Thin film analysis cookbook

Pavel Souček

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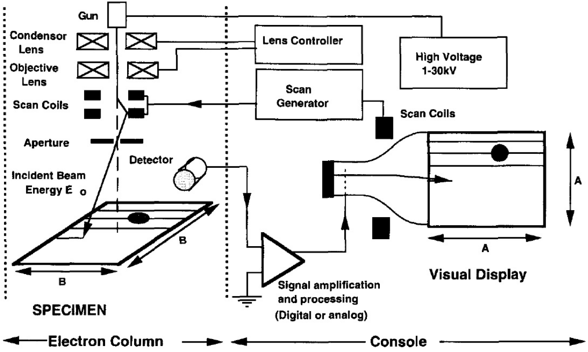
Scanning Electron Microscopy

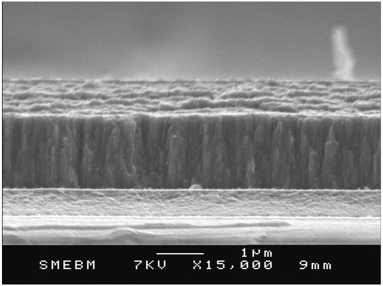
- surface texture, chemical composition and crystal structure analysis
- high energy electrons are impacting the measured sample, where they lose energy and result in several types of signal produced by the sample
- secondary electrons (SE), back-scattered electrons (BSE), characteristic X-rays and visible spectrum radiation (cathodoluminescence)

SEM Microscope



Secondary electrons

- electrons emitted from the irradiated sample, energy 0 to 50 eV
- because of their low energy, they can be emitted from only a thin layer under the surface (3 – 10 nm)
- detection of secondary electrons isn't difficult, they contain information of the sample topography
- resolution can be up to 1 nm



Calotest

- hardened steel ball is turned in order to grind the measured film
- since many films are harder than the steel ball used, additional diamond suspension is placed between the film and the steel ball
- once the film has been abraded off, the projection of surface can be evaluated

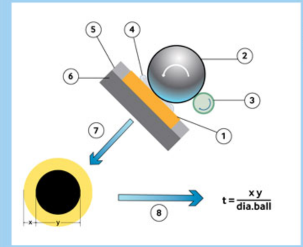


Fig. 10: The Calo test is a simple method to determine the coating thickness.

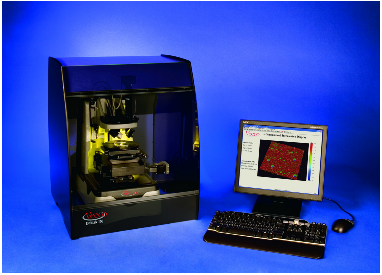
1. Test sample
2. Steel ball
3. Drive shaft
4. Diamond paste
5. Clamp
6. Holder
7. The grinding creates a crater through the coating and in the substrate
8. Imaging software uses a simple formula to calculate the coating thickness

Calotest II

Profilometry

- diamond stylus is moved vertically in contact with a sample and then moved laterally across the sample for a specified distance and specified contact force
- typical profilometer can measure small vertical features ranging in height from 10 nm to 1 mm
- height position of the diamond stylus generates an analog signal which is converted into a digital signal stored, analyzed and displayed
- radius of diamond stylus ranges from 20 nm to 25 μm, and the horizontal resolution is controlled by the scan speed and data signal sampling rate

Profilometer



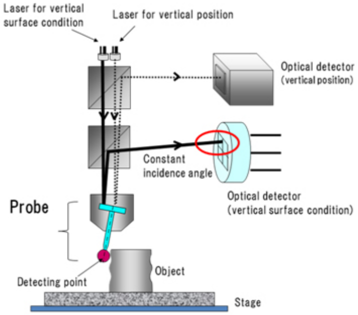
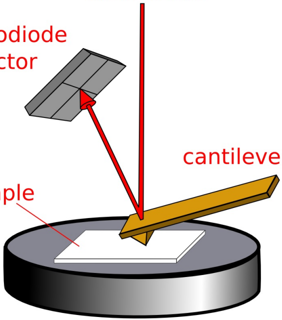


Fig.1 Optical System in Ultrahigh Accurate 3D Profilometer

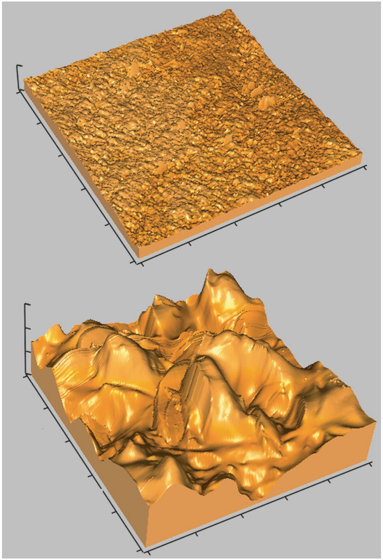
Atomic Force Microscopy

- high-resolution type of scanning probe microscopy
- information is gathered by "feeling" the surface with a mechanical probe



AFM

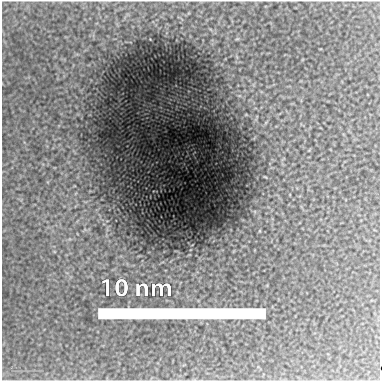
- 2 imaging modes — contact and non-contact
- provides a three-dimensional surface profile, samples viewed by AFM do not require any special treatments, AFM can provide higher resolution than SEM
- much slower than SEM, it cannot normally measure steep walls or overhangs



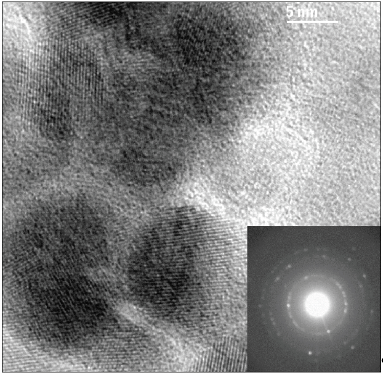
Transmission Electron Microscopy

- structure, chemical composition and crystal structure analysis
- electron beam is interacting with a very thin sample
- contrast is governed by on one hand (for lower magnifications) absorption of electrons in the sample, on the other hand (for high magnifications) by complex interactions of electron matter waves and the sample
- two imaging modes - imaging and diffraction

TEM Images



[a]

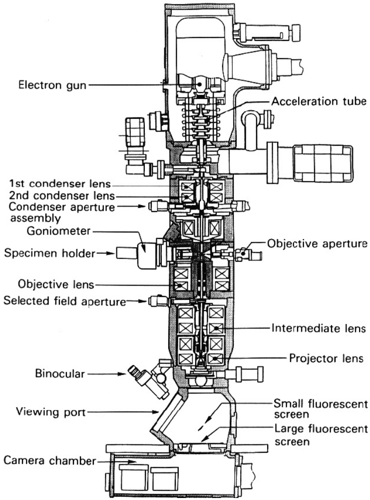


[a]

[a]http://www.temwindows.com/default.asp

[a]http://spie.org/x17488.xml?ArticleID=x17488

TEM Microscope



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[1] FULTZ, B., HOWE, J.M. *Transmission Electron Microscopy and Difractometry of Materials*

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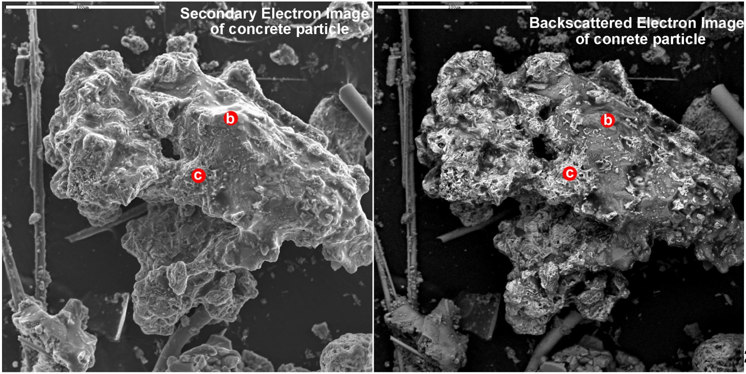
SEM on back-scattered electrons

- electrons that leave the sample with energy from 50 eV to the energy of incoming beam
- electrons that were scattered at angles nearing 180°
- backscattered electrons yield strongly depends on the proton number pand so BSE imaging is suitable for viewing image of chemical composition
- detection of BSEs is relatively difficult due to their high energy
- detector has to be precisely positioned and has to be large enough

SE vs. BSE

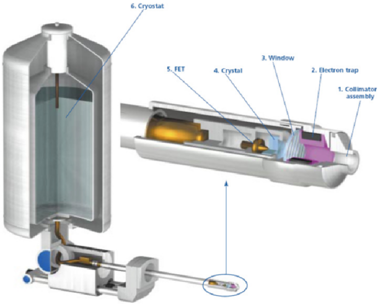


[2]

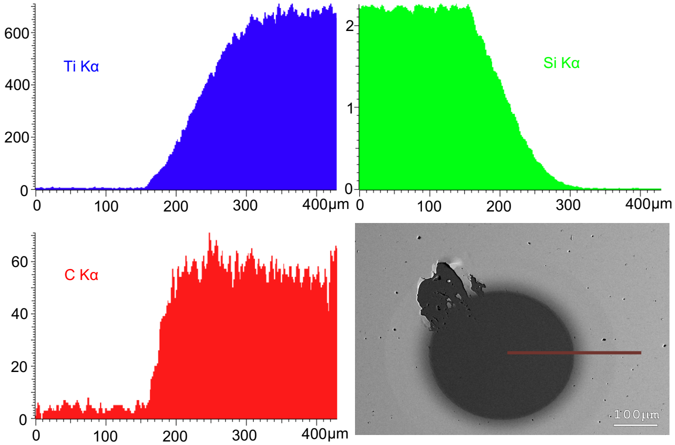
[2] http://pubs.er.usgs.gov/

Energy-dispersive X-ray Spectroscopy

- qualitative and quantitative analysis of chemical composition
- based on detection of characteristic X-rays
- characteristic X-rays have high penetration depth, so this method is a volume measurement
- elements lighter than beryllium cannot be detected
- fast method, part of SEM microscopes, no need for difficult sample preparation, not very precise

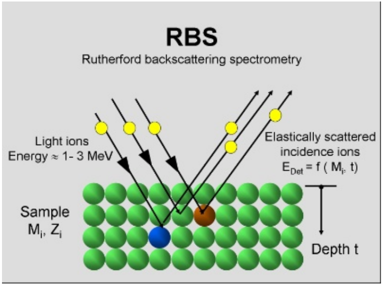


EDX Measurement

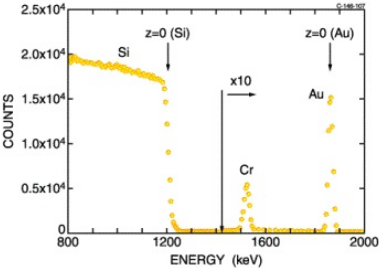


Rutherford backscattering spectrometry

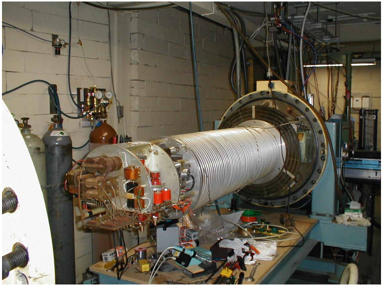
- light ions (protons, alpha particles) at high energy (1 – 3 MeV) are impacting atoms in measured sample
- part of them is backscattered at angle nearing 180°
- energy distribution of these electrons is measured
- can perform depth analysis of chemical composition
- can't measure hydrogen



RBS

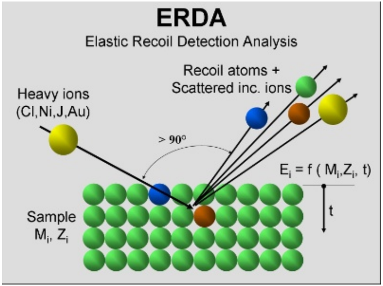


2.5x10^4
2.0x10^4
1.5x10^4
1.0x10^4
0.5x10^4
0
COUNTS
z=0 (Si)
z=0 (Au)
Si
x10
Au
Cr
800
1200
1600
2000
ENERGY (keV)



Elastic recoil detection analysis

- similar method to RBS
- only difference is that heavier ions are used and energy distribution of recoiled atoms is measured



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Scratch test

- common method of testing the adhesion of coatings to substrates
- diamond tip is scratched across the coated surface of a substrate at a constant velocity whilst a load is applied with a constant loading rate
- load on the diamond causes stresses to be increased at the interface between the coating and the substrate that can result in delamination or chipping of the coating to occur

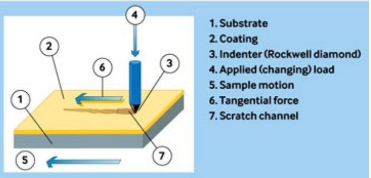
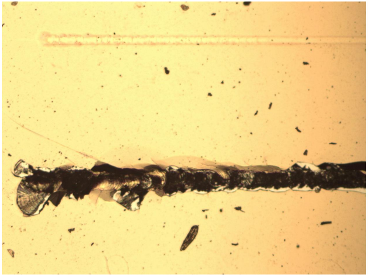
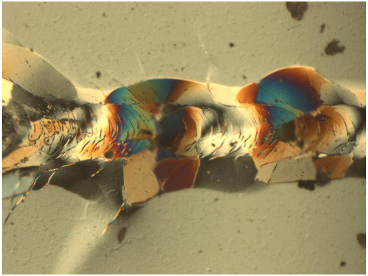


Fig. 9: Schematic representation of a surface scratch test with progressive load.

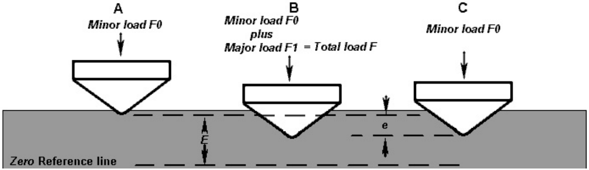
Scratch test



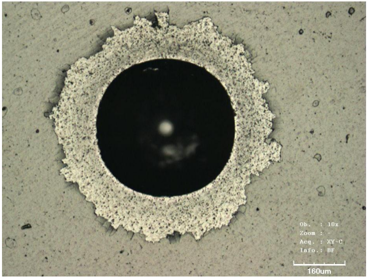


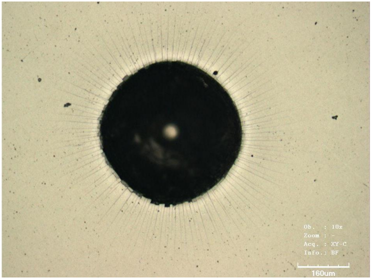
Rockwell test

- method consists of indenting the test material with a diamond cone or hardened steel ball indenter
- permanent increase in depth of penetration, resulting from the application and removal of the additional major load is used to calculate the Rockwell hardness number
- also adhesion of the coating can be deduced from the shape of the coating near the indentation crater



Rockwell test

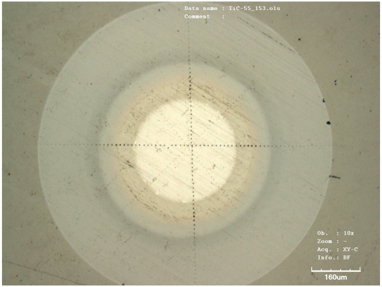


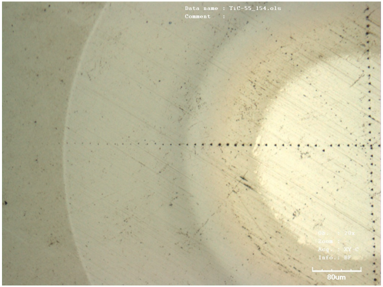


Nanoindentation

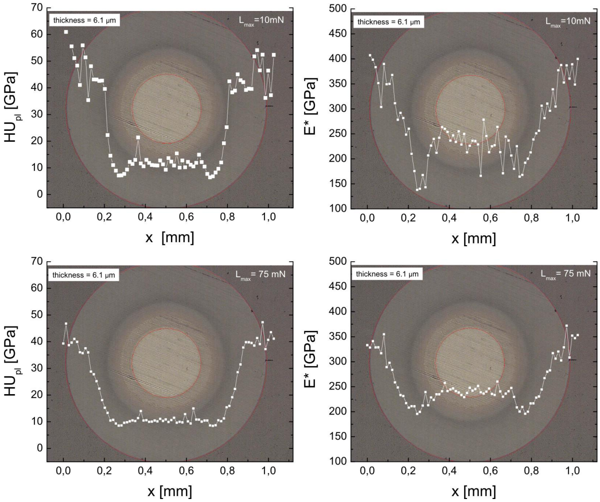
- mechanical properties analysis – mostly hardness and Young's modulus
- uses *depth sensing indentation* method – doesen't use the measurement of area of the residual indent, but measures the force and depth of the indent throughout the whole measurement

Nanoindentation





Nanoindentation



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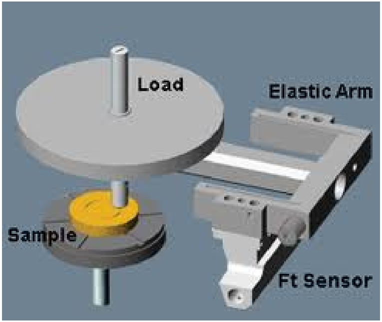
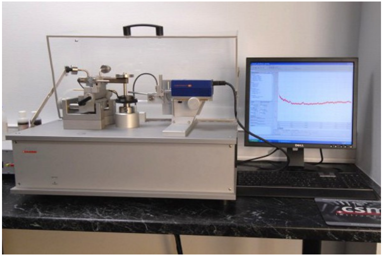
Tribotest

- tribological quantities, such as coefficient of friction, friction force, and wear volume, between two surfaces in contact are measured
- most used types of tribometers — Pin on disc and Bouncing ball





Thank you for attention

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