

BL11 における軟 X 線 XAFS 測定

岡島敏浩, 大谷亮太

九州シンクロトロン光研究センター

2 から 4keV 程度のエネルギー領域にはリン (P, 2.1keV), イオウ (S, 2.5keV), 塩素 (Cl, 2.8keV), カリウム (K, 3.6keV) など, 生命科学や高分子材料といった材料科学にとって重要な元素の吸収端が存在する. このエネルギー領域の X 線は大気による吸収が大きく, 大気中での実験が困難であり, もっぱら真空下での測定がおこなわれている. これに加え, S において

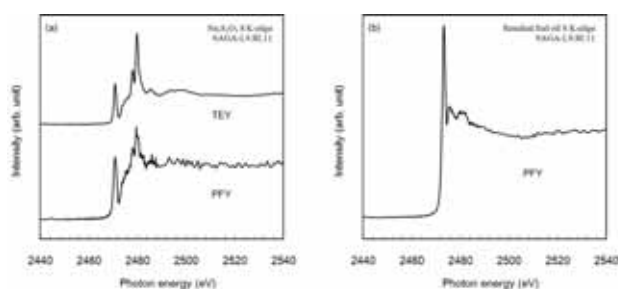


図 標準試料 ($\text{Na}_2\text{S}_2\text{O}_3$) (a) 及び重油 (b) から得られたイオウ K 吸収端領域 (S K-edge) の XANES スペクトル

は, その化学結合状態を決定するには, 一般に使われている FT-IR や NMR といった手法が使えず, X 線吸収端近傍構造 (XANES) 測定がほとんど唯一の手法である. 今回我々は, ビームライン最下流の X 線出射ポート以下, 試料測定チャンバーまでの光軸上のパス全てを大気圧の He ガスにより置換し, 大気による吸収を受けることなく試料まで X 線を導くシステムを開発した. さらに, 電子収量法, 蛍光収量法による同時計測を可能とし, これにより試料表面とバルクの状態を同時に計測することを可能とした. また, 大気圧の He ガスの導入により液体試料の測定も容易に行うことができるようにした. 図は本システムで測定した標準試料 ($\text{Na}_2\text{S}_2\text{O}_3$) と, 重油から得られた S K 吸収端での XANES スペクトルである. 重油試料は原液をポリエチレンの袋に流し込んだだけのものである.



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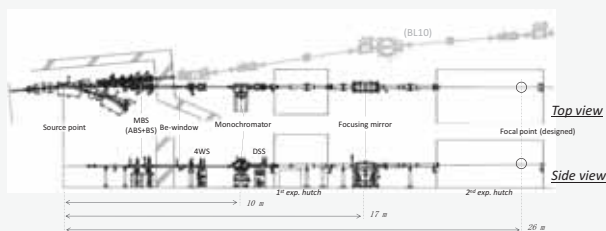
岡島敏浩, 大谷亮太

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New X-ray absorption fine structure (XAFS) measurement system under atmospheric pressure in soft X-ray region has been installed at the beamline BL11 of SAGA Light Source in Japan. XAFS spectrum at lower photon energy region around of 2 to 4 keV could be newly obtained at the beamline. The system was applied to obtain the sulfur (S) and phosphor (P) K-edge XAFS spectra using fluorescence yield and electron yield modes. The spectra was successfully collected. This new system would be anticipated a various application for environmental and biochemical research.

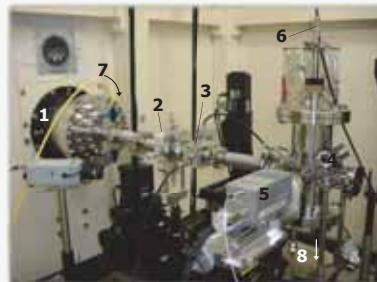
Instruments

Local Structure Analysis Beamline BL11



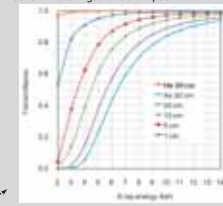
- Source: Bending magnet Critical energy: 1.9 keV, acceptance: 8 mrad (Max)
- Monochromator: Si(111) double crystal, energy range 2.1~23 keV
- Focusing mirror: Bent cylindrical, Rh-coated fused quartz, $R_{\text{sagittal}}=46.94$ mm
- Measurement methods: Transmission, Florescence Yield, Electron Yield
- Typical photon flux: 3×10^9 photons/sec @ 300 mA, $E_{\text{hv}}=7.2$ keV (not focusing)

New XAFS measurement system in BL11



- 1: BL11 beam duct
- 2: Slit chamber
- 3: I_0 ionization chamber
- 4: Measurement chamber
- 5: Silicon drift detector (SDD)
- 6: Sample current signal
- 7: He inlet
- 8: He outlet

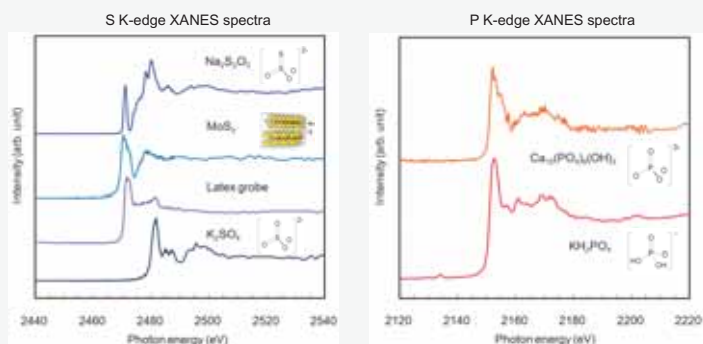
X-ray transmittance at 2 keV maintain over 98% in helium gas of 30 cm path.



- Whole beam pass is filled with He gas with 1 atm.
- The XAFS spectra can be obtained in,
 - Fluorescence yield (Partial Fluorescence Yield; PFY) by SDD.
 - Total Electron Yield (TEY) mode by sample drain current.
 - Conversion Electron Yield (CEY) mode by collection of ionized helium gas.

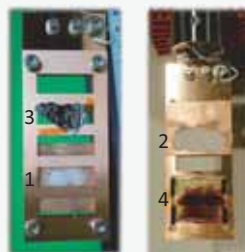
Results

XANES spectra of various materials (PFY mode)

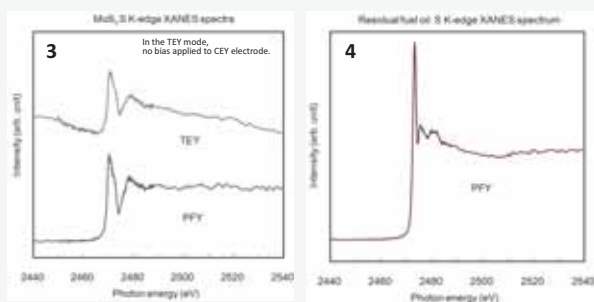


Comparison of XANES structure between chemical states of each sample can be clearly observed at lower photon energy region around of 2 keV.

Specimen preparation

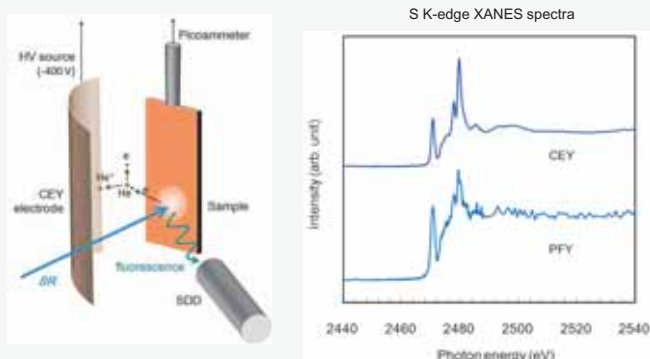


- Powder sample (1,2)
 - > Grind down into fine powder
 - > Suspend in ethanol
 - > Apply over copper plate
 - > Drying
- Foil, sheet, plate (3: MoS_2)
 - > Set onto sample holder by carbon tape or Kapton® tape
- Liquid sample (4: Residual fuel oil)
 - > Enclose into polyethylene bag



Various shape samples can be measured by this system. Contaminated, outgassing, and oily sample is available.

PFY/CEY simultaneous observation



PFY and CEY mode measurements can be performed at the same time. --> It is able to measure the bulk and surface state simultaneously.

Conclusion

We have achieved the XAFS measurements with soft X-ray region by using the new instruments constructed in SAGA-LS BL11. Sulfur and phosphor K-edge XANES spectra by means of TEY/PFY simultaneous measurements were successfully obtained under atmospheric pressure with helium gas. Positive achievements are expected for a various application in the environmental, biochemical research and numerous industrious productions.