

Studies of the self-absorption of boron doublet at 249nm for an accurate CF-LIBS analysis of diboride thin layers.

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With the increasing demand for lightweight refractory materials in the aerospace and machinery industries, transition metal borides (TMBs) have emerged as a promising solution. Coatings of TMBs, particularly those with an AlB₂-type crystal structure, have been shown to enhance material strength significantly [1]. This study aims to accurately quantify the elemental composition of various TMBs using Calibration-Free Laser Induced Breakdown Spectroscopy (CF-LIBS).

LIBS is a well-established technique for characterizing materials through atomic emission spectroscopy, offering rapid and versatile analysis. The sample analyzed is a thin film of zirconium diboride (ZrB₂) deposited on a silicon substrate. Plasma generation was achieved through laser ablation with an Nd:YAG laser operating at 1064 nm. Spectra were recorded at various gate delays and widths using an echelle spectrometer (ME5000 Andor tech.) with a bandwidth of 230-975 nm and a resolution of 4000. The experiments were performed in atmospheric conditions within a chamber designed for raster scanning of the sample. Upon thorough examination of the data hence obtained, the prominent spectral doublet of Boron I at 249.67 nm and 249.77 nm seems to exhibit self-absorption. To address this, an alternative approach involving experimentation in a vacuum UV spectral range under oxygen free atmosphere [2], which would allow observation of additional self-absorption free lines in the vacuum UV range using dedicated optics and vacuum setup. A preliminary study was conducted to observe and address the self-absorption of the aforementioned Boron doublet. An introductory analysis using a mixed TMB (ZrTaB₂) demonstrated accurate transition metal ratios (Ta: Zr), however, the estimation of boron quantity appears to be understated [6]. Accurate elemental quantification thus necessitates correction for the self-absorption of the doublet peak. Various analytical methods for self-absorption correction in LIBS have been utilized in the literature, including the Curve of Growth (COG) [3], Internal Reference Self-Absorption Correction (IRSAC) [4], and one-point calibration [5]. In this work, we are presenting the reconstruction of the self-absorbed peak that remedies the inaccuracy caused by self-absorption and leads to an accurate quantitative analysis of the sample.

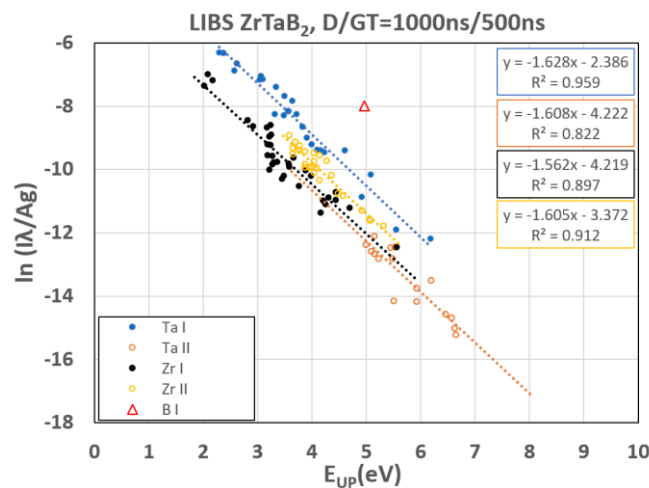


Fig. 1: Boltzmann Plot for ZrTaB₂ with only one peak for Boron from one of our recent works [6].

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