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**Identification of Micro and Nanoparticles by SEM and XRD in Forensic Field.** Marek Kotrly. *Institute of Criminalistics Prague.*
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Experts in forensic laboratories routinely deal with a broad range of materials that they have to be able to identify. Unlike the majority of other analytical laboratories which usually specialize in specific types of materials, in a forensic laboratory, a wide variety of objects can be encountered ranging from materials of natural origin up to industrial technological artefacts. Electron microscopy and X-ray methods are one of the main identification procedures in forensic inorganic microanalysis.
Currently, in forensic facilities, materials containing nanoparticles and nanocomposites are appearing more and more frequently. Identification of these substances is of key importance due to components precisely determining classification of an unknown material (for example a number of forgeries differs only in the content of nanoparticles and nanocomposites). To determine the presence of micro and nanoparticles and identification of unknown material with a reference sample can be used electron microscopy. Field emission electron microscopy allows a direct study of morphology of nano-objects and by using a STEM mode it is possible to employ potentialities of transmission electron microscopy. When analysing materials with the content of nanoparticles, we can also use automated systems for GSR analysis. This is conditioned by the content of elements with a higher atomic number in investigated nano-objects than in a matrix (primary detection by BSE). Mn or Fe are approximately considered as a limit. Through systems with classical thermal cathode it is possible to identify routinely particles with the size of approx. 0,5 micrometers. If the above mentioned conditions are fulfilled, the sufficient content for unambiguous detection by using systems GSR is approx. 50 ppm. By means of field emission systems can be identified the presence of particles from the size about 30 nm, with content approx. 3 ppm. However, the price is rather considerable time consumption (from tens to hundred hours depending on the presence of other components and on conditions of the analysis). Combined systems SEM/FIB facilitate the study of inner structure of GSR, PBR, nanocomposites, etc. (e.g. colour variable pigments - effect paints, protecting elements).
From X-ray methods, a technique of X-ray Powder Microdiffraction (micro-XRPD) has been increasingly putting into practice by means of which the size of analysed areas is approaching to the dimensions examined through other standard methods - optical microscopy, FTIR, SEM, etc. For the possibility to analyse microscopic particles and abrasions on one holder by using different techniques (to rule out the risk of loss, or contamination) various fixation methods were tested. Very promising proves the utilization of conductive zero-background silicon sample holders, whose conductivity approx. 5 ohm.cm-1 suits well even for conditions of SEM in standard vacuum modes. The size of monocrystalic areas in a sample is a particular limiting factor of the exploitation of micro-XRPD. Based on performed experiments, the limiting detectable content of micro and nanoparticles in mixtures can be regarded as the range between 0,1-5% (depending on the amount of material and the symmetry).
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**Symmetry of Bend Contours in Electron Microscopic Images of Nanothin Crystals.** Malkov Vyacheslav[a], Strekalovsky Victor[a], Malkov Andrey[b], Malkov Oleg[b], Puchin Vladimir[c]. [a]*Institute of High-Temperature Electrochemistry, Ural Branch RAS.* [b]*"ROSNA" Scientific and Production Center.* [c]*Institute of Metal Physics, UB RAS, Ekaterinburg, Russia.*
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A characteristic feature of electron microscopic images of nanothin crystals is the presence of patterns of extinction bend contours [1]. In some cases the bend contour patterns exhibit a symmetry. A question arises if it is possible, in accordance with the general physical Curie principle, to judge of the real structure and the orientation of the nanothin crystals under study from the symmetry of bend contour patterns?
An electron microscopic examination was performed concerning the real structure and the orientation of nanothin crystals of hexagonal selenium whose electron microscopic images contained patterns of extinction bend contours with the mirror (Fig. 1a) and the inversion symmetry (Fig. 1b) or asymtric contours (Fig. 1c).

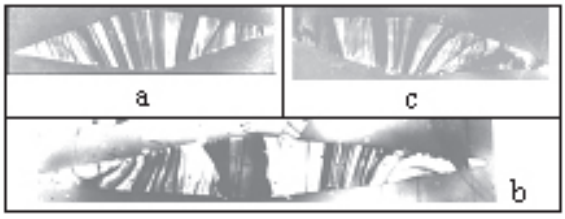


Fig. 1. Microphotographs of nanothin selenium crystals whose contour patterns exhibit different types of symmetry (a ×10000), (b ×15000), (c ×10000)
The microdiffraction analysis of diamond-shaped crystals, whose bend contour pantterns had a mirror symmetry relative to the symmetry plane passing through the short diagonal of the diamond-shaped crystal perpendicularly to its surface, gave mirror-symmetry diffraction patterns of symmetrically equal areas of the crystal. The symmetrically equal areas of the crystals, whose contour patterns exhibited an inversion symmetry or were asymmetrical, gave inversely symmetric or asymmetric electron micropatterns.
Thus, the study of the real structure and the orientation of nanothin crystals of hexagonal selenium, whose electron microscopic images contained bend contour patterns exhibiting different types of symmetry, suggested that the general physical principle of the Curie symmetry can be

used for analysis of the real structure and the orientation of nanothin crystals. A method has been proposed for analysis of nanothin crystals whose electron microscopic images include patterns of bend contours possessing a particular symmetry.

[1] Bolotov I. E., Malkov V. B. *Proc. XI Int. Congr. on Electron Microscopy*. **1986**, Japan, Kyoto, V.1., P.92

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