Exploring Light Elements in Steel via LIBS in the Vacuum-UV (VUV)

John T. Costello School of Physical Sciences & NCPST, DCU



IFATI4, Tokushima University, Japan, Nov 8, 2021.

Talk Outline



Ollscoil Chathair Bhaile Átha Cliath Dublin City University

- DCU Laser Plasma & AMO group
- LIBS Some History
- Why LIBS?
- Why LIBS in the VUV?
- VUV LIBS Experimental setup at DCU
- Single Pulse VUV LIBS Experiments on Steel (2000s)
- Double Pulse VUV LIBS Experiments on Steel (2010s)
- Some Recent VUV LIBS Results on Steel
- Current LIBS Focus and Future Further Developments

LP & AMO Physics Group at DCU (Pls)



Ollscoil Chathair Bhaile Átha Cliath Dublin City University John Costello (LP Spectroscopy & Free Electron Lasers) Emr. Eugene Kennedy (LP Spectroscopy & Synchrotrons) Jean-Paul Mosnier (Synchrotrons) Lampros Nikolopoulos (AMO Theory) Paul van Kampen (Physics Education/Synchrotrons)

Current Postdoctoral and PhD students:

Lazaros Varvarezos, James Campbell, Séamus Cummins, Stephen Durkan, Andrew Foremski, Ross McGarry, Adam Prior and Sadaf Syedah Zehra.

Recent PhD Graduates:

2021: Muhammad Bilal Alli, Hu Lu & Tejaswi Katravulapally 2020: Ben Delaney, Stephen Davitt & Lazaros Varvarezos,

Why Light Elements? e.g., Carbon in Steel...



Ollscoil Chathair Bhaile Átha Cliath Dublin City University Carbon steel is an iron- carbon alloy contains 0.005 to 2.1 wt.% carbon

Low Carbon steel (0.005-0.25 wt.%) - Low strength, high ductility -Applications automobile body components, structural shapes (Ibeams, channel and angle iron), pipes, construction and bridge components, and **food cans**.

Medium carbon steel (0.25 – 0.60 wt.%) - Medium strength and ductility - Applications railway tracks, train wheels, crankshafts, and gears and machinery parts requiring this combination of properties.

High carbon steel (0.60– 1.25 wt.%) - High strength , low ductility, Applications Due to their high wear-resistance and hardness, highcarbon steels are used in **cutting tools**, springs high strength wire and dies.

LP & AMO Physics Group at DCU (Pls)

Current (Ex-LIBS) Projects:

- 1. Photoionization of Atoms and Molecules in EUV FEL Fields
- 2. Ultrafast Optical Pump EUV Probes of Atoms and Molecules
- 3. VUV & EUV Photoabsorption via the Dual Laser Plasma Technique
- 4. Photoionization of Small Molecular lons at Synchrotrons
- 5. Development of an Ultrafast Laser Spectroscopy Laboratory

References

Ollscoil Chathair

Bhaile Átha Cliath Dublin City University

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- 2. Clocking Auger electrons, D C Haynes et al, Nature Physics 17 pp.512–518 (2021)
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- 4. Evolution of L-shell photoabsorption of the molecular-ion series SiHⁿ⁺ (n=1,2,3): Experimental and theoretical studies, E T Kennedy et al Phys, Rev. A 2018 97 043410
- 5. <u>https://www.dcu.ie/research/ultrafast-spectroscopy / http://uf-dynamics.org</u>

LIBS – Some History



Ollscoil Chathair Bhaile Átha Cliath Dublin City University *The first years of laser-induced breakdown spectroscopy,* M Baudelet and B W Smith, J. Anal. At. Spectrom. 28, pp624-629 (2013)



Fig 1. Basic LIBS configuration.

First (Early) LIBS elemental analysis reference: J Debras-Guédon et al. 1963 C. R. Acad. Sci. **257** p3336.

Le laser: Source d'excitation des spectres pour l'analyse qualitative et quantitative par spectrographie

Par J. DEBRAS-GUEDON, N. LIODEC et J. VILNAT, Société Française de Céramique, Paris, France

En concentrant au moyen d'une lentille le faisceau monochromatique (6943 Å) de forte énergie, issu d'un «laser» à rubis, sur des cibles de diverses natures et compositions, avec une durée de contact de l'ordre de 100 μ sec, on obtient non seulement une volatilisation locale de l'échantillon mais également une émission de photons. En recevant ces photons sur la fente d'un spectrographe, on a pu enregistrer des spectres de raies très complets dans lesquels on a même pu observer des raies d'atome ionisé (potentiel d'excitation 18 ev).

Dans certains cas, on a également obtenu des spectres de bandes moléculaires (CN, CO, Al.0)¹).

Ce type d'excitation convient particulièrement pour l'analyse ponctuelle, étant donné les très faibles dimensions de l'impact.

Avec une quantité minime de matière, on obtient grâce à la lecture des spectres enregistrés, un nombre considérable de renseignements sur la composition de celle-ci. La zone spectrale habituellement masquée par les bandes de cyanogène (dues à la combinaison de l'azote de l'air avec le carbone des électrodes, utilisées généralement pour l'excitation des spectres) se trouve dégagée. De plus, du fait de la non utilisation d'électrodes, aucune contamination n'est à craindre. On a pu ainsi identifier des taches de très petites dimensions sur des plaquettes émaillées. Des pointés effectués à différents endroits d'un échantillon permettent de tester son homogénéité.

On a montré également tout récemment qu'il était possible d'obtenir des résultats analytiques quantitatifs. On a ainsi dosé dans des aciers, du chrome à des teneurs comprises entre 0,02 et 1%, la précision obtenue permet de donner, par exemple: $0,91\% \pm 0,04$.

Ces essais ont été effectués avec un «laser» du Laboratoire Central des Télécommunications, et l'aide technique de MM. PAUTHIER et FRICHOT, de ce laboratoire.

LIBS – Some History



Ollscoil Chathair Bhaile Átha Cliath Dublin City University *The first years of laser-induced breakdown spectroscopy,* M Baudelet and B W Smith, J. Anal. At. Spectrom. 28, pp624-629 (2013)





Laser Probe Excitation in Spectrochemical Analysis 11: Investigation of Quantitative Aspects, S D Rasberry, B F Scribner and M Margoshes, APPLIED OPTICS 6, pp87-93 (January 1967) First (Early) LIBS elemental analysis reference:

J Debras-Guédon et al. 1963 C. R. Acad. Sci. 257 p3336.

Le laser: Source d'excitation des spectres pour l'analyse qualitative et quantitative par spectrographie

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LIBS – Some History: Growth in papers since 1980s

DCU Ollscoil Chathair Bhaile Átha Cliath

Dublin City University



Why LIBS?



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- Simple principle underlying a potentially powerful analytical technique: Spectral line wavelength acts as the identifying fingerprint of an element (classification) and the same spectral line intensity acts as a proxy for concentration determination
- Samples need little or no preparation, thereby removing the risk of contamination or loss of analyte associated with a preparation process
- LIBS can be made minimally ablative, so a negligible amount of the sample is destroyed
- Samples can be in any phase: Solid, liquid or gas, they can be conductors, semiconductors, or insulators

D A Cremers and L J Radziemski. *Handbook of laser-induced breakdown spectroscopy*. John Wiley & Sons (2013)

Why LIBS?





- Simple and economical experimental setup
- Highly flexible laser can be directed toward/focused on remote targets or targets in extreme or hazardous environments
 - Easy plasma light collection (mirrors, lenses & fibers)

D A Cremers and L J Radziemski. *Handbook of laser-induced breakdown spectroscopy*. John Wiley & Sons (2013)

Why LIBS in the VUV?

REMINDER.

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- **Qualitative analysis:** Each element (and ion-stage) in a sample produces a unique spectral signature, hence elements present in the target material can be identified
- Quantitative analysis: Elemental concentrations can (with effort) be determined from the intensity of spectral lines

But why work in the (awkward) VUV spectral range?

- 1. The spacing between spectral lines in the VUV region is significantly larger compared to the visible and infrared regions. As result the spectra are less cluttered with more distinct, and easier to select, spectral lines
- 2. Working in the VUV allows access to an abundance of resonance (connected to the ground state) transitions in atoms and ions at which are, in general, stronger than transitions between excited states. Hence one obtains spectra with better signal-to-noise ratio (SNR), and signal-to-background, ratio (SBR)

Why LIBS in the VUV?





>VUV gA values 1 to 2 orders of magnitude greater than vis-IR (e.g. S III and S I)

SIII

 λ (nm) gA (s⁻¹) lower upper 68.07 1.4x10¹⁰ 3s²3p² 3s²3p3d 286.35 5.5x10⁸ 3s²3p4p 3s²3p4d

SI λ (*nm*) *gA* (*s*⁻¹) *lower upper* 130.31 4.8x10⁸ 3s²3p⁴ 3s²3p³4s 861.72 1.6x10⁷ 3s²3p³4s 3s²3p³4p



Ollscoil Chathair Bhaile Átha Cliath Dublin City University Spectroscopy of Steels At Atmospheric-Pressure and in Air, J Belliveau, L Cadwell, K Coleman, L Huwel, H Griffin, Applied Spectroscopy 39 pp. 727-728 (1985)

Work on steel continues to today :

- 1. Aguilera J.A, Aragon C, Campos J., 1992 Appl. Spectrosc. 46 pp. 1382-1387.
- 2. Aragon C, Aguilera J.A, Campos J., 1993. Appl. Spectrosc. 47 pp. 606-608.
- **3.** J M Vadillo, C C Garcia, S Palanco, J J Laserna, 2009. Spectrochimica Acta Part B-Atomic Spectroscopy **13**, pp.793-797
- **4. P Porizka**, J Klus, D Prochazka, E Kepes, A Hrdlicka, J Novotny, K Novotny and J Kaiser, 2016. *Spectrochimica Acta Part B-Atomic Spectroscopy* **123**, pp.114-120.
- 5. Sturm V, Erben B, Fleige R, Wirz W., 2019 Opt. Express 27 pp. 36855-36863.
- 6. Zhang, Y., Sun, C., Yue, Z., Shabbir, S., Xu, W., Wu, M., Zou, L., Tan, Y., Chen, F. and Yu, J., 2020 Optics Express **28** pp. 32019-32032.
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 D Pedarnig, 2020, Spectrochimica Acta Part B-Atomic Spectroscopy 169, Article No. 105884
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Spectroscopy of Steels At Atmospheric-Pressure and in Air, J Belliveau, L Cadwell, K Coleman, L Huwel, H Griffin, Applied Spectroscopy 39 pp. 727-728 (1985)

Work on LIBS applied to *steel* continues to today:



Intensity (a.u.)*10³



Steel analysis with laser-induced breakdown spectrometry in the vacuum ultraviolet, V Sturm, L Peter and R Noll, Applied Spectroscopy 54, pp. 1275-1278 (2000) – Single channel (PMT) detection

Time integrated laser-induced plasma spectroscopy (LIPS) in the vacuum ultraviolet for quantitative elemental characterisation of steel alloys, M A Khater, P van Kampen, J T Costello, J P Mosnier and E T Kennedy, J Phys. D: Appl. Phys. 33, pp2252-2262 (2000) - IPDA



FIG. 6. Calibration curve for sulfur for the emission line 180.73 nm. For abbreviations, see Fig. 4.

TABLE II. Results of the calibration measurements with LIBS: limit of detection (LOD_{3s}), background equivalent concentration (BEC), concentration range of the calibration, and square of the correlation coefficient (r^2) .

Element	LOD_{3s} (µg/g)	BEC (µg/g)	Concentration range (µg/g)	r ²
С	7	530	0-1800	0.999
Р	9	640	0-600	0.998
S	8	210	0-500	0.987
Mn	9	320	0-14 000	0.999
Ni	6	500	0-700	0.991
Cr	7	330	0-1000	0.991
Si	11	680	0-6000	0.990



Figure 5. Relative emission intensity of the C^{2+} 97.70 nm line as a function of the carbon content. The axial distance from the target surface is 1.5 mm.

Table 4. Limits of detection for the five carbon lines used.

	Limit of detection (ppm)		
Line (nm)	Present work	Balloffet et al [58] ^a	
45.96 ш	148 ± 19	_	
68.73 п	208 ± 28	_	
90.41 п	130 ± 12	_	
97.70 ш	87 ± 10	100	
117.57 ш	154 ± 31	_	

^a Using the sliding vacuum spark.



Dublin City University

in Carbon Detection Limits, M A Khater, J T Costello and E T Kennedy, Applied Spectroscopy, 56, pp970-983 (2002) - CCD

Optimisation of the Emission Characteristics of Laser Produced Steel Plasmas in the Vacuum Ultraviolet: Significant Improvements

TABLE II. Detection limits for carbon in steels measured using various direct analytical techniques. Also shown is the *carbon* spectral line used in each case.

Author	Reference [year]	Method/detection limit, µg/g
Balloffet and Romand	28 [1955]	Vacuum Spark/100 (C(III) 97.7 nm)
Aguilera et al.	17 [1992]	LIPS/65 (C(I) 193 nm)
Khater et al.	1 [2000]	LIPS/87 ± 10 (C(II) 97.7 nm)
Sturm et al.	18 [2000]	LIPS/7 C(I) 193 nm)
Hemmerlin et al.	19 [2001]	LIPS/5; Spark/1 (C(II) 133.6 nm)
Khater et al.	Present [2001]	LIPS/1.2 ± 0.18 (C(III) 97.7 nm)









Ollscoil Chathair Bhaile Átha Cliath Dublin City University Eoin O'Leary, PhD Thesis, Dublin City University, (2007) VUV Laser-Induced Plasma Spectroscopy For Low Level Sulphur Detection In Steel (CCD)

Line: S⁴⁺ (78.65 nm) Sulphur/Steel in N₂ Background Gas



Limit of Detection of 1.7 ppm achieved using the S V emission line at 78.65 nm

Double Pulse VUV LIBS Experiments on Steel (2010s)





Fig. 1. Schematic of experimental apparatus. M1 and M2: Mirror 1 and mirror 2, L1 and L2: Lens 1 and lens 2, GG: gas gauge, Gr: grating, GCA: Glass Capillary Array, CCD: charge-coupled device.



Fig. 4. Recorded emission spectra under different pressures of argon. Double-pulse excita tion in the collinear reheating mode with an inter-pulse delay of 100 ns. $C^{\circ} = 0.041\%$.

Dual-Pulse Laser Induced Breakdown Spectroscopy with Ambient Gas in the Vacuum Ultraviolet: Optimization of Parameters for Detection of C and S in Steel, X Jiang, P Hayden, J T Costello and E T Kennedy, Spectrochimica Acta Part B: Atomic Spectroscopy **901** pp106-113 (2014)

Table 3

Measured data for the LOD calibration for C and S in steel under various conditions with the improvement factor denoted by α . DP: double-pulse, SP: single-pulse.

		Ambient	Linear fit	LOD	
Line	Mode	Gas type	R ²	(ppm)	α
C III	SP	Vacuum	0.9715	54.0	
		N^2	0.8749	9.7	
		Ar	0.9953	12.6	
		He	0.9607	17.2	
	DP	Vacuum	0.9883	13.6	3.97
		N^2	0.9786	2.9	3.34
		Ar	0.9978	3.6	3.5
		He	0.9880	5.7	3.0
S V	SP	Vacuum	0.9615	51.2	
		N^2	0.9890	7.3	
		Ar	0.9960	9.8	
		He	0.9869	12.4	
	DP	Vacuum	0.9721	8.9	5.75
		N^2	0.9980	1.5	4.87
		Ar	0.9937	2.4	4.08
		Не	0.9865	3.4	3.65



Syedah Sadaf Zehra, PhD Thesis, DCU (2021 - in correction), *Time Resolved and Time Integrated VUV LIBS for the Detection of Carbon in Steel* (CCD)

Change of focus – from point to linear laser plasma plumes



Spectral range: 10 - 35 eV (120 - 35 nm) / 1m ARC normal incidence, Andor BI-CCD, Nd:YAG: 6 ns, 460 mJ

From point to linear laser plasma plumes - benchmarking the DCU VUV LIBS System

DEU

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First the bad news !!

Single pulse LIBS – 4mm C(III) 97.7 nm [1]

In vacuo

Previous Studies

LoD = 54 ppm [1]

In air at 0.1 mbar

LoD =1.2 ppm [2]*

Double pulse LIBS in Vacuo – 4mm LoD = 13.6 ppm [1] Improvement Factor $\alpha = \frac{LoD_{sp}}{LoD_{dp}}$

Single pulse LIBS in Vacuo – 4mm C(III) 97.7 nm LoD = 316 ppm [3] $LoD = \frac{3\sigma_B}{S}$

This work

[1] X Jiang, P Hayden, J T Costello and E T Kennedy, Spectrochimica Acta Part B: Atomic Spectroscopy 901 106-113 (2014)
[2] M A Khater J T Costello and E T Kennedy Applied Spectroscopy, 56 970-983 (2002)
[3] SSZ Thesis, DCU (2021)

α = 3.97 [1]



Change of focus – from point to linear laser plasma plumes



Spectrum obtained with the target CRM 12 X 15252P with a carbon concentration of 940 ppm. Red arrow – the 11 pixels integrated around the C III 1s²2s2p $({}^{1}P_{1})$ to $1s^{2}2s^{2}$ $({}^{1}S_{0})$ line at 97.7 nm. Black arrow – the background was obtained by integrating 11-pixel values around 104.4 nm.

Change of focus – from point to linear laser plasma plumes

TISR VUV LIBS Emission for C/Steel Calibration



Bhaile Átha Cliath Dublin City University



Carbon in steel intensity calibration curve. Six different samples with carbon concentrations of 50 ppm to 940 ppm were used to construct the curve.

$$LoD = \frac{3\sigma_B}{S}$$

where σ_B standard deviation of the background

S slope of the calibration curve (slope sensitivity)

LoD = 50 ppm

TISR VUV LIBS - Line Plasma



Ollscoil Chathair Bhaile Átha Cliath Dublin City University

Case 1: TISR point plasma VUV LIBS target at 2 mm TISR point plasma VUV LIBS target at 4 mm LoD = 316ppm SBR= 2.8 $R^2 = 0.9998$

TISR line plasma **VUV LIBS** LoD = 50 ppmSBR= 4.4 $R^2 = 0.9998$ $\frac{3\sigma}{c}$ LoD =

Improvement Factor

 $\alpha = \frac{LoD_{TISR \ pointplasma}}{LoD_{TISR \ line \ plasma}}$

TISR VUV LIBS (Target at 4 mm)

LoD gain, $\alpha = 6.3$



ANC

Back to the Future – Single channel TR (SR) VUV LIBS

V Sturm, L Peter and R Noll, Applied Spectroscopy 54, pp. 1275-1278 (2000) – Single channel (PMT) detection



Time resolving VUV detector, showing the flange NW16 containing the plastic scintillator and photomultiplier tube (Tr < 5ns).



Back to the Future – Single channel TR (SR) VUV LIBS

Time Resolved LIBS Emission



Time resolved plasma emission at 97.7 nm (carbon line) and 104.4 nm (continuum) for the steel target with a carbon concentration of 2080 ppm.



Back to the Future – Single channel TR (SR) VUV LIBS

TR LIBS Emission – Carbon Calibration



Time resolved VUV LIBS calibration curve for spectra recorded with the *target located 2 mm from spectrometer optical axis.*

$$LoD = \frac{3\sigma_B}{S}$$

LoD = 56 ppm





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Some Conclusions on VUV LIBS

- 1. VUV LIBS has shown low single digit (in ppm) LoDs
- 2. The LoD is very dependent on laser, focusing conditions and target geometric parameters
- The absolute LoD is difficult to compare from experiment to experiment as 'S' is very dependent also on detector SNR, spectrometric system throughput and 'σ' on VUV photon and background fluctuations (Poisson), along with considerations in 2 above
- Line focus gains in (relative) LoD depend on having area detectors and quasi-stigmatic spectrometric optics to take advantage of the change in focus geometry
- 5. So a (VUV) LIBS std for steel is hard (not impossible)

Current LIBS Focus and Future Developments



Bhaile Átha Cliath **Dublin City University**

Theme: LIBS on 'Materials for the Energy Transition' **Focus:** Wind Energy (*Batteries, Hydrogen, Nuclear, etc.*)





insulators



EUVL Resist Materials (Synthesis & Dynamics)

Spectroscopy



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Current LIBS Focus and Future Developments

Early view: VUV LIBS Spectra of Selected Blades









Current LIBS Focus and Future Developments



Theme: *LIBS for Future Pharmaceutical PATs* Focus: ML- Assisted Chemometrics (P Hayden (now UCD) + IBM)

(NA) Multivitamin A) Disprin Units 0.8 Panadol Sweet Intensity Aribitrary L 0 70 70 90 108 98 100 102 104 106 110 112 Wavelength (nm) Intensity Aribitrary Units (AU) -Multivitamin B) Disprin Panadol Sweet 98 100 102 104 106 108 110 112 Wavelength (nm)

M B Alli, PhD Thesis, DCU (2021)

Table 5.4: % Accuracy ,comprehensive study, 160 spectra tested.

Wavelength Range			
Technique	65 nm	105 nm	Average
SOM 2x2	52.50	0.00	26.25
SOM 3x3	81.25	10.63	45.94
SOM 4x4	73.13	32.50	52.81
SOM 5x5	81.25	13.75	62.50
PCA	66.25	41.88	54.06
COMP	69.38	0.63	35.01
CNN	95.00	91.88	93.44

Acknowledgements – VUV LIBS Students/PDs





M Khater

E O'Leary

Xi Jiang



Muhammad B Alli





Sadaf Zehra

Ross

Stephen / Séamus / James





Ollscoil Chathair Bhaile Átha Cliath Dublin City University

Acknowledgements - Funding

Higher Education Authority – Programme for Research in Third Level Institutes (IV and V)

Science Foundation Ireland – Investigator, Infrastructure and Frontiers Programmes

Irish Research Council (PhD Scholarships / Postdoctoral Fellowships)

EU FP7 Erasmus Mundus Joint Doctorate 'EXTATIC' and Marie Sklowdowska Curie programmes

SEAI - Sustainable Energy Authority Of Ireland - Research, Development & Demonstration (RD&D) programme











Seai Sustainable ENERGY AUTHORITY OF IRELAND



Ollscoil Chathai Bhaile Átha Cliath **Dublin City University**

Some Conclusions of UV LIBS VUV LIBS has shown love Solution light (in ppm)

- 1. VUV LIBS has shown lov e digit (in ppm) LoDs
- 2. The LoD is very de focusing conditions and target geor
- 3. The absol are to mexperiment to expine 50 on detector resolu on VUV σ' on VUV Coisson), along with photon consi
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EXTRA SLIDES

EXTRA SLIDES





Deco Ollscoil Chathair Bhaile Átha Cliath Dublin City University

EXTRA SLIDES



EXTRA SLIDES



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