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An XPS method for layer profiling of NbN thin films

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Abstract. Layer chemical and phase profiling of niobium nitride thin films on a silicon substrate oxidized on air was performed with the help of a method designed by us. The method includes: a new method of background subtraction of multiple inelastically scattered photoelectrons considering depth inhomogeneity of electron inelastic scattering; a new method of photoelectron line decomposition into component peaks considering physical nature of different decomposition parameters; joint solution of the background subtraction and photoelectron line decomposition problems; control of line decomposition accuracy with the help of a suggested performance criterion; calculation of layer thicknesses for a multilayer target using a simple formula.

The interest in thin films of niobium and its compounds is caused by their superconductive properties used in cryogenic electronic instrumentation. For example, niobium nitride thin films are attracting rising attention in recent submillimeter and THz receiver research [1]. As an oxidation result of films of niobium or its compounds (originally homogeneous) multilayer and multiphase films are built. The superconductive properties of niobium compounds become worse with increase of surface niobium oxide layer thickness. Oxide films of niobium and its compounds are covered by a great amount of works, i.e. [2-5]. However, there are no works covering layer phase profiling of oxidized thin films of niobium compounds.

This work presents layer phase profiling of oxidized thin films of niobium nitride on a silicon substrate by the means of XPS. Thin niobium nitride films were sputtered onto a silicon substrate by the magnetron method. The films were oxidized during unload from the vacuum chamber. X-ray photoemission spectra were obtained with the electron-ion spectroscopy module based on the platform Nanofab 25 (NT-MDT). In the analytical chamber, an ultrahigh oil-free vacuum about 10^{-8} Pa was achieved. The spectra were recorded with an electrostatic semi-spherical energy analyzer SPECS Phoibos 225.

Layer profiling on the base of XPS spectra of multilayer multicomponent thin films interpretation is a complex inverse problem with a lot of unknown parameters. For correct solution of this problem new methods and approaches designed by the authors are suggested:

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- the background subtraction method considering the difference between surface and volume energy losses;
- using of parameters that are constant in all the range of XPS for calculation of background and line profiles;
- using of parameters of line profiles defined from spectra of the Handbook of X-Ray Photoelectron Spectroscopy for pure homogeneous targets;
- calculation formulae for natural peak widths of photoelectrons from chemically bound elements;
- joint interpretation of different lines of one element using the same model;
- calculation of layer thicknesses and chemical and phase compound in the multilayer target model considering the results of decomposition of various lines.

On the base of the designed methods, layer chemical and phase profiling of air oxidized niobium an niobium nitride films on a silicon substrate was performed. The results of this analysis:

- a *niobium film 100 nm thick*: on the film surface there is a hydrocarbon layer 1.4 nm thick, oxide Nb_2O_5 layer 9.6 nm thick, under this layer there is a suboxide Nb_2O layer 1 nm thick, then there is metallic niobium.

- a *NbN film 10 nm thick*: on the film surface there is a hydrocarbon layer about 1 nm thick. Then there is a multiphase oxide layer of NbO_x (70% Nb_2O_5 , 7% Nb_2O_3 , 23% NbO) with a total thickness of 2 nm. This layer is generated after unloading the target from the magnetron chamber into air. During the NbN thin film oxidation process the phase state of niobium nitride changes. Under the oxide layer a niobium nitride layer of another phase NbN_x (probably Nb_5N_6) 1.5 nm thick is generated. Then there is an unchanged layer of NbN.

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