| Day | Activity | Time | Task |
|---|---|---|---|
| Day 1 | a) 3D surface profilometry on Zygo Zegage;<br>b) Surface property measurements on Hysitron TI 950 Tribo-Indenter | 09:00 - 10:30 | Learning how to use the Zygo Zegage for 3D surface profilometry |
| | | 10:30 - 12:00 | Learning how to use Bruker TI 950 for nanoindentation and scratch |
| | | 12:00 - 13:00 | Lunch Break |
| | | 13:00 - 15:00 | Group A on Zygo; Group B on Bruker |
| | | 15:00 - 17:00 | Group B on Zygo; Group A on Bruker |
| Day 2 | a) Surface imaging of metal parts on an ZEISS EVO MA10 SEM;<br>b) Surface imaging using scanning probe microscopy on Bruker Multimode 8-HR AFM | 09:00 - 10:30 | Learning how to obtain images on an SEM |
| | | 10:30 - 12:00 | Learning how to obtain images on an AFM |
| | | 12:00 - 13:00 | Lunch Break |
| | | 13:00 - 15:00 | Group A on SEM; Group B on AFM |
| | | 15:00 - 17:00 | Group B on SEM; Group A on AFM |
| Day 3 | a) Surface chemical composition using EDS on ZEISS EVO MA10 SEM;<br>b) Organizing the results obtained from the previous scans on the other machines;<br>c) Discussion on the experience | 09:00 - 10:30 | Learning how to perform EDS on an SEM |
| | | 10:30 - 12:30 | Group A on SEM; Group B organizes results from Day 1 and Day 2 measurements |
| | | 12:30 - 13:30 | Lunch Break |
| | | 13:30 - 15:30 | Group B on SEM; Group A organizes results from Day 1 and Day 2 measurements |
| | | 15:30 - 17:00 | Discussions on the experience and Q&A session |

ENGINEERING
TEXAS A&M UNIVERSITY

# Surface Engineering

Instructor: Prof. Satish T.S. Bukkapatnam
Teaching Assistant: Akash Tiwari

# Schedule

- **Day 1**
  - 3D surface profilometry on Zygo Zegage
  - Surface property measurements on Hysitron TI 950 Tribo-Indenter
- **Day 2**
  - Surface imaging of metal parts on an ZEISS EVO MA10 SEM
  - Surface imaging using scanning probe microscopy
- **Day 3**
  - Surface chemical composition using EDS on ZEISS EVO MA10 SEM

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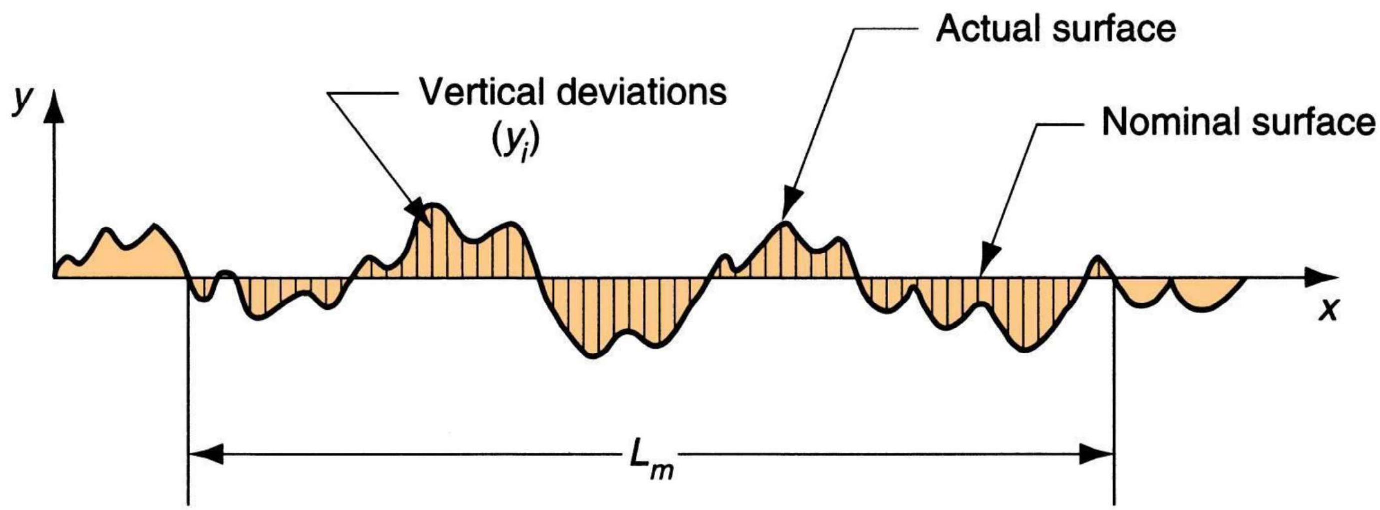
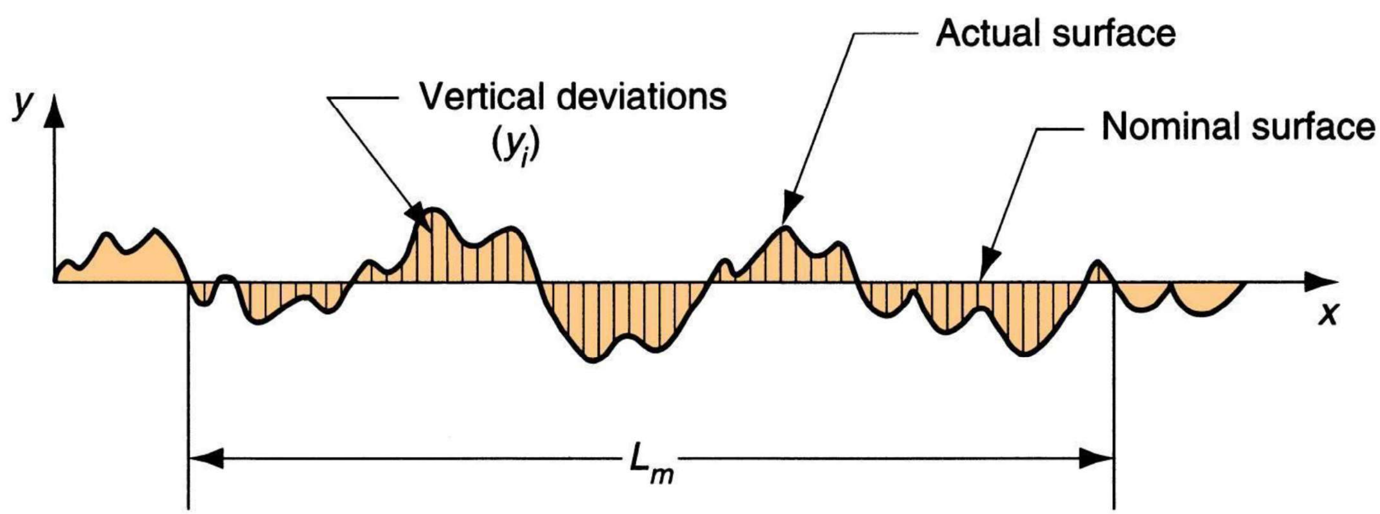
# Surface characteristics

- **Surface Roughness**
  - Sometimes also called “surface finish” or just “surface”. Acceptable surface roughness depends on the applications.
  - A laser mirror requires a very smooth surface whereas an orthopedic titanium implant requires a rough surface. Surface roughness is calculated from the asperities (high and low points) of a surface.




Surface profile along a scanned line

- **Surface Roughness**
  - After collecting the amplitude $y_i$'s all points $i$'s along an axis, the common surface roughness values are defined as:
    - Maximum Valley depth: $R_v = \min(y_i)$
    - Maximum Peak depth: $R_p = \max(y_i)$
    - Average roughness: $R_a = \frac{1}{n}\sum_i^n |y_i|$
    - Root mean squared $R_q = \sqrt{\frac{1}{n}\sum_i^n y_i^2}$



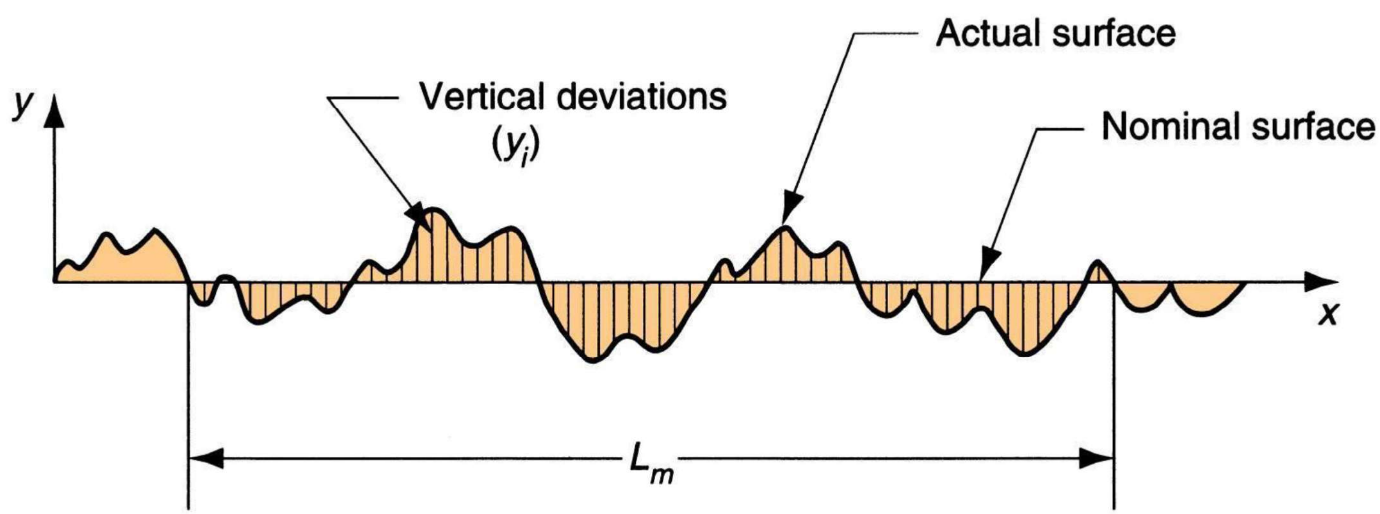

Surface profile along a scanned line

- **Surface Roughness**
  - Total roughness $R_t$ from the highest peak to $t$ he lowest valley points. It is also referred to as $R_t$ or $R_{max}$:

$$R_{max} \equiv R_t = R_p - R_v$$

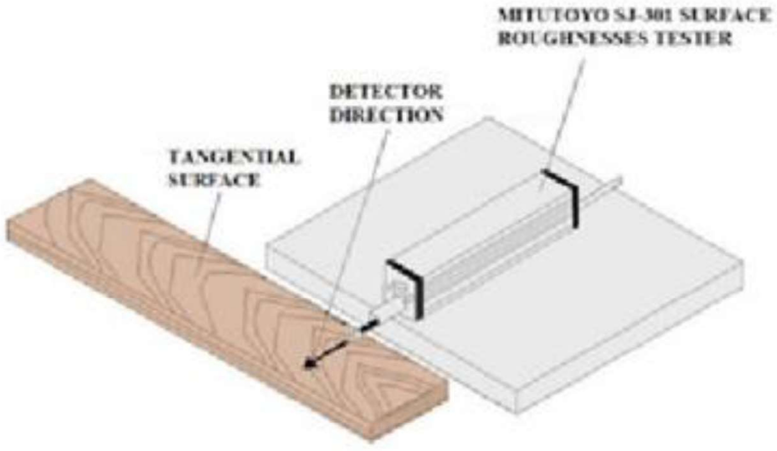
  - Average consecutive peak-valley roughness $R_z$. This is the average of 5 largest consecutive peak-valley distances

$$R_Z = \frac{1}{5}\left[\sum_{i}^{5}(R_{pi} - R_{vi})\right]^2$$

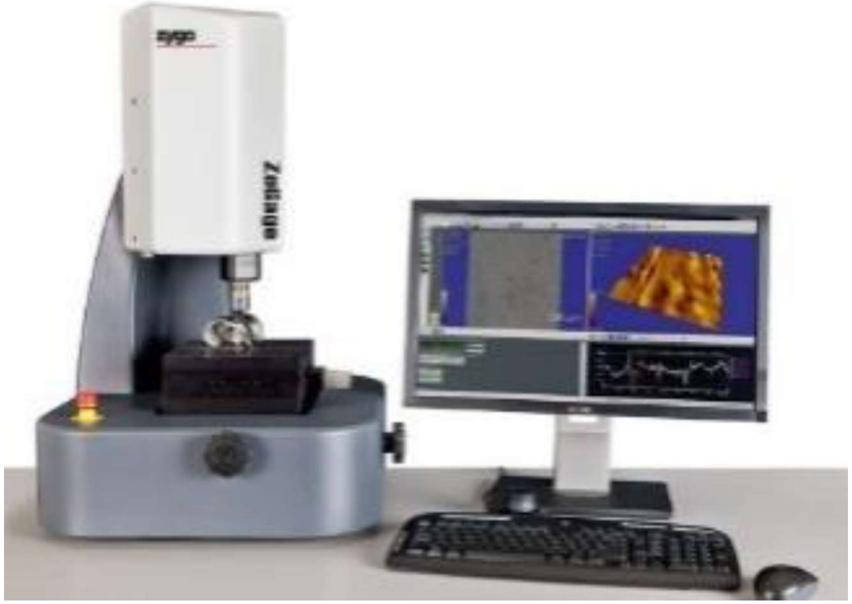



Surface profile along a scanned line

- **Surface roughness**



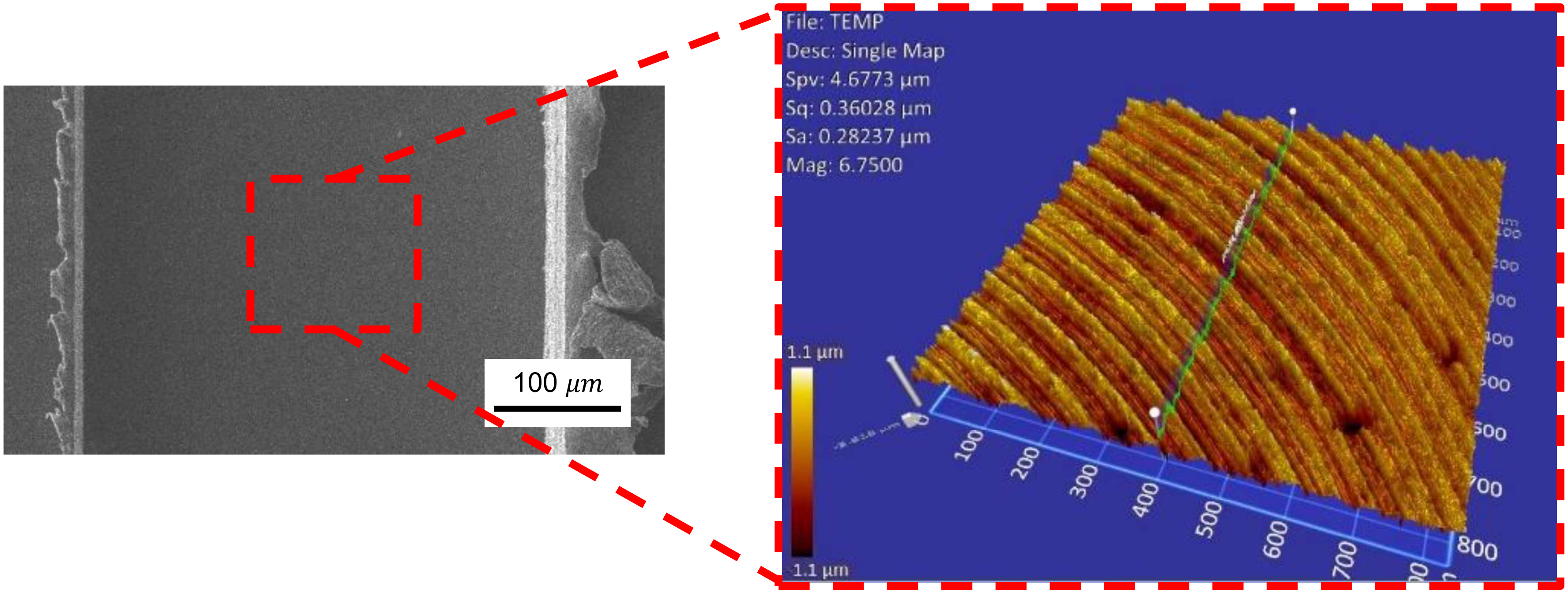

Surface finish measurement with a (contact type) profilometer



Surface finish measurement with a noncontact optical interferometer [www.zygo.com]

  - Surface texture means integrity of surface which includes finish and defects at or below surface.
  - For a 2D surface, similar calculations are performed but the results are labeled with a letter ‘S” as in $S_a$, $S_q$, $S_z$… rather than $R_a$, $R_q$, $R_z$… for line roughness measurement.

- **Profilometry**
  - A method to extract topographical data from a surface.
  - Instrument used for this purpose is known as Profilometer.

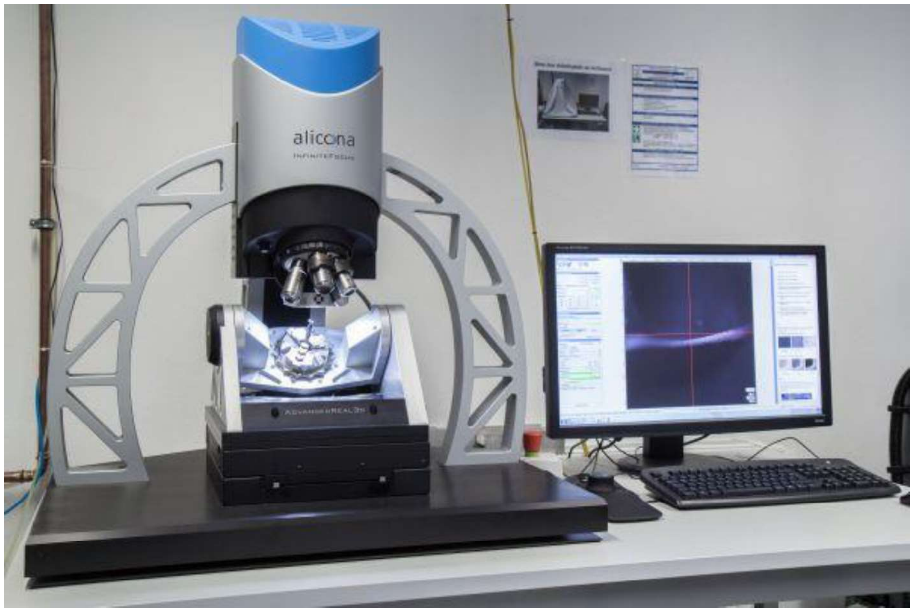


# Profilometry

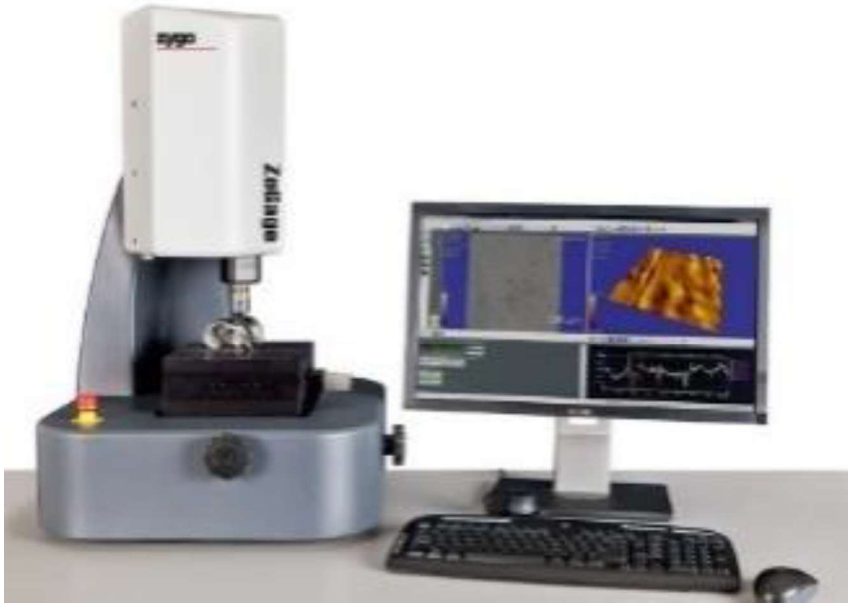
- **Purpose of using profilometer**
  - How rough is surface?
  - What is the density of defects?
  - What is the area of voids?
  - What is the height of the features

- **Functionality of profilometer**
  - Measure surface profile/morphology and defects/voids
  - Generate quantifiers (surface roughness) for surface characteristics
  - Questions: what are the approaches for getting the profile?
    - It can be a single point, a line scan or even a full three-dimensional scan

- **Contact/Non-contact profilometers**
  - Digital holographic microscopy
  - White light interferometry
  - Phase shifting interferometry
  - Advantages:
    - Prevent surfaces from scratches
    - High lateral resolution
    - High speed when requirement is of small steps

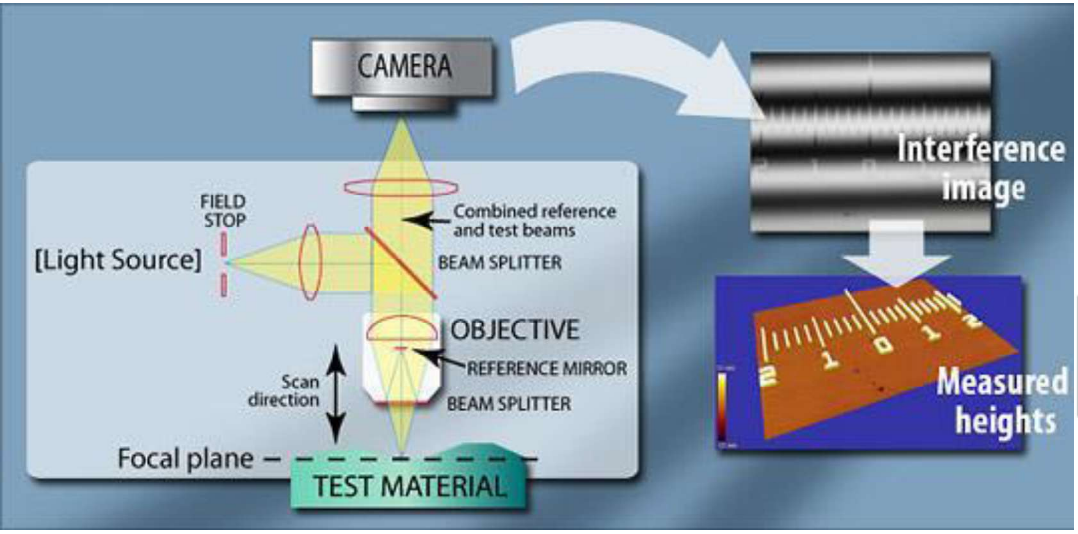


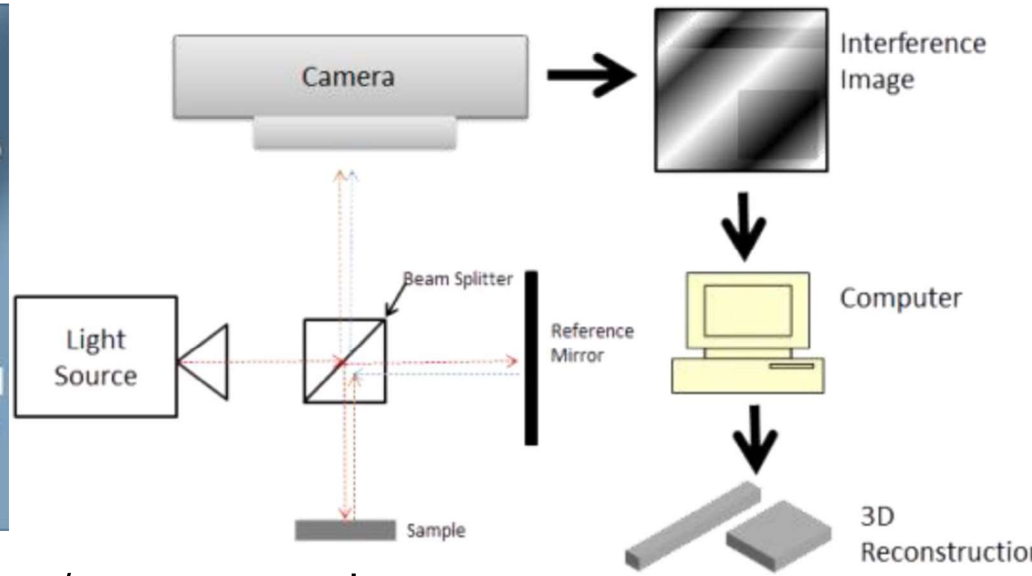

http://www.isf.de/en/institut/ausstattung/alicona.html



Surface finish measurement with a noncontact optical interferometer [www.zygo.com]

# Principles of Optical Profilometry



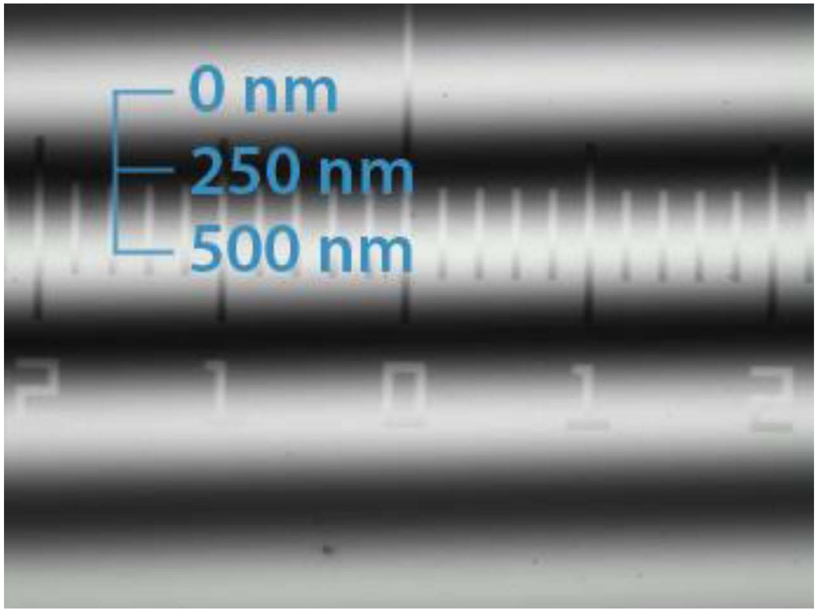




Courtesy of www.zygo.com/www. nanoscience.com

– Light beam is splits, and then reflection from reference and test material occurs, resulting in the interference
– Formation of inference fringes(light and dark bands) can be seen
– Constructive inferences areas are the lighter ones and destructive inference areas are darker ones

# Principles of Optical Profilometry

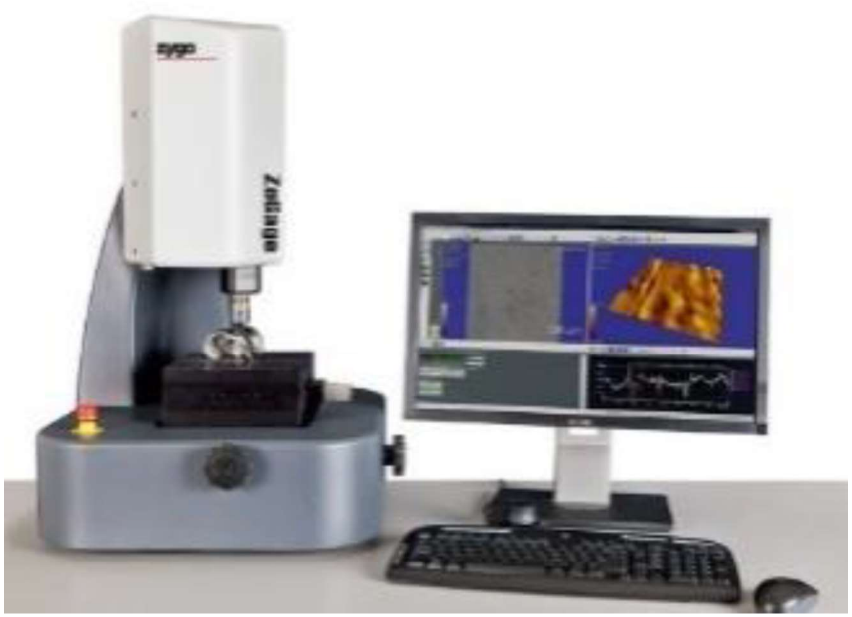
- Wavelength of difference between reference and test path is equal to distance between consecutive fringes of same color
- Height variance on the test surface causes optical path differences
- Out of focus area means less inference
- Higher the contrast means better the focus



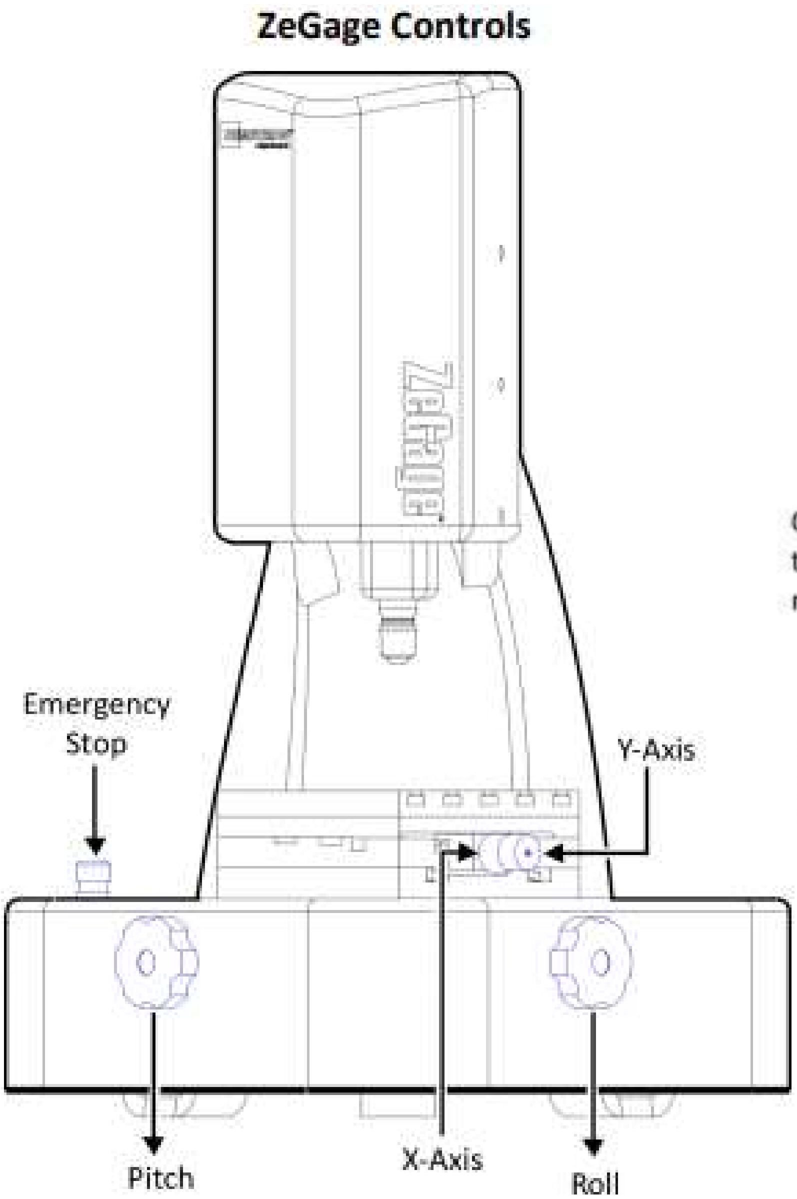

www.zygo.com

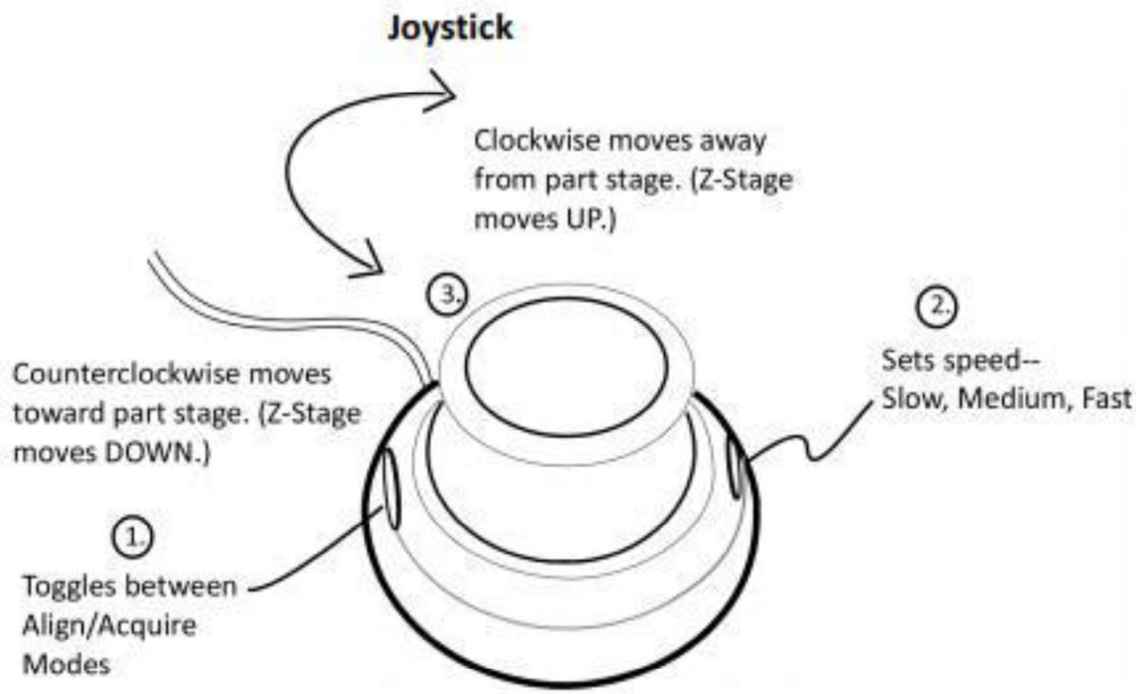
# Profiolometer

- **ZeMaps Software**
  - It has a visually rich interface enabling you to see what is happening at virtually every step in the process
  - Each 3D measurement provides one million data points, making it possible to evaluate the effects of surface processing
  - ISO roughness parameters are standard with the software as are a variety of profiling, plotting, filtering and other interactive data analysis tools



# ZeGage Profilometer



## Open ZeMaps

1. Open the ZeMaps software by double-clicking on the ZE icon on the desktop. Wait for the initialization routine to be completed.
2. If log in is required for your system, there are two locations from which you can access the login dialog.
   - In the menu bar at the top of the screen, select:

     File→Logout

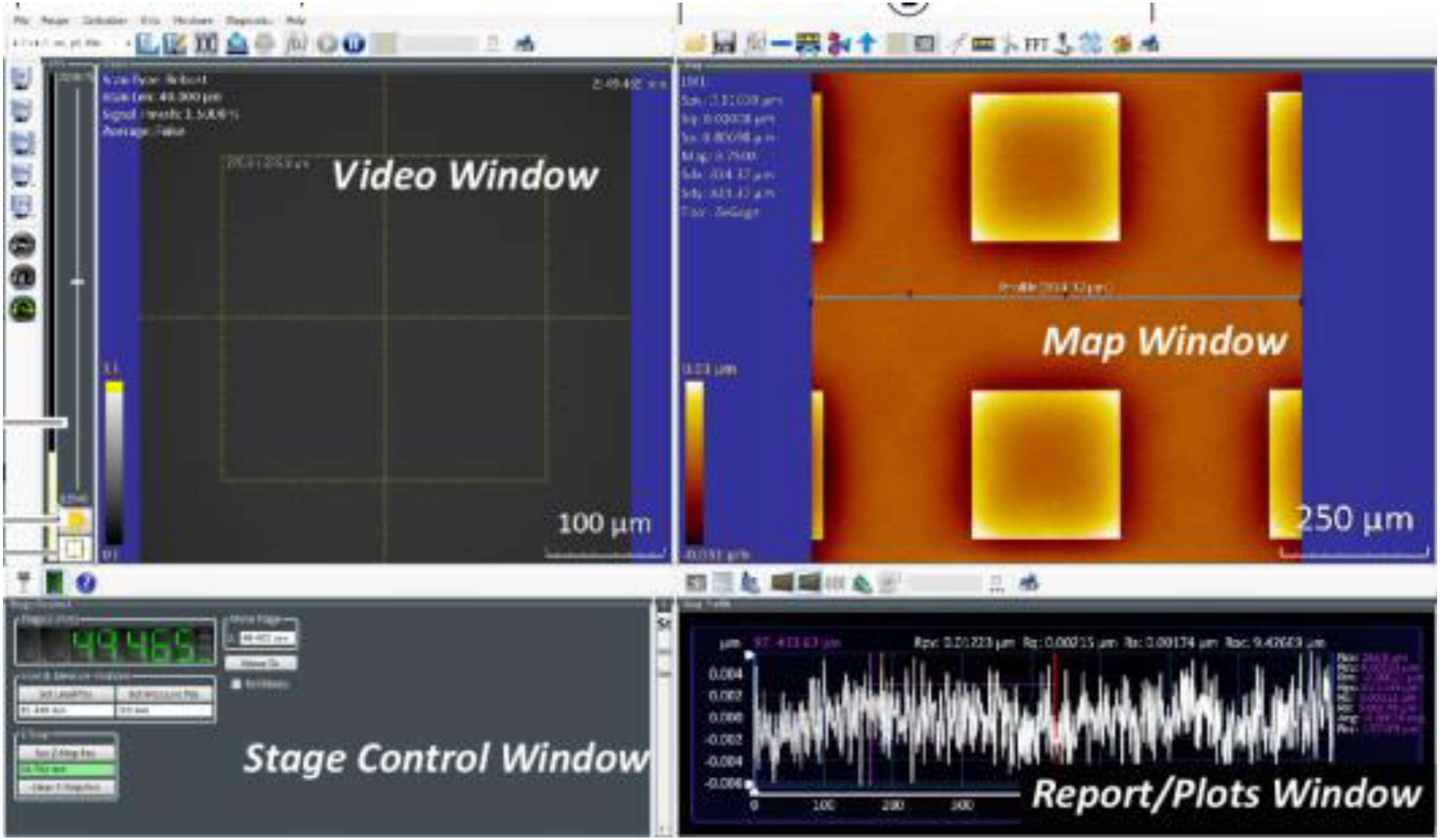
     **OR,**
   - Click on the Login/Logout icon in the Stage Control Window.
3. Enter Name and Password and click Ok.


**Software Icon**


**Login Dialog**

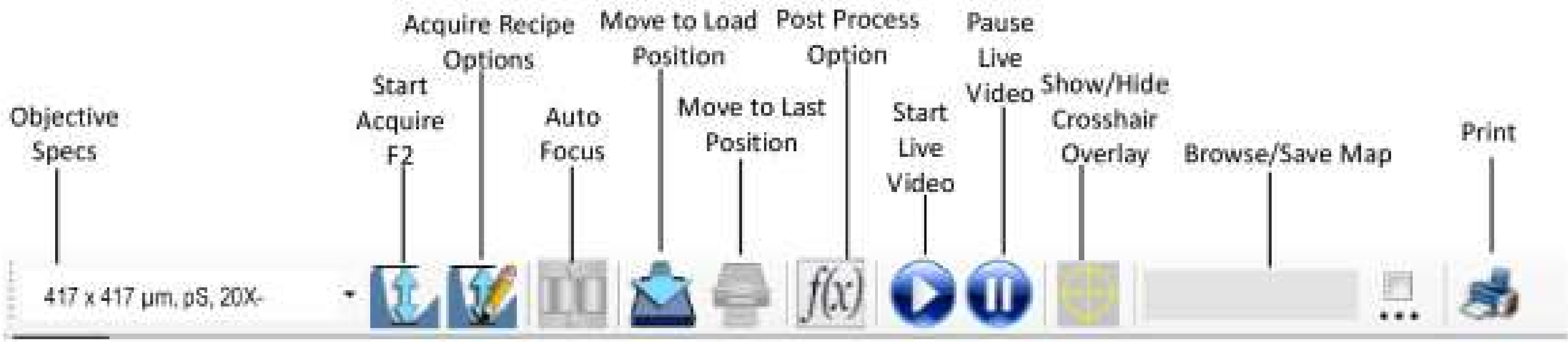
**Log In/Logout Icon**

# Understanding ZeMaps

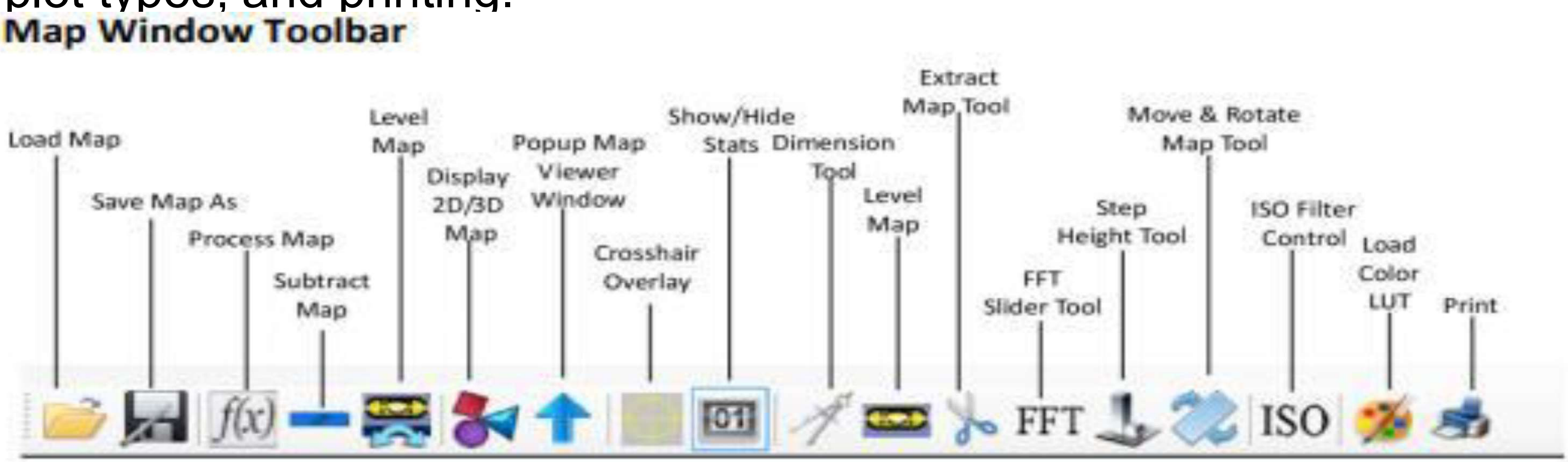


# Understanding ZeMaps

- **Video Window-** This window provides access to controls for focus and alignment, data acquisition, viewing, locating areas of interest on a test part, and saving files.

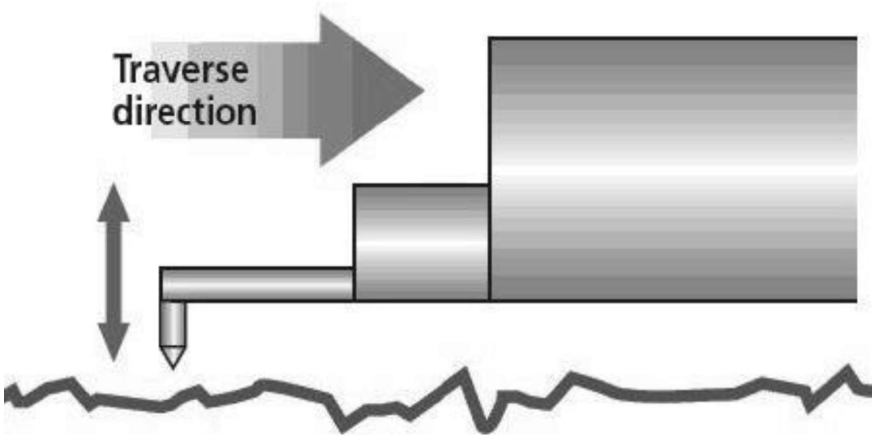


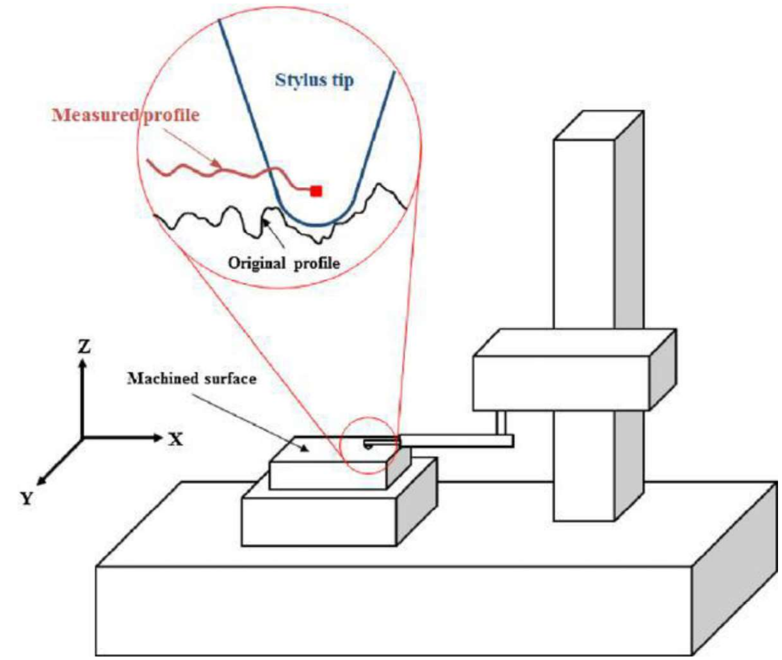

- **Map Window -**This window displays 2D and 3D maps of surface data. There are options for saving and loading maps, processing data, changing plot types, and printing.



# Contact profilometers

- Exemplary equipment
  - Stylus profilometer
  - Atomic force microscopy
  - Scanning tunneling microscopy
- Advantages
  - Standards of surface finish are mostly written using contact profilometers as benchmark examples
  - Direct technique and modeling is not required
  - “Analog” data- Resolution is very high
  - Independent of the surface and environment contaminants



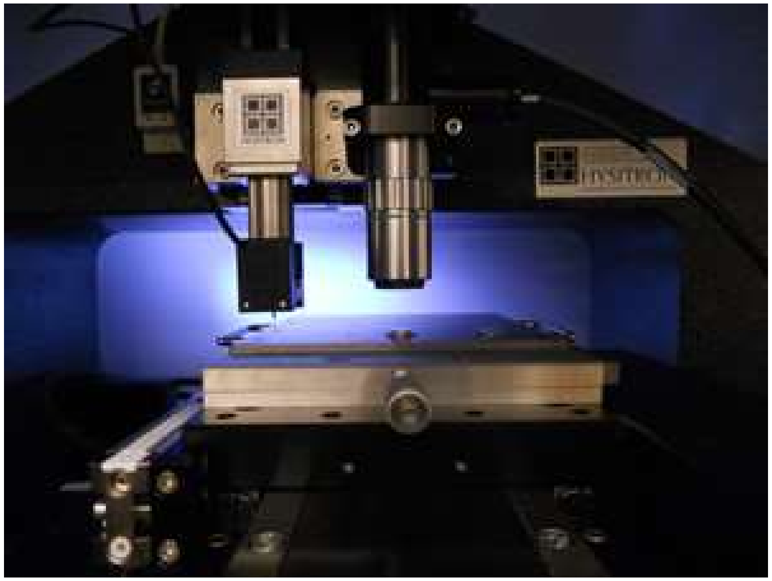




Profile data acquisition by a stylus-type profilometer ( Credit: Dong-HyeokLee, MST, 2012, J. Rusnák’ et. al, 2010 )

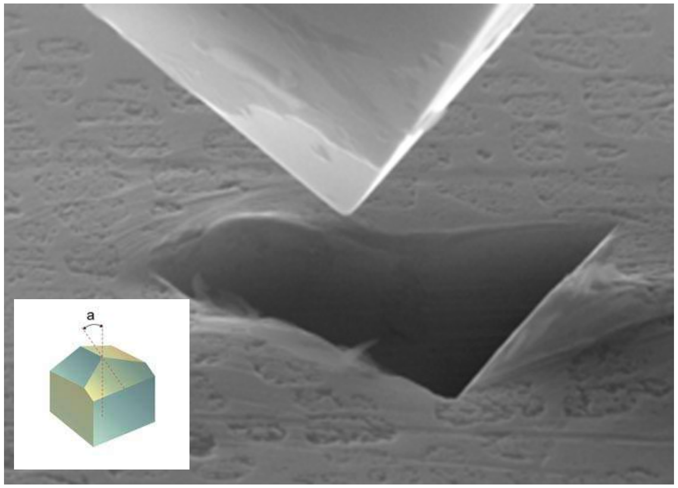
# Nanoindentation for Hardness Testing

- **Hardness**

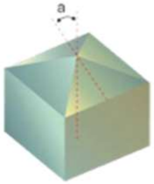
- *Hardness* of a material is defined as its resistance to permanent indentation (or) scratching or wear.
- Nanoscale hardness is important consideration in thin-film coatings for application in MEMS and optical devices.
- Standardized tests for hardness include – Brinell, Rockwell, Vickers and Knoop.



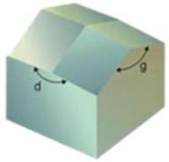
Hysitron TI 950 Triboindenter



Bruker – Berkovich tip



Knoops

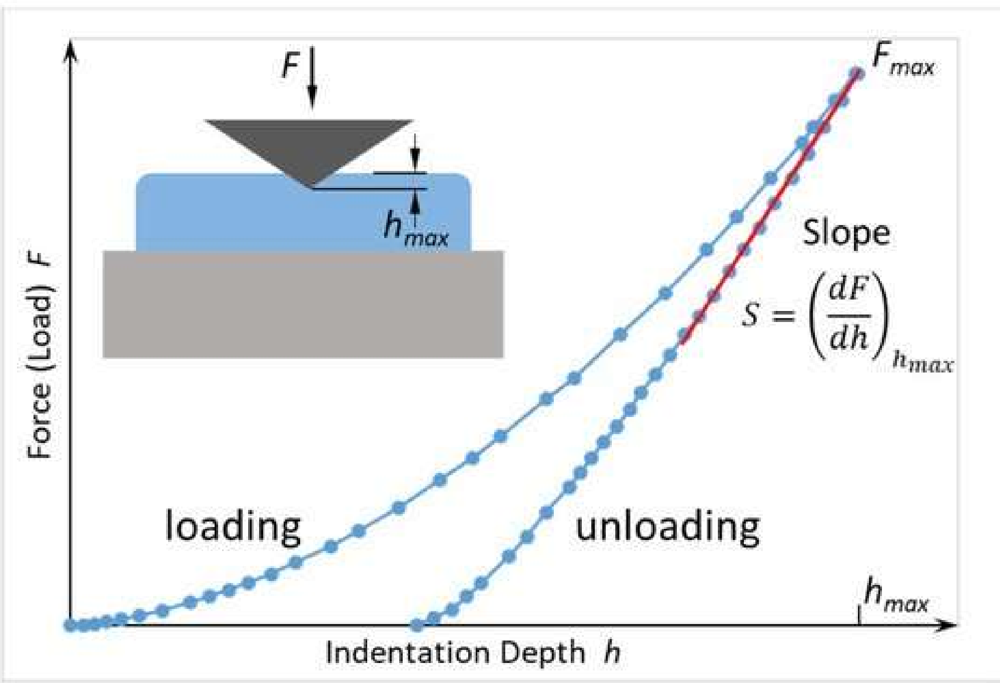


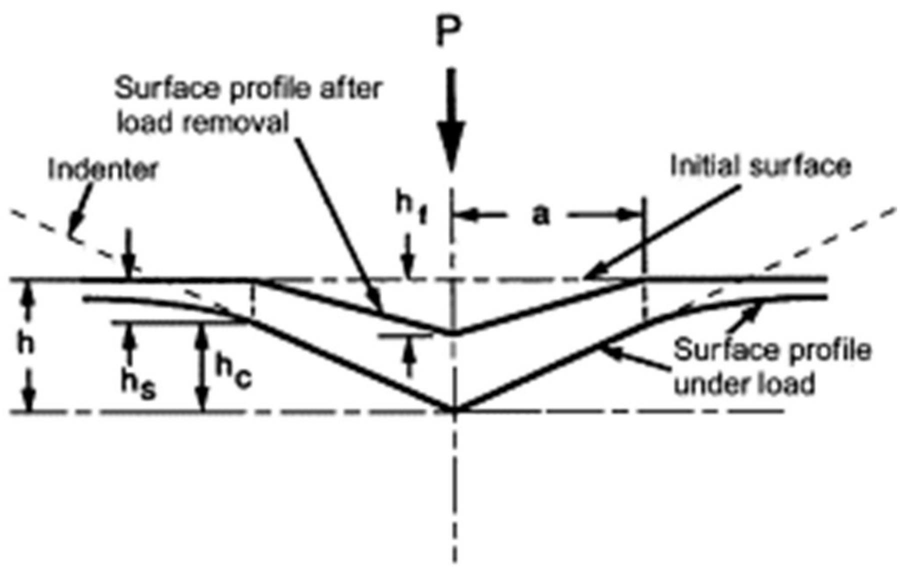
Vickers

# Principles of measurement for hardness

- Hardness measurement is based on Area of Contact, Contact depth and force experienced by the indenter.
- Oliver-Pharr method is applied to obtain hardness value:

Hardness: $$H = \frac{P_{max}}{A(h_c)} \quad \begin{cases} P = A(h - h_f)^m \\ h_c = h_{max} - 0.75\dfrac{P_{max}}{S} \end{cases}$$



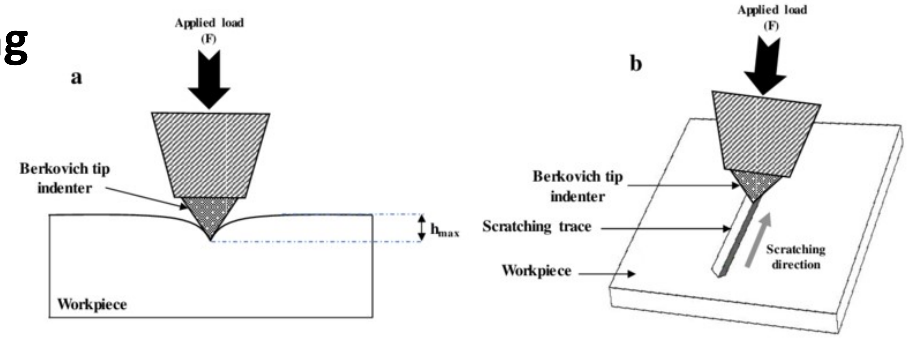



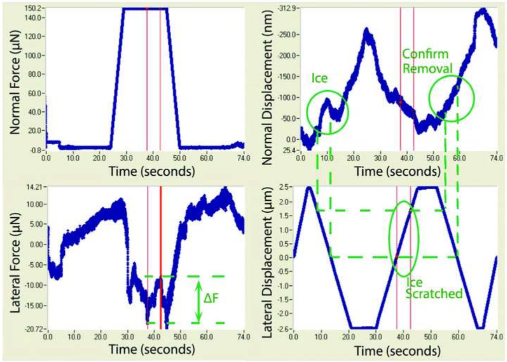
- **Coefficient of friction**
  - *Coefficient of friction* $\mu$ at the interface is defined as

$$\mu = \frac{F}{N} = \frac{\tau A_r}{\sigma A_r} = \frac{\tau}{\sigma} = \frac{\tau}{\text{Hardness}}$$

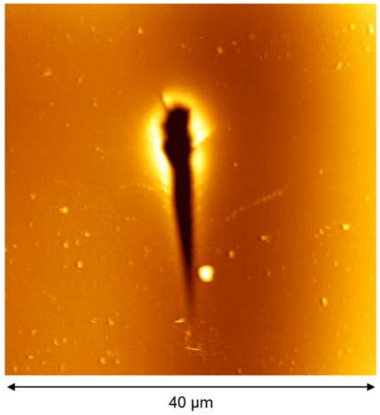
  - $\mu$ can be reduced by reducing shear stress or increasing Hardness.

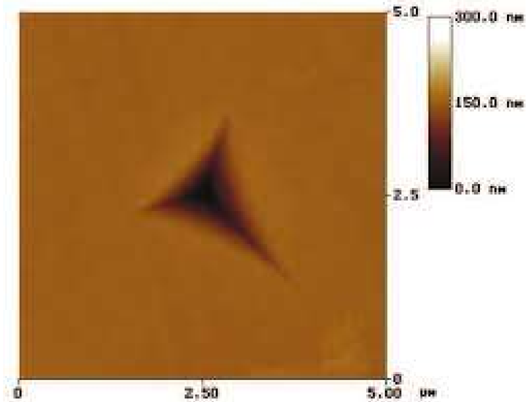
**Scratch testing**



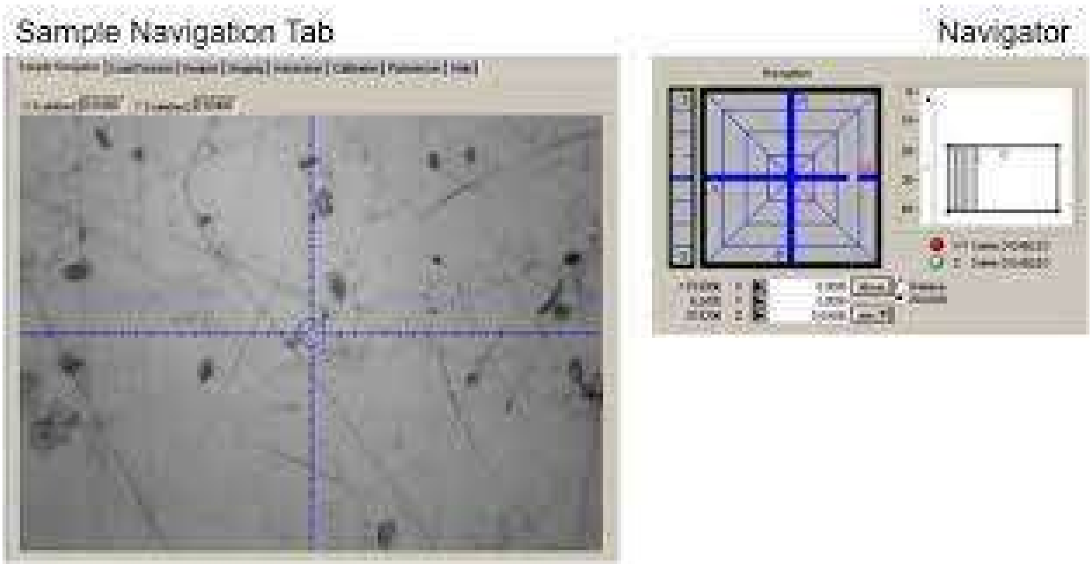

**Imaging**



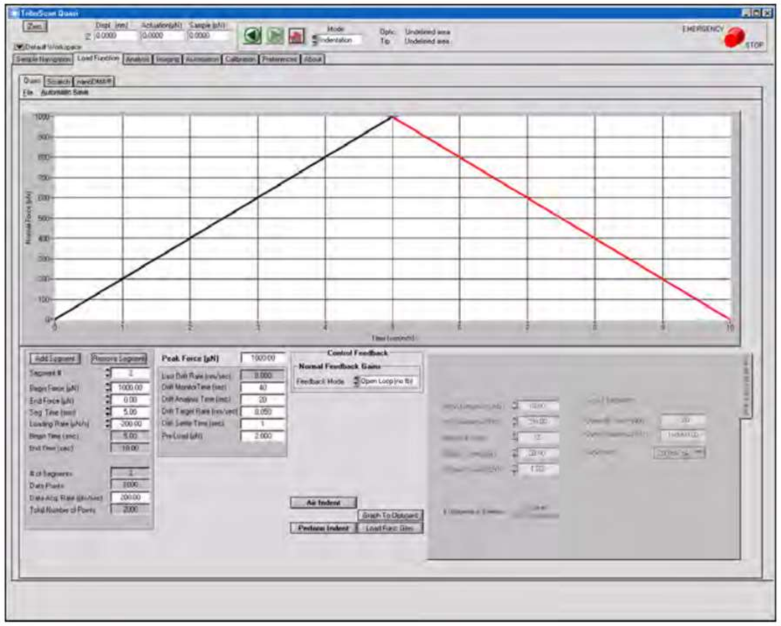



# Machine Operation

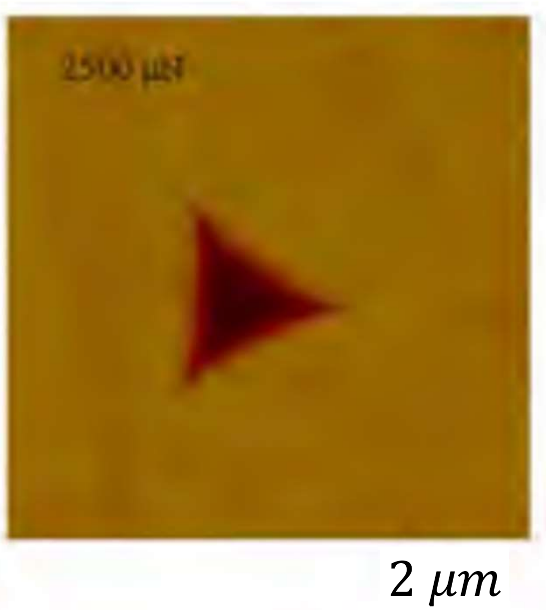
## Navigating Sample



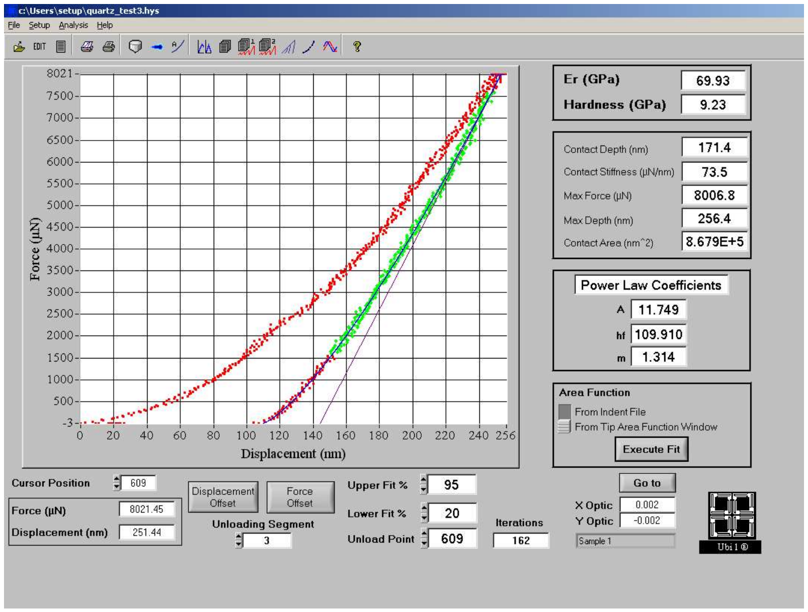

## Setting Load Curves



## Performing Indents and scratch




## Analysis



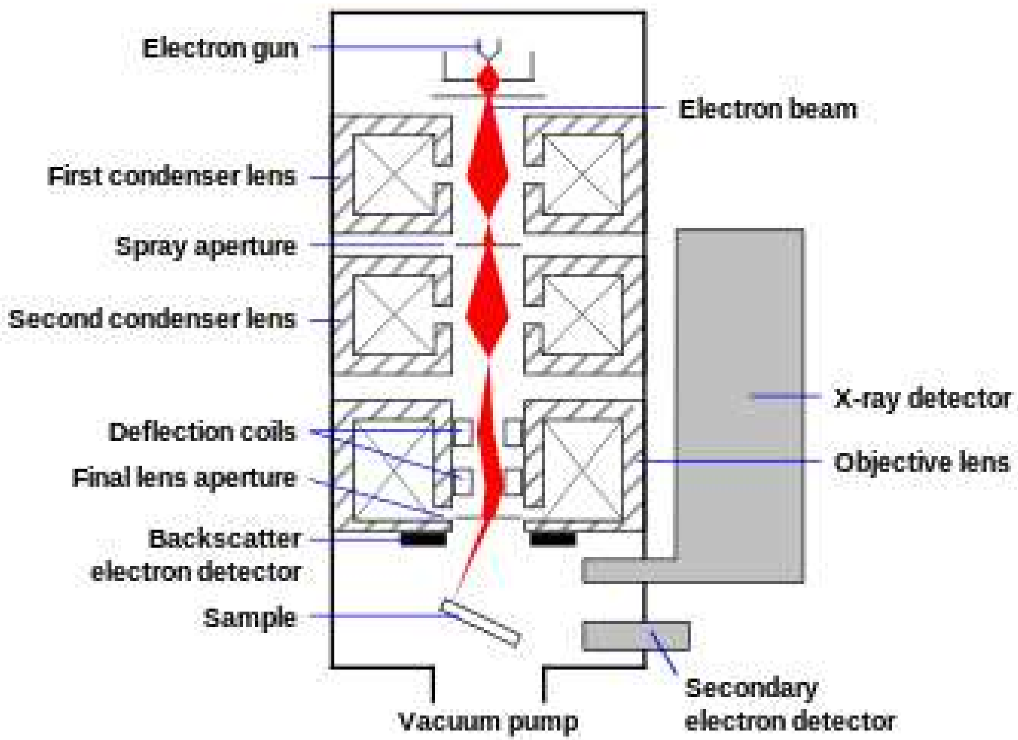
# Scanning Electron Microscopy SEM

- **Functions of SEM**
  - Tiny electron beam scanned across surface of specimen
  - Magnification range 15x to 200,000x
  - Resolution of 50 Å
  - Wide range on depth of field
  - Specimen should be conducting (or coated with thin conductive layer)
  - Specimen size limited by size of sample chamber

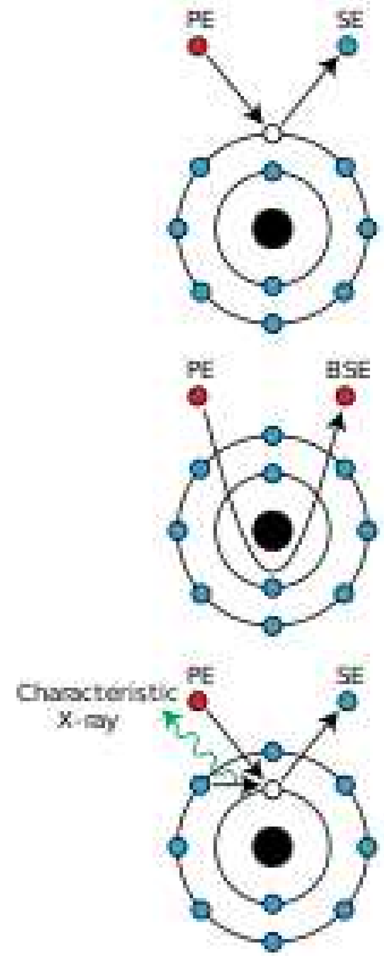

https://www.imaging-git.com/products/electron-and-ion-microscopy/carl-zeiss-reveals-high-definition-fe-sem-sigma-hd

# Electron microscopy-SEM

- **Scanning Electron Microscopy (SEM)**
  - Scanning process and image formation




Schematic of an SEM

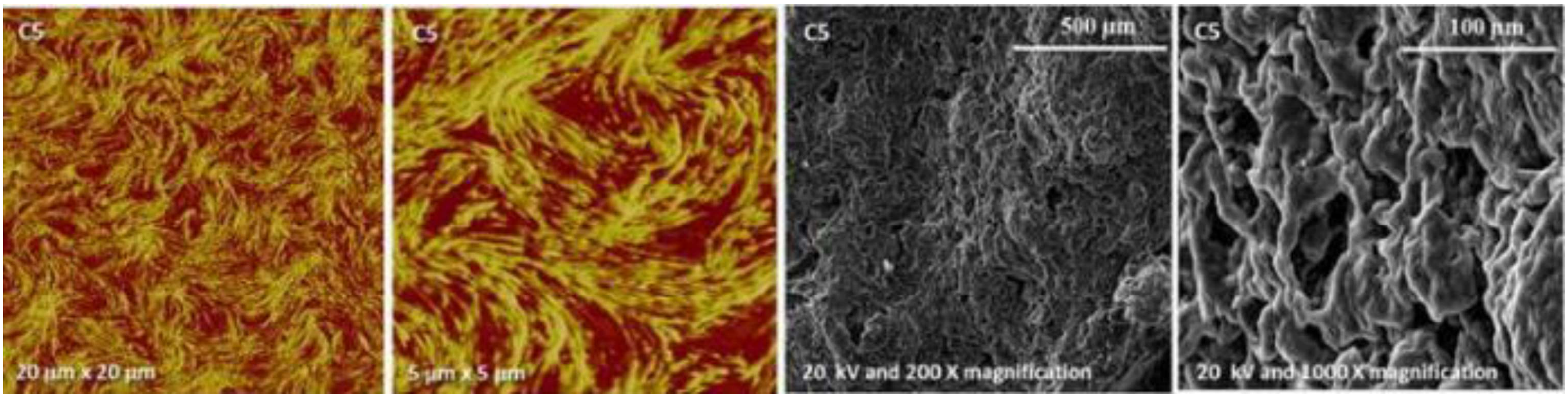



Mechanisms of emission of secondary electrons, backscattered electrons, and characteristic X-rays from atoms of the sample

https://en.wikipedia.org/wiki/Scanning_electron_microscope

# SEM vs AFM

| | SEM | AFM |
|---|---|---|
| *Imaging Advantage* | High Depth of Field | High Contrast |
| *Dimensions* | 2-D | 3-D |
| *Measurements* | Chemical Composition | Physical Properties |
| *Environment* | Vacuum | Vacuum, Air, Liquid |

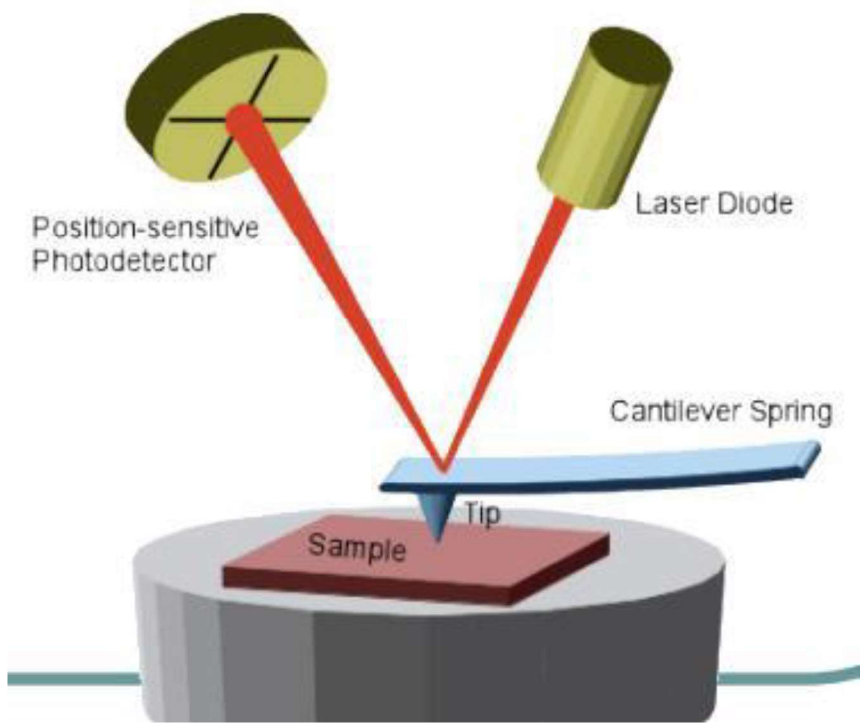


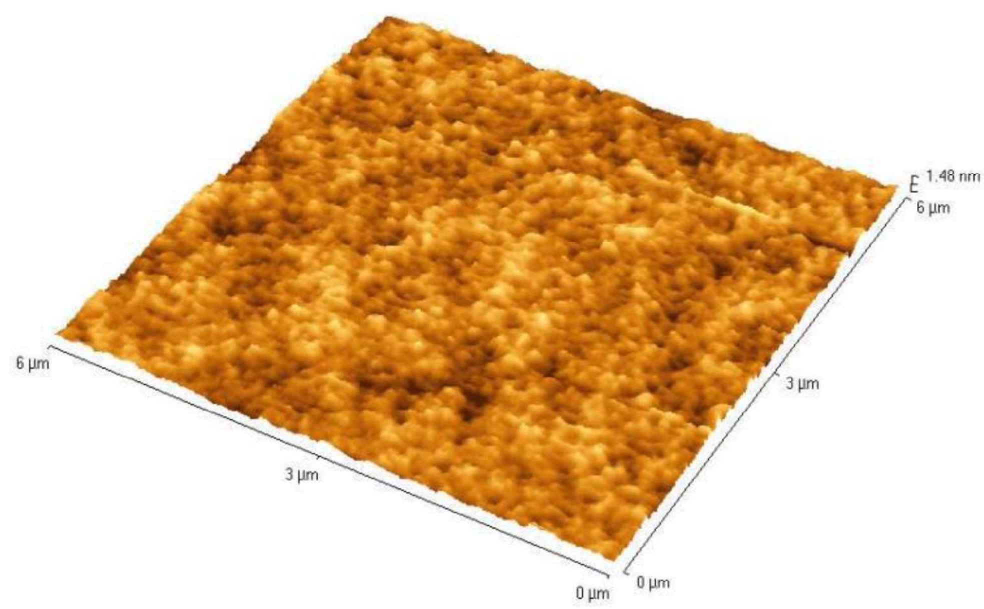

AFM (left) and SEM (right) micrograph corresponding to lithium complex (C5) and lithium–calcium complex soap (C6) greases

Credits-Tribology Letters, 2016, Volume 63, Number 2, Page 1

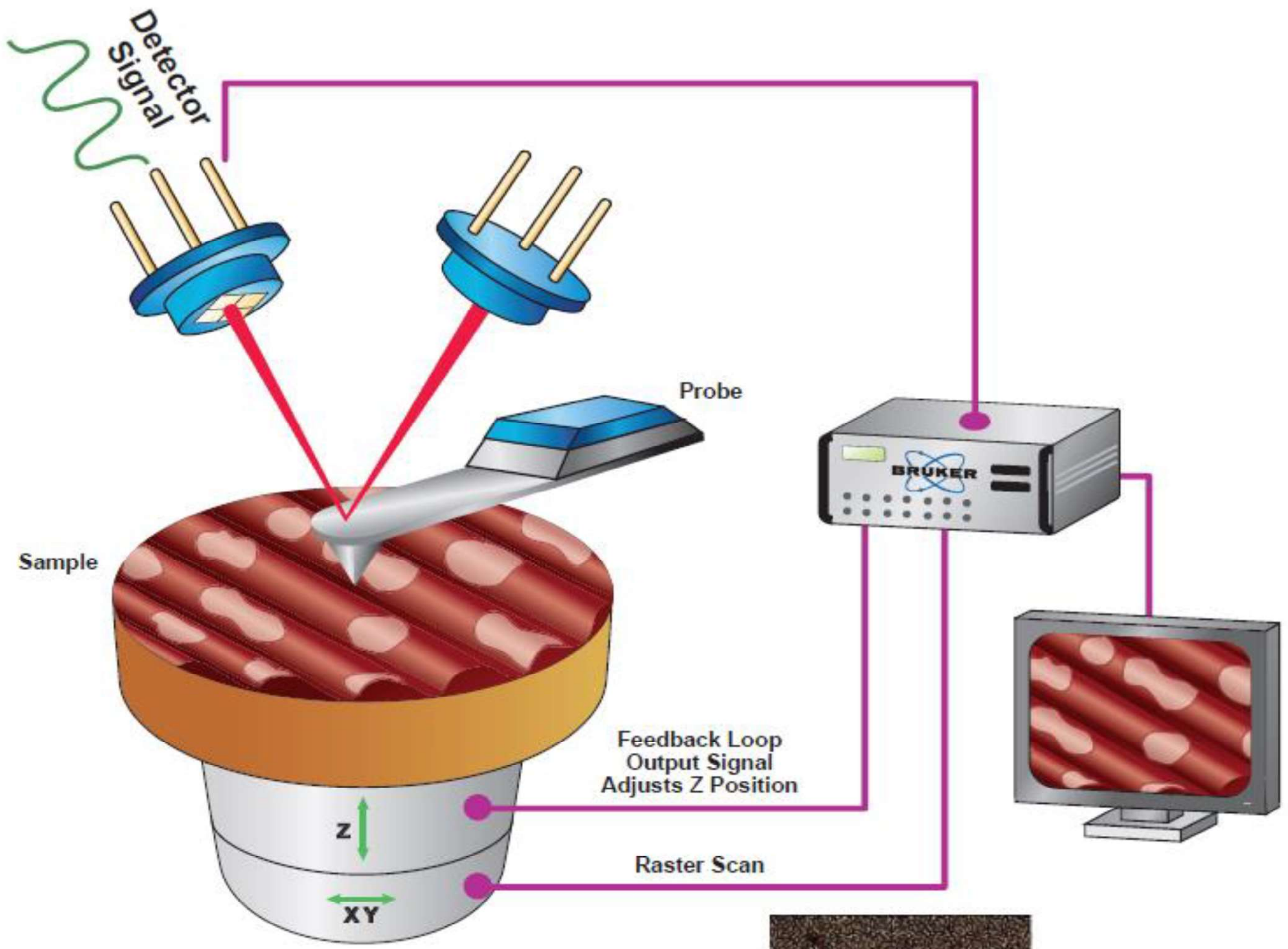
# AFM(Atomic Force Microscopy)

- **AFM**
  - Belongs to the family of Scanning Probe Microscopy
  - AFM senses inter atomic forces that occur between a probe tip & substrate
  - It has very high resolution and can be used in topographical imaging of samples such as DNA molecules, protein adsorption



# Working principle of AFM




Protein molecules in liquid before and after force pulling with AFM.

https://www.bruker.com/products/surface-and-dimensional-analysis/atomic-force-microscopes/campaigns/afm-microscopes.html

# Notes

# Notes