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The XAFS Phase Isolation and Characterization of Dispersion Phase Structure

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Abstract: According to Lu Kunquan's XAFS formula for mixing phase system, it is impossible to get the true structure of this kind of system by usual data analysis. A method which combines Lu Kunquan's XAFS formula with XRD was proposed to isolate XAFS of crystalline and dispersed phases. NiO/ γ -Al₂O₃ system is prepared by mixing NiO and completely dispersed NiO/ γ -Al₂O₃ and dealt by this method. The obtained coordination parameters of dispersed phase is just as same as the actual value. It demonstrates that the method of the phase isolation is reasonable and practicable.

1. Method

According to Lu Kunquan's XAFS formula [1] for mixing phase system, if the ions studied on a dispersion system exist in both dispersed phase and crystalline phase, the XAFS functions can be expressed as $\chi = f_c \chi_c + f_d \chi_d$. Here the f_c and f_d are the fractions of crystalline phase and dispersed phase respectively. It reveals the relationship of χ between the system and its ingredients.

For a dispersion system of oxides or salts, if the backscattering amplitude, phase shift and most coordination parameters etc. except the coordination number, have no considering changes in dispersing process, the XAFS formula of mixing phase can be simplified as $N_{\text{apparent}} = f_c N_c + f_d N_d$. This formula shows that this kind of dispersion system can even be regarded as a homogeneous system which can be treated simply.

But it also can be seen that it is still impossible to get the true structure from mere χ got from experiment regardless of dealing with single shell or multishell process. The major difficulty is that the fractions of ingredients are unknown generally. The principle of XRD phase quantitative analysis can be used to get the crystalline amount in a sample. Apparently, it can also be used to determinate the threshold (dispersion capacity) appearing crystalline phase and the fraction of surface phase in the samples by extrapolating the line of crystal phase of a dispersion system [2]. It is based on the assumption that when content increase, the dispersed amount will increase until it reaches the threshold, which is proved by many experiments. So, it can be supposed, if the Lu Kunquan's theory can combine with XRD phase quantitative analysis, it is possible to solve the problem of surface phase structure. Moreover, by measuring the XAFS of a series of samples above threshold, it seems possible to determinate the threshold and sample structure by isolation and data fitting, here we will not discuss it.

2. Results and discussion

Three series of NiO/ γ -Al₂O₃ system are taken as an example to study the isolation of surface phase XAFS in which the relative content of NiO crystalline phase are different. The first series was prepared by impregnation and calcination at 350°C. In the first three samples JL1-JL3, NiO exists completely in dispersed phase. The last two series were prepared by mixing a dispersed NiO with crystalline NiO mechanically. Their difference is that the dispersed NiO has not reached the threshold before mixing for the second series and in the third series, the dispersed amount has reached the maximum. If the total NiO content in sample is same as of other series, the second series has higher proportion of crystalline NiO. The contents of NiO, the relative amounts of crystalline phase were listed respectively in table 1.

According to the above phase isolation method, the apparent coordination numbers and the true coordination numbers of dispersed phase were got and shown in Figure 1 and table 1. For the last two series, the coordination numbers of dispersed phase of samples with higher NiO content are just the coordination numbers of the first points in the curves. The former is obtained by phase isolation, but the later were directly got from usual data analysis, since these two samples almost contain only the dispersed phase. This proves that the correction of above assumption and practicability of isolation and discrimination of the dispersion phase by phase isolation. It also indicates that the dispersion phase in the sample above the threshold has definite coordination state but it is affected greatly by NiO content for the samples below the threshold.

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Table 1: The contents of NiO, the relative amounts of dispersion phase and crystalline phase

No.	The first series					No.	The second series					No.	The third series				
	NiO content g/g	NiO content of crystal		App. coord. Ni-Ni	coord. No. of disp. phase		NiO content g/g	NiO content of crystal		apparent coord. No.	coord. No. of disp. phase		NiO content g/g	NiO content of crystal		apparent coord. No.	coord. No. of disp. phase
		g/g	relative con.					g/g	relative con.					g/g	relative con.		
JL1	0.05	0	0	0.37	0.37	JNA 1	0.25	0.10	0.4	6.1	2.1	JNA 5	0.44	0.12	0.27	6.1	3.9
JL2	0.10	0	0	0.61	0.61	JNA 2	0.35	0.20	0.57	7.8	2.3	JNA 6	0.55	0.23	0.42	7.3	3.9
JL3	0.15	0	0	1.98	2.0	JNA 3	0.44	0.29	0.66	8.7	2.4	JNA 7	0.60	0.28	0.47	7.7	4.0
JL6	0.30	0.04	0.13	4.3	4.1	JNA 4	0.60	0.45	0.75	9.6	2.4	JNA 8	0.70	0.38	0.54	8.3	4.0
JL10	0.50	0.24	0.48	7.3	4.1	JL3	0.15	0	0	2.40	2.4	JL6	0.34	0.02	0.06	4.4	4.0
JL11	0.55	0.29	0.53	7.8	4.2												
JL13	0.69	0.43	0.62	8.6	4.0

Figure 1: Comparson of JL and JN

A: Apparent coordination number and coordination number of dispersed phase of JL ~ NiO content
B: Apparent coordination number and coordination number of dispersion phase of JN ~ NiO content

Solid point: the third series Hollow point: the second series

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