MSE-215

Mise en œuvre des matériaux II

Spring 2025

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 what-is-this.net, 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 CoatingPaint.com).*

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 on your results and add possible sources of error.

The duration is planned for 2h.

1. Transmission and absorption

Objective & Concepts

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

- Measure the transmission of transparent but colored materials using a laser.
- Use different thicknesses of material to calculate the changes and absorption and reveal the 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 ($n_2 \approx 1.5$) surrounded by air ($n_1 = 1$), the power reflectance R at normal incidence is 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 […] relates the attenuation of light to the properties of the material through which the light is travelling. It states that the absorbance of light is proportional to the thickness, the concentration and the molar absorptivity of the absorbing species. It is commonly applied to chemical analysis measurements and used in understanding attenuation in physical optics, like particles in suspension, neutrons, or rarefied gases.

The Beer-Lambert law can be simplified and rearranged in terms of light intensity after the sample, I:

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

with D, the thickness of material, α the absorption coefficient and I_0 the incident light intensity.

The intensity can be expressed in any unit. It is common to use the output value of the optical detector. The transmission can be calculated using the intensities as

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

If D is known, measuring the transmission allows to determine α, the absorption coefficient.

Adapted from Wikipedia

Experiment example

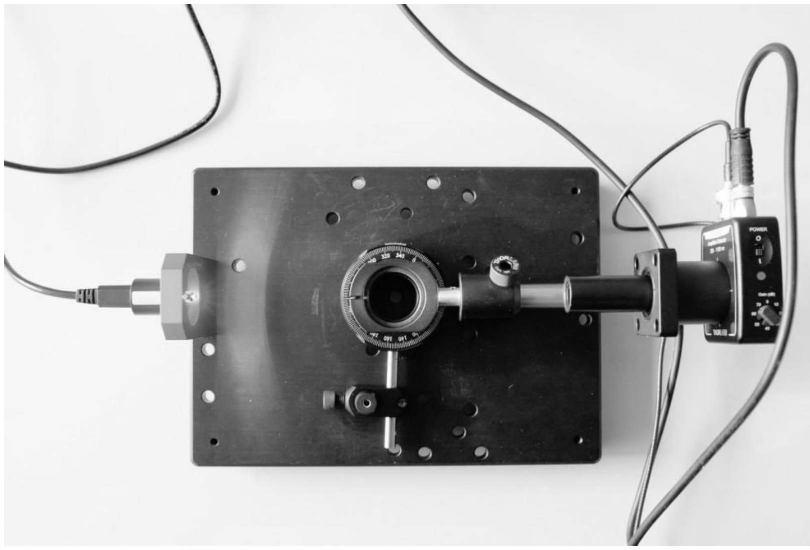
Video link: https://youtu.be/aTBBD0X8fBc
(or search on YouTube: MSE215 TP1)

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. Then run the experiment and fill out tables and discussions with the values you found during the TP.

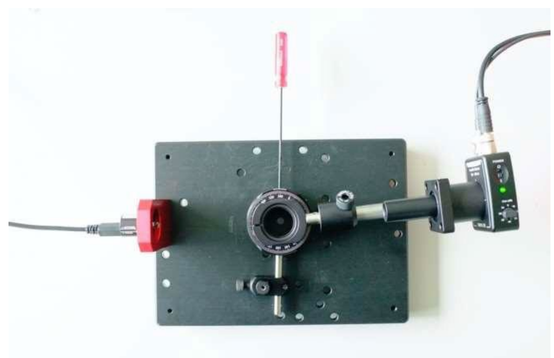
Task one Wavelengths dependence of transmission

- Mount the system as shown below.
- Use the laser and connect it to the USB power supply or a computer USB port.
- Connect the detector to its power supply (+-12 V) and to the handheld multimeter.
- Switch on all elements (laser, detector, power supply, multimeter on V).
- Choose **DC voltage** measurement on the handheld multimeter by clicking the yellow button.

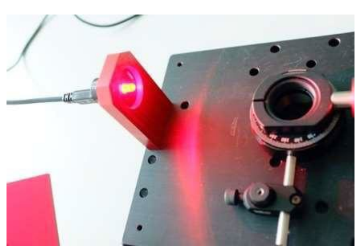
The detector amplifier provides voltages up to 10 V. The detector can easily saturate and adjustment of the AMPLIFICATION is needed for proper operation. For this, use the small rotational knob on the side of the detector (dB scale) to you choose the amplification so that the detector measures a signal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

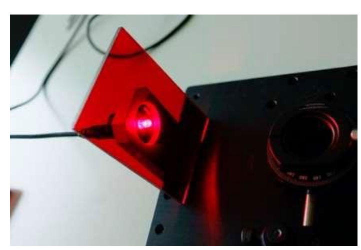


To avoid rotation, the mount can be blocked using the small ball driver as shown below.



- 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 laser and check that there is no signal (< 40 mV).
- Switch the laser on and note the values of the detector signal for the different cases given in the following table, using the color plastic sheets provided. You can hold them in front of the source.





Please fill out the following table (including the units):
$D = 3\ mm$

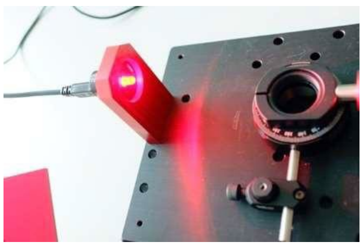
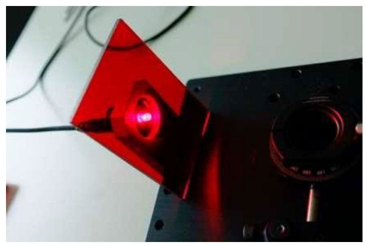
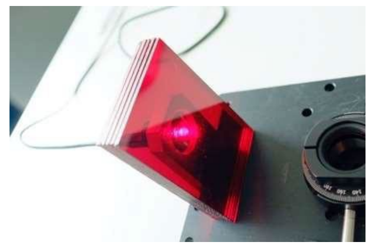
Plastic sheet	**Signal** [____]	**Transmission** (signal I with plastic sheet divided by signal I_0 without plastic sheet)	**Absorption** [____] $\alpha = -\frac{1}{D}\ln\left(\frac{I}{I_0}\right)$
None		1	0
Blue			
Green			
Red			

Discussion:

Task two Beer-Lambert law

To do:
1) Use the laser and the red plastic sheets
2) Measure the light intensity for different thicknesses (0, 3, 6, 9, 12, 15 mm thickness, ie. 0-5 plates)
3) Calculate the absorption coefficient
4) Consider correction of surface reflection

- Use the same setup
- 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 films.

- Measure the transmission for different angles between two polarizers (Malus' law)
- Determine the retardation of Scotch tape (compare your results with the retardation data table provided)
- Take pictures of an injection molded plastic part under crossed polarizers and different rotations. Explain what you see

Introduction

Malus' Law

Malus' law, named after Étienne-Louis Malus, states that when a perfect polarizer is placed in the path of 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

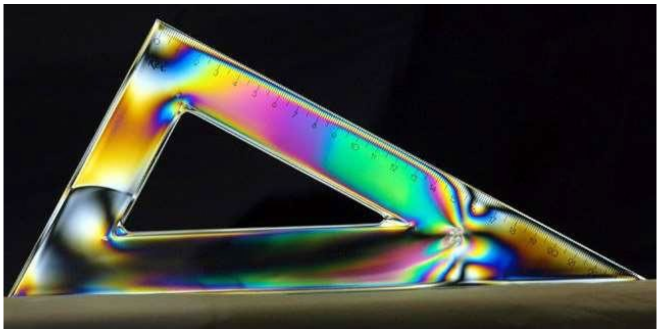
Birefringence is the optical property of a material having a refractive index that depends on the polarization and propagation direction of light. These optically anisotropic materials are said to be birefringent. The birefringence is often quantified as the maximum difference between refractive indices 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, when held between crossed polarizers at 45° with respect to their optical axis, is given by:

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

This causes the appearance of color and allows to quantify the factor $\Delta n \cdot D$, called **retardation**.

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



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

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

Adapted from Wikipedia

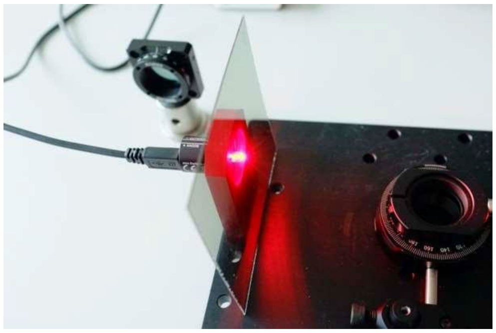
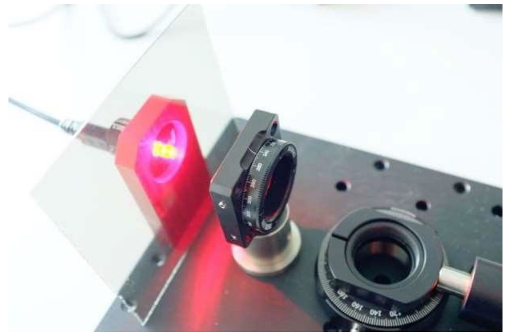
Task one Confirm Malus law

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. The 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.

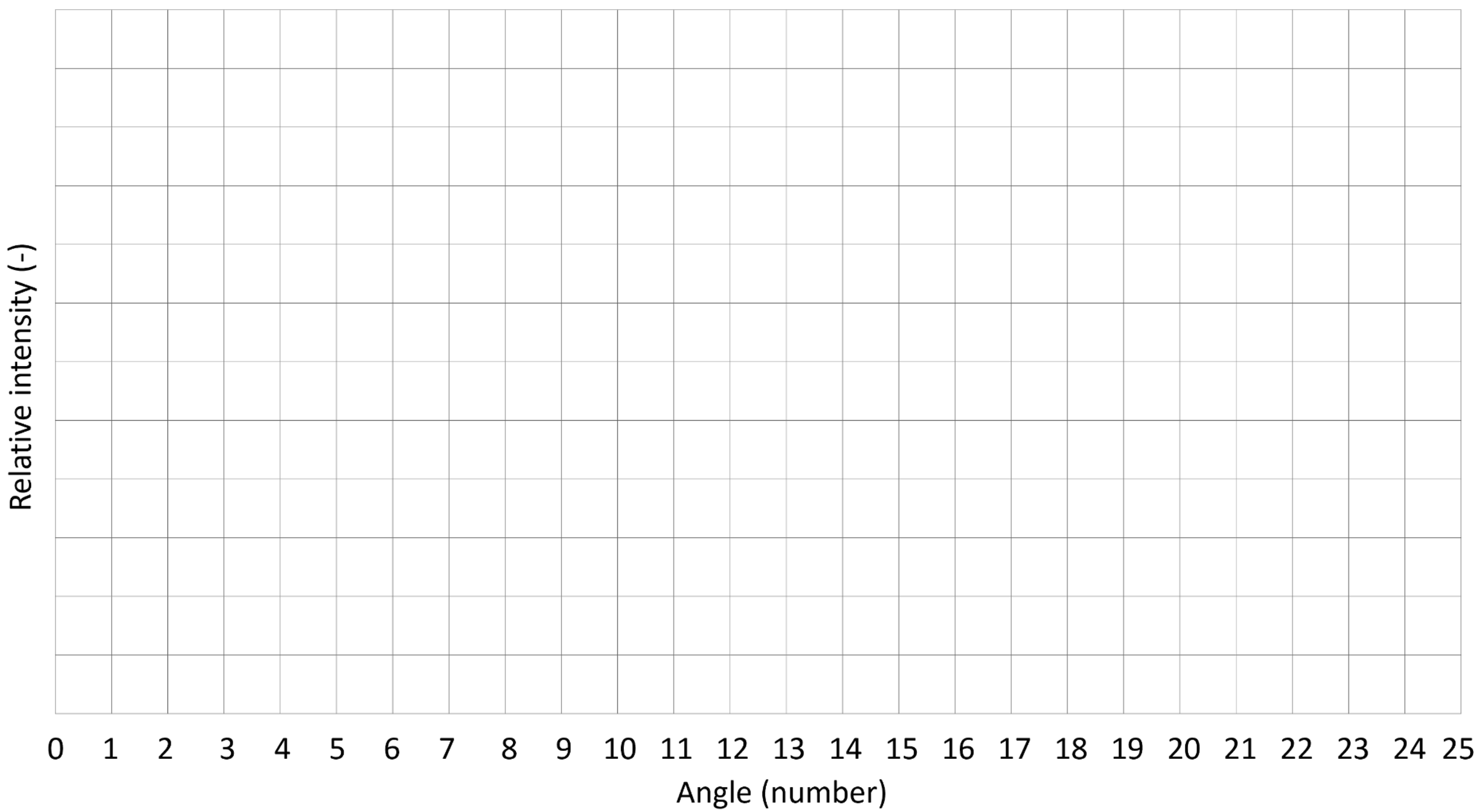
- Use the same setup as given in task one and mount the laser.
- Put a sheet polarizer (grey square plastic sheet) in front of the laser.
- Adjust the amplification of the detectors amplifier to assure high signal but without saturation (signal below 10 V).

- Place the polarizer in a mount in front of the laser.
- You can rotate the polarizer at high precision and read the angle.
- Adjust the position of the rotational polarizer to the maximum intensity, read the angle and note the detector's reading.
- Be careful that your detector is not saturated (Value smaller than 10 V).
- Measure the detector's signal values for every 10° rotation over a cycle of 200 degrees and mark it directly in the graph on the next page.

Discussion:

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

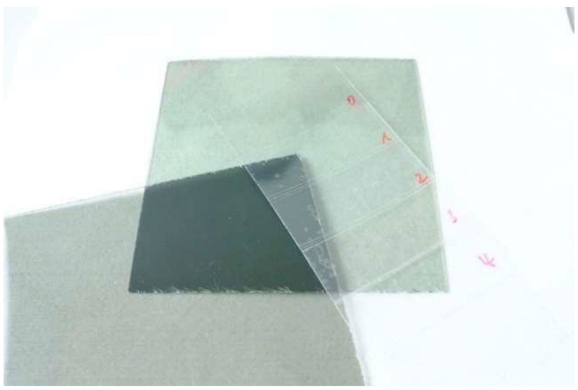

Relative intensity (-)
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25
Angle (number)

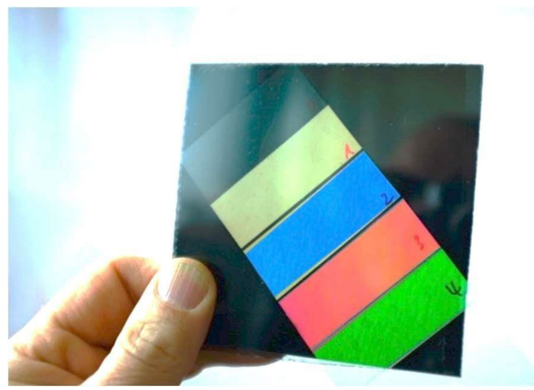
Task two Determine the retardation of scotch tape

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

- Use a non-birefringent, transparent substrate and one layer of tape on it.
- Stick 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.

The sample will look like the picture shown below when observed with a single polarizer and gets colored when put between crossed polarizers. You can use this picture to perform the following analysis, or perform the experiment with the polarizers and Scotch sample provided.

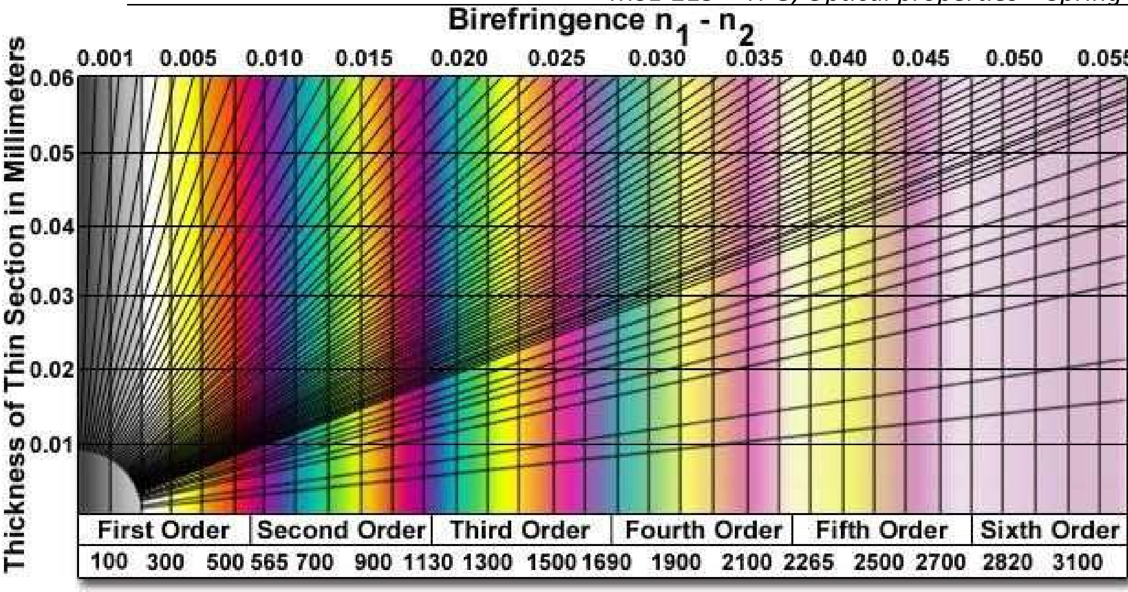




Birefringence colors can be calibrated. With the help of the chart below one can determine the retardation of a single tape layer of your sample. The values are given in the lower column below the orders! Note that each layer adds the same retardation (as it has the same thickness) and so by adding a 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).

Using the sample already prepared, you should proceed as follows:

- Identify the layer without birefringence (it will be 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 these inclined lines have a birefringence attributed to it, as written on the right and on top of the graph. This allows to find the birefringence 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. This means, if for the same color and 60 micron thickness we have a birefringence of 0.01 we will find for 600 micron thickness a birefringence of 0.001. The product is constant for the same color.

In our case **a single layer of the sample is approximately 200 microns thick**. You can look at the 0.02 mm thickness line of the table, find the intersection with the sloped line at your color and **divide the birefringence value by a factor of 10**.

Birefringence of the scotch tape: Δn =

You can **verify your result** by looking at the different colors and layer thickness that are available on your sample.

Discussion:

3. Refractive index and total internal reflection

Objective & concepts

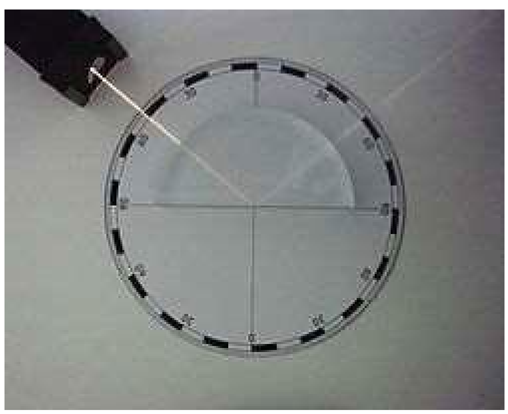
Measure the refractive index of plastic

- Total internal reflection is used to find the limiting angle of the 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_c, 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 undergoes 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

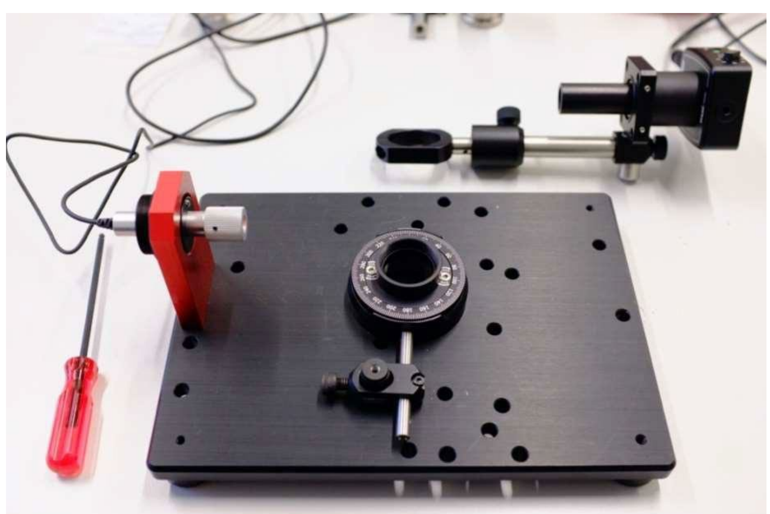
Task Measure the refractive index using the critical angle of total internal reflection

Experiment example

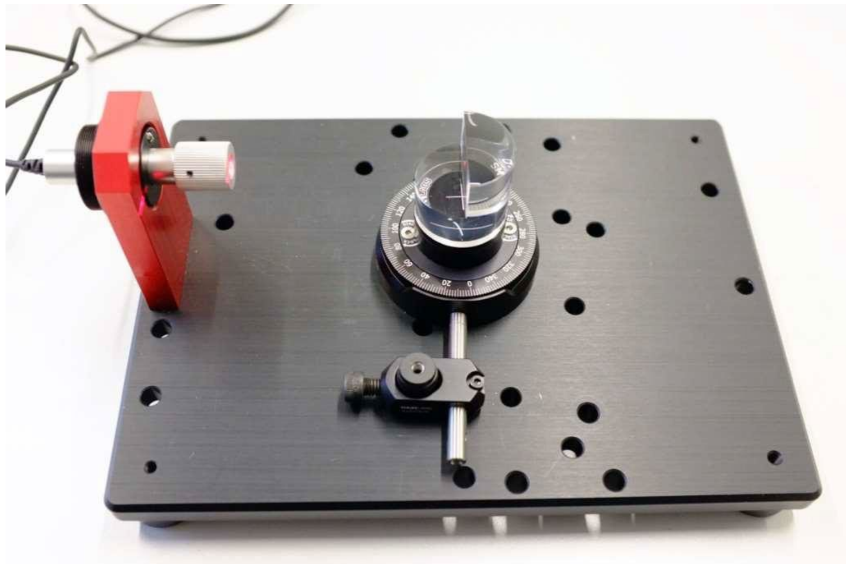
Video link: https://youtu.be/25AF9FlmkS8
(or search on YouTube: MSE215 TP3)

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. Then 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.

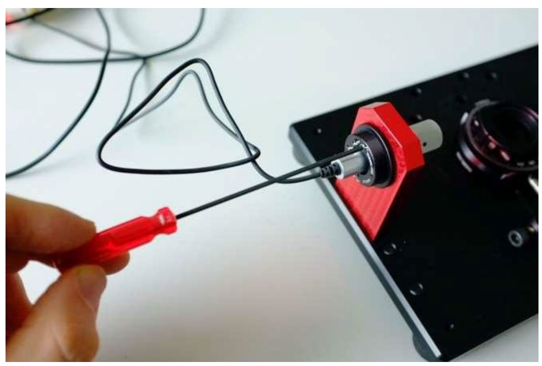
- Simplify the setup by taking away the detector arrangement.



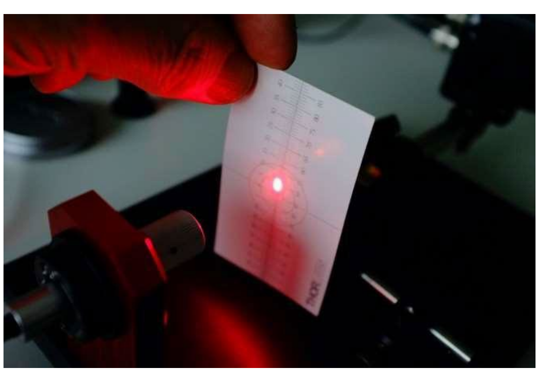
- Mount the cut away half cylinder.

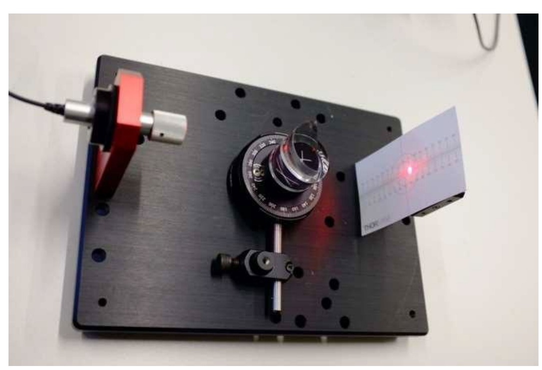


- You can align the laser with the angular movement by adjusting the crews on the mount

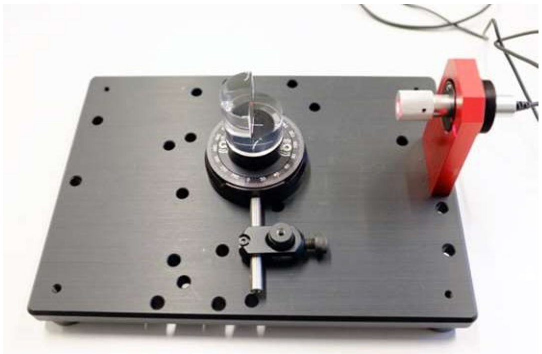


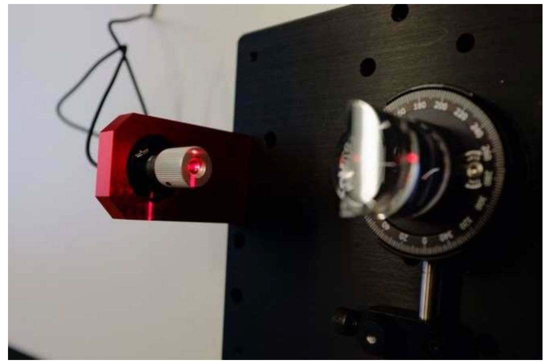
- The laser is at 5 cm height from the breadboard
- Align the laser with the angular adjustment so that you keep this height for the whole breadboard distance



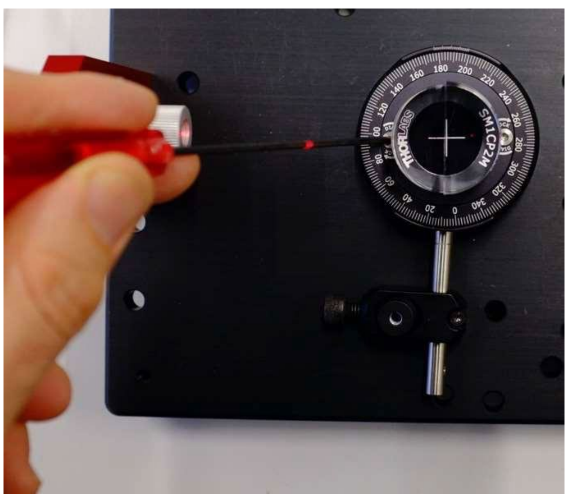


- You can also adjust also the angular position of the cutaway cylinder on the rotational mount.
- Rotate the cutaway cylinder so that the laser is facing the flat side (not the curved side).

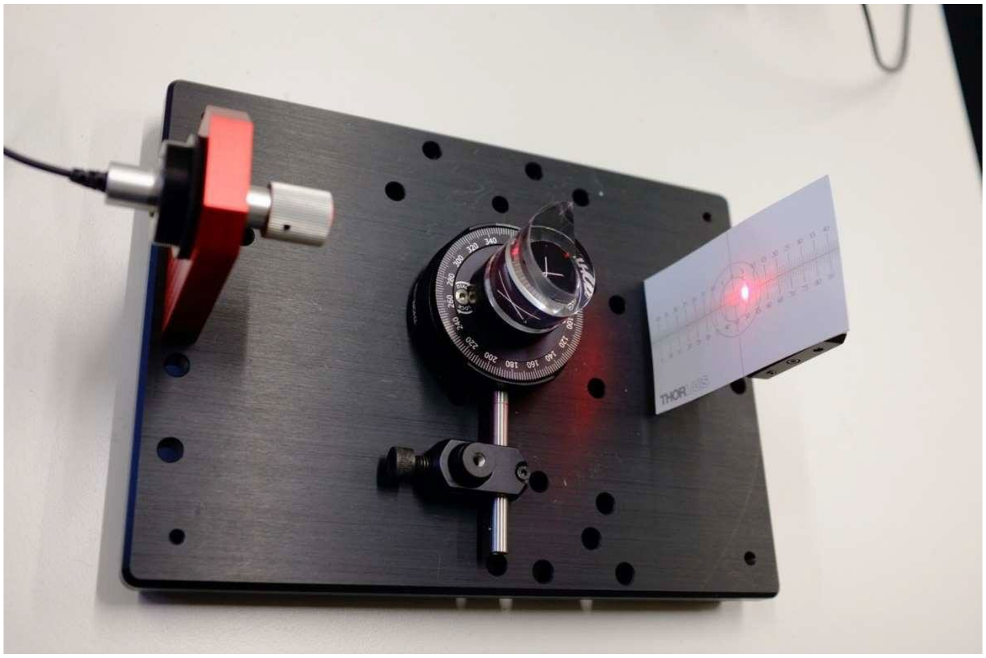




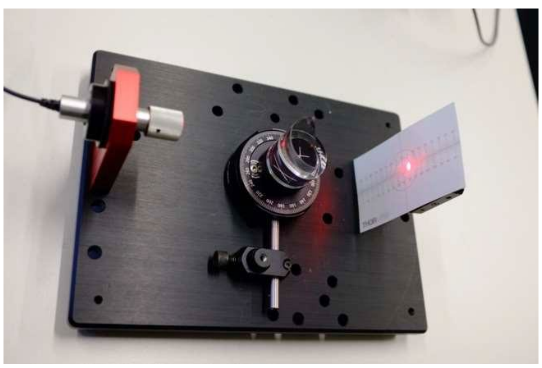
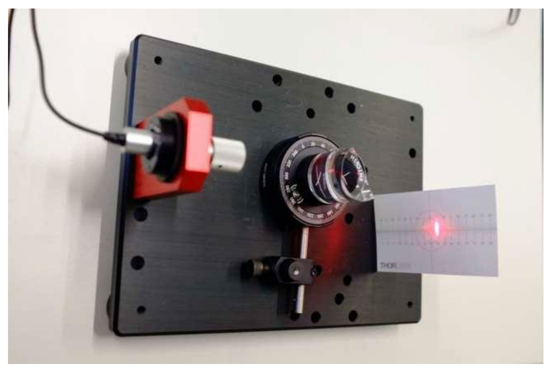
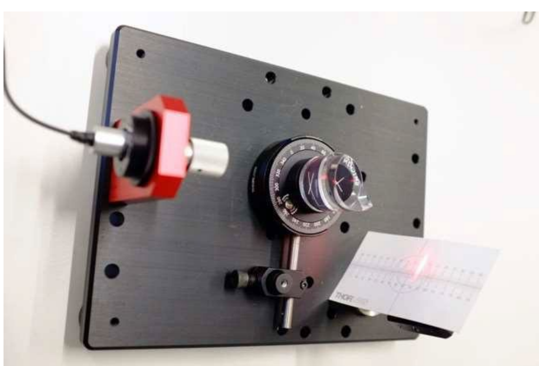
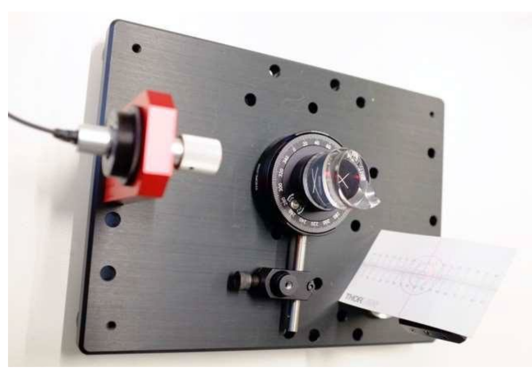
- Rotating the cylinder allows you to exactly determine the position where the light goes back to laser. Now the surface is perpendicular to the lasers' direction of propagation.
- Loosen the screws to adjust the angle of the rotational mount so that it shows 180°.
- Fix then screws.



- Turn the cutaway cylinder in the working position by 180° which means the curved side towards the laser.
- The angle should now be zero! Please check.
- You can observe the laser spot and the light should pass 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 cylinder 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 and use the RIT method
- Compare with the line intercept method

Introduction

General properties of alumina

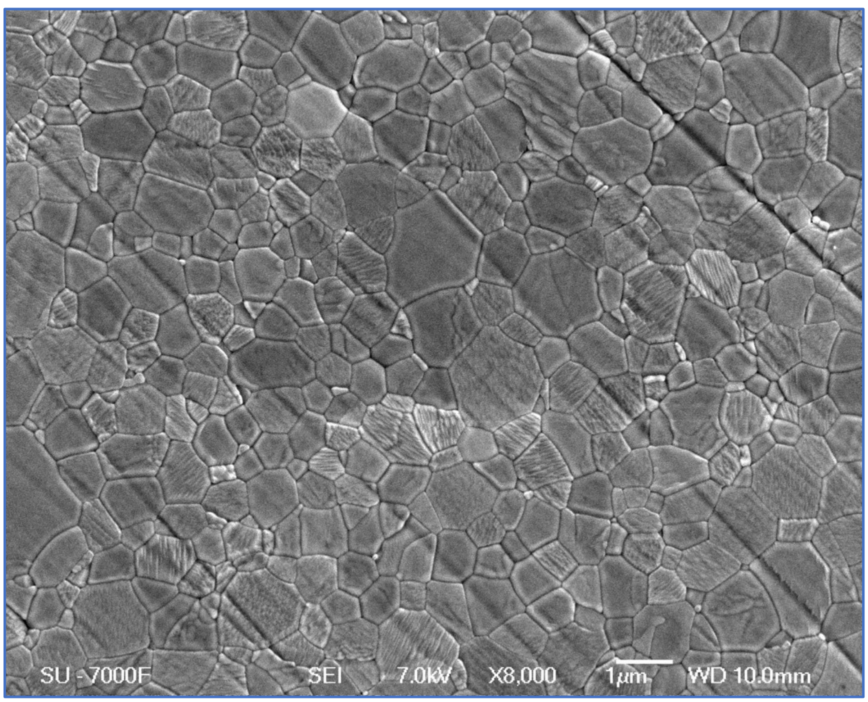


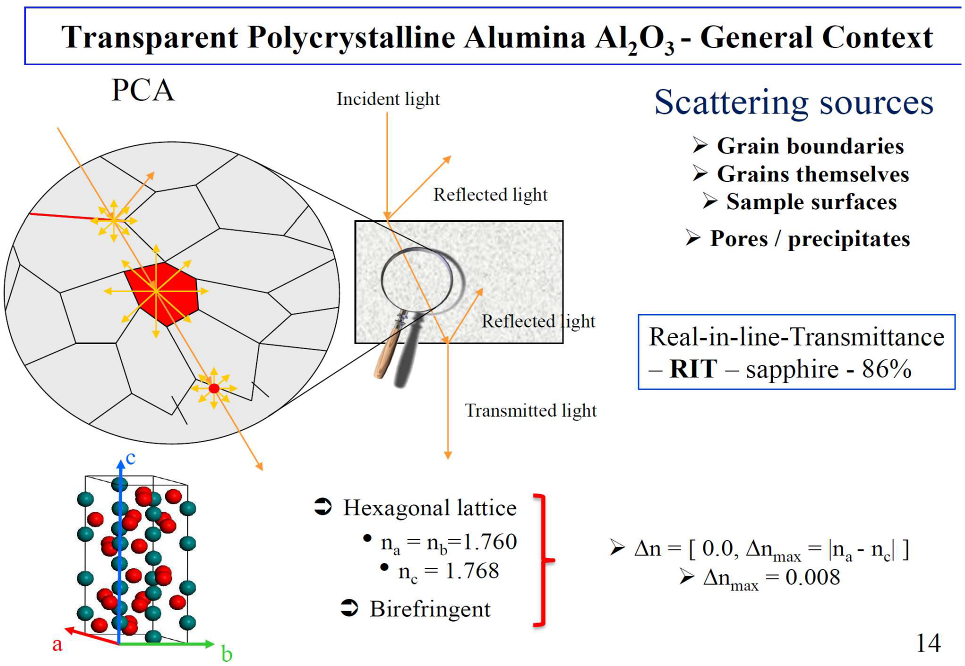
Fig. 1: Scanning electron micrograph of polycrystalline alumina for grain size measurement.

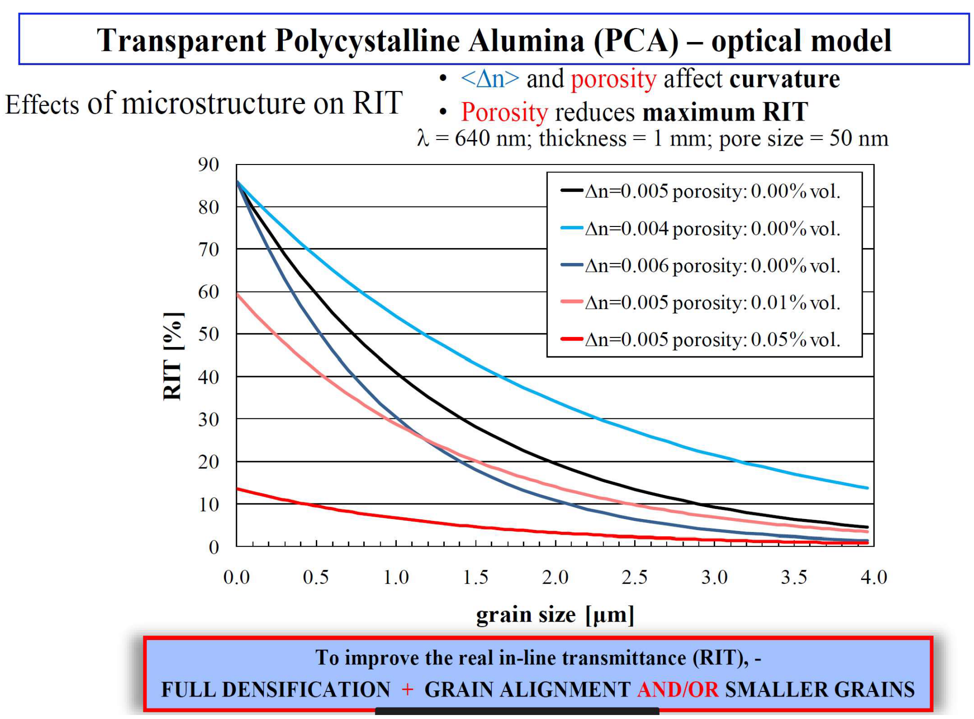
Line intercept method

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

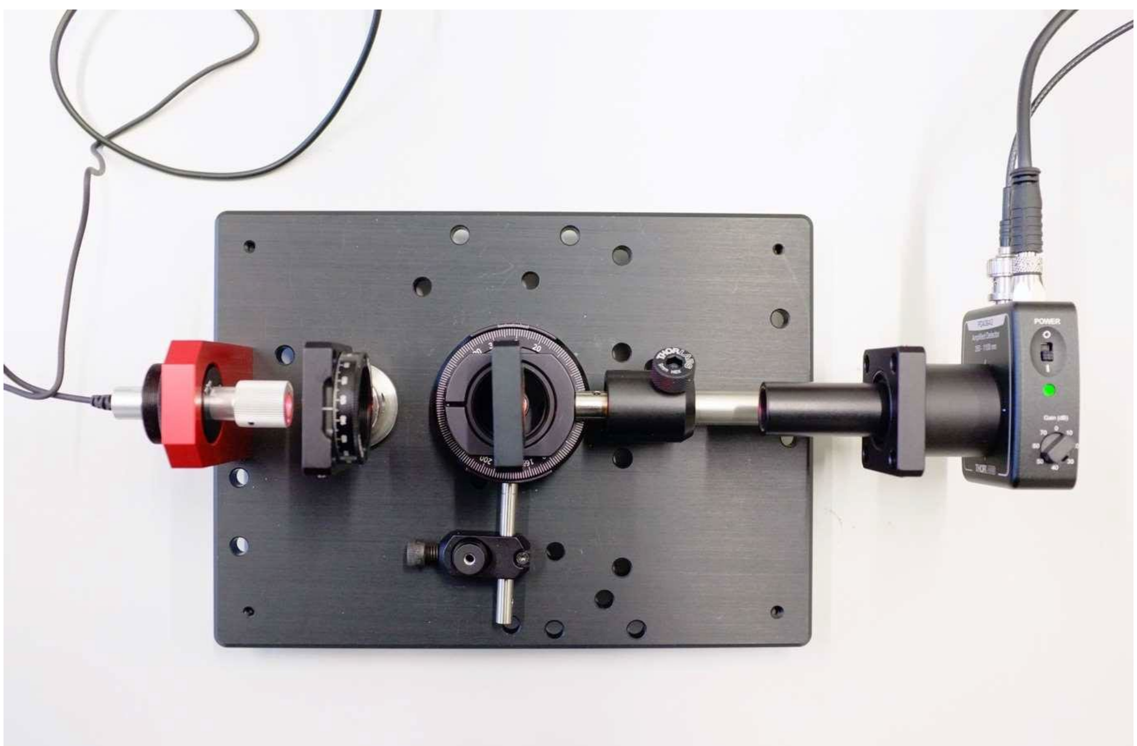
Video link: https://youtu.be/4fdHUGmjDsA
(or search on YouTube: MSE215 TP4)

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. Then run the experiment and calculate the transmittance of polycrystalline alumina using the value you found during the TP.

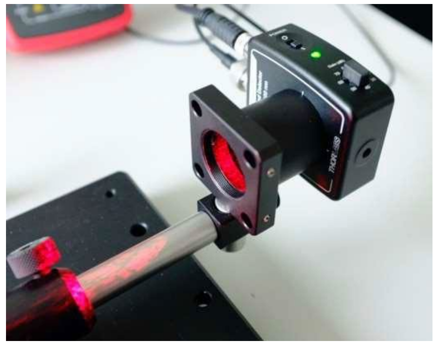
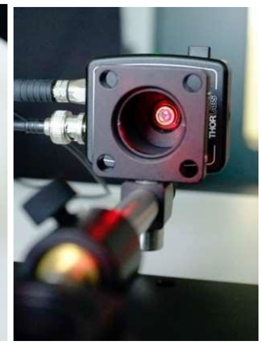
First method: RIT

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 alumina sample in the center of the board.
- Observe the diffusion characteristics (qualitatively) of the laser on alumina.
- **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 (including units):

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