Tampere University

Nordic LIBS 2024

05-06 March 2024

Tampere, Finland

Welcome

Dear Colleagues,

Welcome to the first Nordic LIBS 2024 symposium!

We are delighted to host the first edition of Nordic Laser-Induced Breakdown Spectroscopy (LIBS) symposium in Tampere, Finland, renowned as the technology hub and "the sauna capital of the world". The event is scheduled to take place on March 5th and 6th, 2024. With around 70 participants from 15 countries, we believe that this symposium will mark a significant milestone as the first event in Northern Europe solely dedicated to LIBS.

We express our gratitude to all participants for their valuable contributions in sharing their work and presenting their latest accomplishments. We sincerely thank all invited and regular presenters as well as sponsors for ensuring the success of this event. We cordially acknowledge the support from Tampere University.

Thank you for being a part of our gathering! We hope that you will collect wonderful memories during your time here.

Sincerely,
Organising team of Nordic LIBS 2024

Dr. Vishal Dwivedi **Dr. Jan Viljanen** **Prof. Juha Toivonen**

Organisers

The organising team of Nordic LIBS 2024 is based at the **Applied Optics (AO) research group** at the photonics laboratory of Tampere University in Finland, led by **Prof. Juha Toivonen**.

Organising Team

Dr. Vishal Dwivedi
Postdoctoral Research Fellow

Dr. Jan Viljanen
Postdoctoral Research Fellow

Prof. Juha Toivonen
Professor

Tampere

Tampere, located in the southern part of Finland, is situated between lake Näsijärvi and lake Pyhäjärvi. Tampere is also known as '**the sauna capital of the world**'. Tampere is globally acknowledged as a technology hub and stands out as the most populous inland city in the Nordic countries. Serving as a focal point for cutting-edge technology, research, education, culture, sports, and business, it embodies a diverse spectrum of activities.

Venue

The Nordic LIBS 2024 symposium is scheduled to be held at the **City Centre Campus** of **Tampere University.**

Address: Kalevantie 4, 33100 Tampere

The conference sessions will take place in **Main Auditorium and Auditorium A1** on the 2nd floor of the main building.

Sponsors

LTB Lasertechnik Berlin GmbH
www.ltb-berlin.de

AtomTrace
www.atomtrace.de

Montfort Laser GmbH
www.montfortlaser.com

Spectral Industries BV
www.spectral-i.com

Ab Kimmy Photonics Oy
www.kimmy.fi

Avantes
www.avantes.com

PREIN- flagship, Finland
www.ablatom.com

Sponsors

ABLATOM SAS
www.ablatom.com

Norlab
www.norlab.com

Lightigo
www.lightigo.com

OXXIUS S.A.
www.oxxius.com

Thorlabs Sweden AB
www.thorlabs.com

Program

		Monday, 04 March
18:30	Welcome Evening Place: **Plevna**, Itäinenkatu 8, 33210 Tampere, https://plevna.fi/english/ https://maps.app.goo.gl/D1YrQFtBjWY7FjSk6	

*We would like to welcome you to Tampere by inviting you to the historic site of the first electric light in the Nordics! The **Plevna** brewery restaurant is located at the heart of the old industrial Tampere, offering freshly brewed refreshments and possibility to purchase German-style food, if feeling snacky. The evening reception starts at 18:30 and you will find us close to the entrance.*

		Tuesday, 05 March
08:45	Registration	
09:00	Welcome	**J. Toivonen (FI)**
Session- 1		(Chair: **J. Toivonen**)
09:15	Keynote *LIBS imaging : recent advances and review of breakthrough applications*	**V. Motto-Ros (FR)**
10:05	Coffee Break	
Session- 2		(Chair: **P. Veis**)
10:30	Invited *Automated mineralogy by LIBS for in-situ resource utilization*	**P. Pořízka (CR)**
11:10	Oral *The prospects of laser-Induced breakdown spectroscopy (LIBS) in the analysis of food*	**I. H. Boyaci (TR)**
11:40	Oral *Field-deployable LIBS system for ambient aerosol particle elemental analysis*	**P. Heikkilä (FI)**
12:00	Sponsor *Kimmy Photonics Oy*	**M. Halonen (FI)**
12:15	Lunch	
Session- 3		(Chair: **S. Kaski**)
13:15	Sponsor *LTB Lasertechnik Berlin (Robot-assisted mobile LIBS for geosciences and in-line quality control)*	**V. Merk (DE)**
13:30	Sponsor *AtomTrace a.s.*	**P. Bartko (CR)**

Program

13:45	Sponsor	**M. Sandtke (NL)**	
	Spectral Industries B.V.		
14:00	Sponsor	**S. Sørensen (DK)**	
	Norlab Oy		
14:15	Sponsor	**D. Kopf (AT)**	
	Montfort Laser GmbH		
14:30	Sponsor	**C. Elout (NL)**	
	Avantes B.V.		

14:45 Coffee Break

Session- 4 (Chair: **V. Dwivedi**)

15:10	Invited	**P. Veis (SK)**
	Calibration free LIBS for depth profile analysis of impurities, migrated material and retained fuel in fusion relevant materials	
15:50	Oral	**A. Hakola (FI)**
	Development of LIBS as a tritium monitoring tool for the JET tokamak	
16:10	Oral	**S. Mittelmann (DE)**
	Femtosecond LIA-QMS analysis for the detection of trapped deuterium	

16:30 Poster Session

17:45 Sauna evening and dinner# (Bus at 17:45, from symposium venue)
Place: **Varala**, Varalankatu 36, 33240 Tampere
https://varala.fi/en/saunas/
https://maps.app.goo.gl/rVk5LhS4q1eAG9687

*#The dinner and sauna will take place at **Varala** recreational center's sauna facilities. The sauna is located at the scenic waterfront of Pyhäjärvi Lake. There we have saunas and possibility for swimming/snowbathing, depending on the snow conditions. Please bring your own bathing suit if you want to go to the sauna and swimming. Towels are available at the sauna. A delightful buffet-style dinner with accompanying drinks will be served in the lounge area.*

Varala is located at a short drive from Tampere city center. For your convenience, a bus will depart at 17:45 from the venue with a return bus scheduled for 22:00. If you wish to leave early, there are two options: (i) Varala is next to a local bus stop (~3€ ticket to the city), (ii) using a taxi (~25€ to the city).

22:00 Bus transportation from Varala to Tampere city center

Program

			Wednesday, 06 March
Session- 5			(Chair: **P. Pořízka**)
09:00	Keynote	**R. Noll (DE)**	
	Bringing light into the process - contributions of LIBS for efficient and sustainable industrial production		
09:50	Coffee Break		
Session- 6			(Chair: **P. Suominen**)
10:20	Invited	**J. Kaiser (CR)**	
	LIBS for quantitative imaging of toxic metals in plant tissues		
11:00	Oral	**S. Kaski (FI)**	
	Applications of LIBS in material analysis		
11:30	Oral	**O. Balachninaite (LT)**	
	Comparative studies of ultraviolet and infrared femtosecond laser-induced plasma spectroscopy		
11:50	Lunch		
Session- 7			(Chair: **J. Viljanen**)
12:50	Sponsor	**S. Quetel (FR)**	
	OXXIUS S.A.		
13:05	Oral	**B. Sezer (TR)**	
	Comprehensive cheese analysis through singular laser-induced breakdown spectroscopy spectrum for multiparametric insight		
13:25	Oral	**K. A. Thorarinsdottir (IS)**	
	Use of LIBS analyzers in the aluminum industry and for metallurgical research		
13:45	Oral	**J. Peterson (SE)**	
	Rapid chemical analysis of slag at the production site		
14:05	Oral	**M. Sandtke (NL)**	
	Real-time LIBS analysis of drill cuttings		
14:25	Coffee Break		

Program

Session- 8		(Chair: **P. Heikkilä**)
14:50	Oral	**P. Suominen (FI)**
	RoboAI Green and the needs for industrial LIBS systems in Satakunta region	
15:20	Oral	**I. Laine (FI)**
	Technology of LIBS Imaging	
15:40	Oral	**J. Butikova (LV)**
	LIBS activities at the Institute of Solid-State Physics & Institute of Astronomy, Uni. of Latvia	
16:00	Oral	**J. Viljanen (FI)**
	Soil analysis with laser-induced breakdown spectroscopy	

16:20 Closing

16:30 Laboratory visit## (Bus at 16:30, from symposium venue)
Place: **Hervanta**, Korkeakoulunkatu 7, 33720 Tampere

##The laboratory visit is scheduled at the Hervanta campus (a hub of science and technology) of the University, approx. 7.5 kms from the conference venue. Our visit will include exploration of selected Optics & Photonics laboratories at Tampere University.

18:15 Bus transportation from Hervanta Campus to Tampere city center

Keynote Speakers

Vincent Motto-Ros
Institut Lumière Matière, Université de Lyon, France

Vincent Motto-Ros is an associate professor at the University Claude Bernard Lyon. His outstanding reputation within the global LIBS community stems from his notable contributions to LIBS elemental imaging and related advancements. With an extensive publication record, he is the author of over 120 papers in peer-reviewed journals, holds 2 patents, and has delivered around 70 presentations at national and international conferences. His impact is evident with over 4500 total citations and an impressive H-index of 43 (Google Scholar).

Reinhard Noll
LSA – Laser Analytical Systems & Automation, Germany

Prof. Reinhard Noll is currently Senior R&D Manager at LSA – Laser Analytical Systems & Automation, Germany. He is an excellent visibility among the global LIBS community for his significant contribution in integrating the LIBS method into standard industrial production lines. He has authored more than 100 papers, LIBS related books and book chapters, in peer-reviewed journals. He has over 4400 total citations and an impressive H-index of 32 (Scopus).

Invited Speakers

Jozef Kaiser
CEITEC, Brno University of Technology, Czech Republic

Prof. Jozef Kaiser, a renowned physicist specializing in techniques such as LIBS, LIF, X-ray tomography, etc., holds expertise in diverse spectroscopy methodologies. Apart from his academic role, he actively collaborates with several industrial entities, including Lightigo s.r.o. and Cactux s.r.o. He is esteemed member of various spectroscopy societies. His remarkable contributions extend in advancing LIBS research within the Czech Republic and the global scientific community. With an extensive publication record, he has contributed to about 300 research articles, gaining approximately 7500 citations and H-index of 46. (Google Scholar)

Pavel Veis
DEP, FMPI, Comenius University, Bratislava, Slovakia

Prof. Pavel Veis leads the LIBS research group at the department of Experimental Physics of Comenius University. He is also the director of the Ph.D. and Mgr. study programs at the faculty. His areas of specialization include laser spectroscopy (LIBS, LAS, CRDS, Raman), optical spectroscopy of gas and plasma, metastable states, and surface processes such as plasma wall interaction. With over 80 scientific publications to his name gaining above 1700 citations, he holds an H- index of 24 (Google Scholar). He is the national coordinator of the EUROfusion consortium and Euratom programme.

Invited Speakers

Pavel Pořízka
CEITEC, Brno University of Technology,
Czech Republic

Pavel Pořízka is an associated Professor at Brno University of Technology, Czech Republic. He is leading Laser Spectroscopy laboratory at CEITEC. He has been actively engaged in the LIBS research activities for about past 15 years. His focus has been on comprehensive development in all aspects of LIBS, including instrumentations, data processing, and current as well as future applications. His contributions extend to approximately 80 research articles, with more than 1500 citations and his H index of 23 (Google Scholar).

Speakers

Prof. İsmail Hakkı Boyaci
NANOSENS Industry and Trade Inc., Ankara, Turkey

Paavo Heikkilä
Aerosol Physics & Photonics Laboratory, Tampere University, Finland

Dr. Antti Hakola, D. Sc. (Tech)
VTT- Technical Research Centre of Finland

Dr. Steffen Mittelmann
Max-Planck Institute for Plasma Physics, Germany

Dr. Saara Kaski
Department of Chemistry, University of Jyväskylä, Finland

Dr. Ona Balachninaite
Laser Research Center, Vilnius University, Vilnius, Lithuania

Dr. Banu Sezer
NANOSENS Industry and Trade Inc., Ankara, Turkey

Dr. Kristbjorg Anna Thorarinsdottir
DTE, Iceland

Jonnas Peterson, PhD
Swerim, Sweden

Marijn Sandtke, CTO
Spectral Industries, The Netherlands

Pekka Suominen, PhD
Satakunta University of Applied Sciences, Finland

Ilkka Laine
Aalto University, Finland

Dr. phys. Jelena Butikova
Institute of Solid State Physics, University of Latvia, Latvia

Dr. Jan Viljanen
Photonics laboratory, Tampere University, Finland

Posters

No.		First Name	Last Name	Affiliation	Poster Title
P1	S	Paavo	Heikkilä	TAU, FI	*Field-deployable LIBS system for ambient aerosol particle elemental analysis*
P2		Steffen	Mittelmann	MPI, DE	*Femtosecond LIA-QMS analysis for the detection of trapped deuterium*
P3	S	Ilkka	Laine	Aalto, FI	*Technology of LIBS imaging*
P4		Banu	Sezer	NANOSENS, TR	*Comprehensive cheese analysis through singular laser-induced breakdown spectroscopy spectrum for multiparametric insight*
P5		Kristbjorg Anna	Thorarinsdottir	DTE, IS	*Use of LIBS analyzers in the aluminum industry and for metallurgical research*
P6	S	Doyinsola	Sonoiki	DTU, DK	*Analysis of multilayer marine protective coatings through femtosecond LIBS*
P7		Virginia	Merk	LTB, DE	*Elemental imaging of cacao beans with LIBS*
P8	S	Dominik	Bruzl	AtomTrace, CR	*Advancements in lithium quantification in geological samples using Sci-Trace/M-Trace LIBS technology*
P9		Melina	Gilbert Gatty	Swerim, SE	*Combined real-time temperature and chemical analysis of molten steels for production applications*
P10	S	Juuso	Lehtonen	SAMK, FI	*Investigating the impact of synthetic data on machine learning model performance in LIBS*
P11	S	Eetu	Ojanen	SAMK, FI	*Simulating plasma emission spectra for neural network training: integrating LTE states to correspond with observed LIBS spectra*
P12	S	Luis	Huaman	Aalto, FI	*Studies of LIBS-spectral fingerprints of lithium-ore minerals in different compositional matrixes*
P13	S	Jasper	Ristkok	UT, EE	*Laser-induced breakdown spectroscopy (LIBS) of hydrogen detection in molybdenum at variable gas pressures of argon or nitrogen*
P14		Sari	Romppanen	GTK, FI	*Portable LIBS as a tool to estimate the lithium potential of glaciated terrain*
P15		Matej	Veis	UNIBA, SK	*Calibration Free and Depth Profile LIBS Analysis of Transition Metal Borides*
P16	S	Shweta	Soni	UNIBA, SK	*OH radical analysis in a C_2H_5OH vapour RF discharge plasma for calibration of a VUV spectrometer*
P17		Vishal	Dwivedi	TAU, FI	*Field-deployment of LIBS device for rapid soil carbon monitoring*
P18		Vishal	Dwivedi	TAU, FI	*Elemental analysis and metabolic profiling of medicinally potent members of Zingiberaceae family using FT-IR and LIBS coupled with PLS-DA*
P19	S	Thu	Hoang	TAU, FI	*Moisture calibration in laser-based elemental analysis of soil*
P20		Jan	Viljanen	TAU, FI	*Total soil phosphorus detection using laser-induced fluorescence- assisted laser-induced breakdown spectroscopy*
P21	S	Joni	Ahokas	TAU, FI	*Utilizing laser-induced breakdown spectroscopy in lithium-ion battery recycling*
P22	S	Eetu	Haavisto	TAU, FI	*On-site monitoring of total nitrogen in Finnish soils using LIBS*

S: Student

Abstracts

LIBS imaging: a brief overview of breakthrough applications

V. Motto-Ros

Institut Lumière Matière, iLM UMR5306 Université Lyon 1-CNRS, 69622 Villeurbanne, France

Email: vincent.motto-ros@univ-lyon1.fr

The imaging capability of laser-induced breakdown spectroscopy (LIBS) has a high potential in various domains including biology, industry, geology and medicine (c.f. figure 1) [1,2]. This approach can be distinguished by its ease in use, multi-elemental capability, detection of light elements, as well as operation at ambient conditions. This is furthermore the only all-optical technique providing space-resolved elemental information with ppm-scale sensitivity and µm-range resolution. These advantages, make LIBS imaging very attractive to be used in research laboratories for routine investigations.
However, advanced technological solutions must be found for this application since elemental imaging requires high sensitivity, sharp spatial resolution, high speed of acquisition as well as the ability to process a huge quantity of data. In this presentation, we will summarize the recent progresses made in the Light and Matter Institute concerning the implementation of the LIBS imaging. In particular, different examples of breakthrough applications, such as biomedical (figure 1.a and b) or industrial (figure 1.c and d) will be shown with the aim of illustrating the specificities and the great potential of LIBS imaging. Different perspectives will be finally discussed.

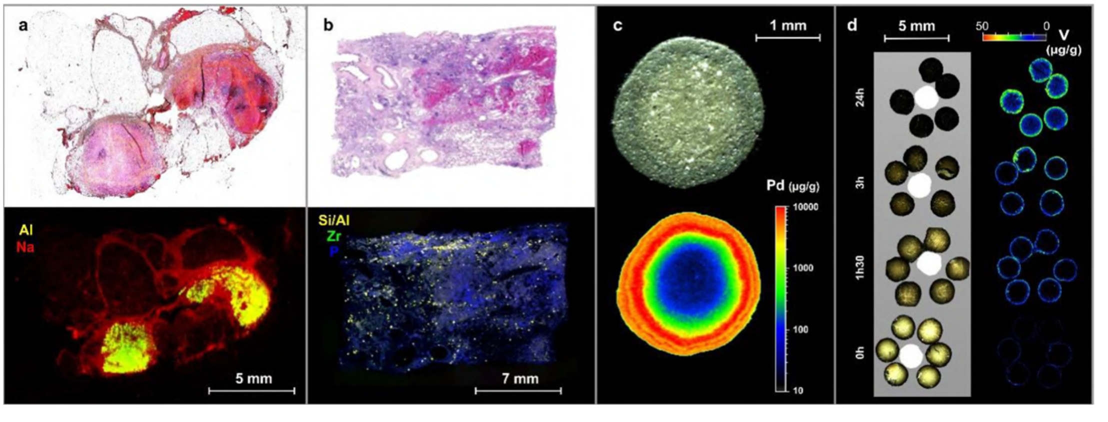


Figure 1. (a), Histologic analysis combined with elemental imaging that identified the metal responsible for a foreign body granuloma in a young patient at CHUGA. (b), Histological and multi-elemental images of a lung biopsy from a patient who worked in the World Trade Center rubble removal. (c) Demonstration of the distribution of the active element Pd of a catalyst supported on alumina, image representing a dynamic of 3 orders of magnitude in concentration. (d) Kinetics of impregnation of a heavy metal, Vanadium, during petrochemical treatments.

References
[1] B. Busser, S. Moncayo, J.-L. Coll, L. Sancey, V. Motto-Ros, Coordination Chemistry Reviews 358, 70-79 (2018).
[2] V. Gardette, V. Motto-Ros, C. Alvarez-Llamas, *et al.*, Analytical Chemistry 95, 49-69 (2023).

Bringing light into the process - contributions of LIBS for efficient and sustainable industrial production

R. Noll, J. Makowe, V. Moerkens, and M. Dargel

Laser Analytical Systems & Automation GmbH (LSA), Gewerbepark Brand 78, 52078 Aachen, Germany

Email: reinhard.noll@lsa-systems.de

Energy and material resources are used in the processing of raw materials, semi-finished and finished products, which are subject to ever greater restrictions in view of the climate crisis. As a result, the requirements for efficient and sustainable industrial production are constantly increasing. The term industrial production is also intended here to include all processes that are required for recycling materials, components and products, including approaches known as inverse production. A comprehensive data set with information on physical and chemical characteristics of the processed materials and goods is crucial for optimizing these processes. Laser-based measurements can shed light on the process by quickly and in-line determining the geometric and chemical characteristics of the processed materials/goods.

The suitability of LIBS for in-line analysis is known. The implementation for industrial applications requires the elaboration of procedures for optical access to the measuring object, control of measuring conditions, and tailored calibration procedures. An overview of LIBS systems routinely used in industry and LIBS systems under development is given: i) scanning high-speed LIBS for spatially resolved determination of microscopic element distributions in metals; ii) in-line monitoring of salt streams; iii) in-line characterization of scrap charges of steel as well as aluminum and lead scraps on conveyors; iv) in-line characterization of refractory materials and sorting.

Automated mineralogy by LIBS for in-situ resource utilization

P. Pořízka [1,2,3], J. Buday [1,2], J. Cempírek [4], J. Výravský [5], J. Vrábel [1,2], D. Prochazka [1,2], and J. Kaiser [1,2,3]

[1] *Central European Institute of Technology, Brno University of Technology (CEITEC BUT), Purkyňova 656/123, 612 00 Brno, Czech Republic*
[2] *Faculty of Mechanical Engineering, Brno University of Technology, Technická 2896, 616 69, Brno, Czech Republic*
[3] *Lightigo s.r.o., Renneská třída 329/13, 639 00 Brno, Czech Republic*
[4] *Department of Geological Sciences, Faculty of Science, Masaryk University, Kotlářská 2, Brno, Czech Republic*
[5] *TESCAN GROUP, a.s., Libušina třída 21, 623 00 Brno, Czech Republic*

Email: pavel.porizka@ceitec.vutbr.cz

This study explores the application of Laser-Induced Breakdown Spectroscopy (LIBS) for automated mineralogy in the context of in-situ resource utilization. As the interest in rare-element deposits is growing, especially in those, containing Li, the need for reliable detection and mapping of light elements (Li, Be, B, F) has increased. Traditional analytical techniques have certain difficulties for this specific application, as the main goal is to provide fast, easy and reliable detection, with the potential of in-situ application. However, Laser-induced breakdown spectroscopy (LIBS) combined with advanced data processing has been proven as a promising solution to this problem [1], [2].

In this work, we analyze four large-scale samples (up to 8x8 cm) using LIBS in combination with Energy-dispersive X-ray spectrometry (EDX) [3]. Utilizing the LIBS we observe space-resolved distributions of selected elements (major, trace, and light) within the whole samples as well as the impact of natural processes resulting in qualitative changes in the spatial elemental distribution. Thanks to the sensitivity of the LIBS, we can acquire spatial distribution of light elements as well, mainly Li or Be, which are out of the reach of the EDX.

Since the samples are composite of various matrices, the k-means clustering algorithm was applied to the acquired LIBS hyper-spectral images to determine different minerals and their transitions within the samples.

The LIBS analysis was extended by EDX due to its extensive libraries for minerals providing exact mineral information on the whole samples. The combination of these two methods reaps the benefits of speed, multi-elemental acquisition, in-situ analysis and detection of light elements for the LIBS, and accuracy and precision in the mineral determination of EDX. The acquired space-resolved results from these methods are combined in the form of cross-correlation for the purpose of detecting specific minerals as well as natural processes that were forming the rock samples. The study demonstrates very good applicability of LIBS for the detection and mapping of major (Si, Al, Fe, Mg, Mn, Ca, Na), light elements (Li, Be, B, F), and trace elements (Ge, Ga, Cu, Zn, Ti, Sr, Ba) in pegmatites, discerning their specific geochemical signatures during a magmatic, metasomatic, and hydrothermal stage of the rock evolution.

Acknowledgements
The authors acknowledge the support of the Czech Science Foundation (23-05186K), Technological Agency of the Czech Republic (FW06010042), and the Brno University of Technology (FSI-S-23-8389).

References
[1] A. Limbeck *et al.,* "Methodology and applications of elemental mapping by laser induced breakdown spectroscopy," *Analytica Chimica Acta*, vol. 1147. pp. 72–98, 2021.
[2] D. Prochazka *et al.,* "Joint utilization of double-pulse laser-induced breakdown spectroscopy and X-ray computed tomography for volumetric information of geological samples," *J. Anal. At. Spectrom.*, vol. 33, no. 11, pp. 1993–1999, 2018.
[3] T. Hrstka, P. Gottlieb, R. Skála, K. Breiter, and D. Motl, "Automated mineralogy and petrology – applications of TESCAN integrated mineral analyzer (TIMA)," *Journal of Geosciences (Czech Republic),* vol. 63, no. 1. pp. 47–63, 2018.

Calibration free LIBS for depth profile analysis of impurities, migrated material and retained fuel in fusion relevant materials

P. Veis [1], S. Soni [1], S. Atikukke [2], M. Veis [1], F. Baiasu [3], P.G. Bhat [1], N. Bolouki [4], P. Dvořák [4], V. Dwivedi [1,5], E. Grigore [3], A. Marín Roldán [1], S.J. Shetty [1], and WP PFC contributors*

[1] *Dept. of Exp. Physics, FMPI, CU, Mlynská dol. F2, 842 48 Bratislava, Slovakia*
[2] *DAPEM, FMPI, CU, Mlynská dol. F2, 842 48 Bratislava, Slovakia*
[3] *NILPRP 409, 077125 Magurele, Bucharest, Romania*
[4] *Dept. of Phys. Electronics, FS, Masaryk Univ., Kotlářská 2, Brno 611 37, Czech Republic*
[5] *Photonics Lab., Phys. Unit, Tampere Univ., Korkeakoulunkatu 3, 33720 Tampere, Finland*
**(see http://www.euro-fusionscipub.org/PFC)*

Email: pavel.veis@fmph.uniba.sk

One of the interesting feature of LIBS is its ability to perform depth profile analysis of thin layer on the surface of plasma-facing materials (PFMs) to quantify the impurities, deposited migrated material and retained fuel [1,2]. The research focuses on evaluating LIBS repro- ducibility over substantial distances and its capability to analyze thin film depth profiling on plasma-facing materials (PFMs). A comparative LIBS study of different W and Be based sample such as W-Zr, W-Cu is performed in two different laser time regimes; ns and ps [3-5]. The CF-LIBS quantification is performed by evaluating the plasma parameters (Te, ne). To enhance depth resolution, control ablation rates and minimise heat affected zone, ps-LIBS and resonant R-LIBS are introduced. For R-LIBS, a tunable OPO ns laser (Ekspla NT342C) is employed, while for ps-LIBS, a 30 ps laser at 532 nm (Ekspla PT403) is utilized. Samples with varying compositions of WTa and WTaD on a Mo substrate are measured under an Ar atmosphere at low pressure. LIBS spectra are acquired using an Echelle spectrometer (Mechelle ME5000, Andor Technology) equipped with an iCCD camera (iStar DH734, Andor Tech.). The results from ablation rated are compared with the standard GDOES method. The comprehensive analysis includes ns-LIBS, ps-LIBS, and R-LIBS for precise quantification and improved depth resolution. A critical aspect involves comparing the results of depth profiling with the standard Glow Discharge Optical Emission Spectroscopy (GDOES) method, providing valuable insights into the effectiveness of LIBS techniques for material analysis.

References:

[1] G.S. Maurya et al, J. Nucl. Mater. 541 (2020) 19.
[2] H. van der Meiden et al, Nucl. Fusion 61 (2021) 125001.
[3] A. Marín Roldán et al, Spectrochimica Acta Part B: Atomic Spectroscopy 177 (2021) 106055.
[4] A. M. Roldan et al, Fusion Engineering and Design, 172(2021), 112898.
[5] V. Dwivedi, The European Physical Journal Plus, 136(2021), 1177.
[6] S. Atikukke et al, Nuclear Materials and Energy 38 (2024) 101558.

LIBS for quantitative imaging of toxic metals in plant tissues

J. Kaiser [1,2,3], L. Čechová [1,4], D. Holub [1,2], P. Modlitbová [1], Z. Kozáková [4], and P. Pořízka[1,2,3]

[1] Central European Institute of Technology, Brno University of Technology (CEITEC BUT), Purkyňova 656/123, 612 00 Brno, Czech Republic
[2] Faculty of Mechanical Engineering, Brno University of Technology, Technická 2896, 616 69, Brno, Czech Republic
[3] Lightigo s.r.o., Renneská třída 329/13, 639 00 Brno, Czech Republic
[4] Faculty of Chemistry, Brno University of Technology, Purkyňova 118/464, 612 00, Brno, Czech Republic

Email: jozef.kaiser@ceitec.vutbr.cz

Toxic metals in the environment pose a great issue worldwide. Toxic metal bioaccumulation in soil can have negative effect on food safety, crop growth and health of soil organisms. Due to bioaccumulation of nutrient elements, toxic metals are bioaccumulated by plants as well. Plants grown in soil contaminated with metals such as cadmium, lead, mercury, or arsenic can exhibit growth reduction, lower biomass or altered metabolism [1].

Quantitative bioimaging of the exact distribution of toxic metals or metal nanoparticles by laser-induced breakdown spectroscopy is paramount for assessment of toxicity and health risks. It gives us information about spatial distribution of studied elements in the sample which enables a retrospective study of pollutant migration within the plant. It is possible to map the whole plants or only chosen parts of plant samples. Additionally, this method is capable of the detection of broad range of elements. Therefore, it is possible to not only study the distribution of toxic metals, but also effect of toxic metals on bioaccumulation and distribution of important macro and micronutrients [2,3].

In this study, LIBS was used as a complementary method to the ecotoxicological tests. After exposition to the contaminated solutions in hydroponic tests, macroscopic toxicological endpoints were measured. These endpoints are the length of the root, the stem, and the whole plant. Spatial distribution of toxic metals was demonstrated by LIBS.

Acknowledgements

The authors acknowledge the support of the Technological Agency of the Czech Republic (FW06010042), and the Brno University of Technology (FSI-S-23-8389).

References

[1] P.C. Nagajyoti, K. D. Lee and T. V. M. Sreekanth, "Heavy metals, occurrence and toxicity for plants: a review" *Environmental Chemistry Letters*, 8, pp. 199-216 (2010).
[2] P. Modlitbová, P. Pořízka, and J. Kaiser, "Laser-induced breakdown spectroscopy as a promising tool in the elemental bioimaging of plant tissues" *Trends in Analytical Chemistry*, 122, pp. 115729 (2020).
[3] T. Brennecke *et al.*, "Imaging the distribution of nutrient elements and the uptake of toxic metals in industrial hemp and white mustard with laser-induced breakdown spectroscopy," *Spectrochim Acta Part B At Spectrosc*, 205, pp. 106684 (2023)

The prospects of Laser-Induced Breakdown Spectroscopy (LIBS) in the analysis of food

Ismail H. Boyaci [1,2]

[1] Department of Food Engineering, Hacettepe University, Beytepe 06800, Ankara, Turkey
[2] NANOSENS Industry and Trade Inc., Ankara University Technology Development Zone, Golbasi, Ankara 06830, Turkey

Email: ihb@hacettepe.edu.tr

Food constitutes the principal reservoir of essential proteins, carbohydrates, fats, and minerals crucial for human nutrition and health. The quantitative composition of these components serves as a key indicator of the overall quality of the consumable, emphasizing the paramount significance of quantifying food constituents in ensuring safety and quality. The escalating need for comprehensive multi-component data in product monitoring underscores the imperative for swift and highly sensitive analytical techniques capable of discerning major and trace components with precision. Producers are increasingly adopting expeditious and dependable eco-friendly technologies to monitor food processes and final products, aligning with global concerns about food safety and the prevention of origin and quality fraud in the context of the globalization of food markets. Various analytical methods have been devised to address these concerns, but they pose primary technological challenges, often involving time-consuming, laboratory-scale methodologies with hazardous chemicals, meticulous pre-processing, and specialized personnel. The burgeoning popularity of rapid, straightforward, and pragmatic online analysis in the food industry is attributed to advancements in spectroscopic analysis. Laser-Induced Breakdown Spectroscopy (LIBS), among these methodologies, demonstrates the capability to ascertain elemental composition, presenting significant potential for expeditious food analysis. Within our research group, LIBS has shown promise in analyzing quality parameters and detecting food adulteration, such as identifying meat species, determining whey-adulterated milk powder, quantifying ash and protein contents in cereals, and analyzing sodium levels in bakery products. Amid the promising outcomes in LIBS applications to food analysis, this study explores future prospects for these methodologies and ongoing investigations concerning food safety and quality.

Keywords: LIBS, rapid analysis, spectroscopy, food safety

Field-deployable LIBS system for ambient aerosol particle elemental analysis

P. Heikkilä [1,2], A. Rostedt [1], J. Toivonen [2], and J. Keskinen [1]

[1] *Aerosol Physics Laboratory, Physics Unit, Faculty of Engineering and Natural Sciences, Tampere University, Tampere, 33720, Finland*
[2] *Photonics Laboratory, Physics Unit, Faculty of Engineering and Natural Sciences, Tampere University, Tampere, 33720, Finland*

Email: paavo.heikkila@tuni.fi

We present the Spectroscopy Platform for Ambient Aerosol analysis (SPAA), which analyses the elemental composition of individual aerosol particles utilizing laser-induced breakdown spectroscopy (LIBS). The SPAA can analyse particles directly from the surrounding air, without filter collection or artificial aerosol generation.
Conducting single particle analysis with LIBS is beneficial in multiple ways:

- Analysis within the carrier gas minimizes matrix effects.
- The energy required for single particle ablation is small, on the order of a few mJ, which leads to compact instrument design.
- LIBS can detect tiny masses of multiple elements with a single pulse, providing fast and detailed information of the aerosol population.

A challenge in analysing aerosols with LIBS is the fact that a particle must be in the same spot as the pulse laser focus. We have presented a method to overcome this challenge utilizing a linear electric quadrupole (LEQ) focusing system on a proof-of-concept level (Heikkilä et al. 2022). Now we present development in which the method has been field-deployed and has a wideband emission detection in the wavelengths of 200 – 1000 nm.
Future development includes increasing the analysis speed of the technique, which is presently at the order of 10 particles/minute. Also, increasing the optical throughput of the LIBS analysis to enhance the signal-to-noise ratio is important when approaching even smaller detectable elementary masses, which presently have a minimum at around 1-100 fg, depending on the analyte element.

This work was supported by the Maj and Tor Nessling Foundation and the Academy of Finland Flagship programmes ACCC and PREIN (decision numbers 337551, 357903, 320165).

References:

Heikkilä, P. et al. *Sci Rep* **12**, 14657 (2022). https://doi.org/10.1038/s41598-022-18349-8

Ab Kimmy Photonics Oy

M. Halonen

Kimmy Photonics, Finland
www.Kimmy.fi

Email: mikko.halonen@kimmy.fi

Our product range is broad and includes, for example, laser light sources, optical measurement systems, and photonics components. We collaborate with leading manufacturers worldwide and actively operate in the Nordic countries and the Baltics.
Some highlights of our product range include Litron Lasers. Litron Lasers specializes in manufacturing high-quality pulsed Nd:YAG lasers. Litron offers high customization of resonator types, allowing the beam quality to be tuned according to the application needed. They are also one of the few manufacturers offering ultra-high-energy pulsed lasers. Kimmy Photonics also provides turn-key femtosecond lasers, HeNe gas lasers, and an extensive selection of lasers chosen based on type, wavelength, power, and pulsed or CW mode of operation.
Our product range also includes confocal Raman imaging microscopes, laser vibrometers, optical surface profilers, digital image correlation material testing systems, laser safety products, digital microscopes, laser beam diagnostics, vibration control, acoustic cameras, high-speed and hyperspectral cameras and other photonics components.
We are keen to share our expertise with you. Feel free to get in touch with us; we are happy to help.

KIMMY

LTB Lasertechnik Berlin

(Robot-assisted mobile LIBS for geosciences and in-line quality control)

V. Merk

LTB Lasertechnik Berlin, Germany
www.ltb-berlin.de/en

Email: virginia.merk@ltb-berlin.de

In this talk we will present a new flexible LIBS system based on a compact measuring head CMH-66. The LIBS measuring head combines laser head, transmitting/receiving optics, sample observation and energy monitoring in one compact housing. Despite being conceived as lightweight at only 7kg and air-cooled only, the CMH-66 still excels with high-quality plasma formation and rapid spectral acquisition. When combined with an industrial robot, the measuring head enables automated LIBS measurements, making the LIBS technique suitable for industrial in-line monitoring. It is also very suitable for analyzing samples that otherwise couldn't be brought to a LIBS instrument. In the presentation we will provide details on how the CMH-66 works and also feature applications such as in-line quality assurance measurements of multi-material adhered surfaces and the analysis of drill cores, to show off the CMH-66s versatility.

Figure 1: LIBS measuring head CMH-66

AtomTrace a.s.

P. Bartko

AtomTrace a.s., Brno, Czech Republic
www.atomtrace.com/

Email: bartko@atomtrace.com

AtomTrace is a Czech vendor of products for chemical and quality analysis of materials using LIBS technology. Our goal is to design and deliver user-friendly and affordable solutions for all kind of users from scientific expert to operator on the production line. The range of application for our products is very wide from Universities and R&D centers through Material engineering to automatic on-line / in-line sensors in sectors such as automotive, mining, battery and food.
We offer following products:
1. Sci-Trace: modular system including unique Vacuum interaction chamber
2. M-Trace: mobile analyzer
3. AtomAnalyzer - SW for spectra processing
4. Tailor-made in-line / on-line analyzers

AtomTrace®

Spectral Industries

M. Sandtke

SPECTRAL Industries, Schieweg 15A, 2627AN Delft, The Netherlands
www.spectral-i.com

Email: m.sandtke@spectralindustries.com

SPECTRAL Industries is a high-tech company that develops unique LIBS systems for non-contact chemical analysis. Applications range from mining to recycling and material processing. SPECTRAL realizes new products by combining academic and practical knowledge and cooperating intensively with high-tech parties around the globe. We focus on realizing unique solutions, where the combination of robustness, mobility and accuracy is of paramount importance.
Scientific spectroscopy is a common tool in laboratories next to the production facilities. Based on sample taking, the production is monitored by these instruments. SPECTRAL envisions these labs will become redundant, as most analysis will take place in-line, during production. This does not require new knowledge – it requires the development of instruments based on existing technology that can operate in a 24/7 harsh environment. SPECTRAL Industries is a frontrunner in this transition from analysis in the laboratory towards analysis in the plant.

SPECTRAL
INDUSTRIES

Norlab Oy

S. Sørensen

Norlab Oy, Denmark
www.norlab.com

Email: simon.soerensen@norlab.com

Norlab is based out of Helsinki with a branch office in Copenhagen. we have over 30 years of experience in selling analytical instruments and we provide full technical support and service in the Nordic countries.
Norlab has a wide range of world leading instruments for material characterization with solutions in the fields of separation science, sample preparation, fluid handling and transfer, spectroscopy and material and pharmaceutical testing.

Montfort Laser

D. Kopf

Montfort Laser GmbH, Austria
www.montfortlaser.com

Email: dk@montfortlaser.com

MONTFORT Laser is specialized in **particularly compact pulsed laser sources** that are unique in their extremely small foot print and laser performance achieved. For example, more than **120 mJ energy in <8 ns laser pulses** are available from **<2 kg all-in-one lasers** including the driver, enabling next generation portable or mobile or even battery powered instrumentation in various fields.

Montfort Laser™

Avantes B.V.

C. Elout

Avantes B.V., The Netherlands
www.avantes.com

Email: c.elout@avantes.com

Avantes is the leading innovator in the development of fiber-optic spectroscopy instruments and systems with nearly 30 years of experience developing customer-defined spectrometer configurations.
With a long history of advising customers worldwide in various sectors and applications, Avantes is an experienced partner, equipped to guide customers who want a solution tailored to their application and research needs. By building best-in-class spectrometers and providing second-to-none customer service, Avantes offers customers the peace of mind that the solutions they purchase will meet, and exceed, their expectations.

Enabling Advances in Technology
Avantes offers spectroscopy solutions (with spectrometers, light sources and fiber-optic sampling accessories) that enable measurements in the ultraviolet, visible, and near-infrared wavelength ranging. Our technology supports a wide range of applications, from analyzing chemical composition and LIBS to measuring absorbance and radiometry.

OEM customers
For OEM customers (original equipment manufacturers), Avantes believes in a partnership approach to tailor our solutions and manufacturing processes to meet the needs of the end-use application.

Why choose Avantes for your LIBS application?
Multi-channel capabilities for high resolution
Excellent timing and triggering
Fast and reliable analysis through our spectroscopy software
Extensive knowledge on LIBS applications

Visit **www.avantes.com** to find more about the industries we serve, the measurement techniques we support, examples of applications and our products.

AVANTES
MEMBER OF THE NYNOMIC GROUP

Development of LIBS as a tritium monitoring tool for the JET tokamak

A. Hakola [1], J. Karhunen [1], J. Likonen [1] , I. Jõgi [2], P. Paris [2], S. Almaviva [3], and P. Veis [4]

[1] *VTT - Technical Research Centre of Finland*
[2] *University of Tartu, Estonia*
[3] *ENEA, Italy*
[4] *Comenius University, Slovakia*

Email: antti.hakola@vtt.fi

In future fusion reactors, thick co-deposited layers can be formed on their inner walls during extended plasma operations. Experiments in present-day fusion devices indicate these layers to consist of eroded wall materials, various impurities in the edge parts of the fusion plasma, and a fraction of the actual plasma fuel particles – most notably the hydrogen isotopes deuterium and tritium. Monitoring the inventory of the radioactive tritium in the reactor vessel is a particularly critical safety issue.

LIBS is one of the few techniques available for monitoring the tritium content and more generally the composition of co-deposited layers during reactor operations and maintenance breaks. The required technical solutions for a LIBS-based tritium monitoring diagnostics for fusion reactors are presently being investigated at the JET tokamak in the UK, now that JET has entered its decommissioning phase.

At VTT, together with the European collaborators, we have actively participated in the JET project and developed methods to assess the amount of deuterium and tritium in samples or wall components removed from JET and other fusion devices. Our system is capable of handling all kinds of materials, including the toxic beryllium presently used as a wall material at JET. Different quantification approaches such as calibration-free LIBS have been successfully tested and reported in laboratory conditions. Presently the technology is being transferred to JET, and the designed setup is in the commissioning phase at VTT. We will report the progress on the efforts and the results obtained from the first measurement campaigns.

Femtosecond LIA-QMS analysis for the detection of trapped deuterium

S. Mittelmann, W. Jacob, M. Mayer, Udo von Toussaint, T. Dürbeck, B. Buchner, and T. Höschen

Max-Planck Institute for Plasma Physics, Germany

Email: steffen.mittelmann@ipp.mpg.de

In the pursuit of learning more about trapped deuterium in plasma facing components of future fusion devices, we present the status of a novel experiment employing femtosecond Laser-Induced Ablation Quadrupole Mass Spectroscopy (fs-LIA-QMS). With regard to already existing LIA-QMS approaches with picosecond and nanosecond lasers this cutting-edge technique promises unmatched capabilities in characterizing material composition with high lateral and depth precision.

Our goal is to have another reliable diagnostic on hand, which is in a line with several other experiments like Nuclear Reaction Analysis (NRA) on our ion accelerator and a Thermal Desorption Spectroscopy (TDS) tool to be able to comprehensively study the behaviour of deuterium and other light nuclear fusion products in the mentioned plasma facing components. Moreover, in combination with Laser-Induced Breakdown Spectroscopy (LIBS) we might be able to find quantitative results on the deuterium content from the emitted plasma spectrum which is a key for an in-situ application of LIBS to the actual inner-wall of the magnetic confinement fusion vessel.

The content of this contribution is a presentation about the planned optical setup that aligns 400fs, 343nm, 150µJ laser pulses towards the studied samples. In a first approach these will be pure polished tungsten samples that are loaded by different techniques with deuterium. In addition, an overview on the design of the quadrupole mass diagnostic and our vacuum chamber is discussed. We aspire to make benchmark experiments on tests samples during this and the next year by gathering results from all the mentioned techniques.

Applications of LIBS in material analysis

S. Kaski

Department of Chemistry, P. O. Box 35 FI-40014 University of Jyväskylä, Finland

Email: saara.kaski@jyu.fi

The LIBS research at University of Jyväskylä has always focused on the innovative applications of laser-induced breakdown spectroscopy in material analysis. Already in the 1990s, LIBS imaging was used for paper analysis to survey the spreading of the coating mixtures in varied industrial conditions. The µLIBS approach with high spatial resolution (<1 µm in vertical (depth) and tens of micrometers in horizontal direction) has been used to construct the maps of the coating and filler distributions in macroscopic areas[1]. Also, the applicability of LIBS for the detection of halogens and sulfur in the vacuum ultraviolet (VUV)[2] and near infrared (NIR) spectral regions has been studied. Recently, we have focused on the optimization of data analysis for finding the critical raw materials; rare earth elements (REEs) and lithium bearing minerals from natural rock samples[3,4]. In this presentation selected results from LIBS and complementary techniques will be presented.

References:

1. Häkkänen, H. & Korppi-Tommola, J. UV-Laser Plasma Study of Elemental Distributions of Paper Coatings, Appl.Spectrosc. **(1995),** 49, 1721–1728. https://doi.org/10.1366/0003702953965894
2. Kaski, S., Häkkänen, H., & Korppi-Tommola, J., Laser-induced plasma spectroscopy to as low as 130 nm when a gas-purged spectrograph and ICCD detection are used, Appl. Opt. **(2003)** 42, 6036-6039. https://doi.org/10.1364/AO.42.006036
3. Romppanen, S., Häkkänen, H., & Kaski, S. Singular value decomposition approach to the yttrium occurrence in mineral maps of rare earth element ores using laser-induced breakdown spectroscopy. Spectrochim. Acta B **(2017)** 134, 69-74. https://doi.org/10.1016/j.sab.2017.06.002
4. Romppanen, S., Pölönen, I., Häkkänen, H. & Kaski, S., Optimization of spodumene identification by statistical approach for laser-induced breakdown spectroscopy data of lithium pegmatite ores, Appl. Spectrosc. Rev **(2023),** 58, 297-317. https://doi.org/10.1080/05704928.2021.1963977

Comparative studies of ultraviolet and infrared femtosecond laser-induced plasma spectroscopy

O. Balachninaitė, G. Zaremba, D. Paipulas, and V. Vaičaitis

Laser Research Center, Vilnius University, Saulėtekio ave. 10, LT – 10223 Vilnius, Lithuania

Email: ona.balachninaite@ff.vu.lt

Laser induced breakdown spectroscopy (LIBS) is a widely used versatile tool for elemental analysis of various materials, both in-situ and in the laboratory. Using femtosecond laser pulses for LIBS has many advantages, like reduced heat-affected zone, low continuum emission, lower ablation threshold and better repeatability. An important parameter of LIBS is the laser wavelength used for plasma induction. The choice of wavelength for LIBS measurements is thought to be more important for longer pulses, closer to the regime of nanoseconds, but in the case of femto - LIBS, the laser-matter interaction is different, the pulse duration is much shorter than material heat transfer time, there is no plasma shielding of the pulse. However, the dependence on wavelength for femto- LIBS has not been extensively studied.

In this work, the effect of femtosecond UV and IR laser radiation on laser-induced plasma characteristics on metals, ceramics and glass samples was investigated. The following plasma characteristics were evaluated: plasma temperature, electron density, spectral line intensity decay and signal-to-noise ratio. Plasma temperature and electron density had no discernible relationship with whether the plasma was induced using a UV or an IR laser wavelength. However, usage of femtosecond UV laser radiation for plasma induction increased plasma lifetime and significantly increased signal-to-noise ratio and correspondingly enhanced limits of detection on all samples studied. Since femtosecond UV laser radiation can be focused to a smaller spot, the sample damage is reduced, enabling higher spatial resolution in applications like LIBS chemical imaging.

OXXIUS S.A.

S. Quetel

Oxxius S.A., France
www.oxxius.com

Email: squetel@oxxius.com

Oxxius, the only French manufacturer of visible lasers, has excelled in the photonics sector for over 20 years, thanks to its innovations in biotechnology and life sciences.
Our product range of diode laser modules and DPSS lasers covers the entire visible spectrum between 375nm and 1064nm.
Our compact laser modules incorporate electronics that enable them to operate autonomously, while maintaining one of the lowest power consumption levels on the market. In biophotonics, our low-noise laser solutions are suitable for applications such as fluorescence imaging, genetic sequencing, confocal and 3D microscopy, optogenetics and more.
We also develop wavelength combiners for microscopy, customizable solutions that can be used on all existing microscopes.
In the field of measurement and spectroscopy, our single-frequency lasers meet the narrow spectral specifications required for applications such as interferometry, holography, Raman spectroscopy. For industrial applications, they are suitable for laser hardening, reprography, and food quality control.
Oxxius is committed to providing cutting-edge solutions, combining innovation, performance and reliability. Our aim is to continue to impact the field of photonics, making significant contributions in both scientific and industrial circles.

Comprehensive cheese analysis through singular Laser-Induced Breakdown Spectroscopy spectrum for multiparametric insight

B. Sezer [1,2] and İsmail H. Boyacı [1,2]

[1] Department of Food Engineering, Hacettepe University, Beytepe 06800, Ankara, Turkey
[2] NANOSENS Industry and Trade Inc., Ankara University Technology Development Zone, Golbasi, Ankara 06830, Turkey

Email: banusezer@nanosens.com.tr

This research explores the potential of Laser-Induced Breakdown Spectroscopy (LIBS) for predicting chemical quality and control parameters in a cheese matrix. Conventional methods for such analyses are often characterized by time-intensive procedures, intricate pre-treatments, and reliance on toxic chemicals, demanding specialized laboratories and personnel with specific analytical skills. In this study, traditional methods were employed to conduct 11 chemical analyses, encompassing moisture, dry matter, salt, total ash, total protein, pH, fat, acidity, water-soluble nitrogen, trichloroacetic acid-soluble nitrogen, and phosphotungstic acid-soluble nitrogen, on 82 full-fat, white pickled, and ripened cheese samples. Furthermore, the parameters of interest were cross-referenced with multi-elemental spectra obtained through LIBS, employing partial least squares regression for analysis. The findings demonstrated a substantial correlation between the chemical parameters analyzed through traditional methods and the LIBS spectra. This correlation suggests the feasibility of utilizing LIBS as either an alternative or complementary method for quality control in cheese production. The study underscores the potential applicability of LIBS in enhancing the efficiency and versatility of quality assessment procedures in the cheese industry, offering a viable alternative to conventional analytical approaches. The new method is expected to provide a simultaneous and multi-parametric analysis of cheese for quality control analyses to be carried out by the food industry and government agencies.

Keywords: LIBS, rapid analysis, spectroscopy, dairy analysis

Use of LIBS analyzers in the aluminum industry and for metallurgical research

K. A. Thorarinsdottir [1], M. Maghsoudi [1,2], A. V. Prudencio [1,3], and K. Leosson [1]

[1] DTE ehf., Lambhagavegur 5, IS113 Reykjavik, Iceland
[2] University of Iceland, Dunhagi 5, 107 Reykjavík, Iceland
[3] Department of Materials Science and Engineering, KTH, Stockholm, Sweden

Email: kristbjorg@dte.ai

We will provide an overview of our commercial deployment of Laser-Induced Breakdown Spectroscopy (LIBS)-based liquid-metal analyzers within the aluminum industry. DTE's state-of-the-art elemental analyzers have found applications in primary aluminum production plants, secondary production of aluminum alloys, and aluminum recycling facilities [1-3].
Not only can LIBS offer a unique real time analysis of the composition on the molten aluminum for the aluminum industry but can also give insight into metallurgical processes and kinetics that occur in aluminum alloys in the molten phase. Using DTE's metal analyzer we can study the formation and dissolution of native solid phases in liquid binary alloys on a time scale of seconds [4], the kinetics of transition metal boride formation, which, for example, can enable in-situ monitoring of the efficiency of boron treatment [5], as well as the behaviour of highly volatile alloying elements in the melt [6].

References

[1] K. Leosson, R. Meirbekova, S.H. Gudmundsson, G. Salloum-Abou-Jaoude, Light Metals 2022 (The Minerals, Metals & Materials Series. Springer, Cham, 2022), pp. 723–727.
[2] S.H. Gudmundsson, B. Björnsdóttir, K. Leosson, Light Metals 2021 (The Minerals, Metals & Materials Series. Springer, Cham, 2021), pp. 491–494.
[3] S.H. Gudmundsson, H. Gudmundsson, K. Leosson, Light Metals 2021 (The Minerals, Metals & Materials Series. Springer, Cham, 2021), pp. 758–762.
[4] K. Leosson, S.K. Padamata, R. Meirbekova, G. Saevarsdottir, S.H. Gudmundsson, Spectrochimica Acta Part B 190, 106387 (2022)
[5] A. V. Prudencio, M. Maghsoudi, K.A. Thorarinsdottir, K. Leosson, submitted to Light Metals (2024).
[6] K.A. Thorarinsdottir, M. Maghsoudi, K. Leosson, Applied Spectroscopy. 78(1):67-75 (2023).

Rapid chemical analysis of slag at the production site

J. Petersson, M. Gilbert-Gatty, D. Malmström, L. Hagesjö, K. Ekström, and A. Bengtson

Swerim AB, Box 7047, SE-164 07 Kista, Sweden

Email: jonas.petersson@swerim.se

Slag is used in steel production both to give the steel its desired properties as well as protecting the molten steel from air and prevent energy losses during the melting. The chemical composition varies between steel type and production step, but the main constituents are CaO, SiO_2, MgO and often also FeO and Al_2O_3. The slag has in its chorused form cement like properties which makes it suitable for several purposes. A simplified slag analysis can therefore be of use for several aspects: process control of the metal production, control of the slag for slag carry-over, and control of the slag for use as secondary raw material.
Swerim have done research with all the above purposes and used both commercial and home built equipment in lab, experimental furnaces and at various industrial facilities. This is further described in the references 1-3 below, and further results will be presented. A main challenge is to handle the heat of the samples both for its influence on the analysis equipment as well as the impact on the emission properties. Other challenges are the varied shapes of crushed slag and the inhomogeneity which makes the comparison with reference analysis difficult.

Figure 1. Examples of installations for slag analysis

[1] Petersson et al., Rapid chemical analysis of steel slag by laser-induced breakdown spectroscopy for near-the-line applications. *J. Anal. At. Spectrom*, **2020**, 35, 1848-1858
[2] Petersson et al., Utökad användning av slagg genom online övervakning av slaggprocesser, *Projektrapport inom innovationsprogrammet RE:Source*, Projektnummer 44320-2, **2022**,
[3] Petersson et al., Near-the-Line Steel Slag Analysis Using Laser-Induced Breakdown Spectroscopy: Traditional Univariate Versus Machine Learning Calibration Methods. *Applied Spectroscopy*, **2023**, Vol. 77(8) 9079–914

Real-time LIBS analysis of drill cuttings

M. Sandtke, J. Akushika, and A. van de Aa

SPECTRAL Industries, Schieweg 15A, 2627AN Delft, The Netherlands

Email: m.sandtke@spectralindustries.com

This presentation shows the design and realization of a LIBS-sensor that is able to measure in real-time the atomic composition of rock drill cuttings during mining operational drilling. The LIBS-sensor was a dedicated design meant to be placed directly on the boom of a drill rig. Shock loads of up to 10G and an ambient temperature range of –20 to +60 degrees Celsius had to be considered. A pre-calibration model for the elements of interest (Cu, Ni, Fe and Mg) was created off-line. A field campaign was arranged at an active Finnish mine to test the LIBS-sensor in real operation. During the field campaign, 19 holes (deeper than 20 meters) were drilled in an operational mining pit with the LIBS-sensor actively analyzing the drill cuttings during drilling. Using the pre-calibration model, it was possible to construct a depth profile of the elements of interest. This allows miners to monitor and differentiate the elemental composition of the ore and waste in real-time and show direct feedback on the shape and value of the orebody. Furthermore, the LIBS data can be used to validate the existing grade control model in real-time. Moreover, we show that the LIBS-sensor can survive the harsh mining environment. This study opens the door for the real-time chemical analysis of other applications in mining like face mapping or conveyer belt analysis of ore. The work presented was a close collaboration between Sandvik Mining & Rock Technology (Tampere, Finland) and SPECTRAL Industries (Delft, The Netherlands).

RoboAI Green and the needs for industrial LIBS systems in Satakunta region

P. Suominen and J. Valio

Satakunta University of Applied Sciences, Finland

Email: pekka.suominen@samk.fi, johanna.valio@samk.fi

RoboAI is a joint research and product development centre of Satakunta University of Applied Sciences (SAMK) and the University of Tampere. RoboAI has about 60 researchers, whose work focuses on regionally important strategic strength areas: automation and robotics, wellness and health technologies, intelligent systems, artificial intelligence, data analytics and optimisation. We complete up to 50 projects per year in collaboration with a wide range of industrial companies.

In 2022, RoboAI launched a new sub-division, RoboAI Green, which focuses on promoting the circular economy of technology metals and battery materials essential for the electrifying society.

The fundamental research question is how to efficiently and intelligently collect, identify and separate devices and components containing valuable technology metals to enable profitable and safe recycling. The main forms of the business-oriented and applied research are modelling, piloting and investigations.

We collaborate closely with a number of metal and battery processing plants in Satakunta. This review presents the identified needs and applications for industrial LIBS systems. The LIBS system developed in RoboAI Green laboratory is also presented.

Technology of LIBS imaging

I. Laine

Aalto University, Finland
Lumo Analytics Oy
libsimaging.net

Email: ilkka.laine@aalto.fi

At Aalto University we have built LIBS scanners capable of scanning large areas (drill core boxes, rocks, anything that fits on a table) quickly with high resolution and autofocus following any sample shape. My thesis presents this technology and what it can achieve.

Laser-Induced Breakdown Spectroscopy (LIBS), better named Laser Spark Spectroscopy, works by shooting a laser to make a spark and then gathering the light from the spark to a spectrometer which reveals all elements, and some molecules, present in the sample.

We present devices capable of scanning LIBS operation and megapixel resolution spectral images acquired with them. Secondly we share a helpful toolset for LIBS analysis from elemental heatmaps to mineral/material mapping using Interesting Features Finder (IFF) with Spectral Angle Mapping (SAM). With these we make beautiful elemental and mineral maps to help geologists do their work. Our methods has been proven in multiple ore exploration projects around Finland and the world.

My thesis presents LIBS Imaging technology in as simple and understandable way as possible, also in website form at libsimaging.net. I explain LIBS Imaging so that everyone will understand, aiming to create “LIBS Imaging for Dummies”, and give out a toolset for others to do their own LIBS analysis.

A spinoff company Lumo Analytics based in Helsinki offers drill core scanning and analysis services to mining industry. Our research team in Aalto University is happy to cooperate on any research effort interested in LIBS Imaging analysis.

LIBS activities at the Institute of Solid State Physics and Institute of Astronomy, University of Latvia

J. Butikova [1], J. Grūbe [1], A. Šarakovskis [1], L. Grīnberga [1], Rashid A. Ganeev [2], and Vyacheslav V. Kim [2]

[1] Institute of Solid State Physics, University of Latvia, Kengaraga 8, Riga, LV-1063, Latvia [jelena.butikova@cfi.lu.lv, jurgis.grube@cfi.lu.lv, anatolijs.sarakovskis@cfi.lu.lv, liga.grinberga@cfi.lu.lv]

[2] Laboratory of Nonlinear Optics, Institute of Astronomy, University of Latvia, Jelgavas iela 3, Riga, LV – 1004, Latvia [rashid.ganeev@lu.lv, vyacheslav.kim@lu.lv]

The Institute of Solid State Physics, University of Latvia (ISSP UL), has been actively participating in a research program within the EUROfusion Consortium. This initiative aims to tackle challenges related to the realisation of clean energy through thermonuclear fusion devices. Laser-induced breakdown spectroscopy (LIBS) emerges as a promising tool for the quantitative, in-situ determination of fuel retention in plasma-facing components (PFC) of magnetically confined fusion devices, such as ITER. Consequently, samples mimicking the PFCs have been analysed to identify the elemental composition of ITER-like deposits.

In the recent studies, the inclusion of different gases, including deuterium, helium, and neon have been determined in Mo:W samples. The depth resolution for ITER-like samples is from 50 nm up to 250 nm per single pulse.

In collaboration with the Institute of Astronomy, we have conducted highly sensitive qualitative analyses of meteorites and geological samples. Pilot studies have demonstrated the effectiveness and unique potential of LIBS in performing sensitive and time-efficient qualitative analyses of the composition of ores and meteorite samples.

Our LIBS system utilizes the fundamental harmonics (1064 nm) of the EKSPLA Nd:YAG laser with a pulse duration of 150 ps and maximum repetition rate 10 Hz. For the detection of spectral information, Andor's Mechelle spectrometer Me5000 coupled with iStar DH334T-18FE3, and Andor's Kymera-381i-D1 coupled with iStar CCD320.

While continuing contributing to the ongoing projects, our future plans involve participation in the LIBS analysis of art objects in collaboration with the Art Academy of Latvia and the Latvian National Museum of Art.

Soil analysis with Laser-Induced Breakdown Spectroscopy

V. Dwivedi, E. Haavisto, M. Fabritius, J. Ahokas, T. Hoang, P. Ryczkowski, **J. Viljanen,** and J. Toivonen

Photonics Laboratory, Physics Unit, Tampere University, FI-33101 Tampere, Finland

Email: jan.viljanen@tuni.fi

Soil and its wellbeing are in crucial role in the climate change, biodiversity, and food security. It has substantial part in the global biochemical cycles acting as a storage and a sink or source of carbon, being the source of the essential nitrogen- and phosphorus-based nutrients, and acting as storage and pathway for water circulation. The current rapidly changing climate and the fast changes in, e.g., fertilizer availability and price are causing demand for extensive soil analysis to assess and optimize the sustainability and cost of land use and food production.

Laser-Induced Breakdown Spectroscopy (LIBS) is a well-established online elemental detection technique that highly benefits from minimal requirement for sample preparation, applicability to solid, liquid, and gaseous samples, and robust experimental arrangement [1]. However, soil sets a new challenge for reliable and consistent elemental detection due to its local and global heterogeneity in multiple parameters including grain size, moisture, and density.

In our work, we have developed an on-site capable LIBS soil measurement prototype [2] and tackled the challenges set by the soil sample matrix by developing new sampling techniques, optimizing the sample handling protocol, and introducing complementing optical measurement techniques. As a result, we have reduced the required time for, e.g., soil total carbon analysis that enable higher sample through put with lower operational costs bringing extensive soil carbon analysis for carbon monitoring and validation available.

References:

[1] D. W. Hahn and N. Omenetto Applied spectroscopy, **66**, pp. 347-419 (2012).
[2] V. Dwivedi, et al., Geoderma **436**, pp. 116550 (2023)

Analysis of multilayer marine protective coatings through femtosecond LIBS

D. S. Sonoiki [1], C. R. Petersen [1,2], M. Schnohr [3], K. Scheibel [3], A. M. Moltke [1], C. R. Smith [1], and O. Bang [1,2]

[1] Department of Electrical and Photonics Engineering, Technical University of Denmark, 2800 Kongens Lyngby, Denmark
[2] NORBLIS ApS, Virumgade 35D, 2830 Virum, Denmark
[3] Hempel GrowHub, Hempel A/S, Lundtoftegaardsvej 90, 2800 Kongens Lyngby, Denmark

Email: doson@dtu.dk

Marine protective coatings are key barriers against harsh environmental conditions, protecting marine structures from fouling, erosion, and corrosion. Nevertheless, they eventually degrade, necessitating replacement. High-pressure medium blasting is a common technique for removing degraded coatings but despite being efficient, the method causes pollution of the environment generating a vast amount of contaminated waste. A more environmentally friendly approach to remove these coatings is laser ablation which involves the use of intense lasers for surface cleaning. Importantly, this method produces no additional waste, and the ablated coating residue can potentially be collected and recycled to minimize the environmental impact. In this work, we investigate laser ablation of multilayer marine coatings on a steel substrate using a femtosecond pulsed laser coupled with ultraviolet/visible (UV-VIS) plasma spectroscopy. The goal was to demonstrate the feasibility of distinguishing individual coating layers on the substrate based on spectral signatures. To this end, a high-resolution fiber-coupled spectrometer (187 nm to 440 nm) was employed to analyze the atomic/ionic emission from the ablation plasma. The ablation process was performed utilizing a 1030 nm laser with 300 fs pulse duration and a pulse repetition rate of 50 kHz delivering pulse energies up to 40 μJ. Layer discrimination was achieved by continuously scanning the coated steel panels back and forth in front of the laser focus, observing the change in spectral signatures depending on the number of scans. This method could potentially be used to both identify the composition of the coating and monitor the efficiency of the ablation process.

Keywords: Marine coatings, UV-VIS, plasma spectroscopy

Funding: Villum Fonden (project no. 00037822: Table-Top Synchrotrons) and the Danish Maritime Fund (LASER-CLEANR, no. 2022-054).

Elemental analysis of Cocoa samples by Laser-Induced Breakdown Spectroscopy

T. Steen [1,2], C. Keil [1], H. Haase [1], and **V. Merk [2]**

[1] *Department of Food Chemistry and Toxicology, Institute of Food Technology and Food Chemistry, Technische Universität Berlin, Straße des 17. Juni 135, 10623 Berlin, Germany*
[2] *LTB Lasertechnik Berlin, 12489 Berlin, Germany*

Email: virginia.merk@ltb-berlin.de

Cocoa beans are essential export products for many African, South and Central American and Asian countries, and fundamental for the confectionary industry. Cocoa beans are a source of many essential minerals and chocolate has the potential to provide significant amounts of minerals in the human diet. The mineral content of cocoa reflects the soil in which it is grown, a manner of concern especially for heavy metals harmful for humans, including Cd and Pb.
Its multi-elemental analysis, fast response, remote sensing, little to no sample preparation, low running costs and ease of use make LIBS a promising technique for the food sector [1].
This research examines the possibilities and limitations of LIBS as a method for quantifying minerals and trace elements in cocoa samples. Cocoa matrix-matching solid calibration standards were produced from high-purity cellulose spiked with increasing amounts of multi-element standard solutions and evaluated for LIBS application. Furthermore, LIBS measurements were performed on longitudinal sections of cocoa beans to investigate the distribution of different elements with high spatial resolution.

References:
[1] M. Markiewicz-Keszycka, X. Cama-Moncunill. Maria. P. Casado-Gavalda, Y. Dixit, R. Cama-Moncunill, P. J. Cullen, C. Sullivan, *Trends in Food Science & Technology*, **65**, 80-93, (2017)

Advancements in lithium quantification in geological samples using Sci-Trace/M-Trace LIBS technology

D. Bruzel, K. Virostková, M. Vejvodová, and J. Valášek

AtomTrace, Czech Republic

Email: bruzl.dominik@gmail.com

In the contemporary landscape of technology and energy, the demand for lithium, a critical component in batteries for electric vehicles, portable electronics, and renewable energy storage systems, is escalating rapidly. This surge is primarily driven by the global shift towards greener technologies and the increasing prevalence of electric vehicles, necessitating efficient and precise methods for lithium extraction and processing. Our study presents advancements in the quantification of lithium in geological samples, utilizing the state-of-the-art Sci-Trace/M-Trace LIBS devices. These instruments have been pivotal in addressing the challenges associated with lithium analysis in diverse geological matrices. The focus of our research was on granites with lithium-rich micas and rhyolites, which are significant sources of lithium. We employed an approach of analyzing lithium content in pressed pellets, a standard technique that enhances the accuracy and reliability of the measurements while also dealing with the effect of sample both chemical and physical (dimensional) heterogeneity. A key feature of our methodology was the performance of measurements under an inert argon atmosphere. This environment is crucial for reducing atmospheric interference, thereby improving the precision of the lithium quantification. The study extensively evaluated three lithium spectral lines: Li I 610.36 nm, Li I 670.78 nm, and Li I 812.60 nm. The selection of these lines was based on their sensitivity and the ability to provide a comprehensive analysis of lithium concentrations in various geological samples.

Combined real-time temperature and chemical analysis of molten steels for production applications

M. Gilbert-Gatty [1], J. Petersson [1], L. Hagesjö [1], A. Bengtson [1], C. Sterner [2], I. Petermann [2], F. Sandberg [3], C. Nilsson [4], R-S Katz [5], and P. Jakobsson [5]

[1] *Swerim AB, Box 7047, SE-164 07 Kista, Sweden*
[2] *RISE Research Institutes of Sweden, Digital Systems, Smart Hardware*
[3] *R&D Metallurgy, Alleima, SE-811 81 Sandviken, Sweden*
[4] *SSAB EMEA AB, Svartövägen 20, Luleå*
[5] *Agellis RHI Magnesita, Tellusgatan 15, SE-224 57, Lund, Sweden*

Email: melina.gilbert-gatty@swerim.se

Progress from recent developments of a lance system for combined in-situ analysis of temperature and chemical composition of molten steels will be presented. This work is done within the project Smart Melt II which is driven by a consortium leading by RISE and including several industrial partners. Despite several efforts, online and real-time assessment of both temperature and chemical composition of metallurgical processes remains a challenging task because of harsh environmental conditions for the sensors added to the difficulties of calibrating sensors for satisfying accuracies [1]. In the present project, the overall aim is to develop a valuable and practical tool for real-time process control of metallurgical processes. A lance system that combines two sensors for online temperature and chemical analysis is developed. The temperature is based on broadband spectral thermometer technology (BST) using the heat radiation to estimate the temperature. The second sensor enables chemical composition analysis using laser-induced breakdown spectroscopy (LIBS). Both temperature and chemical analysis can thus be conducted simultaneously by inserting the lance system directly into the steel melt.

The lance system was developed and tested through several field tests in an experimental facility for both proof-of-concept and calibration. Correlations between the LIBS chemical analysis and reference analysis based on spark OES was investigated. Further we have tested the lance system in real production environments in a tundish. Future development aims to take the lance system to previous metallurgical process steps such as e.g., converter, where real-time information of chemical composition is even more valuable.

[1] Myakalwar, A.K.; Sandoval, C.; Velásquez, M.; Sbarbaro, D.; Sepúlveda, B.; Yáñez, J. LIBS as a Spectral Sensor for Monitoring Metallic Molten Phase in Metallurgical Applications—A Review. Minerals 2021, 11, 1073. https://doi.org/10.3390/min11101073

Investigating the impact of synthetic data on machine learning model performance in LIBS

J. Lehtonen

Satakunta University of Applied Sciences, Finland

Email: juuso.2.lehtonen@samk.fi

This study aims to explore the potential impact of synthetic datasets on the performance of machine learning (ML) models in the context of Laser-Induced Breakdown Spectroscopy (LIBS). Multiple datasets of varying sizes were generated based on National Institute of Standards and Technology (NIST) spectral database to provide a diverse range of training scenarios for ML models. These synthetic datasets were utilized to train neural network models, with the objective of determining whether such data could enhance the accuracy of predicting elemental compositions in measured samples. Central to our research was evaluating how these varied datasets influence the effectiveness of ML models and examining the potential advantages of applying transfer learning strategies. Our methodology included training neural networks with these different datasets to assess the impact of data volume and diversity on the models' predictive accuracy on the measured data. Transfer learning was also investigated as a means to potentially improve model adaptability and efficiency, especially in cases involving limited or specific data types. The study's findings are aimed at providing insights into whether synthetic data from the NIST database can improve the predictive capabilities of ML models in LIBS applications. This approach could offer a strategy for advancing ML models in spectroscopy, potentially reducing the reliance on new physical sample collection.

Simulating plasma emission spectra for neural network training: integrating LTE states to correspond with observed LIBS spectra

E. Ojanen, T. Aaltonen, and P. Suominen

Satakunta University of Applied Sciences, Finland

Email: eetu.2.ojanen@samk.fi

While NIST provides snapshot spectra of Local Thermal Equilibrium (LTE) conditions tailored for Laser-Induced Breakdown Spectroscopy (LIBS) environments, a considerable shortfall exists in current methods for the ongoing simulation of plasma dynamics across time.

In this research, we explore the feasibility of creating simulated data to train neural networks, which typically require a vast amount of training data. Our focus is on simulating the plasma emission spectrum, replicating what is typically measured by current devices constrained by their integration times.

Central to our methodology is the principle that in each time step, the plasma exists in a state of LTE with these states being sequentially integrated over time to simulate an overall exposure. To achieve this sequential integration of LTE states, our study employs a variety of cooling functions for hot plasma, such as linear cooling, with the goal of aligning closely with actual LIBS measurements. The time resolution used in our simulations varies, typically ranging from 10 to 100 nanoseconds.

A key application of this research is to train neural networks to identify elements, particularly those found in lithium-ion batteries.

The resulting simulated data encompasses energy flux, analogous to real measurement exposure, and three-dimensional visualizations of exposures. These visualizations illustrate intensity in wavelength and integration time domain, offering detailed insights into the plasma emission processes.

The simulations are developed based on spectral line and energy level data sourced from the National Institute of Standards and Technology (NIST), a leading repository of atomic data, including A-coefficients and g-values.

Studies of LIBS-spectral fingerprints of lithium-ore minerals in different compositional matrixes

L. Huaman, I. Laine, and J. Leveinen

Department of Civil Engineering, School of Engineering, Aalto University, Espoo, Finland

Email: luis.huaman@aalto.fi

Laser-Induced Breakdown Spectroscopy (LIBS) is a fast-growing technology in the field of core-scanning for the mining industry. Its ability to detect light elements such as Lithium, Beryllium or Carbon in addition to most of the elements detected by other core-scanners makes LIBS the most suitable technique for investigating lithium ores in pegmatites. However, the whole range of potential benefit and limitations of LIBS for mining applications has not yet been brought to light. To address this challenge, 11 pieces of drill cores in different matrixes and selected 14 thin disks from Rapasaari deposit in Kaustinen (Keliber Technology Oy) were scanned in order to study elemental/mineral maps, textural features and potential micrometric aplications. The data was recorded using LIBS scanner from Lumo Analytics in Helsinki, which accounts for two spectrometers measuring wavelength ranging from ultraviolet to visible, 194-469 nm and 445-989 nm. The data processing included a pre-processing step of background removal, followed by initial visualizations of elemental maps with sliderplots and finally mineral maps elaborated with a combination of two algorithms, Interesting Featured Finding "IFF" and Spectral Angle Mapping "SAM". X-Ray diffraction (XRD) and optical microscopy were used to validate and verify mineral classifications. The ultimate goal of this research was to study the practical mining application of the use LIBS as novel core scanner for the mining industry. The results indicate that LIBS can efficiently produce elemental and mineral maps of ore/gangue minerals in time frames according to the needs of the mining industry. Textural features can potentially benefit comminution circuits. Micrometric-scale scan can potential be used to some extent to study rocks at micrometric sizes. These initial findings make of LIBS an efficient core scanner with applications in the mining industry beyond elemental and mineral maps.

Laser Induced Breakdown Spectroscopy (LIBS) of hydrogen detection in molybdenum at variable gas pressures of argon or nitrogen

I. Jõgi, J. Ristkok, and P. Paris

Institute of Physics, University of Tartu, Estonia

Email: indrek.jogi@ut.ee

LIBS is being developed for hydrogen isotope detection in tokamak first wall materials and for this purpose, the present study investigated the spatial and temporal development of Hα line emission in laser-induced plasma plume of molybdenum (Mo) target with hydrogen impurity in argon or nitrogen at variable pressures. An Nd:YAG laser with an 8 ns pulse width was used to ablate the Mo target and the development of the formed plasma plume was investigated by photographs and time and space-resolved emission spectra in the 20 nm range around the 656.28 nm H_α line [1,2]. The experiments were carried out in 3 to 760 Torr argon or nitrogen atmosphere. The temporal development of the plasma plume dimensions was roughly comparable in both argon and nitrogen, while the integral intensity of the plasma plume was stronger in argon. At higher gas pressures, the dimensions of the plasma plume reduced. At all experimental conditions, H_α and Mo I line intensities had different spatial distributions along the axis of plasma plume development: the H_α intensity was highest closer to the target while the Mo I line intensity reached maximum values farther away from the target. The intensity of H_α line decreased with delay time and the reduction of H_α line intensity was faster in nitrogen. Therefore, it's preferable to use argon atmosphere.

[1] I. Jõgi, J. Ristkok et al. Fusion Eng. Des. 179, 113131 (2022)
[2] I. Jõgi, J. Ristkok et al. Nucl. Mater. Energy 37, 101543 (2023)

Portable LIBS as a tool to estimate the lithium potential of glaciated terrain

S. Romppanen, H. Nygård, and J. Kuusela

Mineral Economy Solutions, Geological Survey of Finland (GTK), P.O. Box 96, FI-02151 Espoo, Finland

Email: sari.romppanen@gtk.fi

The expanding need for critical raw materials (CRMs) has increased interest towards lithium prospecting in Europe. Using LIBS in lithium detection is widely acknowledged, and the benefits of LIBS in lithium-bearing spodumene pegmatite analysis have been demonstrated.[1] In the present study, handheld LIBS is applied to glaciated soil (till) to discover hidden lithium resources.

Till is an unsorted mixture of different grain-sized mineral matter which glacier has transported and deposited. The conventional way for till geochemical research is to collect a set of rather big samples in the field and transport those to a laboratory, where the samples are dissolved into acids and analysed with ICP-MS or ICP-OES. The leachability of minerals into acids differs so this approach is not just time-consuming but also requires prior knowledge of the metals of interest and their host minerals.

LIBS offers several advantages for till analysis: short measurement time, reached total elemental composition, and small sample size make LIBS a suitable tool for till geochemical research. All this forms the base of this study where small-sized till samples from a lithium potential area in Kuortane municipality in western Finland were collected and analysed with portable LIBS of a wide spectral range. Challenges related to the representativity of small sample size and the matrix effect of LIBS can be overcome with the right sampling design and robust LIBS data analysis. Our approach presents a non-invasive way to discover the lithium potential of the glaciated area.

Reference

(1) Romppanen, S., Pölönen, I., Häkkänen, H. & Kaski, S., 2023. Optimization of spodumene identification by statistical approach for laser-induced breakdown spectroscopy data of lithium pegmatite ores. Applied Spectroscopy Reviews, 58:5, 297–317, DOI: 10.1080/05704928.2021.1963977

Calibration Free and Depth Profile LIBS Analysis of Transition Metal Borides

M. Veis [1*], S.J. Shetty [1*], S. Atikukke [2], T. Roch [1], and P. Veis [1]

[1] Dept. of Exp. Physics, FMPI, CU, Mlynská dol. F2, 842 48 Bratislava, Slovakia
[2] Dept. of Astronomy, Physics of the Earth, and Meteorology, FMPI, CU, Mlynská dol. F2, 842 48 Bratislava, Slovakia

* Authors with equal contribution
Email: pavel.veis@fmph.uniba.sk

Transition metal borides (TMBs) thin films exhibit distinctive physical and chemical attributes, such as remarkable hardness exceeding 40GPa [2], rendering them suitable for industries demanding high thermal stability and wear resistance, such as aerospace, energy generation and microelectronics. TMBs layers could be useful also in thermonuclear fusion as a gas diffusion barrier layer in the first wall. Ensuring the stoichiometry of the mixture of TM and B is crucial for dedicated applications. A preliminary study was conducted with CF-LIBS to analyse TiB_2 which has excellent thermal properties to withstand temperatures up to 3225 °C [2]. The samples were studied using LIBS which is a well-known analytical technique to quantify all elements including light. CF-LIBS study of different Ta and Zr containing sample such as WTa, WZr were analysed recently by creating laser induced plasma by a ns laser [3-4]. In this work we are analysing the elemental composition of thin ZrB_2 and TaB_2 layers created by pulsed laser deposition (PLD) technique on Si substrate. In this work, the depth profile of the above-mentioned samples together with calibration free (CF) analysis is presented. The accumulation of 20 shots was selected corresponding to the ablation of the layer. Only one B I doublet at 249 nm was experimentally observed and the other doublet was partially self-absorbed. In this work, the self-absorption of the Boron doublet is corrected by the which allows us to accurately perform CF-LIBS analysis. The ns Q-switched Nd:YAG laser (Quantel Brilliant Eazy at 1064 nm) was used for the laser plume generation under air at atmospheric pressure. LIBS spectra are acquired using an Echelle spectrometer (Mechelle ME5000, Andor Technology) equipped with an iCCD camera (iStar DH734, Andor Tech.). The quantitative results by CF-LIBS method are consistent with other standard techniques.

Reference

[1] M. Magnuson, L. Hultman, and H. Högberg: "Review of transition-metal diboride thin films", Vacuum 196 (2022) 110567, doi: 10.1016/J.VACUUM.2021.110567.
[2] S.J. Shetty, S. Soni, S. Atikukke, P. Veis, M. Veis, T. Roch: "Study of the composition of TiB_2 using LIBS in the visible - NIR region", SVK FMFI UK, Bratislava, 2023, p. 163, ISBN 978-80-8147-136-0,
[3] S. Atikukke, M. Veis, P.Veis et al, Resonant LIBS for quantitative elemental depth profile analysis of WTa coating, NME 38 (2024) 101558
[4] A. Marín Roldán, V. Dwivedi, M. Veis, S. Atikukke, H.J. van der Meiden, P. Veis, M. Držík: "Quantification of hydrogen isotopes by CF-LIBS in a W-based material (WZr) at atmospheric pressure: from ns towards ps", Physica Scripta 96 (2021) Art. no. 124061.

OH radical analysis in a C_2H_5OH vapour RF discharge plasma for calibration of a VUV spectrometer

S. Soni [1], J. Viljanen [2], M. Veis [1], H. van der Meiden [3], M. Stano [1], and P. Veis [1]

[1] *Dept. of Exp. Physics, FMPI, CU, Mlynska dol. F2, 842 48 Bratislava, Slovakia*
[2] *Photonics Laboratory, Tampere University, FI-33101 Tampere, Finland*
[3] *DIFFER, 5612 AJ Eindhoven, The Netherlands*

Email: pavel.veis@fmph.uniba.sk

We present a method for the state-of-the-art calibration of the relative spectral response curve in the closed vacuum-UV (VUV) range of a Seya-Namioka Vacuum Monochromator (McPherson, 100nm-300nm) based on the emission spectra of OH radical dimer produced in a plasma that is located inside a quartz tube. The current method is based on the OES from this 13.56 MHz RF plasma generated at low pressure in an Ar gas flow mixed with C_2H_5OH vapour. The CW plasma is produced by capacitive RF discharge connected to a RF power supply maintained at 350 volts. After ignition of the RF discharge with a high voltage source (Tesla, BS275), OES spectra are collected through an unique hollow core fibre with an internal diameter of 0.7 mm and transferred to the VUV monochromator (model 234/32VM, McPherson, f=20 cm, 2400 gf/mm). The spectrometer employed in this study allows the recording of a 30 nm spectral band in a single scan. The entire VUV spectra are recorded for the spectral range 100-300nm by joining 10 successive bands, each averaged for 200 accumulations triggered internally with an exposure time of 0.6 s, resulting in an enhancement in the signal-to-noise ratio by a factor of 14.1. Additional optical and electronic schemes in this experiment are similar to the spectroscopic arrangement used in Ref. [1]. The OH band emission resulting from the $C^2\Sigma^+ \rightarrow A^2\Sigma^+$ electronic transition is observed around 160 - 200 nm (the vibrational 0-0 transition head was at λ00 =179.7nm). The spectral response curve in closed VUV range (160nm -200 nm) is evaluated by the comparison of measured and theoretically calculated molecular band spectra using the known molecular constants [2]. The spectrometer is used for laser-induced breakdown spectroscopic (LIBS) measurements in VUV range for precise low-Z elemental analysis.

[1] J. Jasik et al, Spectrochim. Act. B, 64(2009), 1128.
[2] E. W. Lemmon et al, NIST chemistry webbook, *NIST standard reference database*, 69(2011), 20899

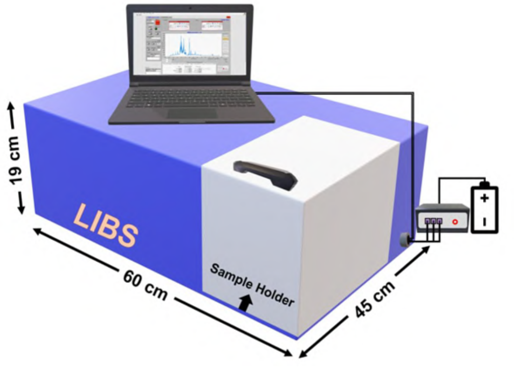
Field-deployment of LIBS device for rapid soil carbon monitoring

V. Dwivedi, J. Ahokas, J. Viljanen, P. Ryczkowski, and J. Toivonen

Photonics Laboratory, Physics Unit, Tampere University, Korkeakoulunkatu 3, 33720 Tampere, Finland

Email: vishal.dwivedi@tuni.fi

In global environmental and carbon management, soil plays an important role. State-of-the-art soil carbon analysis is done with a laboratory-based pyrolysis method, which is expensive and time-consuming as it requires tedious sample preparation, thus not allowing wide scale soil carbon monitoring. Laser-induced breakdown spectroscopy (LIBS) offers rapid and in-situ multi-elemental analysis and holds potential for widespread use. In this project, a compact and field-capable LIBS device is developed for direct on-site characterization of soil, enabling rapid and accurate determination of carbon content and soil elemental composition. Attached figure illustrates the basic design of LIBS device. The device has undergone testing in both laboratory and field settings for various agricultural fields in Finland. The results of the project have the potential to significantly speed-up carbon sequestration practices in cropland soil, leading to substantial societal impact.



Basic design of LIBS prototype at Tampere University, Finland [1]

[1] V. Dwivedi, et al., *Geoderma* **436**, pp. 116550 (2023)

Elemental analysis and metabolic profiling of medicinally potent members of Zingiberaceae family using FT-IR and LIBS coupled with PLS-DA

S. Sohrab [1], P. Mishra [1], **V. Dwivedi [2,3]**, P. Veis[3], A.K. Pathak [4], and S.K. Mishra [1]

[1] Department of Botany, Ewing Christian College, University of Allahabad, Uttar Pradesh, 211003, India
[2] Photonics Laboratory, Physics Unit, Tampere University, Korkeakoulunkatu 3, 33720 Tampere, Finland
[3] Department of Experimental Physics, Comenius University, Mlynská dol. F2, 842 48 Bratislava, Slovakia
[4] Department of Physics, Ewing Christian College, Prayagraj 211003, Uttar Pradesh, India

Email: vishal.dwivedi@tuni.fi

This work explores the potential use of Fourier transform infrared spectroscopy (FT-IR) and laser-induced breakdown spectroscopy (LIBS) for obtaining elemental and metabolic profiles of significant medicinal plants belonging to the Zingiberaceae family. A total of seven samples were collected from two different geographic origins in India for examination. The FT-IR analysis of the methanolic extracts shows the presence of alcohol, aliphatic compounds, esters, ethers, and their derivatives. The spectral characteristics of atomic and molecular species, encompassing both organic and inorganic elements, present in the samples were observed. The LIBS spectra also indicated the presence of heavy metals and trace elements. Furthermore, partial least squares discriminant analysis (PLS-DA) has been used to obtain classification pattern of the samples based on their spectral fingerprints. The study would thus contribute in establishing the pharmacological significance by revealing the organic profile and nutraceutical importance by reflecting the elemental profiles.

Moisture calibration in laser-based elemental analysis of soil

T. Hoang, J. Ahokas, V. Dwivedi, J. Viljanen, and J. Toivonen

Photonics Laboratory, Physics Unit, Tampere University, Finland

Email: thu.hoang@tuni.fi

In this project, we explore how soil moisture impacts elemental analysis using Laser-Induced Breakdown Spectroscopy (LIBS), a technique valued for its detailed insights, compactness, rapid analysis, and minimal sample preparation. LIBS operates by vaporizing a small portion of a soil sample with a pulsed laser, creating a plasma whose electromagnetic spectrum reveals the elements present [1]. We specifically investigate how soil moisture, which tends to reduce LIBS intensity by diverting energy to evaporation rather than plasma heating, affects the calibration of the LIBS signal. Our research includes developing a method to fit the calibration curve of LIBS, ensuring accurate measurements of soil carbon levels regardless of initial soil moisture, thereby facilitating fast and direct soil assessment.

Keywords: laser-induced breakdown spectroscopy, LIBS, soil moisture

1. D.W. Hahn and N. Omenetto, "Laser-induced breakdown spectroscopy (LIBS), part I: review of basic diagnostics and plasma-particle interactions: still-challenging issues within the analytical plasma community.", Applied spectroscopy, **64**(12) 2010.

Total soil phosphorus detection using Laser-Induced Fluorescence-Assisted Laser-Induced Breakdown Spectroscopy

S. Soni [1,2], **J. Viljanen [1]**, and P. Veis [2]

[1] Photonics Laboratory, Physics Unit, Tampere University, FI-33101 Tampere, Finland.
[2] Comenius University, FMPH, Mlynska dolina F2, 842 48 Bratislava, Slovakia

Email: jan.viljanen@tuni.fi

Quantification and monitoring of phosphorus in soil plays a critical role in environmentally friendly agriculture, especially in mitigation of phosphorus leakages to water systems and subsequent risk for eutrophication. On the other hand, deficiency in phosphorus would lead problems in development and growth of cultivated crops. Therefore, monitoring and quantification of phosphorus status in soil is essential.

Laser-Induced Breakdown Spectroscopy (LIBS) is a well-established online elemental detection technique that highly benefits from minimal requirement for sample preparation, applicability to solid, liquid, and gaseous samples, and robust experimental arrangement. It is extensively used in different fields of science and industry for elemental quantification utilizing the atomic emission from the plasma plume produced on the sample with a laser pulse [1]. However, LIBS suffers from relatively poor limit of detection (LoD) for elements that require high plasma temperature for thermal, excitation as the high temperature plasma is available only for a short time and, on the other hand, this is the time when the background continuum radiation is high and most other elements are excited as well with substantially temperature broadened lines causing spectral interferences.

In this work, Laser-Induced Fluorescence (LIF) is applied to the conventional LIBS setup (LIBS-LIF) for sensitive and selective phosphorus detection. The plasma plume is overlapped with another laser pulse that is produced with a wavelength tunable OPO laser unit. The OPO laser pulse is tuned to 253.6 nm that is resonant to the atomic phosphorus transition to re-excite the phosphorus atoms produced in the plasma plume. The experimental arrangement is shown in Fig. 1a. The resulting phosphorus fluorescence is observed at 213.6 nm as shown in Fig. 1b. The LIBS-LIF approach improved the LoD of soil phosphorus detection up to 31-fold compared to phosphorus-optimized conventional LIBS measurement enabling studies in soils containing low amounts of phosphorus. It is demonstrated that calibration curve can be generalised to cover same soil types from different geographical location, thus, improving the applicability and efficiency of the method. As LIF arrangement can be added to any LIBS setup, providing that a suitable light source is available, LIBS-LIF can be utilized in analysis of phosphorus in various environmental samples, such as water reservoirs, that are in the need of more sensitive phosphorus detection schemes with high sample throughput.

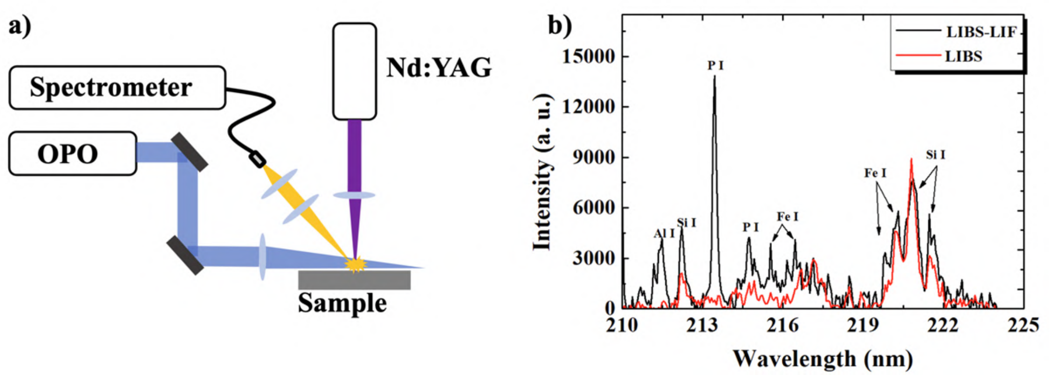


Fig. 1: a) The experimental arrangement, b) the observed phosphorus fluorescence at 213.6 nm with LIBS-LIF on a soil sample containing 2.2 mg/kg of soluble phosphorus.

References
[1] D. W. Hahn and N. Omenetto Applied spectroscopy, **66**, pp. 347-419 (2012).
[2] C. M. Li, C. Mao, Z. M. Zou, X. Y. Yang, Z. Q. Hao, L. B. Guo, X. Y. Li, Y. F. Lu, and X. Y. Zeng. Journal of Analytical Atomic Spectrometry, **29**, pp. 1432-1437 (2014).

Utilizing laser-induced breakdown spectroscopy in lithium-ion battery recycling

J. Ahokas [1,2], H. Rostami [3], U. Lassi [4], and J. Toivonen [1]

[1] *Photonics Laboratory, Tampere University, Korkeakoulunkatu 3, 33720 Tampere, Finland*
[2] *Satakunnan ammatikorkeakoulu, Satakunnankatu 23, 28130 Pori, Finland*
[3] *Kokkolan yliopistokeskus Chydenius, Talonpojankatu 2B, 67701 Kokkola, Finland*
[4] *Oulun yliopisto, Erkki Koiso-Kanttilan katu 1, 90570 Oulu, Finland*

Email: joni.ahokas@tuni.fi

Lithium-ion (Li-Ion) batteries are an indispensable way of storing electrical energy, but unfortunately, the degradation of the batteries can not be avoided. Among other things, the dangerous consequences of the battery malfunctions and failures resulting from aging motivate to look for technologies for prevention and intervention [1]. Also sustainability has become core issue with the Li-Ion batteries. Due to the mining of the scarce resources needed in building the batteries, the recycling of the Li-Ion battery waste will be crucial now and in the future [2].
The recycling procedures of the battery materials bring forth needs of different methods of analysis. Currently several X-ray based methods, as well as methods such as Fourier transform infrared spectroscopy (FTIR), are being used to do elemental characterization of the batteries [3]. These methods come with several issues. They often require that the measurement is done in laboratory conditions, away from the actual recycling center. X-ray based methods also come with limited sensitivity for light elements, which might restrict the potential of these methods [4].
Laser-induced breakdown spectroscopy (LIBS) is a method that uses a laser-induced plasma to reveal multi-elemental information of a target. This information can include elemental composition of said target, as well as quantitative information about these elements. In this work, LIBS is tested as a possible candidate for a technique to solve issues of monitoring Li distributions and contaminations on recycled battery materials. Different LIBS methods are studied, from more traditional calibration models to the calibration-free ones.

References

[1] Diaz D, Hahn DW., *Chemical Characterization Using Laser-Induced Breakdown Spectroscopy of Products Released from Lithium-Ion Battery Cells at Thermal Runaway Conditions*, (2022) Applied Spectroscopy, 76 (8), pp. 967-977
[2] Larcher D., Tarascon J.-M., *Towards greener and more sustainable batteries for electrical energy storage*, (2015) Nature Chemistry, 7 (1), pp. 19 - 29
[3] Verma P., Maire P., Novák P., *A review of the features and analyses of the solid electrolyte interphase in Li-ion batteries*, (2010) Electrochimica Acta, 55 (22), pp. 6332 - 6341
[4] Kappeler M., Basler C., Brandenburg A., Carl D., Wöllenstein J., *Homogeneity Measurements of Li-Ion Battery Cathodes Using Laser-Induced Breakdown Spectroscopy*, (2022) Sensors, 22 (22) pp. 8816

Nordic LIBS 2024

5-6 March 2024

Tampere, Finland