Under the Microscope:
Let’s Focus on Optical Microscopy

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What we’ll talk about today…

Optical Microscopy

- Brief History
- Typical Microscope Parts
- Features (Bright Field, Dark Field, DIC, Stitching)
- Prep Procedures
- Examples
About NSL Analytical

NSL provides independent laboratory testing services to a diverse array of customers within regulated end-markets, where testing speed, accuracy and consistency are mission critical to operations.

Our teams of chemists, engineers and metallurgists provide scientific expertise in materials testing with a focus on metals, alloys and technical ceramics that are utilized in critical end market applications.

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The Microscope
Optical Microscopy

Observations made optically

- Grain size
- Grain orientation
- Cleanliness (Inclusions)
- Surface conditions – e.g., alpha case, (de)carburization
- Porosity/dendrites in castings
- If looking at a failure, may get data on cause of cracking

Optical Microscopy

Optics are dependent on wavelength

- Life-science microscopes depend on transmitted light, where metallurgical microscopes depend on reflected light.
- Theoretical minimum that can be seen is 500nm (at least the length of a wavelength of green light).
- Typical maximum magnification is 1000-1250x.

Wavelength image, courtesy of NASA
Optical Microscopy

Brief history

- Dubious claim - Hans and Zacharius Jensen (1595), two lens compound microscope
- 1st recorded obs. (1619) - Dutch Ambassador Boreel sees one in London in the possession of Dutch inventor Cornelius Drebbel - an instrument about eighteen inches long, two inches in diameter, and supported on 3 brass dolphins
- 1st published account of observations (1625- Stelluti and Cesi)
- Robert Hooke, Micrographia, 1665 (fleas and cork)
- Sorby develops metallurgical microscope to observe meteorites (1863)

From Wikipedia, “History of the Microscope”; Biological Microscope, courtesy University of Cambridge
F.G. 2.
Independent Focussing Device applied to Metallurgical Microscope as used for Photography.
Microscope
Microscope

Optical system of Metallurgical Microscope

- Camera
- Eyepiece lens
- Tube
- Prism/mirror
Microscope

Viewing options

- Light – dark field, light field, DIC, fluorescence
- Visual or camera
- Motorized turret objectives (2.5x to 100x)
- Stage x, y, z motorized (joystick or other)
- Touch screen on front, software controlled, controls in software
Why Optical Microscopy?

Some of the basic foundations of materials science

- The mechanical properties are governed by the structure on a microscopic scale.
- The ability to change the structure predictably allows the engineer to reasonably surmise (in an ideal world) the mechanical properties of the material when processed in a prescribed manner.
- Most materials are not ideal.
Sample Prep and Viewing Unetched Samples
Sample Preparation

Preparation for observation:

- Sectioning
- Mounting
- Polishing
- Observation (unetched)
- Etching (acid, bases, tint to bring out structure)
- Observation (etched)

Mounting Press

Automatic Polisher
Unetched Steel

MnS inclusions in 1214 steel


Al₂O₃ inclusions

Stitching

Oxide on steel surface
Cast Irons

Cast Irons

Type A                Type C                Type D                Nodular (ductile)

Porosity

Pressed and sintered Fe-0.8%C alloy

Mo-1W

Etched Samples
– Non-ferrous
SAFETY FIRST!
Etches are usually made from hazardous chemicals – strong acids, bases, explosive when dry, cyanide

- Steel (Nitric or Picric acids)
- Stainless steel (Oxalic, electrolytic)
- Aluminum (Hydrofluoric, HCl, Nitric)
- Copper (Ammonium hydroxide and H2O2)
- Molybdenum (Potassium ferricyanide and NaOH)
Copper

Cast C11000 copper (0.04% O)

C10200, “oxygen-free” (0.001% O max), cold-worked and annealed

Copper

Using different light techniques

Bright field

Dark field

Cu-12Al bronze, “martensitic” structure

DIC

From: ASM Handbook, Volume 9: Metallography and Microstructures,
G.F. Vander Voort, editor, p332–354
Brass and Titanium

70-30 cartridge brass, hot rolled and annealed

Ti-4.5-3-2-2 with alpha case

Titanium – DIC light

Bright field
DIC

Basketweave pattern, Ti-6-4 alloy

From ASM Handbook, Volume 9: Metallography and Microstructures, G.F. Vander Voort, editor, p775–78
Aluminum Castings

A356

Metallic Mold

A390

Sand Mold

Unknown

Aluminum second-phase ID

A413 –
Si-grey
Al2FeNi – light grey
Mg2Si – black script

A390 –
Si-grey
Al2FeNi – light grey
Mg2Si – black script
Al7Cu4Ni - brown

Etchant – 25% nitric

Refractory Metals

Tungsten

Nitric, HF, Sulfuric - plate
DIC lighting

Molybdenum

Murikami's reagent, wire

Metallic Powder

Water atomized iron powder (SEM image)

Water atomized tool steel powder
Etchant – nitric/picric

Metallic Powder

Gas atomized stainless steel powder (SEM image)

Stainless steel powder at various stages of sintering:
- As-compacted (upper left)
- Sintered (lower right)

Additive Manufacturing

Nickel-chrome alloy powder, SEM image

Nickel-chrome alloy, after laser-powder-bed-fusion (LPBF), showing microcracks

Nickel-chrome alloy with adjusted chemistry, after laser-powder-bed-fusion (LPBF), showing no microcracks

Courtesy NSL/Praxair
Etched Samples
– Ferrous
Steel

Low carbon steel (1008)
Marshall's reagent

Low carbon steel (1008),
50% reduction
Marshall's reagent

Steel

Low carbon steel with Galvalume coating
1% nital

Medium carbon steel (1040), annealed
4% picral

Medium carbon steel (1040), spherodize annealed
4% picral

Pack carburized 1015 steel at 1725 F
Top photo, 1 hr
Bottom photo, 4 hr.
4% picral

Decarburized 4118H steel
4% nital

Stainless Steel

Al-6XN
HCl-Nitric-Acetic
Bright Field

Al-6XN
HCl-Nitric-Acetic
DIC

Stainless Steel

Cast 303, unetched

2205 duplex
20% NaOH

Welds

Welded 22-4 stainless
20% NaOH

A710 steel plate, SAW
15% nitric

Cast Irons

Ductile iron
Unetched

Ductile iron
2% nital

Cast Iron – Polarized Light

- Ductile iron graphite nodule, brightfield
- Ductile iron graphite nodule, polarized
- Grey iron flake, polarized

Microscopy in Failures
Intergranular attack at the surface of a nickel alloy steam generator tube from deposits on the surface.
Failure Analysis

Sensitization, 304 stainless, in MgCl₂
Mixed acids etch

Stress corrosion cracking, 304 stainless, in MgCl₂
Mixed acids etch

Rolling contact fatigue, bearing steel
Unetched

Forging lap in steel ski lift component
Unetched

W.T. Becker, R.J. Shipley, editors, p3-23, 941-956
Summary

- History of optical microscopy
- Parts of a microscope
- Typical observations
- What some metals look like unetched
- What some metals look like etched
Let's Talk Tech!

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Join Us For Our Next Tech Talk!

Practical Approaches to California Proposition 65 Testing
January 14, 2021 at 2:00PM EDT

Hosted By
Dave van der Wiel
Director of Technology Development

Sarah Baskerville
Analytical Chemist