

The Influence of Layer Thickness and Deposition Conditions on Structural State of NbN/Cu Multilayer Coatings

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The influence of the main physical and technological factors of structural engineering (layer thickness, nitrogen atmosphere pressure and bias potential) on the structural-phase state of the NbN/Cu coatings was studied. It was established that with an increase in the thickness of niobium nitride layers from 8 to 40 nm (in the NbN/Cu multilayer composition), the phase composition changes from the metastable δ -NbN (cubic crystal lattice, NaCl structural type) to the equilibrium ε -NbN phase with a hexagonal crystal lattice. At low pressure $P_N = 7 \cdot 10^{-4}$ Torr in thin layers (about 8 nm thick), regardless of the U_b , the δ -NbN phase is formed. The reason for the stabilization of this phase can be the uniformity of the metallic fcc crystal lattice of the δ -NbN phase with the Cu crystal lattice. As the pressure increases from $P_N = 7 \cdot 10^{-4}$ Torr to $3 \cdot 10^{-3}$ Torr, a more equilibrium ε -NbN phase with a hexagonal crystal lattice is formed. An increase in the bias potential during deposition from -50 V to -200 V mainly affects the change in the preferred orientation of crystallite growth. In thin layers of the δ -NbN phase (about 8 nm), a crystallite texture with the [100] axis is formed. In layers with a thickness of 40-120 nm, crystallites of the ε -NbN phase are predominantly formed with a hexagonal (004) plane parallel to the growth plane. At the greatest layer thickness (more than 250 nm), the ε -NbN phase crystallites are predominantly formed with a (110) hexagonal lattice plane parallel to the growth plane. The results obtained show great potential for structural engineering in niobium nitride when it is used as a constituent layer of the NbN/Cu multilayer periodic system.

Keywords: Vacuum arc, NbN/Cu, Period, Bias potential, Phase composition, Structure, Solid solution.

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1. INTRODUCTION

The traditional approach to creating new materials is to select one element as the base [1]. In order to increase the mechanical properties in recent years, carbide [2], nitride [3], or transition metal oxides [4] are used as such basic elements.

Among nitrides, much attention is paid to niobium nitride. This is due to the fact that niobium nitride (NbN) has a high melting point (about 2500 K), which is determined by the high Nb-N bond energy (14.81 eV). Different technologies are used to deposit NbN coatings. These are laser evaporation, ion-beam deposition, magnetron sputtering, and vacuum arc evaporation.

NbN-based coatings exhibit many interesting properties, such as high hardness and electrical conductivity, heat resistance and chemical inertness [5]. NbN coatings are used as a cathode material for auto-electronic emission in vacuum, in microelectronic devices, superconducting detectors [6], and others.

However, the strength properties of materials based on NbN are still far from the expected ones. This is largely determined by the fact that with a decrease in crystallite size of less than 100 nm (and this is the basis for increasing functional characteristics), the determining factor is the border strength. The boundary can be strengthened by introducing a low concentration of impurity atoms with low dissolution in the matrix [7] or by creating a supersaturated solution in the spinodal decomposition stage [8]. In systems based on NbN, additives of aluminum (NbAlN

[9]) or silicon [10] are used.

In this regard, very promising areas for creating new dispersion-hardened materials can be considered the creation of a new class of highly solid materials based on the introduction of the second "immiscible" metal (mainly with a low modulus of elasticity) in order to significantly increase the basic toughness criterion of the coating – H/E ratios. Cu, Ni, Ag, Y, and Si are mainly used as the second "immiscible" metal in the case of nanocrystallites of transition metal nitrides. High efficiency of alloying of coatings by insoluble or slightly soluble elements in equilibrium conditions is due to their thermodynamically controlled segregation along the boundaries of nanograins with the formation of a grain boundary phase limiting grain growth at 7-15 nm. For the most studied CrN-Cu [11] and TiN-Cu [12] systems, the best properties were obtained with a copper content of about 2 %. In this case, the copper content allows the formation of a monolayer release at the boundary [7]. With a copper content of more than 40 %, high mechanical properties for the "metal nitride – copper" system cannot be achieved [13]. In this case, as studies have shown, the most significant physico-mechanical characteristics can be achieved using Cu in multilayer periodic "metal nitride – copper" systems. It should be noted that achievement of the highest hardness with a layer thickness of about 10 nm is typical for multilayer systems based on nitrides [14]. At the same time, for systems with copper layers for their effective medical-biological applications, the layer thickness should be much greater (100-300 nm). In such a large range of

layer thicknesses (10-300 nm), significant changes in the structural state occur [14]. Such changes are the basis of structural engineering and allow to define the deposition regimes to achieve the most optimal structural states (textures, phase composition, micro- and macrodeformation, etc.) [15].

The purpose of this work was to determine the possibilities of structural engineering of NbN/Cu coatings with a layer thickness of 8-600 nm. The following technological parameters were used: the value of the bias potential applied to the substrate (U_b) and the pressure of the nitrogen atmosphere during deposition (P_N).

2. SAMPLES AND RESEARCH METHODS

Multilayer coatings were deposited by the vacuum-arc method on a modernized "Bulat-6" installation [14]. The pressure of the working (nitrogen) atmosphere during the deposition was $P_N = (3-30) \cdot 10^{-4}$ Torr. Deposition was carried out from two sources (Nb and Cu). Deposition was performed on metal substrates in the modes of continuous and discrete rotation of the substrate holder with the substrates. With continuous rotation of samples fixed on the substrate at a speed of 8 rpm, the layer thickness was about 8 nm. In the discrete mode of layer deposition, the stopping time at the target corresponded to the specified layer thickness. The total coating deposition time was 1.5 hours. The coating thickness was about 12 μm . In the deposition process, a constant negative bias potential of $U_b = -50$ and -200 V fed to the substrate. The main modes of coating production are given in Table 1 (I_a is the arc current of the first and second cathodes, respectively, from above and below, U_b is a constant negative bias potential applied to the substrate during deposition, P_N is the pressure of the nitrogen atmosphere).

Table 1 – Cathode material and coating deposition parameters

No series	Cathode materials	I_a , A	U_b , V	P_N , Torr	Layer deposition time, s
1	Cu/Nb	80/85	50	$3 \cdot 10^{-3}$	continuous
2	Cu/Nb	80/85	50	$7 \cdot 10^{-4}$	continuous
3	Cu/Nb	80/85	50	$3 \cdot 10^{-3}$	120/300
4	Cu/Nb	80/85	200	$3 \cdot 10^{-3}$	120/300
5	Cu/Nb	80/100	50	$3 \cdot 10^{-3}$	20/20
6	Cu/Nb	80/100	200	$3 \cdot 10^{-3}$	60/60
7	Cu/Nb	80/100	200	$7 \cdot 10^{-4}$	continuous
8	Cu/Nb	80/100	50	$3 \cdot 10^{-4}$	60/60
9	Cu/Nb	85/90	50	$7 \cdot 10^{-4}$	20/20
10	Cu/Nb	85/90	200	$7 \cdot 10^{-4}$	20/20
11	Cu/Nb	85/90	200	$7 \cdot 10^{-4}$	60/60
12	Cu/Nb	85/90	50	$7 \cdot 10^{-4}$	60/60

The structural-phase state was studied on a DRON-4 diffractometer in Cu- K_α radiation. For monochromatization of the detected radiation, a graphite monochromator was used, which was installed in the secondary beam (in front of the detector). The shooting was carried out in the angle range of 2θ from 20 to 90 degrees. All diffraction peaks from the planes with the highest reticular density of atoms fall into this angular range. Scan step was 0.02 degree.

3. EXPERIMENTAL RESULTS

The layer thickness is an important parameter in multilayer systems with a nanometer period. In this work, the deposition regimes made it possible to obtain coatings with the lowest layer thickness of about 8 nm (using the continuous rotation mode of the substrate holder during deposition) [15]. In cases of the stationary position of the substrate holder, the layer thickness was determined by the deposition time. For the arc parameters used on metal targets, the average deposition rate was 2 nm/s. With the smallest average layer thickness (about 8 nm, continuous rotation of the substrate holder), the resulting multi-period coatings can have superlattice properties (inheritance of the type and orientation of crystal lattices in adjacent layers). According to X-ray fluorescent analysis in coatings obtained with continuous rotation of the substrate holder, the average ratio of metal atoms Nb/Cu $\approx 60/40$. This may be due to different arc currents for Nb and Cu (Table 1).

The diffraction spectra of coatings deposited during continuous rotation of the substrate holder are shown in Fig. 1.

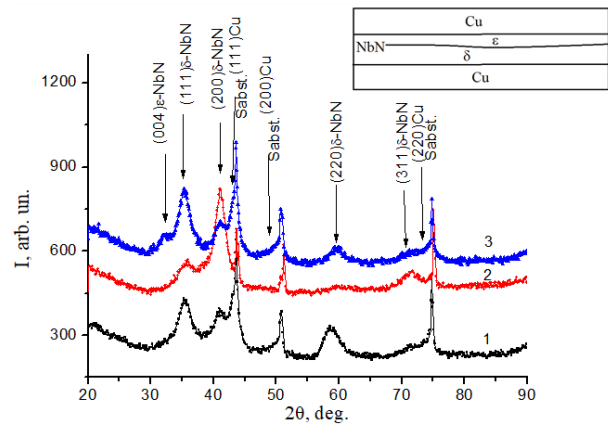


Fig. 1 – XRD patterns of the NbN/Cu multilayer (with a layer thickness of about 8 nm) coatings obtained under different technological conditions: 1 – $P_N = 7 \cdot 10^{-4}$ Torr, $U_b = -50$ V, 2 – $P_N = 7 \cdot 10^{-4}$ Torr, $U_b = -200$ V and 3 – $P_N = 3 \cdot 10^{-3}$ Torr, $U_b = -50$ V

As seen, at a relatively low pressure of $7 \cdot 10^{-4}$ Torr, a metastable δ -NbN phase with an fcc lattice (with a single-type lattice compared to Cu layers) is formed.

In this case, at a small value of $U_b = -50$ V, all diffraction peaks from the δ -NbN lattice planes of the phase are detected. The ratio of the peaks is close to the standard for the fcc lattice (NaCl structural type). This indicates the absence of a pronounced texture. An

increase in the bias potential U_b to -200 V leads to the appearance of the axis [100] of the preferred orientation of crystallite growth (compare spectra 1 and 2 in Fig. 1).

In coatings obtained at a high pressure $P_N = 3 \cdot 10^{-3}$ Torr, a qualitative change occurs in the diffraction spectra. In addition to the formation of the axis of [111] preferred orientation, a diffraction peak from the equilibrium ε -NbN phase with a hexagonal lattice is detected. The appearance of this phase indicates that the deformation factor (associated with the growth of structures with isotypic fcc lattices in adjacent layers) exceeds a critical value. In this case, the tendency of the system to the minimum of free energy stimulates the growth of the equilibrium ε -NbN phase. A schematic view of such a composition is presented in the upper right corner of Fig. 1.

We now consider the second extreme case – the coatings with the thickest layers. These coatings were obtained at deposition times of 120 seconds and 300 seconds (respectively, for Cu and Nb, the layer thickness is about 240 nm and 600 nm). According to X-ray fluorescent analysis, the ratio of metallic elements in the coating is Nb/Cu $\approx 72/28$.

As can be seen from Fig. 2, the equilibrium ε -NbN phase is formed in the nitride layers. Moreover, this state is single-phase for different U_b . The only difference is in different types of preferred orientation of crystallites. In the case of small $U_b = -50$ V, a pronounced texture axis is not detected. At that time, as with large $U_b = -200$ V, the appearance of a texture with the (110) plane parallel to the growth surface is seen (Fig. 2, spectrum 2).

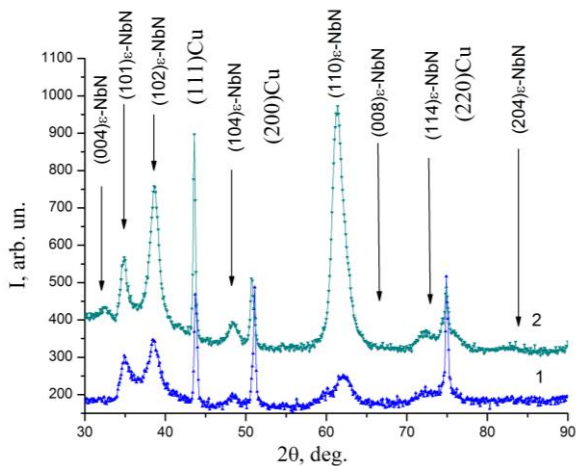


Fig. 2 – XRD patterns of the NbN/Cu multilayer coatings with the greatest layer thickness. 1 – $U_b = -50$ V, 2 – $U_b = -200$ V

The next stage of structural engineering is based on the possibilities of changing the structural state at one pressure. First, we consider the effect on the structural-phase state of the deposition parameters at low $P_N = 7 \cdot 10^{-4}$ Torr (Fig. 3).

For these conditions, we consider the systematization of the two main parameters: U_b and layer thickness. The first series (at $U_b = -50$ V) includes coatings with diffraction spectra 1, 3, and 5 in Fig. 3. The second series (at $U_b = -200$ V) includes coatings with

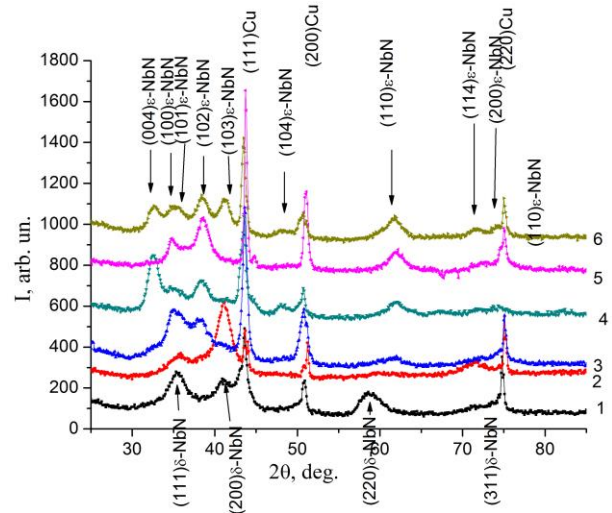


Fig. 3 – XRD patterns of the NbN/Cu multilayer coatings obtained at $P_N = 7 \cdot 10^{-4}$ Torr: 1. $U_b = -50$ V, continuous rotation, 2. $U_b = -200$ V, continuous rotation, 3. $U_b = -50$ V, mode 20/20, 4. $U_b = -200$ V, mode 20/20, 5. $U_b = -50$ V, mode 60/60, 6. $U_b = -200$ V, mode 60/60

diffraction spectra 2, 4 and 6 in Fig. 3. It can be seen that for coatings with the thinnest layers (spectrum 1), a metastable δ -NbN phase with a cubic lattice is formed. An increase in the layer thickness to 40 nm (spectrum 3) leads to the formation of only the equilibrium ε -NbN phase. The formation of this phase is also inherent for the greater layer thickness (about 120 nm, spectrum 5). For the second series (in the coatings obtained at $U_b = -200$ V), a similar change in the phase composition appears. The main difference is the formation of the preferred orientation of crystallite growth. At the smallest layer thickness (about 8 nm), for the δ -NbN phase, a preferred orientation is observed with the [100] axis perpendicular to the growth plane. For thicker layers, in which the ε -NbN phase is formed, it is possible to distinguish a preferred orientation with the (004) plane parallel to the growth plane (at a layer thickness of about 40 nm) and a full spectrum of diffraction peaks without noticeable preferred orientation – at a maximum layer thickness of about 120 nm.

At high pressure $P_N = 3 \cdot 10^{-3}$ Torr (Fig. 4), the first series (at $U_b = -50$ V) includes coatings with diffraction spectra 1, 2, and 4 in Fig. 4. It can be seen that the trend to the δ -NbN \rightarrow ε -NbN transition with an increase in the layer thickness of more than 20 nm is also preserved for this series.

For the second series (obtained at $U_b = -200$ V), the ε -NbN phase is formed in thick layers. However, in this case, parallel to the growth surface of the coating, a preferred growth orientation with the (004) plane (at a layer thickness of about 120 nm, 60/60 mode) and the (110) plane (at the greatest layer thickness, spectrum 5 in Fig. 4) appears.

4. RESULTS AND DISCUSSION

In the course of the research, it was found that two types of phases of niobium nitride can be formed in the layers: a high-temperature metastable δ -NbN phase

(NaCl structural type) and ε -NbN phase equilibrium at room temperature with a hexagonal lattice [16]. The arrangement of atoms in the lattice sites of these phases is shown in Fig. 5.

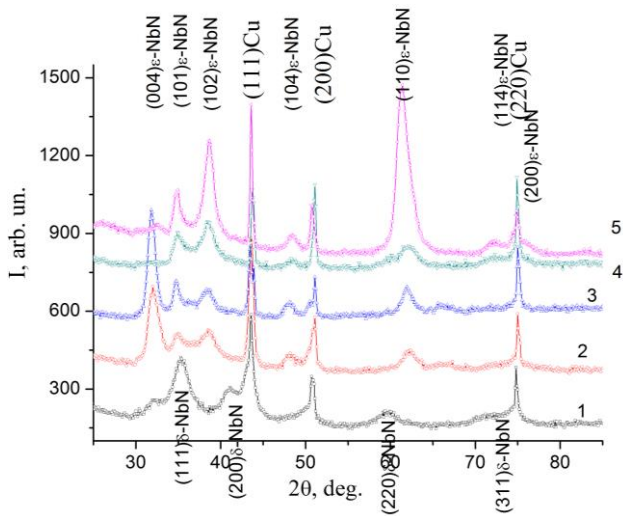


Fig. 4 – XRD patterns of the NbN/Cu multilayer coatings obtained at $P_N = 3 \cdot 10^{-3}$ Torr: 1. $U_b = -50$ V, continuous rotation, 2. $U_b = -50$ V, mode 20/20, 3. $U_b = -200$ V, mode 60/60, 4. $U_b = -50$ V, mode 120/300, 5. $U_b = -200$ V, mode 120/300

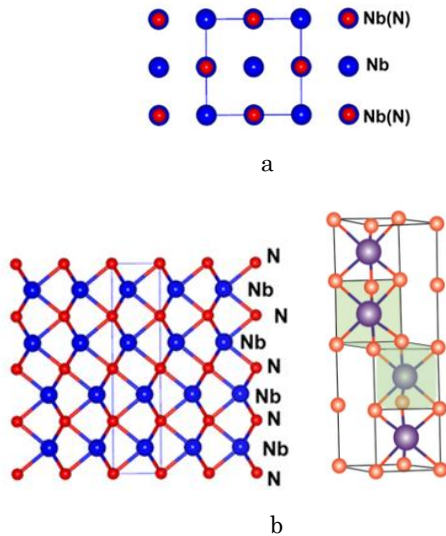


Fig. 5 – The arrangement of Nb and N atoms in the side section of the δ -NbN (a) and ε -NbN (b) phase crystal lattices

The formation of a metastable δ -NbN phase in multilayer systems was previously identified in [17] for the NbN/SiNx system. According to the data of this work, the highest hardness of 32 GPa was achieved with the smallest thickness of 0.4 nm. For the formation conditions of niobium nitride close to equilibrium, the formation of β -Nb₂N and δ -NbN phases (depending on the nitrogen content) has been established. The crystallites of these phases have a hexagonal lattice [18]. Note that the δ -NbN and ε -NbN phases have similar crystal lattices and differ in the dimensional characteristics of these lattices [19]. In addition, the ε -NbN phase has good superconducting properties.

Based on the results obtained in this work, it is possible to stabilize the δ -NbN and ε -NbN phases by changing the layer thickness in NbN/Cu multilayer coatings. In this case, the single-phase δ -NbN state is formed in thin layers (about 8 nm) only at low pressure $P_N = 7 \cdot 10^{-4}$ Torr (Fig. 1 and Fig. 3). In this case, an increase in the potential U_b to -200 V leads to the formation of the [100] texture.

As the pressure increases to $P_N = 3 \cdot 10^{-3}$ Torr, a two-phase state of the δ -NbN and ε -NbN phases appears in the niobium nitride layers (spectrum 3 in Fig. 1). Also, as the pressure increases (which is accompanied by an increase in the nitrogen content in the layers), the [111] texture is formed in the crystallites of the δ -NbN phase.

It should be noted that the difference in the coating deposition at low pressure is the long mean free path of particles without collision. Thus decreases the energy loss of the particles during their transfer from the cathode to the substrate. Therefore, the formation of δ -NbN can be considered to be associated with a higher resistance of materials with this type of crystal lattice (NaCl structural type) to radiation-stimulated defect formation under the action of high-energy ions.

We now consider the coatings with the greatest thickness (more than 250 nm). As the analysis of the diffraction spectra in Fig. 2 shows, in this case, the formation of the single-phase ε -NbN state occurs. With this increase in U_b to -200 V, a texture with the (110) plane parallel to the growth plane is formed. When the layer thickness of niobium nitride is 40-120 nm (middle interval), the formation of the ε -NbN phase also occurs. In this case, an increase in U_b to -200 V leads to the appearance of a texture with the (004) plane parallel to the growth surface (Fig. 4). Earlier in work [20], for the magnetron method of obtaining coatings, it was also established that an increase in U_b does not change the phase composition, but leads to a change in the texture axis.

5. CONCLUSIONS

1. It was established that with an increase in the layer thickness of niobium nitride from 8 to 40 nm (in NbN/Cu multilayer composition), the phase composition changes from the metastable δ -NbN (cubic crystal lattice, NaCl structural type) to the equilibrium ε -NbN phase with a hexagonal crystal lattice.

2. At low pressure $P_N = 7 \cdot 10^{-4}$ Torr in thin layers (about 8 nm thick), regardless of U_b , the δ -NbN phase is formed. The reason for the stabilization of this phase can be the same type of metallic fcc crystal lattice of the δ -NbN phase with the Cu crystal lattice.

3. As the pressure increases from $P_N = 7 \cdot 10^{-4}$ Torr to $3 \cdot 10^{-3}$ Torr, a more equilibrium ε -NbN phase with a hexagonal crystal lattice is formed. This manifests itself even in thin layers and can be associated with an increase in the stability of the ε -NbN phase with increasing nitrogen content in the coating.

4. When the layer thickness of niobium nitride is 40 nm and more, the ε -NbN phase is stabilized and a single-phase state is formed based on this phase. This single-phase state persists to the greatest layer thicknesses (about 600 nm). The copper layer thickness (in

the range of 40-240 nm) also does not affect the change in the phase composition.

5. An increase in the bias potential during deposition from -50 V to -200 V mainly affects the change in the preferred orientation of crystallite growth. In thin layers of the δ -NbN phase (about 8 nm), a crystallite texture with the [100] axis is formed. In layers

with a thickness of 40-120 nm, crystallites of the ε -NbN phase with the hexagonal (004) plane parallel to the growth plane are predominantly formed. At the greatest layer thickness (more than 250 nm), the ε -NbN phase crystallites are predominantly formed with the (110) hexagonal lattice plane parallel to the growth plane.

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Вплив товщини шарів і умов осадження на структурний стан NbN/Cu багат шарових покриттів

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Досліджено вплив основних фізичних та технологічних факторів структурного проектування (товщина шару, тиск атмосфери та потенціал відхилення) на структурно-фазовий стан оболонок NbN/Cu. Встановлено, що при збільшенні товщини шарів нітриду ніобію від 8 до 40 нм (в багат шаровій композиції NbN/Cu) відбувається зміна фазового складу від метастабільного δ -NbN (кубічна кристалічна решітка, структурний тип NaCl) до рівноважної ε -NbN фази з гексагональною кристалічною решіткою. При низькому тиску $P_N = 7 \cdot 10^{-4}$ Торр в тонких шарах (товщиною близько 8 нм) незалежно від U_b відбувається формування δ -NbN фази. Причиною стабілізації цієї фази може бути однотипність металеві ГЦК кристалічної решітки δ -NbN фази з кристалічною решіткою Cu. При збільшенні тиску від $P_N = 7 \cdot 10^{-4}$ Торр до $3 \cdot 10^{-3}$ Торр відбувається утворення більш рівноважної ε -NbN фази з гексагональною кристалічною решіткою. Збільшення потенціалу зміщення при осадженні від -50 В до -200 В в основному впливає на зміну переважної орієнтації зростання кристалітів. В тонких шарах δ -NbN фази (близько 8 нм) формується текстура кристалітів з віссю [100]. У шарах товщиною 40-120 нм переважно формуються кристаліти ε -NbN фази з площиною гексагональної решітки (004) паралельною площині зростання. При найбільшій товщині шарів (більше 250 нм) відбувається переважне формування кристалітів ε -NbN фази з площиною гексагональної решітки (110) паралельною площині зростання. Отримані результати показують великі можливості структурної інженерії в нітриді ніобію при його використанні в якості складового шару багат шарової періодичної системи NbN/Cu.

Ключові слова: Вакуумна дуга, NbN/Cu, Період, Потенціал зміщення, Фазовий склад, Структура, Твердий розчин.