MECHANICAL TESTING TECHNIQUES: FROM MICRO- TO NANOMETER SCALES AS INVESTIGATED WITH SCANNING NANO-HARDNESS TESTER “NANOSCAN-3D”

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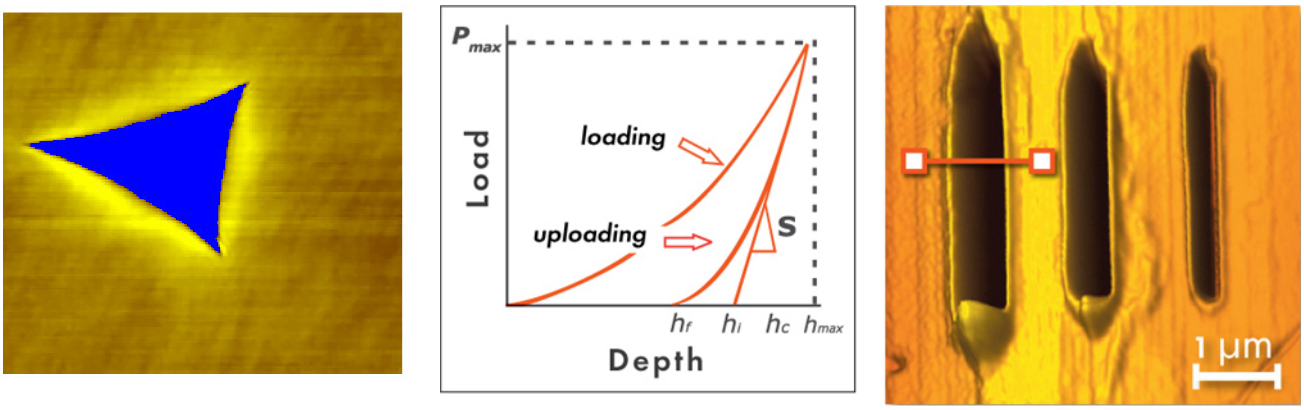
Hardness is always referenced as an empirical value which depends on many details involved in the measuring process, like equipment design, technique, indentor type, measuring conditions, range of applied loads, etc. When a hardness value is mentioned, one should always keep in mind that it is not a physical constant, but some experimental value obtained with known method and depending not only on the material, but on conditions and methods as well.

At the micrometer scale the most trustable method so far is a microhardness test, in which hardness value is determined as a relation of applied load to the projected area of the residual imprint (Fig.1a). Two competing techniques for measuring hardness at the nanometer scale are instrumented nanoindentation (Fig.1b) and scratch hardness test (Fig.1c). Due to easy and fast obtaining of a numerical result, nanoindentation has become the most common way to measure hardness, which is studied carefully in many aspects. This technique underlies an international standard on measuring hardness ISO 14577.



a) Micro-hardness test | b) Nanoindentation | c) Scratch hardness test

Fig.1 Hardness measurement techniques at the micrometer and nanometer scales.

At the same time, the scratch hardness (or sclerometry) testing is used more rarely. However, this technique has several apparent advantages, especially when applied at the nanometer scale. Some of them are less influence of roughness, less effect of elastic recovery of scratch width compared to indentation imprint perimeter, the effect of averaging along the scratch length, variation of attach angle if scratching face forward or edge forward, the possibility of variable load scratch test, easier pile-ups analysis, and the possibility to study the anisotropy of hardness.

Scanning nano-hardness testers of NanoScan series (Fig.2) are intended for investigation of the surface topography, elastic modulus mapping and for measuring the mechanical properties (including hardness and elastic modulus) of bulk materials and thin films on a submicron and nanometer scale. NanoScan has been developed on principles of scanning probe microscopy (SPM). The main characteristic feature of NanoScan is the use of piezoceramic probe sensor having high bending stiffness of the cantilever (~$2 \cdot 10^4$ N/m). High bending stiffness of the cantilever permits to go through the viscous contamination layer when working in an ambient environment until the contact with rigid surface and to make a modification of surface (indentation and scratching). The probe design allows using diamond indentors of different types.

NanoScan allows performing the following measurements:

- Obtaining the surface topography image as a result of surface scanning.

• Obtaining the qualitative mechanical contrast map of the surface simultaneously with the surface topography scanning.

• Performing a micro- and nanoindentation and scratch hardness tests of the material with the subsequent scanning of the indentation area.

• Measuring the numerical value of elastic modulus with submicron spatial resolution using the method of force spectroscopy.

All the measurements are made in the open air without any special vacuum or thermal sample preparation.

For investigation of mechanical testing consistency the hardness values measured on different depth scales (from microscopic down to nanometer), with different techniques (microhardness test, nanoindentation and scratch hardness test) on the materials of different structure (amorphous, metallic, covalent crystalline, polycrystalline) were compared to each other. The experiment conditions for all measurements were the same, i.e. one and the same equipment, with the same diamond indentor was used. The loading velocity and the hold time at the peak load for all methods was also the same.

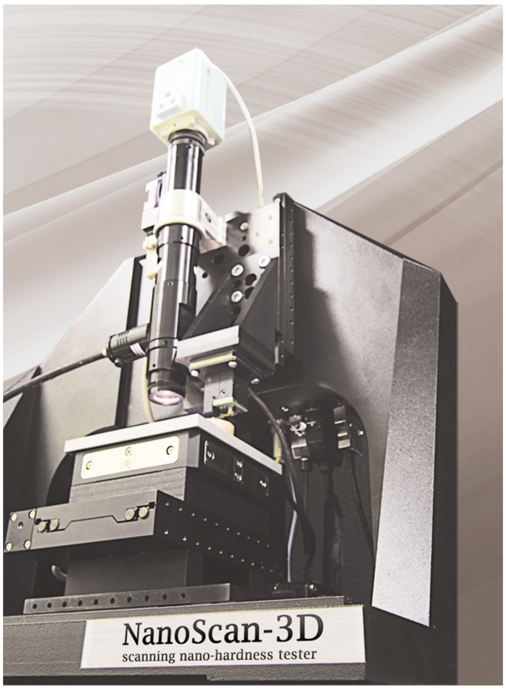


Fig.2 Scanning nano-hardness tester NanoScan.

During the microhardness test the imaging was performed in SPM mode immediately after the unloading. The area of the imprint was measured on the image, as shown on Fig.1a.

Some critical aspects of both scratch hardness, micro- and nanoindentation applications were considered for different test scales. It is shown that one of the most commonly faced source of error in micro-hardness test and nanoindentation is the presence of so-called ‘pile-ups’ – the plastic raise of the material along the perimeter of the imprint. The topography imaging is a powerful tool for observation and analysis of this artifact. The size of pile-up affects the true contact area that indentor had during the loading process and subsequently the measured hardness value. This is especially important for metallic and crystalline materials which tend for form pile-ups after indentation. For example for titanium the ratio of projected contact area with/without counting for pile-up can reach up to 1,5 (Fig.3). This means up to 50% potential error during hardness evaluation. The same problem exists for nanoindentation method which does not imply residual indentation imprint observation. Some suggestions are made on the correction to the area function used in nanoindentation technique based on the data obtained after imprint analysis.

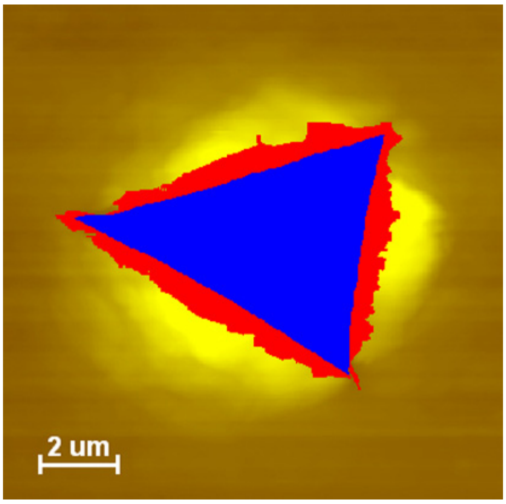


Fig.3 The topography of the residual imprint in titanium. Red color shows pile-up projected area, blue color shows imprint projected area.

It is estimated that the width of scratches with depth as small as 1-2 nm can be measured reliably and used in scratch hardness measurements. The mean-square deviation of width for such scratches on sapphire was estimated 15 %. It is important to note that average roughness of the sapphire surface was 0.45 nm, i.e. 20-30 % of scratch depth. This makes scratching a very promising technique for nanoscaled measurements, since such results are hardly achievable with nanoindentation.

Microhardness test shows significant increase in hardness at relatively small depths for all three materials. With the increasing of the indentation depth, the hardness values tend to its reference value. This is the expected behavior, which is usually associated with the indentation size effect. However, in present study the effect of elastic recovery of the imprint is most probable reason for such increase.

Microhardness technique is evidently not applicable at nanometer scale, first of all, due to significant effect of imprint elastic recovery. Scratch hardness technique shows good results of hardness measurements for the whole depths range and samples involved in the experiment. Imaging of the surface before test and imaging of the imprints after measurements is extremely important for nanoscaled mechanical tests, i.e. one should be aware of what the material is really doing during the deformation.

The variable load scratch test was applied for hardness evaluation of different materials. The indentor shape function as a dependency of load on scratch width was calculated over the reference scratch before the measurements. It is estimated that shape function depends on the loading rate during the scratch: the higher the loading rate the wider the scratch.

It is shown that sclerometry test accompanied by the surface topography imaging option is a powerful method of mechanical properties characterization. Variable-load scratch test is a very promising way to estimate hardness as a function of indentor penetration into the sample surface.

However, scratch test is very sensitive to the loading rate and attack angle of the indentor. In this regard, constant-load and variable-load scratches cannot be exactly related to each other in terms of obtaining the numerical hardness value. When applied to the thin films analysis the variable scratch tests allows estimation of several mechanical parameters in single test (Fig.4).

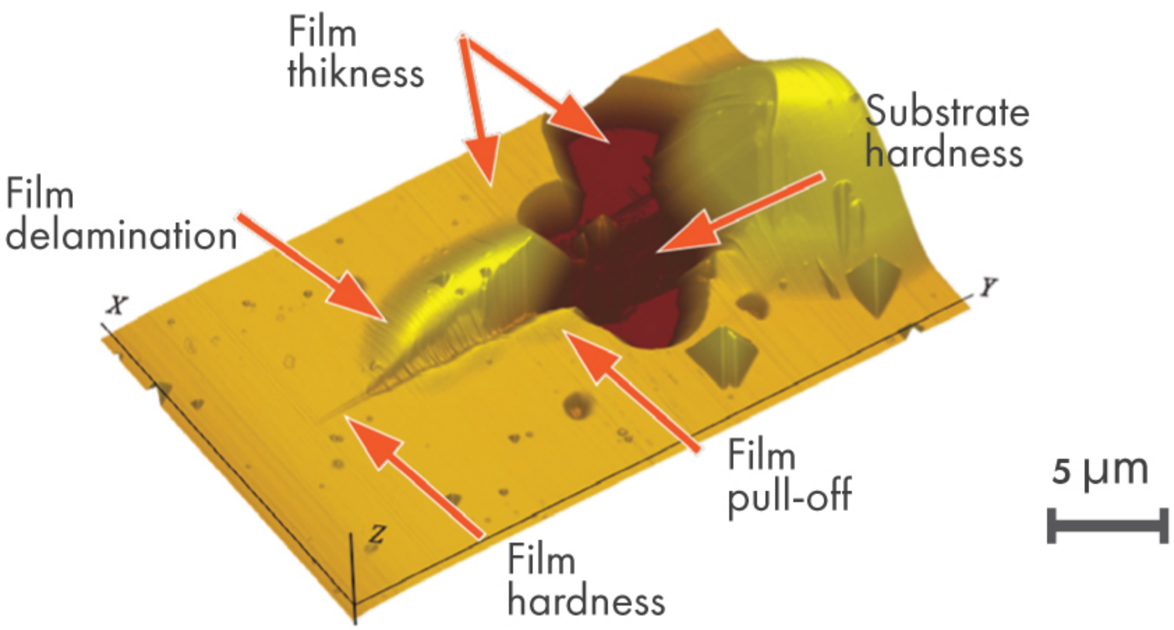


Fig.4 Variable load scratch test on the thin DLC film.

Combination of different mechanical analysis techniques in single testing equipment makes material properties investigation much more informative.