Precise Millimeter-Wave Measurements of Complex Refractive Index, Complex Dielectric Permittivity and Loss Tangent of GaAs, Si, SiO_2, Al_2O_3, BeO, Macor, and Glass

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Abstract—**Highly accurate continuous spectra of the complex refractive index and complex dielectric permittivity are given in the millimeter range for a variety of potentially useful materials. The absorption coefficient is found to increase monotonically with increasing frequencies. Small amounts of glassy inclusions or water were found to increase losses at all frequencies, but impurities and radiation damage (except in semiconductors) have not yet proved to be detrimental to performance. Materials have been found for which the millimeter-wave losses can be tolerated when used as dielectric waveguide, high-power windows, and other applications. Nominal consideration must be given, however, to the conditions of preparation and the nature of contaminants. The measurements were made in a modular, polarizing, dispersive Fourier-transform spectrometer.**

I. Introduction

ALMOST NO information is available from the literature on low-loss millimeter-wave materials. Data of high reliability and, preferably, of high accuracy are required for such applications as millimeter-wave dielectric waveguides, windows and lenses for high-power millimeter-wave sources and systems, quarter-wave plates, and other elements analogous to those commonly used in optical systems. We report here continuous spectra of absorption coefficient α, refractive index n, real ϵ' and imaginary ϵ'' parts of the dielectric constant, and loss tangent $\tan\delta$ on a wide variety of materials as a function of wavenumber $\bar{\nu}$ in cm^{-1}, frequency ν in gigahertz and wavelength in millimeters. These materials are known to be nearly transparent in the microwave regions, but exhibit near-opaque characteristics in the submillimeter-wave regions. In the intermediate millimeter-wave region, one needs to know accurate values of optical and dielectric parameters in order to judge their suitability for the device application and, subsequently, to design the devices. We report refractive index data of accuracy better than five decimal places,

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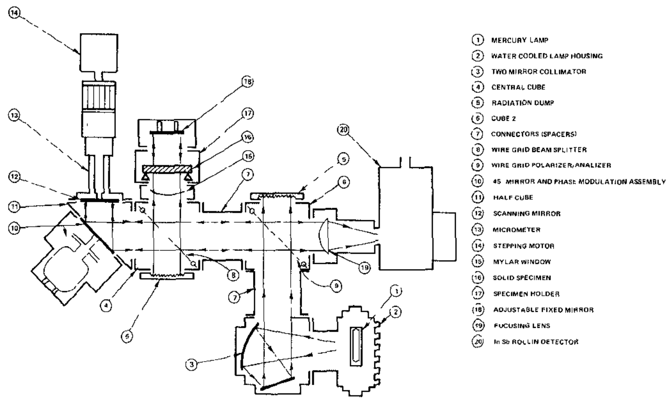

Fig. 1 Modular polarization interferometer for millimeter-wave measurements of complex refractive index and complex dielectric permittivity of low-loss solids.

and absorption coefficient better than one percent. Although any of these low-loss materials are suitable for devices, we intend to provide optical and dielectric parameters of many other potential materials, whenever we can borrow adequate specimens of them. Results reported in this paper cover the wavelength range 5–0.66 mm (2–14 cm^{-1}).

II. Experimental

For broad-band continuous data we have employed a free-space optical technique, namely, dispersive Fourier-transform spectroscopy (DFTS) developed earlier by Afsar [1]–[5]. This perfected [6] DFTS technique reduces markedly all random and systematic errors arising from the system, including reflection and multiple-reflection contributions at specimen surfaces, electronic instability, and gain settings. It is necessary to use a highly responsive and sensitive InSb Rollin hot electron detector and free-standing wire-grid polarizing beam splitters [1]–[5], in order to cope with low-power output of the mercury-vapor lamp source at millimeter wavelengths. The modular design of our interferometer shown in Fig. 1 uses the National

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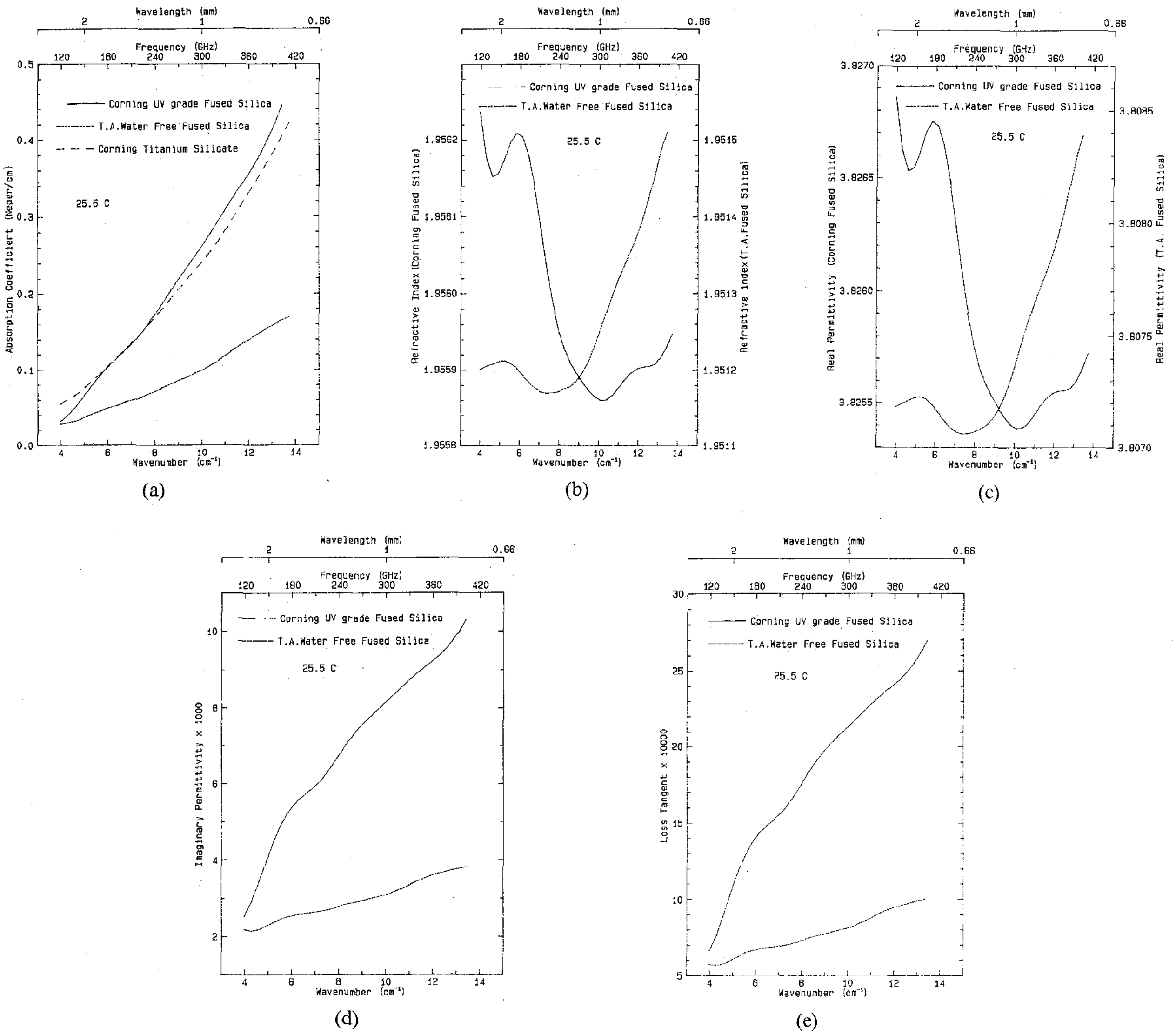

Fig. 2. (a) Absorption coefficient spectra obtained with Corning UV-grade fused silica, Corning titantium silicate, and Thermal American water-free fused silica at 25°C. (b) Refractive index spectra for Corning UV-grade fused silica and Thermal American water-free fused silica. (c) Comparison of real part of permittivity of UV-grade and water-free fused silica. (d) Comparison of ϵ'' spectra of UV-grade and water-free fused silica. (e) Loss tangent spectra for UV-grade and water-free fused silica.

Physical Laboratory, UK-type cube configuration. In the polarization interferometric mode [1]–[4], with a pair of wire-grid polarizers (made with 10-μm diameter tungsten wire), the transmissivity is constant up to a cutoff frequency which is inversely proportional to the grid spacing. With wire spacing of 25 μm, the cutoff wavenumber becomes 200 cm^{-1} (wavelength of 50 μm). In DFTS, the specimen rests in one of the active arms of the two-beam interferometer and produces a phase change as well as the attenuation in the interference pattern. The ratio and the difference of Fourier-transformed modulus and phase spectra of interferograms obtained with and without the specimen lead to the determination of the continuous absorption coefficient and refractive index data simultaneously. Once the initial spectra are obtained, it is necessary to use a rigorous iterative procedure in order to separate out reflective and transmissive contributions [4]. Separation of reflective and transmissive contributions in a conventional FTS is extremely difficult. Moreover, the specimen also acts as source of radiation when it is placed in the passive arm of an FTS interferometer. Thus, a conventional FTS provides erroneous values of the absorption coefficient [2], [4]. (The refractive index cannot be determined directly by conventional FTS.) For a low-loss material, uncorrected transmission spectra may contain as much as 90-percent surface-reflective loss contributions. Once the basic optical parameters, the absorption coefficient, and the refractive index are determined on an absolute scale, both real and imaginary parts of dielectric permittivity and loss tangent can be derived via the following equations:

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

$$\epsilon'' = n\alpha/2\pi\tilde{\nu}$$

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

The DFTS method requires a highly stable reproducible interferometric system in order to generate precise phase information. Extreme care is therefore taken to keep interferometer arms and room temperature constant. The inter-

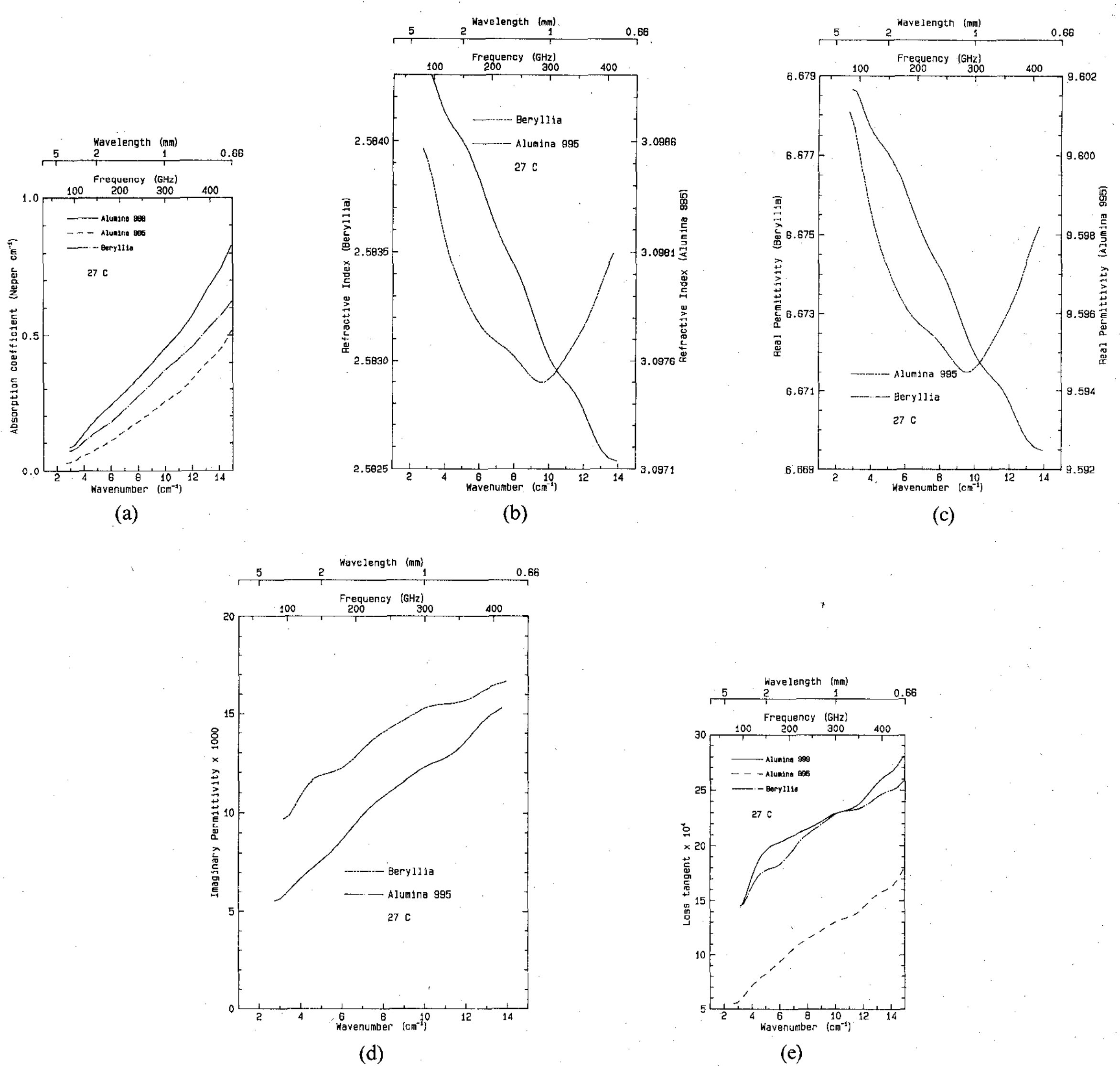

Fig. 3. (a) Comparison of absorption coefficient spectra of alumina 995, alumina 999 (vistal), and beryllia at 27°C. (b) Refractive index spectra for alumina 995 and beryllia at 27°C. (c) Comparison of ϵ' spectra of alumina 995 and beryllia at 27°C. (d) Comparison of ϵ'' spectra of alumina 995 and beryllia at 27°C. (e) Loss tangent spectra for alumina 995, alumina 999, and beryllia at 27°C.

ferometer temperature is controlled by a fluid from a temperature control unit. The main part of the interferometer is evacuated; the specimen chamber is flushed with dry N_2 during measurement. Specimen surfaces were flattened to 1/4 wavelength in the visible and made parallel to about 1 s. It is also necessary to employ stable electronic components. A step change in the attenuator or the gain knob in a lock-in amplifier, and inclusion of the marked gain factor in the calculation, can lead to a systematic change in absorption data. We keep the gain unchanged during sets of measurement and resolve the interference signal by higher digital discrimination (22 bits, ± 2.000001 V).

III. Results

The data presented in this section show that water-free fused silica and high-resistivity single-crystal GaAs have the lowest loss in the 1–3-mm wavelength range. Then, single-crystal Al_2O_3 (sapphire), ceramic alumina (Al_2O_3), beryllia (BeO), UV-grade fused silica (SiO_2), and silicon exhibit somewhat higher losses. The Corning 7971 fused silica containing 7-wt% TiO_2 did not exhibit higher loss than UV-grade, proving that heavy-ion contamination is not detrimental. Neither did moderate neutron irradiation of Al_2O_3 and BeO prove to be detrimental, so far. Different ceramic binders and OH^- inclusions appear to introduce severe losses in specimens that are otherwise nominally identical. Measurements on Corning Macor and Corning 9616 green glass revealed a consistently higher loss across the spectrum by an order of magnitude.

A. Fused Silica and Titanium Silicate

Fig. 2(a)–(e) shows results for fused silica and titanium silicate. Fig. 2(a) represents absorption coefficient spectra for Corning 7940 UV-grade fused silica, Corning 7971 titanium silicate, and Thermal American water-free fused silica over the wavenumber range 4–14 cm^{-1} (2.5–0.66 mm). It appears that titanium silicate with 7-wt% TiO_2 did not change the absorption profile of SiO_2. Water-free fused silica from Thermal American Fused Quartz Co. shows much lower loss in the entire region. This illustrates that some foreign molecules can contribute to the absorption value of two nominally identical materials. Fig. 2(b) and (c) gives refractive index and ϵ' spectra for both fused silica. Refractive index values can be read to the fifth decimal position from the scale. The dielectric permittivity and refractive index are constant and identical in both materials up to three figures. In the fourth and fifth figures, significant differences can be seen both in level and in features. The absolute value of the refractive index is accurate to ± 0.00001. It is now possible to measure differences from specimen to specimen, batch to batch, and in the deviant specimen from its siblings in a batch by observing the highly resolved refraction profile and fine features. In the case of a low-loss material, ϵ' is essentially the square of the refractive index. It repeats all of the features found in the refractive index. Fig. 2(d) and (e) shows imaginary parts of the dielectric permittivity (ϵ'') and loss tangent spectra for both types of fused silica. The term ϵ'' is a function of both the refractive index and the absorption coefficient. When the absorption coefficient values of material are low, the refractive index values also play an active role in ϵ'' and subsequently in $\tan\delta$. It appears from this, that for low-loss materials, the absorption coefficient is a reliable "figure of merit" when different materials are compared. The ϵ'' and $\tan\delta$ are contaminated by n and these parameters (ϵ'' and $\tan\delta$) are not highly reliable figures of merit.

B. Alumina and Beryllia

Both alumina and beryllia are potential candidates for windows in high-power source applications such as gyrotrons. Fig. 3(a)–(e) shows results for alumina and beryllia over the wavelength range 3–0.66 mm. We see in Fig. 3(a) the absorption coefficient for all three ceramics (alumina 995, alumina 999, and BeO) increases monotonically with decrease in wavelength. Alumina 995 exhibits lowest loss among these ceramics followed by BeO and alumina 999. The higher loss in alumina 999 (vistal) may be caused by the ceramic binder. We have not yet investigated differences among specimens of alumina 995. Refractive index, real (ϵ') and imaginary (ϵ'') parts of dielectric permittivity, and loss tangent ($\tan\delta$) spectra for alumina 995 and beryllia are shown in Fig. 3(b)–(e). Again, the highly resolved refractive index and the ϵ' spectra show details of these ceramics. Loss tangent values for alumina 995 are very small. This is because of the division of the ϵ'' term by high ϵ' values of alumina 995. As in the case of fused silica, ϵ'' and $\tan\delta$ are not reliable as figures of merit for these low-loss ceramics.

C. Comparison of α of Fused Silica with Alumina 995 and Crystal Sapphire

We have noticed earlier that water-free fused silica exhibited low loss (absorption coefficient) compared to Corning 7940 UV-grade fused silica. It is believed that inclusion of a foreign molecule, perhaps OH^-, has caused the extra loss in UV-grade fused silica. Fig. 4 shows the comparison of the absorption coefficient of sapphire with alumina 995 and fused silica. Thermal American water-free silica still stands as the lowest loss material, but is now followed closely by sapphire. Further investigation on crystal sapphire down to the 2-cm^{-1} region is in progress. Neutron irradiated alumina 995 and beryllia (irradiation of up to 10^8 rad) have not exhibited any significant difference compared to virgin specimens of alumina 995 and BeO, although BeO changed its color from white to grey and alumina 995 changed from white to yellow [7].

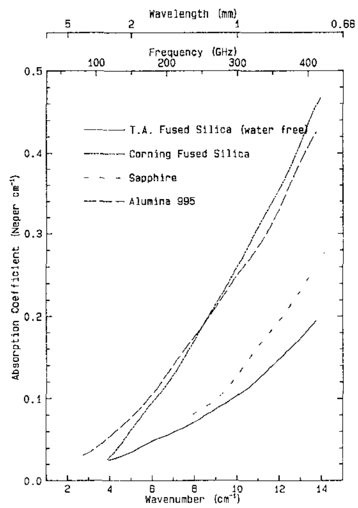

Fig. 4. Comparison of absorption coefficient of Thermal American water-free fused silica, crystal sapphire, alumina 995, and Corning UV-grade fused silica at ambient temperature.

D. Corning Macor and Corning Green Glass

Corning Macor machinable ceramic is a potential candidate for a window material in applications where precision machined elements are needed. Corning 9616 is the lithium alumino-silicate green glass. Both of these materials are found to exhibit one order-of-magnitude higher absorption coefficient than the common low-loss ceramics described earlier in this paper. Data for the absorption coefficient over the range 5–0.66 mm (2–14 cm^{-1}) are shown in Fig. 5(a). Like alumina and beryllia, the absorption coefficient increases with an increase in frequency. Refractive index and ϵ' data are shown in Fig. 5(b) and (c). For both materials, refractive index values fall sharply with an increase in frequency. Since Macor and green glass are not low-loss materials, ϵ' values are not simply the square of the n values. The loss term $(\alpha/4\pi\bar{\nu})^2$ in ϵ' now plays a strong role. The imaginary part of the permittivity (ϵ'') and the loss tangent ($\tan\delta$) are shown in Fig. 5(d) and (e). Now ϵ'' and $\tan\delta$ act better as "figures of merit" for representing comparative losses than was the case for low-loss alumina and BeO. It is consistent therefore that we find the loss tangent for glass and Macor to be almost exactly one order-of-magnitude larger than was measured for the low-loss ceramics. It may be difficult to find significant submillimeter-wave applications for the glass, but Macor may still be used at millimeter wave-

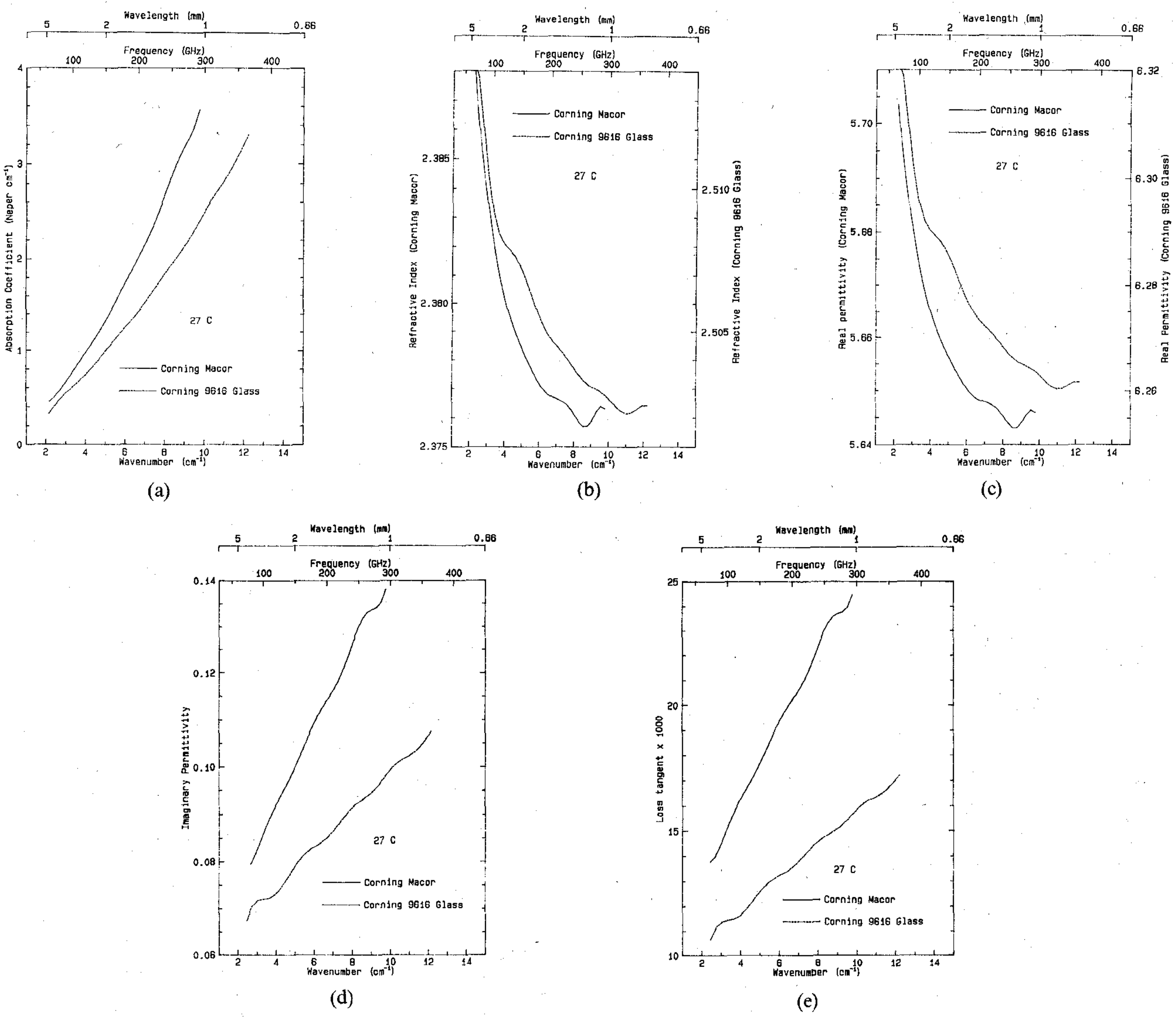

Fig. 5. (a) Absorption coefficient spectra for Corning Macor and Corning 9616 green glass. (b) Refractive index spectra for Corning Macor and Corning 9616 green glass. (c) Comparison of ϵ' spectra of Corning Macor and Corning 9616 green glass. (d) Comparison of ϵ'' spectra of Corning Macor and Corning 9616 green glass. (e) Loss tangent spectra for Corning Macor and Corning 9616 green glass.

lengths where precision machined elements are needed if the higher loss could be tolerated.

E. Gallium Arsenide and Silicon

Semiconductors like GaAs and silicon are in extensive use as diodes, transistors and field-effect transitors, and in integrated circuits. Both of these materials are also potential candidates for windows and dielectric waveguides in the millimeter-wavelength region. We have measured two GaAs specimens and one silicon specimen. One of the GaAs specimens was obtained from Microwave Associates Company (M/A-Com). It is a single-crystal Cr-doped (concentration of $5\times10^{15}/\text{cm}^3$) specimen with crystal orientation of [100]. Its resistivity is greater than 5×10^7 $\Omega\cdot$cm, and the room temperature mobility is 2500 cm^2/V-s. The second GaAs specimen is also a high resistivity Cr-doped single-crystal specimen ($\rho\approx7.8\times10^7$ $\Omega\cdot$cm, Cr-concentration $=2\times10^{16}/\text{cm}^3$) obtained from Hughes Research Laboratory, Malibu. The silicon specimen is an undoped high-resistivity ($\sim$ 8000 $\Omega\cdot$cm) monocrystal specimen purchased from General Diode Corporation. Absorption coefficient spectra of these three specimens are shown in Fig. 6(a). Silicon shows a flat absorption coefficient in the entire region except the appearance of a small bulge around 4.5 cm^{-1} (2.8 mm). This is not surprising for a material with such a high resistivity [8], [9]. M/A-Com GaAs exhibits almost similar behavior as ceramics and SiO_2. The absorption increases with an increase in frequency. Hughes high-resistivity GaAs shows a great potential since a plateau or a flat low-loss absorption is observed over the range 3–1.3 mm. At wavelengths shorter than 1.3 mm, the behavior is similar to M/A-Com GaAs. Refractive index and ϵ' spectra are shown in Fig. 6(b) and (c). All three semiconductor specimens show less refraction variation compared to low-loss ceramics and SiO_2. Shapes of n and ϵ' curves for both GaAs samples are similar. First it falls, then there is a near-flat region, and then it falls again. For silicon, initially the refractive index rises from

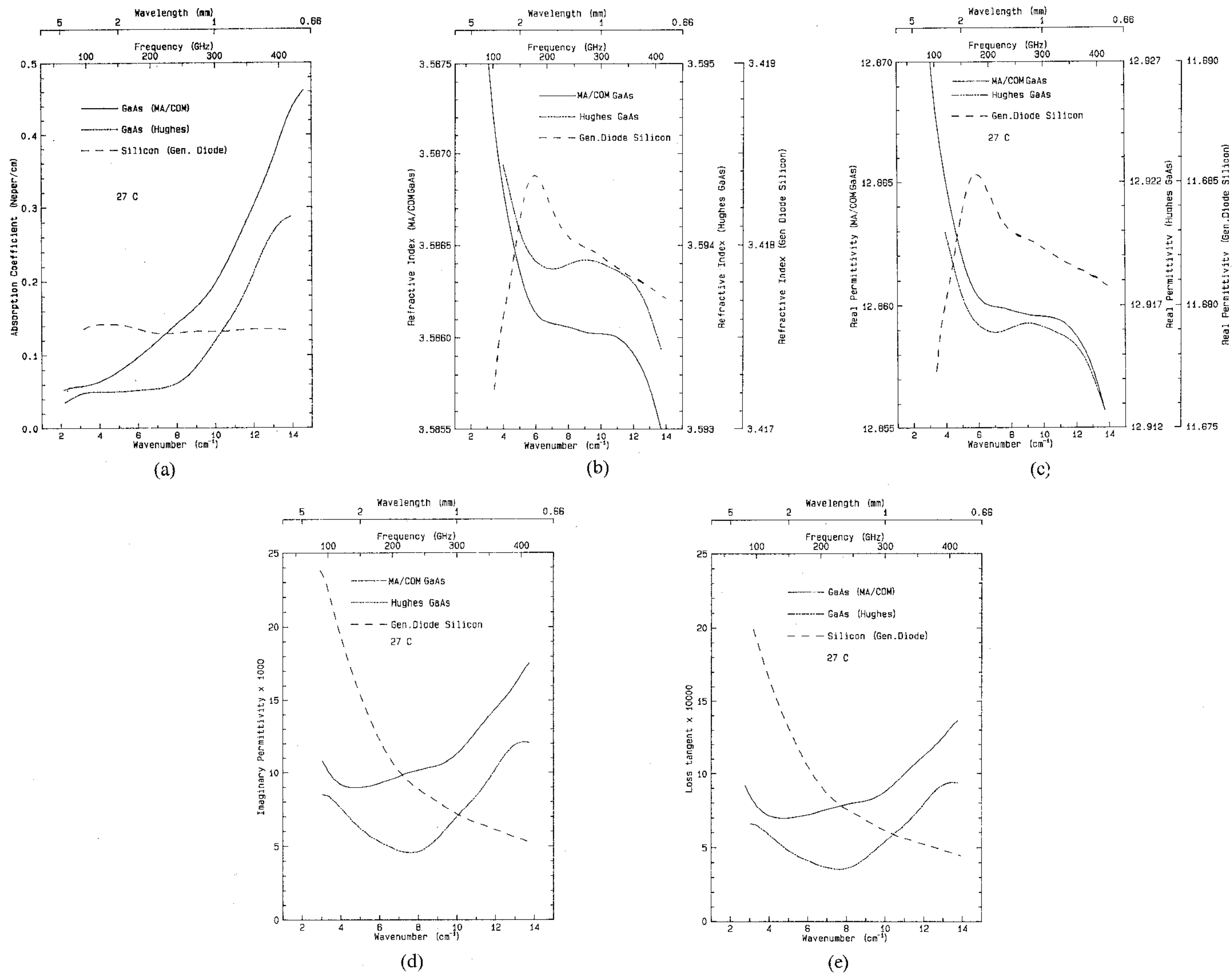

Fig. 6. (a) Comparison of absorption coefficient spectra of single crystal M/A-Com GaAs, Hughes high-resistivity (~ 8×10^7 Ω·cm) single-crystal GaAs, and General Diode undoped high-resistivity (~ 8000 Ω·cm) single-crystal silicon at 27°C. (b) Refraction spectra for single-crystal GaAs and silicon at 27°C. (c) Real part of dielectric permittivity spectra for single crystal GaAs and Silicon. General Diode silicon has resistivity of the order of 8000 Ω·cm. (d) Imaginary part of dielectric permittivity spectra for single-crystal GaAs and silicon at 27°C. (e) Comparison of loss tangent spectra of M/A-Com single-crystal GaAs, Hughes high-resistivity single-crystal GaAs, and General Diode high-resistivity single-crystal silicon at 27°C.

3.4166 at 3 cm^{-1} to 3.4184 at 5.8 cm^{-1}, then falls slowly to 3.4177 at 14 cm^{-1}. Fig. 6(d) and (e) shows spectra for imaginary permittivity and loss tangent. The refractive index factor again plays a dominant role in all these semiconductor specimens, particularly at the low-frequency end of ϵ'' and $\tan\delta$ curves. Like alumina, loss tangent values are low. This is because of the ratio of ϵ'' values to large ϵ' values of semiconductors.

IV. Specimen Description

All specimens we have measured are polished and flattened to about 1/4 wavelength in the visible and made parallel to about 1 s. This is important for reproducibility of refractive index data and for proper correction of surface reflection contributions. The Corning 7940 UV-grade fused-silica specimen is 50 mm in diameter and 17.068 mm thick. The Corning 7971 titanium silicate specimen is 75 mm in diameter and 14.871 mm thick. The Thermal American water-free fused-silica specimen is 75 mm in diameter and 21.6476 mm thick. Alumina 995 specimens were obtained from GTE WESGO- alumina 999 (vistal) specimens were obtained from Coors Porcelain Co., CO. Thicknesses of the alumina 995 and alumina 999 specimens used in present measurements are 49.942 mm and 27.1248 mm, respectively. The beryllia specimen is 75 mm in diameter and 35.8636 mm thick. It was obtained from Ceradyne, Inc. The sapphire specimen was obtained from Crystal Systems, Inc. Its thickness is 6.3656 mm. Corning Macor and Corning green glass specimens are 18.6864 mm and 13.6522 mm thick, respectively. Results of M/A-Com (#1089) GaAs presented in this paper were obtained with a 75-mm diameter and 54.5154-mm-thick specimen. The other high-resistivity GaAs was obtained from Hughes Research Lab., Malibu, CA. The specimen (#D3) dimensions are 75-mm diameter and 14.5465-mm thickness. High-resistivity silicon specimen was obtained from General Diode Corporation. Its dimensions are 75-mm diameter and 11.8125-mm thickness.

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