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# IN-SITU ENERGY-DISPERSIVE XAFS STUDY ON THE DECARBONYLATION PROCESSES OF $Mo(CO)_6$ IN Y ZEOLITES

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## Introduction

The transformation of metal complexes to metal species on inorganic oxides is a key issue to develop efficient catalytic systems. Especially, zeolites are expected to provide highly dispersed homogeneous metal species. $Mo(CO)_6$ is often used to prepare highly dispersed Mo species in the Y-type zeolite and highly dispersed molybdenum species were prepared in the channel of zeolites by the decarbonylation of $Mo(CO)_6$.

We investigated the dynamic decomposition behavior of the molybdenum carbonyl species encaged in NaY and HY zeolites by using energy-dispersive XAFS (DXAFS) technique [1,2]. DXAFS is an experimental method to acquire XAFS data in a short period using a bent crystal as a polychromator and a position sensitive detector. The X-rays in the whole energy range were monitored simultaneously in this set up and the data acquisition time is about 1 s in the present study.

## Results and Discussion

DXAFS measurement was carried out using synchrotron radiation at BL-9C at KEK-PF. A triangle-shaped Si(311) bent crystal was used to focus polychromatic X-ray beam and a 1024-pixel position sensitive detector was used to record DXAFS spectra. $Mo(CO)_6$ was engaged in Y zeolites by chemical vapor deposition (CVD) method.

Figure 1 shows Fourier transformed $k^3$-weighted DXAFS functions of $Mo(CO)_6$/NaY during the temperature-programmed decarbonylation under vacuum at a heating rate of 5 K $min^{-1}$ in the temperature range 293-623 K. The curve fitting analysis [1] revealed that the stable intermediate species $Mo(CO)_3(O_L)_3$ ($O_L$: zeolite framework oxygen atom) are formed during the decarbonylation of $Mo(CO)_6$/NaY between 440 and 490 K and that $Mo(CO)_3(O_L)_3$ species changes to $Mo_2(C)O_x$ at 500-550 K.

Figure 2 shows Fourier transformed $k^3$-weighted DXAFS functions of $Mo(CO)_6$/HY during the temperature-programmed decarbonylation. According to the curve fitting analysis, $Mo(CO)_3(O_L)_3$ is the intermediate also in HY zeolite during the decarbonylation and $Mo(CO)_3(O_L)_3$ species was easily oxidized by the zeolite protons. Oxomolybdenum species after the decarbonylation of $Mo(CO)_6$/HY at 623 K can be fitted without Mo-Mo contribution. During the transformation of $Mo(CO)_3(O_L)_3$ to $MoO_x$, oxidation of the molybdenum species occurs by zeolite OH group.

The present study demonstrates that the DXAFS technique is powerful and useful to monitor the dynamic structure change in metal carbonyl species during the temperature-programmed decarbonylation process in a 1 s time scale.

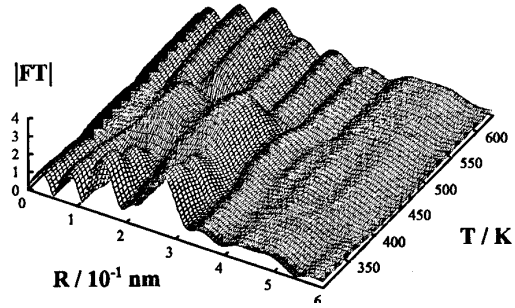


Figure 1 Fourier transforms of DXAFS spectra of $Mo(CO)_6$/NaY during temperature-programmed decarbonylation

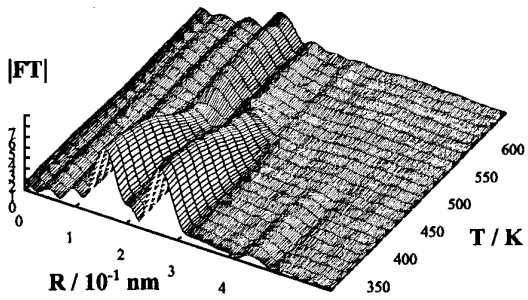


Figure 2 Fourier transforms of DXAFS spectra of $Mo(CO)_6$/HY during temperature-programmed decarbonylation

## References

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