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Experimental studies on epitaxially grown TiN and VN films

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Abstract

TiN and VN single-layers were grown on (100) and (111) MgO substrates as model systems for future studies of V diffusion and understanding the evolution of the lubricous V_2O_5 phase on coating surfaces. The present study aims at elucidating the epitaxial quality and microstructure of TiN and VN films grown by unbalanced d.c. magnetron sputtering from Ti and V targets in Ar+N_2 on (100) and (111) MgO substrates. X-ray diffraction and electron back-scatter diffraction give evidence for epitaxial growth of the films on both MgO substrate orientations. Transmission electron microscopy studies reveal a cube-on-cube orientation for all film/substrate combinations. The higher dislocation density and larger fraction of misoriented grains for VN films on MgO is explained by the higher growth rate and lattice mismatch compared to TiN. Nanoindentation measurements of the (100) oriented single-crystal films reveal hardness values of ~35 GPa for TiN and ~23 GPa for VN.

Keywords: Epitaxial growth; TiN; VN; Hardness; EBSD; TEM

1. Introduction

Among the transition metal nitrides, TiN-based hard coatings are dominating in improving the wear resistance of tools, thus increasing their lifetime. In the last years, incorporation of vanadium by forming TiAlN/VN multilayers or Ti(Al,V)N solid solutions has been intensively investigated. For example, several studies indicate high hardness depending on the layer thicknesses of TiN/VN superlattice coatings [1–4]. However, TiN/VN multilayers start to interdiffuse and V shows a pronounced surface segregation tendency at elevated temperatures [5,6]. This approach has been used recently to obtain a lubricious effect for TiN-based hard coatings [7]. The out-diffusion of V to the coating surface, forming the V_2O_5 phase, drastically reduces high-temperature friction. This lubrication effect is based on both the easy shearable lattice planes (solid lubrication) and the low melting point of V_2O_5 of 680 °C (liquid lubrication) [8–10]. The effect has been well described for TiAlN/VN superlattice as well as Ti(Al,V)N solid solution coatings [11,12]. However, the mechanism of V out-diffusion through the TiN-based lattice is not clear up to now.

To bring more light into this topic, fundamental work on V diffusion in model coatings is necessary. Thus, within this work the possibilities to synthesize well-defined epitaxially grown TiN and VN model layers of different orientations are explored, as a pre-requisite to future interdiffusion studies. Both, TiN and VN show a B1 NaCl face-centered cubic structure with similar lattice parameters of 0.424 nm and 0.413 nm [13], respectively. This facilitates epitaxial growth on MgO substrates (a=0.421 nm [13]) as well as on each other [4,14–16].

In the present study, TiN and VN single-layers were grown on MgO (100) and MgO (111) substrates by magnetron sputtering in order to elucidate the epitaxial quality for film thicknesses in the micron range. The microstructure of the films is analysed in detail by X-ray diffraction (XRD), electron back-scatter diffraction (EBSD), and transmission electron microscopy (TEM). In addition, mechanical properties of single-crystal TiN and VN films were deduced from nanoindentation experiments.

2. Experimental details

MgO substrates of two different orientations, (100) and (111), were used for deposition. They were ultrasonically cleaned in acetone and ethanol for 5 min. TiN and VN single-layers were grown from Ti and V targets in Ar+N_2 using an unbalanced d.c.

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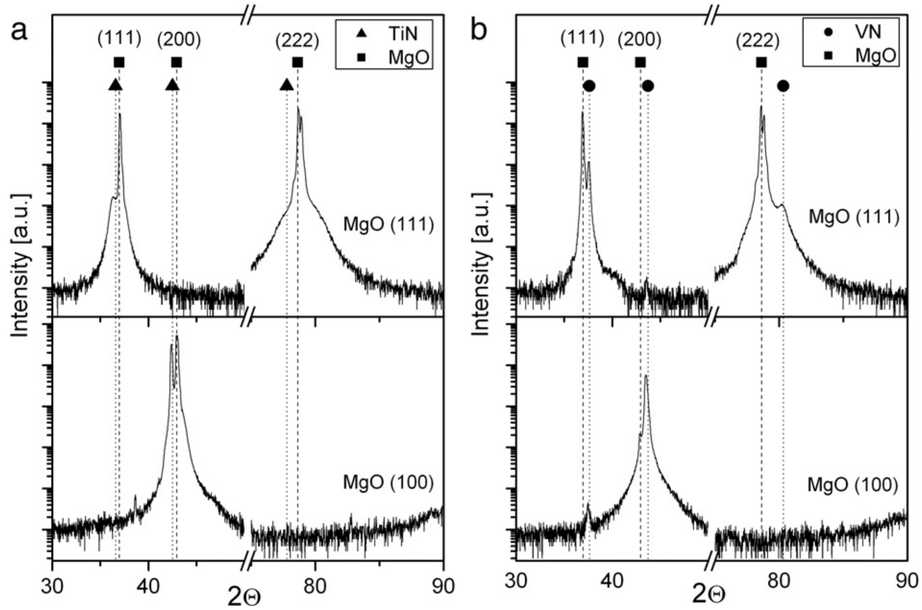

Fig. 1. XRD patterns of (a) TiN and (b) VN films grown on MgO (100) and (111) substrates.

magnetron sputtering system. The working gas pressure $p_{Ar}+p_{N_2}$ was kept constant at 0.25 Pa; the N_2 partial pressure was 30% of the total pressure in case of the TiN and 24% in case of the VN. The substrate temperature during deposition was 580 °C, the deposition current was 0.5 A. The deposition time was set to 60 min resulting in a film thickness of 0.65 μm for TiN and 1.1 μm for VN as found by cross-sectional TEM studies.

XRD analyses in Bragg–Brentano mode have been performed on a Siemens D500 diffractometer applying Cu Kα radiation. EBSD measurements in a scanning electron microscope (SEM)

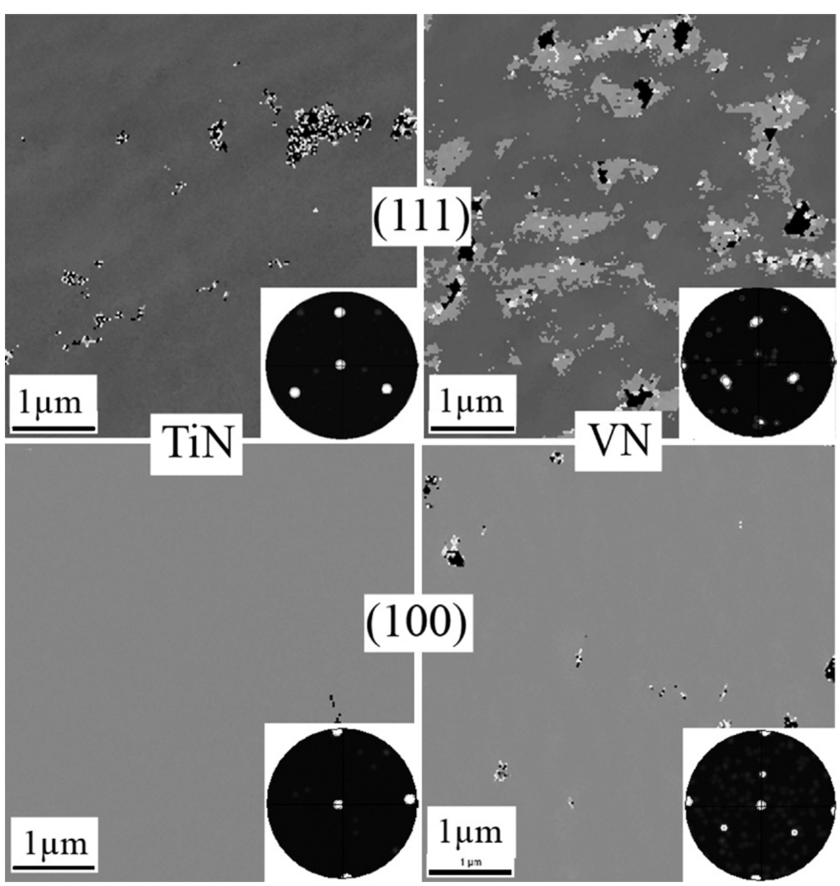

Fig. 2. EBSD analyses (scanned area of 5 × 5 μm^2) and (001) pole figures (large area scan of 850 μm^2) of TiN and VN films grown on MgO (100) and (111) substrates.