# DS 7 Mechanical properties of thin films

Time: Monday 16:00–18:00

# DS 7.1 Mon 16:00 GER 38 $\,$

Characterization, mechanistic study and mechanical properties of diamond/β-SiC nano-composite thin films — •VENKATA SATYA SIVA SRIKANTH VADALI, THORSTEN STAEDLER, and XIN JIANG — Institute of Materials Engineering, University of Siegen, Paul-Bonatz-Str. 9-11, 57076 Siegen, Germany

Diamond thin films often feature poor adhesion on many substrates due to high mechanical stresses induced by a difference in the thermal expansion coefficient between the diamond film and the substrate. One option to overcome this difficulty is to prepare composite films consisting of diamond and a second phase. In this work the synthesis of smooth nanocrystalline diamond/ß-SiC composite films, which are prepared by a microwave plasma assisted chemical vapor deposition process usually used to deposit pure diamond films in combination with the introduction of tetramethylsilane (TMS), will be reported. The composition of the films is controlled by adjusting the TMS concentration in the reaction gas. Changing the TMS flow during a deposition process even allows for gradient composite layers. The morphology and the crystallographic structure of these composite films were characterized by SEM, XRD and Raman spectroscopy. The surface roughness of the films was analysed by AFM. Both, hardness and friction co-efficient values of films prepared with various TMS flow rates were obtained using nanoindentation techniques and will be discussed.

## DS 7.2 Mon 16:15 GER 38

Phase stability in ultra thin multilayer systems containing Zr, Ta, Nb, Mo and Fe or Fe(Zr) alloy layers — •ANDREAS GROB, MOHAMMAD MUJEEBUDDIN, and ULRICH HERR — Universität Ulm, Abteilung Werkstoffe der Elektrotechnik, Albert-Einstein-Allee 47, 89081 Ulm

The properties of binary ultra thin multilayer systems undergo significant changes with respect to the bilayer period. Depending on the selected material combination for the constituent single layers structural, elastic and magnetic properties are affected. Various investigation techniques such as x-ray reflectometry, x-ray diffraction, magnetometry and surface acoustic wave measurements have been carried out on multilayer systems prepared by DC magnetron sputtering under high vacuum conditions. The systems studied consisted of Zr, Ta, Nb and Mo as one layer material and Fe or an iron-rich Fe-Zr alloy as the other layer respectively. The results indicate the stabilization of amorphous phases due to the large interface to volume ratio. With increasing layer thickness a transition from amorphous to crystalline phase is observed in many cases. For some material combinations the phase transitions are accompanied by large changes in elastic properties compared to continuum elasticity theory calculations of the average elastic properties expected for the corresponding multilayers. Additionally a thermodynamic model is used to predict the phase stability for different single layer thickness.

#### DS 7.3 Mon 16:30 GER 38

Evaluation of Adhesion of Diamond/Tungsten carbide Composite Films Prepared by Microwave Plasma Assisted CVD. — •HISHAM ABU SAMRA, RUIJIANG HONG, THORSTEN STAEDLER, and XIN JIANG — Institute of Materials Engineering, University of Siegen, Siegen, Germany

Diamond coatings are in demand in a wide range of applications. Unfortunately, the adhesion issues of this film system in the context of many technical substrates are still to be solved. One proposed solution is to increase the adhesion strength by depositing a functional gradient diamond/carbide composite film. In this study, we investigate the adhesion strength of the following coating systems: (i) diamond (ii) diamond/tungsten carbide and (iii) diamond with diamond/tungsten carbide composite as an interlayer on tungsten and WC-Co substrates. The films were prepared in a single microwave plasma assisted chemical vapour deposition (MWCVD) process. The adhesion strength was assessed by employing indentation tests using a Brinell indenter with loads up to 1225 N. In addition, Finite-Element-Method (FEM) simulations were carried out to calculate the stress state and the stress distribution of these systems. The simulation results are correlated with the experimental findings. Room: GER 38

DS 7.4 Mon 16:45 GER 38  $\,$ 

Yield strength of soft and brittle porous materials by nanoindentation — •MATTHIAS HERRMANN<sup>1</sup>, NORBERT SCHWARZER<sup>2</sup>, and FRANK RICHTER<sup>1</sup> — <sup>1</sup>Solid State Physics, Institute of Physics, TU Chemnitz, Germany — <sup>2</sup>Saxonian Institute of Advanced Surface Mechanics, Eilenburg, Germany

Yield stress and Young's modulus of the above mentioned materials have been investigated in order to understand the material behaviour under typical loading conditions for instance during the chemical-mechanical polishing process.

For the characterization of such soft and brittle layers, no adequate method exists. Two usable methods for the determination of yield stress of porous materials will be demonstrated which are based on Pharr's concept of the effectively shaped indenter. It assumes the plastically/elastically deformed zone beneath the indenter as an equivalent indenter and enables one to evaluate the complete elastic stress field during nanoindentation. One of us (N.S.) has proposed an extension of Pharr's concept which allows the evaluation of the yield stress for the case of higher amounts of inelastic deformation even at low applied loads.

Our investigations were done on mesoporous  $SiO_2$  xerogel as well as MSQ-based films on silicon with porosities of 30 up to 57 volume percent. The film thickness was in the range of 600 nm. The yield strength was in the range of 75 - 150 MPa and the Young's modulus between 1 and 4 GPa, depending on material (xerogel or MSQ) and porosity.

DS 7.5 Mon 17:00 GER 38

**Stress in TiO**<sub>2</sub> **thin films** — •JANIKA BOLTZ, DIETER MERGEL, NICOLAS WÖHRL, and BUCK VOLKER — Thin film working group, Physics Department, University Duisburg-Essen, 45117 Essen

Thin films of  $TiO_2$  have been prepared on silicon substrates by rf diode sputtering. Substrate temperature, sputter pressure and oxygen content in the sputter gas have been varied to obtain a variety of microstructures.

For every sample, mass density, film stress and crystallinity have been determined. The mass density ranges between 3.2 and 4.2 g/cm<sup>3</sup>. The films exhibit amorphous, anatase, rutile and mixed structures depending on the preparation conditions.

The film stress is in the range +0.2 GPa (tensile) to -2 GPa (compressive). It is mainly determined by the sputter pressure during deposition and the mass density of the films. By measuring the temperature dependence of the film stress, intrinsic and thermal stress can be distinguished.

## DS 7.6 Mon 17:15 GER 38

Cubic boron nitride coatings (c-BN) on cemented carbide cutting tools — •ULRIKE SPRINGBORN<sup>1</sup>, SUNG-TAE PARK<sup>1</sup>, ERIC WIE-MANN<sup>2</sup>, KAI WEIGEL<sup>1</sup>, MARTIN KEUNECKE<sup>1</sup>, and KLAUS BEWIL-OGUA<sup>1</sup> — <sup>1</sup>Fraunhofer Institute for Surface Engineering and Thin Films, Bienroder Weg 54 E, D-38108 Braunschweig — <sup>2</sup>Institute for Machine Tools and Factory Management, TU Berlin, Pascalstraße 8 - 9, D-10587 Berlin

Cubic boron nitride is the 2nd hardest of all known materials. In combination with other promising properties, like very high wear resistance, c-BN is a very attractive tool coating material. With a modified PVD sputter technique from a boron carbide target c-BN coatings with thicknesses over 1  $\mu$ m could be deposited on pre-coated cemented carbide cutting inserts. After a first adhesion layer of a hard material, like TiN or TiAlN, a boron carbide layer is deposited. In the following B-C-N gradient layer deposition the c-BN nucleation takes place. The total coating thickness is in the range of 3 - 4  $\mu$ m with a 1 to 2  $\mu$ m thick c-BN top layer. The coating process and results from different mechanical and tribological characterisation methods will be presented in detail. The mechanical properties of the super hard, nano-crystalline c-BN coating systems with hardness above 60 GPa and Young's modulus about 550 - 800 GPa are comparable with c-BN bulk material. The feasibility of c-BN coating systems as a super hard tool coating will be verified by results of cutting tests with coated cemented carbide cutting inserts.

#### DS 7.7 Mon 17:30 GER 38

Residual Stress Control in Nanocrystalline Diamond Films — •NICOLAS WÖHRL and VOLKER BUCK — Thin Film Technology Group, Dept. of Physics, University of Duisburg-Essen, Universitätsstr. 3-5, 45141 Essen, Germany Nanocrystalline diamond films were deposited with a microwave CVD plasma source. The nanocrystalline films shown here were deposited in a pressure range between 200 and 300 mbar from an Ar/H<sub>2</sub>/CH<sub>4</sub> plasma. The films were characterized by two wavelength scanning micro Raman spectroscopy, FTIR and SEM measurements.

Residual stress is a critical parameter in thin film deposition and crucial for technical applications of nanocrystalline diamond films because the adhesion of the films on the substrate is affected by the intrinsic stress. High residual stress can lead to cracking or even to delamination of the film from the substrate. An ex-situ optical device (SSIOD 'Surface Stress Induced Optical Deflection') was used to measure the curvature of silicon substrates coated with nanocrystalline diamond films. With respect to Stoney's equation one can calculate the residual stress from the curvature of the substrate. Taking the different thermal expansion coefficients of the diamond and the silicon substrate into account the intrinsic stress was determined from the stress measurements. It is shown that the intrinsic stress in the substrate can be varied in a wide range just by controlling the deposition parameters. A possible explanation for the origin of the intrinsic stress is given based on the data taken from Raman Spectroscopy and FTIR Spectroscopy.

## DS 7.8 Mon 17:45 GER 38

Thermomechanical Properties of Thin  $\alpha$ -Iron Films Above Room Temperature — •THOMAS WÜBBEN, ANDREAS SCHNEIDER, GUNTHER RICHTER, and EDUARD ARZT — Max-Planck-Institut für Metallforschung, Stuttgart

The mechanical properties of thin metal films as compared to their bulk counterparts have been in the focus of materials science in the recent years. Owing to their technological importance, almost only metals with a face centered cubic structure like copper and aluminum have attracted scientific interest. Thin films made of bcc metals, on the other hand, have been largely neglected. However, from a scientific point of view, the mechanical properties of bcc metals are of special interest. As an example, the yield stress of bcc metals is strongly temperature dependent for low temperatures, while it shows a behavior similar to fcc metals for higher temperatures. The mechanisms for this so-called brittle to ductile transition (BDT) are still not understood in full detail. A major problem is the understanding of dislocation dynamics in bcc systems. As a model system we chose iron with a BDT temperature slightly above room temperature. A first step is to verify in thin films that for temperatures above the BDT the thermomechanical behavior of bcc metals resembles that of fcc metals. To test this, we applied the substrate curvature method under vacuum to thin iron films for temperatures from 40°C to 540°C. We will present results of these measurements obtained with iron films with thicknesses down to 100 nm and compare these to the thermomechanical behavior observed for metals with fcc crystal structure.