

O 75: Oxides and Insulators: Epitaxy and Growth

Time: Thursday 15:15–17:15

Location: MA 042

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Preparation of High-Quality Ultrathin Free-Standing Bismuth Films — •THOMAS PAYER, MANUEL LIGGES, IVAN RAJKOVIĆ, PING ZHOU, DIETRICH VON DER LINDE, MICHAEL HORN-VON HOEGEN, and FRANK MEYER ZU HERINGDORF — Universität Duisburg-Essen, FB Physik, Duisburg, Germany

High-quality ultrathin free-standing metal films are used in many applications like X-ray filters or TEM/TED sample preparation. Deposition on top of sodium chloride templates is a well-known method to grow such films. Typically the crystals are either cleaved in UHV and material is deposited immediately afterwards yielding textured films. Alternatively crystals are cleaved in air making the preparation easier but yielding only lower-quality films. Due to step bunches from the cleaving only small area films can be grown. Our new preparation technique yields high-quality films on a millimeter scale.

We start from large (25mm and above) commercially sold sodium chloride single crystals. As sold, the sodium chloride disks are polished for optical applications in the infrared. The surface exhibits a large roughness and 100nm particle contaminations. Using a water rinse on a spin coater, the contaminations are completely removed. After an additional propanol rinse, AFM shows the presence of atomically flat terraces and regularly spaced atomic steps on the surface. Transfer into UHV yields the clean NaCl LEED pattern. Bismuth films of 20nm thickness are deposited in UHV at room temperature. Ex-situ AFM showed a roughness of only 1.5nm and crystallite sizes of 200nm. Additionally the crystallinity was studied with TED and TEM

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EBSD measurements and optimization of MBE-growth of Aluminum — •RUDOLF NÜSSL, TORSTEN SULIMA, and IGNAZ EISELE — Universität der Bundeswehr, Institut für Physik, Werner-Heisenberg-Weg 39, 85577 Neubiberg

The analysis of microstructure has become an important link between the science and the technology of materials. Since a few years the microstructure-analysis has been revolutionized by a new technique called Electron-Backscatter-Diffraction (EBSD). In this lecture it is shown, how EBSD can be used to optimize metallization systems of microelectronic devices by determining the epitaxial properties of the metallization layers. The metal especially treated in this report is Aluminum, which is deposited on substrates of LiTaO₃ using MBE. When Aluminum is deposited directly on the substrate the metal will grow polycrystalline. In contrast, if a thin matching-layer of Titanium is added on the crystalline substrate, Aluminum shows highly textured or even epitaxial growth. Additionally the texture quality depends on substrate-heating during deposition. The minimum thickness of the Aluminum film to obtain accurate EBSD-measuring-results was determined to be 200nm.

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Thin film growth of Fe₂O₃ on Ag(111) and Al₂O₃(0001) — •MAIKE LÜBBE, ALEXANDER M. GIGLER, and WOLFGANG MORITZ — LMU, Department für Geo- und Umweltwissenschaften, Theresienstr. 41, D-80333 München

Hematite, α -Fe₂O₃, is an interesting and important iron oxide, most notably due to its magnetic and catalytic properties. However, the bulk material is a semiconductor with a wide band gap, $E_g = 2.1$ eV, which renders electron spectroscopic measurements very difficult.

We therefore grew Fe₂O₃ thin films on Ag(111), a system similar to Fe₂O₃ on Pt(111) (see e.g. [1]), which has not been reported up to now. The thin films were grown by MBE at low substrate temperatures, $T_{\text{sub}} \leq 100^\circ\text{C}$, in an O + O₂ atmosphere, $p \approx 10^{-7}$ mbar, using an atomic oxygen source. The samples were characterised by different methods including Auger spectroscopy, XRD, AFM and Raman spectroscopy.

For comparison, we also grew Fe₂O₃ thin films on Al₂O₃(0001), a more commonly known system (see e.g. [2]). We had to choose higher substrate temperatures, $T_{\text{sub}} \approx 500^\circ\text{C}$, to get reasonable results. Analysis revealed that thin films grown on Al₂O₃ are much smoother than those grown on Ag.

XRD measurements that will help to figure out structural differences between Fe₂O₃ thin films grown on Ag(111) or Al₂O₃(0001) are still in progress.

[1] A. Barbier *et al.*, Phys.Rev. B **72**, 245423 (2005)[2] I. J. Lee *et al.*, J. Vac. Sci. Technol. A **23**, 1450 (2005)

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Growth and characterization of ultrathin CeO_x films on Pt(111) — JAN MARKUS ESSEN, TOBIAS PERTRAM, •CONRAD BECKER, and KLAUS WANDEL — Institut für Physikalische und Theoretische Chemie, Universität Bonn, Wegelerstrasse 12, 53115 Bonn, Germany

The use of cerium oxides in catalysis is mainly motivated by its good redox properties and oxygen storage capability. The reproducible preparation of well characterized thin films of CeO_x on a conducting material is important for using these films as model catalyst for the investigation of simple reactions with surface sensitive techniques. In this study we investigated CeO_x films grown on Pt(111) with CO-TPD and HREELS. In order to produce CeO_x thin films we followed three different routes: 1) Oxidation of Pt-Ce surface alloys at different temperatures; 2) oxidation of pure vapor deposited Ce films on Pt(111); and 3) evaporation of Ce in an oxygen atmosphere followed by annealing in oxygen. For Pt-Ce surface alloys oxidized at 900 K HREELS shows a pure CeO₂ film composition. Oxidation at 700 K and 1000 K however, does not lead to full oxidation of Ce, but a mixed oxide is suggested by the phonon spectra. CO-TPD measurements show that the pure CeO₂ films on Pt(111) are not closed. In contrast, exposing the Ce-covered Pt(111) surface to oxygen at 90 K followed by annealing to 1000 K does generate a fully covered surface, but again with a mixed oxide. Evaporating Ce in oxygen atmosphere also produces fully oxidized CeO₂ films but they show no good long-range order.

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Growth of well-ordered Mn_xO_y films on Pt(111): An in-vivo STM/STS study at elevated temperatures — •BENJAMIN BOCHMANN, CHRISTIAN HAGENDORF, STEFFEN SACHERT, and WOLF WIDDRA — Institute of Physics, Martin-Luther-Universität Halle-Wittenberg, Halle

Growth behavior as well as atomic and electronic structure of ultrathin epitaxial manganese oxide films on Pt(111) have been studied using STM/STS and LEED. The films have been prepared by reactive deposition of manganese in an oxygen atmosphere of 10^{-8} to 10^{-6} mbar. STM measurements performed during growth (in vivo) at elevated temperatures (400-600 K) reveal three different well-ordered monolayer structures depending on the preparation conditions: At lowest oxygen pressure a (19×1) uniaxially reconstructed MnO(100)-like layer is formed. STM data which resolve all atoms within one sub-lattice reveal details of the reconstruction. At higher oxygen pressure, an intermediate Mn_xO_y monolayer grows. It is complex, but characterized by sharp LEED pattern which corresponds with atomically resolved STM images of the periodic $1.5\text{ nm} \times 1.5\text{ nm}$ unit cell structure. Under highly oxidizing conditions a quasi-hexagonal monolayer with MnO₂ stoichiometry is formed. LEED shows sharp satellite peaks which can be understood as a Moiré structure with 7% misfit to the Pt(111) substrate. STM images indicate a wagon wheel like reconstruction. The characterization of thicker Mn_xO_y films up to 6 ML is performed by STM and by noncontact-AFM. The latter allows also imaging of insulating films.

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Vibrational and electronic properties of ultrathin MnO(100) films — •SEBASTIAN POLZIN¹, STEFFEN SACHERT¹, KRASSIMIR KOSTOV², and WOLF WIDDRA¹ — ¹Martin-Luther-Universität Halle-Wittenberg, Institute of Physics, Halle — ²Bulgarian Academy of Science, Sofia, Bulgaria

The vibrational properties of ultrathin MnO(100) films on Pt(111) have been studied using high-resolution electron energy loss spectroscopy (HREELS). It was found that the strong optical phonon of the 1st monolayer at 368 cm^{-1} shifts to 382 cm^{-1} with increasing coverage up to 2 ML. At coverages above 1 ML a new phonon at 547 cm^{-1} is observed and identified as Fuchs-Kliwer phonon. Isotopic ¹⁶O/¹⁸O substitution reveals that a collective vibration of the oxygen against the manganese sublattice causes both phonons. For film thickness' up to 13 ML the Fuchs-Kliwer phonon shifts down to 539 cm^{-1} . Electronic excitations in the energy range of 0-7.5 eV show well defined

and narrow d-d band excitations for MnO(100) films from 1 to 10 ML. They converge nicely with increasing thickness towards MnO(100) single crystal data [1].

[1] B. Fromme et al., Phys. Rev. B 58, 9783 (1998).

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Structure of epitaxial cobalt oxide films on Ir(100)-(1×1)
— •DANIELA HOCK, WOLFGANG MEYER, KERSTIN BIEDERMANN, MATTHIAS GUBO, LUTZ HAMMER, STEFAN MÜLLER, and KLAUS HEINZ — Lehrstuhl für Festkörperphysik, Universität Erlangen-Nürnberg, Staudtstr. 7, 91058 Erlangen

Epitaxial cobalt oxide films were prepared on unreconstructed Ir(100). Visual LEED shows the resulting films to be of (111) orientation with, dependent on preparation details, either a distorted or ideal hexagonal unit mesh. In the distorted case there is, above 320 K, a (1×1) phase. The ideally hexagonal phase is accompanied by a (2×2) superstructure. In both cases the film thickness prohibits scattering contributions from the iridium substrate.

LEED intensities were taken for both the distorted (1×1) and the undistorted (2×2) phase. They were analysed by TensorLEED using phaseshifts alternatively for ions and neutral atoms. The distorted (1×1) phase turns out to be of the CoO(111) rocksalt structure with, however, substantial layer relaxations. Also, a stacking fault near the surface appears to be essential to get a good fit quality. In contrast to the distorted (1×1) phase, the undistorted (2×2) structure is due to a spinel-type Co₃O₄(111) surface terminated by an oxygen layer with 1/4 ML Co on top. For both phases convincing comparison between experimental and model intensities is achieved (Pendry R-factor 0.19 and 0.13, respectively). The replacement of ionic by atomic phase

shifts results in the same structural parameters within the limits of errors and similar best-fit R-factors.

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Phases of epitaxial cobalt oxide films on Ir(100)-(1×1) — •KERSTIN BIEDERMANN, MATTHIAS GUBO, DANIELA HOCK, WOLFGANG MEYER, LUTZ HAMMER, and KLAUS HEINZ — Lehrstuhl für Festkörperphysik, Universität Erlangen-Nürnberg, Staudtstr. 7, 91058 Erlangen

The unreconstructed (metastable) Ir(100)-(1×1) surface was used as support for the formation of thin epitaxial films of cobalt oxide. They were prepared by deposition of different amounts of cobalt and simultaneous exposure to an oxygen atmosphere at 320 K followed by annealing at higher temperatures. Dependent on exposure and annealing various structures can be observed. LEED shows that all of them own a hexagonal unit mesh which, however, can be slightly distorted. As a consequence, the oxides formed must consist of polar bilayers.

Ultrathin films (1-2 bilayers) form a c(10×2) superstructure which, according to its appearance in the STM, can be interpreted as rocksalt-type CoO(111) bilayers. Thicker films (≥4 bilayers), when oxygen rich and annealed at up to about 720 K, exhibit an ideal hexagonal unit mesh with a (2×2) superstructure. By further annealing at higher temperatures this transforms (irreversibly) to a ($\sqrt{3} \times \sqrt{3}$)R30° phase with the basic hexagonal unit mesh slightly distorted. As monitored by TDS the transformation is accompanied by the loss of about a quarter of the oxygen content. This suggests that structurally this is a transition from spinel-type Co₃O₄ to rocksalt-type CoO as in fact corroborated by independent quantitative LEED analyses. At about 340 K the ($\sqrt{3} \times \sqrt{3}$)R30° structure undergoes a reversible transition to a (1×1) phase.