In-situ TEM Observation of Gold Particles in Water using an Environmental Liquid Cell

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A closed-type environmental transmission electron microscope (ETEM) is capable in-situ observation of specimens immersed in liquids or gases. In this equipment, diaphragms, which are set at the top and the bottom of a space for the specimen to isolate from the vacuum, are one of the most important components. Although silicon nitride (SiN) films are generally used for this purpose [1], charging effect on the diaphragms is inevitable because of the insulating SiN. In order to solve this problem, we have developed a carbon/SiN hybrid diaphragm which consists of amorphous carbon film as conductive base material and amorphous SiN thin layer coated on it [2]. In this study, our developed diaphragms were applied to *in situ* TEM observation of motion of gold colloidal particles in water.

The specimen used in the experiment was gold colloidal particles 80 ± 5.7 nm in diameter (Fig. 1(a)), dispersed in water in $1.1{\times}10^7$/μl concentration. The liquid specimen was sandwiched by two C/SiN diaphragms supported on dedicated Cu grids with controlling distance between them by 240 nm with a spacer. The diaphragms of the carbon and the SiN layers were formed by a dedicated vacuum deposition to 9.3 nm thickness and pulsed laser deposition (PLD) to 5.9 nm thickness, respectively. The sandwiched liquid specimen was set in an environmental-cell specimen holder [3-4], in which the specimen was in contacted with ambient air through two pipes, as shown in Fig. 1(b). TEM observations were done in a condition of low electron density ($\sim$0.1A/cm^2) using an H-8000 microscope (200kV; Hitachi High Technologies).

Figures 2 show results of dynamic observations of motions of the gold particles in water. A gold particle in Fig. 2(a) migrated slowly at a speed of 10 ~ 30 nm/s, as shown in a quantitative analysis of variation of the moving speed in Fig. 2(b). This is not a Brownian motion because the gold particle drifted in a single direction. In contrast, a gold particle in Fig. 2(c) moved quickly just after starting the electron beam illumination at high speed of more than 1200 nm/s. Although a next short movement occurred after the first quick motion, the gold particle subsequently stopped, as shown in Fig. 2(d). There different types of the motion mean driving force of the movement of the gold particles is not unique. In the present cases, it is considered that an effect of water flow and coulomb force by charging due to the electron beam irradiation caused the motions of the particles in Fig. 2(a) and (c), respectively.

References
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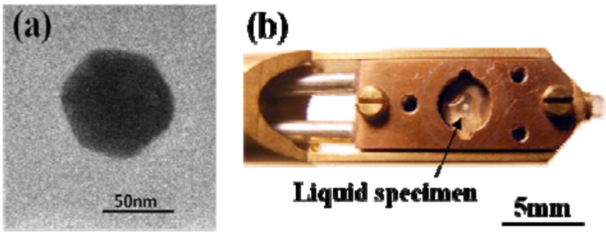

FIG. 1

(a) TEM image of a gold colloidal particle.

(b) Liquid specimen of (a) dispersed in water dropped in the E-cell specimen

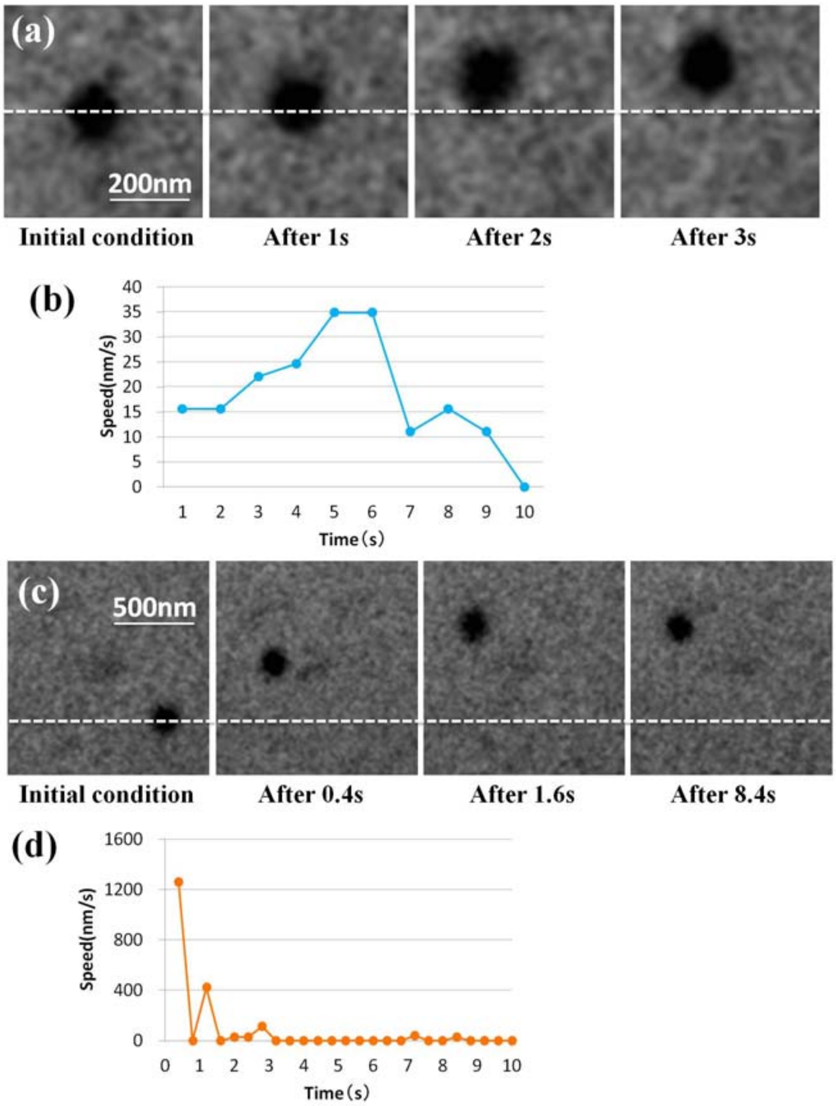

FIG. 2 TEM images of motion of gold particles ((a) slow and (c) quick movement)

(b), (d) variation of speed of motions in (a) and (c), respectively.