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EXAFS Analysis for Nano HDS Catalysts on Nano-Alumina Support

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Introduction

Owing to an environmental legislation, hydrotreatment is the most important process to remove sulfur compounds from oil for the production of ultra-low sulfur diesel fuel by refiners. So the Hydrodesulfurization (HDS) catalysts serve as very important roles to decrease sulfur to lower than 10 ppm in fuel oils, especially in diesel fuel. The HDS catalysts generally composed of sulfided molybdenum promoted by nickel or cobalt supported on alumina support. Improving the performance of HDS catalysts is still one of the most important issues in petroleum industry. In this study, we have synthesized nano-alumina as a useful support for the HDS catalysts [1]. Then a series of HDS catalyst loaded on nano-alumina supports were synthesized and finally they were tested in the hydrodesulphurization of gasoil under operating conditions similar to those of industry over prepared catalysts. Mo K-Edge and Co K-Edge EXAFS analyses were achieved to know the coordination number, bond length and geometry of Mo and Co atoms in the CoMoS catalytic sites.

Experimental

The preparation of nano-alumina support was performed in the same way as Ref. [1]. Then the Co and Mo metals were impregnated on the support. Measurements of extended X-ray absorption fine structure (K-edge EXAFS of each element) were carried out at the Photon Factory in the Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK–IMSS–PF). The measurements were made in a fluorescence detection mode using a Lytle detector with Kr gas. The EXAFS spectra were analyzed with the UWXAFS package. The curve-fitting analysis was carried out using the FEFFIT program in the R-space.

Results and Discussion

Fig. 1 shows the k^3-weighted Mo K-edge EXAFS Fourier transforms and curve fitting results for CoMo/Nano-Alumina, where the Mo-S, Mo-O, Mo-Mo shells appear. In the fitting process of EXAFS functions, the value of the amplitude factor S_0^2 was fixed at 0.72 for Mo-O and Mo-S and 1.18 for Mo-Mo.

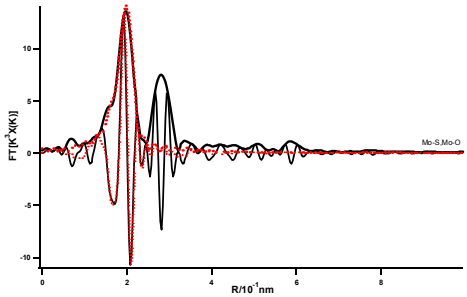

Fig. 1 k^3-weighted Mo K-edge EXAFS Fourier transforms and curve fitting results for CoMo/Nano-alumina.

The results of curve-fitting of CoMoS/nano-alumina are summarized in Table 1, in comparison with the result of CoMoS/micro alumina [1]. Each shell was fitted separately. It is clearly seen that impregnation on the nano-alumina support resulted in the higher Mo-S coordination number, indicating that sulfidation of Mo is achieved to a larger extent on the nano-alumina support.

Table 1 Curve-Fitting results of the Fourier-transformed EXAFS data (Mo-S, Mo-O, Mo-Mo shells) for CoMo/nano-alumina and micro-alumina measured at room temperature functions of the spent catalysts.

Samples	Shell	CN	R (1×10^{-1} nm)	DW (1×10^{-4} nm^2)	S_0^2	R_f (%)
CoMo/ nano- alumina	Mo-O	1.44	1.691	1.3	0.72	0.57
	Mo-S	5.2	2.412	3.8	0.72	0.57
	Mo-Mo	1.88	3.170	4.2	1.18	0.97
CoMo/ micro- alumina	Mo-O	0.70	1.722	4.3	0.72	0.42
	Mo-S	3.90	2.406	3.1	0.72	0.42
	Mo-Mo	1.27	3.167	4.6	1.18	1.46

References

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