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Properties of niobium nitride coatings deposited by cathodic arc physical vapor deposition

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Abstract

Thin films of niobium nitride were deposited on high speed steel substrate by cathodic arc physical vapor deposition using −150 V, −200 V and −250 V bias voltages at 1 Pa nitrogen pressure. X-ray diffraction data showed that hexagonal δ-niobium nitride was formed for all bias voltages. Fracture cross-sections revealed the existence of the transition zone structure in all coatings. The maximum hardness was found to be 39 GPa. The Rockwell C adhesion and the scratch test were used to compare adhesion properties. In Rockwell adhesion test the best adhesion was obtained at −150 V bias voltage and in scratch test the cracks were occurred approximately at 3–5 Ns.

Keywords: Niobium nitride; Cathodic arc physical vapor deposition; Microstructure; Mechanical properties; Hardness; Adhesion

1. Introduction

Transition metal nitrides and carbides exhibit an attractive mixture of physical, chemical and mechanical properties which make them promising candidates for many technical applications. Especially as thin films, transition metal nitrides on metals are well-suited for improving the performance of cutting tools and drills due to their high hardness and wear resistance. Among nitride coatings, niobium nitride (NbN) and NbN-based coatings are of increasing interest because of their high hardness, wear resistance and superconducting properties. NbN is well-known for its superconductive behavior (superconducting transition temperature is approximately 17 K) so NbN films could be used in several superconducting microelectronic applications. In industrial applications, NbN films are also preferred for their good thermal expansion which matches well with commonly used tool steels. They are also used as a layer in super lattice coatings such as, chromium nitride/niobium nitride (CrN/NbN), titanium nitride/niobium nitride (TiN/NbN) which exhibit very high hardness because of the individual layer materials in the coating [1–6].

Several methods such as reactive magnetron sputtering technique [6–12], ion beam assisted processes [13–15], unbalanced magnetron [16], pulsed laser deposition [17] and vacuum arc deposition [1–3,18] are used for producing NbN thin films. Experimental data shows that the structure and the properties of the NbN coatings are influenced by the deposition process and the process parameters. However, compared to other techniques, there is only limited information on NbN films produced by vacuum arc deposition. Zhitomirsky et al. [1,2] deposited NbN coatings on cemented carbide bars using vacuum arc deposition with a bias voltage of −40 V at different nitrogen pressures. They reported that the chemical composition and mechanical properties of NbN coatings were affected by the nitrogen pressure and a maximum microhardness of 42 GPa for mixed phases (cubic δ-NbN and hexagonal β-Nb_2N). Bendavid et al. [3] reported that the properties of NbN coatings prepared by filtered arc deposition were affected by the nitrogen pressure and bias voltage (V_{bias}). The maximum hardness was found to be 48.5 GPa at a substrate bias of approximately −100 V and 0.65 Pa nitrogen pressure. Rutherford et al. [18] studied the influence of V_{bias} at 2 Pa nitrogen pressure on the phase composition of vacuum arc deposited coatings and reported that V_{bias} influenced not only on the phase composition but also the texture.

In previous experimental studies the maximum bias voltage used for arc deposition system was reported as −200 V. The aim of this study is to investigate the effect of bias voltage on microstructure and mechanical properties of NbN thin films

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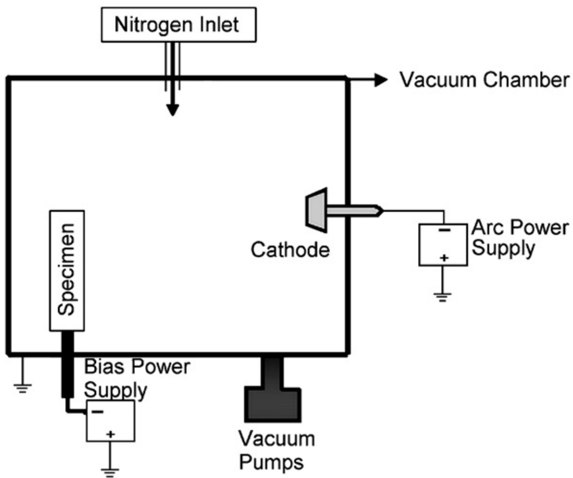

Fig. 1. Schematic representation of vacuum arc deposition system.

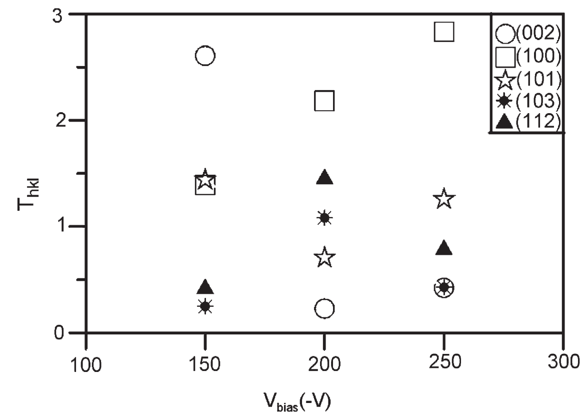

Fig. 3. Texture coefficients of δ-NbN as a function of bias voltages.

deposited on High Speed Steel (HSS) substrate by vacuum arc physical vapor deposition (PVD) using bias voltages of −150, −200 and −250 V at 1 Pa nitrogen pressure.

2. Experimental details

2.1. Coating procedure

20 × 30 × 6 mm HSS samples were used as substrate material. The surfaces of the samples were prepared by standard metallographic techniques. The final polishing was done with 1 μm diamond paste. Before the coating procedure, the specimens were degreased ultrasonically in acetone and alcohol and then fixed on a stationary holder opposite to the cathode in a Novatec-SIE, Model:NVT-12 coating system. Cathodic arc system used in this study is represented in Fig. 1. Pure Nb metal (99.9%) was used as the target material. In order to improve the substrate-coating adhesion, the substrate was bombarded (−1000 V) with accelerated Nb ions and as a result, the substrate temperature raised to ~573–623 K. Other parameters, such as the nitrogen pressure (1 Pa), arc current for evaporation of Nb cathode (65 A) and deposition time (1500 s) were kept constant. Bias voltages, −150 V, −200 V, −250 V, were applied to the substrate during the deposition process.

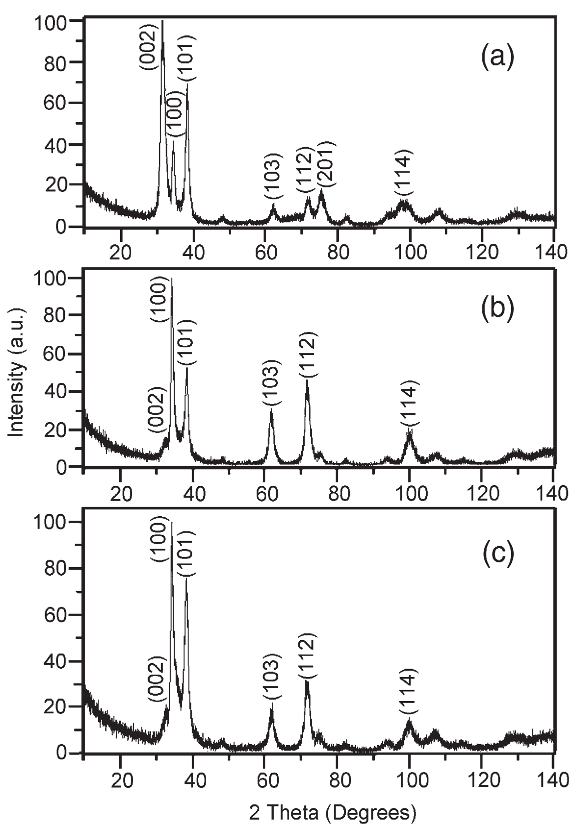

Fig. 2. XRD spectra of NbN coatings at substrate bias voltage of a) −150 V, b) −200 V and c) −250 V.

2.2. Characterization of NbN coatings

The phase structure of the coatings was analyzed by X-ray diffractometer (XRD) equipped with a thin film attachment (Philips Model PW3710) using Cu Kα radiation. Phases in the film were identified by matching the diffraction peaks with those of JCPDS card [19]. The degree of preferred orientation was calculated by the Harris texture coefficients (T_{hkl}) using the peak intensities in the XRD spectra [20]. For a given orientation, the greater T_{hkl} values indicate a higher frequency of occurrence compared to random orientation where the coefficients were in unity.

The morphology of the coatings deposited on mild steel samples were investigated by examining the cross-section view of the fracture with a JEOL JSM-6335F Field Emission Scanning Electron Microscopy (SEM). The thickness of the coatings was determined by ball-cratering (Calotest) using a steel ball with of 20 mm diameter.

The hardness and Young modulus were determined with a nanoindentation (CSM NHT, SN:06-0177) unit. Hardness values were taken from about 200 nm indentation depth in order to avoid influence of the surface roughness and the substrate. At least ten indentations on different locations of the film surface were performed using Berkovitch-type pyramidal diamond tip and average hardness values were calculated.