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Characterization of electronic configuration and structural differences on some special spin transition and mixed valences metal complexes (Zero-point Calibration and Charge Density Case Study)

I. J. Hsu[1] (6948), Y. C. Lin[1](9079), B. N. Wang[1](14681), C. F. Sheu[1](9194), Yu Wang[1*](6950)
Jey-Jau Lee[2](6947),Mau-Tsu Tung[2](4865)
[1]Department of Chemistry, National Taiwan University, Taipei, 106, Taiwan, R.O.C.
[2]Synchrotron Radiation Research Center, Hsinchu, Taiwan, R.O.C.

The major purposes of this study are (1) to do zero-point shift calibration of IP curvature using standard compound LaB_6, (2) to collect low temperature $KNiF_3$ powder data for charge density analysis, and (3) to collect low temperature data of spin crossover compound for structure determination. Following is a short summary of the present results.

The angle dependent of zero-point shift of LaB_6 is displayed in the Figure 1. Although the fitting results are good, yet we are not able to transfer the fitting coefficients to the data collected on other compounds. When the coefficients are applied to the data of $KNiF_3$ and do the Rietveld refinement, the fitting process is not successful at all. This implies that the coefficients are not transferable. It indicates that the deviation between two consecutive holder- mounting is probably larger than the instrumental error. Therefore, the best way to do zero-point shift correction is to use an internal standard, if possible. This implies we need to collect data with and without internal standard compound if we want to have high accuracy data for further analysis.

The Rietveld refinement of $KNiF_3$ is accomplished by using Jana2000 program, because it includes an angle dependent zero-point shift correction according to $A\cos\theta+B\sin 2\theta$ function. The results are listed in the Table 1. Although the fitting results are quite good, the total electron density map after maximum entropy analysis still gives out some not so smoothed surface at low-density level in comparison with the results obtained from theory. So far, we do not have a reasonable explanation for this yet.

The present instrumental resolution is about 0.02° limited by the present IP reader. For the present case studies, the FWHMs of the LaB_6 and $KNiF_3$ are 0.05° and 0.08° respectively below 2θ = 20° at wavelength of 0.55 Å. This indicates there are only 2 or 3 points within the FWHM range. In such case, the Rietveld refinement based on the profile may not yield the accurate intensity. If we want to do charge density analysis, the present IP resolution may not be suitable for these two samples.

For the spin crossover compound, the high spin state data is collected successfully at room temperature. The solvent extracted experiment was carried out but failed due to the incomplete dehydration and a mixed phase was detected.

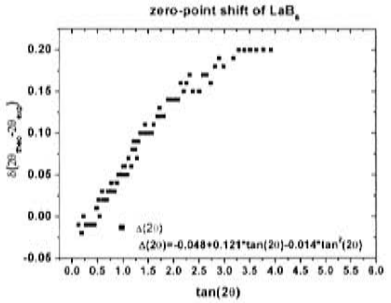

Figure 1 Angle dependent of zero-point shift. The square points are the differences between theoretical and experimental data. The solid line is the fitting results.

Table 1 Rietveld refinement results of powder $KNiF_3$ at 300K and 120K using Jana2000.

Formula	$KNiF_3$	$KNiF_3$
Formula Weight	154.80	154.80
λ (Å)	0.55	0.55
Temperature (K)	300	120
Space Group	Cubic, P m -3 m	Cubic, P m -3 m
a (Å)	4.02529 (4)	4.01402(1)
V (Å^3)	65.2216(6)	64.6753(2)
F(000)	74	74
$2\theta_{max}$(deg)	75	75
# meas. points	3491	3501
Parameters	50	50
# unique refins.	104	104
Rp	2.09%	2.32%
wRp	3.09%	3.47%
R_I	2.43%	2.00%
R_{wF}	3.03%	3.49%

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Anomalous X-ray Reflectivity Study of Fe/GaAs(001) Interface

Tae Chul Kim, Su Nam Kim, Do Young Noh
[1]Department of Materials Science and Engineering,
Gwang-Ju Institute of Science and Technology (GIST), Korea

Fe/GaAs(001) magnetic thin film has been extensively studied for applications in spin-injection device as a new memory system. However the role of interface formation for spin-transport is not clearly demonstrated by considering of magnetic anisotropy effect, elastic strain field, and chemical composition. We think that the composition of interface can play a crucial role in the interaction with ferromagnetic Fe film. Therefore, we focused on composition profile of the interface of Fe/GaAs(001) to determine the alloy structure at interface.

Anomalous x-ray reflectivity can be provided detailed information of buried interface. Using a monochromatic beam tuned to various x-ray energies at SPring-8, we measured x-ray reflectivity curve near absorption edge of Ga and As.

Figure 1 shows the anomalous x-ray reflectivity curve. The curve was normalized with Fresnel reflectivity and fit by Parrat formalism. To illustrate detailed information of the chemical structure at interface, we must consider the atomic alloy structure with varying atomic configurations. This work is now in progressing, but we previously performed normal x-ray reflectivity curve fitting to find a guideline of anomalous x-ray reflectivity curve fitting procedures.

To fit these curves, we setup a three layers model system, top layer is porous Fe oxide, and second layer is Fe and third layer is Fe/GaAs interlayer. Figure 2 show the model system and the fixed parameter such as roughness, Fe and GaAs reflective index with varying x-ray energy. After that the considering parameters at fitting procedures are reflective index of Fe/GaAs interface and porous Fe oxide.

Figure 3 shows the fitting results of delta values, directly related to the density of materials. At the Ga edge, the interface density is relatively enhanced than the quantity at the As edge. Therefore, we may consider Ga is relevant atom with Fe at forming interface-alloy than As. We are currently studying anomalous x-ray reflectivity fitting method. to reveal detailed structure of Fe/GaAs(001) interface.

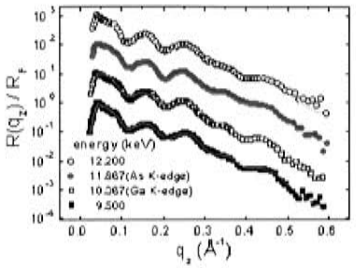

Figure 1 Anomalous X-ray reflectivity curve

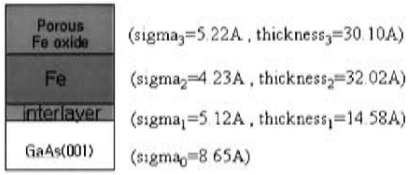

Figure 2 Fitting model system fixed fit parameteres

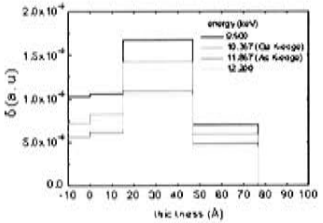

Figure 3 Fitting results of delta values