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Albert Feldman, Deane Horowitz, and Roy M. Waxler

Inorganic Materials Division
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and

Irving Malitson and Marilyn J. Dodge

Optical Physics Division
Institute for Basic Standards
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Abstract

We have measured the following parameters of chemical vapor deposited polycrystalline ZnSe (CVD ZnSe): Refractive index and change of index of refraction with temperature (dn/dT) over the wavelength range 0.5 μm to 18 μm using the method of minimum deviation; the coefficient of linear thermal expansion and dn/dT at 10.6 μm using Fizeau interferometry; and the elastic moduli and photoelastic moduli using Fizeau and Twyman-Green interferometry. A sensitive technique has been developed for measuring stress-optical constants of materials that exhibit a small stress-optical effect.

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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. 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. 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. 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. We obtain dn/dT 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 specimens.

We measure stress-optical coefficients and elastic compliances using a combination of Twyman-Green and Fizeau interferometers. From the shift of fringes in specimens subjected to uniaxial or hydrostatic compression, we obtain the necessary data for determining all the stress-optical constants and elastic compliances. In materials with small stress-optical constants or in materials that cannot withstand large stress, we measure the stress-optical effect with a modified Twyman-Green interferometer, which has a sensitivity of about 0.01λ at 10.6 μm.

1.3   Technical Results

The following measurements have been made on polycrystalline ZnSe that has been grown by the chemical vapor deposition technique: a) the refractive index was measured at 20 °C for 30 wavelengths and at 30 °C for 20 wavelengths in the range from 0.51 μm to 18.2 μm. From these data we obtained a dispersion curve of the change of index with temperature. b) An equation for the change of thickness as a function of temperature was obtained. c) The change of index with temperature was measured interferometrically at 0.6328 μm and at 10.6 μm. At 10.6 μm we made a total of six measurements on three specimens. Our results were highly reproducible, however, they differed significantly from data reported by AFCRL. The discrepancy may be due to specimen differences. d) We measured the stress-optical constants q_{11} and q_{12} at 10.6 μm. These data, which are presented together with earlier data at 0.6328 μm and 1.15 μm, suggest that the dispersion in the coefficients from 1.15 μm to 10.6 μm is small. e) We calculated the elastic compliance component s_{11} from interferometric measurements on a specimen under hydrostratic pressure and from previously reported values of s_{12}, q_{11} and q_{12} obtained at 0.6328 μm.

We built a specially modified Twyman-Green interferometer for the stress-optic measurements at 10.6 μm. This was necessary because the change in optic path at 10.6 μm for the maximum permissible applied stress is less than λ. The estimated resistivity of the interferometer is 0.01 λ at 10.6 μm.

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, 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.

In using the data we have obtained on CVD ZnSe, some caution must be applied. For example, we have treated the ZnSe as an isotropic material, whereas other workers indicate that the material is not isotropic. We are, therefore, measuring the stress-optical constants and elastic compliance on new material to ascertain whether one can expect significant differences in the coefficients of different specimens. Preliminary results indicate that these coefficients may differ in specimens obtained from different batches.

2. Technical Report – Some Properties of Polycrystalline Zinc Selenide

2.1 INTRODUCTION

This report will discuss the measurement of refractive index, change of index with temperature, thermal expansion coefficient, stress-optical constants and elastic compliances in high power laser window materials. These parameters are needed for calculations that predict the optical distortion that occurs in windows subjected to high energy laser radiation [1-5]. In particular, we are investigating these properties in polycrystalline ZnSe that has been grown by the chemical vapor deposition (CVD) technique.

We have measured the refractive index, n, of CVD ZnSe on a prism spectrometer using the classical minimum deviation method. Data were obtained at 20 °C for 30 wavelengths and at 30 °C for 22 wavelengths in the range from 0.51 μm to 18.2 μm. At 10.6 μm, n = 2.4027 at 20 °C.

The change of refractive index with temperature, dn/dT, was measured by two methods. In the first method we measured n by the method of minimum deviation at both 20 °C and 30 °C. This method permitted us to calculate dn/dT at room temperature. At 10.6 μm this value was 7.9×10^{-5}/°C. In the second method we measured the shift of Fizeau fringes in a parallel plate at discrete wavelengths as a function of temperature from 25 °C to 200 °C. With these data, together with our measured value of the thermal expansion coefficient we calculated dn/dT. From a linear least squares fits to the data we obtain dn/dT = 6.4×10^{-5}/°C at 10.6 μm. The differences between the values obtained by the two methods and the differences of both these values with measurements at other laboratories is, as yet, unexplained.

The value of the coefficient of linear thermal expansion, α, has been obtained using Fizeau interferometry. We find that at 25 °C, $\alpha = 7.79 \times 10^{-6}$/°C. An air correction term was used in the calculation.

In our previous report we reported measurements of the absolute stress-optical constants, q_{11} and q_{12}, of CVD ZnSe at 0.6328 µm and 1.15 µm. These measurements were made with conventional Twyman-Green and Fizeau interferometers. We have now developed a more sensitive technique for measuring absolute stress-optical constants at 10.6 µm. The technique makes it possible for us to detect the shift of 0.01 fringe in a Twyman-Green interferometer. This precision is necessary because the stress-optical effect in ZnSe is very small at 10.6 µm. Preliminary measurements suggest that the absolute stress-optical constants of polycrystalline ZnSe at 10.6 µm do not differ greatly from the coefficients at 1.15 µm.

In addition, values are presented for the elastic compliance components, s_{11} and s_{12}, for polycrystalline ZnSe. In the past, we have obtained the component s_{12} by measuring the shift in fringes of Twyman-Green and Fizeau interferometers as a function of uniaxial stress applied to a specimen. We have now measured the shift of Fizeau fringes of a specimen under hydrostatic compression. From this measurement together with the stress-optical constants and s_{12} we calculate s_{11} and the other related elastic moduli.

2.2 Refractive Index and Change of Index with Temperature by the Method of Minimum Deviation

The refractive index of a prism of CVD ZnSe was measured at 30 wavelengths from 0.51 µm to 18.2 µm at a controlled temperature near 20 °C. The apparatus has been described in earlier work [6-8]. In the visible and near-infrared regions, n, was measured at known wavelengths of mercury, cadmium and helium lamps. At longer infrared wavelengths, measurements were made at known absorption bands of H_2O, CO_2, polystyrene and 1,2,4-trichlorobenzene. We also used a series of narrow-band filters between 3.5 and 10.7 µm. A globar was the infrared source and a thermocouple with a CsI window was the detector.

The experimentally determined index values at 20 °C are represented by the solid curve in Fig. 1. The index decreases from a value of 2.7270 at 0.51 µm to 2.3278 at 18.16 µm, an overall change of 0.4. At 10.6 µm, n = 2.4027. The dispersion data are in the process of being fitted by computer to a Sellmeier-type dispersion equation. A preliminary computer run, suggests that the errors in the raw data are probably less than 5×10^{-5}.

Of the six previously reported investigations of the refractive index of ZnSe between 0.5 µm and 14 µm, four have used deviation methods that employ prismatic specimens. Because deviation methods are considered to be the most accurate, we compare the results of these four workers with our results. Marple [9] has measured n for single crystal ZnSe between 0.48 µm and 2.5 µm with a claimed accuracy of 4×10^{-3}. His values are about 0.01 less than our values. Rambauske [10] studied both single crystal and poly-crystalline ZnSe in the visible region. He found the index of the polycry-
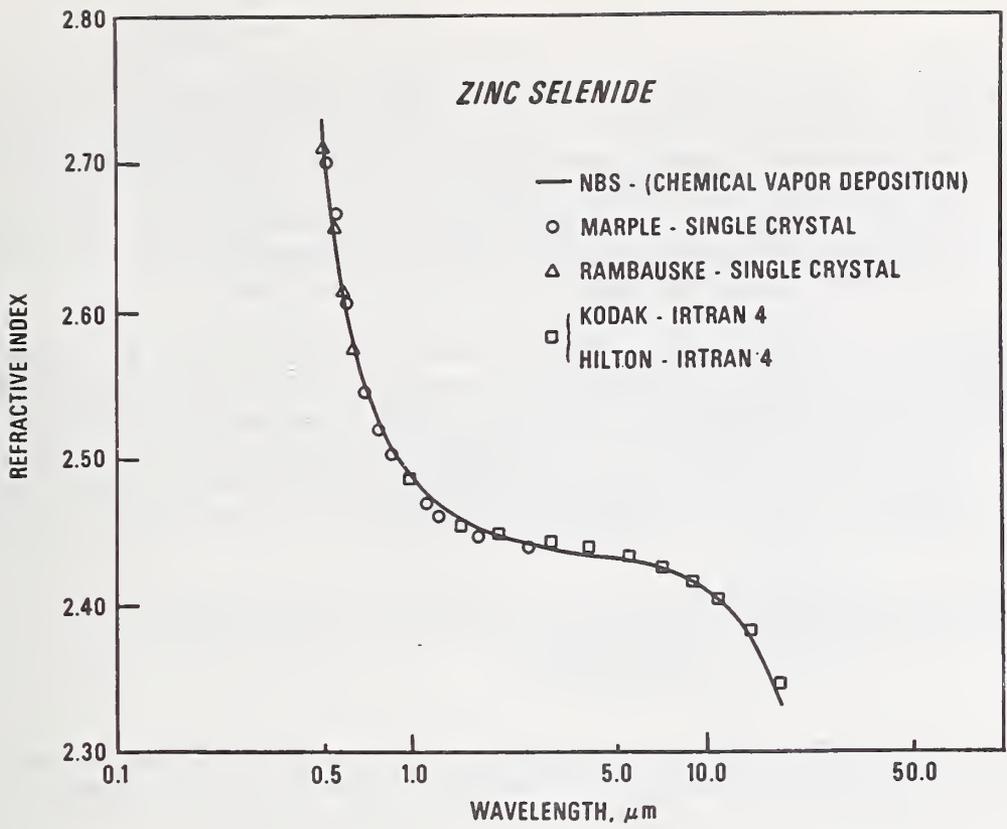
FIGURE 1. REFRACTIVE INDEX OF CVD ZnSe AT 20 °C AS A FUNCTION OF WAVELENGTH.
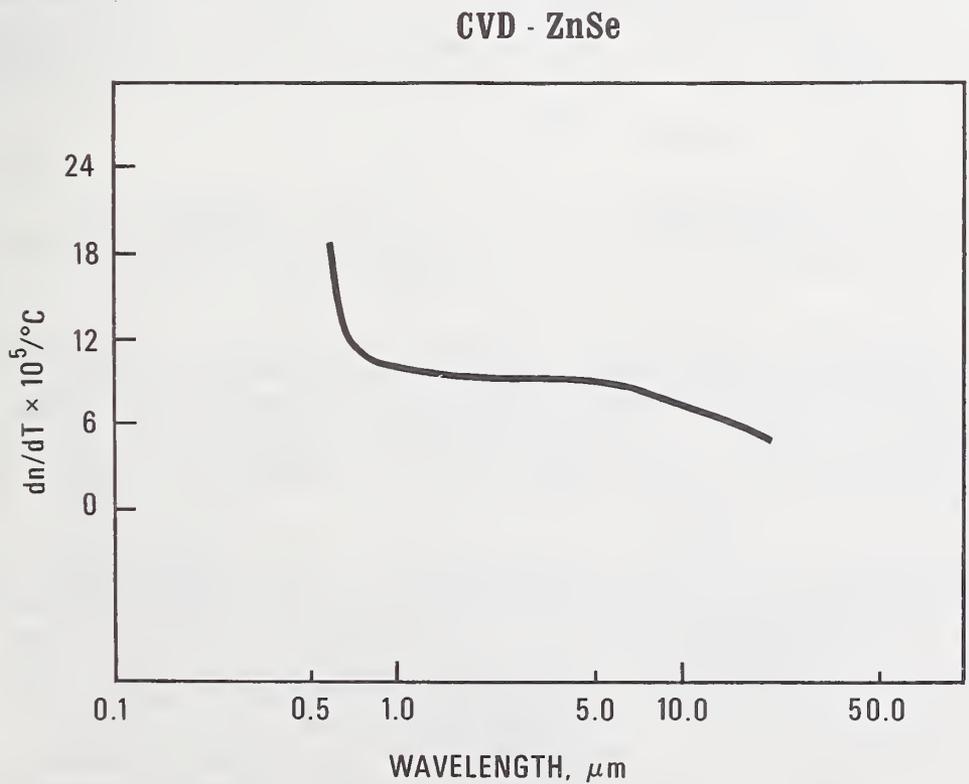
CVD - ZnSe

FIGURE 2. THERMAL COEFFICIENT OF INDEX OF CVD ZnSe AT 25 °C AS A FUNCTION
OF WAVELENGTH.

stalline material to be generally 1% larger than the index of the single crystal material. Kodak has published values of index for IRTRAN 4 [11] which is a hot-pressed polycrystalline ZnSe material. Hilton's measurements on IRTRAN 4 [12] generally agree with Kodak's values to within 1×10^{-3}, but are about 0.2% larger than the indices we measured in CVD ZnSe. To the best of our knowledge, these are the first measurements of n in CVD ZnSe over its practical transmission range on a single instrument.

Measurements of n were repeated at 22 selected wavelengths at a controlled room temperature near 30 °C. These values together with the values at 20 °C were used to calculate dn/dT. Fig. 2 shows dn/dT as a function of wavelength. The value of dn/dT varies from $18.6 \times 10^{-5}/°C$ at 0.5461 μm to $5.5 \times 10^{-5}/°C$ at 18.2 μm. At 10.6 μm, dn/dT = $(7.9 \pm 1.0) \times 10^{-5}/°C$. The fact that dn/dT is positive indicates that the temperature dependence of the electronic polarizabilities predominates over the density effect.

2.3 Interferometric Measurement of Thermal Expansion and Change of Refractive Index with Temperature

In some earlier work [8] we discussed the measurement of the linear coefficient of thermal expansion by the method of Fizeau interferometry. The expression for α is

$$\alpha = \frac{1}{t} \frac{dt}{dT} \tag{1}$$

where T is the temperature in °C, and t is the specimen thickness. We have measured the thermal expansion of a specimen of CVD ZnSe over the temperature range 25 °C - 200 °C and have fitted the data to a quadratic expansion in the temperature. A correction for air was taken into account in the calculation. We then obtained

$$t = t_o[1 + a(T-25) + b(T-25)^2] \tag{2}$$

where t_o is the specimen thickness at 25 °C, a = $7.79 \times 10^{-6}/°C$, and b = $2.1 \times 10^{-10}(°C)^{-2}$. At 25 °C, $\alpha = a$. Data obtained at Raytheon [13] from dilatometer measurements are in good agreement with our data.

In that same earlier work [8] we discussed our method for measuring dn/dT in the visible using Fizeau interferometry. The method has been modified to make use of laser sources. Fig. 3 is a schematic diagram of the system as used at 10.6 μm. A special feature of the system is an As_2S_3 glass plate which acts as a Fizeau interferometer to monitor the mode stability of the laser.

The experimental procedure consisted of heating the specimen, which acts as a Fizeau interferometer, so that the temperature rise was about one degree per minute from room temperature to 200 °C. We counted the fringe minima as a function of the temperature, and then fit the data by a computer to a power expansion in the temperature. We then calculated dn/dT taking into account the thermal expansion of the specimen. At 0.6328 μm we found dn/dT = $1.08 \times 10^{-4}/°C$ at 25 °C. In Table I we present values of dn/dT at 25 °C for several runs at 10.6 μm. Runs #1 and 2 were repeated measurements
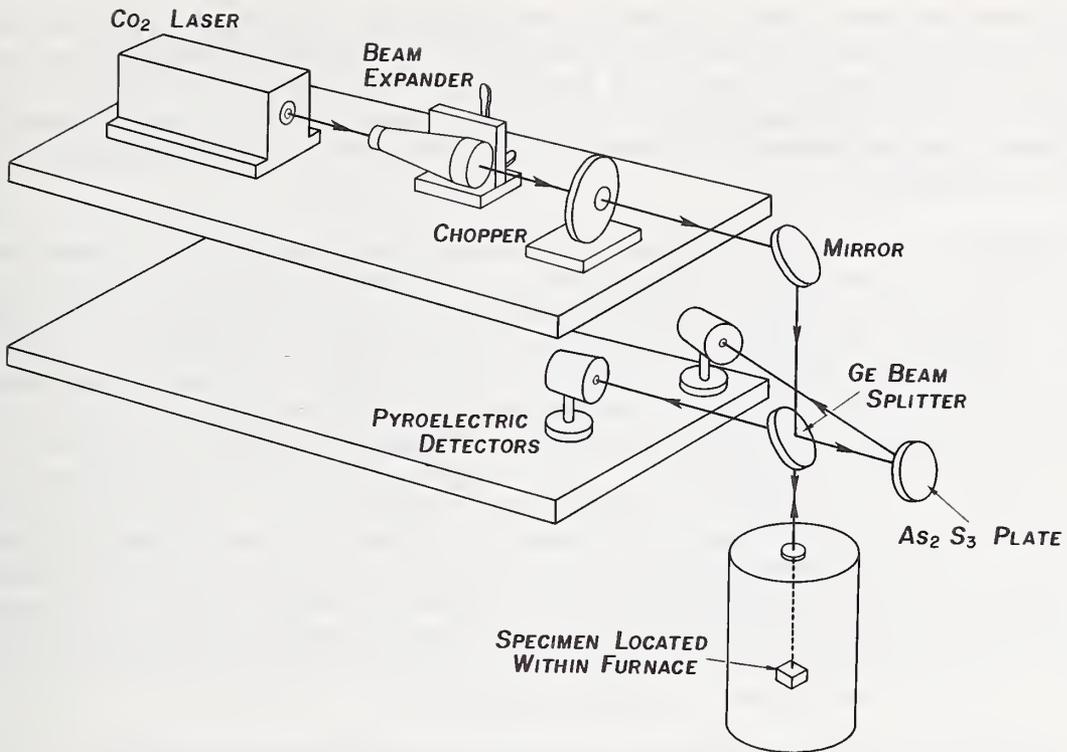
FIGURE 3. APPARATUS TO MEASURE CHANGE OF REFRACTIVE INDEX WITH TEMPERATURE AT 10.6 MICROMETERS.
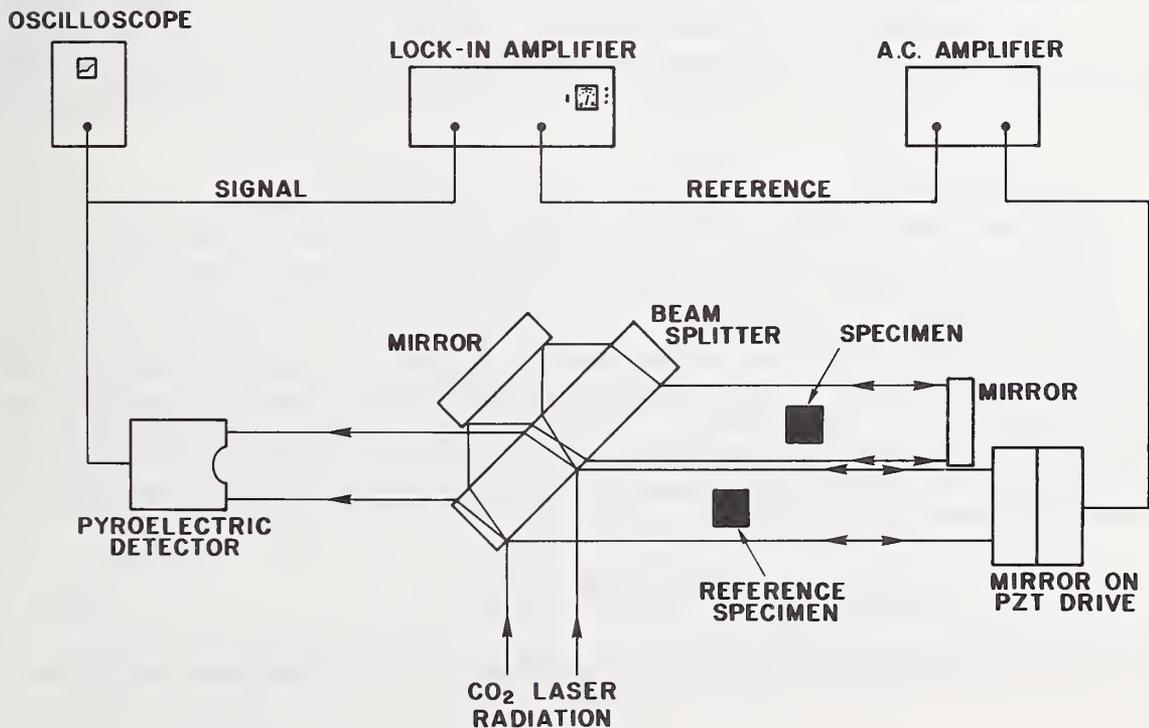

FIGURE 4. MODIFIED TWYMAN-GREEN INTERFEROMETER FOR MEASURING FRACTIONAL WAVELENGTH CHANGES OF OPTIC PATH AT 10.6 MICROMETERS

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on the same specimen. Runs #3 and 4 were made on another specimen but in different directions; in Run #3 the radiation was propagated parallel to the specimen growth direction while in Run #4 the radiation was propagated perpendicular to the specimen growth direction. Runs #5 and 6 were repeated interferometric measurements on the same prism that was used in the refractive index measurements.

The first column in the table was obtained by fitting the thermal expansion data to a linear curve; the second column was obtained by fitting the data to a quadratic curve. The data within a given column are quite reproducible although mean values of the two columns differ by about 7%. The values in both columns appear to differ significantly from the value obtained from the refractive index data. Additionally, values in the literature of dn/dT in polycrystalline ZnSe prepared by different methods vary from $5 \times 10^{-5}/°C$ to $1.0 \times 10^{-5}/°C$ [14]. The origin of these discrepancies is not yet known. We originally thought that a difference in dn/dT might be observed between light propagating parallel to the specimen growth direction and light propagating perpendicular to the specimen growth direction; however, the reproducibility of the data for runs #3 and 4 suggests that this assumption would not explain the discrepancy. The discrepancies might be due to sample variability; however, the consistency of our data for the three different specimens casts doubt on this assumption.

2.4 Measurement of Stress-Optical Constants and Elastic Constants

Previously [8,15] we discussed a method for determining the absolute stress-optical constants and the elastic compliance component s_{12} of a material and data were presented for several materials including CVD ZnSe [8]. Values of q_{11} and q_{12} at 10.6 μm could not be obtained for the ZnSe because the fringe shift at this wavelength was too small to measure with any accuracy. We have now developed a technique for measuring small fringe shifts and have obtained preliminary values for q_{11} and q_{12} at 10.6 μm. These values agree quite well with values obtained at 1.15 μm which suggests that the dispersion of the coefficients in the intervening wavelength range is small.

The apparatus used in this measurement is a Twyman-Green interferometer that has been modified so that fringe location can be determined electronically to a precision of 0.01λ at 10.6 μm. Figure 4 shows a schematic of the apparatus. The two arms of the interferometer are in close proximity, in order to minimize instabilities due to air currents and vibrations. The effects of vibration are also minimized by mounting the diagonal mirror on the same base as the germanium beam splitter and by mounting the two end mirrors on a common base. The end mirror in the specimen arm is mounted on a piezoelectric translator and undergoes a sinusoidal translation along the axis of the interferometer thus modulating the output intensity of the interferometer in the following manner

$$I = \frac{I_o}{2} [1 - \cos \left(\frac{4\pi A \sin \omega t}{\lambda} + \phi \right)], \tag{3}$$

where I_o is the intensity at a fringe maximum, A is the maximum excursion of

Table I. Interferometric Measurements of dn/dT in CVD ZnSe at 10.6 μm and at 25 °C

Run	Linear Fit	Quadratic Fit
#1	6.36×10^{-5}/°C	5.86×10^{-5}/°C
#2	6.35	6.03
#3	6.42	5.95
#4	6.42	5.92
#5	6.34	6.03
#6	6.36	5.91
Average	6.38 ± .04	5.95 ± .07

Value from Refractive Index Measurements 7.9×10^{-5}/°C

Table II. Photoelastic Properties of CVD ZnSe

λ (μm)	0.6328	1.15	10.6
n	2.590	2.473	2.403
$q_{11} (10^{-12} m^2/N)$	-1.32	-1.36	-1.39
$q_{12} (10^{-12} m^2/N)$	0.28	0.62	0.58
$q_{11}-q_{12} (10^{-12} m^2/N)$	-1.60	-1.96	-1.97
p_{11}	-0.10	-0.08	-0.08
p_{12}	-0.01	0.03	0.02

the modulated mirror about its average position, and ϕ is a phase related to the mean optic path difference of the two arms of the interferometer. The reference specimen, which is placed in the other arm of the interferometer, is a device which is made to produce a known optic path change. In our case we use a reference specimen of Ge in a compression apparatus. The Ge is self-calibrating because a shift of many fringes can be produced as a function of stress. Fractional fringes are then obtained by linear interpolation. In operation, the Ge is stressed until the interferometer is at a null which is determined by observing that the first harmonic of the output intensity is zero and the second harmonic is maximum. A given stress is then applied to the unknown specimen, which shifts the interferometer away from null, but which is compensated by applying an incremental stress to the reference specimen. From these data we obtain the change of optic path in the unknown specimen as a function of stress and combined with a knowledge of the elastic compliance component s_{12}, we obtain the stress-optical constant.

In Table II we summarize all our stress-optic data for a specimen of CVD ZnSe. We have slightly modified our previously reported values because of the improved values of refractive index we have obtained. Our new procedure was used to obtain q_{11} at 10.6 µm. We obtain q_{12} at 10.6 µm using the birefringence data $(q_{11}-q_{12})$. It is interesting to note the small differences between the values at 10.6 µm and 1.15 µm.

We have also measured the shift of Fizeau fringes in a specimen of CVD ZnSe as a function of hydrostatic pressure [8] using a HeNe laser source. This data, combined with the known stress-optical constants and the value of s_{12}, permitted us to calculate s_{11}. In Table III we summarize all the elastic moduli we have measured and derived for CVD ZnSe. These moduli have permitted us to calculate the photoelastic constants p_{ij}, which are shown in Table II [16].

In using the data we have obtained, some caution must be applied. Because we have treated the ZnSe as an isotropic material, future measurements are planned on several other specimens in order to ascertain whether one can expect significant differences in the coefficients of different specimens.

2.5 Acknowledgements

Table III. Elastic Properties of CVD ZnSe

Elastic Compliances:	$s_{11} = 14.5 \times 10^{-12} \; \frac{m^2}{N}$
	$s_{12} = -4.0 \times 10^{-12} \; \frac{m^2}{N}$
Elastic Constants:	$c_{11} = 8.8 \times 10^{10} \; \frac{N}{m^2}$
	$c_{12} = 3.4 \times 10^{10} \; \frac{N}{m^2}$
Poisson's Ratio:	$\nu = 0.28$
Young's Modulus:	$E = 6.9 \times 10^{10} \; \frac{N}{m^2}$
Rigidity Modulus:	$G = 2.70 \times 10^{10} \; \frac{N}{m^2}$
Bulk Modulus:	$K = 5.20 \times 10^{10} \; \frac{N}{m^2}$

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15. SUPPLEMENTARY NOTES

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.*)

We have measured the following parameters of chemical vapor deposited polycrystalline ZnSe (CVD ZnSe): Refractive index and change of index of refraction with temperature (dn/dT) over the wavelength range 0.5 μm to 18 μm using the method of minimum deviation; the coefficient of linear thermal expansion and dn/dT at 10.6 μm using Fizeau interferometry; and the elastic moduli and photoelastic moduli using Fizeau and Twyman-Green interferometry. A sensitive technique has been developed for measuring stress-optical constants of materials that exhibit a small stress-optical effect.

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*)

Coefficient of thermal expansion; elastic constants; infrared laser window materials; photoelasticity; polycrystalline ZnSe; refractive index; stress-optical constants; thermal coefficient of refractive index.

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