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FINAL REPORT

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PROPERTIES OF MATERIAL IN THE SUBMILLIMETER WAVE REGION

(Instrumentation and Measurement of Index of Refraction)

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PROPERTIES OF MATERIAL IN THE SUBMILLIMETER WAVE REGION

The "Properties of Materials in the Submillimeter Wave Region" study was initiated at Catholic University, Electrical Engineering department to instrument a sytem and to make measurements of the complex Index of Refraction in the wavelength region between 0.1 to 1.0 millimeters. While refractive index data is available for a number of solids and liquids there stil exists a need for an additional systematic study of dielectric properties to add to the existing data, to consider the accuracy of the existing data, and to extend measurements in this wavelength region for other selected materials. The materials chosen for consideration would be those with useful thermal, mechanical, and electrical characteristics. The data is necessary for development of optical components which, for example, include beamsplitters, attenuators, lenses, grids, all useful for development of instrumentation in this relatively unexploited portion of the spectrum.

At the outset of this study a literature search was made to determine the extent of the index data in the region between 3 mm and 50 microns. Attention was also given in this review to the type of instrumentation and the precision. The more recent method of index measurement in this wavelength region is typically by use of spacial interference patterns familiar in optics. Both the Michelson and Fabret Perot Interferometer have been popular choices. In gathering the information it was found that until the middle 70's instruments for dielectric measurements in the millimeter wavelength region mostly used methods based on the Prism or the Interferometer; including Fourier transform spectroscopy (FTS) and later dispersive fourier transform spectrocopy (DFTS). This instrumentation covered a wavelength range from 50 to 2000 microns.

The FTS instrument system generally includes a "black body" source and depending on the wavelength region a cooled detector as the major components in a Michelson Interferometer. Even though the energy available from black body source is weak in the submillimeter region, with the addition of the cooled detector, precision measurements are possible. The important feature of this FTS method is that it permits data collection over a continuous wavelength range. Analysis of the data is accomplished in the following manner. The detected output of the two path interferometer without sample is fourier transformed to give a spectral distribution of the instrument. This spectral distribution to be studied is then modified by introduction of the sample into one of the two paths. Both transmission and reflection methods are used. The detected signal from sample insertion contains modified spectral contributions as the signal strength V(x) at the detector varies with the variations of path length. The transform from the space to the frequency domain, Stone (13), is given by:

$$B_0(\nu) = \int_{-\infty}^{+\infty} F_0(x) \cos\left(2\frac{\pi\nu}{\sigma} x\right) dx$$

In order to determine $B_o(\nu)$ which is directly related to $n(\nu)$ the index of refraction, a relation between V(x), the voltage output, of the detector and $F_o(x)$ must be determined. This is complicated because of asymmetries of the instrument between the functions V(x) and F(x) with reference to zero path difference. In such cases the extraction of $B_o(v)$ requires extensive calculations. Generally the instrumentation is computer controlled which allows for both automatic data acquisition as well as analysis.

The recent availability of the laser source with its relatively high power and monochromatic output in the submillimeter wave length region provides for the possibility of precision measurement at discrete wavelengths. Since the signal to noise ratio with this method for the longer wavelength, is high, this data can also be used to check the accuracy of the broadband FTS data. Stone (13), at UK National Physical Laboratory NPL using a Mach-Zehnder interferometer configuration with laser source were able to make index measurements on both "weak and strong" absorbing materials. Depending on the type of material either transmission or reflection methods are adopted to measure both the real and imaginary parts of the dielectric permativity. With the availability of a calibrated attenuater (rotating grid type discussed later) loss measurements are considerably facilitated.

The Mach Zehnder interferometer is an amplitude splitting two beam device containing two beamsplitters (see figure 1). The two beams formed by the first splitter propagate along separate paths and are finally recombined at the second beam splitter after which a resulting "fringe" pattern may be detected. For the index measurement a sample of either fixed or variable thickness is placed in one leg of the interferometer resulting in fringe shift. Measurement of this shift leads directly to determination of the index.

In somewhat more detail the measurements are made as follows: attenuation measurements are made on samples of varying thickness (liquids or solids) using one leg of the Mach Zehnder Interferometer. This is accomplished by placing the sample in series with calibrated attenuator both of which are placed in one arm of the interferometer. The detected signal is maintained at constant level by adjustment of the attenuator as the thickness of the sample is varied. With the attenuator in series, a rotation θ of the center grid of the attenuator allows the sample output power versus sample thickness, t, to be constant. The attenuation α in the sample is related by

$$\ln(\cos\theta) = \alpha l + \text{constant}$$

The angle θ represents rotation of the center grid of the attenuator with respect to the reference grids.

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14. The Far Infrared Performance and Application of Free Standing Grids Wound From 5um Siameter Tungsten Wire", W. G. Chambers, Infrared Physics, Vol. 19, 1979, pp. 437-442.

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17. "Self Calibrating Grid Attenuator for use at Submillimeter Wave Lengths", J. Stanforth and J. Allnutt., Proc. IEE, Vol. 118, No. 2, 1971, pg. 343.

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Listing of available real part - Table I and Loss Tangent part - Table II of dielectric constants from the literature study.

TABLE I - REAL PART OF THE DIEELECTRIC CONSTANTS

MATERIAL/FREQ.	a 230 GHZ	b 343 GHZ	c 250/450 GHZ	c 850 GHZ	d 353 GHZ	c 1,300 GHZ	c 2,000 GHZ
Fiberglass			4.34				
Plexiglass		2.61	2.62				
Polyethylene		2.31	2.27	2.333	2.359	2.335	2.34
Polyethylene*					2.298		
Polystyrene		2.57	2.48	2.520	2.528	2.531	
Ebonite					1.665		
Pyrex			4.46				
Rexolite		2.54	2.52	2.525		2.520	2.52
Teflon	2.06	2.09	1.97	2.042	2.033	2.033	2.05
Delrin				2.80		2.78	2.80
Fused silica	3.88			3.28		3.27	3.31
Dynasil							3.85
TiO2 ceramic	88.36						

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d - Chigryal, E.E., "Measurement of Refractive index of Solid and Liquid Dielectrics at Submillimeter Wavelength. Institute of Radio engineering and Electronics, Academy of Sciences of the USSR, Moscow. Translated from Pribory i Tekhnika Eksperimenta, No. 6, pp. 146-148, Nov.-Dec. 1974

TABLE II - DATA* LOSS TANGENT (X1000)

MATERIAL/FREQ.	400 GHZ	500 GHZ	600 GHZ	700 GHZ	800 GHZ	900 GHZ	1000 GHZ
Fiberglass	41	41	43				
Plexiglass	20	20	36				
Polyethylene	1.9	1.7	1.5	1.4	1.3	1.3	1.3
Polystyrene	3		5				7
Ebonite							
Pyrex	28		40				
Rexolite	1		3				5
Teflon	1		2				2
Delrin	17	20	21	23	24	24	
Fused Silica	1.7	2.7	3.6	5.1	5.9	6.2	6.4
Dynasl							
TiO2 ceramic							

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*Final Report, "Millimeter and Submillimeter Wave Dielectric Measurements with Interference Spectrometer", A. McSweeney and A. Sheppard, Project No. A-934, Georgia Institute of Technology Engineering Experiment Station. Contract Number 991(13), 14 April 1970.

INSTRUMENTATION:

One of the main objectives of this grant was to develop instrumentation for the measurement of complex dielectric properties in the SMMW range. The plan agreed upon with N.McAvoy (Technical officer for this contract), for obtaining the necessary equipment was that CUA would develop the dielectric measurement instrument, purchasing the CO/2 pump laser and miscellaneous equipment thorough the contract and that NASA would supply a SMMW laser Fig. 4a,b which was their design. Mr. McAvoy would coordinate the SMMW laser effort closely with CUA.

After reviewing the literature on dielectric measurements in solids, we decided as indicated in the preceding discussion to use a Mach-Zender interferometer (Fig. 1), Allnutt (15) with modification by Stone (13) to measure the phase and loss properties of the sample. The phaser shifter, attenuator and beam splitter were not commerically available components and therefore had to be developed. The phase shifter (phase delay) was constructed using pairs of mirrors mounted in a parallel "V" arrangement on a translation stage with a differential micrometer drive capable of direct reading to 1/10,000 inches. The phase shifter was constructed and is shown in Fig. 2.

The calibrated attenuator was designed using a technique described by, Stanforth (17), which employes two fixed grids and one rotating grid, Fig. 3. An initial design of the attenuator was assembled using a set of grids with a symmetrical 50 micron period fabricated by J.Lamb of NASA. The unit employs an ORIEL rotating stage with micrometer drive which allows for rotation of the center grid. The 50 micron grid set was the only one available to us at the time and did not result in a workable attenuator because of the large grid size. A final design of the attenuator awaited fabrication of a desired 5 micron grid spacing. J. Lamb was studing methods for producing grids of this spacing for a number of other applications.

A SMMW laser was to be assembled at CUA. The major parts were supplied by NASA. Basically the laser was constructed using a pyrex tube 1-1/2" in diameter with a metal input mirror and a silicon output mirror forming the resonant cavity. The input mirror is metal with gold plating having a 6.3 meter radius of curvature and a 4 millimeter off axis coupling hole. The output mirror has a 6 meter radius with a reflecting coating for 10.6 micron (CO_2), and an antireflecting (AR) coating for the SMMW. The SMMW energy was coupled out of the cavity by transmission through an on axis 4 mm circular aperture. The assembly and alignment of the SMMW laser was closely coordinated with N.McAvoy who participated in bringing this first laser to the operational stage. Since initial study was to be in the range of 100 micron wavelength Methyl Alcohol was selected for the active medium. Methyl Alcohol has a strong SMMW line at 118 microns.

When finally made operational the laser emitted a sufficiently strong 118 micron signal. However, this SMMW signal was quite unstable and very often would completely disappear within one minute. After many adjustments to improve the stability without substantial success, it was decided to continue with another part of the instrumentation, i.e., assemble the Interferometer while continuing to look for a solution to the stability problem. It would not be possible to make measurements until the cause of the system instability was removed.

Subsequently, Mr. McAvoy recommended that a SMMW laser of different design, which had recently been developed by his group at NASA, be substituted for the original SMMW laser system. This new system would perhaps provide improved stability. The new SMMW laser is longer than the original, approximately 1.75 meters compared with 1 meter; provides more power and has an improved cavity tuning adjustment. The tuning head of our original laser was quite difficult to adjust. The new laser was subsequently assembled and put into operation but did not solve the instability problem. It was then considered that the main cause of instability may be in the CO/2 laser system. A phase-lock system was obtained to provide additional stabilization to the CO/2 pump laser. Fig. 4a,b. are photographs of the CO/2 laser and SMMW output tuning used. This new equipment was put into operation, however the stability of the SMMW signal was not substantially improved although there were some periods of several minutes when the SMMW signal remained somewhat constant.

An attempt was made to measure the attenuation/length of teflon at 118 micron without the precision attenuator since grids were not available. The procedure used was to illuminate sample rods of the same diameter of different lengths and measure transmission loss. Assuming detector linearity, a plot of the resulting attenuation versus sample length yields a curve with a slope equal to the attenuation constant of the material. The data obtained in this manner for teflon was not reliable, due to laser instability. Measurements could not be made with this system.

While the system could not be used for making index measurements - rough studies on beamsplitters were also attempted. It was desirable to have a beam splitters for the Mach Zehnde interferometer that would divide the power approximately equally. We initiated a beamsplitter design using grids which from the literature data appeared to have desirable characteristics. NASA supplied us with two 50 micron grids and one 10 micron grid on .002" Kapton for experimentation. Transmission and reflection measurements were made on each grid with the angle of incidence equal to 45 degrees for both perpendicular and parallel polarization conditions. The results indicated that at 118 microns, the 50 micron grid splits the power equally regardless of polarization.

In conclusion, it would be appropriate to review chonologically the major problems encountered which lead to the results obtained:

Contract was awarded September 1976.
Initial ordering of Optical Compontents - January 1977.
Initial ordering of components for SMMW Laser - March 1977.
$CO/2$ Laser arrived - June 1977.
Building components for Mach Zehender Interferometer started and SMMW System in process of Assembly - June 1978.
SMMW Laser assembled and aligned. Start up using 1 methyl alcohol - no signal - March 1979.
Continued effort, and obtained first operative results but with severe stability problem. Attempts at measurements - June 1979.
Substitution of the "new" SMMW Laser from NASA Arrived and assembled - no improvement in stability - June 1980.
Continuing attempts to find source of instability lead to another review of entire system. Temperature stability appeared to be a major problem - Aug. 1980.

Request for renewal without additional funding was approved for the year starting August 1980 and the SMMW effort was continued. Assembly of a temperature control system somewhat improved the stability problem. It was decided that temperature controlled refrigeration system would substantially improve the stability and allow measurements. The contract terminated August 31, 1980 and the effort continued without funding.

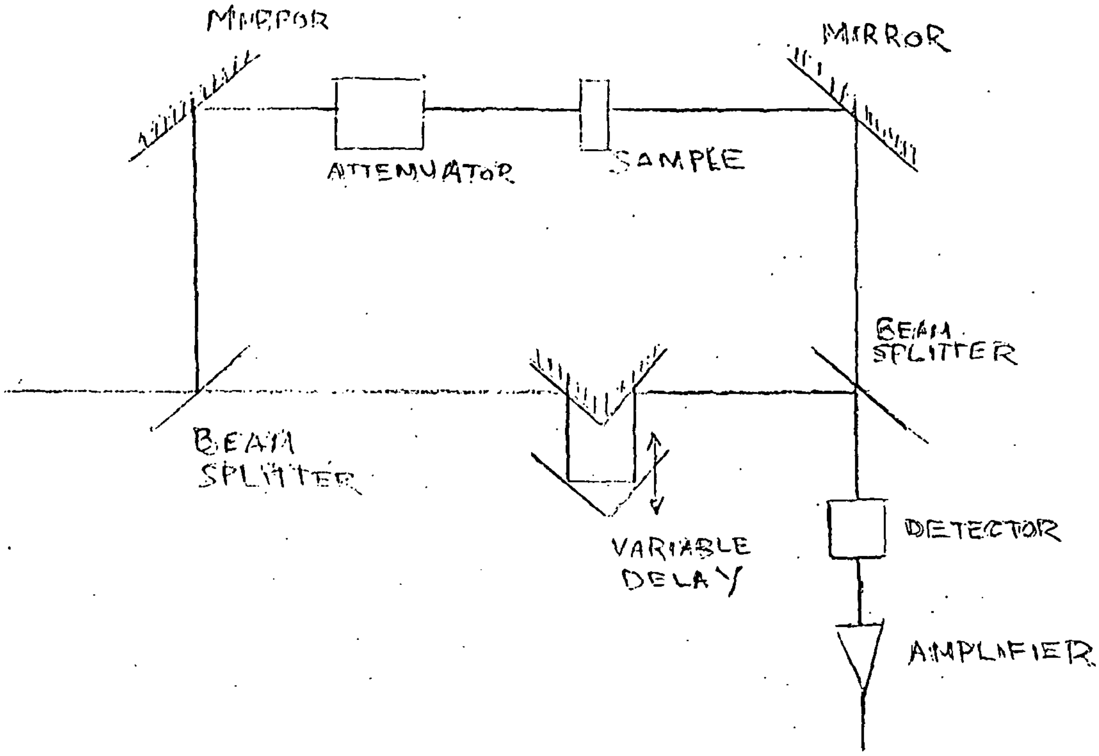

Figure 1 - Mach-Zender Interferometer

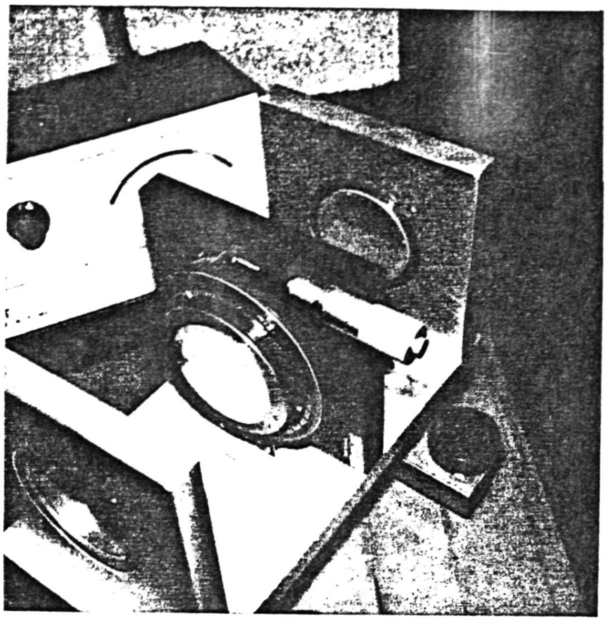

Figure 3 - Variable Attenuator

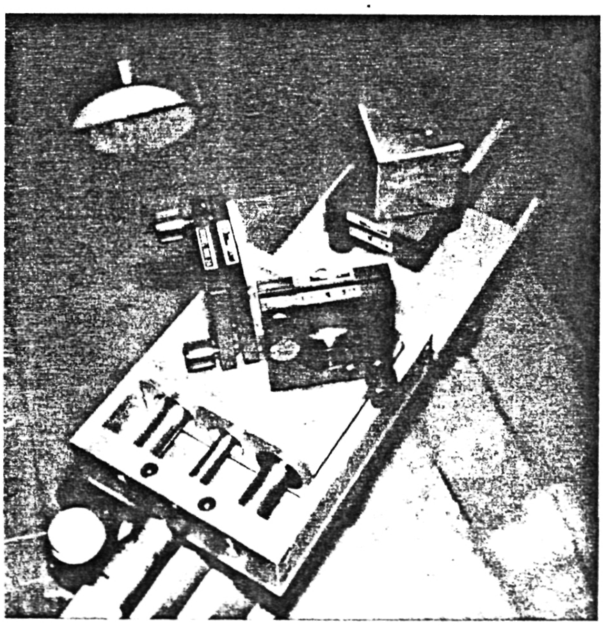

Figure 2 - Phase Shifter

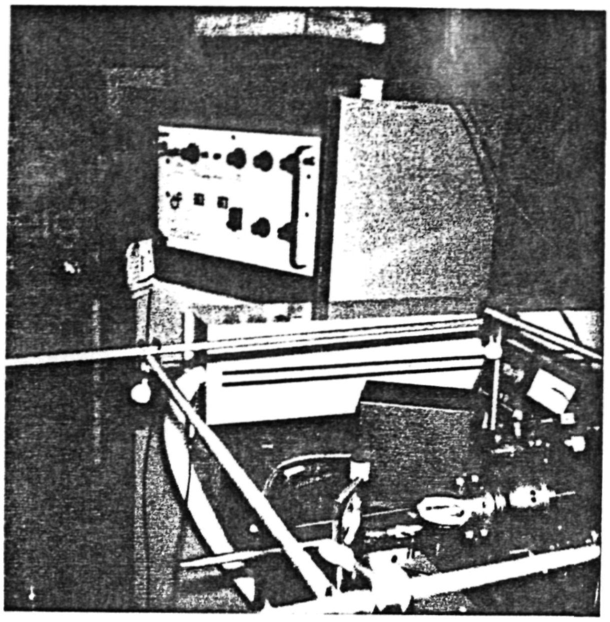

Figure 4a - CO_2 Laser System

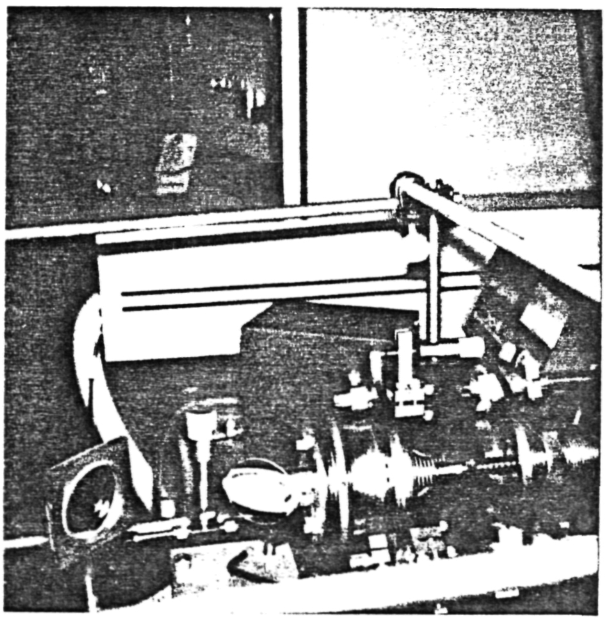

Figure 4b - SMMW Output Window and Tuning Head