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A Few Microscopical Techniques for the Characterization of Materials

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IMS
Long Beach, CA

IMS International Metallographic Society ASM INTERNATIONAL

ASM INTERNATIONAL

Abstract

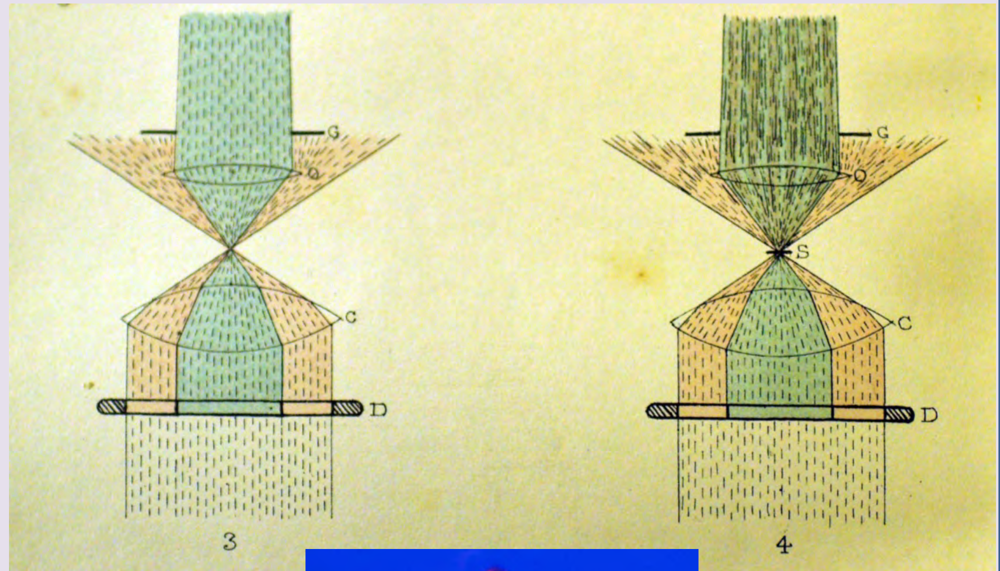
- Three techniques will be discussed to illustrate microscopy as an invaluable tool in material characterization. The first considers that one often seeks to differentiate one component from another or even from the matrix. An old and all but forgotten technique from the 1890s called Rheinberg illumination provides a simple way to accomplish this. Experienced metallographers can observe a cross-sectional sample and qualitatively determine the general directional orientation of a grain or sets of grain. The potential value of knowing grain orientation in regards to understanding material properties (or for interpreting forces having acted on a sample) has resulted in the recent rise of Electron Back-Scatter Diffraction (EBSD) as a technique to provide semi-quantitative to quantitative data in this area of research. A poor man’s alternative to EBSD can be had using multi-image reflected polarized light microscopy which will be shown. Similarly, the ability to convey technical information on characterization to others, both quickly & efficiently often rests in representing a, enormous amount of visual information simply, clearly and distinctly. The last technique demonstrated will involve stacking images to create 3D depth-of-field delivery to aid in this pursuit.

Three Techniques

1. Rheinberg Illumination
2. Grain orientation determination
3. 3D depth-of-field

1.0 Rheinberg Illumination (1896)

- Rheinberg, J. “On the addition to methods of micro research by new way of optically produced colour contrast (as communicated by E.M. Nelson),” *Journal of the Royal Microscopical Society*, pp 373–388, 1896.
- Rheinberg, J. “Note on Coloured Illumination,” *The Journal of the Quekett Microscopical Club,* 2:6, pp 346–347, 1897.
- Rheinberg, J. “Note on a New Modification of Double Colour Illumination,” *The Journal of the Quekett Microscopical Club,* 2:6, pp 438–438, 1897.
- Rheinberg, J. “Notes on Colour-Illumination with Special Reference to the Choice of Suitable Colours,” *Proceedings of the Royal Microscopical Society,* 19:2, pp 142–146, 1899.



Havics, Contrast Methods in Microscopy, Rheinberg Illumination, Microscope, 62, 4, 157-169, 2014

Darkfield

- Transmitted

Darkfield Microscopy

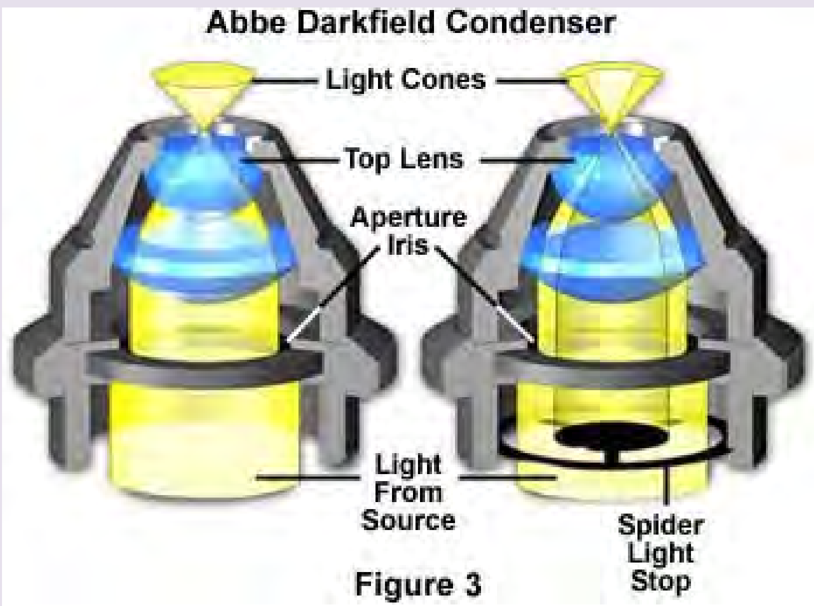


Figure 3

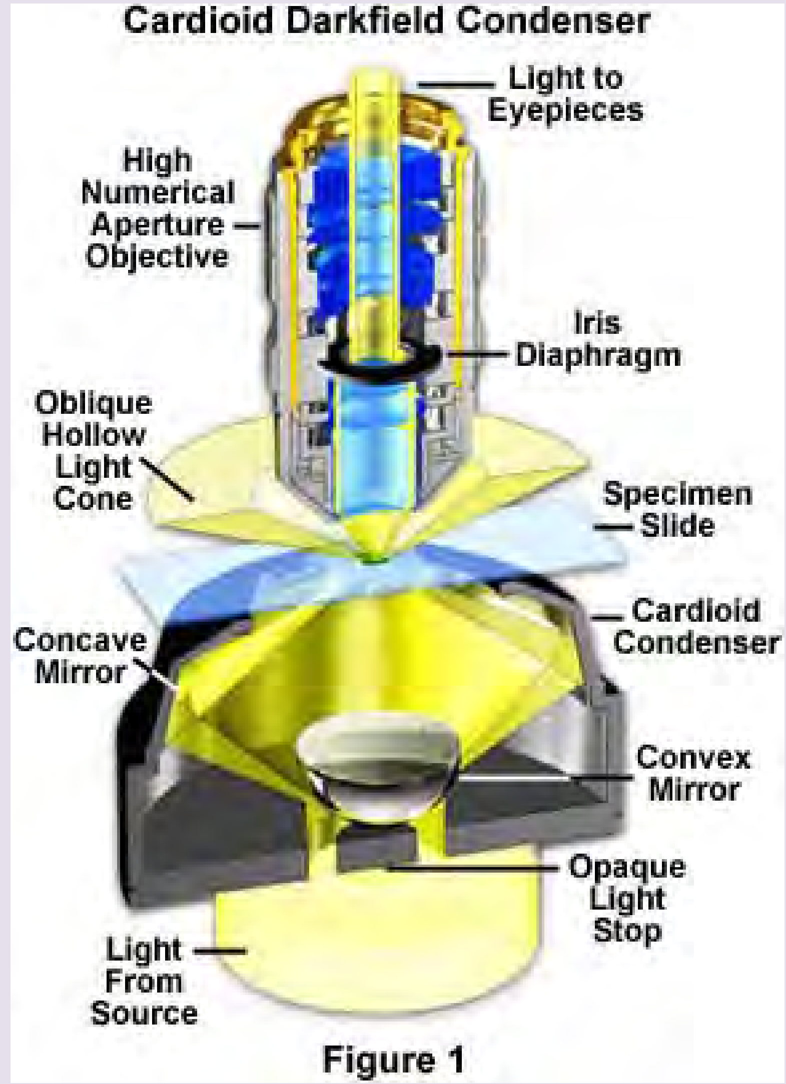


Figure 1

http://www.olympusmicro.com/primer/techniques/darkfield.html

Darkfield

- Reflected Darkfield

Figure 2

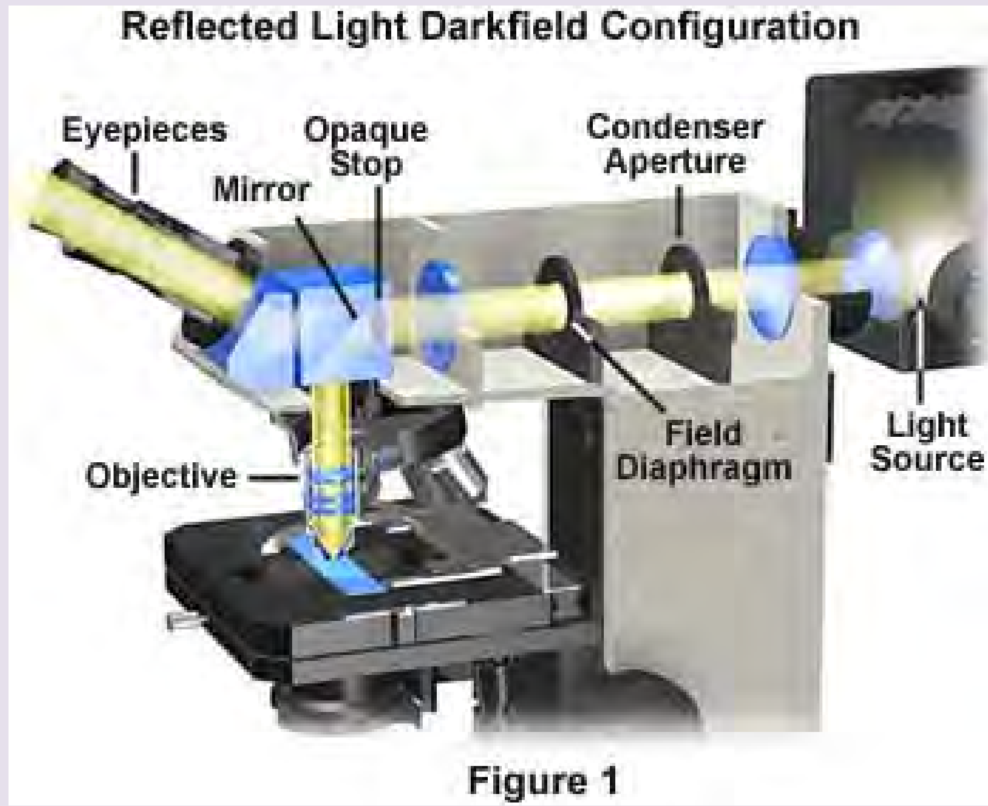
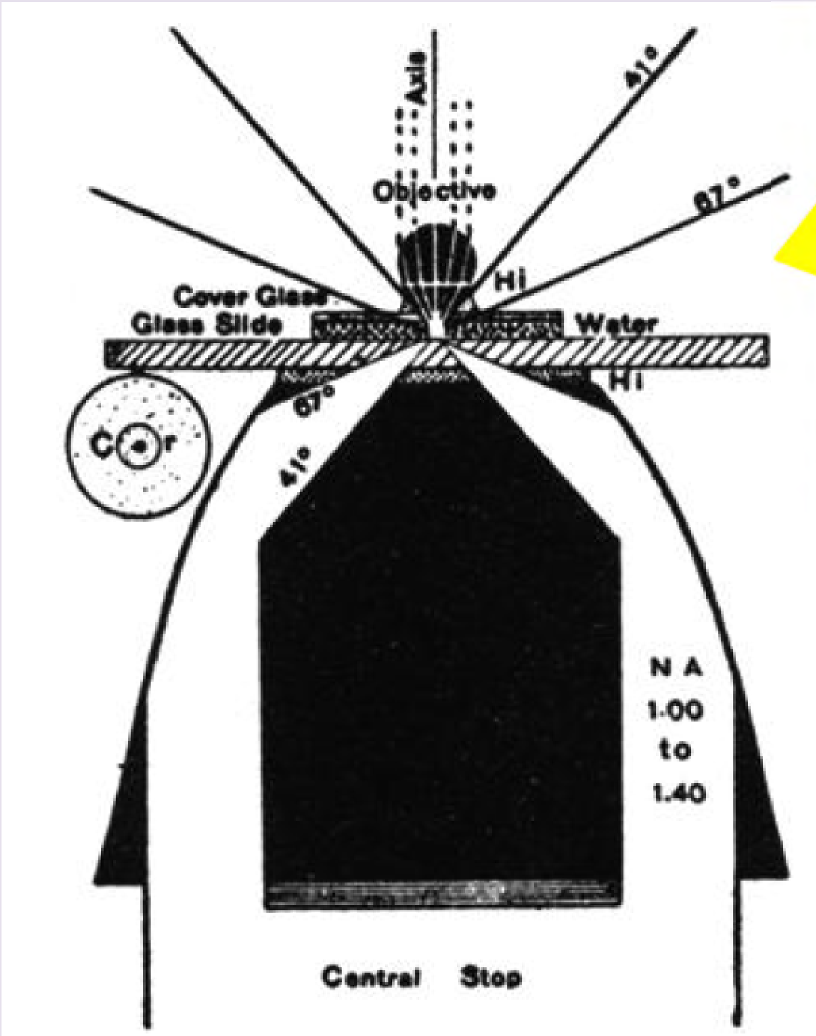


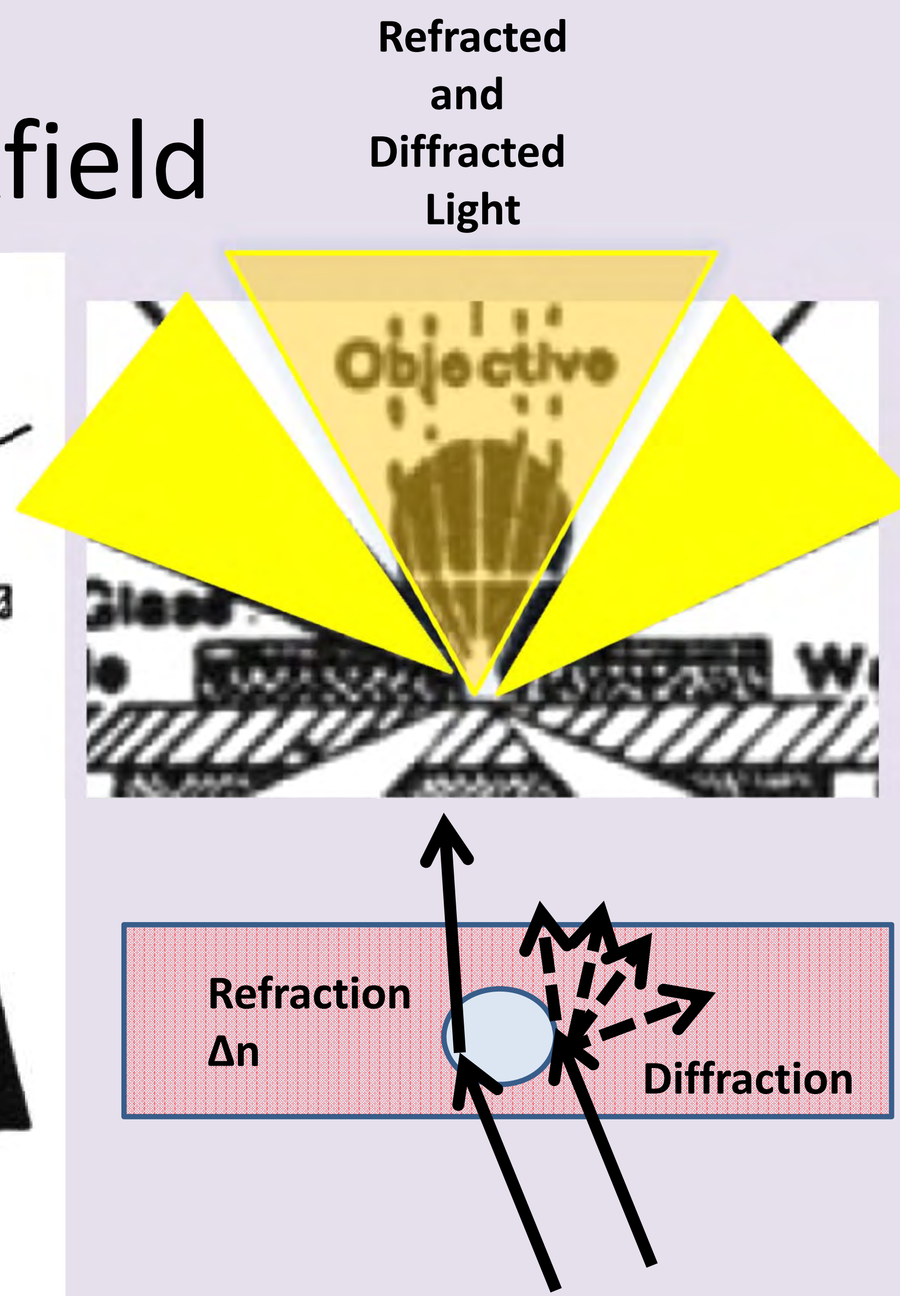
Figure 1

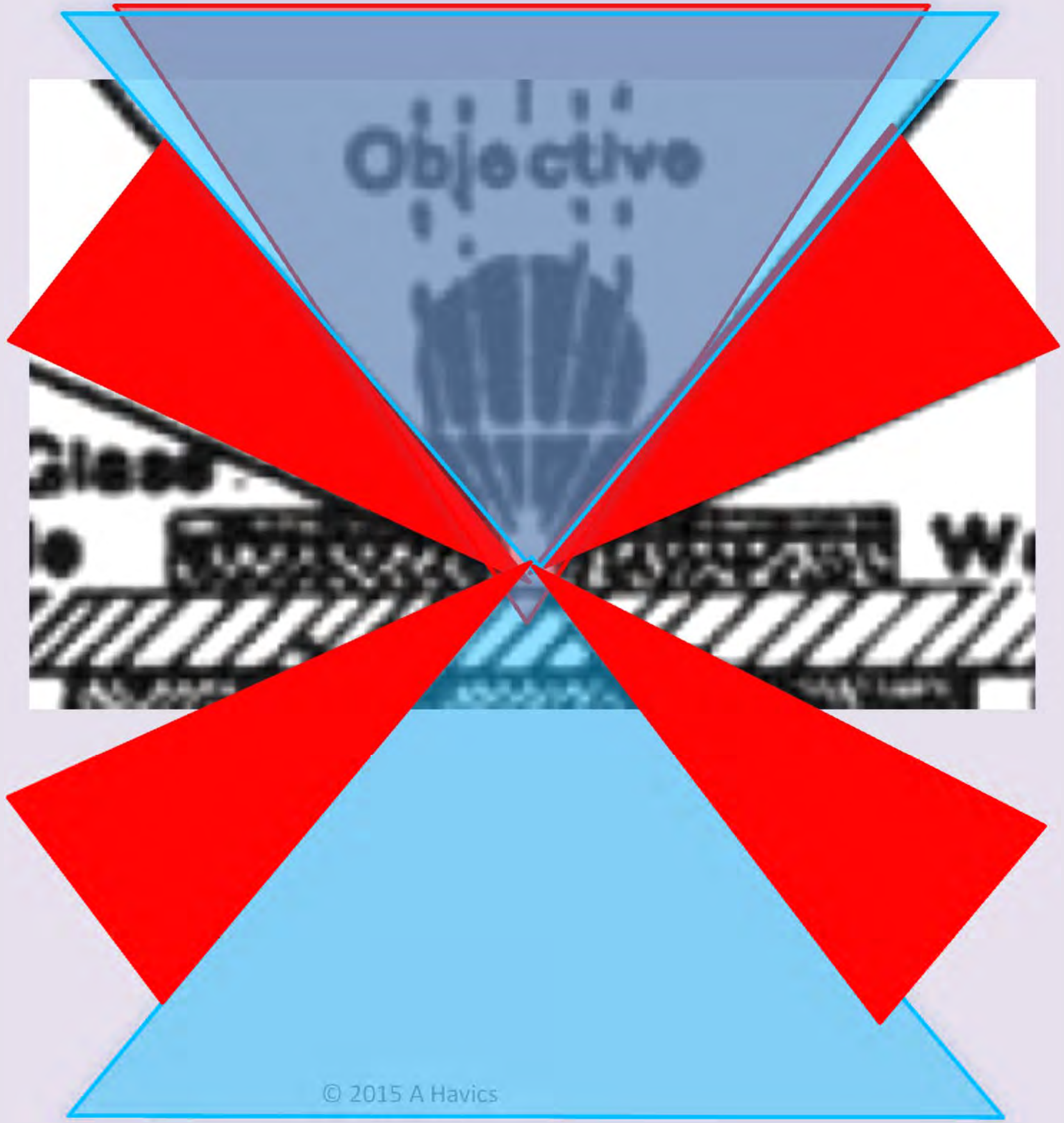
http://www.olympusmicro.com/primer/techniques/darkfield.html

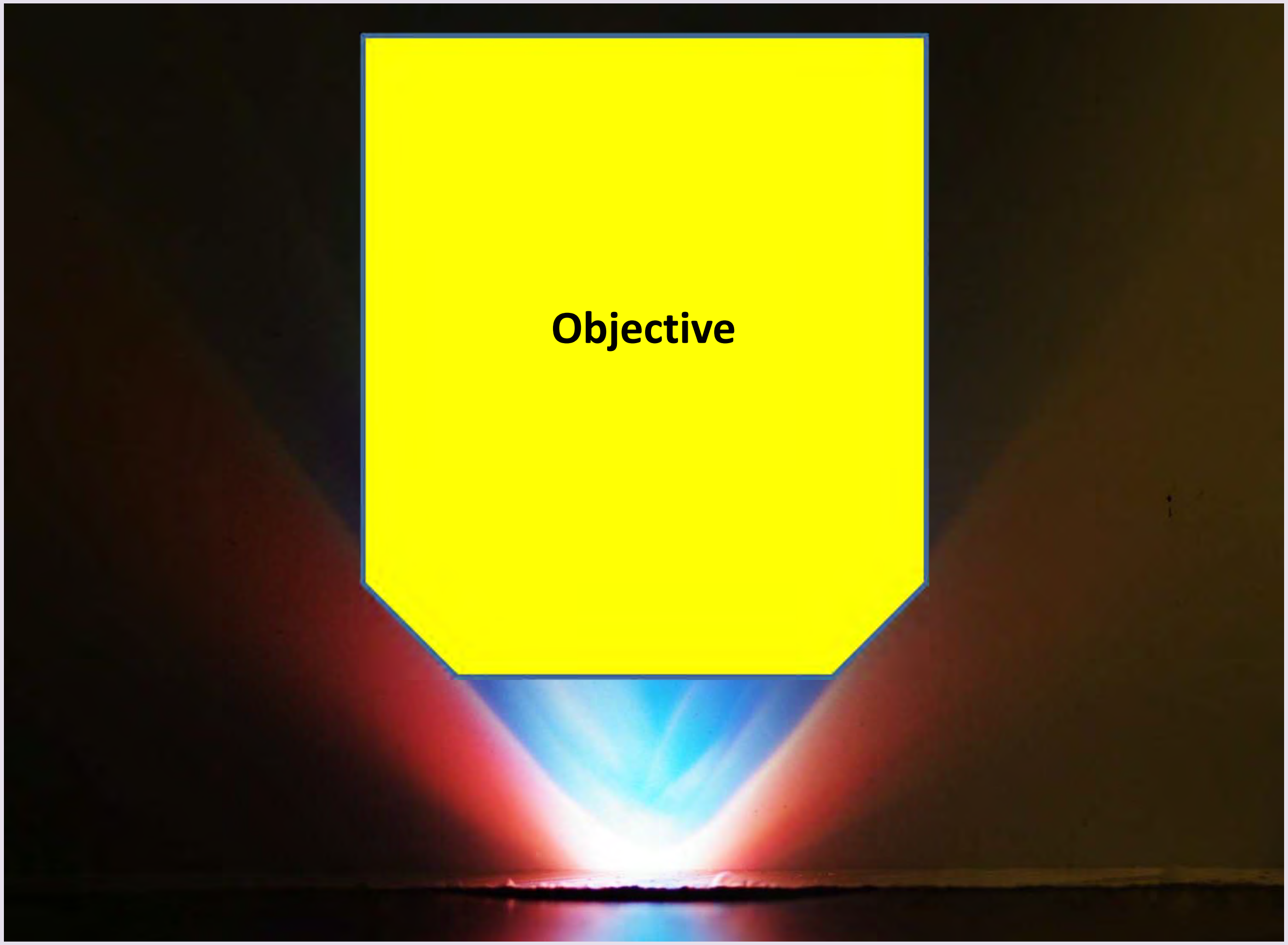
Darkfield



Gage, Modern Dark-Field Microscopy and the History of Its Development, Trans Am Microsc Soc, 39, 2, 95-141, 1920



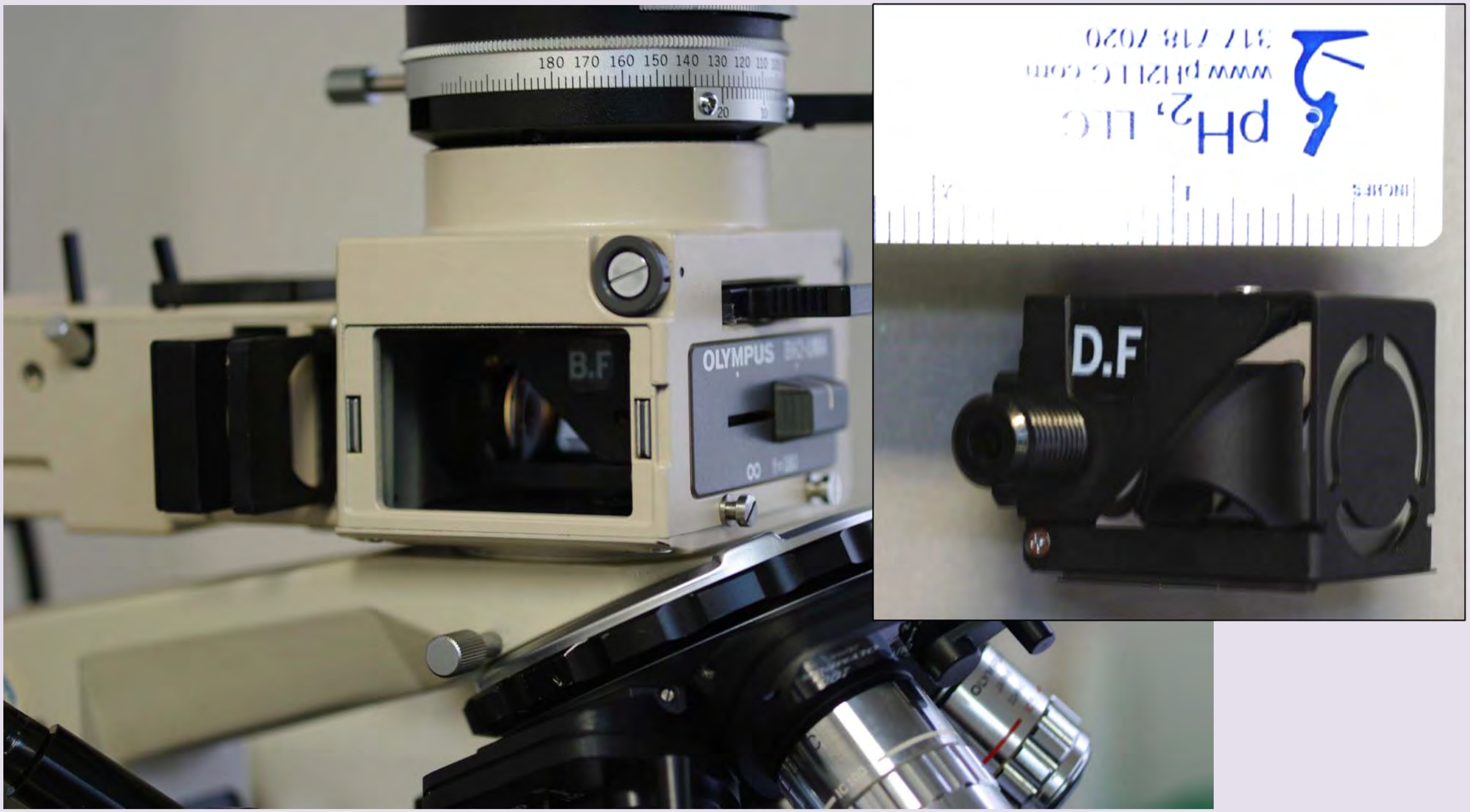

Objective



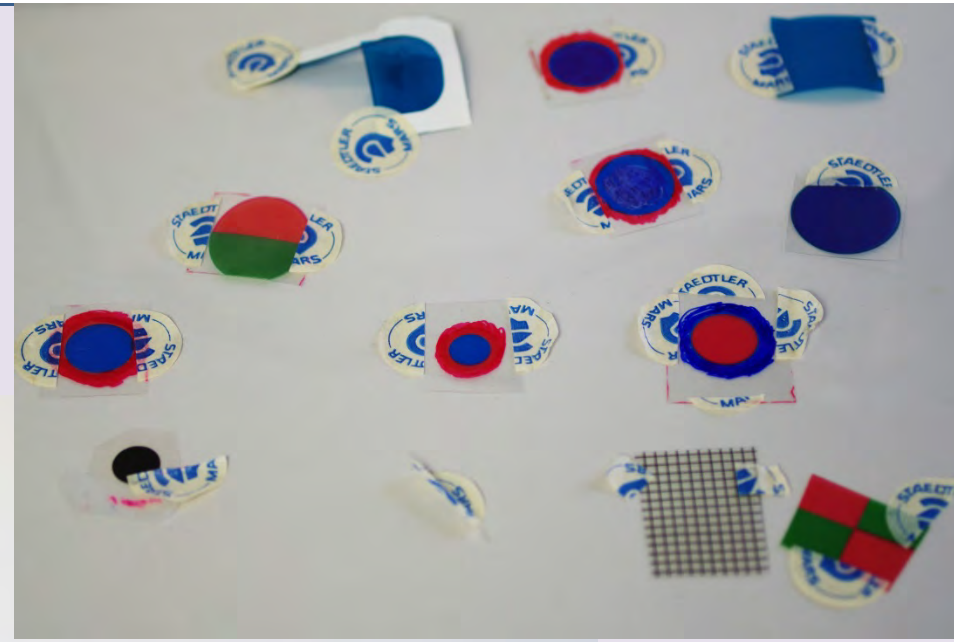
Havics, Contrast Methods in Microscopy, Rheinberg Illumination, Microscope, 62, 4, 157-169, 2014


A seemingly fast but
challenging year will give
many of us time to pause
and reflect on
the value of life
– new and old.

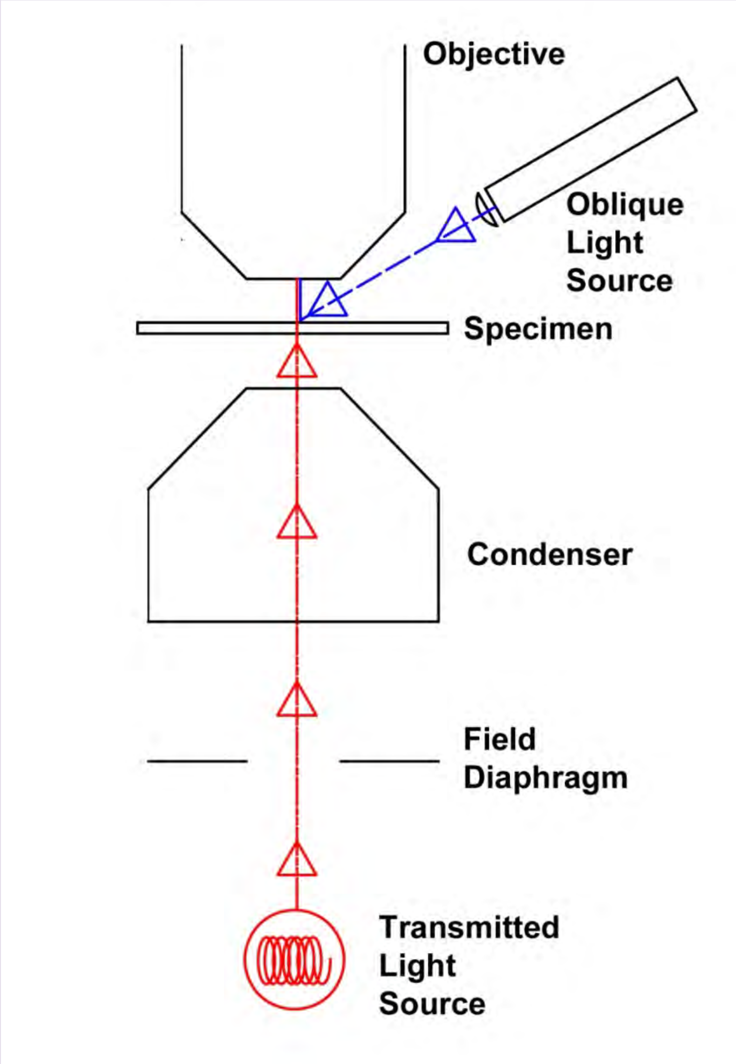
Reflected Darkfield Setup




B.F.

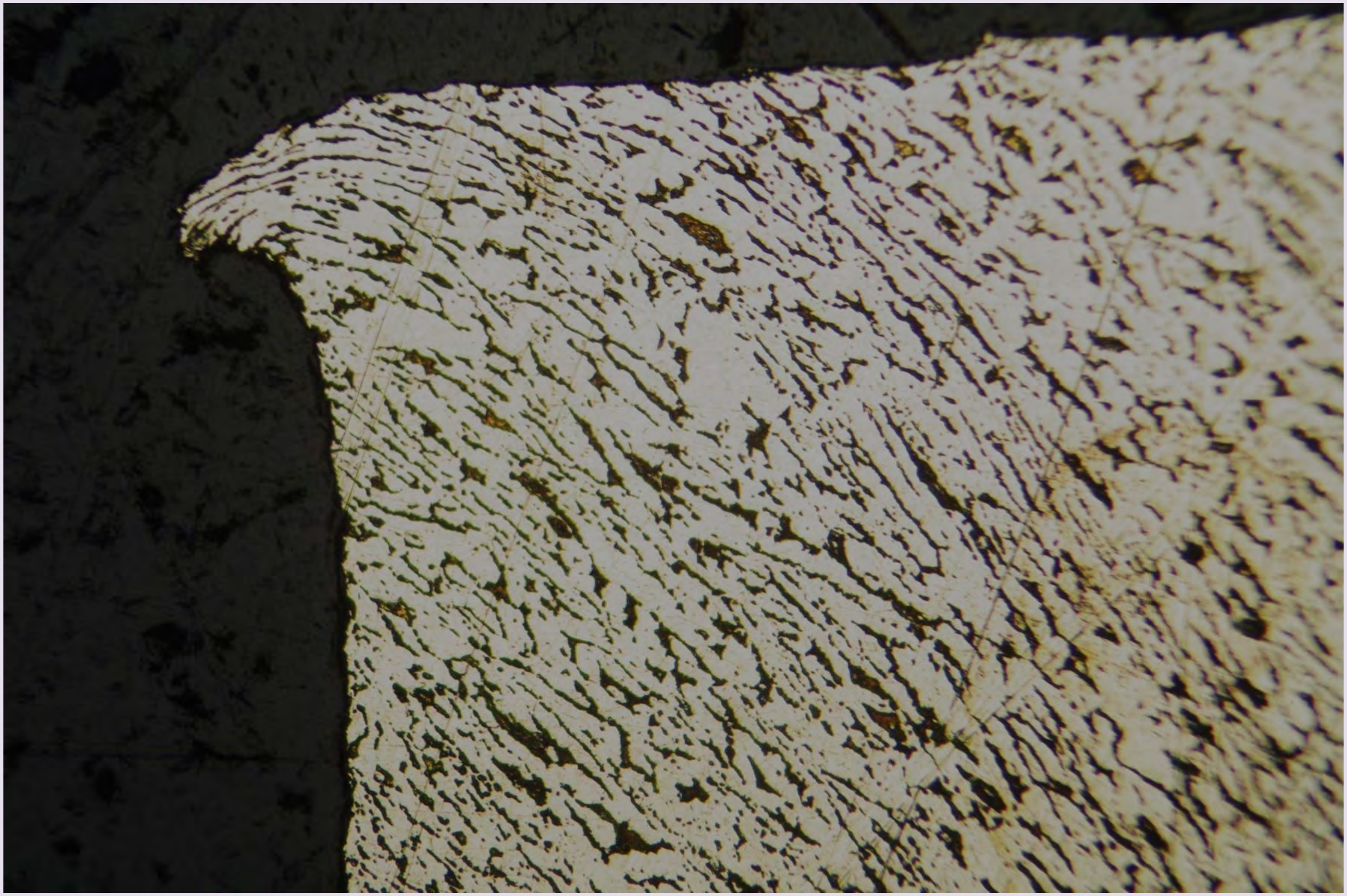

STAEDTLER

Option 2: Sandwich Rheinberg



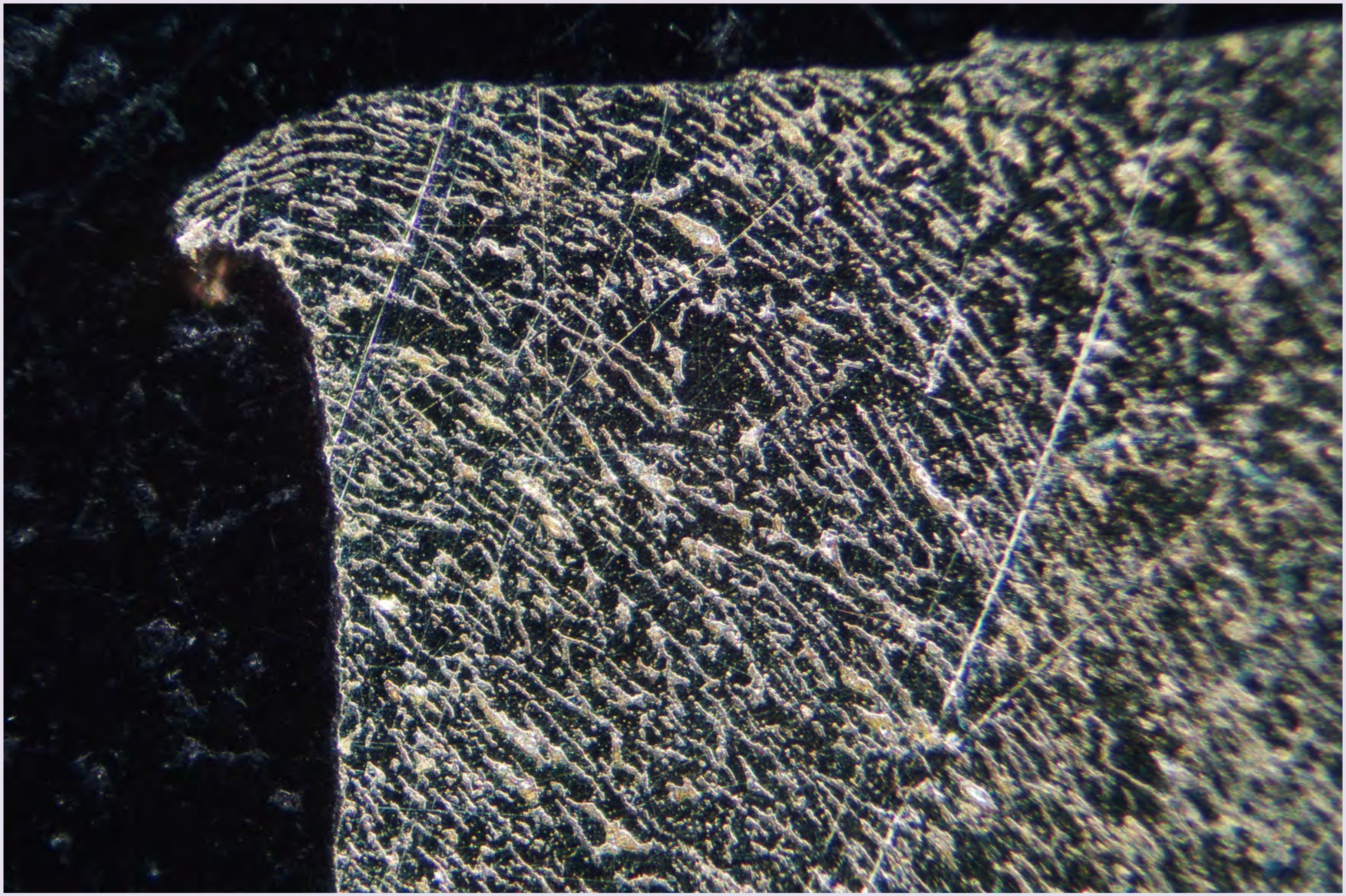
Havics, Contrast Methods in Microscopy, Rheinberg Illumination, Microscope, 62, 4, 157-169, 2014

Brightfield

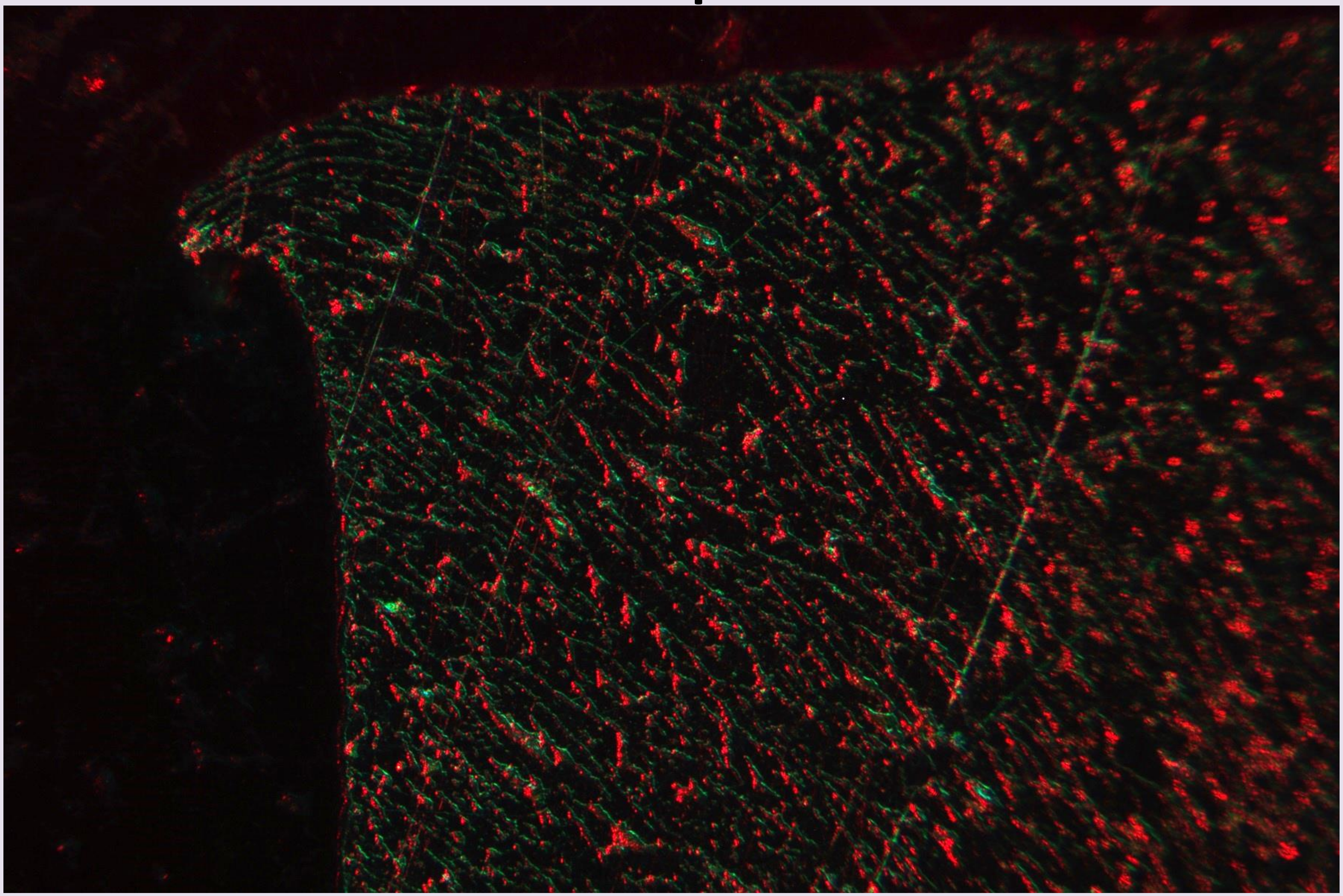


Quick & Dirty prep of Plumbing fitting, unetched

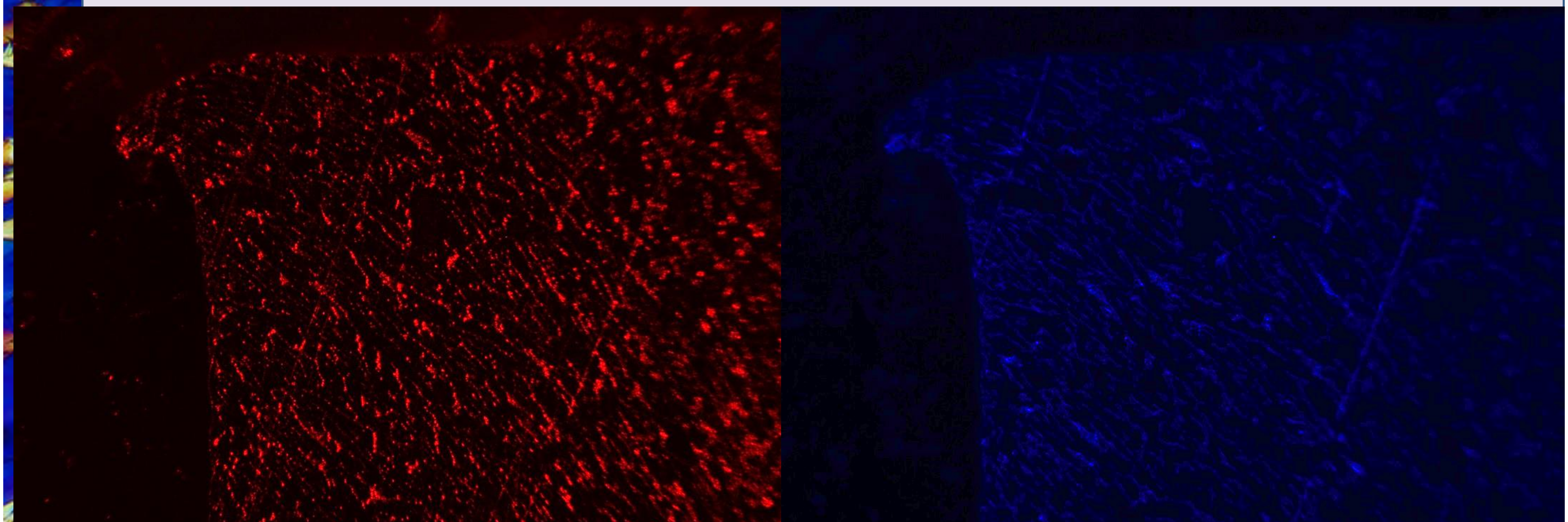
Darkfield



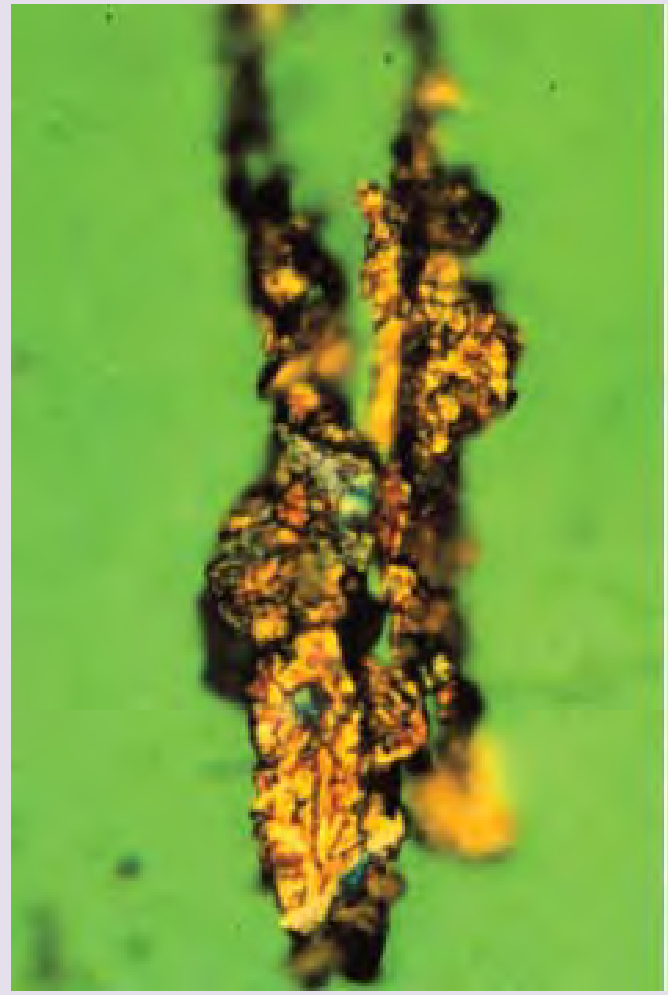
Sandwich Rheinberg: Blue DF Rfl, Red Oblique



Rheinberg Red & Blue Extracts

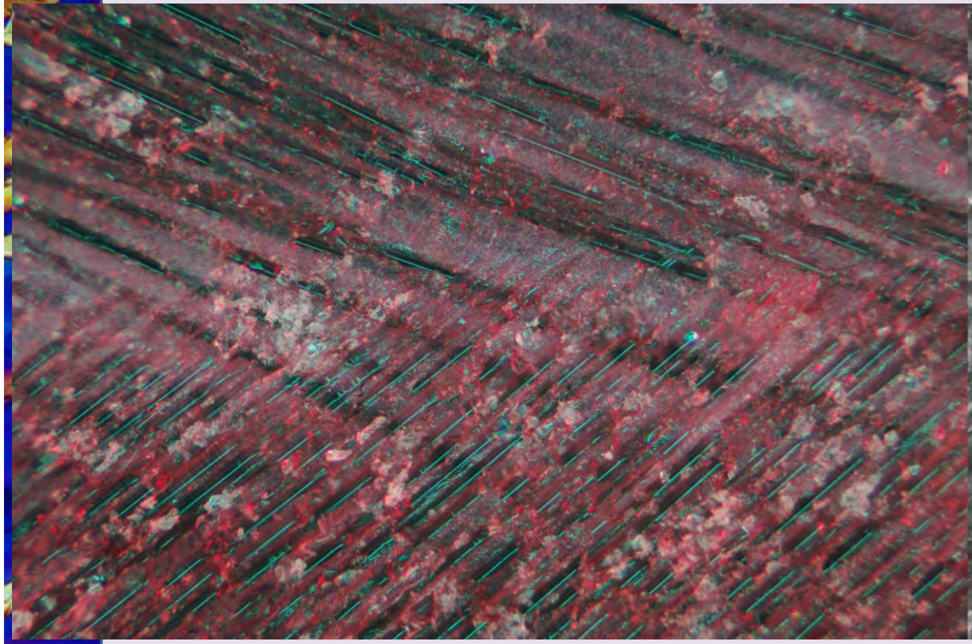


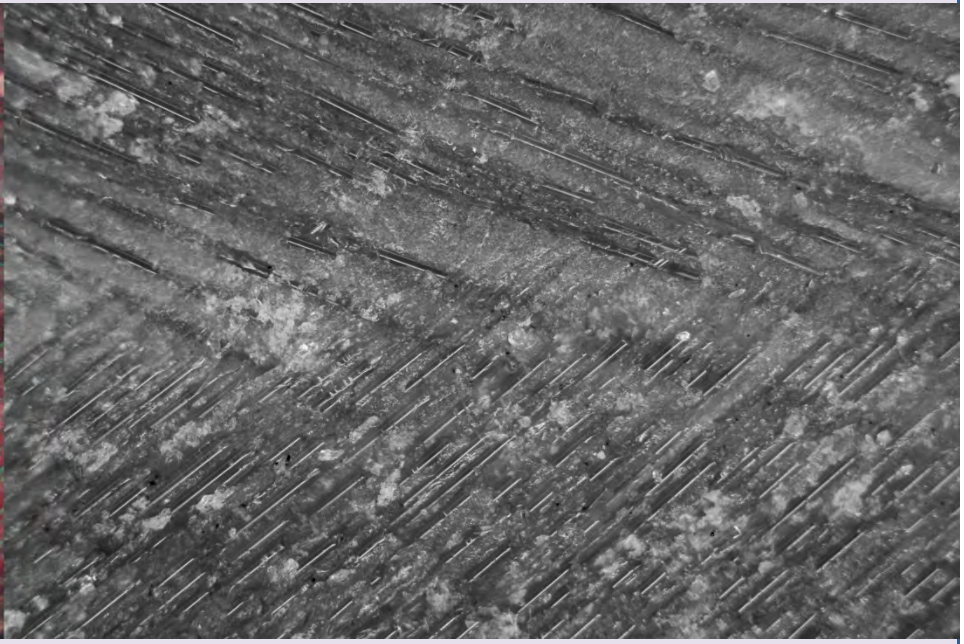
Commercial Sandwich Rheinberg, aka, "Bichromatic"



http://www.spectrosci.com/products/product/t2fm-q500/#features

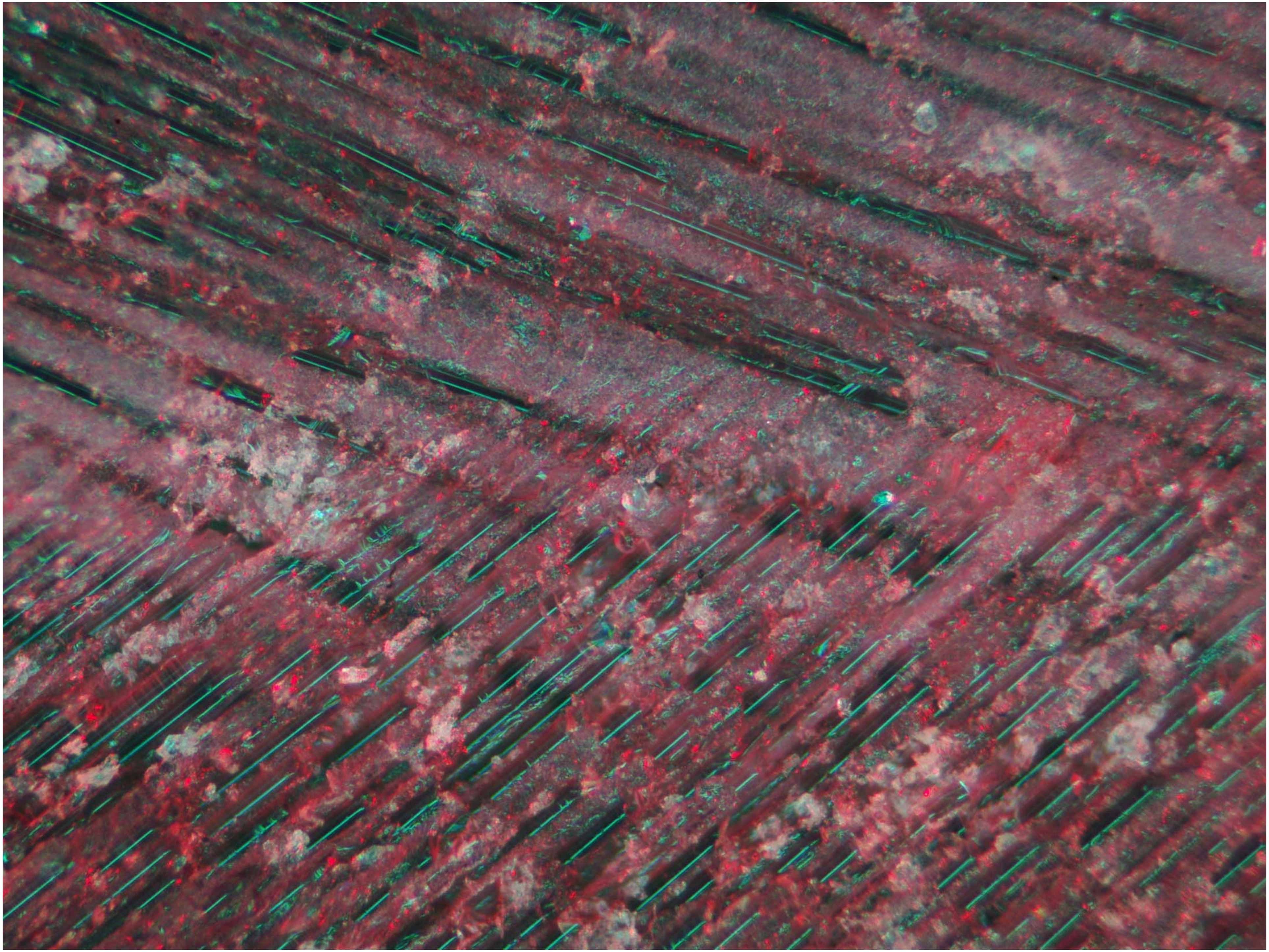
Continuous Spun FG Composite





Bl & Red

G of RGB Extracted



Light Sources

Benefits

- Visual Separation by Highlighting
- Digital Separation
- Reduces severe contrast by Darkfield
- Less visual fatigue

$\phi = t*\Delta\hat{n}$

t = thickness

$\hat{n}$ = refractive index

$\hat{n} = n + ik$

$$I \propto \varphi^2$$

Havics, Contrast Methods in Microscopy, Rheinberg Illumination, Microscope, 62, 4, 157-169, 2014

2.0 Grain orientation determination

~~A Cheap Man’s EBSD~~

~~A Poor Man’s EBSD~~

A Very Po Man’s EBSD

Transmitted Polarized Light

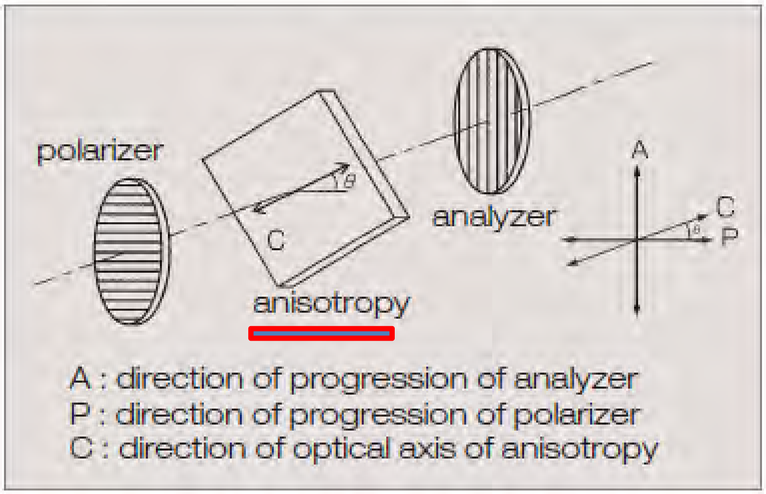


Fig. 2.1 Anisotropy between crossed nicols

$$I(\theta)=I_0 \sin^2 2\theta \cdot \sin^2(\delta/2)$$
$$=I_0 \sin^2 2\theta \cdot \sin^2\left(\frac{\pi R}{\lambda}\right) \qquad (2.1)$$

$$\delta=\delta\lambda/2\pi=d\,(ne\text{-}no) \qquad (1.2)$$

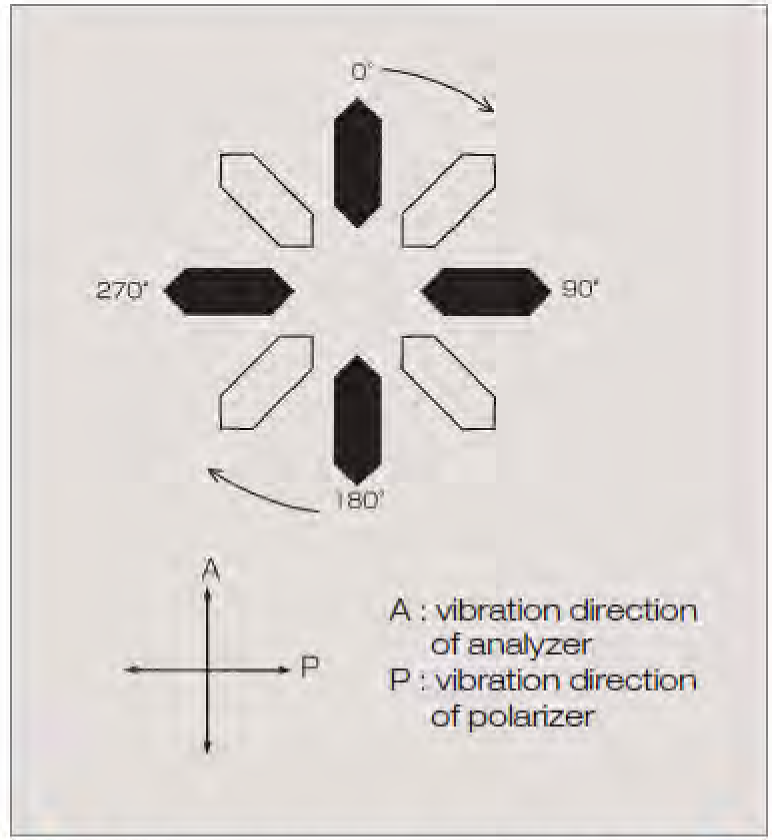


Fig. 2.2 Extinction position and diagonal position of anisotropy

Reflected PLM

$$R = \frac{(n-n')^2+k^2}{(n+n')^2+k^2}$$

R = Reflectance (fraction)
n = index of refraction of material
n' = index of refraction of medium (air)
k = absorption coefficient of material

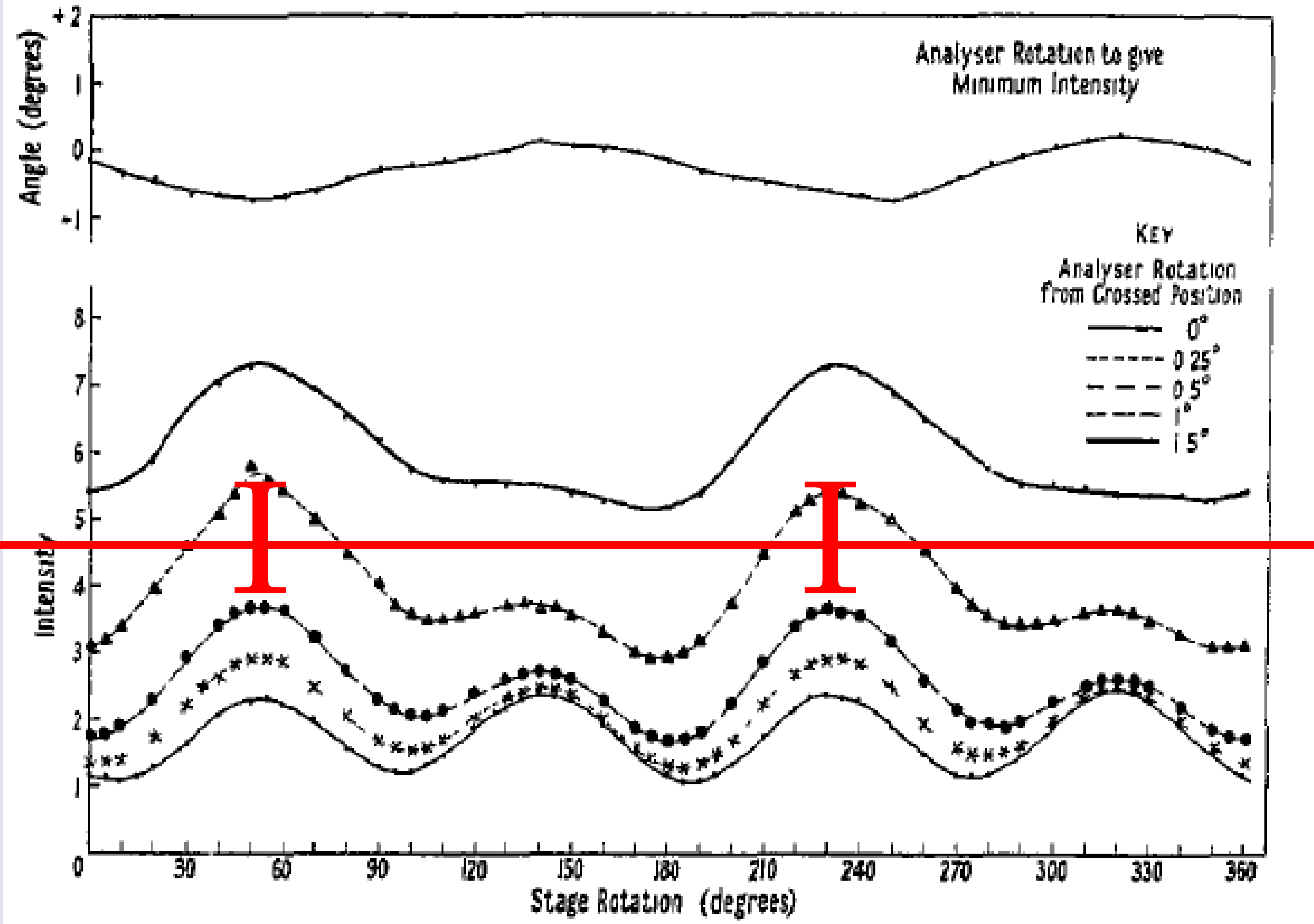


Fig 4 Uranium polished in standard electrolyte.

Woodrow, Analysis of Polarized Light Reflected from Absorbing
Materials at Normal Incidence, Proc Phys Soc, 65, 8, 603-616, 1952

Limitations: Angle of Incidence Effect

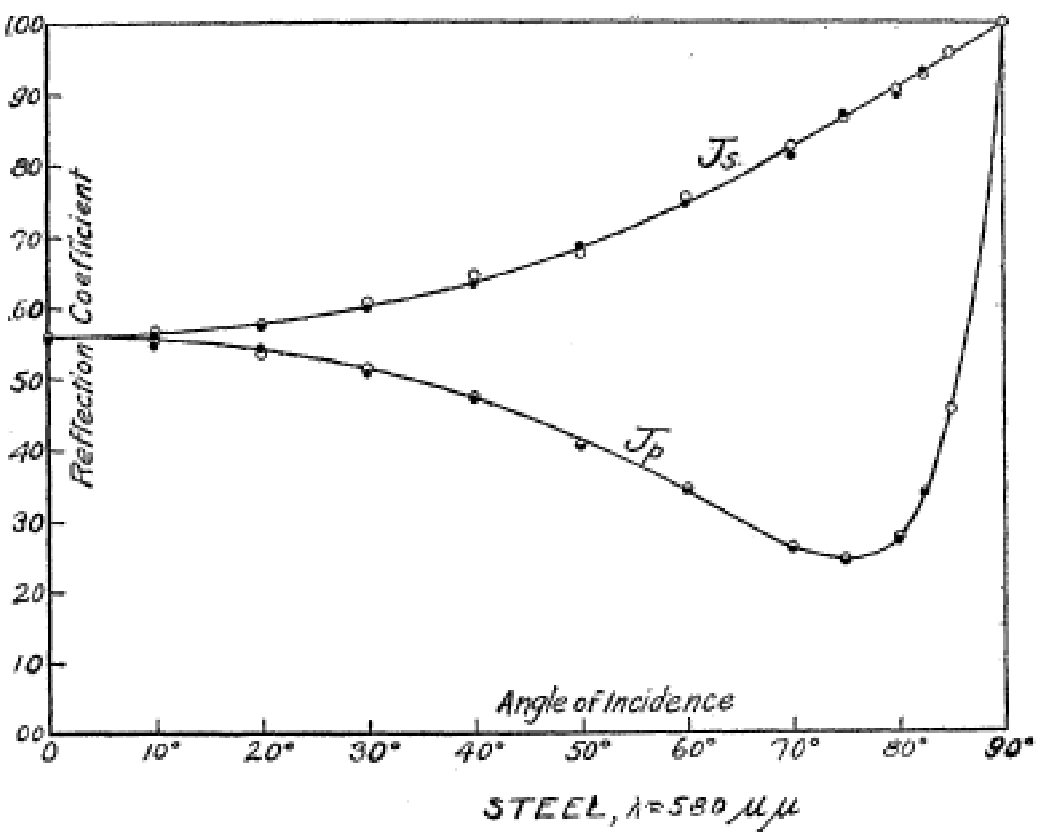


Fig. 3.

Wilsey, The Reflection Coefficients of Metals for the Polarized Components of Light, Phys Rev, 8, 4, 391-401, 1916

Limitations: Wavelength Response

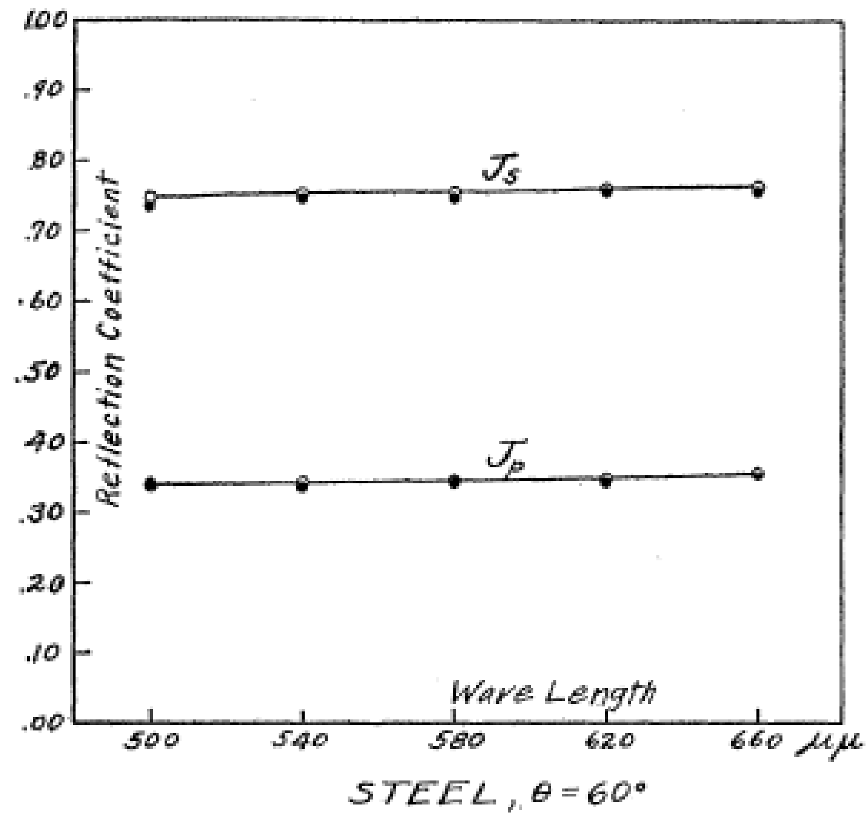
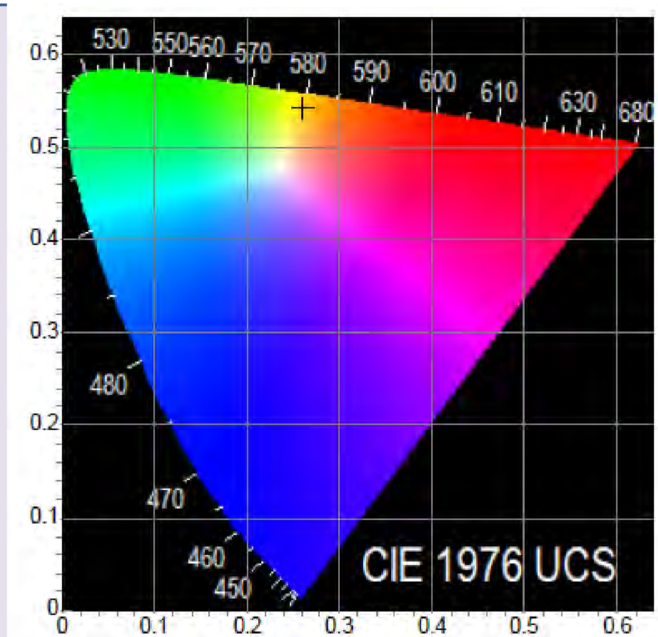
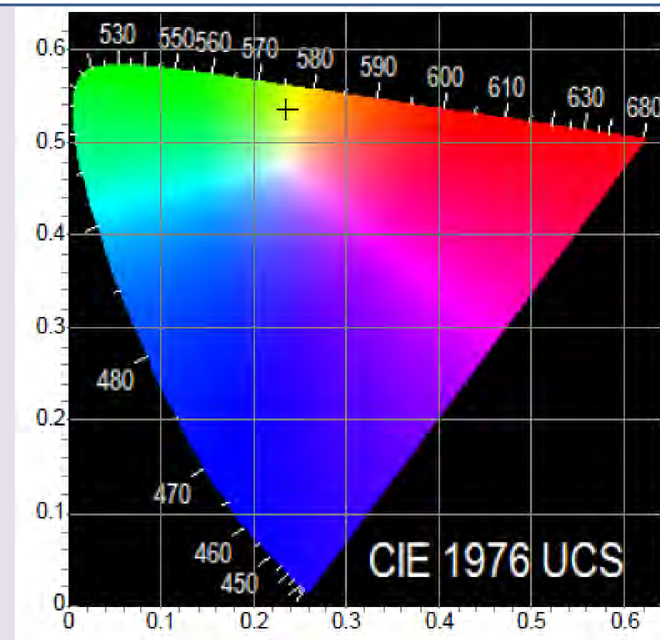
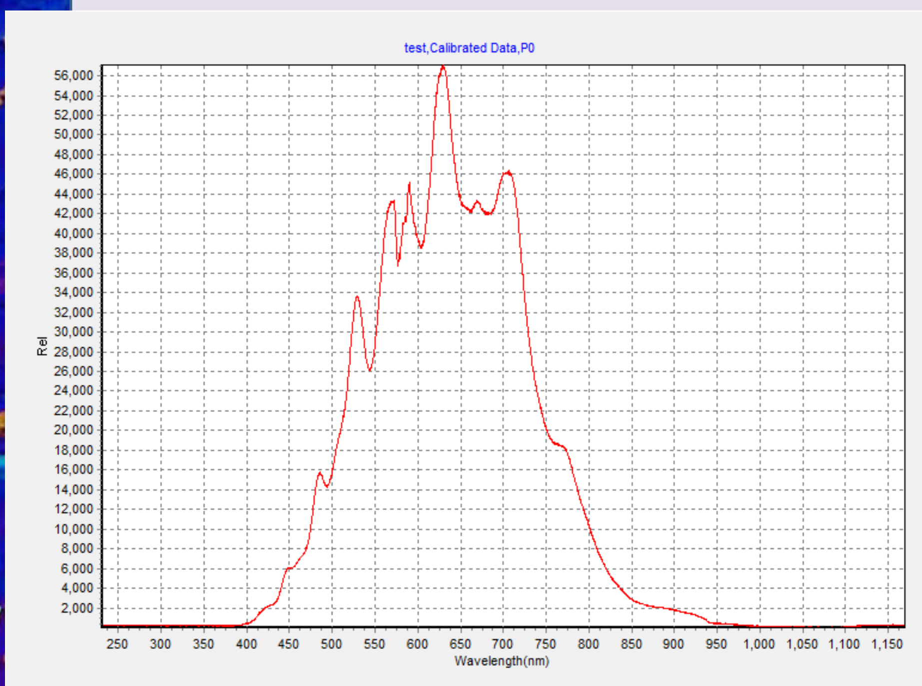


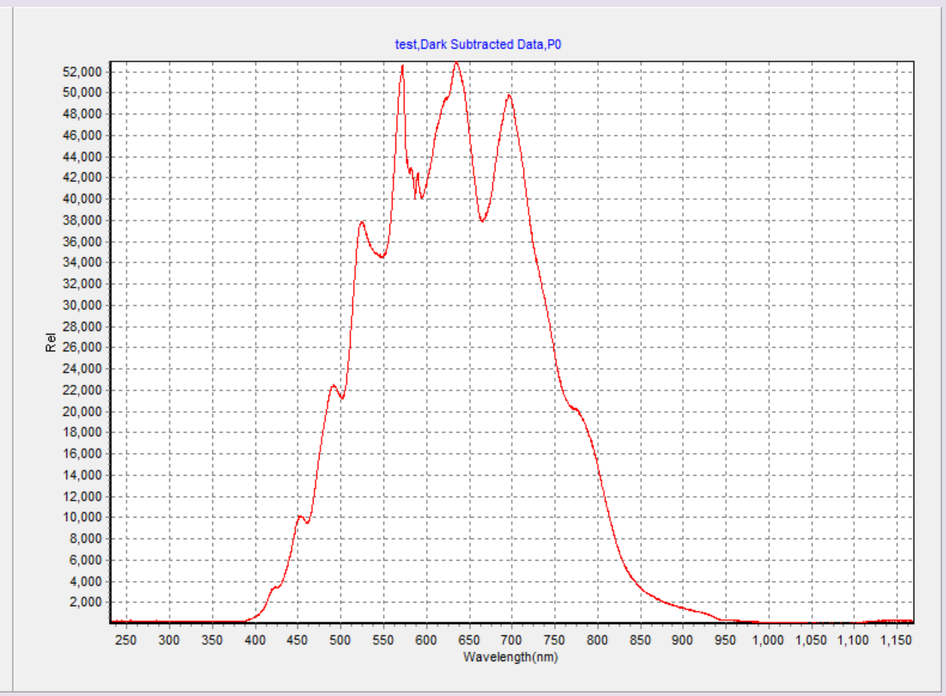
Fig. 4.

Wilsey, The Reflection Coefficients of Metals for the Polarized Components of Light, Phys Rev, 8, 4, 391-401, 1916





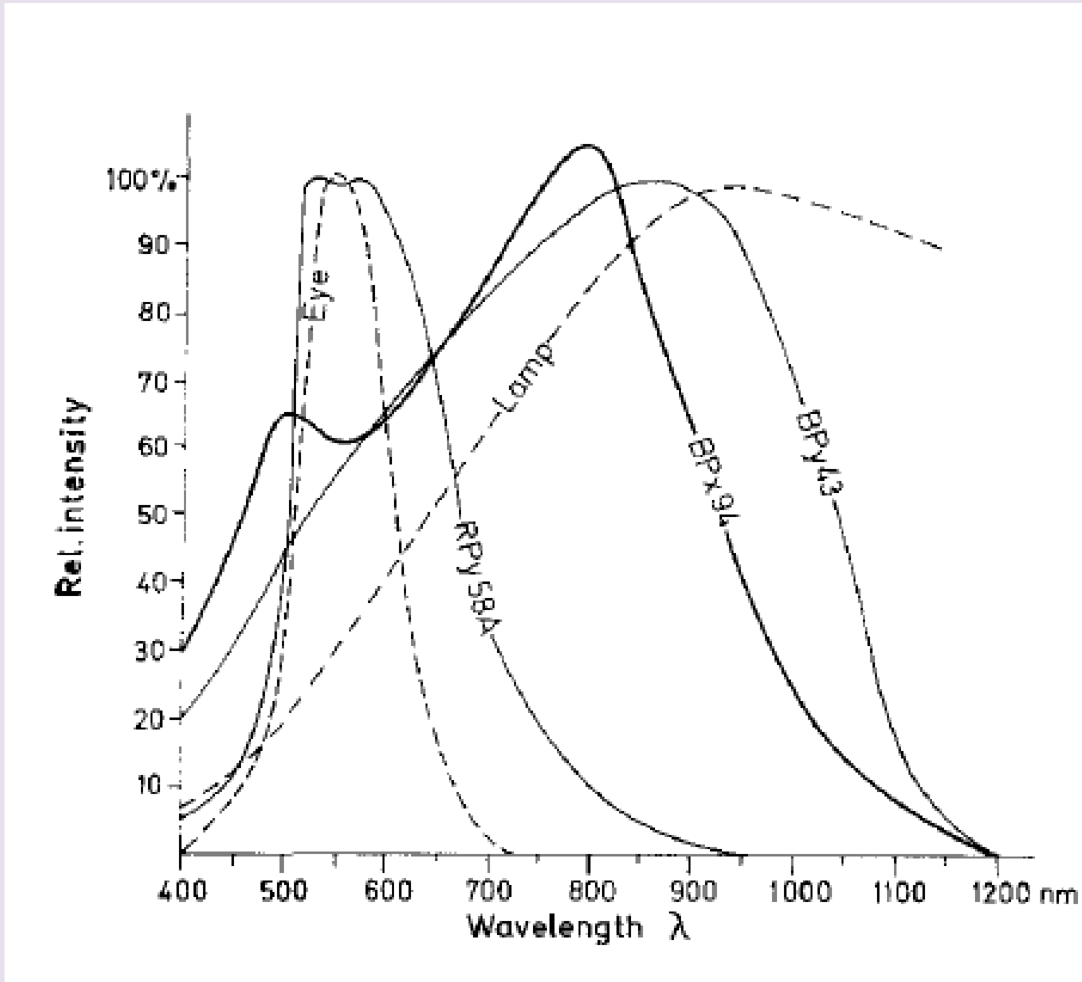




Brass

6061 Aluminum

Limitations: Light Source



Tarkian, A new miniphotometer for teaching and routine work in ore microscopy, Min Mag, 40, 97-103, 1975

Limitations: Difference in Polishing

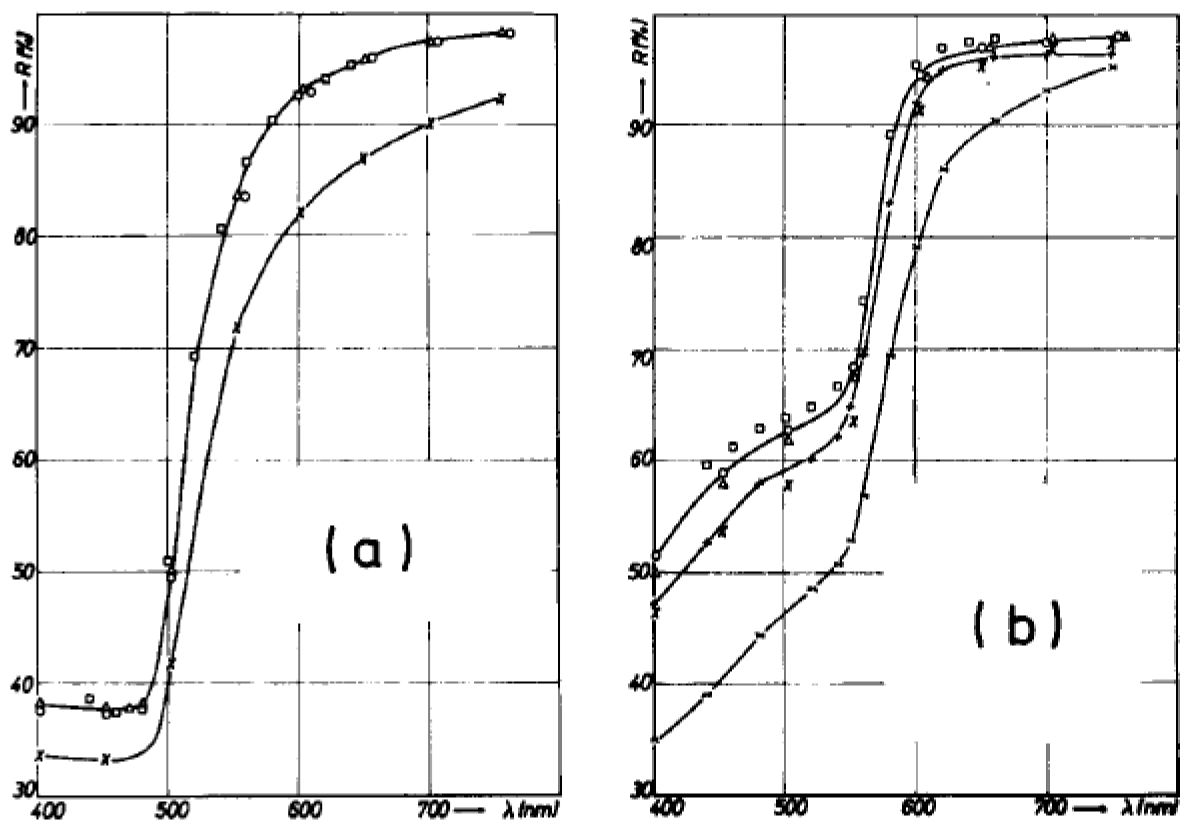
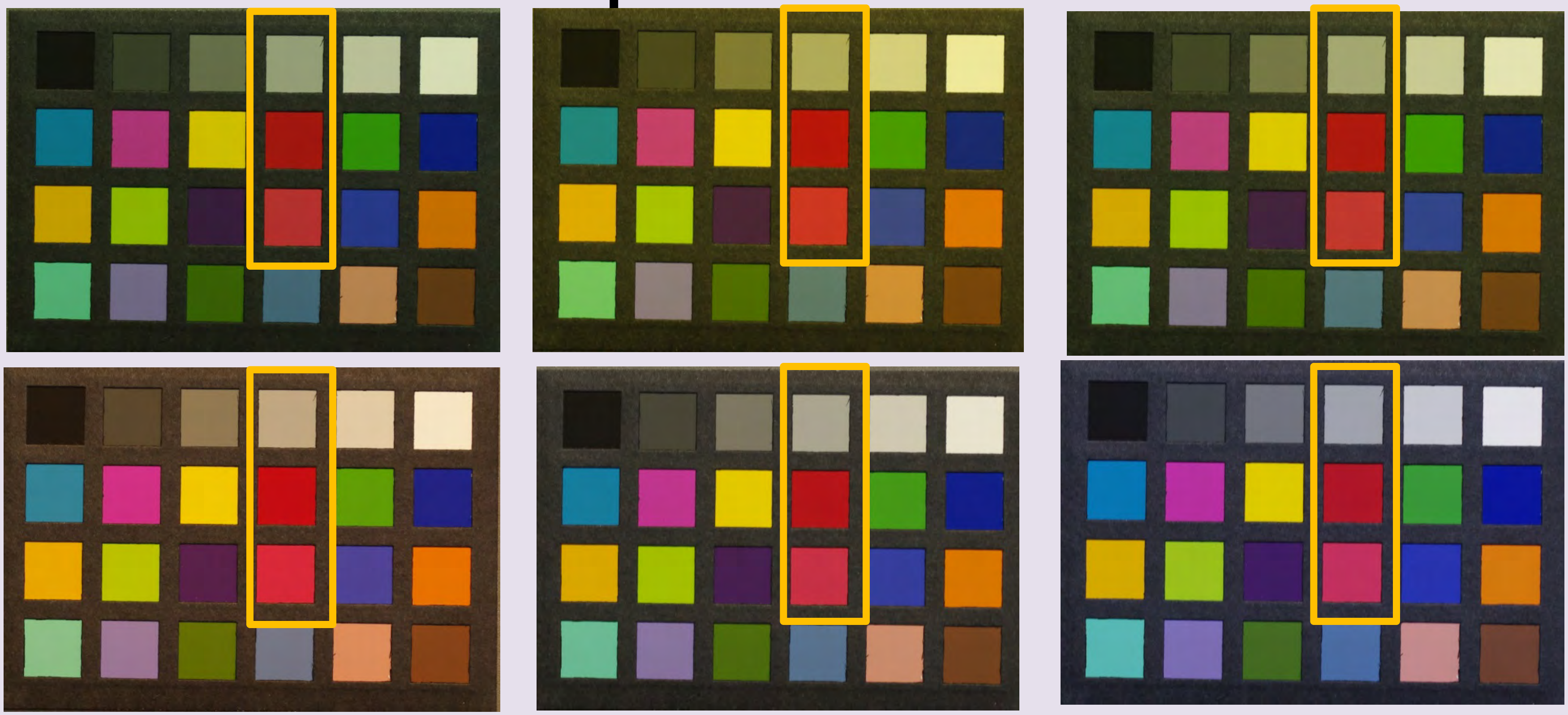


FIG. 2. Reflection spectra of (*a*) gold and (*b*) copper with surfaces polished in different ways. (a) ○ electrolytically polished, △ cut with the microtome, X alumina polished, □ from Otter [6]. (b) ○ electrolytically polished, △ cut with the microtome, + diamond polished, X electromechanically polished, × alumina polished, □ from Otter [6].

Petzow, Application of microreflection in metallography, Metallography, 6, 3, 249-260, 1973

Limitations: Equivalent Photometer



Answer

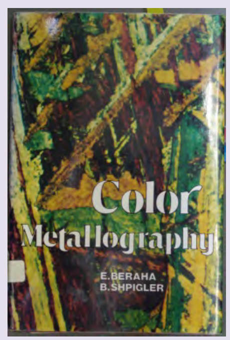
- Same Scope
- Same light source at same voltage
- Same Objective between samples
- Same Preparation Processes
- Ref material (ex. Pyrite)
- Same Camera & Settings
  - F-Stop, Shutter speed

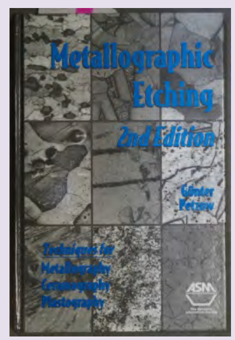
Ways to Differentiate Direction Qualitatively

- Polarized Light Crossed Polarized Light vs Parallel Polarized Light

Anisotropic or Etched Isotropic

- Color Ratios [R:B]
- Texture Etching
- Color Etching
  - material oxide
  - oxide or chemical deposition





Isotropic

Not New: Aluminum

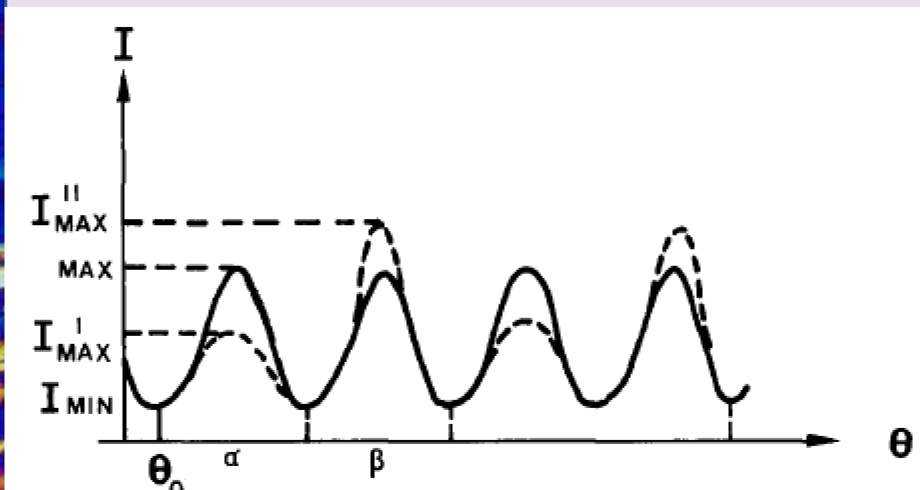


FIG. 2. The variation (schematic) of the intensity I with the angle of rotation θ of th grain around its normal. Solid line = polarizer and analyzer exactly crossed; broken lin = polarizer and analyzer not exactly crossed.

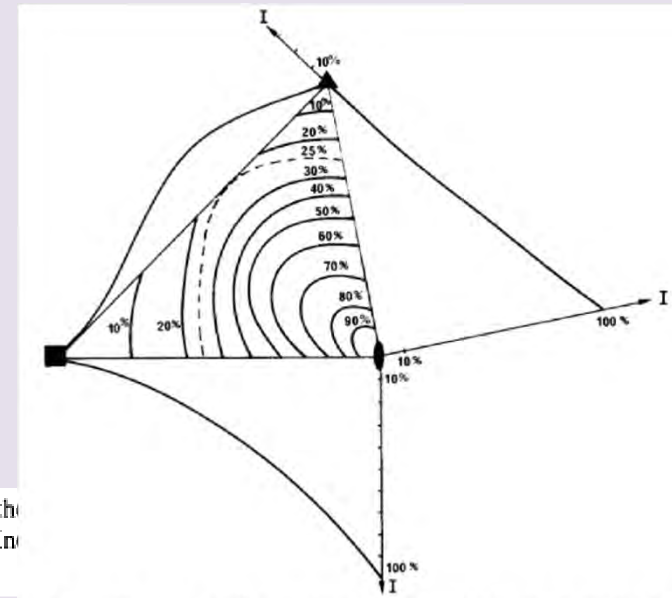


FIG. 5. The variation of the intensity I_{max} with the crystallographic orientation of the grain normal.

Saetre, Variation in polarized light intensity with grain-orientation in anodized aluminum, Metallography, 19, 3, 345-357, 1986

Aluminum

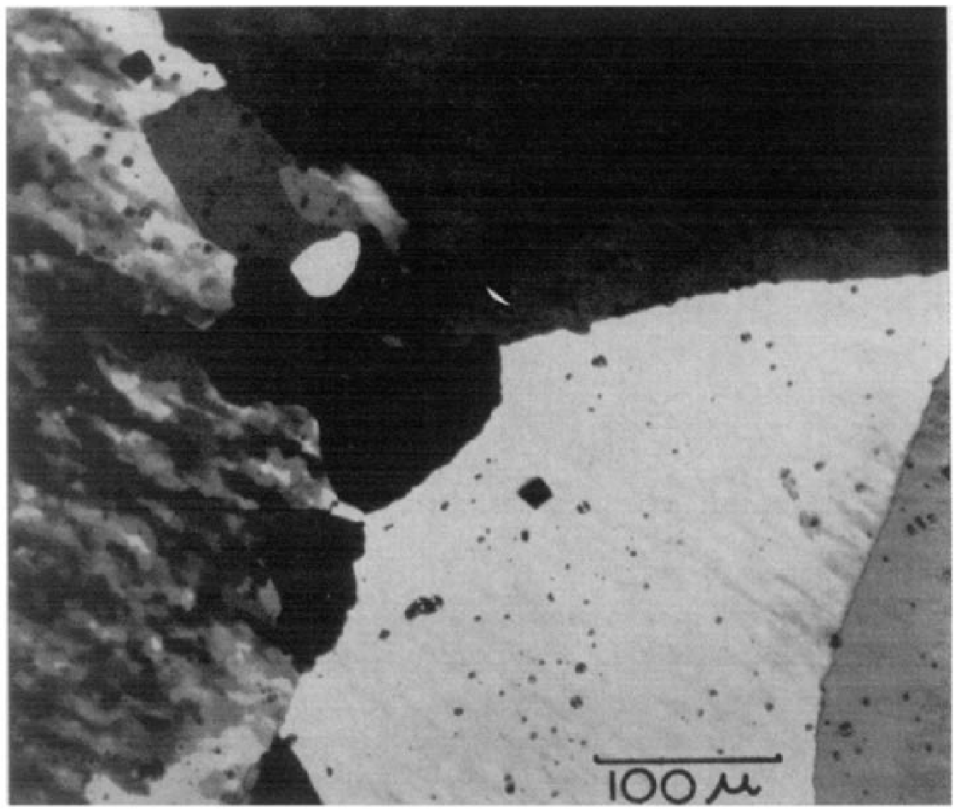


FIG. 2. Specimen electropolished and anodized, viewed with polarized light, showing two microhardness indentation marks.

Ferran, Metallographic preparation and Kossel line studies of aluminum, Metallography, 3, 4, 441-450, 1970

Zinc

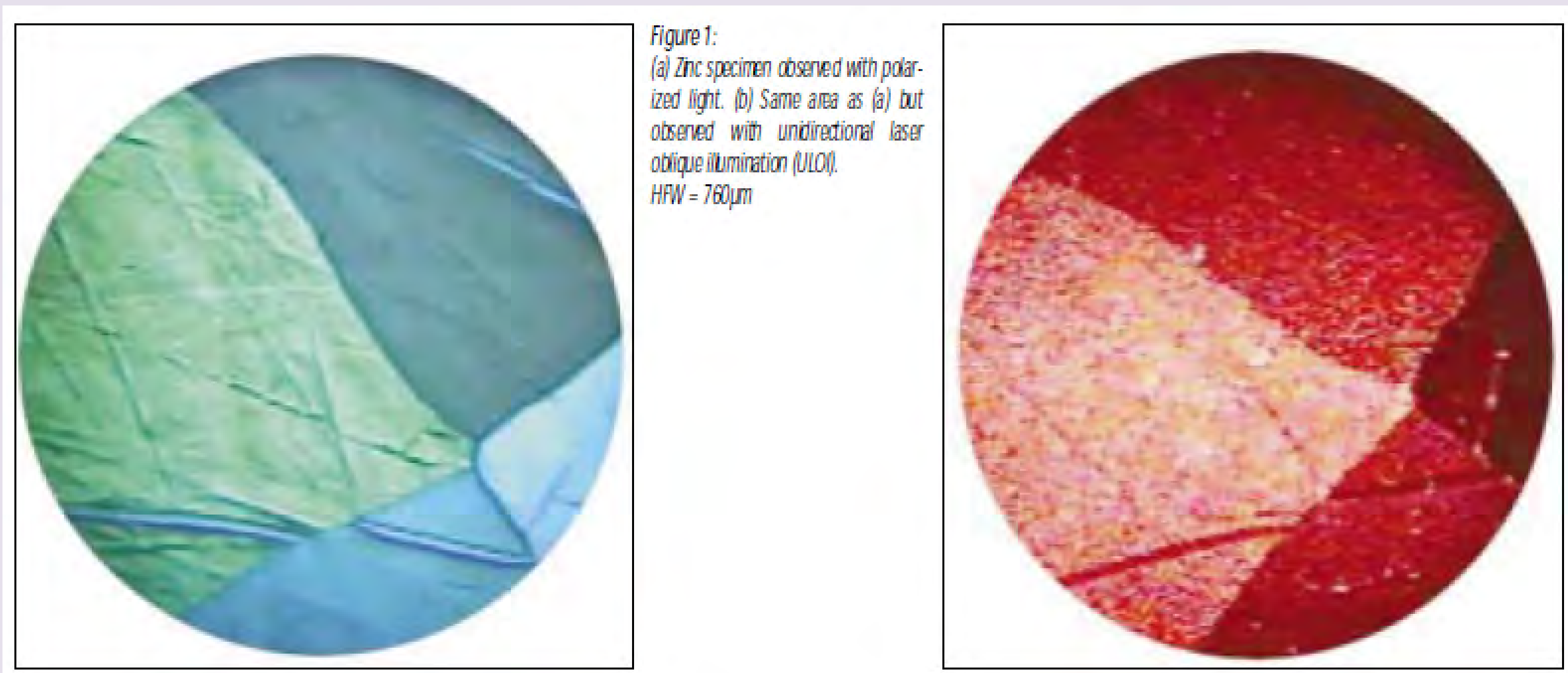
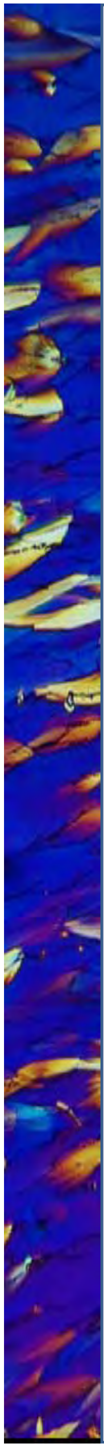


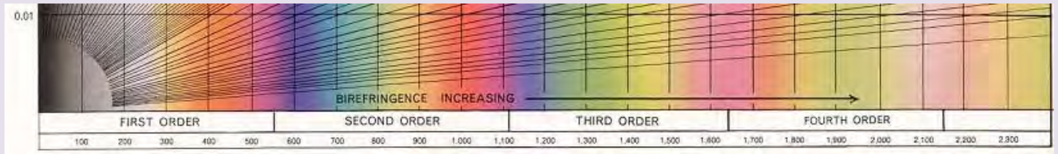
Figure 1:
(a) Zinc specimen observed with polarized light. (b) Same area as (a) but observed with unidirectional laser oblique illumination (ULOI).
HFW = 760μm

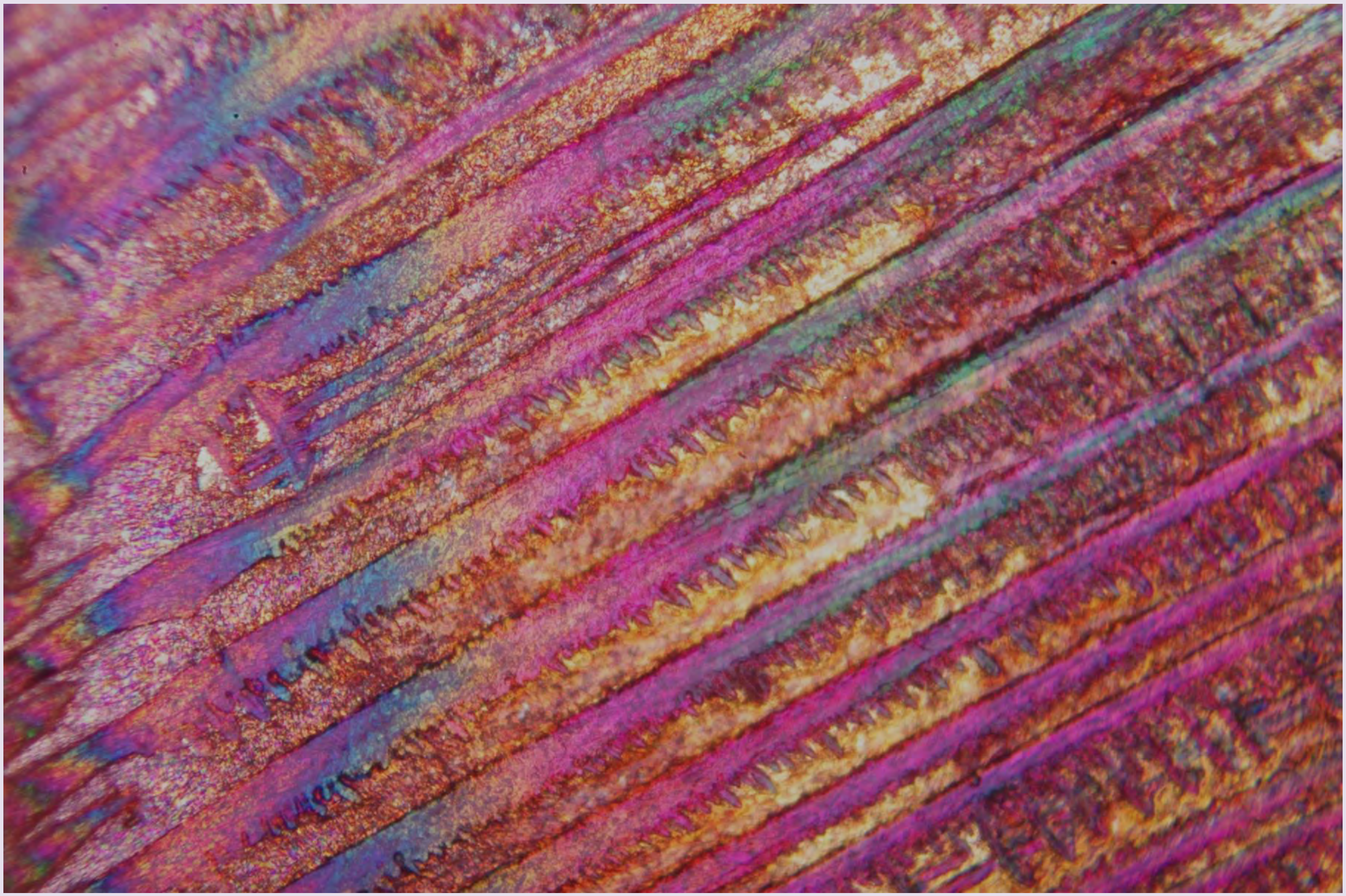
Favret, Comparison of ULOI and Polarized Light Microscopy of Materials , UK Micro Microanal, 20, 5, 27-29, July 2001

New Aspects:

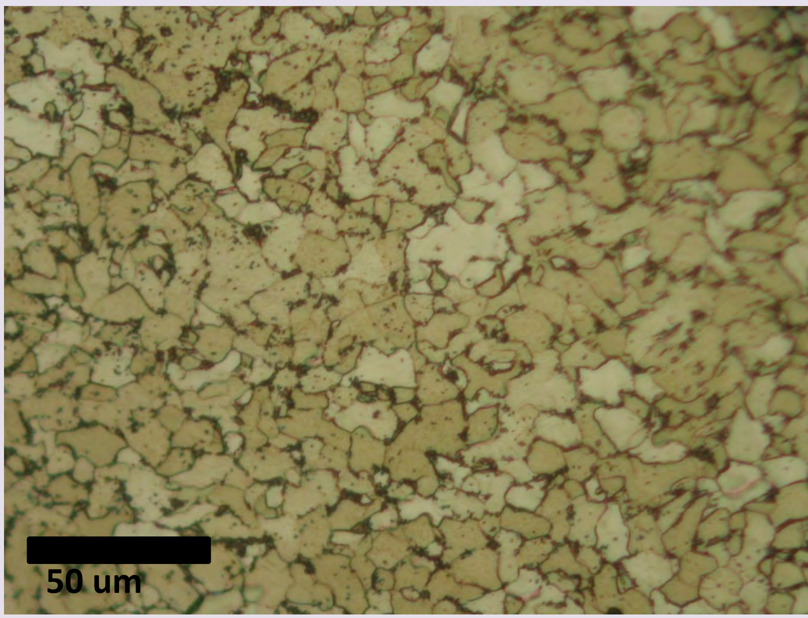
- Digital Separation
- Digital Calculations, e.g., MATLAB
- Digital (programmed) Orientation Representation
- Microspectrophotometry

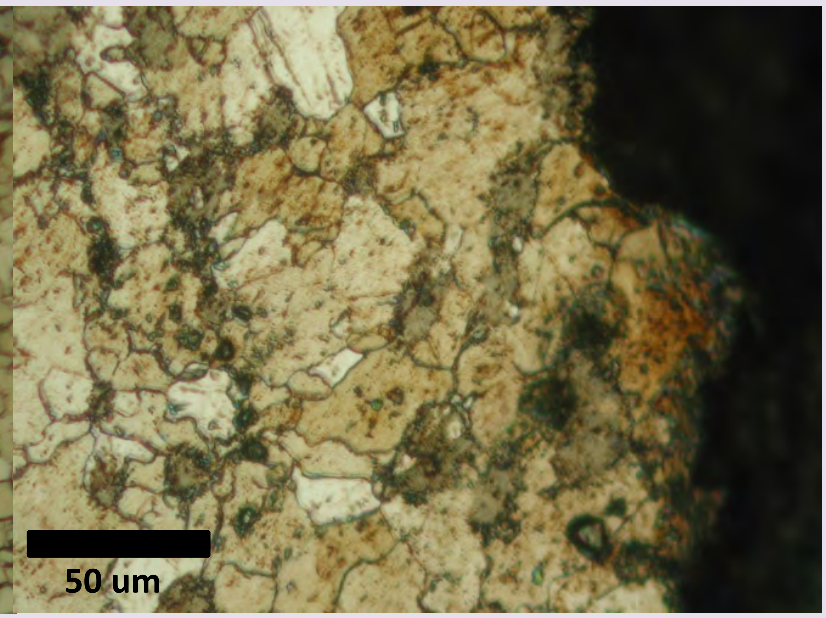



0.01
BIREFRINGENCE INCREASING
FIRST ORDER
SECOND ORDER
THIRD ORDER
FOURTH ORDER



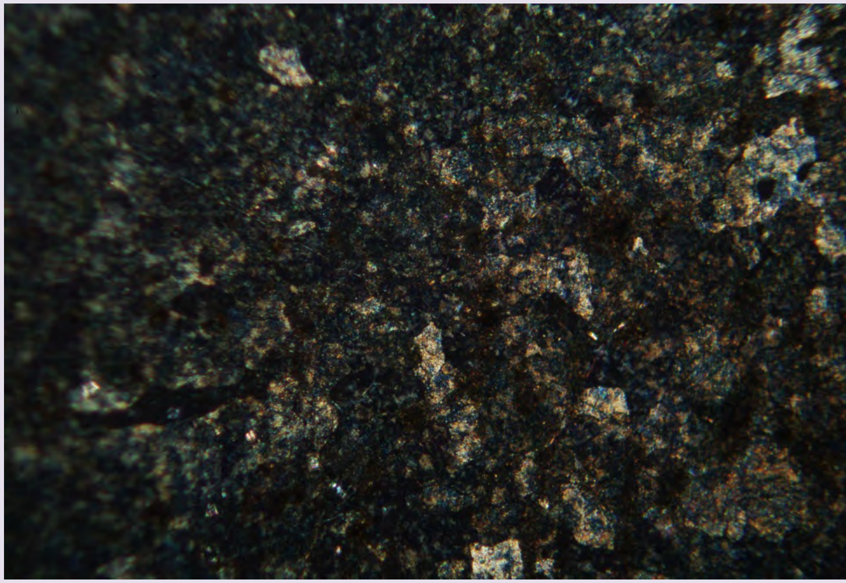
Adjacent to and at Weld



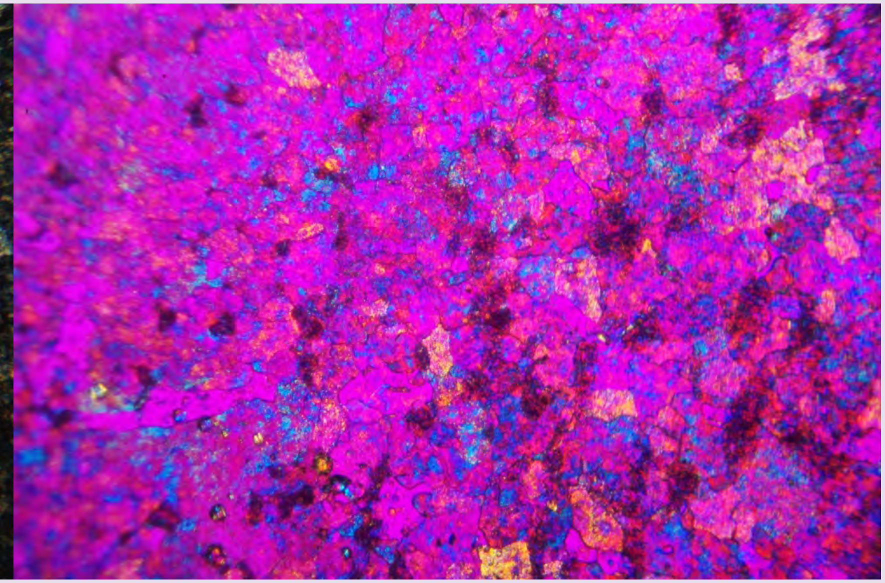


$$R = \frac{(n - n')^2 + k^2}{(n + n')^2 + k^2}$$

at Weld



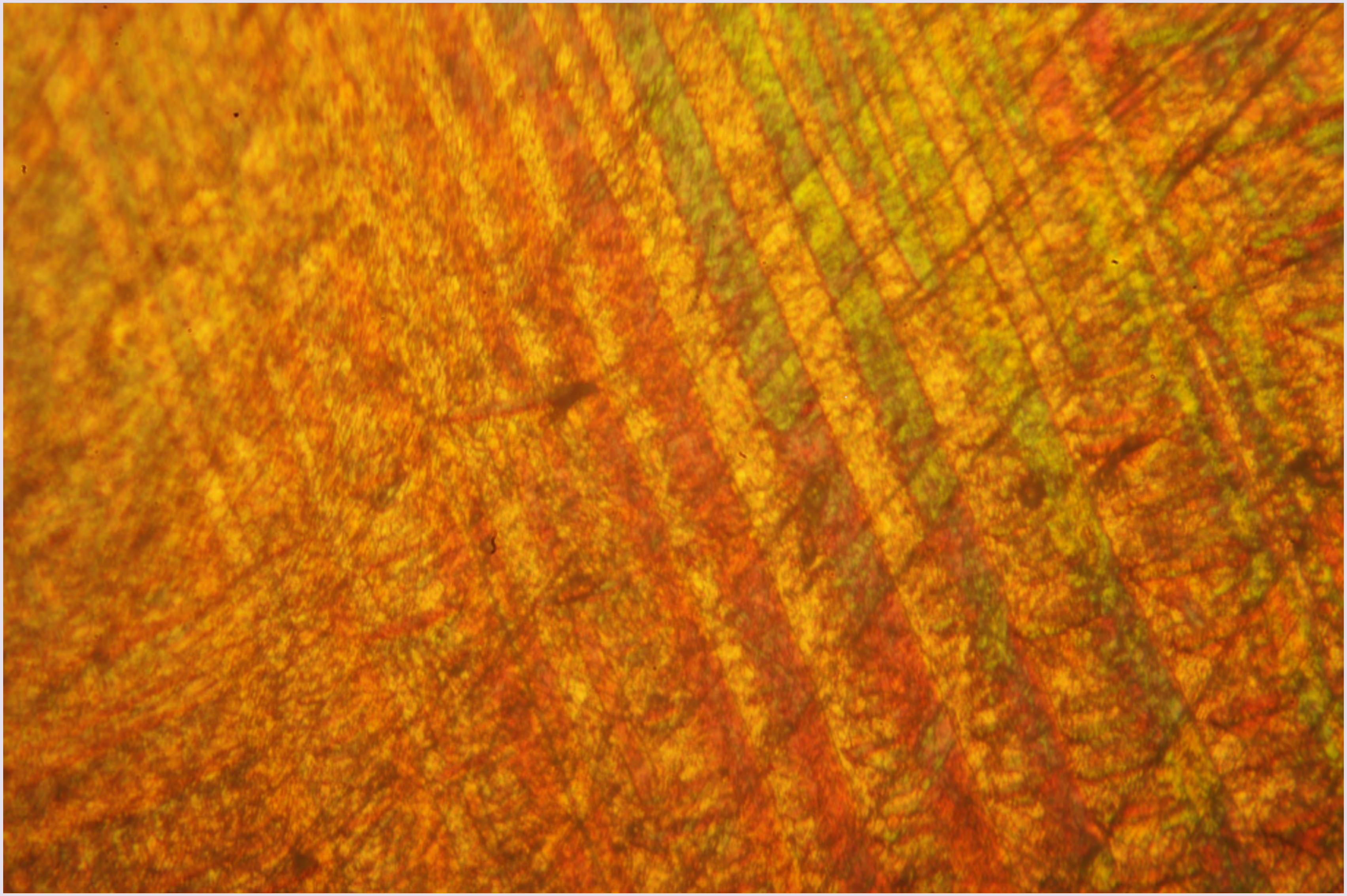
XPL



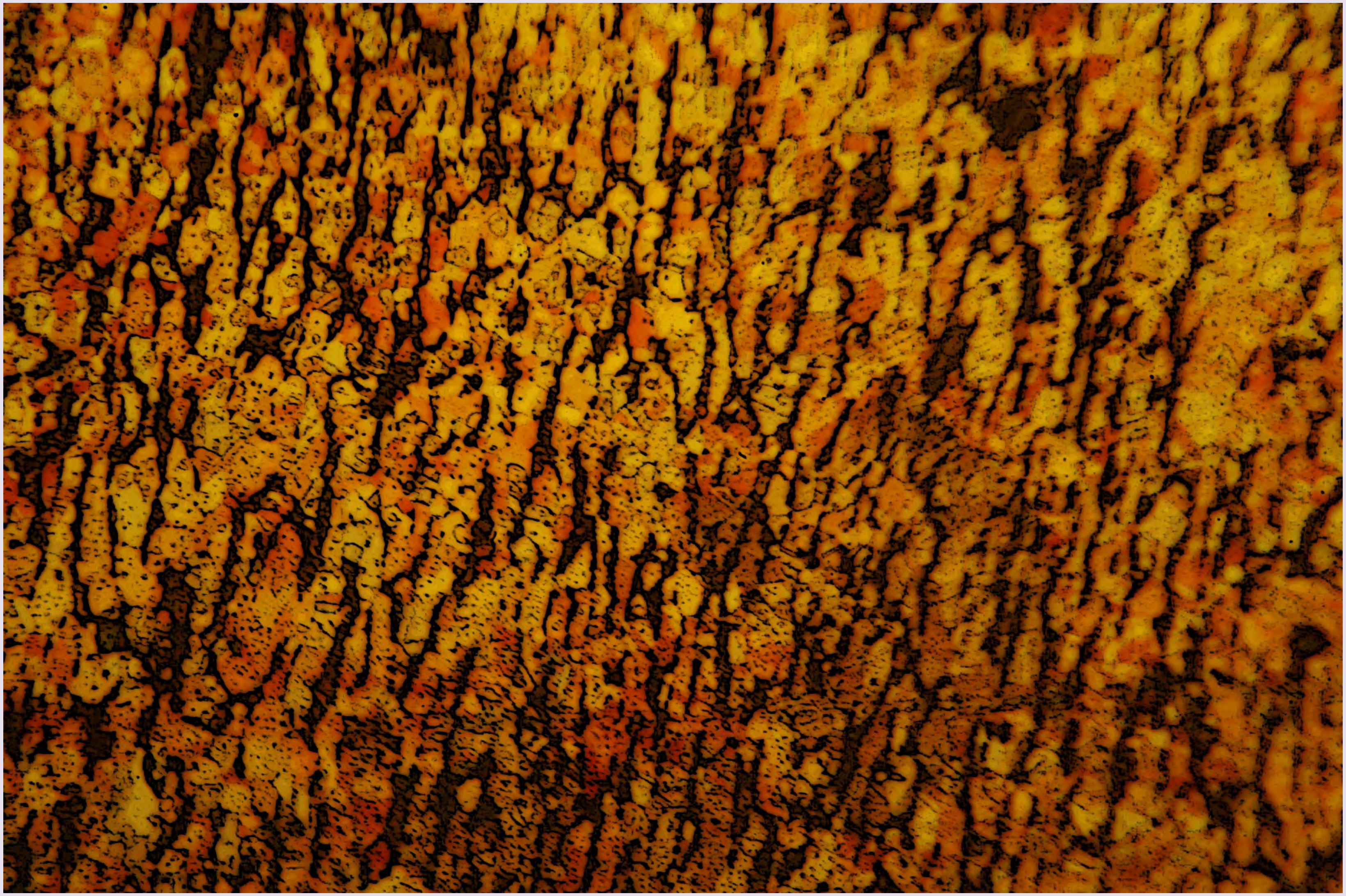
XPL + Red I

Note: Recommend circular polarized light

Copper

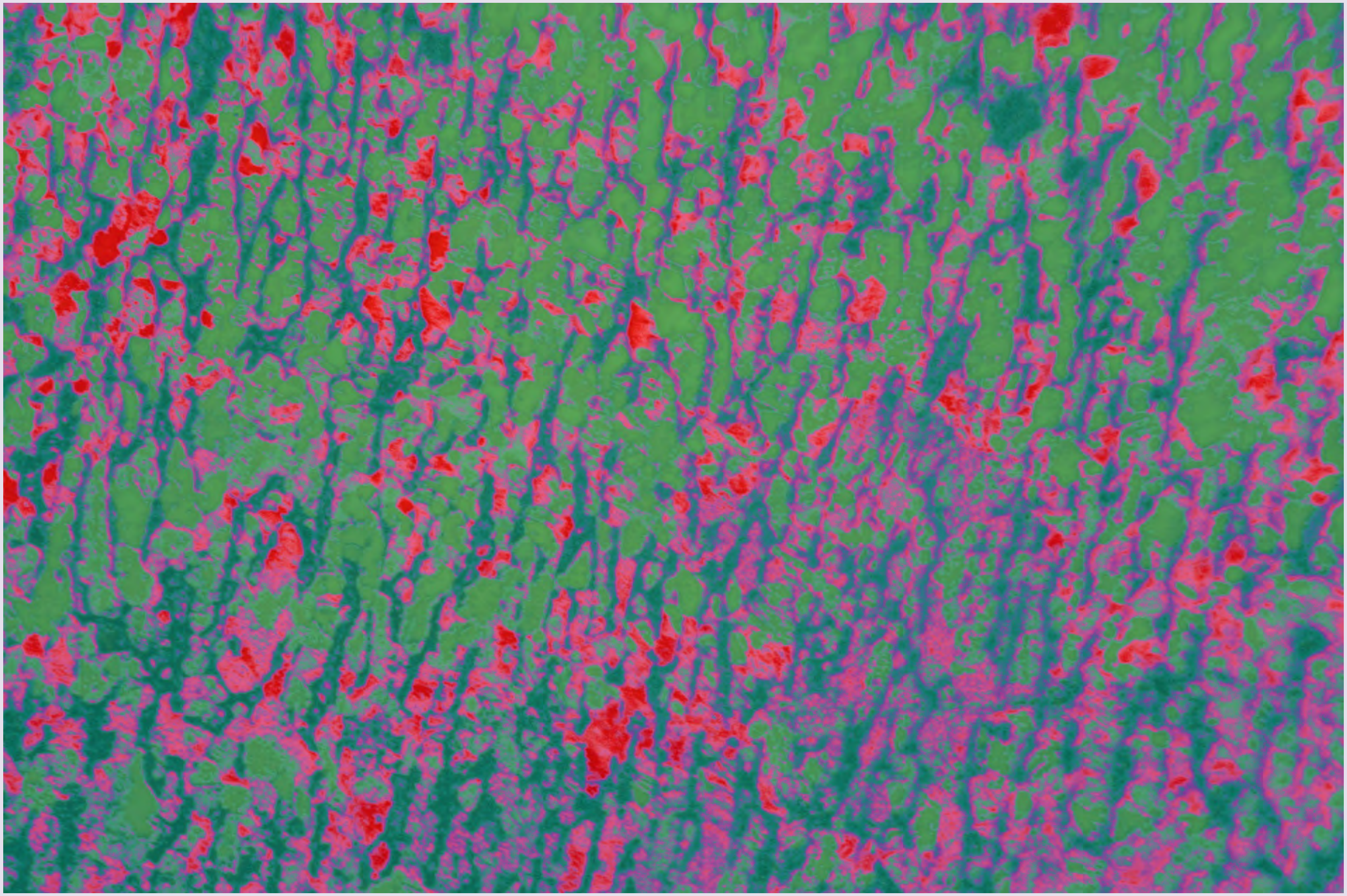


Etch: Klemm



Etch: Klemm

Orientation

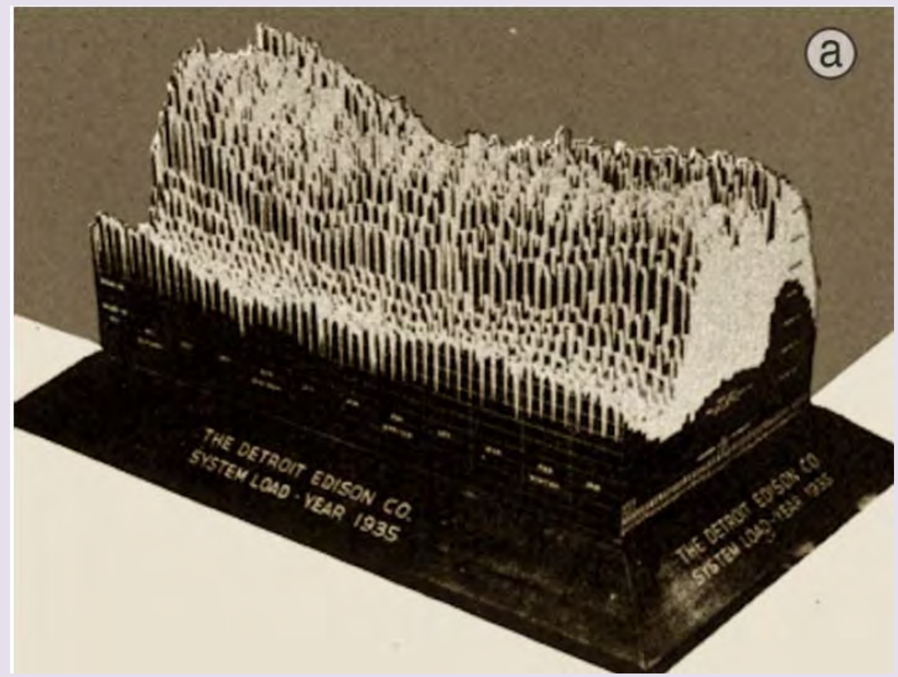


3.0 3D depth-of-field

- What is the value of a 3D Object?

Some Benefits, but not always

- Jansen, et al.: Evaluating the efficiency of physical visualizations, Proc 2013 Ann Conf Human Factors in Comp Sys, 2593-1602, 2013
- Alper et al.: Stereoscopic highlighting, 2d graph visualization on stereo displays, IEEE Trans Vis Comp Graphics, 17, 12, 2325-2333, 2011.
- Ramachandran, et al.: Mayavi: a package for 3D visualization of scientific data



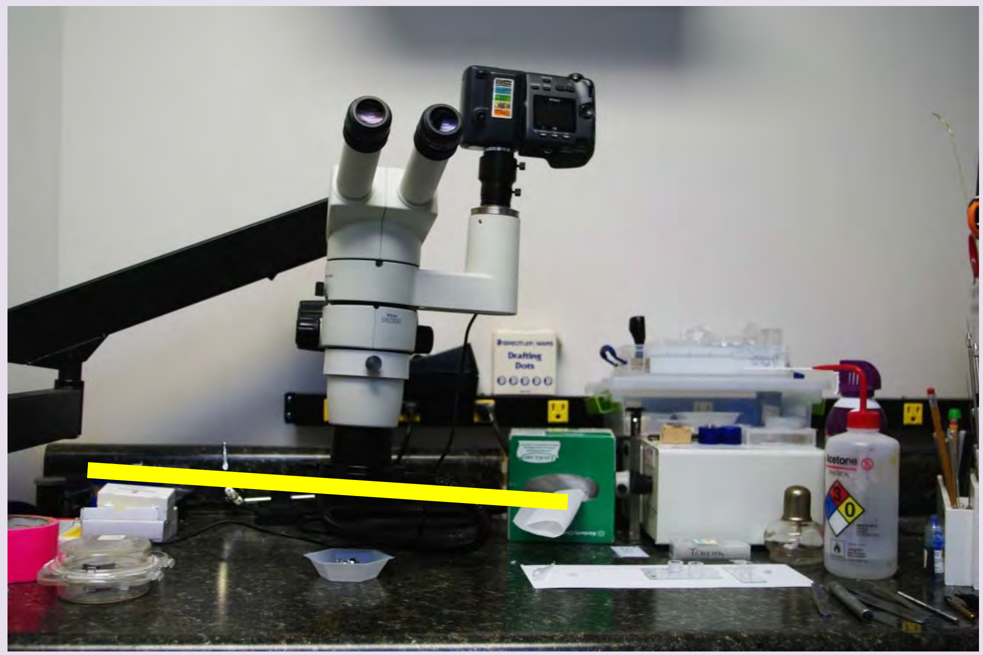
Power Usage, by GE, 1935

Do they see what I see?

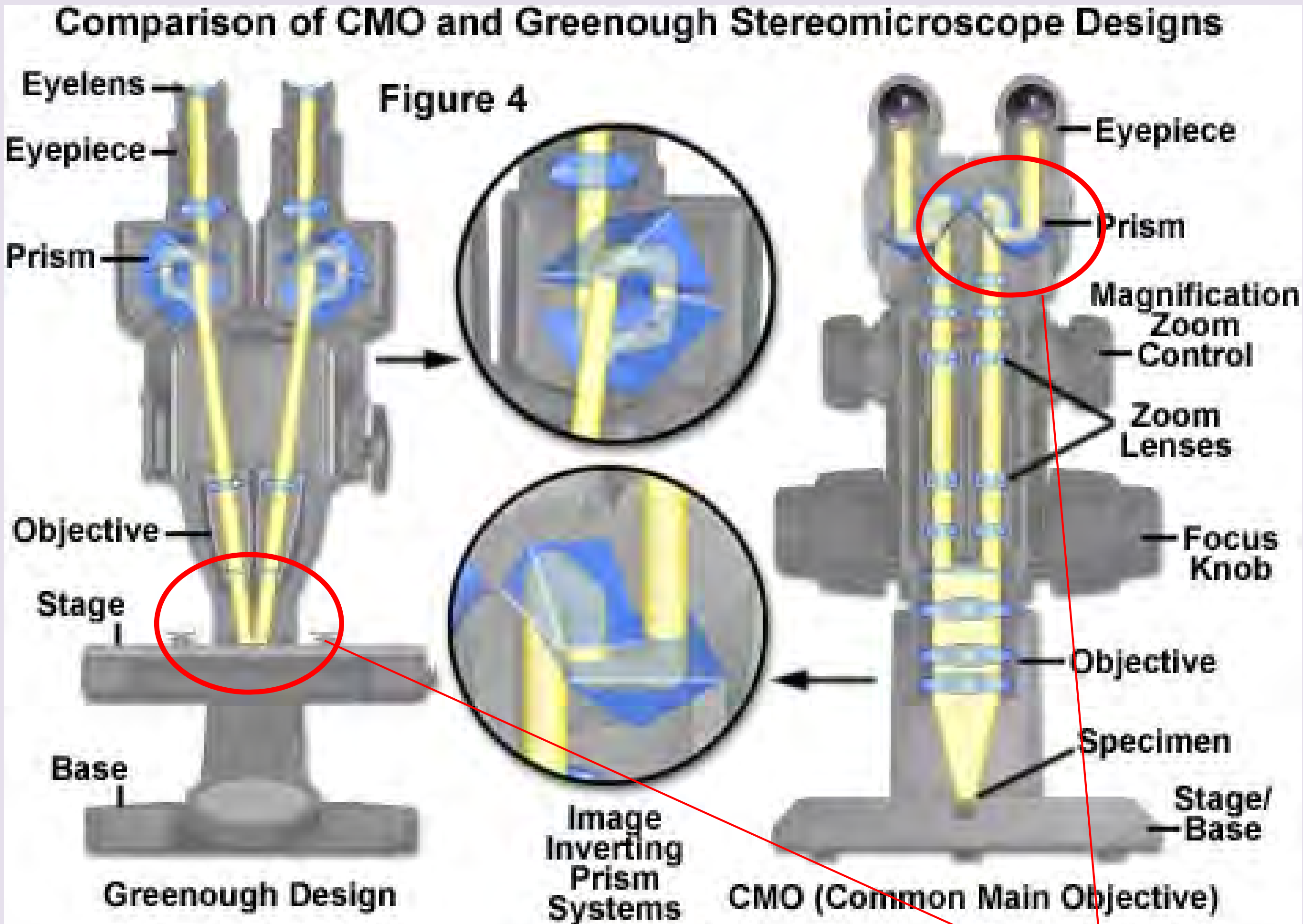
Discussion here: Micro vs. Macro

- Photomacrography reproduction ratios >1:1
- Photomicrography >>>1:1

Stereo & Dissecting



Optical Arrangement

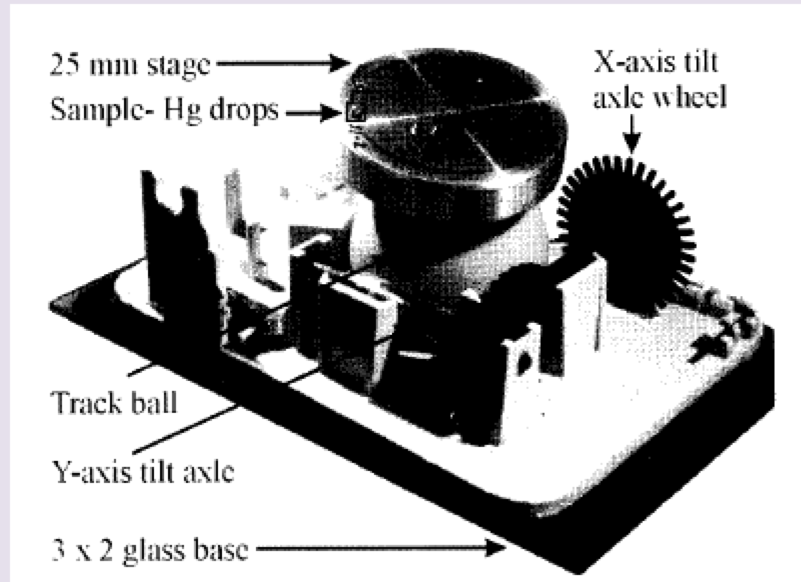


https://microscopyu.com/articles/stereomicroscopy/stereointro.html

12-15 degree angle

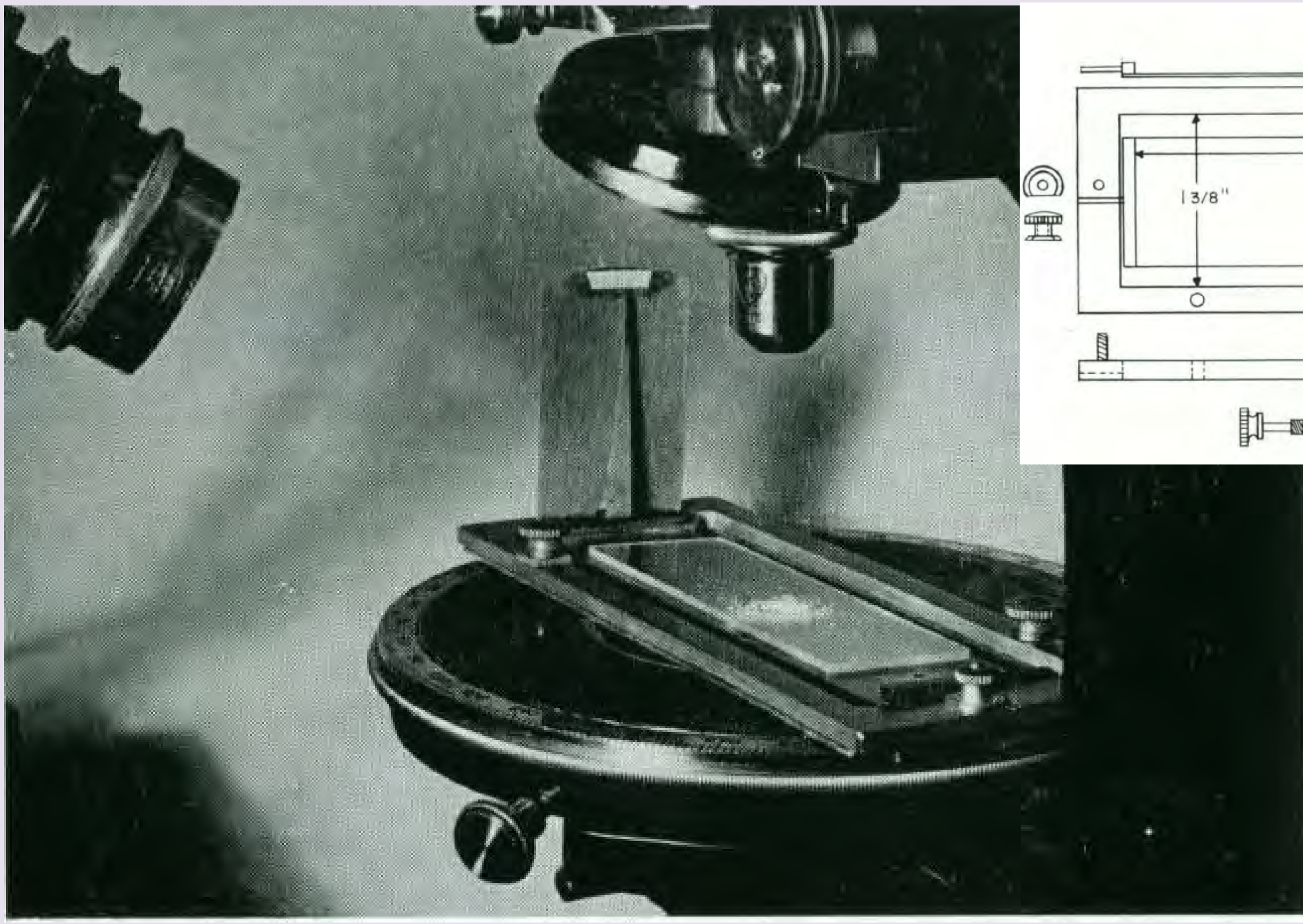
“3D” Representations

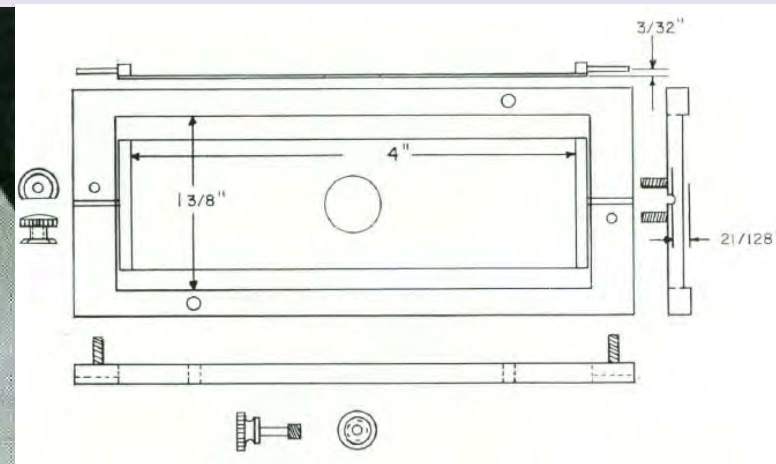
- Stereo Scope
- Stereo Pair Prints
- Anaglyphs [ˈanəˌglifs]
  - Red-Blue or Red-Green
- 3D Stacking



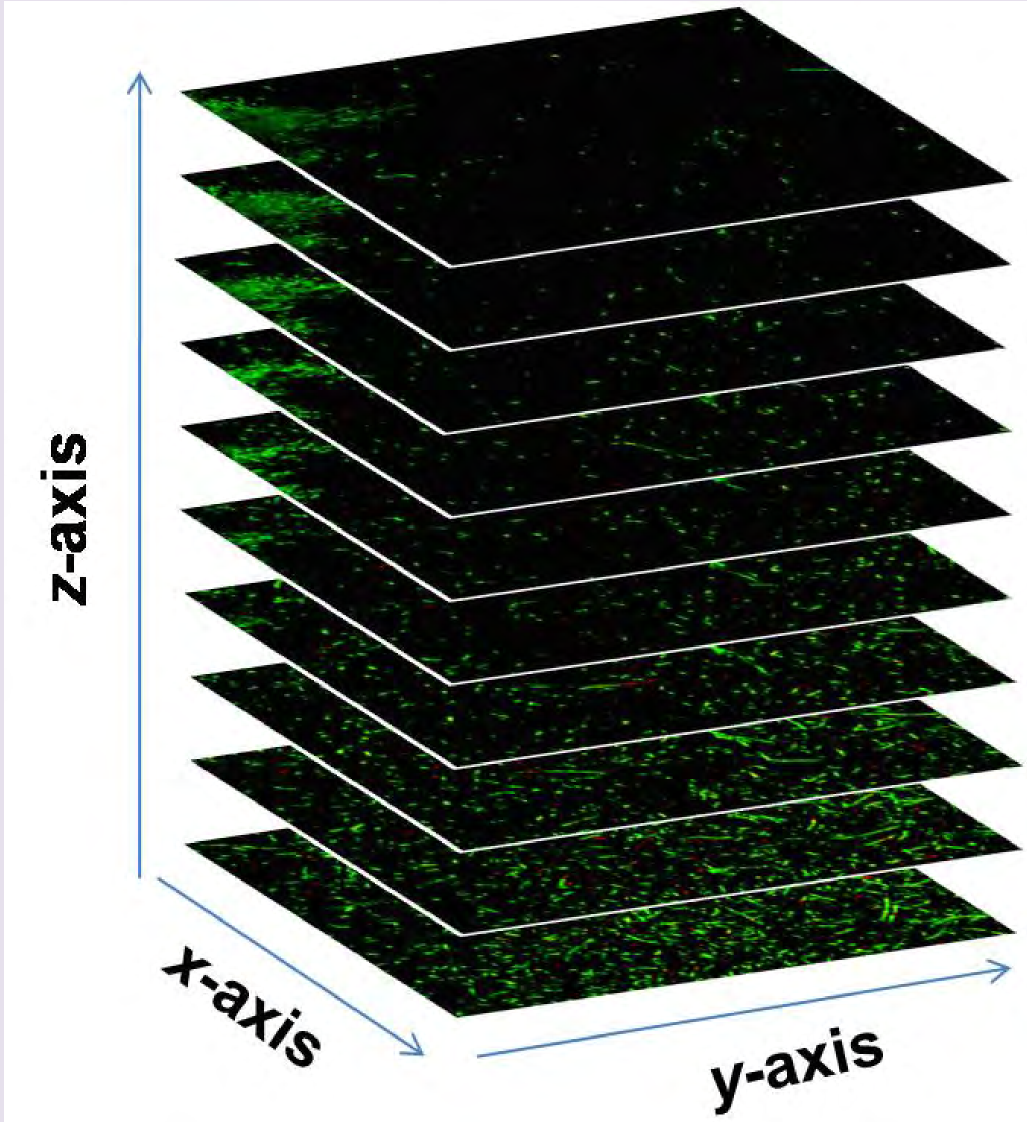
Weaver, Tricks of the Trade, The Universal Tilting Mouse Stage, Microscope, 51, 4, 221-224, 2003

Tilting Stage



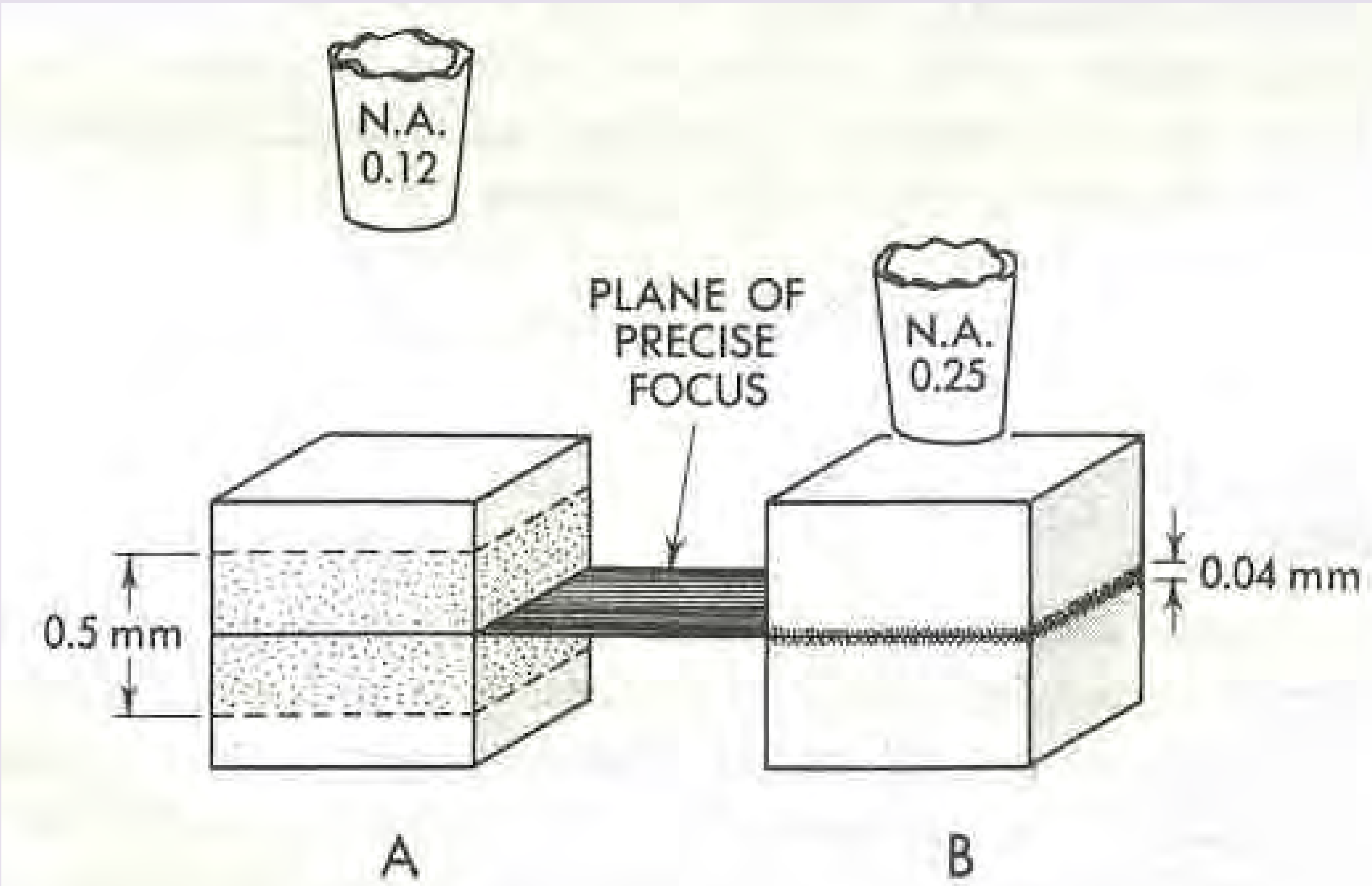


McCrone, Walter: Stereophotomicrography Using the Tilting Stage. Microscope, 14, 11, 429-440, 1965



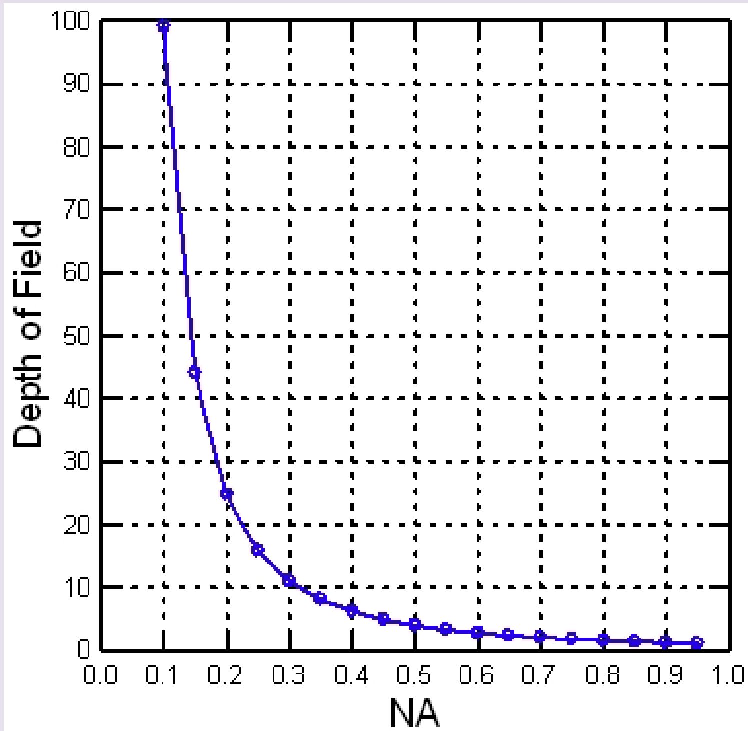
http://leep.clarku.edu/projects/2013/06/10/what-cheese-slices-and-fluorescent-microscopes-have-in-common/

Depth of Field (DOF)



Bloss, F. Donald: *An Introduction to the Methods of Optical Crystallography*, 1961.

How Thin Should a slice be?



$$DOF = 1.77*\lambda/(NA^2)$$

Sheppard, Depth of field in optical microscopy, J Microsc, 149, 1, 73-75, 1988

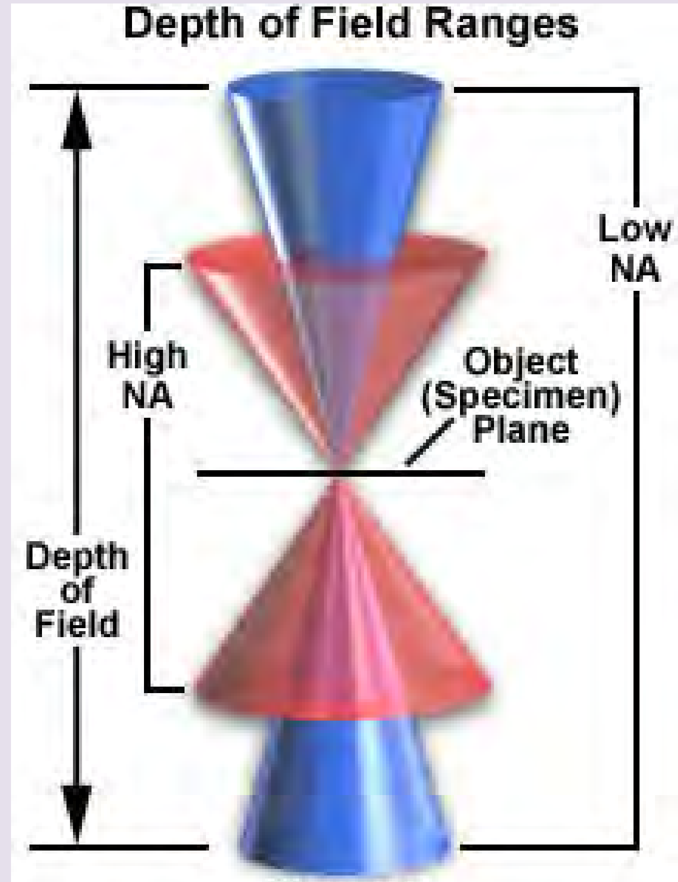
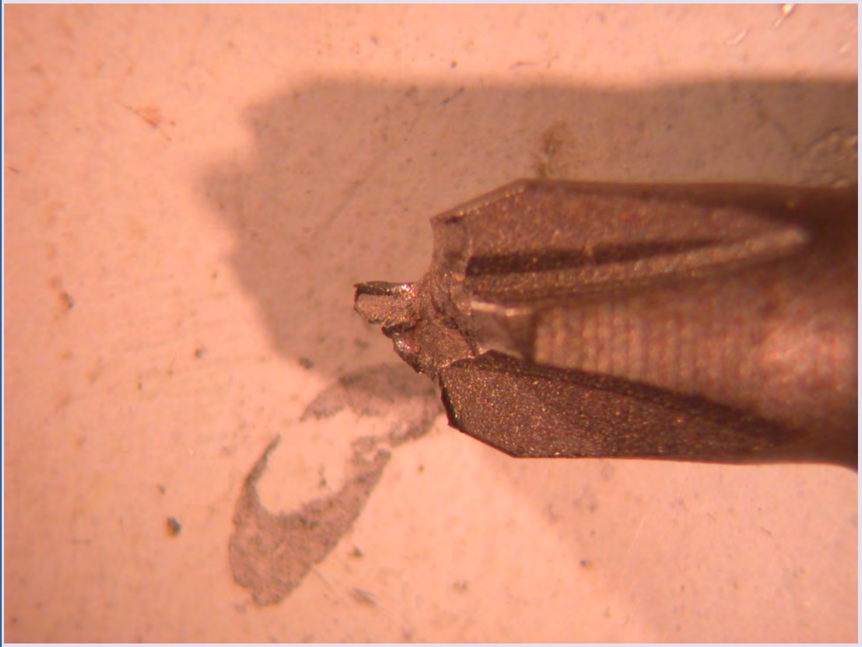
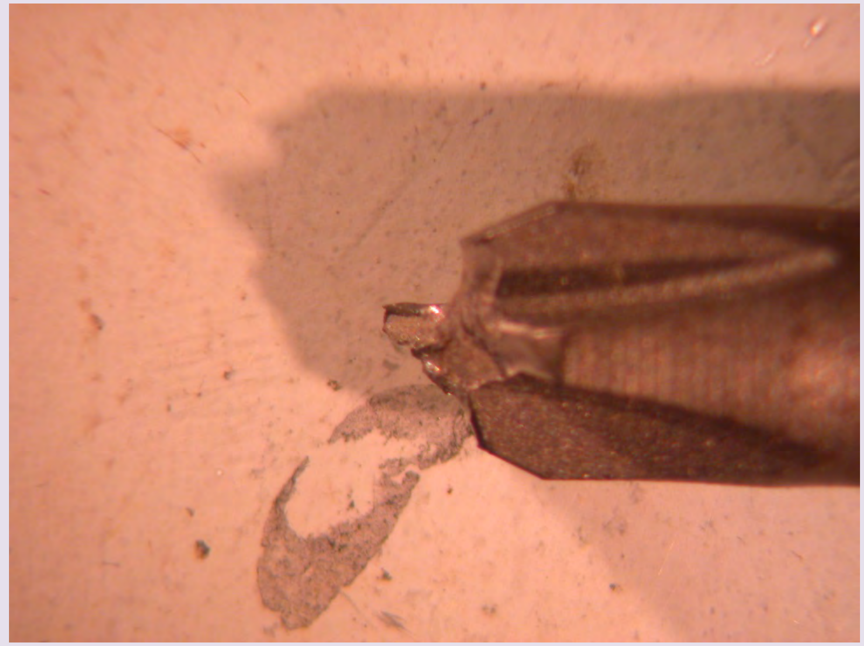


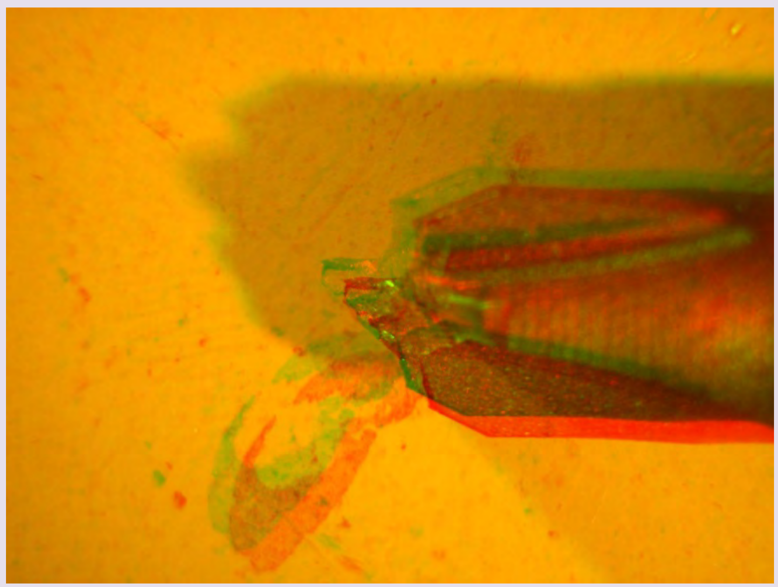
Figure 7

How to estimate ΔDepth for Slices

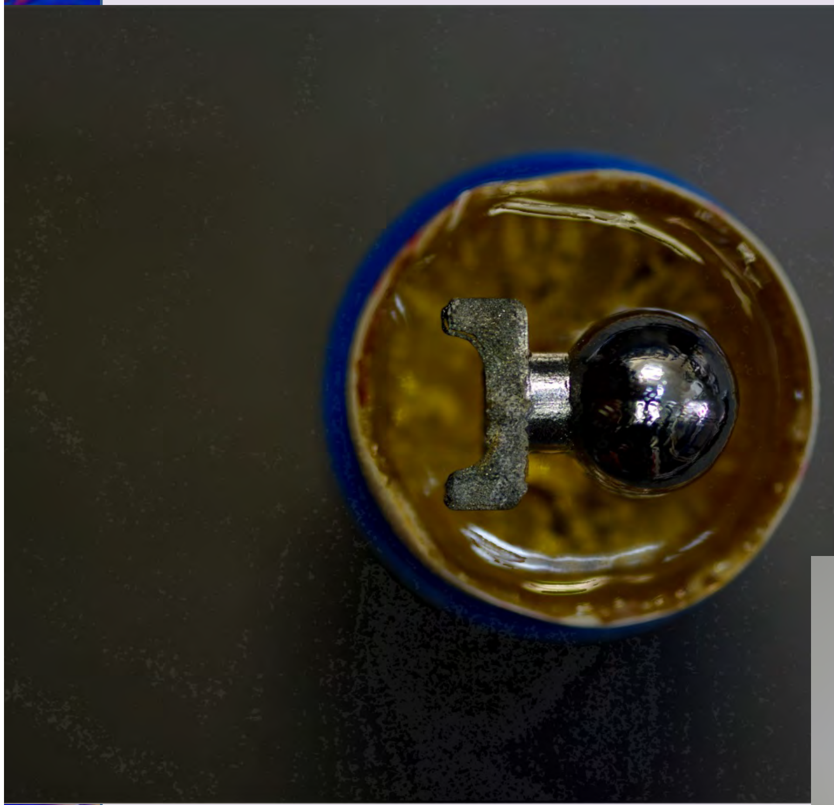
Focus Settings

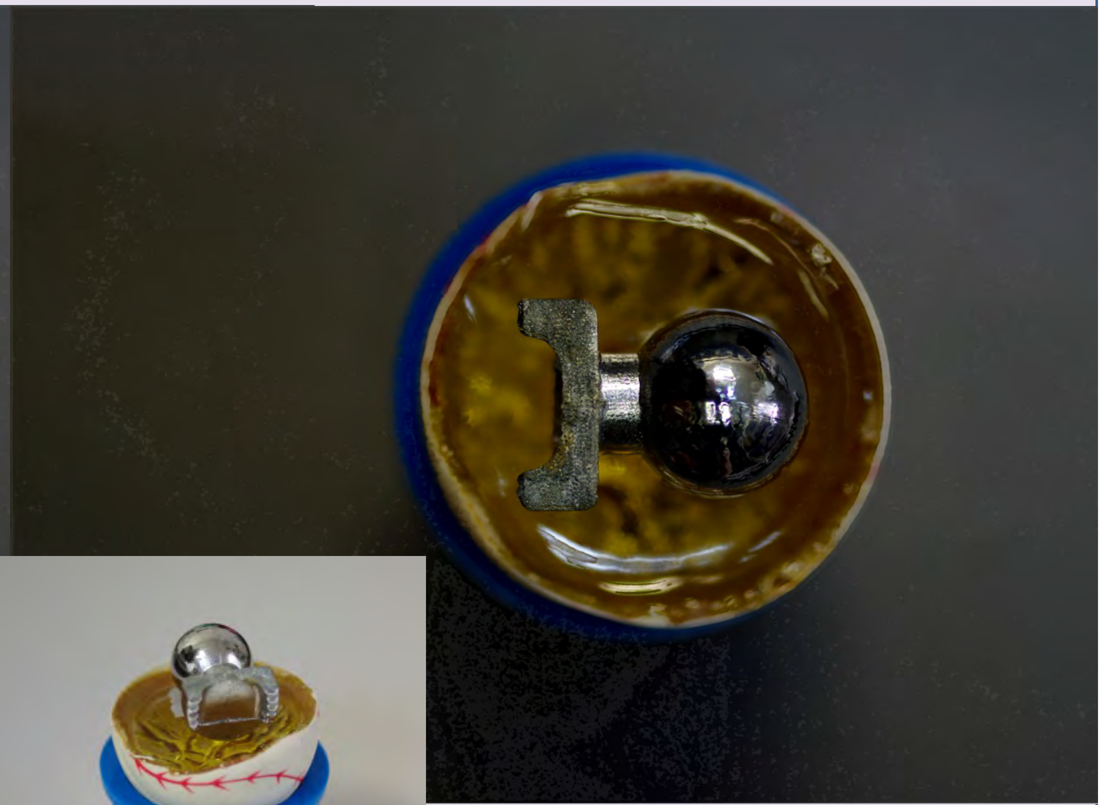


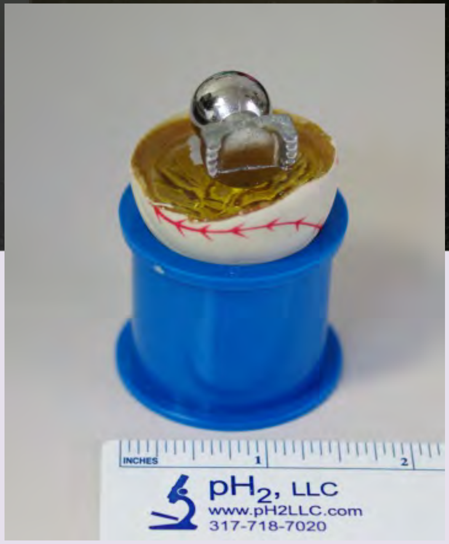




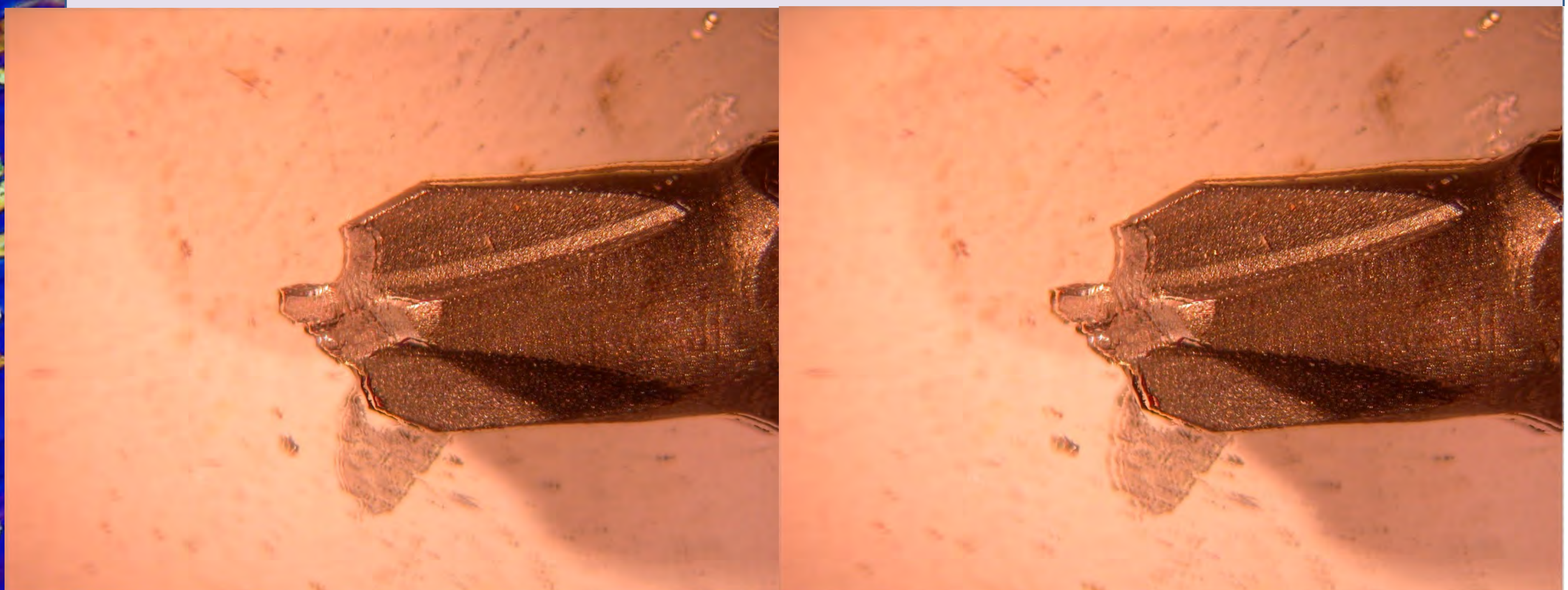
Fracture Pair (ca. 6 degree)





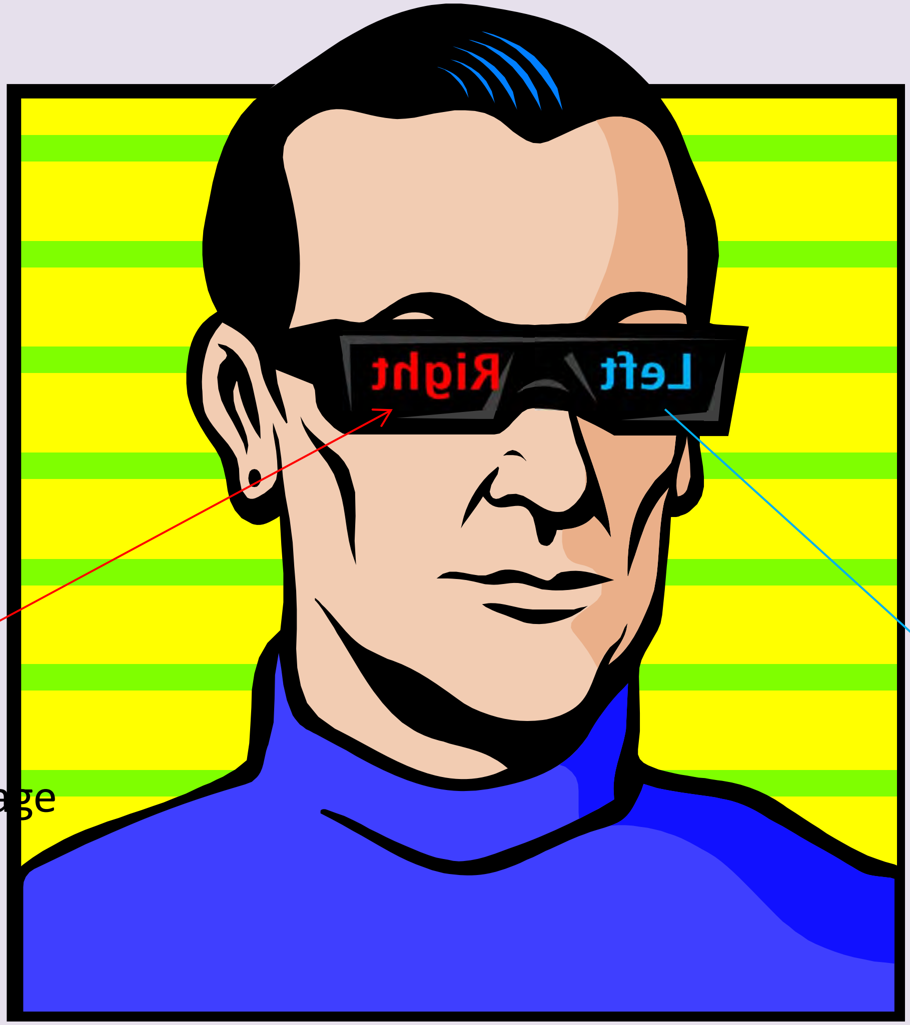


LR Pair



on paper Center of images should be
63-65 mm apart

Anaglyph

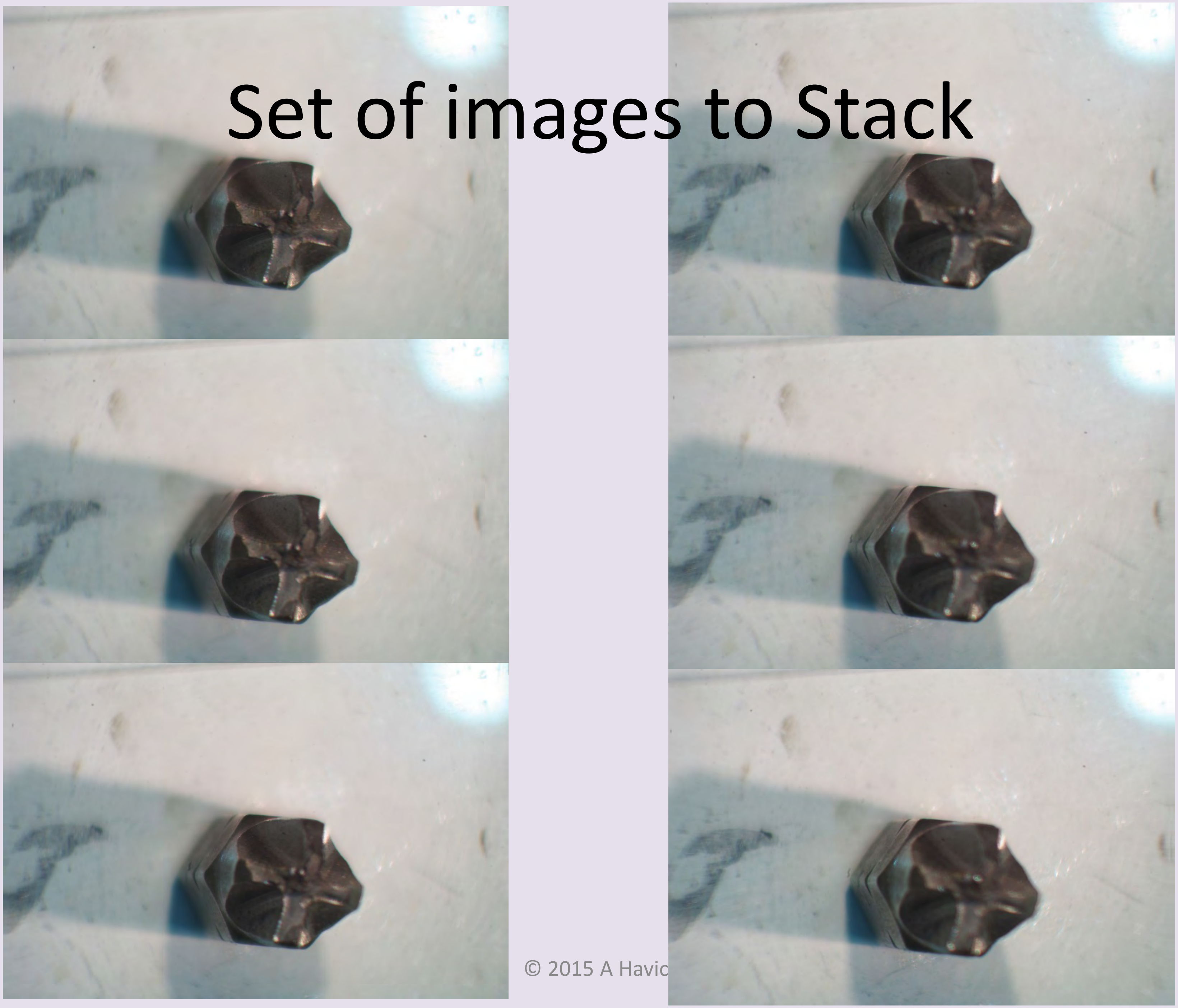


"Right"
mirror image
In Red

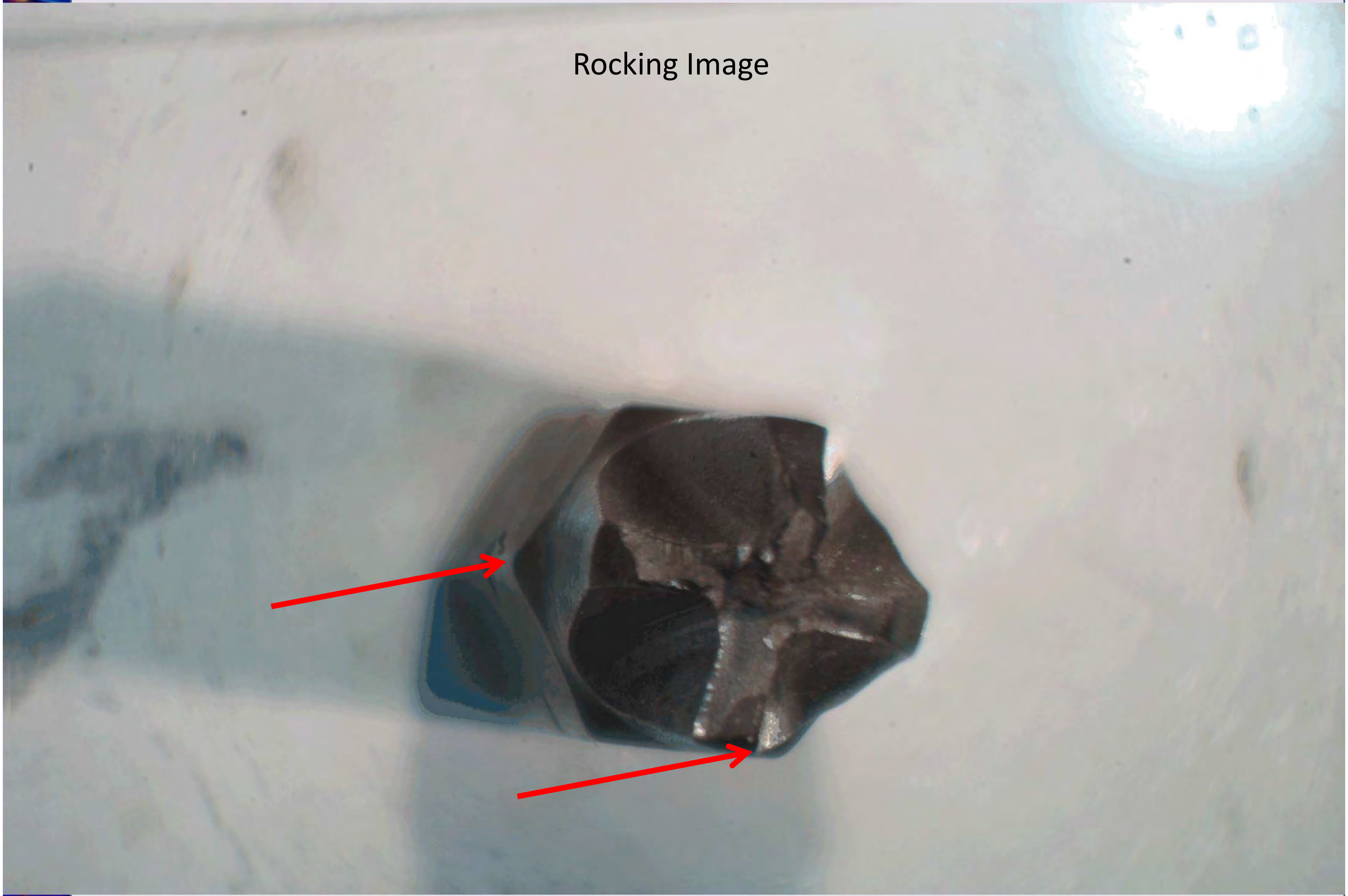
"Left"
mirror image
In Blue

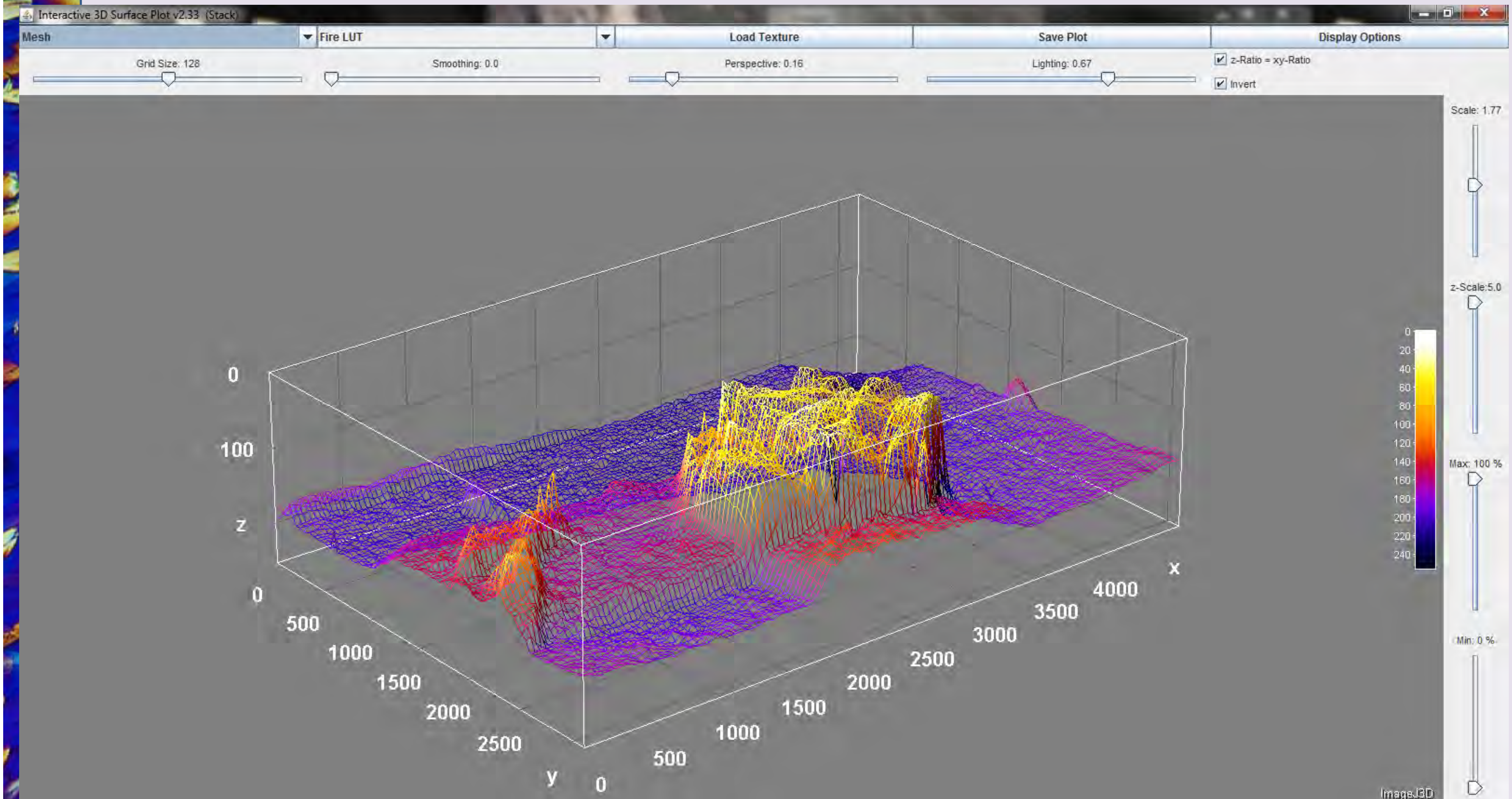
Anaglyph CM

Set of images to Stack

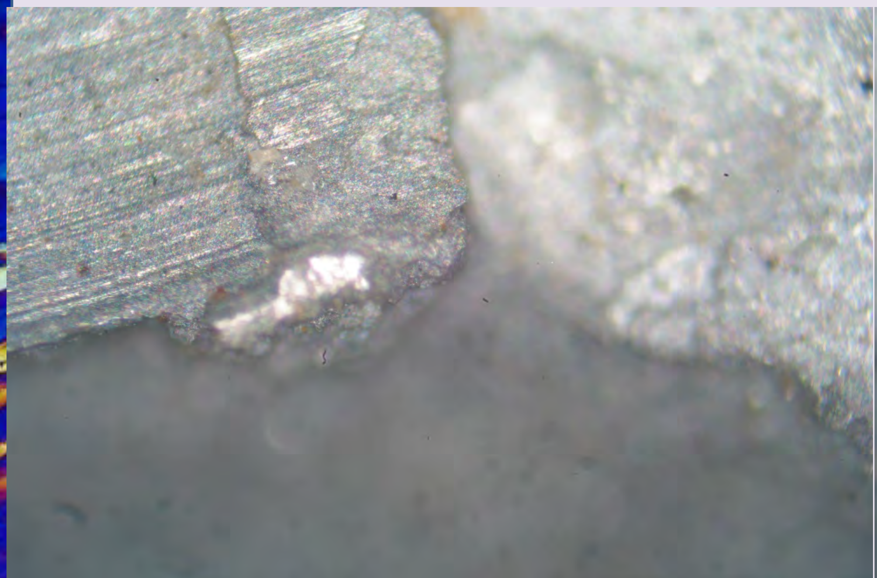


Rocking Image

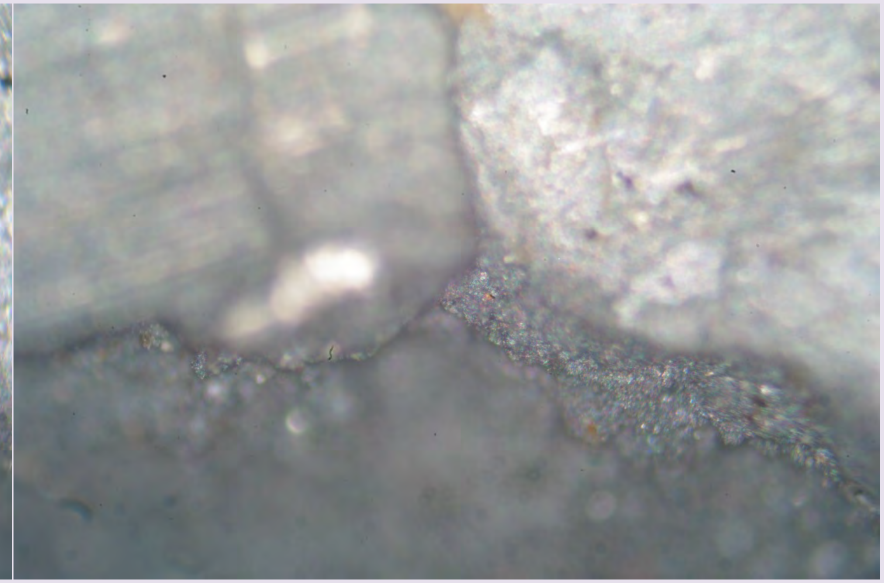



Interactive 3D Surface Plot v2.33 (Stack)
Mesh
Fire LUT
Load Texture
Save Plot
Display Options
Grid Size: 128
Smoothing: 0.0
Perspective: 0.16
Lighting: 0.67
z-Ratio = xy-Ratio
Invert
Scale: 1.77
z-Scale:5.0
Max: 100 %
Min: 0 %
0
100
z
0
500
1000
1500
2000
2500
y
0
500
1000
1500
2000
2500
3000
3500
4000
x
ImageJ3D

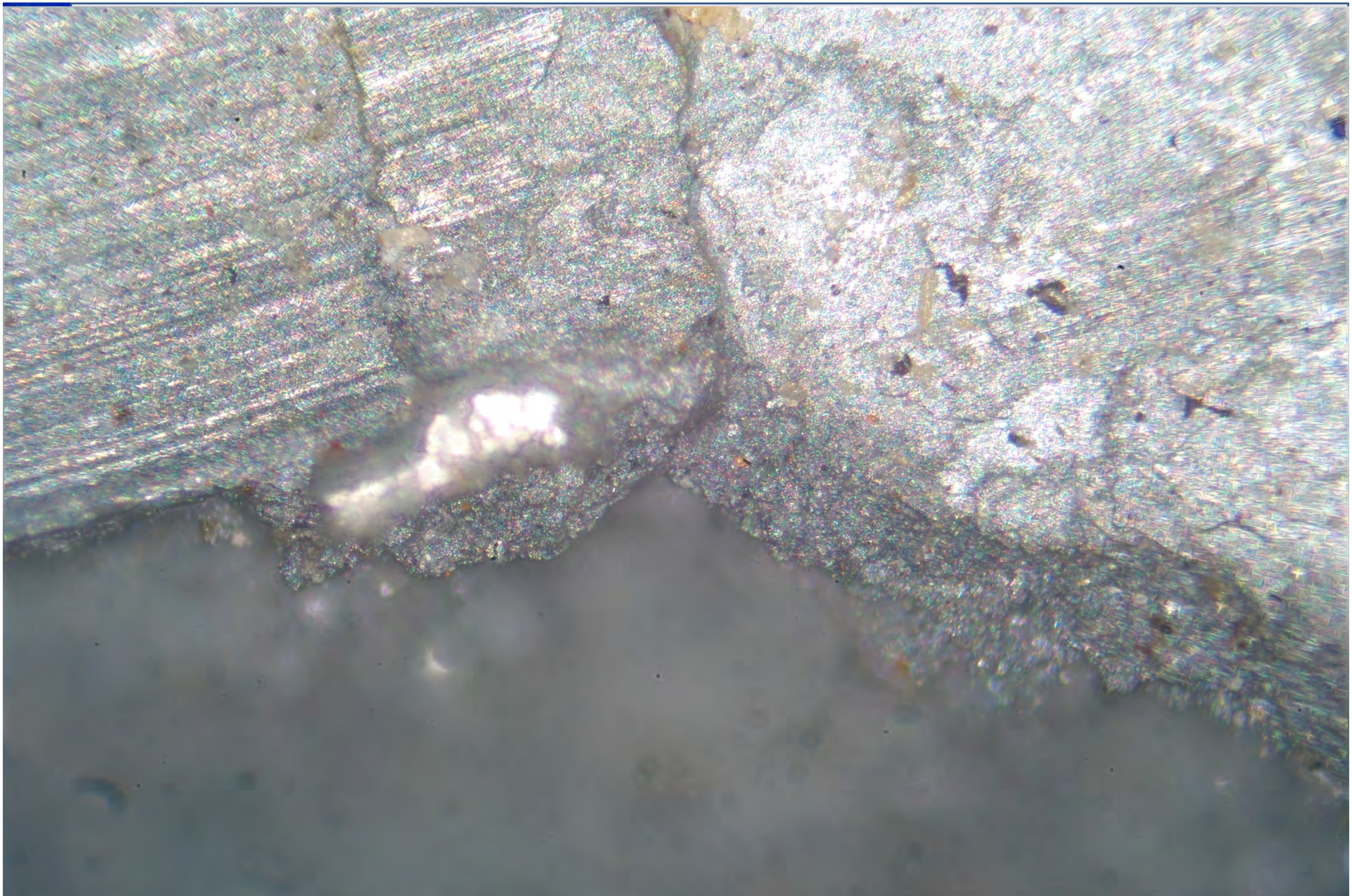
Z-Stacking

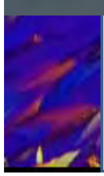


First in Series



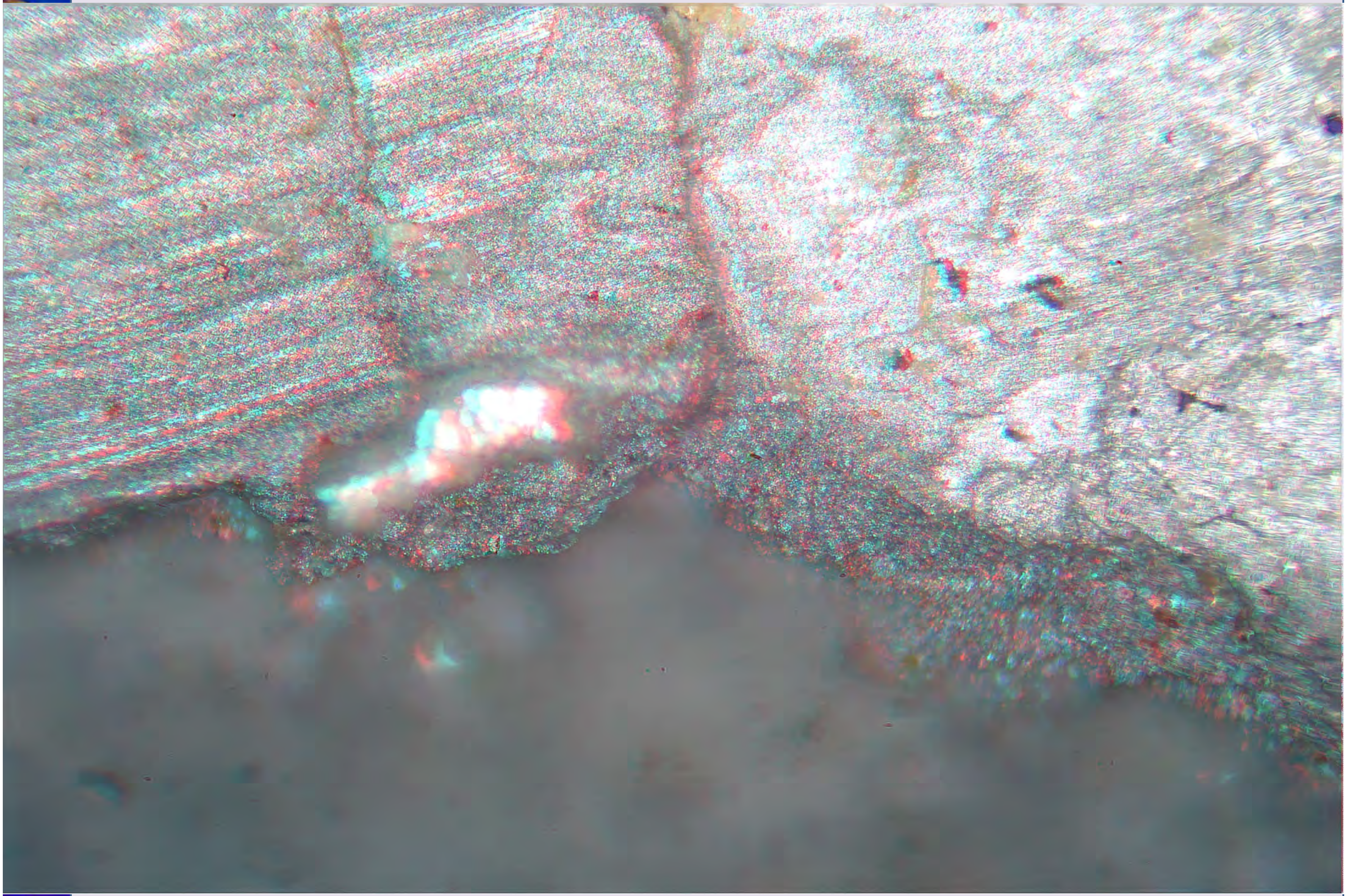
Last in Series





Zinc Fracture

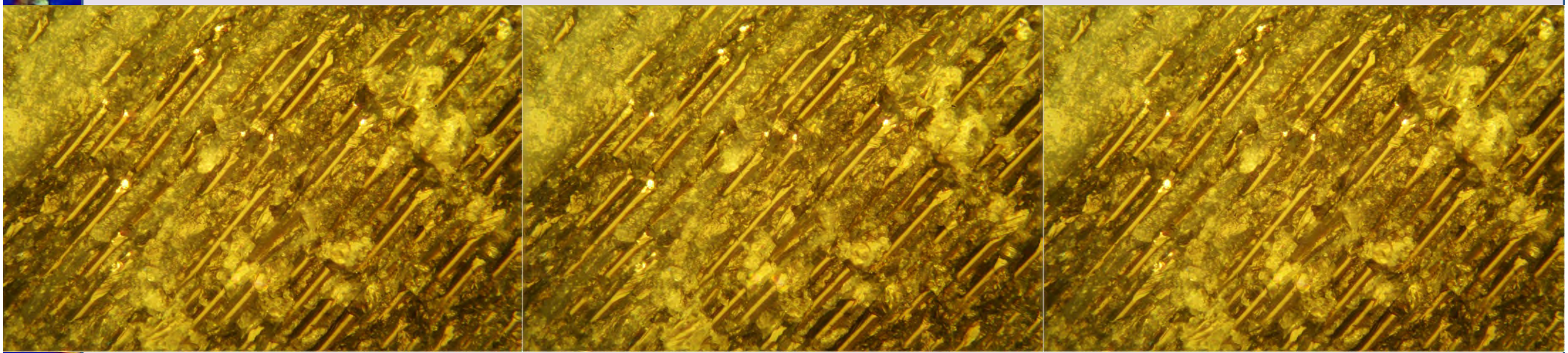
Stack of 17 images



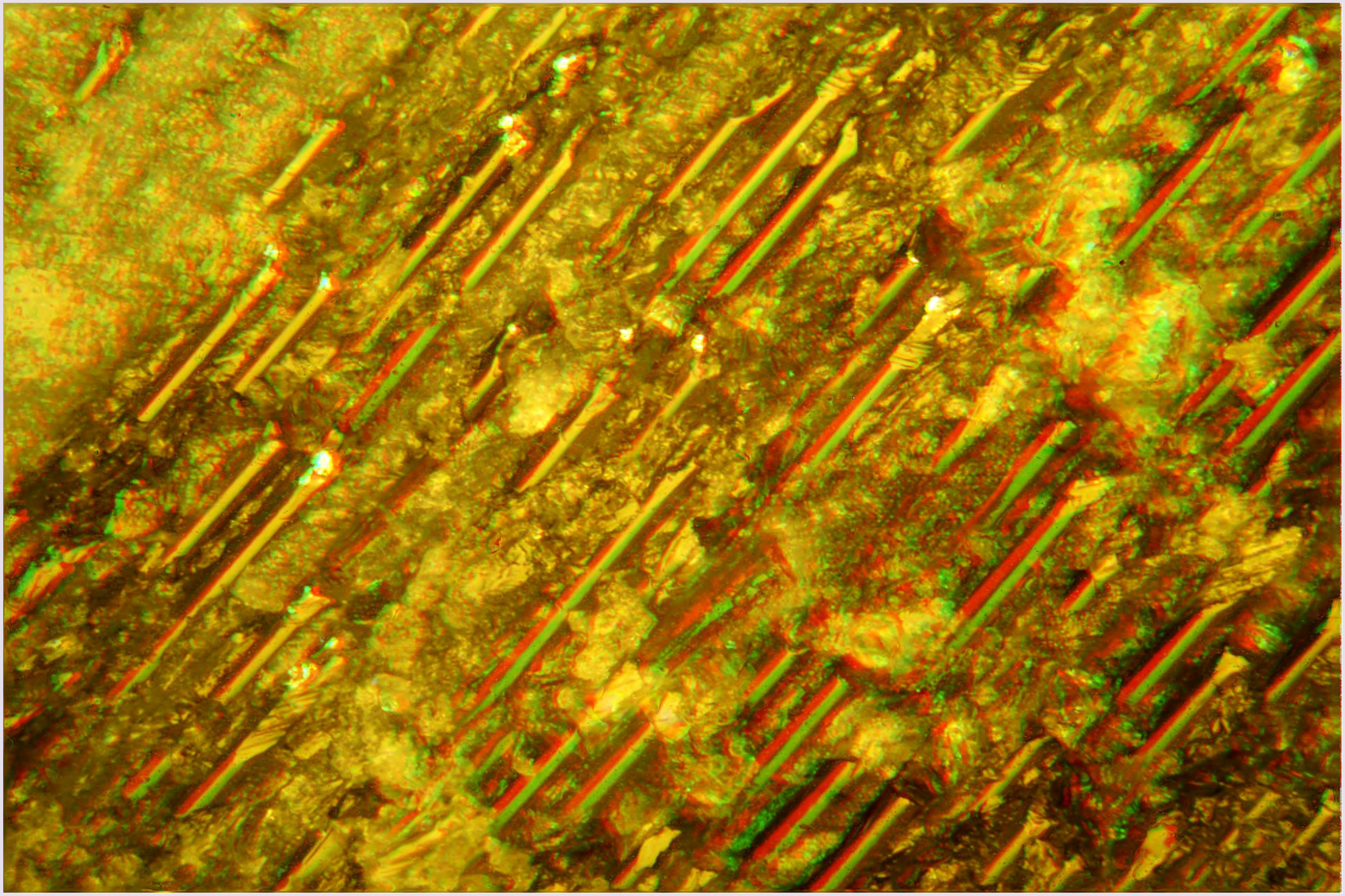
Zinc Fracture

Stack of 17 images

LRL



3D Composite



Questions?