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XAFS study on local structure of supported catalysts prepared by arc-plasma deposition

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1 Introduction

A dry catalyst preparation using plasmas has attracted considerable attentions. The main advantage of the arc-plasma method should be its applicability to bimetallic nanoparticles. Recently, we have reported a novel preparation route of Pd-Fe catalysts using pulsed arc-plasma deposition, which enables the one-step deposition of bimetal nanoparticles from two bulk metals [1]. In the present work, we have applied the dual-mode arc-plasma method for the preparation of Pd-Fe/Al_2O_3 catalysts to study their local structure by means of XAFS.

2 Experiment

Pd-Fe/Al_2O_3 catalysts were prepared by pulsed cathodic arc-plasma method. The bimetal catalysts were prepared with synchronous or asynchronous pulses for the generation of two plasmas from each cathodic arc source, which are denoted as “syn” and “asyn”, respectively.

The structure of the catalyst was characterized by EXAFS. EXAFS was recorded in transmission mode on NW-10A, PF-AR, KEK (Proposal No. 2012G749). The EXAFS data were processed by Athena and Artemis software packages.

3 Results and Discussion

Local structures of Pd and Fe were investigated by XAFS. Figure 1 shows Fourier transforms of Pd K-edge EXAFS for 1.0wt% Pd-1.2wt% Fe/Al_2O_3 prepared by dual-mode arc-plasma method together with two references (Pd foil and PdO) without corrections for phase shifts. When the coordination shells were filtered, the best curve-fitting was obtained and resultant structural parameters including phase shift corrected *r*-values are shown in Table 1. For all of the Pd-Fe/Al_2O_3, the shells indicated two intense peaks attributable to the Pd-O and Pd-Pd bond. However, a shoulder at about 2.2 Å was observed for Pd-Fe/Al_2O_3(syn). The best fit was finally achieved when the contribution of a Pd-Fe shell (r=2.70 Å, CN=0.5) attributed to a Fe_3Pd_1(*Pm-3m*) along with Pd-Pd (r=2.73 Å, CN=0.8) was taken into consideration. This result is in agreement with EDX analysis taken from single nanoparticles in our previous research [1]. Therefore, it is considered that dual-mode arc-plasma method by synchronous pulses should be necessary for the deposition of bimetal nanoparticles. Pd-Fe/Al_2O_3(asyn) exhibited a much weaker peak due to a Pd-Pd shell (r=2.71 Å, CN=0.6) than Pd-Fe/Al_2O_3(syn).

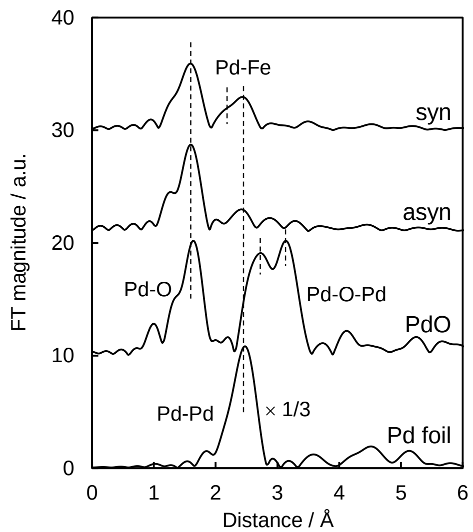

Fig. 1 Fourier transforms of k^3-weighted Pd K-edge EXAFS of Pd-Fe/Al_2O_3.

Table 1 The fitting parameters obtained from Pd K-edge EXAFS analysis of Pd-Fe/Al_2O_3

	shell	CN^a (±0.2)	r/Å^b (±0.03)	$\sigma^2/10^{-2}$Å^{2c} (±0.05)	R/%
syn	Pd-O	2.7	2.01	0.45	2.0
	Pd-Fe	0.5	2.70	0.65	
	Pd-Pd	0.8	2.73	0.59	
asyn	Pd-O	2.7	2.01	0.27	2.5
	Pd-Pd	0.6	2.71	0.59	
PdO	Pd-O	4.0	2.02	0.27	0.5
	Pd-O-Pd	4.0	3.05	0.52	
	Pd-O-Pd	8.0	3.43	0.55	
Pd foil	Pd-Pd	12.0	2.74	0.59	0.1

Interval of k-space to r-space of FT is 3.0-16.0 Å^{-1}.
^{a}Coordination number. ^{b}Interatomic distance. ^{c}Debye-Waller factor.

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References

[1] S. Hinokuma, *et al*., Catal. Today, **201**, 92 (2013).

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