cutting edge developments

# Material testing at nanometer scale

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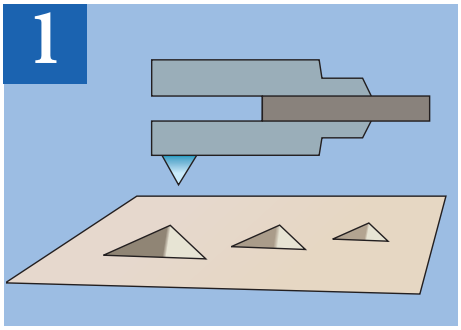
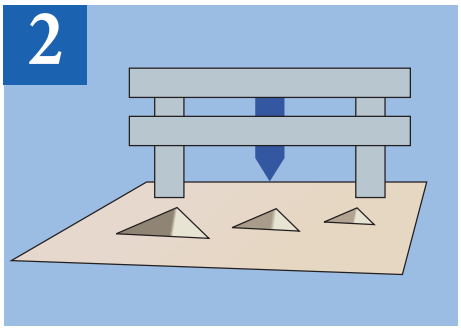


## NTEGRA-based Sclerometry: hard and very hard

· Destructive (Medium loads):
- Nanoindentation
- Nanoscratching
- Nanowearing

· Non-destructive

2

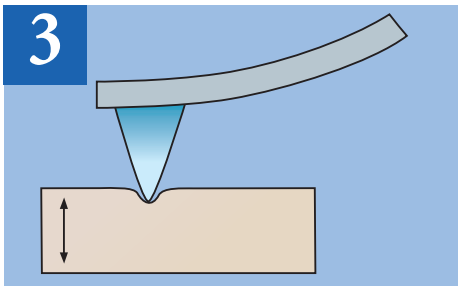


## NTEGRA+Hysitron TriboScope: hard and very hard

· Destructive (Heavy loads):
- Nanoindentation
- Nanoscratching
- Nanowearing

· Non-destructive

3



## Atomic Force Acoustic Microscopy: hard and soft

· Non-destructive (Dynamic testing only)

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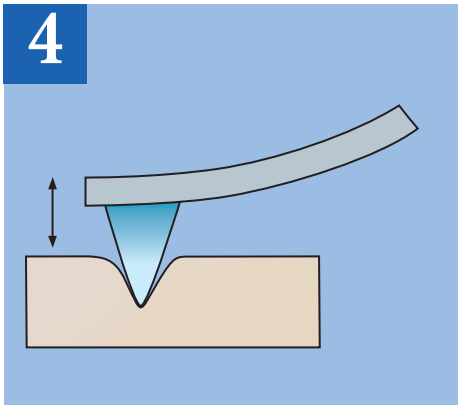
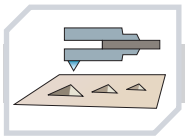


## Atomic Force Spectroscopy: soft and very soft

· Destructive (Light loads):
- Nanoindentation
- Nanoscratching
- Nanowearing

· Non-destructive (Study of living objects in natural environment)



# NTEGRA-based Sclerometry

## Sclerometry: Nanoindentation and nanoscratching

During nanoindentation the surface of a sample is displaced as pressure is applied by the tip of a probe. Analysis of the applied "Force-Displacement" dependence provides data on the hardness of a sample at a given point (fig.1). One may analyze curves as well as topography of the images, by scanning the indented sample (fig.2).

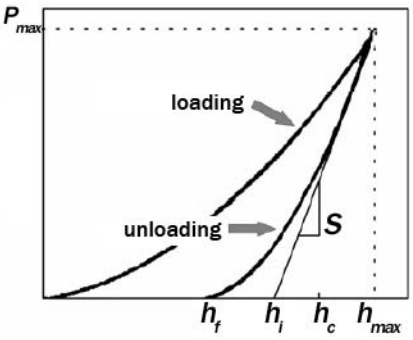



*Fig. 1* **Loading-unloading curves. h - displacement, P - load, S - contact stiffness.**

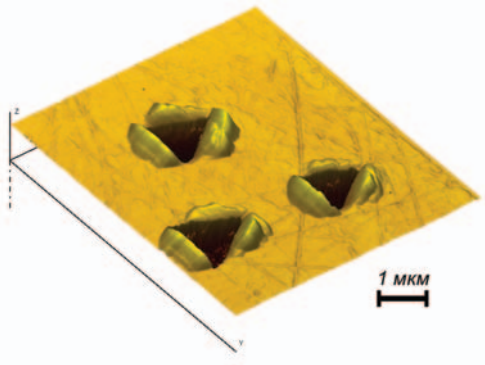
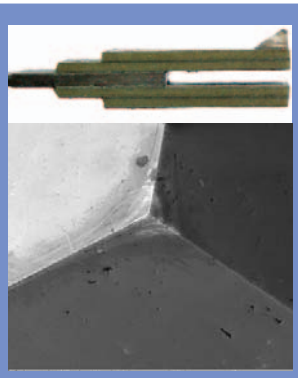
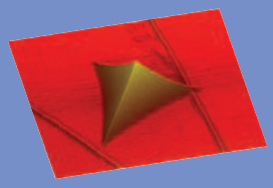



*Fig. 2* **Sapphire surface with indents. Scan size: 5 x 5 µm**



**Piezoresonance probe with a mounted tip & FIB image of the tip apex.**



**Inverted image of the indent in glass (shows sharpness of the probe tip)**

Unlike the cantilever of common silicon AFM probes, the piezoceramic console of the probe for the **NTEGRA**-based Sclerometry has a greater hardness ($10^4$-$10^5$ N/m). This makes the degree of force, applied to a sample much greater than in a usual AFM systems.

Nanoscratching is a technique based on making scratches on the sample surface and measuring their parameters: depth and especially width. This gives an opportunity to evaluate the hardness of materials quantitatively (fig.3,4). In some cases the results obtained can provide more information than that obtained by nanoindentation, because the width of a scratch, as the result of the elastic recovery, modifies less than its depth.

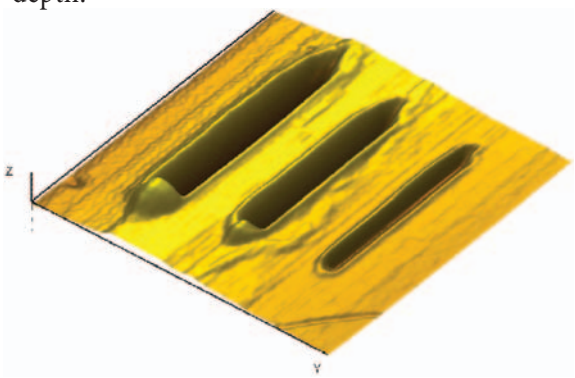


*Fig. 3* **Three scratches of different depths, made in fused quartz. Image size 4 x 4 µm.**

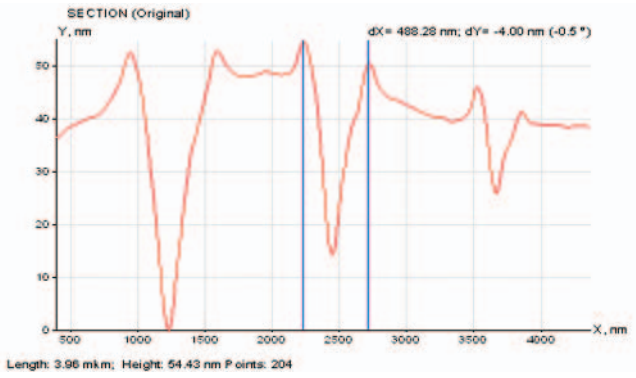



*Fig. 4* **The curve shows depth & width of the scratches in fused quartz.**

## Sclerometry: Dynamic non-destructive testing

A probe is attached to stiff but flexible cantilever, thus amplitude and frequency of forced oscillations of the probe can be used for topography imaging and testing elastic properties of materials (fig. 5). In particular, this method provides a quantitative value of Young's modulus at each point of the scanned sample.

Owing to the high resonance frequency of the piezoceramic probe, it is possible to map hardness and elasticity properties much faster than using standard indentation techniques with a high load (e. g. **NTEGRA**+Hysitron TriboScope). On the other hand, unlike SPMs with conventional silicon probes, an **NTEGRA**-based Sclerometry allows testing very hard materials and films (fig. 6).

The design of the probe used in **NTEGRA**-based Sclerometry allows the use of a variety of prefabricated tips: diamond Berkovich tips, semiconductor diamond tips, etc.

The investigation of thin film adhesion to the substrate can be considered an example of nanotribology applications. Nanotribology involves the scratching of film with an augmentative force and determining the load of film detachment or wear-out (fig.7).

### Why is it important to merge SPM with nanoindentation?

**Because it is possible to make an SPM-image using the same probe, which is essential for:**

1. Finding indents, made with light loading, which are very small and hard to see with usual optics.
2. Accurate quantitative measuring of indentation and scratch parameters and finding defects of indents (pile-ups, etc.).
3. Making sure that the needed object is measured in case it has small size and is not seen in optics, e.g. nanoparticles, nanoscratches on films, etc.

Scanning a modified sample with the same tip is precise since an indent is always wider than the tip due to elastic recovery

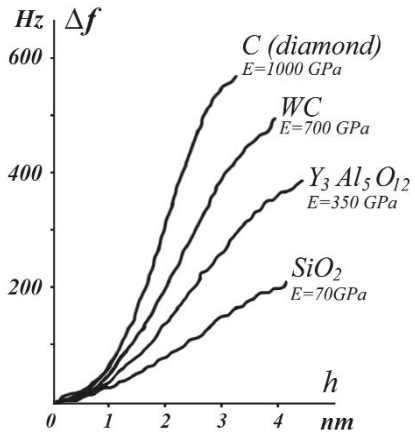



*Fig. 5* **Frequency alteration is recorded as a function of the probe position. Slope of a curve Δf characterizes Young's modulus of a sample.**

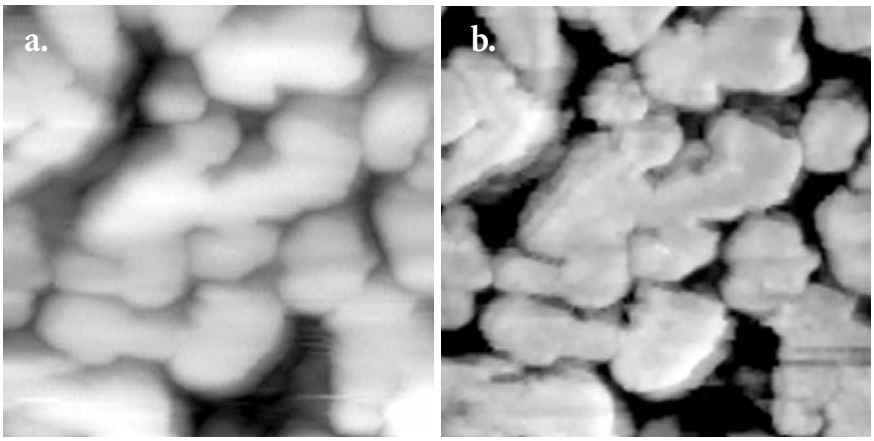



*Fig. 6* **The image of composite (metal + fullerite C60). Average grain size ~0.4–0.8 μm. Image size: 3.5 x 3.5 μm: a) topography of surface; b) Young's modulus map.**

**NTEGRA**-based Sclerometry makes it possible to work with various types of films within a wide range of thicknesses (from several nanometers up to several microns) and hardnesses.

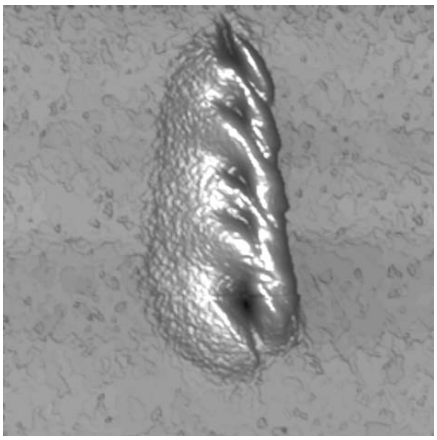


*Fig. 7* **Film of 45°-oriented nanotubes with a trace of a scratch, made perpendicular to the nanotubes slope. Image size: 5.9 x 5.9 µm**

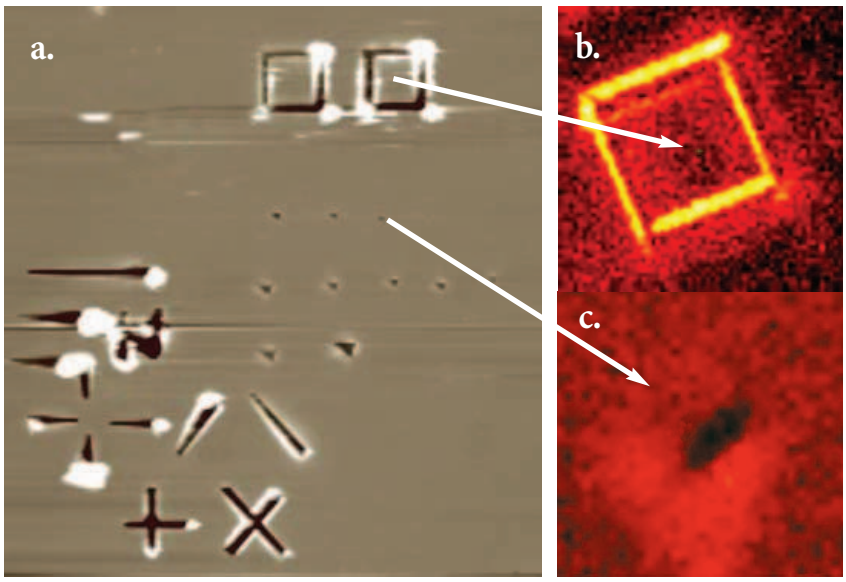
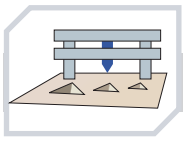


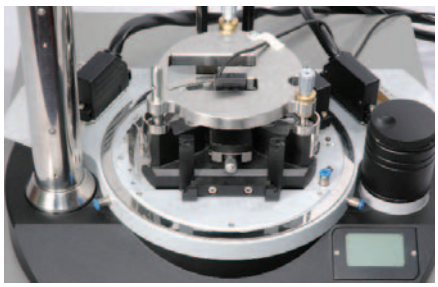

*Fig. 8* **Indents and scratches on the surface of GaAs (a) and images of stress obtained by mapping of Raman spectra shift (b, c). Image sizes: a). 80 x 100 µm; b). 25 x 25 µm; c). 6 x 6 µm.**

**NTEGRA** platform has been specially designed to integrate different techniques in order to give ultimately new and unique methods of material testing. For example, confocal Raman microscopy can be applied to visualize the stress after nanoindentation and nanoscratching (fig.8). Surface modification and examination can be both performed by the same instrument.



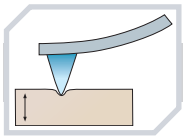
## NTEGRA + Hysitron TriboScope

Any **NTEGRA**-based system can be equipped with Hysitron TriboScope nanoindentation system. It provides high loads (up to 1N) and can be mounted with various commercial probes as well as **NTEGRA**-based Sclerometry. Non-destructive dynamic testing and Young's modulus mapping can be performed as well. All modes of sclerometry – nanoindentation, nanoscratching and nanotribology – can be applied in tests with **NTEGRA** + Hysitron TriboScope integration.



**NTEGRA+Hysitron TriboScope nanoindentation system**

Hysitron TriboScope is a registered trademark of Hysitron Inc.



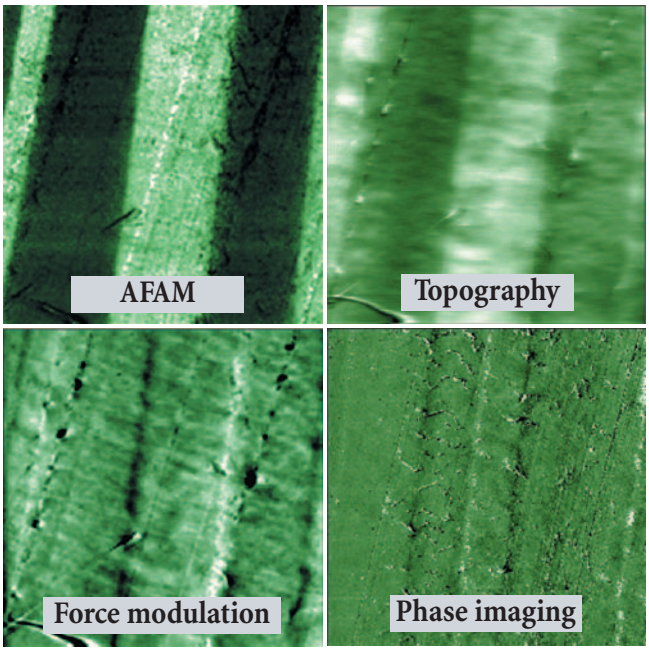
## Atomic Force Acoustic Microscopy (AFAM)

The main idea behind AFAM is the registration of AFM probe oscillations, when a cantilever tip is in contact with an oscillating sample. Simultaneously with acoustic imaging it forms topography as it is done by contact AFM techniques.

Mapping of the Young's modulus does not cause sample destruction (neither indentations nor scratches are left on the surface).

AFAM provides sharp contrast of imaging for hard & soft samples, whereas AFM techniques (e. g. phase imaging and force modulation) support contrast only for relatively soft materials (fig.9,11).

In some cases inner non-homogeneities can be visualized within the sample volume. It is possible because the whole specimen is "shook" with acoustic frequencies and the entire volume is involved in generation of the probe oscillations (fig.10).



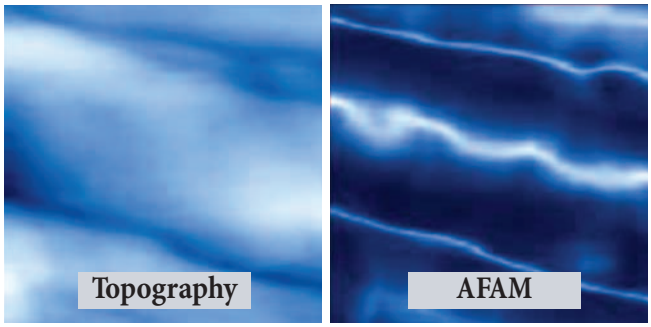




*Fig. 10* **HDD surface. Topography (A) & AFAM (B) A bright line in the middle of the AFAM image marks an inner crack, which is not seen on the topography image. Image size: 0.8x0.8 µm.**

*Fig. 9* **Stripes of low & high density polyethylene with different elasticity. Scan size: 47x47 µm.**

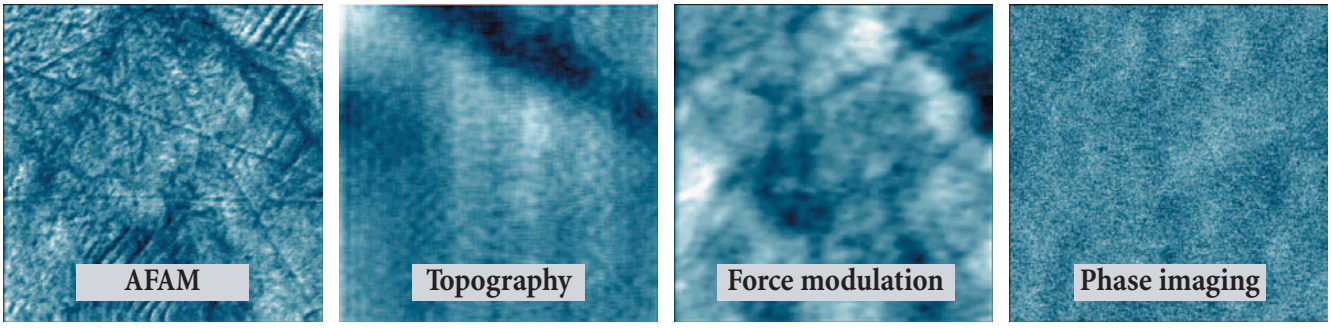
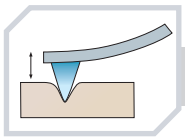



*Fig. 11* **Polished PZT sample. It is seen that the best contrast is obtained with AFAM. Scan size: 4x4 µm.**



## Atomic Force Spectroscopy

When pushing a surface by conventional AFM probe, one may expect a linear dependence of cantilever bending and the applied force. This could be the case, if the sample was absolutely hard and it was not displaced by the probe. Practically, on soft samples the force-distance curve is non-linear. Its parameters can be used to calculate to what degree the surface is displaced, when a particular force is applied. In turn, this is the path to quantitative estimations of Young's modulus (fig.12).

This approach is successful on soft and very soft samples, because the spring constant of conventional AFM cantilevers is relatively small (usually not more than $10^2$ N/m). For studying such subtle objects as living cells and natural cell organelles (fig.13),the cantilever must be as soft as possible to prevent substantial sample deformation. Typical values of the spring constant in this case are $10^{-2}$–$10^{-1}$ N/m.

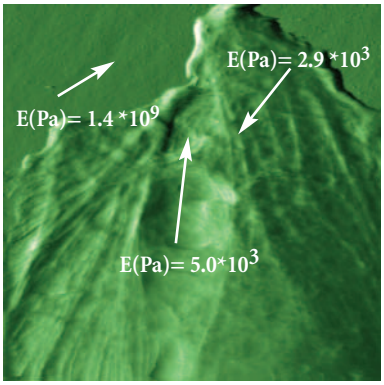



*Fig. 13* **Young's modulus as estimated on a living cell surface and on a Petri dish bottom. One point of the cell surface is almost twice as hard as another one, while the Petri dish is six orders of magnitude harder.**
**Scan size: 25x25 µm**

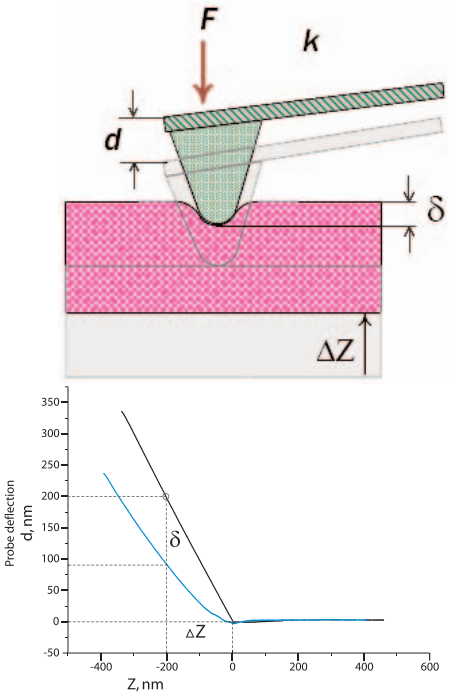



*Fig. 12* **Force curve parameters that are used for quantitative estimation of elastic properties of material.**
**F - load; d - cantilever displacement; k - cantilever spring constant; δ - indentation; ΔZ - sample displacement.**

## Comparison table of the techniques

| | Hardness range | Destruction | Hardness of samples | Maximum loads |
|---|---|---|---|---|
| **NTEGRA**-based Sclerometry | 0.1 - 100 GPA | Destructive & non-destructive | Hard & very hard | 200 mN |
| **NTEGRA** + Hysitron TriboScope | 0.1 - 100 GPA | Destructive & non-destructive | Hard & very hard | 1 N |
| AFAM Option | 10 kPa - 10 GPa | Nondestructive | Hard & soft | — |
| AFM | 1 kPa - 1 GPa | Destructive & non-destructive | Soft & very soft | 2 mN |