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Tytuł rozprawy doktorskiej: Opracowanie metodyki preparowania wielowarstwowych nanostruktur do badania za pomocą nowoczesnych mikroskopów elektronowo-jonowych

ABSTRACT

The doctoral dissertation was carried out as part of the "Implementation Doctorate Programme" and focuses on the development of methodologies for the preparation and characterization of cross-sections of multilayered nanostructures intended for examination using modern electron-ion microscopes. This subject is closely related to the operations of Nanores, the company employing the Author. The primary challenge lies in the sample preparation for SEM analysis, which must not introduce undesired alterations to the original structure's properties. Unfortunately, in many scientific publications, the description of sample preparation for SEM is either omitted or inadequately detailed, particularly concerning the fabrication of multilayer nanostructures.

The research problem addressed in this dissertation involves analyzing the properties of complex structures in the form of multilayer coatings with nanometric dimensions. The research plan included the characterization of three series of multilayered nanostructures based on titanium and vanadium (i.e., Ti/V/Ti), molybdenum and niobium (i.e., Mo/Nb/Mo), and tungsten and hafnium (i.e., W/Hf/W). These nanostructures were designed and fabricated using magnetron sputtering specifically for this doctoral research. The choice of elements with similar atomic numbers Z (Ti(22) and V(23), Mo(42) and Nb(41), as well as W(74) and Hf(72)) was deliberately made to complicate the analysis, as material contrast in SEM is closely related to atomic number. Additionally, these three pairs of elements were selected to allow the assessment of coating properties composed of elements with low, medium, and high atomic numbers. The construction of the multilayers themselves was also intended to pose a challenge. Consequently, in each of the three sample series, the top and bottom layers had the same thickness of 200 nm, while the middle layer varied in thickness from 100 nm to as thin as 5 nm.

Extensive experimental studies were conducted as part of the dissertation, including: a) analysis of the impact of sample preparation methods, such as conventional fracturing, ion beam preparation using Ga, and Xe plasma, on the microstructure of multilayer nanostructures; b) evaluation of the effect of protective coatings (carbon or platinum-based) on the accuracy of multilayer nanostructure analysis using SEM/EDS; c) investigation of the influence of atomic number on SEM image contrast for nanostructures with layers composed of elements with similar atomic numbers. These studies culminated in the development of an enhanced methodology for the preparation of multilayer nanostructures for examination using modern Dual Beam microscopes, namely SEM/Ga-FIB and SEM/Xe-PFIB. The results of these studies have already been implemented at Nanores, which commercially offers, among other services, the characterization of advanced nanomaterials using SEM/EDS with high-end dual-beam microscopes.

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