Hard Coatings and Vapor Deposition Technologies
Room California - Session B4-4-WeA

Properties and Characterization of Hard Coatings and Surfaces IV

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2:00pm **B4-4-WeA-1 Effect of Ti Interlayer on Stress Relief of ZrN/Ti Bilayer Thin Films on Si Substrate*, Jia-Hong Huang*,** *T Zheng,* National Tsing Hua University, Taiwan

Pure metal interlayers have been widely used to enhance adhesion and relieve residual stress in hard coatings. However, the design of interlayer thickness for stress relief was mostly empirical without quantitative basis. The objectives of this study were to investigate the effect of metal interlayer on stress relief of hard coatings, and to establish a physical model correlating plastic deformation of the interlayer with stress relief. ZrN/Ti bilayer thin films on Si substrate was chosen as the model system. ZrN/Ti specimens with different interlayer thicknesses and with ZrN coatings deposited at different bias voltages were prepared using unbalanced magnetron sputtering. Wafer curvature method and average X-ray strain combined with nanoindentation technique [1,2] were employed to accurately measure the residual stresses in the entire specimen and individual layer, respectively. Experimental results showed that the extent of stress relief, ranging from 59.7 to 80.4%, increased with interlayer thickness, while decreased with increasing stress transferring from top ZrN layer. The efficiency of stress relief decreased with increasing interlayer thickness, but varied irregularly with the stress transferring from ZrN layer. A physical model was developed to account for the stress relief due to plastic deformation of the interlayer, based on the energy balance between elastic stored energy in ZrN and plastic work of metal interlayer. The upper limit of stress relief by the interlayer was assumed to be the necking strain of the interlayer under equibiaxial stress state. The model was verified by the experimental results. Furthermore, a critical experiment was conducted and confrimed that the model could provide a conservative estimation on stress relief for practical applications. The proposed model also indicated that the stress relief was mainly due to plastic deformation of Ti interlayer. Using the model, we could quantitatively estimate the allowable stress relief with a specific interlayer thickness or the required interlayer thickness to relieve certain amount of stress.

[1] A.-N. Wang,C.-P. Chuang,G.-P. Yu, J.-H. Huang, Surf. Coat. Technol., 262 (2015) 40.

[2] A.-N. Wang, J.-H. Huang, H.-W. Hsiao, G.-P. Yu, Haydn Chen, Surf. Coat. Technol., 280(2015) 43.

2:20pm **B4-4-WeA-2 In-situ Observation of Stress Fields during Crack Tip Shielding in Loaded Soft-hard Micro-Cantilevers using Cross-sectional X-ray Nanodiffraction*, Michael Meindlhumer,*** Montanuniversität Leoben, Department of Physical Metallurgy and Materials Testing, Austria*; J Todt, J Zálešák,* Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria*; S Klima, N Jäger,* Montanuniversität Leoben, Department of Physical Metallurgy and Materials Testing, Austria*; M Rosenthal, M Burghammer,* ESRF Grenoble, France*; H Hruby,* voestalpine eifeler Vacotec GmbH, Germany*; C Mitterer, R Daniel,* Montanuniversität Leoben, Department of Physical Metallurgy and Materials Testing, Austria*; J Keckes,* Montanuniversität Leoben, Austria

In recent years, cross-sectional X-ray nanodiffraction (CSnanoXRD) with a resolution down to ~30 nm has been proven to resolve depth gradients and residual stresses within individual sublayers of multi-layered thin films. In this work, the in-situ CSnanoXRD setup was used to perform micromechanical testing on a 20 µm thick film composed of four alternating hard CrN and soft Cr sublayers, each 5 µm thick, on a high-strength steel substrate. Notched freestanding and clamped cantilevers of the film thickness and a length and width of 150 and 35 µm and 200 and 40 µm, respectively, machined using focused ion beam (FIB), were tested. Multiaxial stress distributions were evaluated in stationary, stepwise loaded and unloaded cantilevers with a spatial resolution of 200 x 200 nm^2 in a cross-sectional area of ±20 µm around the FIB-fabricated notches.

While the freestanding cantilever was nearly relaxed before loading, the clamped cantilever exhibited residual stresses up to -4 GPa within the CrN sublayers and up to -500 MPa in the Cr sublayers. During the loading, high in-plane, out-plane and shear compressive stresses up to ~10 GPa were observed in CrN under the indenter and at the crack tip. In the Cr sublayers, the loading resulted in a formation of cross-sectional stress gradients and compressive-tensile-compressive stress switching at different stages of the experiments. Interestingly, it was observed in both cantilevers that the multiaxial stress concentrations at the crack tips in CrN and in Cr sublayers were blunted when approaching CrN-Cr interfaces. After the unloading, only minor changes in residual stresses in CrN were observed, compared to the stress state before loading.

In summary, the pioneering in-situ micromechanical approach coupled with X-ray nanodiffraction screening was used for the first time to determine multiaxial X-ray elastic strain/stress fields in the vicinity of notches/cracks with sub µm-resolution. The experimental data provide an understanding of the nanoscale deformation processes in nanolaminates and will be integrated into finite element simulations dealing with the evolution of crystallize size- and interface-dependent stress concentrations during elasto-plastic deformation.

2:40pm **B4-4-WeA-3 Experimentally Parameterized Simulation of an Instrumented Dry Milling Arrangement – Parameter Study Identifying Damage-relevant Coating Properties for End Mills*, Andreas W. Nemetz,*** *W Daves, T Klünsner, W Ecker,* Materials Center Leoben Forschung GmbH, Austria*; C Praetzas,* Institute of Production Management, Technology and Machine Tools (PTW), Germany*; C Czettl, J Schäfer,* CERATIZIT Austria GmbH, Austria

Solid hard metal end mills, both coated and uncoated, are used to machine difficult-to-cut materials, such as titanium alloys. Experience shows that the tool life varies greatly depending on the applied coating and the chosen process parameters. At the end of the tool life, the cutting situation is not the same as at the beginning of the process and the product quality suffers. Therefore it is of high relevance to recognize and interpret the signals, given within the process to assess its quality and estimate the remaining tool life. Especially in the field of process monitoring, temperature monitoring is a valuable addition to the prevailing force measurement. Within an instrumented milling arrangement, the measured temperature inside the coated end mill is documented. The process is numerically simulated to relate the measured core temperature in the end mill to the temperature at the cutting edge. This information is essential for the model based tool health monitoring. The synergetic use of 2D thermo-mechanical milling models and 3D thermal and mechanical models enables the prediction of the transient temperature field in milling tools. The material models describing the hard coatings and the WC-Co hard metal substrate are parameterized experimentally. The influence of hard coatings on the evolution of the evolving temperature field in end mills is investigated during dry milling. A parameter study identifies improved thermal coating material properties.

3:00pm **B4-4-WeA-4 Mechanical Properties and Cutting Performance of AlCrSiN and AlTiCrSiN Hard Coatings*, Liang-Chan Chao,*** *Y Chang,* National Formosa University, Taiwan

Transition metal nitride coatings based on Cr, Ti and Al, such as AlTiN, AlCrN and AlCrSiN, have been used as protective coating materials of cutting and forming tools due to their high hardness and thermal stability. In this study, AlCrSiN and AlTiCrSiN coatings were deposited onto high-speed steels and tungsten carbide tools using AlTi, TiSi, Cr and ternary AlCrSi alloy targets in a Cathodic-arc evaporation (CAE) system. Optimal design of interlayers of the AlCrSiN and AlTiCrSiN can offer an efficient way of controlling residual stress, improving adhesion strength and enhancing toughness. During the coating process of AlCrSiN and AlTiCrSiN, AlCrN and AlTiN were deposited, respectively, as interlayers with different structures to control residual stress, toughness and adhesion strength between the coatings and substrates. By controlling the different interlayers and negative bias voltages, the AlCrSiN and AlTiCrSiN possessed different microstructures and mechanical properties. The microstructure of the deposited coatings was investigated by field emission scanning electron microscope (FE-SEM) and field emission gun high resolution transmission electron microscope (FEG-HRTEM), equipped with an energy-dispersive x-ray analysis spectrometer (EDS). Glancing angle X-ray diffraction was used to characterize the microstructure and phase identification of the coatings. Mechanical properties, such as the hardness and young's modulus, were measured by means of nanoindention. The adhesion strength of the coatings was evaluated by a standard Rockwell indentation test. In order to evaluate the impact fatigue behavior of the coated samples, an impact test was performed using a cyclic loading device with a tungsten carbide indenter as an impact probe. For the cutting experiment, 316L stainless steel was machined by the coated end mills under oil mist condition using a

CNC milling machine. The design of AlCrTiSiN coatings were anticipated to increase the hardness, toughness, thermal stability and impact resistance by optimizing the interlayers and bias condition of the deposition.

Keywords : Mechanical property; Cutting; Hard coating; Interlayer

3:20pm **B4-4-WeA-5 Erosion, Corrosion Resistance and Hydrophobicity of Nano-layered and Multi-layered Nitride Coatings***, **Qi Yang**, L Zhao, P Patnaik,* National Research Council of Canada, Canada

Nano-layered CrTiN, CrTiAlN, CrAlTiN, multi-layered CrAlTiN-CrN and CrAlTiN-AlTiN coatings were deposited on 17-4 PH stainless steel substrate by the cathodic arc evaporation technique. Solid particle erosion tests were performed to investigate their erosion resistance. All of these coatings, CrAlTiN and CrAlTiN-AlTiN coatings in particular, demonstrated higher resistance to solid particle erosion at both low and high impingement angles when compared to the single layered CrN coating. For example, the mass erosion rates of the nano-layered CrAlTiN coating were less than 1/3 and 1/7 of the corresponding erosion rates of the CrN coating at 30° and 90° respectively. The excellent erosion performance of the coatings was attributed to the improved hardness. Potentiodynamic polarization tests of the coatings in 3.5% NaCl aqueous solution indicated that these coatings had higher corrosion potentials and much wider passive ranges with comparable or lower current densities, when compared to 17-4 PH. Water contact angle measurements illustrated that the nitride coatings also had good hydrophobic characteristics.

3:40pm **B4-4-WeA-6 Microstructure and Thermal Stability of Al-rich Ti-Al-Mo-N Protective Coatings***, **Christina Wüstefeld**,* Institute of Materials Science, TU Bergakademie Freiberg, Germany*; M Motylenko,* Technische Universität Bergakademie Freiberg, Germany*; S Berndorf,* Institute of Materials Science, TU Bergakademie Freiberg, Germany*; M Pohler, C Czettl,* CERATIZIT Austria GmbH, Austria*; D Rafaja,* Technische Universität Bergakademie Freiberg, Germany

Ti-Al-N coatings are known as protective coatings in metal cutting applications. Further improvements of the mechanical properties and thermal stability of Ti-Al-N coatings are aspired by the addition of alloying elements like tantalum, niobium or molybdenum and by the adjustment of the microstructure via the deposition parameters. In this study, the Mo content and the bias voltage were varied in order to investigate the impact on the microstructure and thermal stability of Al-rich Ti-Al-Mo-N coatings. The coatings were deposited using cathodic arc evaporation (CAE) in nitrogen atmosphere from $Ti_{32}Al_{65.5}Mo_{2.5}$ and $Ti_{28}Al_{62}Mo_{10}$ targets at different bias voltages that ranged between -40 V and -120 V. In order to study the influence of the Mo addition on the thermal stability, selected samples were annealed in argon atmosphere up to 1000 °C. The as deposited and annealed Ti-Al-Mo-N coatings were characterized by using a combination of X-ray diffraction and transmission electron microscopy (TEM) with high resolution and in scanning mode. TEM in scanning mode was supplemented by electron energy loss spectroscopy and energy dispersive spectroscopy.

The microstructure of the coatings was described in terms of the chemical and phase composition, stress-free lattice parameters of the fcc-phase, macroscopic residual stresses as well as the sizes and preferred orientations of fcc crystallites. It could be shown that the bias voltage can be used to control the fractions of fcc-(Ti,Al,Mo)N and wurtzitic AlN in Al-rich Ti-Al-Mo-N coatings. The correlation between the microstructure characteristics, the parameters of the deposition process and the hardness is discussed.

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