Development of the New Sample Cooling System for the Full-Field Imaging Soft X-ray Microscope

Kishin Usui[1], Takuji Ohigashi[2], Hidetoshi Namba[1], Kuniko Takemoto[3], and Hiroshi Kihara[3]

[1]*Department of Physics, Ritsumeikan University, 1-1-1 Noji Higashi, Kusatsu, Shiga 525-8577, Japan.*

[2]*Research Organization of Science & Engineering, Ritsumeikan University, 1-1-1 Noji Higashi, Kusatsu, Shiga 525-8577, Japan.*

[3]*Department of Physics, Kansai Medical University, 18-89 Uyamahigashi, Hirakata, Osaka 573-1136, Japan.*

It is one of the remarkable features of the full-field imaging soft x-ray microscope (SXM) that a highly resolved image of the sample in water can be observed with sufficient contrast by using the water window region, between K-edges of oxygen and carbon, 2.3-4.4 nm. However, radicals of water molecules in the sample which are produced by excitation or ionization by the x-ray radiation cause damage on biological structures [1]. In order to decrease radiation sensitivity, the sample cooling system (SCS) was developed to avoid diffusion of the radicals by freezing the sample [2]. Recently, the sample stages of the SXM at BL-12 have been improved to perform the computed tomography so that the proper SCS is required. In this report, development of the new SCS and the improvement of its performances are shown.

The schematic image of the SCS is shown in Fig. 1 (a) and the photo is shown in Fig. 1 (b). Liquid nitrogen stored in the Dewar vessel flows into the cooling housing *via* a solenoid valve. Then, the liquid nitrogen evaporates and a cooling housing is filled by the cold gas. This cooling housing was designed to be compact and easily detachable to the SXM for its restriction of narrow space of the SXM for the sample. The head parts are made of Macor^{TM} (Corning Inc.) whose thermal expansion is small. In this SCS, the cooling housing is independent of the sample stages not to be affected by bubbling of the liquid nitrogen. The cooling housing is made of high heat insulating material (STYRO FORM^{TM}, EK-2, The Dow Chemical Co., the thermal conductivity coefficient: 0.028 W/m·k) and copper punching hole plate and heat sinks were set inside the housing to reduce drift of the sample by convection flow of the cold gas.

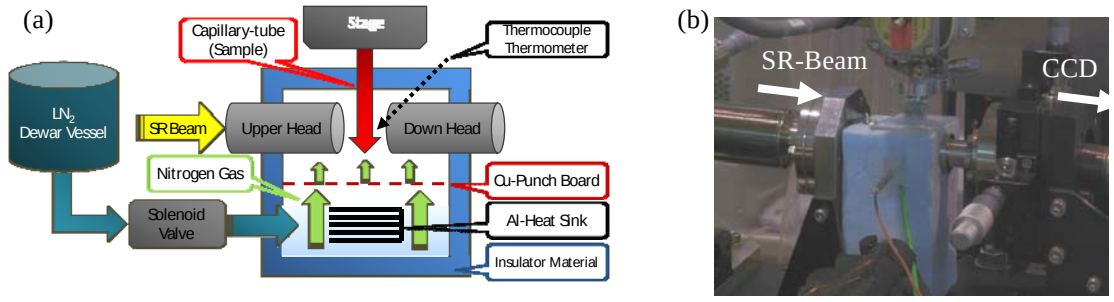

Fig. 1: (a) Schematic image of the sample cooling system and (b) a photo of the sample cooling system

The controlling processes are as follows. A temperature inside the cooling housing is measured by a thermocouple gauge. The controlling program written in LabVIEW (National Instruments Co.) compares this temperature with the preset one and sends a TTL signal to open or close the solenoid valve to the handmade valve controller *via* USB-6008 (National Instruments Co.). The valve controller also has a switch to open the solenoid valve manually for safety and convenience. Performances of this SCS system are shown in Figs. 2. In Fig. 2 (a), the temperature in the cooling housing was cooled down from room temperature to -40 °C within about 3 min. The temperature was maintained under fluctuation smaller than ±1 °C (shown in Fig. 2 (b)). When the supply of liquid nitrogen was stopped at the temperature at -40 °C, the temperature was maintained under 0 °C during 6 min (shown in Fig. 2 (c)).

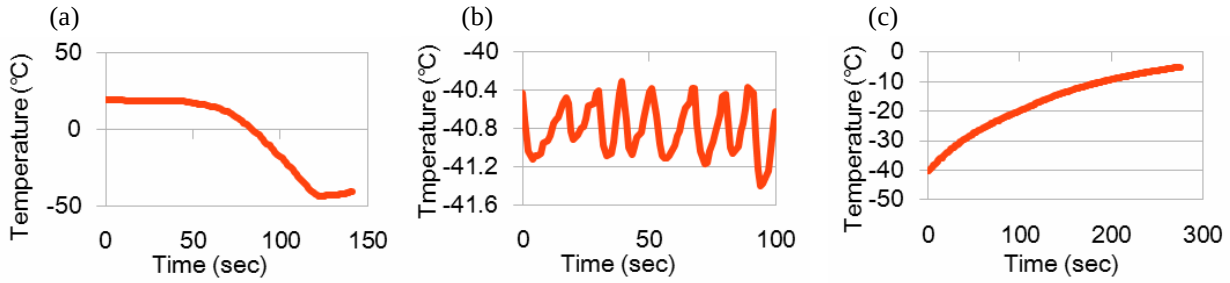

Fig. 2: Performances of the sample cooling system, (a) cooling down from room temperature, (b) keeping temperature and (c) increasing temperature after stopping the supply of liquid nitrogen.

For the evaluation of drifting of the sample, the sample cell, an extended glass capillary tube, was observed. When the temperature was kept at -40 °C, clear image was not obtained because of high absorption of nitrogen gas. Therefore, with stopping supplies of liquid nitrogen, the 2-dimensional positions of a tip of the sample cell were observed from -20 °C every 10 s with binning mode of 2 × 2. The positions of the tip are plotted in in Fig. 3. The mean drifting speed was 16 nm/s and the drifting in vertical direction was much larger than that in horizontal direction. In horizontal direction, the drifting was drastically decreased from 5.8 to 3.5 nm/s by setting the copper punching hole plate and the heat sinks inside the cooling housing. During the stoppage of the liquid nitorogen, the temperature was changed from -40 to -20 °C in 70 s by stopping the supply of liquid nitrogen so that the thermal

expansion occurred in the vertical direction of the sample. The thermal insulation of the SCS still has a problem and improvement of the problem is our future work. Finally, the drifting speed slower than 1 nm/s [3] is expected.

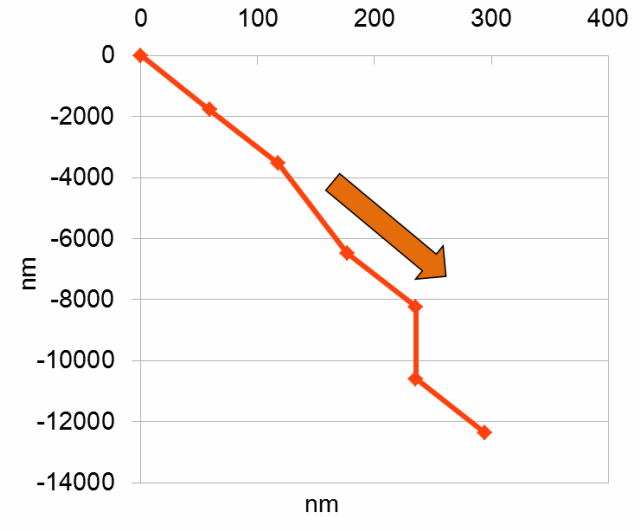

Fig. 3: 2-dimensional positions of the drifting tip of the sample cell are plotted every 10 s. The plots were started from the origin.

References

[1] O. Oksmo and T. Brustad, *Z. Naturforschg.*, **23 b**, 1968, 962-968.

[2] M. Kimura, K. Takemoto, Y. Ohashi, T. Ohigashi, K. Nakanishi, H. Namba and H. Kihara, *Jpn. J. Appl. Phys.*, **50**, 2011, 022601.

[3] D. Sayre, J. Kirz, R. Feder, D.M. Kim and E. Spiller, *Ultramicroscopy*, **2**, 1977, 337-349.