10B/2000G238

Structural study of transition metal chalcogenide microclusters confined in the zeolite pores

Kenji MARUYAMA*[1], Makoto YAMAZAKI[1], Hideoki HOSHINO[2], Takafumi MIYANAGA [2], Ryouta MARUKO[2]
[1]Niigata University, 8050 Igarashi-2, Niigata, 950-2181, Japan
[2]Hirosaki University, 1 Bunkyo-cho, Hirosaki, 036-8560, Japan

Introduction

It is interesting to investigate the structure and properties of microclusters confined in the zeolite cages. In the previous study, we showed that the Se atoms exist in the chain structure in the channels or in ring structure in cages and its structural parameters, such as the bond length and the bond angle, are different from those of bulk Se. These samples also show the photo darkening phenomenon at low temperature.

Recently we have success to make transition metal chalcogenide microclusters in the zeolite cages by heating the ion exchanged zeolite sample, which contains Se or Te clusters, in H_2 gas. The appearance of new magneticity is expected for these magnetic metal chalcogenide, such as Ni-Se/Te, microclusters. In this study we have performed EXAFS measurements for Te-Ni microclusters confined in the cages of zeolite A in order to investigate the structure of these microclusters.

Experimental

The Ni type zeolite A was obtained by an ion exchange of 4A zeolite with $Ni(NO_3)_2$ aqueous solution. The number of Ni ions per cage was determined by using an ICP mass analyzer. The zolite powder was dehydrated by heating up to 400°C under the vacuum of 10^{-6} Torr. The weighed Te was heated with the zeolite powder up to 450 °C in order to make the Te clusters in the cages. Thereafter the sample was heated in H_2 gas up to 400°C for half an hour. Then the Te-Ni microcluster was formed in the cages. The obtained sample was described as Ni_xTe_y(red), where x and y are the number of Ni and Te atoms per cage, respectively.

The EXAFS measurements around the Te K-edge were performed by using the BL10B beam line during the 3 GeV ring operation. The powder sample was packed in a Teflon cell in a copper holder. This sample block was cooled down to 20K with the standard cryocooler.

Results and discussion

Fig. 1. shows the EXAFS oscillation $\chi(k)$ of Ni-Te microclusters in zeolite cages at 20K and room temperature. The $\chi(k)$ of crystalline Te (c-Te) was also shown for comparison. The profile is very different from each other especially in low wave number region. This means Te atoms are binding with Ni ions. The amplitude of $\chi(k)$ is enhanced at 20K. Fig. 2 shows the temperature dependence of radial distribution function around Te atom $|F(r)|$, which was derived by a Fourier transform of $\chi(k)$ for $Ni_{1.9}Te_{3.8}$(red). The first peak around 2.2Å, which is corresponds to Te-Te bonds, has large tail in its longer r side, which means the Te-Ni bonds are formed. The second peak around 3.4Åis much enhanced with decreasing temperature. The detailed analysis for Te-Ni microslusters is in progress now.

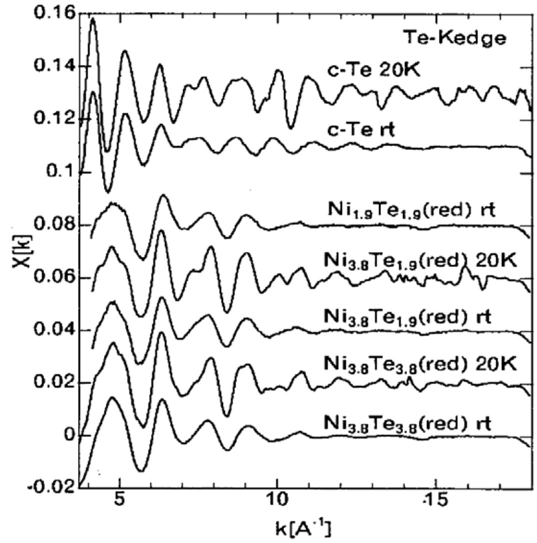

Fig. 1 The EXAFS oscillation $\chi(k)$ for NiTe microclusters of several composition.

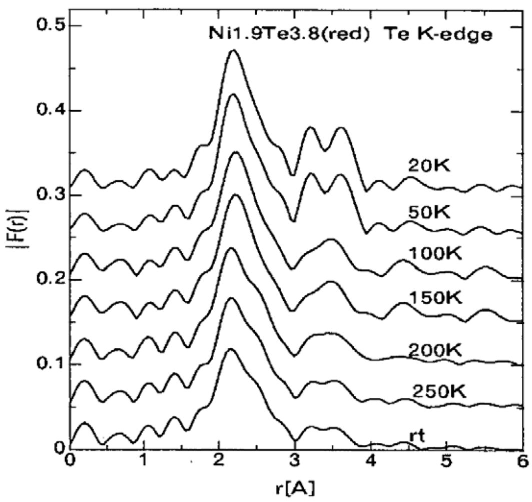

Fig. 2 Temperature dependence of $|F(r)|$ around the Te atoms for $Ni_{1.9}Te_{3.8}$(red)

*maruken@sc.niigata-u.ac.jp