LIBS Instrumentations

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LIBS Instrumentations

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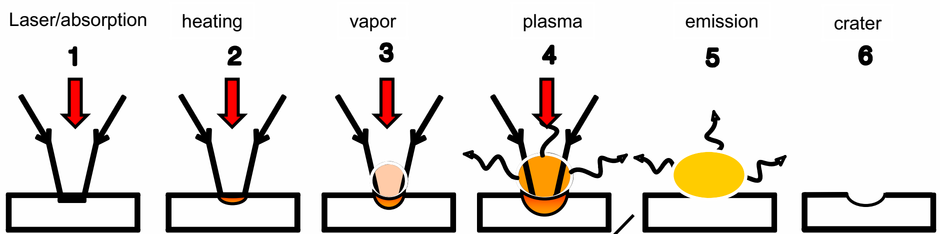
National Research Council Canada Conseil national de recherches Canada Canada

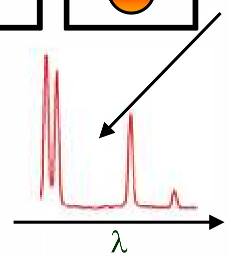
Laser-Induced Breakdown Spectroscopy (LIBS) Instrumentations

- Overview on the LIBS technique development
- Plasma characterization by atomic emission spectroscopy
  - Temperature and electron density
  - Spectrochemistry
- LIBS components: Laser, Detector, Spectrometer
  - Laser
    - Influence of the laser parameters on the LIBS analytical performance
    - Selection of a laser for LIBS application
  - Desirable requirements for atomic emission spectroscopy
  - Spectrometer
    - Conventional spectrometer
    - Paschen-Runge spectrometer
    - Echelle spectrometer
  - Detector
    - Solid-state detectors (ICCD, Interline CCD, IPDA, etc.)
    - Photomultiplier
  - Comparison of detection systems
- LIBS tools
  - Enhancement of LIBS sensitivity
  - Enhancement of LIBS reproducibility
  - Diagnostics of failure of the LIBS system
- Comparison with conventional techniques
- *Conclusions*

Ablation Mecanisms

⇨ **Nanoseconde regime**





LIBS

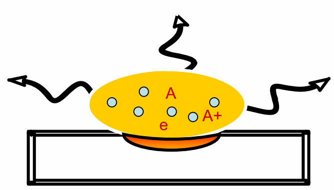
Laser ablation

5

Emission
Or
Ablated mass

⇨ Chemical composition by optical emission

⇨ Chemical composition by ablated mass coupled to another source of excitation



Laser ablation coupled to inductively coupled plasma (ICP) or Mass spectrometer

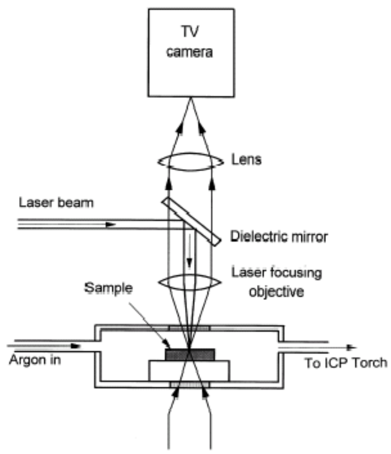
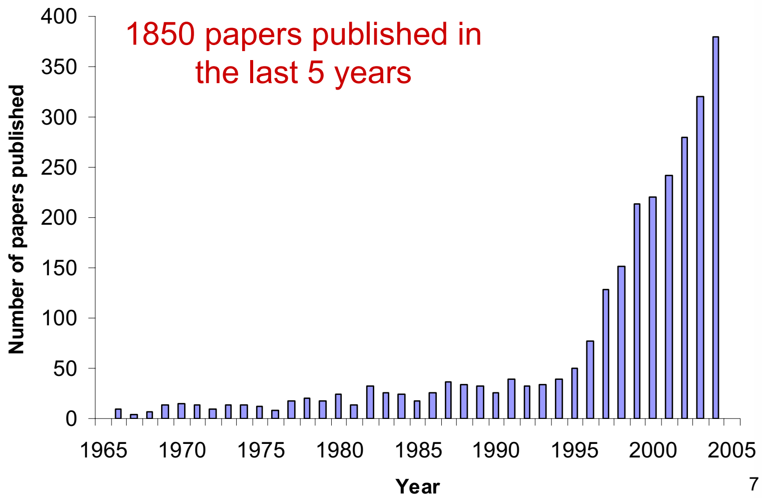


Fig. 1. Schematic of a laser ablation system.

Laser Induced Breakdown Spectroscopy: *Acronyms*

- LIPS: Laser Induced Plasma Spectroscopy
- LIBS: Laser Induced Breakdown Spectroscopy
- LIESA: Laser Induced Emission Spectral Analysis
- LA-OES:Laser Ablation Optical Emission Spectroscopy
- LM-OES:Laser Microanalysis-OES
- LM-OES:Laser Microprobe-OES
- LMSA:Laser Micro-Spectral Analysis
- LSS: Laser Spark Spectroscopy
- LISWPS: Laser-Induced Shock Wave Plasma Spectroscopy
- LPS: Laser Plasma Spectroscopy

LIBS papers evolution



Distribution of LIBS papers in the literature

1143 papers published in the period 1997-2002

Name of the periodical journal	Number of papers	Percentage
Spectrochimica Acta B	110	9.6%
Applied Spectroscopy	109	9.5%
Journal of Analytical Atomic Spectrometry	63	5.5%
SPIE Proceeding	62	5.4%
Applied Surface Science	53	4.6%
Analytical Chemistry	25	2.2%
Fresenius' Journal of Analytical Chemistry	24	2.1%
Applied Physics A	21	1.8%
Total	**467**	**40.8%**

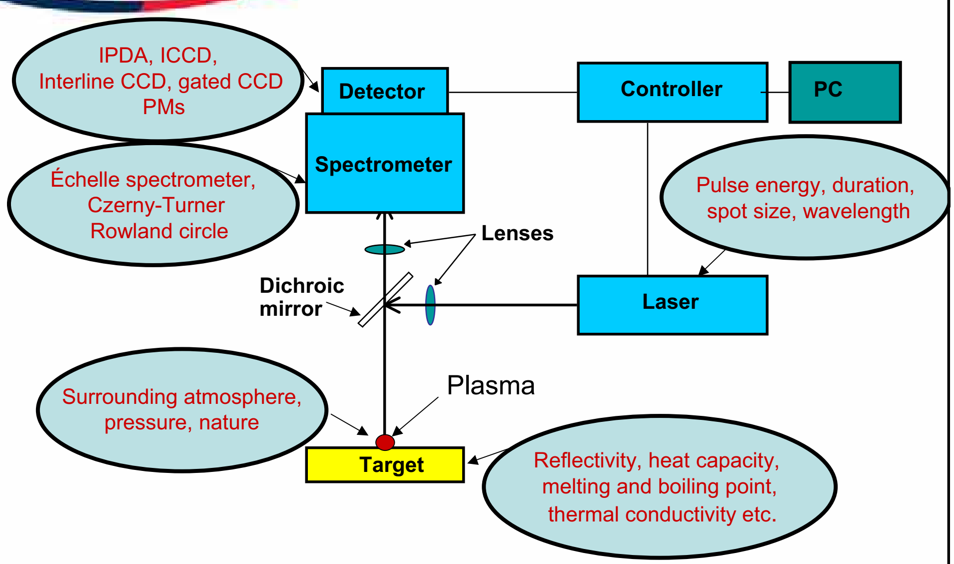
Development of the LIBS technique

1962 to 1980: First experiments
- Inadequate instrumentation (lasers, detectors)
- Quantitative measurement difficult

1980 to 1990: Evolution in laboratory
- Lasers and detectors become more reliable
- Better analytical performances
- Quantitative analysis demonstrated

1990 to date: Applications emerge
- Industrial lasers, intensified detectors and echelle spectrometers enter commercial market
- Growth in research activity

LIBS components



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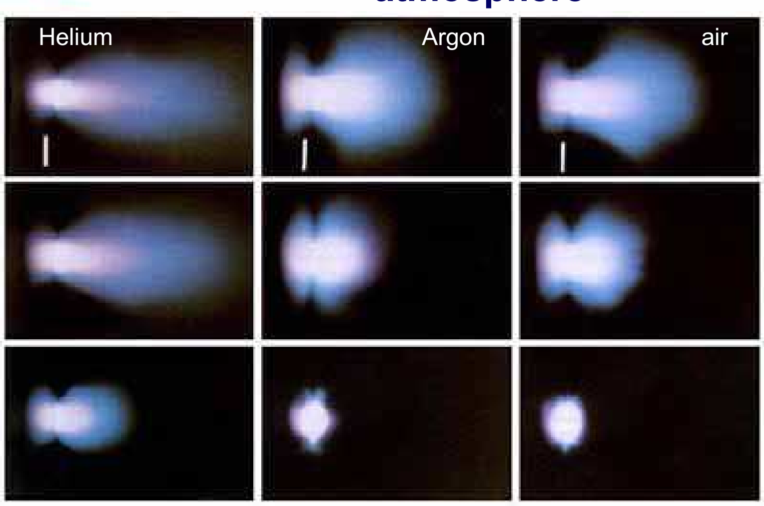
Influence of the surrounding atmosphere



10 torr

100 torr

760 torr

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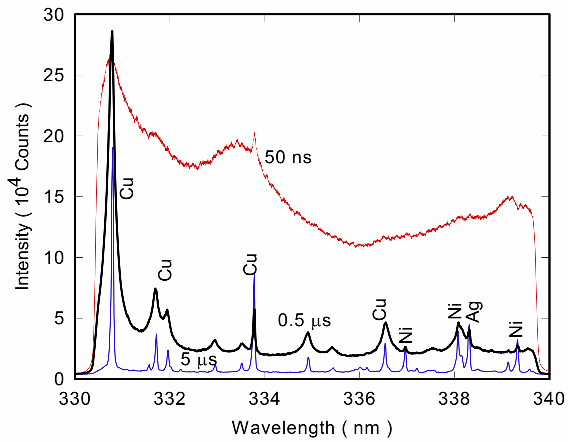
Time-resolved LIBS spectra on copper alloy sample: Spectroscopic aspects



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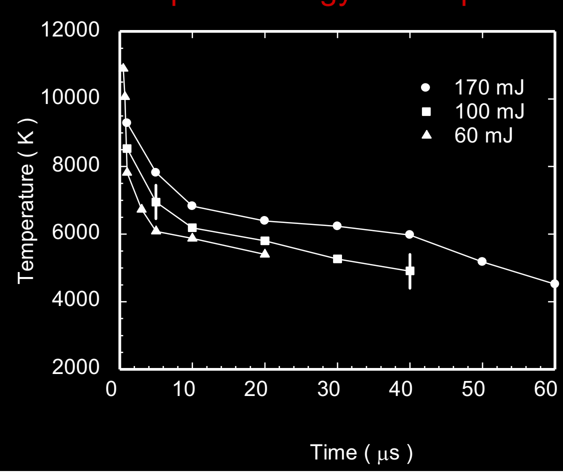
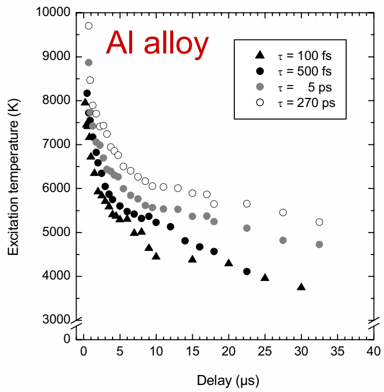
Determination of Plasma Temperature

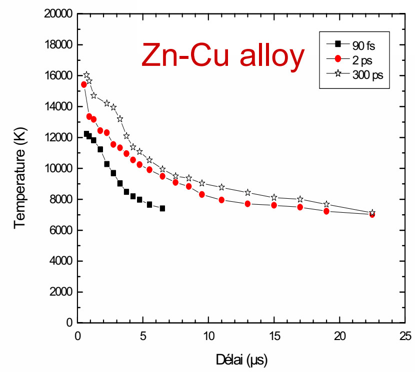
Influence of laser pulse energy on the plasma temperature



Plasma Characterization/ Temperature

Influence of laser pulse duration on the plasma temperature

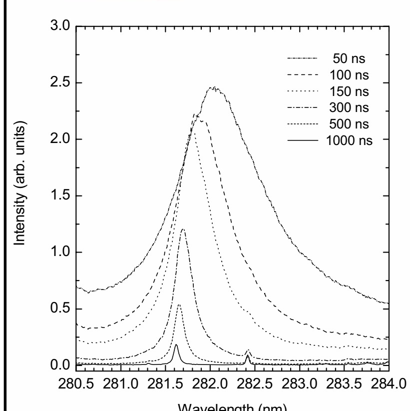


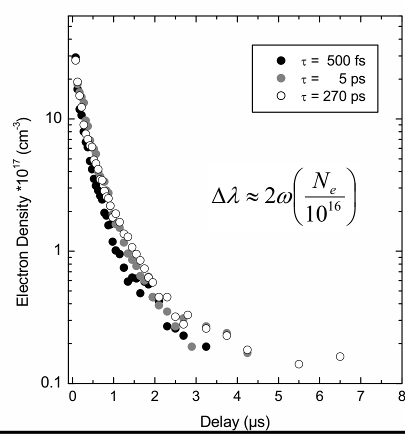


B. Le Drogoff, J. Margot, F. Vidal, S. Laville, M. Chaker, M. Sabsabi, T. W. Johnston, and O. Barthélemy, *Influence of the pulse duration on laser-produced plasma properties*, Plasma Sources Science and Technology **13** (2), 223-230 (2004).

Plasma Characterization/ Electron Density

Influence of laser pulse duration on the electron density





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LIBS technique

- Line intensity
- Ratio of two lines

$$I_{mn} = \frac{A_{mn} N g_m}{U(T)} \cdot \frac{hc}{\lambda_{mn}} \exp\left(-\frac{E_m}{kT}\right)$$

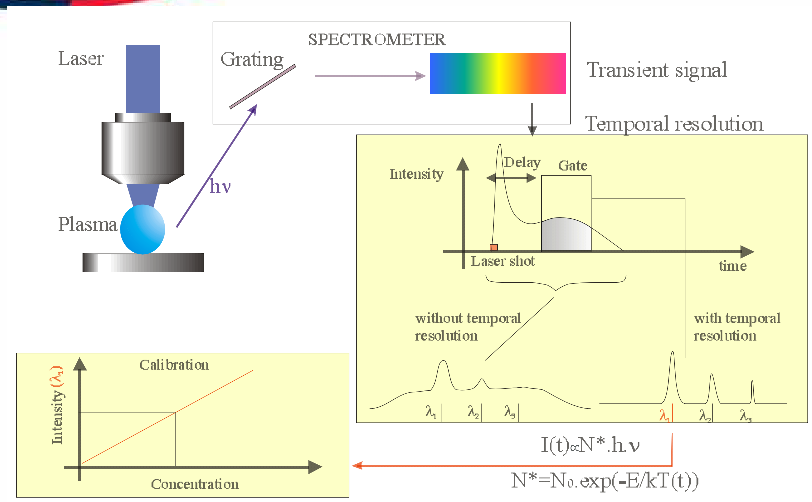
$$\frac{\varepsilon_t}{\varepsilon_m} = \alpha \cdot \frac{N_t}{N_m} \cdot f(T)$$

with

$$f(T) = \exp\left(-\frac{\Delta E}{KT}\right)$$

$$\alpha = \frac{g_1 A_1 U_2 \lambda_2}{g_2 A_2 U_1 \lambda_1}$$

Fundamentals of LIBS



- The combination of spectrometer and detector is an important factor to consider in optical emission spectroscopy for any plasma characterization or analytical spectrochemistry.
- The experimentalist must choose the type of measurements to be made, the concentration range to be monitored.
- Spectroscopist's need :
  - Reproducibility
  - Sensitivity

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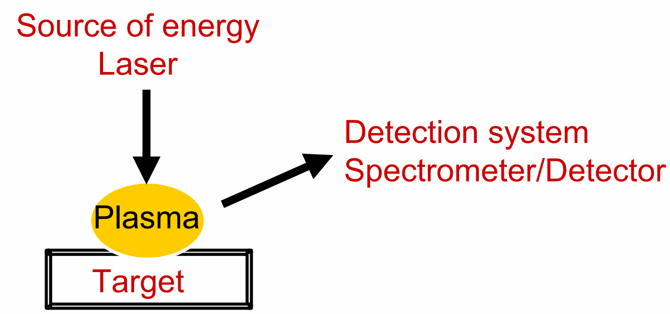
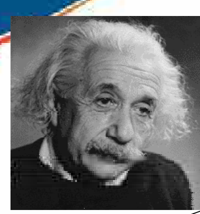
LIBS components



Laser development



1917 Einstein First theorized about stimulated emission, the process which makes lasers possible

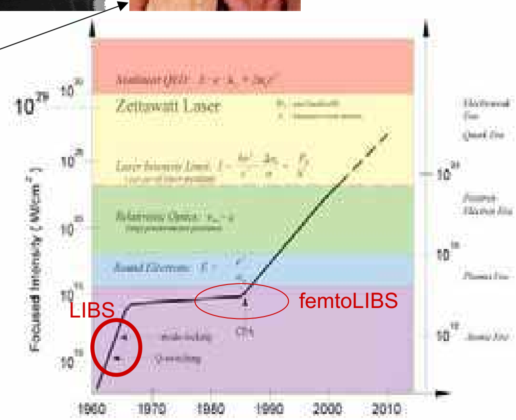
1958 Shawlow, Tawns Theorized about a visible laser

1960 Maiman Built first ruby laser with optical pumping

1962 Brech, Cross Birth of LIBS: detection of spectrum from ruby laser induced plasma

1964 Runger et al. First direct spectrochemical analysis by LIBS

1985 Mourou Chirped Pulse Amplification (CPA)



Selecting a Laser for LIBS applications

- Pulse energy

  Focusing conditions, spot size, laser energy density, stability.
- Wavelength

  Absorption, reflectivity, thermal conduction, laser-induced plasma shielding, ablated mass.
- Pulse duration

  Ablation regime, ablated mass, plasma spectrochemistry, thermal conduction, laser-induced plasma shielding etc.
- Repetition rate and beam quality

  Reproducibility, rapidity of analysis
- Divergence

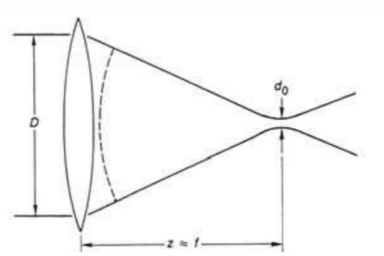
  Focusing conditions, spot size

Laser parameters

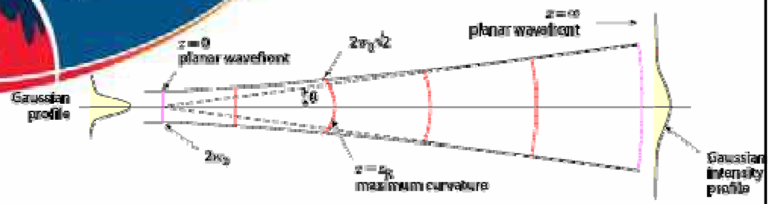
Beam focusing **:**
A laser beam can be focused into a small spot using a positive lens, thus increasing the power/energy density in the lens' focal plane.

Assuming a laser beam with diameter D, the minimum (diffraction limited) spot diameter d_0 is given by:

$$d_0 \approx \frac{1.27 f\lambda}{D}$$



where f is the lens' focal length.



Actual laser beams are never perfectly Gaussian. To address the issue of non-Gaussian beams, a beam quality factor, M^2, has come into general use.

For a theoretical Gaussian beam, the value of the radius-divergence product is: $w_0\theta = \lambda/\pi$

For a real laser beam, we have: $w_{0M}\theta_M = M^2\lambda/\pi > 1$

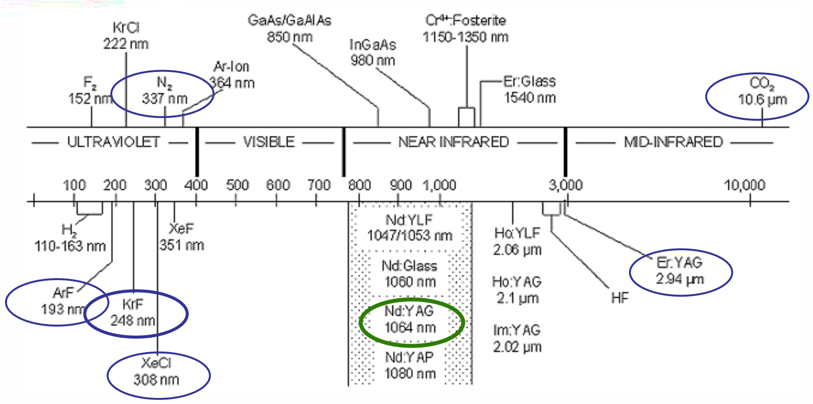
where w_{0M} and θ_M are the $1/e^2$ intensity waist radius and the far-field half-divergence angle of the real laser beam, respectively. The beam quality factor M^2 can therefore be defined by:

$$M^2 = \frac{w_{0M}\theta_M\pi}{\lambda}$$

Typical values of M^2 for laser systems:

- HeNe: $1.0 < M^2 < 1.1$
- Ion lasers: $1.1 < M^2 < 1.3$
- Collimated TEM_{00} diode lasers: $1.1 < M^2 < 1.7$
- High energy multimode lasers: M^2 up to 3 - 4

LIBS components:
Laser

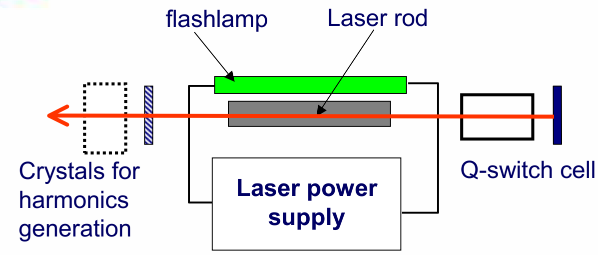


Energy/pulse: 10 µJ-500mJ
Nd:YAG and its harmonics 2w, 3w, 4w are used the most in LIBS applications.

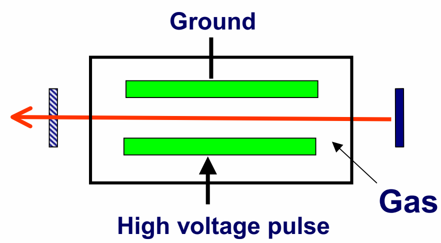
Cost: 3-250k$

Lasers used for LIBS

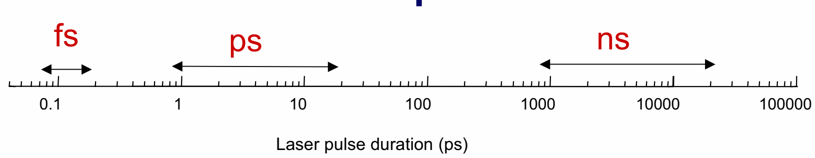
Q-switched Nd-YAG



Excimer laser
TEA CO2 laser



Literature on LIBS with ultra-short laser pulses



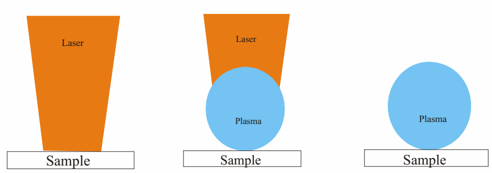
- In the last 5 years we counted more than 1500 papers on LIBS.Most of them are dealing with ns laser pulses.
- Only 50 papers were devoted to LIBS with ultra-short laser pulses.

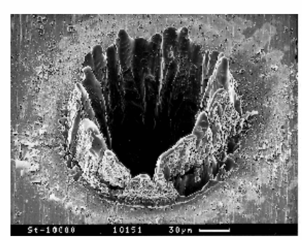
General conclusions from literature comparing fs to ns

- For femtosecond regime in the 10^{13}-10^{14} W/cm^2, the pulse duration is shorter than the plasma expansion, the heat conduction time and the electron to ion energy transfer time.
- No thermal effect (without collateral damage to the rest of the target)
- No plasma shielding
- No thermal stress
- Cutting and drilling holes with high precision
- Improved spatial resolution
- Fs laser is 10 times higher in price than ns laser

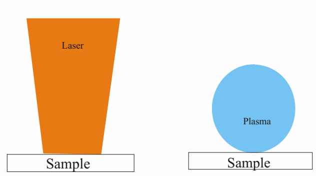
Why doing LIBS analysis by ultra-short pulses?

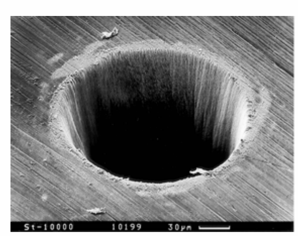
- nanosecond interaction 10^{-9}s





- femtosecond interaction 10^{-15}s

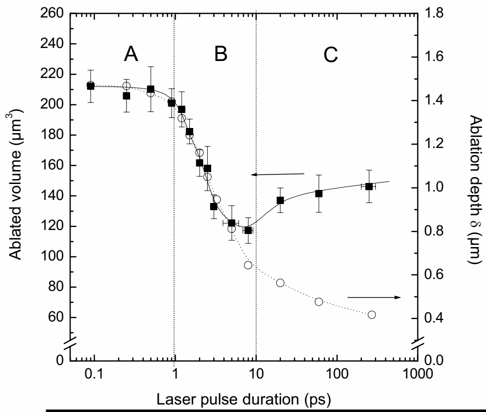


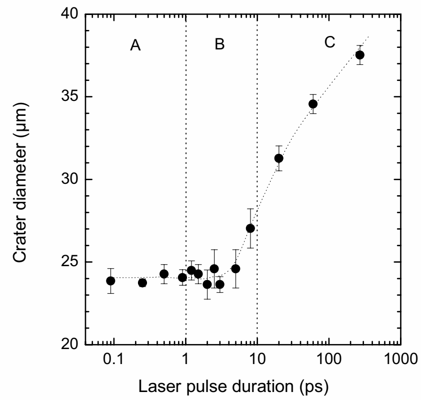


B. N. Chichkov, C. Momma, S. Nolte, F. von Alvensleben and A. Tunnermann, *Appl. Phys.*, 1996, **63**, 109–115.

Plasma Characterization/ Ablation Study

Influence of laser pulse duration on the crater diameter

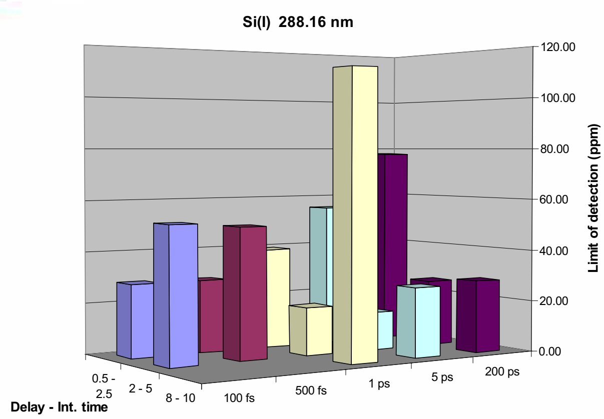




B. Le Drogoff, M. Chaker, T. W. Johnston, S. Laville, F. Vidal, O. Barthélemy, J. Margot and M. Sabsabi, *Laser ablated volume and depth as a function of the pulse duration in aluminum targets*, Applied Optics 44, (2005), pp. 278-281.

Spectrochemistry

Analytical figure of merit: sensitivity



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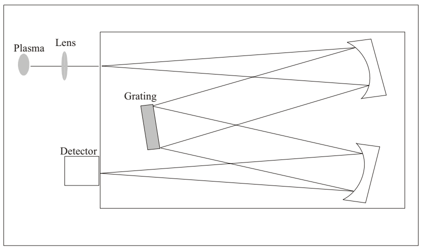
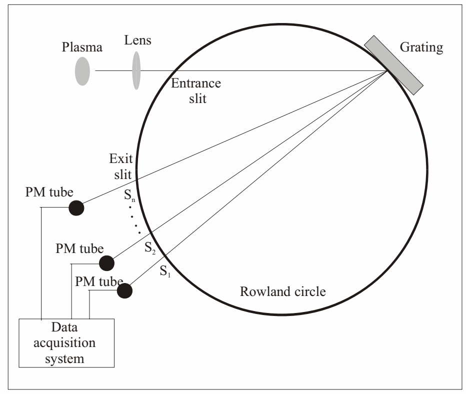
Desirable requirements for atomic emission detectors

The requirements for the ideal spectrometer and detector to provide for simultaneous determination of any combination of elements in the spectrum include :

- A high resolution of 0.01-0.003nm to resolve the lines of interest and avoid interferences.
- Wide wavelength coverage is needed, typically from 165 nm to 800-950 nm to be able to detect simultaneously several elements.
- A large dynamic range is necessary to provide the optimum signal-to-noise ratio (SNR) for a large range of elemental intensities; the detector has to have a wide dynamic range, typically 6 to 7 orders of magnitude.
- The spectrometer/detector combination should have a high sensitivity and a linear response to radiation.
- The detector has to have high quantum efficiency particularly in the near infrared and UV and low noise characteristics.
- For rapid analysis the read out and data acquisition time should be shorter at least less than the time lap between the laser pulses.

Paschen-Runge and Czerny-Turner spectrometer systems





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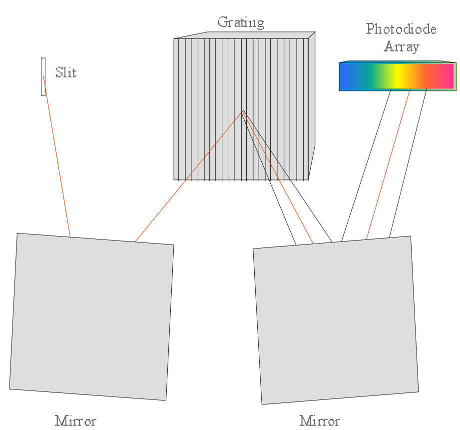
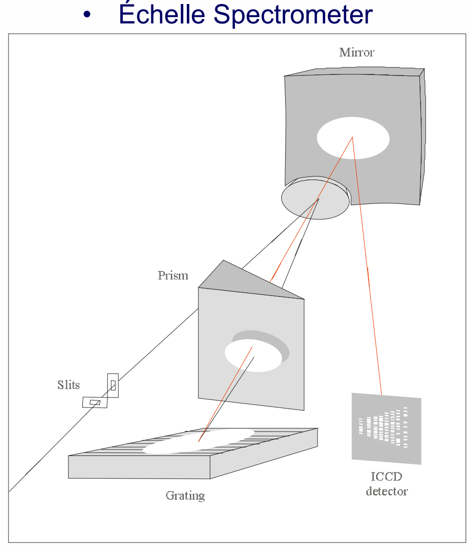
Spectrometer set-up

- Czerny Turner Spectrometer



- Échelle Spectrometer



Comparison of spectrometers

$\sin \alpha \pm \sin \beta = kn\lambda$

Where α and β are respectively the incidence and diffracted angle, n is the grating density (number of grooves/mm), λ is the wavelength and k is diffraction order. For example, if we take $\sin\alpha+\sin\beta=1$ and λ=333 nm, the product kn will be 3000: for k=1, n=3000 grooves/mm, while for k=50, n=60 grooves/mm.

Comparison of a conventional grating and an echelle grating in terms of spectral features

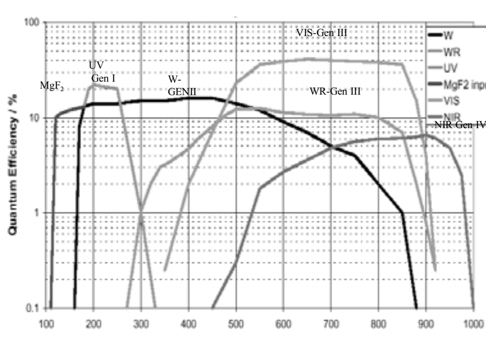
Feature	Conventional grating	Echelle grating
Focal length (m)	0.5	0.5
Grating density (grooves/mm)	1200	79
Diffraction angle	10°22′	63° 26′
Width (mm)	52	128
Spectral order at 300nm	1	75

Detectors

- Intensified CCD or Intensified PDA

  Poor quantum efficiency (below 20%), limited wavelength coverage, high gain, 16 bits dynamic range, simultaneous measurements, high cost.

Cost: 25-50 k$



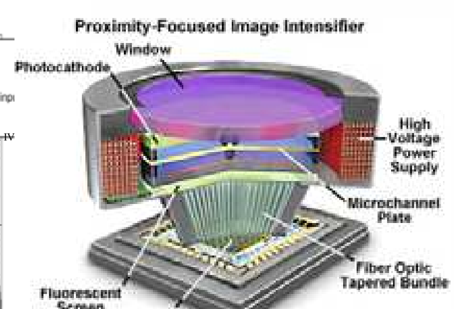
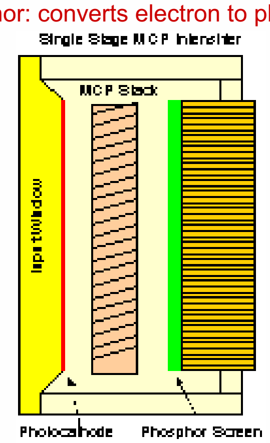
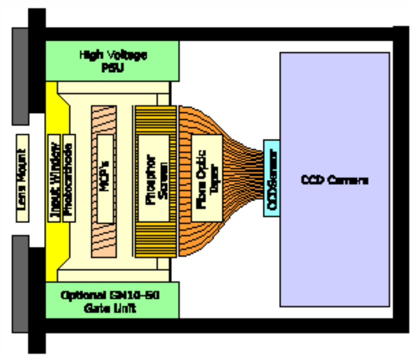


Figure 1

Basics of ICCD

1-Photocathode: converts photons to electrons
2-Microchannel Plate (MCP): amplifies the photoelectrons
3-Phosphor: converts electron to photons





CCD development

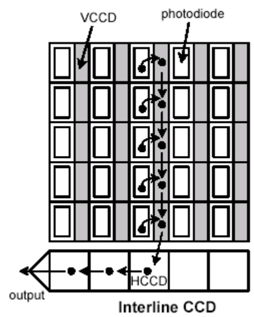
- 1970 CCD invented by Smith and Boyle, Bell NJ
- 1973 introduced by Fairfield
- 1979 Scientific applications by RCA.
- 1983 Use in spectroscopy
- 1990 Integrated in ICP instrumentations by several companies.
- Intensifier coupled to photodiode array in the mid 80's and coupled to CCD in the beginning of 90's.

Detectors

- Interline CCD, Gated CCD

Larger wavelength coverage, high quantum efficiency, 12-14 bits dynamic range, less expensive.

Cost: 1-20 k$



Characteristics	Typical Range	Limitations
Full-well capacity	10000-500000 electrons	Defines dynamic range
Pixel dimensions	6-30μm	Dictate spectral or spatial resolution
Array format	Related to pixel size and number	Dictates the active area
Number of pixels	58X512 to 2048X2048	Dictates number of resolution elements and acquisition time
Quantum efficiency	0-80%	Defines ultimate sensitivity limit
Blooming	Present with materials having strong lines in the range 200-900nm	Cannot observe weak lines near strong lines, ghost lines
Read noise	Few electrons	Excess noise limits weak light detection

Detectors

- Photomultiplier PM

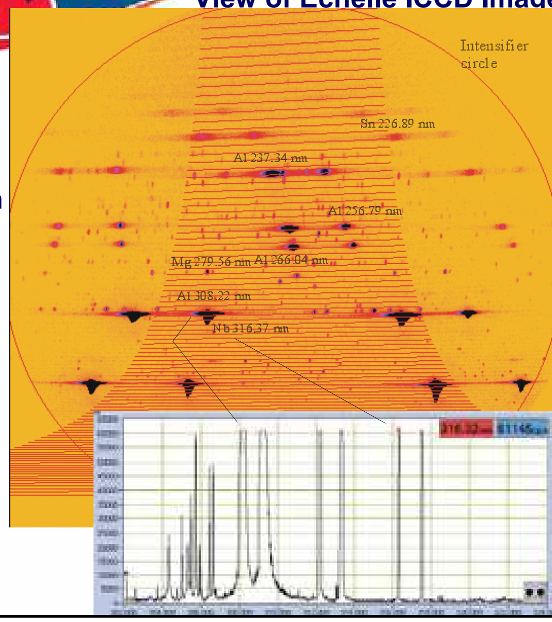
Monochromatic, low quantum efficiency, bulky, larger wavelength coverage, high dynamic range, cheap.

Cost: 1-3 k$

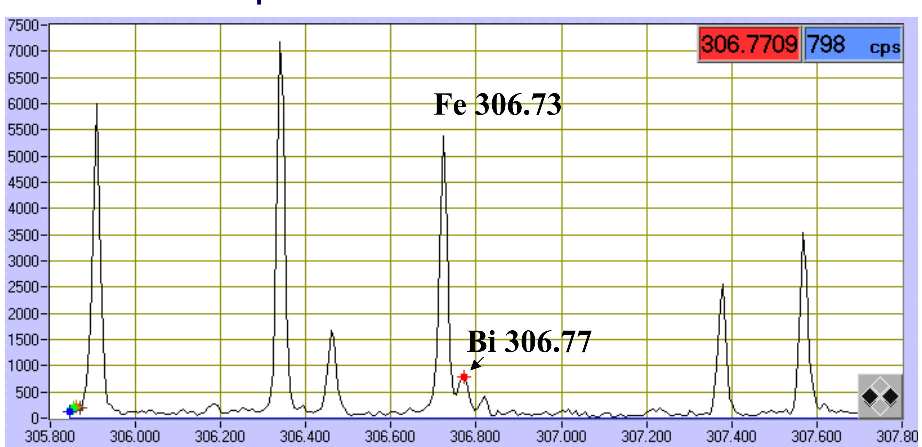
- Thorne, A.P. In *Spectrophysics,* Chapman and Hall, New York, 1988.
- Demtroder, W. In *Laser Spectroscopy : Basic Concepts and Instrumentation*, Second edition, Springer-Verlag, Berlin, 1981.
- Busch, K.W.; Busch, A.M. *Multielement Detection Systems for Spectrochemical Analysis*, Wiley, New York, 1990
- Mika, J.; Torok, T. *Analytical Emision Spectroscopy: Fundamentals*. A.P. Floyd, Trans. Crane, Russak & Company, New York, 1974, pp. 473-496
- Sweedler, J.V.; Ratzlaff, K.; Denton, M. B. In *Charge Transfer Device in Spectroscopy*, VCH pub., New York, 1994

View of Échelle ICCD Image

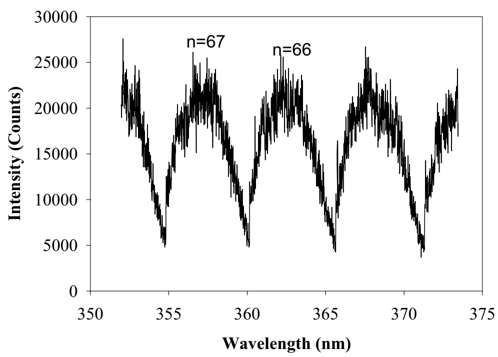
- Aluminum spectra



Échelle spectrometer LIBS spectrum from copper ore sample



Typical evolution of echelle spectrometer response in a given diffraction order



- It needs calibration for absolute measurements

LIBS components: échelle spectrometer/detector

Echelle spectrometer ESA3000, LLA
http://www.lla.de/echelle.html

Echelle spectrometer SE200
Catalina with Roper, http://www.libs.info/

Andor spectrometer 7500
http://www.andor-tech.com/

LTB echelle spectrometer
http://www.ltb-berlin.de/

Cost:35-80k$

NRC·CNRC Industrial Materials Institute

LIBS equipment

Ocean Optics and Spectrolaser use gated CCD

Pharmalaser uses an interline CCD
http://www.pharmalaser.com/

ARL laser spark;
Pachen-Runge spectrometer
with PM; www.arl.ch

Fraunhofer Institut Lasertechnik

Pachen Runge LS-5 with MCI of up to 64 PM, 1000Hz

Marwan Technology uses
echelle spectrometer and dual pulse laser

NRC·CNRC Industrial Materials Institute

Detectors

- Intensified CCD or Intensified PDA

  Poor quantum efficiency (below 20%), limited wavelength coverage, high gain, 16 bits dynamic range, simultaneous measurements, high cost.

- Interline CCD, Gated CCD

  Larger wavelength coverage, high quantum efficiency, 13 bits dynamic range, less expensive.

- Photomultiplier PM

  Monochromatic, low quantum efficiency, bulky, larger wavelength coverage, high dynamic range, cheap.

- New EMCCD and EBCCD in the market but not presently appropriate for LIBS applications.

Cost: 1-35 k$

Advantages

- Very high resolution equivalent to 2m Czerny-Turner spectrometer with 2400 grooves/mm
- Enhanced spectral range
- Powerful tool for identification of complex matrix
- Compactness
- Choice of several lines for the same element
  - Increase the dynamic range
  - Flexibility to choose a line for analysis
  - Rapidity of measurements, normalization
  - Complete analysis by single shot

Limitations

- Correction of the instrument response within an order of diffraction and between orders for absolute measurements
- Blooming
  - Ghost line apparition
  - Difficulties in measuring weak line close to strong line
- Dynamic range
- Small gap for the low orders (in the visible)
- Acquisition rate, 1Hz (ICCD limitation)
- High cost

Portable LIBS System

Recent interest in LIBS has focused on the miniaturisation of the components and the development of compact, low power, portable systems. This direction has been pushed along by interest from groups such as NASA, ESA as well as the military.

Portable LIBS systems are more sensitive, faster and can detect a wider range of elements (particularly the light elements) than competing techniques such as portable x-ray fluorescence

- LIBS is portable; it may weigh less than 2 kg.
- Battery powered
- Multi-element analysis for any element

Weight 570 g

Advantages

- It can be applied on:
  - solid, liquid or gaseous
- No sample preparation
- Small mass required (sub-µg)
- Ambient air at atmospheric pressure
- Remote and real time analysis
- Single step for vaporization and excitation
- High spatial resolution (2 µm)
- Simultaneous multi-element analysis
- Portable system

Drawbacks

- Small volume of analysis
- Destructive technique
- Surface analysis could be different from bulk
- Sensitivity is in the ppm range

Comparison of some methods based on plasma spectrochemistry

Attribute	LIPS	ICP	LA-MS	LA-ICP-MS	ICP-MS
Detection limits	Lowppm/ high ppb	Mid to low ppb	10^{-20}g	ppb	ppb to ppt
Preparation	Little or none	must be liquid	Little or none	Little or none	must be liquid
Atmospheric conditions	Air, He, Ar vacuum	Air, Ar,	High vacuum	High vacuum	High vacuum
Cost	50k$+	50k$-200k$	120k$-500k$	170k$-300k$+	120k$-300k$
Commercially available	Yes	Yes	Yes	Yes	Yes

Laser-Induced Breakdown Spectroscopy (LIBS) Instrumentations

- Overview on the LIBS technique development
- Plasma characterization by atomic emission spectroscopy
  - Temperature and electron density
  - Spectrochemistry
- LIBS components: Laser, Detector, Spectrometer
  - Laser
    - Influence of the laser parameters on the LIBS analytical performance
    - Selection of a laser for LIBS application
  - Desirable requirements for atomic emission spectroscopy
  - Spectrometer
    - Conventional spectrometer
    - Paschen-Runge spectrometer
    - Echelle spectrometer
  - Detector
    - Solid-state detectors (ICCD, Interline CCD, IPDA, etc.)
    - Photomultiplier
  - Comparison of detection systems
- LIBS tools
  - Enhancement of LIBS sensitivity
  - Enhancement of LIBS reproducibility
  - Diagnostics of failure of the LIBS system
- Comparison with conventional techniques
- *Conclusions*

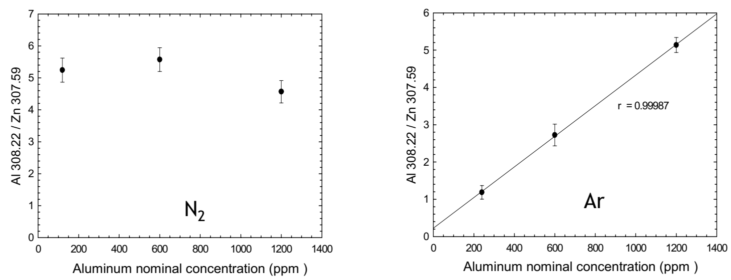
Influence of the various components of the LIBS system on the analytical performances

	laser	optics (spectro)	detection detector	atmosphere
precision	x		x	x
selectivity		x	x	x
limits of detection	x	x	x	x
stability	x	x		

Procedure to evaluate the LIBS analytical performance

- *Ionic to atomic line intensity ratio measurements*
- *Sampling approach and optimization*
- *Practical resolution measurements*
- *Scale of limit of detection*
- *Drift diagnostics*
- *Figures of merit*

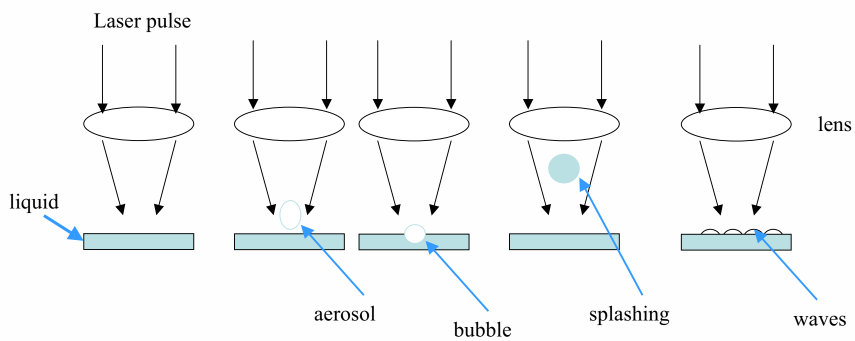
Analysis of aluminum and iron in molten zinc for the galvanizing process



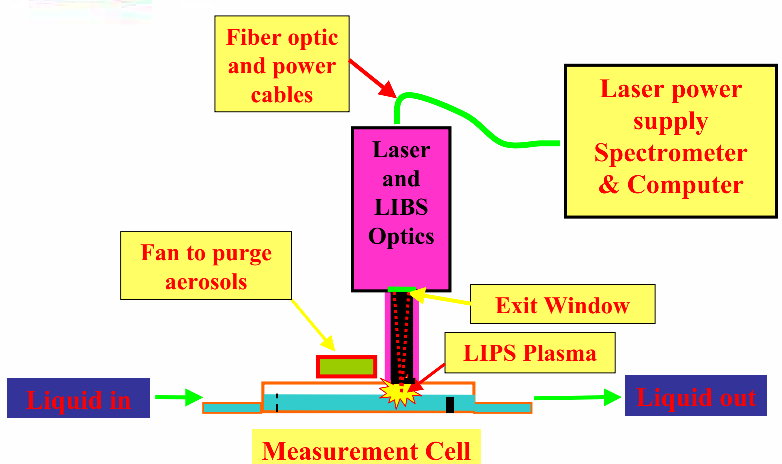
Problems and difficulties

- *Problems related to the laser-liquid interaction*
  - Splashing of the liquid caused by powerful laser pulses
  - Generation of waves perturbing the surface
  - Aerosols and particles ejected
- *Difficulties related to the molten metal*
  - Surface is not representative of the bulk
  - Diffusion of some elements to the surface
  - The sensor should be sufficiently rugged for plant use

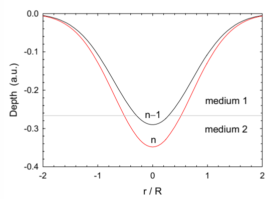
Laser-liquid interaction

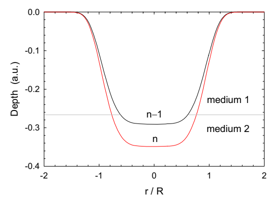


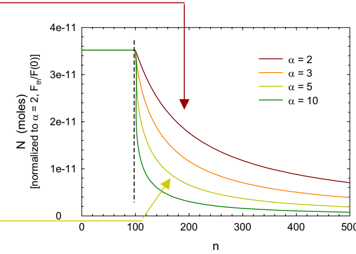
LIBS for Continuous Analysis of Liquids



Profilometry
Spatial distribution of the laser beam







$\alpha = 2$ $\alpha = 5$

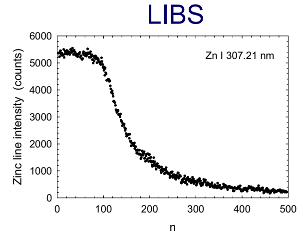
$$F(r) = \frac{1}{\Gamma(1+2/\alpha)}\frac{E}{\pi R^2}\exp\left[-\left(\frac{r}{R}\right)^{\alpha}\right]$$

(R is the radius of the beam at 1/e)

$$D(r) = Y\ F(r)$$

Ablation threshold (cm^3/J)
empiric parameter

LIBS



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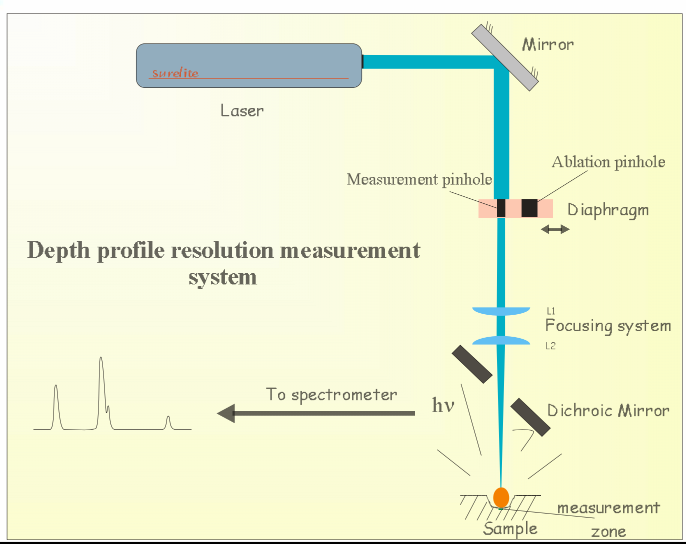
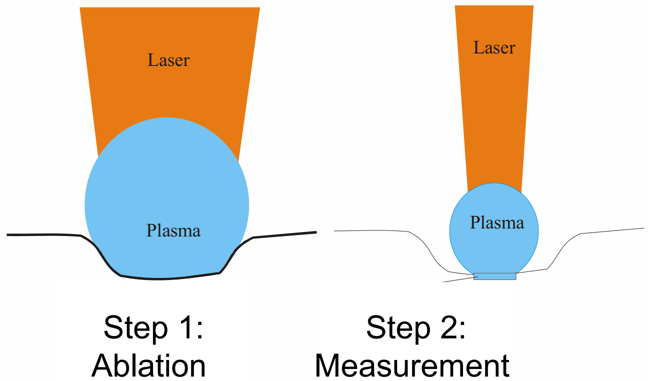
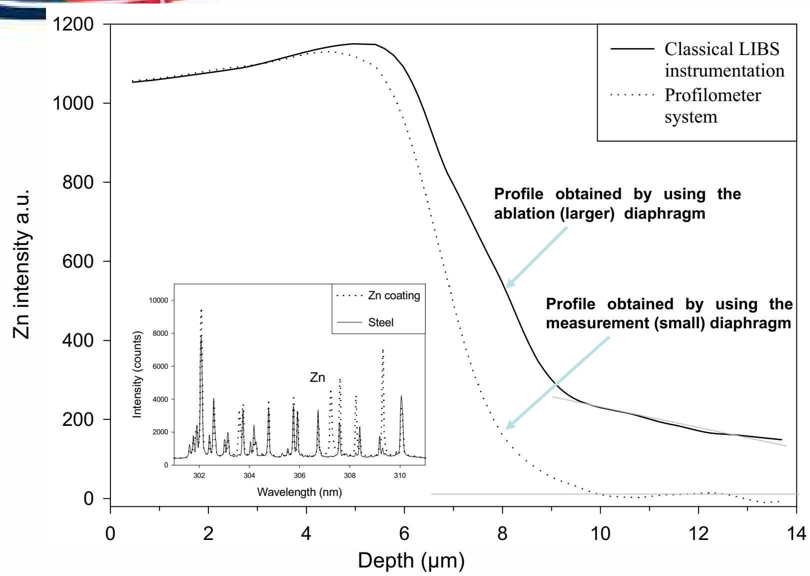
LIBS setup for depth profilometry



Profilometry using 2 diaphragms



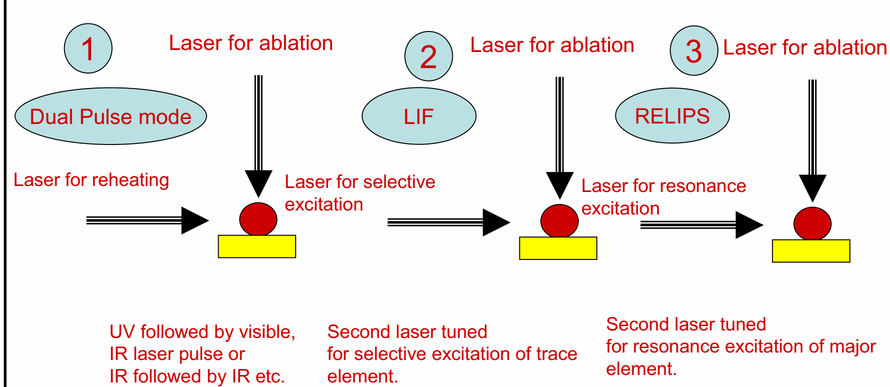
Profilometry



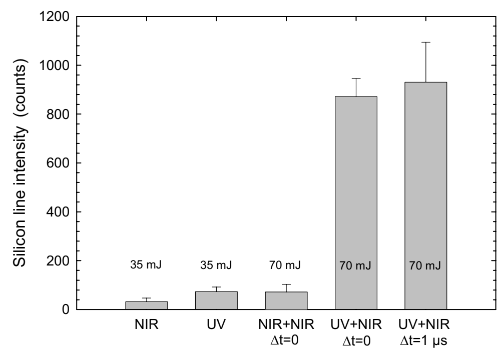
Detection of Gold: moisture effect

Gold concentration (ppm)	Measurement conditions
64.8	Dry sample
2.2	Immediately after watering
68.3	After 5 minutes
63.2	After 10 minutes
65.4	Watering followed by blowing

Approaches to enhance the LIPS sensitivity



Enhanced LIPS using mixed wavelength laser pulses



Conclusion

- The LIBS technique is a useful tool for quantitative analysis of materials.
- The results obtained in terms of accuracy are comparable to conventional techniques.
- LIBS will gain a wide acceptance for process control in a broad range of industrial applications.
- The advent of new detectors and spectrometers promises a bright future for the LIBS technique.
- The LIBS sensitivity can be enhanced by different approaches but at additional cost.

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Thank you for your attention
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