

## OBTAINING THE COATINGS OF NIOBIUM FROM ITS MIXED LIGAND COMPLEX BY THE CHEMICAL VAPOR DEPOSITION

*Sliusarchuk L. Zheleznova L.*

V. I. Vernadsky Institute of General and Inorganic Chemistry NAS of Ukraine  
32/34, Palladina prospect, Kyiv, 03142, Ukraine  
e-mail: [l.sliusarchuk@yahoo.com](mailto:l.sliusarchuk@yahoo.com)

The possibility of obtaining a variety of functional materials containing niobium, due to a number of useful physicochemical and technological properties, makes it suitable for use in nuclear energy and space technology. For example, at a relatively low density, niobium is characterized by mechanical strength, thermal stability, and high plasticity from 200°C to 2000°C. The production of thin films and layers of niobium requires special energy and technical costs (magnetron sputtering, vacuum physical deposition, etc.). In this regard, of particular interest is the prospect of using the advantages of chemical vapor deposition (CVD) [1] to obtain niobium and its compounds. The use of volatile compounds of niobium with subsequent fragmentation of the precursor molecules, which allows the formation of structured sediments, is a prerequisite for mitigating the conditions for the deposition of niobium and its oxides [2-5].

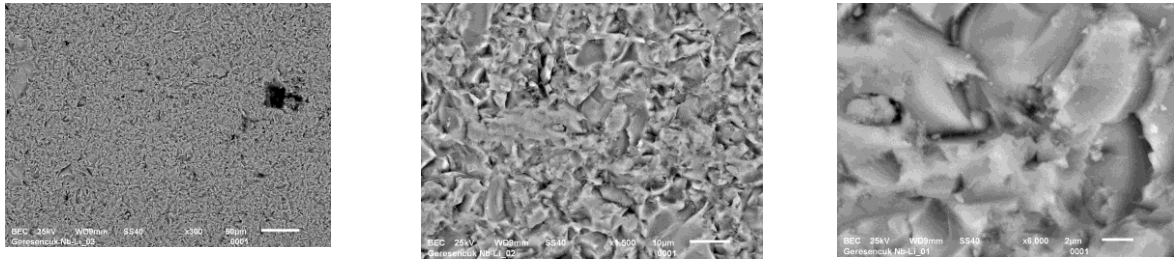
The aim of the work is to determine the synthesis conditions by the CVD method from volatile niobium-containing precursors, thin niobium films on a quartz substrate to study its morphology, structure and elemental composition.

A coordination compound  $[\text{Nb}(\text{OCH}_3)_2\text{Ac}]\text{Cl}_2$  was used in the synthesis of niobium-containing films.

The deposition of plasma activated chemical vapor deposition (PACVD) was carried out using "Plasma 600-TM". A low-temperature plasma discharge with a power of 0.2–0.7 kW and a frequency of 13.5 MHz was used; it was excited by the capacitive method at a working chamber pressure of 40–266 Pa (0.3–2 Torr). Argon was used as the plasma gas. The complex  $[\text{Nb}(\text{OCH}_3)_2\text{Ac}]\text{Cl}_2$  was placed in a quartz ampoule of an evaporator, from which the precursor gas flow supplied to the atmosphere of a carrier gas (argon) entered the discharge chamber onto a heated substrate. During thermal destruction of the coordination compound, a film is formed on the substrate from the decomposition products of the complex. The exposure time was 15 minutes.

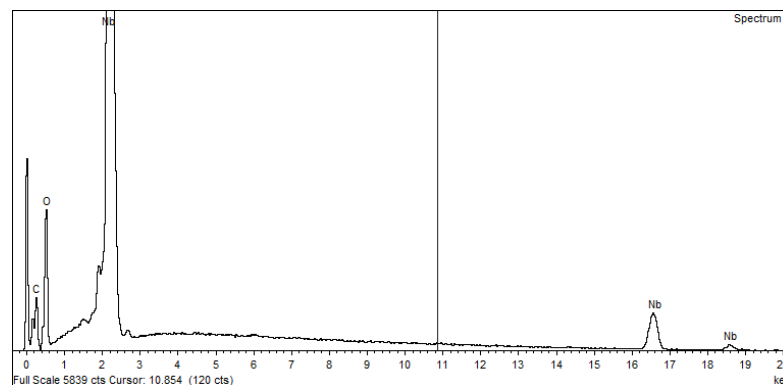
Analysis of the results obtained by scanning electron microscopy and high-resolution electron microscopy showed that a uniform coating is formed, which has the form of flakes with a size of 2-5  $\mu\text{m}$ . The kinetic conditions for the formation of such coatings indicate a high deposition rate at given values of the precursor partial pressure. SEM-images of the surface of the sample taken at different degrees of device resolution are shown in the Fig. 1. The corresponding parameters are shown at the bottom of each of the image fragments.

The morphological features and distribution of elements in the resulting coating are studied. The composition of the coatings was analyzed using a combined technique: scanning electron spectroscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). This made it possible to directly estimate the elemental composition and structure of the coating layer. Studies have shown that the coating consists of a niobium phase and phases of oxide or carbide of niobium.



**Figure 1** - SEM image showing the morphology of coatings, obtained by the method of chemical vapor deposition of the precursor (PACVD)  $[\text{Nb}(\text{OCH}_3)_2\text{Ac}]\text{Cl}_2$ , with a magnifications of  $300^\times$ ,  $1500^\times$ ,  $6000^\times$  and a scale bar representing  $50\ \mu\text{m}$ ,  $10\ \mu\text{m}$ ,  $2\ \mu\text{m}$ , respectively.

The EDX measurement was made for 14 different areas of the coatings. Secondary processing of the spectral peaks belonging to different parts of the sample showed the presence of niobium, oxygen, and carbon. In some samples, trace amounts of chlorine are observed, resulting from the decomposition of the main niobium-containing precursor. The most probable information is obtained by measurements on the part covering the largest surface area of the coating (Fig. 2).



**Figure 2** - The spectrum of X-ray energy dispersion in the selected area of the sample with elemental assignment.

The coating consists of metallic niobium in a mixture with the phases of  $\text{Nb}_2\text{O}_5$ ,  $\text{NbO}_{2.47}$ , and  $\text{NbO}_{2.42}$ ,  $\text{NbO}_2$ ,  $\text{NbO}$ ,  $\beta$ -phase -  $\text{Nb}_2\text{C}$ ,  $\delta$ -phase -  $\text{NbC}$ , as evidenced by the evaluation of the stoichiometric modeling of the obtained results. Based on the ratio of peak intensities, it can be concluded that the metallic niobium phase dominates in content. This opens up the possibilities for obtaining coatings of niobium by the CVD from volatile niobium-containing precursors and their application in various branches of technology.

#### References:

- [1] Gerasimchuk A.I., Mazurenko E.A. Volatile Metals Coordination Compounds as Precursors for Functional Materials Synthesis by CVD-Method // *Journal de Physique IV France*. - 1995. - Vol. 5. - P. C5-547-C5-551. - DOI: 10.1051/jphyscol:1995564.
- [2] Guo Y., Tao J., Jiang J., Zhang J., Yang J., Chen S., Chu J. Low temperature solution deposited niobium oxide films as efficient electron transport layer for planar perovskite solar

cell // *Solar Energy Materials and Solar Cells*. - 2018. - Vol. 188. - P. 66-72. - DOI: 10.1016/j.solmat.2018.08020.

[3] Zaitsev S.V., Gerasimenko Yu.V., Saltykov S.N., Hoviv D.A., Hoviv A.M. Formation of ultrathin Nb<sub>2</sub>O<sub>5</sub> films on quartz substrates // *Inorganic Materials*. - 2011. - Vol. 47. - P. 468-472.

[4] Muchiri P.W., Mwalukuku V.M., Korir K.K., Amolo G.O., Makau N.W. Hardness characterization parameters of Niobium Carbide and Niobium Nitride: A first principles study // *Materials Chemistry and Physics*. - 2019. - Vol. 229. - P. 489–494.

[5] Yang Y., Lu X., Tan W., Xiao L., Zhu L., Xie D. Study on laser annealing of niobium films deposited on copper for RF superconducting cavities // *Nuclear Inst. and Methods in Physics Research, A*. - 2020. - Vol. 964. - P. 163803. - DOI: 10.1016/j.nima.2020.163803.