Correlating atomic-resolution structure to the properties of transition metal nitride coatings

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Introduction

The industrial applications of transition metal nitrides require an in-depth understanding of the structures and their correlations to properties. This has led to numerous experimental and theoretical research [1-5]. Utilizing modern electron microscopy and spectroscopy, our understanding of nitride structure and property relations and its deformation has been significantly advanced. This presentation will briefly summarize our recent atomic-resolution studies on transition metal nitride coatings using spherical aberration-corrected transmission electron microscopy (TEM).

Methods

The film used in this study was prepared by direct current magnetron sputtering using a custom-made lab-scale magnetron sputter deposition system. The film was grown on MgO(001) or Si substrate at 700 ℃, and the potential was kept floating during the deposition. Specific deposition conditions for different films are slightly different. Nanoindentation tests were conducted on a KLA Nanoindenter G200 equipped with a diamond cube-corner indenter tip to obtain H and E by the conventional analysis first proposed by Oliver and Pharr. The tests applied the continuous stiffness measurement method with a constant indentation strain rate ▯ of 0.05 s−1. The cross-sectional TEM lamella of the as-deposited and indented films was machined by focused ion beam (FIB) using an FEI Helios NanoLab 660 workstation. The lamella was cut along the <110> direction of the substrate and then transferred to a Cu TEM grid. The lamella was polished using accelerating voltages from 30 to 2 kV and ion currents ranging from 20 nA to 7 pA. Some TEM specimens were also prepared using a standard approach, i.e., grinding, polishing, dimpling, and Ar ion-milling.

For high-resolution TEM (HRTEM) characterization, a JEOL 2100F and FEI Titan Themis 60-300 cubed TEM, both equipped with an image-side Cs-corrector, were adopted. The scanning TEM (STEM) images and energy-dispersive X-ray spectroscopy (EDXS) were acquired by a 300 kV field emission TEM (JEOL ARM300F) equipped with double Cs-correctors. The microscope has two windowless detectors, each with an active area of 100 mm2.

Results

The extensive high-resolution transmission electron microscopy (HRTEM) observations on TaN/TiN multilayer reveal that the dissociation of full dislocations results in the network of stacking faults (SFs) and the formation of Lomer-Cottrell lock arrays inside the TaN layer. Consequently, the high density of stacking faults dramatically strengthens the TaN/TiN multilayer [1]. Using valence electrons and inner shell electron spectroscopy, a combined experimental analysis on a multilayered structure of CrN/AlN allowed mapping of the multilayer's mechanical properties (bulk modulus) at the nanometer scale [2]. Moreover, we found that the presence of oxygen impurities causes a remarkable reduction of the bulk modulus of rs-CrN while having no significant effect on the bulk modulus of the stable wurtzite structure wz-AlN layers. The findings have been validated by theoretical calculations [2].

We observed an atomic-scale intermixing phenomenon in the nanoscale TiN/AlN multilayer by coupling the cross-sectional FIB cutting with detailed atomic resolution electron microscopy analyses. A new solid solution phase was created during nanoindentation, which could be unambiguously shown by mapping the electronic structure differences (i.e., using Ti-

L2,3 edge). This also raises some concerns about understanding experimentally measured hardness values in multilayers using the nanoindentation method. By using atomic-resolution energy dispersive x-ray spectroscopy, we further corroborated that such a homogenous solid solution zone formed upon loads [3]. We recently found a new deformation mechanism of a high vacancy-mediated WN_x/TiN multilayer through quantitative electron microscopy analysis, theoretical calculations enabled by ab initio molecular dynamics, and combined with mechanical testing [5].

Conclusions
Advanced electron microscopy methods reveal the atomic and electronic structures of transition metal nitride coatings, which allows correlating the structure with properties at the atomic scale and leads to new findings. These exciting findings shed light on the design of novel multilayer coatings for applications.

Keywords:

Nitride, HRTEM/STEM, EELS, deformation

Reference:

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