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Investigation of structure and phase formation in multilayer coatings and their thermal stability

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A B S T R A C T

Multilayer coatings were deposited on carbide cutting inserts with an Arc-PVD method using a cathodic vacuum arc evaporation apparatus equipped with three disposed flat arc cathodes (Ti–Al, Zr–Nb and Cr). The samples were obtained under different deposition conditions: an arc current at the Zr–Nb cathode, a rotation speed of the samples coated relative to evaporated cathodes and a substrate bias. The mechanism of formation of the structure and phases in the material of the Ti–Al–N/Zr–Nb–N/Cr–N coating are established depending on the parameters of cathodic vacuum arc ion-plasma evaporation. It is established that the introduction of an additional Zr–N-based barrier layer to a multilayer nanostructure between the mutually soluble Ti–Al–N and Cr–N layers results in the enhancement of its thermal stability. The coatings are characterized by high hardness, Young’s modulus and the work of plastic deformation.

1. Introduction

Ti–N/Cr–N multilayer coatings have been thoroughly studied and are successfully used in industry to prevent premature wear of cutting tools due to the combination of high physicomechanical and tribological properties [1,2]. Meanwhile, a significant deterioration in their properties is observed at high temperatures that may be attained in the process of cutting, which is associated with low thermal stability of these coatings [3].

In this respect, the enhancement of thermal stability of the multilayer structure of these coatings is an important task. On the basis of the analysis of state diagrams of multicomponent systems consisting of refractory metal nitrides, it was ascertained that ZrN nitride is characterized by limited solubility in TiN and CrN at temperatures ~1580 °C and ~1430 °C, respectively. The use of ZrN as a barrier layer between the mutually soluble TiN and CrN may have a significant effect on diffusion processes occurring at the interlayer boundaries.

This work was aimed at studying structure and phase formation in Ti–Al–N/Cr–N multilayer coating in the presence of an additional Zr–N barrier layer and investigating its effect on thermal stability of Ti–Al–N/Cr–N multilayer coating.

2. Experimental

Coatings were deposited using a Bulat-NNV 6.6-I1 cathodic vacuum arc evaporation apparatus. We used a three-cathode evaporation system with Ti–Al [VT5 (Ti–Al) alloy], Zr–Nb (composed by the zirconium host with a niobium insertion at the area ratio Zr: Nb = 3: 1), and Cr cathodes. Prior to loading into the coating chamber, the substrates were initially cleaned in an ethanol bath. The chamber was pumped down to a base pressure of 3×10^{-3} Pa. Argon ion etching of the substrate was carried out at a pressure of 5 Pa and bias voltage of −1 kV followed by metal Ti ion etching at a bias voltage of −800 V. The coatings were deposited using nitrogen as the reaction gas. The varied parameters of the process were the rotation speed of the samples coated relative to evaporated cathodes (n), the current (I_{ZrNb}) of the evaporation arc on the zirconium-niobium cathode, and negative electrical bias (U_b) supplied to the substrate, which varied from 1 to 3 rpm, from 135 to 170 A, and from −80 to −160 V, respectively. The deposition time of the coatings was approximately 60 min. Replaceable polyhedral carbide inserts of VK6 (WC-Co) and TT10K8B (WC-TiC-Co) grades were used as substrates.

The thickness of the deposited coatings was controlled by the deposition time; it was approximately 5 μm.

The phase composition and the substructure of coatings were investigated by the X-ray diffraction method using a DRON-4 diffractometer. Recording was performed in CoK_α radiation applying the graphite monochromator.

The chemical composition and the bond energy of coating phases were investigated by X-ray photoelectron spectrometry (XPS) using a PHI 5500 spectrometer (Physical Electronics, United States). The level-by-level distribution of elements in the coating was investigated during sputtering on the surface by argon ions with energy of 2 keV.

The structure of coatings was studied using a Quanta FEG 3D high-resolution multifunctional scanning electron microscope (SEM) (FEI, The Netherlands) at an accelerating voltage $U = 20$ kV and by transmission electron microscopy (TEM) using a JEM-2100 device (JEOL, Japan).

The hardness (H), the Young’s modulus (E), and work of plastic deformation (W_p) of coatings were determined by indentation under small loads according to the Oliver and Farr technique, which was carried out on a Micro-Hardness Tester

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Table 1
Chemical composition of multilayer coatings.

Deposition parameters		Element content in the multilayer coating, at.%					
I_{ZrNb}, A	U_b, V	Ti	Al	Cr	Zr	Nb	N
135	−80	17	2	26	7	1	47
135	−100	17	2	26	7	1	47
135	−120	17	2	27	8	1	45
170	−120	16	2	25	12	2	43
135	−160	17	1	28	9	2	43

microindentometer (CSM Instruments, Switzerland) under a fixed load of 300 mN. The indenter penetration depth was controlled in such a manner so as to be smaller than 10–20% of the coating thickness to eliminate any effects of the substrate.

3. Results and discussion

3.1. The element composition of coatings

It was demonstrated via investigation of the element composition of the deposited multilayer coatings that they contained 25–28 at.% of Cr, 16–17 at.% of Ti, 7–12 at.% of Zr, 43–47% of N, 1–2 at.% of Al, and 1–2 at.% of Nb, depending on deposition parameters (Table 1).

An increase in the current of the evaporation arc on the zirconium–niobium cathode from 135 to 170 A resulted in a rise in niobium and zirconium contents in the multilayer coating from 1, 7 to 2, 12%, respectively. The concentrations of the other elements decreased in a corresponding manner. The rise in the bias potential supplied to the substrate from −80 to −160 V, resulting in an increase in energy of evaporated ions, caused a decrease in nitrogen content in the multilayer coating from 47 to 43 at.% due to the fact that being the lightest element within the multilayer coating, nitrogen was preferentially sputtered by heavy metal ions.

3.2. The phase composition and texture of coatings

The presence of ZrN, TiN and Cr_2N phases within the multilayer coating was ascertained via X-ray diffraction analysis. One can assume that the absence of aluminum and niobium phases within the multilayer coating is a result of their dissolution in ZrN and TiN nitrides yielding the corresponding solid solutions.

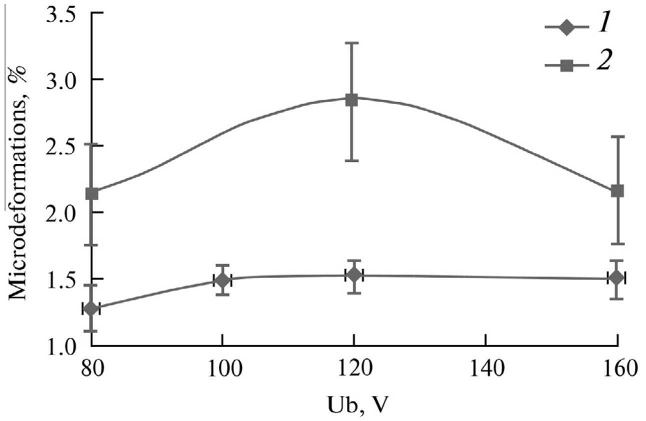

Fig. 2. Microdeformations of the crystal lattice of the phases of ZrN- (1) and TiN-based (2) multilayer coatings as a function of the bias potential on the substrate.

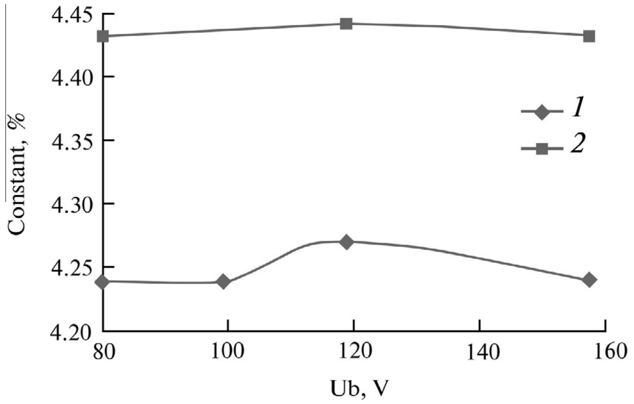

Fig. 3. The period of the crystal lattice of the phases of ZrN- (1) and TiN-based (2) multilayer coatings as a function of the bias potential on the substrate.

Additional information on the phase composition of multilayer coatings was found from an analysis of the high-resolution photoelectron spectra for the Cr2p, Zr3d and Ti2p electrons, which are shown in Fig. 1.

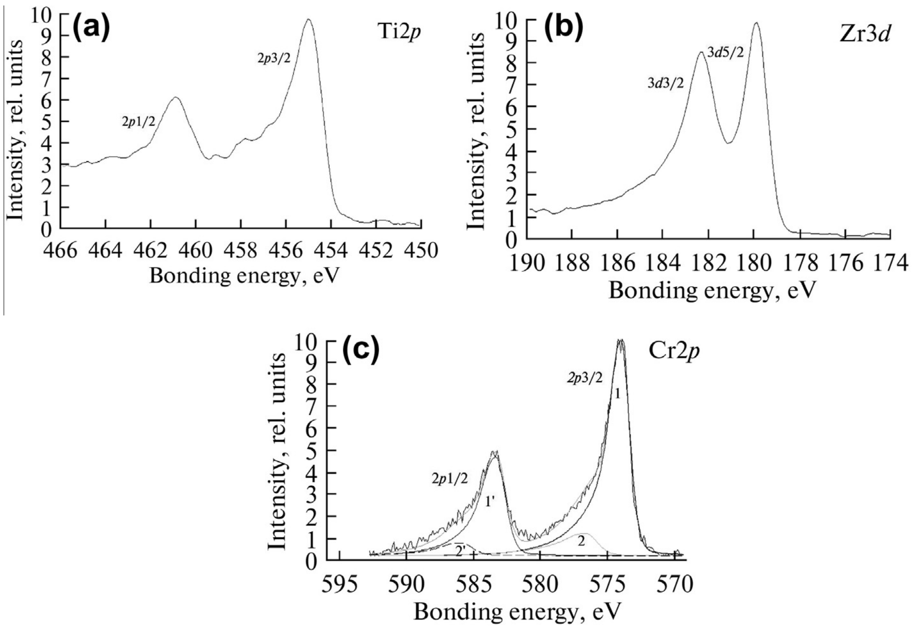

Fig. 1. High-resolution photoelectron spectra for (a) Ti 2p, (b) Zr 3d (b), and (c) Cr 2p electrons of a multilayer coating.