

Abstract

This work presents a comprehensive analytical study of the candidate first wall materials for nuclear fusion reactors, which include tungsten (W) based materials, and Transition metal borides (TMBs). It contains complementary elemental analysis spanning from depth profiling of thin film of borides to trace level detection in liquid samples.

Transition metal borides (TMBs) are a distinct class of ultra-hard refractory ceramics known for their hardness, high melting points, and resistance to thermal and chemical degradation. These properties make them well-suited for applications such as protective coatings, plasma-facing components in fusion reactors, and aerospace materials. Accurate boron detection in these thin-film materials is particularly important in nuclear fusion research, where boronization is used to condition the vessel surface and reduce plasma contamination. Standard electron probe techniques, including EDS and WDS, fail to reliably detect boron owing to its low atomic number, strong self-absorption of soft X-ray, and significant matrix effects, creating a critical gap in the characterization of TMB thin films and fusion wall deposits.

This thesis addresses the above-mentioned problem by applying Laser-Induced Breakdown Spectroscopy (LIBS) in the vacuum ultraviolet (VUV) spectral range, where boron produces strong emission lines that are otherwise not observed under atmospheric conditions. The VUV-LIBS setup achieves noticeably better sensitivity and accuracy for boron detection and quantification. The work also includes the application of LIBS to screws from the walls of the COMPASS tokamak, on which depth profiling and CF-LIBS were performed to analyze deposits. Additionally, an upgrade to an acoustic levitator-assisted LIBS configuration (LIBS-AL) is developed, in which the levitated droplet volume is determined to establish the preconcentration factor accurately, enabling trace boron detection in aqueous samples.

Beyond these core measurements, the thesis explores machine learning methods to extract chemical composition from dimensionality-reduced LIBS spectra of fusion materials. Alongside LIBS, X-ray scattering techniques, including Bragg-

Brentano diffractometry, grazing incidence X-ray diffraction (GIXRD), X-ray reflectometry (XRR), and texture pole figure measurements, are used to characterize the structure and phase composition of the boride thin films. Combining VUV-LIBS for compositional trend analysis with X-ray methods for structural and phase information produces a well-rounded analytical approach for studying TMB thin films, with practical implications for fusion research, materials science, and environmental studies.