Investigation of Structure of Coatings Based on NbN by Methods of Scanning Electron Microscopy and X-Ray Diffraction Analysis

D.S. Vershinin, M.B. Ivanov, Yu.R. Kolobov, O.I. Klykov and T.N. Vershinina

Center of NanoStructured Materials and Coatings of BSU, Pobedy St.,Belgorod,308015, Russia, Phone:+7(4722) 30-11-92, Fax:+7(4722) 30-11-92, E-mail: Vershinin@bsu.edu.ru

Abstract – The results of experiments on deposition of niobium, its nitride and multilayer protective composite coatings based on indicated compounds by method of condensation with ion bombardment on titanium alloy VT-20 samples are presented. At the same time the preliminary cleaning of the samples in glow discharge in Ar atmosphere was made. The usage of ion assistance during deposition allows to widely modify chemical and phase composition of coatings which satisfy to conditions of limited thickness, uniformity and adhesion. The obtained coatings were investigated by methods of scanning electron microscopy and X-ray diffraction analysis. It is shown that realization of metal-nitride multilayer coating allows finally to remove technological defects of coating. Thus, the main problem of decreasing of coating defectiveness was solved – the microdroplet fraction was minimized or fully excluded, and rough dendrite structure was shattered. At the same time the phase composition of coatings corresponds to required composition and conforms with state diagram of metal-nitride.

1. Introduction

It is known that niobium and its combinations had found wide application in many branches of industry (chemical, atomic, engineering industry etc.) not only as alloying elements but as coatings. In particular, the coatings based on NbN have high wear-resistance, high-temperature strength and high transition temperature to superconductive condition [1].

This work presents the results of investigation of NbN based coatings produced with vacuum arc usage. The coatings were deposited onto alloy VT20. Depending of experiment the composition of coating was Nb, NbN or their alternation for obtaining multilayer structure.

2. Materials and methods of investigation

Topographical investigations of coatings quality were made by the method of scanning electron microscopy.

The scanning electron microscope Philips SEM 515 with accelerating voltage 30 kV was used. Local qualitative element analysis was made with usage of adapter to scanning electron microscope EDAX Econ IV for energy-dispersion microanalysis.

Investigations of phase composition of NbN coatings obtained by ion-plasma deposition method were carried out with X-ray diffractometer XRD-6000 Shimadzu on Cu-K_α irradiation in grazing X-ray diffraction regime with angle α= 7°, range 2θ=25÷90 (NbN), velocity 1°/min and step 0,02°. A computer program PowderCell was used for diffractograms interpretation. The program allows to perform qualitative and quantitative phase analysis; to calculate grids parameters; to define a degree of preferred orientation, values of areas of coherent scattering (ACS) and elastic microstresses (ε).

3. The method of coatings deposition

Deposition of Nb coatings and multilayer Nb-NbN-Nb-NbN-Nb coatings was done by method condensation with ion bombardment on standard installation NNV-6.6-I1. The installation was equipped by original ion source based on glow discharge with hollow cathode. The source was used for preliminary cleaning of samples by Ar^+ and N^+ ions before deposition, for increasing of ionization degree of reactive gas (in our case – nitrogen) and for ion-beam assistance during deposition process.

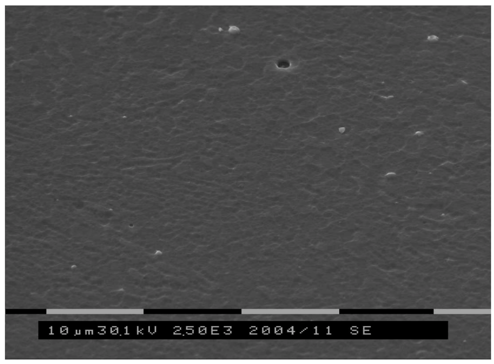

Fig. 1. Surface relief of NbN coating. Scanning electron microscopy

4. Results of investigations

The coatings of chemical composition of Nb and multilayer coatings based on Nb and NbN are characterized by practically total lack of discontinuity flaws caused by microdroplet fraction of vacuum-arc deposition (Fig. 1).

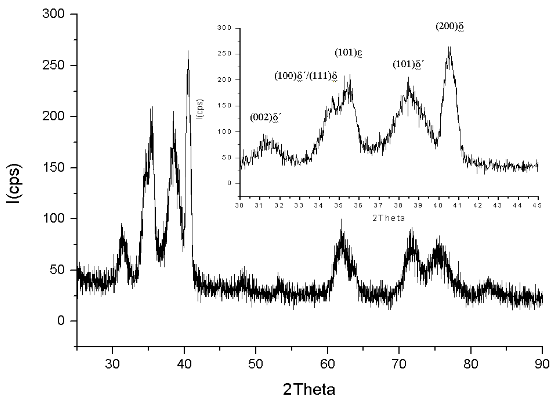

Fig.2. Diffractogram of NbN coating

X-ray-diffraction investigations have shown that coatings based on NbN are characterized by low size of ACS and high level of microstresses (ε) (Fig. 2). Phase composition of NbN coatings is represented by three main components: NbN (δ) (space group $Fm\overline{3}m$), NbN (ε) (space group $P6_3/mmc$) and NbN (δ') (space group $P6_3/mmc$), their quantitative correlation, grid parameters and ACS are presented for two regimes in Table I.

Table 1. Parameters and bulk fractions of phases of NbN coating

Phase	Bulk fraction, %	Lattice parameters, Å		Texture		ACD, nm	ε, E
		a	c	(hkl)	degree, %		
NbN (δ)	32,7	4,3507		(001)	54	10	$5\cdot10^{-3}$
NbN (ε)	7,5	2,9186	11,1700	(101)	77	26	$1\cdot10^{-3}$
NbN (δ')	59,8	2,9176	5,5500	(101)	30	10	$5\cdot10^{-3}$

It is necessary to note that δ'-phase appears during the process of δ-phase decay at low temperatures. Formation of δ-phase could be consequence of its formation directly from plasma. But more convincing version is the stabilization of high-temperature δ-phase of niobium nitride by soluted oxygen and carbon that could be both in initial cathode material and in residual vacuum of installation with diffusion pumping. As niobium carbide NbC and oxide NbO have fcc lattice, their solubility in hcp phases is low, hence, the stabilization of δ-phase of NbN is probable.

In general the phase conditions in system Nb-N formed by deposition method with ion bombardment are conformed with results of well-known investigations both Russian and foreign.

Analysis of fractured surface of samples with multilayer coating has shown that total thickness of coating is up to 5 μm (Fig. 3). The coating is charactèrizes by very good adhesion.

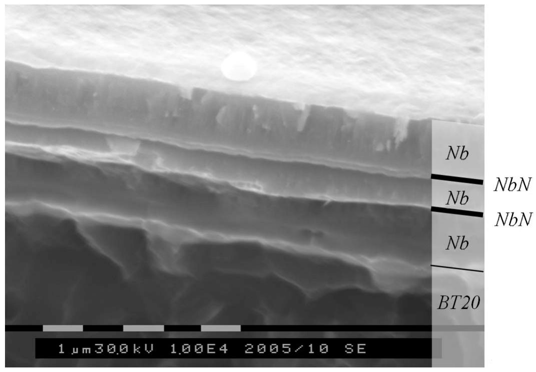

Fig. 3. The view of fractured surface of multilayer coating Nb-NbN-Nb-NbN-Nb. Scanning electron microscopy

5. Conclusion

Thus, the vacuum-arc nitride coatings based on niobium (including multilayer structure coatings) were produced. The coatings have very good adhesion to titanium alloy VT20 substrate. Main problem of decreasing of coatings defectiveness: minimized or fully excluded microdroplet fraction and shattered rough dendrite structure. At the same time the phase composition of coatings is corresponded to required composition and agreed with equilibrium metal-nitrogen diagrams [2].

References

[1] A.N. Zelikman, B.G. Korshunov, *Metallurgy of rare metals: Handbook*. M.: Metallurgija, 1991. – 86–124 pp.

[2] *Equilibrium diagrams of binary metallic systems: Handbook: In 3 vol.: Vol. 3.Book.1.*/Edited by N.P. Lyakishev. – M: Mashinostroenie, 2000 – 872 p.