R01A38B1-0027N BL38B1

Measurement of the copper ion concentration for the front end cooling water using a total-reflection X-ray fluorescence technique

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The cooling water for the front end components, which is pure water whose specific resistance is more than 4MΩ·cm, is very important to handle the intensive heat load by the synchrotron radiation. As two years ago we have encountered the matter that the copper oxide was separated out on the strainers of the cooling system, we have been monitoring the copper ion concentration periodically for the main cooling water in the system. Consequently, we have found there is a possibility that the front end components, made of copper, dissolved into the cooling water. In this experiment we examined whether there are dissoluble components or not by measuring the copper ion concentration for the branch cooling water passed through each components of BL29XU.

Samples were prepared from the absorber (ABS), the graphite filter (GFIL) and the XY slit. Each of samples were picked at the experimental mode, and the shut down modes after few minutes (①) and four days (②) from the end of the experimental mode. The sample from only the absorber was picked for both open and close status of the absorber at the experimental mode.

The experiment was carried out at BL38B1. We expected that the copper ion concentration was too low to measure the copper Kα X-ray for the liquid condition, because many photons scattered by the liquid sample were detected and these were the large background noise. In order to reduce these scattered photons, we dried all samples on the silicon wafers and used a total-reflection X-ray fluorescence technique. Using this technique, incident photons could be reflected to the forward direction. A SSD, located in the vertical direction against the incident photon, was used to detect the X-ray.

In order to determine the copper ion concentration, five kinds of standard solutions of copper were used. Figure 1 shows the relationship between the copper ion concentration and the intensity of the copper Kα X-ray for these standard solutions. The cooper ion concentrations of the cooling water were calculated by the fitted line in Fig.1. Table1 shows the results for the cooling water. From the table, the cooper ion concentrations was the highest for all components at the operation mode of shut down ②. Especially the difference between the experimental mode and the shut down ② in the case of absorber was remarkable.

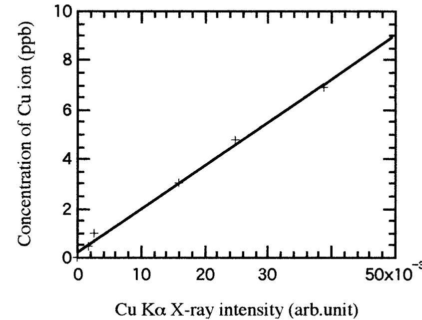

Fig.1 Copper ion concentration for the Kα X-ray intensity of standard solutions.

Sampling date	Location	Operation mode	MBS Status	Concentration(ppb)
6/1	ABS	Experiment	Open	3.5
6/1	ABS	Experiment	Close	2.9
6/1	ABS	Shut down①	Close	2.5
6/5	ABS	Shut down②	Close	9.1
6/1	GFIL	Experiment	Open	2.2
6/1	GFIL	Shut down①	Close	2.2
6/5	GFIL	Shut down②	Close	2.6
6/1	XY Slit	Experiment	Open	2.4
6/1	XY Slit	Shut down①	Close	2.1
6/5	XY Slit	Shut down②	Close	3.7

Table1 Cooper ion concentrations for the cooling water.

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Application of Fluorescence EXAFS to Diluted Mn Systems

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Diluted magnetic semiconductor based on GaAs, $Ga_{1-x}Mn_xAs$, attracts much attention owing to a potential for spin-electronic devices. Magnetic properties of these materials are affected by the chemical environment where Mn atoms are located. A submonolayer plane of Mn embedded in GaAs, which is called the δ-doped Mn layer, is an interesting structure because Mn atoms are highly concentrated in the lateral direction while they are extremely diluted in the normal direction with respect to the interface. In the present study, the X-ray absorption fine structure (XAFS) was employed to analyze the local structure around the δ-doped Mn in GaAs.

Two samples were subjected to measurements. Sample I consists of five periods of 0.2 ML Mn layers grown on GaAs(001) at a temperature of 220 °C. The spacing between neighboring Mn layers was 200 nm. The topmost surface of the sample was capped by 20 nm-thick GaAs. Sample II was a 20 nm-thick MnAs layer epitaxially grown on GaAs(001).

Expected yield of the fluorescent X-rays from Mn is given by the following equation:

$$Y = I_0 \alpha \sigma \frac{\Omega}{4\pi} S \int \rho(z) e^{-(\mu_1+\mu_2)z} dz \qquad (1)$$

where α is the fluorescence probability (approximately 0.5), σ the scattering cross section of a Mn atom (7000 barn for 6.5 keV X-rays), S the illuminated area on the surface (0.3×3 mm^2), $\rho(z)$ the distribution function of Mn atoms, μ_1 and μ_2 the linear absorption factors for incident and fluorescent X-rays, Ω the solid angle of a detector (1.4 sr) and I_0 the incident photon flux. For samples I and II, the integration in the Eq.(1) is calculated as 6×10^{14} / cm^2 and 3×10^{16} / cm^2, respectively. The incident flux was estimated from the current of the ionization chamber used for I_0 monitor to be 2×10^{10} cps. Consequently, the yield is expected to be 40 cps for sample I and 2000 cps for sample II.

Measured intensity of Mn fluorescence was cosistent with the above-mentioned estimation. Since the yield for sample I was too small to complete EXAFS measurements, we focused on measuring near-edge XAFS spectra. Figure 1 shows the yield of Mn K X-rays as a function of the energy of incident X-rays. Because of considerable statistical error, conclusive discussion will be postponed to future work.

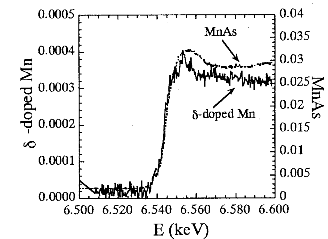

Figure 1: XANES spectra for δ-doped Mn and for MnAs.