X-RAY Spectrometry

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News

Sciences

Recent progress of U-shaped rotating anode X-ray source (November 5, 2010)

A U-shaped design for rotating anode X-ray sources is one method for enabling high brilliance, and was first proposed by Professor N. Sakabe (KEK, Tsukuba, Japan) in 1995. Unlike ordinary rotating anode X-ray sources, the electron beam goes beyond the outside surface of the rotating anode and then reverses its direction so that it can hit the inside surface. In this case, because of the centrifugal force of the rotating anode, the surface can be much smoother than usual even near the melting point, enabling the production of more X-ray photons. A KEK research group has published a report on recent progress with this type of X-ray source. According to their simulation, by optimizing both the bending and the steering magnets, the beam size can be 0.45 mm (horizontal) × 0.05 mm (vertical) for a 120 keV/75 mA beam. The effective brilliance is about 500 kW/mm^2. For more information, see the paper, ''Research and development of an electron beam focusing system for a high-brightness X-ray generator'', T. Sakai *et al*., J. Synchrotron Rad. **18,** (2011) (Published online, DOI:10.1107/S0909049510029948).

Calculation of $L_{II, III}$ X-ray absorption spectra for 3d transition metals (November 3, 2010)

Professor P. Blaha (Technische Universität Wien, Austria) and his colleagues have recently calculated the X-ray absorption spectra at the $L_{II, III}$ edges of the early 3d elements by solving the Bethe-Salpeter equation (BSE). Under the independent particle approximation (IPA), X-ray absorption spectra are usually considered as proportional to the unoccupied part of the projected density of states weighted by the momentum matrix elements between the core and the conduction states. However, due to the localization of the core wave function, the interaction of the exited electron with its hole is not negligible. In particular, the branching ratio between the L_{II} and L_{III} edges cannot be explained. Though the IPA gives 1 : 2, the observed ratios are much closer to 1 : 1 or even higher for K, Ca, Sc, Ti, and V. They detailed this problem based on their BSE calculation. For more information, see the paper, ''Understanding the L2,3 x-ray absorption spectra of early 3d transition elements'', R. Laskowski *et al*., Phys. Rev. **B 82**, 205104 (2010).

Calculation of X-ray emission from doubly ionized neon (October 25, 2010)

When an X-ray source with extremely high brilliance such as an X-ray free electron laser is used for X-ray spectroscopy experiments, we have to consider the significant population of the two core-hole states because of two-photon x-ray absorption (see, for example, the previous news article, ''''Hollow'' neon atom created by X-ray laser excitation'' in X-ray Spectrometry, Vol. 39, No.5 (2010)). A research group led by Professor F. Gel'mukhanov (Royal Institute of Technology, Sweden) has recently published the calculation of the resonant X-ray emission from a neon atom induced by the two-photon population of a double-core-hole excited state. They studied mainly 2 cases; an off-resonant intermediate single-core-hole state, and a resonant core-ionized intermediate state. For more information, see the paper, ''Two-photon-induced x-ray emission in neon atoms'', Y-P. Sun *et al*., Phys. Rev. **A 82,** 043430 (2010).

A laser-plasma base synchrotron radiation source (October 24, 2010)

A number of new methods have been proposed of late to produce X-ray photons with high brightness and short pulse duration, namely extremely high order harmonics from a laser, inverse Compton scattering and laser plasma acceleration. Dr. S. Kneip and his colleagues have recently applied the last of these types of technology to a table top synchrotron X-ray source. The method uses the phenomenon whereby the passage of an intense laser pulse through an underdense plasma generates a so-called plasma wakefield, which can have intrinsic fields of 1,000 times or more the best achievable by conventional accelerator technology. Some readers might recall previous news articles, ''Table-top soft X-ray undulator source'' in X-ray Spectrometry, Vol. 39, No.1 (2010) and ''A compact synchrotron light source driven by pulse laser'', in X-ray Spectrometry, Vol. 37, No.2 (2008). Both are pioneering reports on wakefield-driven synchrotron radiation. After generating high-energy electrons by laser pulse, they tried to transport the beam to an undulator, which is a magnet array and usually known as an insertion device placed at the straight section of the storage ring. In contrast, the electron beam in the plasma accelerator undergoes transverse (betatron) oscillations when subject to the focusing fields of the plasma wave, and the radiation wavelength can extend to the hard X-ray region as well. The research group succeeded in enhancing the brilliance of this betatron radiation by optimizing the wakefield conditions. They were able to accelerate electrons upto 230 MeV with a 5 mm path, and the critical energy of the obtained radiation was 6–10 keV. For more information, see the paper, ''Bright spatially coherent synchrotron X-rays from a table-top source'', S. Kneip *et al*., Nature Physics, (2010) (Published online, DOI:10.1038/nphys1789).

Phase retrieval in highly strained crystals (October 20, 2010)

One of the hottest topics in X-ray structural analysis is coherent X-ray diffraction measurement to obtain real-space images of nanoscale crystals. The key here is the method of phase retrieval. Until now, iterative projective algorithms have been frequently employed to recover the phase information from the amplitude measured in reciprocal space. The analysis relies on experimental data to be oversampled, and there have been difficulties in the case of highly strained structures, where information is below the Nyquist frequency. A research team led by Professor I. K. Robinson (University College London, UK) has recently reported a new method, called a density modification phase reconstruction algorithm, to solve this problem. This is a successful extension of the recent compressive sensing theory and works well in solving the nonconvex phase retrieval problem for highly strained crystalline materials. For more information, see the paper, ''Phase retrieval of diffraction from highly strained crystals'', M. C. Newton *et al*., Phys. Rev. B 82, 165436 (2010).

$K\beta/K\alpha$ intensity ratio in Ti_xCo_{1-x} alloy (October 17, 2010)

Dr. I. Han (Ağrı İbrahim Çeçen University, Turkey) and his colleagues have published a paper on the relationship between the $K\beta/K\alpha$ X-ray fluorescence intensity ratio and valence-electron configurations in Ti_xCo_{1-x} ($x = 0.7, 0.6, 0.5, 0.4$, and 0.3). For more information, see the paper, ''Relative K x-ray intensity studies on valence-electron structure of Ti and Co in Ti_xCo_{1-x} alloys'', I. Han *et al*., Phys. Rev. **A82**, 042514 (2010).

Separation of diffuse scattering in specular X-ray reflectivity measurement (October 8, 2010)

A research group led by Professor H. Zabel (Ruhr-Universität Bochum, Germany) has recently published an interesting paper discussing the solution to a well-known problem in X-ray reflectivity. The technique is for layered thin films, and can give the layer thickness, surface/interface roughness and correlations of the interface roughness parallel and perpendicular to the interface. Due to the finite size of the receiving detector slit, it will always collect not only pure specular reflection but also diffusely scattered radiation. For many years, the separation of the diffuse contribution to the intensity of specular reflection has been an important topic for reliable data analysis. The researchers propose several measurements using different slit openings for specular scans, and show

some applications to realistic systems, such as periodic V/Fe multilayers prepared on MgO substrate, with V and Pd capping layers. For more information, see the paper, ''Separation of the diffuse contribution to the specular x-ray scattering of multilayer films'', V. P. Romanov *et al*., Phys. Rev. **B 82,** 165416 (2010).

Aerosol analysis by soft X-ray spectromicroscopy near carbon absorption edge (September 30, 2010)

A research group at Lawrence Berkeley National Laboratory, USA, has recently published an interesting report on an automated data analysis method for submicrometer atmospheric particles containing organic and inorganic material. The main idea is the use of X-ray spectral features in the energy range from 278 to 320 eV, which is near the carbon K-edge and potassium LII and LIII edges. The method provides quantitative mapping of the spatial distribution of elemental carbon, organic carbon, potassium, and noncarbonaceous elements in particles of mixed composition. It was recently applied to analyze differences in over 1000 particles collected at various times and locations in Mexico City to examine the effects of atmospheric aging on internally mixed atmospheric aerosol particles. For more information, see the paper, ''Automated Chemical Analysis of Internally Mixed Aerosol Particles Using X-ray Spectromicroscopy at the Carbon K-Edge'', R. C. Moffet *et al*., Anal. Chem. **82,** 7906 (2010).

Professional

JPCM's special section on surfaces and buried interfaces research by X-rays and neutron techniques (December 1, 2010)

In Issue 47, vol. 22 (2010) of Journal of Physics: Condensed Matter, a special section features a compilation of articles on exploring surfaces and buried interfaces of functional materials by advanced X-ray and neutron techniques. Many of the authors are members of a group established in the Japan Applied Physics Society, and this is their 9th collection of articles since 2001. Unlike many other surface-sensitive methods, these techniques do not require ultra high vacuum, and therefore, a variety of real and complicated surfaces fall within the scope of analysis. It must be particularly emphasized that the techniques are capable of seeing even buried function interfaces as well as the surface. Furthermore, the information, which ranges from the atomic to mesoscopic scale, is highly quantitative and reproducible. Such features are fairly attractive when exploring multilayered materials with nanostructures (dots, tubes, wires, etc), which are finding applications in electronic, magnetic, optical and other devices. Visit the Web page to download the papers in this collection, http://iopscience.iop.org/0953-8984/22/47

National Geographic's news column on 115-year-old X-ray photo (November 8, 2010)

W. Rontgen took the world's first X-ray photo on November 8, 1895, thereby creating the very famous X-ray image of his wife's fingers. Ker Than has written a short article in National Geographic News on this X-ray photo. For more information, see the article, ''115-Year-Old X-Ray'', http://news.nationalgeographic.com/news/2010/11/photogalleries/101108-x-rays-google-doodle-115th-anniversary-years-science-pictures/?now=2010-11-08-00:01

The 5th Asada award (October 22, 2010)

The recipient of the 5th 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. Tsutomu Kurisaki (Fukuoka Univ., ''Development of a novel soft X-ray absorption spectroscopic measurement apparatus and structural analysis of various metals ions and metal complexes in aqueous solution''). The ceremony was held during the 46th Annual Conference on X-Ray Chemical Analysis, Japan, at the Hiroshima Prefectural Information Plaza, Hiroshima.

New Products

PerkinElmer's new X-ray flat panel detectors (November 15, 2010)

PerkinElmer has announced two new products in its Flat Panel Detector family, the XRD 0822 and XRD 1622, with effective area sizes of 20 cm^2 and 41 cm^2, respectively. The detectors are designed mainly for non destructive testing, and support an energy range above 20 keV. The latitude range and the frame rate are ca. 13 bit and 100 Hz, respectively. For further information, visit the web page, http://www.perkinelmer.com/

Rigaku's application notes for the analysis of petrol (October 17, 2010)

Rigaku Corp has published three application reports for the petroleum market. Two explain the elemental analysis of P, S, Ca, Zn, Mg, Ba, Cu and Cl in a typical lubricating oil formulation using respective empirical and fundamental parameters (FP) calibration methods. The third details the measurement of sulfur in ULSD (ultra-low sulfur diesel) by ASTM D7220. In the reports, calibration summaries and typical detection limits are presented, and instrument repeatability is demonstrated. For further information, visit the web page, http://www.rigaku.com/

Corporate

PANalytical's new office in Houston, USA (October 11, 2010)

PANalytical Inc. has announced the opening of a new office with demonstration laboratory and meeting space near Houston, TX, USA. For further information, visit the web page, http://www.panalytical.com/

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For additional news about X-ray analysis and other spectroscopy sciences, please browse the Wiley website. http://www.SpectroscopyNow.com

Kenji Sakurai
News Editor
sakurai@yuhgiri.nims.go.jp
or sakurai@pas.tsukuba.ac.jp

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News

Sciences

X-rays unveil the mystery of color changes in Van Gogh's paintings (February 14, 2011)

Recently, a European international research group led by Professor K. Janssens (Antwerp University, Belgium) has succeeded in solving the scientific mechanism of color darkening in the paintings of Vincent van Gogh. Some readers may remember a previous news article, "Synchrotron XRF revealed Van Gogh's hidden painting", No.5, Vol. 37 (2008), which explained how synchrotron X-ray spectroscopy and imaging are powerful tools in the analysis of such paintings. In the present work, the research group discusses the change in color from yellow to dark brown in two Van Gogh paintings, *Bank of the Seine* (1887) and *View of Arles with Irises* (1888). They also systematically studied the aging process of artificial samples using pigments. The chrome yellow pigment is chemically lead chromate ($PbCrO_4$), which may include some amount of $PbSO_4$ and/or PbO. During their research based on X-ray micro-spectroscopy, it was found that part of the material is transformed into hydrated chromium oxide ($Cr_2O_3 \bullet E2H_2O$), which is known as viridian, i.e., a blue-green pigment under sunlight or UV light irradiation. They also noted the formation of other Cr(III) compounds. Their conclusion was that the color change is due to the reduction from Cr(VI) to Cr(III) on the surface of the paintings, and the formation of a thin layer containing Cr(III). This would be the reason for the brownish color. Most of the experiments were done at beamline ID21 at the European Synchrotron Radiation Facility (ESRF, Grenoble, France). For more information, see the papers, "Degradation Process of Lead Chromate in Paintings by Vincent van Gogh Studied by Means of Synchrotron X-ray Spectromicroscopy and Related Methods. 1. Artificially Aged Model Samples" and "2. Original Paint Layer Samples", L. Monico *et al.*, Anal. Chem., **83** 1214–1231 (2011).

High-resolution V Kα spectra of catalysts (February 8, 2011)

A Japanese research group led by Professors J. Kawai (Kyoto University) and T. Yamamoto (Tokushima University) has recently published a series of high-resolution X-ray fluorescence spectra for supported vanadium oxide catalysts. The measurement was done with a double crystal spectrometer (Si 220 reflections with (+, +) arrangement), and the typical energy resolution was around 2.5 eV. The authors were successful in discussing quantitatively the difference in chemical states among the catalysts supported on the different oxides, amorphous SiO_2, γ-Al_2O_3, and TiO_2 (anatase/rutile = 7/3). For more information, see the paper, "Quantitative Chemical State Analysis of Supported Vanadium Oxide Catalysts by High Resolution Vanadium Kα Spectroscopy", T. Yamamoto *et al.*, Anal. Chem., Article ASAP (DOI: 10.1021/ac102681z Publication Date (Web): February 8, 2011).

Calculation of L X-ray production cross sections of heavy metals (February 4, 2011)

Dr. J. M. Fernandez-Varea (Universitat de Barcelona, Spain) and his colleagues have recently studied the emission of Lα, Lβ, and Lγ characteristic X-rays by the impact of electrons on Hf, Ta, W, Re, Os, Au, Pb, and Bi atoms. They calculated the ionization cross sections of the LI, LII, and LIII subshells of these atoms within the distorted-wave Born approximation, and compared them with the published experimental data. For more information, see the paper, "Lα, Lβ, and Lγ x-ray production cross sections of Hf, Ta, W, Re, Os, Au, Pb, and Bi by electron impact: Comparison of distorted-wave calculations with experiment", J. M. Fernandez-Varea *et al.*, Phys. Rev. **A83,** 022702 (2011).

Femto-second coherent X-ray diffraction imaging of tiny proteins using X-ray free electron laser (February 3, 2011)

Two very exiting experimental reports have been published on the application of an X-ray free electron laser (XFEL) at Linac Coherent Light Source (LCLS, Stanford, USA). An international research team led by Dr. H. Chapman (DESY, Hamburg, Germany) and Professor J. Hajdu (Uppsala University, Sweden) has demonstrated a new advanced stage of protein crystallography, which uses only tiny proteins instead of preparing large-size crystals. This could open up new possibilities for the analysis of proteins that have been difficult or even impossible to prepare so far. The technique has been basically known as coherent X-ray diffraction imaging. The present research is the first experimental application of extremely brilliant femtosecond XFEL pulses. In addition to the demonstration of snapshots of nano-crystalline proteins, they have reported the first single-shot images of intact viruses. For more information, see the papers, "Femtosecond X-ray protein nanocrystallography", H. N. Chapman *et al.*, Nature, 470, 73 (2011) and "Single mimivirus particles intercepted and imaged with an X-ray laser", M. M. Seibert *et al.*, Nature, 470, 78 (2011).

Theoretical models for molecular imaging under significant damage by X-ray free electron laser (February 1, 2011)

One of hottest topics related to the application of an X-ray free electron laser (XFEL) is how to determine the structure of non-crystalline membrane proteins. There has been a clear conflict between the incident brightness required to achieve diffraction-limited atomic resolution and the electronic and structural damage induced by such illumination. Professors K. A. Nugent and H. M. Quiney (ARC Centre of Excellence for Coherent X-ray Science, University of Melbourne, Victoria, Australia) have recently published their theoretical research on this problem. They have improved the imaging model by using optical coherence theory and quantum electrodynamics, and concluded that the analysis is far more tolerant of electronic damage than believed so far. For more information, see the paper, "Biomolecular imaging and electronic damage using X-ray free-electron lasers", H. M. Quiney *et al.*, Nature Physics, 7, 142 (2011).

Total-reflection inelastic X-ray scattering (January 18, 2011)

Many readers of this news column are familiar with total-reflection X-ray fluorescence (TXRF). They also know that experiments can be done with a wavelength-dispersive mode, besides ordinary measurement with a silicon drift detector or a Si(Li) detector. If the spectrometer is optimized to see inelastic X-ray scattering spectra, what happens? Very recently, a research team led by Dr. P. H. Fuoss (Argonne National Laboratory, USA) published a very interesting report. The experiment used soft X-rays to observe the electronic structure of a 10-nm-thick $La_{0.6}Sr_{0.4}CoO_3$ epitaxial layer grown on a $SrTiO_3$ substrate. By comparing data acquired under total X-ray reflection and penetrating conditions, it was found that the O K-edge spectra from a 10 nm thin film and that from the underlying substrate can be separated successfully. For more information, see the papers, "Total-Reflection Inelastic X-Ray Scattering from a 10-nm Thick La0.6Sr0.4CoO3 Thin Film", T. T. Fister *et al.*, Phys. Rev. Lett. 106, 037401 (2011).

Continuous Wavelet transform of XRF spectra (January 11, 2011)

Dr. S. Arzhantsev (Center for Drug Evaluation and Research, US Food and Drug Administration, St. Louis) and his colleagues have published some very interesting research. The research group is engaged in the determination of toxic metals in pharmaceutical materials using hand-held XRF spectrometers. It is extremely important with respect to toxic metal contamination to establish a reliable technique for classifying a

large number of samples. As the procedure is basically a kind of pattern recognition, the problem that needs to be overcome is finding a suitable filter for signals and noises in XRF spectra. The research group chose a continuous Wavelet transform, which is an extension of short-time Fourier transform (STFT) and is capable of constructing a time-frequency representation of a signal that offers very good time and frequency localization. In the paper, they discussed the comparison of the signal-to-noise ratios at the energies of the elements of interest obtained by wavelet filtering and those obtained by the conventional empirical method. The results were evaluated in a collaborative study that involved 5 different hand-held XRF spectrometers used by multiple analysts in 6 separate laboratories across the United States, leading to more than 1200 measurements. The detection limits estimated for arsenic, lead, mercury, and chromium were 8, 14, 20, and 150 μg/g, respectively. For more information, see the paper, ''Rapid Limit Tests for Metal Impurities in Pharmaceutical Materials by X-ray Fluorescence Spectroscopy Using Wavelet Transform Filtering'', S. Arzhantsev *et al*., Anal. Chem., **83,** 1061 (2011).

Professional

Synchrotron X-ray analysis of star dust brought back by Japanese satellite *HAYABUSA* (February 2, 2011)

Scientists in Japan have been using two synchrotrons, the SPring-8 and the Photon Factory, to analyze the dust particles collected by the *HAYABUSA* Asteroid probe, which returned from Asteroid Itokawa on June 13, 2010. *HAYABUSA*, which means ''Falcon'' in Japanese, was launched from the Uchinoura Space Center in Japan on May 9, 2003, and arrived at Itokawa in September 2005. The *HAYABUSA* particles were initially analyzed using electron microscopes, and then forwarded to the above synchrotron facilities in January 2011. Many interesting 3D images were collected at BL20XU, SPring-8, and the structure and chemical compositions were also analyzed at BL-3A, Photon Factory, KEK. For more information on the *HAYABUSA* project, visit the web page of the Japan Aerospace Exploration Agency (JAXA), http://www.isas.jaxa.jp/e/enterp/missions/hayabusa/index.shtml

Two US and two Japanese scientists awarded 2011 Japan Prize (January 25, 2011)

The Science and Technology Foundation of Japan has announced that Japanese and US scientists have been named as laureates of the 2011 (27th) Japan Prize. Dr. Dennis M. Ritchie, 69, Distinguished Member of Technical Staff Emeritus, Bell Labs, and Dr. Ken Thompson, 67, Distinguished Engineer, Google Inc., have received the prize in this year's category of ''Information and Communications'' for developing the operating system (OS), UNIX, in 1969. Dr. Tadamitsu Kishimoto, 71, Professor Emeritus, Osaka University and Dr. Toshio Hirano, 63, Dean of the Graduate School of Medicine, Osaka University, were selected in the other prize category of ''Bioscience and medical science'' for their discovery of interleukin 6 (IL-6), a cell-signaling molecule in the immune system and its application in treating diseases. They will each receive a certificate of recognition and a commemorative gold medal at an award ceremony during Japan Prize Week in Tokyo on April 20, 2011. A cash award of 50 million Japanese yen (approximately US$600,000) will also be given to each field - this year the two laureates in each field will split the prize equally. The prize categories for the 2012 (28th) Japan Prize will be ''Environment, Energy, Infrastructure'' and ''Healthcare, Medical Technology''. For further information, visit the web page, http://www.japanprize.jp/en/index.html

New Products

PANalytical's new energy-dispersive XRF spectrometer (January 31, 2011)

PANalytical has announced its new range of Epsilon 3 benchtop spectrometers. For further information, visit the web page, http://www.panalytical.com/

New Niton FXL for mining and exploration (January 31, 2011)

Thermo Fisher Scientific Inc. has announced the launch of the Niton FXL field X-ray lab as the newest member of its family of X-ray fluorescence analyzers. For further information, visit the web page, http://www.thermoscientific.com/niton

Corporate

PANalytical acquires XRF laboratories from British Geological Survey (February 2, 2011)

PANalytical (Almelo, the Netherlands) has acquired the XRF laboratories from the British Geological Survey (BGS). For further information, visit the web page, http://www.panalytical.com/

On Semiconductor acquires CMOS image sensor business unit from Cypress (January 27, 2011)

On Semiconductor (Nasdaq: ONNN) and Cypress Semiconductor Corp. (Nasdaq: CY) have announced that a definitive agreement has been signed for ON Semiconductor to acquire the CMOS Image Sensor Business Unit (ISBU) from Cypress in an all cash transaction for approximately $31.4 million. The transaction is expected to close by the end of the first quarter of 2011. For further information, visit the web page, http://www.onsemi.com/

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Kenji Sakurai
News Editor
sakurai@yuhgiri.nims.go.jp
or sakurai@pas.tsukuba.ac.jp

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News

Sciences

Trace determination of perchlorate in water by TXRF (April 4, 2011)

Recently, a research group led by Professor N. Kallithrakas-Kontos (Technical University of Crete, Greece) reported successful total-reflection X-ray fluorescence (TXRF) analysis of perchlorate. In the present research, perchlorate anions were concentrated on anion-selective membranes prepared on a mirror-polished quartz substrate. Then the quartz reflectors were taken out of the solution and analyzed by measuring Cl Kα intensity under the total-reflection condition, using a copper X-ray tube and helium atmosphere. The effects of many experimental parameters were discussed in detail, and even the possible capability of discrimination between chloride and perchlorate anions was suggested. The minimum detection limit was lower than 1 ng/mL. For more information, see the paper, ''Determination of Trace Perchlorate Concentrations by Anion-Selective Membranes and Total Reflection X-ray Fluorescence Analysis'', V. S. Hatzistavros *et al*., Anal. Chem., Article ASAP (DOI: 10.1021/ac103295a Publication Date (Web): April 4, 2011).

Confocal XRF imaging of forensic samples (March 25, 2011)

A Japanese group led by Professor K. Tsuji (Osaka City University, Japan) recently reported an interesting application of 3D micro X-ray fluorescence (XRF) imaging. One should note that their research employed low-power laboratory X-ray sources (30-50 W, Mo tube) instead of synchrotron X-rays. They also used two polycapillary lenses for both incoming and outgoing directions to limit the viewing volume in 3D. The research group measured some forensic samples such as multilayered automotive paint fragments, leather samples etc., which have different color coatings. They analyzed 3D profiles of many elements (Si, S, Cl, K, Ca, Ti, Mn, Fe, Zn, and Ba) and discussed the relationship with the coating. For more information, see the paper, ''Depth Elemental Imaging of Forensic Samples by Confocal micro-XRF Method'', K. Nakano *et al*., Anal. Chem., Article ASAP (DOI: 10.1021/ac1033177 Publication Date (Web): March 25, 2011).

Observation of laser-driven electron acceleration (March 13, 2011)

It is extremely important to develop new X-ray sources for future X-ray spectrometry. One promising direction is a table-top synchrotron X-ray source, which consists of a high-power pulse laser and an undulator. The method uses acceleration of electrons by pulse laser photons. The idea becomes realistic once the energy reaches GeV and other properties such as stability, emittance etc are improved sufficiently. For such development, it is indispensable to establish the method for quantitatively investigating the structure of the electron beam in time and space. Recently, a German group succeeded in taking snapshots of the magnetic field generated by an accelerated electron bunch and simultaneously of a plasma wave by a combination of two techniques: time-resolved polarimetry and plasma shadowgraphy. For more information, see the paper, ''Real-time observation of laser-driven electron acceleration'', A. Buck *et al*., Nature Physics (Published online, March 13, 2011 DOI:10.1038/nphys1942).

How to use XFEL to determine the structure of a single molecule (March 9, 2011)

One of the hottest topics in X-ray crystallography in the early 21st century is coherent X-ray diffraction imaging and its application to the determination of atomic structures of non-crystalline materials - the ultimate goal can be a single molecule. The technique appears to require non-ordinary coherent photon sources, such as X-ray free-electron lasers (XFEL), which are now in operation at Stanford. On the other hand, there are several challenging questions basically concerning sample damage, Coulomb explosion, and the role of nonlinearity. Recently, Dr. A. Fratalocchi and his colleague published their calculations showing that XFEL-based single-molecule imaging will only be possible with a few-hundred long attosecond pulses, due to significant radiation damage and the formation of preferred multisoliton clusters which reshape the overall electronic density of the molecular system at the femtosecond scale. For more information, see the papers, ''Single-Molecule Imaging with X-Ray Free-Electron Lasers: Dream or Reality?'', A. Fratalocchi *et al*., Phys. Rev. Lett. 106, 105504 (2011).

X-ray spectrometry aids understanding of how iron gall ink degrades paper (March 4, 2011)

A French-Belgian joint group led by Dr. V. Rouchon (Centre de Recherche sur la Conservation des Collections, MNHN-MCC-CNRS, France) and Professor K. Janssens (Universiteit Antwerpen, Belgium) recently published an interesting paper on the application of X-ray spectrometry to cultural heritage. For many years, in Europe, iron gall inks have been used for writing manuscripts, and they could damage the paper via two major ways: (i) acid hydrolysis, enhanced by humidity, and (ii) oxidative depolymerization provoked by the presence of oxygen and free Fe(II) ions. The present research aimed to give some quantitative evidence for each contribution by studying depolymerization of cellulose under various environmental conditions, with viscometry and related changes in the oxidation state of iron determined by X-ray absorption near-edge spectrometry. It was found that residual amounts of oxygen (less than 0.1%) promote cellulose depolymerization, whereas the level of relative humidity has no impact. For more information, see the paper, ''Room-Temperature Study of Iron Gall Ink Impregnated Paper Degradation under Various Oxygen and Humidity Conditions: Time-Dependent Monitoring by Viscosity and X-ray Absorption Near-Edge Spectrometry Measurements'', V. Rouchon *et al*., Anal. Chem., **83,** 2589 (2011).

Application of energy-dispersive 2D detector to X-ray color imaging (February 28, 2011)

A German group recently developed an X-ray fluorescence imaging system with a pnCCD-based camera. They performed a test using a laboratory 30 μm microfocus X-ray tube and synchrotron radiation at the BAM beamline, BESSY II. It was found that the system simultaneously records ca. 70,000 spectra with an energy resolution of 152 eV (at Mn Kα) with a spatial resolution of 50 μm over a viewing area of 12.7 mm squared. For more information on pnCCD detectors, for example, the following Web page could be useful, http://www.pnsensor.de/Welcome/Detector/pn-CCD/index.html For more information on the whole system for X-ray fluorescence imaging, see the paper, ''Compact pnCCD-Based X-ray Camera with High Spatial and Energy Resolution: A Color X-ray Camera'', O. Scharf *et al*., Anal. Chem., **83,** 2532 (2011).

In-situ X-ray spectrometry of reacting catalysts (January 19, 2011)

Recently, a research group at Lawrence Berkeley National Laboratory reported an interesting application of X-ray absorption spectrometry to studies on the oxidation states of Co and CoPt nanoparticles in the presence of H_2 andO_2 at a controlled pressure. The key to the research lies in the specially developed gas reaction cell. For more information, see the paper, ''In-situ X-ray Absorption Study of Evolution of Oxidation States and Structure of Cobalt in Co and CoPt Bimetallic Nanoparticles (4 nm) under Reducing (H_2) and Oxidizing (O_2) Environment'', F. Zheng *et al*., Nano Lett., **11,** 847 (2011).

New theoretical expression of X-ray reflectivity by using Green function (September 22, 2010)

X-ray reflectivity is one of the most power analytical tools for observing the layered structures of thin films. So far, many calculations have been done by combining Parratt's recursive formalism with Nevot-Croce corrections on the Fresnel coefficients. The technique basically provides detailed information on the roughness of the surface and interfaces, in addition to the precise thickness values of each layer. However, the analysis of the roughness has not been always straightforward, because it is also necessary to consider multiple diffuse scattering. Recently, Dr. A. M. Polyakov (National University of Science and Technology 'MISiS', Russia) and his colleague published an interesting paper describing a novel approach to the calculation of X-ray reflectivity. Their method is based on the Green function formalism using Kirchhoff's integral equation for describing the X-ray wavefield propagation through a random rough surface separating vacuum and medium. Readers would find it interesting that the influence of multiple diffuse scattering effects upon grazing X-ray specular scattering is essential for the correlation lengths that are of the order of, and/or less than, the X-ray absorption length. Although the present calculation is only valid for the random surface heights described in the frame of Gaussian statistics, the present approach can be further extended in the future. For more information, see the papers, "X-ray specular scattering from statistically rough surfaces: a novel theoretical approach based on the Green function formalism", F. N. Chukhovskii *et al.*, Acta Cryst., **A66,** 640 (2010).

Professional

Influence of the M9 class earthquake on synchrotron facilities in Japan (March 11, 2011)

As a result of the Tohoku Region Pacific Coast Earthquake in Japan, which took place on March 11, 2011, nearly 30,000 people were killed or are still missing. As can be clearly seen from the map of the magnitude of shaking intensity (see, for example, http://www.scientificamerican.com/article.cfm?id=fast-facts-japan), several research facilities were affected by this disaster. Very strong quakes took place in Tsukuba, Ibaraki prefecture, where the Photon Factory, a synchrotron source, is located. However, first of all, the map does not correspond very well to the loss of lives and damage to buildings, roads, railways and other infrastructure. While the coastal areas of Miyagi, Iwate and Fukushima prefectures were destroyed by the tsunami, many cities and towns in the inland area were quite safe. In spite of the largest earthquake since scientific surveys started, damage was minimal. No lives were lost, and no buildings were completely destroyed in the campus of the Photon Factory. The detailed status of the facility is available in the following Web page, http://pfwww.kek.jp/whats_new/earthquakeinfo/announce_e.html.

All beamtime allocated in the term from May to September has been cancelled. On the other hand, another Japanese synchrotron radiation facility, SPring-8 had no damage, because the location is far from the source of the earthquake. The SPring-8 plans to accept some users of the Photon Factory for experiments. For further information, visit the Web page, http://www.spring8.or.jp/en/urgentnews/110401

NSLS-II passes the halfway stage of construction (March 22, 2011)

In 2009, the U.S. Department of Energy's Brookhaven National Laboratory started construction of the National Synchrotron Light Source II (NSLS-II), which is a new advanced synchrotron X-ray source with a 3 GeV storage ring and around 30 beamlines. Construction has now passed the halfway stage, and magnet installation has just started. The completion of the facility is expected in 2015. For further information, visit the web page, http://www.bnl.gov/nsls2/

New Products

Princeton releases new direct detection X-ray cameras for extremely low flux applications (March 25, 2011)

Princeton Instruments has announced the new PIXIS-XB series of direct detection cameras. The PIXIS-XB series is designed for extremely low flux imaging and spectroscopy applications in the X-ray energy range of 3–20 keV. The new PIXIS-XB models feature front-illuminated and back-illuminated, deep depletion CCDs with a wide variety of formats from 1340 × 400 to 1340 × 1300 pixels. For further information, visit the web page, http://www.princetoninstruments.com/

Bruker's new handheld XRF spectrometer (March 14, 2011)

Bruker has announced the introduction of the new TRACER IV-GEO handheld XRF system, which is equipped with 30 mm^2 Silicon Drift Detector (SDD), achieving about three times the sensitivity of previous products. For further information, visit the web page, http://www.bruker-axs.com/

Corporate

Oxford Instruments allies with Vericheck in handheld XRF business (April 3, 2011)

Oxford Instruments Industrial Analysis has announced an agreement with Verichek Technical Services based in Jefferson Hills, PA USA, to sell the X-MET handheld XRF analyzer in the scrap metal and positive material identification (PMI) markets. For further information, visit the web page, http://www.oxinst.com/

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Kenji Sakurai
News Editor
sakurai@yuhgiri.nims.go.jp
or sakurai@pas.tsukuba.ac.jp

X-RAY Spectrometry

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News

Sciences

Grazing-incidence inelastic X-ray scattering: lattice dynamics in thin films (May 19, 2011)

Inelastic X-ray scattering is a powerful modern tool to study lattice dynamics of condensed matter. Recently an international team led by Dr. J. Serrano (Polytechnic University of Catalonia, Spain) has tried to extend the technique to several micron-thick systems by introducing grazing-incidence geometry. Their sample is indium nitride grown on a sapphire substrate with a gallium nitride buffer layer inbetween, but X-rays only probe the surface, and not the substrate underneath. The analysis was combined with *ab initio* calculations to determine the complete elastic stiffness tensor, the acoustic and low-energy optic phonon dispersion relations. This finding could be a help in developing new types of solar cells. For more information, see the paper, ''InN Thin Film Lattice Dynamics by Grazing Incidence Inelastic X-Ray Scattering'', J. Serrano *et al*., Phys. Rev. Lett. **106,** 205501 (2011).

First-principle calculation of resonant X-ray emission spectra (May 16, 2011)

An interesting theoretical paper on the calculation of K edge resonant X-ray emission spectroscopy has been published recently. The crystalline band structure was calculated using a quasiparticle self-consistent GW implementation, and then coherent spectra were obtained in the Kramers-Heisenberg formalism. The calculated results for ZnO were compared with experiments. For more information, see the paper, ''First-principles calculation of resonant x-ray emission spectra applied to ZnO'', A. R. H. Preston *et al*., Phys. Rev. **B83,** 205106 (2011).

Pump-probe X-ray microscopy: both time and spatial resolution (May 10, 2011)

A research team led by Professor J. Stohr (SLAC National Accelerator Laboratory, Stanford, USA) has recently performed time-resolved scanning transmission X-ray microscopy measurements to study the current-induced magnetization switching mechanism in nanopillars exhibiting strong perpendicular magnetic anisotropy. Because of both the short-time (70 ps) and high-spatial (25 nm) resolutions of this imaging technique, the detailed mechanism has become clear as follows; after an incubation time of $\sim$ 1.3 ns, a 100 $\times$ 300 nm^2 ellipsoidal device switches in $\sim$ 1 ns via a central domain nucleation and opposite propagation of two domain walls toward the edges. For further understanding, micromagnetic simulations were done and shown as being in good agreement with experiments. For more information, see the paper, ''Nonuniform switching of the perpendicular magnetization in a spin-torque-driven magnetic nanopillar'', D. P. Bernstein *et al*., Phys. Rev. **B83,** 180410(R) (2011).

X-ray spectra reveal color changes in historical paintings (May 9, 2011)

A research team led by Dr. L. Robinet (Synchrotron Soleil, Saint Aubin, France) has recently published an interesting paper describing how the blue pigment, smalt, has faded in many famous paintings such as ''The Heavenly and Earthly Trinities (The Pedroso Murillo)'' by Bartolome Esteban Perez Murillo. The experiment was basically X-ray absorption spectroscopy near the Co K edge. The samples were tiny pieces taken from the original paintings archived in the National Gallery, London and the Louvre Museum, Paris. It was found that in intense blue particles the cobalt is predominantly present as Co^{2+} in tetrahedral coordination, while in colorless altered smalt the Co^{2+} coordination number in the glass structure is increased, and there is a transformation from tetrahedral towards octahedral coordination. This change correlates with the alkali content as well, indicating that it is caused by leaching of potassium cations, which act as charge-compensators and stabilize the tetrahedral coordination of the cobalt ions that is responsible for the blue color. For more information, see the paper, ''Investigation of the discoloration of smalt pigment in historic paintings by Co K-edge micro X-ray absorption spectroscopy'', L. Robinet *et al*., Anal. Chem., Article ASAP (DOI: 10.1021/ac200184f Publication Date (Web) May 9, 2011).

Dedicated optical tweezers for synchrotron experiments (May 4, 2011)

Optical tweezers are widely used because they are capable of trapping small materials by highly-focused laser beams. They are highly useful for manipulating single fragile objects. Recently compact optical tweezers have been designed and developed specifically for synchrotron X-ray diffraction experiments. Samples of a few micrometers up to a few tens of micrometers size can be trapped easily. The selection and positioning of single objects out of a batch of many can be performed semi-automatically by software routines. For more information, see the paper, ''Optical Tweezers for Synchrotron Radiation Probing of Trapped Biological and Soft Matter Objects in Aqueous Environments'', S. C. Santucci *et al*., Anal. Chem., Article ASAP (DOI: 10.1021/ac200515x Publication Date (Web) May 4, 2011).

Near edge X-ray absorption imaging of geo-samples (April 18, 2011)

In most cases, rocks and geomaterials are chemically and structurally inhomogeneous. The use of X-ray absorption spectro-microscopy is one promising solution, but the very long measuring time for scanning large samples with a tiny beam poses a limit for detailed analysis. At the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, scientists recently performed much more efficient and feasible experiments by coupling near-edge X-ray absorption spectroscopy and full-field transmission radiography with a large X-ray beam. The method basically consists of the repeated acquisition of X-ray images as a function of X-ray energy near the absorption edge (in the present case, iron K edge). The research group also combines this with polarization contrast imaging. By looking at the Fe^{3+}/Fe(total) image, some redox variations were found in the single mineralogical phase of complex metamorphic rocks. The research group also analyzed bentonite analogue by separating the spectra into those of 5 simple minerals. The material is a candidate for the storage of nuclear waste and CO_2, and the information is helpful in designing such applications. For more information, see the paper, ''Submicrometer Hyperspectral X-ray Imaging of Heterogeneous Rocks and Geomaterials: Applications at the Fe K-Edge'', V. De Andrade *et al*., Anal. Chem., Article ASAP (DOI: 10.1021/ac200559r Publication Date (Web) April 18, 2011).

Full-field phase contrast imaging with coded aperture and laboratory X-ray source (April 18, 2011)

Phase contrast X-ray imaging is a promising method for low Z samples which cannot always be properly imaged by conventional absorption and scattering imaging. Recently Professor R. D. Speller (University College London) and his colleagues reported a novel way using a laboratory X-ray source outfitted with a pair of coded apertures; one in front of the sample for imaging and one behind it. They were offset slightly to remove scattering background. Readers might be aware that the method is quite similar to X-ray Talbot interferometry (for example, see the previous news article, ''Micro-structure imaging using visibility contrast'', No.5, Vol. 39 (2010)), when a 2D grating is used as a coded-aperture. The technique could open up many interesting opportunities through its application to a wide range of fields, such as nano-bio technologies, because the experiments can be done with an ordinary incoherent X-ray source. For more information,

see the paper, "Noninterferometric phase-contrast images obtained with incoherent x-ray sources", A. Olivo *et al*., Appl. Optics, **50,** 1765 (2011).

Use of zone plates to make X-ray microscopy more quantitative (April 8, 2011)

A group led by Professor C. Chang (University of Pennsylvania, USA) has recently reported a quantitative X-ray phase imaging method that can be readily implemented on existing facilities. This technique utilizes Fresnel zone plates both as imaging optical elements for magnification and as second-order grating structures for phase-shifting interferometry. For more information, see the paper, "Quantitative x-ray differential-interference-contrast microscopy with independently adjustable bias and shear", T. Nakamura *et al*., Phys. Rev. **A83,** 043808 (2011).

Synchrotron micro X-ray diffraction of cement samples (April 4, 2011)

A German group led by Professor U. Panne (Humboldt University, Berlin) has recently reported the successful application of the micro X-ray diffraction technique to the evaluation of the durability of cements against reaction with sodium sulfate. The experiments were done with a Debye-Scherrer camera equipped with a large-size CCD camera (3072 × 3072) and monochromatic micro beam (11.6 keV, 10 μm). By moving the sample along the X-ray path, it is possible to obtain information at different depths, and the team could therefore eventually reconstruct the profile of each crystalline phase along the depth from the surface. It was found that phase transformations proceeded during damage caused by penetration of sulfates. For more information, see the paper, "Deciphering the Sulfate Attack of Cementitious Materials by High-Resolution Micro-X-ray Diffraction", M. C. Schlegel *et al*., Anal. Chem., **83,** 3744 (2011).

Professional

Recovery commissioning at the Japanese synchrotron, The Photon Factory (May 31, 2011)

As reported in the previous news article, "Influence of the M9 class earthquake on synchrotron facilities in Japan", No.3, Vol. 40 (2011)), the Photon Factory, located to the north of Tsukuba city in Ibaraki prefecture, had to cancel all beamtime allocated in the term from May to September 2011. However, scientists have devoted a great deal of time and effort to recovery work, and on May 16, the ring became capable of storing electron beams, and generating synchrotron radiation. Recovery commissioning at each beamline started in the 4th week of May. Many users are involved in test experiments with their own samples. Some readers may be interested in the status of BL-4A, which is the beamline for X-ray fluorescence spectroscopic analysis. Recovery at the beamline appears more or less complete. Some data taken on March 10, one day before the earthquake, were reproduced almost perfectly. Commissioning will continue until early July. For further information, visit the Web page, http://www.kek.jp/ja/news/highlights/2011/PF_recovery.html (only in Japanese language).

Collaboration of DESY, XFEL and Brazilian LNLS (May 5, 2011)

In the presence of German President Christian Wulff and Brazilian President Dilma Rousseff, the three directors of DESY, the European XFEL, and LNLS have signed a cooperation agreement in Brasilia. For further information, visit the Web page, http://www.xfel.eu/news/2011/brasilian_german_agreement/

9th Ewald Prize - E. Dodson, C. Giacovazzo and G.M. Sheldric (April 26, 2011)

The International Union of Crystallography (IUCr) has announced that Professor E. Dodson (Department of Chemistry, University of York, UK), Professor C. Giacovazzo (Institute of Crystallography-CNR, Bari, Italy) and Professor G.M. Sheldrick (Lehrstuhl fur Strukturchemie, Gottingen, Germany) have been awarded the ninth Ewald Prize for the enormous impact they have made on structural crystallography by designing new methods and providing these in algorithms and constantly maintained, renewed and extended user software. Their invaluable contributions to the computational side of the field have led to leadership with the program suites CCP4, SIR and SHELX, respectively. The presentation of the prize will be made during the Madrid Congress Opening Ceremony in August 2011. The Ewald Prize consists of a medal, a certificate and an award of USD 30,000. Former recipients are D. Sayre (USA, 2008), P. Coppens (USA, 2005), M. M. Woolfson (UK, 2002), G. N. Ramachandran (India, 1999), M. G. Rossmann (USA, 1996), N. Kato (Japan, 1993), B. K. Vainshtein (Russia, 1990), J. M. Cowley (USA) and A. F. Moodie (Australia) in 1987.

Japanese X-ray free electron laser (March 29, 2011)

RIKEN and the Japan Synchrotron Radiation Research Institute (JASRI) have announced the start-up of the X-ray Free Electron Laser (XFEL) facility in Harima, named "SACLA" (SPring-8 Angstrom Compact Free Electron Laser). For further information, visit the Web page, http://xfel.riken.jp/eng/index.html

New Products

PANalytical's extension of XRD machine for SAXS application (April 26, 2011)

PANalytical has released new hardware and software for small-angle X-ray scattering (SAXS) measurements on their range of X-ray diffraction (XRD) systems. For existing users, the SAXS capability is a straightforward addition to the instrument. With the release of software, EasySAXS v2.0, nanoparticle analysis could become easier. For further information, visit the web page, http://www.panalytical.com/

Corporate

Canon sells Xradia's 3D X-ray microscopy system (May 16, 2011)

Xradia, Inc., has announced a partnership agreement with Canon Marketing Japan, Inc., which will become the exclusive distributor of X-ray computer tomography system for industrial applications, research laboratories and synchrotron facilities throughout Japan. For further information, visit the web page, http://www.xradia.com/

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Kenji Sakurai
News Editor
sakurai@yuhgiri.nims.go.jp
or sakurai@pas.tsukuba.ac.jp

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News

Sciences

Absolute K-shell photoionization for gas-phase atomic nitrogen (July 11, 2011)

So far, because of the difficulty of creating a target of neutral atomic nitrogen, there have been no reports on the details of high-resolution K-edge spectra. Recently, scientists at Lawrence Berkeley National Laboratory have performed both experimental and theoretical studies on the strong $1s \rightarrow np$ resonance features throughout the threshold region. The absolute value of the K-shell binding energy was experimentally obtained for the first time, and it was 409.64 ± 0.02 eV. For more information, see the paper, "K-Shell X-Ray Spectroscopy of Atomic Nitrogen", M. M. Sant'Anna *et al.*, Phys. Rev. Lett. **107,** 033001 (2011).

Real-time analysis of martensitic phase transition of cobalt (June 30, 2011)

Professor K. F. Ludwig (Boston University, USA) and his colleagues have recently reported their real-time X-ray scattering studies on heterogeneous microscale dynamics in the martensitic phase transition of cobalt. During the transformation of the high-temperature fcc phase to the low-temperature hcp phase, first, a rapid local transformation happens, and then, strains are relaxed slowly. The research group employed coherent X-ray scattering measurements to see the latter part of the transformation. It was found that the kinetics is dominated by discontinuous sudden changes - avalanches. The spatial size of observed avalanches varies widely, from 100 nm to 10 μm, the size of the X-ray beam. For more information, see the paper, "Direct Measurement of Microstructural Avalanches during the Martensitic Transition of Cobalt Using Coherent X-Ray Scattering", C. Sanborn *et al.*, Phys. Rev. Lett. **107,** 015702 (2011).

3D micro analysis of the Dead Sea Scrolls (June 29, 2011)

The Dead Sea Scrolls are a collection of 972 texts from the Hebrew Bible and extra-biblical documents found between 1947 and 1956 at Khirbet Qumran on the northwest shore of the Dead Sea from which it derives its name, in the British Mandate for Palestine, in what is now named the West Bank. Recently, a research group led by Professor B. Kanngiesser (Technische Universität Berlin, Germany) has investigated the feasibility of merging two X-ray techniques, ordinary micro XRF and confocal 3D micro XRF for optimized analysis of highly inhomogeneous samples such as the Dead Sea Scrolls. Ordinary micro XRF lacks information on the depth, but the measurement is efficient and rather quick. On the other hand, confocal 3D micro XRF has depth resolution, but the measurement takes very long. The authors found that the reliability of the analysis of highly heterogeneous samples can be improved by quantitatively combining both data. For more information, see the paper, "3D Micro-XRF for Cultural Heritage Objects: New Analysis Strategies for the Investigation of the Dead Sea Scrolls", I. Mantouvalou *et al.*, Anal. Chem., Article ASAP (DOI: 10.1021/ac2011262 Publication Date (Web): June 29, 2011).

3D micro X-ray spectroscopic analysis of natural diamond formed in ultra-deep underground (June 27, 2011)

A research group led by Professor L. Vincze (Ghent University, Belgium) has recently reported the interesting analysis of 1–20 μm sized inclusions in natural diamond crystals from Rio Soriso (Juina area, Mato Grosso State, Brazil). The crystals are called ultra-deep diamond, because they were formed in the astenospheric upper mantle, the transition zone (410–670 km), and even the lower mantle (>670 km) of the Earth. The experiment is basically 3D imaging by confocal X-ray fluorescence suing synchrotron radiation. By scanning X-ray energy near the Mn and Fe K absorption edges, the authors obtained chemical information on the inclusion cloud in the crystal. It was found that the observed Fe-rich inclusions were ferropericlase (Fe,Mg)O, hematite and a mixture of these two minerals. Another finding was that significant overprint of inclusions along pre-existing planar features is possible without changing their outer shape. For more information, see the paper, "Three-Dimensional Fe Speciation of an Inclusion Cloud within an Ultradeep Diamond by Confocal μ-X-ray Absorption Near Edge Structure: Evidence for Late Stage Overprint", G. Silversmit *et al.*, Anal. Chem., Article ASAP (DOI: 10.1021/ac201073s Publication Date (Web): June 27, 2011).

Resolution enhancement in ptychographic X-ray diffraction microscopy (June 13, 2011)

Ptychographic X-ray diffraction microscopy is known as an extension of so-called X-ray diffraction microscopy, which is a lensless X-ray imaging technique based on coherent diffraction measurements and iterative phasing methods. The technique employs sample scanning to see a large viewing area, but so far, the spatial resolution has been rather limited mainly because of positioning errors due to the drift between the sample and illumination optics. Recently, Professor Y. Takahashi (Osaka University, Japan) and his colleagues have published an experimental way to resolve the problem. The research group has developed a method of correcting positioning errors, and made it possible to illuminate a highly focused hard X-ray beam at the exact position on the samples. The spatial resolution achieved is as good as 10 nm or even better in a viewing area of larger than 5 μm. For more information, see the paper, "Towards high-resolution ptychographic X-ray diffraction microscopy", Y. Takahashi *et al.*, Phys. Rev. **B83,** 214109 (2011).

Theory of Kα emission efficiency under the irradiation of ultra short pulse laser (June 6, 2011)

When laser light hits thin solid foil, one can obtain soft X-rays, and this is sometimes called a laser plasma X-ray source. When the peak power of the laser becomes extremely high by shortening the pulse duration, it is also possible to observe hard X-ray spectra including Kα and Kβ emission. A team at Sandia National Laboratory has recently reported some calculations on the efficiency of Kα emission. The conversion efficiency of laser energy into Kα X-ray energy is clearly a critical parameter for designing an X-ray source. Basically the value is fairly small, but the team's simulations indicate that an enhancement of efficiency greater than tenfold over conventional single targets may be possible by introducing a two-phase target concept. For more information, see the paper, "Efficiency Enhancement for Kα X-Ray Yields from Laser-Driven Relativistic Electrons in Solids", A. B. Sefkow *et al.*, Phys. Rev. Lett. **106,** 235002 (2011).

Quantitative chemical imaging of trace elements using advanced synchrotron X-rays (May 31, 2011)

A Swiss group has recently published many interesting chemical images of trace elements in heterogeneous media. The authors combined several techniques; laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS), synchrotron radiation based micro-X-ray fluorescence and extended X-ray absorption fine structure spectroscopy. The analysis was done for Opalinus clay, which has been proposed as the host rock for high-level radioactive waste repositories. 2D images were shown for the matrix elements Ca, Fe, and Ti, as well as for the trace element, Cs. The synchrotron experiments were performed at Sector 20 (PNC-CAT), Advanced Photon Source (APS), and microXAS beamline at the Swiss Light Source (SLS). The beam size was $4 \times 3\ \mu m^2$ and $3 \times 3\ \mu m^2$, respectively. For more information, see the paper, "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", H. A. O. Wang *et al.*, Anal.

Chem., Article ASAP (DOI: 10.1021/ac200899x Publication Date (Web): May 31, 2011).

Tissue imaging with X-ray excited optical luminescence (May 27, 2011)

Professor J. N. Anker (Clemson University, South Carolina, United States) and his colleagues have recently reported an interesting application of optical luminescence excited by X-rays. So far, the spatial resolution of conventional florescence microscopy for tissue has been fairly limited. This is mainly due to the spread of the excitation light, which is scattered by the sample itself, particularly in the case of thick tissue. The novel idea is to use X-ray excited optical luminescent light from the scintillator plate placed at the back of the tissue. X-rays are not scattered very much even in thick tissue, and such a small spread leads to high-resolution chemical imaging of the tissue. The authors demonstrated an interesting application as a pH imager using methyl-red dyed paper. For more information, see the paper, ''High-Resolution Chemical Imaging through Tissue with an X-ray Scintillator Sensor'', H. Chen *et al.*, Anal. Chem., **83**, 5045 (2011).

Pump-probe X-ray reflectivity (March 10, 2011)

A research team led by Professor J. Larsson (Lund University, Sweden) has recently performed time-resolved X-ray reflectivity measurements with 100 picosecond resolution at ID09B, at the European Synchrotron Radiation Facility (ESRF). The experiment is a so-called pump-probe measurement, i.e., the repetition of the measurement with systematic change of the delay time of the pump (laser light) and probe (X-ray) pulses. In their research, amorphous carbon films with a thickness of 46 nm were excited with laser pulses (100 fs duration, 800 nm wavelength, and 70 mJ/cm^2 fluence). Here, the laser-induced stress caused a rapid expansion of the thin film followed by a relaxation of the film thickness as heat diffused into the silicon substrate. The researchers succeeded in measuring changes in film thickness by X-ray reflectivity with a short X-ray pulse (100 ps duration). It was observed that thermal stress generated by laser excitation causes the film to rapidly expand and increases the surface roughness substantially. The subsequent relaxation of film thickness is governed by heat diffusion into the substrate. For more information, see the paper, ''Picosecond time-resolved x-ray reflectivity of a laser-heated amorphous carbon film'', R. Nuske *et al.*, Appl. Phys. Lett. 98, 101909 (2011).

Professional

Ultra fast X-ray camera designed for European XFEL (July 27, 2011)

An innovative X-ray camera, designed to record bursts of images at an unprecedented speed of 4.5 million frames per second, is being built with the help of the UK's Science and Technology Facilities Council (STFC) and will be delivered to the European XFEL (X-ray Free-Electron Laser) in 2012. For further information, visit the Web page, http://www.stfc.ac.uk/About%20STFC/36221.aspx

Design improvement at European XFEL (June 30, 2011)

The European XFEL under construction at Hamburg in Germany aims to have the first beam ready in 2015. Very recently, it was found that the design parameters can be further improved. The first revision is to the energy range. This will now be 260 eV - 25 keV, while the 2006 design was 800 eV – 12.4 keV. The second is X-ray pulse duration. This will become of variable duration from a few femtoseconds (fs) to about 100 fs, instead of about 100 fs only. Such upgrades will be realized by improving electron beam quality by building on the experience with the X-ray free-electron laser at Stanford. For further information, visit the Web page, http://www.xfel.eu/news/2011/x_ray_flashes_revised/

New Products

JEOL's new large angle SDD for TEM samples (July 7, 2011)

JEOL has developed a new generation of energy-dispersive X-ray spectrometer, Centurio, for the element analysis and mapping of electron microscope samples. The detector is a silicon drift detector that collects X-rays from samples at an unprecedented large solid angle of up to 0.98 steradians from a detection area of 100 mm^2. For further information, visit the web page, http://www.jeolusa.com/

Amptek's new detector for low energy X-ray spectra (March 7, 2011)

Amptek Inc has introduced a new series of low energy X-ray windows. The new ''C Series'' represents a new technology that extends the low energy response of the Amptek Super SDD down to carbon. These windows can be used in SEMs, benchtops, or hand-held analyzers. For further information, visit the web page, http://www.amptek.com/press.html

Corporate

GE moves X-ray business to China (July 25, 2011)

General Electric Healthcare has announced the transition of the 115-year-old X-ray business from Waukesha, Wisconsin to Beijing, China. This is the first GE business to be headquartered there. For further information, visit the web page, http://www.genewscenter.com/Press-Releases/GE-Healthcare-Global-X-ray-Business-Announces-Leadership-Move-to-China-324f.aspx

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Kenji Sakurai
News Editor
sakurai@yuhgiri.nims.go.jp
or sakurai@pas.tsukuba.ac.jp

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News

Sciences

Evaporation of water by extremely strong X-rays (26 September 2011)

A Korean group led by Professor J. H. Je (Pohang University of Science and Technology) has recently reported some interesting experiments on water vaporization by X-ray photons. The experiments were performed at the undulator beamline, XSD 32-ID, Advanced Photon Source in Argonne, USA. It was found that water is vaporized at a rate of 5.5 pl/s at a 100 ms exposure of 0.1-nm-wavelength (~13 keV) X-ray irradiation of around 10^7 photons/μm^2 (10 photons/nm^2), which corresponds to a dose rate of ~50 kGy/s. They also confirmed that water vapor is reversibly condensed during pauses in irradiation. This result suggests that photoionization induces vaporization through the reduction of the surface tension of water. For more information, see the paper, 'X-ray-induced water vaporization', B. M. Weon *et al.*, *Phys. Rev.* **E84**, 032601 (2011).

X-ray emission spectroscopy clarifies local magnetism of iron-based superconductors (22 September 2011)

Professor Young-June Kim (University of Toronto, Canada) and his colleagues have recently reported an Fe Kβ X-ray emission spectroscopy study on iron-based superconductors, such as PrFeAsO, $Ba(Fe,Co)_2As_2$, LiFeAs, $Fe_{1+x}(Te,Se)$, and $A_2Fe_4Se_5$ (where A = K, Rb, and Cs). They found that the materials possess local magnetic moments even in their paramagnetic phases. By analyzing Fe $K\beta_{1,3}$ and Kβ′ spectra with the use of the integrated absolute difference method, the local moment size for each sample was determined. It was found that the value is independent of temperature or carrier concentration but varies significantly across different families. Specifically, all iron pnictide samples have local moments of about $1\mu_B$/Fe, whereas FeTe and $K_2Fe_4Se_5$ families have much larger local moments of ~$2\mu_B$/Fe and ~$3.3\mu_B$/Fe, respectively. Such differences point to the importance of considering the contribution of multi-orbital physics in describing magnetism of these compounds. For more information, see the paper, 'Revealing the dual nature of magnetism in iron pnictides and iron chalcogenides using X-ray emission spectroscopy', H. Gretarsson *et al.*, *Phys. Rev.* **B84**, 100509(R) (2011).

X-ray photon correlation analysis of martensitic transformation (1 September 2011)

A German group led by Professor U. Klemradt (Aachen University) has recently performed an X-ray photon correlation spectroscopy (XPCS) experiment on martensitic transformation of a $Au_{50.5}Cd_{49.5}$ single crystal. XPCS experiments basically consist of the observation of a time-dependent speckle pattern caused by scattering of coherent X-ray photons, and give information on the dynamics of phase transformations in soft and hard condensed matter at atomic length scales. The measurement was carried out at ID 10A, European Synchrotron Radiation Facility (ESRF) in Grenoble, France. A standard Bragg scattering geometry was employed to see the fluctuations of the symmetric (0 0 1) Bragg reflection from the polished surface of the Au–Cd single crystal. The research team observed slow non-equilibrium-dynamics in a narrow temperature interval in the direct vicinity of the otherwise athermal phase transformation. For more information, see the paper, 'Slow aging dynamics and avalanches in a gold–cadmium alloy investigated by X-ray photon correlation spectroscopy', L. Muller *et al.*, *Phys. Rev. Lett.* **107**, 105701 (2011).

Application of micro X-ray diffraction mapping to the exposure of counterfeit drugs (30 August 2011)

Professor M. D. Ward (New York University, USA) and his colleagues have recently proposed an interesting and effective application of the micro X-ray diffraction technique to anticounterfeit protection of pharmaceutical products. Counterfeit drugs have been a global threat to public health, and they undermine the credibility and the financial success of the producers of genuine products. There have been great demands for some good methods for rapid and nondestructive screening of the products. The research team's idea is the use of barcodes and logos fabricated on drug tablets using soft-lithography stamping of compounds that can be read by X-ray diffraction mapping but are invisible to the naked eye or optical microscopy. The materials used were suspensions of rutile powder mixed with corn syrup in a 1:2.5 (w/w) ratio or zinc oxide powder mixed with corn syrup at a 1 : 10 (w/w) ratio. It was demonstrated that the technique is feasible for realistic screening, because of its nondestructive, automated, and user-friendly properties. For more information, see the paper, 'Anticounterfeit protection of pharmaceutical products with spatial mapping of X-ray-detectable barcodes and logos', D. Musumeci *et al.*, *Anal. Chem.*, Articles ASAP (DOI: 10.1021/ac201570r Publication Date (Web): 30 August 2011).

Fine structures in Kβ spectra of chromium (26 August 2011)

A research team led by Professor I. Tsuyumoto (Kanazawa Institute of Technology, Japan) has recently studied chromium Kβ spectra and found that the intensity of Kβ″ satellite, which is observed at the higher energy side of the main $K\beta_{1,3}$ peak, is strongly correlated with the pre-edge peak of the X-ray absorption near edge structure specific for chromium (VI) compounds, such as CrO_3, $Na_2CrO_7\ 2H_2O$, $Na_2CrO_4\ 4H_2O$, $K_2Cr_2O_7$, K_2CrO_4, $Zn_2CrO_4(OH)_2\ 2H_2O$, $PbCrO_4$, and $BaCrO_4$. For more information, see the paper, 'X-ray Fluorescence Analysis of Hexavalent Chromium Using Kβ Satellite Peak Observed as Counterpart of X-ray Absorption Near-Edge Structure Pre-Edge Peak', I. Tsuyumoto *et al.*, *Anal. Chem.*, Articles ASAP (DOI: 10.1021/ac201606c Publication Date (Web): 26 August 2011).

X-ray tomography reveals how Chinese jawless fish evolved (18 August 2011)

Most living vertebrates are jawed vertebrates (gnathostomes), and only scarce information on the evolutionary origin of jaws is available from living jawless vertebrates (cyclostomes), hagfishes, and lampreys. The extinct bony jawless vertebrates, or 'ostracoderms', have been regarded as precursors of jawed vertebrates and provide an insight into this formative episode in vertebrate evolution. Very recently, Chinese scientists employed synchrotron radiation X-ray tomography in an effort to analyze the cranial anatomy of galeaspids, a 370–435-million-year-old 'ostracoderm' group from China and Vietnam. For more information, see the paper, 'Fossil jawless fish from China foreshadows early jawed vertebrate anatomy', Z. Gai *et al*., *Nature* **476**, 324 (2011).

Quantitative X-ray reflectivity analysis of growing thin films (15 August 2011)

Professor J. R. Engstrom (Cornell University) and his colleagues have recently published a detailed comparative study on surface morphology obtained from *in situ*, time-resolved X-ray reflectivity, which is extremely feasible as a tool for investigating surface and interfaces during thin film growth, but requires some modeling of the growth process for the interpretation. The research group prepared two sets of organic thin films (pentacene/SiO_2 and diindenoperylene SiO_2) for each system, giving a total of four films, grown to different thicknesses, under nominally identical conditions. The X-ray reflectivity data were analyzed on the basis of three different models, and the obtained parameters were directly compared with atomic force microscope data. It was found that all models employed can give good agreement between the surface morphology obtained from fits with the actual morphology at early times. On the other hand, this agreement deteriorates at later times, once the root-mean squared film roughness exceeds about one monolayer. It was also found that the best fits to reflectivity data, corresponding to the lowest values of χ^2, do not necessarily yield the best agreement between simulated and measured surface morphologies, simply because the model reproduces all local extrema in the data. For more information, see the paper, 'Quantitative modeling of *in situ* X-ray reflectivity during organic molecule thin film growth', A. R. Woll *et al*., *Phys. Rev*. **B84**, 075479 (2011).

Extension of coherent X-ray diffractive imaging (12 August 2011)

A research team led by Professors V. Holý (Charles University, Czech Republic) and T. Baumbach (ANKA—Institute for Synchrotron radiation, Germany) have recently performed some extension of coherent X-ray diffractive imaging for high-resolution strain analysis in crystalline nanostructured devices such as layered nanowires and/or dots. Their research successfully determined the strain distribution in (Ga,Mn)As/GaAs nanowires. The key was their improvement of the phase-retrieval algorithm, i.e. separation of diffraction signals in reciprocal spaces. It was found that individual parts of the device can be reconstructed independently by this inversion procedure. The method is effective even for strongly inhomogeneously strained objects. For more information, see the paper, 'Selective coherent X-ray diffractive imaging of displacement fields in (Ga,Mn)As/GaAs periodic wires', A. A. Minkevich *et al*., *Phys. Rev*. **B84**, 054113 (2011).

Quantitative analysis of photoreduction process in Cu metalloproteins (1 August 2011)

X-ray spectroscopy is an extremely strong tool for metal speciation at the molecular level in biological and environmental samples, especially for metalloproteins. When samples are quite easily influenced by photoreduction, however, analysis has not been straightforward. Recently, a Chinese group has studied, in detail, soft X-ray induced photoreduction in organic Cu(II) compounds. The research team measured XANES spectra at Cu-LIII, O–K, and C–K edges to see how the valence state of Cu changes. A scanning transmission X-ray microscopy was also employed to look at specific radiation damages. It was found that reducing the radiation dose to 0.1 MGy effectively prevented the photoreduction of organic Cu(II) compounds. For more information, see the paper, 'Soft X-ray induced photoreduction of organic Cu(II) compounds probed by X-ray absorption near-edge (XANES) spectroscopy', J. Yang *et al*., *Anal. Chem*., Article ASAP (DOI: 10.1021/ac201622g Publication Date (Web): 1 August 2011).

X-ray parametric down-conversion for atom-scale resolution in the extreme-ultraviolet region (17 July 2011)

Parametric down-conversion is a quantum-optical process in which a 'pump' photon splits spontaneously into two (the 'signal' and 'idler') in a nonlinear optical medium. Recently, Professor T. Ishikawa (RIKEN, Harima, Japan) and his colleagues reported their experiments with X-ray photons. They have visualized three-dimensionally the local optical response of diamond at wavelengths between 103 and 206 Å with a resolution as fine as 0.54 Å. This corresponds, to a resolution from λ/190 to λ/380, an order of magnitude that is the best ever achieved. For more information, see the paper, 'Visualizing the local optical response to extreme-ultraviolet radiation with a resolution of λ/380', K. Tamasaku *et al*., *Nature Physics* **7**, 705 (2011).

Professional

Memorandum of Understanding between the European Molecular Biology Laboratory and the European X-ray Free Electron Laser (13 September 2011)

The European Molecular Biology Laboratory and the European X-ray Free Electron Laser have signed a memorandum of understanding, thereby laying the foundation for close future collaboration in deciphering the structure and dynamics of biomolecules. For further information, visit the Web page, http://www.xfel.eu/.

Denver X-ray conference awards (3 August 2010)

The following awards were presented during the plenary session of the 60th Annual Denver X-Ray Conference: The 2011 Barrett Award was presented to Dr. Juan Rodriques-Carvajal, Institute Laue-Langevin, Grenoble, France, to honor his exceptional contributions to the field of X-ray diffraction, in particular for his work on characterization of the structural and magnetic properties of strongly correlated oxides using diffraction techniques and for writing and freely disseminating FULLPROF, the most widely used Rietveld refinement program for analysis of crystallographic and magnetic structures. The 2011 Jenkins Award was given to Dr. Paul K. Predecki to honor his contributions to the development of X-ray methods for a wide variety of materials and to honor his generosity in teaching and inspiring others in X-ray

materials analysis both at the University of Denver and through organization and management of the Denver X-ray Conference. The 2011 Jerome B. Cohen Student Award was given to Vallerie Ann Innis-Samson, University of Tsukuba, Ibaraki, Japan, for her work, 'X-ray Reflection Tomography: A New Tool for Surface Imaging'. For further information, visit the Web page, http://www.dxcicdd.com/.

New products

Vacuum system for open X-ray tube (22 September 2011)

X-RAY WorX GmbH has introduced electronically controlled venting valves for open X-ray tubes. This avoids the manual venting typically performed during X-ray tool maintenance. The new venting method considers the turbo pump's rotation speed and automatically optimizes tube venting. For further information, visit the Web page, http://www.x-ray-worx.com/.

SII's new X-ray particle inspection system for battery applications (30 August 2011)

SII Nano Technology Inc. SIINT) has announced that it has recently developed an X-ray inspection system that enables detection of any metal particles of about 20 μm diameter contained in fuel cell electrodes and lithium ion rechargeable battery electrodes within a few minutes, as well as carrying out automatic elemental analysis. For further information, visit the Web page, http://www.siint.com/en/.

Corporate

FUJIFILM's new subsidiary in Vietnam (21 September 2011)

FUJIFILM Corporation has announced that it has established FUJIFILM Medical Systems Vietnam Co., Ltd., in Ho Chi Minh City, Socialist Republic of Vietnam to further expand its business in the fast-growing economy of Vietnam. The business operation will launch in October 2011. For further information, visit the Web page, http://www.fujifilm.com/.

PANalytical expands with X-ray tube factory in Eindhoven, The Netherlands (15 September 2011)

PANalytical has relocated its X-ray tube manufacturing to a new factory in Eindhoven, The Netherlands. For further information, visit the Web page, http://www.panalytical.com/.

Bruker acquires Center for Tribology (12 September 2011)

Bruker Corporation has announced the signing of a purchase agreement to acquire Center for Tribology, Inc. (CETR), which is a privately held corporation located in Silicon Valley in Campbell, CA, USA. Bruker intends to continue to operate CETR in Silicon Valley and to integrate its business management, operations, research and development, sales and support with the current atomic force microscope and stylus and optical metrology businesses in the Bruker Nano Surfaces division. For further information, visit the Web page, http://www.bruker.com/.

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Kenji Sakurai
News Editor
sakurai@yuhgiri.nims.go.jp
or sakurai@pas.tsukuba.ac.jp