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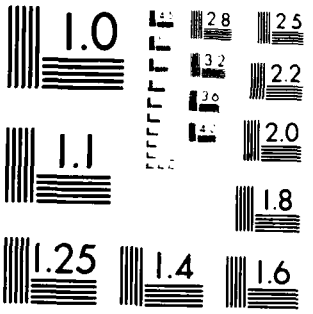

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DEVELOPMENT OF A HIGH-RESOLUTION THERMOPROBE

Final Technical Report

January 1983

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A method is being developed for measuring temperature with very high spatial (0.5 μm) and temporal (10 ns) resolution. This method, which exploits the light absorption characteristics of semiconductors, is of interest for studying the temperature rises associated with high-rate material deformation processes such as rapid crack propagation, sliding friction and wear, and shock-induced detonation of explosives.

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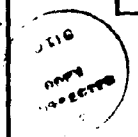
This report discusses the analytical and experimental examination of the temperature response of CdS single crystals and vapor-deposited polycrystalline films. A temperature sensitivity of 0.03K and a range of 300K were obtained. These values depended strongly on film thickness, film vapor-deposition processes, and post-deposition annealing, showing that sensitivity and range can be tailored somewhat for specific applications.



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I INTRODUCTION

Many long standing questions concerning material behavior under applied dynamic loads might be resolved by adopting a thermal, rather than a mechanical, investigative approach. The reason is that plastically deforming or fracturing material releases heat in proportion to the mechanical energy absorbed in the failure process. If the rate of deformation or fracture is low, the conversion from mechanical to thermal energy proceeds nearly isothermally, and no measurable rise in temperature occurs. At faster rates the conditions become more adiabatic, and material near the site of deformation or fracture may experience elevated temperatures for a brief time. Thus temperature histories, if they could be measured, would provide useful, and in some cases heretofore unattainable, information about material behavior under high loading rates.

Little attention has been given to the thermal approach, primarily because of the difficulty in making temperature measurements with sufficient spatial resolution. However, a novel optical method reported by Wieder[1,2] provides significantly higher spatial resolution and promises to be useful in studies of material failure behavior.

The technique makes use of the change in the ability of a semiconductor film to absorb light at different temperatures. Certain semiconductors exhibit a precipitous change in phototransmissibility with wavelength, Figure 1, and the location of this change, called the absorption edge, varies with temperature and film thickness. Thus, a thin film of the semiconductor attached to a specimen can be irradiated by a monochromatic beam of appropriate wavelength λ_o, and the change in light intensity ΔI after transmission through the film will indicate the temperature history of the specimen, Figure 2. High spatial resolution (to about one wavelength) can be

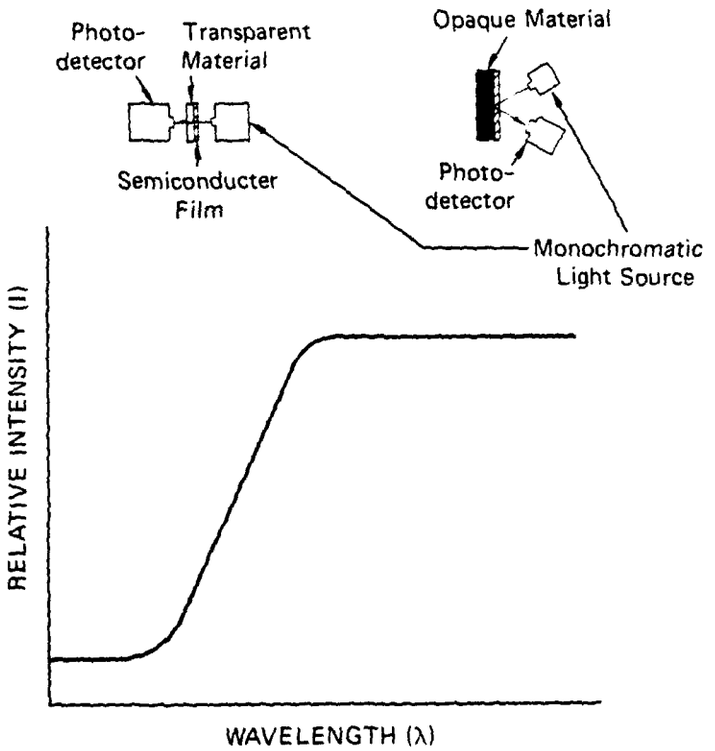

FIGURE 1 VARIATION OF INTENSITY WITH WAVELENGTH OF MONOCHROMATIC LIGHT TRANSMITTED THROUGH A SEMICONDUCTOR

Insets show arrangements for measuring temperatures of transparent and opaque specimen materials.

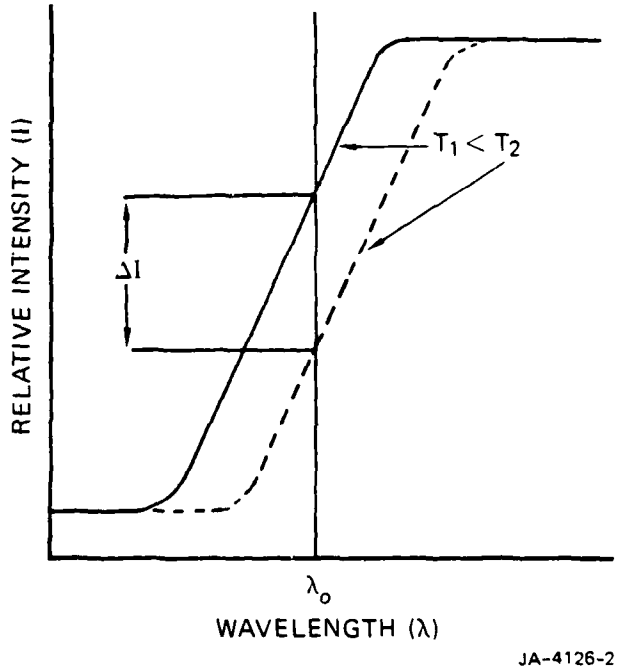

FIGURE 2 PRINCIPLE OF OPTICAL THERMOPROBE

The intensity of monochromatic light transmitted through a semiconductor film changes with temperature.

achieved by focusing the monitoring beam, and nanosecond time resolution is attainable. A thermal sensitivity of several hundredths of a degree can be realized. The useful temperature range can be several hundreds of degrees, depending on film thickness and wavelength.

This report describes efforts to develop the optical thermoprobe and to adapt it for studies of high rate material behavior. The theoretical foundations for the light absorption phenomena are summarized, and confirming experimental results on single crystals and vapor-deposited polycrystalline films of CdS are presented. Problems encountered in producing and tailoring the response of the practically more useful vapor-deposited films are discussed.

II THEORETICAL CONSIDERATIONS

The absorption of light passing through a medium of thickness d can be described by the equation:

$$I = I_o \exp(-\alpha \cdot d) \qquad (1)$$

where I_o and I represent the intensity of the incident and transmitted light, respectively, and α is the absorption coefficient of the medium.

Dutton[3] showed that the temperature dependence of α for CdS single crystals was well described by an expression of the form:

$$\alpha(\lambda,T) = \alpha_1 \exp\left[-\frac{\beta}{\kappa T}\left(E_o - \frac{hc}{\lambda}\right)\right] \qquad (2)$$

where λ is the wavelength of a monochromatic light beam, T is the absolute temperature, h is Planck's constant, κ is Boltzmann's constant, c is the velocity of light under vacuum, and α_1(= 2.17), β (= 1.798×10^9 cm^{-1}), and E_o are empirical constants. The values of E_o for incident light polarized perpendicular and parallel to the crystal axis are 2.586 and 2.608 eV, respectively.

Equations (1) and (2) describe the absorption edge dependence on temperature and crystal thickness and predict the following characteristics:

(1) The midpoint of the absorption edge (at the location $I/I_o = 0.5$) lies between 490 and 530 nm for sample thicknesses between 10^{-5} and 1 mm at room temperature. The relationship between the thickness of the CdS film and the wavelength at which the absorption edge occurs at 300K is shown in Figure 3 for light polarized perpendicular and parallel to the crystal

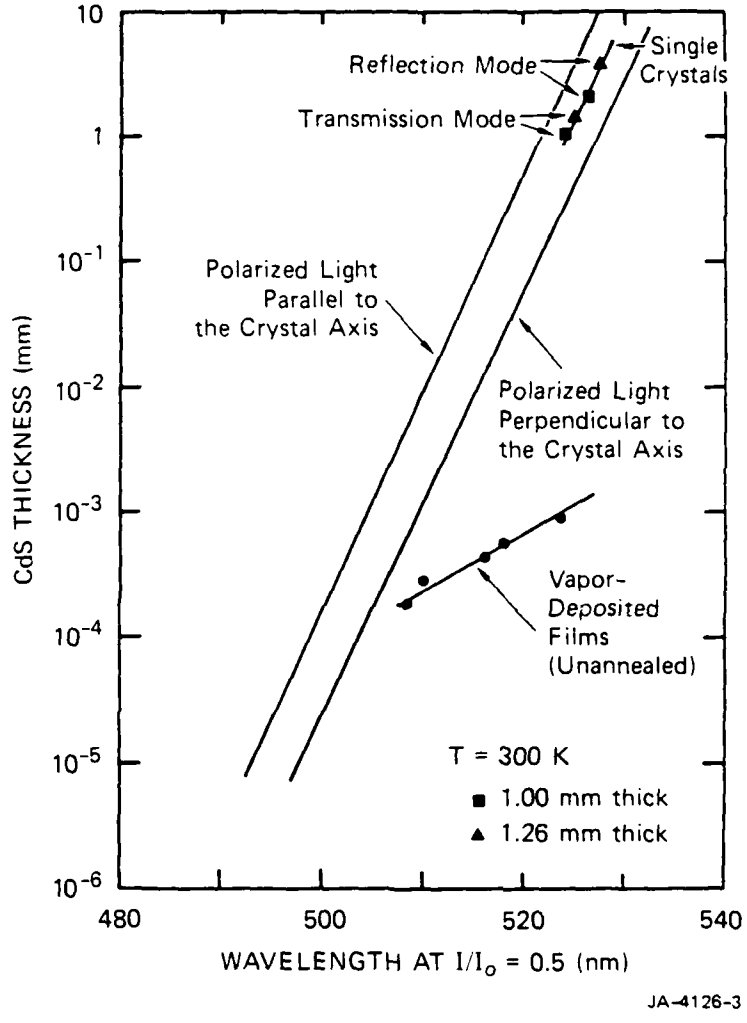

FIGURE 3 PREDICTED RELATIONSHIP BETWEEN WAVELENGTH AT MIDPOINT OF ABSORPTION EDGE AND CRYSTAL THICKNESS; COMPARISON WITH MEASUREMENTS

axis. These curves are useful in selecting the proper CdS film thickness for a given wavelength light source or in selecting the proper wavelength for a given thickness of CdS film.

(2) The steepness of the absorption edge, $\Delta I/\Delta\lambda$, depends slightly on crystal thickness as shown below.

Crystal Thickness d (mm)	Steepness $\Delta I/\Delta\lambda$ (nm^{-1})
10^{-4}	0.120
10^{-3}	0.117
10^{-2}	0.114
10^{-1}	0.112
1	0.110

(3) Higher temperatures shift the absorption edge to longer wavelengths. The amount of shift for various thicknesses for a 1K temperature rise is shown in Figure 4. The corresponding decrease in intensity per 1K is also indicated.

The measurable temperature range is governed by the steepness of the absorption edge. An approximate linear relationship between the change in transmitted intensity and temperature change can be expected, if measurements are done in an intensity interval ΔI between 0.9 I_o and 0.1 I_o, where I_o is the intensity of the incident light. The measurable temperature range is slightly dependent on crystal thickness as shown below.

Crystal Thickness d (nm)	Temperature Range (K)
10^{-4}	91
10^{-3}	75
10^{-2}	63
10^{-1}	54
1	48

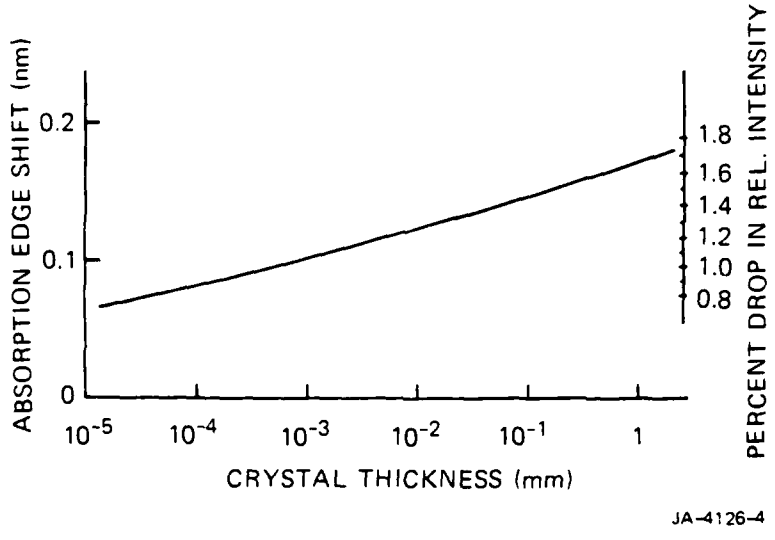

FIGURE 4 ABSORPTION EDGE SHIFT AND PERCENT DROP IN RELATIVE INTENSITY PER 1°C TEMPERATURE RISE AS A FUNCTION OF THICKNESS

Thus, the steeper the absorption edge, the greater the temperature sensitivity and the smaller the temperature measurement range.

These analytical predictions guided the development efforts described in the next section.

III EXPERIMENTAL PROCEDURES

The absorption characteristics of CdS single crystals and vapor-deposited films were determined with a Leiss double-dispersion monochromator with flint prisms using a 100-W tungsten halogen quartz lamp as a light source and two photomultipliers as light intensity detectors. Figure 5 shows the experimental setup. One photomultiplier measured the light intensity of the incident beam; the second measured the intensity of transmitted light through the CdS layer. The beam was focused on a circular area of 0.05 mm^2. The wavelength of the light beam was varied continuously from 425 to 680 nm in 20 s by rotating the prism of the monochromator with a synchronous motor. The monochromator dial reading was marked automatically with an photoelectronic sensor.

The output signals from the photomultipliers were directed into preamplifiers with an adjustable band width and recorded with a digital oscilloscope. The recorded data were processed with a desk top calculator interfaced with the oscilloscope. Relative absorption was plotted as a function of light wavelength. This experimental setup can be used for transparent or opaque surfaces by proper positioning of the active photomultiplier as shown in Figure 5.

The temperature dependence of the absorption edge was characterized by monitoring relative absorption as a function of temperature at a fixed wavelength and by monitoring absorption as a function of wavelength at a fixed temperature. In the first case the differential outputs from two photomultipliers were added together to cancel the disturbances of the light source during the experiment, and temperature was measured with a thermocouple attached to the face of the CdS layer.

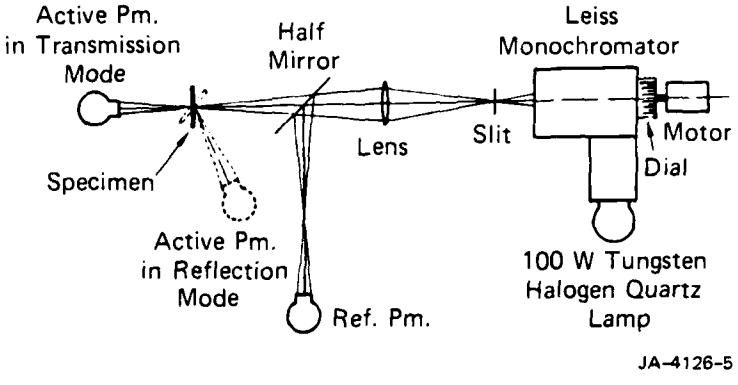

FIGURE 5 EXPERIMENTAL SETUP FOR ABSORPTION EDGE CHARACTERIZATION

Results obtained on single crystals are presented in the next section.

The results from vapor-deposited polycrystalline films are reported in Section V.

IV CdS SINGLE CRYSTALS

Single crystals of CdS provided by Dr. G. Fahrenbruch of Stanford University were attached to transparent glass and to polished steel substrates and examined by the procedures described in the previous section.

Figure 6 shows the relative intensity as a function of wavelength for crystals of two different thicknesses evaluated in the transmission and reflection measurement modes at room temperature. The wavelengths corresponding to the mid-points of the absorption edges ($I/I_0 = 0.5$) were determined from the curves and are plotted in Figure 3. Since the light used was nonpolarized, the data fall between the analytical curves for perpendicular and parallel polarized light. The agreement between the analytical and experimental curves is excellent.

The temperature dependence of the absorption edge for the 1-mm-thick crystal was determined in the reflection mode. Figure 7 shows the shift produced by a temperature change of 32.4K, and indicates a sensitivity of 0.17 nm per degree Kelvin at the absorption edge midpoint, a result that agrees well with the analytical prediction in Figure 4.

A second temperature characterization was accomplished by fixing the light wavelength at 526.2 nm and observing the light intensity change due to the temperature-induced shift of the absorption edge. Figure 8 shows that the relative intensity changed linearly with temperature during a heating and cooling cycle.

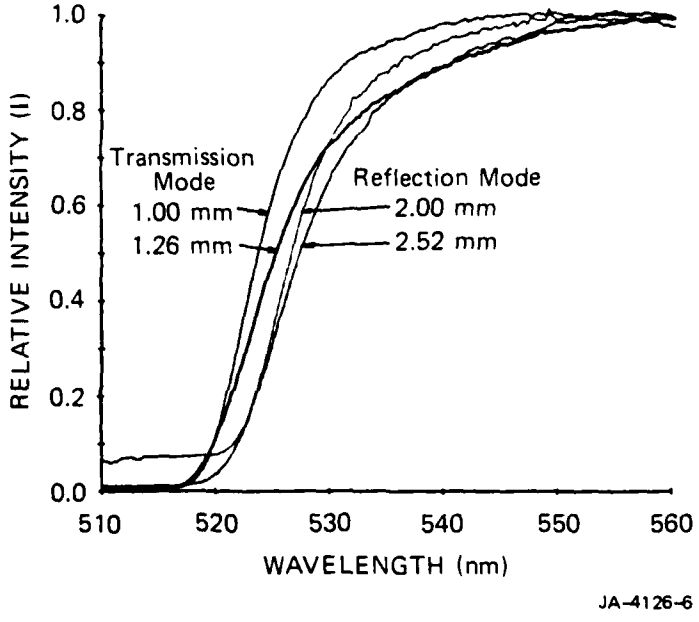

FIGURE 6 ABSORPTION EDGE CHARACTERISTICS OF CdS SINGLE CRYSTALS OF DIFFERENT THICKNESSES

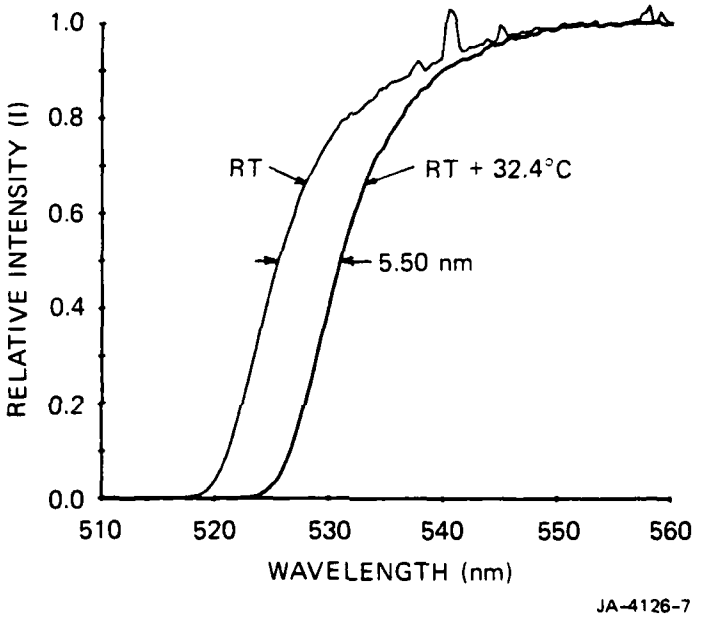

FIGURE 7 ABSORPTION EDGE SHIFT OF CdS SINGLE CRYSTAL DUE TO 32.4°C TEMPERATURE RISE

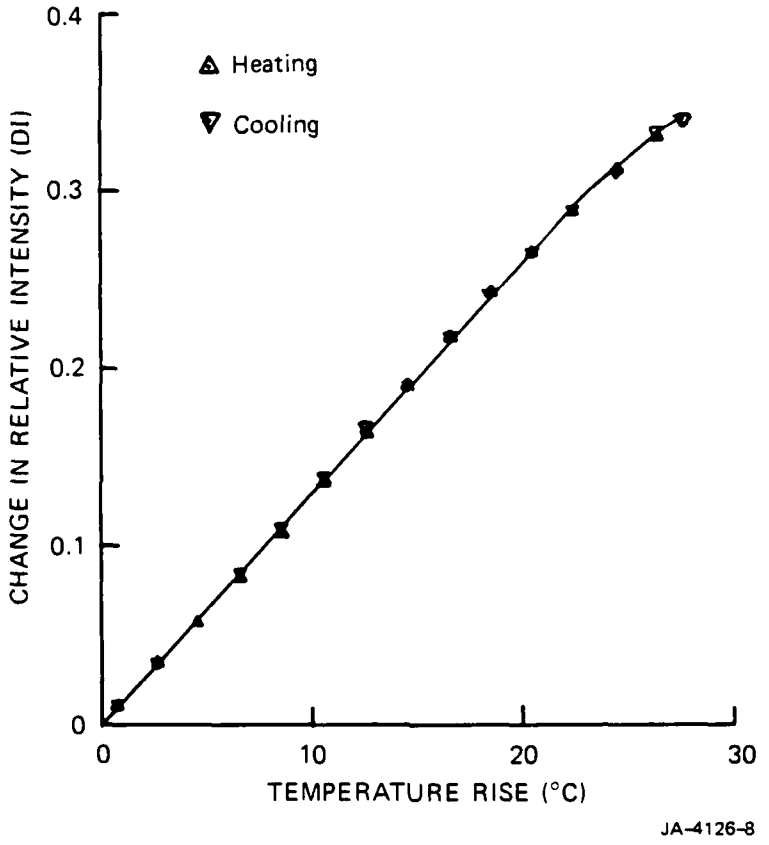

FIGURE 8 RELATIVE INTENSITY CHANGE OF CdS SINGLE CRYSTAL DURING HEATING AND COOLING CYCLE OBSERVED AT A FIXED WAVELENGTH OF 526.2 nm

V VAPOR-DEPOSITED CdS FILMS

CdS films were vapor-deposited onto glass and polished steel substrates by placing 99.99% pure optical quality CdS powder in a molybdenum boat and evaporating in a vacuum chamber. Substrate temperatures between 200° and 300°C were necessary to produce optical quality films that adhered to the substrate.

Figure 9 shows the absorption characteristics of vapor-deposited films of various thicknesses measured at room temperature. The absorption edge wavelengths (at $I/I_o = 0.5$) were determined from these curves, and the values are plotted in Figure 3. It is apparent that the behavior of vapor-deposited CdS is quite different from that of single crystals. The absorption edge of the vapor-deposited film occurred at a longer wavelength than that for single crystals. Furthermore, the observed steepness of the absorption edge, $\Delta I/\Delta\lambda$, was less than one-third of that for single crystals.

The temperature sensitivity of the vapor-deposited CdS films is shown in Figure 10. The amount of shift observed was 0.044 nm per one degree Kelvin temperature change--about one-third of the single crystal response.

The cause of this anomalous behavior of vapor-deposited CdS film was suspected to be nonstoichiometry,[4] so we attempted to achieve more nearly stoichiometric conditions through heat treatment.[5,6] Best results were obtained by packing the samples in CdS powder and heating them for one hour to about 600°C in an argon gas environment. Figure 11 shows the absorption characteristics of a heat-treated film. The absorption edge shifted to a shorter wavelength, and temperature sensitivity improved to the level of a single crystal.

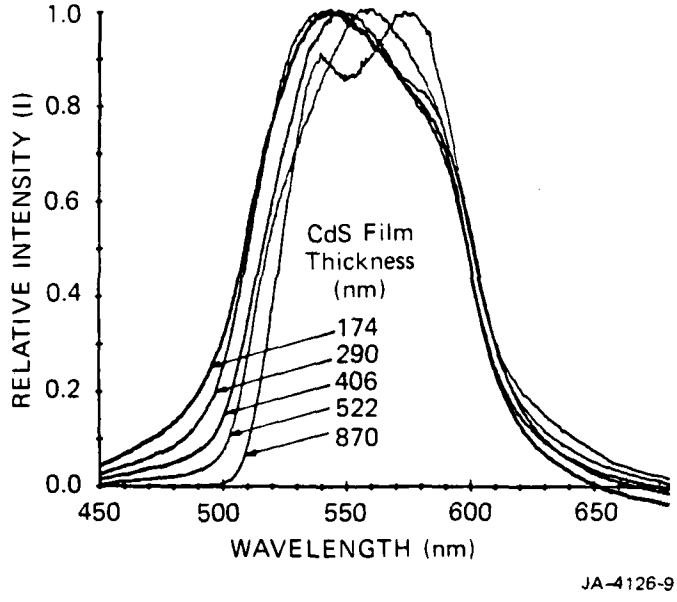

FIGURE 9 ABSORPTION EDGE CHARACTERISTICS OF VAPOR-DEPOSITED CdS FILMS OF DIFFERENT THICKNESSES

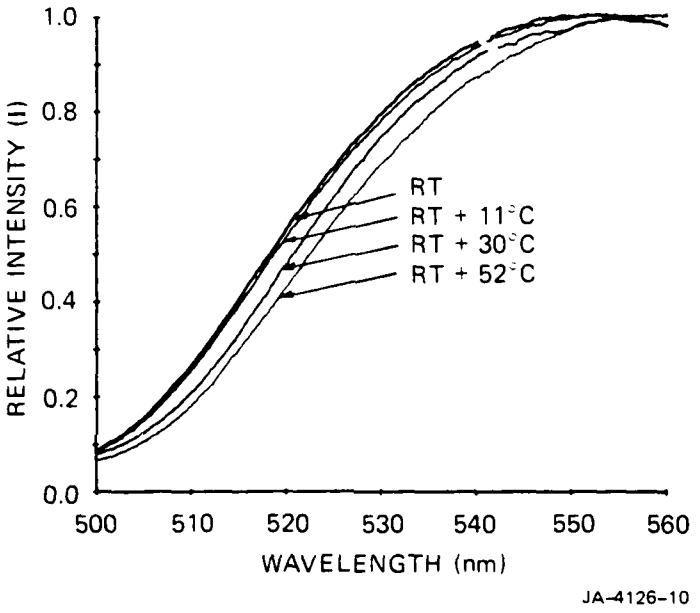

FIGURE 10 ABSORPTION EDGE SHIFT OF VAPOR-DEPOSITED CdS FILM DUE TO INCREMENTAL TEMPERATURE CHANGES

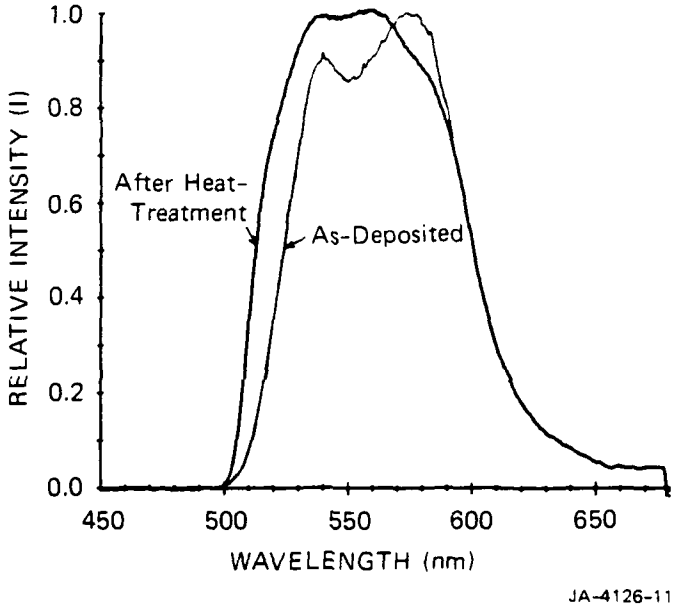

FIGURE 11 ABSORPTION EDGE CHARACTERISTICS OF VAPOR-DEPOSITED CdS AS DEPOSITED AND AFTER HEAT-TREATMENT

VI DISCUSSION

Two methods of measuring the temperature were demonstrated: the entire absorption edge characterization method and the method of measuring intensity at a fixed wavelength.

The first method is suited only for quasi-static temperature changes since it took approximately 20 s to sweep 450-680 nm. Because this method does not depend on the intensity change due to the shift of absorption edge, the temperature measurement range is limited only by the break-down of crystals.

The second method, which entails measuring the intensity change at a fixed wavelength, is suited for dynamic temperature measurements, but requires a more intense light source than the monochromator. Ar^+ lasers (514.5 nm), Kr^+ lasers (520.8 and 530.9 nm), or tunable CW dye lasers are suitable light sources for CdS films because they lase at wavelengths within the absorption edge.

The absorption edge can be adjusted by tailoring the thickness of the CdS layer. We experienced difficulty in thinning single crystals down to about 0.03 mm for the Ar^+ laser. However, suitably thin CdS films can be produced by vapor-deposition.

The achievable temperature resolution is governed by the signal and noise levels. For the photomultiplier/monochromator system, the signal level was approximately 130 mV and the noise level was $\pm$ 0.5 mV, which corresponds to $\pm$ 0.4% relative intensity change. The signal-to-noise ratio is characteristic of the photomultiplier. The relative intensity change of $\pm$ 0.4% corresponds to a temperature change of $\pm$ 0.3K. Experiments using the Ar^+ laser and photodiodes produced a signal level of 480 mV and a noise level of $\pm$ 0.2 mV. This improved the resolution to $\pm$ 0.04% intensity change and $\pm$ 0.03 K.

An advantage of vapor-deposited CdS films is that films of desired thickness (to match the wavelength of the light source) are easily produced. Since the response time of CdS film is proportional to the product of temperature difference and square of the thickness of the film, thinner vapor-deposited films show better temporal response.

A disadvantage is that as-deposited CdS films have poor temperature response and the high substrate temperatures required to achieve a suitably sharp absorption edge may change the substrate properties. Further research is needed to establish appropriate processes for fabricating good quality CdS films.

VII SUMMARY

The optical thermoprobe concept has been further developed and appears applicable to investigations of dynamic material behavior. Theoretical predictions of light absorption behavior and its dependence on light wavelength, semiconductor thickness, and temperature were substantiated by experiments on single crystals and vapor-deposited films of CdS. The suitability of the technique for opaque surfaces was demonstrated.

Better ways are needed to produce single crystal layers in appropriate thicknesses and to attach them to the surfaces whose temperatures are to be measured. Requirements on layer thickness and adherence to substrate are conveniently met by vapor-deposition or sputtering, but care must be taken to achieve stoichiometry. Additional work is needed to address these concerns.

Future work is also recommended to determine the useful temperature range and the tradeoff between range and temperature sensitivity, to characterize other semiconductors such as GaAs, and to develop scanning and imaging techniques. Experiments should be conducted to apply the technique in its present state of development to measure temperature gradients produced by sliding friction.

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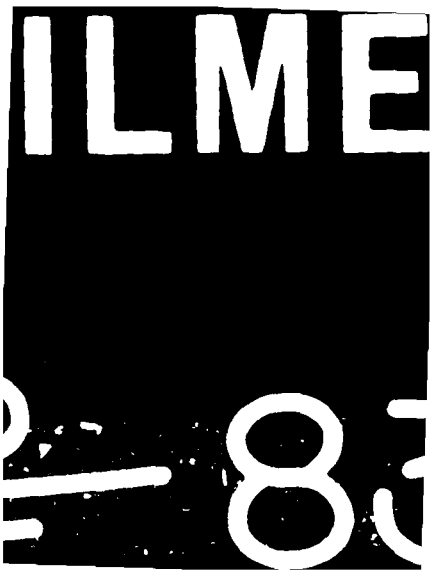
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