Materials Science

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In-situ Observation of Formation of Ag Particles in Tween20/Water/Ionic Liquid Microemulsions

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Introduction

Microemulsions consisting of the ionic liquid (IL), surfactant and water have been intensively investigated by Gao et al [1-3]. For example, the ternary system consisting of Tween20/water/1-butyl-3-methylimidazolium hexafluorophosphate ([bmim][PF_6]) was prepared, and three regions of the microemulsions, i.e., water-in-[bmim][PF_6], bicontinuous, and [bmim][PF_6]-in-water, were identified. Tween20/water /[bmim][PF_6] microemulsions was found to solubilize salt species into the microemulsion droplets [3]. In this study we have synthesized Ag particles by the photoreduction of silver perchlorate ($AgClO_4$) in the microemulsions, Tween20/water/1-octyl-3-methylimidazolium tetrafluoroborate ([omim][BF_4])/CO_2 system, and have investigated the electronic structure of Ag particles by *in-situ* EXAFS measurements. Here we have applied the high pressure CO_2 to the ternary system in order to improve the mobility of ions and particles.

Experimental

Ag particles were synthesized in a high-pressure SUS 316 cell (inner volume of ca. 10 mL) equipped with four optical windows: two of them were CVD diamond windows for *in-situ* EXAFS measurements and the other two were quartz windows for the irradiation of Hg lamp. Tween20/water/[omim][BF_4] microemulsions containing $AgClO_4$ were prepared by adding CO_2 into the cell which contained a mixture of $AgClO_4$ aqueous solution, Tween20, [omim][BF_4], and benzoin for photoreduction. The weight fraction of Tween20 was 0.33, and the [omim][BF_4]-to-Tween20 molar ratio (R) and the water-to-Tween20 molar ratio (w) was fixed to 8.7 and 0.62, respectively. The concentration of [Ag^+] in the ternary system was 4.4 or 21.1 mM. The cell was kept at 35°C and 25 MPa for 1 h with continuous stirring to form microemulsions. After stirring, the microemulsion was photo-irradiated for 5 h to investigate the reduction of Ag^+ and the formation of Ag particles.

The *in-situ* EXAFS measurements were carried out in a fluorescence mode at NW10A beam line in PF-AR. EXAFS spectra at Ag-K edge were collected by a Ge solid-state detector to evaluate the electronic state and the coordination number of Ag particles. Data analysis was performed by REX2000 ver. 2.0.7 (Rigaku Co.).

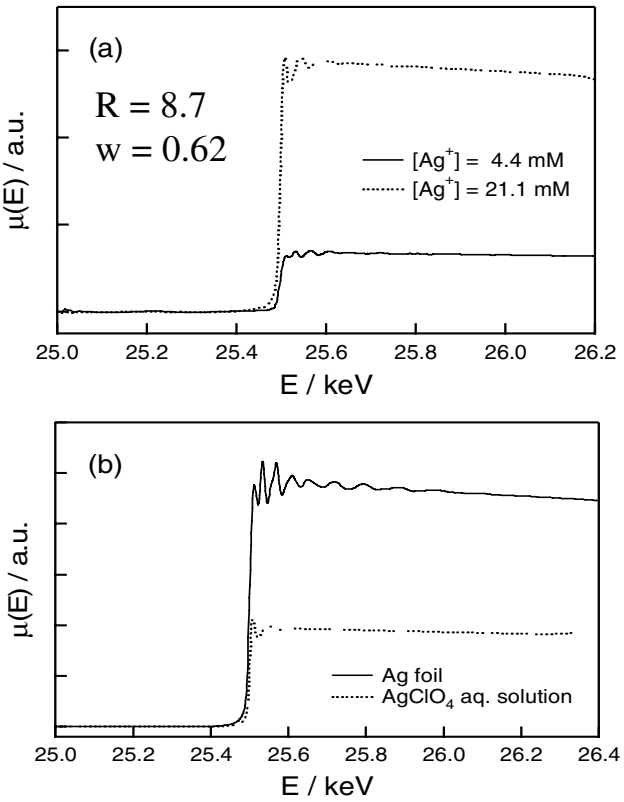

Fig. 1. X-ray absorption coefficient μ(E) at the Ag-K edge for (a) the Ag solutions prepared by 5h photoreduction and (b) the reference of Ag foil and $AgClO_4$ aqueous solution.

Results and Discussion

Fig. 1(a) shows the EXAFS oscillations of Ag colloidal solutions prepared by photoirradiation. Compared with those obtained from the reference compounds (Fig. 1(b)), the reduction of Ag^+ completes after irradiation time of 5 h in the [Ag^+] concentration equal to 4.4 mM, although a large amount of unreduced Ag^+ ions remains in the higher concentration of [Ag^+]. This confirms the creation of Ag-Ag bond and the subsequent formation of Ag particles in the dilute concentrations. The detailed analysis is in progress.

References

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