The research of structural parameters, mechanical properties and efficiency of the cutting tool with multilayer coatings based on niobium nitride

V Tabakov¹, A Chikhranov¹²*, Y Dolzhenko² and S Vlasov³

¹Ulyanovsk State Technical University, 432027, Ulyanovsk, Russian Federation
²Institute of Civil Aviation named after Chief Marshal of Aviation B.P. Bugaev, 432071, Ulyanovsk, Russian Federation
³Research Nuclear University MEPhI (Moscow Engineering Physics Institute), 115409, Moscow, Russian Federation

*chihranov@mail.ru

Abstract. The paper presents the results of studies phase composition, structure parameters, mechanical properties and crack resistance of multilayer coatings based on niobium nitride. The relationship of these characteristics with the coating design and the composition of functional layers is shown

1. Introduction
The use of wear-resistant coatings, including multi-layer coatings, allows to increase efficiency of cutting tools significantly [1–10]. At the same time, increasing the productivity of machining and durability of cutting tools makes it necessary to further research in searching of new compositions of wear-resistant coatings. However, the currently used single-layer and multi-layer coatings are based on modifications of titanium nitride, the possibilities of which are almost exhausted. In this regard, niobium nitride can be considered as one of the promising materials that can be used as the coating of the cutting tool. A few publications [11–16] show that niobium nitride differs in its mechanical and thermophysical properties for the better from titanium nitride, which suggests higher efficiency characteristics of multilayer coatings based on it. The purpose of this work is to study the phase composition, structure parameters, and mechanical properties of multilayer coatings based on niobium nitride.

2. Solution Methods
Coatings were deposited on a Bulat-6 machine on MK8 carbide plates manufactured by «MKTC-HERTEL» (Russia), HT110 by Mitsubishi Carbide (Japan) and 20X13 steel plates. The coating structure parameters (crystal lattice periods \(a\) and \(c\), X-ray diffraction line half-width \(\beta_{004}\)) and residual stresses \(\sigma_0\) were determined on the "ДРОН-3М" diffractometer. The relative microdeformation of the crystal lattice \(\Delta a/a\) and block sizes of coherent scattering regions (CSR) \(D\) were calculated were calculated using the method [17, 18]. The chemical composition of the coatings was determined by the method of quantitative X-ray spectral analysis on a MAP-4 facility taking into consideration ZAF corrections. Microhardness of coatings \(H_a\), the elastic modulus \(E\) and the stress intensity coefficient \(K_{1C}\) were determined using the method described in [19] on the Mitutoyo NH-125 microhardness tester using the Knupp pyramid. The adhesive strength of the coatings was estimated by the value of the adhesion
coefficient \( K_0 \), that was determined on a TK-2M hardness tester using the method [1]. The cyclic crack resistance of the coatings was determined by the method of [20].

3. Research Results and Discussion

Two-layer coatings with the top layers based on three-element nitrides were studied: NbN-NbTiMeN, where Me – Zr, Cr, and Al. The choice of two-layer architecture and composition of functional layers of coatings was made on the basis of recommendations of works [8, 9]. Coatings were deposited using niobium cathodes, composite titanium cathodes with zirconium and chromium plates, and titanium and aluminum alloy cathodes. The chemical composition of the top layers of two-layer coatings is presented in table 1.

Table 1. Chemical composition of coatings

| Top layer of coating | Ti   | Nb   | Zr   | Al   | Cr   |
|----------------------|------|------|------|------|------|
| NbTiZrN              | 46.5 | 41.9 | 11.6 | –    | –    |
| NbTiAlN              | 49.2 | 40.0 | –    | 10.8 | –    |
| NbTiCrN              | 43.8 | 48.1 | –    | –    | 8.1  |

Comparative analysis of diffractograms and phase composition of coatings NbN-NbTiZrN, NbN and NbTiZrN is shown in Figures 1, 2 and Tables 2, 3. As can be seen from Figure 1, a, niobium nitride has a hexagonal crystal lattice, as evidenced by the diffraction peak of maximum intensity at the angle 2\( \theta \), equal to 62 deg., corresponding to the crystal plane (110) of the hexagonal structure. The three-element coating NbTiZrN has a tetragonal structure. The diffraction peak, which characterizes the tetragonal structure of maximum intensity, is observed at an angle of 2\( \theta \) equal to 41.4 deg. (crystal plane 004) (Figure 1, b).

![Figure 1](image1.jpg)

**Figure 1.** The diffractograms of coatings NbN (based on 20X13) (a) and NbTiZrN (based on HTi10) (b).

Analysis of the diffractogram (Figure 2) and the phase composition of the two-layer coatings (Table 3) shows the presence of diffraction peaks on them, corresponding to both hexagonal and tetragonal structures. This is explained by the presence in the architecture of a two-layer coatings of the lower layers of NbN nitride with a hexagonal lattice, and the top layers based on three-element nitrides with a tetragonal lattice.
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As can be seen from the diffractograms, the diffraction peaks of the tetragonal structure have a higher intensity, which indicates the advantage of this structure for two-layer coatings. Furthermore, the offset of diffraction peaks of the hexagonal structure of maximum intensity towards smaller sliding angles than the NbN coating is typical for them. At the same time, the diffraction peaks of the tetragonal structure of maximum intensity for both single-layer and two-layer coatings are observed at approximately the same sliding angles. For example, for a single-layer NbN coating (Figure 1, a), the diffraction peak of the maximum intensity is located at an angle of $2\theta$ equal to 62 deg. (crystal plane 110), and for NbN-NbTiZrN – at an angle of 31.2 deg. (crystal plane 002). At the same time, there is also a diffraction peak of the hexagonal structure corresponding to the crystal plane 110 on the diffractogram of the two-layer coating, which is located almost at the same angle with The NbN coating $2\theta$ equal to 61.8 deg., but has a lower intensity. The diffraction peaks of the tetragonal structure of maximum intensity of the single-layer NbTiZrN coating (Figure 1, b) and the two-layer NbN-NbTiZrN coating (Figure 2) are located at almost identical angles of $2\theta$ equal to 41.4 deg. and 41.3 deg., respectively, and correspond to the same crystalline plane 004.

Analysis of single-layer and two-layer coatings diffractograms shows that two-layer coatings are characterized by a lower intensity of diffraction peaks of both hexagonal and tetragonal structures. This may be due to the smaller thickness of the functional layers of two-layer coatings in comparison with the thickness of single-layer coatings. The observed offset of the diffraction peaks of the hexagonal structure of maximum intensity towards smaller sliding angles can be also associated with a smaller thickness of the NbN layer in the two-layer coating. It can be assumed that a niobium nitride of hexagonal structure corresponding to crystal plane 002 or, in the case of large thicknesses (5…6 μm), to plane 110, is formed during the deposition of NbN layer with a small thicknesses (about 2…3 μm).

**Table 2.** Phase analysis of coating NbTiZrN (based on HTi10)

| Diffraction angle $2\theta$ (deg.) | Interplanar distance $d_{exp}$ (nm) | Relative intensity | HKL | Interplanar distance $d_{tab}$ (nm) | Coating phases | Substrate phases | Coating structure |
|----------------------------------|------------------------------------|-------------------|-----|------------------------------------|----------------|-----------------|------------------|
| 31.6                             | 0.28313                            | 0.35              | 001 | 0.2820                             | –              | WC              | –                |
| 35.1                             | 0.25566                            | 0.01              | 112 | 0.25236                            | NbN            | –               | tetragonal       |
| 35.7                             | 0.25150                            | 0.65              | 100 | 0.250                              | –              | WC              | –                |
| 41.4                             | 0.11809                            | 1.0               | 004 | 0.11708                            | NbN            | –               | tetragonal       |
| 48.4                             | 0.19906                            | 1.0               | 101 | 0.1870                             | –              | WC              | –                |
Table 3. Phase analysis of coating NbN-NbTiZrN (based on HTi10)

| Diffraction angle 2θ (deg) | Interplanar distance \( d_{exp} \) (nm) | Relative intensity | \( HKL \) | Interplanar distance \( d_{tab} \) (nm) | Coating phases | Substrate phases | Coating structure |
|---------------------------|-----------------------------------------|-------------------|--------|-----------------------------------|----------------|-----------------|-----------------|
| 31.2                      | 0.28667                                 | 1.0               | 002    | 0.2768                            | NbN            | –               | tetragonal       |
| 35.3                      | 0.25425                                 | 0.13              | 112    | 0.2524                            | NbN            | –               | tetragonal       |
| 41.3                      | 0.21860                                 | 1.0               | 004    | 0.2171                            | NbN            | –               | tetragonal       |
| 48.3                      | 0.18843                                 | 1.0               | 101    | 0.2870                            | –              | WC              | –               |
| 61.8                      | 0.15012                                 | 0.25              | 110    | 0.1484                            | NbN            | –               | hexagonal        |

Structure parameters depend on the coatings composition (table 4). A decrease in residual compressive stresses, half-widths of the X-ray line, an increase in the relative microdeformation of the crystal lattice and the sizes of CSR blocks are observed in the case of single-layer coatings when passing from a single-element coating NbN to a three-element NbTiZrN. The crystal lattice periods of single-layer three-element coatings and two-layer coatings with corresponding top layers have approximately the same values. Two-layer coatings NbN-NbTiCrN and NbN-NbTiAlN, deposited on different instrumental substrate, have higher values of the half-width of the X-ray line in comparison with three-element coatings NbTiCrN and NbTiAlN. The increase in the \( \beta_{004} \) value for two-layer coatings ranged from 9.6% to 44% depending on the composition of top layer and tool base. The exception was the NbN-NbTiZrN coating, for which the half-width of the X-ray line was practically unchanged or even smaller when deposited on hard-alloy plates compared to single-layer NbTiZrN coatings.

Table 4. Structural parameters and residual stresses of coatings (based on HTi10)

| Substrate | Coating       | \( a \) (nm) | \( c \) (nm) | \( D \) (nm) | \( \Delta a/a \times 10^3 \) | \( \beta_{004} \) (deg) | \( \sigma_0 \) (MPa) |
|-----------|---------------|--------------|--------------|-------------|-----------------------------|--------------------------|---------------------|
| 20X13     | NbN           | 0.2994       | 0.5594       | 9.2         | 8.1                         | 1.13*                    | -2893±94            |
|           | NbTiZrN       | 0.4402       | 0.8744       | 10          | 10.7                        | 0.95                     | -1236±161           |
|           | NbN-NbTiZrN   | 0.4438       | 0.8744       | 10          | 10.8                        | 0.95                     | -1901±380           |
|           | NbTiCrN       | 0.3089       | 0.5698       | 16          | 8.79                        | 0.6                      | -                   |
|           | NbN-NbTiCrN   | 0.3011       | 0.5422       | 9           | 15.1                        | 1.0                      | -3491±227           |
|           | NbTiAlN       | 0.4417       | 0.8684       | 13          | 7.95                        | 0.71                     | -5305±286**         |
|           | NbN-NbTiAlN   | 0.4386       | 0.8664       | 10          | 10.5                        | 0.92                     | -5658±117**         |
| MK8       | NbTiCrN       | 0.4392       | 0.8686       | 16          | 6.78                        | 0.61*                    | -                   |
|           | NbN-NbTiCrN   | 0.4356       | 0.8644       | 9           | 11.58                       | 1.03                     | -                   |
| HTi10     | NbTiZrN       | 0.4389       | 0.8724       | 11          | 9.78                        | 0.86                     | -543±230***         |
|           | NbN-NbTiZrN   | 0.4420       | 0.8744       | 12          | 8.8                         | 0.78                     | -347±93***          |

Note: * - measured at peak 110; ** - hexagonal structure; *** - macrostresses in a carbide base
Residual stresses were determined in multilayer coatings deposited on 20X13 steel plates. It should be noted that the residual stresses in multilayer coatings, even deposited on these substrates, were not always able to be measured due to the absence of corresponding peaks in the diffractograms.

It was found that two-layer coatings have higher values of residual compressive stresses in comparison with the corresponding single-layer three-element coatings (by 33% ... 53% depending on the coating). Higher compressive residual stresses in two-layer coatings compared to single-layer three-element coatings are explained by the presence of a lower NbN layer with high compressive residual stresses in them. For example, for the NbN-NbTiZrN coating deposited on 20X13 steel base, the residual compressive stresses measured from the diffraction peaks of the tetragonal structure are equal to 1901±380 MPa, and for single-layer NbTiZrN coatings 1236±161 MPa. Similar data were obtained for residual stresses measured by the peaks of the hexagonal structure. As can be seen from table 4, they are equal to −5305±286 MPa and to 5658±117 MPa, respectively, for NbTiAIN and NbN-NbTiAIN coatings deposited on a 20X13 base. It should be noted that the value of the residual stresses measured by the hexagonal structure significantly exceeds the stresses measured by the tetragonal structure. For example, for the NbTiAIN coating deposited on 20X13 steel plates, they are equal to −1828±124 MPa and to −5305±286 MPa, respectively.

Mechanical properties of multilayer coatings in comparison with single-layer coatings are presented in table 5.

**Table 5. Mechanical properties of coating (based on MK8)**

| Coating       | $H_a$ (GPa) | $E$ (GPa) | $H_a/E$ | $H_a^2/E^2$ (GPa) | $K_0$ | $K_{IC}$ (MPa-m$^{1/2}$) | $t$ (min) |
|---------------|-------------|-----------|---------|-------------------|-------|------------------------|----------|
| NbN           | 29.8        | 467       | 0.064   | 0.121             | 0.10  | 9.78                   | 18.85    |
| NbTiAIN       | 32.1        | 455       | 0.071   | 0.160             | 0.22  | 12.76                  | 28.87    |
| NbN-NbTiAIN   | 34.0        | 463       | 0.073   | 0.183             | 0.15  | 13.54                  | 46.42    |
| NbTiZrN       | 32.9        | 496       | 0.066   | 0.145             | 0.29  | 11.84                  | 27.12    |
| NbN-NbTiZrN   | 34.6        | 477       | 0.073   | 0.182             | 0.16  | 12.62                  | 43.27    |
| NbTiCrN       | 33.4        | 448       | 0.075   | 0.186             | 0.26  | 12.69                  | 26.41    |
| NbN-NbTiCrN   | 34.9        | 461       | 0.076   | 0.200             | 0.16  | 13.47                  | 40.28    |

It was established that the microhardness $H_a$, the elastic modulus $E$, the resistance of coatings to abrasive wear $H_a/E$ and plastic deformation $H_a^2/E^2$, accordingly, as well as the stress intensity coefficient of two-layer coatings, practically do not differ from the corresponding values of single-layer coatings, which are the top layers of two-layer coatings. At the same time, the stress intensity coefficient of two-layer coatings is higher by 29...38%, depending on the composition of the top layer of multilayer coatings compared to NbN coating.

As can be seen from table 5, three-element coatings have a lower adhesive strength with a solid-alloy base compared to NbN coatings. This is evidenced by their higher adhesion coefficient values, which are 1.6 times higher compared to NbN coatings. Multi-layer coatings have higher adhesion strength compared to single-layer coatings used as top layers. The adhesion coefficient of these coatings is less by 32 ...45 %, depending on the composition of the top layer.

A strong correlation between the time of cyclic crack resistance $t$ and the efficiency of a cutting tool with single- and multi-layer coatings is shown in [9, 14, 20]. Therefore, the assessment of the efficiency of the cutting tool with coatings was carried out by the value of the cyclic crack resistance in this paper. It was found due to research that the time of cyclic crack resistance of two-layer coatings compared to single-layer three-element coatings is 1.5...1.7 times higher, and compared to NbN it is 1.5...2.5 times higher, depending on the composition of the upper layers of coatings.
4. Inference
Analysis of the structure parameters and mechanical properties of multilayer coatings based on niobium nitride allows us to conclude about the effectiveness of their use as coatings for cutting tools in order to improve their efficiency.

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