X-RAY Spectrometry

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# News

## Sciences

### Observation of non-linear resonances of inner-shell electrons by X-ray free electron laser (30 November 2011)

Recently, a very stimulating paper has been published discussing experimentally the fundamental processes of photo-absorption and excitation of electrons by using extremely high-fluence, ultra-short X-ray pulses. The research was performed for the electron system in inert Ne gas at LCLS (Linac Coherent Light Source), Stanford, USA, which is the world's first hard X-ray free-electron laser facility. The scheme is as follows: an intense single X-ray pulse of sub-10-fs duration at 848 eV first strips a 2p electron from Ne and, at this stage, because the X-ray energy is below the binding energy of a 1s electron in neutral neon, 870 eV, a 1s hole cannot be produced, but because of the above 2p hole, the next pulse can excite the 1s electron, leading to 1s-2p resonance in the $Ne^+$ ion and, finally, stimulated emission (2p-1s) competes with Auger decay to refill the 1s hole. The results have indicated that intense X-ray pulses of sub-10-fs duration can modify and even control the Auger decay process. For more information, see the paper, 'Unveiling and Driving Hidden Resonances with High-Fluence, High-Intensity X-Ray Pulses', E. P. Kanter *et al.*, Phys. Rev. Lett. 107, 233001 (2011).

### Soft X-ray absorption spectroscopy aids understanding on ferroelectric $BaTiO_3$ (28 November 2011)

$BaTiO_3$ is a promising candidate ferroelectric material for magnetoelectric composites and layered film structures. Recently, some interesting soft X-ray absorption spectra at Ti-$L_{II, III}$, O-K, and Ba-$M_{IV, V}$ edges have been discussed mainly from a theoretical point of view by a German group. For more information, see the paper, 'High-resolution x-ray absorption spectroscopy of $BaTiO_3$: Experiment and first-principles calculations', A. Chass é *et al.*, Phys. Rev. B84, 195135 (2011).

### Theory for multi-wavelength anomalous diffraction with X-ray free electron laser (14 November 2011)

Multi-wavelength anomalous diffraction (MAD) has been widely employed to determine phase information in X-ray crystallography. The method uses the contrast of the scattering power of heavy atoms at the absorption edges. However, when the X-ray source becomes extremely brilliant, the sample encounters severe electronic radiation damage, especially to heavy atoms, which makes the interpretation of MAD rather difficult. Recently, a theoretical paper discussing this problem has been published. The theory uses a Karle-Hendrickson-type equation in the high-intensity regime and demonstrates the calculation of relevant coefficients with detailed electronic damage dynamics of heavy atoms. For more information, see the paper, 'Multiwavelength Anomalous Diffraction at High X-Ray Intensity', S-K.Son *et al.*, Phys. Rev. Lett. 107, 218102 (2011).

### Monochromator optics for sub-meV resolution (11 November 2011)

Scientists at Argonne National Laboratory, USA have recently reported a novel set of optics for X-ray monochromators, which combine the effect of angular dispersion and anomalous transmission of X-rays in Bragg reflection from asymmetrically cut crystals. The optics employ a five-reflection, three-crystal arrangement, and it was found that the spectral contrast, the bandwidth and the angular acceptance are approximately 500, 0.5 meV, and 0.1 mrad, respectively, for 9.1 keV X-rays. The new optics could be a foundation for next-generation inelastic X-ray scattering spectrometers. For more information, see the paper, 'Using angular dispersion and anomalous transmission to shape ultramonochromatic x rays', Y. Shvyd'ko *et al.*, Phys. Rev. A84, 053823 (2011).

### Combination of ptychography and near-edge resonance (8 November 2011)

One of the key aspects of progress in X-ray microscopy is the advent of coherent diffractive imaging, which basically does not use any lenses. Ptychography is one improved version of a series of techniques using coherent X-ray beams and allows reconstructions of both strongly and weakly scattering samples. A further extension of this method has recently been published by a German group led by Professor T. Salditt (Georg-August-University Göttingen). The research introduced chemical contrasts based on near-edge X-ray absorption fine structures. The group demonstrated that two different molecules in a biological system are distinguished visually by using the contrasts near the oxygen K edge. For more information, see the paper, 'Chemical Contrast in Soft X-Ray Ptychography', M. Beckers *et al.*, Phys. Rev. Lett. 107, 208101 (2011).

### Mini electron-probe X-ray fluorescence instrument (21 October 2011)

Professor J. Kawai (Kyoto University, Japan; Associate editor of *X-ray Spectrometry*) and his colleagues recently developed a novel tiny X-ray instrument equipped with a pyroelectric $LiTaO_3$ crystal as an electron source, a sample stage and an X-ray detector. The research group found that adequate X-ray fluorescence spectra can be measured for 0.03 $mm^2$ titanium, iron, and nickel wires. For more information, see the paper, 'Development of Miniaturized Electron Probe X-ray Microanalyzer', S. Imashuku *et al.*, Anal. Chem., 83, 8363 (2011).

### Influence of pH treatment on Kβ/Kα intensity ratio in ZnCo alloys (18 October 2011)

Empirical and semiempirical K-shell fluorescence yields (ωK) and Kβ/Kα intensity ratios for ZnCo alloy with various chemical compositions, leading to differences in pH, have recently been published. The samples were excited by 59.5-keV γ rays from a

$^{241}$Am annular radioactive source, and X-ray fluorescence spectra were measured by an Ultra-LEGe detector. For more information, see the paper, 'Effect of pH treatment on K-shell x-ray intensity ratios and K-shell x-ray-production cross sections in ZnCo alloys', N. Kup Aylikci *et al*., Phys. Rev. A84, 042509 (2011).

### Quantitative synchrotron X-ray fluorescence analysis of buried nanolayer (3 October 2011)

A German group led by Dr. B. Beckhoff [Physikalisch-Technische Bundesanstalt (PTB), Berlin] recently analyzed quantitatively the buried $B_4C$ nanolayer on a silicon substrate by using synchrotron radiation at BESSY II. The thickness and elemental composition were successfully determined by reference-free X-ray fluorescence spectrometry under conventional and grazing-incidence conditions. For more information, see the paper, 'Complementary Characterization of Buried Nanolayers by Quantitative X-ray Fluorescence Spectrometry under Conventional and Grazing Incidence Conditions', R. Unterumsberger *et al*., Anal. Chem., 83, 8623 (2011).

### Coherent X-ray diffraction to look at stress in nano particles (25 September 2011)

A research team led by Professor I. Robinson (London Centre for Nanotechnology, University College London) recently analyzed how gold nanocrystal changes after the adsorption of organic molecules because of the strain field. So far, it has been difficult to observe such influence of adsorbed molecules on the particle structure. The team employed the coherent X-ray diffraction method, which is extremely sensitive to displacement of atoms, and therefore to adsorption-induced near-surface stress in a single nanocrystal. It was discovered that the stress generated by thiol adsorption on gold has a fundamentally different nature in the curved, nominally spherical, regions of the crystal surface than in its flat facets. The magnitude of surface stress was also quantitatively analyzed and discussed. The experiments were performed with coherent X-rays of 8.92 keV from the 34-ID-C beamline of the Advanced Photon Source (APS), Argonne, USA. For more information, see the paper, 'Differential stress induced by thiol adsorption on facetted nanocrystals', M. Watari *et al*., Nature Materials 10, 862 (2011).

## Professional

### Synchrotron X-ray analysis of an unfinished self-portrait by Rembrandt van Rijn (2 December 2011)

On Friday 2 December 2011, an unknown painting by Rembrandt was presented in the Rembrandt House Museum (Amsterdam). The small panel, Old Man with a Beard, was painted by Rembrandt around 1630, at the end of his time in Leiden. A research group led by Professor K. Janssens (University of Antwerp) and Professor J. Dik (Delft University of Technology) has performed experimental studies on this painting with a synchrotron beam at the European Synchrotron Radiation Facility (ESRF) and the Brookhaven National Laboratory (BNL), and has unveiled a hidden, unfinished self-portrait below the painting. For further information, visit the web page, http://webh01.ua.ac.be/mitac4/rembrandt/index_301111.html

### The 6th Asada Award (28 October 2011)

The recipient of the 6th Asada Award, which is presented by the Discussion Group of X-ray Analysis, Japan, in memory of the late Professor Ei-ichi Asada (1924–2005) to promising young scientists in X-ray analysis fields in Japan, is Dr. Takashi Yamamoto (Tokushima Univ., 'Studies on pre-edge peak in XANES spectra of transition metals for empirical chemical state analysis'). The ceremony was held during the 47th Annual Conference on X-Ray Chemical Analysis, Japan, at Kyushu University, Fukuoka.

### MOU between European XFEL and Spanish Laser Institute (10 October 2011)

European XFEL and the Spanish Center for Ultrashort Ultraintense Pulsed Lasers (CLPU) in Salamanca will pool their efforts to promote research into high-energy density science and to develop new ultrafast lasers to analyze physical and chemical processes in conjunction with the X-ray beams of the European XFEL. Both research institutions signed a memorandum of understanding at the European XFEL headquarters in Hamburg. In the framework of this cooperation, an optical laser expert from CLPU has now joined the European XFEL Optical Lasers Group for an initial period of 6 months. For further information, visit the web page, http://www.xfel.eu/

## New products

### New handheld XRF spectrometer from Oxford Instruments (14 November 2011)

Oxford Instruments has released a new faster, lighter and smaller handheld XRF analyzer – the X MET7000, which is designed for industrial markets, including positive material identification, metals and alloys, scrap sorting and precious metals analysis. For further information, visit the web page, http://www.oxinst.com/

## Corporate

### Collaboration between Bruker and Symphony Environmental on the XRF analysis of plastic (23 November 2011)

After a 1 year cooperative development, Bruker Corporation and Symphony Environmental Ltd have signed a contract for the exclusive supply of Bruker's X-Ray Fluorescence (XRF) technology to Symphony for the identification of pro-degradant, anti-microbial and anti-fungal additives in plastic. For further information, visit the web page, http://www.bruker-axs.com/

### JEOL ASEAN Technical Center in Thailand (2 November 2011)

JEOL Ltd. has announced a plan to open the JEOL ASEAN Technical Center, in Thailand. The center will launch in January 2012. For further information, visit the web page, http://www.jeol.com/

### Rigaku acquires handheld Raman products from BaySpec (13 October 2011)

Rigaku Americas Corporation has announced the acquisition of the handheld Raman technology and product lines from BaySpec, Inc. and the concurrent formation of a new

division, Rigaku Raman Technologies Inc., for R&D, engineering, production, marketing and distribution. For further information, visit the web page, http://www.rigaku.com/

**Horiba's new facility in Paris (16 September 2011)**

Horiba has launched a new facility for R&D in the Paris Saclay cluster. For further information, visit the web page, http://www.horiba.com/

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# News

## Sciences

### Phase transition of carbon induced by X-ray free-electron laser (11 July 2012)

Extremely strong pulses from X-ray free-electron laser (XFEL) can change the material structure. Recently, scientists at Linac Coherent Light Source, Stanford, USA, have reported the amorphous to crystalline phase transition of carbon by femtosecond 830 eV XFEL beam. The research group employed atomic force microscopy, photoelectron microscopy, and micro-Raman spectroscopy to discuss the change of the $sp^2/sp^3$ ratio (graphitization), as well as the change of local order of the irradiated sample area. It was found that the phase transition threshold fluence is $282 \pm 11$ mJ/cm$^2$, and also, the transition is mainly due to thermal activation rather than a nonthermal mechanism such as ionization and so on. For more information, see the paper 'Amorphous to crystalline phase transition in carbon induced by intense femtosecond X-ray free-electron laser pulses', J. Gaudin *et al.*, Phys. Rev. B86, 024103 (2012). DOI:10.1103/PhysRevB.86.024103

### Extension of resonant X-ray scattering by using K–K relationship in soft X-ray reflectivity (10 July 2012)

Resonant X-ray scattering is a powerful technique for the study of electronic structure at the nanoscale. However, the optical properties of the constituent components of a material must be known prior to modeling of the scattered intensity. Professor J. B. Kortright (Lawrence Berkeley National Laboratory, USA) and his collaborator have recently proposed a method of refining electronic structure, in the form of optical properties, simultaneously with physical structure, in a Kramers–Kronig (K–K) consistent manner. This technique has been applied to specular reflectivity from a $SrTiO_3$ single crystal, and both a nonresonant surface contaminant layer and a modified $SrTiO_3$ surface region have been evidenced. For more information, see the paper 'Kramers–Kronig constrained modeling of soft X-ray reflectivity spectra: Obtaining depth resolution of electronic and chemical structure', K. H Stone *et al.*, Phys. Rev. B86, 024102 (2012). DOI:10.1103/PhysRevB.86.024102

### Detection of characteristic X-ray photons from a single atom (8 July 2012)

Several electron-microscopist groups have recently reported that a Si drift detector with a 60–100 mm$^2$ effective area can be used to detect characteristic X-rays from a single atom in nanomaterials such as silicon and platinum in monolayer and multilayer grapheme, as well as erbium in a $C_{82}$ fullerene cage supported in a single-walled carbon nanotube. They employed a tiny electron beam of 0.1 nm in the aberration-corrected scanning transmission electron microscope. As will be clear for readers of *X-ray Spectroscopy* journal, the discussion is a kind of major and/or minor component analysis of extremely small volume rather than so-called ultra trace element analysis. The signal intensity was apparently very weak but was in the order of some counts/s according to the reports. Such high sensitivity points to the significant potential of the energy dispersive detector system. On the other hand, further detailed analysis including the estimation of parasitic background will be necessary. For more information, see the papers 'Single atom identification by energy dispersive X-ray spectroscopy', T. C. Lovejoy *et al.*, Appl. Phys. Lett., 100, 154101 (2012) DOI:10.1063/1.3701598 , and 'Detection of photons emitted from single erbium atoms in energy-dispersive X-ray spectroscopy', K. Suenaga *et al.*, Nature Photonics, advanced online publication DOI:10.1038/nphoton.2012.148

### Femtosecond imaging with X-ray free-electron laser (29 June 2012)

One very interesting outcome at Linac Coherent Light Source, Stanford, USA has recently been published. The experiment was single-shot imaging of ferromagnetic, nanoscale spin order taken with femtosecond XFEL pulses. For more information, see the paper 'Femtosecond single-shot imaging of nanoscale ferromagnetic order in Co/Pd multilayers using resonant X-Ray holography', T. Wang *et al.*, Phys. Rev. Lett. 108, 267403 (2012). DOI:10.1103/PhysRevLett.108.267403

### Table-top soft X-ray laser (8 June 2012)

High-harmonic generation (HHG) is a universal response of atoms and molecules in strong femtosecond laser fields and can be used to generate coherent photons in the soft X-ray region. Simply speaking, HHG is the coherent version of an X-ray tube; instead of accelerating thermal electrons emitted from the filament and generating incoherent X-rays by hitting a metallic target, HHG begins with tunnel ionization of an atom in a strong laser field. The portion of the electron wave function that escapes the atom is accelerated by the laser electric field and, when driven back to its parent ion by the laser, can coherently convert its kinetic energy into a high-harmonic photon. So far, for many cases, around 100 near-infrared laser photons have been combined to generate bright, phase-matched, extreme ultraviolet beams when the emission from many atoms is added constructively. Recently, a team led by Professor H. C. Kapteyn and Professor M. M. Murnane (University of Colorado at Boulder, USA) has employed a mid-infrared femtosecond laser in a high-pressure gas and succeeded in getting ultrahigh harmonics up to orders greater than 5000, resulting in a bright continuum spectra ranging from 0.2 to around 1.6 keV. The energy has still not yet reached the hard X-ray regime, but this would be a very attractive coherent ultra short pulse source for soft X-rays. For more information, see the paper 'Bright coherent ultrahigh harmonics in the keV X-ray regime from mid-infrared femtosecond lasers', T. Popmintchev *et al.*, Science, 336, 1287 (2012). DOI:10.1126/science.1218497

### Quantitative grazing incidence small-angle X-ray scattering analysis (7 June 2012)

Dr D. Babonneau (PhyMat, CNRS UMR 6630, Université de Poitiers, France) and his colleagues have recently analyzed morphological characteristics of nanoripple patterns prepared by broad beam-ion sputtering of $Al_2O_3$ and $Si_3N_4$ amorphous thin films as well as two-dimensional arrays of Ag nanoparticles obtained by glancing angle deposition on $Al_2O_3$ nanorippled buffer layers. They employed three-dimensional (3D) reciprocal space mapping in the grazing incidence small-angle X-ray scattering geometry. For more information, see the paper 'Quantitative analysis of nanoripple and nanoparticle patterns by grazing incidence small-angle X-ray scattering 3D mapping', D. Babonneau *et al*., Phys. Rev. B85, 235415 (2012). DOI:10.1103/PhysRevB.85.235415

### Continuous flow sample cell for X-ray absorption spectroscopy (22 May 2012)

A Swiss group has reported on the design and performance of a novel high-temperature and high-pressure continuous-flow reactor, which allows for X-ray absorption spectroscopy or diffraction in supercritical water and other fluids under high pressure (up to 30 MPa) and temperature (up to 500 °C). For more information, see the paper 'Design of a continuous-flow reactor for in situ X-ray absorption spectroscopy of solids in supercritical fluids', M. Dreher *et al*., Rev. Sci. Instrum. 83, 054101 (2012). DOI:10.1063/1.4719921

### Real time observation of copper corrosion by synchrotron X-ray diffraction (9 May 2012)

A research team led by Professor A. Adriaens (Ghent University, Belgium) has developed a number of useful techniques based on synchrotron X-ray diffraction to see the growth of synthetic corrosion layers in real time. The observation was performed for copper, and the final products were identified as mixtures of nantokite (CuCl), cuprite ($Cu_2O$), and paratacamite ($Cu_2(OH)_3Cl$). The team employed a highly sophisticated instrument for growing corrosion using a spin coater, and it could be used for many other similar applications. Experiments were performed at both SRS, Daresbury, and ESRF, Grenoble. For more information, see the paper 'The use of synchrotron X-rays to observe copper corrosion in real time ', M. Dowsett *et al*., Anal. Chem. 84, 4866 (2012). DOI:10.1021/ac300457e

### Synchrotron XRF mapping of large paintings (7 March 2012)

An Australian team has reported on its study of a historical self-portrait by Sir Arthur Streeton (1867–1943) with fast-scanning X-ray fluorescence (XRF) microscopy using synchrotron radiation. They employed the event-mode Maia X-ray detector, which has the capability to record elemental maps at megapixels per hour with the full XRF spectrum collected per pixel. The 25 megapixel elemental maps were obtained across the $200 \times 300\,mm^2$ scan area. The size of the beam used was $10 \times 10\,\mu m^2$. As heavy brushstrokes of lead white overpaint conceal the portrait, the excitation energy was chosen as 12.6 keV to avoid the influence of extremely strong Pb L fluorescence as well as Raman inelastic scattering. For more information, see the paper 'High-definition X-ray fluorescence elemental mapping of paintings', D. L. Howard *et al*., Anal. Chem. 84, 3278 (2012). DOI:10.1021/ac203462h

### How to reconstruct 3D chemical map from confocal micro-XAFS (18 January 2012)

A research group led by Professor B. Kanngießer (Technische Universität Berlin, Germany) has reported on a new approach for chemical speciation in stratified systems using 3D micro-X-ray absorption fine structure (XAFS) spectroscopy. As XRF mode in XAFS measurement generally leads to distorted spectra because of absorption effects, they developed a reliable reconstruction algorithm. For more information, see the paper 'Reconstruction procedure for 3D micro X-ray absorption fine structure', L. Luhl *et al*., Anal. Chem. 84, 1907 (2012). DOI:10.1021/ac202285d

## Professional

### Denver X-ray conference awards (8 August 2012)

During the plenary session of the 61st Annual Denver X-Ray Conference, 2012 Birks Award was awarded posthumously to John Criss, and 2012 Jerome B. Cohen Student Award was given to Magnus Menzel, Institut für Anorganische und Angewandte Chemie, Universität Hamburg, Hamburg, Germany, for his work, 'Confocal μ-XRF XANES Analysis of the Cathode Electrolyte Interface of Lithium-ion Batteries'. For further information, visit the web page, http://www.dxcicdd.com/

### 15th international conference on X-ray absorption fine structure (28 July 2012)

The 15th international conference on X-ray absorption fine structure was recently held in Beijing, China, from 22 to 28 July 2012. In addition to many applications of the XAFS technique in a variety of scientific fields, reports and discussions were held on progress in theory and software, as well as some advanced experiments such as time-resolved XAFS. The next conference will take place at Karlsruhe, Germany in summer 2015. For further information, visit the web page http://www.ixasportal.net/ixas/index.php?option=com_content&view=article&id=90&Itemid=134

### 2012 workshop on buried interface science with X-rays and neutrons (28 June 2012)

The 2012 workshop on buried interface science with X-rays and neutrons was held at KEK, Tsukuba, Japan, on 26–28 June 2012. This was the latest in a series of 18 workshops held since 2001. There are increasing demands for sophisticated metrology to observe multilayered materials with nanostructures (dots, wires, etc), which are finding applications in electronic, magnetic, optical, and other devices. X-ray and neutron analysis is known for its ability to observe in a nondestructive manner even 'buried' function interfaces as well as the surface. In addition to such inherent advantages, recent remarkable advances in micro analysis and quick time-resolved analysis in X-ray reflectometry are extremely important. The latest progress in novel quantum beam technologies, such as XFELs, ERLs, as well as many other table-top laser-like machines could push such techniques towards further sophisticated applications. The present workshop gathered together those with different research backgrounds, i.e. from semiconductor electronics to chemical biomaterials, and even theoretical groups were invited to give insights into unsolved problems on buried interfaces.

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## New products

**Bruker's micro-CT accessory for scanning electron microscopes (30 July 2012)**

Bruker has introduced its new micro-computed tomography (CT) accessory for scanning electron microscopes (SEM). Micro-CT for SEM can add 2D and 3D high-resolution X-ray imaging capabilities to third-party SEMs, allowing the nondestructive analysis of internal microstructures of specimens. For further information, visit the web page http://www.bruker-axs.com/

**5th generation of Rigaku's MiniFlex (15 January 2012)**

Rigaku Corporation has announced additions to its MiniFlex series of benchtop X-ray diffraction analyzers. For further information, visit the web page http://www.rigaku.com/

## Corporate

**Varian launches on-line X-ray product finder (25 June 2012)**

Varian Medical Systems has launched an on-line product finder tool giving customers the ability to quickly locate needed replacement X-ray tubes as well as flat panel detectors for digital imaging. The product finder showcases all of Varian's X-ray product offerings and can be found on the company's website at http://www.varian.com/us/xray/products/

**e2v sells three businesses to Baird (16 May 2012)**

e2v technologies plc has announced the sale of e2v Scientific Instruments, e2v microsensors, and its industrial gas sensing businesses, to SGX Sensortech Limited, a company backed by Baird Capital Partners Europe. Those companies provide a range of professional sensing products for X-ray spectroscopy and gas sensing for automotive and environmental safety applications. For further information, visit the web page http://www.sgxsensortech.com/

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