Thin Films Tuesday

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High-resolution elemental depth profiling of PIII&D deposited multilayer coatings by ion beam techniques combined with EFTEM — ◆FLORIAN SCHWARZ<sup>1,2</sup>, JÖRG LINDNER<sup>1</sup>, MAIK HÄBERLEN<sup>1</sup>, GÖTZ THORWARTH<sup>1,2</sup>, CLAUS HAMMERL<sup>2</sup>, WALTER ASSMANN<sup>3</sup>, and BERND STRITZKER<sup>1</sup> — ¹Institut für Physik, Universität Ausgburg, 86135 Augsburg, Germany — ²AxynTeC Dünnschichttechnik GmbH, Am Mittleren Moos 48, 86167 Augsburg, Germany — ³Sektion Physik der LMU München, Am Coulombwall 6, 85748 Garching, Germany

The emergence of multilayered and nanostructured coatings requires analysis methods capable of high spatial resolution as well as high depth range. While traditional ion beam analysis methods are capable of accurate, standards-free determination of sample composition, methods such as energy filtered transmission electron microscopy (EFTEM) offer the desired short-range resolution, yet are deficient in the quantitative assessment of the elemental contributions. We demonstrate the combination of IBA (ERDA, RBS) measurements with EFTEM data for analysis of two protective multilayer-type coatings grown by plasma immersion ion implantation and deposition (PIII&D) resulting in high resolution elemental depth profiles.

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Preparation of TEM cross-sections and HRTEM structure determination of thin  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  films — •Thomas Riedl, Thomas Gemming, and Klaus Wetzig — IFW Dresden, P.O. Box 270116, D-01171 Dresden, Germany

For the determination of lattice distortions of crystalline films by means of HRTEM well prepared TEM specimens are required. The quality of specimen preparation can be quantified in terms of amorphization, impurity content and specimen morphology. Conventional preparation using ion milling as well as the focussed ion beam H-bar technique have been applied to produce cross-sections of La<sub>0.7</sub>Sr<sub>0.3</sub>MnO<sub>3</sub> /SrTiO<sub>3</sub> samples interesting for magnetoelectronics. Thickness maps near the specimen rim indicate that under the applied parameters particularly the Bal-Tec RES ion mill produces large thin areas with small wedge angles and bending. Low-energy milling at 0.5keV reduces amorphized rims below 1nm leading to an enhanced atomic-column contrast in HRTEM images. The lattice distortions within the La<sub>0.7</sub>Sr<sub>0.3</sub>MnO<sub>3</sub> films have been studied by the analysis of HRTEM geometric phase [1]. As expected the lattice planes perpendicular to the interface are expanded whereas the parallel planes are compressed relative to the bulk [2].

M. J. Hytch et al.: Ultramicr. 74 (1998) 131

[2] We acknowledge the DFG for financial support via FOR 520, project GE 1037/8.

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Modeling asymetric polarization hysteresis of BaTiO<sub>3</sub>-ZnO heterostructures —  $\bullet$ V. M. VOORA<sup>1</sup>, N. ASHKENOV<sup>1</sup>, T. HOFMANN<sup>2</sup>, M. LORENZ<sup>1</sup>, M. GRUNDMANN<sup>1</sup>, and M SCHUBERT<sup>2</sup> — <sup>1</sup>Institut für Experimentelle Physik II, Universität Leipzig, Leipzig, Germany — <sup>2</sup>CMRA, University of Nebraska-Lincoln, Lincoln, USA

The spontaneous polarizations of appropriately oriented wurtzite and perovskite material layers cause bound charges at their interfaces. Whereas the wurtzite-type polarization is inherently tied to one distinct lattice direction, the spontaneous polarization can be reversed within the perovskite lattice upon application of external electric fields. We have successfully grown high-quality Pt-BaTiO\_3-ZnO-Pt layer structures by Pulsed Laser Deposition on Si-substrate and investigated the structural, electrical, and optical properties of these structures. The asymetric polarization hysteresis of the Si-Pt-BaTiO\_3-ZnO-Pt heterostructures show distinct fingerprints of a Schottky-type junction formed at the BaTiO\_3/ZnO interface. For positive voltage direction the hysteresis is dominated by a clear reverse diode behavior, whereas for the negative voltage direction the clear switching behavior of BaTiO\_3 is present. A quantitative model analysis of the electrical measurements is presented.

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Coherent X-Ray Reflectivity at the Energy Dispersive EDR-Beamline at BESSY II — ◆TOBIAS PANZNER¹, GUDRUN GLEBER¹, TUSHAR SANT¹, IVAN VARTANYANTS², and ULLRICH PIETSCH¹ — ¹Universität Siegen, Fachbereich 7, Festkörperphysik, Emmy-Noether-Campus, Walter-Flex-Str. 3, 57068 Siegen — ²DESY Hamburg

3rd generation storage rings provides partly coherent radiation allowing for new kind of x-ray experiments. Adapting knowledge and tech-

niques from the photon correlation spectroscopy with visible light (PCS) many successful experiments are published where sample became under investigation which are opaque in PCS. The advantage of coherent x-ray experiments is the reconstruction of surfaces on micrometer to nanometer length scale (static speckle experiments) or the observation of dynamic processes (XPCS) at surfaces and interfaces on the same length scale. One major drawback of standard x-ray experiments is that only intensities can be measured. In case of coherent x-ray scattering this problem can be overcome by reconstruction of the missing phase information by the so-called phase retrieval procedure. In our poster we show this procedure for energy-dispersive coherent scattering where the development of phase is considered along the whole beam passage from the incoming pinhole through the scattering by sample up to the detector. Taking the known phase information of the pinhole into account we are able to reconstruct the true surface of the illuminated sample area more precisely.

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FT-IR studies of Ag/MgO(001) — ◆FANZHEN MENG, DANIEL SEIBEL, GERHARD FAHSOLD, and ANNEMARIE PUCCI — Kirchhoff-institut für Physik, Heidelberg University, Im Neuenheimer Feld 227, D-69120 Heidelberg, Germany

We present the IRRS (Infrared reflection spectra) of Ag films measured during their growth on MgO(001) at room temperature, at 100 K, and at 50 K. We get a reflectance minimum at a certain thickness. This thickness is comparable to the percolation threshold that we know from our previous IR transmittance measurements [1]. Beyond the percolation threshold, the IRRS of room temperature prepared films show structures quite different to those grown at low temperature. Also, we will show the effect of gas exposure during metal deposition on IR spectra and film morphology at room temperature. For the Ag/MgO(001) system, CO does not show an effect, different to Cu/MgO(001) [2]. However, we detected that hydrocarbon exposure leads to enhanced film roughness. From surface enhanced IR absorption (SEIRA) of adsorbates we get additional information on film morphology.

[1] F. Meng, G. Fahsold and A. Pucci, Phys. Stat. sol.(c), accepted.

[2] M. Lust, A. Priebe, G. Fahsold and A. Pucci, Surf. Interface Anal.33, 487 (2002).

DS 24.38 Tue 15:00 P2

Effect of film thickness on the microstructures of Indium - Indium oxide composite films — • Deniz Deger and Kemal Ulutas — Istanbul Univ., Science Faculty, Physics

Pure indium metal thermally evaporated in the presence of oxygen atmosphere, with partial pressure of  $5\mathrm{x}10^{-4}$  Torr, onto glass substrates and onto C-Cu grid at room temperature. The structural characteristics of these optically transparent and electrically conducting thin films were investigated using XRD and TEM techniques and the results are discussed on the base of the differences in their morphologies and thicknesses. Cubic  $\mathrm{In_2O_3}$  and tetragonal In phases, with crystal structures and lattice parameters as reported in the literature, have been identified in the thinnest film having 1000 Å thickness. The tendency for amorphization of the cubic and tetragonal phases becomes evident as the film thickness increases.

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Direct Observation of Intermediate Phases of Pyrolytic Carbon by Atomic Force Microscopy — ◆ANDREAS PFRANG¹, YONG-ZHONG WAN¹, and THOMAS SCHIMMEL¹,² — ¹Institute of Applied Physics, University of Karlsruhe, D-76128 Karlsruhe, Germany — ²Institute of Nanotechnology, Forschungszentrum Karlsruhe, D-76021 Karlsruhe, Germany

Although it is technologically highly relevant, the mechanism of pyrolytic carbon deposition is not yet fully understood. Especially the role and even the existence of intermediate phases of carbon during deposition are not clear. In our experiments, islands and layers of pyrolytic carbon were deposited on planar substrates in a hot-wall reactor from methane / argon mixtures. Combined scanning force techniques were applied to reveal two types of islands by different chemical contrast. This observation can be interpreted in terms of an intermediate phase of pyrolytic carbon [1]: for deposition in a regime where the nucleation mechanism dominates, an intermediate phase of pyrolytic carbon was predicted which is expected to have deviating mechanical properties in good agreement with our results of island removal experiments carried out using atomic force microscopy.

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Moreover, on layers deposited at sufficiently high methane pressures where adsorption saturation is reached, additional carbon structures exhibiting different chemical contrast were found. This is further experimental evidence for the existence of an intermediate phase of carbon postulated for deposition in the nucleation mechanism.
[1] Z.J. Hu, K.J. Hüttinger. Carbon 40 (2002), 617-636

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FTIR-ATR study of the interface between Al<sub>2</sub>O<sub>3</sub> and H-terminated SiC(0001) and Si(111) — ◆F. Speck, K.Y. Gao, K. Emtsev, Th. Seyller, and L. Ley — Lehrstuhl für Technische Physik, Universität Erlangen-Nürnberg, Erwin-Rommel-Str. 1, D-91058 Erlangen, Germany

Aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) is an insulator which can be regarded as an alternative to thermally grown SiO<sub>2</sub> as gate dielectric for MOSFETs on Si as well as on SiC. We have studied the composition of the interface between the dielectric Al<sub>2</sub>O<sub>3</sub> and the semiconductors Si and SiC. Al<sub>2</sub>O<sub>3</sub> films were grown by atomic layer deposition (ALD) on hydrogenterminated SiC(0001) and Si(111) substrates. Surface hydrogenation of SiC(0001) was performed by annealing in ultrapure hydrogen. On Si(111) a wet-chemical treatment by etching in NH<sub>4</sub>F was employed. The interfaces were investigated for Si-H bonds by Fourier-transform infrared attenuated total reflection spectroscopy (FTIR-ATR). The spectra show that on both SiC(0001) and Si(111) Si-H entities are present at the interface after the ALD process. The characteristic absorption line of the Si-H stretching vibration is broadened and red-shifted as compared to Si-H modes on the hydrogenated substrates. Shift and broadening are probably due to electrostatic interactions at the interface. The presence of Si-H bonds suggests that substrate atoms not connected to the aluminum oxide remain saturated by hydrogen atoms.

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Ultra thin Aluminium oxide films on silicon — •Mandana Roodbari Sh. and Ali Bahari — Physics Department, Mazandran University. Iran

Ultra thin aluminium oxide films, have been identified as potential candidates to replace conventional silicon oxide gate dielectrics in current and future CMOS. Because a shrinking of the silicon oxide thickness with one atomic layer for the next generation will lead to a couple of orders of magnitude increase in tunneling current. Another critical issue for future generations is gate oxide degradation due to boron penetration into the oxide from the poly-silicon gate electrode. We have demonstrated a number of new processes to grow ultra thin aluminium oxides. These sudies have demonstrated a number of new processes to grow ultra thin aluminium oxides.

Two step processes have been employed including evaporation of aluminium to less than monolayer coverage followed by oxygen oxposure. For these investigations of nano-properties and atomic growth processes, the availability of synchrotron radiation with high quality and stability, as met at ASTRID, Aarhus in Denmark, has been important.

Therefore, the present method can be used to deposit uniform aluminium oxide layers of the relevant effective thickness for coming generations of devices directly on silicon surfaces, with atomically sharp interfaces.

DS 24.42 Tue 15:00 P2

Abscheidung von siliziumhaltigen Schichten auf Mikroteilchen in dielektrisch behinderten Plasmen unter Atmosphärendruck — •MARCEL HÄHNEL, VOLKER BRÜSER, and HOLGER KERSTEN — INP Greifswald, F.-L.-Jahn Straße 19, 17489 Greifswald

Die vorliegende Studie befaßt sich mit der Abscheidung von homogenen und geschlossenen  $\mathrm{SiO_2}$ -haltigen Schichten auf Mikroteilchen. Diese Schichten wurden aus Hexamethyldisiloxan (HMDSO) und Tetraethylorthosilicat (TEOS) unter Beimischung verschiedener Gaszusammensetzungen deponiert. Die Untersuchungen zur Abscheidung solcher  $\mathrm{SiO_2}$ -haltigen Schichten erfolgte auf Kaliumbromidpulver in der Größenordnung von 10 bis 80 Mikrometer. Für die Beschichtung wurde eine dielektrisch behinderte Oberflächenentladung verwendet, die durch Modifikationen auch für eine kontinuierliche Arbeitsweise geeignet ist. Die Entladung wurde gepulst mit Spitzenspannungen von 14 kV bei einer Pulswiederholrate von 10 kHz betrieben. Als Spannungsquelle diente ein Fourier-Synthese Impulsgenerator mit einer Ausgangskapazität von 200 pF.

Die Bewertung der Schichten erfolgte durch Oberflächenanalytik (FTIR, REM), sowie makroskopischer Tests zur Bestimmung der physikalisch-chemischen Eigenschaften.

DS 24.43 Tue 15:00 P2

Electrophysical properties of TiN thin films deposited by plasma treatment — •ELENA SHCHERBAKOVA — Minsk, Belarus

In this work the dependence of resistivity of titanium nitrides thin films upon changes in their structure and phase composition as a result of processing with hydronitrogen plasma was found. By means of transmission-electron microscopy and electron diffractometry regularity of structural and phase transformations in titanium thin films irradiated with plasma of arc discharge were investigated. Conditions of processing by plasma for formation of titanium nitrides thin films with resistivity 50  $\mu\rm{Ohm/cm}$  were determined.

The results of studies show that the titanium films obtained have resistivity  $\approx\!110~\mu\mathrm{Ohm/cm}.$  This films polycrystalline and fine-grained, with the average grain size of 5-10 nm. When the films is exposed to hydronitrogen plasma at 500 °C, TiN and Ti<sub>2</sub>N are formed and resistivity is increased to 210  $\mu\mathrm{Ohm/cm}.$  As treatment temperature is increased to 600°C, the nitride phase having a small amount of nitrogen disappears, and a film of golden colour, consisting entirely of TiN, is formed. Further increasing of temperature to 750°C does not change phase composition, but the average grain size is increased to  $\approx\!120$  nm. At temperatures up 600 to 750°C titanium nitride films had a small resistivity of 50-60  $\mu\mathrm{Ohm/cm}.$ 

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Structural evolution in reactively sputtered copper nitride films — •F. USLU<sup>1</sup>, M. LUYSBERG<sup>2</sup>, K. SARAKINOS<sup>1</sup>, P. KARIMI<sup>1</sup>, and M. WUTTIG<sup>1</sup> — <sup>1</sup>I.Physikalisches Institut, RWTH Aachen, 52056 Aachen — <sup>2</sup>IFF, Forschungszentrum Jülich, 52425 Jülich

Early transition metal nitrides such as TiN or ZrN are well known for their applications, which include hard coatings due to their high hardness and high melting temperatures. Much less is known about the physical properties of late transition metal nitrides such as copper-nitride. Reactive dc magnetron sputtering has been applied to prepare coppernitride films on glass and silicon substrates as well. To elucidate the microstructural features of copper nitride films several methods such as X-ray diffraction, grazing incidence geometry and X-ray reflectometry have been employed. In addition transmission electron microscopy has been utilised to obtain a thorough understanding of the microstructural evolution in copper-nitride films. To this end specimens deposited at two different nitrogen flow rates of 12 and 50 sccm N<sub>2</sub> respectively, were analysed. The x-ray investigations reveal that the (111) and (200) grain orientations are stronger than the other ones, where the (111) orientation is dominant. It was possible to decrease this (111) preferred orientation and increase of the (200) orientation by increasing the sputtering current. This is attributed to an enhanced incident ion flux and hence to a bombardment with nitrogen ions. A further effect of this bombardment is reflected in the mechanical properties, where the films reveal compressive stress. The enhanced bombardment leads to an increase of the cell size with increasing nitrogen flow rate.

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Self-organised pattern formation upon femtosecond laser ablation — •OLGA VARLAMOVA $^{1,2}$ , FLORENTA COSTACHE $^{1,2}$ , MARKUS RATZKE $^{1,2}$ , and JÜRGEN REIF $^{1,2}$ —  $^1 LS$  Experimentalphysik II, BTU Cottbus, Karl-Wachsmann-Allee 1, 03046 Cottbus —  $^2 IHP/BTU$  Joint-Lab, Karl-Wachsmann-Allee 1, 03046 Cottbus

Upon multi-shot femtosecond laser ablation from different materials, self-organised regular patterns are observed at the crater bottom. By irradiation with linearly polarised light, it has been shown that long periodic ripples with many bifurcations develop, the orientation of which is determined by the polarisation direction, though the fundamental nature of this correlation is not yet known. To investigate this phenomenon closer, we performed corresponding experiments using circularly and elliptically polarised light. Surface morphology investigation reveals that, again, a variety of self-organised patterns is obtained, from arrays of nanoparticles to bifurcating longer lines. Experiments with laser beams of elliptical polarisation have shown that ripples' orientation is sensitive to the major axe of the polarization ellipse. However, for circularly polarized light the orientation of these structures is random. Furthermore, electrical measurements done with a Scanning-Probe Microscope on the ablated area reveal the existence of a spatial variation in the electric field response correlated with the patterns' modulation on the crater bottom.