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METALLURGICAL COATINGS
AND THIN FILMS

PROGRAM AND ABSTRACTS

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ADVANCED SURFACE ENGINEERING DIVISION
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in the wear rate. When both Ti and graphite targets were pulsed at 100 kHz and 5.0 micros, the film exhibits a high hardness of 32 GPa, a friction coefficient of 0.28 and a low wear rate of 2.2x10^-7 mm^2N^-1m^-1.

**Triboology and Mechanical Behavior of Coatings and Thin Films**

**Room: California - Session E2-1**

### Mechanical Properties and Adhesion

**Moderator:** R. Chromik, McGill University, L. Davies, Caterpillar, N. Jennett, National Physical Laboratory

8:00am **E2-1-1 Effect of Surfactants on the Mechanical Properties of Electroless (Ni - P) Coating, R. Elansezhian (elansezhian@yahoo.co.in), B. Ramamoorthy, P. Kesavan Nair, Indian Institute of Technology Madras, India**

Electroless nickel (EN) coating is a well established surface engineering process that involves deposition of a metal-metallloid alloy coating on various substrates. Although a variety of metals can be plated, electroless Ni-P coating has received widespread acceptance as it provides high hardness and excellent resistance to wear, abrasion and corrosion. Electroless methods offer high-quality ultrathin films that are compatible with high-resolution patterns such as sub-100 nm interconnects, contacts and vias contacts for ULSI and in high aspect ratio structures for MEMS. The effect of surfactants on the surface roughness, microhardness and microstructure of electroless Nickel - Phosphorus (EN) surface protective coating obtained from an alkaline bath is presented in this paper. In this study the influence of surfactants sodium dodecylsulfate (SDS) and cetyl trimethyl ammonium bromide (CTAB) on the surface roughness, microhardness and microstructure of coated samples are investigated. The variation on surface morphology was examined using high resolution scanning electron microscope (HRSEM) and surface roughness values were measured using a stylus instrument. It was observed that the surface roughness and surface morphology of Ni-P coating were affected by addition of surfactants SDS and CTAB. Different surfactant concentrations were added to the electrolyte bath. It was found that without surfactant mean surface roughness (Ra) value was 1.885 Å. When SDS concentration was lower, the mean roughness (Ra) value was found to be much higher. When SDS concentration increased, the roughness value was found to be improving and stable. For lower CTAB concentration mean roughness (Ra) value was deteriorating and when CTAB concentration increased the roughness value was observed to be improving. It was found that without surfactant the micro hardness value of as-coated condition was 450 Hv. The micro hardness value was increasing with increased SDS and CTAB concentrations. When SDS concentration increased, beyond its critical micelle concentration (CMC) the hardness value has stabilized and found to be better. When CTAB concentration increased, the hardness value was stable and observed to be improving. A 100% increase in micro hardness was observed in the presence of surfactant. The complete experimental details, results obtained and their analysis is presented in this paper.

8:20am **E2-1-2 Mechanical Properties of Gradient and Multilayered TiAlSiN Hard Coatings, Y.-Y. Chang, Mingdao University, Taiwan, S.-J. Yang (kim3042002@yahoo.com.tw), National Chung Hsing University, Taiwan, D.-Y. Wang, Mingdao University, Taiwan, W. Wu, National Chung Hsing University, Taiwan, D. Yang, National Chung Hsing University, Taiwan, D. Yang, National Chung Hsing University, Taiwan**

Multicomponent coatings based on different metallic and non-metallic elements possess the combined benefit of individual components leading to further improvement of coating properties. In this study, TiAlSiN nanocomposite coatings prepared as monolithic single layer, multilayer and gradient films were synthesized by using a cathodic-arc deposition system with lateral rotating arc cathodes. Chromium and AlSi alloy cathodes were used for the deposition of TiAlSiN coatings. In this study, field emission scanning electron microscope, transmission electron microscope and X-ray diffraction using Bragg-Brentano and glancing angle parallel beam geometries were used to characterize the microstructure of the as-deposited and high temperature annealed films. The mechanical properties including hardness, elastic modulus, and fracture toughness of the deposited and annealed TiAlSiN coatings were analyzed by a nanoindentation with Berkovich indenter tip. It has been found that the mechanical properties of the deposited and annealed films were correlated with the gradient and multilayered structures.

8:40am **E2-1-3 The Strength of Thin Films, Small Structures and Materials Under Localised Stresses, B.J. Hespanhol (d.hespanhol@qmul.ac.uk), Queen Mary University of London, United Kingdom**

Critical thickness theory explains an excess yield stress in thin films inversely proportional to the film thickness. However, in other contexts, an excess yield stress is often observed which is proportional to the inverse square-root of a relevant dimension. Work-hardening coefficients show both square-root and inverse dependencies. We report on recent experimental work which has demonstrated these effects unambiguously, and discuss the extent to which they can be theoretically explained.

9:00am **E2-1-5 The Effects of Alloying on the Mechanical Behaviour of In,Ga,As, S. Korte (sk511@cam.ac.uk), I. Farrer, W.J. Clegg, University of Cambridge, United Kingdom**

The mechanical behaviour of In,Ga,As quantum wells is of interest in its own right, although these systems have also attracted interest as model systems for studying deformation behaviour, such as the effect of coherency strains. However, to the knowledge of the authors, only very little information is available on the deformation behaviour of InGaAs over the full compositional range from GaAs to InAs. Existing data on a range of systems suggests that a maximum in the yield stress or measured hardness occurs at some intermediate composition, for instance in ZnInSe. In recent studies on InGaAs, a dependence of mechanical properties on composition has only been carried out in the range of normal dopant concentrations. Here, nanindentation with different tip geometries was used to investigate the mechanical properties of InGaAs over its full compositional range, going into greater detail to either side of the maximum hardness at x ≈ 0.25. Yield stress from spherical indentation, Berkovich hardness and Young modulus are reported for all samples. Stress-strain curves from spherical indentation suggest a different yield behaviour depending on composition and dislocation density of the volume under the indenter. These measurements are compared with observations using transmission electron microscopy.

9:40am **E2-1-7 Investigation of Magnetron Sputtered Titanium-Nickel Nitride Thin Films for Use as Mould Coatings, H.R. Stock (stock@iw-bremen.de), M. Dieseller, H.-W. Zoch, Stiftung Institut für Werkstofftechnik, Germany**

Reactive magnetron sputtering of substoichiometric TiNx films (x < 1) with different amounts of nickel ranging from 8 to 32 at.% was undertaken. Moulding tools made of X42Cr13 (AISI 420) steel were used as substrates, ground and polished to a roughness of Ra = 12 nm. By varying the nitrogen gas flow, nickel content and deposition temperature the properties of the films change significantly. The morphology of the coatings is adjustable between amorphous and columnar and the universal hardness increases between 7 and 21 GPa. After deposition the samples were heat treated at temperatures up to 700°C. Both chemical composition and heat treatment temperature were found to have a considerable effect on resulting hardness. Moulding experiments with PMMA (polymethylmethacrylate) revealed a remarkable performance of the coated tools. No delamination of the coating was observed. The only wear detectable was some abrasion occurring at the near surface region of the coating, also known as polishing wear. This results in a decrease of the roughness and even better surface smoothness of the moulded PMMA parts.

10:00am **E2-1-8 Structure and Mechanical Characteristics of Amorphous Carbon Film Deposited on Poly Aryl-Ether-Ether-Ketone (PEEK) Substrate, M. Xu, Z. Wei, S.H. Pu, P.K. Chu (pckhu@cityu.edu.hk), City University of Hong Kong**

Poly aryl-ether-ether-ketone (PEEK) is a modern, high-temperature and radiation-resistant thermoplastic material used in a variety of applications in the aerospace industry, automotive industry as well as electronic and medical devices. However, the use of PEEK is limited because of its low hardness, poor scratch resistance, and high susceptibility to gases and chemicals. Surface modification by depositing an amorphous carbon film is one of the promising methods to improve the surface properties of the polymeric materials because amorphous carbon films have many desirable properties including improved hardness, excellent chemical, thermal stability, and so on. Recently, a number of techniques have been used to coat polymer substrates with amorphous carbon films, including plasma-enhanced chemical vapor deposition (PECVD), ion beam assisted deposition (IBAD), and pulsed laser deposition (PLD). In this work, plasma immersion ion implantation and deposition (PIII&D) is used for the first time to produce the amorphous carbon film on PEEK substrate. Medium to high energy ion bombardment which is intrinsic to PIII&D provides ion mixing ensuring successful depositing of a hard film on a soft substrate in spite of the large discrepancy in the mechanical properties between the two types of materials. We will report the structure and mechanical properties of...
the coated PEEK materials. The film structure is evaluated by X-ray photoelectron spectroscopy and glancing angle X-ray diffraction. The surface morphology is determined by atomic force microscope and hardness by nanoindentation measurements. The scratch resistance of the a-C:H/PEEK is assessed by nano-scratch tests.

10:40am E2-1-10 Surface Morphology of Compressively Stressed Film Deposited on a Soft Polymer Using PECVD. M.W. Moon, J.W. Yi, K.-R. Lee (krlie@kist.re.kr), Korea Institute of Science and Technology, Korea

It has recently been shown that focused ion beam (FIB) irradiation induces a wrinkled skin on the surface of polydimethylsiloxane (PDMS), providing a robust technique for creating wrinkling patterns on selective areas of PDMS. However, a key limitation of this technique is the size of the area exposed to ion beam (confined to 500 micron 500 micron maximum due to the magnification limitation of the FIB system). This restriction hinders the application of this technique where the desired area size of the created pattern is in the order of square millimeter or even square centimeter (e.g. cell templates for tissue engineering and surface modification for enhancing the wear characteristics of polymers). This restriction is overcome in our recent experiments by coating the compressively stressed thin film on soft polymer surface where ordered wrinkles can be created over a large area of the polymeric substrate. Using plasma enhanced CVD technique (PECVD), a diamond-like carbon (DLC) film with a elastic modulus of ~100GPa was deposited on a soft PDMS with the range of 1~10MPa in elastic modulus. The wavelength of the wrinkling patterns of DLC films was controlled by varying the thickness of DLC film and the deposition condition of the implanted PET. The adhesion strength of the coated PET was much better than that of DLC coating on the PET without ion implantation. Detailed experiment results will be presented.

11:00am E2-1-12 Nano and Micro Indentation and Scratch Tests of Mechanical Properties of Thin Films. N. Gitis (NGitis@ctcrt.com), L. Hermann, S. Kairy, Center for Tribology, Inc.

Experimental comparison of common nano and micro scales for hardness evaluation has been performed by atomic force microscopy and hardness measurement of the films. The indents under 5-10% of the film thickness have produced repeatable and apparently substrate-independent results.

In the micro-indentation and nano-indentation tests, the indents under 5-10% of the film thickness have produced repeatable and apparently substrate-independent results. In the micro-scratch and nano-scratch, the scratches under 30-35% of the film thickness have produced repeatable substrate-independent results. In the micro-indentation tests, traditional Rockwell and Vickers hardness tests produced more data variability than the instrumented-hardness tests with the same indenters in the same test setup, though the statistics requires more data.