T10

Semiconductor

Spectroscopy is the study of electromagnetic spectra and can be used to interrogate allowed energy transitions and the band structure of many material types. The first part of the lab will introduce you to spectroscopic equipment and give you an opportunity to explore the relationships between the visible wavelength ranges and our perception of colour. In the second part of the lab you will use the transmission spectra semiconductors to extract values for film thickness and bandgap energy, and of 'doped' glasses typical of those used in lasers and fibre optic amplifiers.

Schedule

Preparation time	:	3 hours
Lab time	:	3 hours

Items provided

Samples	:	colour filters, semiconductor samples, rare earth doped and thin film coated glass.
Safety equipment	:	Nitrile gloves
Equipment	:	Ocean Optics Spectrometer (USB2000) and LS1 Tungsten Halogen Light Source
Software	:	Spectrasuite

Items to bring

Essentials. A full list is available on the Laboratory website at
https://secure.ecs.soton.ac.uk/notes/ellabs/databook/essentials/

Before you come to the lab, it is essential that you read through this document and complete *all* of the preparation work in section 2. If possible, prepare for the lab with your usual lab partner. Only preparation recorded in your laboratory logbook will contribute towards your mark for this exercise. There is no objection to several students working together on preparation, as long as all understand the results of that work. Before starting your preparation, read through all sections of these notes so that you are fully aware of what you will have to do in the lab.

> **Academic Integrity** – *If you undertake the preparation jointly with other students, it is important that you acknowledge this fact in your logbook. Similarly, you may want to use sources from the Internet or books to help answer some of the questions. Again, record any sources in your logbook.*

Revision History

September 12, 2012   Darren Bagnall (dmb)   First version of this lab January 10, 2014

1   Aims, Learning Outcomes and Outline

This laboratory exercise aims to:

- Provide a practical introduction to spectroscopy
- Explore the relationships between electromagnetic spectra and colour
- Investigate the transmission spectra of semiconductors
- Explore the spectra of glass containing 'rare earth' atoms used in many lasers
- Provide an opportunity to extract parameters (such as semiconductor bandgap or film thickness)from transmission spectra

Having successfully completed the lab, you will be able to:

- Apply appropriate laboratory techniques to measure semiconductor properties
- Develop analytical approaches to understanding complex physical systems

Allowed energy transitions within materials determine the optical transmission, reflection and absorption characteristics of those materials. Spectroscopy is the study of electromagnetic spectra and can be used to interrogate the energy transitions and the band structure of materials. This lab aims to introduce you to spectroscopy and semiconductor spectroscopy.

The first part of this lab provides a simple introduction to the spectrometry apparatus; you will use this apparatus to first obtain transmission spectra for some simple optical filters with different transmission spectra.

For the second part of the lab you will be provided with a series of semiconductor thin film samples. You will obtain the transmission spectra for each type of sample and use your data to determine the semiconductor thickness and bandgap energies.

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2   Preparation

Read through the course handbook statement on safety and safe working practices, and your copy of the standard operating procedure. Make sure that you understand how to work safely. Read through this document so you are aware of what you will be expected to do in the lab.

2.1   The Nature of Colour

Using the Web or another resource, obtain a CIE (the International Commision on Illumination, 1931 see http://en.wikipedia.org/wiki/CIE_1931_color_space) colour triangle, and research its use.

> ⬦ *Why are the three primary colours significant to human vision, and how does a plasma screen (for instance) achieve a full-colour output?*

> ⬦ *Why is it that when you mix red, green and blue light you produce white light, but if you mix red, green and blue paint you get black?*

2.2   USB2000 and Spectrasuite

Read through the Appendix 1 (page 8) to gain insight into the equipment and software you will be using. ***This is particularly crucial to making good use of your time in the laboratory.***

2.3   Transmission Spectra

Read Appendix 2 (page 10) and any other suitable material you can find, to familiarise yourself with the principle features of absorption within semiconductors and the characteristics of semiconductor transmission spectra.

◇❓   *What is the Beer-Lambert formula?*

2.4 Film thickness determination

The transmission and reflection spectra of thin film materials often exhibit interference fringes. These fringes are the result of the phase differences that exist between light reflected from the front and back surfaces of the films. Taking into account the physics of the interference of light derive a methodology that will allow you to estimate the film thickness from the interference fringes observed in <u>transmission</u> spectra. **Note:** you will be provided with suitable values of the refractive index values of the films. You may find http://en.wikipedia.org/wiki/Fabry%E2%80%93P%C3%A9rot_interferometer useful – note that the 'bull's eye' pattern shown here and in many references is only seen in strongly diverging light beams.)

◇❓   *When do these 'Fab<u>r</u>y-Perot' interference fringes occur; what is the relationship between film thickness, refractive index, and fringe spacing?*

2.5 Bandgap energy determination

Using material provided in appendix 2 derive a methodology that will allow you to determine the bandgap of a thin film semiconductor sample. You will need to use transmission values obtained as a function of wavelength to extract values for the absorption coefficient as a function of energy (using the Beer-Lambert formula.) To correct for the effect of reflection, re-scale your transmission values so that in the region where it transmits, the value is 100%, as in this region there is only reflection and no absorption..

◇❓   *What exactly is meant by the 'bandgap'; what is the difference between 'direct' and 'indirect' bandgaps?*

2.6 The location of discrete energy levels.

The obscure 'rare earth' elements (lying between Lanthanum and Lutetium in the sixth row of the periodic table – and much loved by Tom Lehrer ☺ http://en.wikipedia.org/wiki/The_Elements_%28song%29) added at low concentration, typical ~1%, to glasses and crystals have well defined discrete energy levels characteristic of the particular element. These materials are crucial to modern lasers and Internet fibre optic amplifiers. You will use the measured absorption spectrum to locate the energy levels, and if possible identify the particular element used. Obtain a brief idea of the devices these materials are used for, the absorption spectra of such materials and how 'energy level diagrams' are drawn for them (on the web). (Note that you do *not* need to understand the rather complex labelling used for these levels.)

◇❓   *What are the 'rare earths'; what are they used for; can you find an energy level diagram for them?*

3   Laboratory Work

Having made sure that you have read and understood how to use the spectrometer and light source your first task is to establish that your spectrometer is functioning correctly (Appendix 1)

and understand some of its requirements and limitations. Time spent ensuring that you really understand the spectrometer will pay off later; do not be tempted to start looking at samples until you feel confident about this.

You should then proceed to optimise the apparatus settings (integration time, boxcar and average). Optimised systems should provide data with appropriate resolution, high signal to noise ratio across a suitable range. Most of these features will benefit by taking and average of many measurements taken with long integration times, but too much integration will lead to saturation of the signal and in the context of this lab you don't really want to wait too long between measurements because conditions may 'drift'.

Once you are happy with your apparatus settings you should take your light reference by allowing light from the source to enter the spectrometer with nothing in the sample holder. Take the dark reference in a similar manner with the light source (briefly) turned off.

Note: You should not use the printer during this lab, instead you should sketch the spectra you obtain in your log book and save the data from the spectrometer electronically. The act of sketching the spectra will help you to consider and highlight the principle features you observe.

3.1   Colour filter transmission measurements

The first sample you will measure is simply a piece of colourless glass. Within the visible region and nearby infrared and ultraviolet, it does not absorb light, but its transmission is not quite 100%. Why is this? What optical property can be roughly deducted from the observed transmission, and what value do you obtain? Does it vary with wavelength?

Proceed to measure the transmission spectra of at least 3 of the colour filters provided.

Sketch the spectra you obtain. Using the CEI colour chart correlate the spectra you obtain to the colour you perceive. Consider how the colour correlates with your measurement.

(Over time the temperature of light source, the spectrometer and the electronic systems you are using will change, these changes will require occasional re-calibration of light and dark reference levels. You should always carefully recalibrate light and dark reference levels before taking important results).

3.2   Semiconductor transmission measurements

Two semiconductor samples are provided, these samples are labelled by type and sample number. Take samples one at a time from the collection point and return them immediately after use. They are quite fragile, quite expensive, and in one case somewhat toxic; please do not chew the samples ☺ . The toxic one is a hard solid and will not easily transfer material to you; but it as an additional precaution wear the nitrile gloves provided when handling them, only touch the metal sample holder, not the semiconductor itself, and it is good practise to wash your hands afterwards, and not eat before doing so. Furthermore fingerprints destroy samples (they can also be used to track you down!)

You should take accurate noise-free transmission data for each semiconductor sample type. For each sample type sketch the transmission characteristic and estimate the bandgap energy of the semiconductor.

An accurate calculation of the bandgap energy, will require good data in the region of very low-transmission close to the band-edge. You may wish to increase the sampling intensity of the signal in this region (even if this means that other parts of the spectra are beginning to saturate).

Once you are satisfied with the data being collected, copy it to Excel (or any program you find useful) and store the data. You can do this by using the '**Copy Spectra Data**' and '**Copy Graphical Data**' buttons in the toolbar. Make sure you label the data accordingly.

3,3 Discrete energy levels.

Measure the spectrum of the rare earth element doped glass (pink-ish in colour.) Locate the discrete energy levels, and draw a diagram of them with energies in electron volts, eV. These energy levels are highly characteristic of the rare earth concerned – using web sources, can you identify which element it is?

3.4 Thin films.

Two samples of thin film niobium pentoxide, Nb_2O_5 (n=2.42 at 500nm, falling to 2.31 at 800nm), on glass (n=1.5) are provided with different thickness. Glass and niobium pentoxide should not absorb over the wavelength range of your measurements, but you may see some evidence of absorption due to manufacturing difficulties.

3.5 Data extraction

Using spectral data from one sample and using the methodologies devised in your preparation:

1) Use interference fringes from data in the long-wavelength region to estimate the film thickness

2) Use data in the low-transmission region to calculate the bandgap value

Finding a straight line in α^2 data is famously difficult, you will always have more absorption than you should at low energies and less than you should at high energies, so your "straight line" will be curved at both ends. A short straight-ish region somewhere in the middle is normally the best you can do.

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4   Optional Additional Work

Marks will only be awarded for this section if you have already completed all of Section 3 to an excellent standard and with excellent understanding.

Measure the very thin glass sample; with care interference fringes can be seen, most clearly in the 800-900nm region. If n=1.5 how thick is the glass? Why are the fringes most easily seen at the longest wavelength available?

Measure the spectrum of the so-called 'didymium' glass. There is actually no rare earth element called didymium! You will easily find on the Internet that it is a mixture of two real elements; can you identify which absorption features belong to which element? Can you draw energy level diagrams for the levels you observe for those elements?

A variety of thin film semiconductor samples are available. These give more complex spectra, showing both band-gap and interference effects. Measure two or three, select one in which both effects are clear, and try to derive both film thickness and band gap value for that sample.

Appendix 1: Using SpectraSuite

The USB2000 spectrometer and LS1 Tungsten Halogen Light Source can be used together to take measurements of various spectra in the range of wavelengths between 400nm and 1000nm.

The light source must be handled with extreme care. Once hot, the light source and its casing must be handled with care to avoid burns.

The spectrometer connects to your PC via a USB port, and draws power from the PC, eliminating the need for an external power source. Light enters the spectrometer, passes through a filter which selects the 400nm-1000nm range, then reflects off a mirror towards a diffraction grating. This diffraction grating separates the incident light into separate colours, similar to the effect seen when white light is dispersed by a prism. The light is then focused onto a CCD detector via another mirror, which converts optical data into electrical and then sends it to the PC, enabling the light to be viewed as a spectrum on the screen.

FIGURE 1: The USB2000 spectrometer

FIGURE 2: The LS1 Tungsten Halogen Light Source

In order to set up the equipment, plug in the spectrometer to the USB port on the PC. Then in the start-up menu navigate to the EQUIPMENT folder, then the OCEAN OPTICS folder and run the **SpectraSuite** program. If everything is connected and working correctly, you should see a signal on the screen similar to that shown in Figure 3. If not, make sure that everything is connected and restart SpectraSuite.

In order to understand how to operate SpectraSuite, we need to know how it calculates transmission.

The software calculates the amount of light a sample absorbs at wavelength λ using the following equation:

$$A_\lambda = -\log_{10}\left(\frac{S_\lambda - D_\lambda}{R_\lambda - D_\lambda}\right) \tag{4}$$

and the percentage of light transmitted through a sample at wavelength λ by

$$\%T_\lambda = 100 \text{ x}\left(\frac{S_\lambda - D_\lambda}{R_\lambda - D_\lambda}\right) \tag{5}$$

S_λ is the sample intensity at wavelength λ, D_λ is the dark intensity at wavelength λ, and R_λ is the reference light intensity at wavelength λ.

Thus we need to make three measurement:

Firstly, with the light beam blocked, D_λ; block the light beam, close the lid, and press the 'black lightbulb' to collect the data. D_λ results from leakage currents in the detector and any stray light hitting it.

Secondly, R_λ, the signal with the light totally transmitted. Unblock the beam, close the lid, and press the 'yellow light bulb' to collect data.

> ⟨?⟩ *What causes the shape of the Rλ curve, its variation with wavelength? What is the influence of the silicon CCD detector sensitivity and the light bulb, whose filament temperature is ~2800K?*

These only need to be done once for a series of samples; but if *any parameter is changed* (integration time, averaging, boxcar) or if a long time has elapsed (due to signal drift) *they must be collected again.*

Finally A_λ or T_λ are collected by pressing the appropriate button.

Note: The method that Spectrasuite uses to calculate absorption makes the assumption that any light which isn't transmitted is absorbed, thus ignoring reflection. However, reflection is significant in some cases.
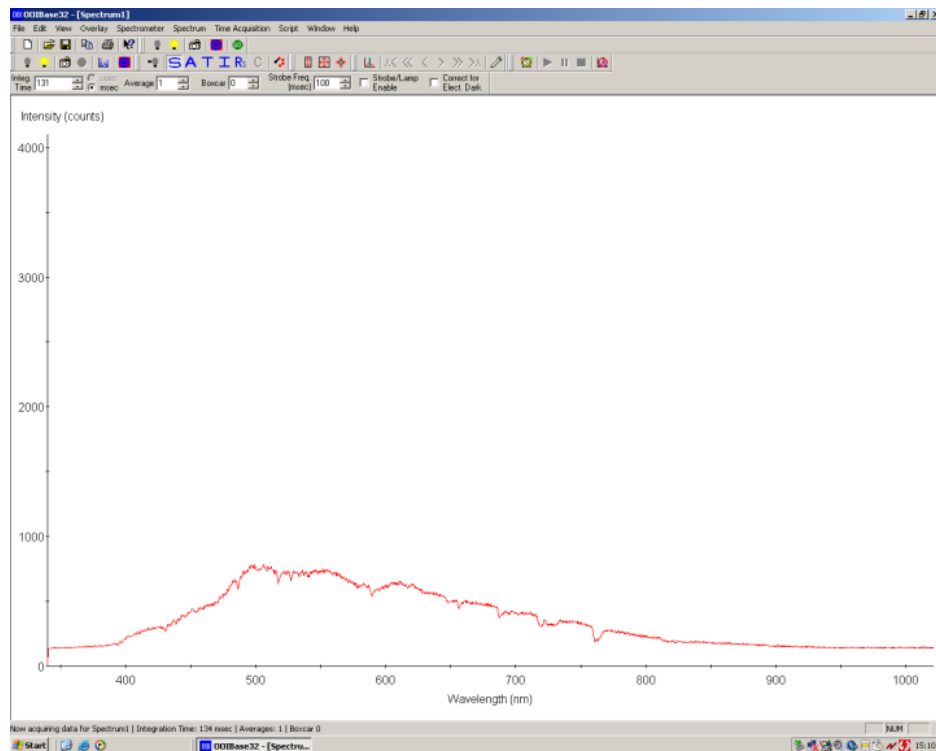

FIGURE 3: Expected graph obtained on start-up OOIBase32

If the spectrum similar to the one above on your screen appears to be saturating (flat top to the spectra), decrease the integration time (top left hand corner). If it is very small, increase the integration time. A peak intensity of below 3500 counts is best for good measurements.

Adjusting the variables labelled "Boxcar" and "Average" will smooth out your waveform, gradually eliminating the spectral noise you may see on the screen – but reducing the detail that can be seen.

"Boxcar" works by taking a number of wavelengths close to each other at any given time and taking the average of those wavelengths to produce the spectrum. It is best to keep "Boxcar" under thirty for 'smooth' spectra, if it is any higher the spectral resolution of the system will be reduced. We recommend using 2 for the value of Boxcar to begin with. Be especially cautious about using too high a 'boxcar' setting when measuring the rare earth doped samples, or the thin glass sample, whose interference fringes are fine.

"Average" takes an average of many spectral readings over a period of time before producing its results. It is best to keep this value under twenty so the spectra regularly "refreshes" itself,

otherwise it can become time consuming. An average of 10 should be sufficient to begin; unlike 'boxcar' there is no loss of data quality – it just uses more time.

Some common sense should also be used in taking these measurements, for instance, in transmission mode, you should be cautious of any values over 100% or below 0% in either case you probably need to reset your reference levels.

Before starting to use the device, look at the signal S_λ and T_λ obtaining with no sample. It should be a flat 100%. Is it? What is happening below ~450nm and above ~950nm and why?

Try opening the cover of the instrument and observing the T_λ with no sample; once again it should be a flat 100%. Is it? What is happening here?

Appendix 2: Estimating the Semiconductor Bandgap

Most of the common features of semiconductor transmission spectra can be seen in figure 4.
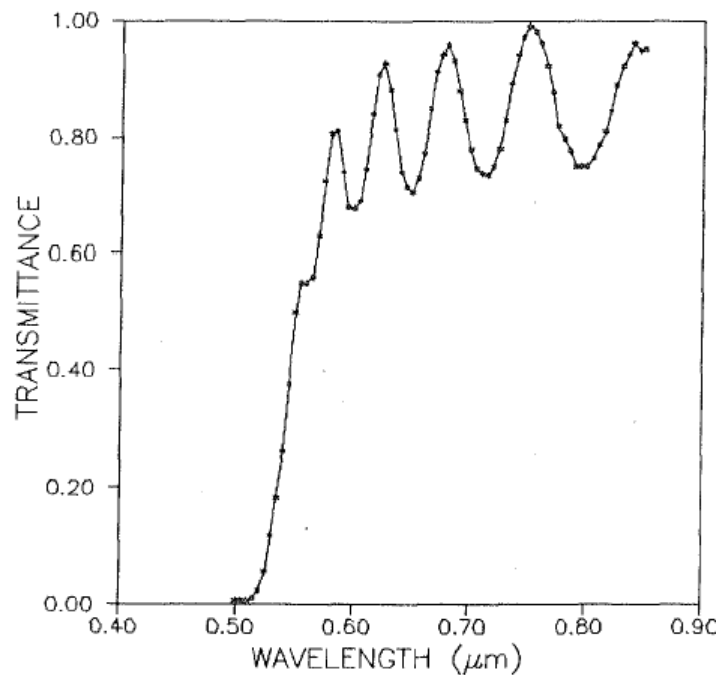

FIGURE 4: Example of the transmission spectrum from a thin film semiconductor sample

The transmission spectra show two distinct regions, a long-wavelength region where the photon energy is below the bandgap and where there is relatively high transmission (low absorption), and a short-wavelength region where the photon energy is much greater than the bandgap energy and most of the light is absorbed. Thus, the bandgap of a semiconductor can be readily estimated.

Transmission spectra can also show interference fringes (figure 4).

An accurate bandgap value can only be obtained by plotting the square of the absorption coefficient (α) against photon energy and extrapolating a straight line. For a direct band gap semiconductor:

$$\alpha = B(h\nu - E_g)^{\frac{1}{2}}$$

where B is a constant, approximately equal to 3×10^4 cm^{-1}. For an indirect semiconductor, the equation is:

$$\alpha = B(h\nu - E_g \pm E_p)^2$$

as phonon energy (E_P) must be accounted for. Adding phonon energy accounts for phonon absorption, subtracting for phonon emission.

Using the above equations, it is possible to calculate the band gap of any semiconductor. You should attempt to form a $y = mx + c$ plot, where c is $-E_g$. To do this, you should rearrange the equations, obtain suitable x and y values, plot them, and then obtain an approximate y-intercept using a line of best fit.

$$\alpha = B(h\nu - E_g \pm E_p)^2$$

as phonon energy (E_P) must be accounted for. Adding phonon energy accounts for phonon absorption, subtracting for phonon emission.