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Atomic Resolution Investigation in Liquid by Frequency-Modulation (Non-Contact) AFM

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High resolution Frequency-Modulation AFM (FM-AFM) imaging had been limited to a vacuum environment. Fukuma et al. (2005) succeeded in obtaining true atomic resolution in a liquid by FM-AFM [1]. Subsequently, insoluble crystals have been mainly observed in liquid by FM-AFM. However, to observe the soluble crystals with atomic resolution is very important for discussing about mechanism of crystal growth process. We observed soluble crystals such as alkali halide or protein crystals [2].

FM-AFM images were obtained using a modified commercial AFM (Shimadzu, SPM-9600) with open fluid cell. We intentionally observed the soluble crystals in undersaturated solution at first. However, the concentration of solution gradually increased because of evaporation of water. Therefore, dissolution, near equilibrium, or growth conditions could be observed depending on time. By using this method, crystal growth speed of vertical direction was suppressed and atomic resolution images could be obtained.

In the case of KCl(100) cleaved surface in solution, we could observe periodic structure, in which the wavelength is 6.29 A. Therefore, only one kind of atom was considered to be imaged in liquid environment as well as ultrahigh vacuum environment.

In the case of lysozyme (110) in solution, the surface unit cell (black rectangle in fig. 2b, 11.2 x 3.8 nm) involves four molecules with the four unique orientations, which make two kinds of zigzag structures (circles and triangles) along the [001] direction. We obtained the AFM images with the individual four molecules in the unit cell and the image have higher resolution than images obtained by conventional contact mode or amplitude-modulation (tapping) mode AFM[3].

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[3] J. H. Konnert et al., Acta Crystallogr. D 50, 603 (1994).

Keywords: AFM, Crystal growth