Three-dimensional iron mapping of cosmic dust samples using subtraction microtomography.

Akira Tsuchiyama[1], Kentaro Nakamura[1], Kentaro Uesugi[2], Tsukasa Nakano[3], Takaaki Noguchi[4], Keiko Nakamura[5], Tomoki Nakamura[6], and Yoshio Suzuki[2]

1) Department of Earth and Space Science, Osaka University, 1-1 Machikaneyama-cho, Toyonaka 560-0043, JAPAN, akira@ess.sci.osaka-u.ac.jp. 2) JASRI/SPring-8, Mikazuki, 679-5198 JAPAN. 3) AIST/GSJ, Tshukuba, 305-8567 JAPAN. 4) Depart. Material and Biological Sci., Ibaraki University, Mito 310-8512, JAPAN. 5) Graduate School of Science, Kyushu University, Fukuoka, 812-8151, JAPAN. 6) NASA/JSC, Houston, TX, 77058, USA.

We have performed 3-D Fe mapping of cosmic dust nondestructively using a SR projection microtomography system at BL47XU of SPring-8. Extraterrestrial materials of < 1 mm are called cosmic dust in contrast to meteorites (> 1 mm). As they have a wider variety of origin than primitive meteorites, specific information that cannot be obtained from meteorites alone could be extracted from cosmic dust.

Distribution of a specific element can be obtained by X-ray CT using two energies just above and below the absorption edge of the element. This subtraction method has been applied to mapping of heavy elements. Lately, high-resolution quantitative 3-D Cs mapping was obtained for a Cs-doped molten granite sample (this volume and [1]). Fe and Ni are naturally abundant and heavy elements. However, their edge energies are so low that the method has not been applied to natural samples.

In the present study, using cosmic dust of <100 micron combined with microtomography of the resolution of <1 micron gave Fe mapping of the natural samples successfully. One standard sample (olivine) with known Fe content and density and three cosmic dust samples were imaged at X-ray energies just above and below the Fe K-edge (7.124 and 7.098 keV, respectively) with the pixel size of 0.2 micron. To minimize the effect of harmonics a monochrometer was detuned and a helium path for reducing the decay of ~7keV X-ray beams by air was used. Comparison of linear attenuation coefficients (LACs) of some standard materials with their theoretical LACs shows that the effect of harmonics was negligibly small. Two sets of CT images at the different X-ray energies were slightly shifted each other three-dimensionally even by a small shift of X-ray path. The shift was adjusted using an appropriate algorithm before obtaining subtraction images. 3-D Fe concentration map was obtained for the olivine sample directly from the subtraction images. The average Fe concentration in the Fe map was almost the same as the real concentration. 3-D Fe concentration maps of the cosmic dust samples were obtained by assuming a density – Fe content relation for common minerals in cosmic dust. The Fe contents of Fe-rich regions in the Fe maps are generally smaller than those analyzed by an electron probe micro analyzer. The discrepancy is probably due to the effect of Fe fluorescent X-ray generated above the Fe edge energy.

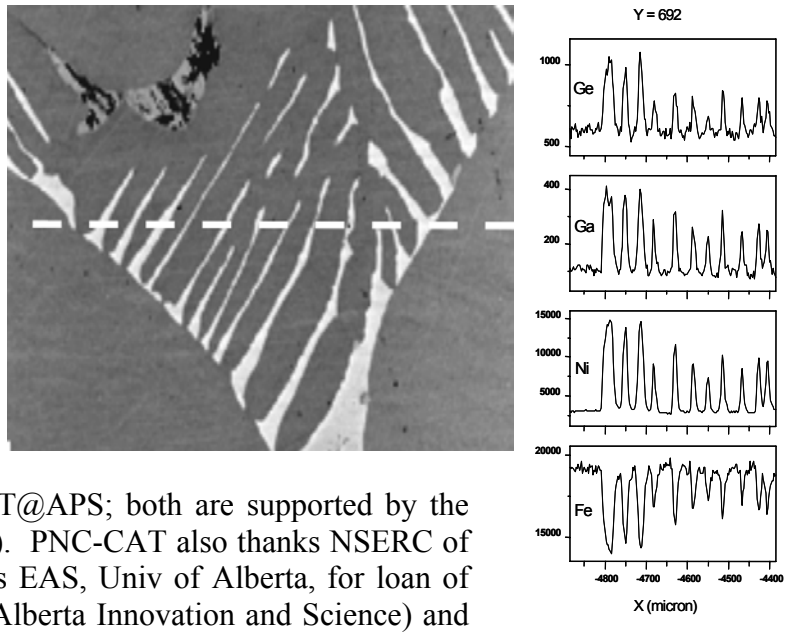
Reference: [1] Ikeda et al. (2004) Am. Mineral., 89, 1304-1313.

Hard X-ray Spectromicroscopy for Chemical and Structural Analysis of Selected Meteorites: Challenging Inhomogeneous Materials.

Ronald G. Cavell[1,2], Renfei Feng[2], M. Adam Webb[2], Alistair McCready[2], Christopher Herd[3] and Robert A. Gordon[4]

[1]Dept of Chemistry, Univ of Alberta, Edmonton, AB, Canada, T6G 2G2
[2]Alberta Synchrotron Institute (ASI), Univ of Alberta, Edmonton, AB, Canada, T6G 2E1
[3]Dept of Earth and Atmos. Sciences, Univ of Alberta, Edmonton, AB, T6G 2E3,
[4]Pacific Northwest Consortium (PNC-CAT at APS) and Dept of Physics, Simon Fraser University, Burnaby, BC, Canada, V5A 1S6

A focussed, micrometer dimensioned, hard X-ray beam is used to generate a two-dimensional map of the distribution of the elements present in typically inhomogeneous meteoritic samples including even those elements present in trace concentrations at (<100) ppm levels. These trace elements are used for the classification of the meteorites. Subsequent application of XANES and EXAFS to selected areas of interest provides confirmation of element identity, the chemical valence state and local structure information about the element. The inhomogeneous character of the samples can be illustrated by the image (Fig) of a sample with two intermixed Ni/Fe (either 30/70 or 7/93 ratios) phases. The area is approximately 450 micrometers square. The concentration profile across the sample (dash line) shows feature sizes of 10 to 50 micrometers. We also find (via EXAFS) that the trace elements (Ga and Ge) are components of the major phases and not grain boundary impurities.



[Data was obtained at PNC-CAT@APS; both are supported by the US Department of Energy (DoE). PNC-CAT also thanks NSERC of Canada for support. RGC thanks EAS, Univ of Alberta, for loan of meteorites and ASI (funded by Alberta Innovation and Science) and NSERC for support.]

Study of fault rocks by X-ray microscopy

Y. F. Song[1], S. R. Song[2], M.-T. Tang [1], G. C. Yin [1], and K. S. Liang[1]

[1] *National Synchrotron Radiation Research Center, 101 Hsin-Ann Rd., Science-Based Industrial Park, Hsinchu 300776, Taiwan, R. O. C.*
[2] *Department of Geosciences, National Taiwan University, P. O. Box 13-318, Taipei 106, Taiwan, R. O. C.*

We have installed a new transmission X-ray microscope at beamline BL01B of the Taiwan Light Source, National Synchrotron Radiation Research Center (NSRRC). The X-ray source is a superconducting wavelength shifter operated at 5T. The X-ray microscope has been shown to provide 2D imaging and 3D tomography at energy 8-11 keV with a spatial resolution of 30-60 nm, and is equipped with the Zernike-phase contrast capability for imaging light materials such as biological specimens. Employing this X-ray microscopy, we investigate the fault rocks from the cores of the Taiwan Chelungpu-fault Drilling Project (TCDP), which drilled in the fault zone of 1999 Chi-Chi earthquake. The characterization of particle size distribution, porosity and 3D structure of the fault rocks in transition from the fault core to damage zone are related to the comminuting and fluid behaviors and energy in the earthquake faulting[1,2,3]. The results may ascertain the implication of the nucleation, growth, transition, structure and permeability of the fault zones[4,5,6]. Furthermore, it may be possible to infer the mechanism of physical and chemical property of the fault, and the nucleation of the earthquake.

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Measurement of the strain for the InGaAs layers grown on the step structure using x-ray microdiffraction

K. Izumi[1)], T. Koyama[2)], Y. Tsusaka [2)], and Y. Kagoshima[2)]

[1)]*Fundamental and Environmental Research Laboratories, NEC Corporation*
[2)]*Graduate School of Material Science, University of Hyogo*

Metal-organic vapor phase epitaxial growth of InGaAs/InP system is widely used for fabricating photonic devices, because the multilayer thickness, composition, and lattice strain can be precisely controlled by only controlling flow rate of the source material gasses. In this work, we measured the position and the channel-width (Wc) dependence of the strain for the InGaAs (001) layers grown on the step structure of InP substrate(Fig. 1).

Experiment was carried out at the BL24XU in SPring-8 using a new high-resolution microdiffraction system [1]. The size of the focused beam is 0.32 μm vertically and 1.3 μm horizontally at a photon energy of 15 keV.

Figure 2 shows the position and channel-width dependence of the perpendicular strain determined from the peak shifts of the measured rocking curves. The positions shown in the Fig. 2 are the distances from the center of the step structure (Fig. 1). It can be seen that the strain changes largely at around the step position for large channel-width.

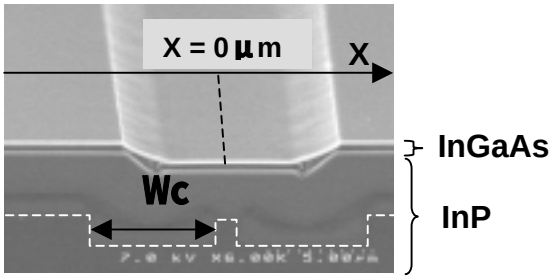

Fig.1 SEM image of sample

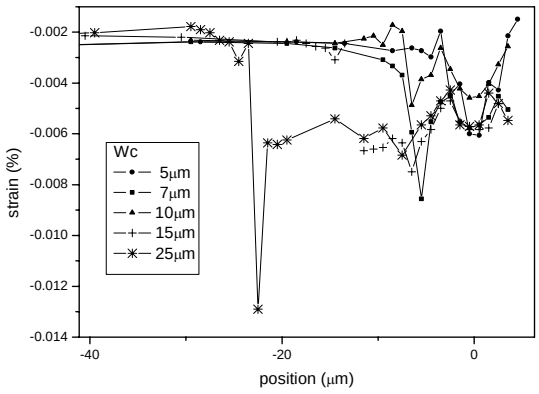

Fig.2 perpendicular strain of the InGaAs layer

[1] K. Izumi et al., SPring-8 User Experimental Report, No11, 320(2003A).

Mapping very similar chemical components in micron-scale organic rods by Scanning Transmission X-ray Microscopy

Noriyuki Iwata[1,2], Katsuhiko Tani[1], Atsuyuki Watada[1], *Hiromi Ikeura-Sekiguchi[3], Tohru Araki[4], Adam P. Hitchcock[4]

[1]*Ricoh Co Ltd., 16-1 Shinnei-cho, Tsuzuki-ku, Yokohama 224-0035, Japan*
[2]*Graduate School for Science, Chiba University, 1-33 Yayoi-cho, Inage-ku, Chiba 263-8522, Japan*
[3]*Reserch Institute of Instrumentation Frontier, AIST, Tsukuba, Ibaraki, 305-8568 Japan*
[4]*BIMR, McMaster University, Hamilton, ON Canada L8S 4M1*

Scanning transmission X-ray microscopy (STXM) is powerful tool for analysis of organic materials because it provides two- and three-dimensional [1] chemical component mapping based on the near edge X-ray absorption fine structure (NEXAFS) spectra of the components. It is very interesting to test the capability of STXM to distinguish very similar organic components since subtle differences are often used to control materials properties. This work reports a C 1s STXM study of micro organic rods constructed from two kinds of polyester (called polyester A and B). Their chemical structure is very similar, but the function of polyester A is to form the overall shape of the rod whereas polyester B is not. Quantitative information about the overall composition and internal spatial distributions of components is very helpful in process development.

Reference spectra of the pure components and C 1s image sequences of microtomed sections of a micro organic rod were measured using the STXM at beam line 5.3.2 [2] at the Advanced Light Source. The C 1s NEXAFS of polyester A and B (**Fig. 1**) are very similar; however small but reproducible differences between 288 and 291.5 eV provide sufficient contrast for mapping. **Fig. 2** shows an optical density image at 285 eV and quantitative maps of the two polyester components of one micro organic rod section. **Fig 1** compares the C 1s spectra extracted from regions of high A and B polyesters to validate the analysis. This study has shown that STXM can provide quantitative chemical component maps even for species with very similar NEXAFS spectra, such as these two polyesters.

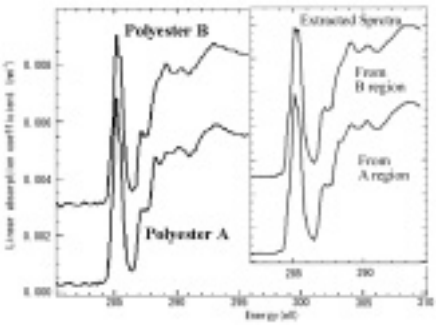

Fig 1 C 1s NEXAFS of two polyesters

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2) Kilcoyne, A.L.D. et.al.,J.Synchrotron Rad. **10** (2003) 125-136
3) Research supported by Ricoh Co. Ltd., NSERC, Canada Research Chair. The ALS is supported by US DoE under contract DE-AC03-76SF00098

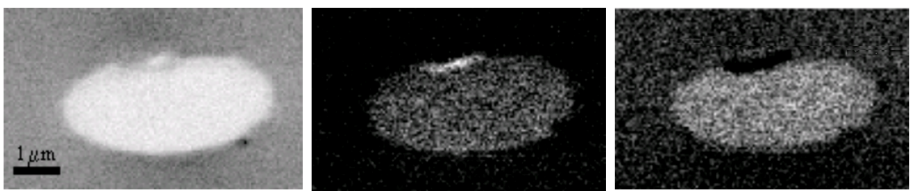


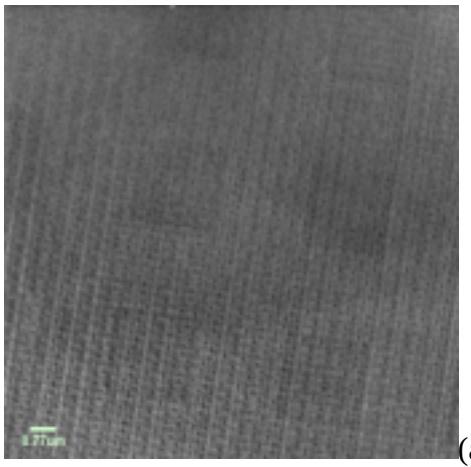
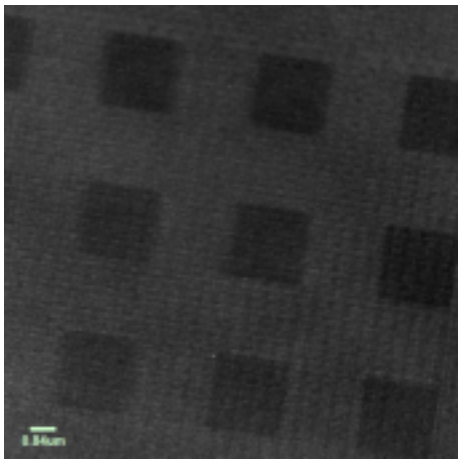
Fig. 2 (left) Optical density image at 285.1 eV. Component maps of polyester A (centre) and B (right)

An energy tunable hard x-ray microscope for IC investigation

Gung-Chian Yin[1,3], Yen-Feng Song[1], Mau-Tsu Tang[1],Fred Duewer[2], Wenbing Yun[2] and Keng S. Liang[1]

[1] National Synchrotron Radiation Research Center, 101 Hsin-Ann Road, Hsinchu 30077, Taiwan
[2] Xradia Inc, 4075A Sprig Dr, Concord, CA 94520, USA
[3] Department of Photonics, National Chiao Tung University, 1001, University Road, Hsinchu 30077, Taiwan

An energy tunable tomographic transmission hard X-ray microscope with spatial resolution better than 60nm was developed by NSRRC and Xradia Inc. The x-ray energy is from 8 keV to 11 keV provided by the superconducting wavelength shifter source at NSRRC. The microscope utilizes three different zone plates for different energies. The chosen energy range is well suited for most IC materials; Cu, Ta, W, Ga, As and Ge etc. With tunable energy microscopy, the materials of the IC sample can be identified with respect to different absorption edges as shown in Fig (a) and (b). These two pictures show Cu pads imaged at two different energies. The Cu contrast is enhanced when imaged by x-rays above the Cu absorption edge. Contrast enhancement of copper interconnects of 110 nm width is observed. Elemental distribution in three dimensions will be presented by comparing the tomography data at different energies.


(a)

(b)

Figure(a) Cu pad under 8kev (b) Cu pad under 9.1Kev

Chemical Analysis of Rust on Japanese Smoked Roof Tiles using Soft X-Ray Spectroscopy

Yasuji Muramatsu[1], Mika Hirose[2], Muneyuki Motoyama[3], Eric M. Gullikson[4], and Rupert C. C. Perera[4]

[1] *Department of Materials Science and Chemistry, University of Hyogo, 2167 Shosha, Himeji Hyogo 671-2201, Japan*
[2] *Matsuoka Roofing Inc., 2039-1 Funatsu-cho, Himeji, Hyogo 679-2101, Japan*
[3] *Center for Corporate Relations, University of Hyogo, 1-3-3 Higashikawasaki-cho, Chuo-ku, Kobe, Hyogo 650-0044, Japan*
[4] *Center for X-Ray Optics, Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, California 94720, USA*

We investigated the surface carbon films of Japanese smoked roof tile, "Ibushi-Kawara," by soft x-ray spectroscopy using synchrotron radiation to understand their properties from a chemical-bonding point of view and to improve quality control [1-3]. In this study, we analyzed the rust that rarely forms on the carbon films. Figure 1(a) shows the Kawara sample piece with a rusted portion. Soft x-ray absorption measurements that cover the C *K* to Fe *L* absorption edges and mapping measurements of the absorption peaks were performed at beamline BL-6.3.2 of the Advanced Light Source (ALS). In the mapping measurements, the spot size of the incident beams at the sample position measured 40 micron^{V} x 350 micron^{H}. By comparing the absorption spectra of the rusted Kawara with various iron oxides, the chemical formula of the rust is confirmed to be Fe_2O_3. Figure 1(b) shows the mapping spectrum measured by monitoring the Fe*L* absorption peak intensity. This shows that Fe_2O_3 gradually spreads from the upper point. Therefore, it is estimated that the rust forms by the oxidation of the iron in the basal sintered soil with water soaked through pinholes of the carbon films. Then the Fe_2O_3 can ooze to the surface through the pinholes.

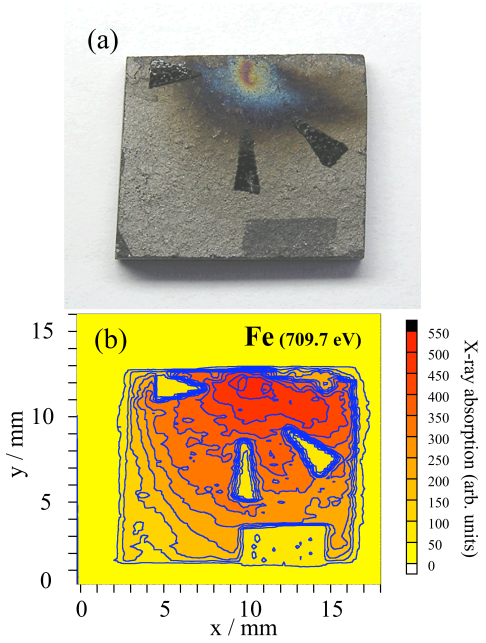


Fig. 1 Photograph of the rusted Kawara piece (a) and the mapping spectrum monitored Fe*L* absorption peak (b).

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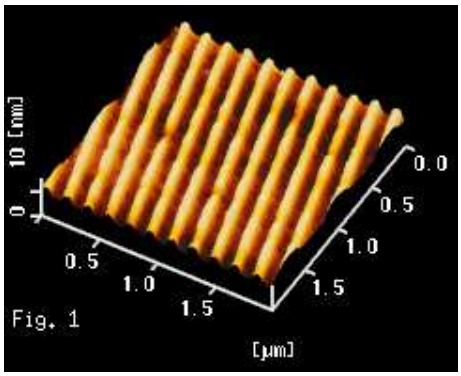
Direct Micromachining of Inorganic Transparent Materials Using Laser Plasma Soft X-Rays

Tetsuya Makimura[1], Hisao Miyamoto[1], Satoshi Uchida[1], Hiroyuki Niino[2], and Kouichi Murakami[1]

1) Institute of Applied Physics, University of Tsukuba, Ten'nodai 1-1-1, Tsukuba, Ibaraki 305-8573, Japan; 2) Photonics Research Institute, National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki 305-8565, Japan



Inorganic transparent materials are highly valued for their use in the fields of nanometric chemical analysis or chemical reactions in medicine and biotechnology, and for optical materials such as gratings, photonic crystals and optical waveguides. Although a limited number of materials can be machined, it is required to machine a wide variety of materials precisely at low cost. We have investigated direct micromachining of inorganic transparent materials using laser plasma soft X-rays. The soft X-rays were generated by irradiation of Ta targets with 532 nm Nd:YAG laser light with a pulse duration of 7 ns, at an energy density of $\sim 10^4$ J/cm. The soft X-rays were focused on specimens, using an ellipsoidal mirror that we desined so as to focus soft X-rays at around 10 nm efficiently. We found that synthetic quartz glass, fused silica, Pyrex, LiF, CaF_2, Al_2O_3, $LiNbO_3$ can be machined smoothly. Typically, quartz glass is ablated at 40 nm/shot, and has a surface roughness less than 10 nm after 10 shots. In order to investigate lateral resolution, we fabricated a WSi contact mask with 200-nm-pitch line-and-space patterns on quartz glass. Figure 1 shows an atomic force micrograph of the quartz glass plate after a single shot of laser plasma soft X-rays and etching the WSi mask. We found that quarts glass plates can be machined at a resolution less than 100 nm. With further development of imaging optics, nanomachining of inorganic trasparent materials should be achieved by direct irradiation of laser plasma soft X-rays.

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