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Editors

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UNDER THE AUSPICES OF

prof. Ing. Michal Frivaldský, PhD. dean of Faculty of Electrical Engineering and Information Technology, University of Žilina,

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Introduction

The international conference on

ADVANCES IN ELECTRONIC AND PHOTONIC TECHNOLOGIES (ADEPT 2025)

is the 13th conference focused on the latest results of research and development in the area of novel materials, structures, devices and systems for micro/nano- electronics, sensors and photonic solutions. Its goal is to bring together leading experts with young generation of researchers from universities, as well as institutes interested in progress of advanced technologies.

The conference attracted young and senior scientists from Slovakia and other countries to submit 69 abstracts. In review process 64 papers have been accepted for publishing in conference proceedings as 63 contributed and 1 invited paper divided in 36 oral and 28 poster presentations. At this point, we would like to thank all reviewers for review activities and valuable comments.

The objective of ADEPT 2025 is to provide a forum for researchers, teachers and engineers involved in general areas of electronics, photonics and material engineering to disseminate their research results, exchange views on future research directions and to create partnership for further fruitful collaboration in specified fields. We believe that ADEPT will create this new space for scientist from field of electronics and photonics.

Editors

APPLICATION OF VACUUM UV LIBS FOR THE ELEMENTAL QUANTIFICATION OF SAMPLES CONTAINING BORON.

S. J. Shetty¹, M. Veis¹, T. Roch², P. Veis^{1*}

Abstract Laser-induced breakdown spectroscopy (LIBS) in the vacuum ultraviolet (VUV) region enables enhanced detection of light elements like boron in thin transition metal boride films. By performing LIBS within a vacuum chamber and collecting plasma emission using specialized optics like MgF2, emission lines below 200 nm are accessed, overcoming absorption and sensitivity limitations of conventional UV–NIR setups. This configuration improves the limit of detection (LOD) and signal-to-noise ratios (SNR) for boron. Implementing calibration-free LIBS (CF-LIBS) algorithms allows quantitative analysis without matrix-matched standards using the emission intensities [1]. When applied to thin films of transition metal borides, VUV LIBS achieves accurate elemental quantification, and these advances facilitate rapid, in situ monitoring of boron content in boride coatings critical for high-temperature and wear-resistant applications.

Keywords: Vacuum UV, LIBS, elemental quantification, Boron.

1. INTRODUCTION

Transition metal borides (TMBs) comprise a family of ceramic-like materials combining boron with early transition metals, belonging to groups 4-6 of the periodic table. These compounds exhibit an outstanding combination of high melting points (> 2,000 °C), extreme hardness (up to 35 GPa), excellent thermal and chemical stability, and favorable electrical and thermoelectric properties [2]. Among the various stoichiometries, the AlB₂-type TMB₂ phase has attracted special interest. This topology imparts high stiffness, good electrical conductivity, and remarkable resistance to corrosion and wear, making TMB₂ films and coatings promising for cutting tools, protective layers, electronic interconnects, and energy-conversion devices [3].

However, characterizing TMB thin films poses significant analytical challenges, predominantly due to the difficulty quantifying light elements such as boron. The mechanical performance, oxidation resistance, and electronic behavior of TMB2 are highly sensitive to the exact B:M ratio. Yet, conventional techniques, such as standard EDS and WDS, suffer from low sensitivity to low-Z elements, especially when present in nanometric films or at trace levels.

Laser-Induced Breakdown Spectroscopy (LIBS) has emerged as a powerful tool for rapid, in-situ elemental analysis with minimal sample preparation. While conventional LIBS is effective across a broad atomic number range, it is inadequate to detect boron in the UV–VIS–NIR region, whose intense emission is in the vacuum-UV range, which is absorbed by atmospheric oxygen. To overcome this limitation, experiments has to be carried out in vacuum chamber with inert-gas ambience, thereby preserving VUV radiation and enhancing detection limits for light elements. A similar approach by Veis et al.

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demonstrated quantitative measurement of B, C, Si, and S in He environments, employing Saha–Boltzmann analysis of multiple spectral lines to obtain plasma temperature and electron density, which underpin accurate concentration determination [4].

Complementary techniques, including X-ray diffraction (XRD), energy-dispersive spectroscopy (EDS), wavelength-dispersive spectroscopy (WDS), X-ray photoelectron spectroscopy (XPS), and Auger electron spectroscopy (AES), are often employed alongside LIBS to provide a more comprehensive characterization of boride thin films. These methods enable the verification of LIBS results and offer additional insights into the crystallographic structure, and surface composition of the materials.

The primary objective of this paper is to develop and demonstrate an advanced methodology for the accurate analysis of transition metal boride thin films, with a particular focus on the detection and quantification of light elements such as boron. By integrating VUV-LIBS with CF-LIBS algorithms and complementary XRD analysis, the study aims to overcome the limitations of conventional spectroscopic techniques and provide a robust framework for the characterization of boride materials. The research seeks to improve the limit of detection, enhance the signal-to-noise ratio, and enable efficient, calibration-free quantitative analysis, thereby facilitating the design and optimization of boride-based materials for critical technological applications.

2. METHODOLOGY

The plasma is generated by focusing a pulse generated by an Nd:YAG laser. The experiment is conducted in a vacuum chamber filled with inert gas to avoid absorption of VUV radiation. The collection of plasma emissions in the VUV range necessitates specialized optical components. Quantitative elemental composition is determined using Calibration-Free LIBS (CF-LIBS) methods [1].

2.1 Experimental setup

Fig. 1 shows the experimental setup for simultaneous VUV and UV-NIR LIBS. A Nd:YAG laser operating at 1064nm with a pulse width of 7ns and a repetition rate of 10 Hz was utilized to ablate the sample. The plasma radiation was captured by a Mechelle 5000 and McPherson 307 VUV spectrometer, both cascaded with a high-speed ICCD camera (DH437, andor Tech), which are aligned to different ports of the vacuum chamber. Additionally, a high-resolution spectrometer (THR 1500) was employed for accurate deterimination of emission lines. Dedicated optics made of MgF_2 are utilized to transmit VUV radiation from the plasma to the VUV spectrometer, owing to the absorption of VUV radiation incurred by conventional optical fiber cables.

2.2 Analysis, Results and Conclusion

Fig. 2 presents a comparison of LIBS simulated emission spectra from NIST LIBS database for different B ionizations. A high number of spectral lines in the VUV region for B neutral and first ionic lines against the number of lines in the UV region can be observed. The response curves of both Mechelle 5000 and McPherson spectrometers are shown in red and, together with simulated LIBS spectra information provide information that the most intense boron lines are detected in VUV range.

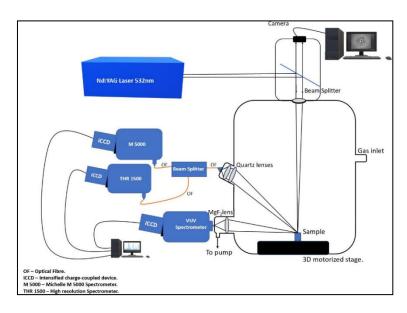


Fig. 5 Experimental setup for simultaneous VUV and UV-NIR LIBS.

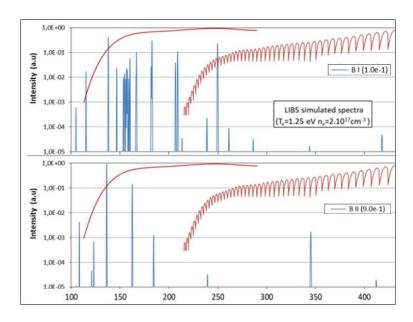


Fig. 6 Simulated VUV-NIR LIBS spectrum of Boron for plasma temperature of 1.25 eV and electronic density 2.10^{17} cm⁻³ with spectral sensitivity of VUV and UV-NIR echelle spectrometer (red curve) [4-5].

The majority of simulated Boron lines presented in VUV range are 3 orders of magnitude stronger that those presented in standard UV-NIR spectral range (except the Boron B I doublet at 249 nm). For the quantification of Boron one doublet could be sufficient if the laser induced plasma is in local thermodynamical equilibrium (LTE), but line should not suffer interference with other spectral lines and in addition these lines are resonant so often self-absorbed [6]. For testing the possible interference, LIBS spectra of (ZrB₂+TaB₂)/Si were measured in air at atm. pressure (Fig. 3).

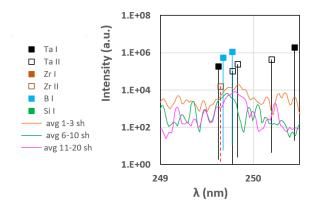


Fig. 3 Measured part of ns LIBS depth profile spectrum of (ZrB₂+TaB₂)/Si sample at to spectral position of only one detectable doublet of Boron neutral lines [6].

It can be concluded that for LIBS spectra of (ZrB₂+TaB₂)/Si, multiple lines suffer interference, and more results will be achieved through the combination of VUV LIBS.

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