Research of the effect of bias voltage on the morphology, structure and lattice spacings of the Nb coatings deposited by inverted magnetron

A A Lozovan, S Ya Betsofen, A S Lenkovets, I A Grushin, A A Labutin and Yu S Pavlov

Moscow Aviation Institute (National Research University), 125993, Moscow, Russia

E-mail: s.betsofen@gmail.com

Abstract. Using the metallography and X-ray diffraction analysis the effect of the bias voltage on the substrate on the structure, morphology, texture, and lattice parameters of Nb coatings deposited by an inverted cylindrical magnetron has been investigated. It is shown that the coatings are characterized by increased lattice spacings compared with pure niobium, which can be associated with both oxygen and hydrogen saturation and the presence of compressive residual stresses.

1. Introduction
The article [1] emphasized that in order to effectively use the inverted cylindrical magnetron it is necessary to optimize the parameters of its operation. To use this magnetron, the authors suggested for the deposition of layers of composites of refractory metals and their compounds. However, many works have long been established (see for example [2, 3]) that the properties and structure of thin films of refractory metals are largely determined by the gas impurity background present in the working chamber during their deposition by evaporation or sputtering. Special attention in these studies was given to niobium as an effective getter, despite the fact that for the manufacture of superconducting Nb films, it was necessary to ensure the minimal presence of impurity atoms in their composition [4].

Also very well-known is the very significant role in the process of film deposition, which is played by the bias voltage on the substrate. A negative bias voltage increases the energy of ions bombarding the growing film, leading to a higher compaction of the film, an increase in compressive stress, and a change in the structure and texture of the coatings. The substrate temperature is also important, which also has a significant impact on the structure and properties of coatings.

In this work, we investigated the effect of the bias voltage on the substrate on the structure, morphology, and lattice parameters of Nb coatings deposited by an inverted cylindrical magnetron presented in [1].

2. Materials and research methods
Coatings were sprayed onto tubes with a diameter of 10 mm from copper M1, the surface of which was polished, polished and washed in acetone and alcohol. The deposition of Nb coatings was carried out according to the modes listed in table 1.
Table 1. Modes of spraying Nb coatings.

| Sample | $I_{\text{cathode}}$ (A) | $U_{\text{cathode}}$ (V) | $U_{\text{substrate}}$ (V) | $t$ (h) | $T_{\text{substrate}}$ ($^\circ$C) | $P_{\text{Ar}}$ (Pa) |
|--------|-----------------|-----------------|-----------------|--------|-----------------|-----------------|
| Sample 1 | 0.5             | 260             | 0               | 5      | 250             | 0.2             |
| Sample 2 | 0.5             | 260             | –100            | 5      | 300             | 0.2             |
| Sample 3 | 0.5             | 260             | –200            | 6      | 330             | 0.2             |

3. Results of research and discussion

The appearance of the coatings is presented in figure 1. With an increase in the voltage of mixing, the microstructure of the coating evolved from a large columnar to a dense columnar, and then to a glare-free structure with a reduced surface roughness. The phase composition, crystallographic texture, and Nb lattice periods of the coatings obtained by the magnetron method at bias voltages of 0, –100 and –200 V were studied using CuKα X-ray diffraction analysis. In addition to reflections from the bcc niobium lattice (figure 2), no X-rays were detected on X-ray diffraction patterns phases, however, the texture and periods of the niobium lattice change with variations in the bias voltage.

Figure 3 shows the values of the lattice spacing for the three coatings and the dashed line shows the lattice spacing for pure niobium ($a_{\text{Nb}} = 0.3300$ nm). It can be seen that the lattice spacing of the coatings substantially exceed the lattice spacing of pure niobium, and the maximum value of the lattice spacing corresponds to the coating applied without a bias voltage ($a = 0.3360$ nm). As the bias voltage...
increases, the period decreases to \(a = 0.3343\) nm for a bias voltage of \(-100\) V and to \(a = 0.3314\) nm for a bias voltage of \(-200\) V.

\[
\begin{array}{c}
\text{(a)} \\
\text{(b)} \\
\text{(c)}
\end{array}
\]

Figure 2. X-ray diffraction patterns of Nb coatings deposited at different bias voltage: (a) \(-U_{\text{substrate}} = 0\) V; (b) \(-U_{\text{substrate}} = -100\) V; (c) \(-U_{\text{substrate}} = -200\) V.

An analysis of the influence on the magnitude of the lattice spacing of niobium of the interstitial and substitution elements based on the data of the Pearson monograph [1] showed that the saturation of niobium with oxygen and hydrogen can only increase the lattice spacing by \(0.0025\) nm. An additional increase in the lattice period can be given only by compressive stresses acting in the tangential direction of the cylindrical surface of the coating, which lead to an increase in the lattice spacing in the direction of measurement, i.e. in the radial direction. For niobium with a Young's
modulus of ~100 GPa for an increase in the lattice spacing of the missing 0.0011 nm, a compressive stress of ~1 GPa can be given.

![Figure 3. Lattice spacing of Nb coatings deposited at different values of the bias voltage: : $a_{Nb}$ – lattice spacing of pure Nb.](image)

Figure 3. Lattice spacing of Nb coatings deposited at different values of the bias voltage: $a_{Nb}$ – lattice spacing of pure Nb.

Figure 4 shows the inverse pole figures for the three coatings, and figure 5 pole density histograms for 5 reflections for these three coatings. These results indicate that in the coating applied with a zero bias voltage, the texture is weakly expressed (figure 3(b)). For coatings deposited with a non-zero bias voltage, the texture is characterized by the location along the radial direction of the crystallographic direction <111> (figure 4(c), (d)), while the texture intensity in coating with a bias voltage of ~100 V is higher than the coating with a bias voltage of ~200 V. It should be noted that niobium has a “negative” anisotropy of elastic modulus and the <111> direction is the direction for which the value of the Young's modulus is minimal.

![Figure 4. Standard stereographic triangle (a) and inverse pole figures of Nb coatings on a copper substrate, deposited at different values of bias voltage: (b) – $U_{substrate} = 0$ V; (c) – $U_{substrate} = -100$ V; (d) $U_{substrate} = -200$ V.](image)

Figure 4. Standard stereographic triangle (a) and inverse pole figures of Nb coatings on a copper substrate, deposited at different values of bias voltage: (b) – $U_{substrate} = 0$ V; (c) – $U_{substrate} = -100$ V; (d) $U_{substrate} = -200$ V.

![Figure 5. Pole density for (hkl) reflections on the IPF of Nb coatings deposited at different values of the bias voltage.](image)

Figure 5. Pole density for (hkl) reflections on the IPF of Nb coatings deposited at different values of the bias voltage.

References

[1] Lozovan A A, Lenkovets A S, Ivanov N A, Alexandrova S S and Kubatina E P 2018 XV International scientific and technical conference Rapid solidification materials and coating (RSMC–2018) (Moscow: Probel-2000) pp 152–7

[2] Sosniak J 1968 *The J. Appl. Phys.* 39 4157–63

[3] Perry A, Sartwell B and Valvoda V 1992 *J. Vac. Sci. Technol.* 10 1446–52
[4] Wilde S, Valizadeh R and Malyshev O 2018 Phys. Rev. Accel. Beams 21 073101

[5] Pearson W 1958 A Handbook of Lattice Spacing and Structures of Metals and Alloys (New York: Pergamon Press) 1044 p