High-resolution High-Speed LIBS Imaging

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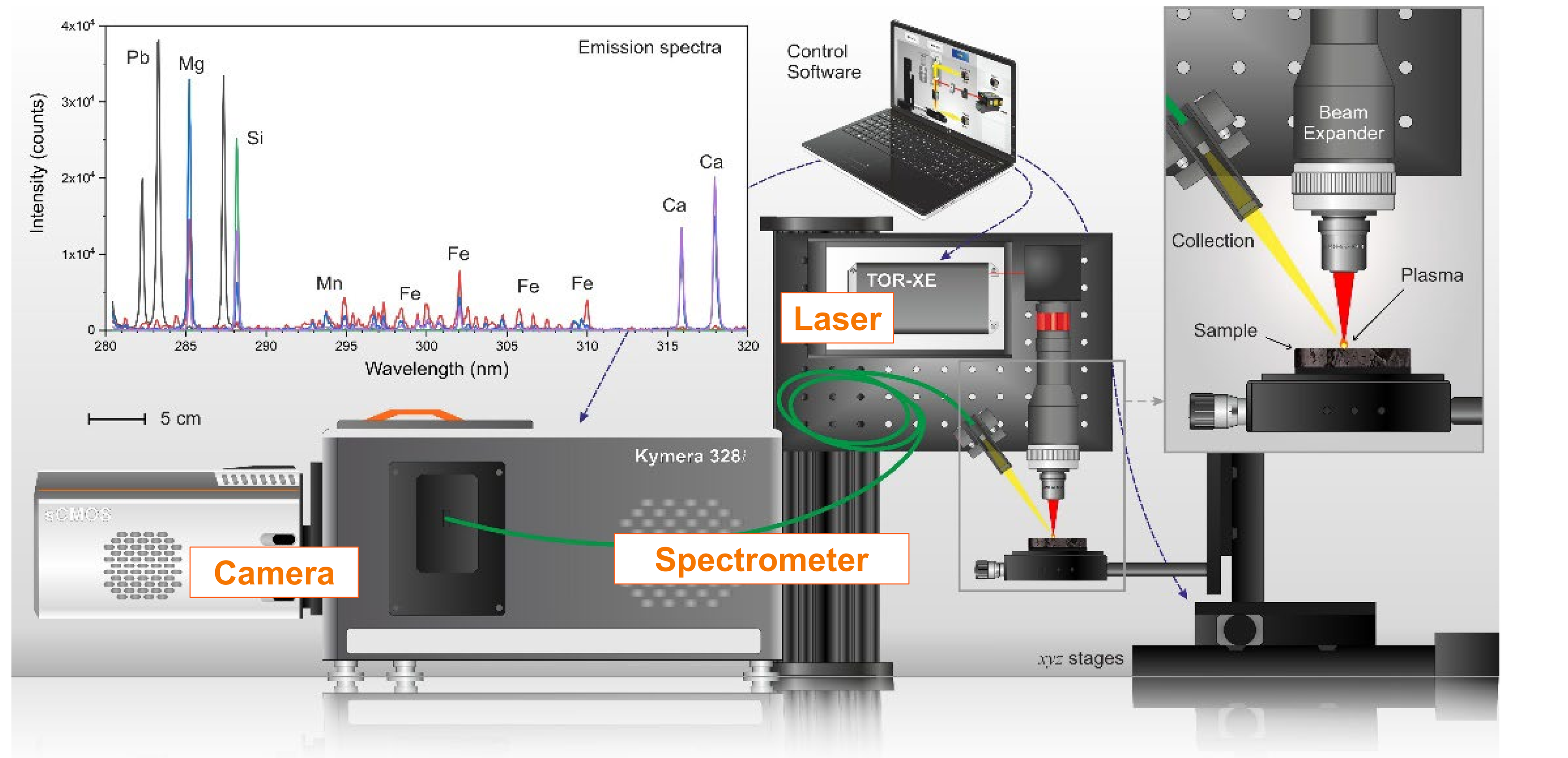
Motivation

Laser Induced Breakdown Spectroscopy (LIBS) is becoming increasingly prevalent as an analytical technique for material content due to its high sensitivity and the capability to detect any element of the periodical table with nearly no requirements or limitations on sample dimensions, consistency, and/or surface quality. **LIBS imaging** has expanded the capabilities of LIBS technique even further [1]. LIBS imaging enables the reconstruction of elemental or mineralogical maps which show the distribution of the elements or minerals within an analyzed sample. Typically, excitation sources, such as lasers, with a repetition rate between 10-100 Hz are used for LIBS analysis. These systems have traditionally provided a sufficient balance of sampling frequency and laser pulse energy. However, for high-resolution µm-scale LIBS images of large sample areas (several cm²) a higher repetition rate is required to avoid unreasonably long acquisition times. This work demonstrates an approach to reduce the acquisition time for **high resolution µ-LIBS imaging** by using a laser operating in the kHz frequency range [2]. The faster laser repetition rate enabled the development of a **µ-LIBS imaging microscope** capable of resolving images with about **10 µm resolution at < 20 min/cm²**. For the first time, **images of such high resolution** showing detailed elemental distribution within an analyzed sample have been achievable. The capability of the system to perform fast LIBS analysis over large samples and revealing the spatial distribution of elements within the analyzed area opens opportunities for application within a wide variety of fields of research including **biomedical and geological material analysis**, as well as **in industrial settings** such as mining.

µ-LIBS imaging microscope

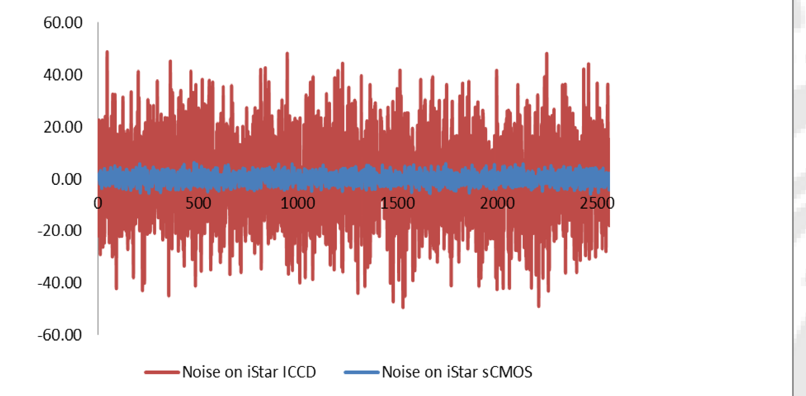


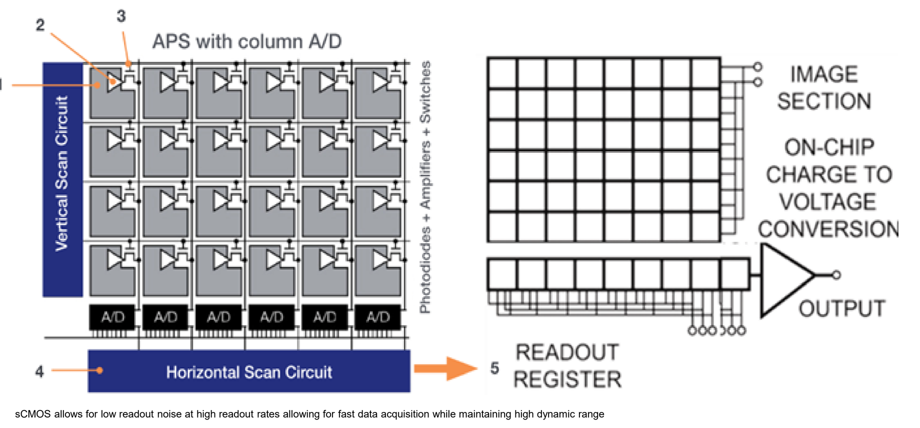
The proposed µ-LIBS imaging system is **a custom-made LIBS µ-microscope** capable of resolving images with about 10 µm resolution at < 20 min/cm². The system is based on a compact nanosecond Nd:YAG Q-switched 1064 nm laser (Cobolt Tor™ XE), with a pulse energy of 0.5 mJ and a pulse duration of 2.5 ns. The laser beam is focused down to 10 µm spot. The plasma emission lines are recorded with a spectrometer (Kymera 328i from Andor Technology) coupled to a sCMOS camera (iStar from Andor Technology).

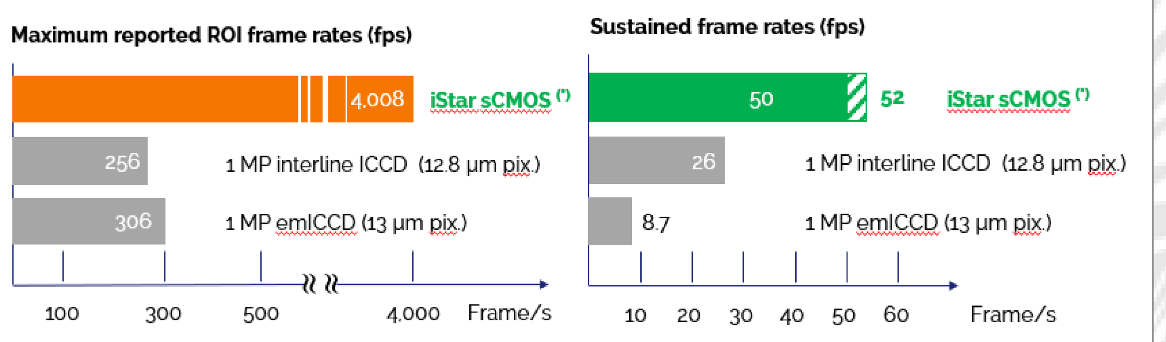
iStar from Andor Technology

Fast gated camera based on sCMOS sensor technology

	iStar sCMOS (13 µm pix [2x2 bin])	1 MP Interline ICCD (12.8 µm pix.)	1 MP emICCD (13 µm pix.)
Pixel read noise (full frame max. rate)	5.2 e-	70 e-	90 e-
Gain to overcome sensor noise floor	~X31	~ X420	~ X540* *dual MCP and EM gain
Pixel well depth	120,000 e-	130,000 e-	400,000 e- (EM register)
Dynamic range	~3,870:1	~310:1	~740:1







Latest generation fast gated cameras based on sCMOS sensor technology provide significant enhancement in terms of high acquisition rates and simultaneously high dynamic range compared to CCD, Interline or EMCCD-based gated detectors.

Cobolt Tor™ XE

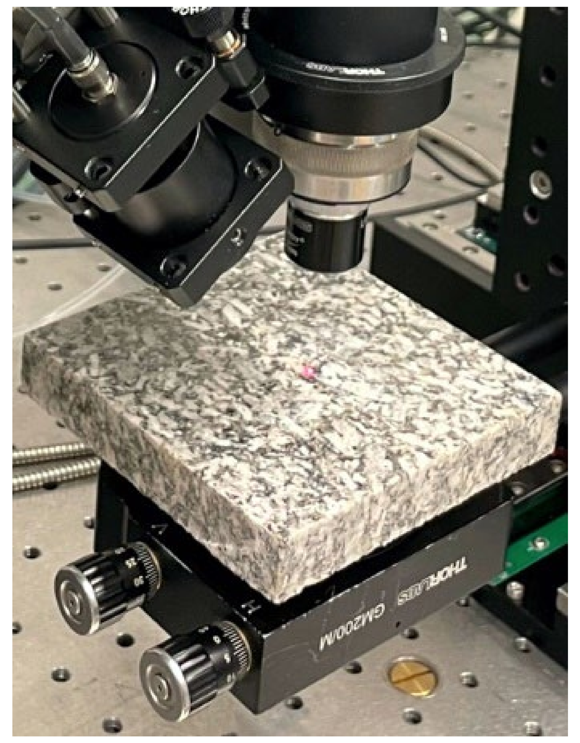
Compact air-cooled ns-pulsed laser

Cobolt Tor™ XE

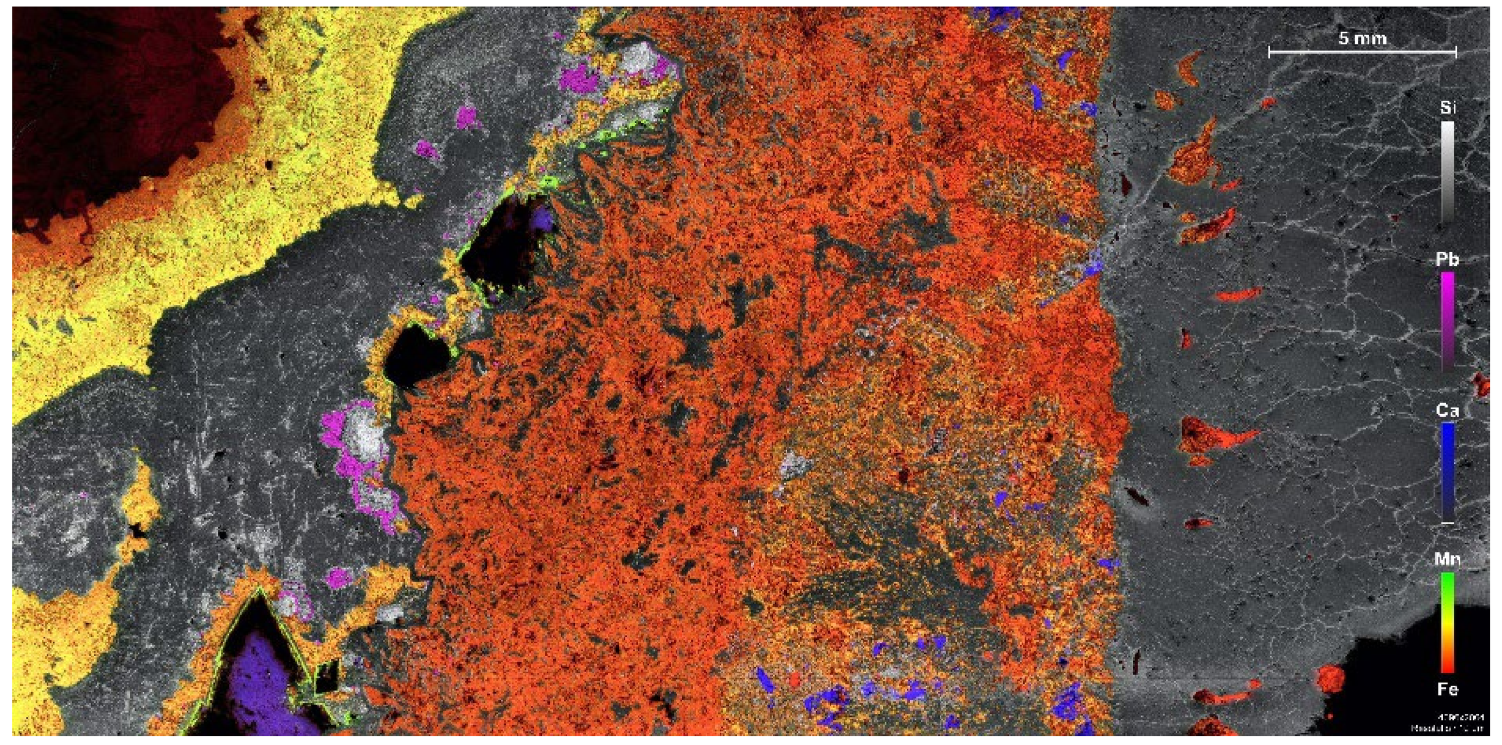
Pulse energy (µJ) @ 1064 nm	500 ± 50
Pulse length (ns)	1 - 3
Spatial mode (TEM00), M²	< 1.3
Pulse jitter (µs)	< 2
Power stability (8 hrs ± 3°C)	< 3
Beam diameter (mm)	0.7 ± 0.1
Repetition rate (kHz)	up to 1 kHz
Trigger source	External, Internal, Gated

In LIBS systems, the choice of the laser used for material ablation plays an important role. LIBS measurement repeatability can be linked to laser performance as the plasma generation relies on non-linear processes. Any fluctuations of laser parameters such as **pulse energy** (or **pk-to-pk stability**), **pulse duration, and beam quality**, could produce fluctuations in ablated mass, plasma temperature, and electron density, which would be reflected in the emission spectrum and could lead to analytical inaccuracies. Laser performance is even more critical in LIBS imaging, where raster scanning of the sample typically means using a **single laser pulse** per acquisition point. **The quality** of the beam M² is associated with the ability to focus the beam into a spot which defines the spatial resolution of the system, thus, the min.pixel size of an image. **The repetition rate** of the laser defines the maximum acquisition/scanning speed available. **The footprint** of the laser and **the ability to air-cool it**, can be big advantages for system integration.

High-resolution 10 µm high-speed < 20 min/cm² LIBS image



a) Photo of a sample placed under the µ-LIBS microscope.



b) Reconstructed µ-LIBS image showing the distribution of silicon (Si), lead (Pb), calcium (Ca), magnesium (Mn), and iron (Fe) in a sample.

The **high-speed, high-resolution µ-LIBS imaging results above were enabled by gated sCMOS technology and an ultra-stable pulse kHz laser.** To demonstrate imaging capabilities of the µ-LIBS imaging microscope over large sample areas, a **rock sample with an area of about 6 cm²** was used. The sample was polished with SiC paper under water solvent to obtain a planar and clean section. The image shows the distribution of silicon (Si) for quartz, lead (Pb) for galena, calcium (Ca) and magnesium (Mn) for carbonate, and iron (Fe) for pyrite in a sample **with 10 µm resolution. The reconstructed µ-LIBS image was completed in less than 2 hours (< 20 min/cm²)**, to put it into perspective, similar image would take 20 hours to complete with a laser operating at 100 Hz [3].

Summary

- **A high-speed high-resolution µ-LIBS imaging system, µ-LIBS microscope,** capable of reconstructing elemental compositional images with **10 µm resolution over a sample area of 6 cm² in a dramatically reduced time of < 2 h** which equates to < 20 min/cm².
- **A drastically reduced time for material content analysis** of relatively large sample area with µm-resolution can ultimately **provide unique capabilities** and **opens new perspectives for LIBS imaging and LIBS analysis** in many application fields, and in geological material analysis specifically.

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