Study of the influence of substrate bias voltage on the texture of Nb coatings deposited on a Cu substrate

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Abstract. The phase composition and texture of Nb coatings deposited using an inverted magnetron at voltages on the Cu substrate from 0 to –300 V were investigated by an X-ray method. With an increase in the voltage on the substrate, the thickness of the coating decreases from 9–10 μm at voltages up to –100 V and decreases to 2 μm at ~250 V. The texture of the coatings varies non-monotonically from (110) to (111), while at high voltages on the substrate in the range from –250 to –300 V, the Nb coating is characterized by a textureless state.

1. Introduction

The substrate bias voltage significantly affects the film deposition process, as well as its structure and properties [1–3]. Negative bias voltage increases the kinetic energy of ions and leads to compaction of the film, an increase in residual stresses, and a change in the structure and texture of the coating. The temperature of the substrate is of great importance; a simultaneous increase in the temperature and voltage of the substrate can also have a significant effect on the structure and properties of coatings.

Investigations of the effect of substrate bias voltage on the texture of Nb coatings deposited on a Cu substrate are carried out in this paper. The deposition of Nb coatings on a Cu substrate was studied in detail with the aim of fabricating Nb-coated copper resonators for CERN [4]. The studies presented below were carried out in the framework of a series of experiments on the creation of multilayer composite thin-walled small-sized shell structures, where Nb is one of the candidates for the material of one of the layers [5].

2. Materials and research methods

Nb deposition on a copper (M1) tube Ø10 mm was performed on a specialized sputtering unit with a cylindrical inverted cavity magnetron [6]. The unit has an oil-free pumping system as a part of a turbomolecular and spiral pre-vacuum pumps. Argon with a purity of at least 99.9 % was used as a working gas. The cathode material consists of niobium (Nb) and has a purity of ≈ 99.9 %. Before sputtering, the outer surface of the tube was polished, then the surface of the tube was cleaned in an ultrasonic bath in gasoline and isopropanol. Then the tube was installed on the rod for vertical movement of the samples and the working chamber of the device was evacuated to a residual pressure of 10⁻⁵ Pa. Before sputtering, the tube surface was cleaned by treatment in a glow discharge at an argon pressure of 5 Pa and a voltage on the substrate of 1100 V for 15 minutes. Next, 13 deposition
modes were performed, changing the bias voltage on the substrate, which are shown in table 1. The coating deposition time is 2 hours.

X-ray phase analysis and determination of texture were performed on a DRON-4 X-ray diffractometer in filtered CuKα radiation with wavelengths $\lambda_{\text{Kα}} = 1.54178$ Å.

Table 1. Modes of deposition of niobium on a copper substrate.

| Sam. № | $U_m$, V | $I_m$, A | $-U_{\text{sub}}$, V | $I_{\text{sub}}$, A | $P_{\text{Ar}}$, Pa | $T$, °C |
|--------|-----------|----------|----------------------|-------------------|------------------|--------|
| 1      | 254       | 1        | -                    | -                 | 0,2              | 295    |
| 2      | 260       | 1        | 25                   | 0,02              | 0,2              | 300    |
| 3      | 260       | 1        | 50                   | 0,04              | 0,2              | 315    |
| 4      | 260       | 1        | 75                   | 0,04              | 0,2              | 360    |
| 5      | 260       | 1        | 100                  | 0,08              | 0,2              | 370    |
| 6      | 250       | 1        | 125                  | 0,12              | 0,2              | 385    |
| 7      | 245       | 1        | 150                  | 0,2               | 0,2              | 400    |
| 8      | 245       | 1        | 175                  | 0,2               | 0,2              | 450    |
| 9      | 245       | 1        | 200                  | 0,2               | 0,2              | 502    |
| 10     | 240       | 1        | 225                  | 0,18              | 0,2              | 538    |
| 11     | 245       | 1        | 250                  | 0,17              | 0,2              | 565    |
| 12     | 250       | 1        | 275                  | 0,16              | 0,2              | 590    |
| 13     | 260       | 1        | 300                  | 0,14              | 0,2              | 630    |

Note: $U_m$ and $I_m$ are the voltage and current of the magnetron discharge, $U_{\text{sub}}$ and $I_{\text{sub}}$ are respectively the bias voltage and current on the substrate.

Inverse pole figures (IPF) were obtained by taking X-ray diffraction patterns in the angular range $2\theta = 30–150°$. The pole density of 6 independent reflections $hkl$ on the stereographic triangle: 001, 011, 013, 111, 112, 123, was determined from the equation:

$$P_{hkl} = \frac{n}{\sum_{i=1}^{N} (I_{hkl}^{\text{ref}}/I_{hkl}^{\text{ref}})}$$

where $I_{hkl}^{\text{ref}}$ and $I_{hkl}^{\text{ref}}$ are the integral intensities of the reflections $hkl$ for the textured and textureless (reference) sample, respectively; $n$ is the number of independent $hkl$ reflections ($n = 6$).

3. Experimental results

In addition to reflections from the BCC niobium lattice (Fig. 1), the X-ray diffraction patterns contained lines of foreign phases, presumably niobium oxynitride, as well as FCC reflections from the copper substrate, which intensity increases with increasing bias voltage. This indicates a decrease in the coating thickness due to an increase in the fraction of sputtered Nb atoms with an increase in the energy of the deposited coating particles. These results make it possible to evaluate the change in coating thickness with increasing bias voltage using the following equation:

$$T = \ln(I_{0}/I_{T}) sin \theta / 2\mu,$$

where $I_0$ and $I_T$ are the intensity of the reflections of the uncoated and coated substrate; $\mu$ is the X-ray absorption coefficient in Nb.

The dependence of the coating thickness on the bias voltage is shown in figure 2, which shows that the coating thickness is $\sim 9–10$ μm at bias voltages $\sim 100$ V and decreases to $\sim 2$ μm with an increase in this voltage to $\sim 250$ V.
Figure 3 shows the inverse pole figures for the substrate (figure 3, a) and coatings applied at –25 V (figure 3, b), –50 V (figure 3, c), –100 V (figure 3, d). Figure 4 shows the dependences of pole densities for reflections (111) and (110) for all coatings. These results indicate that the texture of the coatings changes non-monotonically with increasing bias voltage. As a result, only two regularities can be noted – the first is that most coatings are characterized by texture (111). The second is that for the highest values of the bias voltage ($U_{\text{sub}} = –250, –275, \text{and} –300$ V), the texture is weakly expressed, as shown in figure 4 it is clearly seen that for these values of the bias voltage, the pole densities of both reflexes are close to one.

![Figure 1](image1.png)

**Figure 1.** X-ray diffraction patterns of Nb coatings on a Cu substrate: (a) Cu substrate; (b) Nb coating, $U_{\text{sub}} = 0$ V; (c) Nb coating, $U_{\text{sub}} = –225$ V.

![Figure 2](image2.png)

**Figure 2.** Dependence of the thickness of Nb coatings on the voltage on the substrate.
Figure 3. Standard stereographic triangle of a cubic lattice (a) and reverse pole figures for Nb coatings on a copper substrate applied at different voltage values ($U_{sub}$):

$U_{sub} = -25$ V (b), $U_{sub} = -50$ B (c), $U_{sub} = -100$ B (d).

Figure 4. Dependence of the pole density for orientations (111) and (110) on the substrate voltage.

References
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