siloxanes have higher cooling capacities but also higher dielectric loss than the 3M fluorocarbons. The fluorocarbons are available in several chemical compositions and generally exhibit as low dielectric loss as we have seen in the best solids.

Dispersion Effects

Normally the magnitude of the refractive index, $n \simeq (\varepsilon')^{\frac{1}{2}}$ is limited in accuracy and reproducability to three, sometimes four, significant figures in most measurements. This dielectric constant, ε' is indeed nearly constant as a function of frequency and is adequate for all engineering applications. Nevertheless, serious problems arise when one must assess the engineering consequences of differences among siblings in a batch of material, different sources of the same material, different methods of preparation, different aging processes such as neutron irradiation or high-power electromagnetic radiation, and environmental changes in properties caused by assimilation of water vapor of chemical pollutants. These problems can only be solved by using the dispersive Fourier transform spectrometric method which uniquely provides the refractive index to six significant figures at millimeter wavelengths in low loss materials. Our refraction spectra of fused silica, alumina, beryllia, gallium arsenide, silicon, zinc sulphide, zinc selenide,&fluorocarbon fluids show massive features as a function of frequency in the fifth significant figure and fine structure in the sixth figure. The differences can be determined simply by inspection.

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