FM-AFM on ice–brine interface at sub-zero temperature

Z. Lu,[1,#] R. Yanagisawa,[1] S. Moriguchi,[1,2] T. Ueda,[3] K. Nakamoto,[3,4] T. Minato,[3,*] and H. Onishi[1,3,5,*]

[1] *Department of Chemistry, Kobe University, Kobe, 657-8501, Japan*
[2] *Shimadzu Techno-Research Incorporated, Kyoto, 604-8436, Japan*
[3] *Institute for Molecular Science, National Institutes of Natural Sciences, Okazaki, 444-8585, Japan*
[4] *Center for Materials Research Platform, Nara Institute of Science and Technology, Nara, 630-0192, Japan*
[5] *Research Center for Membrane and Film Technology, Kobe University, Kobe, 657-8501, Japan*

Presenting author's e-mail: 243s227s@stu.kobe-u.ac.jp

We have successfully examined the operation of atomic force microscopy (AFM) at sub-zero temperatures for the purpose of characterizing ice–liquid interfaces. Topographic imaging in amplitude-modulation mode and force curve measurements in contact mode were conducted on ice films under antifreeze organic solvents, using a Bruker microscope (Dimension XR Icon) [1]. A Shimadzu microscope (SPM-8100FM) equipped with frequency-modulation force detection capability was maintained at low temperatures in an acoustic enclosure (Fig. 1). Topographic imaging and force curve measurements were accomplished on graphite in 1-octanol liquid cooled to as low as − 15 °C [2]. In the present study, the operation of FM-AFM is examined on ice under aqueous solutions of NaCl. Figure 2 illustrates a two-dimensional map of the frequency shift of cantilever oscillation (Δf), where the cantilever scanned vertically from the bulk solution to the ice substrate. At the ice–solution interface, a layered modulation of Δf was identified, suggesting an uneven density distribution within the solution.

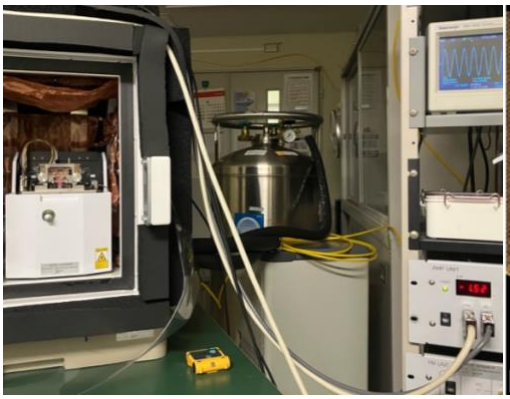

Figure 1. (left) The SPM-8100FM microscope installed in the enclosure for sub-zero temperature operation.

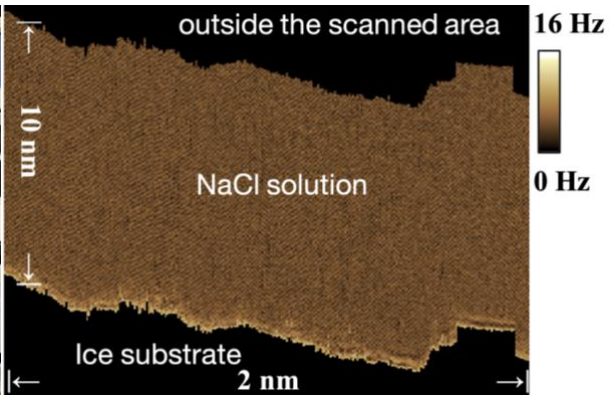

Figure 2. 2D (X-Z) frequency-shift map over ice under an aqueous solution of NaCl cooled at -2 °C. Cantilever oscillation amplitude: 0.79 nm. A large (or small) positive Δf is depicted using a bright (or dark) color.

References

[1] R. Yanagisawa *et al*. *J. Chem. Phys.* **161**, 024702 (2024).

[2] Z. Lu *et al. Jpn. J. Appl. Phys.* **64** 05SP05 (2025).