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Is dielectrophoresis effective for increasing local concentration of particles in liquid-cell transmission electron microscopy?

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Liquid cell transmission electron microscopy (LC-TEM) is a technique for observing a solution sample by sandwiching it between two membranes and introducing it into the vacuum chamber of a TEM without volatilizing it. This technique has a capability to capture nucleation, which is the initial process of crystallization, from an aqueous solution with sufficient spatial and temporal resolution, and has provided insights into the process [1,2]. However, it is still technically difficult to capture nucleation because control of supersaturation, which is the driving force for crystallization, in the liquid cell is challenging. One way to control the supersaturation is to use radiolysis. Radiolysis can precipitate metal particles by reducing metal ions [3] or produce ions that are not present in the initial solution, and to precipitate crystals involving those ions [4,5]. Thus, radiolysis has been used to control supersaturation to drive crystallization in certain systems. In order to control supersaturation in a wider range of systems, it is necessary to develop a new method. Here, we focused on dielectrophoresis (DEP) [6]. DEP is the phenomenon in which particles in a solution are moved by the inhomogeneous electric field gradient created around an electrode when a voltage is applied to the electrode. By adequately applying DEP force to the particles in solution, the particles can be collected around the electrode, which increases the local concentration of particles and is directly linked to the control of supersaturation. The particles that can be collected by DEP are relatively large in size, such as protein molecules [7]. DEP has been mentioned in studies involving LC-TEM [8], but phenomena related to DEP, such as particle collection, have not been observed in LC-TEM. In this study, we applied DEP to control the local concentration of the particles in solution using a custom-designed silicon chip equipped with a silicon nitride membrane, electrical isolation structures, and electrodes design and manufactured by Norcada, and observed it by optical microscopy and TEM to investigate the effectiveness of DEP.

Colloidal solutions were used as samples to investigate the DEP inside the liquid cell. We used optical microscopes and a TEM (JEM-2100F) with a field-emission gun at an acceleration voltage of 200 kV. We used an external liquid-cell holder for observation by optical microscopes and an LC-TEM holder (Poseidon Select). Both holders are equipped with channels for flowing solution and applying electric signals to the liquid cell. We used two types of silicon chips with electrodes that can be used in the holders (Fig. 1). One is a commercial product used for electrochemical experiments, and the other is a chip with two electrodes with a gap of a few microns (Fig. 2). The latter was fabricated to increase the electric field gradient around the electrodes and to increase the DEP force on the particles in the sample solution. An ac signal was applied by a function generator.

Liquid cells were assembled using each of the two types of chips, and the sample solution flowed into the cell. During the observation, the ac signal was applied to the electrode. We observed the colloidal

particles exerted by the DEP force and whether the particles collected around the electrodes. Under the optical microscope, the colloidal particles were collected around the electrodes by optimizing the frequency and voltage of the applied ac signal, no matter which chip was used. In TEM observation, it was difficult to collect the particles around the electrode when using the commercial silicon chip. This is because there is not enough DEP force exerted on the particles to collect them around the electrode. This may be due to the attenuation of the signal at the electrode. The silicon chip fabricated in this study was designed to allow a high electric field gradient, which allowed particles to be collected around the electrode. Our observations suggest that DEP can be used to locally increase the concentration of particles in LC-TEM.

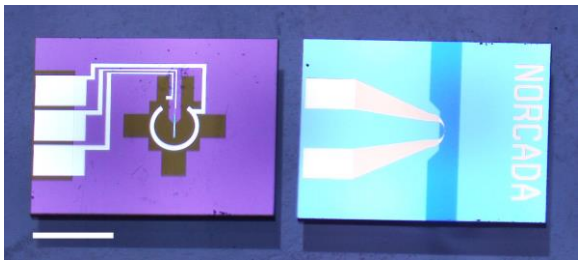

Figure 1. Silicon chips for the liquid cell. (Left) A commercially available chip for electrochemical experiments. (Right) A newly developed chip for increasing the DEP force. The scale bar is 2 mm.

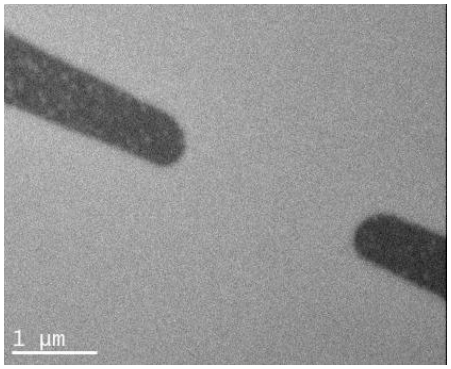

Figure 2. A TEM image of a newly developed chip. The rods on the right and left of the image are the electrodes. The gap between the electrodes is about 2.3 µm.

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