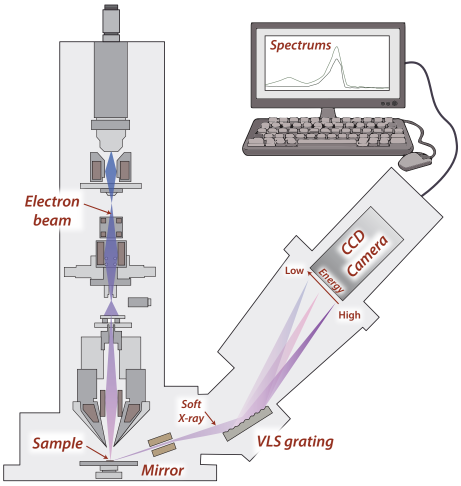
Soft X-Ray Emisssion Spectroscopy for EPMA/SEM application

Principle and features



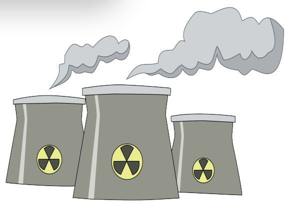
Characteristic X-rays are generated by the electron beam from the sample and guided, by the focusing mirror, to the newly developed diffraction varied-line-spacing (VLS) grating. X-rays diffract from the grating according to their wavelength; different characteristic X-rays are detected simultaneously by a parallel detection system (CCD camera) and displayed as a spectrum.

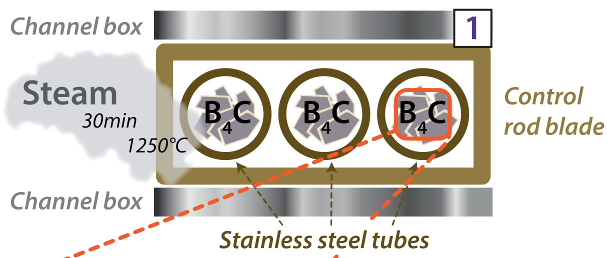
Benefits:

- **Extreme spectral resolution** → **chemical state analysis** of light elements
- Excellent **light element detection** (Li, Be, B, C, etc..)
- **High sensitivity** → a few Boron ppm in steel can be detected
- **High stability** and **reproducibility**
  → No moving parts, possibility to switch between gratings

Study case: chemical state mapping of degraded B_4C Control Rod

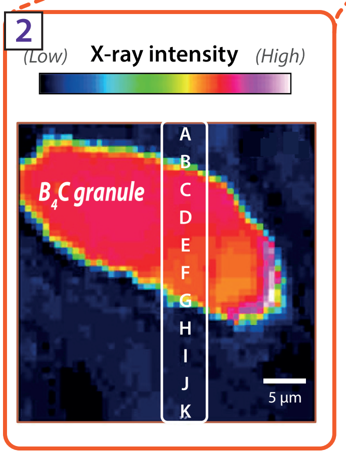
Control rods are used to contain the reaction of nuclear fission processes in power plants, such as in the Fukushima Daiichi nuclear power plant. Control rods consist of stainless steel tubes filled with B4C known for their excellent neutron absorption and high melting point. However, the melting point of B4C can be decreased due to interactions with stainless steel, resulting in its evaporation by reacting with steam at higher temperatures. In this context, a precise understanding of the location and chemical state of B in the fusion core is necessary to secure the nuclear plant.





- Methodology

A control bar model containing three sets of stainless steel tubes filled with B_4C was used for this study (Fig 1). The high temperature steam oxidation test was performed on the model for 30 minutes at 1250 °C. It was cut in the axial direction and then polished. Finally, elemental mapping on EPMA-SXES illustrating the chemical distribution of B was performed (Fig 2).

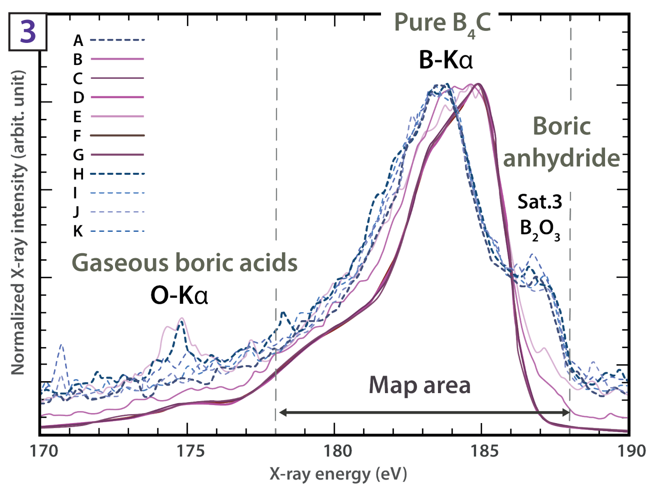


- Results: maps and spetcra

The map in Fig 2 clearly shows that the B_4C composition is no longer homogeneous after steam oxidation. A transect of spectra (points from A to K) intersecting the map vertically is shown in the graph of Fig 3. Two different chemical states are clearly identified by the SXES analyses:

- **1** Only one pure B_4C (materialized by the B-Kα peak Fig 3) granule remains in the center of the bar: red area in Fig 2 and continuous line spectra (*B, C, D, E, F and G*) in Fig 3.

- **2** Around the granule, the oxidation of B_4C generates different oxides that are formed by reacting with the stainless steel tubes: blue area in Fig 2 and dotted spectra (*A, H, I, J and K*) in Fig 3. Three types of oxides have been identified: boric anhydride (B_2O_3; sat.3 peak Fig 3) and gaseous boric acids (HBO_2 and H_3BO_4; O-Kα peak Fig 3).



- In summary

This study shows that SXES have an **excellent energy** and **spatial resolution** for boron, despite the fact that it is an element with a very low atomic number: two different **chemical states** are clearly identified (pure B_4C and three different oxides) at a **very high resolution spatial distribution**.

Ref: *Kasada, R., Ha, Y., Higuchi, T., & Sakamoto, K. (2016). Chemical State Mapping of Degraded B 4 C Control Rod Investigated with Soft X-ray Emission Spectrometer in Electron Probe Micro-analysis. Scientific reports, 6(1), 1-6.*