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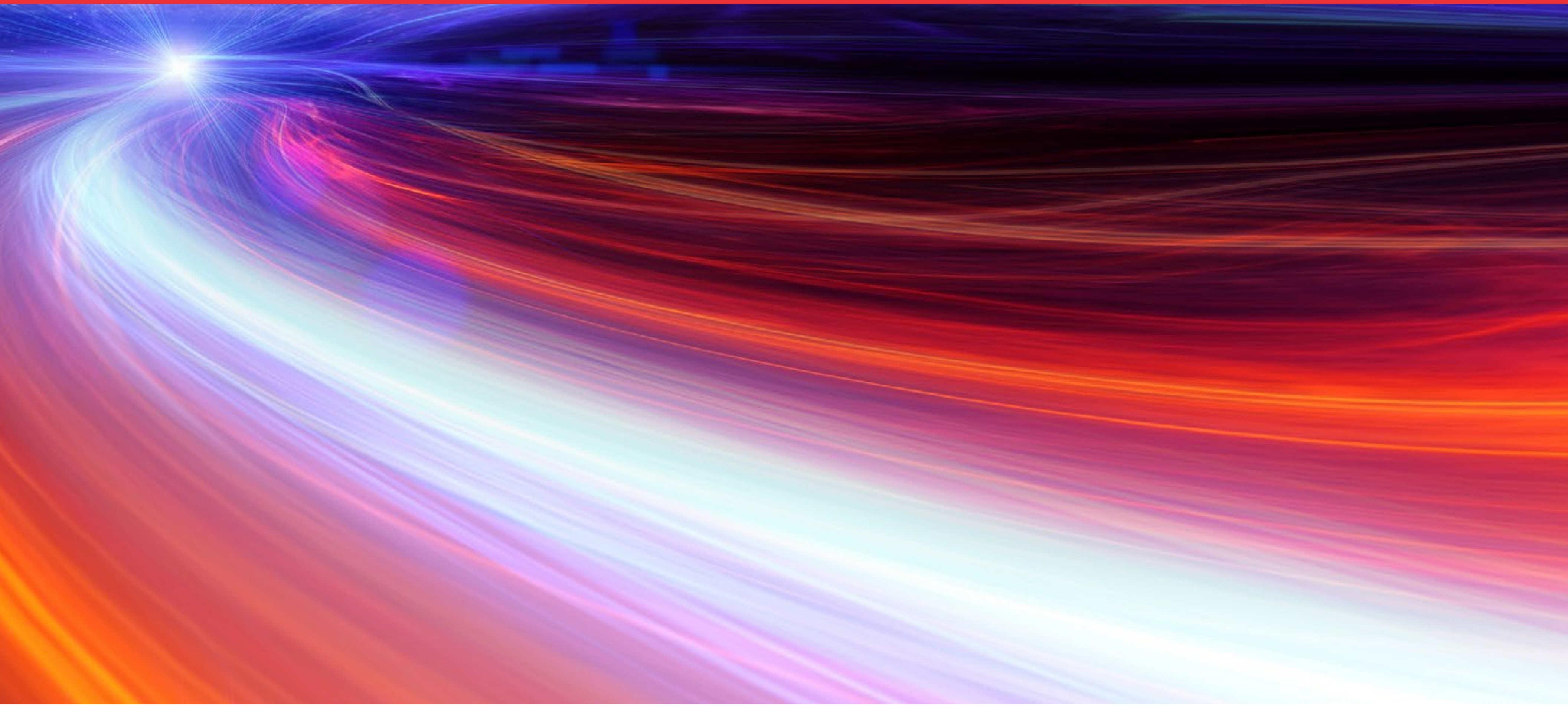

LIBS technology for non-scientists

Laser induced breakdown spectroscopy

ThermoFisher SCIENTIFIC

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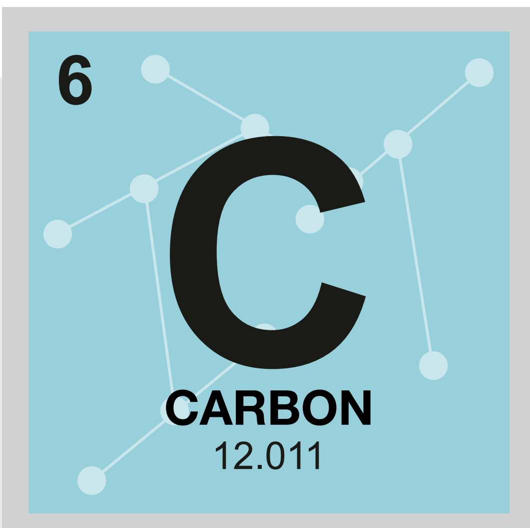
Overview

What is LIBS?

Overview

Laser Induced Breakdown Spectroscopy (LIBS) is the analytical technique using a high-focused laser to determine the chemical composition of materials. LIBS has been around for many years and is a technique used primarily in laboratory equipment. With recent advances in technology, the technique has now been developed into a portable handheld analyzer capable of measuring elements, including carbon, in the field for material identification.



Each element of the periodic table produces a unique LIBS spectral peak. By using a detector to measure the unique characteristics of light emitted, it is possible to detect what elements are present within the sample. By measuring the peaks of light and their intensities in the sample, the chemical composition can be rapidly determined and quantified in weight percent concentrations (%).

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Exploring the periodic table

Number of protons = Atomic Number (different for each element).

Number of electrons typically = number of protons (so that the atom is neutral).

Number of neutrons is variable and is what allows some atoms to have isotopes.

Electrons in shells closest to the nucleus are most strongly bound to the atom. Binding energy increases with atomic number. The higher the number, the higher the weight.

An isotope of an element has the same number of protons but a different number of neutrons.

Chemical elements are a species of atoms, where all atoms contain the same number of protons in the nucleus. This is a substance that cannot be broken down into any simpler substance and still retain the nature of the element.

The elements are arranged in increasing order of their number of protons and electrons.

1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba		72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra		104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn	113 Nh	114 Fl	115 Mc	116 Lv		

57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

DID YOU KNOW?

The periodic table was created in 1869 by Dmitry I. Mendeleev.

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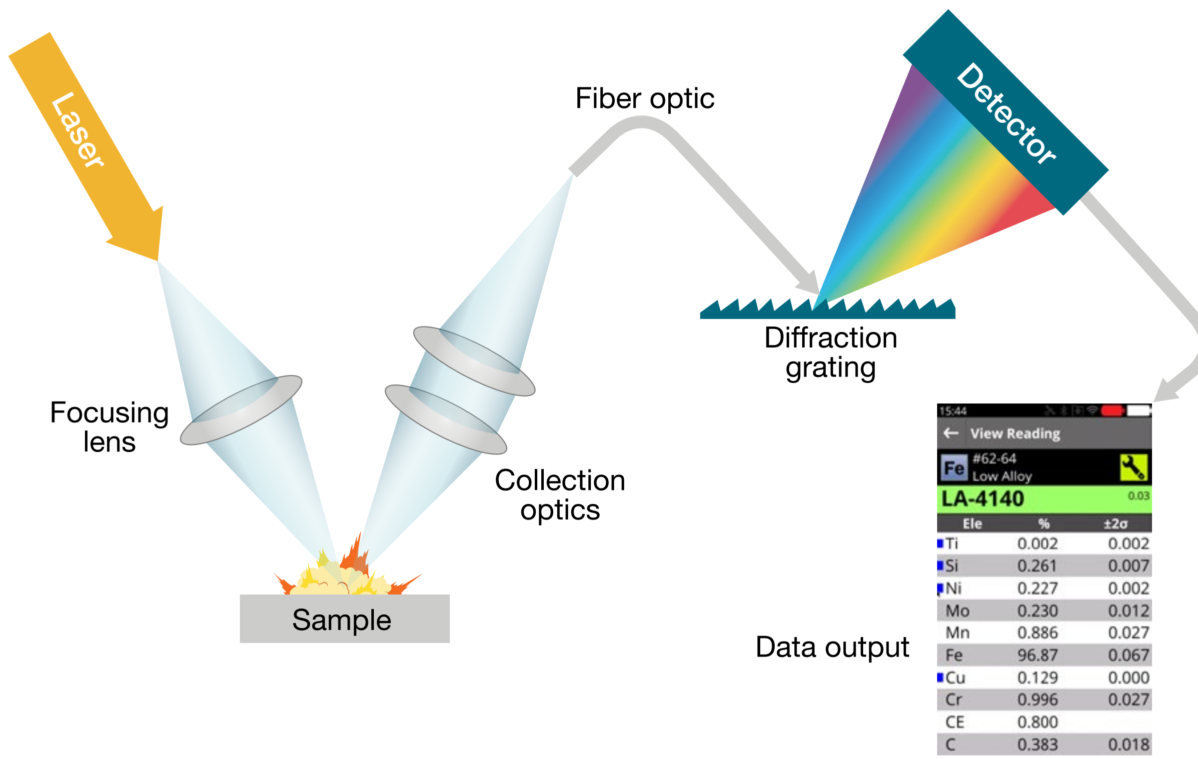
The LIBS analysis process

The LIBS technique utilizes a high-focused laser that interacts with the surface of a material and forms a plasma in which the material is broken down into single elements.

1. A laser pulse is produced by the analyzer and pointed at the sample surface.
2. The surface is ablated and enters the plasma. The plasma atomizes the samples, and the excited atoms emit light as they return to ground state.
3. The emitted light is transferred through fiber optics and enters the spectrometer through a slit.
4. The light interacts with a diffraction grating where it is split into single wavelengths/ colors.
5. The single wavelengths/ colors hit the detector and produce spectral data.
6. The central processing unit (CPU) analyzes the spectral data and determines the concentration of each element present in the sample.
7. Composition data and identified alloy grade are displayed and stored via memory for later recall or download to an external PC.



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Where is LIBS used?

Oil and gas

For positive material identification (PMI) of piping, pressure vessels, valves, pumps, and finished welds, or to grade unknown materials to regain traceability.

To calculate carbon equivalency prior to welding to determine heat affected zone hardenability (HAZ).

Metal fabrication

To validate Material Test Reports (MTRs) prior to adding value during the fabrication process.

For quality control and quality assurance (QA/QC) of outgoing finished goods.

Scrap metal recycling

For fast and accurate sorting of low alloy steel grades.

Enhanced separation of stainless steel grades based on carbon.

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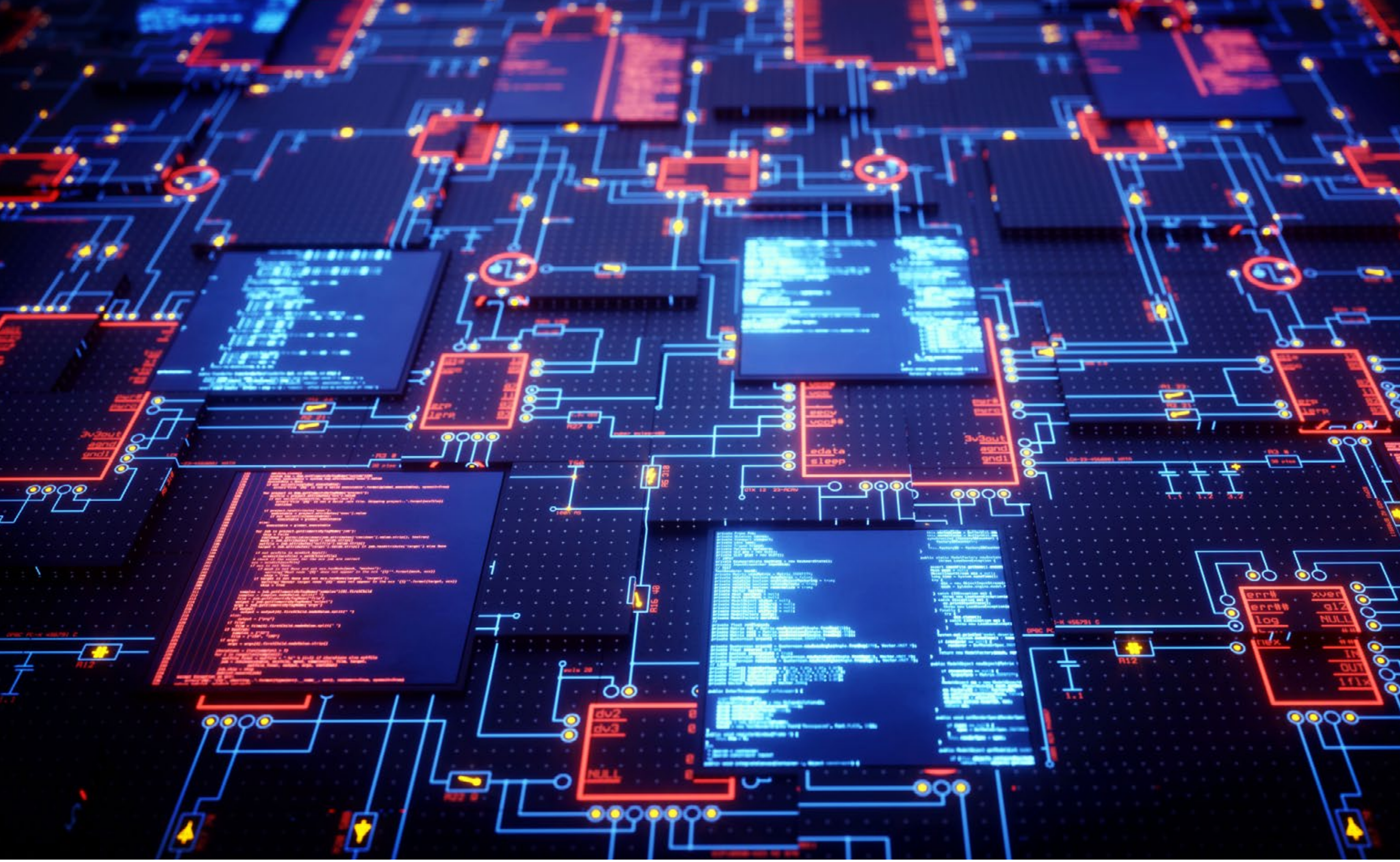
PDF For more information, download the application note: **Analysis of Carbon Equivalents in Steel Components**



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Technology

The handheld LIBS instrument

Handheld LIBS analyzers contain the following components:

Laser
Ablates the sample surface

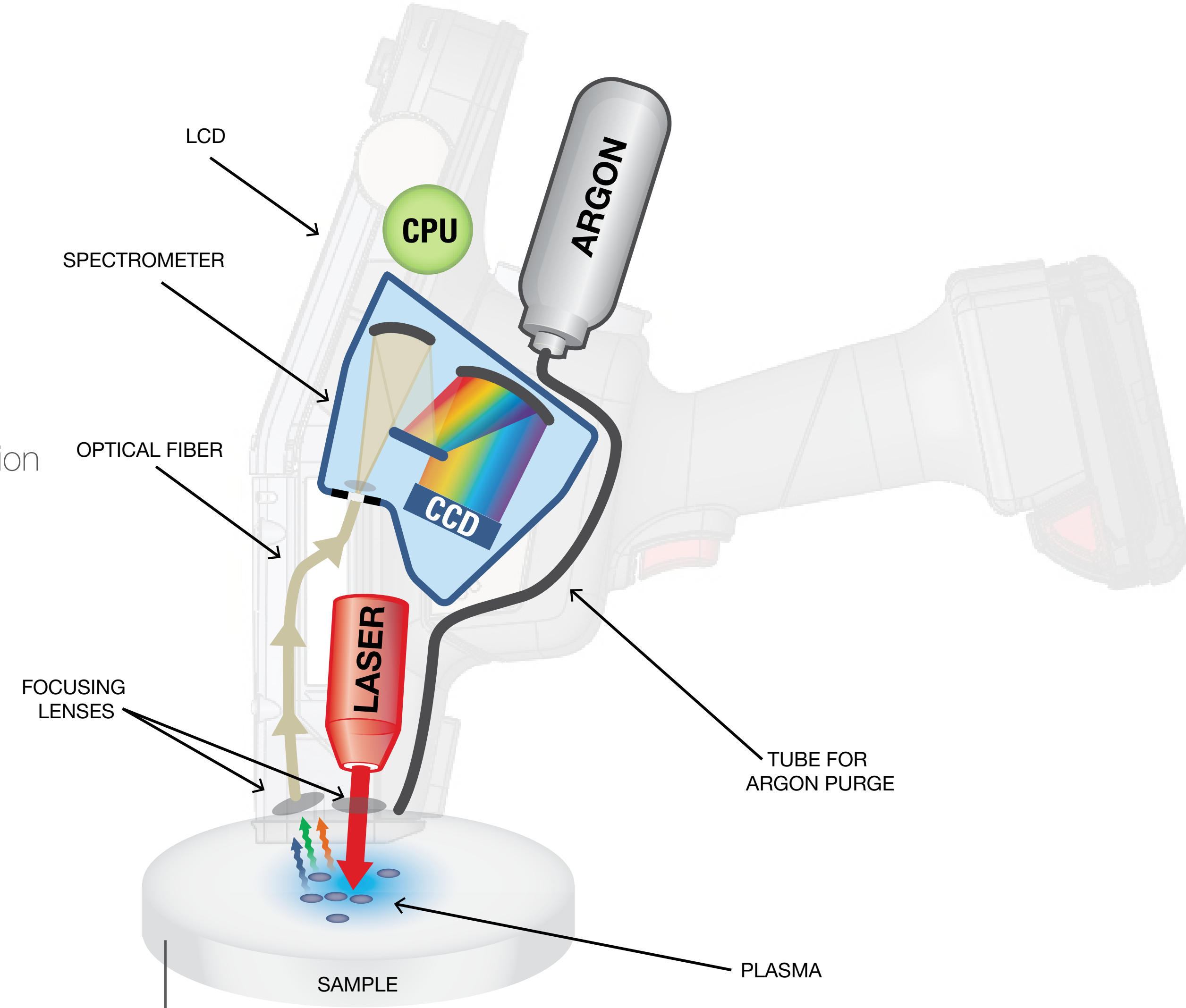
Optical Fiber(s)
Transfers light to the spectrometer

Spectrometer(s)
Measures sample spectra

Argon Cartridge
Assists with plasma formation and stabilization

Optical Lens(es)
Focuses the laser and collects emitted light

Central Processing Unit (CPU)
Provides signal processing



Sketch of a handheld LIBS instrument

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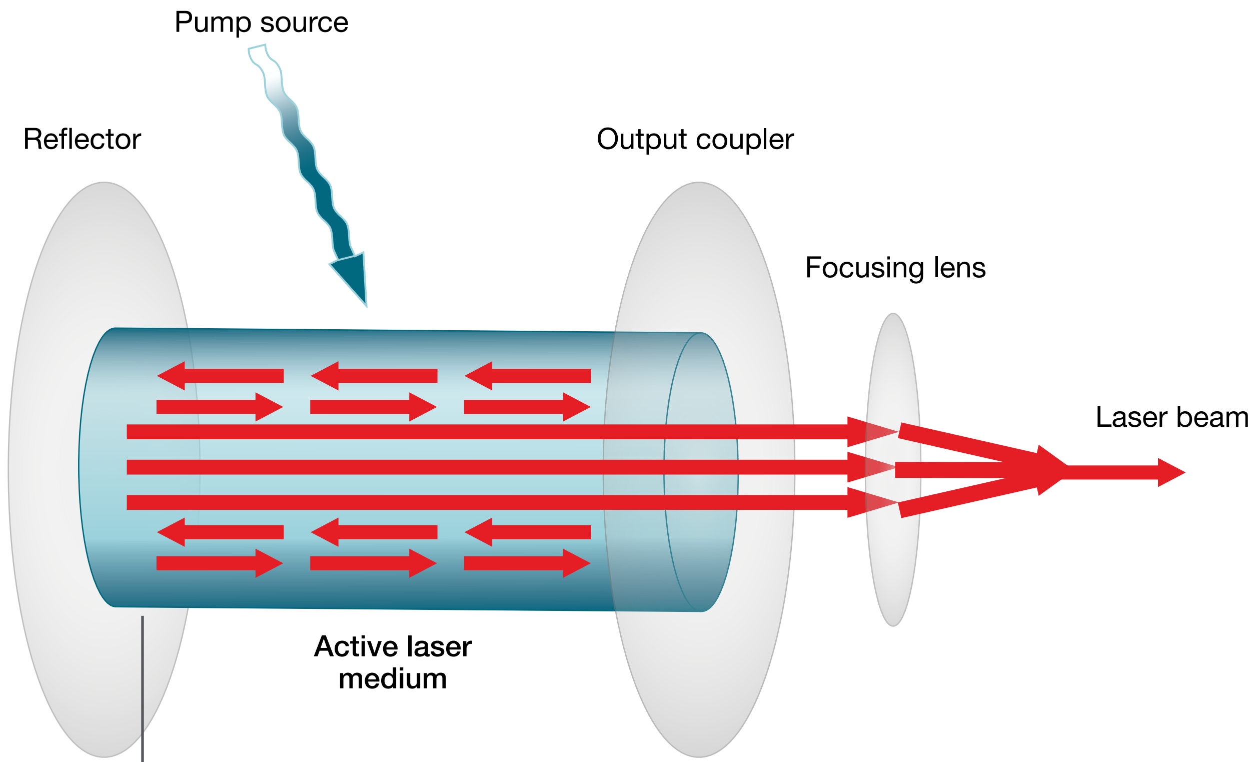
Lasers

Most handheld LIBS analyzers utilize a pulsed laser with a wavelength of 1064nm. Short pulses (nanoseconds) of high energy produce enough power per unit of area to ablate a small amount of material (about a nanogram) and produce a plasma at the sample surface.



Working principle of laser

DID YOU KNOW?

LASER is the acronym for Light Amplification by Stimulated Emission of Radiation.

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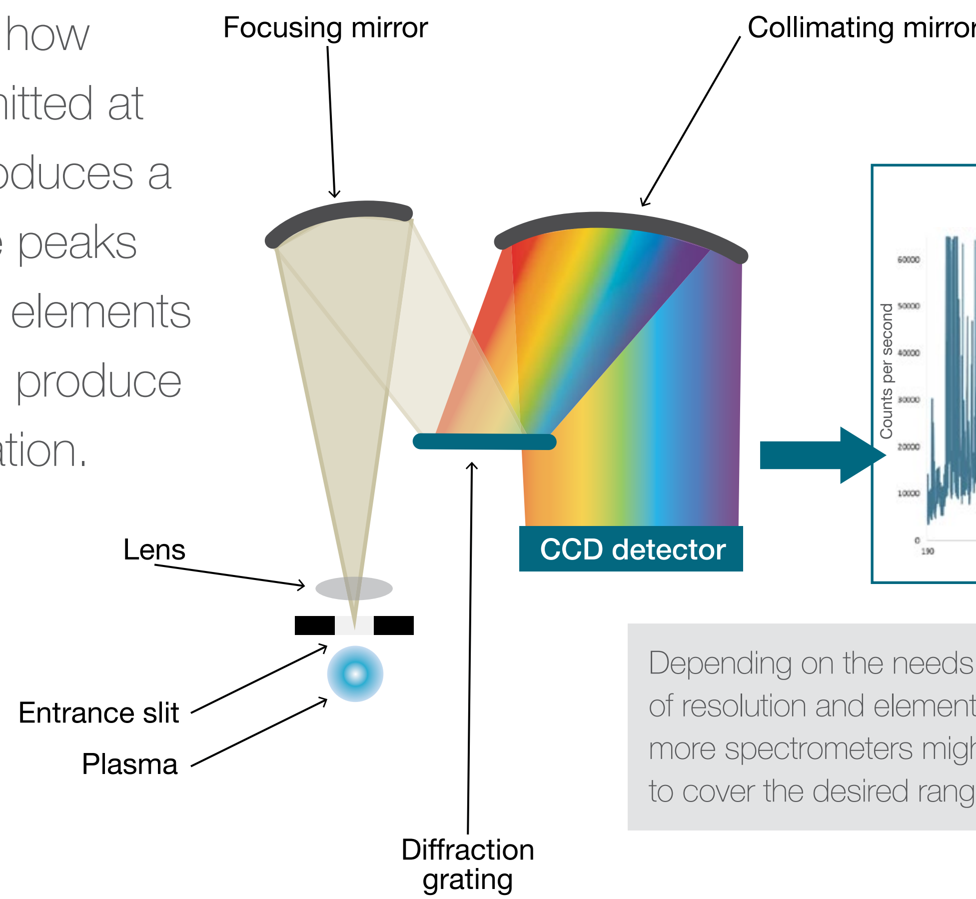
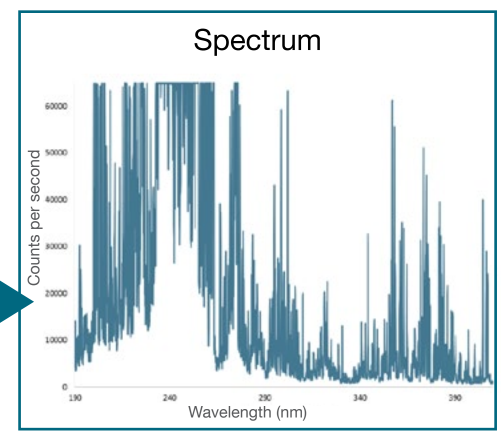
Spectrometers

The light from the plasma is polychromatic (white light), which means it contains multiple different wavelengths. This white light is split into its component wavelengths by the diffraction grating, much the same way that white light is split into a rainbow of individual colors passing through a prism. Different elements emit specific wavelengths of light, and the intensity of that light is directly proportional to the concentration of element present.

The spectrometer measures how many photons of light are emitted at specific wavelengths and produces a spectrum of the sample. The peaks that are representative of the elements of interest are measured and produce a result to indicate concentration.





Depending on the needs of the application in terms of resolution and elements to be measured, one or more spectrometers might be built in the instrument to cover the desired range of wavelength.

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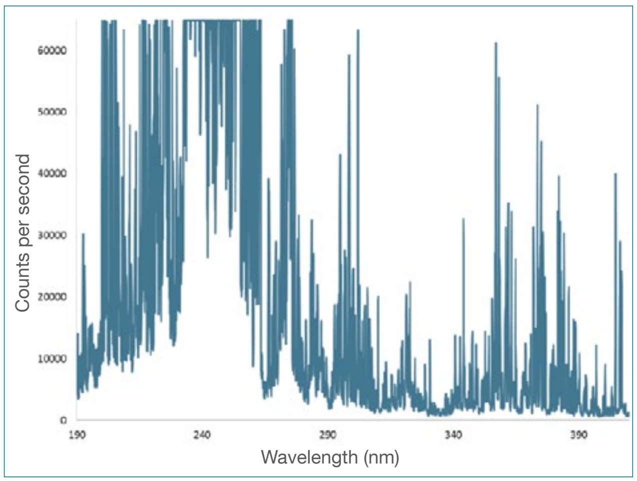
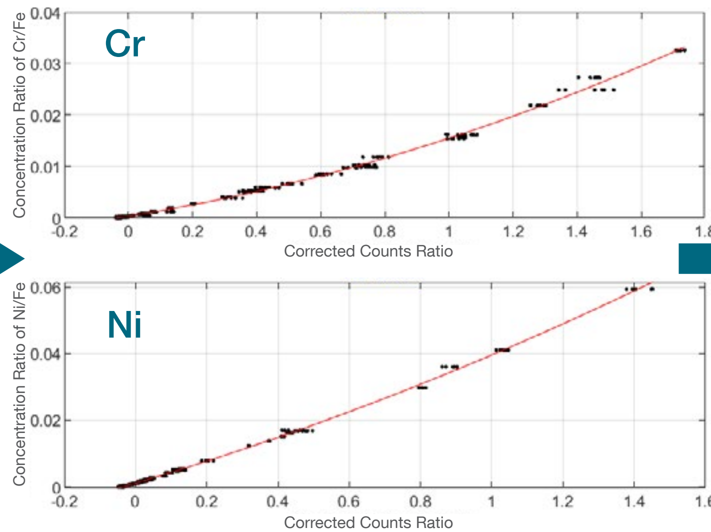
Signal processing

During LIBS analysis the measured intensity for an unknown sample is compared to the intensity recorded during the calibration process for standard samples with known concentrations. The concentration of the unknown sample is calculated for every element from the respective calibration curve and is displayed on the analyzer screen.

Spectrum



Calibration curves



Displayed results

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Calibration

In LIBS analysis, a calibration is a mathematical model that converts the measured intensity for a given element wavelength into units of concentration % (weight percent).

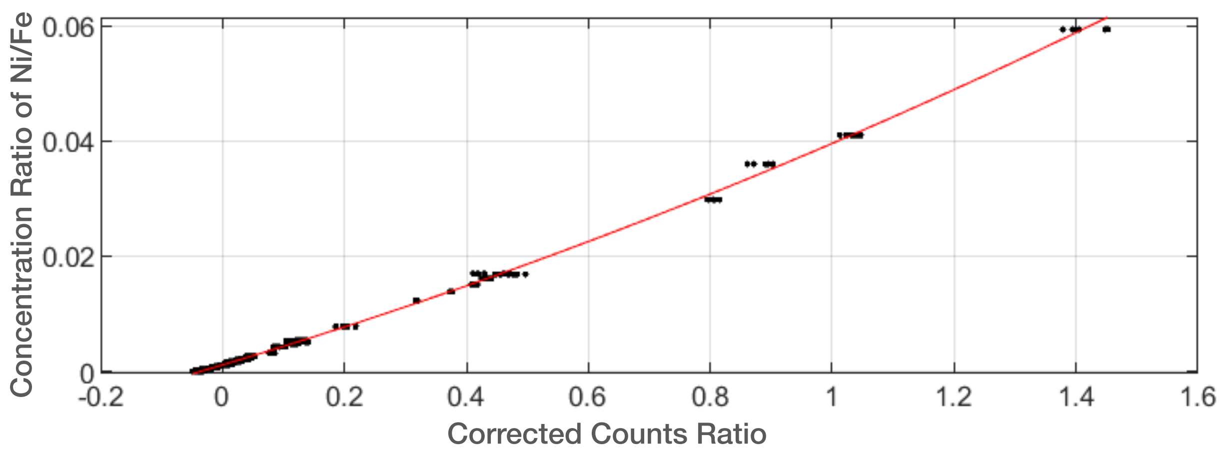
Empirical calibration

Empirical calibration utilizes experimental data to establish a mathematical model that is calculated after measuring the intensities at wavelength of the elements of interest for samples containing known concentrations.

Drift correction / Re-standardization

Drift correction requires measuring set up samples (SUS) and readjusting the wavelength scale as well as the response of the instrument to ensure the accuracy and consistency of results.

Calibration curve of nickel in steel



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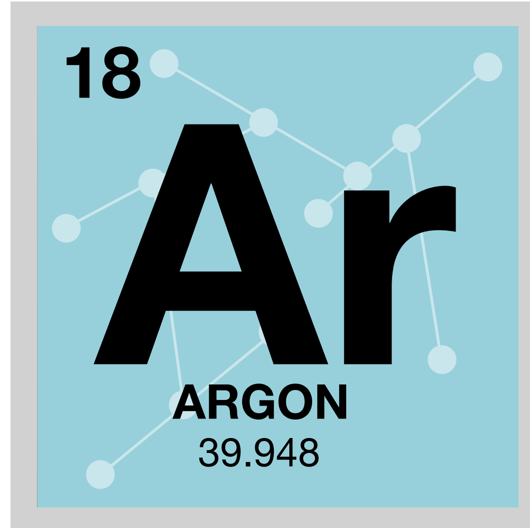
Argon

Argon is used to stabilize and promote plasma formation during LIBS analysis. Additionally it flushes the volume around the plasma to allow the detection of carbon that emits short wavelengths.



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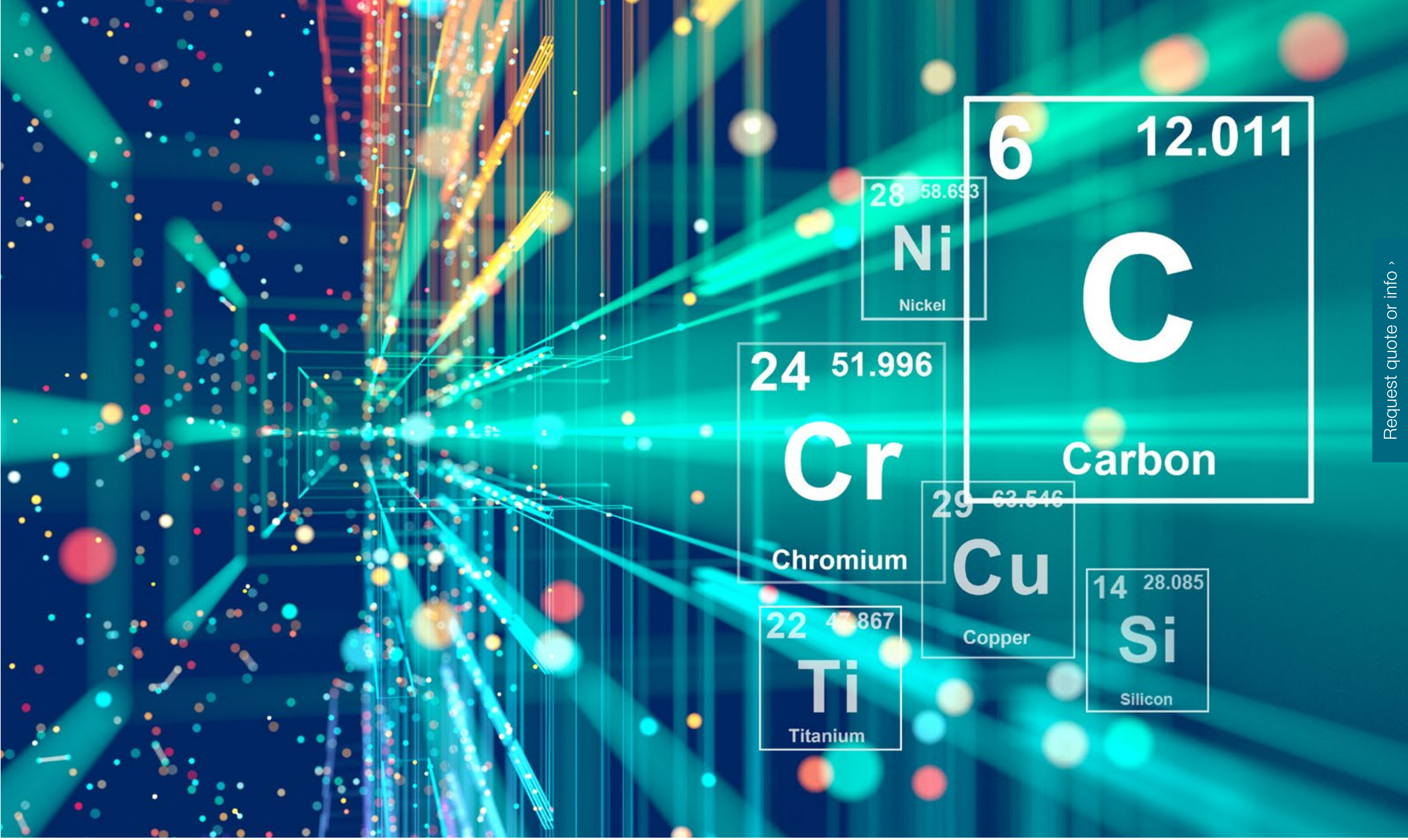

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The science behind LIBS

Plasma

Plasma is a partially ionized gas containing molecules, radicals, atoms, ions and free electrons resulting from the coupling of energy with matter in the gaseous state. Plasmas have very high temperatures in their cores ranging from several thousands to hundreds of millions of degrees celsius. In LIBS, the beam of a pulsed laser is focalized and absorbed by the surface of a sample causing the vaporization of the material. The laser beam further interacts with the vaporized matter producing a ball of ionized gas, a laser induced breakdown.

The laser induced breakdown is a plasma where the key processes of LIBS take place.

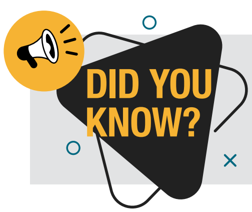
Atomization: at the beginning of the laser pulse, the vaporized material from solids is broken down into single elements (atoms).

Ionization: parts of the atoms lose an electron from their outer shell to form ions.

Excitation: atoms and ions get into states of high energy- so called “excited” states.

Emission: after the laser pulse terminates, atoms and ions come back to states of lower energies or (ground states) by emitting patterns of light (spectral lines) that are characteristic to each element.

The temperature of LIBS plasmas is typically 5,000 to 20,000 degrees celsius.



Beside solids, liquids and gases, plasmas form the fourth state of matter and are the most abundant matter in the universe. Stars like the sun are plasmas! Other common examples of plasmas are phenomena such as lightings or discharges in neon tubes.

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Anatomy of an atom

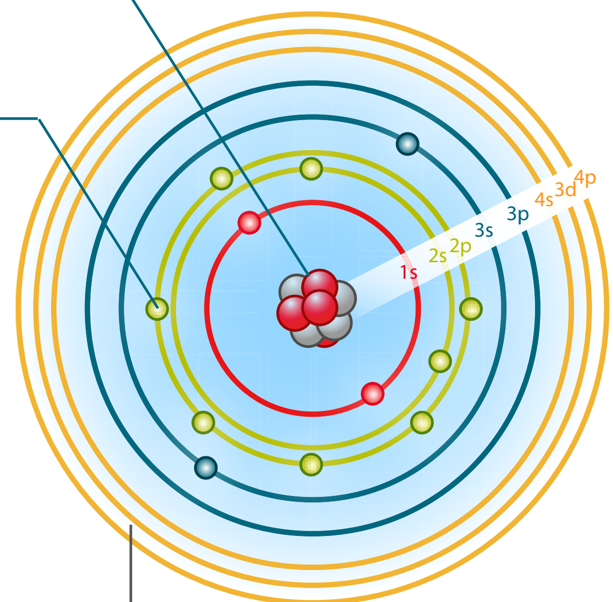
LIBS is a method of optical emission spectrometry. The key process takes place at an atomic level.

In the center of the atom is the nucleus, made up of **protons** and **neutrons**. Each proton carries a positive electrical charge, but neutrons carry no electrical charge. The nucleus of an atom is positively charged because of its protons.

Electrons are particles that orbit the nucleus at a high speed and carry a negative electrical charge. This balances the positive electrical charge of the protons inside the nucleus. Since the total negative charge of electrons is equal to the positive charge of the nucleus, an atom is neutral.

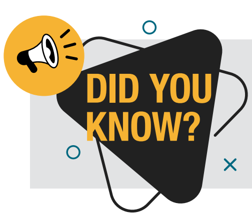
The negative electrons are attracted to the positive protons, so the electrons stay around the nucleus in discrete shells. These shells are labeled as 1s, 2s, 2p, 3s, 3p, 4s, 3d, 4p, etc. The LIBS process involves the electrons of the outermost shell of atoms or ions. Ions are atoms that have lost one electron.

Magnesium atom



Ground State

Atoms are not really combined of concentric circles of electrons. We just draw them that way to illustrate how electrons orbits around the nucleus.



The word atom comes from the ancient Greek word atomo, meaning “that can’t be cut”.

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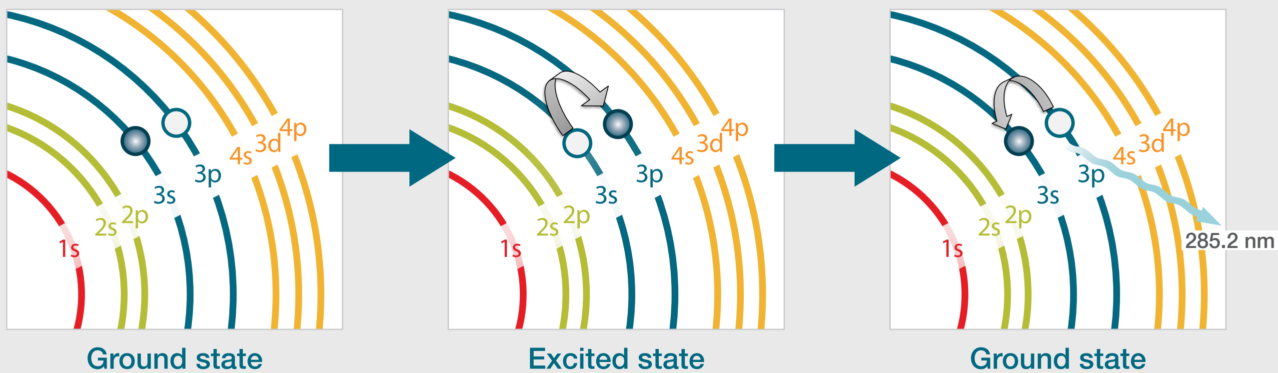
What happens at the atomic level?

1. In a highly energetic source such as a plasma, electrons of the outer shells of atoms in the ground state move to shells of a higher energy.
2. The atom is then in a so-called excited state.
3. The electron moves from the shell of higher energy to a shell of lower energy.
4. The atom then returns to a ground state by emitting discrete spectral lines corresponding to the energy difference of the evolved states. This difference in energy is unique to each element.

The atom of magnesium emits a very sensitive line at 285.2nm. This corresponds to a difference of energy between shells 3p and 3s.



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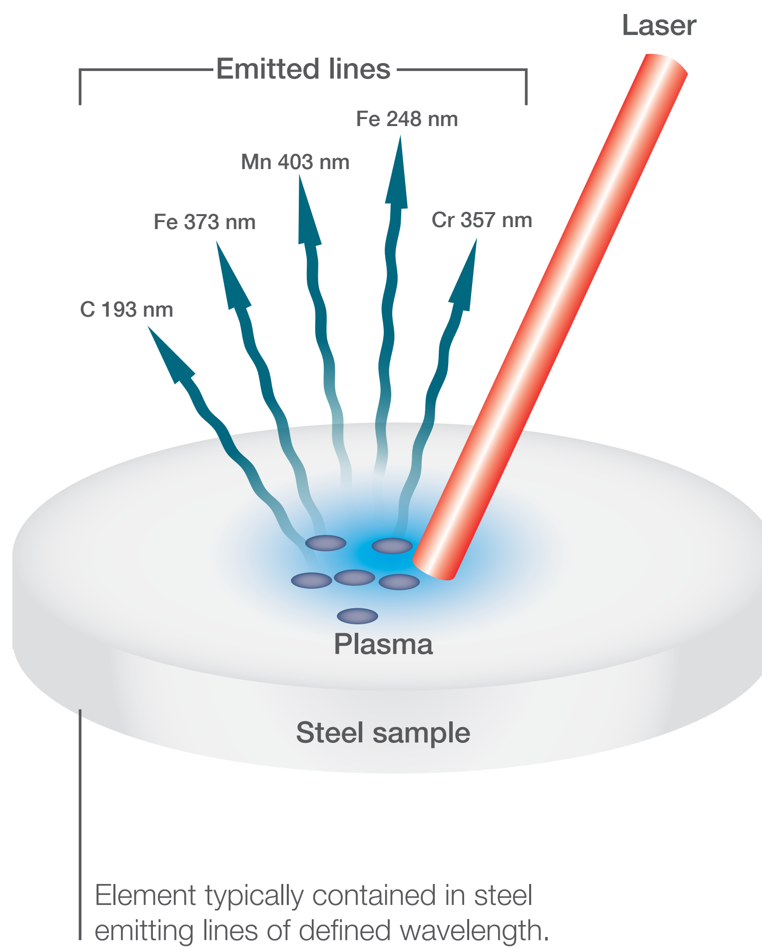
LIBS spectrum

Similar to a fingerprint, every element of the periodic table emits a set of characteristic lines in the plasma. The overlay of the lines emitted by elements contained in the sample form the spectrum.

The spectrum is comprised of two components:

- **Wavelength** - provides information regarding the elements present in the sample **(qualitative analysis)**.
- **Count rate** - also known as intensity, provides information about the quantity of those elements **(quantitative analysis)**.

The measurement of intensity at the wavelengths of the emitted lines is the basis of LIBS analysis.



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Equipment

10 features to look for when purchasing a handheld LIBS analyzer

1. **Analytical Range.** Ensure that the analyzer can measure key elements, such as carbon (C).
2. **Repeatability**. Variances in measurements will result in irregularities of finished goods. By verifying repeatability, you can guarantee the quality of your processes.
3. **Lightweight.** Fatigue is likely to set in sooner with instruments weighing over 7 pounds.
4. **Hot Swap Battery.** Reduce downtime with a hot swap battery when power is depleted.
5. **Cameras.** A micro camera can precisely pinpoint your exact measurement, while a macro camera can collect sample imagery.
6. **Miniaturized Geometry.** Evaluate whether you'll be able to access tight welds, corners, and joints.
7. **Safety Interlocks.** Check for multiple robust laser safety interlocks to ensure that users are protected during operation.
8. **WiFi Connectivity.** Off-site workers may need access to key analytical information. Determine if you'll be able to obtain data with WiFi connectivity.
9. **Easy Navigation.** A tilting, color touchscreen and directional keys make it easy for users to navigate through menus and sample readings. Ensure that touchscreens can also be operated with gloves on.
10. **Splash/ Dustproof.** Look for a minimum rating of IP54 to ensure splash and dustproof claims are validated.

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Handheld LIBS analyzer

Thermo Scientific™ Niton™ Apollo™ Handheld LIBS Analyzer

Confidently perform elemental analysis with the Niton Apollo handheld LIBS analyzer. When carbon detection and mobility are top of mind, industrial businesses rely on the Niton Apollo for superior performance and enhanced productivity. Identify low alloy/ carbon steels and L and H grade stainless steels. The Niton Apollo handheld LIBS analyzer transforms a traditional cart-mounted Optical Emission Spectroscopy (OES) system into a highly portable, easy-to-use handheld analyzer.

Product details ›

To learn more, browse **Niton Apollo FAQs**

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For more information, download the specification sheet: **Laser Focused Carbon Analysis**

Handheld XRF analyzers

For versatile applications requiring additional elemental coverage, from Mg-U.

Thermo Scientific™ Niton™ XL5 Handheld XRF Analyzer

The Niton XL5 handheld XRF analyzer is the lightest, smallest, most powerful portable XRF analyzer available for elemental determination.

Product details ›

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The Niton XL2 Plus handheld XRF analyzer provides operators a robust, versatile analyzer ready for the toughest industrial environments.

Product details ›

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Learn more about **Using XRF + LIBS Together: When and How It Makes Sense** thermoscientific

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