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Al *K*-edge XAFS study for Ag-zeolites

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Introduction

In the fully Ag^+-exchanged 4A zeolite (Ag-4A) the $12Ag^+$ ions are present inside zeolite cages, as needed to balance the anionic charge of the zeolite framework. When the Ag-4A zeolite are dehydrated in the cavity under heating at 500°C or vacuum, the Ag clusters are formed by reducing Ag^+ ions[1]. Thereafter, when these samples are hydrated in the cavity in atmosphere at room temperature, their structures are maintained and the Ag clusters are destroyed partially. We reported that during they cool down to room temperature under atmospheric condition, there is a intermediate state in the range from 70°C to 50°C. The intermediate species can be considered to give a prominent photoluminescence at 1.9 eV [2]. To reveal the mechanism of the formation of the luminescent species, Al K-edge EXAFS analyses should be useful. In the present study, we report the results of the XAFS measurements on the Al *K*-edge for the fully exchanged Ag-4A zeolite at several temperature (unheated, 100, 200, 300, 400, 500°C) under atmosphere in comparison with Na-4A as standard samples.

Experimental

The Ag-4A ($Ag_{12}[(Al_2)_{12}(SiO_2)_{12}]27.5H_2O$) powder samples were prepared by immersing Na-4Azeolite in an aqueous $AgNO_3$ solution at 25°C [1]. The sample was prepared by heating Ag-4A under atmosphere for 24h at several temperature (100-500°C). After the heating, the sample was cooling down to room temperature. Al *K*-edge EXAFS spectra were measured at BL11A of the Photon Factory in KEK. Silicon drift detector (SDD) was used for the fluorescence detection. These EXAFS interference functions extracted from the absorption spectra was Fourier transformed by XANADU code [3].

Results and Discussion

Figure 1 shows the Al *K*-edge XAFS spectra for Ag-4A and Na-4A zeolites for various heating temperatures. Fairly good quality data were obtained. We analyze the EXAFS data extracted from the Fourier transformed data and performed non-liner least square fitting. Figure 2 shows the plots of first nearest Al-O interatomic distances of Ag-4A and Na-4A for various heating temperatures. The right plot is for unheated one. For Na-4A zeolites, the interatomic distance of the first nearest Al-O does not depends on the heating conditions. This is quite natural because no cluster can be formed in Na-4A zeolite by the heating.

On the other hand, the Al-O distance depends linearly to the heating temperatures: The higher temperature, the longer Al-O distances. This means that the Ag cluster formed inside the cavity of the zeolite repels the electrons on O atoms in the framework of the zeolite.

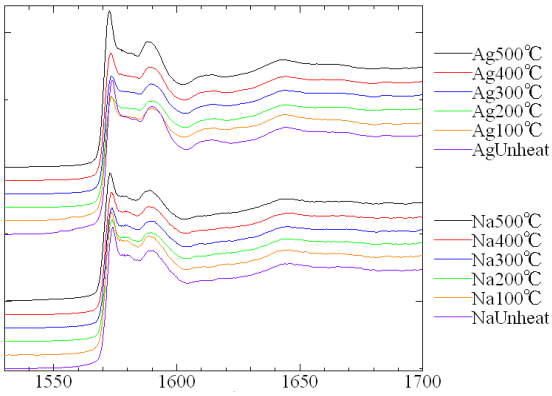

Fig.1 *K*-edge XAFS spectra measured at room temperature for Ag-4A and Na-4A zeolites for various heating temperatures.

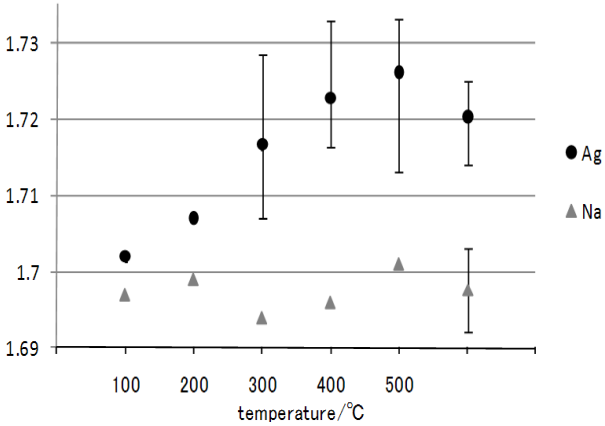

Fig.2. Plots of first nearest Al-O interatomic distances of Ag-4A and Na-4A for various heating temperatures.

References

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