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Depth-Resolved Detection of Deuterium in Tungsten-Based Materials Using High-Resolution LIBS

S. Atikukke[1*], M. Veis[1], P. Ďurina[1] , E. Grigore[2], F. Baiasu[2], P. Veis[1]

[1] *Comenius University, Faculty of Mathematics, Physics and Informatics, Bratislava, Slovakia*
[2] *NILPRP 409, 077125 Magurele, Romania ,*

*corresponding author: sahithya.atikukke@fmph.uniba.sk

INTRODUCTION

Tungsten based coatings are widely investigated as plasma-facing materials for fusion devices because of their thermal stability and role in impurity control. However, retention of hydrogen isotopes within these coatings can influence fuel inventory, material performance, and plasma–wall interactions [1,2,3, 4].

In this work, depth-resolved ns-LIBS was applied to deuterium-loaded W coatings deposited on Mo substrates. High-resolution Hα and Dα measurements were combined with confocal crater profilometry to determine the ablation rate, estimate the coating thickness, and evaluate the depth distribution of retained deuterium [5].

EXPERIMENTAL SETUP

A Laser-Induced Breakdown Spectroscopy (LIBS) setup was employed using a Nd:YAG laser (Brilliant Eazy,Quantel) operated at a pulse energy of 75 mJ and 8 ns pulse width. The laser beam was directed onto the sample surface using a laser-line mirror and focused inside an AtomTrace vacuum chamber using a focusing lens. Prior to the measurements, the chamber was evacuated and subsequently filled with argon at pressure of 2.5 mbar. The sample was mounted on a translation stage to allow controlled movement during ablation measurements.

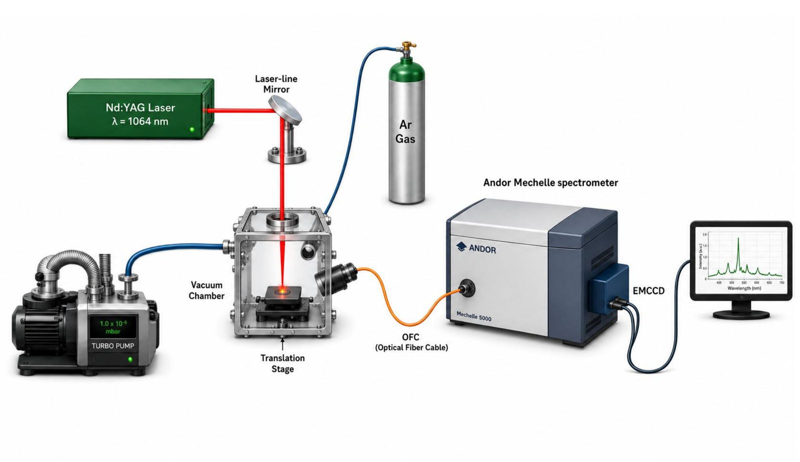

Figure 1: Schematic experimental setup using THR spectrometer

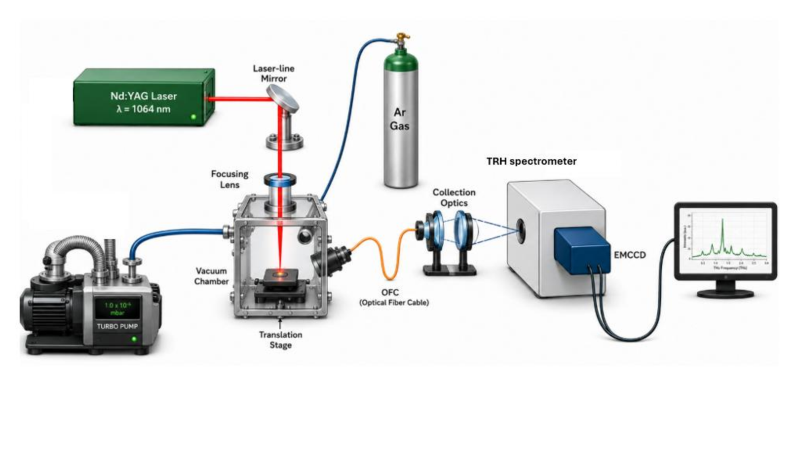

Figure 2: Schematic experimental setup using Andor Mechelle 5000

The plasma emission generated during laser ablation was collected using optical collection optics and analysed with two spectroscopic systems: a high-resolution THR spectrometer (HORIBA Jobin Yvon) equipped with an Andor EMCCD detector, and an Andor Mechelle 5000 echelle spectrometer for broadband spectral acquisition equipped with an ICCD (iStar DH743, Andor Tech). The THR system was wavelength-calibrated using Hg/Ar lamp lines, and different slit widths were investigated to optimize the spectral resolution.

Under the optimized conditions, a resolution of approximately 0.05 nm was achieved, enabling reliable separation of the closely spaced Hα and Dα emission lines. The high-resolution configuration was therefore used for hydrogen and deuterium analysis in the Balmer-α region, while the Andor Mechelle 5000 provided complementary wide-range spectral information from the W coatings. The investigated sample consisted of a W–Al–D coating containing 72% W, 18% Al, and 10% D deposited on a Mo substrate, with a nominal deuterium-containing layer thickness of 7.5 µm.

RESULTS

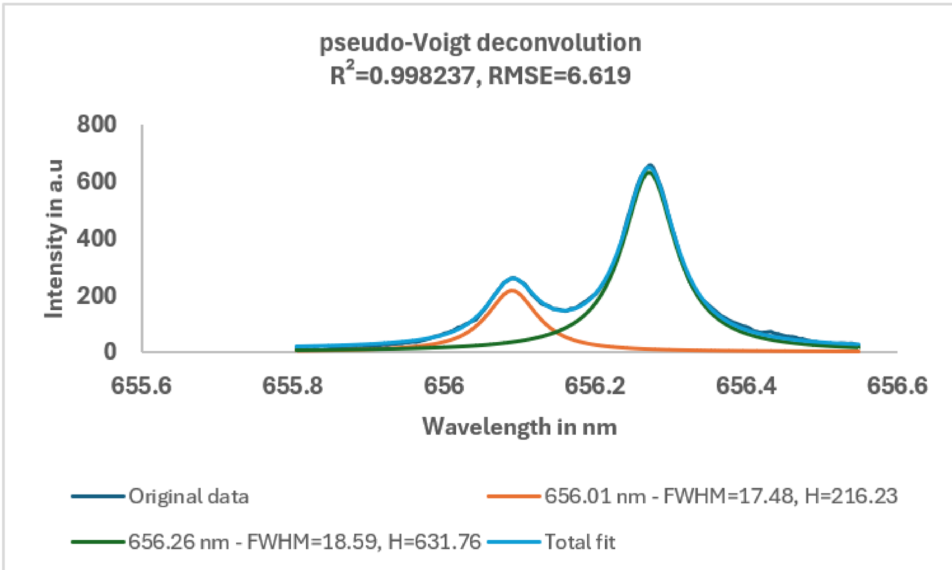

Figure 3. Pseudo-Voigt deconvolution of the Hα–Dα spectral region, showing the original spectrum, fitted components centred at 656.01 and 656.26 nm, and the resulting total fit. The fit shows excellent agreement with the experimental data.

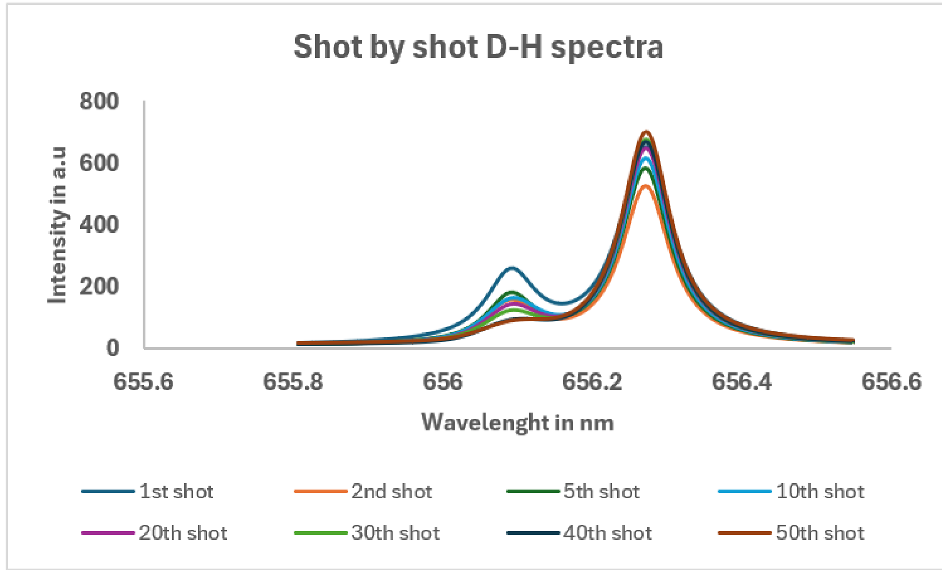

Figure 4. Shot-by-shot evolution of the Dα–Hα region, showing a progressive decrease in the Dα signal with increasing laser shots.

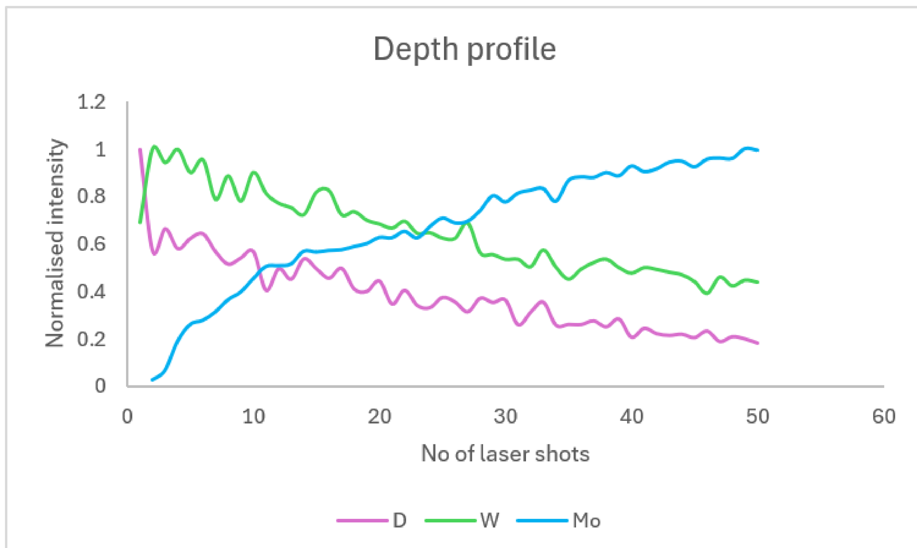

Figure 5. D, W, and Mo depth profiles measured using the THR spectrometer.

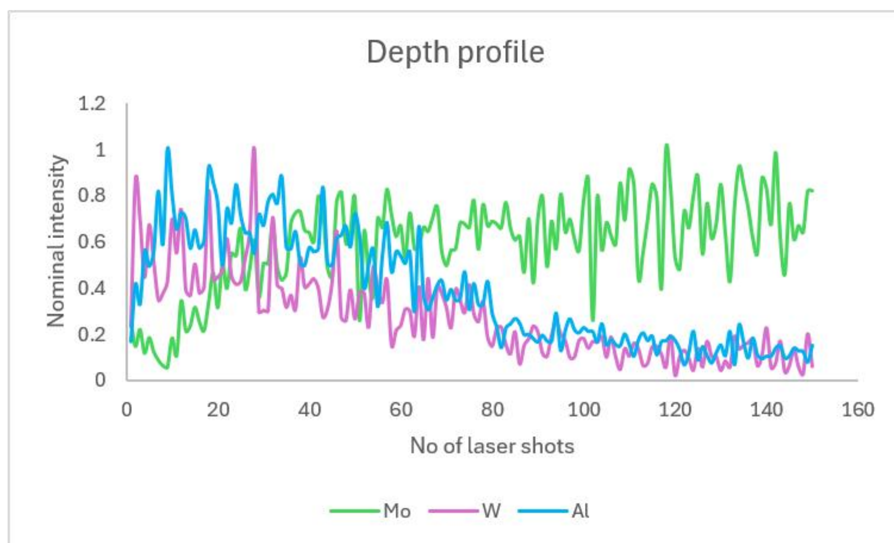

Figure 6. W, Al and Mo depth profiles measured using the THR spectrometer.

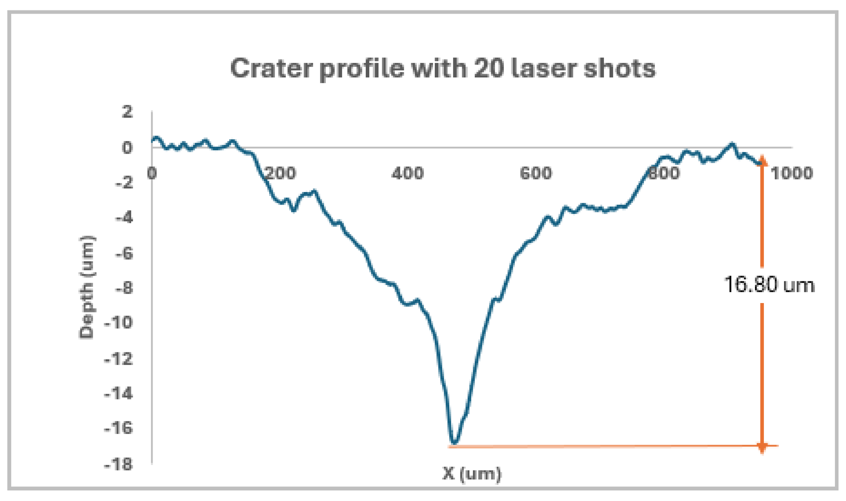

Figure 7. Crater depth profile after 20 laser shots, showing a maximum ablation depth of approximately 16.8 µm.

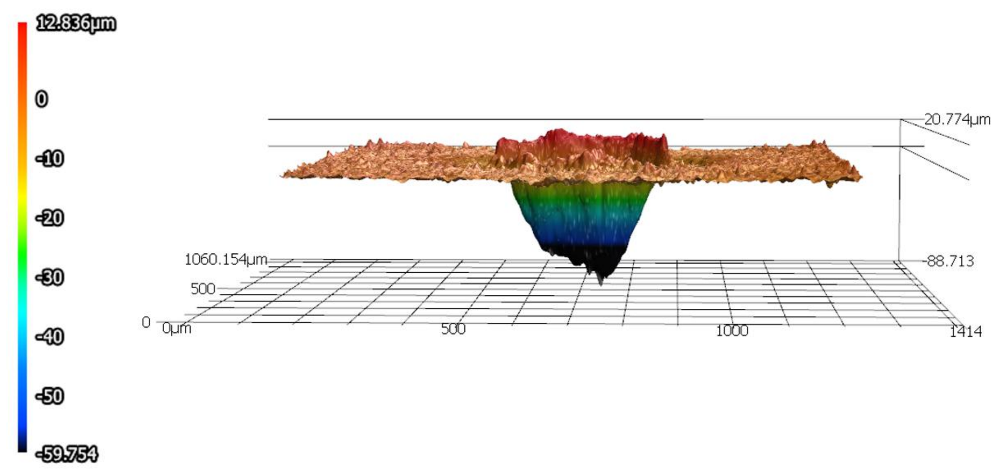

Figure 8. Crater depth profile after 150 laser shots, showing a maximum ablation depth of approximately 82.7 µm.

RESULTS:

Confocal profilometry showed that the crater depth increased almost linearly with the number of laser pulses. The slope corresponds to an average ablation rate of approximately 0.544 µm /pulse, which was used to convert the pulse-resolved LIBS data into a depth profile. High-resolution measurements in the Balmer- α region clearly resolved the Hα and Dα emissions, confirming the presence of retained deuterium in the W coating. The Dα signal progressively decreased with increasing pulse number and approached the background after 10 laser pulses, indicating that deuterium was predominantly confined to a near-surface region with an estimated thickness of 7.3 µm, compared with the nominal value of 7.5 µm, showing good agreement/a deviation of approximately 3% . The experimentally determined W and Al-coating thickness was approximately 18 µm. The appearance of Mo emission at greater depths further confirmed the transition from the W coating to the substrate. As a next step, quantitative analysis will be performed to determine the deuterium concentration and its depth-dependent distribution within the coating using the data from the broadband Andor Mechelle 5000 spectrometer .

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RECOVERY AND RESILIENCE PLAN

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