


Characterization of Construction Materials
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Lecture-34
Optical and Scanning Microscopy- Features and Functions - Part 1




Hello everybody. So, we started off talking about differences between different types of microscopy.

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Background reading 

- <http://www.visioneng.com/resources/history-of-the-microscope/>
- <https://www.microscope.com/education-center/microscopes-101/history-of-microscopes> ←
- https://en.wikipedia.org/wiki/Optical_microscope
- <https://www.fei.com/introduction-to-electron-microscopy/history/> ←
- https://en.wikipedia.org/wiki/Electron_microscope
- <http://micro.magnet.fsu.edu/index.html> ←

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Techniques available 

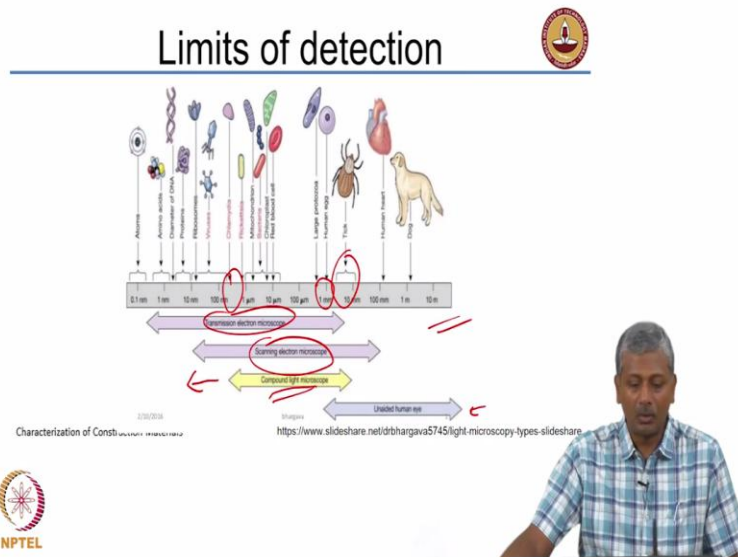
- Optical microscopy ✓
- Scanning electron microscopy ✓
- Transmission electron microscopy
- Scanning tunneling microscopy
- Scanning probe microscopy
- Infrared microscopy
and several others!

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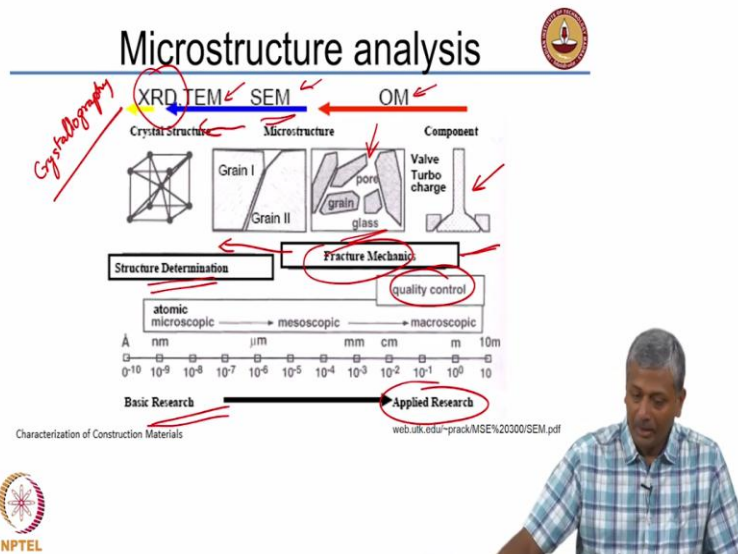
We were looking at the typical techniques of microscopy that are available.

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And in general, what the major aspects that you can actually observe with different kinds of techniques are.

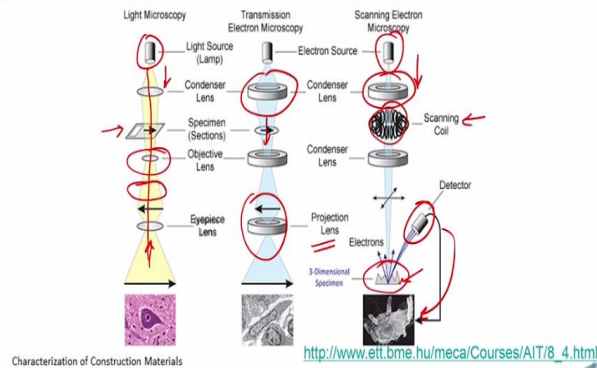
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We also saw what the reasons for doing different types of microscopy were. What kind of answers we can get with different types of microscopy.

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Comparison between techniques



We also looked at primary differences between the techniques in terms of optical microscopy, scanning electron microscopy and transmission electron microscopy.

(Refer Slide Time: 00:49)

Specimen preparation for microscopy



- Probably more important than microscopy itself!!
- Fractured specimens – no real ‘preparation’; just mount and observe; not suitable for optical microscopy, only SEM
- Polished specimens – common procedures for optical microscopy and SEM

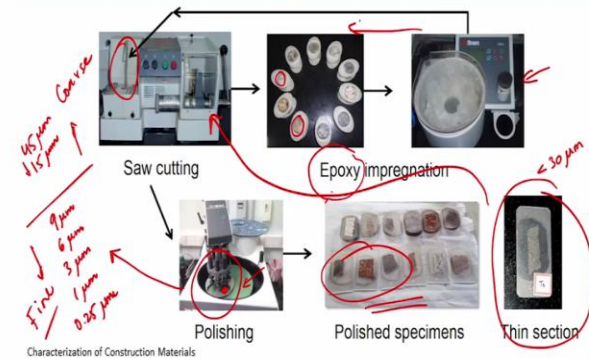
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And then we talked briefly about specimen preparation for microscopy, which I stated is very crucial in the process of getting a good image from microscopy technique.

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Preparation of polished specimens of porous building materials



So, there are several aspects involved in terms of polishing specimens in porous building materials.

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Slicing paste samples



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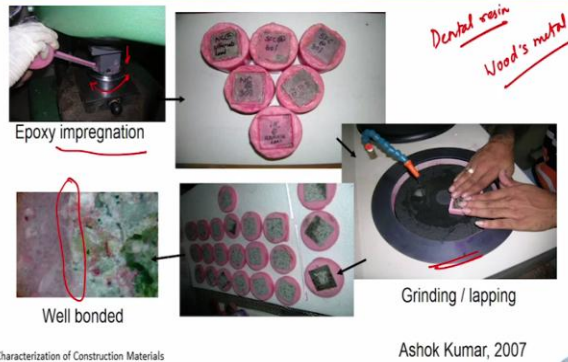
Scrivener et al. 2015



And also sometimes you can prepare materials specifically for the purpose of using for microscopy.

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Preparation of loaded specimen



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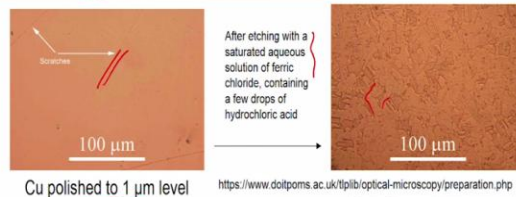
Ashok Kumar, 2007



We also looked at some cases where loaded specimens could be used and you can actually preserve the structure under loading by using an appropriate epoxy that is able to encapsulate and impregnate the porosity and the cracks to ensure that the cracks do not close after unloading.

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Etching – for optical microscopy

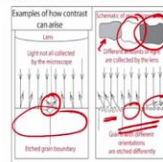


Cu polished to 1 μm level

<https://www.doitpoms.ac.uk/tpib/optical-microscopy/preparation.php>

Etching is used to reveal the microstructure of the metal through selective chemical attack. In alloys with more than one phase etching creates contrast between different regions through differences in topography or the reflectivity of the different phases. This results in a surface relief that enables different crystal orientations, grain boundaries, phases and precipitates to be easily distinguished. Etching could be chemical / plasma

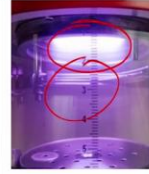
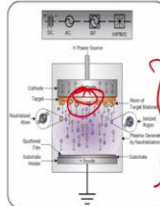
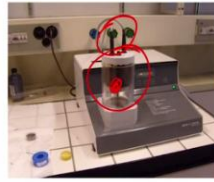
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We talked about certain additional aspects of specimen preparation. One is etching, etching is basically selective chemical attack which exposes certain phases and exposes the grain boundaries in a much better fashion than if you do just polishing.

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Coating - for electron microscopy



Coating is ~~sometimes~~ necessary because of charging of non-conductive specimens. Charging leads to:

- Deflection of SE's
- Increased emission of SE's in cracks
- Periodic SE bursts
- Beam deflection

<http://www.semicon.com/what-is-sputtering>

<https://www.labtech.com/em/high-purity-sputter-coater-targets>

Coating methods:

- Sputter coating with C, Cr, or Au-Pd
- Carbon tape, carbon paint, In foil

ESEM - no need for coating!!

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And finally, we talked about coating which is recommended for samples that are non-conductive, because in non-conductive samples when you use it in scanning electron microscope, the electron beam tends to charge up on top of the specimen because there is nothing to conduct the beam down towards the rest of the metallic parts of the microscope. So, this charging is not very good for imaging. So, because of that, we need to have a conductive surface, which is typically obtained by sputter coating by an element like carbon, chromium or gold-palladium, which can be applied as a layer on top of the specimen to ensure that the surface gets conductive.

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Optical microscopy

Involves passing visible light transmitted through or reflected from the sample through a single or multiple lenses to allow a magnified view of the sample. The resulting image can be detected directly by the eye, imaged on a photographic plate or captured digitally.

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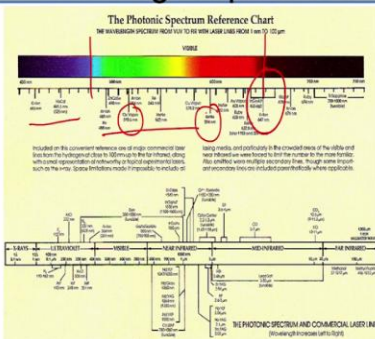
So, moving on to the next aspect, let us start discussing now, the aspects of optical microscopy. Now, this is something that many of you are familiar with because you have probably used

microscopes in your school days. The idea is to use the assessment of the specimen by passing visible light, transmitted through or reflected from the sample, depending on how the sample is, whether it is transparent, translucent or opaque, in one case, you may have to rely on through-transmission light microscopy and in other cases, you may have to work with reflected light microscopy. So, idea is to allow a magnified view of the sample and the resulting image is either detected directly by the eye or can be captured photographically.

Now, it is quite similar in some ways to your principles of photography. In photography also, you are looking at an object and you are trying to capture the image of the object on the camera. And today, of course, with all the sophistication is that you have in the cameras, like an optical zoom or a digital zoom, you can actually lead to magnified views of the object, in many ways, quite similar to what you actually do with a microscope. Of course, you cannot get to the resolution of the microscopes, but you can certainly get somewhere close

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Visible light spectrum



The Photonic Spectrum Reference Chart
THE WAVELENGTH SPECTRUM FROM 100 TO 10000 nm (100 μm)


Included on this document reference line of single commercial laser lines from the hydrogen chloride 100 up to the far infrared, along with some representative wavelengths of typical applications such as the visible. Some wavelengths are impossible to include, or some details, and particularly in the crowded areas of the visible and near infrared are given to list the number to the main families. This content was created by Paul Robinson, Purdue University and is available under a Creative Commons Attribution-NonCommercial-ShareAlike license.


THE PHOTONIC SPECTRUM AND COMMERCIAL LASER LINES
(Wavelength in nanometers)



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Source: Paul Robinson, Purdue University






So again, just to make you remember the visible light spectrum, please remember that the white light is composed principally of 7 colors (the rainbow colors) and these are distinguished by their wavelength. So, if you look at the visible light spectrum, we are basically in this range (shown in Figure in slide). So, you have violet, indigo, blue, green, yellow, orange and red because that is the order of the wavelength. So, the violet light has the smallest wavelength and red has the largest wavelength (close to about 700 nm).



So, what is also given in this chart is that these are actually the discharges produced by different types of metals, for example, you have here copper vapor which can produce a wavelength in the range of about the green light. So, several elements actually emit radiation and that is in the range in the visible range in some cases. For example, here you talk about helium and neon. We are releasing it in the red wavelength. So, this is just to give you an idea about what are the different categories of wavelengths and what kind of characteristic emissions from metals correspond to those wavelengths.

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Basics of light 

- Absorption – decrease in intensity upon transmission through a medium (different colours, i.e. specific wavelengths, may get absorbed depending on the object phases)
- Refraction – change in direction from one medium to another (short wavelengths bent more than long)
- Diffraction – bending around edges
- Dispersion – separation of light into constituent wavelengths upon entering a transparent medium – such as rainbow spectrum
- Dual nature of light – wave and particle

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So, if you look at the basics of light, which is something you might have learned in your high school physics, you know that light can be absorbed, refracted, reflected, diffracted, dispersed or scattered. And you also know that light has a dual nature, we discuss often about the particle nature of light as well as the wave nature of light. As a particle, it vibrates in all directions, as a wave it has got a well defined characteristic wavelength and so on.

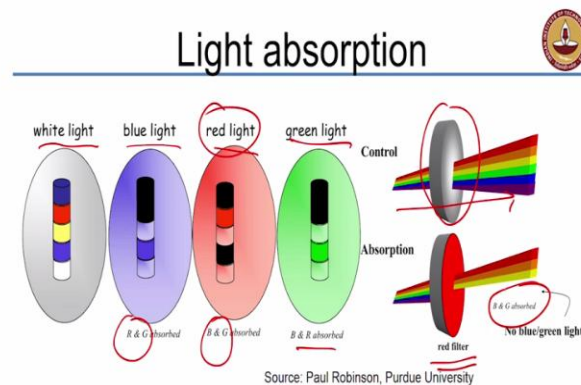
What are these characteristics that we are talking about? Absorption, is when you try to transmit light through a medium or an object, some amount of that light gets absorbed by the medium. So, what might happen is based on the characteristics of medium, some specific wavelengths from the light may get absorbed. So visible light has several wavelengths, based on the characteristics of the material it can absorb, probably, let us say blue or green light for instance, and allow the rest of the light to pass through. That is called absorption.

Refraction is change in direction from one medium to another. And it depends obviously on the characteristics of the medium itself, primarily the refractive index. And in general, when visible light travels through a different medium, the spectra of different wavelength tend to travel differently. For example, the short wavelengths are bent more than the long wavelengths.

Diffraction is a feature which happens whenever you have slits or edges, which may tend to bend the rays of light. So bending around the edges or when light tries to pass through a slit. It tends to again bend there are some beams that go straight but there are several beams that bend. That bending is called diffraction.

Dispersion, on the other hand, is the separation of light into constituent wavelengths upon entering a transparent medium, such as your rainbow spectrum. So, why is the rainbow caused? It is caused because of dispersion of light through water droplets. And you might have also done a high school physics experiment in which you take a triangular prism and then you try to actually look at the dispersion of light when white light crosses this prism. And of course, we already talked about the dual nature of light as a wave and a particle.

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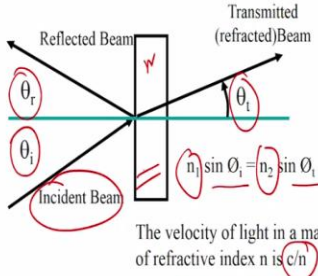
So, now, what is absorption? For example, if you have here white light, a blue light implies that you are absorbing the other primary colours, that is red and green. Red light, on the other hand, implies that blue and green are getting absorbed. So, if you have a filter for instance, which can absorb these wavelengths and only transmit the red, for example, if it absorbs blue and green and

transmits the red, then you call it a red light. A green light, on the other hand, is when you absorb the blue and red.

So, essentially what is happening here, for example, a control spectrum has all the colors, but in the case of a red filter, what you are doing is absorbing the blue and green and only transmitting the rest of the colors. So, essentially, absorption simply means that based on the characteristics of the material, it absorbs certain wavelengths and allows the other wavelengths to pass through.

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Reflection and refraction



- **Snell's Law:** The angle of reflection (θ_r) is equal to the angle of incidence (θ_i) regardless of the surface material
- The angle of the transmitted beam (θ_t) is dependent upon the composition of the material

The velocity of light in a material of refractive index n is c/n

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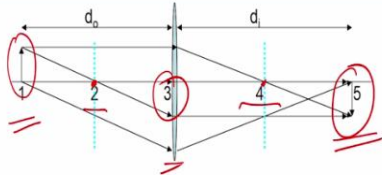


Now, Reflection and Refraction, so here you have an incident beam of light, which goes to a different medium, part of that beam is obviously reflected and you know that angle of reflection is equal to angle of incidence. But based on the refractive index 'n' of the material, you may have this beam which transmits through the material, but in a bent fashion. So, this bending produces a transmitted beam angle. And the relationship between incident beam angle and transmitted beam angle depends on the refractive indices of the 2 media. So, when light travels from air through glass, air has a refractive index of 1 and then glass may have a different refractive index. So, (n_1/n_2) will be essentially the ratio of the refractive indices of the air to the glass and your angle of refraction will depend on how different n_1 and n_2 are. Now, if you look at the velocity of light itself, in a material of refractive index 'n', it will be (c/n), where 'c' is the actual velocity of light ($c=3*10^8$ m/s).

Now, we know this law which relates the reflection angle to the incidence angle, which is called Snell's law. And of course, we also know that the refractive index is defined by the degree to which the light is bent on travelling to another medium, which is dependent on the composition of the material. So, already we have some basis for characterization using light. That basis is simply that if different phases in a material have different refractive indices, the light travelling through these phases will be bent to different degrees. Also, based on the composition of the material itself, the amount by which the light gets reflected or the energy of the reflected light will also be different for different types of phases. That is something we can relate to with respect to characterization. So, that is how we will use light microscopy to distinguish the different types of phases that are present in our sample.

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Optics - basics



1 - object plane
 2 - front focal plane
 3 - lens plane
 4 - back focal plane
 5 - image plane

Lensmaker's equation: $1/f = 1/d_i + 1/d_o$
 Magnification $m = d_i/d_o$
 Assumes that focal length is equal on both sides - OK for a thin lens
 If $d_o < f$, image will be on the same side as object

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So, this is ray diagram which is quite familiar to you probably from your middle school physics. So here you have a lens, and then you have an object here which needs to be imaged on the other side. And you know that the rays travelling through the object go through a point in the object plane which is known as the focal point. And of course, we also have parallel rays which go then through the lens on the other side, again it passes through the focal point. Always the rays tend to converge on this point known as the focal point, and then you form the image on the other side. So this is a simple single lens setup. For example, if you use a magnifying lens, you will be able to see objects on the other side which are magnified based on the property of your lens itself. So, this is the simplest experiment that you can do.

In the figure given, 1 is called the object plane, 2 is called the front focal plane, 3 is obviously the lens plane, 4 is the back focal plane and 5 is the location where the image is formed - the image plane. And again from high school physics, we know that the Lensmaker's equation relates your focal length to the distance of the image (d_i) and the distance of the object (d_o).

$$\frac{1}{f} = \frac{1}{d_i} + \frac{1}{d_o}$$

The magnification itself is nothing but the ratio of the image distance to the object distance.

$$\text{Magnification, } m = \frac{d_i}{d_o}$$

The farther the image from the lens, the greater will be the magnification. Now, when you observe things with a magnifying glass, you will also see that in some locations, you are actually getting a magnified image which is upright, but if you actually turn the magnifying glass away from your eye, you will also reach a distance where the object on the other side looks inverted. Quite a simple experiment, we must have done this several times in childhood. When you observe the magnifying glass very closely, you will see a magnified image on the other side, which is upright. But if you take it slightly further away from your eye, you will see an inverted image, which will not be magnified. So, that is a simple experiment, you can try that out, if you have forgotten it. But that is basically again related to the properties of the lens and the location of the focal point. That is about it. Of course, if your object distance is less than the focal distance, then the image will form on the same side as the object. If the object distance is less than the focal length your image formation will happen on the same side. Again, of course, you if you can take up the schoolbooks of 8th or 9th standard children, you will probably find a lot more explanations than we are actually having time to get into at this stage.

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Microscope optics



Compound lens system

Magnification $M = m_1 \times m_2$

With a single lens, a range of focal lengths can be used because the lens has no fixed position; also, the eye is itself an adjustable lens (variable f)

Eyepiece of a microscope – not so flexible!! Specific distance \rightarrow important for photo or video recording of image

Images that are in focus for a photo may appear out of focus to the eye!

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What about a microscope? A simplest microscope works with 2 lenses, you have the objective lens which is close to the object, and then you have the eyepiece lens through which you are actually viewing the object. So, what is happening here is that the intermediate space between the objective and the eyepiece lenses is where an intermediate image plane exists.

So, the initial ray diagram tends to form the image here (in place of intermediate image plane). But then when I try to view through the eyepiece, I need the image to form in my eye, or if I am keeping a camera here instead of the eyepiece, the image has to form in the camera. So, the overall image is actually the one that is getting formed here. This is the intermediate image which is formed because of the single objective lens. So depending upon the spacing between your objective and the eyepiece, you may be able to form images at different locations which are magnified to some extent when they come to the eye.

Now here since we are using a compound lens, that is more than 1 lens, the magnification is simply a multiplication of the magnification of the first lens and the second lens, obviously, more number of lenses you have, the greater the magnification. So in this case, your magnification is simply a multiplication of the 2.

$$\text{Magnification, } m = m_1 * m_2$$

Now, what happens is, when you have a single lens, you can actually use a range of focal lengths, because lens has no fixed position. If you take a magnifying glass, you can keep

changing the magnifying glass. That is why I said that when you hold the magnifying glass close, you will actually see a magnified view upright. When you take it further away, you will see a not magnified view which is inverted. Now, that is happening because you are playing with the focal length and the eye itself has an adjustable focal length. Your eye has a muscle inside which can actually adjust and focus at different lengths. I am trying to focus on the first bench here I can also focus on the last bench. It is not the same with a specific lens, which has only a fixed focal length. So, idea is simply that when you are trying to view with a single magnifying glass, you have the flexibility of changing the focal length.

So, when you work in a microscope system, on the other hand, you need to define the tube length, which is the length or distance between the objective and the eyepiece. And that is usually fixed, that distance is usually fixed. So, microscope has a specific distance, which is important for recording of the image to capture the image, either photo recording or video record. And one thing that you need to also understand is because the eye has a flexible, focal length, that means you can actually strain your muscles or relax your muscles to change the length that you are actually trying to view. When you actually observe under the microscope, the images that are clear to the eye may not be clear to the camera that you placed there because camera has to be fixed at one particular location. So, images that appear in focus in camera may appear out of focus to the eye and vice versa. Eye is the best lens possible. Because of the muscles that you have, you can actually adjust it to view anything. But in a camera, when you set up the microscope to capture the images on a camera, you need to ensure that you adjust to the cameras focal length, not to the eye's focal length.

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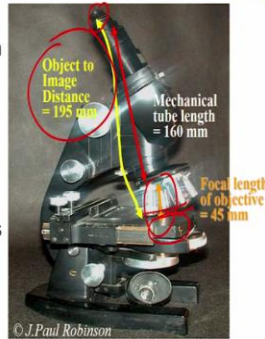
Microscope basics



- Originally conformed to the German DIN standard
- Standard required the following
 - real image formed at a tube length of 160mm
 - the parfocal distance set to 45 mm
 - object to image distance set to 195 mm
- Currently we use the ISO standards
- Modern microscopes are now infinity not 160mm

Source: Paul Robinson, Purdue University

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© J. Paul Robinson



Now because of this inflexibility in the objective to eyepiece distance, in most cases, the old microscopes had a very clear cut distance of the object to image of 195 millimeters. So, this is the eyepiece here on top, and that is the objective lens system and that is your specimen, which is being viewed (in figure in slide). So, in the old microscopes, these were conforming to the German standard (DIN standards).

So, what happened here in this case, the real image is formed at a tube length of 160 millimeters. So, this is the tube length from eyepiece to the top of the objective lens, that is basically your tube, the light tube which is carrying the light rays from the objective to the eyepiece, that tube length was 160 millimeters. The focal distance was set to 45 millimeters. So, your intermediate image plane should capture the light coming through the objective lens at a distance of 45 millimeters. So, object image distance was set to 195 mm.

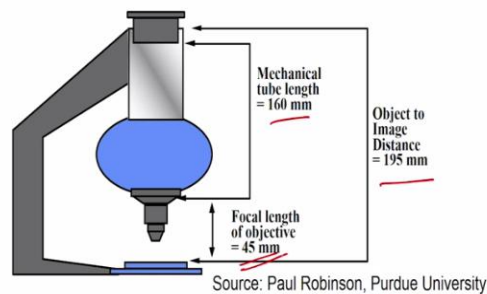
But today we have a lot of intermediate optics in the microscopes, which makes it possible for us to actually improve the quality of imaging. Modern microscopes do not have this fixed tube length to 160 millimeters, but they have what is known as an infinity focus. Now, what do you mean by infinity focus? You all realize this when you actually look at objects. When you try to get an object very close to your eye. You have to strain to read things. No I am not talking about reading things which are far off, which are extremely small letters. But what I am saying is, when you are trying to focus on something close by, it is quite difficult. It puts a

strain in your eye. So, here in the old microscopes, you had to focus your eye exactly at that length.

But now, in modern microscopes, what they do is, they adjust the tube with additional lenses, which makes it look as if you are peering into infinity. Now, if I am trying to focus on something very far away, an object that is extremely far away, I do not have any strain in my eye only when I am trying to see very close, I put my eye under strain. If I am trying to focus far away, infinity, I do not really have that kind of strain in my eye. So modern microscopes are embedded with light tubes that have multiple lenses, which makes it possible for your eyes to actually focus as if it is focusing on infinity, it is looking very far. You may actually get that sense, sometimes when you actually look into the modern microscopes. You may think that you are actually viewing an object that is far off. But in reality, it is right there. That is all because of the intermediate optics.

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Conventional microscope



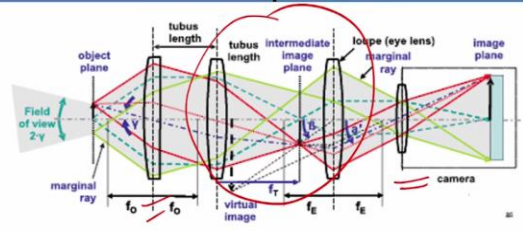
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So again, this is just a graphical description of what I just talked about. In the old microscopes tube length of 160 mm, object to image distance of 195 mm, and the focal length of the objective lens was 45 millimeters.

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The real picture 😊



<https://enacademic.com/dic.nsf/enwiki/75817>

In all microscopes the image is viewed with the eyes focused at infinity (mind that the position of the eye in the above figure is determined by the eye's focus). Headaches and tired eyes after using a microscope are usually signs that the eye is being forced to focus at a close distance rather than at infinity.

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Now, the real picture, because you have all these compound lenses inside, is like this (Figure in slide). So you have your objective lens here, and you have your eyepiece lens here, but all these are the intermediate lenses that are present in your tube. Now, without really getting into the wave optics there because it is quite complicated, the idea is simply that this kind of a system which processes multiple lenses inside the tube, helps your eye to relax and focus on something which is at infinity that means you are looking at an object that is quite far away, and that makes it quite easy for you to look at things without straining your eyes.

So, generally when you use a microscope, which has this fixed length, you will end up with headaches and tired eyes. So, whenever you are trying to focus on a close distance, you always get a headache or tired eyes, but if you are focusing on something far away, you do not have that problem. Which is why when you read books, people say that the optimal distance of the book should be about 25 centimeters, at least 25 cm should be the distance at which you keep your book. If you try to read very close, you are obviously going to be straining yourself.