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Preferentially oriented vanadium nitride films deposited by magnetron sputtering

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ABSTRACT

Because of solid state lubricious properties of vanadium oxides, wear resistant coatings based on nitrides and carbides of that metal are still of interest for research teams. The aim of this report is to show phase composition evolution from metallic vanadium through intermediate phases up to δ-VN phase supersaturated with nitrogen in thin films deposited by reactive, pulsed magnetron sputtering from vanadium target. This analysis is completed by remarks on preferential orientation, lattice constant and crystallite size. Presented work is a part of research on composite hard coatings for woodworking tools where vanadium nitrides and carbides are considered as a component reducing friction.

1. Introduction

Properties of vanadium nitride belonging to the group of hard, transition metal nitrides are much less explored than single- or multi-component nitrides of titanium, aluminium, zirconium or tungsten. However, due to its catalytic properties [1] as well as recently reported potential applications as electrode material in lithium batteries [2], vanadium nitride based coatings attract increasing attention.

Their high hardness opens the way to applications where wear resistance and chemical stability are required [3,4] A particular property of vanadium nitride is that its oxidation leads to generation of surface layer acting as solid lubricant [4–6].

There is a well established knowledge on phase equilibrium, crystallographic and thermodynamic data for the vanadium–nitrogen system [7,8]. However modern thin film deposition techniques, particularly plasma based, offer deposition conditions which are very far from thermodynamic equilibrium. It opens technological window for deposition of meta-stable and supersaturated phases.

Vanadium nitride films have been deposited by reactive magnetron sputtering [6], reactive electron beam evaporation [3], pulsed laser ablation [9] and ion beam assisted deposition [10]. Their structure has been studied in a wide range of V/N ratio but particular attention was paid to the most stable phases known as hexagonal β-V_2N and cubic δ-VN. Some consideration on relations between deposition conditions and structure of coatings were also reported [10–12].

We explored a wide range of nitrogen concentrations in the processing atmosphere during reactive, pulsed magnetron deposition of vanadium nitride films. The aim of this report is to show a phase composition evolution from metallic vanadium through intermediate phases up to δ-VN phase, supersaturated with nitrogen. This analysis is complemented by remarks on preferential orientation, lattice constant and crystallite size.

2. Experimental

Vanadium nitride coatings have been deposited by pulsed, reactive magnetron sputtering in N_2/Ar atmosphere. The vacuum chamber of about 25 cm in diameter was used and the substrate holder was placed 60 mm from the planar, 100 mm (4 in.) magnetron source equipped with vanadium cathode. The vacuum chamber was pumped down to the ultimate pressure 10^{-3} Pa by standard system equipped with a diffusion pump.

As the substrates, mirror polished ($R_a = 35$ nm) discs, 28 mm in diameter, made of 440 C steel were used. Before deposition, substrates were heated up to 350±10 °C and ion cleaned for 15 min in argon DC glow discharge at 0.6 kV and argon pressure of 35 Pa.

Magnetron sputtering source was driven by pulsed power supply (1 kHz with 100 kHz modulation) operated in unbalanced mode controlled by external electromagnet coils. Mean power dissipated at the source was 0.9 kW. Mean deposition rate, depending on nitrogen flow, was about 70 nm/min.

The parameters changed for different sets of samples were: substrate bias (kept at potential of −70 or −100 V) and Ar and N_2 pressure in Ar/N_2 mixture (Table 1).

3. Results and discussion

Deposited coatings were examined using a XRD spectrometer equipped with cobalt lamp and working in Bragg–Brentano ($\theta - 2\theta$) geometry.

For low partial pressures of reactive gas (sample sets 1–3) films containing metallic bcc-V, tetragonal $VN_{0.09}$ (ICDD 71-1230), hexagonal

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