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The physical properties of the nanocrystal strontium titanate under high-pressure by x-ray diffraction and absorption methods

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I、The results of data analysis

In-situ XAS measurements of palladium nanocrystals (2.7 to 50 nanometers) and bulk were performed at BL12B2 beamline during Oct. 14~21, 2004. EXAFS study is especially useful in providing structural information about the crystalline and amorphous materials. The analysis of EXAFS data can determinate the local structural parameters, such as interatomic distance and Debye-Waller factor, etc.

Figure 1 shows the Fourier transform of Pd nanocrystalline and bulk EXAFS spectra at different size. It was found that the Fourier transform of Pd nanocrystal and bulk K edge of the 1st shell distance decreased as the size increased, shown in Fig. 2, and the Debye-Waller factor decreased as the size increased, shown in Fig. 3.

II、Conclusion

Our experimental results indicated that the bond length decreased as the size increased and the Debye-Waller factor decreased as the size increased. We could attribute this observation as the bond strength became weak associated with the size increased, and therefore the lattice vibration became stronger as the size increased.

The further analysis will reveal their relation precisely.

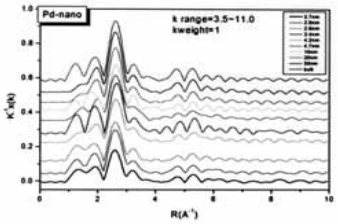

Fig. 1 The Fourier transform of EXAFS spectra of the Pd nanocrystals at different size.

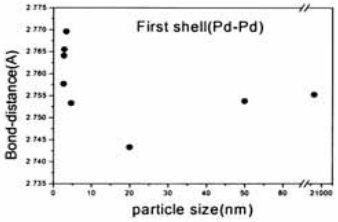

Fig. 2 The first neighborhood distance vs. the size in Pd nanocrystals.

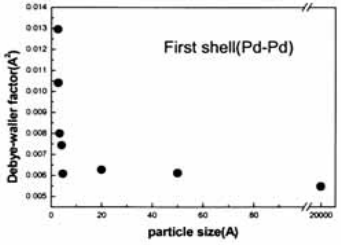

Fig. 3 The Debye-Waller factor of the 1st neighborhood distance vs. the size in Pd nanocrystals.

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Solvent Extracted Powder Diffraction Experiment

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$Fe(btr)_2NCS_2{\cdot}H_2O$ is a spin crossover complex in two dimensional framework with a hysteresis of about 25K ($T_{\downarrow 1/2}$ = 119.8K, $T_{\uparrow 1/2}$ = 145.1K). The existence of the water molecules is the vary reason for the spin crossover phenomenon which was first mentioned by W. Vreugdenhil et. al.[1]. This is consistent with our previous x-ray absorption studies. The crystal was destroyed while the water molecules were extracting out of the lattice. Therefore the main goal of this work is to solve the structure without water by the powder diffraction.

In order to see the difference between structures with and without water $Fe(btr)_2NCS_2{\cdot}H_2O$ was first measured at room temperature for 1 hour. The same sample was then heated up to about 115 °C by the heating gun and the data was collected for another 3 hours. All the experiments were measured in oscillation mode. The powder patterns were shown in Figure 1. It is apparent that the pattern does change a lot due to the water extraction.

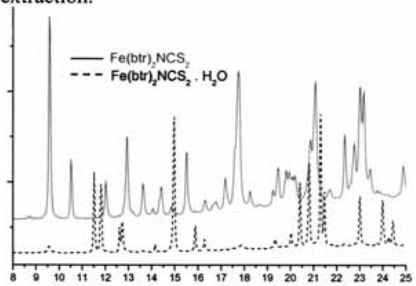

Fig. 1. Powder pattern of structure (---) with and (—) without packing water molecules.

The Rietveld refinement was performed by Jana2000 program taking the angle dependent zero-point shift correction by using Acosθ + Bsin2θ function. Certain constrains on btr ligand were applied in the refinement of $Fe(btr)_2NCS_2{\cdot}H_2O$, the fitting results are shown in Figure 2 and Table 1. Slight enlargement of the unit cell was observed in all three directions. The mismatch between data points and calculation lines in the range between 20 ~ 22° (2θ) indicate that the refinement might be not good enough so far. The further refinement is in progress.

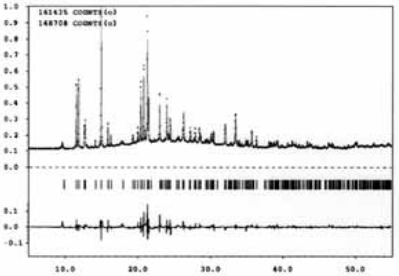

Fig. 2. Results of Rietveld refinement. (+): Experiment data points. (—): Calculation of fitting model.

The indexing in the powder pattern of the species without water molecules is still now in process. The change in structure seems to accompany with the decreasing in symmetry.

Table. 1. Comparison between structures obtained from previous in-house measurement and spring8 powder pattern.

Formula	$FeC_{10}N_{14}OH_{10}S_2$	
Formula Weight	Crystal	Powder
λ (Å)	0.71073	1.45855
Temperature	293	293
Space Group	C 2/c	C 2/c
a (Å)	11.116(1)	11.3232(2)
b (Å)	13.173(1)	13.3992(2)
c (Å)	13.073(2)	13.2833(4)
β (°)	91.851(1)	92.016 (3)
V (Å³)	1913.4(4)	2014.10(7)
sinθ/λ	0.595	0.317
# meas. Points / param.		2501 / 88
Rp	0.033	0.063
wRp	0.042	0.043
Rf		0.065
wRf		0.043

[1] W. Vreugdenhil, S. Gorter, J. G. Haasnoot and J. Reedijk, *Polyhedron*, **1985**, *4*, 1769