Materials Science

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Quick XAFS Measurements for Ag Clusters in Zeolite 4A

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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, time resolved structural analyses are important. In the present study, we report the results of the Quick XAFS measurements on the Ag *K*-edge for the vacuum-dehydrated fully exchanged Ag-4A at 300°C.

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 vacuum of 10^{-6} Torr for 24h at 300°C. After the heating, the sample was cooling down to room temperature under vacuum and then the pressure is increasing to 760 Torr. Ag *K*-edge Quick EXAFS and Normal EXAFS spectra were measured at beamline NW10A of the Photon Factory in KEK. A Si (311) double-crystal monochromator was used. These spectra were recorded in transmission mode using ionization chamber detectors. The measurement condition of Quick XAFS: The Quick EXAFS spectra were recorded continuously for 50 sec per one scan. The date number is 3300 and the data accumulation time is 0.015s per point. These EXAFS interference functions extracted from the absorption spectra was Fourier transformed by XANADU code [3].

Results and Discussion

Figure 1 shows the Quick EXAFS and Normal EXAFS spectra, $k^2\chi(k)$ for several conditions: (a) it was measured at 300°C in vacuum by Quick XAFS [Q-300°C] and (b) that of Normal EXAFS [N-300°C], (c) measured at room temperature in vacuum by Quick EXAFS [Q-Vac] and normal EXAFS [N-Vac] . And then the samples were exposed under atmosphere [Q-atm and N-atm] measured at room temperature. The oscillations are detectable in the *k* region up to 14 $Å^{-1}$. For Quick EXAFS spectra ((a), (c) and (e)) there are some noises in the high wave number. It was found that the Quick EXAFS spectra was reproduced the Normal EXAFS for the same condition except for the noise in high *k*-range.

Figure 2 shows the Fourier transforms F(r) of Normal EXAFS and Quick EXAFS spectra. The Quick EXAFS spectra are almost same as the Normal EXAFS for the same condition.

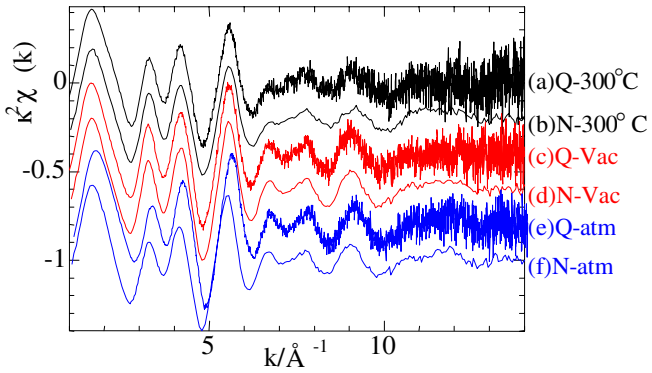

Fig. 1 Ag *K*-edge Quick EXAFS and Normal EXAFS $k^2\chi(k)$, functions for three different Ag-4A zeolites. (a) Q-300°C (b) N-300°C, (c) Q-Vac (d) N-Vac (e) Q-atm (f) N-atm.

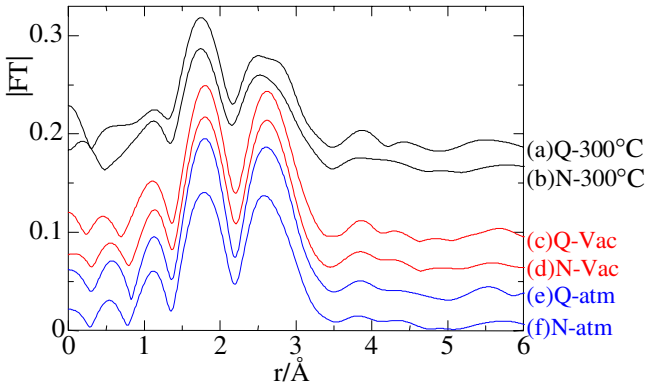

Fig. 2 Fourier transforms of Quick EXAFS and Normal EXAFS, F(r), for three different Ag-4A zeolites as shown in Fig.1.

References

[1] T. Miyanaga, *et al.*, J. Synchrotron Rad. **8**, 557 (2001).
[2] H.Hoshino *et al,* J. Phys. Soc. Jpn., in press (2008).
[3]H. Sakane, *et al.*, Jpn.J. Appl. Phys. **32**, 4641 (1993).

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