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Technical Note

A versatile interaction chamber for laser-based spectroscopic applications, with the emphasis on Laser-Induced Breakdown Spectroscopy☆

J. Novotný [a,b,*], M. Brada [a,b], M. Petrilak [a,b], D. Prochazka [a], K. Novotný [a,c], A. Hrdička [c], J. Kaiser [a,b]

[a] *Central European Institute of Technology, Brno University of Technology, Technicka 3058/10, 616 00 Brno, Czech Republic*
[b] *Institute of Physical Engineering, Faculty of Mechanical Engineering, Brno University of Technology, Technicka 2896/2, 616 69 Brno, Czech Republic*
[c] *Central European Institute of Technology, Masaryk University, Kamenice 753/5, 625 00 Brno, Czech Republic*

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ABSTRACT

The technical note describes the interaction chamber developed particularly for the laser spectroscopy technique applications, such as Laser-Induced Breakdown Spectroscopy (LIBS), Raman Spectroscopy and Laser-Induced Fluorescence. The chamber was designed in order to provide advanced possibilities for the research in mentioned fields and to facilitate routine research procedures. Parameters and the main benefits of the chamber are described, such as the built-in module for automatic 2D chemical mapping and the possibility to set different ambient gas conditions (pressure value and gas type). Together with the chamber description, selected LIBS application examples benefiting from chamber properties are described.

1. Introduction

A modern analytical method known as the Laser-Induced Breakdown Spectroscopy (LIBS) utilizes the optical emission of the plasma plume to determine the elemental composition of the analyzed material [1]. The plasma plume is induced on the sample surface by the focused laser pulse. This type of the atomic emission spectroscopy technique, where the excitation energy is in the form of a laser pulse, brings number of significant advantages to the field of material analysis. LIBS is able to identify a wide spectrum of chemical elements including those with low atomic numbers. There is no need for any special sample preparation and all states of matter can be analyzed. Results of the analysis are available in a few seconds and one measurement may contain spectroscopic traces of all chemical elements present in the sample.

Despite the fact that there still exist drawbacks, e.g. problematic quantitative analysis, matrix effect and relatively poor limits of detection (LOD, generally 1–100 ppm), LIBS has become a respected technique, complementary to the well-known techniques of chemical material analysis, such as Atomic Absorption Spectroscopy (AAS), X-Ray Fluorescence (XRF), Inductively Coupled Plasma Mass Spectroscopy (ICP-MS), and many others [2].

From the origins of the LIBS technique in the beginning of the 80's until present, the growing interest is noticeable particularly in the last two decades. LIBS applications and LIBS technique itself are the research subjects of many university laboratories and research institutes. Applications of the LIBS technique as an analytical tool for the fast chemical analysis are under intensive investigation in the fields of steel industry, food industry, environmental diagnostics, archeology and cultural heritage, geology, forensic analysis, etc. The rapidness of LIBS analysis and the possibility to measure samples in-situ and remotely enable measurements to be carried out in the places and situations inaccessible for the conventional techniques.

Thorough information overview including the references about the state and results of the research in all application fields can be found in LIBS books [1,3] and in the recent review articles [4–8]. Until now a number of modifications of the standard LIBS technique have been published, usually with the goal to enhance the LODs, accuracy, repeatability, spatial resolution or with the purpose of adapting to various environmental conditions of the actual measurement. Double-Pulsed LIBS, LIBS + LIFS (Laser-Induced Fluorescence), RLIBS (Resonance Laser-Induced Breakdown Spectroscopy), CF-LIBS (Calibration-Free LIBS), μLIBS, Liquid-LIBS, Stand-Off LIBS, Remote LIBS, and R-FIBS (Remote Filament-Induced LIBS) can be mentioned among others.

Even though the assembly of the typical LIBS setup is relatively simple, high demands are placed on the parameters of the individual components. Development of the LIBS and its applications is highly dependent on the development progress in the fields of pulsed lasers, spectrometers and optical radiation detectors. Besides these basic

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* Corresponding author at: Institute of Physical Engineering, Faculty of Mechanical Engineering, Brno University of Technology, Technicka 2896/2, 616 69 Brno, Czech Republic. Tel.: +420 776758292; fax: +420 54114 2821.
E-mail address: novotny.j@fme.vutbr.cz (J. Novotný).

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components, the LIBS setup consists of many other peripheral devices — e.g. pulse generators, photodiodes, power meters, a control PC, optical assemblies, and optomechanical parts. One of them could be the chamber enclosing the laser–sample interaction.

The LIBS laboratory at Brno University of Technology (Brno, Czech Republic) has been dealing with the LIBS method for more than 15 years and gained considerable experience that served as an impulse for the design of the interaction chamber. The main purpose was to extend the capabilities of the table-top LIBS setups, to simplify the necessary mechanical alignment procedures, to speed up the measurements and to enable automated acquirement of the series of spectroscopic data (e.g. for chemical mapping).

2. Experimental set-up

The interaction chamber (Fig. 1) for the laser-based spectroscopic applications with special focus on LIBS has been developed. It meets the requirements of the LIBS method and its modifications mentioned above.

The chamber basically consists of the manipulator, chamber body, series of optomechanical components and the pressure regulating system. In order to ensure high level of versatility, most of the construction parts mentioned in the following sections have been designed in the form of a cage system. Thanks to that, these mounts are assembled from the standardized optical and mechanical parts and can be easily adjusted, rebuilt or modified.

2.1. Manipulator

In the center of the chamber there is a motorized 3-axis manipulator (no. 1 in Fig. 2). It is mounted on the chamber door that can be slid out of the chamber up to 200 mm distance to guarantee easy access to the analyzed sample. Three electromotors for the orthogonal *x*, *y* and *z* axes have altogether the movement range of 80 × 60 × 50 mm. Movement resolution is below 2 μm. The stage on the manipulator has one centering and two mounting threaded holes to attach several types of the sample holders. The sample stage can carry more than 2 kg. Electromotors are able to operate in the vacuum down to Ultra High Vacuum (UHV).

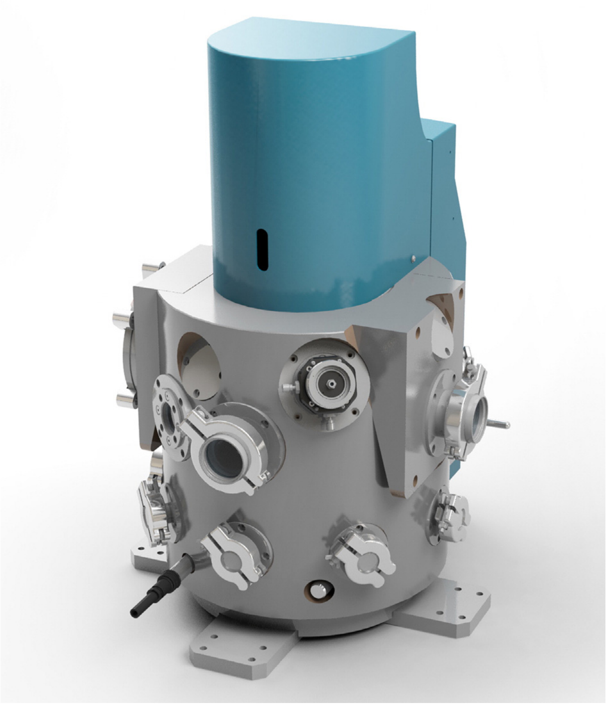

Fig. 1. The interaction chamber.

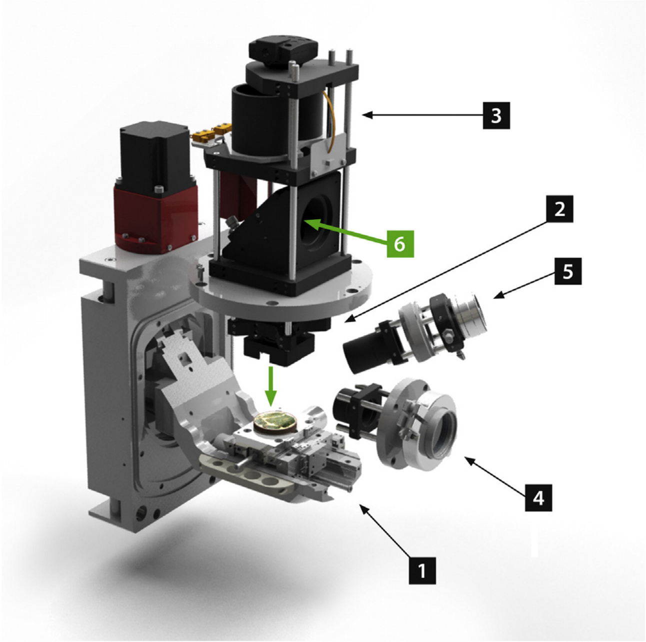

Fig. 2. Chamber inner view — 1. motorized manipulator, 2. primary laser focusing optomechanical assembly, 3. sample view optomechanical assembly, 4. secondary laser focusing optomechanical assembly, 5. plasma radiation collecting optomechanical assembly and 6. direction of the primary laser pulse.

2.2. Primary input

Primary input for the laser beam is situated on the top of the chamber (no. 2 in Fig. 2), so the optical axis is perpendicular to the sample surface. The input is equipped with the optomechanical assembly for the focusation of the laser beam. The distance to the sample allows utilizing the lens with the focal length up to 75 mm. Using a standard available adapters it is possible to mount microscopy objective to realize μLIBS analysis. In the default configuration the focusing lens is an air-spaced doublet with suppressed spherical aberration and AR (anti-reflective) coating optimized for the Nd:YAG laser harmonics wavelength 1064 and 532 nm. With 32 mm focal length lens and 532 nm wavelength the diameter of the craters is lower than 80 μm. The front side of the focusing assembly is covered by the protective window with AR coating.

2.3. Sample view

The primary laser pulse (no. 6 in Fig. 2) is reflected into the primary input by the dichroic mirror. Above the mirror — and thus above the primary input, there is an optomechanical assembly for the sample view (no. 3 in Fig. 2). It contains a tandem of view objectives for electronically switchable field of view (1.5 and 7.5 mm) and CMOS camera (1280 × 1024, color, 25 fps) with the protective shutter. The sample is illuminated by the LED ring.

The sample view is used not only for better orientation on the sample surface, but also for the laser autofocus system as the data provider. The developed autofocus algorithm [9] analyzes the sharpness of the captured sample view image based on a Fourier transform of the image matrix. In a few iterative cycles it is able to place the manipulator to the focal plane of the focusing lens with the accuracy of ±50 μm.