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

Takafumi MIYANAGA[1]*, Yushi SUZUKI[1], Hideoki HOSHINO[2], Naoyuki MATSUMOTO[1], Takeshi AINAI[2]

[1] Faculty of Science and Technology, Hirosaki University, Hirosaki, Aomori 036-8561, Japan

[2] Faculty of Education, Hirosaki University, Hirosaki, Aomori 036-8560, Japan

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 ($Na_{86}[(AlO_2)_{86}(SiO_2)_{106}]246.2H_2O$) zeolite in an aqueous $AgNO_3$ solution at 25℃[2,3]. We prepared two kinds of sample that 86 (Ag_{86}-13X) and 66 Ag^+ ions (Ag_{66}-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 monochrometer 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].

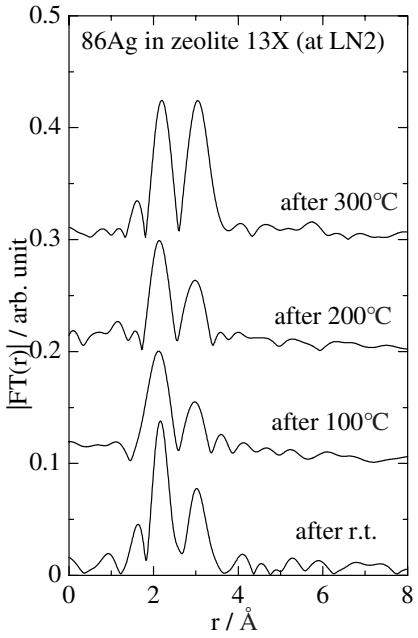

Fig. 1 Fourier transforms of EXAFS for Ag_{86}-13X measured at 77K in vacuum after heated at various temperatures.

Results and Discussion

Figures 1 and 2 show the Fourier transforms (FT) of EXAFS for Ag_{86}-13X and Ag_{66}-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_{86}-13X 300C and in Ag_{66}-13X at 200C, respectively. For Ag_{12}-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.

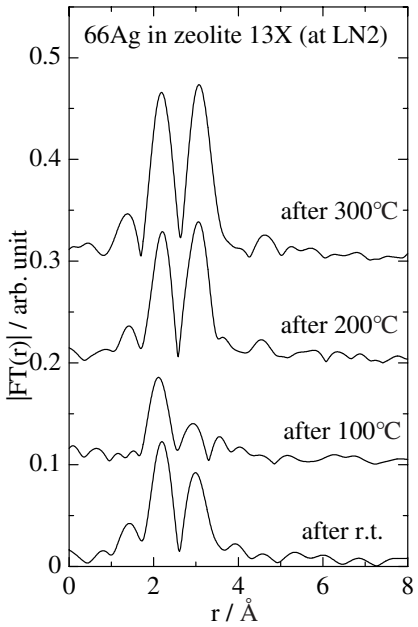

Fig. 2 Fourier transforms EXAFS for Ag_{66}-13X measured at 77K in vacuum after heated at various temperatures.

References

[1]T.Sun and K.Seff, (1994), *Chem. Rev.* **94(4)**, 857-870.

[2]T.Miyanaga, H.Hoshino and H.Endo (2001), *J. Synchrotron Rad.* **8,** 557-559.

[3]H.Hoshino, (1996) *Bulletin of the Faculty of Education, Hirosaki University*, **75**, 29-37 .

[4]H.Sakane, T.Miyanaga, I.Watanabe, N.Matsubayashi, S.Ikeda, S. and Y.Yokoyama, (1993), *Jpn. J. Appl. Phys.*, **32**, 4641-4647.

* takaf@cc.hirosaki-u.ac.jp