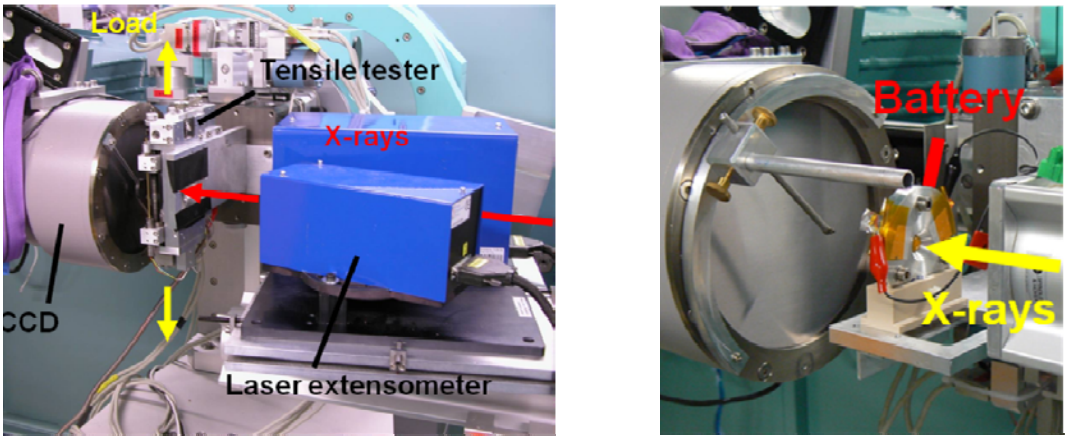
Synchrotron-based *in situ* testing techniques

P. A. Gruber

An accurate measurement of the mechanical properties is necessary to obtain a better understanding of the plasticity of ultra-thin metallic films. Therefore synchrotron-based *in situ* tensile testing techniques were developed and optimized in order to characterize the biaxial stress evolution in ultra-thin metallic films on compliant polymer substrates during isothermal tensile tests. Experimental procedures for polycrystalline (P. Gruber, Acta Mater., 2008-I) as well as single crystalline films (P. Gruber, Acta Mater., 2008-II) were established. Thereby, the variable wavelength of the synchrotron radiation was the key feature for the experiments. For polycrystalline films, monochromatic X-rays of defined energy were necessary to adapt for the very strong <111> fiber texture in the films, whereas a polychromatic X-ray beam was used for single crystalline films. The stress determination during the tensile tests was realized in transmission geometry. Thus, no sample rotation or tilting was necessary and the whole information could be recorded on a big CCD area detector during a single X-ray exposure (15 to 120 s, complete Debye-Scherrer rings for polycrystalline films and Laue patterns for single crystalline films). In this way, the complete stress tensor of polycrystalline as well as single crystalline films with film thicknesses down to 20 nm could be measured with a strain resolution of 10^{-4} and at strain rates up to 10^{-4} s^{-1}. By these techniques, a unique field of testing parameters for ultra-thin metallic films, like different microstructure (unpassivated, passivated, polycrystalline, single crystalline), temperature (-150 to 250°C) and high total strain (up to 25%) was established.



(a) (b)

Fig. 1: Experimental setup at MPI-MF beamline at ANKA (Ansgtrömquelle Karlsruhe) for (a) tensile testing of thin metallic films on polyimide substrates and (b) *in situ* investigation of electrochemical Li intercalation into the electrode material of a Li ion battery cell (cell design by S. Indris, INT, Research Center Karlsruhe)

Since October 2007, these techniques have been extended to nanocrystalline thin films of Pd and Pd alloys (see DFG Research group 714 Project 5) as well as Li ion battery cells (cooperation with R. Mönig, IMFII, H. Gesswein, J. Binder, IMFIII and S. Indris, INT, Research Center Karlsruhe). For nanocrystalline films, and in order to study the evolution of defects during deformation, the *in situ* diffraction method was optimized to record several diffraction peaks simultaneously. A similar approach was made for the Li ion battery cells with the goal to investigate the lattice distortion and possible phase transformations in the electrode material

during electrical loading and unloading. Experiments were carried out on prepared and cycled batteries to clarify which processes limit the reversibility of the electrochemical reactions. Distinct changes in the X-ray spectra (reversible and persistent) during deformation of the Pd films and electrical loading of Li ion batteries were found. Currently, we are developing data evaluation routines for both experiments based on classical Williamson-Hall, Warren-Averbach and Rietveld methods to extract microstructural information like crystallite size, microstrain and defect structure of the materials.

In July 2008, we submitted a proposal for microdiffraction experiments at the Advanced Light Source in Berkeley, CA, USA. It is planned to establish experimental routines for *in situ* testing of single crystalline nanowires and micropillars. This will be done in cooperation with W.D. Nix (Stanford University), N. Tamura (ALS, LBNL Berkeley) and A.M. Minor (NCEM, LBNL Berkeley). By combining these experiments with *in situ* TEM and FIB/SEM experiments on identical samples, it is believed that a more detailed picture of the deformation mechanisms at the submicron and nanoscale can be revealed.