

MM 31: Nanocharacterization

Time: Wednesday 10:15–12:15

Location: H 1029

MM 31.1 Wed 10:15 H 1029

HR(S)TEM and TAP analyses of embedded Pb nanoparticles — ●ANNA MOROS, HARALD RÖSNER, and GERHARD WILDE — Westfälische Wilhelms-Universität Münster, Institut für Materialphysik, Wilhelm-Klemm-Str. 10, 48149 Münster

In the present work nanoscaled Pb particles embedded in an Al(Ga) matrix with 3 at.% Ga were processed by melt spinning. Using the ChemiSTEM technology integrated into a FEI Tecnai Osiris 200 kV microscope, elemental mappings have been performed and revealed Ga segregation at grain boundaries. This is a well known process (grain boundary poisoning) leading to an intergranular embrittlement of the Al matrix. However, Ga segregation also occurred at the nanoscaled embedded Pb particles. In order to investigate the particle-matrix interface precisely, high resolution micrographs were taken at the JEOL JEM-ARM 200F. Geometric phase analysis has been used to evaluate the strain state of the heterogeneous particle-matrix interface. It is shown, that the lattice parameter mismatch between the Pb particle and the Al(Ga) matrix is reduced in comparison with the AlPb composite. In order to verify whether the Ga is alloying with Pb or not, Tomographic Atom Probe (TAP) was used. These experimental results are presented and critically discussed. Funding by DFG is gratefully acknowledged.

MM 31.2 Wed 10:30 H 1029

Characterization of nano-particle size and orientation using auto-correlation analysis of electron microscopy images — ●THOMAS WEISS, DIETER AKEMEIER, and ANDREAS HÜTTEN — Universität Bielefeld, 33615 Bielefeld, Germany

The determination of the size, in particular the size-distribution, of nano-particles has been carried out so far by counting each particle. This method is time consuming, whence an automated method is desired to avoid this procedure.

Images of nano-particles taken by electron microscopy are analysed by auto-correlation analysis. Using auto-correlation analysis it allows one to determine the concentration of the particles, the size-distribution and, if existent, the oriented alignment. In order to compare these results nano-particles on an image plane are simulated, where the further analysis has been made by auto-correlation.

This method can be applied to images of any kind, for example of nano-particles observed in electron microscopy or cross sections of magnetic beads.

MM 31.3 Wed 10:45 H 1029

Iron L- and M-edges of iron containing minerals measured by inelastic x-ray scattering — ●ALEXANDER NYROW¹, CHRISTIAN STERNEMANN¹, MAX WILKE², CHRISTOPH SAHLE¹, KOLJA MENDE¹, LAURA SIMONELLI³, ROBERT GORDON⁴, NOZOMU HIRAOKA⁵, FLORIAN WIELAND¹, METIN TOLAN¹, and JOHN TSE⁶ — ¹Fakultät Physik/DELTA, Technische Universität Dortmund, D-44221 Dortmund, Germany — ²Deutsches GeoForschungsZentrum, Section 3.3, Potsdam, Germany — ³European Synchrotron Radiation Facility, F-38043 Grenoble Cedex, France — ⁴PNC-SRF, Dept. of Physics, Simon Fraser University, Burnaby, BC, Canada — ⁵National Synchrotron Radiation Research Center, Taiwan — ⁶University of Saskatchewan, Department of Physics and Engineering Physics, Saskatoon, Canada

Iron is one of the most important elements which form the Earth's mantle. Most physical properties of the Earth's interior have been extracted from seismological observations or structural laboratory studies of phases attendant in the Earth's body. Depending on pressure, temperature and oxygen fugacity the Fe²⁺/Fe³⁺ ratios and the Fe spin-state of iron-bearing minerals can vary significantly. Thus, a quantitative determination of the oxidation state of iron is crucial for the understanding of the thermodynamic properties of these minerals at the conditions of the deep Earth. In this study Fe L_{2/3} and M_{2/3}-edges of different iron oxides and iron containing minerals measured by x-ray Raman scattering are presented in comparison with results of soft x-ray absorption and electron energy loss spectroscopy. Furthermore, the momentum transfer dependency of the Fe M_{2/3}-edge is discussed.

MM 31.4 Wed 11:00 H 1029

X-ray Raman scattering: A spectroscopic tool to study low Z elements at extreme pressure and temperature — ●KOLJA

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In situ studies of materials under conditions of geological relevance, i.e. high pressure and high temperature, can only be performed using diamond anvil cells in combination with resistive or laser heating. Due to highly absorbing sample environments, the study of absorption edges for binding energies between 10 eV and 2 keV of low and intermediate Z elements such as sodium, aluminum, silicon, etc. is hardly possible using electron or soft x-ray spectroscopy. Here, x-ray Raman scattering (XRS), an energy loss spectroscopy using hard x-rays as a probe, provides a unique experimental method. XRS yields similar information as soft x-ray absorption and electron energy loss spectroscopy. It is very sensitive to changes of the electronic and local atomic structure and allows to probe different excitation channels by variation of the momentum transfer. With this method, the partial unoccupied density of states can be determined. The capabilities of this experimental technique for geophysical applications are discussed for selected examples.

MM 31.5 Wed 11:15 H 1029

Probing intermediate valence in rare-earth compounds by X-ray excitations goes along with significant final state effects — ●K. KUMMER¹, YU. KUCHERENKO², S. DANZENBÄCHER², C. KRELLNER³, C. GEIBEL³, S. L. MOLODTSOV⁴, C. LAUBSCHAT², and D. V. VYALIKH² — ¹ESRF, Grenoble, France — ²Institut für Festkörperphysik, Technische Universität Dresden, Germany — ³Max-Planck-Institut für Chemische Physik fester Stoffe, D-01187 Dresden, Germany — ⁴European XFEL GmbH, Hamburg, Germany

Interaction with itinerant valence states can cause an instability of the 4f shell in rare-earth intermetallics. The resulting intermediate valence of the rare-earth ions often depends on applied pressure, temperature or chemical doping and is closely related to the magnetic and transport properties of the material. X-ray spectroscopies like 4f photoemission, X-ray absorption, or resonant inelastic X-ray scattering are very sensitive to different 4f configurations and have thus become a standard tool to study intermediate valent behavior. However, comparing results of the different X-ray spectroscopic techniques among each other and with numbers obtained with low-energy excitation methods one often finds discrepancies larger than the accuracy of each of the employed techniques. We performed theoretical simulations which reveal that final state effects lead to non-linear relations between the the weight of a 4f configuration in the ground state and its spectral weight. Valence determination from X-ray spectroscopic data requires a quantitative characterization of those final state effects which we demonstrate here at the example of intermediate valent Yb compounds.

MM 31.6 Wed 11:30 H 1029

Structural investigations on the interfacial layer between diamonds and metal matrices in diamond tools via X-ray scattering — ●ANDRE STEFFEN¹, MICHAEL PAULUS¹, CHRISTIAN STERNEMANN¹, MANUEL PINHO FERREIRA², CHRISTIAN KRONHOLZ^{2,4}, RALPH WAGNER³, WOLFGANG TILLMANN², and METIN TOLAN¹ — ¹Fakultät Physik/DELTA, TU Dortmund, D-44221 Dortmund — ²Institute of Materials Engineering, TU Dortmund, D-44221 Dortmund — ³Fachbereich C - Abteilung Physik, Bergische Universität Wuppertal, D-42097 Wuppertal — ⁴Benteler Tube Management GmbH, D-33104 Paderborn

Diamond grinding tools have widely established its usage in machining and cutting of hard materials such as natural stone and concrete. These diamond metal composites are mainly fabricated powder-metallurgically. The sintered metal serves as a boundary matrix for the embedded diamond grains. Therefore the bonding type of the diamonds in the metal matrices is of essential relevance. So it is of important interest if the interfacial area between the diamonds and metal matrices consist of metal-carbides, solid solutions of carbon in metal or even graphite. In this work diamond metal (Co, Fe, Cr) composites have been investigated by X-ray absorption near-edge fine structure spectroscopy (XANES) and X-ray diffraction (XRD) in order to anal-

use the structure of the interfacial layer between the diamonds and the metal matrices. First analysis of the XANES data indicate changes in the local structure due to the sintering process. XRD studies show the formation of graphite.

MM 31.7 Wed 11:45 H 1029

Material Processing with Femtosecond Laser Pulses — ●STEFFEN FIEDLER, ROBERT IRSIG, ANNA ONISZCZUK, JOSEF TIGGESBÄUMKER, CONRAD SCHUSTER, ANNA SVANIDZE, NEEKE ROTHE, STEFAN LOCHBRUNNER, and KARL-HEINZ MEIWES-BROER — Institute of Physics, University of Rostock, Germany

Material modifications on the μm -scale are required in many applications, e. g. in micromechanics or modern medical implant technologies. Pulsed laser light sources are powerful tools dedicated to replace conventional methods of mechanical processing in many cases.

In laser machining the working area is confined to the laser focus with minimal effects on the functionality of the surrounding surface. Even higher precision becomes available for ultrashort femtosecond laser pulses by a reduction of heat transfer into the material due to the short interaction time [1]. Furthermore the high intensity of femtosecond laser pulses leads to a different ablation mechanism that in principle allows for processing arbitrary materials. In order to achieve this it is necessary to adapt the laser parameters for each sample. Especially when applying shaped laser pulses an improved precision is accessible [2].

The goal of this project is to optimize the working conditions and the quality of the machining process for different specimens and their implementation in medical applications in particular.

[1] B. N. Chichkov et al., Appl. Phys. A 63, 109 (1996)

[2] L. Englert et al., Appl. Phys. A 92, 749 (2008)

MM 31.8 Wed 12:00 H 1029

Mechanism of nanostructure formation during in-situ consolidation of mechanically-milled copper — ●MOHSEN SAMADI KHOSHKHOO^{1,2}, S. SCUDINO¹, H. BAHMANPOUR³, A. KAUFFMANN¹, J. FREUDENBERGER¹, R. SCATTERGOOD³, M. J. ZEHETBAUER⁴, C. C. KOCH³, and J. ECKERT^{1,2} — ¹IFW Dresden, P.O. Box 270116, D-01171 Dresden, Germany — ²TU Dresden, Institut für Werkstoffwissenschaft, D-01062 Dresden, Germany — ³Department of Materials Science and Engineering, North Carolina State University, Campus Box 7907, Raleigh, NC 27695-7907, USA — ⁴Faculty of Physics, University of Vienna, Boltzmanngasse 5, A-1090 Wien, Austria

Bulk nanocrystalline Cu samples have been produced by in-situ consolidation during mechanical milling. The effect of milling temperature and milling intensity on the structure of the samples was studied in detail and particular attention was paid to reach the optimal conditions for successful in-situ consolidation. Microstructural evolution during milling was monitored using X-ray diffraction analysis assisted with the whole powder pattern modeling (WPPM) technique. The results show that the dislocation density increases continuously with milling time, reaches a maximum and then decreases with further milling. Transmission electron microscopy (TEM) investigations carried out before and after the observed maximum of dislocation density reveal that dynamic recrystallization is responsible for the reduction of dislocation density. The mechanism of nanostructure formation through dynamic recrystallization was studied in detail using high-resolution TEM analysis and scanning electron microscopy.