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LOCAL STRUCTURE OF Cr CLUSTERS BY EXELFS SPECTROSCOPY

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Abstract: Crystalline structure of small chromium clusters, with diameters ranging from some tens to a few hundred Å, have been studied by extended energy loss fine structure (EXELFS) in the transmission mode beyond the $L_{2,3}$ edges of chromium using electron microscopy. The analysis of EXELFS data indicates a nearest-neighbor distance contraction of about 5% for the smallest clusters.

1. Experimental apparatus and Results

The experimental apparatus used in our EXELFS measurements (Cr $L_{2,3}$ edges) consists of a magnetic sector energy analyzer (Gatan 607) attached to a Philips EM430 transmission electron microscope operating at a primary beam energy $E_0 = 250\text{keV}$ with counting times of about 1 sec per eV channel. Clusters were prepared by electron-beam evaporation of chromium (99% pure) on copper grids, covered with a carbon layer and protected against oxidation by an ultrathin carbon film, in an ion-pumped high-vacuum system at a base pressure of about 3.5×10^{-6} Pa. The particle mean diameters were measured with an image analysing computer (IBAS2).

The EXELFS, superposed to a smooth background, above the core ionization edge is due to final state interference effects between the outgoing electron wave ejected from the ionized atom and the waves backscattered by the surrounding atoms [1]. The oscillatory part of the fine structure is given by:

$$\chi(k) = \sum_j \frac{1}{k} \frac{N_j}{R_j^2} F_j(k, \pi) e^{-2\sigma_j^2 k^2} e^{-2R_j/\lambda} \sin[2kR_j + \phi_j(k)]$$

where k is the ejected electron wave vector, N_j is the number of atoms in the shell j , R_j is the distance of shell j from the absorbing atom, σ_j^2 is the mean-square deviation of the interatomic distance R_j , λ is the inelastic mean-free-path and $F_j(k, \pi)$ is the backscattering function. After the data reduction [1], the Fourier integration of $\chi(k)$ directly gives the radial distribution function $F(R)$. The main peak of this function is related to the interatomic distance of the first surrounding shell from the excited atom minus a phase shift $\phi(k)$, due to the central and backscattering atom potentials [2].

We have verified that the analysis of Cr EXELFS signals beyond the $L_{2,3}$ is not affected by the presence of an interfering edge (L_1), localized at 120 eV higher in energy loss.

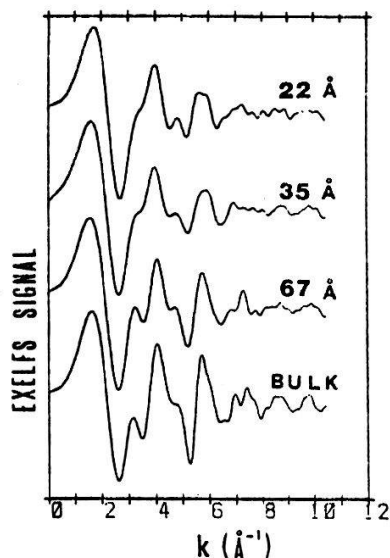


Fig. 1. EXELFS above the Cr $L_{2,3}$ edge

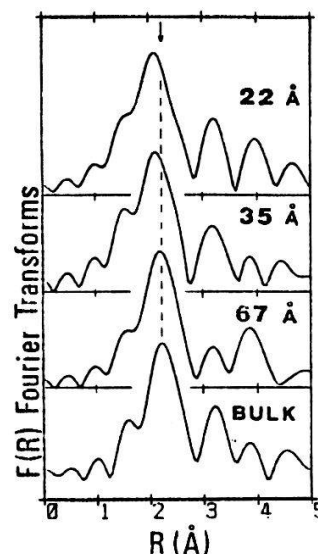


Fig. 2. Fourier transform of EXELFS

Figure 2 shows the radial distribution function obtained from the data of fig. 1. For the bulk, the main peak is located at 2.25 ± 0.02 Å, in good agreement with the results obtained by Leapman et al. [3]. It fairly corresponds, after the phase shift correction, to the bcc Cr lattice parameter ($R = 2.49$ Å) [4]. We note from Fig. 2 that the main peak position of the $F(R)$ shifts towards lower distances when the film thickness decreases. For thinnest films we observe a contraction of about 5.2% in the nearest neighbor distance.

This contraction is explained in terms of surface stress following the macroscopic liquid drop model, for which the contraction ΔR is given by: $\Delta R = -4KR_b f / (3D)$ where k is the bulk compressibility, R_b is the bulk nearest-neighbor distance and D is the particle mean diameter [5].

2. References

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