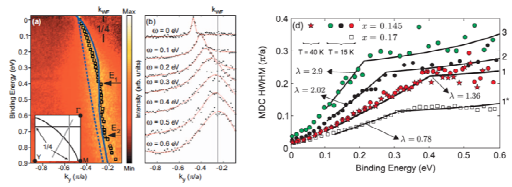
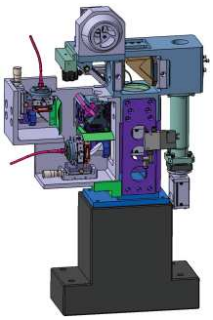
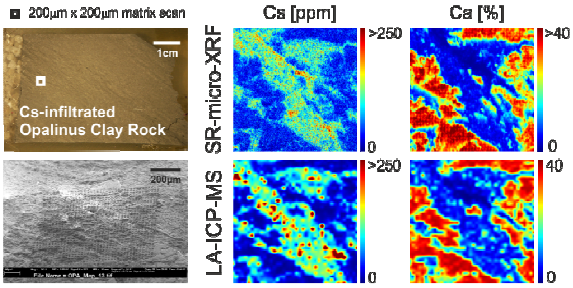
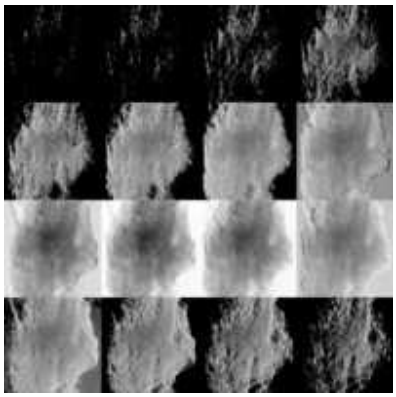
SWISS LIGHT SOURCE









SLS Symposium on

Complimentary and Extended Analyses

Tuesday, July 3, 2012

10:00 to 12:15, WBGB/019

10:00 Light and neutron spectroscopies applied to strongly correlated metals
Johan Chang, N.B. Christensen, S. M. Hayden, C. Monney, J. Mesot, M. Shi, Ch. Niedermayer, H. Rønno and, T. Schmitt

10:30 Third Generation of On-axis in situ Optical Spectroscopy: Extending the Scope of Macromolecular Crystallography
Florian Dworkowski, G. Pompidor,V . Thominet, C. Schulze-Briese and M. Fuchs

11:00 Coffee

11:15 High Spatial Resolution Quantitative Chemical Imaging by Complementary Techniques
H. A. O. Wang, D. Grolimund, L. R. Van Loon, C. N. Borca, J. Shaw-Stewart, P. Karvinen and D. Günther

11:45 Accessing reciprocal space with hard X-ray grating interferometry
Peter Modregger, M. Kagias, F. Scattarella, B. R. Pinzer, C. David, R. Bellotti, and M. Stampanoni

Light and neutron spectroscopies applied to strongly correlated metals

J. Chang[1,2], N.B. Christensen[3], S. M. Hayden[4], C. Monney[2], J. Mesot[1,2], M. Shi[2], Ch. Niedermayer[5], H. Rønnow[2], T. Schmitt[2].

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Metals have played an important role in the past millenniums of human history. Periods, like the bronze and iron age, have been named after the most important metals used at the time. Harnessing semiconductors like silicon has enabled the creation of computers and revolutionized our daily life. The impact of material research is therefore also reflected in Nobel prize nominations. In the past couple of decades, more than a handful of Nobel prizes have been awarded to the discovery or understanding of metal physics such as superconductivity (1987, 2003), colossal magnetoresistance (2007), quantum hall effect (1998), and most recently the discovery of graphene (2010). Landau theory of Fermi liquids and its notion of quasiparticles underlies much of our understanding of how electron interactions affect the properties of a metal. There is, however, a large class of so-called strongly correlated electron systems that defy a description within the Landau Fermi liquid concept. In this talk I will discuss how new insights into these strongly correlated metals can emerge from complementary spectroscopy techniques that can be performed at the Swiss light source (SLS) and neutron spallation source (SINQ). In particular spin and charge excitations of these metallic materials will be discussed.

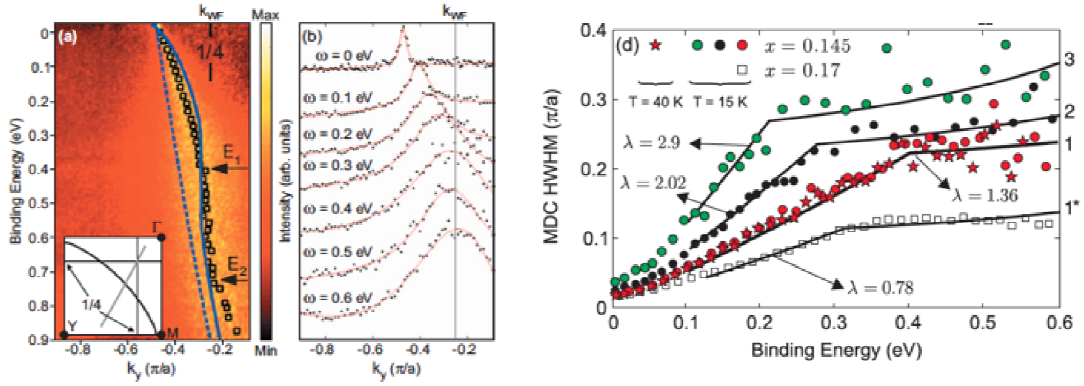

Figure 1: ARPES spectra and analysis (J. Chang et al. PRB 2008) recorded on the strongly correlated metal $La_{2-x}Sr_xCuO_4$ with x = 0.145.

Third Generation of On-axis *in situ* Optical Spectroscopy -- Extending the Scope of Macromolecular Crystallography

Florian Dworkowski*, Guillaume Pompidor*, Vincent Thominet*, Clemens Schulze-Briese+, Martin Fuchs*

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X-ray diffraction based structure determination of biological macromolecules is one of the fundamental tools of a structural biologist. However, the electron density maps obtained by this method are limited to elucidate the three-dimensional structure of a macromolecule, but do not yield information on, for example, the chemical state of co-factors, the redox state of metal centers or disulphides, or the identity of bound ligands. Many of these observations can be directly or indirectly linked to X-ray radiation induced damage to the sample, one of the central limitations of synchrotron x-ray diffraction data collection [1]. To be able to better quantify the extent of this effect and obtain additional complementary data on the sample, the on-axis geometry for *in-situ* combination of optical spectroscopic methods with the diffraction experiment has proven highly effective [2].

At beamline X10SA at the Swiss Light Source (SLS) we now are commissioning the third generation of an on-axis multi-mode micro-spectrophotometer (SLS-MS3). It is fully integrated into the new D3 experimental endstation and designed to remain always online at the beamline, thereby strongly increasing users' acceptance for spectroscopic radiation controls by dramatically reducing setup times. Since the SLS-MS2 instrument started user operation in 2011 we now support UV/Vis, Fluorescence and also Raman and Resonance Raman spectroscopy. With the new instrument we introduced a new modular concept which makes it even more accessible to users with a structural biology background to use all those possibilities.

We present quantitative mappings of the on-axis sampling geometry to demonstrate its advantages for aligning the X-ray and optical beams and thereby sampling volumes. Especially in combination with the high sampling rate provided by the PILATUS 6M pixel detector, this setup is also ideal for kinetic crystallography experiments with observation of reactions on the minute timescale.We show applications of UV/Vis spectroscopy and resonant and non-resonant Raman spectroscopy toward monitoring the photo reduction of metal centers, ligand abstraction and bond breakage. Recently the methodology proved also very useful for determination of specific reaction mechanisms inside of proteins, showing unexpected photo-switching behavior and metal coordination [3,4].

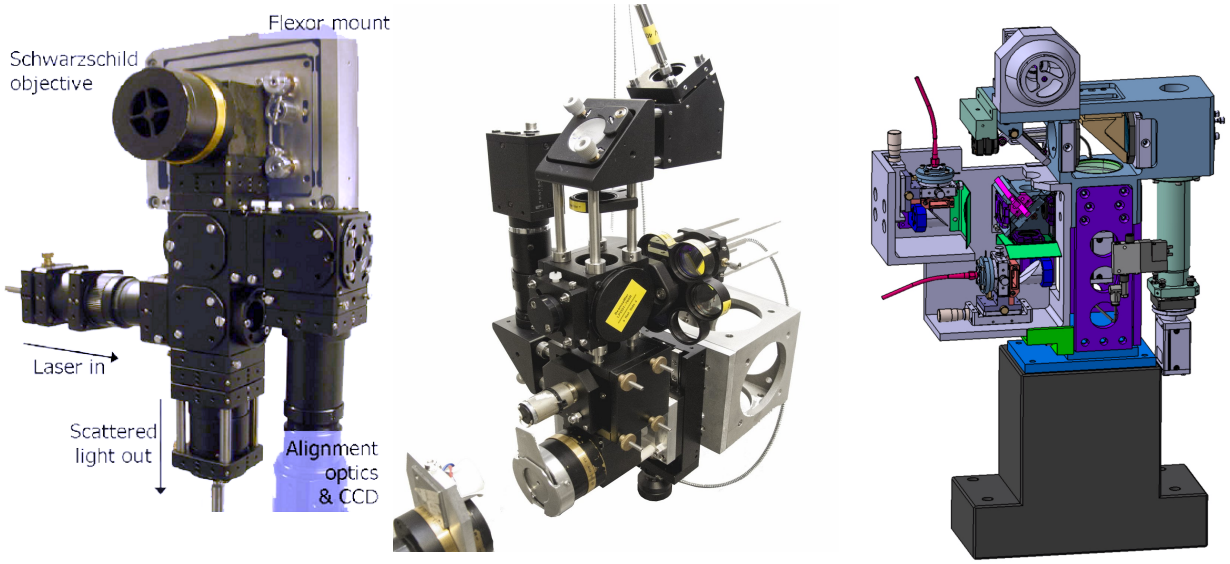

Figure 1: Evolution of the Micro-Spectrophotometer: SLS-MS (UV/Vis), SLS-MS2.6 (UV/Vis, rRaman, Raman, Fluorescence), SLS-MS3 (full endstation integration).

[1] Garman, E. F. and M. Weik. *Journal of Synchrotron Radiation* (2011), **18**(3): 313-317

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[3] Regis Faro, A., P. Carpentier, *et al.*, *Journal of the American Chemical Society* (2011) **133**(41), 16362-16365

[4] He, C., M. R. Fuchs, *et al.*, *Angewandte Chemie International Edition* (2012), **51**(18), 4470-4473

High Spatial Resolution Quantitative Chemical Imaging by Complementary Techniques

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A broad range of scientific and industrial research applications require **quantitative chemical images** with **high spatial resolution**, e.g. for the identification of chemical processes or reaction rate quantification. Trace elements quantification is normally a difficult task in X-ray fluorescence analysis (XRF), also inevitable in high flux Synchrotron-based microXRF (SR-microXRF). However, the high spatial resolution and high throughput of SR-microXRF are advantageous over other chemical analysis techniques. In this work, we combined it with laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS), which provided quantitative output on trace elements, but with a reduced spatial resolution.

We applied this complementary approach on a test sample to investigate the migration of a Cs contamination plume into a heterogeneous geological material (Opalinus clay rock). Both techniques yield 2D chemical images which correlated between the two techniques (Figure 1). Quantitative LA-ICPMS images were utilized to 'cross-calibrate' the qualitative SR-microXRF results, which resulted in high spatial

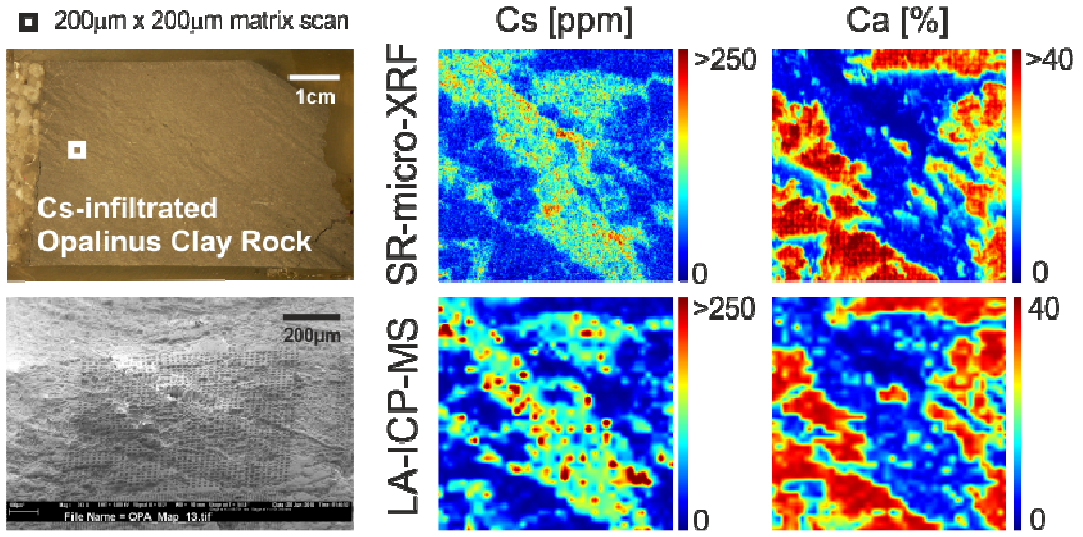

Fig. 1 High spatial resolution chemical imaging of a Cs contamination plume in a heterogeneous geological medium.

resolution quantitative 2D chemical images.

The importance of micro-/nano- properties and chemical relativities in macroscopic materials is gaining more and more recognition. The developing need to give a detailed picture of such (sub-)microscopic structures and features does thrust the development of quantitative chemical analysis with advanced spatial resolution. Recent development of hard X-ray SR-nanoXRF allows chemical imaging with approximately 200 nanometers spatial resolution by a pair of high-efficiency linear zone plates. In parallel, the aerosol transport systems used for LA-ICPMS were studied in detail and optimized accordingly. These modifications provide now the capabilities to ablate materials at a spatial resolution close to 1µm, while maintaining access to trace element concentrations. The complementary application of these two “sub-micron” techniques was tested on micro-structured thin film pattern. Highly correlated chemical images provide evidence of successful high-resolution 2D quantitative chemical imaging based on cross-calibration using microLA-ICPMS and SR-nanoXRF.

References:

H.A.O. Wang, D. Grolimund, L.R. Van Loon, K. Barmettler, C.N. Borca, B. Aeschlimann, D. Günther, Quantitative Chemical Imaging of Element Diffusion into Heterogeneous Media Using Laser Ablation Inductively Coupled Plasma Mass Spectrometry, Synchrotron Micro-X-ray Fluorescence, and Extended X-ray Absorption Fine Structure Spectroscopy, *Anal. Chem.*, **83,** (2011) 6259-6266.

H.A.O. Wang, D. Grolimund, L.R. Van Loon, K. Barmettler, C.N. Borca, B. Aeschlimann, D. Günther, High Spatial Resolution Quantitative Imaging by Cross-calibration Using Laser Ablation Inductively Coupled Plasma Mass Spectrometry and Synchrotron Micro-X-ray Fluorescence Technique, *CHIMIA*, **66,** No 4, (2012) 223-228.

Accessing reciprocal space with hard X-ray grating interferometry

***P. Modregger*[1,2], *M. Kagias*[3], *F. Scattarella*[4,5], *B. R. Pinzer*[1], *C. David*[6], *R. Bellotti*[4,5], *and M. Stampanoni*[1,3]**

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X-ray grating interferometry (GI) constitutes phase contrast imaging technique that provides a particular high sensitivity towards electron density variations in the sample. This characteristic renders GI especially suitable for imaging soft tissue material and, thus, GI has experienced an increasing interest from researchers in the bio-medical field [1]. Up to now, X-ray imaging with GI was regarded to deliver information only in real space. Recently, we have demonstrated that the ultra-small angle X-ray scattering (USAXS) distribution [2] and, thus, reciprocal space information can be accessed with GI as well, and this even simultaneously with real space sensing. This was performed by introducing an alternative approach to the contrast formation process of GI and using the appropriate data analysis procedures for the experimental data. In doing so, we increased the number of complementary image modalities provided by GI from previously three to hundreds.

Compared to established techniques for reciprocal space mapping such as scanning small-angle X-ray scattering (SAXS), USAXS with GI is able to finely resolve reciprocal space around its origin. This implies that USAXS with GI delivers information, which is complementary to techniques like SAXS. However, due to the small sample to detector distance the relationship between the USAXS signal, which we defined as the scatter signal at the detector, and the reciprocal space of the sample is not straight forward. As a consequence we have experimentally observed the occurrence asymmetric scattering distributions. Further, the comparably small momentum transfer indicates that multiple scatter events have to be taken into account if GI-USAXS is to be combined with tomography. In the presentation, we will discuss our recent theoretical investigations into these challenges and show our first experimental examples.

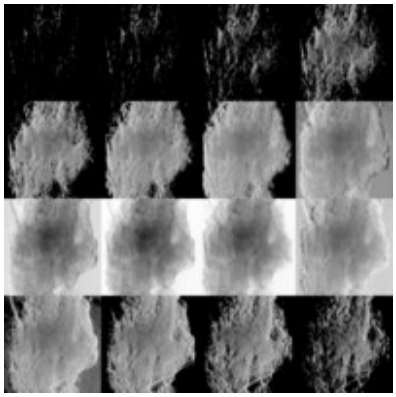

Fig. 1: Scatter images of a human calcified heart valve derived nodule. Each image shows the relative scattering strength for different scattering angles.

References

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