

XAS of BN once contacted with fluoride melts

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Introduction

In order to evaluate the chemical stability of BN matrix in sample pellet used for XAFS measurements at high temperatures, chemical environment around B and N atoms in the pellets are investigated by B-K edge (188 eV) and N-K edge (409.9 eV) X-ray absorption spectroscopy. Since K-edge XANES spectrum of B and N show clear dependence on the chemical environment around the absorbers [1], distinct change in the spectrum would be expected if BN matrix have chemically reacted to sample reagents and atmosphere.

Experimental

The B-K edge and N-K edge spectra are corrected by the total electron yield method. Samples are BN, B₂O₃, SrF₂+ BN pellet and EuF₃+ BN pellet, where the pellets have been used for high temperature XAFS measurements. The reason why these pellets are selected in that XAFS experiments on SrF₂ has been carried out at the highest temperature (1750 K) among our experiments and chemical instability of Eu³⁺ was confirmed in EuF₃ XAFS measurement at 1573 K. The emitted spectra are normalized by incident photon fluxes.

Results and discussion

These XANES spectra are shown in Fig. 1. As shown

in the figures, one can notice that small peaks in BN matrix powder in the pellets appear at ca. 194 eV. These peak are attributed to the transition of B-1s electron to unoccupied B-2p_z (π*) states for B₂O₃ [2]. Therefore, B₂O₃ can be produced by heating in the electric furnace. However, the broad specific peak at ca. 203 eV of B₂O₃ can not be confirmed in the pelleted samples. N-K edge spectra do not show any recognizable difference, and the shapes of the spectra are very similar to typical hexagonal BN structure [1]. Thus, the environment around a nitrogen atom must be unchanged by the heat treatment. Therefore, it is conjectured that the 1s → 2p_z (π*) transition peak in B-K XANES is emphasized even if a tiny amount of B₂O₃ is mixed in the sample. Consequently, the BN is enough stable at the high temperature region at which the XAFS measurements have been carried out [3].

References

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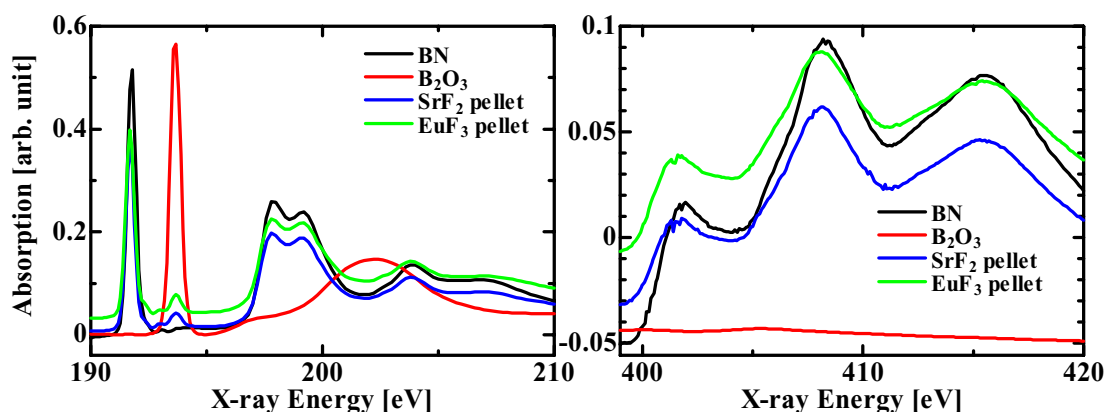


Fig. 1 B-K edge (a) and N-K edge (b) XANES spectra of standard sample (BN and B₂O₃) and used pellet (SrF₂, EuF₃ + BN) for high temperature XAFS measurements.

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