

Optical Spectroscopy of Transparent Conducting Oxides from the UV to  
near-IR and a Method for Determining the Refractive Index of Transparent  
Thin Films

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### **Abstract**

Over the course of approximately one year I rebuilt and enhanced an optical spectrometer located in Weniger hall, room 118 at Oregon State University. Throughout this time I became familiar with the spectrometry concepts of thin films and powders. Generally, simple transmission and reflection measurements were made on thin films by positioning a detector in the path of the reflected or transmitted light, but occasionally an integrating sphere was used in order to collect scattered light from these films or from powders. An Oriel 70323 lead-sulfide detector was added to the system in order to extend the spectrometer's long wavelength limit from 900nm out to 2.6 $\mu$ m. Measurements on CuScO<sub>2+y</sub> films were taken in this IR region and reflectances and transmittances of 20% and 70%, respectively, were found for such a film named sm16d. At the end of my work, I wrote and developed a Microsoft Excel Workbook that calculates the refractive index of thin films. A refractive index of approximately 2.1 was measured in the visible region for sm16d using this workbook. Unfortunately, there are no other measurements of the refractive index of this film to compare this number to, but this does not seem to be an unreasonable value.

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## I. Introduction

Recently at Oregon State University there has been a considerable amount of research in the field of transparent conductors. A transparent conductor is simply a material that is transparent to visible light, and also conducts electricity. Tin-doped indium oxide, commonly known as ITO, is the current industry standard. ITO is excellent because it has a transparency of ~90% in the visible region, and a relatively high conductivity. However, like nearly all other transparent conductors known before 1997, ITO is n-type (the charge carriers are negative). In order for transparent conductors to become more useful, p-type (positive charge carriers) conductors should be found so that the electronics industry can create diodes and transistors followed by more complex transparent circuits. Just this winter, John Wager's group published an article on the use of zinc oxide to fabricate a transparent thin film transistor (TTFT) [Ref. 1]. This transistor was important because, in comparison to other groups, it possessed a high level of transparency combined with good electrical performance. Because of this, it represents a substantial step forward in the engineering of transparent electronics. The uses for transparent electronic devices range from improved efficiency of computer monitors and photo-voltaics to decreasing the size of portable electronic devices by placing some of the circuitry over the display of the device.

I began my research in the winter of 2002 by becoming familiar with an optical spectrometer system that I took over from Derek Tucker. A spectrometer is an optical device that measures the wavelength dependence, or wavelength spectrum, of the transmittance or reflectance of a sample. The transmittance of a sample is the fraction of light that is transmitted through the sample while the reflectance is the fraction that is

reflected from it. Generally, features of the transmittance and reflectance spectra are used to determine other properties of the sample, including the index of refraction, the absorption or the band-gap. So, although the spectrometer measures only the transmittance and/or reflectance of a sample, this data can be used to determine other, potentially more useful, properties of the sample.

When I began, the spectrometer was capable of taking reflection and transmission spectra from 200-900nm. It also included an integrating sphere that could be used to gather scattered light from a film or powder sample. As time passed I became increasingly familiar with the system and began to make improvements. I started by rebuilding the box in which many of the system's components were housed. When I did this I made room for components I anticipated adding. Once this was done I realigned the optical components and added an infrared (IR) detector along with an extensive number of components necessary to retrieve meaningful data from this detector. Later I added an optical cryostat, but it is not discussed in this document. Finally, I spent a great deal of time creating a Microsoft Excel Workbook that calculates the index of refraction of thin films. This workbook, started by David McIntyre, evolved through many stages but finally became a powerful tool for obtaining the index of refraction for the films. In the end I obtained meaningful data from the spectrometer in the IR. A thin film of oxygen-intercalated  $\text{CuScO}_{2+y}$ , a p-type transparent conductor, was used as an example throughout this document. Called sm16d, this sample had a reflectance of ~20% and a transmittance of ~70% in the IR region. Using the Excel Workbook, I calculated an index of refraction for this sample to be approximately 2.1 in the visible region and approximately 2.0 in the near IR. Similar calculations were done on other materials.

I believe that the most valuable lesson I learned throughout my work was what it means to do research. I found that most of the time research involves making approximations and error corrections, ordering materials, trying to figure out why a data set doesn't look like we think it should, plumbing, debating with one's professor and generally – but not always – finding out the professor was right, tracking down parts, troubleshooting the system and software, keeping logs of *everything*, and repetition... lots of repetition. I learned that very rarely does research involve brilliant revelations of new understanding, and that when this does occur it is generally after a great deal of hard work. In spite of the repetition, I generally found my research very interesting. I was fortunate enough to work on the project over the summer of 2002, and unlike many undergraduate research projects, the work I was doing did not require constant supervision by a professor or graduate student. I had a lab virtually to myself and a spectrometer system that I was encouraged to tinker with and enhance.

In this thesis I have outlined and explained some of the complications and intricacies that I encountered along my way. My hope is that this format will not only represent what I have learned and spent most of my time doing, but that it will also prevent others from spending too much time figuring out what I've already figured out. There may be a lot of repetition in research, but the less the better.

## II. Basics of Spectrometry and the UV-Visible Region

In this section I begin by exploring the basic concepts of spectrometry. This includes definitions of terms and basic equations but most importantly this means understanding what the terms reflectance and transmittance mean. I then continue on to

discuss UV-visible measurements wherein the idea of absorptance is introduced.

Equations are then derived for determining the absorption coefficient – a fundamental, intrinsic parameter of materials – from data collected using the spectrometer. At the end of the section I discuss sources of error and potential methods for reducing the error.

### II.1. Thin Film Spectrometry: Transmittance and Reflectance Spectra

The basic idea in thin film spectrometry is to shine light of a particular wavelength, or energy, at a sample, and determine the percentage of the light that it reflects, transmits and absorbs at that wavelength. One then repeat this process for many different wavelengths to obtain a “spectrum” of the sample. In general the word “spectrum” has many different uses and might widely be defined as the distribution of a characteristic of a physical system as a function of some variable [Ref. 2]. This definition works here when “variable” is either wavelength or energy and “characteristic” is usually transmission or reflection.

Nearly all the data that I collected was a transmission, reflection or reference spectrum. It is important to understand that every optical component of the spectrometer has its own spectrum. Every lamp, lens, mirror, monochromator grating, filter, detector, *etc.* has its own wavelength dependent spectrum. For example, the tungsten halogen lamp does not emit light below ~240nm while the xenon arc lamp emits all the way down to ~175nm. Furthermore, fused silica lenses were used instead of standard grade silica because standard grade silica absorbs in the UV, while fused silica does not. A lamp that emits in the UV does little, besides creating toxic ozone gas, if the lenses absorb the UV light before it reaches the detector.

Grating's Blaze Wavelength	Wavelength Region
0.25 $\mu\text{m}$	0.175-0.7 $\mu\text{m}$
0.5 $\mu\text{m}$	0.3-1.0 $\mu\text{m}$
1.0 $\mu\text{m}$	0.6-2.0 $\mu\text{m}$
2.0 $\mu\text{m}$	1.2-4.0 $\mu\text{m}$

**Table 1:** In our lab we used the grating's blaze wavelength as the name of that particular grating i.e. we referred to the grating in row 1 as "the .25 $\mu\text{m}$  grating". The usable wavelength region of each grating is displayed in the second column.

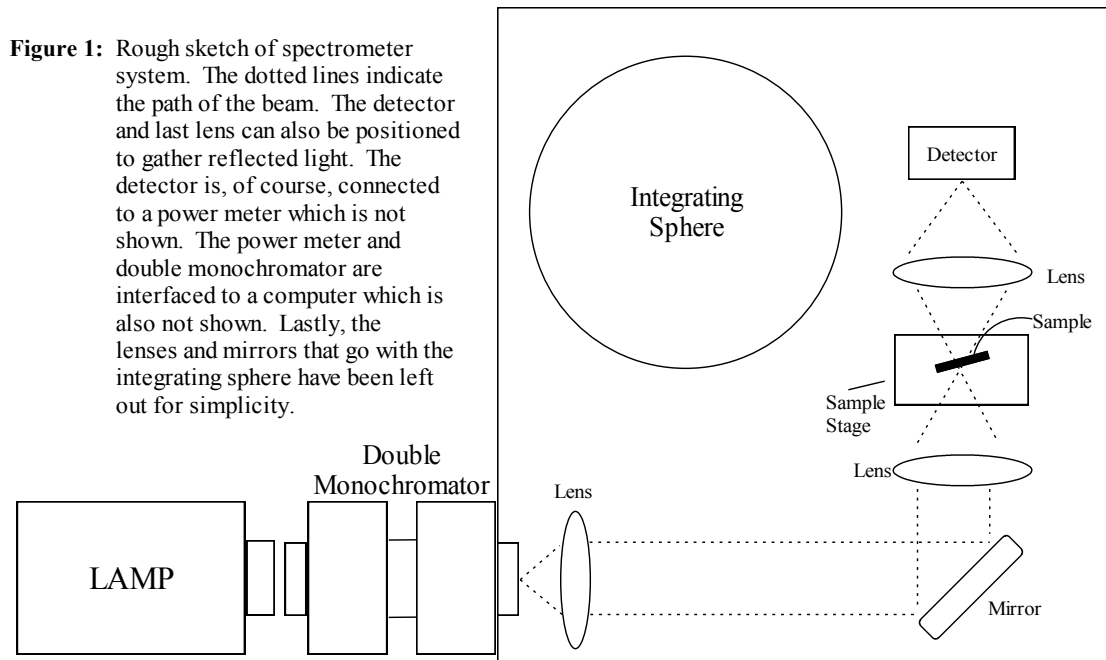
The UV-visible configuration includes a silicon photo-detector, xenon arc lamp and two 0.25  $\mu\text{m}$  gratings in the double monochromator. A grating is an optical component with tightly spaced microscopic parallel lines cut into it. The lines diffract

white light into a spectrum where wavelength is a function of angle. Table 1 lists the gratings we used labeled by their blaze wavelength along with the region over which they were useful. The "blaze wavelength" corresponds to the wavelength at which the grating has its highest reflectivity. A monochromator is a device that adjusts the orientation of a grating so that only a single wavelength of light passes out of the monochromator. More simply, a monochromator takes white light (broadband) in and outputs monochromatic light, hence the name monochromator.

Table 1 shows that the 0.25 $\mu\text{m}$  grating is the only one that goes below 0.3 $\mu\text{m}$ . Fortunately, this grating also responds well out to 0.7 $\mu\text{m}$  and produces some signal at 0.9 $\mu\text{m}$ . The silicon photo-detector works very well up to about 1 $\mu\text{m}$  since that is where silicon's band gap is. The xenon arc lamp emits light all the way down to  $\sim 175\text{nm}$ , while the tungsten-halogen bulb does not provide light below  $\sim 240\text{nm}$ . Interestingly, but perhaps unfortunately for spectrometry, oxygen in the air begins to absorb at about 200nm and thus this becomes the lower bound of our spectra rather than the xenon lamp's lower limit. Fortunately, the films we studied are already strongly absorbing by this point and thus we never need to go any further.



In understanding that each part of the spectrometer has a different spectrum, we begin to see that a raw transmission or reflection spectrum contains information about the system as well as the sample. Fortunately we don't need to know the spectrum of each component of the system - we simply need the spectrum of the entire system to compare to the spectrum of the sample in the system. Fig. 1 is a sketch of the spectrometer that shows the important elements.



With this new understanding of optical components and the diagram above we can begin to address data collection. But first, we make some reasonable assumptions: 1) The response of the spectrometer components is linear with intensity and 2) neither the intensity of the lamp nor the response functions of the system components change between measurements. Then, the fractional transmission of the sample for a given wavelength is the ratio of the intensity of the light at the detector when the sample is in place to that without the sample. This fractional transmission is called the transmittance,  $T$  of the sample:

$$T(\lambda) = \frac{I_t(\lambda)}{I_{ref}(\lambda)} \quad \text{Eqn. 1a}$$

where  $I_{ref}$  is the intensity without the sample in place,  $I_t$  is the intensity with the sample in place and  $\lambda$  is wavelength. But this equation can also be extended to include the reflectance,  $R$ , so that in general:

$$F(\lambda) = \frac{I_f(\lambda)}{I_{ref}(\lambda)} \quad \text{Eqn. 1b}$$

where  $F$  is either the transmittance,  $T$ , or the reflectance,  $R$ , of the film and  $I_f$  is the corresponding intensity. Since these equations hold for all  $\lambda$ , one merely divides one spectrum by the other to obtain  $T$  or  $R$  as long as the previously stated conditions hold over the wavelength range. The spectrum which results from use of *Eqn. 1* is called a “normalized” spectrum and the process of separating a spectrum of a sample from the spectrum of the system, through the use of *Eqn. 1*, is called normalization.

Two important things should be noted here. 1) When a raw reflection spectrum,  $I_r$ , is taken the detector is moved into the path of the reflected beam. However,  $I_{ref}$  is the same spectrum as in the transmission case. This is because  $R$  is the ratio of the reflected intensity to the incident intensity and  $I_{ref}$  always represents this incident intensity. 2) A thin film sample consists of a film and a substrate. The material of interest exists as the film on the surface of the substrate (generally quartz). Because of this, a spectrum of a sample is a spectrum of the film *and* the substrate, the film cannot be physically separated from the substrate. If the substrate spectrum has structure then this structure will be included in the spectrum of the sample. It is possible to take a spectrum of the substrate and use this as  $I_{ref}$  in *Eqn. 1* if the substrate has large amounts of structure. However, if one thinks of the sample as a series of interfaces of materials than the interface between

the sample and the substrate is not properly accounted for by simply dividing out the spectrum of the substrate. Instead, it turns out, it is better to use a bare lamp spectrum for  $I_{ref}$  in Eqn. 1 and then account for the substrate mathematically. This turns out to be possible because the generally well known optical properties (such as the index of refraction) of the substrate can be used to separate the spectrum of the film from the substrate more accurately than simply normalizing the spectrum of the sample to that of the substrate. I do not go into the details of performing these calculations here, but the formulas can be found in Appendix B and plots of the reflectance and transmittance spectra based on these calculations are included in the index\_calc.xls workbook.

## II.2. UV-Visible Measurements: The Absorption Coefficient and Band Gap

The most important configuration of the spectrometer system is the one for measuring spectra in the UV-Visible region. Transparent conductors have their band gaps in the near-UV (~350nm). Furthermore, the films studied had thicknesses of the order of the wavelength of visible light, so they produce thin film interference fringes in the UV-visible region. Because of these features the UV-visible spectra contain information which can help to determine the size of the band gap and the refractive index - some of the most important properties of the materials. Later we will explore the refractive index, but for now we focus on absorption and the band gap.

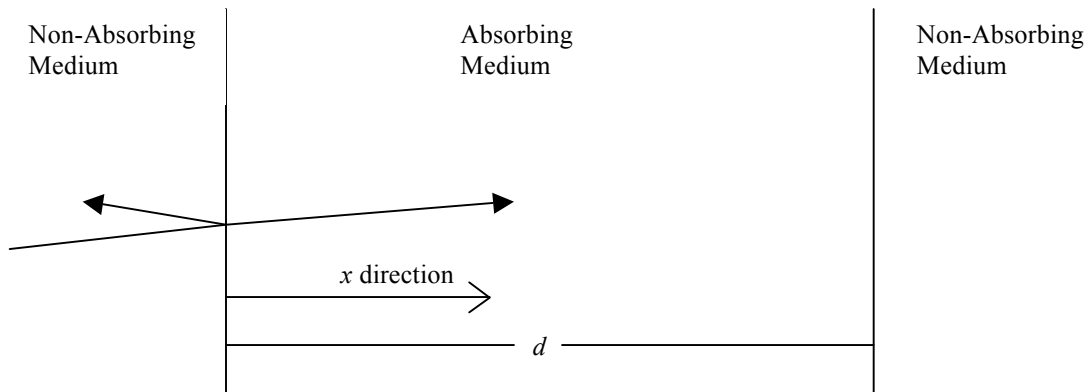
In the previous section we discussed transmittance and reflectance, but absorption also plays a role. For this reason we define an absorptance,  $A$ , as the fraction of light that is absorbed by the sample. If we now assume that this absorption single-handedly covers all intensity loss not accounted for in T and R then we may write the equation:

$$T + R + A = 1 \quad \text{Eqn. 2}$$

This makes determining the absorptance trivial once the transmittance and reflectance are known. Of more interest is the absorption coefficient because it is an intrinsic property of the material, whereas the absorptance depends on the thickness of the film. The absorption coefficient,  $\alpha$ , is a parameter that defines the decay rate of the intensity of light as it passes through a uniformly absorbing medium. It is defined by the equation:

$$I(x) = I(0)e^{-\alpha x} \quad \text{Eqn. 3}$$

where  $I(x)$  is the intensity of the light as a function of  $x$ , and  $I(0)$  is the intensity of the transmitted wave at  $x=0$ . If we now envision light incident on a layer, or film, of material that absorbs light uniformly, and define the origin on the incident boundary with the  $x$  direction perpendicular to this boundary, as in Fig. 2, then we are prepared to determine the absorption coefficient.



**Figure 2:** Ray diagram of electromagnetic field at boundaries of an absorbing medium of thickness  $d$ .

Looking closely at Eqn. 3 and Fig. 2 we recognize that  $I(0)$  is the intensity of the light just after entering the film, and it should therefore not include the reflected light. This means that we may say that:  $I(0) = 1 - R$ . If we then let  $I(x)$  be the intensity at a general point within the film, then for a film of thickness  $d$ ,  $I(x)$  becomes  $I(d)$  at the back

boundary. However, the intensity at the back boundary of the film is just the transmitted intensity, and so we see that:  $I(x) \rightarrow I(d) = T$ . Using these new definitions, and rearranging *Eqn. 3*, we may write [Ref. 3]:

$$\frac{I(x)}{I(0)} \rightarrow \frac{I(d)}{I(0)} = \frac{T}{1-R} = e^{-\alpha d} \quad \text{Eqn. 4a}$$

One can then solve the last equality to obtain an expression for the absorption coefficient in terms of experimental quantities:

$$\alpha = \frac{-\ln\left(\frac{T}{1-R}\right)}{d} \quad \text{Eqn. 4b}$$

It is important to understand that both  $T$  and  $R$  are functions of wavelength, and thus so is the absorption coefficient. This is as we expect because the magnitude of the absorption coefficient should increase in regions of high absorption and decrease in regions where the absorbance is low. In particular, a sharp decrease in absorbance marks the band gap. As a follow up to *Eqn. 2* we can use *Eqn. 4a* and *Eqn. 2* to rewrite the absorbance in terms of the absorption coefficient:

$$A = (1-R)(1 - e^{-\alpha d}) \quad \text{Eqn. 5}$$

For many purposes, *Eqn. 4* works well for determining the absorption coefficient. However, it is not exactly correct because it does not take multiple reflections within the film into account. Each reflected wave will be attenuated according to the physical absorption coefficient. The above calculation for the absorption coefficient assumes all of this attenuation occurs in one pass through the material (in one distance  $d$ ). Because of this, the absorption coefficient as calculated above will be too high. There are many

different ways to correct for this error, but the good ones require one to know the index of the film. Because we are able to calculate the index of the film, as discussed later, this is possible but is not discussed in this document.

As can be seen above, the calculations involved in these measurements are relatively trivial. However, it is important to understand that these calculations are a limit of more complex equations that treat the system correctly. For a more precise analysis of the absorption coefficient see Ref. [3].

One thing that I learned while doing my research is that it can be difficult to be sure how accurate a data set actually is. The first, and perhaps foremost, complication that affects the accuracy of our data is the fact that the analysis assumes the light will be transmitted directly through or reflected specularly from the sample. The experimental reality is that some of the light will be scattered. Since the detector is a large distance from the sample, much of this scattered light will not strike the detector and will become a part of neither  $T$  nor  $R$ . Under the assumptions of *Eqn. 2*, this light will be attributed to absorption and the end result of this error is an artificially high absorptance,  $A$ , and absorption coefficient,  $\alpha$ .

Furthermore, according to Lord Rayleigh, scattering occurs more strongly at lower wavelengths. The wavelength dependence of this scattering means that not only will the magnitude of our spectra be affected, but their shapes could be as well! Fortunately this problem can be addressed. A large-area detector placed as close to the sample as possible will capture most scattered light. This method works well for transmission, but for reflection the detector cannot be too close to the sample without

blocking the incident light. Another solution is to use an integrating sphere. This method is outlined in the next section.

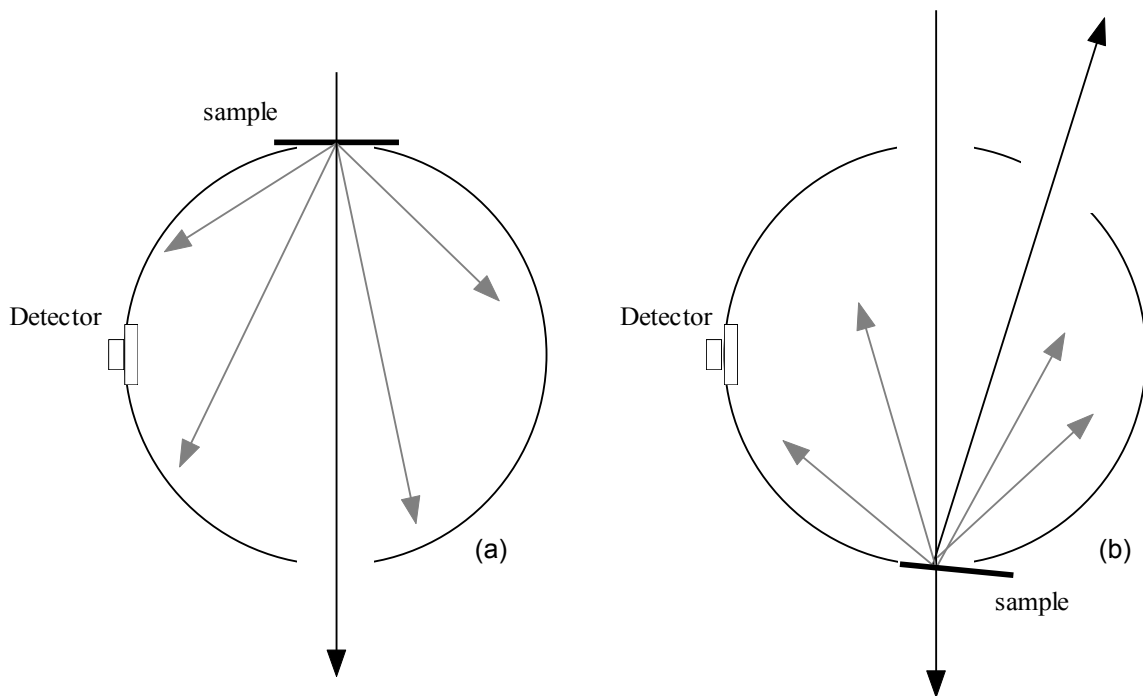
### III. The Integrating Sphere

An integrating sphere collects all of the light from a sample (regardless of the sample's scattering properties) and directs it onto a detector. Our sphere, an Oriel 70491, has an 8 in. diameter and an inner surface coated with a rough layer of barium sulfate ( $\text{BaSO}_4$ ), a material that is strongly reflecting from the near UV through the visible spectrum and into the near IR. The rough surface reflects light diffusely, which means that reflected light is scattered into a wide angle rather than in a single direction. After several bounces in the sphere, light is uniformly distributed. This section begins with the discussion of the loss of scattered light problem brought up at the end of the previous section, but quickly moves into the issue of normalizing a spectrum taken with the integrating sphere. Various configurations of samples in the sphere, and their usefulness, are discussed including powder and film samples. Near the end of the section a theory of the absorption coefficient of powdered samples is discussed, analyzed and applied.

#### III.1. Diffuse Reflection and the Integrating Sphere

Perhaps the best way to determine how much error is due to scattered light when using the spectrometer in the standard (no sphere) configuration is to simply measure the intensity of the scattered light. The intensity of the scattered light can be measured by allowing the direct transmitted and specularly reflected light to pass out of the integrating sphere while capturing the scattered light (Fig. 3). Besides indicating how much error is introduced when not using the sphere, scattering also indicates how uniform a film is – an

amorphous film is likely to scatter more strongly than a uniform one. D. Tucker used scattered light spectra to show the  $1/\lambda^4$  dependence of Rayleigh scattering from the grains in the film [Ref. 3]. Once the scattered light has been measured this information can be used to determine whether more precise measurements are required, whether a spectrum can be corrected, or – in the case of very little scattering – whether the previously obtained data is accurate enough.

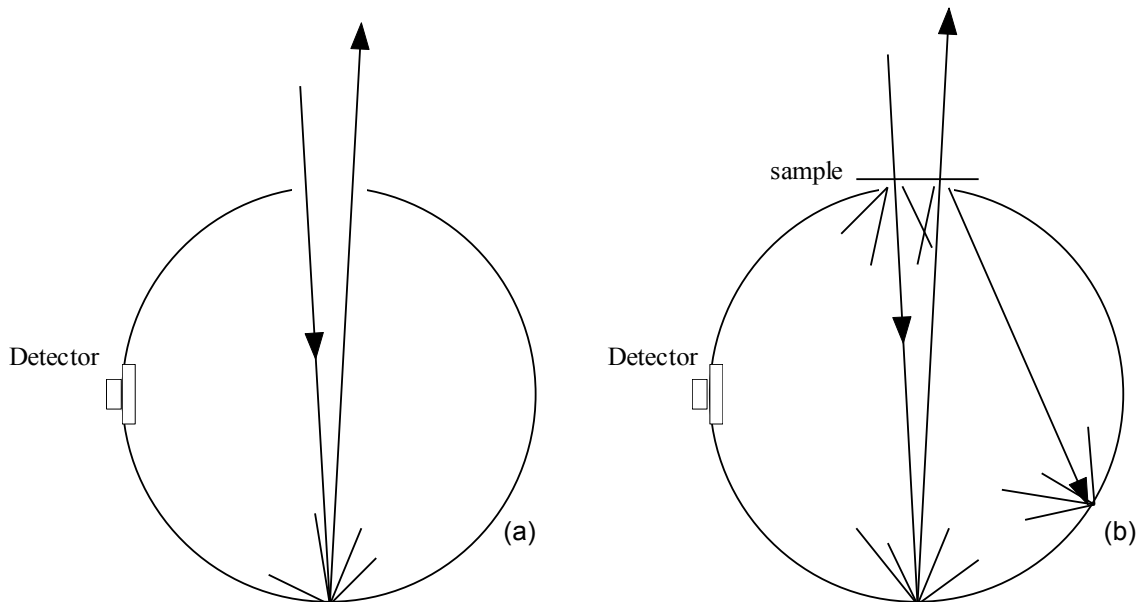


**Figure 3:** Configurations for gathering only the scattered light (grey arrows) while allowing the direct transmitted and specularly reflected light (black arrows) to pass out of the sphere. a) Collect the light that is “transmission-scattered” from the film. b) Collect the light that is “reflection-scattered” from the film.

If scattered light measurements indicate that more precise measurements are required, the integrating sphere can be used to capture all of the light – scattered and direct/specular – as in Fig. 4. The trouble with the sphere is that proper normalization becomes difficult. The most obvious source of error when using the sphere, but not necessarily the most significant, comes from the requirement that an additional plug, the



plug that allows transmitted light to pass out of the sphere, be removed in order to obtain a reflection spectrum. Furthermore, though the sphere is designed to distribute the light evenly, there remains the “first bounce” problem. This is the complication wherein a disproportionate amount of the light is reflected specularly from the point at which it initially comes in contact with the sphere, rather than being reflected evenly in all directions. The simplest case of this becoming a problem is when a reference spectrum is taken off of the bottom of the sphere, and then a sample is placed over the hole for a transmission spectrum as in Fig. 4.



**Figure 4:** a) Diagram showing the “reference spectrum” or “lamp spectrum” configuration of the integrating sphere. Because the sphere surface does not scatter light uniformly, the specularly reflected ray (shown) has a higher intensity than scattered rays. b) Diagram with a sample in place for a transmission measurement that gathers all of the light. Notice that the film could be “trapping” additional light that was not included in the reference spectrum. This means that the transmittance as measured in this configuration could, theoretically, be higher than 1! Of course we know, from conservation of energy, that this is an error of the measurement, rather than a property of the material.

Fig. 4 makes it apparent that when the sample is placed over the hole, additional light (light that was *not* trapped in the reference spectrum) is trapped inside the sphere because the sample reflects some of the light back. Admittedly, the magnitude of this problem is probably very small since the amount of light reflected off of the back of the

sample is small, but analyzing this problem is instructive in understanding some of the nuances of the integrating sphere.

I believe that if care is taken these problems can be overcome rather easily to make the sphere a more precise tool. Unfortunately I ran out of time and was unable to properly characterize the sphere. Partly for this reason, and also because the sphere is less convenient, more than 90% of my data is taken not using the sphere.

### III.2. Powder Measurements and the Integrating Sphere

Generally we use the integrating sphere to measure the diffuse reflectance of powders. The diffuse reflectance of a powder has many qualitative features in common with the transmittance of a more homogeneous sample of the same material. For a homogeneous sample, light is specularly reflected at the smooth surface, and all other light is transmitted (or absorbed), and one simply picks which portion to measure. The powdered sample, on the other hand, consists of roughly packed crystallites and air pockets. Light reflected at the rough interface can make its way via refraction into the body of the powder and exit the surface by the same mechanism. This non-trivial path involves transmission through some crystallites and therefore the diffuse "reflection" spectrum contains characteristics of the transmission spectrum of the homogeneous sample.

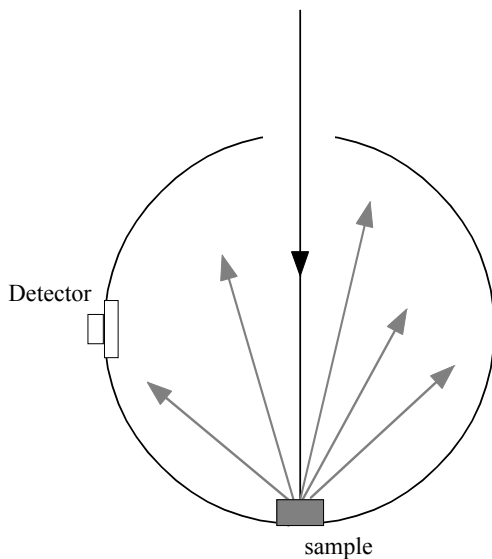
Fig. 5 gives a diagram of the powdered sample configuration of the integrating sphere. With powdered samples we assume that light is either reflected from the sample

or it is absorbed by it<sup>\*</sup>. Keeping this in mind we recognize that the fraction of light incident on the sample that is not reflected by it must be absorbed by it. In other words, if we define the reflectance of the powder like we did for films in *Eqn. 1b*:

$$R(\lambda) = \frac{I_R(\lambda)}{I_{ref}(\lambda)} \quad \text{Eqn. 6}$$

Then the absorptance becomes:

$$A = 1 - R \quad \text{Eqn. 7}$$



**Figure 5:** Integrating sphere with a powder sample. The sphere collects the light scattered from the sample and it eventually ends up on the detector.

This is merely a restatement of *Eqn. 2* without a transmittance term. Unfortunately, the distance  $x$  in *Eqn. 3* has little meaning in a powder measurement. In theory  $x$  would be the path length of the light in the powder. However, this distance can be very difficult to determine because it is complicated by air pockets within the powder and scattering from each

particle. Fortunately, a theory has been developed by Kubelka and Munk [Ref. 4] that treats diffuse reflectance from powders. The primary assumptions of this theory are that the powder is weakly absorbing and that each particle of the powder scatters uniformly in all directions. Though the former assumption is not always accurate in our experiments

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\* It is possible to obtain a transmission spectrum of a powdered sample if the powder is in a transparent container or on a glass slide but since diffuse reflectance is already so similar to transmission this does not necessarily contribute that much additional information and is therefore not usually necessary.

(the band-gap corresponds to the onset of high absorption) the Kubelka-Munk theory is still very useful because it gives a qualitative picture of the absorption coefficient rather easily. This qualitative picture is known as the remission function,  $f(R)$ , and is defined as:

$$f(R) = \frac{(1-R)^2}{2 \cdot R} = \frac{k'}{s} \quad \text{Eqn. 8}$$

Where  $k'$  and  $s$  are parameters from the Kubelka-Munk theory which are related to the absorption coefficient and the average particle diameter respectively. Later, Simmons published a paper in which he posited a theory for the following relationships [Ref. 5]:

$$\begin{aligned} s &= l^{-1} \\ k' &= \frac{2}{3} \cdot k \cdot n^2 \end{aligned} \quad \text{Eqn. 9}$$

Where  $n$  is the real part of the index of refraction of the powdered material,  $l$  is the average particle diameter, and  $k$  is the extinction coefficient (the imaginary part of the complex index) which is related to the absorption coefficient by the equation:

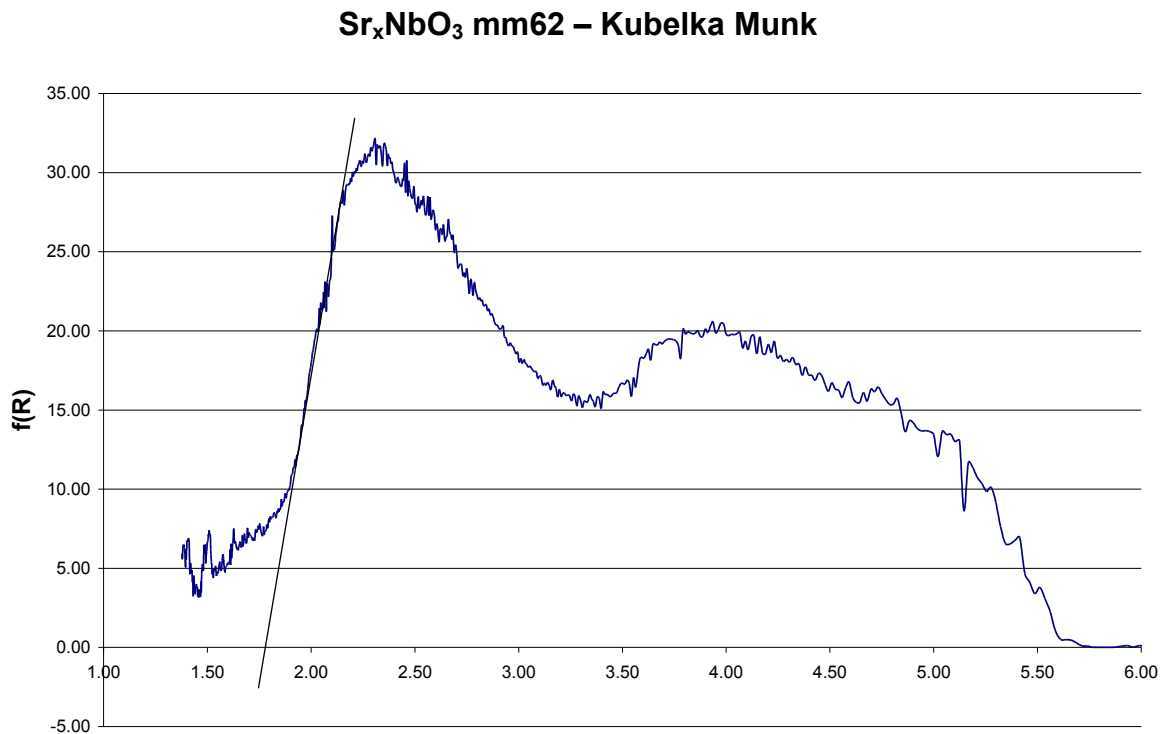
$k = \alpha \cdot \lambda / (4\pi)$ . In Simmons' theory,  $s$  is precisely the inverse of the particle diameter, and  $k'$  is proportional to the extinction coefficient provided  $n$  is constant over the range of interest. Combining Eqn. 8 and Eqn. 9, we get:

$$f(R) = \frac{(1-R)^2}{2 \cdot R} = \frac{2}{3} \cdot k \cdot l \cdot n^2 \quad \text{Eqn. 10}$$

Simmons proposed that diffuse reflectance measurements could be used to determine either  $l$ ,  $k$ , or  $n$ , provided the other two were known or determined elsewhere but we do not generally know any of these parameters for powder films. However, even though  $n$  is not constant in the region of the gap the remission function still looks similar to the

absorption coefficient, and it seems to be useful in determining an approximate value for the band-gap of the material.

Some powder samples from Reed College were brought down by two undergraduate students and analyzed using our spectrometer. Though the powders were strongly absorbing (and hence it would seem should not qualify for Kubelka-Munk analysis), the students had been using this theory and it did seem that it was possible to obtain an approximate value of the gap. In Fig. 6 we recognize that the sharp rise at  $\sim 2\text{eV}$  corresponds to the onset of absorption due to the band gap.



**Figure 6:** The remission function of a  $\text{Sr}_x\text{NbO}_3$  powdered sample named mm62 that was made at Reed College. Note the sharp rise at  $\sim 2\text{eV}$  which corresponds to the band-gap.

A linear extrapolation applied in Fig. 6 yields an estimate of the band gap of  $1.75 \pm 0.25$  eV, where I have included a wide margin of error due to the hand-waving above.

Because the majority of the data I took was on films, the Kubelka-Munk analysis was the only non-trivial analysis I performed on powders. I have included this section primarily

as an overview of the usefulness and limitations of the Kubelka-Munk analysis in the event that it should be used in the future. I think this analysis could be a useful tool and should be explored more thoroughly.

With powders, as was the case when using the sphere with films, normalization is once again an issue. The specifics are, however, somewhat different. The problem comes from the question: what is  $I_{ref}$  in Eqn. 6? In general  $I_{ref}$  is the amount of light incident on the powder. However, because we are using the sphere,  $I_{ref}$  must be taken in such a way as to include the spectrum of the sphere as well. This in turn means that a reference material must be positioned at the bottom of the sphere to scatter the incident light in approximately the same way that the sample will. However this reference material must have a reflectance very close to 1. The reason for this is understood more clearly when one understands that  $I_{ref} = R_{ref} \cdot I_{lamp}$ . Where  $R_{ref}$  is the reflectance of the reference material and  $I_{lamp}$  is the spectrum of the rest of the system. So that Eqn. 6 becomes:

$$R(\lambda) = \frac{I_R(\lambda)}{R_{ref}(\lambda) \cdot I_{lamp}(\lambda)} \quad \text{Eqn. 11}$$

where again,  $I_R$  is the reflection spectrum intensity of the sample, and  $R$  is the reflectance of the sample. As long as  $R_{ref} = 1$ , then  $I_{lamp} = I_{ref}$  and  $R$  is the spectrum of the sample relative to the spectrum of the system. However, for  $R_{ref} < 1$  (it cannot be greater than one),  $R$ , as measured in this experiment, will be greater than the “true” reflectance of the powder. This means that in order to have a good diffuse reflectance spectrum of a sample powder, a reference powder must be found with a diffuse reflectance spectrum,

$R_{ref}$ , equal to 1 over the region of interest. This, in turn, means that the reference powder must have zero, or at least very little, absorption in the region of interest.

For the UV-Visible region either a BaSO<sub>4</sub> plug or MgO powder was placed in position as the reference material. Partly because it was thought that the MgO scattering properties were more similar to those of the sample (because MgO and the sample are both powders), and partly because some of the people using the system were unaware of the BaSO<sub>4</sub> plug, the MgO powder has been used more often than the BaSO<sub>4</sub> plug. In spite of these mostly historical considerations, the BaSO<sub>4</sub> plug has the advantage of being the same material as the lining of the sphere and thus contributes no additional structure to the spectrum. Because of this I believe that the BaSO<sub>4</sub> plug should be used instead of the MgO powder. Occasionally, the MgO powder was mixed with the sample or used as a backdrop for the sample when the sample is either too dark or too small. In these cases it may be more accurate to use the MgO powder as the reference material.

#### IV. Infrared Measurements and Wavelength Sorting Filters

The most significant addition I made to the spectrometer system was the extension of its range into the near infrared. For the UV and visible measurements, a silicon detector was used. However, due to silicon's band gap of approximately 1.14 eV (~1090nm), it has a poor response beyond approximately 900nm, and none at 1100nm. Though the range from 650-900nm does represent a tiny piece of the IR region, it is not enough to be considered significant. In order to extend this IR range, a lead-sulfide detector was purchased. Lead-sulfide has a band gap of approximately 0.41eV (~3.0 $\mu$ m), and we were thus able to extend our analysis out to approximately 2.6 $\mu$ m. This limit

(rather than the 3.0  $\mu\text{m}$  of the detector) is primarily due to the fact that quartz lenses absorb strongly at this wavelength. The short wavelength limit arises from a sharp rise in noise level below 800 nm. Furthermore, the noise level of this PbS detector is “significantly increased by exposure to UV or blue light”, this noise level “usually recovers in the dark, but this can take many hours” [Ref. 6]. In order to keep this from happening every time the detector is exposed to room light, a low-pass (low frequency, long wavelength) filter was fastened to the front of the detector that blocks light below  $\sim 0.8\mu\text{m}$ .

$2 \cdot d \cdot \sin(\theta) = m \cdot \lambda = 600\text{nm}$	
$m$	$\lambda$
1	600
2	300
3	200

**Table 2:** A table of values of  $m$  and  $\lambda$  which all satisfy Eqn. 12 for a single value of  $d$  and  $\theta$ . This means that for a given grating (given value of  $d$ ) multiple wavelengths will diffract at the same angle (a single value of  $\theta$ ). Since  $\theta$  is the variable that the monochromator adjusts in order to change the wavelength,  $\lambda$ , we obtain multiple values of  $\lambda$  at one monochromator setting of  $\theta$ .

Unfortunately, when we began taking these IR measurements we did not have a single grating that worked for the region from 0.8 to 2.6 $\mu\text{m}$ . From table 1, one can see that both the 1.0 $\mu\text{m}$  and 2.0 $\mu\text{m}$  gratings were necessary. This in turn meant that two entire sets of data needed to be taken in order to span this range. Later, an

additional grating was purchased with the hope that it would be capable of spanning this entire region alone. However, even though this grating was rated to span this range, I was unable to get enough signal at the long wavelength end (beyond  $\sim 2.4\mu\text{m}$ ) to obtain decent spectra. As a result, in order to maximize the range and minimize the noise, two gratings must be used. Fortunately, there are still several things that can be tried in order to get one grating to work for the entire IR range (see Appendix C).

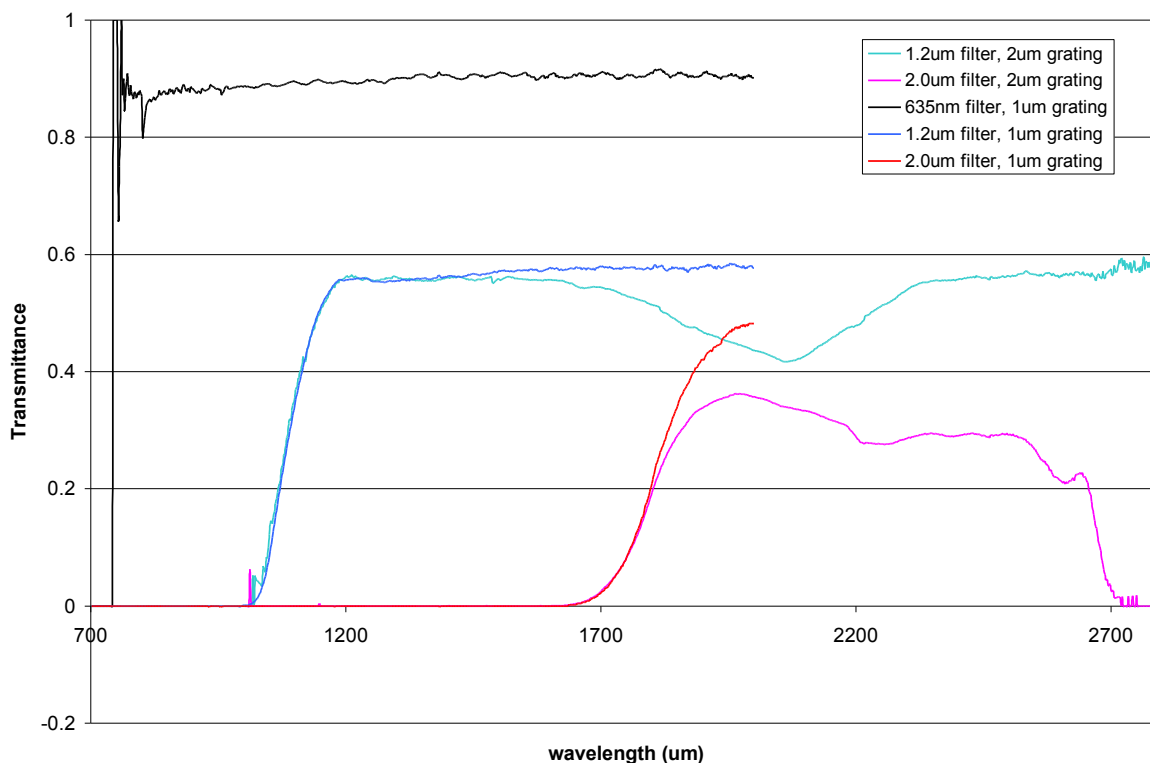


Another issue, which existed in the UV-visible region, but becomes more of a factor in the IR is that of higher order wavelengths at the monochromator output. This is a direct result of grating physics. The grating equation is:

$$m \cdot \lambda = 2 \cdot d \cdot \sin(\theta) \qquad \text{Eqn. 12}$$

where  $\lambda$  is the wavelength,  $d$  is the grating spacing,  $\theta$  is the angle from the grating normal, and  $m$  is an integer that denotes the order of the light. Unfortunately, a problem becomes apparent when one understands that in a simple diffraction grating the maximum intensity occurs at  $\theta=0$  where, by the grating equation,  $m=0$  and  $\lambda$  is arbitrary. In other words the point of greatest intensity occurs where all wavelengths are piled on top of one another (white light). This is not at all useful for spectrometry and so gratings are “blazed” in order to shift the intensity peak onto the first order,  $m=1$ . However, the grating equation still holds for higher orders. For a given value of  $\theta$ , ( $d$  is of course fixed) the right hand side of *Eqn. 12* is fixed but there are still multiple values of  $m$  and  $\lambda$  that satisfy the equation as illustrated by table 2. From this, one can see that at a given angle,  $\theta$ , multiple wavelengths of light are transmitted by the monochromator. This is undesirable in spectrometry and so filters are used to block the higher order, lower wavelength, light. This is less of an issue in the UV-visible region because the lamp intensity is so low at the second and third order wavelengths. In the IR region, however, the lamp intensity of the second order light is of comparable magnitude to the first order light and can thus alter a spectrum significantly.

## Various Wavelength Sorting Filters Normalized to Lamp



**Figure 7:** Spectrum of various filters normalized to bare lamp spectrum. Notice the structure in the spectrum of the filters when using the 2 $\mu$ m grating. This arises because the 2 $\mu$ m grating has second order light in this region. In order to obtain a proper spectrum of these filters one should take their spectra using a spectrometer which uses filters in it already so that second order light does not affect the spectrum of the filters. These spectra have been shown to help demonstrate the need for the filters.

From Fig. 7 it becomes obvious that filters are labeled by the shortest wavelength at which the transmission of that filter reaches its maximum value. In the case of the 635nm filter this maximum value is about 90%, but in the 1.2 and 2.0  $\mu$ m filters it is more like 55% and 50% respectively. Closer examination shows that the longest wavelength at which a filter has zero transmittance occurs at half the wavelength at which the next filter has its maximum transmittance. For example, the 1.2  $\mu$ m filter, which has a maximum transmittance at 1.2  $\mu$ m, has no transmittance below 1.0  $\mu$ m so that no second order light sneaks into the spectrum when one scans, at say 1.95  $\mu$ m, just before the 2.0  $\mu$ m filter is put in place. Based on these arguments, it becomes apparent that each filter should be swapped at the wavelength at which that filter is labeled. This ensures

that the signal is maximized in order to produce clean data while still blocking all second order light.

Furthermore, one notices structure in the 2  $\mu\text{m}$  grating spectra above 1.5  $\mu\text{m}$  that does not exist in the 1  $\mu\text{m}$  grating spectra. This structure indicates the existence of the 2<sup>nd</sup> order light that we theorized earlier. When a bare lamp spectrum is taken using the 2  $\mu\text{m}$  gratings (no filters in place), the second order light strikes the detector and creates an abnormally high reference spectrum. Then when a spectrum is taken with a filter in place, this second order light is blocked. When one divides the filter spectrum, by the lamp spectrum the result is a spectrum that is not only the spectrum of the filter, but a convolution of the filter and the inverse of the spectrum of the second order light!

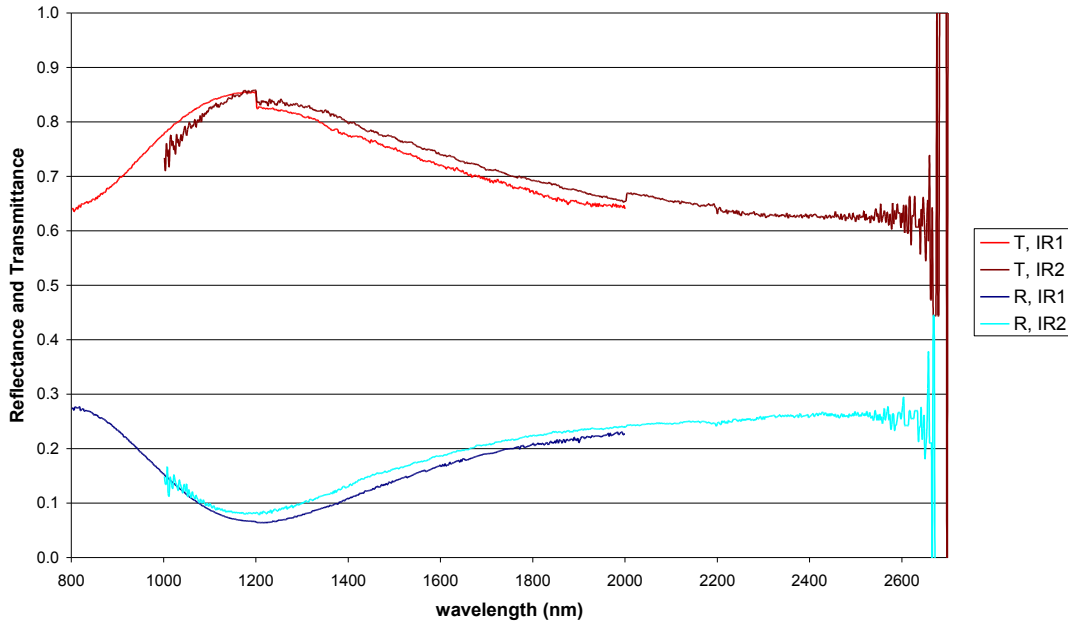
So now the question becomes: why don't the spectra of the filters using the 1  $\mu\text{m}$  grating have this same structure? The answer is simple: the 1  $\mu\text{m}$  grating does not have any second order light in this wavelength range. Recall that each component of the spectrometer system has its own spectrum. Well, this rule goes not only for the 1<sup>st</sup> order light from a grating, but for the 2<sup>nd</sup> order light as well. In our case, the 2  $\mu\text{m}$  grating happens to have second order light from  $\sim 1600\text{nm}$  to at least  $2700\text{nm}$  (of course this light is actually of wavelength  $800\text{--}1350\text{nm}$ ), while the 1  $\mu\text{m}$  grating has no second order light in this range.

Upon closer examination of the spectra, one notices that when using the 2  $\mu\text{m}$  grating and 1.2  $\mu\text{m}$  filter the effects of the second order light begin to diminish at about 2.0  $\mu\text{m}$ . This makes sense because this corresponds to the point at which the 1.2  $\mu\text{m}$  filter begins to let the second order light pass through again. On the other hand, the 2.0  $\mu\text{m}$  filter blocks all of the second order light to nearly 3.4  $\mu\text{m}$  and so we see the inverse

spectrum of the second order light continue on the spectrum of the 2.0  $\mu\text{m}$  filter. Finally, one notices that the spectrum of the 2.0  $\mu\text{m}$  filter goes to zero at 2.7  $\mu\text{m}$ , rather than becoming noisy as one would expect when one has a 0/0 data set. This is because the lamp signal without the filters in place is not zero due to second order light, while the filtered spectrum is.

A set of data taken using the IR detector is shown in Fig. 8. The reflectance of this film was taken to be approximately 20% because in a bulk sample the interference fringe would not exist. The transmittance is taken to be approximately 70% if the fringe did not exist, however if absorption were a factor in these region for this material, 70% transmittance would not apply to a bulk sample.

### sm16d $\text{CuScO}_2$ - IR



**Figure 8:** Transmittance and reflectance of the  $\text{CuScO}_2$  film sm16d in the IR Region from 800 to 2600nm. The bump and dip in the transmission and reflection spectra respectively is the first interference fringe. The noise at 2600nm represents the limit of the range of this system.

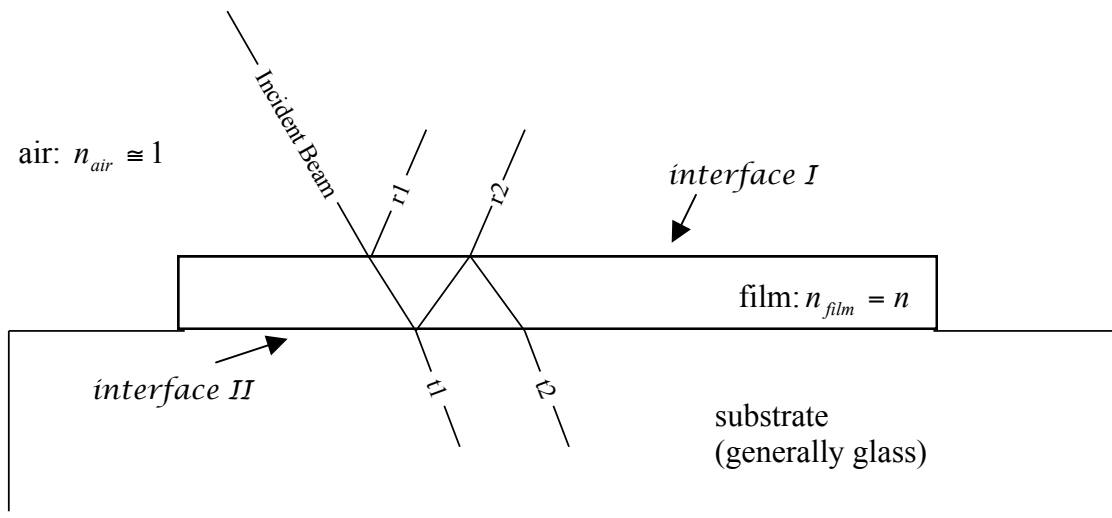
Notice, in Fig. 8, that the two spectra that are taken do not line up particularly well. This is a problem to which I was unable to find an answer. Also notice the discontinuity in the transmittance spectrum at 1200nm and 2000nm. This is where the filters were changed, but it should not be there if the spectra are properly normalized. Both of these problems *could* be due to nonlinearities in either the lock-in or the PbS detector but I am not sure. Whatever the cause, these problem should be solved if the IR detector is going to be considered a precision device.

Other additions were necessary for the IR spectra, including the addition of a chopper wheel, a chopper wheel interface panel, a lock-in and a power-supply. The chopper wheel was necessary because the lead-sulfide detector has no DC functionality, instead it utilizes AC input and output signals. The lock-in was then necessary to obtain the amplitude of this AC signal from the detector and make it a DC signal for the computer software. The rest of the components surrounding IR measurements, and how to use them, are outlined in the spectrometer.doc file which is included with this document as Appendix A.

## V. Refractive Index

In this section, the theoretical basis for determining the index of refraction from thin film interference fringes is introduced but details are left to referenced publications. Instead, this section focuses on the details of obtaining the index of refraction from a thin film's reflectance and transmittance spectra. The first few pages of this section are consumed with a seemingly trivial question. Is the index of the film higher or lower than the index of the substrate, which is generally quartz? In the end we find that the index of

the films we study is higher than that of the glass, as we probably could have guessed, but proving it is necessary when your sources of information are limited. Once we have determined this, we are able to obtain data points for the index of refraction at the wavelength of the maxima and minima. From here on it is a matter of finding a functional form for the index and fitting its parameters to match the data points. Fortunately, the Sellmeier equation is widely accepted and accurate, so we use it, with reasonable approximations, to obtain a functional form of the index of refraction. Along the way we will need a way to determine the thickness,  $d$ , but fortunately we will find a method for doing this within a reasonable margin of error (about 20%). Also, throughout this section the analysis is developed assuming that one does not have an IR spectrum as in Fig. 8 of the previous section. Though having an IR spectrum would in some ways make this analysis easier, it is neglected to show that the index of these thin films can be determined based on UV-visible spectra alone.



**Figure 9:** Ray diagram of the first few reflections in a thin film – other reflections have been excluded for simplicity. The back boundary of the substrate is not shown because the substrate is assumed to be very thick (much thicker than the coherence length of the light). When  $r1$  and  $r2$  interfere constructively a maximum occurs in the reflection spectrum.

A thin film is defined here as a layer of material whose thickness is of the order of the wavelength of visible light ( $\sim 0.5 \mu\text{m}$ ). Because of this property, interference fringes appear in the visible spectrum of thin films. In this section a method is described for determining the index of refraction of the film based on the location of its thin film interference fringe maxima and minima. Taking a look at Fig. 9, we see that when  $r_1$ , light reflected from the first surface of the film, interferes destructively with  $r_2$ , light reflected from the second surface, a minimum results in the reflection spectrum, and of course a maximum in the transmission spectrum.

Conversely, when  $r_1$  interferes constructively with  $r_2$  we get a maximum in the reflectance spectrum, and a minimum in the transmittance. When the total phase difference between two waves at a given point is an odd multiple of  $\pi$ , destructive interference occurs, and when the total phase difference is an even multiple of  $\pi$  constructive interference occurs. This means that determining a theoretical phase difference between two waves can lead one to find an equation for the location of the interference fringes. The derivation of such an equation can be found in introductory optics texts [Ref. 7] and is given here as\*:

$$\lambda_{\text{max,min}} = \frac{2 \cdot n \cdot d}{m} \qquad m = 1, \frac{3}{2}, 2, \frac{5}{2}, 3, \frac{7}{2} \dots \qquad \text{Eqn. 13}$$

$d = \text{film thickness}$

But which  $m$ 's correspond to a maximum and which to a minimum? The answer depends upon the relative indices of refraction between the substrate and the sample.

When a wave is incident on a material whose index is higher than the index in which the wave is propagating, a  $\pi$  phase change occurs between the incident and reflected waves.

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\* This equation assumes normal incidence. This is a valid assumption for small incidence angles, and is reasonable for our incidence angle of  $\sim 15^\circ$ .

This phase change does not occur when a wave is incident on a material with a lower index.

Noting that the index of any solid is higher than that of air, we recognize that a  $\pi$  phase change occurs at *interface I*. So now we need to know whether the index of the film is higher or lower than that of the substrate – generally quartz – in order to determine whether a phase change occurs at *interface II*, but the index of the film is exactly what we are trying to determine in the first place! So in order to find the index of the film, it would appear that we would need to know it beforehand!

Because there are only two possibilities for the relative indices at *interface II* (i.e.  $n_{film} > n_{glass}$  or  $n_{film} < n_{glass}$ ) the best way to solve this problem is to try each and see which one produces a reasonable result. The optical path length difference between  $r_1$  and  $r_2$  is  $\Lambda = 2 \cdot d \cdot n$ , and therefore the total phase difference between  $r_2$  and  $r_1$  is (Ref [6]):

$$\delta = \frac{4 \cdot \pi \cdot n \cdot d}{\lambda} - \delta_I + \delta_{II} \quad \text{Eqn. 14}$$

where  $\delta_I$  is the phase change at *interface I* and  $\delta_{II}$  is the phase change at *interface II*.

We know that  $\delta_I = \pi$ , but we do not know  $\delta_{II}$ . Now, we know that when the total phase difference between  $r_1$  and  $r_2$  is an integer multiple of  $2\pi$  we get a maximum in the reflection spectra, and when it is an odd multiple of  $\pi$  we get a minimum, so we have:

$$\begin{aligned} \delta_{R-\max} &= 2 \cdot \pi \cdot m' \\ \delta_{R-\min} &= 2 \cdot \pi \cdot (m' - \frac{1}{2}) \end{aligned} \quad m' = 1, 2, 3, 4 \dots \quad \text{Eqn. 15}$$

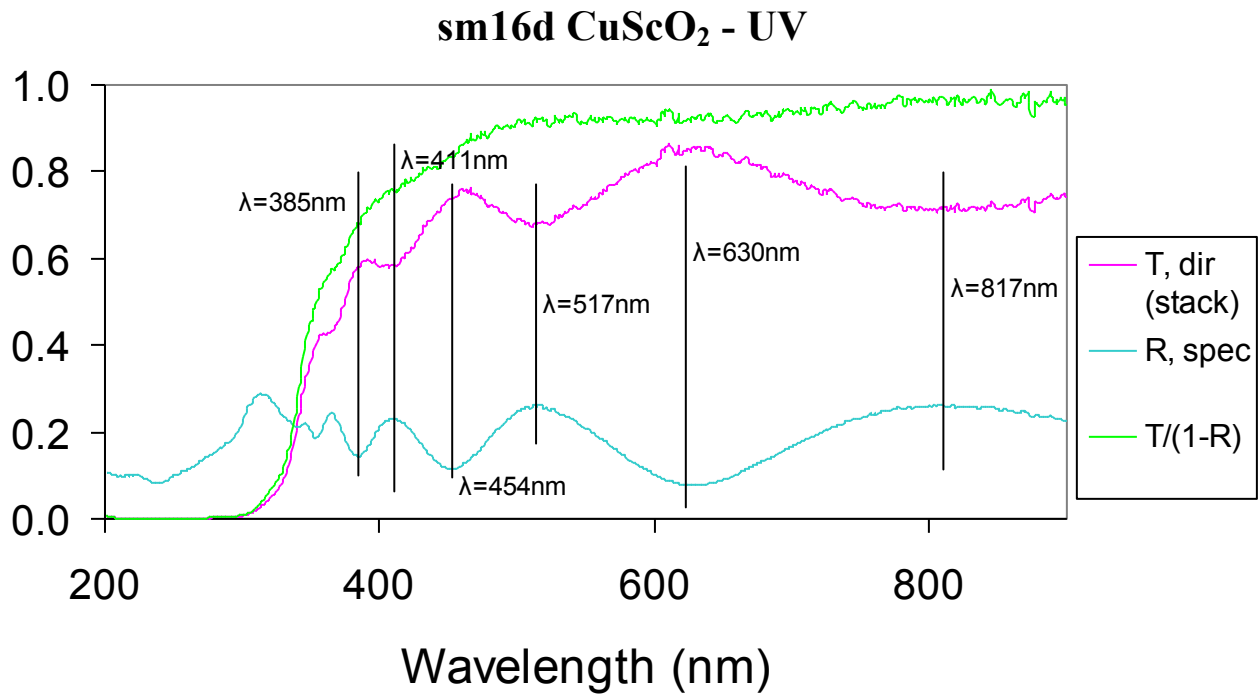
Setting *Eqn. 15* equal to *Eqn. 14* and solving for the index we get:



$$n_{R-\max} = \frac{\lambda_{R-\max} (m' + \frac{1}{2} - \delta_{II} / 2\pi)}{2 \cdot d} \quad m' = 1, 2, 3, 4 \dots \quad \text{Eqn. 16}$$

$$n_{R-\min} = \frac{\lambda_{R-\min} (m' - \delta_{II} / 2\pi)}{2 \cdot d}$$

where the *R-max* and *R-min* subscripts denote maxima or minima, respectively, in the reflectance spectrum. Now we look to the optical spectra to determine the locations of the fringes, as demonstrated in Fig. 10, and create a table of these values, as in column 3 of table 3.

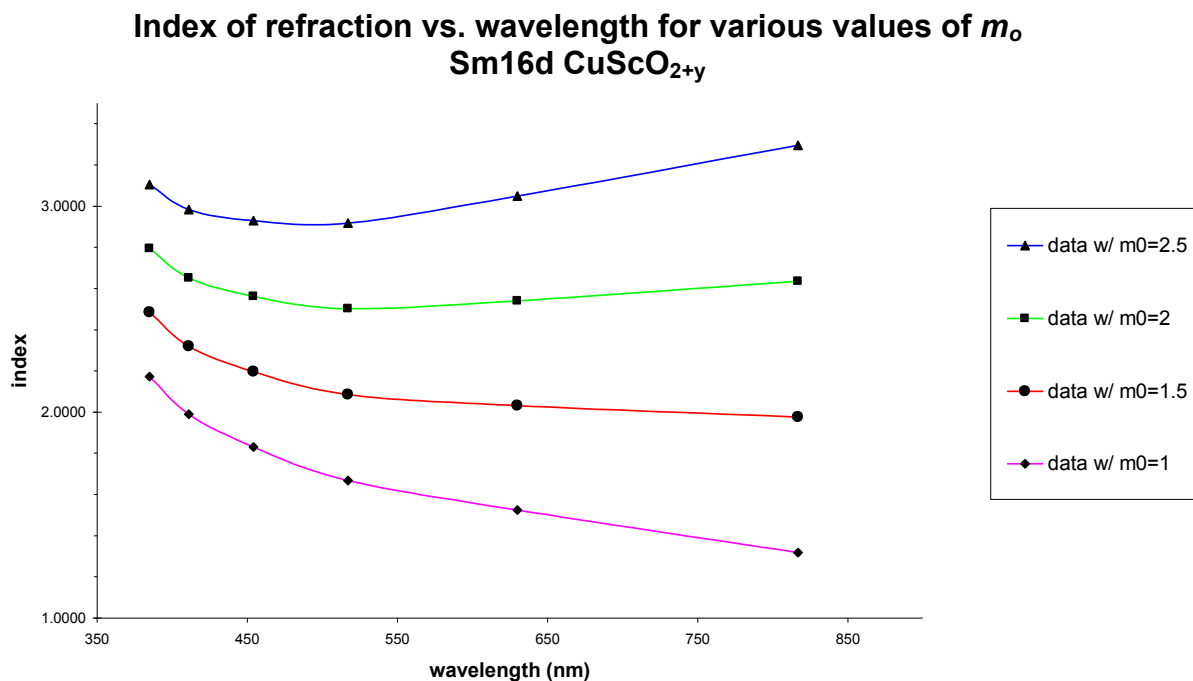


**Figure 10:** Typical transmission, reflection and T/(1-R) spectra of a CuScO<sub>2+y</sub> film (named sm16d). Note that the band gap is below the visible region and that the film is approximately 70% transmitting in the visible region. The maxima and minima of the interference fringes are labeled. These numbers are used to help calculate the index of the film.

Solving Eqn. 13 for *n* and changing the *counting* index (not the *refractive* index) from *m* to *j* we get:

$$n_{\max,\min} = \frac{(m_0 + \frac{j}{2}) \cdot \lambda_{\max,\min}}{2 \cdot d} \quad j = 0, 1, 2, 3, 4 \dots \quad \text{Eqn. 17}$$

Under this indexing scheme,  $m_o$  is a guess at the order ( $m$  value) for the first (highest wavelength) fringe, in Fig. 10 this is the fringe at 817nm.  $j=1,2,3,4\dots$  correspond to the maxima or minima at successively lower wavelengths. We now guess a few values for  $m_o$  and use this equation to generate data points of  $n$  at all values of  $j$  for which we have a maximum or minimum. Table 3 is an example of such a calculation with  $m_o=1.5$ . We then plot  $n$  vs.  $\lambda$  for different values of  $m_o$ , as in Fig. 11 and look to see which set of data is physically reasonable.



**Figure 11:** Plots of the index of refraction for various values of  $m_o$ . It is important to understand that these are only guesses. Only one, if any, of these data sets corresponds to the actual values of the refractive index; the others have no physical significance.

From Fig. 11 it is clear that  $m_o = 2$  and  $2.5$  are wrong because the index begins to increase with increasing wavelength which disobeys most common dispersion relations\*.

We see that  $m_o = 1$  could be good, but it seems to be approaching  $n = 1$ . It looks as though  $m_o = 1.5$  is the correct curve, but let's make sure. Setting Eqn. 17 equal to Eqn.

16, we get a relationship between  $m_o, j, m'$ , and  $\delta_{II}$ :

$$\begin{aligned} m_o + j_{R-\max}/2 &= m'+1/2 - \delta_{II} / 2\pi \\ m_o + j_{R-\min}/2 &= m' - \delta_{II} / 2\pi \end{aligned} \quad \text{Eqn. 18a}$$

But looking at Fig. 10, and remembering that  $m_o=1.5$ , we see that:  $j_{R-\max} = 0,2,4\dots$  and  $j_{R-\min} = 1,3,5\dots$  so that no matter what the case, the relationship between  $m', m_o$ , and  $\delta_{II}$  is:

$$m' = m_o + a - \frac{1}{2} + \delta_{II} / 2\pi \quad a = 0,1,2,3\dots \quad \text{Eqn. 18b}$$

But since we know from Eqn. 16 that  $m'$  is always an integer than either  $m_o$  or  $\delta_{II}/2\pi$  must be a half integer, which means that for  $m_o = 1$ ,  $\delta_{II} = \pi$ . However, the index of the  $m_o = 1$  curve is not less than the index of glass for all  $\lambda$ , and so we do not always expect a  $\delta_{II} = \pi$  phase change. Instead, if we use  $m_o = 1.5$  then  $\delta_{II} = 0$ , which makes sense, because we predict that the index of the film is higher than that of glass for all  $\lambda$ , and thus there is no  $\pi$  phase change at the film-glass boundary! Since we have already ruled out the  $m_o = 2$  and  $2.5$  solutions, we see that  $m_o = 1.5$  is correct. Unfortunately, when the films become very thick ( $>600\text{nm}$ ) it can become difficult to determine the value of  $m_o$  because the

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\* 'Dispersion' is a fancy word for the wavelength dependence of the index of refraction. Also, the index does increase with wavelength in certain regions, but this is called "anomalous dispersion", and occurs only in regions of strong absorption due to a resonance. In the cases of  $m_o=2$  and  $2.5$  in Fig. 10, the increase in the index with wavelength appears to occur between 500 and 800 nm, but there is no significant resonance in this region and so we conclude that these are not the correct data sets.

fringes become numerous and tightly spaced. When this is the case it may be necessary to take IR data in order to determine the order of the fringes.

In general the index of the film is higher than the index of glass which means that the phase change at the film-glass interface (*interface II*) is  $\delta_{II} = 0$ , so in such cases the mess above can be avoided\*. Understanding this allows us to restate *Eqn. 13* in a form that differentiates between maxima and minima:

$$\lambda_{\text{max,min}} = \frac{2 \cdot n \cdot d}{m} \qquad m_{R\text{-max}} = \frac{3}{2}, \frac{5}{2}, \frac{7}{2} \dots \qquad \text{Eqn. 19}$$

$$m_{R\text{-min}} = 1, 2, 3, 4 \dots$$

Table 3 lists the index of a film at the wavelengths corresponding to its reflection spectra maxima and minima using *Eqn. 19*. These numbers are useful numerical values of the

$d = 304.1 \text{ nm}$	$m$	$\lambda \text{ max/min (nm)}$	$n$
$m_o$ :	1.5		
	1.5	817	2.01
	2	630	2.07
	2.5	517	2.13
	3	454	2.24
	3.5	411	2.37
	4	385	2.53

**Table 3:** Table of values of the index of refraction calculated using *Eqn. 17*. The wavelength of the maxima and minima of the reflection spectrum must first be identified and put in the column labeled  $\lambda \text{ max/min}$ . This data is for the sm16d film who's R and T spectra are pictured in Fig. 9.

index around each data point, but a functional form of the index is often desirable. Now that we know that the  $m_o = 1.5$  curve is the correct one, it is simply a matter of fitting the index data from *Eqn. 17* to the Sellmeier equation in order to obtain a

wavelength dependent form of the index. The Sellmeier equation has the form:

$$n = \sqrt{1 + \sum \frac{\lambda^2 \cdot G_k}{\lambda^2 - \lambda_k^2}} \qquad \text{Eqn. 20a}$$

where the  $\lambda_k$  correspond to resonance wavelengths and  $G_k$  are the corresponding amplitude coefficients. The Sellmeier equation was derived by Sellmeier in 1871. It is

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\* Notice that we are talking about glass, or quartz, here only. If a different substrate is used with a higher index of refraction one should be careful that this phase shift is accounted for.

based on a classical forced oscillator model of the atom being driven by the electric field of the incident light wave. The model envisions the atoms as being small and close together relative to the wavelength of light [Ref. 7]. Though this equation takes neither the damping nor the effect of fields from adjacent atoms into account, it does seem to work here based on the close fit that can be seen in Fig. 12.

Now, if we assume that there is only one resonance close to the region of interest (presumably the band-gap) then this equation can be simplified. Letting  $\lambda_g$  correspond to the resonance within our region of interest, and let  $\lambda_a$  and  $\lambda_b$  correspond to resonances far above and below (in wavelength space) our region of interest respectively, the Sellmeier equation becomes:

$$n = \sqrt{1 + \frac{\lambda^2 \cdot G_g}{\lambda^2 - \lambda_g^2} + \sum_a \frac{\lambda^2 \cdot G_a}{\lambda^2 - \lambda_a^2} + \sum_b \frac{\lambda^2 \cdot G_b}{\lambda^2 - \lambda_b^2}} \quad \text{Eqn. 20b}$$

However, with our assumption that  $\lambda_g$  is the only resonance wavelength close to our region of interest, we have two remaining possibilities:  $\lambda_a \gg \lambda_g$  or  $\lambda_b \ll \lambda_g$ . For  $\lambda_a \gg \lambda_g$  the term in the first sum of Eqn. 20b approaches zero and hence the whole sum goes to zero. On the other hand, for  $\lambda_b \ll \lambda_g$  the term inside the second sum approaches  $G_b$ . This means that the second sum is reduced to a sum of the constants,  $G_b$ , which is itself a constant. Combining this sum of constants with the 1 at the front of the square root, Eqn. 20b reduces to:

$$n \cong \sqrt{A + \frac{\lambda^2 \cdot G_g}{\lambda^2 - \lambda_g^2}} \quad A = 1 + \sum_b G_b \quad \text{Eqn. 20c}$$

So that we have approximated an equation with an unknown number of undetermined coefficients to one that has three and works very well over the region in which we are interested. Now all that is left is to fit the data to this equation. Unfortunately this

equation is not capable of being reduced to a form in which its parameters can be found through any kind of closed form linear *or* non-linear regression analysis that I am aware of. Instead, a more brute-force guess-and-check numerical analysis is required.

Fortunately, Microsoft Excel contains the solver add-in which uses a powerful “Reduced Gradient (GRG2)” nonlinear optimization code developed by Leon Lasdon, University of Texas at Austin, and Allan Waren, Cleveland State University [Ref. 8]. The `index_calc.xls` workbook has been written to use this solver to fit the parameters  $A$ ,  $G_g$  and  $\lambda_g$  to the data points obtained using *Eqn. 19*.

This analysis, however, requires one to know the thickness of the film beforehand because the data points of the index depend on the thickness in accordance with *Eqn. 19*. When the thickness is not known beforehand, it is possible to use a simpler dispersion relation in order to compute the thickness. This simpler form, which is a rather drastic approximation to *Eqn. 20*, is:

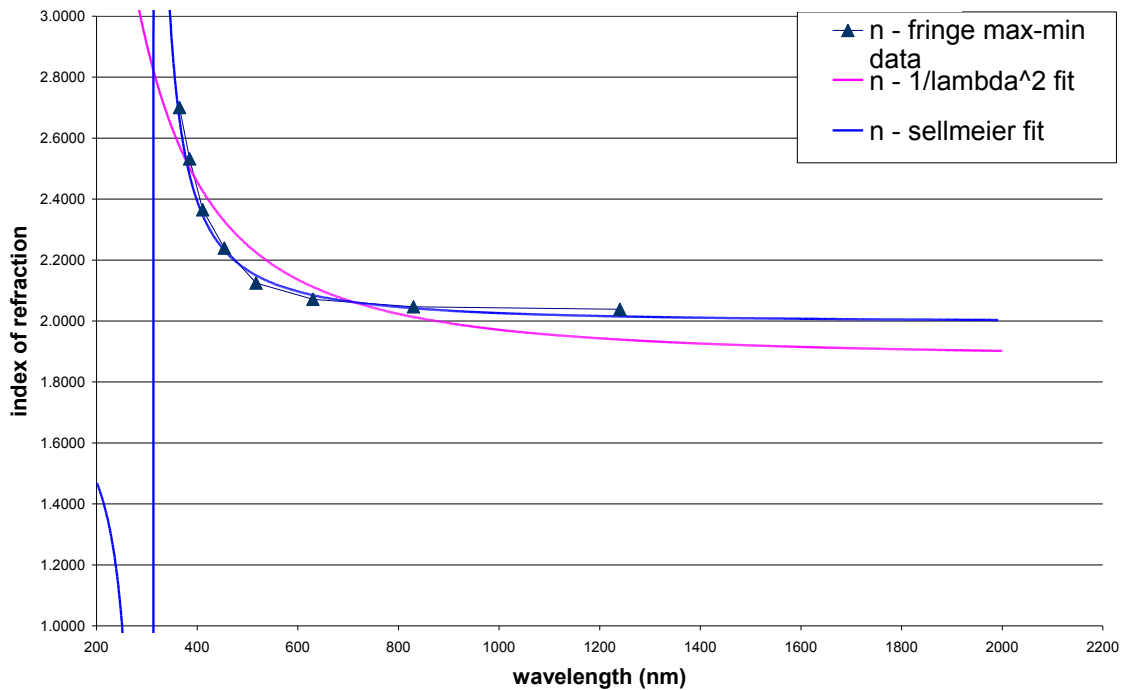
$$n = int + slp \cdot 1/\lambda^2 \quad \text{Eqn. 21}$$

Where *int* and *slp* are parameters that are fit to the data in a simple linear regression of the index vs.  $1/\lambda^2$ . The advantage of this form is that its parameters are determined easily and quickly. With this new approximate dispersion relation we are now prepared to calculate reflection coefficients for *interface II* in Fig. 9 (the film-substrate interface), which will in turn allow us to produce a calculated reflectance of the film plus the substrate.

Once a calculated spectrum has been obtained using this analysis, one can adjust the thickness parameter of the calculated spectrum until the calculated spectrum matches the experimental data as well as possible. Generally this means that the interference

fringes line up with one another. With this newly determined thickness parameter it is now possible to go back and find the parameters in *Eqn. 20c* using the solver. Once this index is found, one can go back and recalculate the thickness using the new index, followed by readjusting the index parameters with this new thickness. It may seem that one could recalculate the thickness followed by the refractive index indefinitely. However, if this calculation is performed more than once, the parameters begin to diverge from, rather than converge onto, the correct solution. For this reason the refractive index and thickness are recalculated only once rather than multiple times.

### Index of Refraction vs. Wavelength sm16d - CuScO<sub>2</sub>



**Figure 12:** In this graph, the data points, the simple  $1/\lambda$  fit and the Sellmeier fit are all plotted as a function of wavelength. Notice how much closer the Sellmeier fit, from *Eqn. 20c*, is than the  $1/\lambda$  fit. Also notice the discontinuity in the Sellmeier fit at 317 nm. This should in theory be the band gap, but it is off by approximately 50nm.

The index\_calc.xls\* workbook does all of this rather efficiently, but requires a high speed computer to perform the calculations. Using a Dell Optiplex GX260 with a 2.26 GHz Pentium 4, it took approximately 10 minutes to compute the thickness and the refractive index. Although the error in these calculations has not yet been properly determined a reasonable estimate is about 5 to 10 percent. It should also be noted that the

Parameter	Value
$d$	304.1 nm
$A$	2.91
$\lambda_g$	1.07
$G_g$	317.97

**Table 4:** Parameters calculated by solver in the index\_calc.xls workbook for the CuScO<sub>2</sub> film sm16d.  $d$  is the thickness of the film and the other parameters define the wavelength dependence of the refractive index according to Eqn. 20c.

index\_calc.xls workbook calculates a reflectance and transmittance of the film only (without a substrate) as discussed at the end of section II.1. This result could be useful at some point, but unfortunately the magnitudes of the fringes are generally too large to be correct. As a result, the error in these calculated spectra seem to be quite significant and I am thus unsure how accurate or

useful they are.

For more understanding of how these calculations are done, see the index\_calc.xls help document, which is included in this paper as appendix B. The calculations contained therein are based primarily on the work of Hishikawa et al [Ref. 9].

## VI. Conclusion

A flexible spectrometer system capable of measuring from 200nm to 2.6 $\mu$ m has been developed and many of its components analyzed. In the beginning this work focused on proper normalization and methods for obtaining more accurate data while discussing other basic theories of spectrometry. The integrating sphere was shown to be

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\* The index\_calc.xls workbook assumes that the film is on a quartz substrate. If the substrate is a different material the index\_calc.xls workbook should be modified to reflect this, see Appendix B for details of the index\_calc.xls workbook.



a useful tool for gathering all light reflected or transmitted from a sample. The Kubelka-Munk remission function was applied to a set of powder samples brought down from Reed College and an approximate value of the band-gap,  $1.75 \pm 0.25 \text{ eV}$ , was obtained for a  $\text{Sr}_x\text{NbO}_3$  sample named mm62. The extension of the systems range into the near-IR was moderately successful and reflectances of  $\sim 20\%$  and transmittances of  $\sim 70\%$  were seen for  $\text{CuScO}_{2+y}$  films. Finally, a theory for determining the refractive index of films, based on thin film interference and the Sellmeier equation, was introduced and applied to a  $\text{CuScO}_{2+y}$  film named sm16d. Discrete values of the index of refraction of this material can be found in table 3, while a wavelength dependent form is demonstrated by using the parameters in table 4 in *Eqn. 20c*.

There are still many bugs that need to be worked out of this system including resolution issues and aligning the spectra from one measurement to the next. Every effort should be made to improve the accuracy, reliability and ease-of-use of this spectrometer in the future. It seems to me that the systems versatility has been enhanced so far that these other factors have been stretched a bit thin. Fortunately perhaps, I do not see many more avenues down which this system's versatility could be increased so that more attention can be given to refining what already exists. Finally, future users must be careful not to let this system fall into neglect or over-use. Because the system is as versatile as it is it requires maintenance and realignment from time to time. This, I suppose, is one of the drawback of creating a versatile system such as this one rather than purchasing a factory device.

I feel very fortunate to have had the opportunity to do graduate level research as an undergraduate. The skills and experience that I gained during this time will

undoubtedly carry with me in the future. I just hope that these documents make it possible for the next person to learn the system easily, and use it efficiently and accurately.

## Acknowledgements

I would like to thank Professors Janet Tate and David McIntyre for the opportunity to be involved in their work. Both professors were consistently available for consultation.

Professor Tate was my main advisor and her guidance on a range of topics throughout my undergraduate career has been invaluable. Her insistence on accuracy and integrity has made clear to me that research, perhaps even more than other parts of life, demands both. I cannot possibly thank her enough for all of the helpful, straightforward advice she has given me.

Professor McIntyre was always very enthusiastic and helpful when I encountered many of the problems that I did. As my optics advisor, I usually consulted him when faced with optics related problems. Our discussions rarely ended without finding a potential solution to the problem at hand.

I would also like to thank Derek Tucker for showing me the spectrometer system originally. The work that he did with this system made it possible for me to make the progress I did. Furthermore, his thesis was not only a guide for my own, but also a reference.

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# Appendix A

## Operation Procedure for Wngr-118 Spectrometry System

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\* Much of this document has been “borrowed” and updated from a document written by Derek Tucker about an older but similar system.

## **Introduction**

The spectrometry system can be divided into several major components; power supplies, light sources, a double monochromator, a series of lenses and mirrors, an integrating sphere, and one of two detectors attached to their correct interfacing devices. Spectra are measured using a computer interface and one of two HP\_VEE programs called UV\_scan.vee or IR\_scan.vee. These two files, and this document can be found in c:\spectrometer\, (this directory has a shortcut on the desktop titled “spectrometer”) on the Dell PC in WNGR 118.

## **Power Supplies & Light Sources**

- ❖ There are two power supplies for this system. One for a tungsten filament, halogen quartz (HQ) lamp. The second powers a xenon (Xe) lamp. The useful spectrum for the HQ lamp is approximately 400nm to 3000nm. The useful spectrum for the Xe lamp is approximately 325nm to 900nm.
- **CAUTION:** The Xe lamp produces ozone and extended use of this source without ventilation can be dangerous. If any unfamiliar smell is noticed or you feel lightheaded, it may be ozone, so open the door periodically to get some fresh air and be sure ventilation is adequate.
- **Caution:** The Xe lamp is also under extreme pressure (~50 atmospheres). For this reason it is very fragile and very dangerous. If you are going to open the lamp housing always wear a face shield and wear clothes that don't leave skin exposed. If you are going to change, move or otherwise shift the Xe lamp directly **consult the safety instructions first!**

## **Double Monochromator**

- ❖ A monochromator is a device that uses a diffraction grating to separate the incoming “white” light into a spectrum of wavelengths, a rainbow. There are two monochromators aligned in series. As the grating angle is changed, the wavelength of the exiting light changes accordingly. The end result is that polychromatic light enters and approximately monochromatic light exits. The wavelength of the exiting light is a whole number multiple of the number displayed on top of the monochromators, the multiplier varies depending on which grating is in the monochromator.

Grating ( $\mu\text{m}$ )	multiplier
.25	1x
.5	1x
1	2x
2	4x

This wavelength can be varied manually with the monochromator control box under the monochromators or automatically through the computer interface.

- ❖ Operating the Monochromator:
  - The monochromator control box under the monochromator has two switches labeled “slew”; these are the primary switches you will use, in general don’t touch anything else. The “fast” switch adjusts the monochromator quickly. The “slow” switch adjusts the monochromator slowly for precision. Use the fast switch to get close, and the slow switch to get exact.
  - When adjusting for a scan be sure to use the fast switch to go about 10nm below your target, then bring it back up to 1-3nm before your target, then finally use the “slow” switch to bring it to your target exactly. This coming at it from

underneath/behind, technique is necessary to compensate for the play in the monochromator gears, mechanisms etc.

- The other switches of importance are, the power switch and the knobs on the far left. These knobs adjust the max that the monochromator will go. If you go past this, the monochromator will freeze and a red light will go on next to the knob. In order to adjust the monochromator you will have to turn the knob down.
- The other switches are not necessary for our use, do not touch them.

#### ❖ Changing Monochromator Gratings

- First put on a pair of lab gloves, so that there is no chance of getting finger prints on the grating surface.
- Start by using the “fast” switch on the monochromator control box to slew down to about 200. This sets the monochromator up so that it is easiest to pull the gratings out.
- Remove the sheet metal housing from the monochromator.
- Locate the grating in the monochromator. It is on the near side facing away from you.
- Pull back the spring-loaded lever which pushes against the back of the grating.
- Grab the metal tab on the grating mount and carefully remove the grating such that it **does not get scratched**. Place the grating in its slot in the plastic grating case.
- Now grab the new grating and – again carefully – place it so that it seats nicely against its mount. Once you believe it is seated, gently jockey it up and down and side to side to make sure. You can also peer into the monochromator to try and



see if it is seated if you are not sure. This is the trickiest part of changing the gratings. You will have to get the feel for it.

- Let the spring-loaded lever down against the back of the grating mount.
- Put the sheet metal housing back on the monochromator.
- Repeat these steps for the other monochromator and grating.

**Inside the Box—Lenses and Mirrors, Integrating Sphere, Sample Mounts (see below for detectors)**

- ❖ The cardboard box is used to block out external light “noise” that might interfere with accurate measurements. All measurements should be taken with the lid of the box closed as completely as possible.
- ❖ The lenses and mirrors are arranged to gather as much light as possible onto the sample and the detector.
- ❖ The filter-wheel inside the box is used to cut out light below the wavelengths indicated next to each filter.
  - The filter-wheel can be operated manually by the filter-wheel control panel or by computer interface.
  - Note: four “steps” on the panel represents one filter change. The wheel can also be turned physically by hand. This is sometimes convenient because when you feel the wheel turn one notch, this is one filter change, rather than a quarter when the control panel “step” is used.
  - The filter-wheel is necessary because the monochromator not only outputs light of the specified wavelength, but also  $\frac{1}{2}$ ,  $\frac{1}{3}$ ,  $\frac{1}{4}$ , (etc.) of that wavelength. Therefore we use the filter-wheel to block light of these wavelengths.

- ❖ The integrating sphere is used to study powders and to measure diffuse reflection and transmission of films.
- ❖ There is one kinematic stand holding a mirror inside the box. If this stand is placed on its mount, the light is directed towards the sphere for powder and diffuse measurements. When the stand is not on its mount, the light is allowed to pass to the direct transmission and specular reflection part of the system.

## **Detectors**

- ❖ **Silicon Detector**: UV and visible light (up to ~900nm)
  - The silicon detector is a small round photo-detector, approximately 1 inch in diameter. The circular black area on the front of the detector is the receptive part of the material. On the back of the detector a co-axial cable may be attached.
  - Ideally the incident light image will fall completely within the boundary of the sample material and should be within the receptive area of the photo detector to ensure that the resulting spectrum is accurate.
  - Positioning the silicon detector:
    - For reflection and transmission spectrum the silicon detector can be placed in a mount that is on a stand with a short focal length lens (~2.5cm). The purpose of this lens is to gather all incoming light and focus it onto the detector.
      - This stand can now be placed up against the “kinematic stops” (stand base platforms) in either the reflection position or the transmission position.
      - Finger-tighten the stand down with the sliding clamps.

- For diffuse and powder measurements the detector can be placed on the sphere at the port which is on the equator of the sphere (consider the two opposing ports to be along the sphere's axis).

❖ **Lead-Sulfide detector**: near infrared light (~800-2700nm)

- The lead-sulfide detector is a large rectangular box, approximately 2"x2"x5". The back of the detector has two connectors. The coaxial connector is the signal-out. The serial connector is the power-in. On the front of the detector is a filter that keeps light below approximately 800nm out. This filter is in place in order to protect the detector from high frequency light that can bounce around in the detector and create problems. Do not remove this filter with any room lights on.
- The lead sulfide detector requires a DC power source. The power supply for the lead-sulfide detector is located to the left of the (Kepco) power supply for the Halogen-Quartz lamp. The power supply is a gray box with a black switch and a cable coming out of it.
  - The cable that leaves this power supply then goes into another little gray box that interfaces the power supply cable with the detector cables. Be sure that the detector cables go in such that "+" goes in red, "gnd" in green, and "-" in black.
- The lead sulfide detector requires an AC signal. For this reason a chopper wheel has been placed in front of the lamp to generate an AC signal. See: Power Meters and Computer Interface → Lead-Sulfide interface, for more on the chopper wheel.

- The lead-sulfide detector has a gain switch. Normally we have too much signal rather than not enough so, we use the 2x position. Assume this is the case unless otherwise stated or you don't have enough signal.
- Positioning the Lead-Sulfide detector:
  - The infrared detector must be placed carefully so that the maximum amount of light is striking the detector. This is very difficult because the detector is small (3X3mm) and can not be seen behind the filter.
  - The transmission signal must be as closely proportional to its reflection signal as possible. I.e. for quartz, we expect ~6% light reflected and ~94% transmitted (in the IR). However, if the detector is not placed properly when measuring reflection than when the data is normalized to the lamp we may not get correct data. Usually it is not a problem between measuring lamp and transmission because the detector can simply stay where it is, but for reflection, the detector must be moved.
    - The best way to deal with this problem is to hook up the detector to an oscilloscope and turn on the system so that you get a signal out of the detector.
    - Now move the detector in the plane perpendicular to the incident light and watch carefully for where the signal is strongest. Do this for both the reflection and transmission positions with a sample mount that will never move.

- Once you have found the location of strongest signal, set up a kinematic system that ensures that the detector will be in the same place the next time you move it.
- Note: in order for this to work, the detector must never move in it's stand, the kinematic stops must never move, the sample position must never be moved or rotated, and all of the lenses/mirrors prior to the sample must never move. In this case, I have already done this for you, and I have set up the kinematic stops so that they are the same kinematic stops for the silicon detector.
  - The infrared detector can also be mounted on the sphere, however, it should be supported by a stand because it is so long and heavy.

## **Power Meters and Computer Interface**

### **❖ Silicon Detector Interface:**

- The silicon detector should be attached to the Newport optical power meter (model 835) with a coaxial cable. There is a notch cut into the back of the box that the coaxial cable and other cables run through. This meter measures the power generated by the light incident on the photo detector. This meter uses a GPIB interface with the computer that works very well and gives a real time power measurement on the console of the UV\_scan.vee program.

### **❖ Lead-Sulfide Interface:**

- The lead-sulfide detector's interface is a bit more complicated. Because it has an AC signal, it requires a lock-in in order to determine the magnitude of this signal.
- Chopper Wheel

- The chopper wheel is located outside of the box and is what creates the AC signal for the detector. The chopper wheel merely spins and “chops” the light with its “fins” in order to create such a signal.
- The chopper wheel has a control panel to go with it. However, the panel does not control the chopper wheel’s speed! The chopper wheel’s speed can only be changed by opening it up and moving the rubber belt from one set of pulley’s to another.
- The control panel actually controls very little. It is used to produce a reference signal for the [lock-in](#). Producing a reference signal:
  - Plug in the chopper wheel. This starts the wheel spinning at a non-variable speed.
  - Turn on the chopper wheel panel, and put all switches in the “up” position. This turns on two led’s in the chopper wheel and also detects the light from them and generates a reference signal.
  - Plug one end of a coaxial cable into the “channel 2” port. This is assuming you are using the upper, (shorter/smaller) holes on the chopper wheel. If the light is passing through the lower (larger/longer) holes on the chopper wheel then use the “channel 1” port. Note: it is recommended that you use the shorter/smaller holes because a higher frequency AC signal yields cleaner data than a lower frequency one.
  - Plug the other end of the coaxial cable into the lock-in reference input.
  - Now you have your reference signal at the lock-in.

➤ **Lock-in**

- The lock-in works by taking a reference signal and an input signal and using the reference signal to find the amplitude of the input signal at the frequency of the reference signal. It is basically doing a fourier transform.
- The lock-in has many different knobs, switches and interfaces.
  - The left hand side is the signal-input. This is where the detector is plugged in. This is also where the voltage scale is chosen. Normally, use 250mV (the maximum), or at least start with this. I have tried to configure the monochromator, lamp etc. in order to make this the scale to choose. More often, the problem will be too much signal rather than not enough, and therefore we end up using the 250mV scale.
  - To the right of the signal-input area is the reference signal and phase adjustment area of the panel. Plug the chopper wheel signal in here ([see above](#)).
  - The 0-90-180-270° phase switch is used to flip the signal in 90 degree increments. The 0-100° knob is used to zero the signal. Once the signal has been zeroed, flip the signal  $\pm 90^\circ$  with the 0-90-180-270° phase switch to get a, preferably positive, signal.
  - To the right of the reference interface, is the voltmeter which has a 0-1, and a 0-2.5 scale to make it easier to read when you adjust the sensitivity. However, on the signal output, the range is from 0-1 volts regardless of the scale chosen for the signal input. (The output signal can and does go higher than this, but I am not sure of how accurate it is after 1 volt. Presumably, the device is linear up to 1 volt, but not necessarily after.)

- To the right of the voltmeter panel is the signal output interface.
  - ◆ The red switch is the lock-in power switch.
  - ◆ The zero offset knob is used in conjunction with the switch below it labeled: “10x full scale”. This switch is used to choose the direction that the knob adjusts the signal. This knob and switch are used primarily for large displacements in signal amplitude (as 10x full scale implies), rather than micro-adjustments.
  - ◆ The larger black knob controls the time constant of the device. Higher time constant produces a smoother looking curve, but this curve has less resolution. A lower time constant produces better resolution, but doesn’t filter as much noise. The trick is finding a time-constant that gives low noise and high resolution, this may mean slowing down the monochromator scan speed.
  - ◆ The DC-prefilter is just another filter similar to the time constant, but it only filters the output signal, rather than the signal that can be read on the voltmeter of the lock-in. Generally, leave this set to “out” and adjust the time constant knob to filter noise.
- From time to time check the phase by making sure it is still zero when the signal is flipped 90°, and certainly never assume that it will be accurate from day to day.
- The lock-in interfaces to the computer through the box to the left of the computer, channel 0.

❖ **Software**



- This spectrometer uses the programming/interfacing language HPvee. The two programs that we use are UV\_scan.vee and IR\_scan.vee. The primary difference between the two is:
  - UV\_scan.vee is configured to use the GPIB interface with the Newport optical power meter.
  - IR\_scan.vee uses “get single value” to retrieve data from the card located to the left of the computer. More on get single value:
    - Get single value has two inputs:
      - ◆ The “hsubsys” input must be connected to the “A/D config” box.  
Click on configure on the “A/D config” box and set it to channel 0.
      - ◆ The “channel” input also requires a value of 0.
    - The “value” output of the get single value box is the value received from the card in volts.
    - The “hsubsys” output is a trigger and a configuration output that is not essential.
  - Other minor differences between the two programs exist.
- When using either of these programs you must select your ending and beginning wavelength on the panel.
- Both programs also display the wavelength in real-time and a plot of the data when the program is finished.
- A “userObject” called “scan” is used to advance the monochromator in both programs. This userObject uses “put single value” with the card to the left of the computer to control the monochromator. “Put single value” is similar to “get

single value” except the value input pin is your digital word input, and use “digital out config” to configure “put single value”. Note: the color wheel is controlled by “put single value” also.

- When using either piece of software you may choose to unselect “rotate filters”. This removes the rotation of the color wheel from the program. The only use for this is when you want to measure the bare grating/lamp spectrum and when you want to measure a filter spectrum. Normally, leave this box checked! If you do not, your data will be inaccurate because of the halving wavelengths.
- When using IR\_scan.vee, you must tell the program which grating you are using. Input the blaze wavelength number here in microns, i.e.: 0.25, 0.5, 1, 2. The program will automatically adjust the monochromator and the wavelength data that is output to the file to compensate for the grating you specify. When you start the program, it will then calculate what the monochromator should read and asks if you have it set to this number.

## **UV step-by-step Run Through**

### ❖ Power up the Equipment

- First, unplug the cord labeled “Chopper Wheel” from the blue power strip located on the lowest ceiling rack above the lamp unit on the north side of the spectrometer float table.
- Now turn this blue power strip on. This turns on the cooling fan for the lamps and should **always be done before powering up either lamp**. You should be able to

feel the fan blowing by putting your hand against the chrome grill on the back of the lamp unit.

- Next, turn on the **XE Lamp**
  - The Schoeffel LPS251 Lamp Power Supply is located on the top ceiling rack above the south side (computer side) of the float table.
  - First, be sure the black power level dial is set to 150W XE and that the fine tuning knob is turned all the way to the left.
  - Second, flip the small metal power toggle on the left side of the face. The voltage needle should rise to about 120V.
  - Now, push and hold the red START button for about one second (the buzz sound is normal). The voltage should drop to about 20V and the current reading should now read about 4.75V. If the current reading remains at zero, try pushing the start button again (you may not have held it in quite long enough).
  - Let the lamp warm up for about 25-45 minutes before taking any spectra. This is how long it takes the lamp to get to a steady state. See [lampstartup.xls](#) for a picture of this.
- Turn on the power to the Newport Optical Power Meter Model 835 using the red push-button. The meter is located on the lowest ceiling rack above the cardboard box at the west end of the float table.
- Using the small metal toggle, turn on the power to the monochromator control box located directly beneath the monochromator stand. There are two other

adjacent toggles, fast and slow, that allow you to manually change the wavelength.

- If the CPU adjacent to the float table is not on already, turn it on and open [UV\\_scan.vee](#).
- Turn on the filter-wheel control panel.

#### ❖ Initial Setup

- Align the chopper wheel so that light passes through one of the holes. The best way to do this is:
  - Put on sunglasses (if you have them).
  - Start by closing the iris down which is between the chopper wheel and monochromator.
  - Now use an allen wrench or similar tool to move the fins of the chopper wheel back and forth until you can tell that the spot projected onto the iris is not being blocked by either fin of the chopper wheel.
- Put the .25 $\mu$ m gratings in the monochromators. See [changing monochromator gratings](#) above (pg 3).
- Adjust the wavelength of the light exiting the monochromators until the two dials read exactly 520nm. See adjusting the monochromator above (pg 2).
- Be sure that the coaxial cable from the detector (out of the back of the box) is plugged into coaxial plug on the front of the Newport optical power meter labeled “detector”.

#### ❖ Align the Optics in the Box

- Open the lid of the cardboard box and remove the front.

- Adjust the filter-wheel by pushing the “step” button until one of the filters or filter holes is lined up with the monochromator slit (remember there are four “steps” per filter change). Now manually (grab the filter-wheel with your hand) turn the wheel until the hole just before the “.345” filter is aligned with the monochromator. Leave it like this, the first thing the program does is move the filter-wheel to the correct filter for your scan range
- You should see a beam of green monochromatic light. If you don’t see it adjust the shutter on the top of the lamp unit with the knurled metal knob. This shutter blocks one lamp at a time. If this does not work be sure that the gratings are in the monochromator and that they are seated properly.
- **Linear Transmission**
  - Be sure that the mirror on the kinematic mount is removed and set aside.
  - Place the stand with the mount for the silicon detector firmly against the “stops”, in the transmission position, and use the clamps to lock it down.
  - Place the silicon photo-detector in its mount on the detector stand and connect the coaxial cable that is in the box to the back of it.
  - Use an allen-wrench to snug the clamps just a bit tighter than finger tight. (There is no reason to tighten more than this).
  - Note: This is where you position the detector if you are going to take lamp spectra as well.
  - Now put your sample in the side of the sample clip/holder closest to the lamp. Also, orient the sample such that the film, rather than the blank substrate face, is facing the lamp as well. I.e. do not orient the sample such that the back of

the substrate is facing the lamp. Note: If you are doing a lamp spectrum, do not insert a sample.

➤ **Specular Reflection**

- Be sure that the mirror on the kinematic mount is removed and set aside.
- Place the stand with the mount for the silicon detector firmly against the “stops”, in the reflection position, and use the clamps to lock it down.
- Place the silicon photo-detector in its mount on the detector stand and connect the coaxial cable that is in the box to the back of it.
- Use an allen-wrench to snug the clamps just a bit tighter than finger tight. (There is no reason to tighten more than this).
- Now put your sample in the side of the sample clip/holder closest to the lamp. Again, orient the sample such that the film, rather than the blank substrate face, is facing the lamp as well. I.e. we want to reflect off the film face, not the substrate face.

➤ **Diffuse/Powder Measurements**

- Place the mirror on the kinematic mount onto the kinematic base plate.
- Place the silicon detector in the port along the equator of the sphere. (Imagine the two opposing ports are the axis.)
- Close the iris in front of the 200mm lens down so that the beam at the top of the sphere is ~3mm in diameter (should be ~5mm at bottom of sphere). (Do not change this once you have begun taking measurements. If you do you will have to re-take a normalization spectrum.)
- Diffuse transmission

- Place a sample in the circular spring-loaded sample holder.
- Swing it into position above the top (north) port of the sphere. Get it as close to the top of the sphere as possible.
- Center the sample so that the beam of green light is touching only the film.
- Place the plug for the off-axis port in its port.
- Diffuse Reflection
  - Place a sample in the circular spring-loaded sample holder.
  - Swing it into position below the bottom (south) port of the sphere. Get it as close to the bottom of the sphere as possible.
  - Center the sample so that the beam of green light is touching only the film.
  - Remove the off-axis plug and orient the sample so that the reflected light goes out this off axis port.
- Powder Measurements
  - Place the powder in a small (~1inch diameter) dish.
  - Mount the adjustable dish holder on the bar to the left of the sphere so that it can swing all the way under the sphere.
  - Place the powder dish in the holder.
  - Swing the powder under the sphere and slide it up as close to the bottom of the sphere as possible.
  - Center the beam in the middle of the powder.
  - Be sure the off axis port plug is in place.
- Reference Spectra

- Find the other BaSO<sub>2</sub> port plug and use an Allen wrench to tighten it onto the bottom (south) port.
  - Make sure that the off axis port plug is in place.
- Put the front, and then the lid, on the box.

### ❖ **Operating the Software**

- Open the file [UV\\_scan.vee](#) in c:\spectrometer\.
- In the UV\_scan.vee program window, type in your desired start and stop points for the wavelength sweep.
- With the optics and your sample aligned inside the closed cardboard box, be sure that the monochromator is still set to 520nm.
- Hit the button labeled “run program”. The program will make sure that you have set the monochromator correctly, and then you will be prompted to choose a file name for your data. Name the spectrum file and save it to YOUR OWN FOLDER to eliminate clutter and confusion.
- After clicking SAVE, the monochromators will begin a sweep of the wavelengths between the limits you specified. This will take about 5-10 minutes. When the sweep is done, the monochromators will return to 520nm and the filter wheel will cycle back to wherever it was to begin with.
- Next, open MS Excel and open your file. A series of dialog boxes will open, which allow tab-delimited files to be opened. Click “Next” at the first box; at the second box, click the “Comma” check box, then “Finish”. Your file should now be shown as a column for wavelength and a column for the raw data measured by the Newport Optical Power Meter.



- Finally, click “FILE > Save As” and save the file as a MS Excel 97 worksheet (.xls) making sure to remove the quotes and .vee from the file name.
- From this point, you can plot it or run band-gap calculations, etc. in the spreadsheet. See the section of this document on analyzing the data.
- Note: there is also a button on the UVscan.vee panel to “rotate filter wheel ‘X’ filters” this is used to turn the filter wheel a specified number of filters without getting out of your chair. If you use this be sure to set it back to the hole just before the .325nm filter before running spectra.

❖ **Shutdown**

- Save all of your files and shut down the programs you used.
- Flip the metal power switch on the xenon lamp power supply to OFF.
- Flip the metal toggle on the monochromator control box to OFF.
- Turn off the Newport power meter.
- Turn off the filter-wheel control panel.
- Turn off the blue power strip last. Give the fan about 8-15 minutes to cool off the powered down lamps before you flip the fan power off.
- Be sure to close the lid on the box before you leave to prevent dust buildup.

## IR step-by-step Run Through

### ❖ Power up the Equipment

- First, be sure the cord labeled “Chopper Wheel” is plugged into the blue power strip located on the lowest ceiling rack above the lamp unit on the north side of the spectrometer float table.
- Now turn this blue power strip on. This turns on the cooling fan for the lamps and should **always be done before powering up either lamp**. You should be able to feel the fan blowing by putting your hand against the chrome grill on the back of the lamp unit. This also gives power to the chopper wheel and the monochromator control box.
- Next, turn on the **HQ Lamp** (Tungsten filament, Halogen Quartz Lamp)
  - The Kepco Power Supply is located on the lowest ceiling rack above the north side of the cardboard box. **BE SURE THE CURRENT KNOB IS TURNED ALL THE WAY TO THE LEFT BEFORE YOU TURN THE POWER ON.**
  - With the current set at zero, flip the linked power switches to ON. The light above the current knob should now be on.
  - Now, watching the voltmeter on the front of the power supply, turn the current knob until the meter reads about 1V, wait a minute, turn the current knob until the voltmeter reads about 3V, wait a minute, turn the current knob until the voltmeter reads about 5V, etc. until the voltmeter reads 10V. The goal is to ramp the voltage up slowly so that we do not stress the lamp filament. The voltage should hold at  $10V \pm 0.03V$ . **CAUTION:** *Keep an eye on the Kepco dial meter just in case the multi-meter is malfunctioning. The current should*

*not rise beyond about 6 amps. The power level will be:*

$$P=I*V=6A*10V=60W$$

- Using the small metal toggle, turn on the power to the monochromator control box located directly beneath the monochromator stand. There are two other adjacent toggles, fast and slow, that allow you to manually change the wavelength.
  - Turn on the lock-in.
  - Turn on the filter-wheel control panel.
  - Turn on the chopper-wheel control panel.
  - Be sure that the power cables for the lead-sulfide detector are plugged into the small gray box behind the large black box, and be sure that these power cables are plugged into the detector.
  - Turn on the detector power supply. It is the gray box located just to the left of the lamp power supply.
  - Turn on the CPU adjacent to the float table if it is not on already.
- ❖ Initial Setup
- Check to make sure you are using the correct grating. See changing gratings in section on the double monochromator → [changing gratings](#) above (pg 3).
    - Use the 1 $\mu$ m grating for scans from ~800-2000nm
    - Use the 2 $\mu$ m grating for scans from ~1000-2700nm
  - Adjust the wavelength of the light exiting the monochromators until the two dials read exactly 520nm. See [adjusting the monochromator](#) above (pg 2).

- Plug the end of the coaxial cable that is inside the box into the lead-sulfide detector.
- Plug the other end of this cable into one of the signal inputs on the lock-in and use the small metal switch next to these coax-ins to select the proper signal (note: never use the “A – B” position for this switch).
- Be sure that the reference signal input from the chopper wheel control panel is plugged into the reference input on the lock-in.
- Turn the 0-100° knob on the lock-in reference area so that the signal on the lock-in voltmeter reads 0 volts. If you can not, turn the 0-90-180-270° switch and try again. Repeat this process until you manage to zero out the signal.
- Now turn the 0-90-180-270° switch 90° so that the voltmeter reads a positive signal.
- At this point the overload light on the face of the lock-in might be on. Use the aperture between the chopper wheel and the monochromator to adjust the signal so that it reads ~0.9 (on the 0-1.0 scale). Now the lock-in is configured to take measurements.

❖ Align the Optics in the Box

- Open the lid of the cardboard box and remove the front.
- Adjust the filter-wheel by pushing the “step” button until one of the filters or filter holes is lined up with the monochromator slit (remember there are four “steps” per filter change). Now manually (grab the filter-wheel with your hand) turn the wheel until the hole just before the “.345” filter is aligned with the

monochromator. Leave it like this, the first thing the software does is adjust the filter wheel to the correct filter.

- You should see a beam of green monochromatic light. If you don't see it adjust the shutter on the top of the lamp unit with the knurled metal knob. This shutter blocks one lamp at a time. If this does not work be sure that the gratings are in the monochromator and that they are seated properly.
- **Linear Transmission**
  - Be sure that the mirror on the kinematic mount is removed and set aside.
  - Place the stand with the lead-sulfide detector firmly against the “stops”, in the transmission position, and use the clamps to lock it down.
  - Use an allen-wrench to snug the clamps just a bit tighter than finger tight. (There is no reason to tighten more than this).
  - Note: This is where you position the detector if you are going to take lamp spectra as well.
  - Note: Be **very careful** with the lead-sulfide detector when moving it around in this way. Not only is it fragile, but the active area of the detector is only 3X3mm. This means that the lens and detector are lined up very precisely, and any jarring or bumping of the detector stand could mess up this alignment.
- **Specular Reflection**
  - Be sure that the mirror on the kinematic mount is removed and set aside.
  - Place the stand with the lead-sulfide detector firmly against the “stops”, in the reflection position, and use the clamps to lock it down.

- Use an allen-wrench to snug the clamps just a bit tighter than finger tight.  
(There is no reason to tighten more than this).
- **Diffuse Measurements**—The alignment for these measurements is a work in progress. (Updates to follow)
- Now put your sample in the side of the sample clip/holder closest to the lamp.  
Also, orient the sample such that the film, rather than the blank substrate, face is facing the lamp as well. I.e. do not orient the sample such that the back of the substrate is facing the lamp. Note: if you are doing a lampspectrum, do not insert a sample.
- Put the front, and then the lid, on the box.
- ❖ **Operating the Software**
  - Open the file IR\_scan.vee in the folder c:\spectrometer\.
  - In the IR\_scan.vee program window, type in your desired start and stop points for the wavelength sweep.
  - Input which grating you will use, i.e.: “1” (for 1 $\mu$ m grating) or “2” (for 2 $\mu$ m grating) etc.
  - With the optics and your sample aligned inside the closed cardboard box, be sure that the monochromator is still set to 520nm.
  - Hit the button labeled “run program”. The program will ask that you have set the monochromator correctly, and then you will be prompted to choose a file name for your data. Name the spectrum file and save it to YOUR OWN FOLDER to eliminate clutter and confusion.

- After clicking SAVE, the monochromators will begin a sweep of the wavelengths between the limits you specified. This will take about 5-10 minutes. When the sweep is done, the monochromators will return to 520nm and the filter wheel will cycle back to wherever it was to begin with.
- Next, open MS Excel and open your file. A series of dialog boxes will open, which allow tab-delimited files to be opened. Click “Next” at the first box; at the second box, click the “Comma” check box, then “Finish”. Your file should now be shown as a column for wavelength and a column for the raw data measured by the Newport Optical Power Meter.
- Finally, click “FILE > Save As” and save the file as a MS Excel 97 worksheet (.xls) making sure to remove the quotes and .vee from the file name.
- From this point, you can plot it or run band-gap calculations, etc. in the spreadsheet. See the section of this document on analyzing the data.
- It is also important to note that the lock-in/detector have a DC offset the source of which I am not exactly sure. At any rate, it will be important to account for this by either taking data where the signal should be zero or finding a part of your data that should be zero and adjusting the entire spectrum for this factor. I.e. if it is a negative DC offset, add this value to your data so that zero signal is actually 0. This matters allot where the spectra are small. If it is not accounted for you will see your normalized data tail off near the ends.

❖ **Shutdown**

- Save all of your files and shut down the programs you used.

- Follow the reverse of the turn on procedure on the Kepco power supply to shut off the halogen-quartz lamp. I.e.: slowly ramping down the current knob over a few minutes time and then let it sit a zero current for a few minutes so that the power supply fan can cool it down. Then flip the linked power switches to OFF.
- Flip the metal toggle on the monochromator control box to OFF.
- Turn off the lock-in.
- Turn off the filter-wheel control panel.
- Turn off the blue power strip last. Give the fan about 8-15 minutes to cool off the powered down lamps before you flip the fan power off.
- Be sure to close the lid on the box before you leave to prevent dust buildup.

### **Data Analysis**

- ❖ There are various templates created for analyzing data so that all you have to do is cut and past your data into columns. When using a template:
  - Do not save over the template. Always save the file as a different name than the template name!
  - Always rename the graph titles, worksheet names, and all other titles to reflect the correct sample name.
- ❖ There are many different templates, some have been created for specific purposes. I recommend creating your own templates so that you better understand them, and so that you know what it is for.
- ❖ The most common analysis is transmission and reflectivity calculations.
  - Be sure that you have a lamp spectrum (a lamp spectrum should always be taken with the samples).



- To get transmission:
  - Divide the transmission spectrum by the lamp spectrum.
- To get reflection:
  - Divide the reflection spectrum by the lamp spectrum.
- In this way you are finding what percentage of the light is being transmitted through the material, and what percentage is being reflected off the surface of it, respectively.
- To see this graphically, simply make a plot of these “normalized” transmission and reflection spectra vs. wavelength.
- ❖ Other analyses include band-gap calculations, alpha measurements, etc.

# Appendix B

## **Index\_Calc.xls Help Document**

This document and the index\_calc.xls file were  
written by: Levi Kilcher

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### Terminology

A “workbook” is an entire \*.xls file, while a “worksheet” refers to a single sheet within such a file. “*R*” refers to the reflectance of a film, and “*T*” to the transmittance. *Solver* is a powerful numerical analysis tool that is used throughout this workbook in order to find various parameters. The word solver has been italicized in order to make it clear what is being discussed.

### History

The index\_calc.xls workbook started as a small template file for calculating and plotting R and T from data taken on the spectrometer system in Weniger 118. Originally,

this system was only capable of measuring spectra from about 200-900nm. Prof. Janet Tate added a few simple calculations and plots for computing the absorption coefficient ( $\alpha$ ), and band-gap. Later, a lead-sulfide (PbS) detector was added to the system to extend its range into the near-IR (~2500nm), at this point the workbook was enhanced to include the IR spectra. In this way as the spectrometer evolved so did the workbook.

When it became apparent that a method for measuring the index of refraction was needed Prof. David McIntyre began modifying the workbook. Later I enhanced, updated, made more accessible and generally re-wrote what he had started to create the workbook that exists now. Along the way, we found that it may be possible to determine the thickness of the film from our data as well.

The index\_calc.xls workbook is a Microsoft Excel workbook designed to calculate the index of refraction of thin films based on reflection and transmission spectra. Originally the idea of this workbook was to create a template in which one would only need to input the raw reflection, transmission and lamp spectra, in order to calculate the index of refraction and thickness of the film. In the end I believe that the workbook achieved this goal relatively well. Although there are a few intermediate steps before these parameters are obtained they are straight forward once you learn to navigate your way through the workbook.

## Getting Started

The best way to get started using this workbook is to familiarize yourself with what each worksheet does and where the parameters are defined. The issue of becoming

familiar with each worksheet is outlined below in the workbook overview section. Go to the “Help” worksheet in order to find links to the location of each parameter.

One of the most important things about this workbook is that you should never need to enter any values or data into a cell unless that cell is blue. I have labeled some boxes in green, to indicate that they are reference variables, but reference variables have no dependents, and thus should have no reason to be changed. Reference variables are placed in locations where they can be compared to other variables conveniently.

Once one has become familiar with the worksheets and parameters of this workbook, it may be instructive to begin to look at each calculation more closely. Most of the more complicated calculations are done in the calc-index, calc-n\_fixed and calc-n\_variable worksheets. Understanding these worksheets should be done last and may require reading more literature than this document. The next section outlines each worksheet in the workbook and goes over some of the more complex worksheet functions and their uses.

## Workbook Overview

Each worksheet is described below in detail. Each worksheet name (except the “Help” and “info” worksheets) has two parts. The first part tells you what type of worksheet it is; i.e. a data (dat) worksheet contains data that is obtained from an external source, a graph (gph) worksheet contains a graph, and a calculation (calc) worksheet contains calculations based on the external data and the formulas contained within the worksheet. The second part of the name further classifies what is happening in the worksheet.

- I) **graph-mstr** – Master graph of all normalized spectra contained in worksheet
- 1) This graph displays the Transmission, Reflection and  $T/(1-R)$  data normalized to the bare lamp.
  - 2) Transmittance spectra are in reddish colors, reflectance in blues, and  $T/(1-R)$  in greens.
  - 3) The data for this graph is contained in various “dat” worksheets.
  - 4) Things to watch out for:
    - A) Be sure the data range contains all of the data you want it to.
      - (i) Here and in all graphs of spectra, the data ranges are, by default, set wide so that, unless the data set is huge, all the data is plotted. If the data range does become huge, you may need to adjust it.
      - (ii) Data near either end of a spectra might be high in noise. You should cut this data from the graph by shrinking the data range appropriately.
      - (iii) To change the data range right click on the graph and select “Source Data...” then click on the “Series” tab, then click the appropriate series, and adjust its range.
- II) **info** – This worksheet is where the film name and material of the film is placed to change the graph labels and other text boxes globally throughout the workbook. Also, insert the thickness measured externally here. Other plot display text is also calculated here.
- 1) ALWAYS BE SURE TO INPUT THE FILM NAME! The importance of this cannot be overemphasized, I recommend getting in the habit of doing this when you paste your first set of data into the workbook.

- 2) Note: the measured thickness is not used in any calculations by default (except when you use the “calc n w/ d measured” button on the calc-index worksheet). It is merely displayed throughout the workbook as a reference. However, it can be used if you wish to, its name is  $d_{meas}$ . Most calculations are done in nm, so it should be input in these units.
- 3) There is also a location where you can input the name of the data collector and relevant dates, such as the date the data was taken and the date it was analyzed. This information will appear in the upper-left hand corner of all of the graphs.

III) **Help** – A worksheet that contains this text file, along with tables of variable and data locations.

- 1) This document is placed within the workbook template for convenience and to ensure that it is not misplaced. However, if one is familiar with the workbook, it need not be included in every workbook... just DON'T DELETE THIS DOCUMENT FROM THE MASTER WORKBOOK TEMPLATE.
- 2) Each variable and data location is hyperlinked in the table. How do you like that! Of course you can also click on the “Name Box” on the “data input bar” and scroll down to find any name you want without being in the “Help” worksheet, but there is no description. Use this table to help get you started.

IV) **gph-UV** – A graph of data taken in the UV-visible region using the silicon detector.

- 1) Often, this will be the only spectral range for which you have taken data.
- 2) As in all graphs the data range is the most important thing to keep track of. By default the data range is set wide.
- 3) The data for this graph is contained in the “dat-UV” worksheet.

V) **gph-IR** – A graph of all data taken in the Infrared region.

- 1) As in all graphs the data range is the most important thing to keep track of. By default the data range is set wide.
- 2) The data for this graph is contained in the various IR data sheets.

VI) **dat-UV, dat-IR1, dat-IR2** – Data worksheets for data collected from the UV through the visible and into the near-infrared.

- 1) Paste the data into the appropriate worksheet in the appropriate “raw data” column. The best way to do this is to copy the data from the source location and then use “paste special...” from the edit menu:
  - A) Select data at source location and copy it.
  - B) Return to appropriate “dat” worksheet. Click on the cell at the top of where you want the data pasted.
  - C) Click Edit, Paste Special.
  - D) Click the “Values” radio button.
  - E) This will paste the correct values into this column without changing the formatting or deleting any comments.
- 2) Things to be careful of:
  - A) Make sure that the formulas are the same length as the data set.
  - B) By default, common wavelength values have been placed in the “wavelength” column. However, if you change the scanning increment or scan range, be sure to change these wavelength values as well.

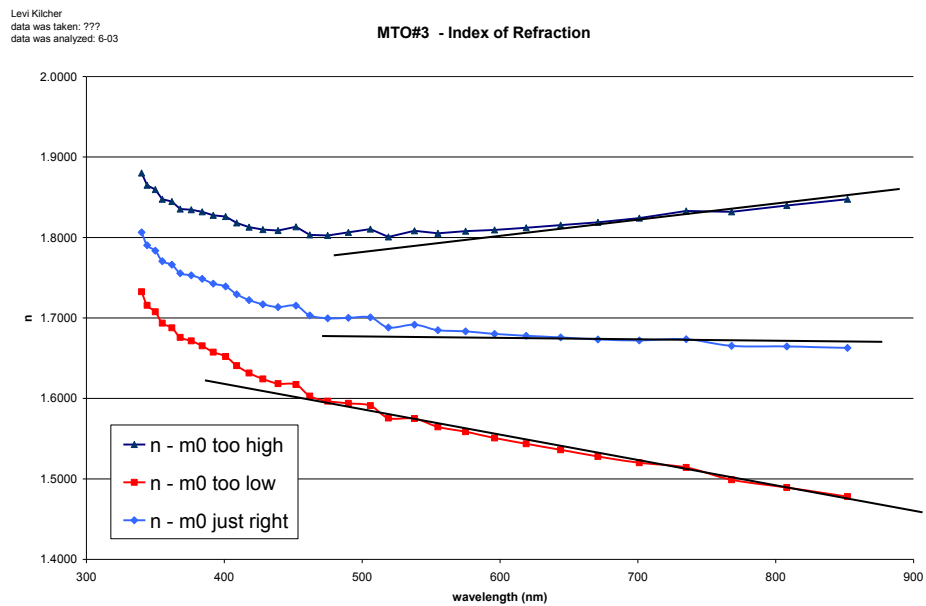
- 3) dat-IR1 and dat-IR2 refer to different sections of the near-IR region. They are necessary because each diffraction gratings does not span the entire region of interest.

VII) **calc-index** – Worksheet that calculates the index of refraction and thickness (if you want it to) of the film.

- 1) The first thing a person should do in this worksheet is input the locations of the fringes and determine their corresponding  $m$  values.
- 2) By looking at the various graphs of the spectrum, place the max and min points of the interference fringes in the blue part of column K with the first fringe (highest wavelength) at the top of the column.
- 3) If you can see the first order (highest wavelength,  $m=1$ ) fringe than merely set  $m_o=1$ . Higher order (larger  $m$ ) fringes occur at decreasing wavelength.
- 4) However, if you cannot see the first fringe, as is generally the case. Determining the value of  $m_o$  can be more difficult.
  - A) The following relationships work as long as the index of the film is higher than the index of the substrate (which is generally the case with our films on quartz substrates).
    - (i)  $m = \text{integer}$ : maximum in T (minimum in R)
    - (ii)  $m = \text{integer} + \frac{1}{2}$ : minimum in T (maximum in R)
  - B) With this simplification, one should merely try different values of  $m_o$  and then look at the graph of the data points on the gph-index worksheet.
    - (i) If the set of data looks like it is curving up as  $\lambda$  increases than you have guessed a value of  $m_o$  too high.



- (ii) If the set of data looks like it becomes linear as  $\lambda$  increases and this line has negative slope than  $m_o$  is too low.
- (iii) If the set of data appears to be becoming linear as  $\lambda$  increases and this line has zero slope (in other words if  $n$  looks as though it becomes constant as  $\lambda$  increases) than you have guessed the correct value of  $m_o$ .
- (iv) The plot below is an example of what is discussed in parts i, ii, and iii:



Notice that the  $n$  axis of this plot has been magnified. The difference between the three curves will generally be greater than this and it will thus be easier to tell which value of  $m_o$  is correct. This plot is shown as an example of the worst case scenario.

- (v) Note:  $m_o$  should be either an integer or half integer and which of these it should be can be determined from the relationships in part A just a few lines up. This makes determining the correct value of  $m_o$  considerably easier.

- 5) The worksheet then calculates the index at that fringe according to the following equation:

$$n = \frac{m \cdot \lambda}{2 \cdot d}$$

- Where  $m$  is the fringe number talked about above,  $\lambda$  is the wavelength of the fringe and  $d$  is the thickness of the film in the same units as  $\lambda$ . This is repeated for each fringe and these data points are displayed in the table labeled “input of fringe max/min points and associated calculations”.
- 6) Before doing anything else, be sure to set up the fit range and reference page:
- A) Look at the experimental reflectance data and determine a fit range. Here are some basic guidelines:
- (i) Generally the upper bound of the fit range will be the upper limit of the data set in the *reference\_page*. i.e. if your data goes from 200-900nm, your upper bound will be 900nm. However, if one does wish to fit the data less strongly to this part of the spectrum that is totally reasonable.
  - (ii) The lower bound of your fit should be a bit above the band gap. If a fringe or two exist in the band gap it is probably best to not include these in the fit range.
- B) Return to the calc-index worksheet.
- C) Input the values for the fit range in their proper locations.
- D) Type the name of the reference worksheet that contains the experimental data you are fitting to.

- 7) All data entry for this worksheet is essentially done. Now all that we need to do is perform the calculations necessary in determining the information we seek.
- 8) When the film thickness is not known beforehand: calculating the thickness,  $d$ , and the refractive index,  $n$ . Be sure that you have set up the fit range correctly as described above and then click the “**calc n&d**” button in order to begin the process.
- A) The first thing that the “**calc\_n&d**” button does is obtain an approximate value of the thickness. It does this by adjusting  $d$  in order to fit a theoretical reflectance curve to the experimental one. However, a form of the refractive index is needed. This initial form is the simple approximation to the Sellmeier equation:

$$n = int + slp \cdot 1/\lambda^2$$

This simple form comes from a linear regression fit of  $n$  vs  $1/\lambda^2$ . The data points used come from the location of the fringes as discussed above. This simple form is used because its parameters can be determined without the *solver*.

- (i)  $\text{Chi}_{2\_1}$  is the variable that defines the error in between the theoretical and experimental reflectance spectra.
- B) Now that a value of the thickness,  $d$ , has been found, *solver* adjusts the parameters of the Sellmeier equation to fit the parameters of the Sellmeier equation to the set of data points for  $n$ . The approximation to the Sellmeier equation used here is and derived in the body of this document in section V

(refractive index) is:

$$n \cong \sqrt{A + \frac{\lambda^2 \cdot G_g}{\lambda^2 - \lambda_g^2}} \quad A = 1 + \sum_b G_b$$

In this second stage, *solver* adjusts the parameters A,  $G_g$  and  $\lambda_g$  in order to fit the equation above to the data points obtained from the location of the fringes and the previously determined film thickness.

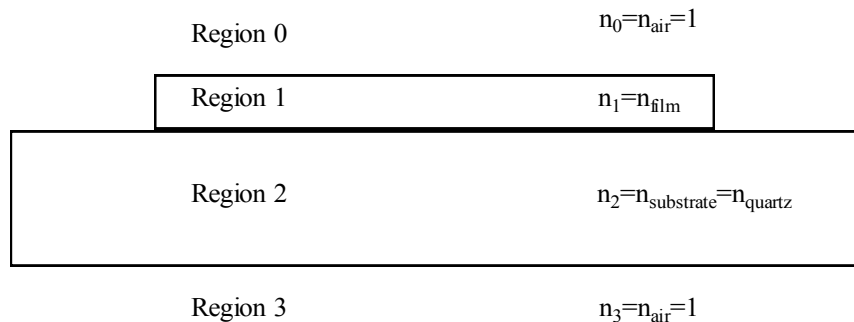
- C) Now that we have a more exact form of the refractive index, *solver* is used to re-adjust the thickness to fit a new reflectance spectrum (which uses this more exact index) to the experimental data.
- (i) Chi\_2\_2 is the error between the theoretical and experimental reflectance spectra over the fit range. In other words, it is what *solver* tries to minimize.
- D) Finally, with this more exact value of the thickness, *solver* once again fits the Sellmeier equation to the index data points (which have changed because  $d$  has changed) in order to determine a final approximation of the refractive index.
- E) One might think that the iterations performed in parts C and D above could be repeated indefinitely. However, if they are repeated the solution diverges from, rather converge to the correct answer. This is why I have done this many iterations and not more.
- 9) When the film thickness is known beforehand: calculating the refractive index,  $n$ .  
Click the “**calc n w/ d\_measured**” button in order to begin the process. This will do two things:

- A) First this will set the thickness variable,  $d$ , equal to the measured thickness,  $d_{meas}$ , defined on the info page. Because of this, one must have a measured thickness in the appropriate cell in the info worksheet.
- B) Second, this will fit the parameters of the Sellmeier equation defined above to the data points using the *solver* to produce a functional form of the refractive index.
- (i)  $\chi^2$  is the error between the theoretical and experimental reflectance spectra over the fit range.
- 10) It is also possible to calculate the refractive index based on an arbitrary thickness or guess. To do this simply put your guess of the thickness into the thickness cell and click “**calc n w/ arbitrary d**”. This will merely fit the Sellmeier parameters to the data points determined by this value of  $d$ .
- 11) Adjust the *reference\_page* and fit range (*fitr\_min* and *fitr\_max*) in this worksheet as well.
- A) The *reference\_page* (reference worksheet) name is the worksheet which contains the data that you wish to fit the theoretical spectrum to. All that one must do is change the value of this cell (calc-index!G3) so that it matches the name of the worksheet to which you want to compare your theoretical data. All values within the calc-R<sub>n</sub>-Sellmeier and calc-R<sub>n</sub>-simple worksheets will adjust to this new reference worksheet.
- B) Only dat-UV, dat-IR1 and dat-IR2 are valid entries for the reference page cell.
- C) In general, you will fit the data to the UV-visible data, and so the worksheet is set to dat-UV by default.

- D) Adjust the fit range, in nm, so that it extends over most of the fringes, and not into the region of the band gap.
- E) The *delta\_lambda* name is also defined in this worksheet. It is simply the increment in wavelength of the data set. It is used to calculate the fit range in column Z. It is necessary because the wavelength increment is not necessarily 1nm.
- 12) The parameters *A\_sell*, *G\_sell*, and *lambda0* correspond to the parameters *A*, *G<sub>g</sub>* and  $\lambda_g$ , respectively, in the Sellmeier equation defined in part VII.7.B above. In general you should not need to change them manually, but they could be changed without creating problems (*solver* will just change them back to whatever they should be). The one time one might want to change them occurs when:
- A) Sometimes *solver* will converge on a local minimum when obtaining the parameters of the Sellmeier equation rather than the true minimum. If this happens I recommend that you put in a value for *lambda0* ( $\lambda_g$ ) that corresponds to the location of the band gap. If the problem persists, use common values for *G* and *A* such as:  $1 < A < 10$  and  $0 < G < 10$ .
- 13) At the far right of the worksheet, the refractive index is calculated based on the simple  $(1/\lambda^2)$  form of the index and the more complicated Sellmeier form from 0.2 to 2.0 microns so that it can be plotted in the worksheet “gph-index”.
- VIII) **gph-index** – Graph of index as a function of wavelength.
- 1) This worksheet plots the index as a function of wavelength ( $\lambda$ ) for the calculations based on the fixed fringe locations *and* the *solver* solution.

IX) **calc-R\_n-Sellmeier** – This worksheet calculates a theoretical transmittance and reflectance based on the Sellmeier equation form of the index. This calculated spectrum can then be fit to the experimental data by adjusting the thickness parameter  $d$  in order to obtain a value for  $d$ , as described in the section about the calc-index worksheet.

- 1) Before doing anything else, drag all formulas on this worksheet so that they are the same length as those in the reference worksheet (see below).
  - A) In the upper left hand corner, the parameters of the Sellmeier equation are displayed for convenience along with the sum of  $\chi^2$  which indicates the error between the theoretical and experimental data
- 2) Below is a column by column record of the equations in this worksheet. Here is a diagram of a film stacked on a substrate and some labels that will clarify some of the notation:



- A) Column A uses the “concatenate” function followed by the “indirect” function. Concatenate is a function that “joins several text strings into one text string” [Ref. 11]. Here it is used to create a text form of the location of the wavelength data in column A of the *reference\_page*. The “indirect” function returns the reference indicated by a text string. This simply links this

cell to the reference indicated by the joined text. The concatenate and indirect worksheet functions are used together repeatedly in this worksheet.

- B) Column B is just the wavelength in column A converted to  $\mu\text{m}$ .
- C) Column C is the energy in electron-volts of columns A and B.
- D) Column D is the index of refraction of quartz as a function of wavelength.

This column uses the Sellmeier equation with the following constants:

$$n = \sqrt{1 + \sum_k \frac{\lambda^2 \cdot G_k}{\lambda^2 - \lambda_k^2}}$$

$G_1 = 0.6962$	$\lambda_1 = 0.0684 \mu\text{m}$
$G_2 = 0.40794$	$\lambda_2 = 0.11624 \mu\text{m}$
$G_3 = 0.89748$	$\lambda_3 = 9.89616 \mu\text{m}$

Here  $\lambda$  is in  $\mu\text{m}$ .

- E) Concatenate and indirect are again used to link this column to the column containing the  $T/(1-R)$  data in the *reference\_page* worksheet.
- F) This is simply the index of refraction of the film as determined by fitting coefficients of the Sellmeier equation to the index at the fringe locations.
- G) This is the normal incidence reflection coefficient at the film-substrate interface according to the below equation where  $n_f$  is the index of the film and  $n_s$  is the index of the substrate. Note that in the  $r_{ab}$  notation the beam is incident on medium b and reflected in medium a.

$$r_{fs} = \frac{n_f - n_s}{n_f + n_s}$$



H) Here, an intermediate variable of the absorption coefficient,  $x$ , is calculated that takes complex interference effects of the film into account.

$$x = \left( \frac{T}{1-R} \right) \frac{1 - \left( \frac{T}{1-R} \right)^2 r_{fs}^2}{1 - r_{fs}^2} = e^{\alpha d}$$

I) In column I  $\alpha$ , the absorption coefficient, is calculated from the intermediate variable,  $x$  in column H according to the equation:

$$\alpha = \frac{\ln(x)}{d}$$

J) Here the imaginary part of the complex index,  $k$ , is calculate from alpha according to:

$$k = \frac{\alpha \cdot \lambda}{4\pi}$$

K) In column K, the complex index,  $n_{\text{cmplx}}$ , is created from  $k$  and the real part of the index,  $n$ .

L) The next few columns (L-S) simply calculate the reflection and transmission coefficients for each interface using the normal-incidence fresnel equations. The notation in the diagram above, as used by Hishikawa et. al. [Ref. 9], is used here to label each column. Where  $r_{ab}$  is the reflection coefficient of light incident on material b and reflected in material a. And  $t_{ab}$  is the transmission coefficient from material a into material b.

$$r_{ab} = -r_{ba} = \frac{n_a - n_b}{n_a + n_b} \quad t_{ab} = \frac{2 \cdot n_a}{n_a + n_b}$$

T) Column T is an intermediate exponential variable. In Hishikawa [Ref. 9], it is labeled  $e_1$ , and so I use this notation here.

$$e_1 = e^{\frac{i \cdot 2\pi \cdot n_{\text{cmplx}} \cdot d}{\lambda}}$$

U) The next set of columns calculate transmittance and reflectance for various stacks. The equations are straight out of Hishikawa et al. [Ref. 9]. The notation  $T_{ab}$  is the transmittance from layer  $a$  through all intermediate layers to layer  $b$ , and  $R_{ab}$  is the reflectance off of all interfaces between layer  $a$  and layer  $b$ . Column U calculates the reflectance,  $R$ , of the films interfaces only:

$$R_{02} = \left| r_{01} + \frac{e_1^2 \cdot t_{01} \cdot t_{10} \cdot r_{12}}{1 - e_1^2 \cdot r_{12} \cdot r_{10}} \right|^2$$

V) Column V calculates the reflectance backwards through the film interfaces:

$$R_{20} = \left| r_{21} + \frac{e_1^2 \cdot t_{21} \cdot t_{12} \cdot r_{10}}{1 - e_1^2 \cdot r_{12} \cdot r_{10}} \right|^2$$

W) Column W calculates the transmittance through the film interfaces:

$$T_{02} = T_{20} = \frac{n_2}{n_0} \left| \frac{e_1^2 \cdot t_{01} \cdot t_{12}}{1 - e_1^2 \cdot r_{12} \cdot r_{10}} \right|^2$$

X) This column is the calculated reflectance from the entire stack ( $R_{03}$ ), and is again taken from Hishikawa et. al.

$$R = \frac{T_{20}^2 \cdot R_{23}}{1 - R_{20} \cdot R_{23}} + R_{02}$$

- Y) This column uses the concatenate and indirect worksheet functions again to obtain the experimental reflectance data from the *reference\_page* worksheet. It is used to compare to the above calculated total reflectance.
- Z) This column is simply the  $\chi^2$  error of the data from the theoretical value. At the top of this column, a sum of this error is calculated over the fit range specified by the user. The concatenate and indirect functions are again used to establish the fit range based on the fit range parameters defined in this page.
- AA) Here, the total transmittance through the stack is calculated according to the equation in Hishikawa et. al.:

$$T = \frac{T_{23} \cdot T_{02}}{1 - R_{20} \cdot R_{23}}$$

- BB) Column AB is the experimental transmittance – once again using concatenate and indirect. It is here to be plotted and compared to the theoretical values. Note: there is no  $\chi^2$  value calculated for the transmittance as there was for the reflectance.

IX) **calc-R\_n-simple** – This worksheet calculates a theoretical transmittance and reflectance based on the simple form of the refractive index. This calculated spectrum can then be fit to the experimental data by adjusting the thickness parameter  $d$  in order to obtain a value for  $d$ , as described in the section about the calc-index worksheet. *Slope* and *intercept* are the variables that define the index of refraction according to the equation:

$$n = \textit{intercept} + \textit{slope} \cdot \frac{1}{\lambda^2}$$

- 1) Before doing anything else, drag all formulas on this worksheet so that they are the same length as those in the reference worksheet.
- 2) The calculations performed on this worksheet are exactly the same as those in the calc-n\_fixed worksheet except that the index of refraction,  $n$ , in column F is determined by the simple form of the index rather than the more complicated Sellmeier form.

XI) **gph-fit\_R** – Graph of the experimental and theoretical reflectance.

- 1) This worksheet is used to look and see how good the fit is.
- 2) As usual make sure that the plot range as defined in source data is correct for the data set you are looking at.
- 3) Generally the amplitude of the theoretical fringes will be larger than the experimental ones. However, we do hope that the fringes line up with one another, if they do I believe it should be considered a decent fit.

XII) **gph-fit\_T** – Graph of the experimental and theoretical transmittance.

- 1) This worksheet is constructed simply so that one can see how well the calculated transmittance compares to the experimental.
- 2) Again, the plot range must be correct for the data set.
- 3) Generally this plot will look better than the reflectance plot, but remember that the transmittance spectra is not used to fit the data.

XIII) **calc-gap** – This worksheet does some simple calculations to help determine the band gap and absorption coefficient of the material.

- 1) The values to which these calculations are done come from the *reference\_page* worksheet as defined in the calc-index worksheet. In general this should be over

- the UV-visible range. If it is not you will need to change the ranges on the gph-alpha and gph-gap worksheets.
- 2) This data doesn't mean anything unless one has both reflectance, R and transmittance, T, experimental spectra in the reference worksheet.
  - 3) The d\_cm name is defined and is used here in these calculations. By default it is linked to the d\_nm1 name, but this can be changed to be any thickness value that you have for the film. Just be sure that the units are in centimeters.
  - 4) The alpha and wavelength data come straight from the calc-n\_fixed worksheet. The gap calculations are then done based on this.
  - 5) The gap calculations are for the gph-gap worksheet. There is nothing in this worksheet that says "this is the gap". Instead, one looks at the gph-gap worksheet and attempts to determine where the gap is (see the section on the gph-gap worksheet for more info.)

XIV) **gph-alpha** – Graph of the absorption coefficient,  $\alpha$ , as a function of energy.

- 1) This graph is an approximate graph of the absorption coefficient. There are two curves, but it is the same data, one is a linear and the other a logarithmic vertical scale.
- 2) As in all graphs, one should make sure that the source data plot range is correct. Furthermore, one should also be sure that the horizontal scale is correct.

XV) **gph-gap** – Graph that can be used in determining the band gap of a material.

- 1) This graph has two plots. One is used to determine the direct band gap, and the other to determine the indirect gap.

$$data = (\alpha(\lambda), E(\lambda))$$

A) The data for the direct gap plot is determined from the following equation:

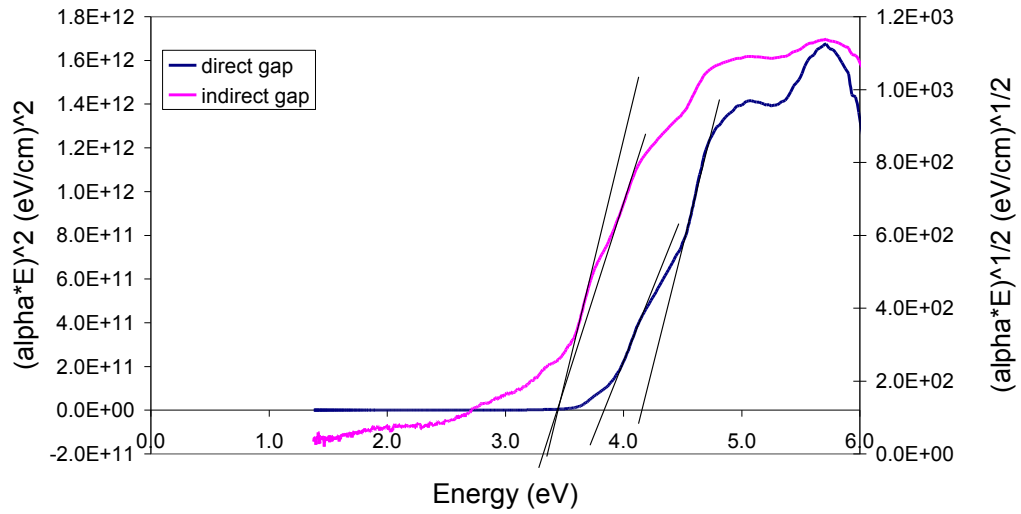
Where alpha is the absorption coefficient as a function of wavelength and E is the energy of a photon at that wavelength,  $E = h \cdot c / \lambda \approx 1240 \text{ nm} \cdot \text{eV} / \lambda$ .

B) The data for the second plot is determined from this equation:

C) Both of these plots are plotted as a function of energy.

2) In order to determine the gap of the material, one simply looks at the plot and attempts to see the regions where the data is linear. Then a line is drawn through this linear region, and the point where this line intersects the horizontal axis is considered the band-gap. Sometimes there is more than one linear region on each curve, and this may indicate more than one gap. Lines drawn based on the indirect gap data correspond to indirect gaps, and lines drawn for the direct gap data correspond to direct gaps. Here is a picture of these calculations being done:

sm16d CuScO2 - Gap



Interestingly in this case, the lines based on the indirect gap data seem to intersect at approximately the same point on the horizontal axis, whereas the lines drawn based on the direct gap data have two distinct intersection points.

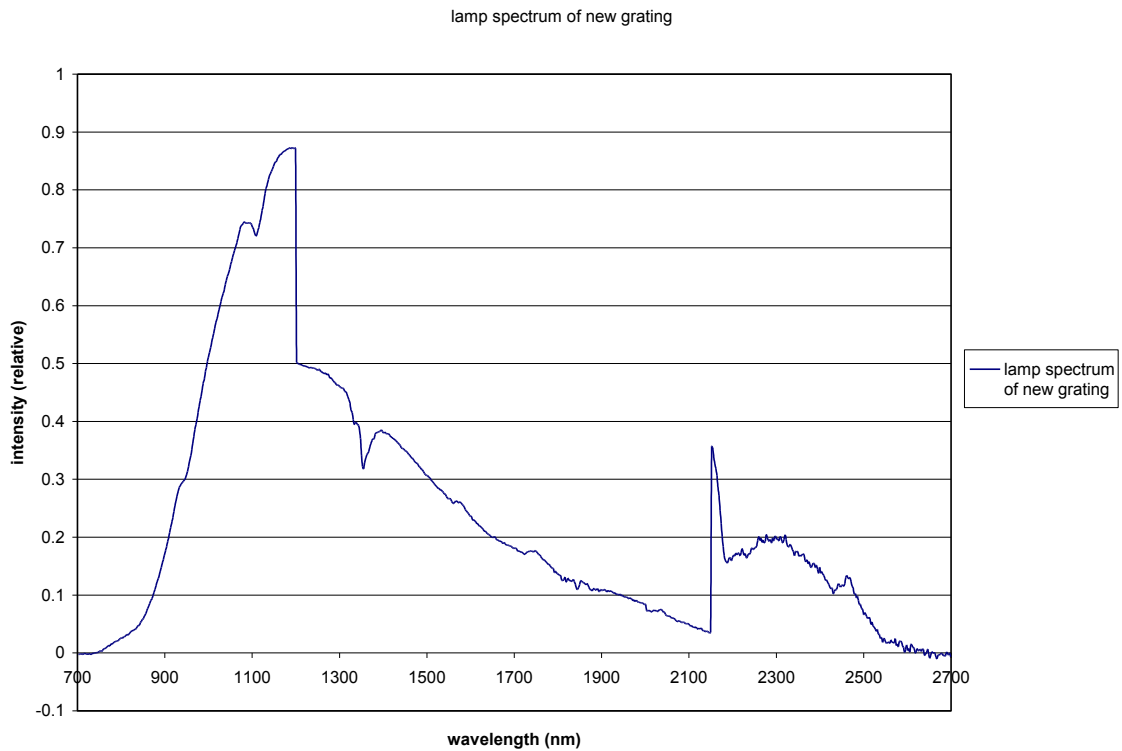
## Appendix C

### Getting one grating to work for the IR range from 0.8 to 2.6 $\mu\text{m}$

The problem here is that two gratings are currently required in order to maximize the wavelength range of the signal. The 1 $\mu\text{m}$  grating, as in Table 1, works well up to 2 $\mu\text{m}$  but will not go beyond this because of the mechanical limit of the monochromator – the monochromator will not go beyond 1000 increments, and the 1 $\mu\text{m}$  grating has a multiplier of 2 nm/increment. On the other hand, the 2 $\mu\text{m}$  grating has a 4nm/increment multiplier but provides no signal below approximately 1 $\mu\text{m}$ . Because the range in which we are interested is from 0.8 to 2.6  $\mu\text{m}$ , neither of these gratings appear to be sufficient. For this reason another grating was purchased with the hope that it would be able to span this range.

Unfortunately, this grating did not work as we had hoped. The root of this problem, I believe, comes from the curvature of this gratings reflectivity as a function of wavelength. A lamp spectrum of this grating from 7 to 2.7 $\mu\text{m}$  is shown in Fig. C-1. Because of the peak at 1.2 $\mu\text{m}$  narrow slits must be used in order to attenuate the signal enough that it doesn't overload the lock-in when the detector and lock-in gains are set at their minimum values. This has the adverse effect of making the signal (and therefore the S/N) detrimentally small at the far ends of this range. The fact that the lamp spectrum of the system in this configuration has a large peak, and the fact that the lock-in will overload is at the heart of the noise issue at the edges of the spectrum.





**Figure C-1:** Intensity spectrum of new grating from 700-2700nm. Notice the high peak at 1200nm where the 1.2 $\mu$ m filter is engaged. Also notice the jump up at 2150nm where the gain of the lock-in was increased. This was done with the hope that the noise was coming from the lock-in rather than the system or detector itself and that amplifying the signal would increase the signal to noise ratio. Unfortunately, this did not help and we conclude that the noise must be coming from another source.

One way to solve this problem is to increase the intensity of light reaching the detector at the long and short wavelength ends without increasing the intensity reaching the detector at the peak (1.2 $\mu$ m). Two ways of doing this are suggested here:

**Solution 1-1:**

Begin by placing wider slits in the monochromator. Then program the IR\_scan.vee program to stop at some wavelength, say 1000nm, before the signal becomes too large and insert narrower slits. Now let the program scan over the peak, and then have it stop again at say 1500nm where the wider slits can be reinserted. The problem with this is that it is not much better than using two gratings because you still have to

manually exchange components of the monochromator. On the other hand, this could be a cheap way of determining if solution 1-2, below, could work.

**Solution 1-2:**

Put in a filter that lowers, but does not reduce to zero, the intensity around 1.2 $\mu$ m while leaving the extremities of this range relatively unaffected. Wider slits could then be used without overloading the detector.

Another way to solve this problem would be to:

**Solution 2:**

Purchase a lock-in that is capable of handling higher signals and use wider slits in the monochromator. A lock-in capable of handling higher signals would not overload at the peak if wider slits were used, but should also be capable of detecting the small signals at the far ends of this range.

A final, more complicated solution entails the use of the 2 $\mu$ m grating that had been used previously.

**Solution 3:**

Use the second order light of the 2 $\mu$ m grating. In the first order, the 2 $\mu$ m grating doesn't provide any signal below  $\sim$ 1100nm. However, the 2 $\mu$ m grating does have a decent reflectivity of second order light from  $\sim$ 1600nm-2700nm, but this is actually light of wavelengths from 800nm to 1350nm.

The idea here would be to use a filter that blocks light above 1600nm while scanning the 2.0 $\mu$ m grating from 1.6 $\mu$ m to 2.4. This would effectively use the second order light that exists in this region for this grating to get data on the film from 800nm-1.2 $\mu$ m? The thing that remains to be determined is whether the intensity of the second

order light is high enough at 1600nm (actually 800nm). Conveniently, the third order light (if it exists) would be blocked by the filter on the front of the detector (which blocks light below 800nm) as long as we don't use this method above 2.4um. If the filter that blocked light above 1600nm were in the filter wheel this method would allow us to scan the entire IR range of interest without changing anything in the box.