4.3.17. 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. Two competing techniques for measuring hardness at the nanometer scale are instrumented nanoindentation and scratch hardness test. 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.

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 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 ($\sim 2 \cdot 10^4$ 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 type.

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.

Some critical aspects of both scratch hardness, micro- and nanoindentation applications were considered for different test scales. The variable load scratch test was applied for hardness evaluation of different materials. The indentor shape function was P(w) calculated over the 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. 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.