Using the Hybrid Nanoscope for Non-destructive Control of Nanostructural Materials

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Abstract. In Moscow State University of Information Technology, Radio Engineering and Electronics there was developed a hybrid nanoscope (HN), which is intended for the study and control of nanostructure materials using a variety of microscopes and spectroscopy. When operating as X-ray microscope, it allows to obtain information on micron and nanometer level about internal structure of different composites and hybrid materials without their destruction.

1. Introduction
For comprehensive study of structure and composition of nanostructural materials, the different types of electronic, probe and optical microscopes are used. Electronic and probe microscopes allow mainly getting the image of the surface of objects, but for a considerable part of nanomaterials, many of their properties are related to the underlying structure. To get the information about an underlying structure, edge cuts and fractures that are obtained with some certain methodologies are often studied. In addition, applying the layer etch of surface of an object by ionic bunch, it is possible to get information about the internal structure of objects. But these destroying and expensive methods do not give complete and operative information about an internal structure. There exist some problems, both as with preparation of objects and also with interpretation of the results obtained. Studying the internal structure of materials and getting three-dimensional images is possible by means of the x-rayed radiation. Such radiation does not practically interacts with objects and in many cases does not require special sample preparation. Investigation can be conducted on air, in a liquid phase, and in a vacuum. Because of it’s moderate resolution (20–30нм on synchrotrons⁰¹, 50нм on the demountable x-rayed tubes [2–5] ), high cost, overall largenesses and high running expenses, today's nanofocal x-ray microscopy is rarely used.

2. The design of hybrid nanoscope
For complex control of nanostructured materials, a hybrid nanoscope (HN) has been developed, whose modular design allows in optimal way (with high resolution ) combine the basic types of microscopes and spectroscopic detectors (figure 1). The basic microscope of the hybrid is a scanning electron microscope (SEM) in the desktop version. The main element of HN is an electron-probe module (EPM), which consists of a column with an electron gun, vacuum system components, x-ray electronic detectors, as well as a set of objects tables (figure 2). Column is the main functional element that provides a focused electron beam. In this instrument, column is the basic structural element of EPM. The column is a system of magnetic lenses with deflecting systems inside them. To ensure maximum
density of electronic beam in used range of energy and size of the probe, an optimal focusing lens system was applied [6]. Selection of elastically scattered and secondary electrons was conducted through the objective (the last) lens (OL), which with the help of the detectors before OL focuses the electron beam on the object or target.

**Figure 1.** Schematic diagram of the operation HN in various modes:
TED – transmission electron detector; SED – secondary electron detector; ESED – elastically scattered electron detector; EDXD – energy dispersive x-ray detector; XD(p) – x-ray detector for projection mode; XD(s) – x-ray detector for scanning mode; OL – objective lens; AL – aperture lens; CL – condensor lens; EG – electron gun.
Figure 2. Electron-probe module.

The nanoscope utilize tungsten thermionic cathode operating under accelerating potential 1-40kV and has following basic modes and parameters:

| Mode name                                      | Environment     | Limit (nm) |
|------------------------------------------------|-----------------|------------|
| 1. Raster in secondary electrons              | vacuum          | 2–3        |
| 2. Raster in passed electrons                 | vacuum          | 1–2        |
| 3. Raster in elastically scattered electrons   | vacuum, air     | 10         |
| 4. Projective in X-ray                        | vacuum, air     | 20–30      |
| 5. Scanning in X-ray                          | vacuum, air     | 50         |

3. Modes of transmission X-ray microscope

The design of the scanning microscope has been optimized for operation in air in the mode of transmission X-ray microscopy with nano resolution. Since the X-ray regime is given a special attention, as the basic one an option is selected, when the electron gun is at the bottom, whereas OL and objects are at the top. Then, the objects may simply be put down on the target substrate. Thus, SEM is working in the transmission X-ray microscope (TXM) mode, while setting under the electron beam the thin metal targets on a vacuum tight substrate, which transmits X-rays on the air to the object and the X-ray detectors. For accurate and rapid focusing the electron beam on the target, a detector of secondary electrons from the target is used [7], since the nanoscale electron beams give low X-ray intensity and thus it is practically impossible to provide accurate and timely focusing of X-ray beam. The X-ray microscope can work in projection mode, when the electron beam is at a point on the target, and the X-rays passing through the object is registered with the coordinate-sensitive detector (figure 3a). Besides, while scanning beam on the target and the use of X-ray detectors with variable aperture entrance for X-rays, one can to obtain in raster (inversion) mode the X-ray image of the object (figure 3b), which resolution is determined by the input aperture of the detector and the size of the focal spot.

Using a number of detectors allows to obtain multiple images from different angles, adding them to increase the resolution and get a three-dimensional image. It is possible to use a hybrid system of detectors, in which the coordinate-sensitive detector is located on the axis for the projector mode, and other detectors are located aside, to give raster images at different angles. Moreover, among them can be energy-dispersive detectors.
To enhance X-ray spatial resolution, a near focus mode is used, when using substrates of micron and submicron thickness, a distance between the object and an electron beam \((d_{o-f})\) on a target is minimized. In this case, X-ray flux density is considerably increases on the object and the detector compared to common for X-ray microscopy values for \(d_{o-f}\) of several hundreds microns. This compensates for X-ray intensity reduction at nanoscale focal beams. Modern technologies allow to obtain thin micron and submicron vacuum-tight membranes of Be, Si, Si\(_3\)N\(_4\), C etc. At the film with thickness of the order of 0.1 mkm, electrons with energy of 10-30 keV passe through the film on an air, and backscattered electrons passed backward are registered with special detector. This allows control without a special treatment a surface of materials on an air, like as for atmospheric SEM JASM-6200 (Japan).

Using ultra-thin wafers for the targets allows you to vary within wide limits the distance the object - the focus \((d_{o-f})\) and, if necessary, implement a phase contrast by selection \(d_{o-f}\) in accordance with the focal spot. In addition, if the thickness of the object is greater than the thickness of the substrate, the area of the object at different distances from the focal spot will appear at various zoom levels. This allows you to add pictures of objects made with the calibrated displacements of the focal spot, and receive cross-sectional images of the object, i.e., implement a mode of digital laminography.

**Figure 3.** Projection and raster (with three detectors) modes of transmission x-ray microscope: \(d_o\) – diameter of hole in an object; \(d_{o-f}\) – distance object - focus; \(a\) – target- detector distance; XD- X-ray detector; \(d_d\) – figur hole diameter at the input to the detector.

4. Conclusion

Developing of HN on own initiative is being conducted for several years. The main attention was paid to the design of EPM, that provides optimal formation of nanoscale electron beam in the accelerating voltage range of 1–40 kV. The work was made a small pilot party of devices has been made (figure 4), on which there were tested some design options of EPM. On the first of these, there were obtained some good results in X-rays (0.2 – 0.3 mkm at 15kV) and electrons (less than 10 nm at 15 kV). Images zinc particles of different sizes in the organic film with the thickness of 270 mkm obtained in X-ray and a secondary electron in SEM are shown in figure 5. When comparing these images, the advantage in the X-ray image is apparent. The study with SEM of fracture of the film or layer-etching of the surface with the ion beam does not give a complete and timely information about the internal structure of the film as was done in an X-ray mode. HN in X-ray modes may be efficiently used for obtaining information about the internal structure of the various embodiments of composite and hybrid materials in the micron and nanometer level. The main technical parameters of HN give a possibility to combine the features and functions of most imported different types of microscopes (figure 6) with parameters higher than that of each of them, and at the cost of the desktop microscopes. This is an import-substituting device of high level, that has yet no equals.
Figure 4. Appearance of hybrid nanoscopes (side view).

Figure 5. Object – organic film of 270mkm thickness with Zn particles: a) TXM (X-ray); b) SEM (electrons).
Figure 6. Hybrid nanoscope and devices, features and capabilities of which combines GN.

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