X-RAY SPECTROMETRY

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News Article

Sciences

First all optical synchronization in X-ray free electron laser (January 20, 2015)

At the soft X-ray free-electron laser (XFEL) facility, FLASH, in Hamburg, Germany, all-optical synchronization has finally been achieved. Scientists are reporting that the timing is better than 30 fs rms for 90 fs X-ray photon pulses. As one of the most promising experiments using XFEL is time-resolved analysis based on the pump & probe scheme, it is crucial to synchronize all independent components, including all accelerator modules and all external optical lasers, to better than the delivered free-electron laser pulse duration such as shorter than 100 fs. For more information, see the paper, "Femtosecond all-optical synchronization of an X-ray free-electron laser", S. Schulz *et al.*, Nature Communications, 6, 5895 (2015).

Synchrotron light unveils hidden letters inside a carbonized ancient papyrus scroll discovered in Herculaneum (January 20, 2015)

At the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, scientists from Italy, Germany and France have succeeded in 'reading' letters inside a papyrus roll found in the ancient library, discovered in Herculaneum. The experiments were done at the beamline ID17, and the X-ray phase contrast tomography technique was employed. The team was successful in extracting words under several papyrus layers in a fragment, and finally found that they are the complete Greek alphabet. For more information, see the paper, "Revealing letters in rolled Herculaneum papyri by X-ray phase-contrast imaging", V. Mocella *et al.*, Nature Communications, 6, 5895 (2015). An interesting movie has also been uploaded to Youtube, https://www.youtube.com/watch?v=d3aWBgNYOCU

Recent progress in X-ray ptychography (December 22, 2014)

X-ray ptychography is known as a promising lensless imaging method. Compared with other similar techniques, it can give a rather wide viewing area with the same high-spatial-resolution in nano scale, by combining multiple coherent diffraction measurements from the illumination of several overlapping regions on the sample. However, this apparently has to assume a highly sophisticated scanning/positioning instrumentation. The method may suffer also from partial-coherence effects and fluctuations. Dr. A. Menzel (Paul Scherrer Institut, Switzerland) and his colleagues have recently published an interesting report on fast measurement. The authors discussed ptychographic on-the-fly scans, i.e., collecting diffraction patterns while the sample is scanned with constant velocity. It was found that such a scan can be used as a model for a state mixture of the probing radiation and helps to achieve reliable image recovery. The feasibility of on-the-fly measurements in traditional scanning transmission X-ray microscopy is already known. This time, the research team was successful in applying these to X-ray ptychography, which usually uses reconstruction algorithms assuming diffraction data from a static sample. Such problems were discussed in detail. For more information, see the paper, "On-the-fly scans for X-ray ptychography", P. M. Pelz *et al.*, Appl. Phys. Lett., **105**, 251101 (2014).

Novel approach to see topographic shape of buried interfaces – grazing resonant soft X-ray scattering (December 15, 2014)

A team led by Professor Harald Ade (North Carolina State University, USA) has reported that grazing resonant soft X-ray scattering (GRSoXS), a technique measuring diffusely scattered soft X-rays from grazing incidence, can reveal the statistical topography of buried thin-film interfaces. So far, in wide variety of material systems, the internal structures of layered systems, particularly interfaces between different materials, have been critical to their functions. However, the analysis of buried interfaces has always presented some difficulties. It is known that X-ray electric field intensity distribution along the depth can be controlled by a change of either the incidence angle or the X-ray energy. The research team was able to manipulate it by scanning the X-ray energy, and succeeded in identifying the microstructure at different interfaces of a model polymer bilayer system such as PMMA/PEG. The authors attempted to gauge the feasibility of the technique for further practical systems like an organic thin-film transistor, PS[100 nm]/PBTTT [50 nm]/Si. For more information, see the paper, "Topographic measurement of buried thin-film interfaces using a grazing resonant soft x-ray scattering technique", E. Gann *et al.*, Phys. Rev. **B90**, 245421 (2014).

4 m length inverse-Compton scattering X-ray source (December 1, 2014)

In addition to large-scale X-ray facilities such as synchrotrons and X-ray FELs, there have been increasing demands for much more compact X-ray sources with high brilliance, ultra short pulse properties and coherence. Dr. W. S. Graves (Massachusetts Institute of Technology, USA) and his colleagues have proposed a design for a compact X-ray source based on inverse Compton scattering. The source consists of a 1 m linuc and an ultra short pulse laser. The whole size of the source including X-ray experiment space is nearly 4 m. The colliding laser is a Yb:YAG solid-state amplifier producing 1030 nm, 100 mJ pulses at 1 kHz repetition rate. The calculation shows that X-ray intensity at 12.4 keV is 5×10^{11} photons/second in a 5% bandwidth. For more information, see the paper, "Compact x-ray source based on burst-mode inverse Compton scattering at 100 kHz", W. S. Graves *et al.*, Phys. Rev. STAB, **17**, 120701 (2014).

Generation of copper Kα and Kβ pulses by the use of middle infrared laser system (November 10, 2014)

A German and Austrian group has recently developed a table-top X-ray source based on ultra-short laser pulses. Generation of X-ray pulses by lasers may not be a big surprise for readers (See, for example, "Ultrafast X-ray Pulses from Laser-Produced Plasmas" by M. M. Murnane, Science, 251, 531 (1991), "Microfocus Cu Kα source for femtosecond x-ray science" by N. Zhavoronkov, Opt. Letter, 30, 1737 (2005)). However, so far, the X-ray intensity has not been sufficient for use in practical measurements such as pump-and-probe time resolved X-ray analysis. This time, scientists employed a mid infrared wavelength (3.9 micron) to accelerate electrons from the copper tape target to very high kinetic energy by making use of its comparably long optical period. The pulse width of the laser employed is 80 femto second. It was found that the system gives 10^9 copper Kα photons per pulse generated with pulses of a peak intensity of 6×10^{16} W/cm^2. This is about 25 times higher than that generated by 800 nm wavelength laser pulses. For more information, see the paper, "High-brightness table-top hard X-ray source driven by sub-100-femtosecond mid-infrared pulses", J. Weisshaupt *et al.*, Nature Photonics, **8**, 927 (2014).

Origin of broad N Kα emission spectra (November 10, 2014)

A team led by Dr. T. Jach (NIST, USA) and Dr. W. T. Elam (University of Washington, USA) has recently published an interesting theoretical paper discussing the broadening of N K absorption and emission spectra of NH_4NO_3 and NH_4Cl. The authors studied many-body lifetime effects in valence-band X-ray emission. For more information, see the paper, "Origins of extreme broadening mechanisms in near-edge x-ray spectra of nitrogen compounds", J. Vinson *et al.*, Phys. Rev. **B90**, 205207 (2014).

Chemical imaging by X-ray-excited optical luminescence (November 6, 2014)

Professor A. Adriaens (Ghent University, Belgium) and her colleagues have recently reported on an X-ray-excited optical luminescence microscope using synchrotron light and its applications. The experiments were done at beamlines BM28 and BM26A at the ESRF in Grenoble, France. A broad X-ray beam is used to illuminate large areas of ~4 mm^2 of the sample, and the resulting optical emission is observed by a specifically designed optical microscope equipped with a CCD camera. By scanning the X-ray energy near the absorption edge, the image can obtain the sensitivity of chemical states. The authors studied copper surfaces with well-defined patterns of different corrosion products (cuprite Cu_2O and nantokite CuCl). For more information, see the paper, "Evaluation of an X-ray Excited Optical Microscope for Chemical Imaging of Metal and Other Surfaces", P-J. Sabbe *et al.*, Anal. Chem., 86, 11789 (2014).

Quantitative confocal micro X-ray fluorescence with polychromatic excitation (October 11, 2014)

So far, monochromatic X-rays have been used for 3D micro X-ray fluorescence analysis based on

confocal geometry. Dr. P. Wrobel (AGH University of Science and Technology, Poland) and his colleagues have recently discussed the feasibility of polychromatic excitation. The research group described the full theoretical expression of matrix effects and geometrical effects for polychromatic X-ray photons in confocal arrangement. It was demonstrated that the introduction of effective energy approximation works well. For more information, see the paper, "Depth Profiling of Element Concentrations in Stratified Materials by Confocal Microbeam X-ray Fluorescence Spectrometry with Polychromatic Excitation", P. Wrobel *et al.*, Anal. Chem., 86, 11275 (2014).

Pin-hole X-ray camera (October 4, 2014)

Dr. F. P. Romano and his colleagues have reported full-field X-ray fluorescence imaging based on the principle of the pin-hole camera. The instrument consists of a low power X-ray source (W anode, 50 kV-2 mA), a pinhole (50 micron dia) and a CCD camera (back illuminated type, 1024 × 1024 pixels, pixel size 13 micron square). To obtain good energy resolution (133 eV at 5.9 keV), the research team took a number of images in single photon counting mode. The team also obtained a reasonable spatial resolution down to 30 microns. The system can change the magnification ratio from 0.35 to 6, depending on the viewing area of interest and the required spatial resolution. For more information, see the paper, "Macro and Micro Full Field X-Ray Fluorescence with an X-Ray Pinhole Camera Presenting High Energy and High Spatial Resolution", F. P. Romano *et al.*, Anal. Chem., 86, 10892 (2014).

Professional

Review paper on liquid metal surface studies by using X-rays (December 8, 2014)

Professor P. S. Pershan (Harvard University, USA) has recently published an interesting review paper on X-ray studies of the interface between liquid metals and their coexisting vapor. For more information, see the paper, "Review of the highlights of X-ray studies of liquid metal surfaces", P. S. Pershan, J. Appl. Phys., **116**, 222201 (2014).

The 9th Asada award (October 30, 2014)

The recipient of the 9th 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. Susumu Imashuku (Kyoto Univ.), "Portable electron probe microanalyzer using pyroelectric crystal". The ceremony was held during the 50th Annual Conference on X-Ray Chemical Analysis, at Tohoku University, Sendai.

Multimedia

Popular YouTube video of SLAC's public lecture on X-ray studies on battery materials (May 24, 2014)

Lecture Date: Tuesday May 27, 2014. Dr. Johanna Nelson Weker, SLAC, delivered the SLAC public lecture, "X-rays Reveal Secret Life of Batteries" (https://www.youtube.com/watch?v=V8lSTLRkKEk)

Popular YouTube video of SLAC's public lecture on the application of synchrotron X-ray fluorescence to paintings (October 1, 2013)

Lecture Date: Tuesday October 1, 2013. Jennifer Mass of the Winterthur Museum in Wilmington, Delaware, delivered the SLAC public lecture, "Don't Fade Away: Saving the Vivid Yellows of Matisse and Van Gogh." (https://www.youtube.com/watch?v=RiASAbniQYw)

New products

EDAX's new silicon drift detector (December 11, 2014)

EDAX, Inc., has introduced the Element Silicon Drift Detector (SDD). The chip size is 25 mm^2, and it is designed with a silicon nitride (Si_3N_4) window to optimize low energy X-ray transmission for light element analysis. For further information, visit the web page, http://www.edax.com/

Rigaku launches SmartSite RS, a portable stress analyzer (October 1, 2014)

Rigaku Corporation has announced the release of the Rigaku SmartSite RS portable stress analyzer, which is especially designed for on-site analysis. For further information, visit the web page, http://www.rigaku.com/

Bruker's SDD based handheld XRF analyzer (September 15, 2014)

Bruker Elemental has introduced the new S1 TITAN model 500 handheld XRF analyzer equipped with a large silicon drift detector. For further information, visit the web page, http://www.bruker.com/hhxrf

Corporate

AMETEK acquires Amptek (August 4, 2014)

AMETEK, Inc. (NYSE: AME) has acquired Amptek, Inc. Amptek joins AMETEK as a unit of its Materials Analysis Division within AMETEK's Electronic Instruments Group. For further information, visit the web page, http://www.ametek.com/

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Sciences

Picosecond time-resolved X-ray absorption near edge spectrometer (9 July 2015)

Dr. F. Dorchies (Universite Bordeaux, CNRS-CELIA, France) and his colleagues have recently developed a laser-base X-ray absorption spectrometer covering 0.5–4.0 keV with a time resolution of around 3.3 ps. The spectrometer uses bremsstrahlung caused by the extremely high impact of laser pulses on the metallic target. To perform time-resolved X-ray spectroscopic studies, there have been quite a few challenges. For most research, it is crucial to avoid damaging/destroying samples, and the measuring time should not be very long. In addition, scientists do not like to compromise the signal-to-background ratio of spectral data taken at each time point, even though the quality is not the same as that of ordinary X-ray absorption spectra. The authors seem to believe that they are getting some breakthroughs. Their setup is a combination of a tabletop laser (Ti: Sapphire, 800 nm, 150 mJ, 10 Hz) and a Johan spectrometer equipped with a CCD camera. A set of polycapillary optics were employed as a beamline transport between the X-ray source and the sample (1-m distance) to maintain a clean, independent, and flexible environment for the sample. The X-ray intensity near the Al K edge and Cu L edges is 1.3×10^6 photons/eV/pulse. For more information, see the paper 'Experimental Station for Laser-based Picosecond Time-resolved X-ray Absorption Near-edge Spectroscopy', F. Dorchies *et al*., Rev. Sci. Instrum. **86**, 073106 (2015).

Standardless analysis of heavy elements by electron probe (6 July 2015)

Since the development of electron probe micro analysis by Castaing's PhD thesis in 1951, great efforts have been made to improve the technique. It was believed that the use of standard samples is absolutely indispensable to the determination of the concentration of each element. This can be a limit for some fields, such as nuclear materials application, where the quantification of minor actinides in fresh or spent fuel is demanded with no availability of any standard samples. In France, Dr. A Moy (Universite de Montpellier) and his colleague have recently reported successful standardless analysis of Pb and U in PbS, PbTe, $PbCl_2$, $Pb_5(VO_4)_3Cl$ (vanadinite), and UO_2, by measuring absolute Mα and Mβ X-ray intensity by a wavelength dispersive spectrometer. Experimentally obtained X-ray intensity was converted into absolute X-ray yields by evaluating the detector efficiency and then compared with calculated background X-ray intensity based on Monte Carlo simulations. For more information, see the paper 'Standardless Quantification of Heavy Elements by Electron Probe Microanalysis', A. Moy *et al*., Anal. Chem. **114**, 255501 (2015).

Molecular movie in femtosecond time scale (22 June 2015)

A team led by Dr. M. Minitti (SLAC National Accelerator Laboratory, USA) has recently succeeded in recording the time evolution of a structural change of ring-type 1,3-cyclohexadiene gas molecule to linear 1,3,5-hexatriene. The employment of the X-ray free-electron laser at Linac Coherent Light Source, Stanford, USA allowed them to do ultra fast snapshots of X-ray scattering in several tens of femtosecond (fs) scale. The study is based on pump-and-probe measurement, i.e. X-ray data were collected as a function of the controlled delay time between the UV pump pulse (267 nm, 65 fs, 4–8 μJ, 100-μm size) and X-ray probe pulse (8.3 keV, around 30 fs, 10^{12} photons/pulse, 30-μm square size). The team established that some signals caused by structural change are found as early as 30 fs, and the reaction finishes at 200 fs. For more information, see the paper 'Imaging Molecular Motion: Femtosecond X-Ray Scattering of an Electrocyclic Chemical Reaction', M. P. Minitti *et al*., Phys. Rev. Lett. **114**, 255501 (2015).

Coherent X-ray diffraction imaging of bacteria (26 May 2015)

Coherent X-ray diffraction imaging is one of a number of recently developed lens–less microscopic techniques giving 2D real space structure when combined with phase retrieval data processing. A team in Shandong University in China has recently published an interesting observation of intact unstained magnetotactic bacteria. It was confirmed that the reconstructed images give some intercellular structures, such as nucleoid, polyβ-hydroxybutyrate granules, and magnetosomes, which have been identified by electron microscopy. The team was also successful in quantification of the density, i.e. it was found that the average density of magnetotactic bacteria is 1.19 g/cm^3 from their data. The experiment was carried out with 5-keV X-ray photons at BL29XU, SPring-8, Japan. For more information, see the paper 'Quantitative Imaging of Single Unstained Magnetotactic Bacteria by Coherent X.ray Diffraction Microscopy', Jiadong Fan *et al*., Anal. Chem. **87**, 5849 (2015).

Spectrally narrow X-ray pulses by manipulating Mössbauer resonance (22 May 2015)

A research group led by Professor Jorg Evers (Max Planck Institute for Nuclear Physics, Heidelberg, Germany) has recently reported a method for narrowing the spectral width of X-ray pulses by the use of subluminal light propagation. So far, in visible light, slow group velocity such as 17 m/s has been observed in low-temperature sodium gas at 435 nK [see, L. V. Hau *et al*., Nature, 397, 594 (1999)]. The authors intend a similar effect in X-ray wavelength photons by manipulating the optical response of the 14.4-keV Mössbauer resonance of ^{57}Fe nuclei. The method combines coherent control, as well as cooperative and cavity enhancements of light–matter interaction in a single setup. It was found that the reduced group velocity of the obtained X-ray pulses is lower than 10^{-4} of the speed of the light. For more information, see the paper 'Tunable Subluminal Propagation of Narrow-band X-ray Pulses', K. P. Heeg *et al*., Phys. Rev. Lett. **114**, 203601 (2015).

Chemical state analysis of phosphorus by synchrotron X-ray fluorescence and PIXE (30 April 2015)

A Slovenian group has recently reported the Kα and Kβ emission spectra of phosphorus, measured by monochromatic synchrotron

X-rays (3 keV, at ID26, ESRF) and a 2-MeV proton beam. They also compared them with a Density Functional Theory calculation using StoBe-deMon code (Stockholm-Berlin version of demon). For more information, see the paper 'Chemical State Analysis of Phosphorus Performed by X.ray Emission Spectroscopy', M. Petric *et al.*, Anal. Chem. **87**, 5632 (2015).

Electrochemical X-ray fluorescence now carried out *in situ* (10 April 2015)

Readers may remember that electrochemical X-ray fluorescence developed by Prof. Julie V. Macpherson's group at Warwick University, UK, can analyze sub-ppb level heavy elements in solution [see, news in no. 5, vol. 43 (2014)]. Recently, the research team published their successful extension of the technique to *in situ* time evolution analysis. Their electrode is a freestanding film of boron-doped diamond, and it can work also as an X-ray window. Primary X-rays pass through the back side of the electrode and excite the heavy elements in the electrodeposit on the electrode. In addition to quantitative analysis of a mixed solution of Hg^{2+}, Pb^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, and Fe^{3+}(all at 10-μM concentration), time evolution analysis of electrodeposition can be a very promising application of this unique method. For more information, see the paper 'Direct Identification and Analysis of Heavy Metals in Solution (Hg, Cu, Pb, Zn, Ni) by Use of *in Situ* Electrochemical X.ray Fluorescence', G. D. O'Neil *et al.*, Anal. Chem. **87**, 4933 (2015).

X-ray chemical monitoring of uranium oxide (18 March 2015)

Scientists at Los Alamos National Laboratory have recently reported the X-ray analysis of uranium oxideα-U_3O_8 samples under controlled temperatures and humidities. They found that the combined use of powder X-ray diffraction and U L-III EXAFS can help in identifying temporal changes of uranium oxide stored for a number of years. For more information, see the paper 'Oxidation and Hydration of U_3O_8 Materials Following Controlled Exposure to Temperature and Humidity', A. L. Tamasi *et al.*, Anal. Chem. **87**, 4210 (2015).

Fast X-ray chopper using hard disk drive's rotation (9 March 2015)

A very interesting idea that proposes the use of a motor in a hard disk drive as an X-ray chopper has been recently published. It can produce X-ray pulses of millisecond width and few microsecond rise time. In the research, the system was used to test the response of X-ray detectors such as ionization chambers and photo diodes. For more information, see the paper 'Hard Disk Drive Based Microsecond X-ray Chopper for Characterization of Ionization Chambers and Photodiodes', O. Muller *et al.*, Rev. Sci. Instrum. **86**, 035105 (2015).

Professional

XRF article features in *Spectroscopy's* celebration of 30 years (1 June 2015)

Ms. Laura Bush, who is an editorial director of *Spectroscopy*, has recently published an article on the present and future of X-ray fluorescence on the occasion of *Spectroscopy*'s celebration of 30 years. It is a summary of her interviews with experts. For more information, see the article 'Analysis of the State of the Art: XRF', Laura Bush, Spectroscopy, **30** (6) 86–94 (2015), which can be found online at http://www.spectroscopyonline.com/analysis-state-art-xrf A PDF file can also be downloaded from *iTunes*.

Review paper on crystallography of correlated disorder (21 May 2015)

Professors D. A. Keen (Rutherford Appleton Laboratory) and A. L. Goodwin (University of Oxford) have recently published an interesting review paper on disordered structures. For many years, crystallographers have determined the structures of many complicated crystals with atomic or even sub-atomic resolution. On the other hand, the structures of disordered systems, which lack the crystalline periodic order, are still not well understood because of the limits of the analytical technique. Correlated disorder is a disorder, but maintains crystallographic signatures, that can be used for classifying the type of disorder. For more information, see the paper 'The Crystallography of Correlated Disorder', D. A. Keen and A. L. Goodwin, Nature, **521**, 303 (2015).

Brookhaven National Lab's NSLS II now open for user experiments (10 February 2015)

The National Synchrotron Light Source II of Brookhaven National Laboratory will officially start user runs from the third cycle in 2015. Seven beamlines will be commissioned in September 2015, and a further 21 beamlines will be designed and constructed in the coming years. The facility provides the world's smallest electron beam emittance, resulting in the brightest X-ray source. For more information, visit the web page http://www0.bnl.gov/ps/nsls2/about-NSLS-II.php.

The following YouTube video also gives useful information: https://www.youtube.com/watch?v=AzP8EGHw4BA

Multimedia

Popular YouTube video of SLAC's public lecture on the application of synchrotron X-ray fluorescence to paintings (14 July 2015)

Lecture date: Tuesday, 14 July 2015. Jerry LaRue of SLAC, delivered the SLAC public lecture, 'Caught in the Act! Chemical Reactions Exposed'. (https://www.youtube.com/watch?v=RiASAbniQYw)

Popular YouTube video of 'Science of SLAC' (24 May 2015)

Professor Yi Cui's lecture, 'Batteries for the Future: What's Possible'? (https://www.youtube.com/watch?v=ISEzvNevyck)

New products

Mythen2 X, a new microstrip detector from DECTRIS (16 April 2015)

DECTRIS has introduced a new performance class of microstrip detectors, the Mythen2 X series. The frame rate is 1 kHz, and the

dynamic range is 24 bit. For further information, visit the web page http://www.dectris.com/

World's first commercially available phase contrast CT from Bruker (25 March 2015)

Bruker's new SkyScan-1294 is based on phase-contrast imaging with polychromatic X-rays patented by the Paul Scherrer Institute at the Swiss Light Source (Zurich, Switzerland) and licensed to Bruker for commercialization. For further information, visit the web page, https://www.bruker.com/

Corporate

Shimadzu launches Innovation Center at its US subsidiary (1 July 2015)

Shimadzu Corporation has established the Innovation Center at Shimadzu Scientific Instruments, Inc. (SSI), its wholly owned US subsidiary based in Columbia, Maryland, USA. For further information, visit the web page, http://www.shimadzu.com/

Rigaku acquires former Oxford Diffraction (30 April 2015)

Rigaku Corporation has announced the completion of the acquisition of the X-ray diffraction business from Agilent Technologies Inc. (NYSE: A). Formerly known as Oxford Diffraction within Varian when that company was acquired by Agilent in 2010, the X-ray diffraction group develops single-crystal X-ray instruments for chemical crystallography. For further information, visit the web page http://www.rigaku.com/

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