Compact, self-contained pulsed lasers expand capabilities in LIBS applications

LIBS analysis is now being adopted as an attractive technique in a wide range of industrial applications: from sorting recycled metals to identifying future mining areas. In this white paper, we introduce a very compact, air-cooled, pulsed laser and show it can be used successfully in conventional LIBS experimental set-ups for identifying metals.

LIBS basics

Laser-induced breakdown spectroscopy (LIBS) is an atomic-emission spectroscopy technique that enables rapid chemical analysis of a wide range of materials including metals, semiconductors, glasses, biological tissues, plastics, soils, thin and paint coatings. The technique relies on focusing short high-energy laser pulses onto the surface of a target sample which in turn generates a plasma containing a small amount of the sample (typically, a few nanograms). The high temperatures generated within the plasma cause the ablated material to dissociate into excited atomic and ionic species which emit characteristic spectral lines upon relaxation. The spectrum of the investigated sample is then detected by a spectrometer and is converted into a quantitative or qualitive reading of the material content.

The simplicity of the LIBS analytical method together with the absence of sample preparation requirements makes it a powerful non-contact (or stand-off) optical technique. The versatility of the LIBS technique means that a broad range of application areas can also be addressed. For example, compact hand-held LIBS devices are suited to fast material identification in the field while larger tabletop systems can be used for routine material quality monitoring and research, and even larger industrial LIBS systems address the needs for in-line analysis of metals (e.g. in scrap metal sorting systems, aluminium and steel production processes, slag and lime-mud).

The laser requirements for each of these types of LIBS systems can also vary. For instance, portable handheld systems require compact and power efficient lasers by nature whereas larger industrial LIBS systems require high-energy pulsed lasers and are not necessarily concerned about the dimensions of laser sources. These high energy pulsed lasers are often quite bulky due to the size of the laser heads, drive electronics and cooling systems. They also sometimes provide low-quality beams which are difficult to focus into small spots and consume a fair amount of electrical power. For these reasons, they work well for larger scale LIBS systems with large focal depth and fast-moving samples, but they are less suited for portable devices or compact tabletop systems.

Lasers for compact & fast LIBS systems

An alternative for smaller but still powerful LIBS systems is the Cobolt Tor™ XE. This laser is a compact air-cooled passively Q-switched diode-pumped solid-state laser (DPL) providing 0.5 mJ pulse energies at 1064 nm with kHz repetition rates and in a high-quality beam (M2<1.3). The most critical parameters for LIBS applications is the energy density of the laser pulse and the peak power, which must exceed the ablation thresholds of the analysed material (ref:1,2,3). Due to the excellent TEM00 beam quality of all lasers within Cobolt Tor™ series, and the Cobolt Tor™ XE in particular, it is possible to focus the nearly diffraction limited beam into a small area, and thereby reach sufficiently high pulse energy density and peak power to ablate the material and produce a plasma of excited atoms and ions without melting the material.

Figure 1. Cobolt Tor™ XE laser head (144 × 50 × 70 mm) with integrated electronics on an air-cooled heatsink (176 x 118 x 54 mm). No external electronics or cooling systems are required.

To achieve high signal-to-noise ratios in LIBS measurements it is important that the recording of the LIBS spectra is well timed with the laser pulses. The Cobolt Tor™ XE laser has the advantage that the pulse repetition rate (PRF) can be chosen freely and changed by the user (from a single pulse up to 1 kHz pulse trains) which can help to improve the signal-to-noise ratio. Moreover, the user can choose between three different trigger modes: internal, external, and gated (Read the laser Manual for a full description of the trigger modes).

The variety of trigger modes allows easy synchronisation with other equipment used for LIBS measurements. In addition, the unique cavity design of the laser provides a very stable pulse train, where the timing between the pulses is well defined by very low (for passively Q-switched lasers), pulse-to-pulse timing jitter (<2 µs) even at kHz repetition rates.

The compactness of the laser makes it attractive for integration into portable devices or compact table top systems and is shown in Figure 1. The laser head contains all the drive and control electronics and measures only 144 x 50 x 70 mm and the heatsink 176 x 118 x 54 mm. The laser is manufactured using HTCure technology which ensures long lifetime and robust performance under varying ambient conditions and harsh environments.

Industrial testing of the laser capabilities

In collaboration with Swerim AB, a leading industrial research institute for mining engineering, process metallurgy, material science and manufacturing engineering, the Cobolt Tor™ XE (0.5 mJ, 1064 nm, <3.5 ns) was evaluated for metal analysis using the LIBS technique.

The experimental setup seen in figure 2, included the Cobolt Tor™ XE laser to generate a plasma, a set of optical components for beam shaping and light collection into an optical fiber and a spectrometer for the spectral material content analysis. (Read more about the experiment in our editorial article in Photonics Views).

The LIBS technique can be used for both quantitative and qualitative analysis of materials. For qualitative LIBS analysis, the presence of certain elemental spectral lines is enough to simply identify a sample, whereas for quantitative LIBS analysis, the exact amount of the element within the sample can be measured. This requires the system to be calibrated against reference samples of known concentrations.

Calibration of the LIBS system is done by first measuring the LIBS signals of the reference samples, which are then correlated to the known element contents. Once the calibration is established, the LIBS intensity spectra of the unknown samples can be analysed by not only the material content but also by the concentration of the elements contained.
For a full description of the calibration method please read our editorial article in Photonics Views.

An example of the measured LIBS spectrum for a reference aluminium-based sample with the known concentration of Si, Mg, Mn, Cu as alloying elements is presented in Figure 3.

The detection limit is first defined by measuring the lowest concentration of the elements within the reference sample. These measured values of 0.12 % for Si and 0.06 % for Mn identify the detection limit or as often referred to as the concentration sensitivity of the system.

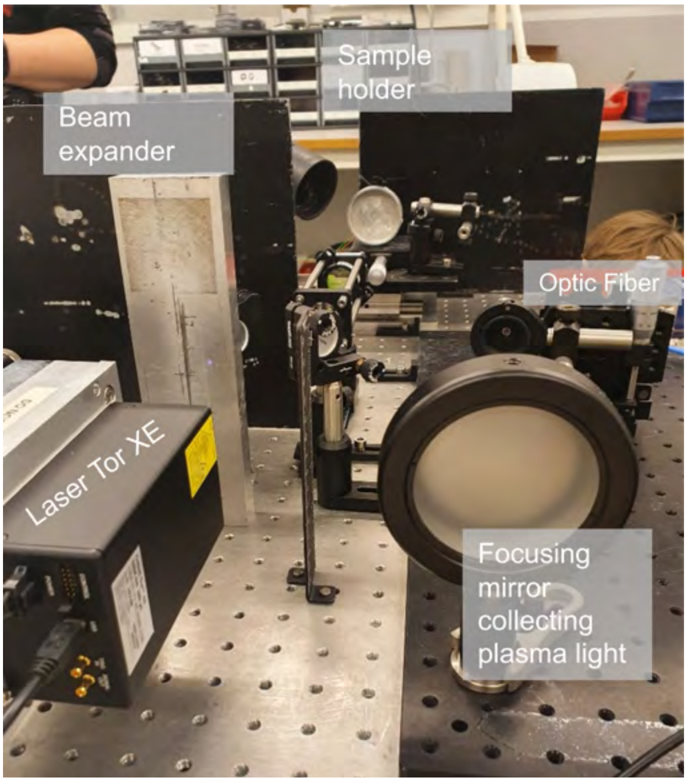

Figure 2. Photo of the experimental setup

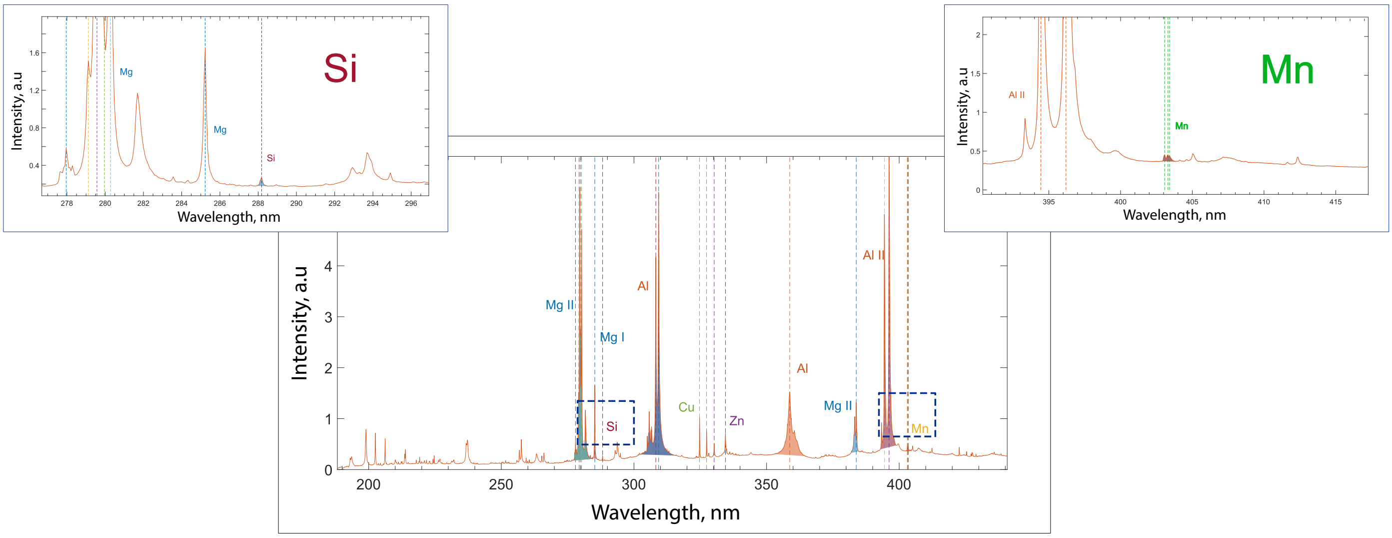

Figure 3. LIBS spectrum of an aluminium reference sample obtained with the Cobolt Tor™ XE laser at 1064 nm. This data is used to identify the detection limit of the system..
Inset: Si emission line with concentration of Si down to 0.12% and the Mn emission line with concentration of Mn down to 0.060%.

LIBS measurements on scrap metals

Qualitative LIBS measurements were performed on aluminium scrap samples with a rough (not planar, variation of ca. 5 mm) and contaminated surface with unknown alloying elements. Detectable qualitative LIBS signals were achieved and are shown in Figure 4.

The spectra of the samples in Figure 4a clearly indicate the presence of different alloy content, for example Mg and Mn. The spectrum of the sample in Figure 4b, indicates the presence of Mn in the Al matrix as well. On top of that, a high sensitivity to surface contamination was observed. Figure 4b indicates that the sample surface is contaminated with dirt, as spectral lines of Ca overlap with the aluminium spectra. The sensitivity to the LIBS signal to the surface contamination can be considered as an advantage for studies related to a surface analysis.

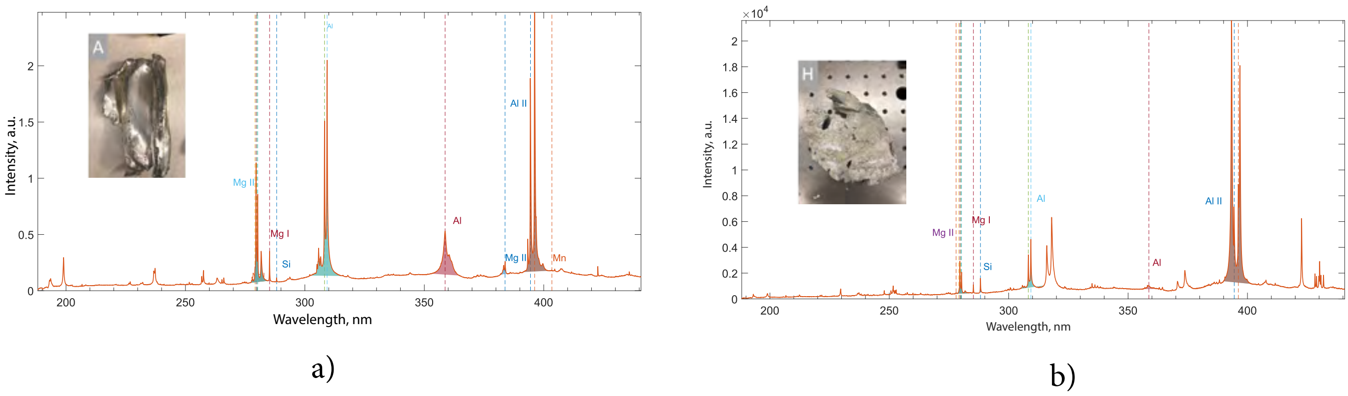

Figure 4. LIBS spectra of aluminium scrap samples

Using the same experimental setup, a representative qualitative LIBS signal was also obtained for a slag sample, which typically has a rough surface and is quite inhomogeneous (Figure 5). The spectral lines of typical slag elements (Ca, Mg, Si, Fe, Al) are clearly observed confirming that this set-up could be used for slag analysis. The slag composition is often a key parameter in both process and product development in steel- and iron-making.

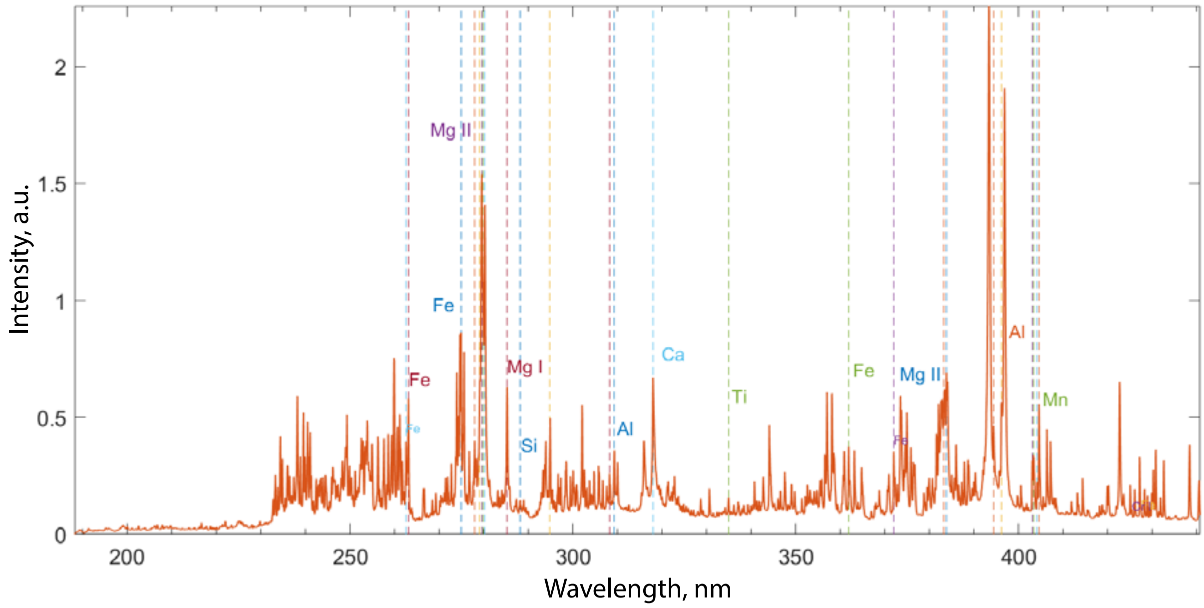

Figure 5. LIBS spectrum of slag material obtained with the Cobolt Tor™ XE

Outlook

The LIBS technology has great potential for being used in various tools for industrial and in-the-field material analysis.
This study has demonstrated how a compact, high-repetition rate, ns-pulsed 1064 nm laser with 0.5 mJ pulse energy in a high-quality beam enables detailed analysis of metal samples using a conventional LIBS set-up. Despite the small number of reference samples, the results obtained are promising and confirm the practical applicability of a LIBS system based on this compact, self contained laser for quantitative LIBS analysis.

The high-quality laser beam with short nanosecond pulses and low pulse to pulse jitter results in high energy densities and efficient synchronisation with detection, thereby providing strong LIBS signals that allow for analysis with a high level of sensitivity and specificity, all with sub mJ pulse energies. The compact size of the air-cooled Cobolt Tor™ XE laser with all electronics integrated into the laser head can drastically reduce the size of the LIBS system making it suitable for use in bench-top analytical instrumentation.

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