EPFL

MSE-215

Mise en œuvre des matériaux II

Spring 2022

TPG – Optical properties

Optical properties of materials: refractive index, absorption, polarization, scattering

On the left is a picture illustrating an interesting property of certain materials: ***birefringence*** *(picture from* *<u>what-is-this.net</u>, downloaded 08.03.2021). On the right, you can see several* ***optical coatings****: thin layers of a variety of materials, deposited onto an optical surface (lens, mirror, etc.) in order to play with the* ***transmission or reflection of light*** *(adapted from <u>CoatingPaint.com</u>).*

At a glance

The aim of this TP is to provide a basic knowledge of material parameters that are important for material analysis, applications and measurement systems. You will treat for subjects:

1. Transmission and absorption
2. Polarization and birefringence
3. Refractive index and total internal reflection
4. Light diffusion of ceramics and grain size determination

For each part, you will follow the tasks by reading this material as well as running the experiments. In the discussion, you should comment your results and add possible sources of error.

The duration is planned for 2h.

1. Transmission and absorption

Objective & concepts

Measure transmission properties of colored plastic sheets and confirm Beer-Lambert law.

- Measure transmission of clear but colored materials with LEDs.
- Plastic of different thickness piled up to calculate absorption and see exponential law.

Introduction

Transmission coefficient

For the case of normal incidence there is no distinction between polarizations. Let n_1 and n_2 be the refractive index on each side of the surface. Thus, the reflectance is given as

$$R = \left|\frac{n_1 - n_2}{n_1 + n_2}\right|^2$$

If there is no absorption A of the material the transmission T is then given as

$$T = 1 - R$$

For common glass (n_2 ≈ 1.5) surrounded by air (n_1 = 1), the power reflectance R at normal incidence can be seen to be about 4% for single side, or 8% accounting for both sides of a glass pane.

Adapted from Wikipedia

Beer-Lambert law

The Beer Lambert law is combined of two laws and each are correlates which state that the absorbance of light is proportional to the thickness of the sample; or absorbance is proportional to the concentration of the sample.

The Beer–Lambert law […] relates the attenuation of light to the properties of the material through which the light is travelling. The law is commonly applied to chemical analysis measurements and used in understanding attenuation in physical optics, for photons, particles in suspension, neutrons, or rarefied gases.

Beer-Lambert law:

$$I = I_0\, e^{-\alpha D}$$

with D the thickness of material, α the absorption coefficient and I_0 the intensity of the light without material.

The intensity can be expressed in any value and usually measured signal of the optical detector is taken. The transmission can be calculated using the intensities as

$$T = \frac{I}{I_0} = e^{-\alpha}$$

Measuring the transmission will give the possibility to determine α the absorption coefficient. The signal for I_0 is without sample and signal I would be with sample introduced.

Adapted from Wikipedia

Experiment example

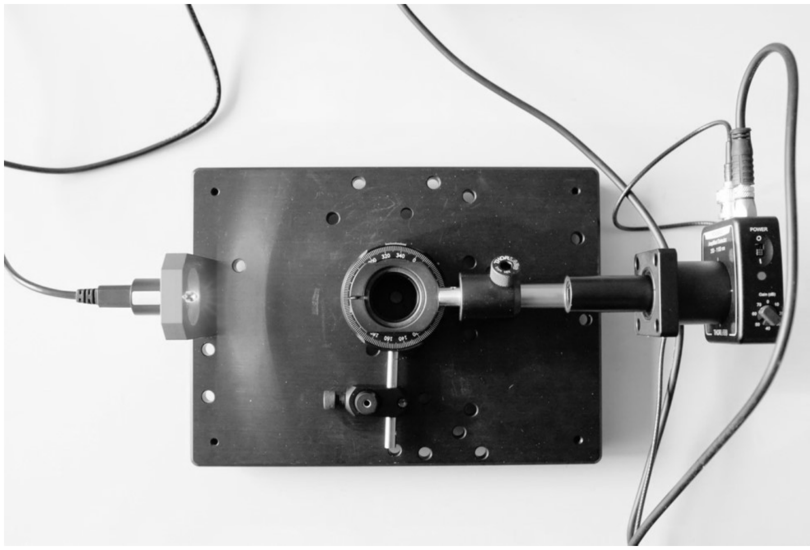
Video link: **https://youtu.be/aTBBD0X8fBc**
(or search on YouTube: MSE215 TP1)

For the following tasks (Tasks one and two) an example is made available in the video TP1. Please read the description below first to get an idea of what you are going to do, run the experiment, and fill out tables and discussions with the values you found during the TP.

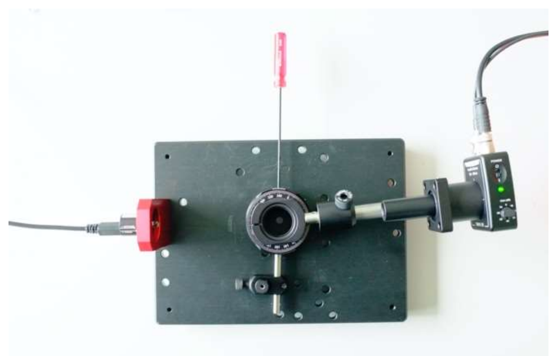
Task one Wavelengths dependence of transmission

- Mount the system as shown below.
- Use the red LED and connect it to the USB power supply.
- Connect the detector to its power supply (+-12 V) and the handheld multimeter.
- Switch all supplies on.
- Choose DC voltage measurement on the handheld multimeter.

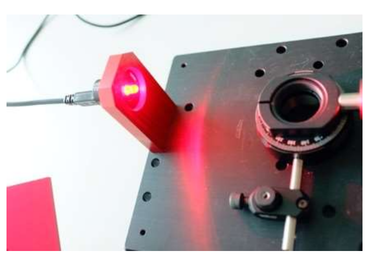
The detector amplifier provides voltages up to 10 V. The detector can easily saturate and adjustment of the AMPLIFICATION is needed for proper operation. The small rotational knob on the side of the detector (dB scale) allows you choose am amplification so that the detector measures a signal without saturation.

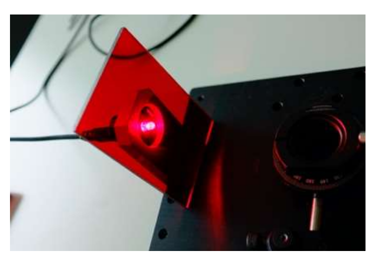


To avoid rotation the mount can be blocked using the small ball driver as shown below.



- Set the detectors amplification to a value so that the multimeter shows a value below 10 V. Try to be as close to 10 V as possible.
- Switch off the light source and check that there is no signal (<40mV).
- Switch the source on and note the values of signal for different cases as given in the table using the color plastic sheets provided. You can put them in front of the source.





Please indicate the color of your LED:

Blue (465 nm) Green (528 nm) Red (635 nm)

Please fill out the following table:

$D = 3\ mm$

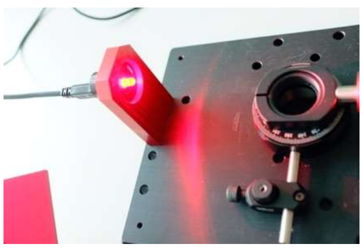
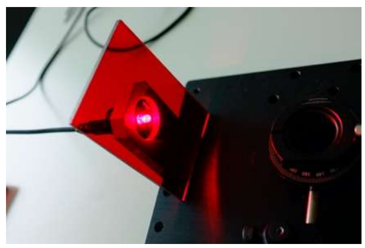
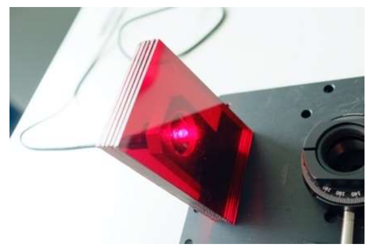
Plastic sheet	**Signal** [___]	**Transmission** (signal I with plastic sheet divided by signal I_0 without plastic sheet)	**Absorption** [___] $\alpha = -\frac{1}{D}\ln\left(\frac{I}{I_0}\right)$
None		1	0
Blue			
Green			
Red			

Discussion:

Task two Beer-Lambert law

To do:
1) Use the red LED and the red plastic sheets
2) Measurement for different thickness (0, 3, 6, 9, 12, 15 mm thickness, 0-5 plates)
3) Calculate the coefficient
4) Consider correction of surface reflection

- Use the same setup and measure for different numbers of plastic sheets.
- For red LED you should use the red plastic sheet.
- Fill in the table with your results and calculate an average absorption coefficient.
- The thickness of the plastic sheet is assumed to be 3 mm.

Please fill out the following table:

Number of plastic sheets	**Effective thickness** D [___]	**Signal** I [___]	**Transmission [___]** $\frac{I}{I_0}$	**Absorption [___]** $\alpha = -\frac{\ln(T)}{D}$
0	0		1	0
1	3			
2	6			
3	9			
4	12			
5	15			

Discussion:

Comment on possible errors of the measurement:

2. Polarization and birefringence

Objective & concepts

Understand the operation of dichroic polarizers and measure the birefringence of plastic sheets.

- Measure the transmission for different angles between two polarizers (Malus's law)
- Determine the retardation of scotch tape – glue several layers parallel on a plastic support and compare the color between crossed polarizers with retardation sheet
- Take photos of an injection molded plastic part under crossed polarizers and different rotations. Explain what you see.

Introduction

Malus Law

Malus law, which is named after Étienne-Louis Malus, says that when a perfect polarizer is placed in a polarized beam of light, the irradiance I of the light that passes through is given by

$$I = I_0 cos^2\theta$$

where I_0 is the initial intensity and θ is the angle between the light's initial polarization direction and the axis of the polarizer.

Adapted from Wikipedia

Birefringent colors

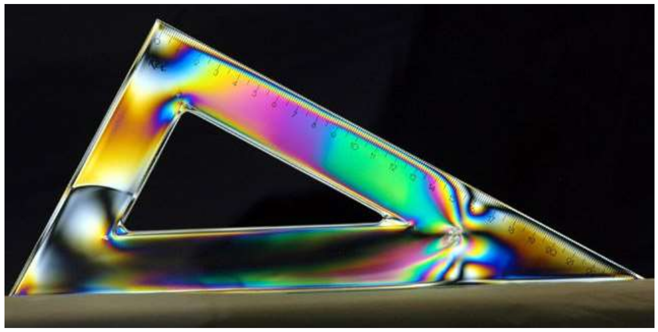
Birefringence is the optical property of a material having a refractive index that depends on the polarization and propagation direction of light. These optically anisotropic materials are said to be birefringent. The birefringence is often quantified as the maximum difference between refractive indices exhibited by the material. Crystals with non-cubic crystal structures are often birefringent e.g. alumina ceramics, as are plastics under mechanical stress.

Transmission T through a birefringent slab with thickness D and birefringence $\Delta n(\lambda)$ as a function of wavelengths λ between crossed polarizers and held at 45° with respect to the optical axis:

$$T = sin^2 \frac{\pi \Delta n(\lambda)\, D}{\lambda}$$

This causes the appearance of color and allows us to judge the factor $\Delta n \cdot D$ which is called RETARDATION.

As an example, we show an image of transparent plastic between crossed polarizers (that is why the background is black). The piece has a distribution of optical axis, thickness variations and birefringence distribution according to the internal stress and creates a multitude of colors.



Picture from https://www.pinterest.co.uk/pin/320248223475892216/, downloaded 13.01.2010

Under known experimental conditions he colors can be used to determine the retardation or birefringence of an object.

Adapted from Wikipedia

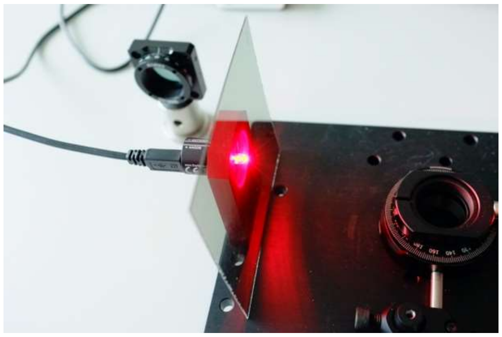
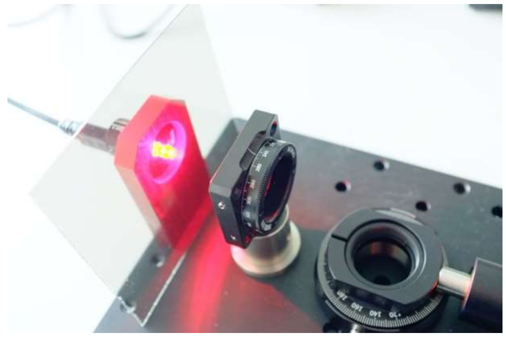
Task one Confirm Malus law

Experiment example

Video link: https://youtu.be/K9vM-rNSgP4
(or search on YouTube: MSE215 TP2)

For the following task an example is made available in the video TP2. Please read the description below first to get an idea of what you are going to do, run the experiment, and mark the graph with the values you found during the TP. The results for this task can be taken from the video TP2, if time left does not allow you to run the practical experiment.

- Use the same setup as given in task one and mount the RED LED.
- Put a sheet polarizer (grey plastic sheet) in front of the LED.
- Adjust the amplification of the detectors amplifier to assure high signal but without saturation (signal below 10 V).

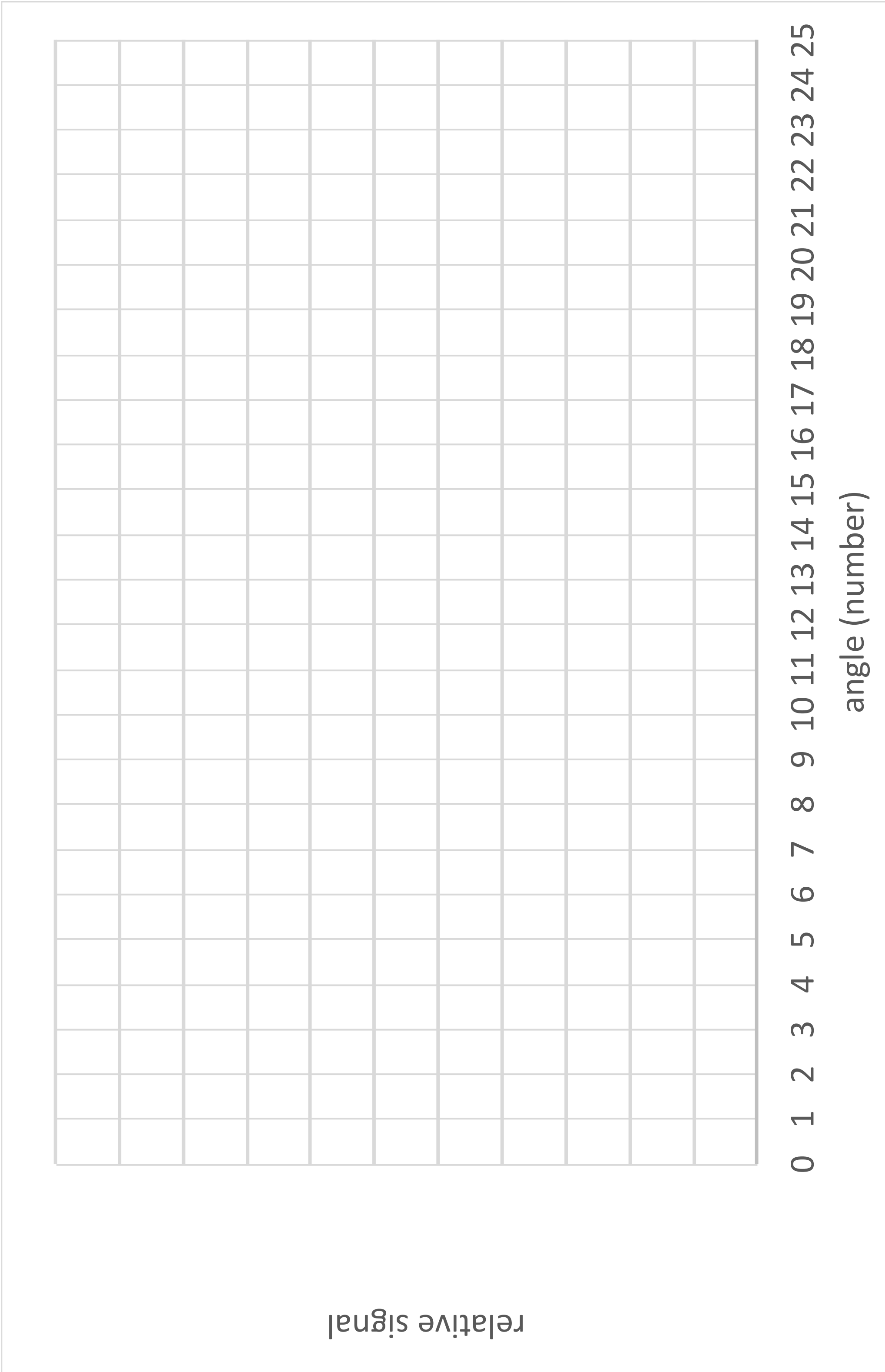
 

- Place the polarizer in the rotational point in front of it.
- You can rotate the polarizer at high precision and read the angle.
- Adjust the position of the rotational polarizer to the minimum intensity and read then first angle and note the value.
- Be careful that your detector is not saturated (Value smaller than 10V).
- Measure the signal values for every 10° rotation over a cycle of 200 degrees and mark it directly in the graph below (see the next page!).

Discussion:

Compare your graph with the theoretical curve of $\cos^2$!

Title:

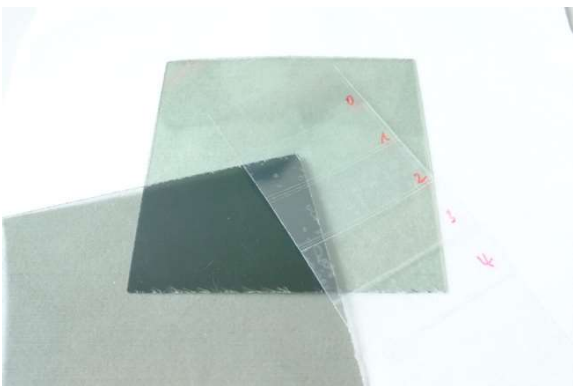
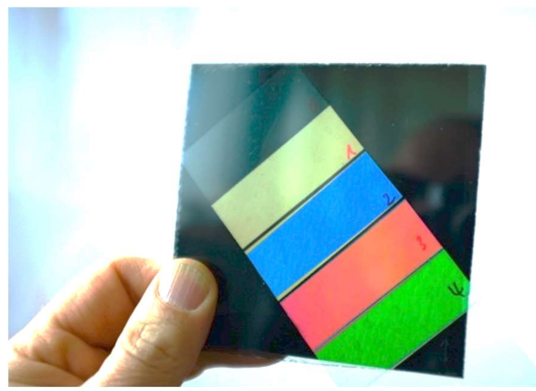

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25
angle (number)
relative signal

Task two Determine the retardation of scotch tape

Due to fabrication auto-adhesive tape is very often birefringent. It is an effect coming from the stretching of the material which leads to orientation of the polymer chains and furthermore a structural anisotropy. When observed between crossed polarizers under 45° with respect to the orientation colors appear. To fabricate a test sample one can proceed as following:

- Use a non-birefringent substrate (transparency from laser printer) and one layer of tape on it.
- Glue a second and a third layer with the same direction so that the tape partially overlaps.
- Apply one tape layer in an ORTHOGONAL direction over all layers.

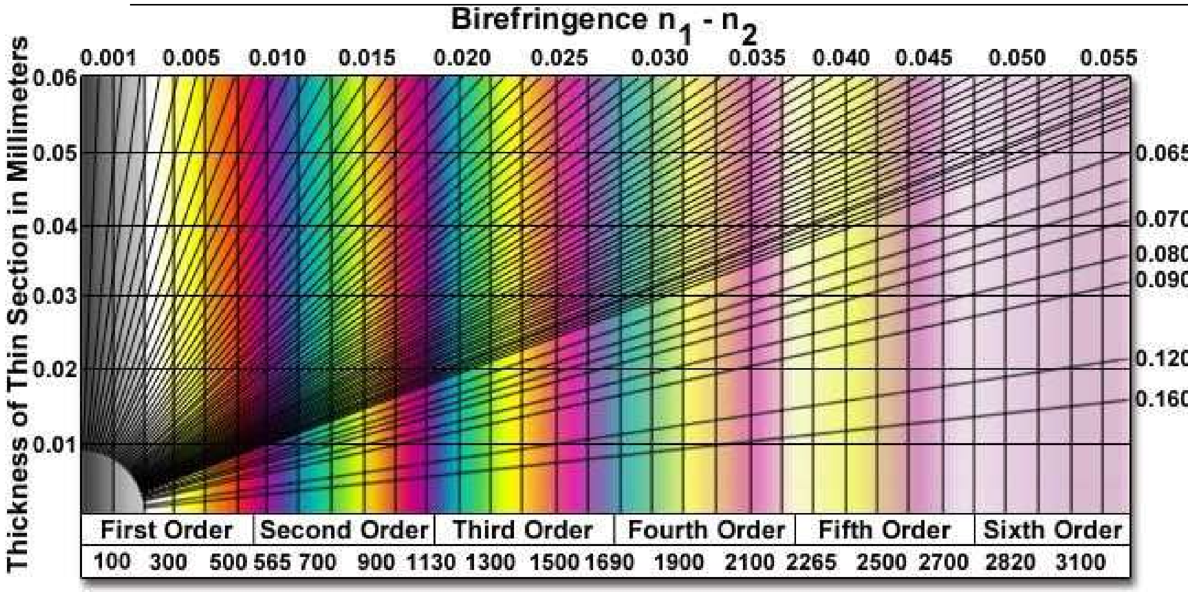
Your sample might look like the picture shown below when observed with a single polarizer and gets colored when put between crossed polarizers. Let’s utilize this picture to perform the following analysis.

Birefringence colors can be calibrated. With the help of the chart below one can determine the retardation of a single tape layer of your sample. The values are given in the lower column below the orders! Note that each layer adds the same retardation (as it has the same thickness) and so by adding a tape layer one moves exactly the same distance on the x-axis of the chart. For instance if you have a yellowish appearance for one layer (300) the next one will be blueish (600) and then orange (900).

The samples are already prepared and you should proceed as follows:

- Identify the layer without birefringence (is black between crossed polarizers).
- Compare the color of the different layers with the color chart below.
- Mark the color for the first, second, third layer etc.



The chart above is done for crystals and used as follows. One identifies a color between crossed polarizers and at axis orientation 45°. One knows the thickness (scale here 0-60 micron) and looks for the intersection of color and thickness (horizontal line). The intersections point will be close to an inclined line. Each of this included lines have a birefringence attributed to it as written on the right and on top of the graph. This allows to find the retardation of the sample. Birefringence multiplied by the thickness D of the sample gives the retardation $\delta = \Delta n \cdot D$ and is constant. So if we want to use the chart for larger thicknesses we need rescaling. Means, if for the same color and 60 micron thickness we have a birefringence of 0.01 we will find for 600 micron thickness 0.001 as birefringence. The product is constant for the same color.

In our case **a single layer of the sample is approximately 200 micron thick**. You can look at 0.02 at the thickness of section, find the intersection slope at you color and you need to **divide the birefringence value by a factor of 10**.

Birefringence of the scotch tape: $\Delta n =$

You can **control your result** for the different colors and layer thickness that are available on your sample.

Discussion:

3. Refractive index and total internal reflection

Objective & concepts

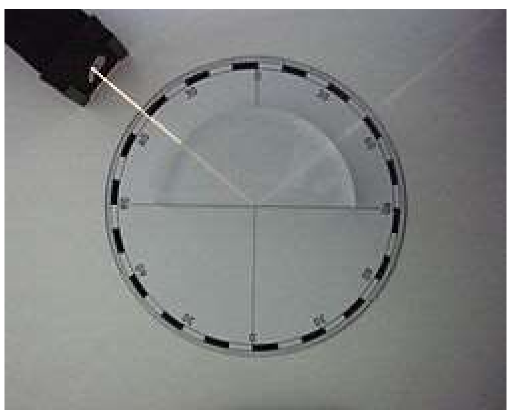
Measure the refractive index of plastic.

- Total internal reflection is used to find the limiting angle of total internal reflection. The angle allows direct calculation of the refractive index.

Introduction

Total internal reflection

Total internal reflection of light can be demonstrated using a semicircular-cylindrical block of common glass or acrylic glass. In the figure below, a "ray box" projects a narrow beam of light (a "ray") radially inward. The semicircular cross-section of the glass allows the incoming ray to remain perpendicular to the curved portion of the air/glass surface, and thence to continue in a straight line towards the flat part of the surface, although its angle with the flat part varies.



Total internal reflection. Picture from Wikipedia.

Where the ray meets the flat glass-to-air interface, the angle between the ray and the normal to the interface is called the angle of incidence. If this angle is sufficiently small, the ray is partly reflected but mostly transmitted, and the transmitted portion is refracted away from the normal, so that the angle of refraction (between the refracted ray and the normal to the interface) is greater than the angle of incidence. For the moment, let us call the angle of incidence θ_i and the angle of refraction θ_t (where t is for transmitted, reserving r for reflected).

As θ_i increases and approaches a certain "critical angle", denoted by θ_c, the angle of refraction approaches 90° (that is, the refracted ray approaches a tangent to the interface), and the refracted ray becomes fainter while the reflected ray becomes brighter. As θ_i increases beyond θ_cc, the refracted ray disappears and only the reflected ray remains, so that all of the energy of the incident ray is reflected; this is total internal reflection. In brief:

- If $\theta_i < \theta_c$, the incident ray is split, being partly reflected and partly refracted;
- If $\theta_i > \theta_c$, the incident ray suffers total internal reflection; none of it is transmitted.

The critical angle θ_c is the smallest angle of incidence that yields total reflection. For an interface of a material with refractive index n against air θ_c becomes

$$\theta_c = arcsin\left(\frac{1}{n}\right)$$

Adapted from Wikipedia

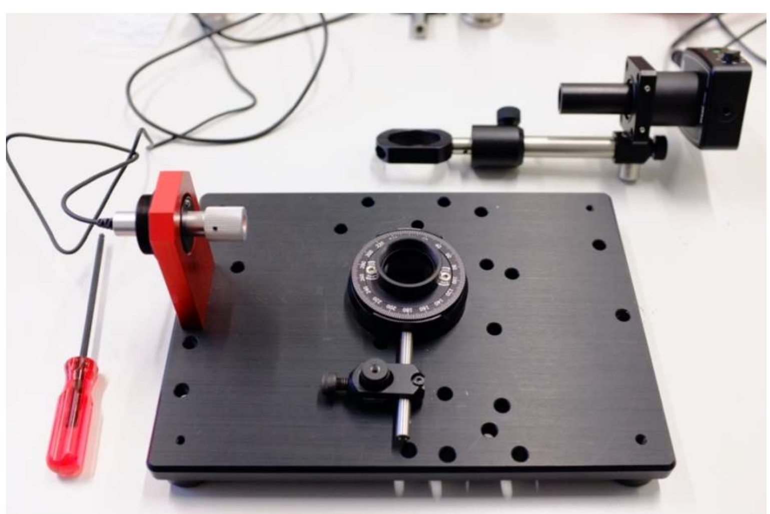
Task Measure the refractive index using the critical angle of total internal reflection

Experiment example

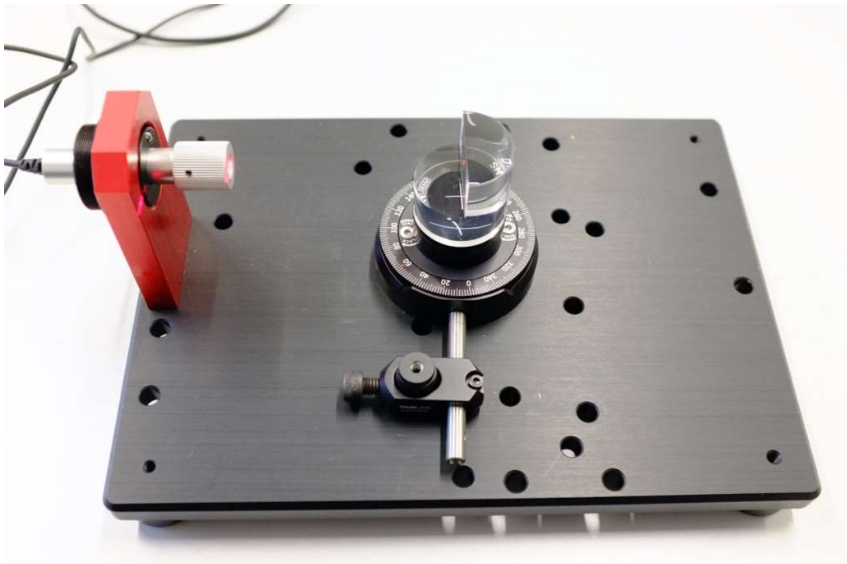
Video link: https://youtu.be/25AF9FlmkS8
(or search on YouTube: MSE215 TP3)

For the following task, an example is made available in the video TP3. Please read the description below first to get an idea of what you are going to do, run the experiment, and calculate the refractive index using the value you found during the TP. The results for this task can be taken from the video TP3, if time left does not allow you to run the practical experiment.

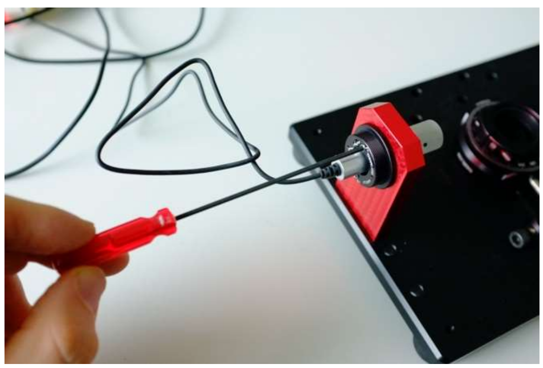
- Simplify the setup by taking away the detector arrangement.
- Mount the laser source.



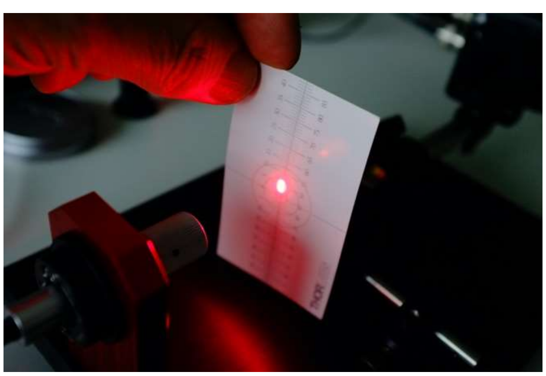
- Mount the cut away half cylinder.

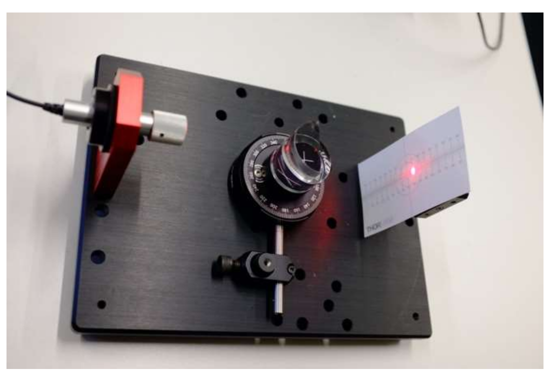


- You can align the laser with the angular movement

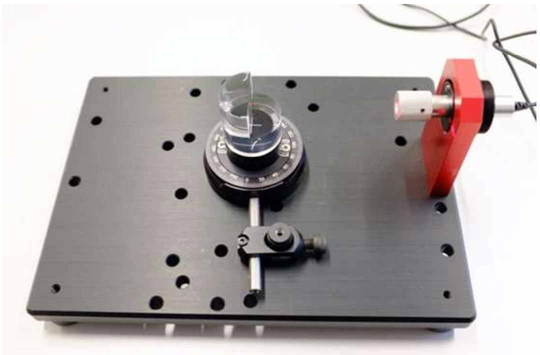
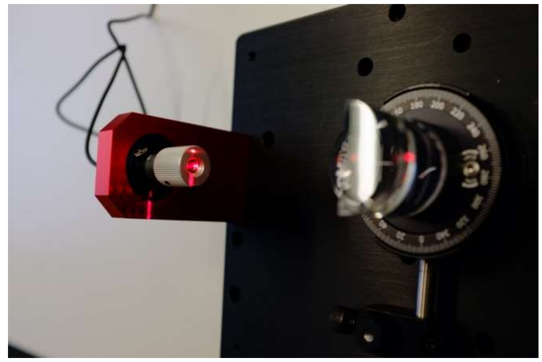


- The laser is at 5 cm height from the breadboard
- Align the laser with the angular adjustment so that you keep this height for the whole breadboard distance.
- Focus in the position by turning the cap of the laser

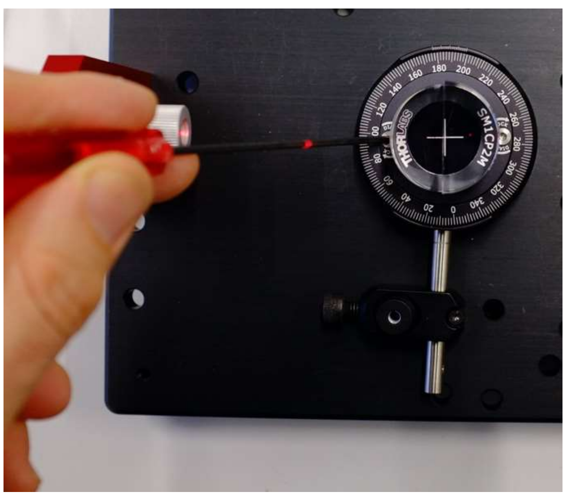




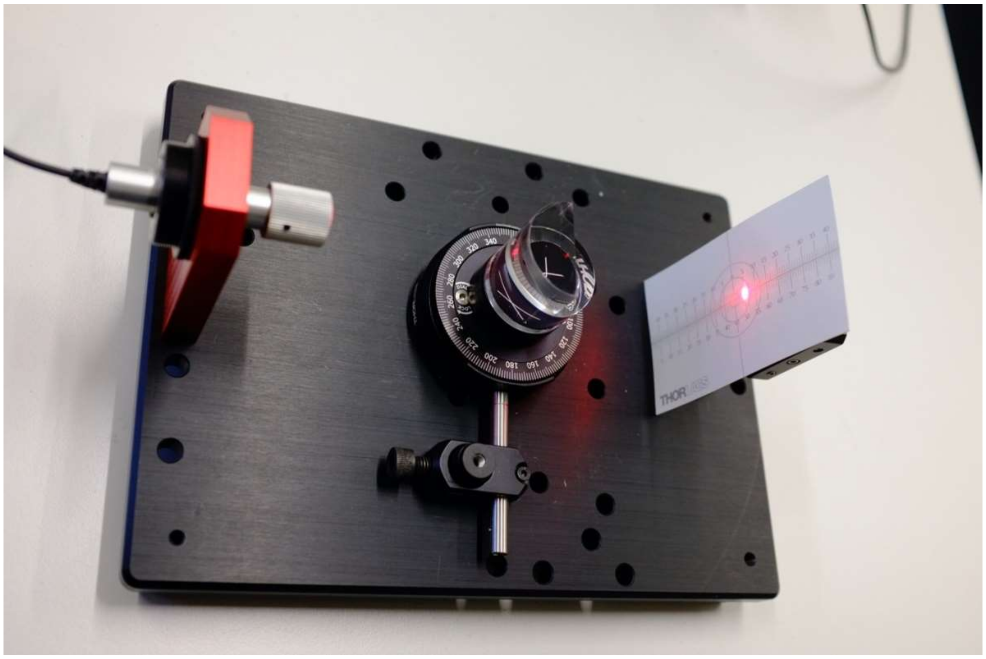
- You can adjust also the scale of the rotational mount.
- Rotate the cutaway cylinder so that the laser is facing the flat side (not the curved side).

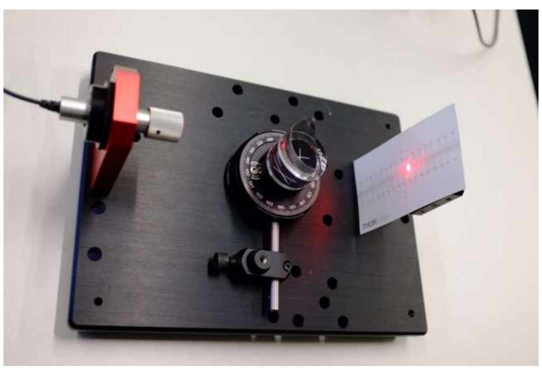
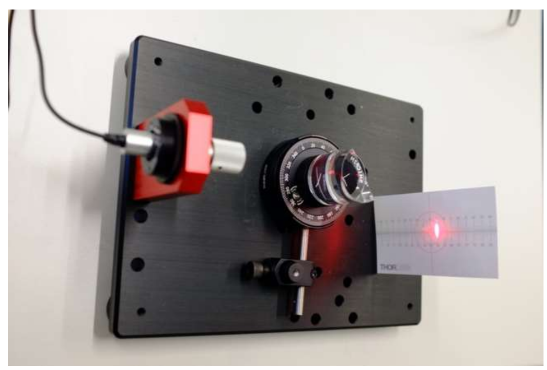
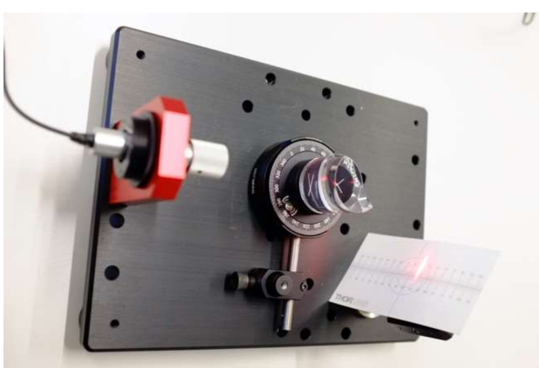
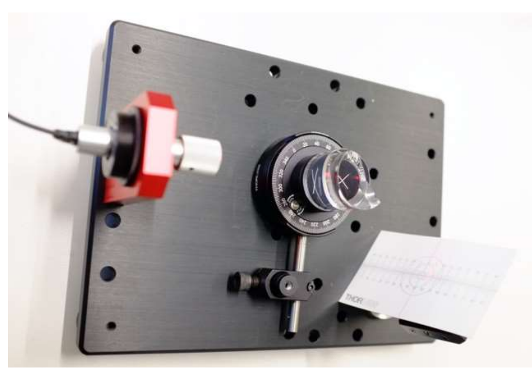
- Rotating the cube allows you to exactly determine the position where the light goes back to laser. Now the surface is perpendicular to the lasers' direction of propagation.
- Loosen the screws for adjustment of the scale and set the scale at 180°.
- Fix then scale.



- Turn the cutaway cylinder in the working position by 180° which means the curved side towards the laser.
- The scale should now be at zero! Please check.
- You can observe the spot and the laser light should run through the rotational center of the cylinder.



- If you turn the cylinder the refracted spot starts to move and at a certain angle will be totally reflected. We have reached the angle of total internal reflection.

- Determine the angle when no light is transmitted as precisely as possible by turning the cube and read the angle on the scale.

- Calculate the refractive index with the formula below.

$$n = \frac{1}{sin\theta_c} =$$

Discussion: **Comment on possible errors of the measurement:**

4. Light diffusion of ceramics and grain size determination

Objective & concepts

Determine the grain size of a transparent alumina sample.

- Measure the transmission of a collimated (straight) laser beam with line Intercept method.

Introduction

General properties of alumina and optical methods

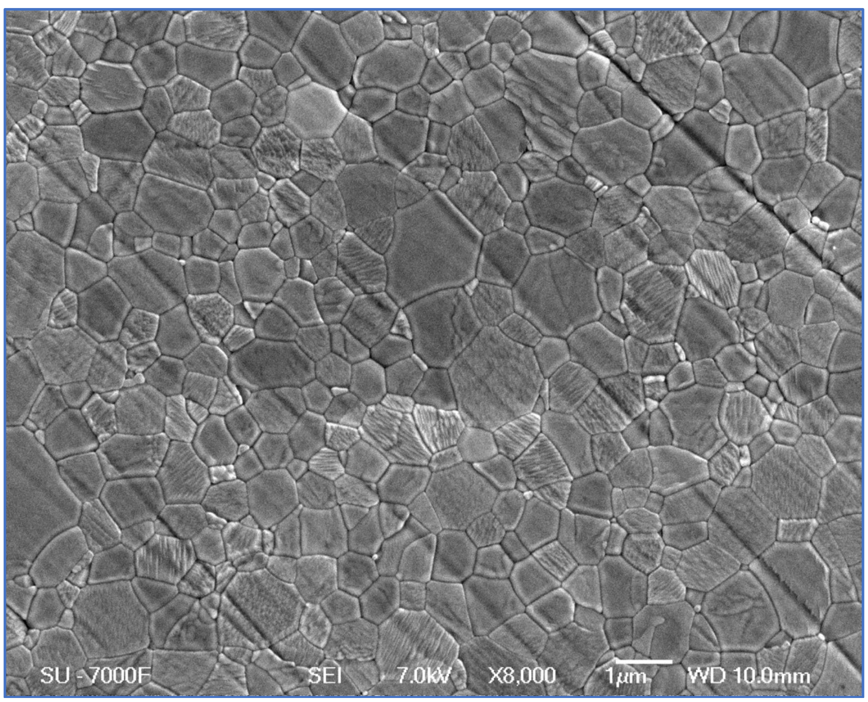
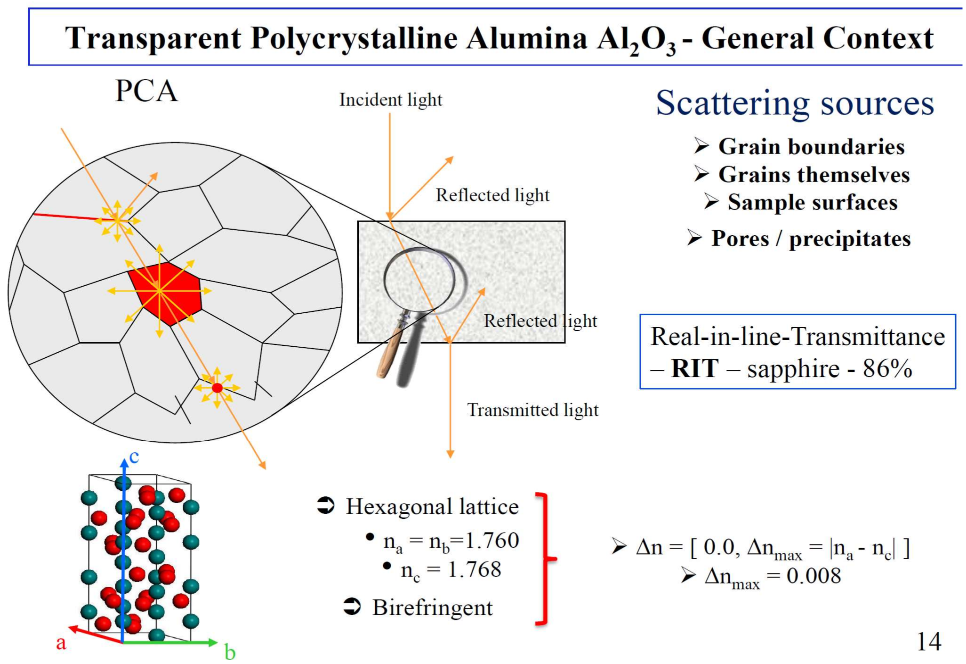


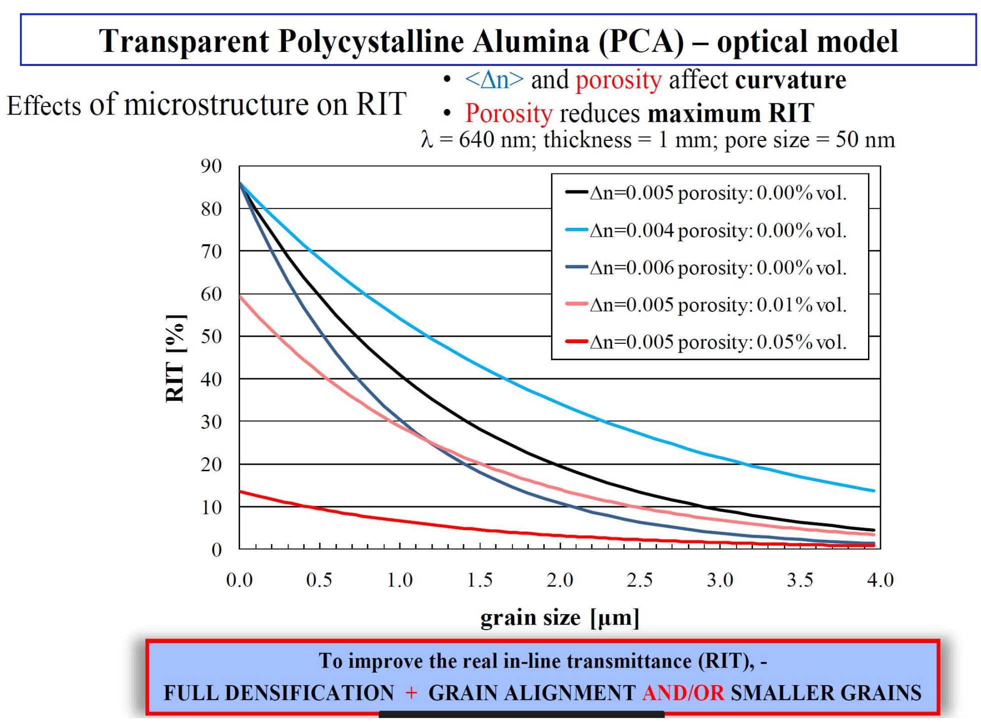
Fig. 1: Scanning electron micrograph of polycrystalline alumina for grain size measurement.

Line intercept method - One of the simplest techniques to estimate an average **grain size** is the **intercept technique**. A random straight **line** is drawn though the micrograph. The **number** of **grain** boundaries intersecting the **line** are counted. The average **grain size** is found by dividing the **number** of intersections by the actual **line length**. You can draw several lines to get a better statistical representation BUT you must not include grains at the edge of the micrograph and a line cannot pass through the same grain twice. Because of the isotropic cut assumption (in 3D grains are equiaxed but we are looking at a 2D projection) normally the grain size is underestimated and the final grain size is given by multiplying by a correction factor of 1.56.

[Mendelson, M.I., *Average Grain Size in Polycrystalline Ceramics.* Journal of the American Ceramic Society, 1969. **52**(8): p. 443-446.]

RIT method





[Paul Bowen, *Mise en oeuvre des matériaux I - Céramiques.* EPFL, 2019]

Task Measure transmission and determine grain size

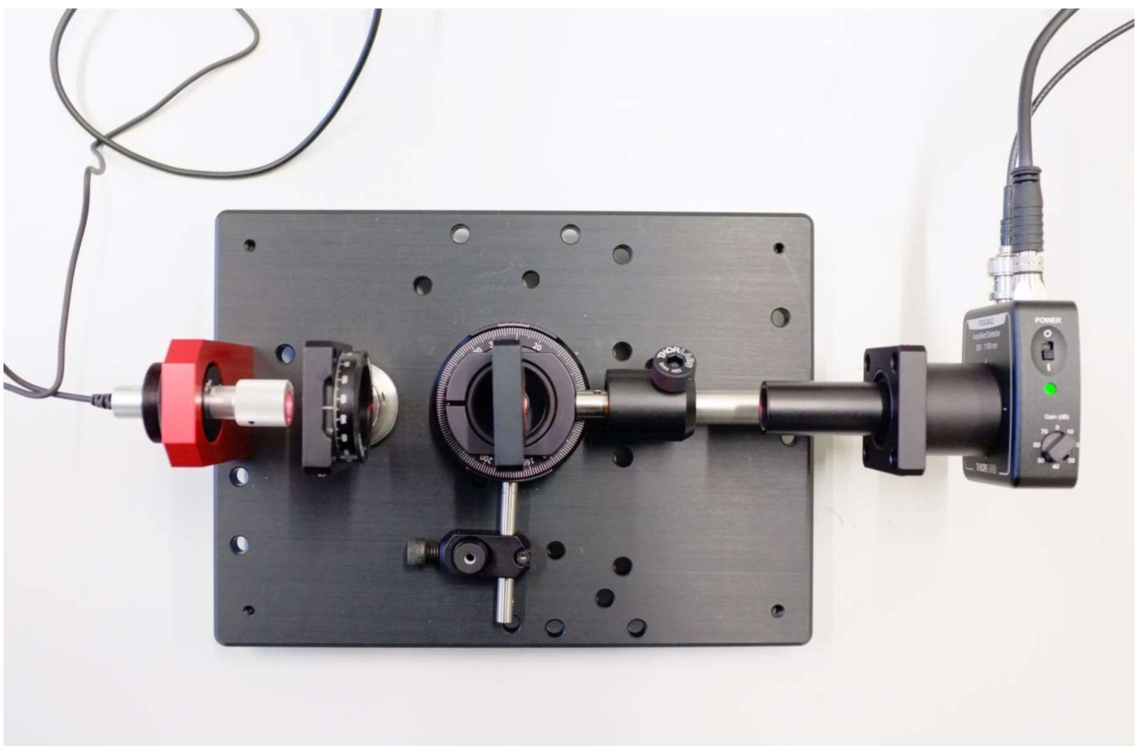
Experiment example

Video link: https://youtu.be/4fdHUGmjDsA
(or search on YouTube: MSE215 TP4)

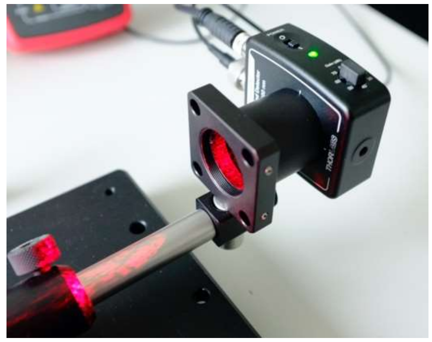
For the following task, an example is made available in the video TP4. Please read the description below first to get an idea of what you are going to do, run the experiment, and calculate the transmittance using the value you found during the TP.

First method: RIT.
We will use the laser and detector to measure the scattering and transmission properties of polycrystalline alumina ceramics.

- Setup the system as shown below. Fix the laser in the mount and put the detector in place. You might also need the polarizer in the rotational mount for intensity adjustment. In the image below we have already put the sample in the middle of the assembly.



- We first need to align the system. Remove any sample and polarizer.
- Remove the scattering light protection tube from the detector so that you can see the detector chip.


- Focus and align the laser on the detector carefully.
- Put the polarizer in the path and turn it to adjust the intensity so that the detector delivers signals below 10 V (no saturation).
- Put your sample.
- Observe the diffusion characteristics (qualitatively).
- **Measure the reduction of signal and calculate the transmittance (I/I_0). The result is directly the real in line transmission RIT.**
- **Evaluate the grain size from the chart above assuming a Δn of 0.005 and zero porosity.**

Grain size found by RIT measurement:

<u>Second method: Line intercept method.</u>

- From the micrograph supplied (Scanning Electron Micrograph) on the first page of this section (Fig. 1), use the line method to evaluate the average grain size.
- You can use the picture *Alumina sample* available in this TPs' folder with **ImageJ** to follow the line intercept method (please see the annex on the last page).

Please fill out the following table:

Line no.	**Number of intercepts**	**Line length** [___]	**Average grain size** [___]
1			
2			
3			
4			
5			
Final average grain size		*Corrected average grain size*	

Discussion:

Compare the value of the two methods!

Comment on possible errors of the measurement:

Annex – ImageJ installation procedure

1. Download ImageJ Fiji : https://imagej.net/Fiji/Downloads
2. Extract Fiji.app
3. Run ImageJ-win64.exe