High-brightness liquid-metal-jet-anode electron-impact hard x-ray source

H. M. Hertz, M. Otendal, T. Tuohimaa, and O. Hemberg

Biomedical and X-Ray Physics, Royal Institute of Technology/Albanova, SE-10691 Stockholm, Sweden
hertz@biox.kth.se

Abstract

Hard x-ray (multi keV) synchrotron sources with high brightness have enabled novel and important imaging techniques, from x-ray microscopy to medical imaging with improved and new contrast. Unfortunately the brightness of conventional compact hard x-ray sources has not improved significantly since the invention of the rotating anode, making it difficult to envision compact systems for these new imaging modalities based on present sources. In this paper we describe a novel compact electron-impact hard x-ray source with potential for much higher brightness than present state-of-the-art rotating-anode sources. The source can enable improved resolution and contrast in medical imaging and non-destructive evaluation, and has applicability for compact hard x-ray microscopy and certain diffraction applications.

For electron-impact sources the brightness is proportional to the electron-beam power density at the anode. Present rotating-anode and micro-focus technology show little potential for further improvement due to intrinsic thermal limitations. We have introduced a new anode concept, the liquid-metal jet. Calculations show that this new anode allows a 100-1000× increase in source brightness compared to today’s compact hard x-ray sources1,2. We presently operate it with a 20-50-µm diameter liquid-tin jet at ~70 m/s with a 50 kV, 600 W electron beam focused to 40-50 µm FWHM. The resulting electron-beam power density is 200-400 kW/mm², which is approx 10× higher than on present sources. The emitted spectrum exhibits two tin emission peaks around 25 keV and a broad bremsstrahlung background. Conventional absorption imaging shows excellent spatial resolution (tens of microns). Furthermore, the small source allows phase imaging for improved contrast. Initial results compare well with theoretical modelling.

This paper will describe the source system properties as well as show early high-resolution imaging results, with and without phase effects. We will also discuss the source’s extension to lower (10 keV) and higher (>50 keV) energies. Finally, we will elaborate on the new source’s applicability for hard x-ray microscopy and medical imaging.

Development of high-average power extreme ultra violet source by laser produced plasmas

H. Nishimura, K. Nishihara, S. Fujioka, Y. Tao, T. Aota, N. Ueda, T. Ando, Y. Simada\textsuperscript{1}, M. Yamaura\textsuperscript{1}, K. Hashimoto\textsuperscript{1}, S. Uchida\textsuperscript{1}, Q. Gu, K. Nagai, T. Norimatsu, A. Sunahara\textsuperscript{1}, H. Furukawa\textsuperscript{1}, Y. -G. Kang, M. Murakami, H. Yoshida, K. Tsubakimoto, H. Fujita, M. Nakatsuka, N. Miyanaga, Y. Izawa, and K. Mima

Institute of Laser Engineering, Osaka University
2-6, Yamada-oka, Suita, Osaka 565-0871, Japan
\textsuperscript{1}Institute for Laser Technology, Ibid.

Extreme ultraviolet (EUV) radiation from laser-produced plasma has recently attracted particular attention for use in production of the next generation semiconductor devices of node-size below 40 nm. It is expected to generate over 115 W at 13.5 nm within 2\% bandwidth at the repetition rate of 7-10 kHz. Since 2003 a new project of MEXT has started to provide experimental and theoretical databases for clean and efficient EUV generation, and technical guidelines to build-up an EUV-source system for practical use in industry. The main tasks of the project are

(1) Acquisition of databases: Comprehensive experimental databases are provided for a wide range of parameters of lasers and targets. These experimental data will be utilized to benchmark a radiation hydrodynamic code including equation-of-state solvers and advanced atomic kinetic models dedicated for EUV plasma prediction.

(2) Target design and fabrication: Various types of targets have been proposed such as gas, liquid, low-density solid or clusters. In addition, innovative targets are desired in order to attain acceptably high conversion efficiency and mitigate debris from targets.

(3) Establishment of laser technology: A 5-kW laser at a repletion rate of 5-kHz system is ready to demonstrate efficient EUV source generation under optimized plasma conditions. Present status of the laser produced EUV source development and future prospects for various applications will be discussed.
Hard X-ray Microscopy at the Advanced Photon Source

Jörg Maser

Advanced Photon Source, Argonne National Laboratory
Center for Nanoscale Materials, Argonne National Laboratory

X-ray microscopy at the Advanced Photon Source has, since its beginning in 1997, developed into a powerful, versatile tool that has been implemented at various APS beamlines and is being applied to a large variety of scientific topics. Due to its high brilliance in the hard x-ray range, the Advanced Photon Source lends itself in particular to scanning probe applications that utilize x-ray fluorescence and x-ray diffraction as contrast forming mechanisms. Hard x-ray microprobes using both Fresnel zone plates and Kirckpatrick-Baez mirrors are in operation, with a spatial resolution typically in the range of 100-300 nm. With the development of x-ray optics of higher numerical aperture and concurrent improvements of the microscopy facilities, a spatial resolution of below 100 nm has been obtained more recently.

Microprobes using x-ray fluorescence are being applied in particular to biological, medical and environmental sciences, where they are used to map the distribution of trace metals in tissues, cells and bacteria. Microprobes using x-ray diffraction are typically used in materials sciences, both for direct study of the crystalline structure of materials, and to map strain, e.g. in systems showing ferroelectric and magnetic order. An emerging application is the quest for novel materials and their applications, namely the study of nanoparticles and their interaction with their environment. We will present an overview over current x-ray microscopy activities at the Advanced Photon Source, and provide an outlook for future plans. We will focus in particular on the hard x-ray nanoprobe beamline that is under construction as part of Argonne’s Center for Nanoscale Materials, and aimed to provide a spatial resolution of 30 nm in the hard x-ray range.

Fe distribution in mycobacterium avium after infection of a mouse peritoneal macrophage.
A) Optical image of macrophage
B) Ca map of macrophage
C) Fe map of bacteria

Recent Developments on the X-ray Phase Contrast Imaging and CT in BSRF

Peiping Zhu, Qingxi Yuan, Junyue Wang, Wanxia Huang, Hang Shu, Ziyu Wu

Beijing Synchrotron Radiation Facility, Institute of High Energy Physics, Chinese Academy of Sciences, 100049
Beijing, China

Abstract

Since 2001 hard x-ray phase contrast imaging and computer tomography (CT) methods have been developed at the Beijing synchrotron radiation facility (BSRF). The most recent advancements of these imaging methods are reported below:

1. A model discussing the mechanism of imaging of cylinder-like structures in inline phase contrast imaging has been proposed. Cylinder-like structures are present in many biological and medical samples, and the correlation among contrast, radius, refraction index difference, distance between sample and detector and spatial coherence of the source have been included in this model.

2. The main parameters of the diffraction enhanced imaging (DEI) equation were investigated, the influence of small angle scattering accepted by the analyzer crystal on imaging is discussed and the term to describe the influence was introduced into DEI equation, some results were given in this contribution.

3. The condition for phase contrast CT has been carefully investigated. In particular we demonstrated that the projection of the function to be reconstructed can not be described as a simple line integral along the x-ray path, and in addition this function has to be rotational invariant. An example of the refraction index gradient of CT is discussed in this manuscript.

The new instrumentation is operative at BSRF for users for both phase contrast imaging and CT experiments. Recent experimental results will be presented and discussed.
Volume zone plate development at BESSY

Stefan Rehbein, Stephan Rudolph and Gerd Schneider

BESSY mbH, Albert-Einstein-Str. 15, 12489 Berlin, Germany

State-of-the-art Fresnel zone plates can be described by scalar diffraction theory neglecting the three-dimensional shape of the zone structures. According to this theory their diffraction efficiency scales as \(1/m^2\) where \(m\) is the diffraction order. While keeping the zone height constant, the aspect ratio of the zones increases inversely with decreasing outermost zone width. For photon energies below one keV, it is shown by applying electrodynamic theory that scalar theory is no longer suited to describe zone plates with outermost zone width below 20 nm and aspect ratios of about 10:1 \([1,2]\).

Full electrodynamic theory - which includes forward and backward diffracted as well as evanescent waves - predicts that the diffraction efficiency decreases continuously if the lateral dimensions of the zone width approach the wavelength used for imaging. This result is obtained for zone structures parallel to the optical axis. Unlike the diffraction properties of parallel zone structures, rigorous coupled wave theory (RCWT) predicts for zone structures tilted to the optical axis according to the local Bragg condition that the diffraction efficiency can be up to 50 \% \([3]\). In addition, RCWT calculations show that similar diffraction efficiency values can be obtained in any high order of diffraction \(m > 1\).

The resolving power of zone plates scales with the order of diffraction \(m\). By applying high orders of diffraction, it is possible to increase the resolution without the need for manufacturing increasingly smaller outermost zone width far below 20 nm. Applying high orders for imaging requires manufacturing tilted zone structures with aspect ratios of about 20:1 \([2]\). To overcome the extremely difficult problem of manufacturing tilted zones with high aspect ratios of 20:1, we propose to manufacture zone plates on top of each other with slightly decreasing zone radii \([3]\). In good approximation – depending only on the number of layers – the zones can be tilted according to the local Bragg condition and each single layer requires only moderate aspect ratio structures (see Fig. 1C). However, the overlay accuracy for e-beam writing is in the nanometer range. Theoretical results on the dependency of the number of layers and their required overlay accuracy will be presented. We will also present the current status of the zone plate development at BESSY.

![Fig. 1: A) Zone plate with zones parallel to the optical axis, B) Zone plate with tilted zones, C) Zone plate where the local tilt angle of the zones is approximated by 5 layers.](image)

Hard X-ray Microscopy with sub-30 nm Spatial Resolution in Taiwan

Mau-Tsu Tang, Yen-Fang Song, Gung-Chian Yin, Jian-Hua Chen, Fu-Rong Chen, King-Long Tsang and Keng S. Liang

National Synchrotron Radiation Research Center
101 Hsin-Ann Road, Hsinchu Science Park, Hsinchu 30077, Taiwan

With the advances in synchrotron radiation and the mature in fabricating high performance X-ray focusing optics, X-ray microscopy has been realized recently as a nano-scale probe that can compete with light and electron microscopy on the common ground of a nondestructive manner. In 2004, under the NSRRC X-ray Microscopy Project, we have installed a full-field transmission X-ray microscope (TXM) to the BL01B end station of an advanced X-ray source generated by a superconducting wavelength shifter. The X-ray microscope equipped with capillary-type condensers and objective Fresnel zone-plates (outermost width 50nm) can provide 2D imaging and 3D tomography at X-ray energy 8-11 keV with spatial resolution 60 nm, and with the Zernike-phase contrast capability for imaging light materials such as biological specimens. While operating the Fresnel zone-plate in third order diffraction, the microscope has almost reached the theoretical resolution limit sub-30nm (Fig.1). In this presentation, we would demonstrate the actual setup of the beamline and microscope. Commissioning results, including the characterization of the microscope, preliminary studies in IC failure, fault rocks, and biological specimens will be presented.

Figure 1: The gold spoke-pattern imaged at BL01B at NSRRC by using 3rd order diffraction of Fresnel zone plate at X-ray energy 8 keV. The spatial resolution is estimated better than 30nm. Exposure time was 10 minutes.
PORTABLE SYNCHROTRON HARD X-RAY SOURCE
"MIRRORCLE-6X" FOR X-RAY IMAGINGS

Hironari Yamada, Daisuke Hasegawa\(^1\), Tohru Hirai, Yoshiko Okazaki, Makoto Sasaki, Taichi Hayashi\(^1\), and Takanori Yamada\(^1\)
Ritsumeikan University, Synchrotron Light Life Science Center,
1-1-1 Nojihigashi, Kusatsu-City, Shiga 525-8577, Japan
\(^1\)Photon Production Lab. Ltd., 4-2-1 (808) TakagaiChōMinami,
Omihachiman-City 523-0898, Japan

MIRRORCLE-6X is a portable synchrotron composed of a 6-MeV microtron injector, and a 60cm outer diameter exactly circular synchrotron ring made of a normal conducting magnet. The injection is performed at 400 Hz repetitions by 100 mA injector peaks current that lead to 3A initially accumulated current. X-rays are generated by a collision of the relativistic electron beam and a small target placed inside the circulating beam. The generated X-ray energy is dominated at around 30-300keV, and the total flux in the 0.1% bandwidth and ±85-mrad spreads is 1000 times higher than a conventional synchrotron light source. A few \(\mu\)m wide target, which defines the X-ray emitter size, produces extremely fine resolution edge enhanced images. Due to the small emitter size the brilliance of MIRRORCLE reaches \(10^{16}\) photons\([s, \text{mrad}^2, \text{mm}^2, 0.1\% \text{ band width}]\) at any point within ±85-mrad. The image field can be more than 30 cm wide at 2 m distances from the source point. This machine provides highest quality non-destructive testing of heavy constructions. Due to the phase contrast effect this machine also enables imaging of soft tissues. When the imaging device is set at the distance from the sample we can take magnified images. We have already obtained 10 times magnified fine resolution images, and are challenging to 100 times magnification that forms a novel X-ray microscope without optical elements. MIRRORCLE-6X opens up new frontiers of X-ray imaging in medical, biological, commercial and industrial uses.

The observed X-ray beam quality, brilliance, coherence, and challenge to the sub-micron size target will be discussed.
A fast-readout CCD system for configured-detector imaging in STXM

G R Morrison¹, A Gianoncelli¹, B Kaulich², D Bacescu², J Kovac³

1. Dept of Physics, King’s College London, Strand, London WC2R 2LS
2. ELETTRA - Sincrotrone Trieste, I-34012 Trieste-Basovizza, Italy
3. Jozef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia

The use of a transmitted x-ray detector in scanning transmission x-ray microscopy that has a software configurable response function makes possible the use of a number of simultaneous imaging modes, allowing both absorption and phase contrast to be derived from a single scan of the specimen [1].

This paper describes a practical system based around an electron-multiplying CCD system (iXon DV860 from Andor Technology) that combines fast frame-transfer readout with very high sensitivity. The detector consists of a Peltier-cooled CCD array of 128 by 128 sensors, and makes use of visible light coupling to a phosphor screen, to make it easy to operate over a wide range of photon energies, from the oxygen K edge upwards. It has been tested successfully on the Twinmic end-station at the Elettra synchrotron.

In essence, such a detector system records, for every pixel in the STXM raster, a map of the intensity distribution in and around the brightfield cone of illumination produced by the focusing optic in the STXM, resulting in a large volume of data from a single raster scan. Simple real-time processing of these data yields absorption and differential phase contrast image signals, but more elaborate processing can subsequently be applied to the full 3-D dataset generated by the STXM scan. In combination with through-focal series, and the acquisition of image data on either side of an x-ray absorption edge, a configured detector system can provide a wealth of information about the complex transmittance of the sample.

QUANTITATIVE X-RAY PHASE-CONTRAST MICROSCOPY AND MICROTOMOGRAPHY USING AN SEM

S.W. Wilkins¹, D. Gao¹, T.E. Gureyev¹, S.C. Mayo¹, P.R. Miller¹, Y. Nesterets¹, D. Paganin¹,², D. Parry¹, A. Pogany¹ & A.W. Stevenson¹

¹ CSIRO, Manufacturing & Infrastructure Technology, PB33 Clayton Sth, Clayton, Vic 3169, Australia.
² School of Physics & Materials Engineering, Monash University, Clayton, Vic 3168, Australia.

The projection method for X-ray microscopy is very simple in principle and enables X-ray imaging at sub-optical resolution without the need for any focusing X-ray optics. The use of an SEM to provide an electron beam focussed on a target as a source of X-rays for imaging was described as early as 1945¹. In this and subsequent works, the mechanism for contrast formation and interpretation was based on absorption.

Our approach to projection X-ray microscopy is to take advantage of the phase distortions imposed on an X-ray wavefront passing through an object, which, in combination with Fresnel diffraction leads to phase contrast in the resulting X-ray image.²³ We have developed an X-Ray ultra-microscope (XuM) based on an SEM as host (in the present case an FEI XL-30 S-FEG SEM) which is designed to take advantage of both phase and absorption contrast and has the capability for X-ray imaging at around 50 nm line pair resolution with X-rays from 0.8-15keV in energy. This system uses a scientific grade CCD in direct mode to give high signal to noise and optionally also an energy resolving capability for the transmitted beam. Images acquired using this system can be post-processed using combined phase-retrieval and deconvolution algorithms to produce very high resolution quantitative information about the sample⁴⁶.

This technique has particular strengths which are applicable to a wide range of microscopy- and microtomography-based studies of materials and devices. A number of examples of microtomography studies using the XuM will be presented including studies of manufactured devices, nanocomposites based on polymers, corrosion effects and biological samples.

References
Contribution of X-ray Microscopy to Bone Mineral Studies

EICHERT Diane, SALOME Murielle, COMBES Christèle*, BLEUET Pierre, BOHIC Sylvain, REY Christian*, SUSINI Jean

European Synchrotron Radiation Facility, ESRF, X-ray Micro spectroscopy Beamline ID21-22, BP220, 38043 GRENOBLE Cedex, FRANCE
*ENSIACET, Equipe Physico-chimie des Phosphates, 118 route de Narbonne, 31077 TOULOUSE Cedex, FRANCE

Bone, which is formed by the infusion of an organic matrix, principally collagen, with calcium phosphate, performs two major functions in the body. This combination of about 20wt% collagen and 80wt% calcium phosphate provides the biomechanical properties needed for body support and movement. In addition, bone mineral is in metabolic interrelation with body fluids, serving principally as a reservoir for body minerals, storing or releasing them as the need arise, changing in size, distortion and chemical perfection with age, disease and chemical/medical treatment [1-2]. Trace elements have been shown to have a profound influence on the chemistry and solubility of bone as this latter is the accumulation “target” organ for many heavy metals. Consequently, many human diseases and pathological conditions result in changes in bone tissue that affect the rate of bone turnover and its physico-chemical properties. Bone mineral presents then a complex composition, and appears as a very sensitive, reactive and sophisticated material.

Several techniques may be used for the study of bone mineral but often allow a global measurement only without spatial information. However the study of the mineral content of bone at a microscopic scale is of particular interest, since it can give new insights into remodelling activities, mineralization processes, effect of drugs and related mechanical properties.

We report here applications of X-ray microprobe techniques to the analysis of bone and biomaterials (analogous to bone mineral and implants). In particular, we will present the studies carried out on bone mineralization and maturation processes using X-ray micro-fluorescence, micro-XANES and micro-diffraction and micro-infrared [3-4]. Secondly, examples of applications to more specific topics will be shown, namely the study of bone mineral in osteogenesis imperfecta pathology [5], the effect of Sr based drugs against osteoporosis and the integration of titanium implants in bone [6]. Last, recent developments in x-ray micro-tomography allowing in-situ dynamic follow up of the formation and propagation of micro cracks in bone under strain will be presented [7].

[4]: Eichert et al., J. Bone Miner. Res. (2005), submitted
[5]: Eichert et al., in preparation
[6]: Eichert et al., in preparation
[7]: Bleuet et al., SPIE Developments in X-Ray Tomography IV(2004) 5535 : 129
A deep look into polycrystals: X-ray diffraction contrast tomography

W. Ludwig\textsuperscript{1,3}, E.M. Lauridsen\textsuperscript{2}, S. Schmidt\textsuperscript{2}, H.F. Poulsen\textsuperscript{2}, P. Cloetens\textsuperscript{3}

\textsuperscript{(1) GEMPPM - INSA de Lyon, France} \textsuperscript{(2) Risoe National Lab., Roskilde, Denmark} \textsuperscript{(3) ESRF, Grenoble, France}

We present here two possible extensions of X-ray microtomography, capable to characterize the shape and the orientation of individual grains in the bulk of undeformed polycrystalline materials: (i) diffraction contrast tomography in “topo-tomography” alignment \cite{1} and (ii) diffraction contrast tomography in conventional alignment. In both cases, Bragg diffraction (transmission case) gives rise to an additional contribution to the local attenuation coefficient, which in turn can be exploited by means of analytic or algebraic tomographic reconstruction techniques in order to reconstruct the three dimensional grain outlines. The second approach offers in addition the possibility to determine grain orientations.

The imaging principle and related data analysis strategies will be illustrated for both methods with the help of first experimental data, obtained from a coarse grained Al (1050) multicrystal. A comparison of diffraction contrast tomography with respect to the more widely applicable 3DXRD approach \cite{2} will be given. Based on this, we will discuss possible future developments and applications for combined tomographic imaging and diffraction experiments.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig1.png}
\caption{Diffraction contrast tomography (topo-tomo alignment) of an undeformed, coarse grained Al (1050) multicrystal. (a) tomographic slice showing the outline of the cylindrical sample as well as the shape of the diffracting grain with some substructure (inclusions). (b) 3D rendition of the diffracting grain, the Al matrix has been set to transparent.}
\end{figure}

\begin{thebibliography}{9}
\end{thebibliography}
Observation of Anisotropic Disorientation Grain Boundaries in \( \text{Sn}_2\text{O}_3 \) Nanobelts Using X-Ray Nanodiffraction*

Zhonghou Cai and Yanan Xiao

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

Molecules in a thermodynamic system have a spontaneous tendency to seek maximum interactions among themselves for lowering the free energy of the system via variation of density distribution. It is well known that the intensive parameters like temperature and pressure often limit the extent of the interactions and are responsible for the richness of phases that a system exhibits. However, the exploration of the extent of interactions due to the extensive parameters, such as the volume and the number of molecules in a system, and the effect on the density distribution is still in its infancy, due to the limited availability of such systems. A wire-like nanostructure synthesized through one-dimensional growth exhibits the effect due to its condensed phase, which enables molecules to significantly interact, while the small cross-section size limits the extent of the interactions. For instance, the energy needed to form a point defect at the growth front of a nanowire is much less than the energy needed to form the same defect in the bulk, since the lattice elastic deformation and associated strain field quickly die out when the boundary of the nanomaterial is reached. Once the energy can no longer counter the energy due to the change of the entropy associated with the formation of the point defect at the growth temperature, disorder starts to become the favorable configuration. This imposes a fundamental limitation on the smallest cross-section size of a perfect nanowire crystal that can be synthesized through one-dimensional growth. In order to observe the limitation, the internal structures of tin oxide (\( \text{Sn}_2\text{O}_3 \)) nanobelts of various cross-section sizes, from 200 nm x 40 nm down to 22 nm x 8 nm, were individually investigated using x-ray diffraction with a focused beam (7 keV) obtained from advanced zone-plate optics. By mapping the diffraction intensity of the (030) reflection with a CCD area detector along the full length of the nanobelts, we observed disorientation boundaries in the belts of cross-section sizes of 100 nm x 30 nm and smaller. The lattice disorientation of grains inversely relates to the cross-section size of the belts. Moreover, the lattice disorientation boundaries are confined only in the plane of growth front and none of that out of the plane has been observed. Given the fact that a tin oxide nanobelt is bounded by faces of high density and low surface energy, all the observations obtained from the tin oxide nanobelts can be explained consistently with the limited extent of interactions among molecules at the growth front.

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3D Internal Strain Mapping by Tracking Microstructural Features in Tomographic Volumes of Structural Materials

H. Toda\textsuperscript{1}, T. Ohgaki\textsuperscript{1}, M. Kobayashi\textsuperscript{1}, T. Akahori\textsuperscript{1}, M. Niinomi\textsuperscript{1}, T. Kobayashi\textsuperscript{1}, K. Uesugi\textsuperscript{2}, K. Makii\textsuperscript{3} and Y. Aruga\textsuperscript{3}

\textsuperscript{1} Department of Production Systems Engineering, Toyohashi University of Technology, 1-1, Hibarigaoka, Tempaku, Toyohashi, AICHI 441-8580, Japan
\textsuperscript{2} Japan Synchrotron Radiation Research Institute, 1-1-1, Kouto, Mikazuki-cho, Sayo-gun, HYOGO 679-5198 Japan
\textsuperscript{3} Kobe Steel, Ltd., 1-5-5, Takatsukadai, Nishi-ku, Kobe, HYOGO 651-2271, Japan

Synchrotron X-ray microtomography has been utilized for the 3D characterisation of microstructures in several aluminium alloys. Tomographs, consisting of isotropic voxels with a maximum of 0.474 µm edge, were collected mainly at the X-ray imaging beamlines BL20B2 and BL47XU of the SPring-8. A combination of the high-resolution deflection contrast imaging technique and several state-of-the-art application techniques have enabled the quantitative image analyses of internal microstructure, such as micro-pore, intermetallic compound particles and grain boundary as well as the assessment of their effects on deformation and fracture behaviours of the aluminium alloys. The application techniques include liquid metal wetting which enhances nanoscopic microstructural features in term of the absorption contrast, microstructural tracking which enables large scale strain mapping, in-situ observation technique using a material test rig specially designed by the present authors and local area observation for samples larger than available fields of view. 3D finite-element meshes were also generated from the tomographic volumes to monitor local stress and strain distributions, then being used to verify the image analyses.

In terms of the microstructural tracking, in order to evaluate microstructural effects quantitatively, the tomographic dataset was thresholded and labelled utilizing a grey value for each 3D feature of interest. Volume, surface area and centre of gravity of each feature were automatically measured at sub-voxel accuracy using software developed by the present authors. Centre of gravity of each microstructural feature was then utilized as a displacement gauge marker to calculate three-dimensional mechanical parameters of the underlying aluminium, such as strain, stress and crack driving force. Variations during loading in the centroid spacings of the pairs of neighbouring microstructural features may then be separated into three directions by orthogonal decomposition.

The tracking technique has provided a highly effective way of assessing microstructure/property relationships in the structural materials, together with supplementary ways of verifying and interpreting them by visualising and quantifying various mechanical behaviours. The proposed technique has been clearly advantageous compared to the very limited procedures for such measurements available in the current literature, where detailed internal information can only be accessible for limited types of material, such as transparent materials.
Ancient cosmetics and painting analysed by combination of complementary microanalysis techniques

Marine Cotte¹, Philippe Walter², Eléonore Welcomme², Pierre Bleuet¹, Armando V. Solé¹, Jean Susini¹

¹ European Synchrotron Radiation Facility, BP220, 38043 Grenoble Cedex, France
² Centre de Recherche et de Restauration des Musées de France, Palais du Louvre, 14 quai F. Mitterrand, 75001 Paris, France

Our research is focused on two different archaeological issues: the study of the cosmetics used in the Mediterranean geographic area along a period of time (going from the Egyptian antiquity to the Greco-Roman time). Cosmetics were of primary importance in the everyday life, not only in aesthetic context, but also in religious, ritual and medical practises.

Second, the study of the pigments used in Northern European paintings at the beginning of the Renaissance (Cranach, Dürer, Grünewald, Holbein, etc.). The physical-chemical signatures of the pigments, grounds and binding media are necessarily related with relevant practises and art expertise in Europe at that time.

Several similarities exist between these two subjects. First, the chemical nature is quite the same: products are made of pigments, with the possible addition of a binding medium (e.g. oil). Second, the analytical problematics are common: we search to determine the chemical composition of the products (are they natural or artificial compounds? Where they come from?) and the manufacturing process (chemical/mechanical transformation, heating…). More generally, our aim is to identify some characteristic “paint-pot”, ingredients and practices used in a limited time and space. Besides, in addition to supply information on ancient know-how, knowledge about degradation process can be useful for conservators.

Due to the samples complexity and preciousness, a network of various micro-analytical techniques was employed. Experiments were performed at the ESRF, on the beam lines ID21, ID22 and ID18F, which provide a panel of micro-imaging techniques, with high spatial resolution and high sensitivity. Micro X-ray fluorescence was used to identify the trace elements, which enable sometimes to distinguish between natural ores and synthesised products and to determine geographic provenances of minerals. Micro X-ray diffraction is a relevant method to analyse crystallised compounds and identify phases. Micro XANES analyses were performed at the sulphur and manganese K-edge to identify the chemical composition of some cosmetics and paintings. Infrared micro-spectroscopy gives access simultaneously to organic and mineral phases. It was particularly useful to study the interaction products between oil and pigments. More generally, each technique can generate images. This fact is very important since it enables elemental and chemical co-localisations, hence an easier identification of components. In addition, inter-technique correlations are also very fruitful. Finally, the combination of complementary and quantitative synchrotron based techniques is a powerful way to study complex ancient chemicals.