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Optical Materials Characterization

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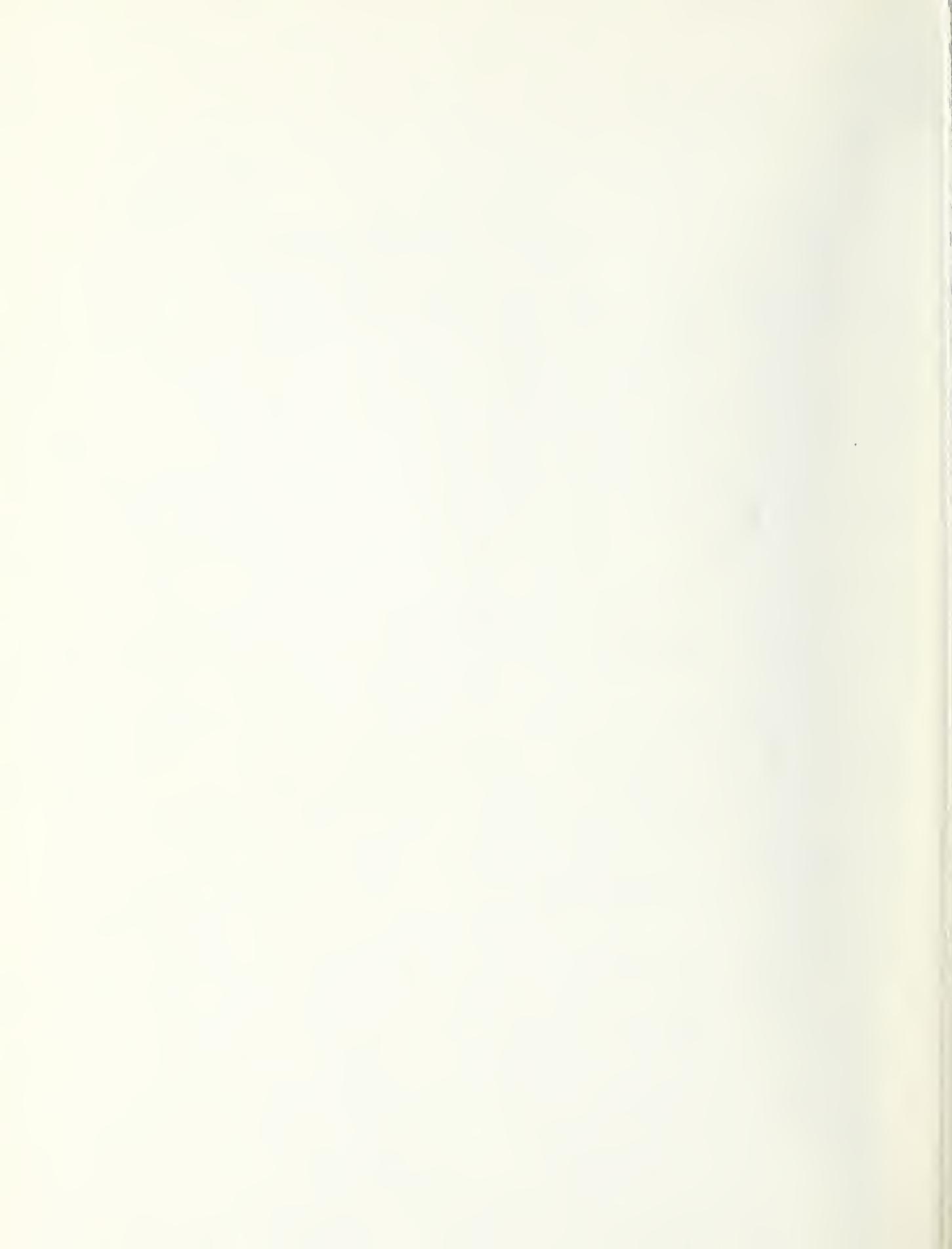Washington, D. C. 20234

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OPTICAL MATERIALS CHARACTERIZATION

Abstract

A program has been established for measuring refractive index, n, stress-optical constants, q_{ij}, change of index with temperature, dn/dT, thermal expansion coefficient, α, and elastic compliances, s_{ij}, of infrared laser window materials. These parameters are necessary for determining the optical distortion that occurs in windows due to heating by the absorption of high power laser radiation. n and dn/dT are measured over a spectral range 0.2 to 50 μm by the method of minimum deviation on precision spectrometers. Twyman-Green and Fizeau interferometers, which operate at 0.6328 μm, 1.15 μm, and 10.6 μm, are used for measuring q_{ij}, α, dn/dT and s_{ij}. Materials currently under study are polycrystalline ZnSe, As_2S_3 glass, chalcogenide glass (Ge 33%, As 12%, Se 55%), and KCl. Results are given for n and dn/dT in KCl, and q_{ij} and s_{12} in ZnSe, As_2S_3 glass, and chalcogenide glass.

1.   Technical Report Summary

1.1 Technical Problem

        Windows subjected to high average power laser radiation will undergo optical and
mechanical distortion due to absorptive heating.   If the distortion becomes sufficiently severe,
the windows become unusable. Theoretical calculations of optical distortion in laser windows
depend on the following material parameters: absorption coefficient, refractive index,
change of index with temperature, thermal expansion coefficient, stress-optical constants,
elastic compliances, specific heat, thermal conductivity and density. Our program has
been established to measure refractive indices, changes of index with temperature, stress-
optical constants, elastic compliances, and thermal expansion coefficients of candidate
infrared laser window materials.

1.2 General Methodology

        Laboratory experiments are conducted for measuring refractive indices, changes
of index with temperature, stress-optical constants, elastic compliances, and thermal expansion
coefficients.

        The refractive indices of prismatic specimens are measured on precision spectrometers
by using the method of minimum deviation [Section 2.2]. Two spectrometers are used. One
instrument, which uses glass optics, is used for measuring refractive indices in the visible
with an accuracy of several parts in 10^6. The other instrument, which uses mirror optics,
is used for measuring refractive indices in the ultraviolet and the infrared to an accuracy
of several parts in 10^5. Using both spectrometers we can measure refractive indices over
the spectral region 0.2 μm to 50 μm.

        We measure the coefficient of linear thermal expansion, α, by a method of Fizeau
interferometry [Section 2.3]. The interferometer consists of a specially prepared specimen
which separates two flat plates. Interference fringes are observed due to reflections from
the plate surfaces in contact with the specimen. We obtain α by measuring the shift of
these interference fringes as a function of temperature.

        The change of refractive index with temperature, dn/dT, is measured by two methods.
In the first method, we measure the refractive index with the precision spectrometers at
two temperatures, 20°C and 30°C, by varying the temperature of the laboratory [Section 2.2.2].
This provides us with a measure of dn/dT at room temperature. The second method may be
used for measuring dn/dT up to a temperature of 800°C [Section 2.3]. We obtain dn/dT from
from a knowledge of the expansion coefficient and by measuring the shift of Fizeau fringes
in a heated specimen as a function of temperature. The Fizeau fringes are due to interferences
between reflections from the front and back surfaces of the specimen.

        Stress-optical coefficients and elastic compliances are measured using a
combination of Twyman-Green and Fizeau interferometers [Section 2.4]. From the shift of
fringes in specimens subjected to uniaxial or hydrostatic compression, we obtain the
necessary data for determining all the stress-optical coefficients and elastic compliances.

1.3 Technical Results

        Measurements of refractive index have been made on a crystal of KCl from
220 nm to 14.3 μm. This is the first time that measurements of refractive index have been
made on a single sample of KCl over this wide a spectral range on a single instrument.
The data compared favorably with earlier compilations of refractive index [see Section 2.2.1].
The refractive index measurements were made at 20°C and 30°C. From these data we obtained
dn/dT. [Section 2.2.2].

The thermal expansion coefficient of polycrystalline ZnSe was measured interfer-ometrically. Data obtained at Raytheon using a dilatometer was in agreement with our data [Section 2.3].

Techniques were developed for measuring stress-optical constants q_{ij} and elastic compliances s_{12} [Section 2.4]. The techniques use Twyman-Green and Fizeau interferometers operating at 10.6 μm, 1.15 μm, and 0.6328 μm. We have measured the stress-optical constants of As_2S_3 glass at 10.6 μm, 1.15 μm, and 0.6328 μm, of a chalcogenide glass (33% Ge, 12% As, 55% Se) at 1.15 μm and 0.6328 μm, and of polycrystalline ZnSe at 1.15 μm and 0.6328 μm. In ZnSe we have measured the stress-induced birefringence at 10.6 μm as well. We have also measured s_{12} in As_2S_3 glass, the chalcogenide glass, and polycrystalline ZnSe. In As_2S_3 glass and the chalcogenide glass, the values of s_{12} agreed with the values obtained by other workers. We have been unable to find measurements of s_{12} in polycrystalline ZnSe by other workers.

1.4 Department of Defense Implications

The Department of Defense is currently constructing high-power infrared laser systems. Criteria are needed for determining the suitability of different materials for use as windows in these systems. The measurements we are performing provide data that laser system designers can use for determining the optical performance of candidate window materials.

1.5 Implications for Further Research

Measurements of refractive index, change of index with temperature, thermal expansion, stress-optical coefficients, and elastic compliances will be continued on candidate laser window materials. More specifically, we plan measurements on polycrystalline KCl, reactive atmosphere process (RAP) grown KCl, doped KCl, polycrystalline ZnSe, and other materials supplied by manufacturers under contract to the Advanced Research Projects Agency and other Department of Defense agencies. An important preliminary to the actual measurements will be the preparation of test specimens. This will require cutting, grinding, and polishing of samples.

The measurement of stress-optical coefficients in materials that possess very small stress-optical coefficients, or in materials that connot withstand large stresses, requires a very sensitive interferometric technique. Such an interferometer is currently under develop-ment.

2. Technical Report

2.1 Introduction

Laser systems of high average power are currently under development. As the output power increases, the optical components in these systems undergo optical and mechanical distortion due to absorptive heating. If the distortion is sufficiently severe, the optical elements become unusable. The parameters necessary for determining this optical distortion [1,2][1] are absorption coefficient, refractive index, change of index with temperature, thermal expansion coefficient, stress-optical coefficients, elastic compliances, specific heat, thermal conductivity, and density.

The temperature change that occurs in a laser window due to absorptive heating will produce three effects: (1) an expansion, (2) a direct change of refractive index, and (3) a system of internal stresses due to thermal gradients, which changes the refractive index through the stress-optical effect.

Although some information is available [3,4] on thermal expansion and change of index with temperature of laser window materials, there is very little data on the stress-optical effect [5]. In the past stress-optical coefficients have been measured in the visible region of the spectrum

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[1]Figures in brackets indicate the literature references at the end of this paper.

using interference methods [6,7]. We have now constructed Twyman-Green and Fizeau interferometers for making these measurements at 1.15 μm and 10.6 μm as well as at 0.6328 μm. This is the first time that interferometers have been used for measuring stress-optical coefficients in the infrared. These interferometers are also useful for measuring thermal expansion, change of index with temperature, and elastic compliance coefficients.

In this paper we present the methods used for measuring refractive index, n, change of index with temperature, dn/dT, linear thermal expansion coefficient, α, stress-optical coefficients, q_{ij}, and elastic compliances, s_{ij}. We also report on results obtained on As_2S_3 glass, a chalcogenide glass (Ge 33%, As 12%, Se 55%), polycrystalline ZnSe, and single crystal KCl.

We discuss measurements of n and dn/dT by using precision refractometry methods in Section 2.2. The quantity dn/dT is obtained near room temperature by measuring n at 20°C and at 30°C. Results are reported for KCl.

In Section 2.3 we discuss the measurement of thermal expansion and dn/dT up to a temperature of 800°C. These measurements involve the use of Fizeau fringes.

In Section 2.4 we discuss the measurement of stress-optical coefficients and elastic compliances. In isotropic materials there are two independent elastic constants and two independent stress-optical coefficients. The use of Twyman-Green and Fizeau interferometers in conjunction with samples under uniaxial and hydrostatic compression supplies us with sufficient data for obtaining all the constants.

In Section 2.5 we present the results of measuring q_{ij} and s_{12} on the chalcogenide glass, As_2S_3 glass, and polycrystalline ZnSe.

2.2 Refractive Index and Change of Index with Temperature

In the search for potential window and auxiliary optical component materials for high-power lasers, KCl appears to be one of the most promising candidates. It has useful transparency from ∼ 0.2 to 20 μm (with 90% transmittance between 0.45 and 14 μm). This covers window utilization in both the ultraviolet and infrared.

2.2.1 Index Data

A KCl prism cut from a commercially grown crystal was used in this study. The prism faces are 10 x 10 mm and the refracting angle near 60°. The refractive index was measured by means of the classical minimum deviation method with two precision spectrometers. The spectrometer schematic shown in figure 1a is designed for visible region refractometry and is capable of accuracy of several parts in 10^6. The spectrometer schematic shown in figure 1b employs mirror optics and is used in the non-visible spectral regions. It is capable of accuracy of several parts in 10^5. Both instruments and procedures for high-precision refractive index measurements have been described in previous publications [8,9].

Refractive index measurements were made at controlled room temperatures near 20°C at 60 wave-lengths from 0.22 μm in the ultraviolet to 14.3 μm in the infrared. The wavelengths were selected from the spectra of emission sources of Hg, Cd, He, Cs and Zn, and from calibrated absorption bands of H_2O, polystyrene, and methylcyclohexane. A series of calibrated narrow-band filters was also used in the infrared in the 3.5 to 10.6 μm region.

The experimentally determined index values at 20°C for KCl are represented by the solid curve in figure 2. The refractive index decreases from a value of 1.6360 at 0.224 μm to 1.4374 at 14.3 μm, an over-all change of 2×10^{-1}. At 10.6 μm the value of the refractive index is 1.4542 at 20°C.

The refractive index measurement program is being supplemented with a compilation of critically evaluated refractive indices of key laser materials. A literature search has turned up fourteen published papers dealing with refractive index of KCl. The data cover the wavelength range from ∼ 0.16 to 220 μm and were obtained by a variety of experimental techniques. It is interesting to note that in the spectral regions of interest to laser designers, the most accurate measurements were made in 1901 [10], 1908 [11], and 1927 [5]. The crystals measured were natural salt, and the minimum deviation method was employed with the sample in prism form. The excellent agreement between the NBS and the early data is shown in figure 2. The NBS values are lower in index by an average value of 2×10^{-4}.
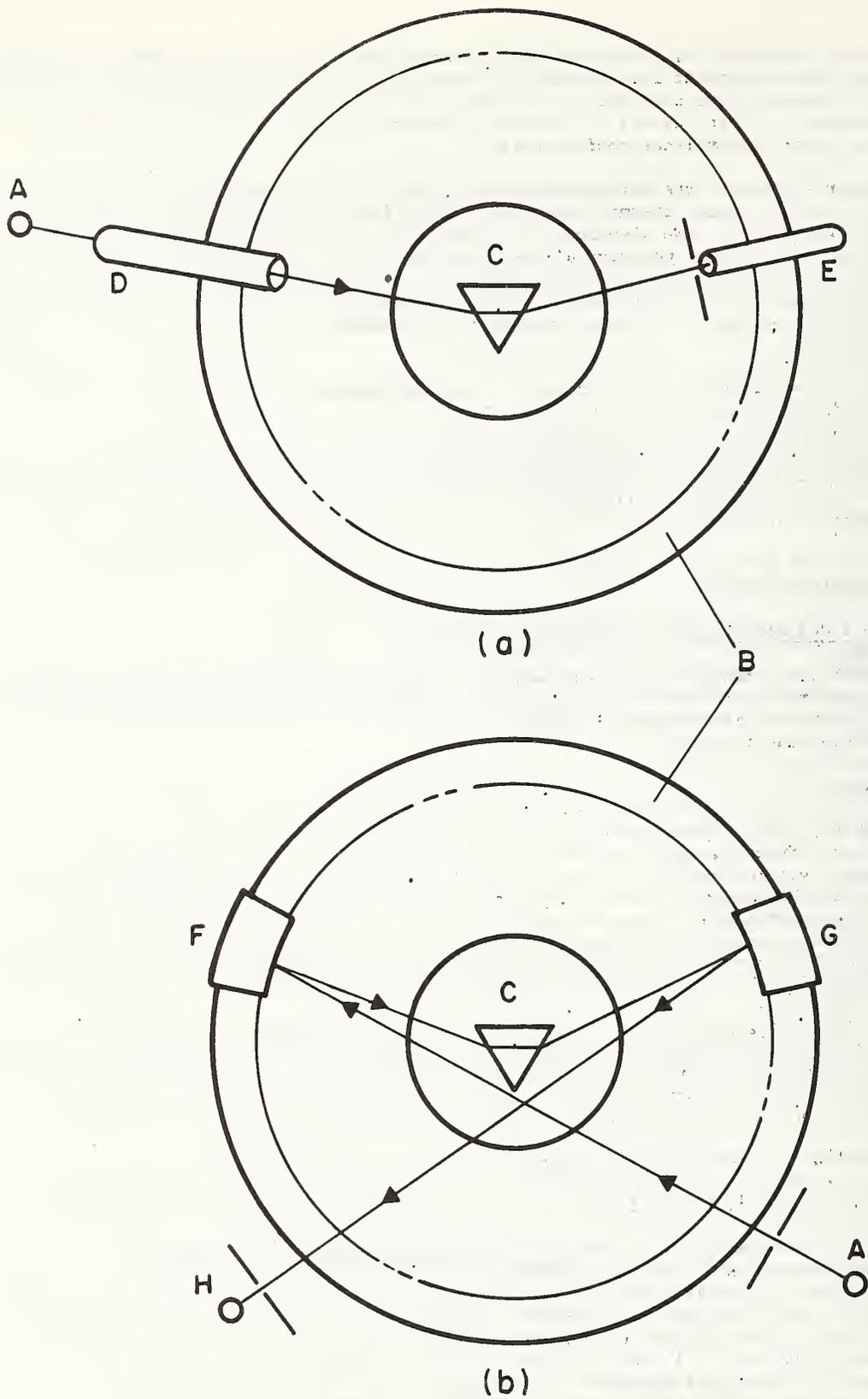
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Figure 1. (a) Schematic of spectrometer used for visible-region
refractometry. A. source; B. divided circle; C. prism table; D. collimator;
E. telescope. (b) Schematic of spectrometer used for nonvisible-region
refractometry. A. source; B. divided circle; C. prism table; F. colli-
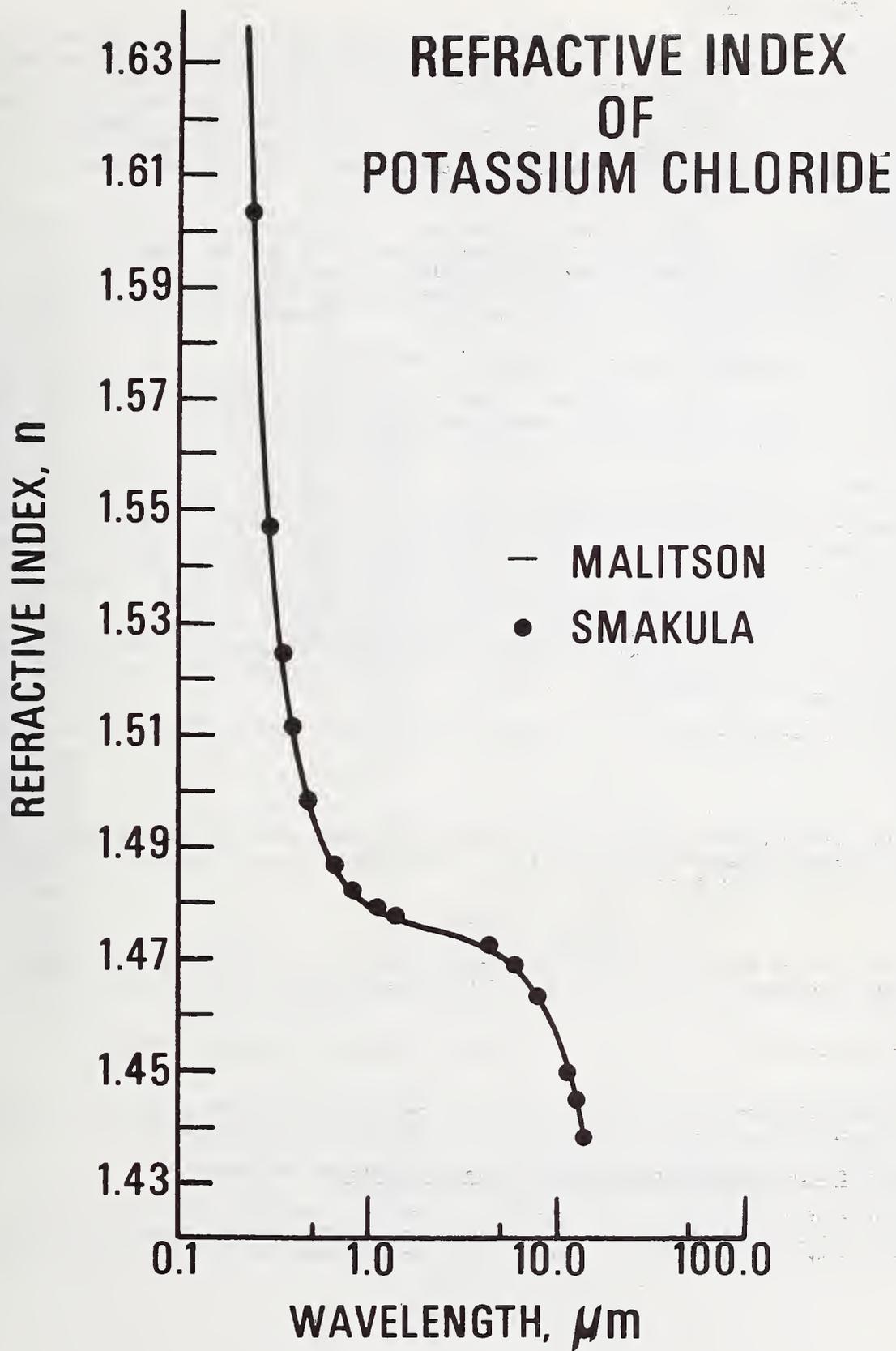mating mirror (fixed); G, movable mirror; H. detector.

Figure 2. Refractive index of KCl at 20°C.

2.2.2 Thermal Coefficient of Index

Refractive index measurements in KCl were repeated at 22 selected wavelengths at a controlled room temperature near 30°C. The refractive index values at 20°C and 30°C were used to calculate the thermal coefficient of index, dn/dT. The dependence on wavelength is shown in figure 3. The value of dn/dT varies from -2.1×10^{-5} K^{-1} at 0.27 μm to -2.5×10^{-5} K^{-1} at 14.3 μm; dn/dT remains relatively constant at about -3.0×10^{-5} K^{-1} in the infrared between 3 μm and 6.0 μm. The fact that dn/dT is negative indicates that the variation in index with temperature is essentially due to the change in density of the crystal. At the extremes, where dn/dT is less negative, the influence of temperature suggests a shift of the ultraviolet and infrared absorption edges to longer wavelengths.

The dn/dT values of Gyulai [12] and Paschen [13] are shown in figure 3 for comparison. Gyulai's values for a mean temperature of 33°C cover the wavelength range 0.20 to 0.61 μm. Paschen's data, for which the mean temperature is unknown, covers the wavelength range 0.59 to 17.7 μm. It can be seen that the absolute values of the earlier work are generally higher than those of NBS. It has been observed in halide crystals that the absolute value of dn/dT increases with increasing temperature.

2.3 Thermal Expansion and Change in Refractive Index with Temperature

The thermal expansion and change of refractive index with temperature dn/dT may be measured up to a temperature of 800°C by an interferometric method that uses Fizeau type fringes [14]. In the visible part of the spectrum these measurements can be made simultaneously.

The interferometer assembly as used in the visible is shown in figure 4. The set of fringes on the left is obtained between the top and bottom surfaces of the lower fused silica plate, T, and is calibrated in terms of temperature so that the fringes serve as an optical thermometer. The center set of fringes is obtained between the top and bottom surfaces of the index sample, R, and is used to calculate the change in refractive index. Beneath this sample is a frosted area on the lower plate so that no fringes are formed at the interface. The third set of fringes is formed by the slight wedge introduced between the upper plate, E, and the lower plate, T, when these fringes are separated by the expansion sample, S, which is cut from the same material as the index sample, R. These fringes are used to obtain the thermal expansion. The assembly is heated by a vertical tube furnace and the shifts in the three sets of fringes are recorded simultaneously on a single strip of 35 mm film as a function of temperature.

The interference pattern generated by the sample of thickness, t, and refractive index, n, is expressed by the equation,

$$N\lambda = 2nt, \tag{1}$$

which relates the optical path, nt, and the fringe number, N, when monochromatic light of wavelength, λ, passes twice through the sample at normal incidence. By differentiating with respect to temperature, T, and rearranging, it is found that

$$\frac{dn}{dT} = \frac{\lambda}{2t} \frac{\Delta N}{\Delta T} - \frac{ndt}{dT} \cdot \tag{2}$$

We calculate the change of refractive index as a function of temperature from the number of fringes that pass a point of reference and from the change of thickness obtained from the thermal expansion sample [15].

Using a modification of the above method, we intend to extend the measurement of dn/dT to the infrared.

The expansion coefficient of polycrystalline ZnSe was measured by the above method. Data obtained at Raytheon using a dilatometer was in agreement with our data. We obtained $\alpha = 7.9 \times 10^{-6}$/K.

2.4 Measurement of Stress-Optical Coefficients and Elastic Constants

The importance of measuring all the stress-optical coefficients in the infrared is not always well understood. Several laboratories have been measuring stress-induced birefringence at 10.6 μm. However, these data are not sufficient for determining the optical wavefront distortion due to thermal
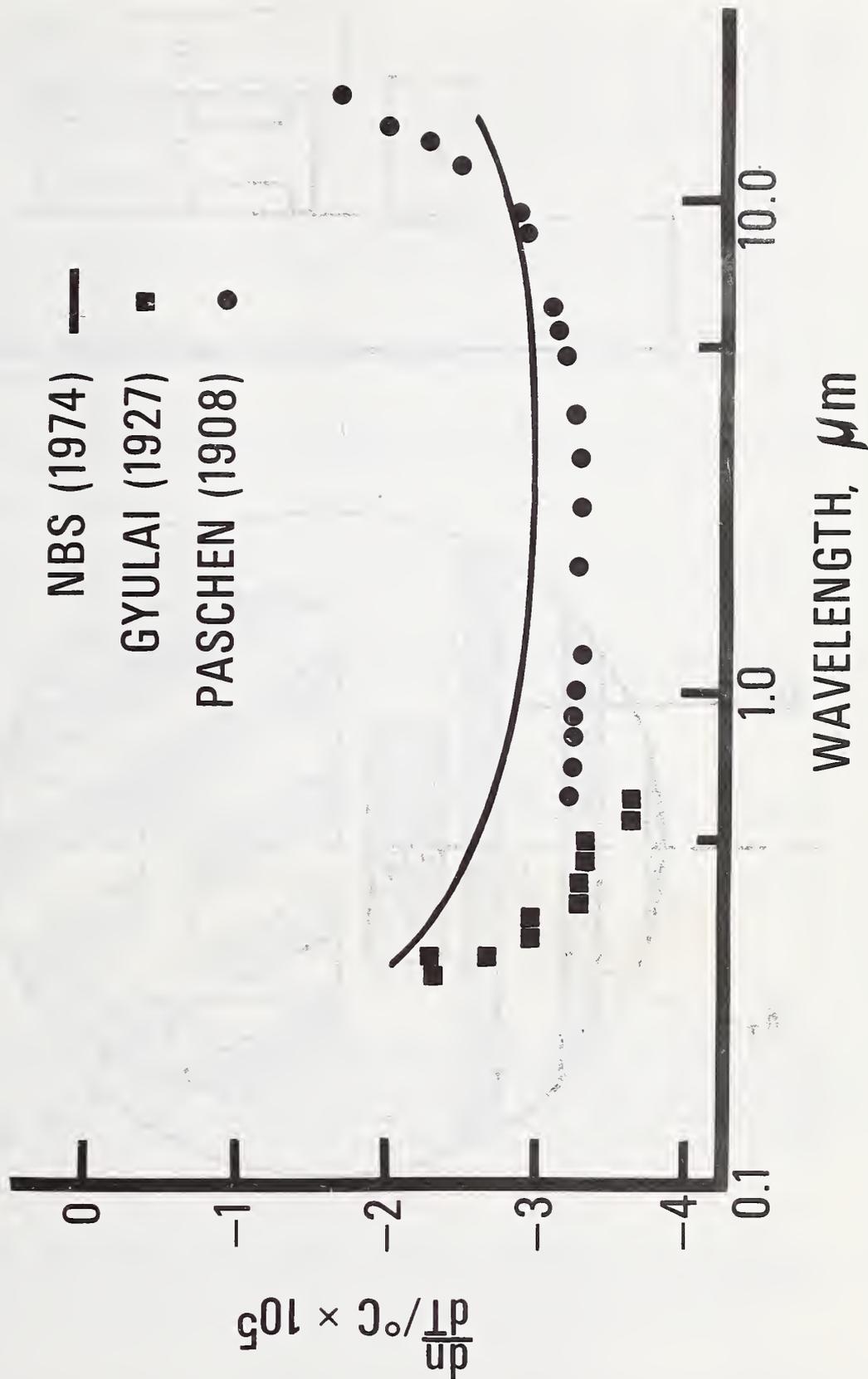
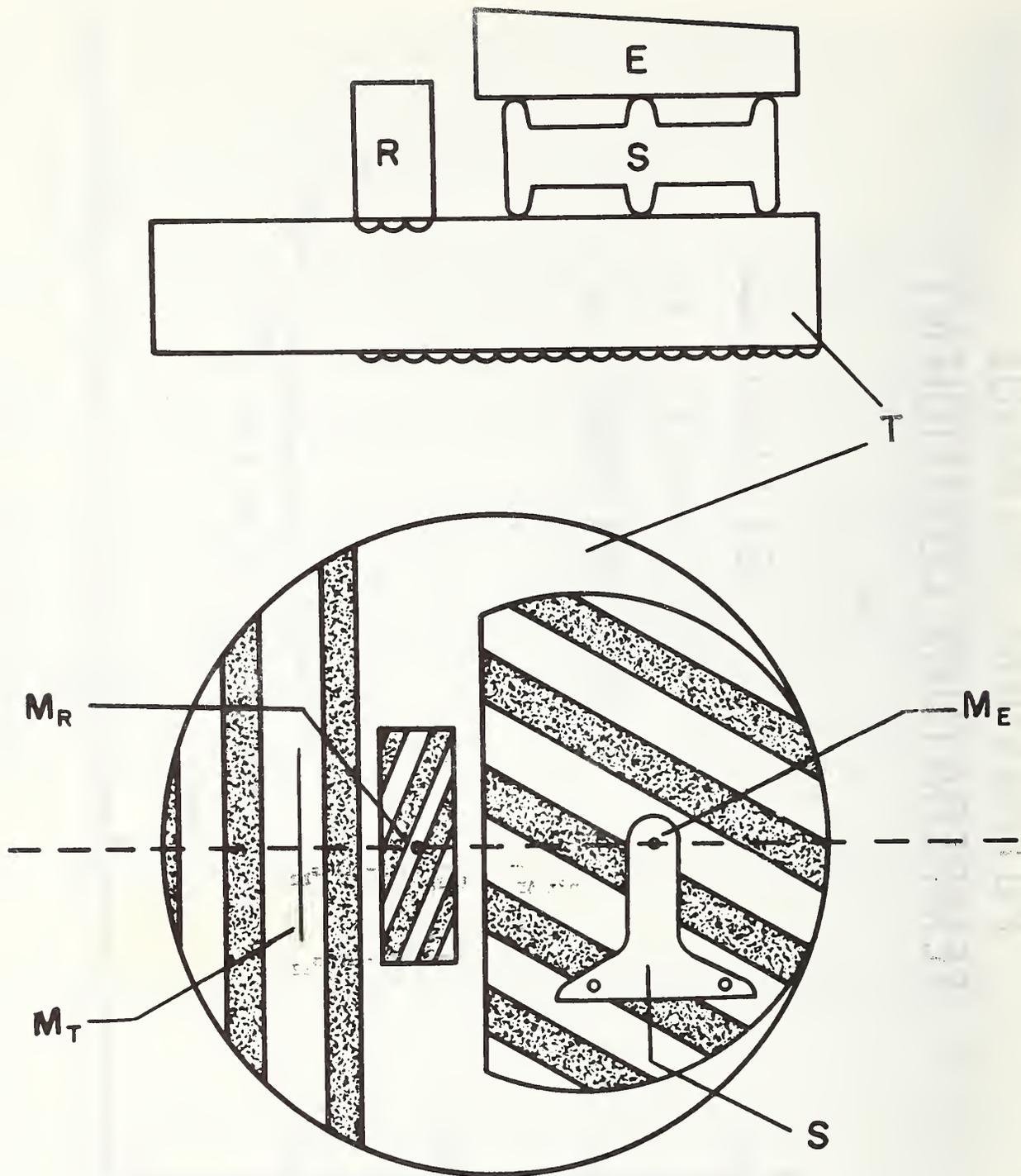Figure 3. Temperature coefficient of refractive index of KCl.

Figure 4. Interferometer assembly for the measurement of thermal expansion and thermal change in refractive index. E. an interferometer plate; S. thermal expansion specimen; R. refractive index specimen; T. interference thermometer; M_E, M_R, and M_T, reference lines for the interference fringes of thermal expansion, refractive index change and temperature change.

stresses, because they measure only the relative stress-optical coefficients [$(q_{11} - q_{12})$ and q_{44} for cubic materials]. We know of only one paper which presents measurements of q_{11} and q_{12} separately at 10.6 μm [5], and these measurements, which were done on Ge, did not give the sign of the coefficients. Both q_{11} and q_{12}, in combination with the elastic constants, are necessary for calculating the optical distortion due to stresses from thermal gradients in optical materials [1,2]. Index changes due to these stresses can be even greater than changes in index due to a rise in temperature. For example, in a material tailored to have $dn/dT = 0$, the changes of index will be due solely to the stress-optical effect.

2.4.1 Apparatus

Conventional Twyman-Green interferometers are used [16]. The Twyman-Green interferometer at .6328 and 1.15 μm uses helium-neon lasers as sources. The fringes are observed with a silicon matrix vidicon camera.

The Twyman-Green interferometer at 10.6 μm uses a CO_2 laser source, a germanium beam expander, and a germanium beam splitter. The fringes are viewed either on a liquid crystal detector or on a commercial thermal image plate.

For uniaxial loading, the specimen, in the form of a rectangular parallelepiped is compressed in a rectangular frame screw clamp equipped with a calibrated load cell and a digital readout voltmeter. A photograph of the stressing apparatus with the voltmeter is shown in figure 5.

2.4.2 Experimental Procedure

The sample is placed within one arm of the Twyman-Green interferometer. The fringe shift per unit of applied stress is measured for stress parallel to the polarization of the radiation, $\Delta N_1/\Delta P$, and for stress perpendicular to the polarization of the radiation, $\Delta N_2/\Delta P$.

The sample, itself, functions as a Fizeau interferometer if the faces are properly polished; interference fringes are obtained from reflections from the sample surfaces. The fringe shifts obtained for stresses parallel and perpendicular to the polarization of the radiation are, respectively, $\Delta N_1'/\Delta P$ and $\Delta N_2'/\Delta P$.

For an isotropic material we obtain

$$s_{12} = \frac{\lambda}{2t} \left(\frac{\Delta N_1}{\Delta P} - \frac{\Delta N_1'}{\Delta P} \right) = \frac{\lambda}{2t} \left(\frac{\Delta N_2}{\Delta P} - \frac{\Delta N_2'}{\Delta P} \right) \qquad (3)$$

$$q_{11} = \frac{\lambda}{n^3 t} \frac{\Delta N_1}{\Delta P} + \left(\frac{2}{n^3} \right) (n - 1) s_{12} \qquad (4)$$

$$q_{12} = \frac{\lambda}{n^3 t} \frac{\Delta N_2}{\Delta P} + \left(\frac{2}{n^3} \right) (n - 1) s_{12} \qquad (5)$$

Additional experimental data can be obtained by measuring the stress-induced birefringence. The experimental arrangement is shown in figure 6. The stress is applied 45° with respect to the plane of polarization. The analyzer is oriented parallel to the incident polarization. The uniaxial load applied to the sample is increased until extinction is obtained. The loaded specimen then acts effectively as a half-waveplate, and considering the double passage of the light through the specimen, the load is that which creates a single pass quarter wave retardation in the sample. The relative stress-optic coefficient is

$$q_{11} - q_{12} = \lambda/(2n^3 Pt). \qquad (6)$$

The value of $q_{11} - q_{12}$ from the measurement of birefringence is more precise than the value of $q_{11} - q_{12}$ obtained from eqs (4) and (5). Thus, we calculate q_{11} and q_{12} from the sum $q_{11} + q_{12}$, obtained by adding eqs (4) and (5), and the difference $q_{11} - q_{12}$ from eq (6). This assures us that the birefringence calculated from the numerical values of q_{11} and q_{12} equals the measured value.
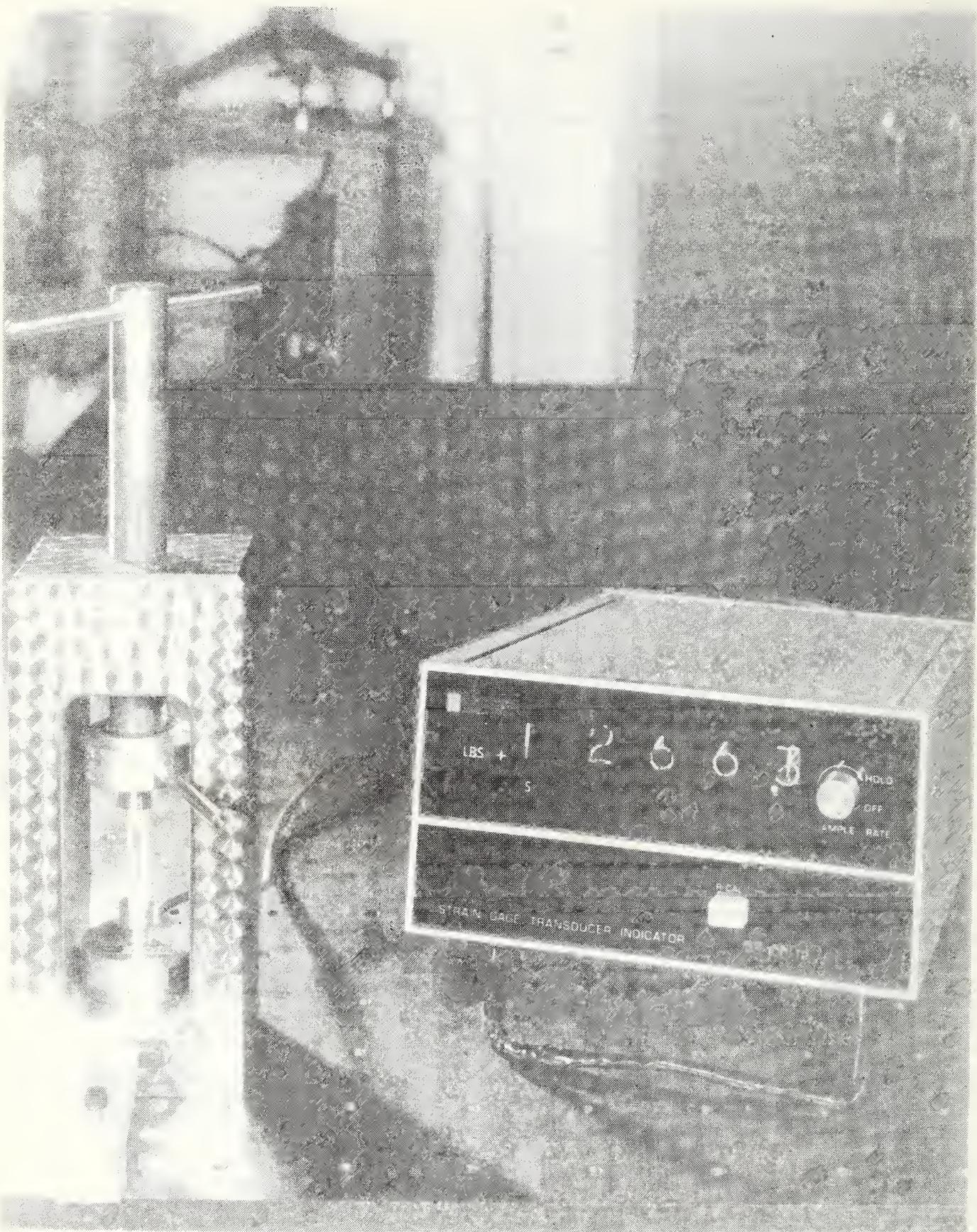
Figure 5.   Stressing apparatus with specimen, load cell, and digital
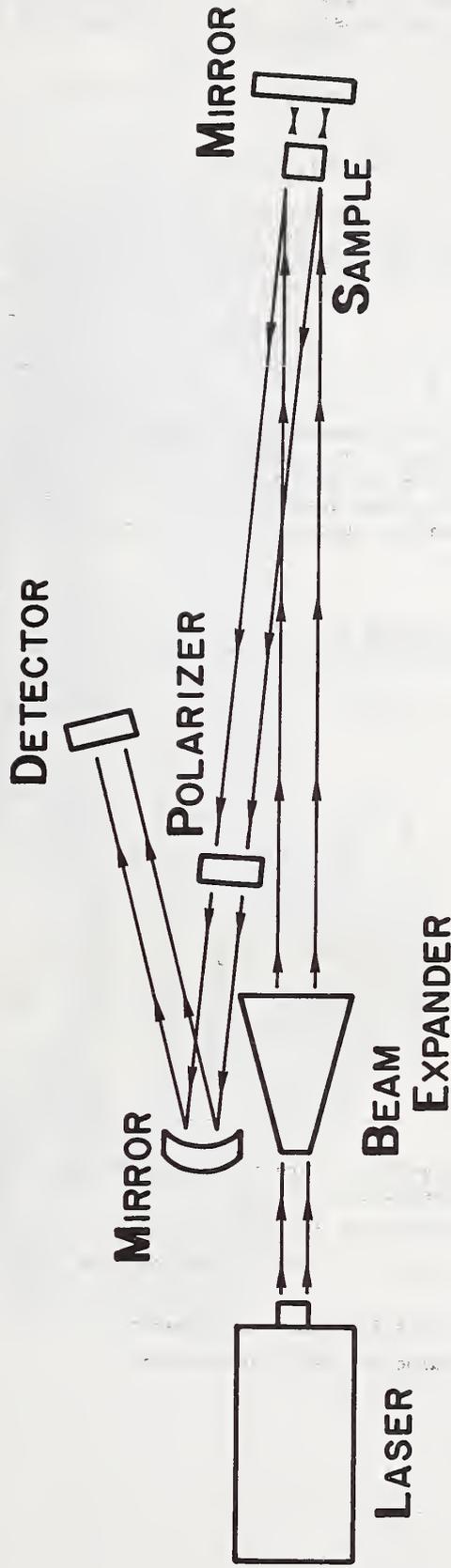voltmeter readout.

Figure 6. Experimental arrangement for measuring stress-induced birefringence.

Equation (3) gives the value for s_{12}. In order to obtain s_{11}, we use a method similar to the method for measuring dn/dT. The sample which acts as a Fizeau interferometer is compressed in a pressure vessel. Figure 7 shows the pressure vessel. Hydrostatic pressure is generated by compressing a fluid. From the shift in fringes per unit pressure, $\Delta N/\Delta P$, we obtain for an isotropic material

$$s_{11} + 2s_{12} = \frac{\lambda}{2nt} \frac{\Delta N}{\Delta P} - \frac{n^2}{2} (q_{11} + 2q_{12}). \qquad (7)$$

Using s_{12} obtained from eq (3) together with eq (7), we can solve for s_{11}.

The procedures described in this section are for measuring stress-optical constants and elastic constants of isotropic materials. These constants can be measured with the same procedures on crystalline materials. Because stresses must be applied along several crystallographic axes, more samples are required for measurements on crystalline materials than for measurements on isotropic materials.

2.4.3 Results

Measurements of absolute and relative stress-optical coefficients have been made on chalcogenide glass, As_2S_3 glass, and polycrystalline ZnSe. The results are given in Table 1. The data for As_2S_3 are presented for three wavelengths, 0.6328 µm, 1.15 µm, and 10.6 µm. Data for the same wavelengths are presented for polycrystalline ZnSe although absolute values are lacking at 10.6 µm. We have assumed the ZnSe to be an isotropic material. Data for the chalcogenide glass are presented at the infrared wavelengths.

Table 1. Stress-optical constants, q_{11} and q_{12}, and elastic compliance, s_{12}, in units of 10^{-12} m^2/N.

	n[a]	q_{11}[b]	q_{12}[b]	$q_{12}-q_{11}$[b]	s_{12}[b]
As_2S_3					- 18.6, - 18.8[c]
0.6328 µm	2.606	7.63	6.36	- 1.27	
1.15 µm	2.452	7.64	6.54	- 1.10	
10.6 µm	2.354	8.23	6.95	- 1.28	
Chalcogenide Glass (Ge 33%, As 12%, Se 55%)					- 12.0, - 12.2[d], - 11.5[e]
1.15 µm	2.5704	5.32	4.89	- 0.43	
10.6 µm	2.4919	5.42	4.94	- 0.48	
Polycrystalline ZnSe				- 4.0	
0.6328 µm	2.578	- 1.34	0.29	1.62	
1.15 µm	2.465	- 1.37	0.62	1.98	
10.6 µm	2.408			1.96	

[a] These values were not measured in this work, but are given because they were used to calculate the q_{ij}. The values for ZnSe are from single crystal data.

[b] Estimated accuracy of numbers from this work is ± 2%.

[c] F. W. Glaze, D. H. Blackburn, J. S. Osmalov, D. Hubbard, and Mason H. Black, J. Res. Natl. Bur. Std. 59, 83-92 (1957).

[d] Ray Hilton, private communication -- value for Texas Instruments.

[e] Ray Hilton, private communication -- value for Bell Laboratories.

TO PRESSURE GENERATOR

STEEL BOMB

SPECIMEN HOLDER

COPPER WASHER

STEEL PLUG

GLASS WINDOW SURFACES OPTICALLY FLAT
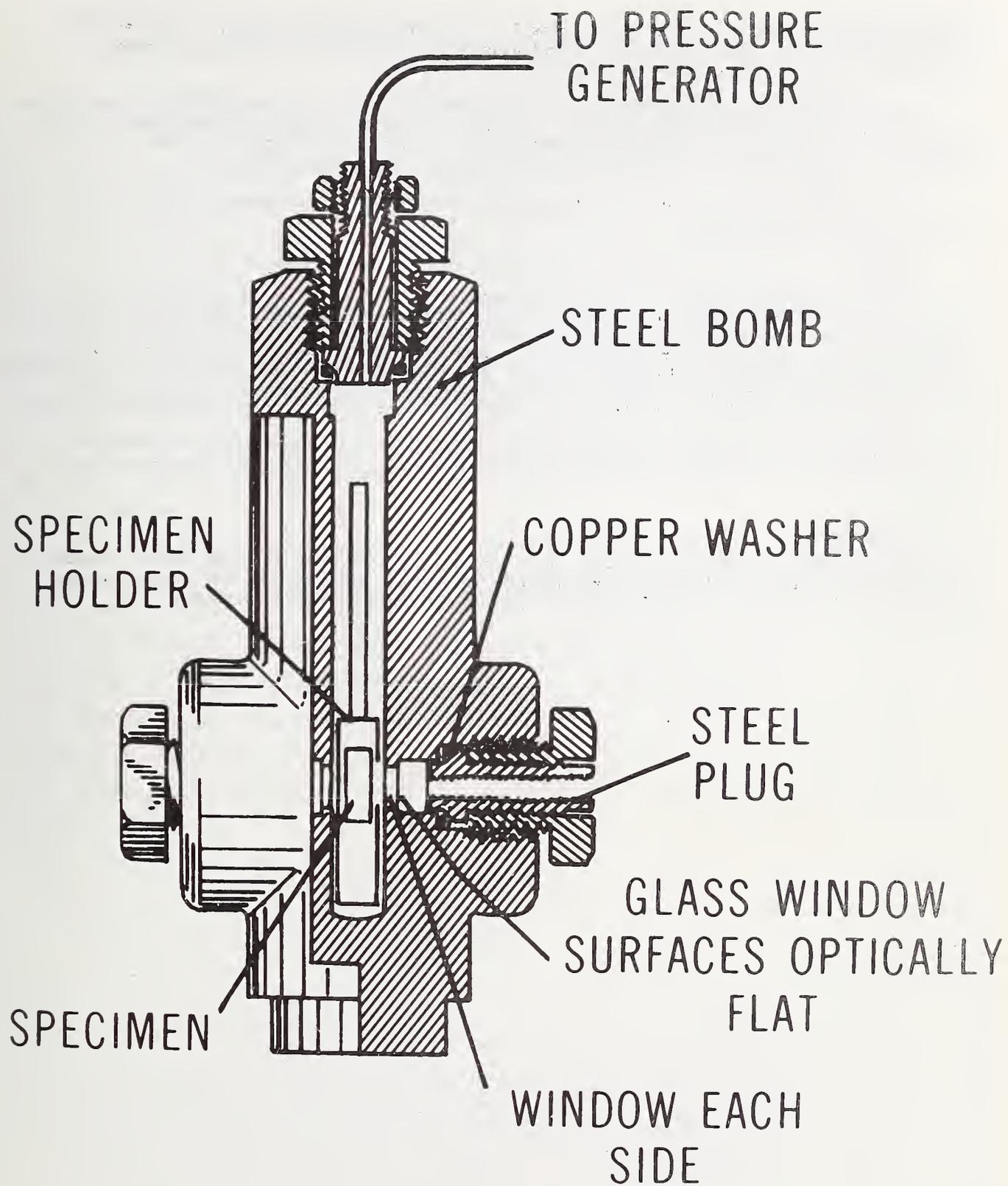
SPECIMEN

WINDOW EACH SIDE

Figure 7. Pressure vessel.

The birefringence of the above infrared glasses is of opposite sign to most optical glasses [17]. The q_{11} is greater than the q_{11} of most optical glasses by a factor of ten, and q_{12} is greater than the q_{12} of most optical glasses by a factor of three [18].

From the theory of photoelasticity and from the data, we expect that the refractive index of the ZnSe will decrease under hydrostatic pressure. This behavior has been observed previously in single crystal ZnS [19]. Since dn/dT for ZnS is positive [20], it would be of interest to observe whether the same is true for ZnSe. In addition, for ZnSe there is a large dispersion in q_{12}.

Also included in Table 1 are the values we have obtained for s_{12}. Agreement with the values of other workers is very good.

2.5 Summary

We have outlined the program of optical materials characterization at the National Bureau of Standards. The properties that we are measuring are refractive index, change of index with temperature, thermal expansion, stress-optical coefficients, and elastic constants. At the present time, primary interest is in laser window materials at 10.6 μm. Data have been presented on chalcogenide glass, As_2S_3, polycrystalline ZnSe, and crystalline KCl. Measurements on these materials and on other infrared laser window materials are in progress. In addition, we are developing more sensitive techniques for measuring the stress-optical coefficients in materials that exhibit a small stress-optical effect.

A similar need exists for optical materials characterization in the ultraviolet. Techniques similar to those we have used in the infrared can be employed in the ultraviolet should the need arise.

2.6 Acknowledgments

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16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

   A program has been established for measuring refractive index, n, stress-optical constants, q_{ij}, change of index with temperature, dn/dT, thermal expansion coefficient, α, and elastic compliances, s_{ij}, of infrared laser window materials. These parameters are necessary for determining the optical distortion that occurs in windows due to heating by the absorption of high power laser radiation. n and dn/dT are measured over a spectral range 0.2 to 50 μm by the method of minimum deviation on precision spectrometers. Twyman-Green and Fizeau interferometers which operate at 0.6328 μm, 1.15 μm, and 10.6 μm, are used for measuring q_{ij}, α, dn/dT and s_{ij}. Materials currently under study are polycrystalline ZnSe, As_2S_3 glass, chalcogenide glass (Ge 33%, As 12%, Se 55%), and KCl. Results are given for n and dn/dT in KCl, and q_{ij} and s_{12} in ZnSe, As_2S_3 glass, and chalcogenide glass.

17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons) As_2S_3; chalcogenide glass; coefficient of thermal expansion; elastic constants; infrared laser window materials; KCl; photoelasticity; polycrystalline ZnSe; refractive index; stress-optical constants; thermal coefficient of refractive index.

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