NenoVision

Application note

In-situ nanoindentation module for AFM-in-SEM LiteScope™

Nanoindentation has become a standard quantitative method that enables to measure mechanical properties such as modulus and hardness using very small amounts of materials. A nanoindentation test is performed by applying a load to a sample in a highly controlled manner with a geometrically well-defined probe. This versatile technique only requires simple sample preparation and can measure properties of various materials ranging from hard superalloys to soft biomaterials.

Nanoindenter module for LiteScope enables quantitative material analysis by SEM-assisted indentation.

The Alemnis nanoindenter module is an optional accessory LiteScope, an atomic force microscope (AFM) designed for swift integration into scanning electron microscopes (SEM).

The resulting combination of three complementary techniques enables micromechanical experiments to be performed while observing the specimen with a superb SEM magnification and analysing the indented specimen with sub-nanometre resolution using a LiteScope. This unique solution is designed for maximum versatility and enables a wide range of novel and complex applications.

Figure 1: Photo of Alemnis nanoindenter module for LiteScope.

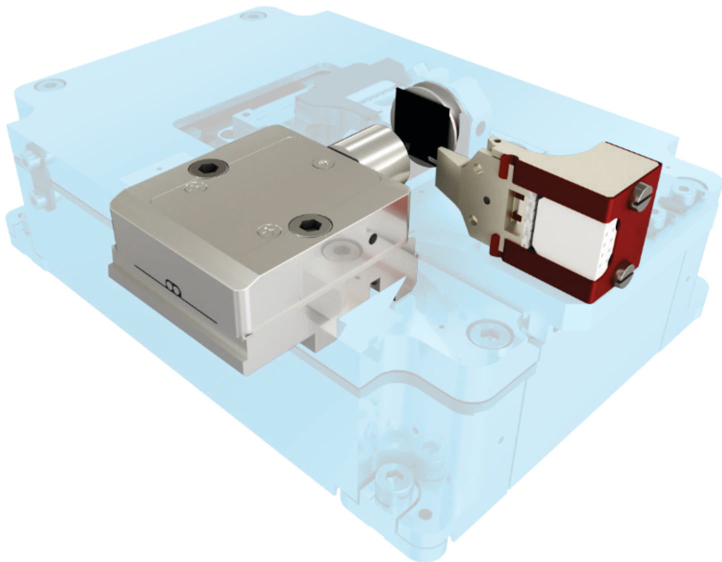

Figure 2: Schema of the nanoindenter module mounted to AFM LiteScope depicts the inverted sample vs AFM probe configuration

Key advantages of the 3in1 solution (SEM + AFM + nanoindenter):

Precision:

- precise localization of nanostructures
- colocalization of related SEM techniques (EBSD, EDX etc.)

Accuracy:

- pre-indentation area survey (roughness, tilt, cracks, impurities, etc.)
- observation of indentation after-effects (pile up, cracking, etc.)

Efficiency:

- fast nanoindentation and AFM targeting using SEM
- easily repeatable localization of the same area

Fast nanoindentation and AFM targeting using SEM

The advantages of merging nanoindenter, AFM and SEM techniques are demonstrated on M3 class 2 high-speed tool steel. The synergy of the three devices enabled facile sample phase identification, precise indentation targeting, and topographical analysis – all in one measurement. Figure 3 shows the results of the etched steel sample analysis. The SEM analysis identified a fine structure of separate phases (grains of MnS and M_6C carbide in a martensitic matrix). Subsequent AFM imaging allowed to measure their roughness. All phases were nanoindented with the standard Berkovich tip using an identical force of 15 mN. The obtained hardness and Young's modulus as well as AFM scans revealed that the softest material is the MnS grain, followed by the martensite, and finally the M_6C. AFM imaging of the indents also revealed a moderate pile-up effect occurring around all the three indents.

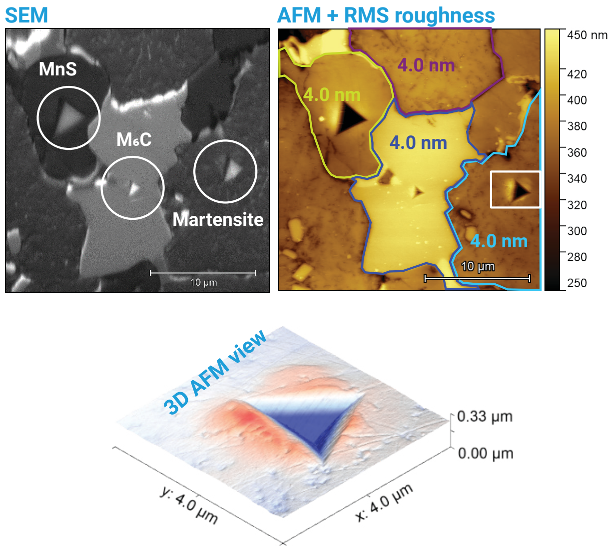

Figure 3: The SEM image identifies individual phases (indents in circles). The combination of instruments allowed easy pre-indentation roughness measurements and further visualization of the significant pile-up effect after the indentation (see 3D AFM visualisation of the matrix indent).

Table 1: Obtained values of material characteristics

Phase	Hardness	Young's modulus
M_6C	13.79 GPa	310 Gpa
Martensite	6.45 GPa	224 Gpa
MnS	3.33 GPa	114 Gpa

Multi-method sample analysis

Properties of Transformation Induced Plasticity (TRIP) steel sample (Figure 4) were analysed by four techniques: EBSD and a combination of SEM, AFM and nanoindentation. TRIP steel possesses a complex microstructure consisting of ferrite-bainite matrix and secondary phases (martensite and retained austenite) inhomogenously dispersed within the matrix. Analysis of advanced TRIP steels phase composition is complicated due to the presence of fine secondary phases and therefore requires a combination of techniques.

The SEM enabled to precisely localize the ROI on the sample previously analysed by EBSD technique and to accurately perform 20 indents into selected phases within a 20x20 µm area. Additionally, thanks to measuring the TRIP steel surface morphology both before and after the indentation, the acquired hardness values were corrected by using the true contact area in the indent.

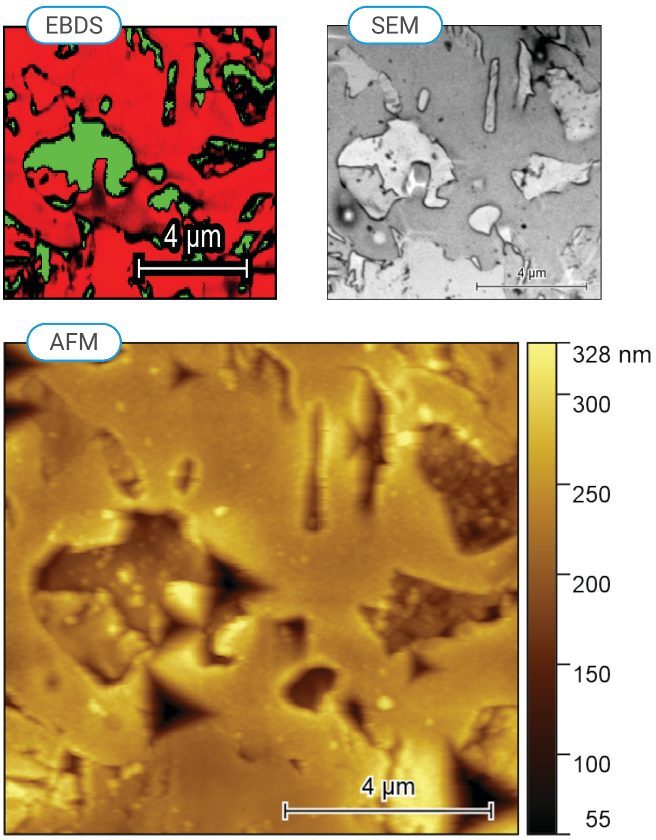

Figure 4: The same sample area analyzed by EBDS and combined SEM, AFM and nanoindentation (less visible indents are circled). Sample courtesy of JFE STEEL CORP.