

Current status and prospects of X-ray microscopy

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Abstract. Design and main constructive features of a new combined device for X-ray electron and X-ray microscopy for nanometer diagnostics are considered in relation to the current status of modern X-ray and electron microscopy.

1. Introduction

Currently, X-ray microscopy enables to conduct non-invasive research on the micron level of the different materials and devices in various fields of technology. With increasing resolution of X-ray machines, they are increasingly being used for scientific research at the nanoscale in X-ray microscopes.

In the X-ray machines, sealed and dismountable X-ray tubes are used. The formers have a low cost, small size, but a limited life time and may provide low resolution, worse than 10 μm . This is due to the fact that all of the current from the cathode is focused on the target. Even the use of scaling down magnetic lenses placed outside the tube does not allow exceeding this limit due to lens aberrations without the appropriate aperture on the tube axis.

In the dismountable tubes for focusing the electron beam after cathode, 2–3 electromagnetic lenses were applied, in which the relevant restrictive and aperture diaphragms are established to optimize the aberration on the axis. In principle, such an electron-optical systems are used in electron microscopes to obtain nanoscale electron beams. In the case of X-ray microscopes, X-ray source is the generation region (focal spot) formed in the target which is being broadening due to the path length of the electrons. Further, X-rays output rate from the target is lower by several orders of magnitude, than the corresponding rates for the secondary electrons. Therefore, X-ray imaging requires high current electron beams, which corresponds to their large size and low resolution.

A high resolution (50 nm) is achieved in laboratory nanoXCT-100 systems (X-Radia, USA) with a rotating anode tube and X-ray optical elements (figure 1). Higher resolution (20–30 nm) on biological objects was obtained at synchrotrons using X-ray optics [1]. A number of foreign companies produce high-voltage microfocus inspection apparatus with collapsible X-ray tubes having a limiting resolution at 0,1–0,5 μm , that allow research and inspection on the micron level. The majority of them are performed in a single module embodiment, when almost all the constituent elements are in a single protective housing. Therefore, they have a large overall size to the size in three dimensions 1–2 m with volumes 6–8 m^3 at 2–3 ton weight. These devices are mainly designed to control large circuit boards. In general, due to their design and dimensions, it is difficult to call such devices as microscopes and they are rarely used in scientific research.

Among these devices outstands a bit XD-7600NT – Dage (England) with the limiting resolution 0,1 μm (figure 2). X-ray tube used in it, is advertised as a revolutionary step in the development of the tubes, since it combines the advantages of conventional sealed and dismountable tubes. The tube is

called sealed-transmissive. In it, a small magnetic drive and a gettering pumps are connected to a system of magnetic focusing lenses. There is also an indoor high vacuum gate through, which the primary backing pumping, is conducted by the manufacturer and launch o high vacuum pumps. In such a tube, high vacuum is maintained permanently and only if necessary, the running of magnetic drive pump occur. The gain in production costs compared to conventional dismountable tubes is minimal, since only the backing pump is excluded from the cost. The pumping time is little only in the case of small intervals between switching on and off. And at large period there is a possibility not to obtain a high vacuum, due to leakage into a vacuum volume. In practice, in some cases it occurs. As a result, it is necessary to send the tube to the manufacturing company to produce a vacuum. In addition, the cost of such a tube is still much higher, than the cost of a sealed tube.



Figure 1. Laboratory nanoXCT-100.



Figure 2. XD-7600NT – Dage (England).

At present, in the Russian Federation, X-ray machines at the dismountable tubes are not made serially, but there is some experience in this area. For example, until the 90s the Vyborg instrument-making plant manufactured X-ray microscopes (MTP-7 and others) with a resolution of 5 μm . It should be noted, that with modern X-ray detectors and transmission targets, a resolution less than 1 mm would be obtainable at them. In addition, at the Moscow technology university MIREA, the X-ray measuring microscope (XMM) was made to control the thickness of layers in carbon like nuclear fuel elements, working at accelerating range 5–30 kV with 0,2–0,3 μm resolution (figure 3).

In principle, it is expedient to manufacture microfocus devices in the modular embodiment as MTP-7, where in in the protective chamber there are only objects moving mechanisms and detectors. Dismountable tube and power supply units are out of the camera. Herewith, the devices will have smaller size and cost. Figure 4 shows an experimental model using a camera on the MTP-7.



Figure 3. XMM.

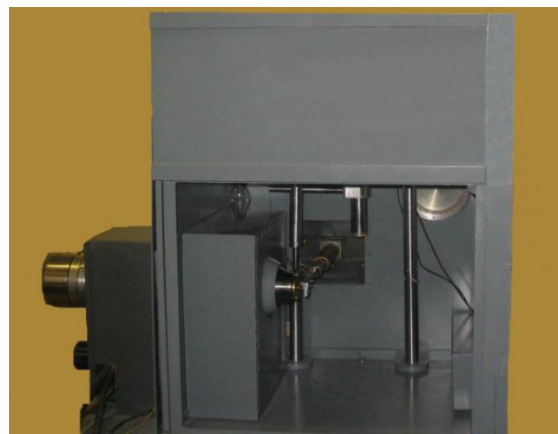


Figure 4. Shielding camera (X-ray dismountable tube – at left, object table – at back, detectors – up or at right).

2. Hybrid nanoscope

For X-rays nanometer resolution at the level of tens of nanometers, it has been developed a hybrid nanoscope (HN), which optimally combines scanning electron microscope (SEM) and X-ray microscope (XM). To maximize the resolution parameters in both modes, an optimized system of magnetic lenses with interchangeable pole pieces was used to focus the electrons [2]. In the SEM mode, an object is set under the electron beam, and with secondary electron detector (SED) before the last (objective) lens in a scanning mode the secondary electrons from the object are recorded. In XM mode, a target on a vacuum-tight substrate is set under the electron beam. For accurate and fast focusing of electrons on the surface of the target, over the entire operating range of resolutions an image of the target surface is obtained in secondary electrons using a SED, while scanning electron at a target [3]. XM can work in projection and scanning (reverse) mode (figure 5).

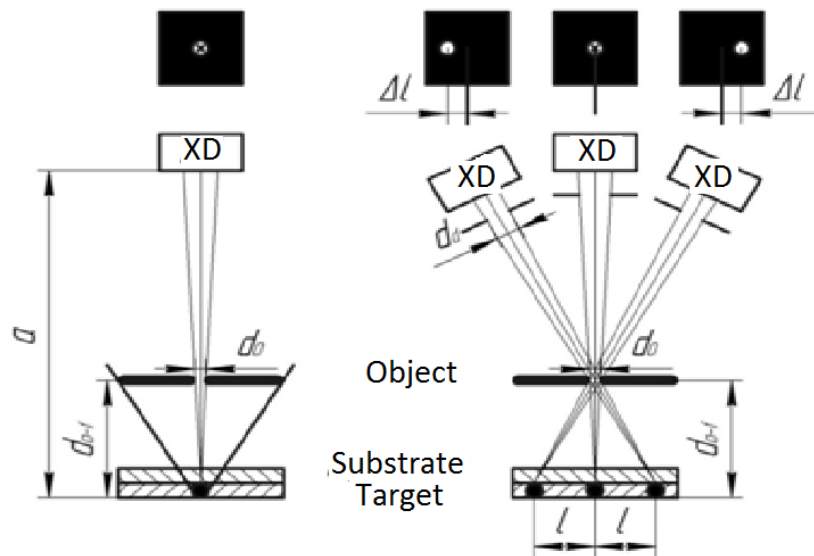


Figure 5. Projective and scanning (with 3 detectors) modes (d_o – diameter of hole in the object); d_{o-f} – object-focus distance; a – target-detector distance; XD – X-ray detector; d_d – hole diameter at the detector input).

To obtain high adsorption contrast for the nanoscale details of objects, it is necessary to use the accelerating voltage on 5–15 kV level. In this case, the resolution level of 20–30 nm can be achieved in near focus mode, when a target is laid on a sub-micron and micron substrate of Be, Si, Si_3N_4 , C, etc. This allows placing objects on micron and submicron distances from the focal spot. In the microfocus X-ray microscopy, there are commonly used the substrates with thicknesses at the level of hundreds of microns. While using thinner substrates, the density of X-ray flux rises at both the object and the detector. This makes it possible to compensate for a decrease in the intensity of X-ray source at the nanoscale focal spot. In addition, for small distances between the subject and the focus, also a phase contrast mode can be effectively realized. The material and the thickness of the target influences greatly on the intensity of the soft X-rays flowing after the substrate. The use of materials with high density, in which electrons have a smaller path length, reduces beam broadening and increases the radiation intensity. If the path of the electrons will be more than the target thickness, a part of the electrons pass through the target without producing X-rays, while this slightly reduced broadening of the electron probe. With the thick targets it may occur that the part of the target, to which the electrons are not reached, will substantially absorb X-rays from the focal spot. Therefore, it is necessary to optimize the thickness of the target in accordance with the accelerating voltage. The resolution in the X-ray is also significantly affected by the thickness and chemical composition of objects. As a result, if you choose the optimal actual test object, the parameters of the target and the accelerating voltage, it is possible to obtain high resolution at the level of tens of nanometers.

3. Results and discussion

The five experimental samples (figure 6) were produced during the development of HN. Good initial results have been obtained in their test. Due to their technical parameters HN's allow to combine the key features and capabilities of the majority of imported electronic and X-ray microscopes. It is not easy an imports substitution, rather it is an import superior nanodiagnostics device. The advantage and performance of the HN can be seen, when comparing the images of objects with approximately the same size (figures 7, 8) made on different devices. And we must remember that the first two shots were taken at the upper level of the capacity. The pictures on the HN are not made under optimal conditions. The projection shooting mode was carried out on X-ray film and the scanning one – by a single detector. Going to X-matrix and the use of 10–20 detectors in raster mode will significantly increase resolution and image performance. It should be noted, that you can significantly increase the resolution due to the use in dismountable tubes of the field emission cathodes, as an electron source.



Figure 6. External view of electron-probe modules of HN (0–40 kV) (left) and low-voltage (0–15 kV) HN (LVHN) (right).

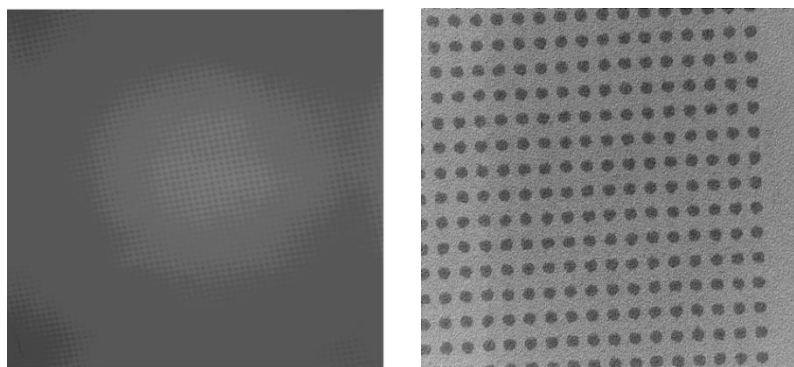


Figure 7. Metal balls with 20 μm diameter: sealed X-ray tube (left) and XD-7600NT (right).

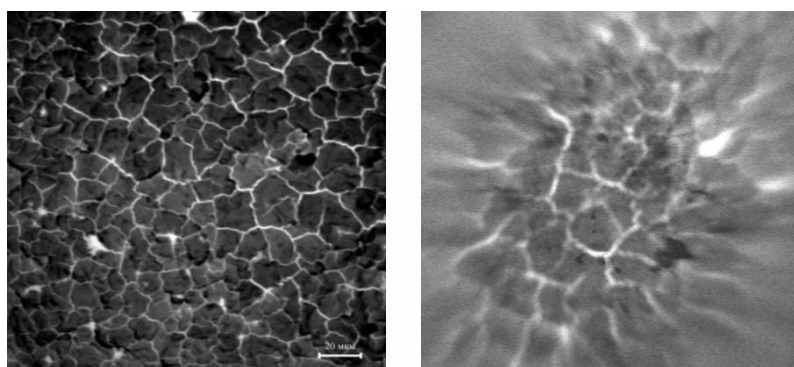


Figure 8. Re-film (grain size: 20–30 μm , gaps between them: 1 μm , film thickness: 2–3 μm): HN-projection mode (left) and HN-scanning mode (right).

4. Conclusion

It should be noted that the improvement of X-ray microscopes goes both in the direction of increasing of X-ray sources performance, parameters, as well as of increasing the parameters of detectors and X-ray optical elements. As a result, one can expect to go in the near future to the level of 10 nm.

References

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