

Tuesday Morning, November 10, 2009

Advanced Surface Engineering

Room: C4 - Session SE-TuM

Hard and Nanocomposite Coatings

Moderator: P.H. Mayrhofer, Montanuniversitat Leoben, Austria

8:00am **SE-TuM1 AlTiN and AlCrN Hard Coatings - Alloying as an Approach to Improve Oxidation and Tribological Properties, C. Mitterer**, University of Leoben, Austria **INVITED**

Metastable transition metal aluminum nitride based hard coatings like AlTiN and AlCrN grown by plasma-assisted physical vapor deposition are nowadays widely used to protect high-performance tools against wear and oxidation. The excellent properties of these coatings arise from their ability to form protective Al₂O₃-based oxide scales and from the decomposition of their metastable face-centered cubic (fcc) lattice, resulting in age hardening.

The aim of this presentation is to give a survey of attempts to alloy AlTiN and AlCrN coatings, with the goals to improve hardness and wear resistance, oxidation resistance, toughness, and to reduce friction. Coatings were synthesized by reactive cathodic arc evaporation, and powder-metallurgically produced TiAl and CrAl targets with the alloying elements V, Si, B, Ta, and Ru have been used. Low Si, B and Ta contents are incorporated in the single-phase fcc solid solutions, enhancing both mechanical properties and oxidation resistance. In particular, for AlTiN coatings the onset temperature for oxidation is shifted to significantly higher values compared to AlTiN, which is related to a reduction of intrinsic stresses in the rutile layer formed underneath the top Al₂O₃ scale. B alloying of AlCrN results in the formation of a nanocomposite structure, with nanosized fcc grains surrounded by a BN-rich grain boundary layer, giving rise to extremely high hardness values and wear resistance. On the other hand, V alloying can be used to form self-lubricious V₂O₅ oxide layers, where their low friction coefficients in the temperature range between 550 and 700°C arise from liquid lubrication, due to the low melting temperature of this phase. Furthermore, low contents of Ru have been shown to increase the toughness of AlTiN coatings, which is assumed to formation of a metallic Ru phase within these coatings.

In summary, alloying of metastable transition metal aluminum nitride based hard coatings enables to design advanced quaternary and multiterinary hard coatings with property combinations meeting the requirements of severe machining processes.

8:40am **SE-TuM3 Pressure Dependence of the Al Ion Energy Distribution Functions during Filtered Cathodic Arc Thin Film Growth in an Ar, O₂ Ambient, A. Atiser, S. Mraz, J.M. Schneider**, RWTH Aachen University, Germany

Charge state resolved ion energy distribution functions (IEDFs) of Al⁺, Al²⁺ and Al³⁺ were measured as a function of Ar pressure in the range from 5.7 × 10⁻⁵ to 2.13 Pa (0.01 to 256 Pa cm). As the pressure distance product is increased, the annihilation of the Al²⁺ and Al³⁺ populations as well as the thermalization of the Al⁺ ion population is observed, resulting in the formation of a close to monoenergetic beam of Al⁺ ions at pressure distance product of 256 Pa cm. The average charge state was reduced from 1.58 to 1.00 as the pressure distance product was increased from 0.01 to 32 Pa cm. Thermalization is also observed in an Ar/O₂ mixture at 128 Pa cm, where stoichiometric γ -alumina films are grown. The IEDFs have been fitted by a shifted Maxwellian distribution. The plasma processing strategy presented here resulting in a monoenergetic Al⁺ plasma beam may through substrate bias potential variations enable effective tailoring of thin film properties such as density, elasticity and phase stability.

9:00am **SE-TuM4 Al-Si-N Thin Films: Nanocomposites and Solid Solutions, A. Pélisson, M. Parlinska-Wojtan, H.J. Hug, J. Patscheider**, EMPA, Switzerland

Thin films consisting of Al-Si-N were prepared by reactive magnetron sputtering from elemental targets in an Ar/N₂ reactive atmosphere at 200°C. The system shows a solubility limit for silicon at around 6 atomic %. Correspondingly the Al-Si-N system forms, as a function of the silicon content, either a solid solution or a two phase nanocomposite structure. To understand the properties and formation of the nanocomposite nanoscaled multilayers were used as a simplified model system. Coatings with a total thickness of about 1 micron and consisting of alternating layers of h-AlN or h-Al_{1-x}Si_xN (5 nm) and a-Si₃N₄ (from 0.25 nm to 2.0 nm) were prepared. The hardness as well as the residual stress state are strongly influenced by the thickness of the Si₃N₄ layer and the silicon content of the

crystalline Al-Si-N layer. Maximum hardness values of 33 GPa are reached for a Si₃N₄ layer thickness of 0.35 nm, whereas the stress state can be tuned between - 1.5 and + 1.5 GPa. Both High Resolution TEM and XRD showed that, for Si₃N₄ layer thicknesses below 1 nm, the Si₃N₄ layers grow heteroepitaxially on AlN. The implication for the hardness of isotropically deposited solid solution and nanocomposite thin films of Al-Si-N will be discussed.

9:40am **SE-TuM6 Development of Water Repellent Metal Oxide Thin Film as Like Organic Polymer, T. Watanabe, Y. Yokota, N. Yoshida**, The University of Tokyo, Japan, Y. Okura, Kogakuin University, Japan

Mechanically durable water repellent thin film composed of only metal oxides such like Al₂O₃, HfO₂, ZrO₂, TiO₂ and CeO₂ were successfully developed by sol-gel process. The key points to enhance dynamic hydrophobicity are reducing surface roughness and increasing chemical homogeneity. Surprisingly to say, developed films show 100 degree of water contact angle and sliding angles of water droplet is only around 20 degree. Water repellency has not been deteriorated even by 500 times abrasion tests of 0.1kg/cm². Water removability on these films is as like that of hydrophobic polymer surface. Fundamentally, intrinsic surface wettability of metal oxide is hydrophilic. However nanometer scale flatness and homogenous surface reduce activation energy of de-wetting process of water droplet. It is considered to cause of higher water removability. Durable hydrophobic surface composed only by inorganic oxide attracts much attention because it can be applied for water repellent automobile glass window and other applications which need higher mechanical strength and durability. Moreover, developed films show several unique properties which cannot be observed on organic polymer surfaces.

11:00am **SE-TuM10 Anti-Wear and Anti-Bacteria Behaviors of Rejuvenated TaN-Cu Nanocomposite Thin Films, J.H. Hsieh, P.C. Liu**, Ming Chi University of Technology, Taiwan, C. Li, Nanyang Technological University, Singapore

TaN-Cu nanocomposite films were deposited by reactive co-sputtering on Si and tool steel substrates. The films were then annealed using RTA (Rapid Thermal Annealing) at 400 °C for 2, 4, 8 minutes respectively to induce the nucleation and growth of Cu particles in TaN matrix and on film surface. Cu nano-particles emerged on the surface of TaN-Cu thin films were then removed after the samples were tested for their anti-wear and anti-bacterial behaviors. The samples were then re-annealed (rejuvenated), and re-tested for their anti-wear and anti-bacterial behaviors. The results reveal that the rejuvenated samples could have similar anti-wear and anti-bacterial behaviors so long as the annealing conditions were well adjusted. However, the hardness of the samples would decrease to a certain extent.

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