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Design and measurements of n for the multicomponent semiconductor

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ABSTRACT

A great deal of research has been performed on refractive index n and extinction coefficient k due to varieties of applications in optical industries. The dispersion equation is described for the photons of varying energies and their interactions with materials since there is a strong correlation of n and k with wavelength. Measurements based on reflectance can be expensive and are very difficult due to compositional variations. We present a low-cost reflectance probe fiber optics designed in-house to determine the absorption coefficients and refractive index of solids. The solutions using a modified Beer-Lamberts Law and merging the concentration and extinction coefficient terms into an absorption coefficient, α, can be given by the equation $I = I_0 \exp(-\alpha^* d)$ where I is the transmitted intensity, I_0 is incident intensity and d is the thickness. We have experimented with several semiconductor compounds for this study.

1. INTRODUCTION

Reflectance is measured by shining light on a sample and measuring the light reflected from the sample. Reflected light consists of specular reflected light and diffuse reflected light, which when combined together is referred to as total reflected light. Specular reflected light is the light reflected from shiny mirror-like surfaces at the same angle as the incident angle. Diffuse reflected light is the light reflected in all directions from rough surfaces, such as paper and powder surfaces. Total reflected light is the total combination of specular and diffuse reflected light and is often measured when measuring samples that have both rough and shiny characteristics, such as plastic and painted samples. Reflectance measurements measure either the relative or absolute reflected light. Relative reflectance measurements calculate the proportional amount of reflected light measured from a sample surface, relative to the amount of reflected light measured from a reference plate, such as barium sulfate or a mirror. The relative reflectance is calculated based on assuming the reference plate has a reflectance of 100 %. Therefore, it is very important to manage reference plates properly because different reflectance values can be obtained if reference plates are substituted or if they become contaminated or change characteristics[1].

$$Relative\ Reflectance\ (R\%) = \frac{Amount\ of\ Light\ Reflected\ from\ the\ Sample}{Amount\ of\ Light\ Reflected\ from\ the\ Reference\ plate}\ x\ 100$$

Absolute reflectance measurements calculate the proportional amount of reflected light relative to the amount of light measured directly from a light source, not using a reference plate such as barium sulfate or a mirror. Reflectance measurement values are based on assuming a 100 % reflectance for air. Absolute reflectance measurements allow determining the true reflectance of samples[1].

$$Absolute\ Reflectance\ (R\%) = \frac{Amount\ of\ Light\ Reflected\ from\ the\ Sample}{Amount\ of\ Light\ Used}\ x\ 100$$

Devices for measuring reflectance such as fiber optic probes and integrating spheres can be expensive and have limited adaptability to materials of different shapes and compositions [2]. Other devices for measuring reflectance are inserts that go into spectrophotometers, but still have limited adaptability to certain types of samples[3]. To mitigate these issues, additive manufacturing is being employed to design and fabricate low-cost devices to obtain reflectance data.

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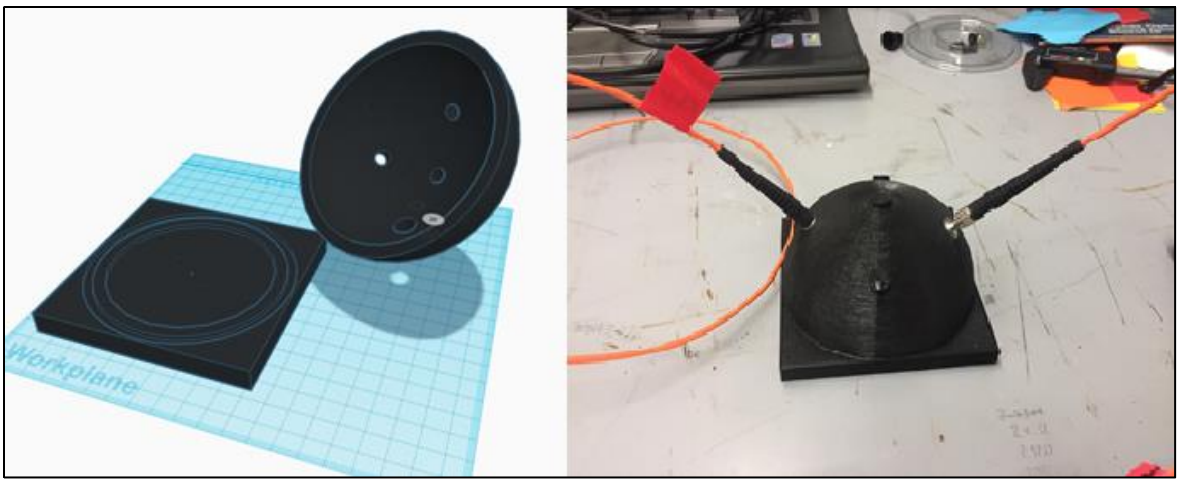
In-house design and fabrication allow for customizability of measurement devices to different samples that can be made cheaply and quickly.
To obtain values of refractive index n and absorption coefficient α from reflectance, several relationships will be employed. A modified Beer-Lamberts law merging the concentration and extinction coefficient terms into absorption coefficient α can be given by the equation $I = I_0 \exp(-\alpha^* d)$ where I is the transmitted intensity, I_0 is incident intensity and d is the thickness [4]. To determine the refractive index from reflectance, the relationship $n = \frac{1+\sqrt{R}}{1-\sqrt{R}}$ is used[5].

2. EXPERIMENTAL METHODS

2.1 Sample holder; For the reflection studies we developed custom designs in-house. We have designed two different designs for holding the samples and reflecting light for the reflection measurements.

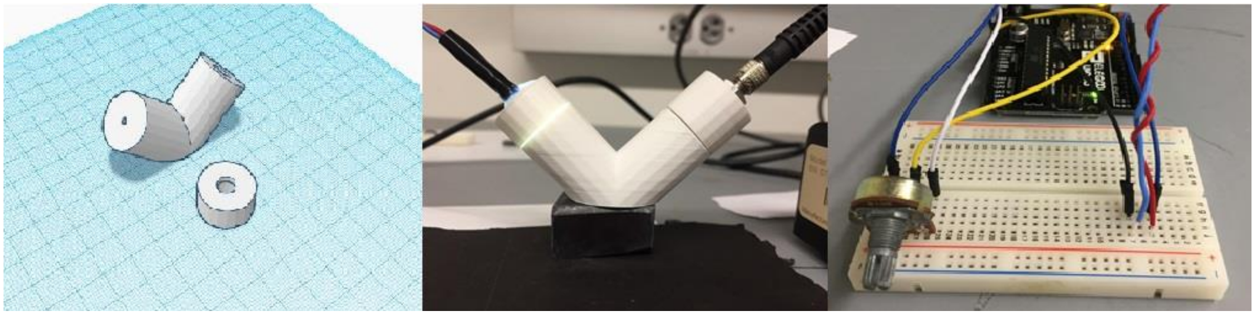
In the first design we developed a dome shape container. The dome design consists of a cover and a base, both made of black polylactic acid (PLA) and made using additive manufacturing. The cover is 100 mm in diameter, 50 mm in height, and 10 mm thick. The cover has four 7.9 mm ports for SMA905 fiber optic connections and one 11.1 mm port for a collimator connection. The cover has one port at normal and the four others at 45° from normal. The base is 105x105 mm with a sample region in the center that is depressed 2 mm to accommodate thin film samples.



(a) **(b)**

Figure 1. (a) 3D model of reflectance dome and (b) 3D printed reflectance dome with fiber optics setup for 45° angle reflectance measurements.

The second design utilized a V shape sample light holder. V design has two ports 45° from normal. One port allows a white LED light with variable intensity controlled by an Arduino, and the other port allows an SMA905 fiber optic connection. For both designs, two reference standards were used to obtain a lamp profile. Glossy photo paper was used for materials with low specularity and a mirror was used for materials with high specularity. Reference standards and light intensity were chosen for each sample to obtain a spectrum that did not saturate the detector while still providing sufficient incident intensity. The detector used was an Edmund Optics BRC-111A-USB-VIS CCD spectrometer with BWTek software.



(a) (b) (c)

Figure 2. (a) 3D model of V shaped design. (b) Printed V shaped design with LED light and SMA fiber optic connections on a solid. (c) Arduino and potentiometer controls for varying LED light intensity.

3. RESULTS AND DISCUSSION

We used an LED lamp as the light source in the present study. The experimental data indicates that the light source does not provide a uniform intensity over the spectrum of interest (420 – 760 nm). However, it provided reasonably good intensity over the visible range to obtain reflectance spectra. We have measured reflectance of several industrially important compounds developed for high operating temperature infrared detection, acousto-optical devices, radiation detection and wide bandgap applications including Tl_4HgI_6, Tl_3AsSe_3, $TlPbI_3$, Tl_4HgI_6, AlSiC, Hg_2Cl_2 and Hg_2Br_2. In this paper we report the data on measurements of $TlPbI_3$ inhouse grown crystal. We have used both polished and unpolished samples to evaluate the effect of polishing. A slab of crystal from a previously grown inhouse Bridgman crystal was cut and measured.

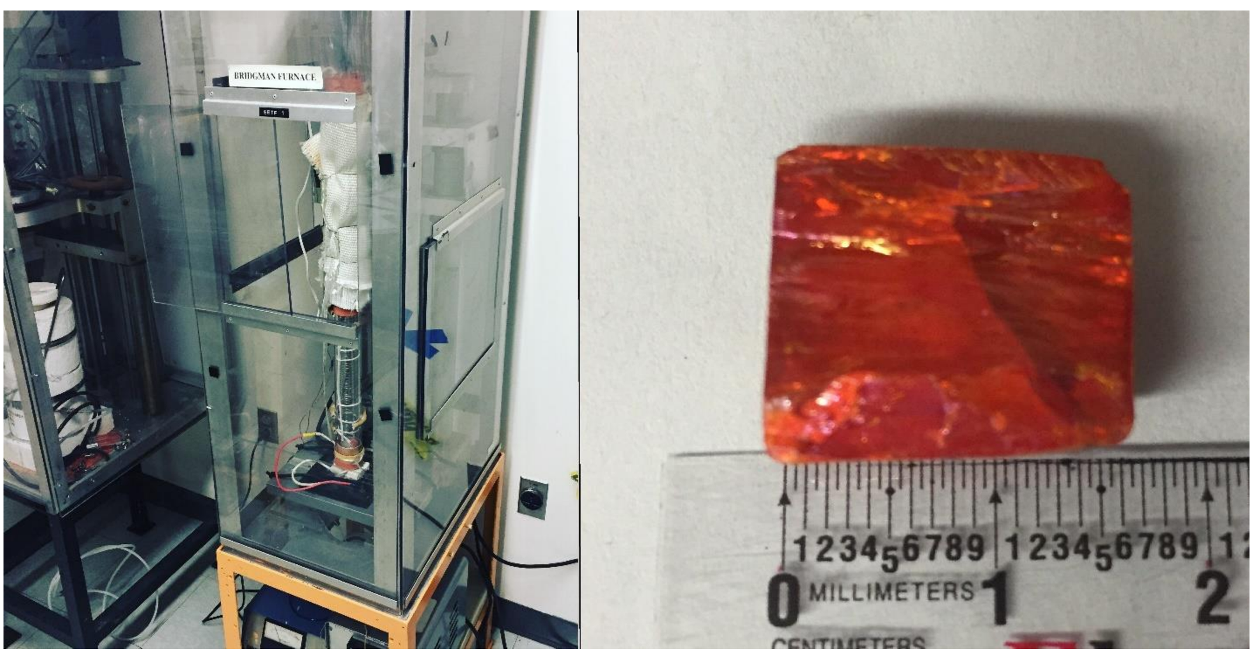


Figure 3. *Left*: Bridgman furnace used for single crystal growth. *Right*: TlPbI sample cut, unpolished

We used the modified Beer-Lambert equation, $I = I_0 \exp(-\alpha^* d)$ for the calculation. Generally this is typically used with thin films and not large solids[6]. The calculations used in this paper assumed that the reflectance incorporated transmission and that $I/I_0 = R$ was an appropriate assumption as it has been shown earlier that TPI does not transmit below 0.61 μm[7]. For opaque samples, however, this may not be appropriate as the width of the entire object was used in the modified Beer-Lambert equation when in reality the incident light may only penetrate a very small distance beyond the surface. This idea is illustrated in Figure 4. The absorption coefficient in Figure 5 shows that α is approximately 4.5 cm^{-1} in the range of 420 – 760 nm. Due to imperfections in the unpolished surface of the TlPbI sample, a large amount of light was scattered causing loss in signal/noise ratio in the spectrum. The material begins to transmit at wavelengths above 0.61 μm, with a sharp peak at 611 nm as shown in Figure 4. Detection limits in this experiment limit the wavelengths investigated up to only 760 nm, so although it is expected that the material will continue to show high transmittance beyond 760 nm, further research in that range is required.

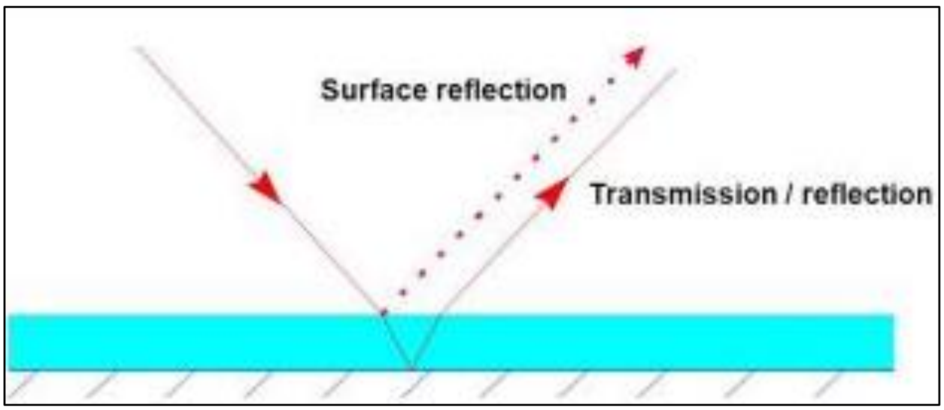


Figure 4. Theory of transmission/reflection in relation to modified Beer-Lambert law [similar to ref.6]

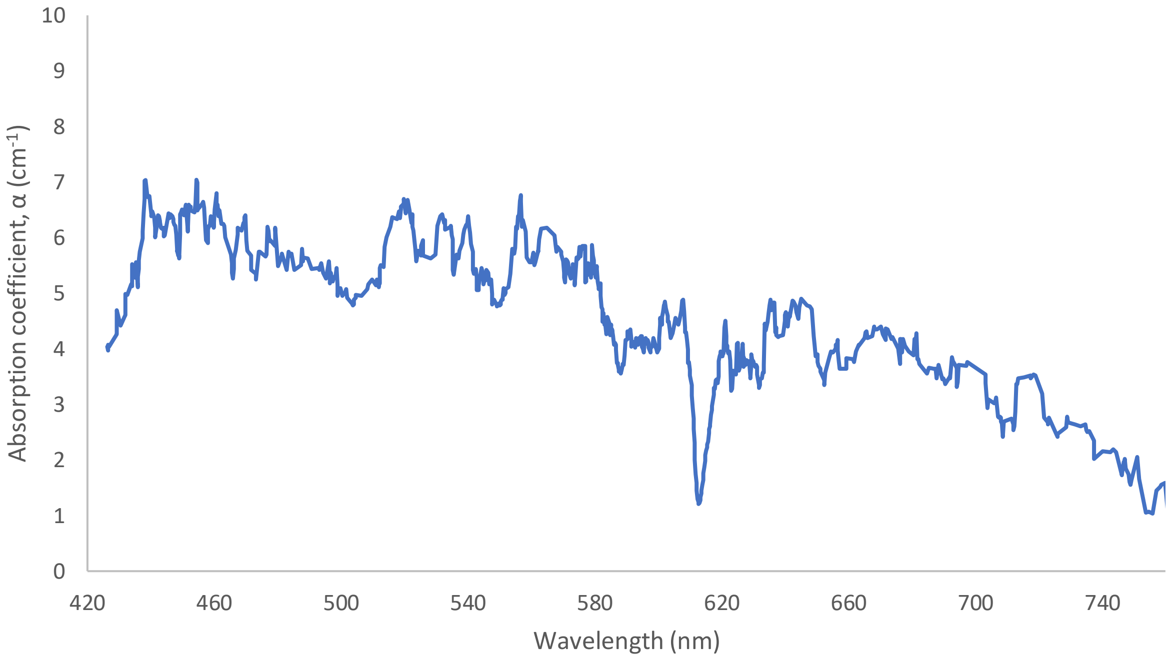


Figure 5. Absorption coefficient of TPI

Because of this discrepancy with how to treat the variable concerning optical path length, d, we compared data with predictions made by using bandgap of the material. Several equations are summarized by Saraf et al [7]. These equations are based on previously published data by several authors [8, 9]. The predictions estimated by using several equations based on bandgap showed refractive index values between 2.2 and 2.8. Figure 6 shows the estimated refractive index using the experimental reflectance data reaches n = 2 at wavelengths where the material begins transmitting, indicating that refractive index estimations made using reflectance data are lower than estimations made using the band gap energy but still comparable. We had synthesized the material using TlI and PbI_2 and purified using directional solidification. Several small absorption peaks may be due to fractured surfaces since we used a cleaved surface for this study. Most data published on optical properties of semiconductor materials are in the NIR-IR range as opposed to the visible range, so published material in the visible range with which to make comparison is sparse. We are continuing this study with several single crystals of semiconductors and results will be communicated in future communication [10].

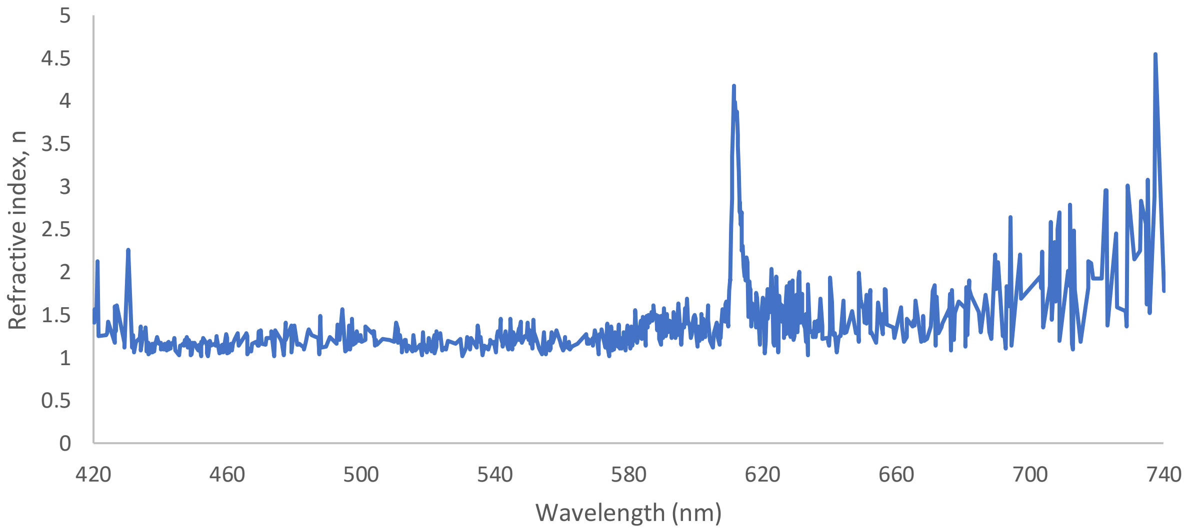


Figure 6. TPI refractive index estimated using measured reflectance data.

4. SUMMARY

A low-cost reflectance fiber optics Probe designed in-house to determine the absorption coefficients and refractive index of solids was fabricated using additive manufacturing. Reflectance reference standards with appropriate specularity are integral to acquiring reflectance spectra, and a mirrored surface works very well as a reference standard for materials with high specularity. Optical constants including absorption coefficient and refractive index were estimated using reflectance data. The modified Beer-Lambert equation and optical dispersion relationships was used to estimate refractive index below transmission region. These values are comparable with refractive indices estimated by various models based on bandgap of the $TlPbI_3$ crystal.

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