OPTICAL MICROSCOPY NOTES®
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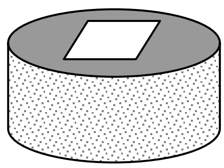
Introduction:

The use of "Optical Microscopes" in "Materials Evaluation" is very popular since it enables the researcher to observe the *"internal structure"* of materials. This information can then be used *to improve material processing and properties* which form the foundation of materials science and engineering (MSE).
The microstructure generally ranges from the atomic scale (0.1nm) to 1mm(1000μm) with the most widely used scale of 1-1000 μm. Practically the optical microscopes can be used up to 2000x at which the resolution becomes so poor that objects smaller than 1 μm cannot be distinguished. But since most materials have grain sizes in the range 1-100μm optical microscope (OM) is a perfect low cost tool. Typical microstructural features are grains(single crystal), precipitates, inclusions, pores, whiskers, defects, twin boundaries, etc. Most of the manufacturers use OMs for process control and R&D.
There are mainly two types of optical microscopes (OM): *Reflected Light* (RL) and T*ransmitted Light* (TL). In the former the light is reflected from the surface of the specimen while in the latter the sample is so thin (20-50μm) that light passes through it. A third but less important type may be the *stereo microscopes* that do not require any sample prep and are generally used to observe fracture surfaces, electronic curcuit boards, etc.

Reflected Light Optical Microscopes are the most widely used machines in materials science. Because they involve light reflection the specimen surface must be very well polished to a "*mirror finish*". In order to have a mirror finish the sample goes through a number of "sample preparation treatments":

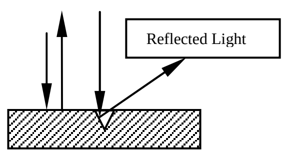
(1) **Cutting:** Specimen should be cut to <1.25 inch with a hacksaw or a disc saw. Care must be taken not to heat the sample during cutting.



(2) **Mounting:** Hot mounting in a press w/phenolic resin or cold mount in polyester. The former is faster and more generally used for metals while the latter is favored for ceramic samples.
(3) **Grinding:** Successively sized SiC grinding papers (180-320-600-1200 Grit) are rotated on a wheel (~50-250 rpm) and the sample is pushed face down while watercooling. Small SiC particles are glued to the grinding paper so these are also sometimes called fixed abrasives. These particles slowly remove chips from specimen surface.
(4) **Polishing:** Same as grinding except the abrasive particles are loose and no watercooling is performed. Diamond or alumina suspension having particles 0.05 - 30μm diameter are used.
(5) **Etching:** Selective removal of certain microstructural features to reveal the internal structure. Use a hood and do not inhale acid vapor. Wash the specimen thoroughly to avoid damage on objective lenses.

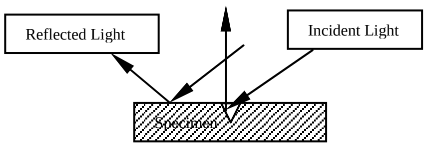
Because OM examination of any material is actually a sampling operation from a larger material, care must be taken not to change the microstructure during sample preparation. It is important that the sample must be *representative*. The sample is examined in OM in two main modes:

(1) **Bright Field (BF):** The polished surface appears bright and the surface irregularities such as grain boundaries appear dark. The reason why grain boundaries appear dark can be

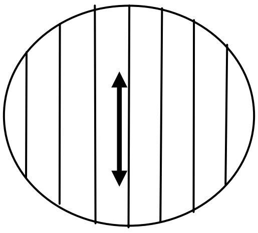


clearly seen in the figure. Light reflected from the grain boundaries do not go back to the objective. The same thing is true for precipitates that also appear dark. BF mode is more commonly used than DF.

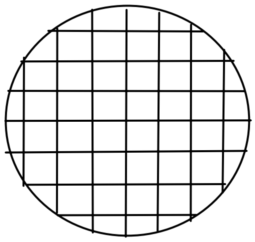
(2) **Dark Field (DF):** The incident light is fed from an angle therefore polished smooth surface appears dark and surface irregularities appear bright. DF is less commonly used. Applications are generally in grain size measurements and in judging the quality of polishing.



BF Illumination is used in the following modes:



(1) Plane polarized light (PP): Most widely used illumination technique in MSE. Grain size measurements, precipitates, inclusions, are all observed in this mode. PP is achieved by inserting a polarizing filter into the incoming light path. This filter kills the wave's electrical wave component's vibration in all directions except in one direction. This direction corresponds to that of the filter orientation. E.g. light passing through the window through the blinds (jaluzi).



(2) Cross polars or cross-nicols (CP): When two polarizing filters are placed at 90^{o} to each other i.e.when they form a cross shape. Then no light can pass and we have extintcion. In extinction, the image is totally black when there is no specimen in the microscope or when the specimen is totally isotropic like single phase austenitic steel. Only materials with a measurable level of "birefringence" can show up in CP mode. *Birefringence* is the difference between the refractive indices of a material in different directions ($\delta=n_x-n_y$). Generally noncubic materials are birefringent. So using this imaging mode one can tell where the cubic and noncubic materials are located throughout the microstructure. This allows the observation of non-cubic phases in the microstructure which may play a major role in some properties like high temperature strength. This information can be very valuable especially in ceramics. CP is less frequently used in RL.

(3) Differential interference contrast (DIC): New technique. Allows surface relief to be shown much like an SEM-SEI (secondary electron image). A wedge-shaped filter (Nomarski Filter) is inserted in the light path that results in the wavelength of the light to be slightly modified. This produces a retardation of the light wave and consequently a relief effect on the observed image.

TL-OM: Used generally by geologists (petrographic microscope), biologists and ceramists but never by metallurgists because metals are opaque.

References:

1) http://www.metallography.com
2) M.Davidson and M.Abramowitz, "Optical Microscopy", a pdf file available at Olympus Co. Web page. http://www.olympusmicro.com/primer/opticalmicroscopy.html
3) http://www.engineeringbookstore.com/smeb/Metallography/Metallography.htm

Some Selected Machines that must be present in a Metallography Lab

Cutting Machine:
A high speed saw that uses 25 cm diameter resin impregnated corundum cutting disc along with watercooling. Disc rotates at around 3000rpm. Coolant is circulated in the system.

Hot Mounting Press
Samples are hot mounted in a phenolic resin to form a 32mm diameter pellet containing the specimen.

Polishing Machine:
Shown in the figure is a fully automatic polishing machine that has a rotating wheel (0-500rpm) and an automatic head.

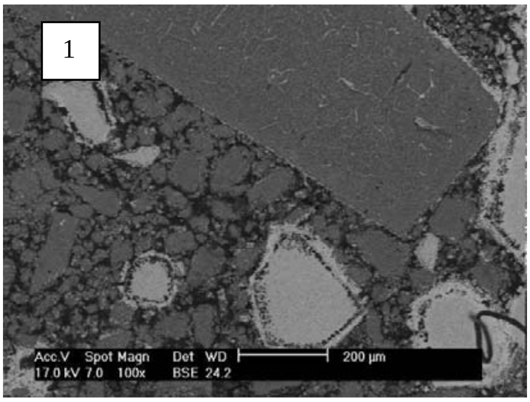
RL-OM: Nikon Epiphot 300 Inverted Reflected Light Optical Microscope

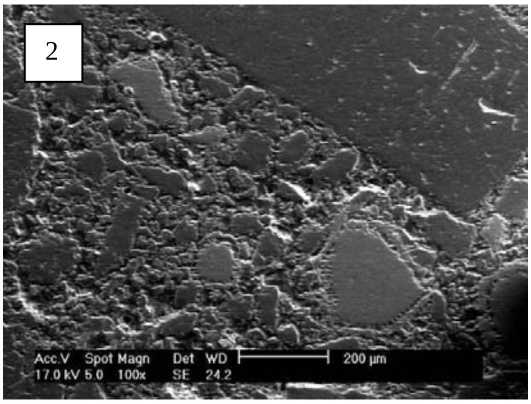
Sample is placed upside down and observed as such. This allows the examination of large samples. Another version of this machine is the upright RL-OM. We have the latter one in our lab.

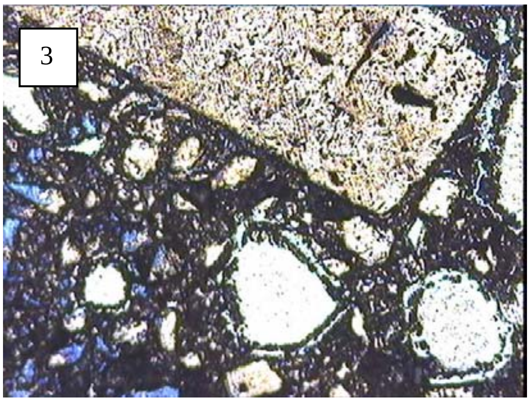
Four Different Image Modes:

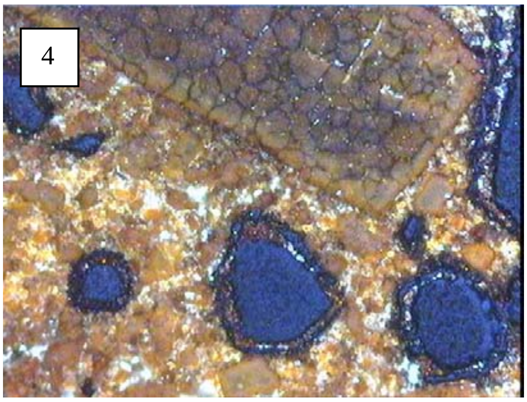
(1) SEM BSE: Scanning electron microscope, Backscattered Electron Image mode,
(2) SEM SEI: Scanning electron microscope, Secondary Electron Image mode,
(3) RL-OM PP: Reflected light optical microscope, plane polarized light mode,
(4) RL-OM CP: Reflected light optical microscope, cross polars polarized light mode.

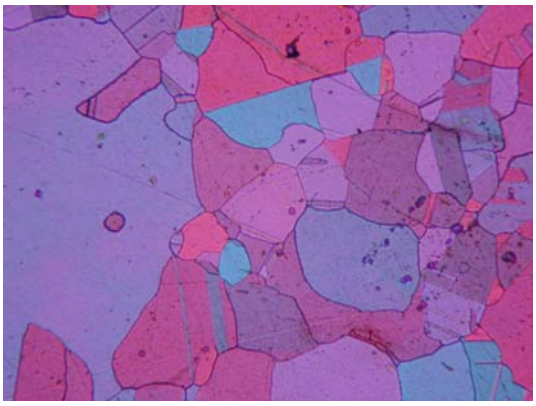
The sample was taken from a commercial refractory brick. White features in figure 1 are chromite grains while the large feature on top half of the micrograph is a periclase grain.











A Ni-Cr Superalloy microstructure:

Straight lines are twin boundaries.
Each color indicates a grain. Occasional Precipitates are visible.
Magnification=~750x
Cross polars. Each color is a sign of different crystallographic orientation.
Sample taken from an aircraft part.

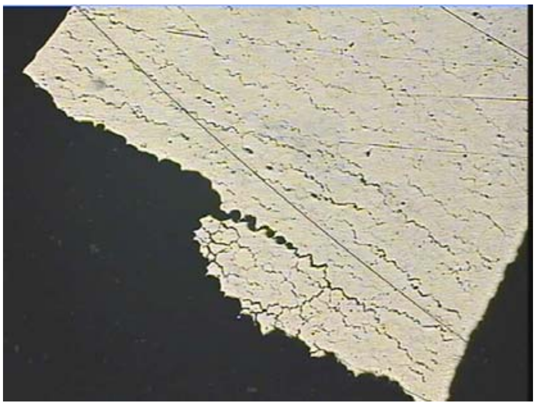


Figure: Creep, An aircraft part (F-110 inner liner) that was used at T>700oC for long periods. This service abuse led to creep cracks in the microstructure. Notice that the grain boundaries are opening up under the stress.

Figure Fatigue crack growth
Notice the progression of crack from bottom right to top left as the fatigue cycle progresses.

Figure: FOD (Foreign object damage) on a J-79 Turbine blade. Notice the bolt-shaped dent created upon impact of the free bolt in the engine.