In-situ XAFS study of Ag clusters in zeolite 13X

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Introduction
In the Ag+-exchanged zeolite the Ag+ ions are present inside of the zeolite cages, as needed to balance the anionic charge of the zeolite framework. By dehydration under vacuum, silver ions are reduced and then silver clusters are formed in the case of zeolite 4A (Ag-4A) [1,2]. In this report, we study the structural change of Ag clusters in zeolite 13X (Ag-13X) by in-situ XAFS measurements.

Experimental
Ag-13X powder samples were prepared by immersing Na-13X (Na,[AlO]3[SiO]3)246.2H2O) zeolite in an aqueous AgNO3 solution at 25°C[2,3]. We prepared two kinds of sample that 86 (Ag,.-13X) and 66 Ag+ ions (Ag+.-13X) are replaced by Na+ ions. The air-dried Ag-13X was set into the in-situ XAFS measurement cell in which the sample can be heated under vacuum. Ag K-edge XAFS spectra were measured at BL-10B at 77K in vacuum. A Si(311) channel-cut monochromator was used, and energy and current of the storage ring were 3.0 GeV and 250-300 mA, respectively. The analyses were performed by XANADU code [4].

Results and Discussion
Figures 1 and 2 show the Fourier transforms (FT) of EXAFS for Ag+.-13X and Ag+.-13X measured at 77K after heated at various temperatures. It is found that two prominent peaks in the FT: First peak corresponds to Ag-O and second one to mainly Ag-Ag [2]. It is noted that the contribution from Ag-Ag increases at in Ag+.-13X 300C and in Ag+.-13X at 200C, respectively. For Ag+.-13X the prominent increase of the second peak is observed at 100C. It is interesting that the temperature corresponding to the formation of Ag clusters shifts to higher temperature with the increase of the amount of Ag+ ions in the zeolite cavity. More accurate determination of structural parameters is in progress.

Fig. 1 Fourier transforms of EXAFS for Ag+.-13X measured at 77K in vacuum after heated at various temperatures.

Fig. 2 Fourier transforms EXAFS for Ag+.-13X measured at 77K in vacuum after heated at various temperatures.

References

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