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APPLICATION NOTE:
LIBS IN THE PHYSICAL SCIENCES

Techniques

Laser Induced Breakdown Spectroscopy

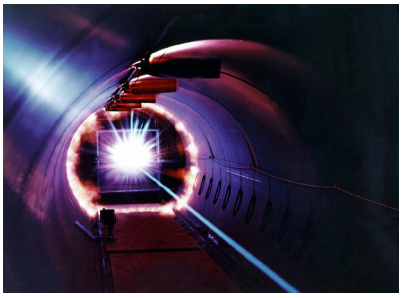
Keywords

- Laser ablation
- Atomic emission
- Material identification
- Plasma

Introduction

Laser-Induced Breakdown Spectroscopy is a type of atomic emission spectroscopy that employs a laser to ablate or vaporize a microscopic layer of a sample's surface. The resultant plasma caused by this laser ablation process emits light as it cools. This light is then collected and analyzed with a spectrometer for quantitative and qualitative material analysis. This virtually nondestructive spectral analysis method has valuable applications across numerous physical science fields. There are several advantages in LIBS compared to other available analytic methods as well, and Avantes is your trusted partner for the development of customized LIBS systems.

LIBS Background



Laser Induced Breakdown Spectroscopy (LIBS) is a process for material analysis that employs a very short-duration pulsed laser (usually a Nd:YAG 1064nm Laser) to excite particles at the sample surface. Such excitation by the laser causes the breaking of chemical bonds and produces vapor, aerosol particulate, and high temperature microplasma.

Plasma, the ionized gas produced in laser ablation, can reach temperatures as high as 15000 degrees Kelvin, but cools rapidly. During the cooling phase, plasma emits light that, when analyzed, reveals spectral peaks much like a chemical fingerprint. Every element on the periodic table emits light in the 200-900 nm spectral range and will exhibit its own unique spectral signature. This spectral signature is the theoretical foundation that allows scientists to use LIBS measurements for material qualification and quantification.

The light emitted by the plasma can be collected with fiber optics and delivered to a spectrometer for spectral characterization. The spectrometer transfers spectra data to the computer control system for data processing and analysis.

When reference spectra are available, comparing the sample against spectra references might be relatively simple. More commonly, LIBS spectra must be analyzed using a variety of multivariate analysis processes to provide for qualitative and quantitative measurements. Then again, theoretical models that depend on fundamental assumptions must always be tested and verified against real world applications as any application will have its own unique calibration curve.

However when reference calibration data is not available or the composition of the sample is unknown, methods have been developed for calibration free approaches. These methods involve multiple rapid sampling and often apply mathematical models that simulate probabilistic optimums.

A variation of the single pulse LIBS

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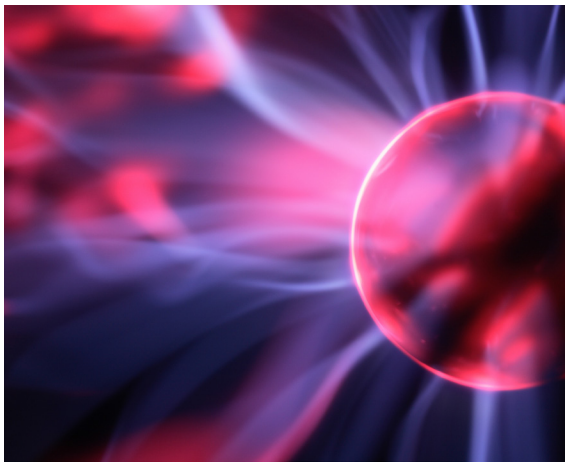
approach has also been developed. The use of a second pulse has become popular for some applications, especially with liquid measurements. In the double pulse method, the first laser pulse creates a minute plasma filled bubble while the second pulse further excites the plasma to produce a more intense light for measurement.



The Advantages of LIBS measurements

Laser Induced Breakdown Spectroscopy is a highly useful research and analysis tool. LIBS analysis is very versatile because it can be used on any material, whether solid, liquid, or gas, and will detect any and all chemical elements in a sample with a single pulse. LIBS is especially sensitive in the detection of light elements like Helium, Lithium, Beryllium, Nitrogen, and Oxygen that are not easily detected by other analytic methods.

Unlike many other investigative tools, LIBS spectroscopy requires little to no sample preparation. This lack of sample preparation supports field applications and real-time, in-situ LIBS measurements.

Furthermore since the sample size per pulse (µg to ng) is so small it may be considered to be virtually non-destructive on a sample's surface. And yet, it is sensitive enough to measure at resolution down to a single grain (below 10 µm) and powerful enough to bore a microscopic crater in a solid sample to target a minute mineral inclusion or individual particle.

LIBS Applications in the Physical Sciences

Interior Nerja Cave. Malaga, Costa del Sol, Spain

Researchers Lebedev and Shestakov of the Institute of Laser Instruments and Technologies, Ustinov, St. Petersburg, Russia produced a series of experiments demonstrating the use of LIBS spectroscopy identification of solids. They document the use of a Q-switched diode pumped Nd3+-YAG laser emitting 20-100 mJ with a repetition rate of generation reaching 30 Hz and a pulse duration of 10ns.

Lebedev's system pairs the excitation laser with the AvaSpec-ULS2048-USB2 spectrometer by Avantes . The AvaSpec-ULS2048 features an ultra-low straylight, symmetrical Czerny-Turner spectrometer with a linear 2048 pixel array CCD detector. This spectrometer is often chosen for LIBS measurements, especially in a multi-channel or arrayed configuration. Each channel in the array measures a short region of the spectrum (a few hundred nanometers) with very high resolution. The AvaSpec-ULS2048 spectrometer is popular for use in many applications in the physical sciences, from geology and metallurgy to environmental and climate science measurements, and scientists worldwide rely on Avantes spectrometers for LIBS measurements.

Rock, Sediment, and Soil Analysis

Minerals are the fundamental building blocks of most rocks and soils with more than 4,000 identified minerals on Earth. Knowledge about chemical composition is essential to understanding the formation and characteristics of any rock or soil body. The field capabilities and prep-less sampling of LIBS measurements explains why this spectroscopic technique is commonly used across many different areas of the geosciences.

Mineral Automated Yield Analyzer MAYA

LIBS spectroscopy was employed in the characterization of stalagmites for Magnesium and Strontium in the Caves of Nerja near Malaga, Spain. Researchers isolated deposits of manganese (Mn), magnesium (Mg), strontium (Sr), calcium (Ca) and iron (Fe) in speleothems taken from the the caves.

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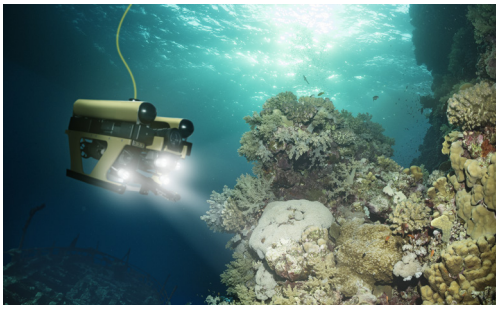


Deep ocean exploration vehicle

Deep Ocean Analysis

The short laser pulse generated in LIBS measurements, when focused on a liquid will cause dielectric breakdown, and the instantaneous heating produces explosive expansion resulting in a gas vapor bubble. The spectral emissions are less intense when using a laser pulse focused into a liquid due to absorption of the energy by water, and from diffraction and scattering due to particles and microbubbles. The plasma is also rapidly quenched. The second pulse LIBS method is particularly useful when performing LIBS analysis in liquids.

Researchers working to develop deep sea LIBS methodology had to overcome challenges posed by environmental conditions. Several spectroscopic parameters are affected by high-pressure, including energy input requirements and spectral emission intensity. Recent developments, however, have lead to the development of LIBS spectroscopy systems rated to 3000 m below sea level and tested to 1000m.

Ice Core Sampling

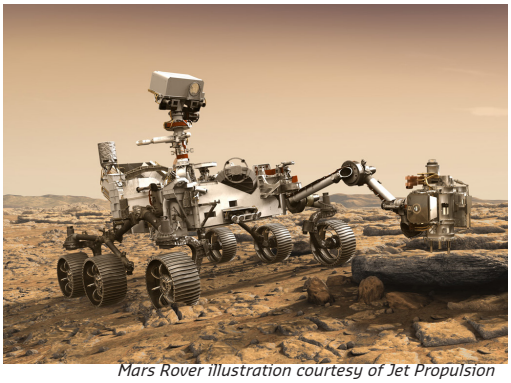
Pollution Monitoring

Climate and environmental testing and monitoring are in-demand fields. LIBS systems have been deployed for continuous, in-line monitoring for industrial pollution in natural waterways where pollution is problematic.

Polar Ice Research

Another area where LIBS technology is finding use is the study of polar and alpine glacial ice. Glacial ice formed over millennia of Earth's history, has trapped air and particles from the time the ice was forming. Using laser-induced breakdown spectroscopy to study the composition of ice core samples contributes to our understanding of the role of atmospheric carbon dioxide (CO2) on Earth's climate.

Space Exploration



Mars Rover illustration courtesy of Jet Propulsion Laboratory

Laser-induced breakdown spectroscopy has been widely used in space exploration in the investigation of extraterrestrial bodies, such as meteorites, using a calibration-free (CF-LIBS) approach. Expect LIBS systems to take center stage on future missions to Mars, as well. It will be critical to future Mars missions to be able to understand Martian ecology with complete mineral and chemical analysis.

Industrial Applications for LIBS

It is true that laser-induced breakdown spectroscopy is a highly regarded research tool, but this measurement technique has important applications in industrial environments as well.

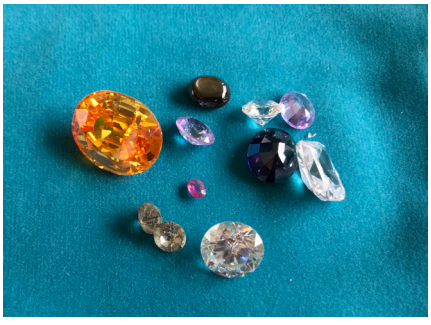
Semiconductor

Metals and metal alloys are used to transport electrical signals in semiconductor components. Manufacturing these increasingly complex devices requires thin film coatings and substrates applied to the wafer. These films which typically are around a few hundred nanometers thick are deposited onto a silicon wafer before and after treatments, such as thermal cycling to strengthen it. Laser-induced breakdown spectroscopy may be used in semiconductor wafer and coating characterization and quality control.

Gemology

As a minimally destructive techniques, LIBS may be used in gemstone validation and characterization. Laser beam shaping allows for micro ablations of materials such that the measurement spot size is 50 microns in diameter. Mining and metallurgy

LIBS is superbly suited to the requirements of the mining and metallurgical industries. The potential for high speed sampling without sample preparation allows for in line analysis of ores and metal alloys during extraction and processing. The Avantes EVO electronics platform facilitates high speed USB3 or gigabit ethernet communication with a control system is well suited for such applications.



Gemstones

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Working with Avantes

Laser Induced Breakdown Spectroscopy, like so many technologies, has experienced rapid advancements over the past decade, and will continue to find new applications and uses in the years to come.

Avantes is a world leader in the design and manufacturing of optical spectroscopy instruments with twenty-five years of experience and tens of thousands of systems in the field around the world. We work collaboratively with our customers to test and design the optimal instrument configurations for their unique applications.

Contact our Louisville, Colorado office today to speak with one of highly knowledgeable sales engineers by calling 303-410-8668 (toll free 1-866-678-4248) today or email us at infousa@avantes.com.

You can also learn more about this and other spectroscopy applications employing Avantes instruments on the web at www.avantesusa.com.

Further Reading

Read our editorial “Laser-Induced Breakdown Spectrocopy: Beyond the Lab” published in Photonics Spectra magazine.