# Ένας ΜικροΗλιος για τον Προδιορισμο Ισχνοστοιχειων

# Εφαρμογες στο Περιβάλλον & την Ιατρική

George Asimellis, Ph.D.

ε΄ κύκλος ομιλιών

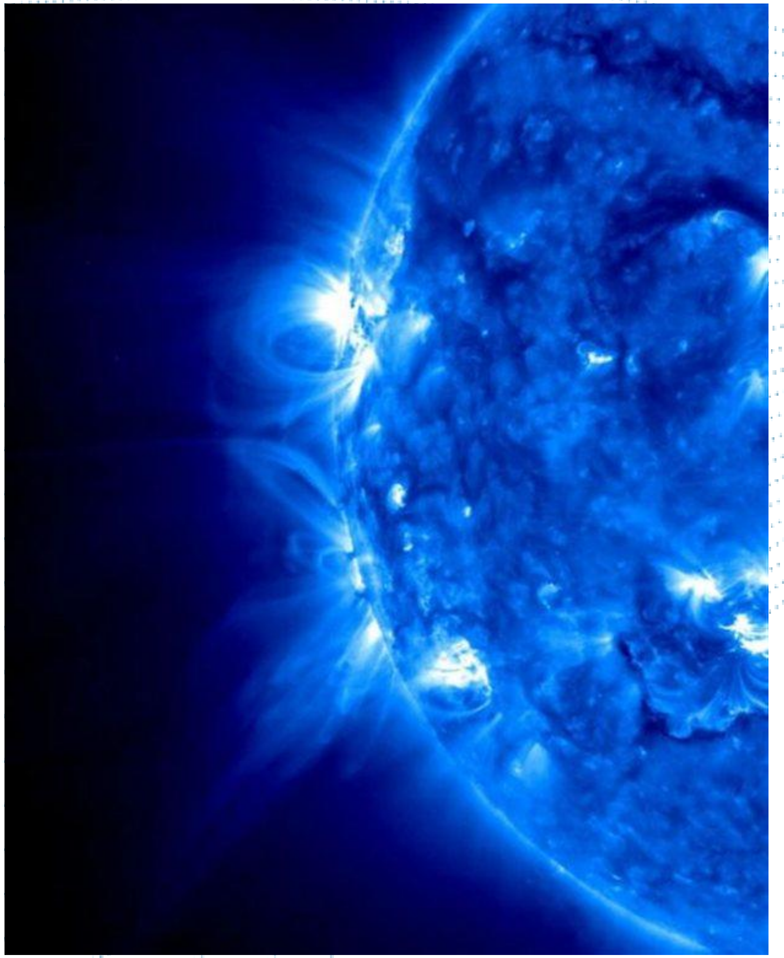
Laser …
μια ακτίνα φωτός
για την Έρευνα,
την Τεχνολογία
και τον Άνθρωπο

Διοργάνωση:
Ινστιτούτο Θεωρητικής
& Φυσικής Χημείας / ΕΙΕ

# Ο Ενάλιθος…

Τί ξέρεις για το φως, Ενάλιθε;
Δάσκαλε, ένα μόνο ξέρω, ότι δεν ξέρω τίποτε..
Καλή αρχή..

# The Sun… Plasma Surface



- Hot, ionized particles interwoven with magnetic fields
- Visible radiation most intense in the yellow-green portion of the spectrum
- Photosphere (effective): 5,778 K
- Corona: ~$5 \times 10^6$ K

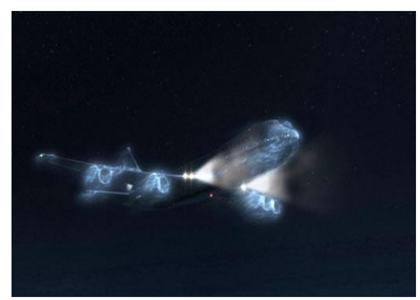
# PLASMA (ΠΛΆΣΜΑ)

**Ionized state**

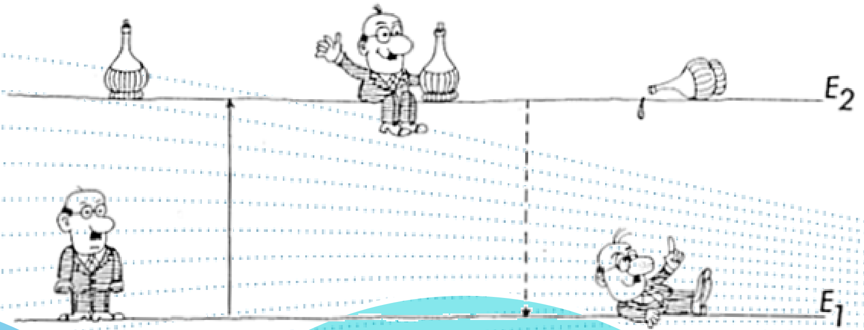
- one of the four fundamental states of matter
- Like gas, plasma does not have a definite shape or a definite volume unless enclosed in a container;
- Unlike gas, under the influence of a magnetic field, it may form structures such as filaments, beams and double layers.

**everyday examples of phenomena made from plasma:**

- Lightning
- electric sparks
- Neon lights
- plasma globe
- Plasma trails

# What 'Color' is Plasma?



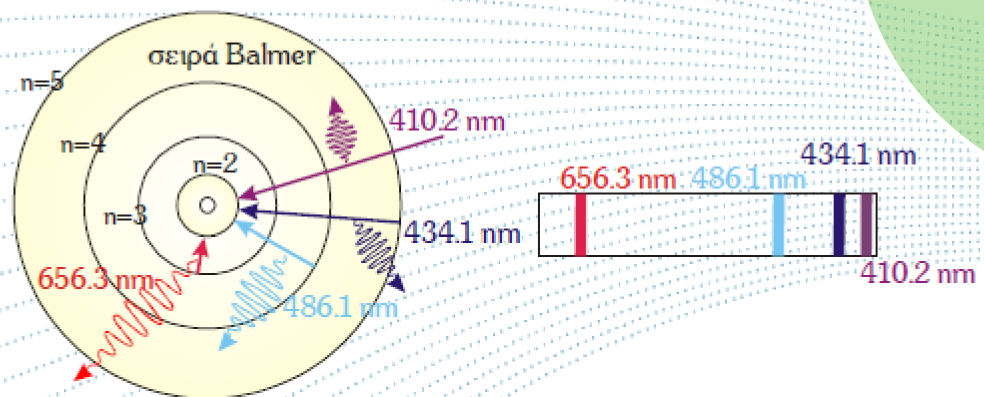

The glowing colors result from electron relaxation in excited states to lower energy states after they recombine

Light is emitted in a spectrum characteristic of the transition

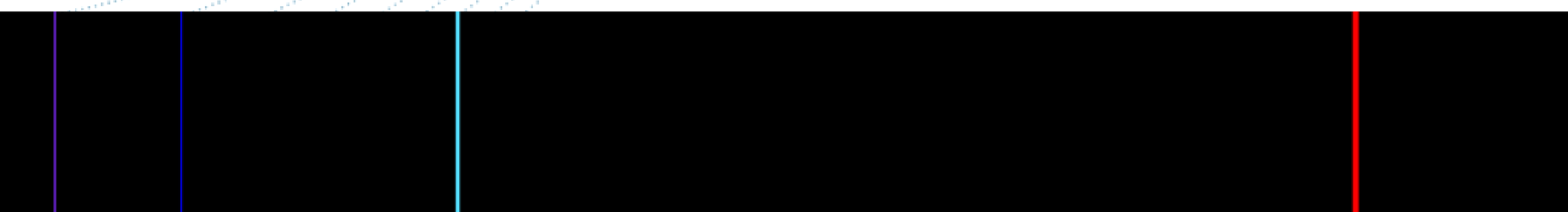
Atomic spectrum: The range of characteristic frequencies of electromagnetic radiation absorbed and emitted by an atom

# Atomic Emission Spectra

'Uniqueness' of atomic structure

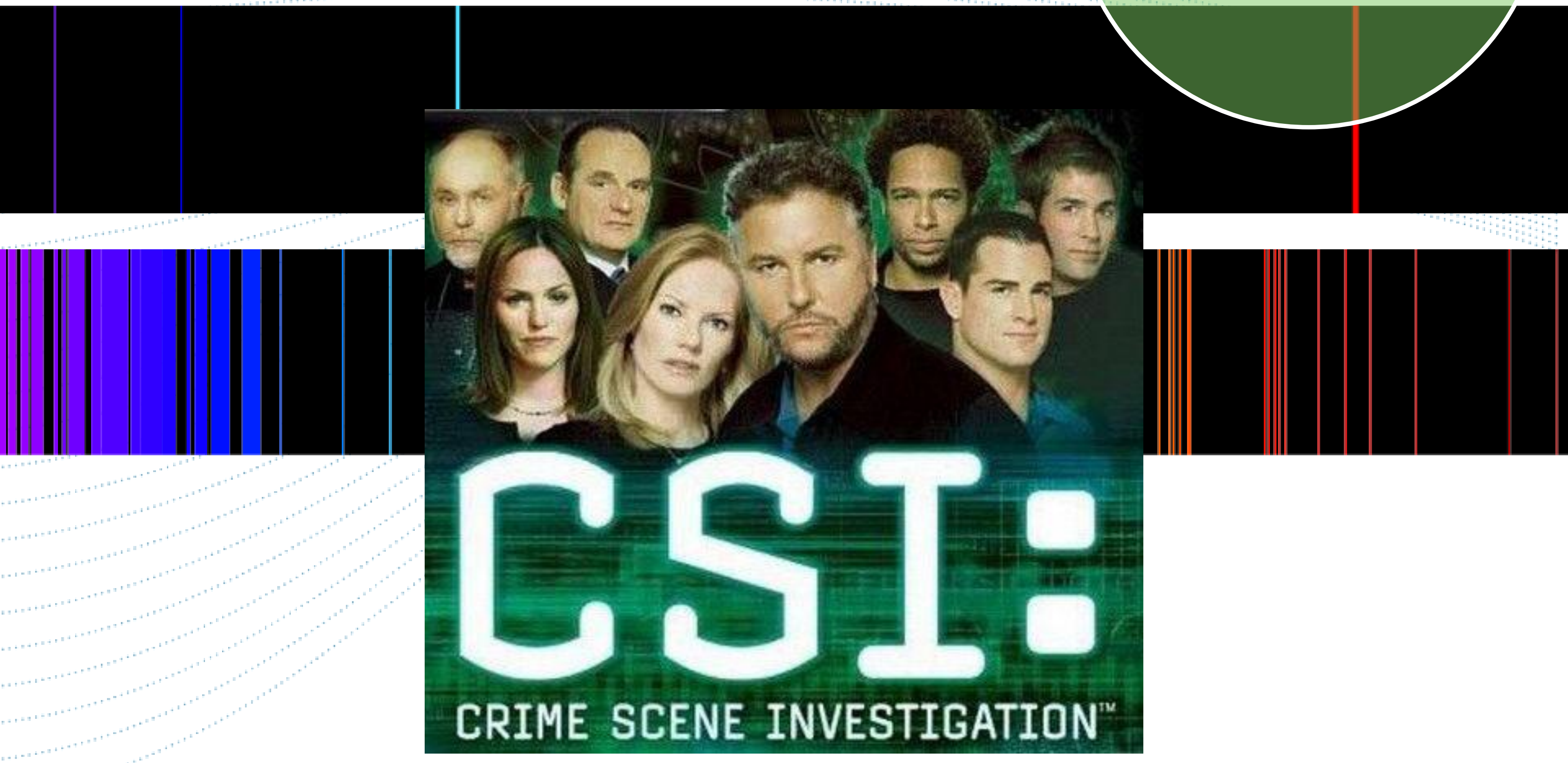



Σχήμα 1-4-2: Ορατό τμήμα από το φάσμα υδρογόνου.



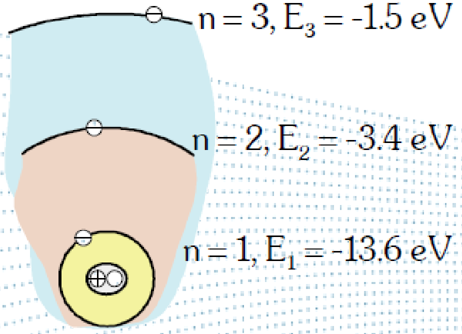
# Atomic Emission Spectra

Identification of atomic spectra: identification of atomic substance

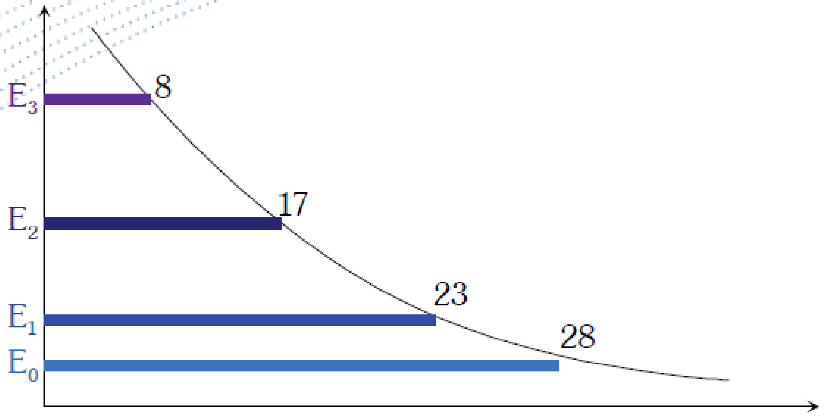


# LASER: ΚΒΑΝΤΙΚΟ ΕΡΓΑΣΤΗΡΙ ΦΩΤΟΣ

Ενεργειακές Στάθμες




Σχετικοί Πληθυσμοί

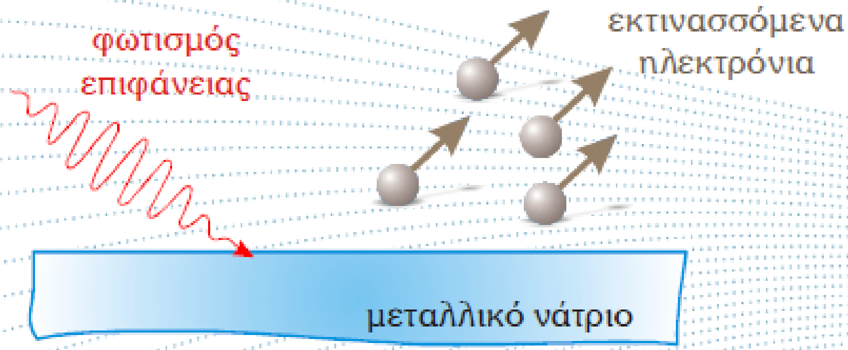


# LASER: THE INITIAL IDEA

από τον ένα ··· λίθο. 1905


[1] *Albert Einstein*, "Über einen die Erzeugung und Verwandlung des Lichtes betreffenden heuristischen Gesichtspunkt", *Annalen der Physik* **17**, 132–148 (1905).

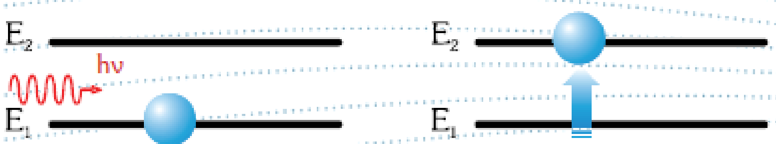
# Trying to Solve the Photoelectric Effect



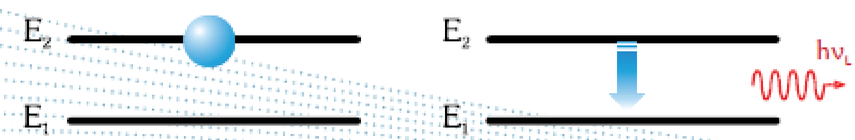


Energy is Quantized!

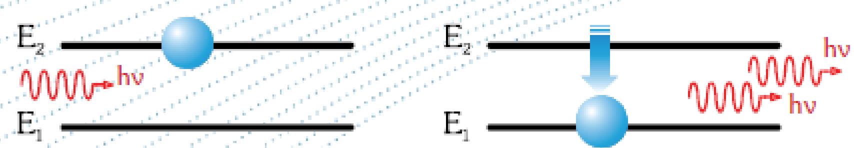
# Laser: Αλληλεπιδρασεις



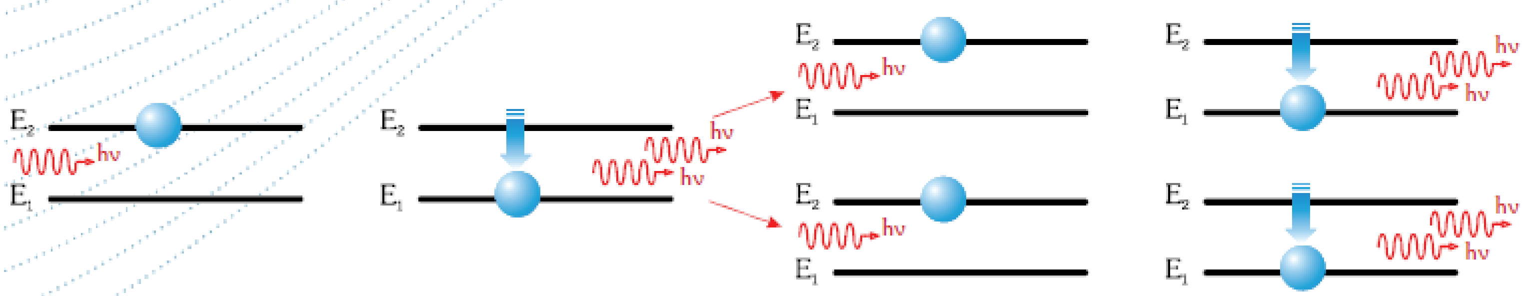

*Σχήμα 7-2-4: Η διαδικασία της κβαντικής απορρόφησης.*




*Σχήμα 7-2-2: Η διαδικασία της αυθόρμητης εκπομπής.*

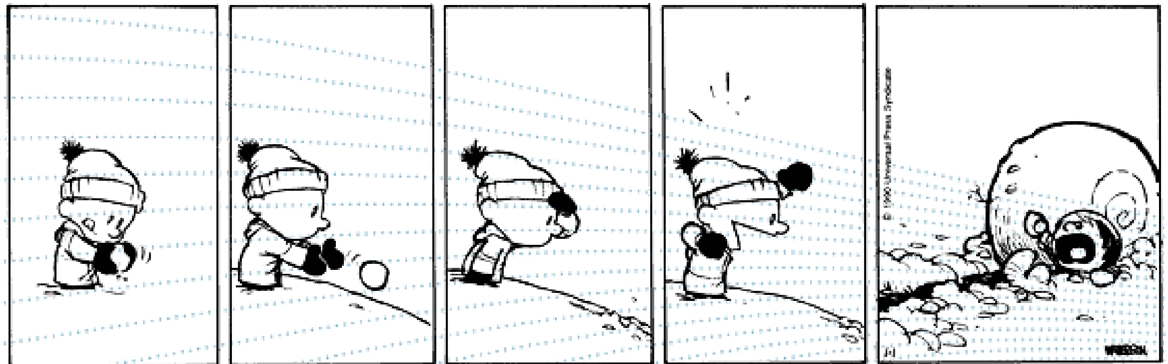



*Σχήμα 7-2-3: Η διαδικασία της εξαναγκασμένης εκπομπής.*

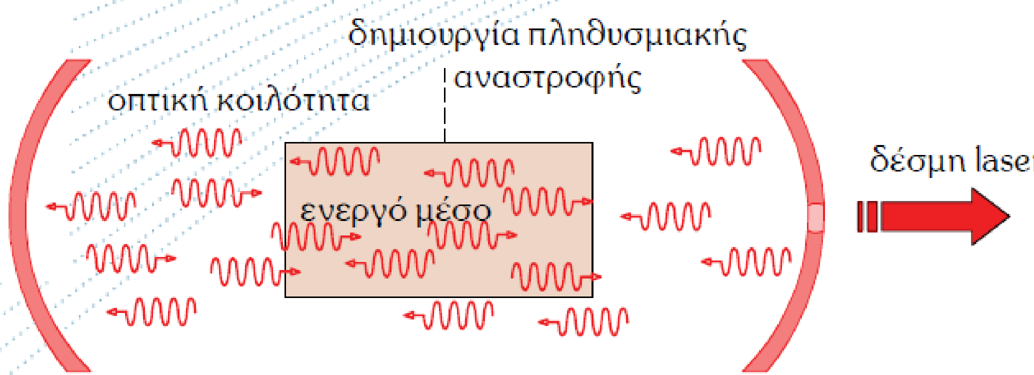



*Σχήμα 7-3-2: Η εκκίνηση της διαδικασίας laser.*

# AMPLIFICATION - PHOTON AVALANCHE



**L**ight
**A**mplification by
**S**timulated
**E**mission of
**R**adiation

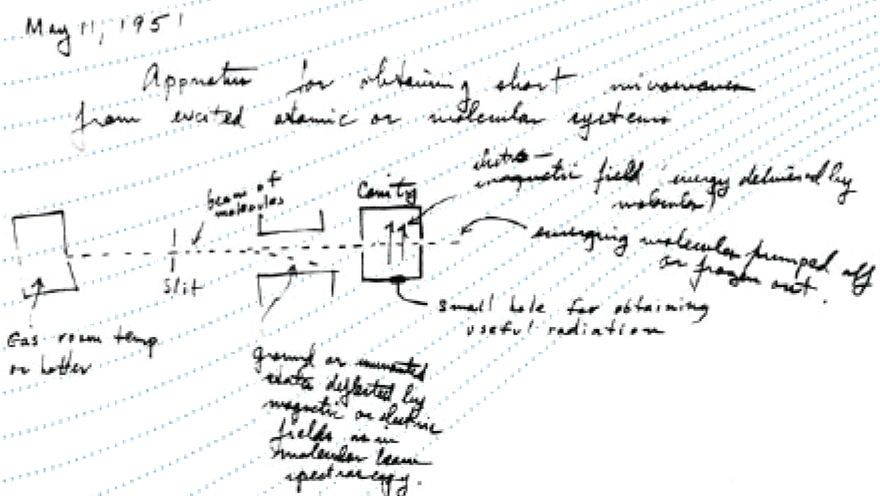



*Σχήμα 7-3-3: Σχηματικό διάγραμμα λειτουργίας του laser.*

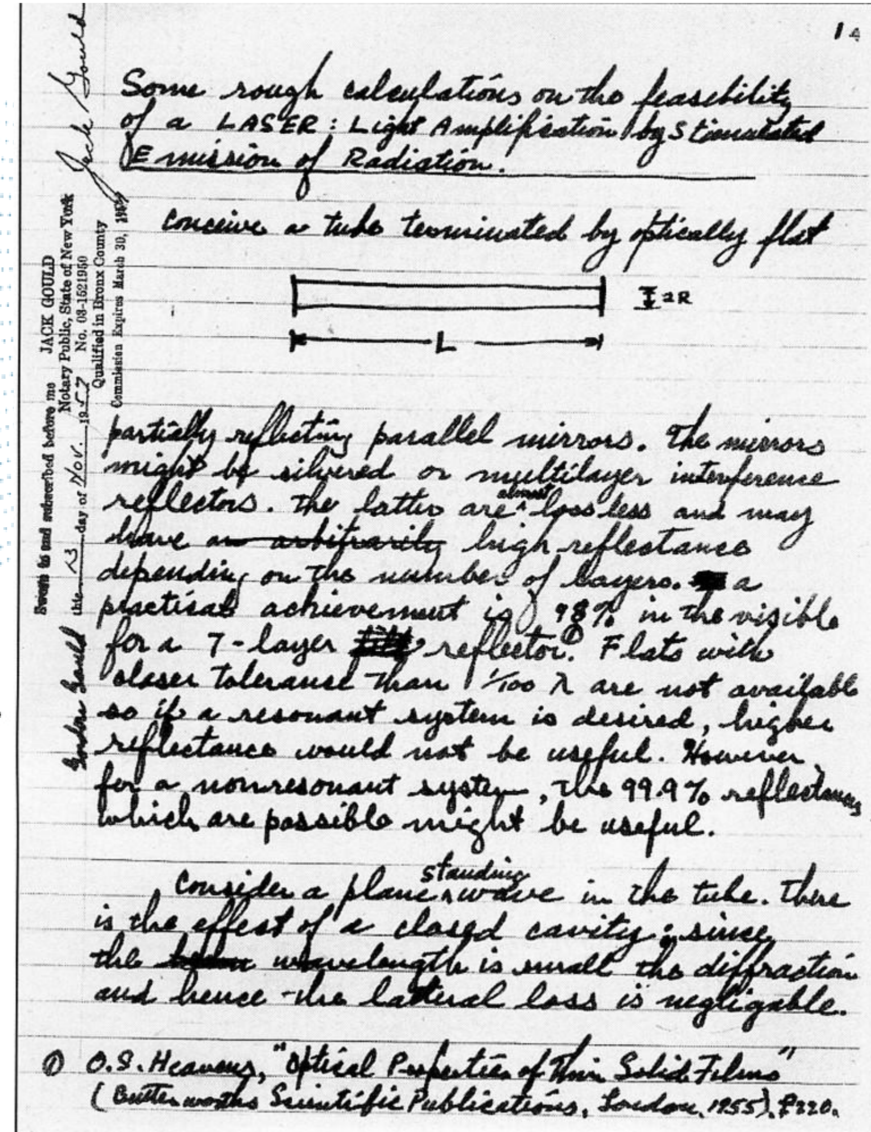
# The LASER Birth Certificate

Николай Геннадьевич βasov
Александр Михайлович Прохоров

Charles Hard Townes
Arthur Schawlow
Gordon Gould




Σχήμα 7-5-1: Η ληξιαρχική πράξη γέννησης του Maser.



14

Some rough calculations on the feasibility of a LASER: Light Amplification by Stimulated Emission of Radiation.

Conceive a tube terminated by optically flat



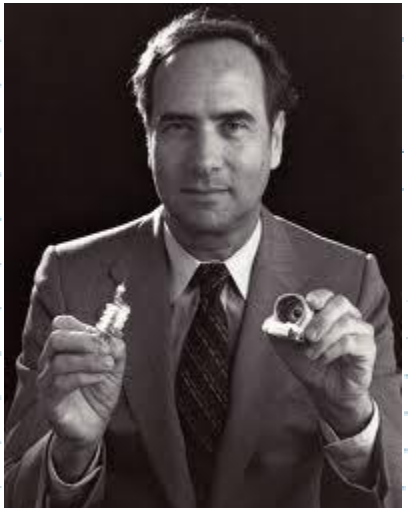
partially reflecting parallel mirrors. The mirrors might be silvered or multilayer interference reflectors. The latter are almost loss-less and may have ~~an arbitrarily~~ high reflectance depending on the number of layers. A practical achievement is 98% in the visible for a 7-layer reflector.① Flats with closer tolerance than 1/100 λ are not available so if a resonant system is desired, higher reflectance would not be useful. However for a nonresonant system, the 99.9% reflectances which are possible might be useful.

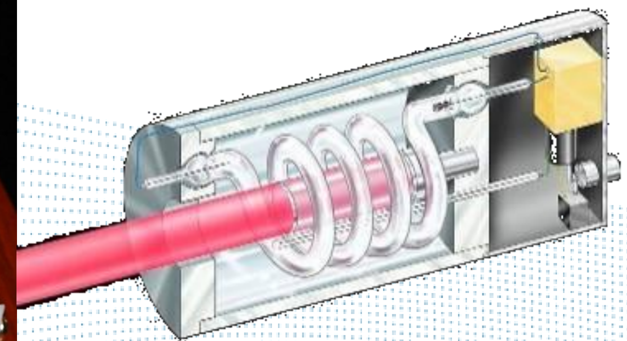
Consider a plane standing wave in the tube. There is the effect of a closed cavity; since the wavelength is small the diffraction and hence the lateral loss is negligible.

① O.S. Heavens, "Optical Properties of Thin Solid Films" (Butterworths Scientific Publications, London, 1955), p.320.

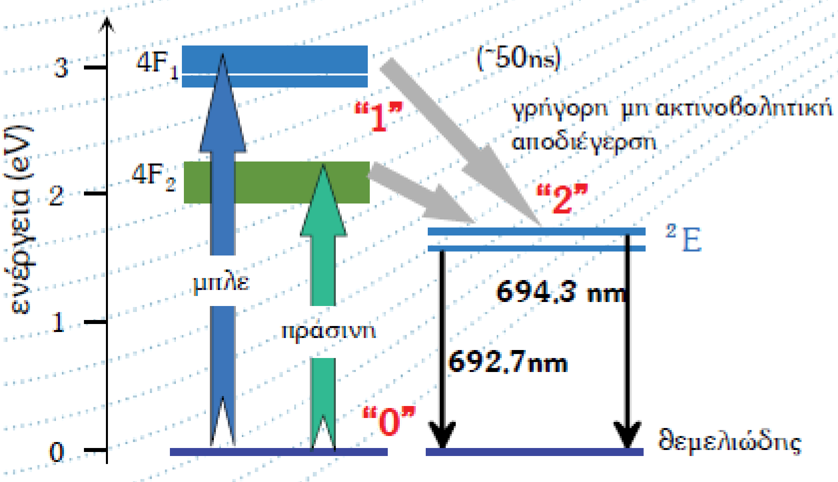
Sworn to and subscribed before me this 13 day of Nov. 1957
Jack Gould
JACK GOULD
Notary Public, State of New York
No. 03-1621950
Qualified in Bronx County
Commission Expires March 30, 1959
Gordon Gould

# The First LASER





Maiman, 1960




*Σχήμα 7-5-7: Το ενεργειακό διάγραμμα laser Ρουβιδίου.*

The broadband optical pumping of a synthetic pink ruby crystal using a flash lamp is capable of raising a substantial fraction of the chromium ions to the upper laser level.

It consisted of a ruby crystal surrounded by a helicoidal flash tube enclosed within a polished aluminum cylindrical cavity cooled by forced air.

The ruby cylinder forms a Fabry-Perot cavity by optically polishing the ends to be parallel to within a third of a wavelength of light.

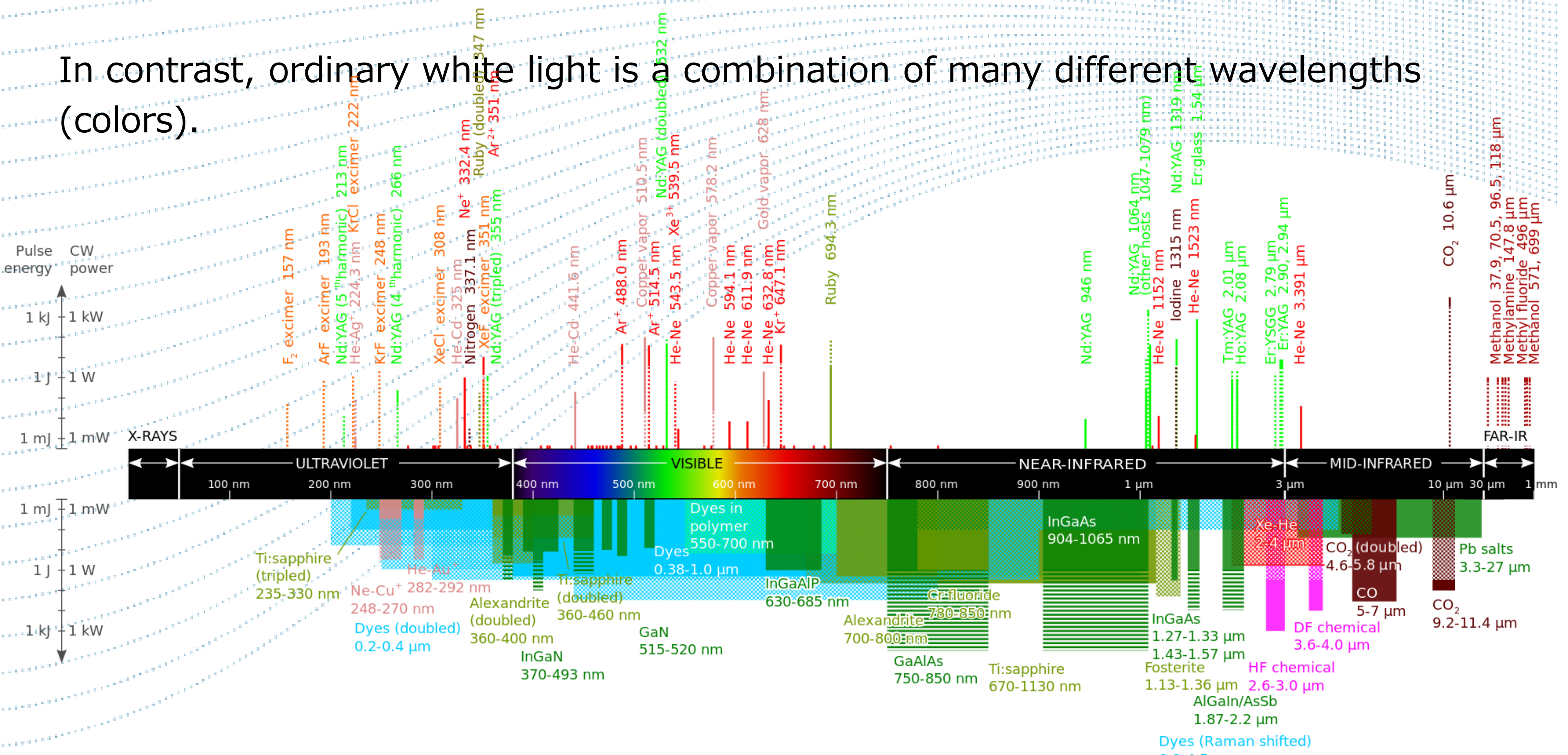
Each end was coated with evaporated silver, one end was made less reflective to allow some radiation to escape as a beam.

# Laser Properties

## Monochromaticity

The light emitted from a laser is monochromatic, that is, it is of one wavelength (color).

In contrast, ordinary white light is a combination of many different wavelengths (colors).



# Laser Properties

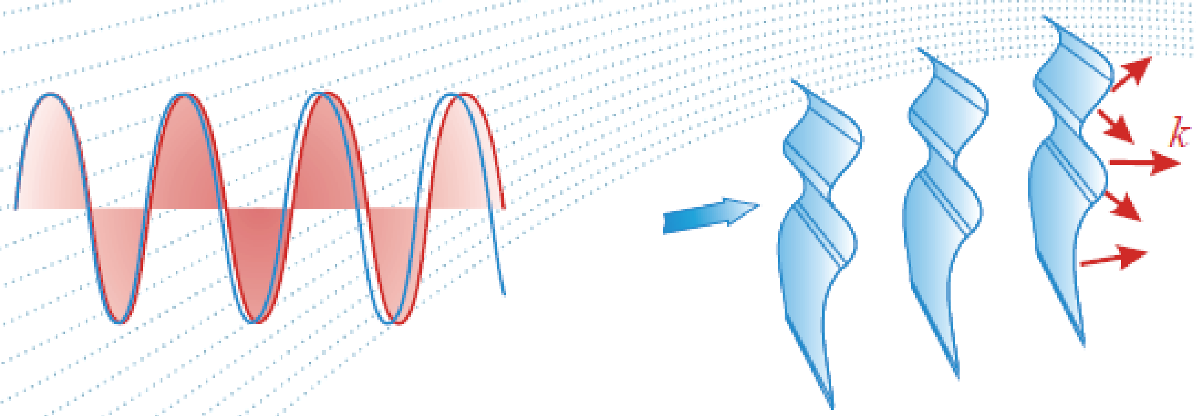
Coherence

# LASER PROPERTIES

Coherence:

**Temporal**
- Relating to 'same frequency'

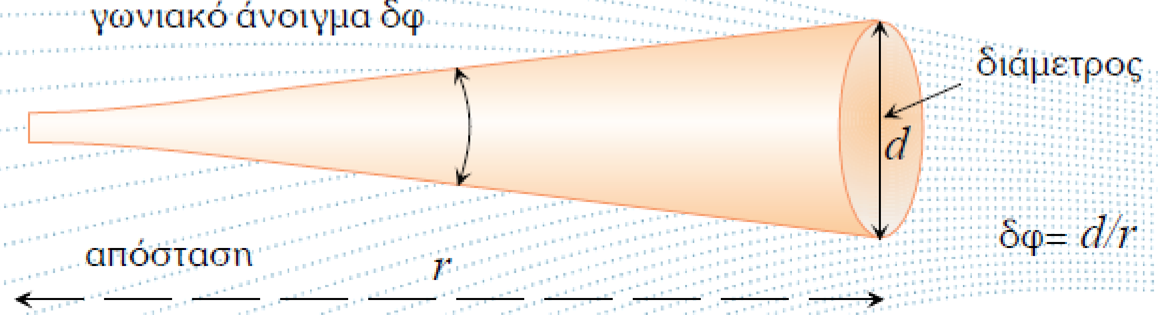
**Spatial**
- Relating to 'small aperture'




Σχήμα 7-3-20: Μερική συμφωνία δύο κυμάτων, χρονική και χωρική.

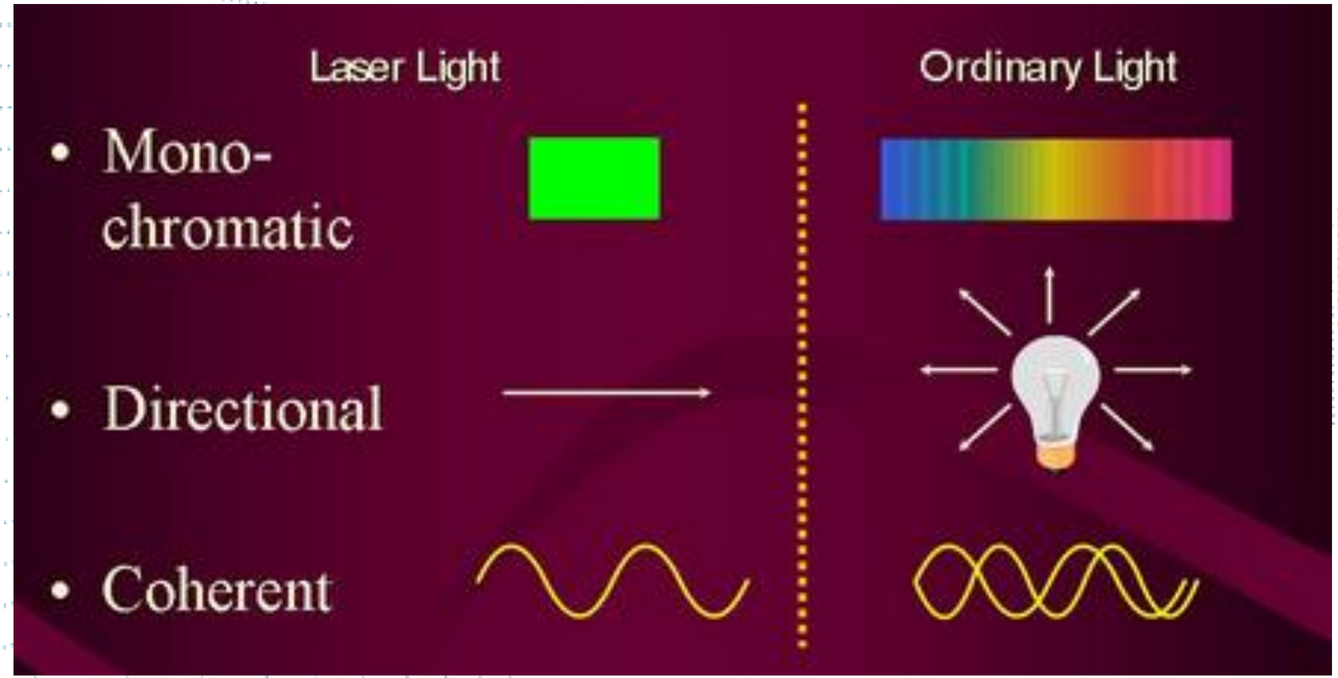
# Laser Properties

Directionality (result of spatial coherence)




*Σχήμα 7-3-21: Ορισμός κατευθυντικότητας.*

# Laser Properties: Synopsis




# Do you Expect me to Talk?


# LOOKING FOR A JOB

*"Initially, the laser was called an invention looking for a job."*
*Harry Stine*

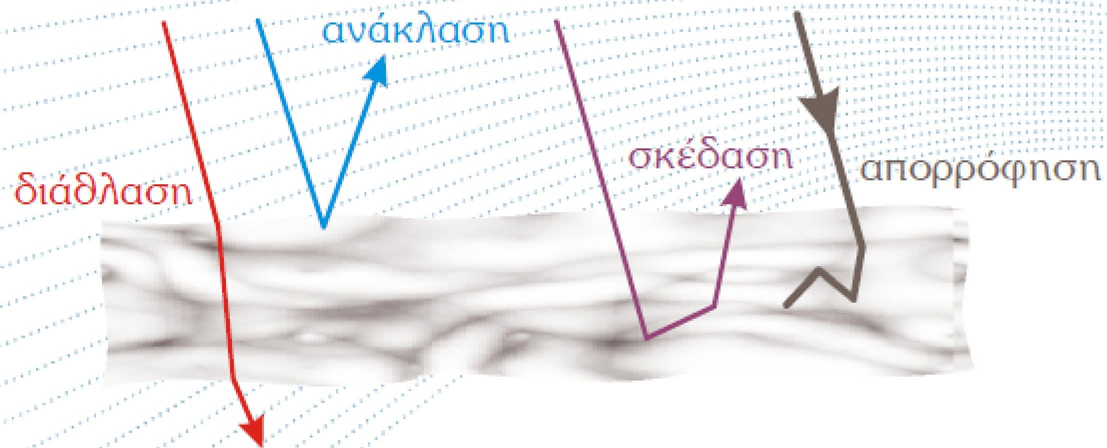
Laser-Matter interaction

Light absorption

We cannot change the laws of Physics

# Laser-Matter Interaction

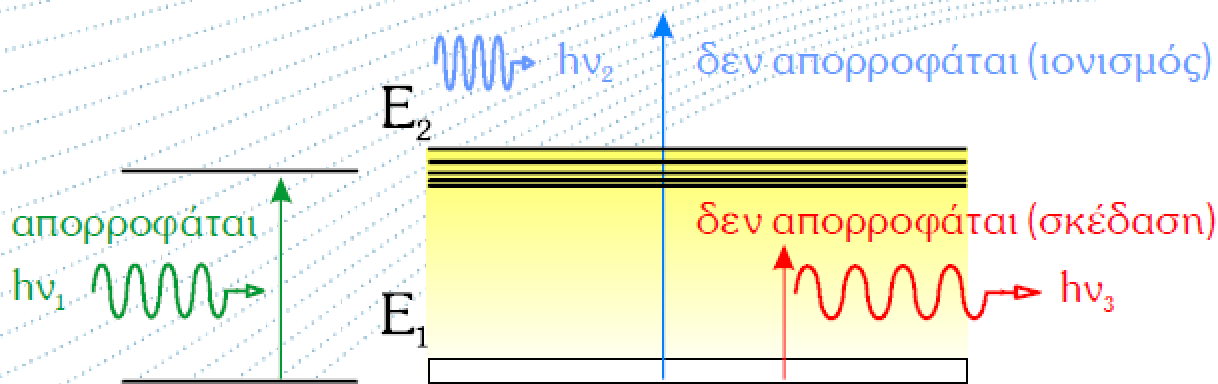
The four basic interactions




Σχήμα 11-2-8: Αλληλεπιδράσεις φωτός με ιστό.

# PHOTON ABSORPTION?

Depends on laser 'frequency' / color




Σχήμα 1-4-3: Η απορρόφηση των φωτονίων εξαρτάται από την ενεργειακή δομή του υλικού στο οποίο προσπίπτει.

# LASER-MATTER INTERACTION

## Absorption - Fluorescence

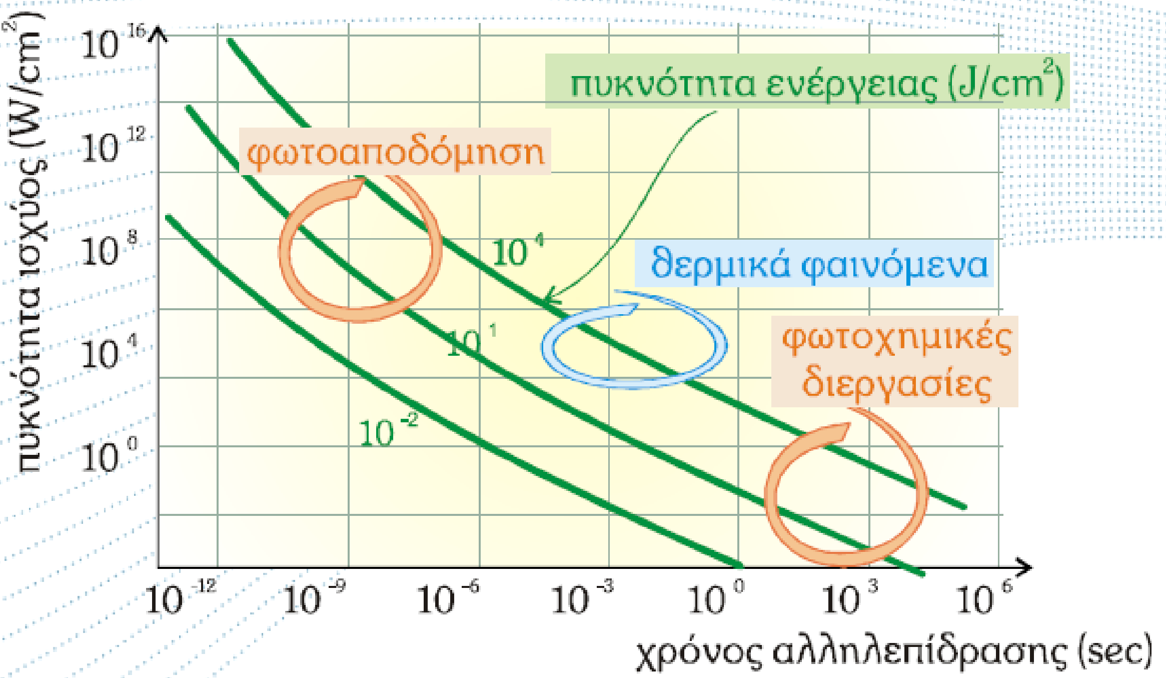

Σχήμα 1-4-6: Μηχανισμός κβαντικής απορρόφησης και φθορισμού.

## Photo-ionization


Σχήμα 1-4-8: Μηχανισμός φωτοϊονισμού.

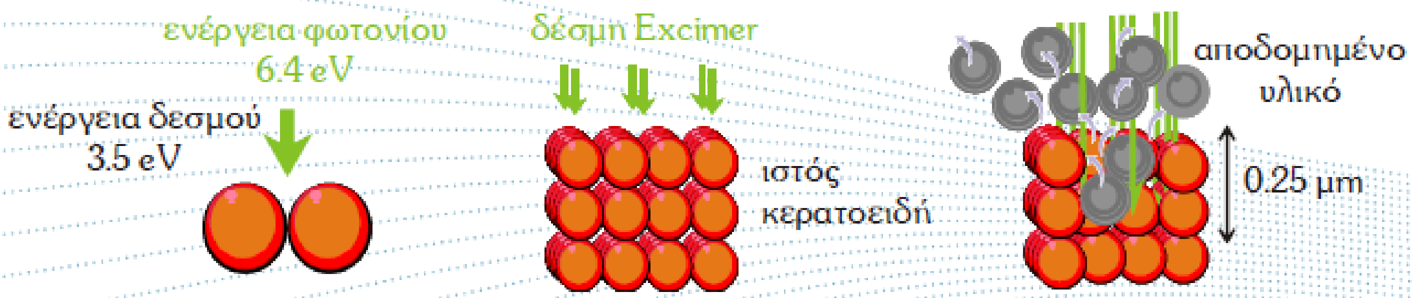
# PULSED LASER PHOTOABLATION

## Absorption - Fluorescence



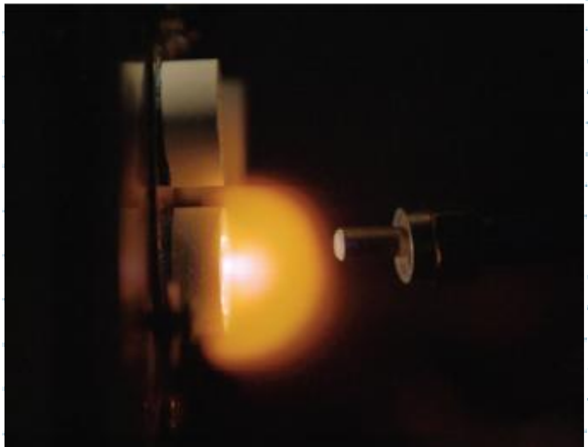

Σχήμα 11-2-9: Οι 'συνέπειες' της απορρόφησης από τον ιστό εξαρτώνται από το χρόνο αλληλεπίδρασης -διάρκεια παλμού- και την πυκνότητα ισχύος.

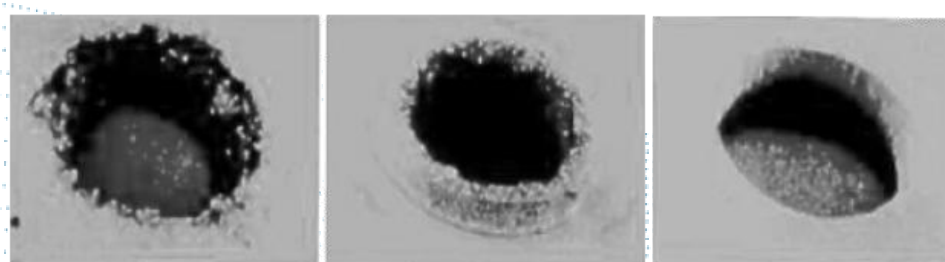
# PHOTODISRUPTION



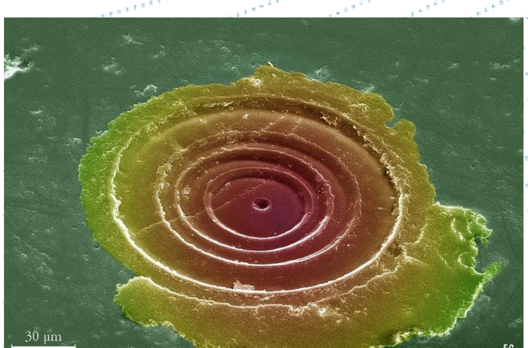

Σχήμα 11-2-11: Φωτόνια από excimer laser, με ενέργεια 6.4 eV, μπορούν να διασπάσουν τους μοριακούς δεσμούς κολλαγόνου, εισερχόμενα σε βάθος 0.25 μm.

# LASER-INDUCED BREAKDOWN





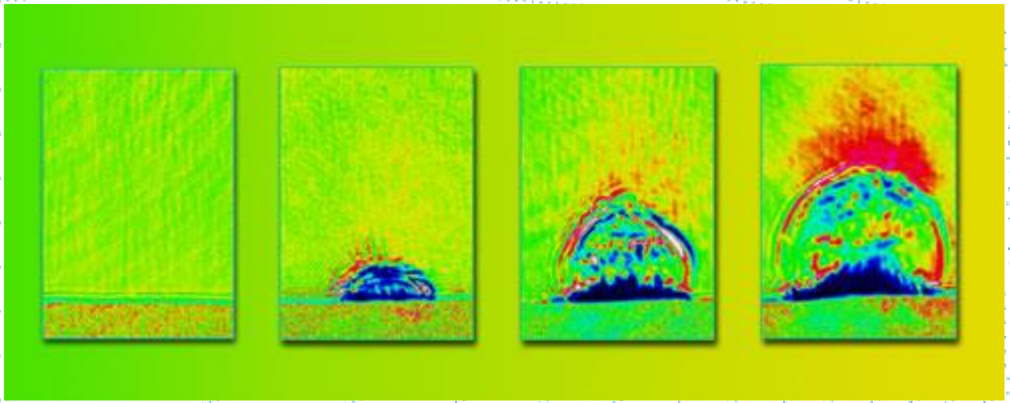
Σχήμα 11-2-13: Κρατήρες από φωτοαποδόμηση με θερμική φωτοαποδόμηση με
Nd:YAG και (β) με $CO_2$, και (γ) με μη θερμική φωτοαποδόμηση με Excimer Laser

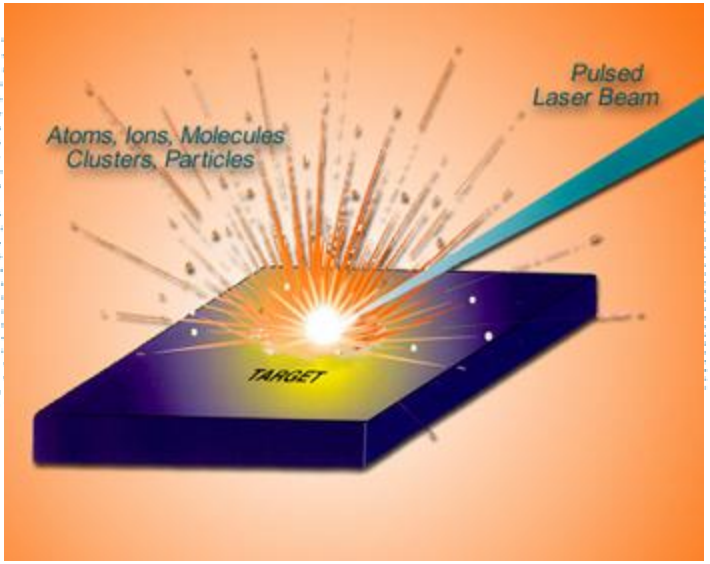




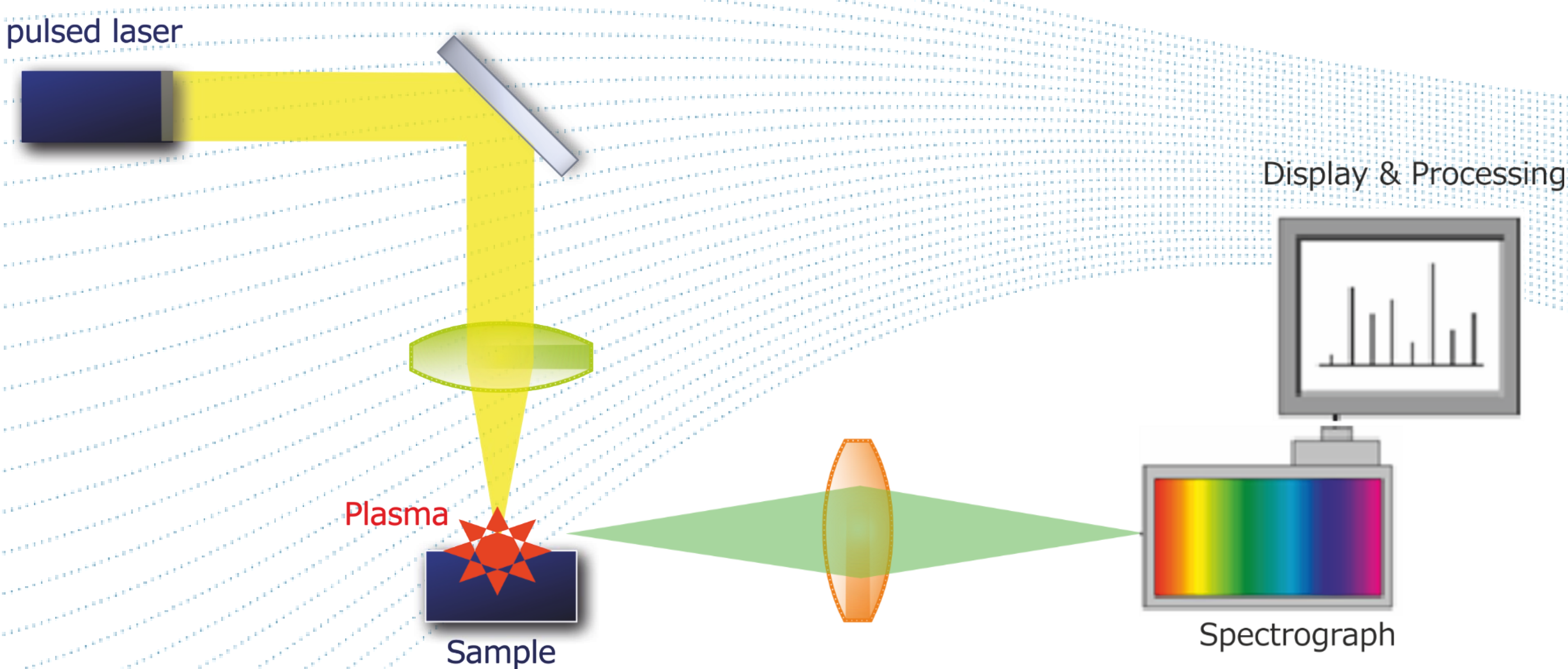
Σχήμα 11-2-7: Χάραξη λογότυπου της εταιρείας Lamda Physik πάνω σε τρίχ

# Laser-Induced Breakdown

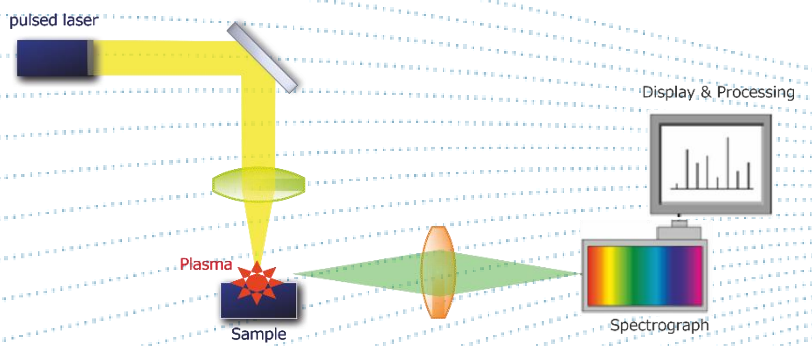





# Laser-Induced Breakdown Spectroscopy



# How LIBS Works




- Pulsed laser focused on to sample ($0.5mm^2$)
- Intense optical energy ablates/excites small amount of sample
- OES techniques used to collect and analyze data
- Spectral data processed
- Results analyzed and displayed via specialized software

# Commercial LIBS Instrument


TRACER™ 2100
66cm H x 41cm D x 74cm W

Autosampler Stage

**Rapid sample preparation**

**No solvents or additives**

**Primary and trace elements (ppm), - high dynamic range**

**Low Z elements (Li, Be, B, C)**

**Solids, liquids and/or slurries**

- Multi-sample, multi-element analysis
- Automated calibration & analysis
- Ruggedized for factory & field

# Recent Commercial Applications

- Oil Exploration & Drilling
  - ✓Chemostratigraphy
- Mining
  - ✓Ore Grading (Phosphates)
  - ✓On-line Process Control – Ore Slurry
- Manufacturing QA
  - ✓Catalyst Coatings – precious Metals (Pt, Pd, Rh)
- Rapid Material Sorting
  - ✓Metal Sorting
  - ✓Ore Sorting - Beneficiation

# LIBS ANALYSIS ON OIL RIGS

On-rig, near real time, LIBS analysis

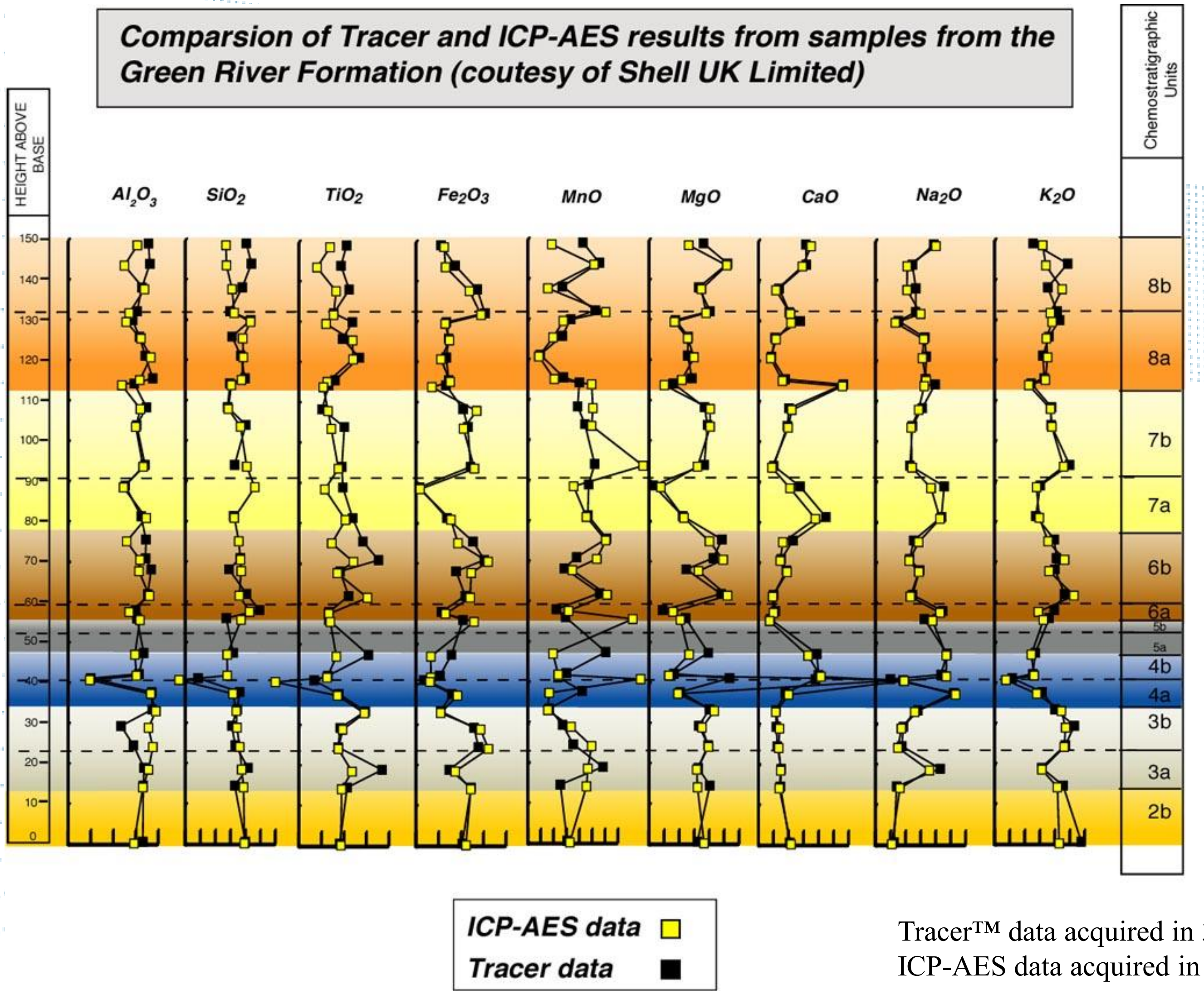
**Tracer analyzes drill cuttings within minutes of reaching the surface.**

**Chemostratigraphy locates the precise strata that is being drilled, allowing:**

- Look ahead for fast drilling
- Accurate casing points
- Accurate total depth
- Directional drilling precision
- Chemosteering

**Operational in North Sea, Canada and in the Gulf Coast**

# CHEMOSTRATIGRAPHY – GEOCHEMICAL ROCK ANALYSIS




Tracer™ data acquired in 3 hours
ICP-AES data acquired in 3 days

# ON-SITE CALIBRATION CURVES IN ROCK MATRICES

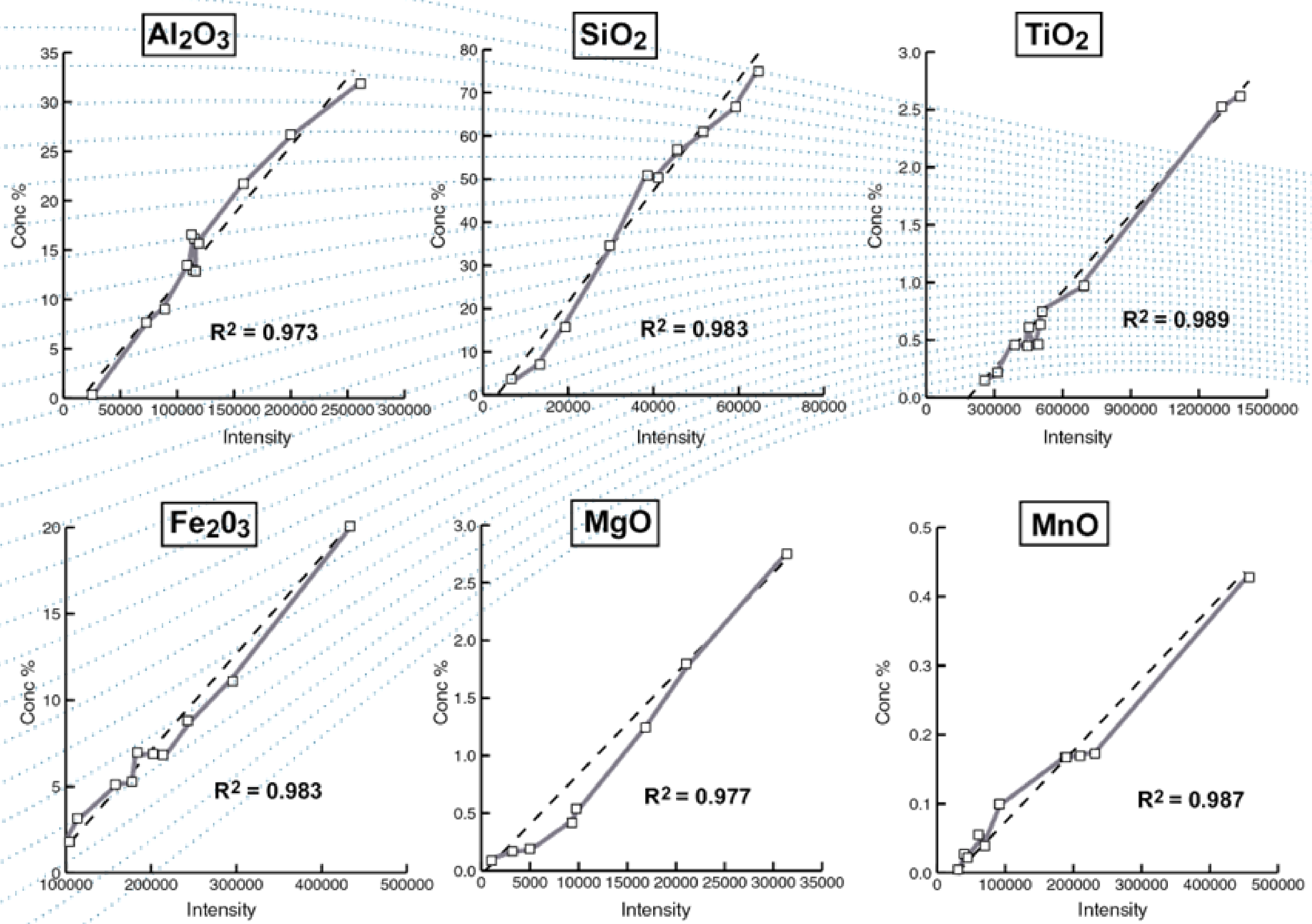



Fig. 6. Calibration curves for selected elements based on the analysis of Standard Reference Materials and In-house standards by Tracer™. Details of the elemental concentrations in individual stards are reported in Table 1.

# On-Site Phosphate Ore Grading

Mine Overview

Sampling Ore

Requirements

- Multiple, bedded seams vary significantly in composition
- Visual distinction is difficult
- Mill feed makeup is critical

# On-site Phosphate Ore Grading

LIBS Applicability

- Simple requirements to operate
- Easy to use
- On-site ore analysis can easily classify ore – high, medium, low grade
- Results returned in minutes

Working in the field

Very Simple Environment

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Spectrochimica Acta Part B 61 (2006) 1253 – 1259

SPECTROCHIMICA ACTA PART B

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Phosphate ore beneficiation via determination of phosphorus-to-silica ratios by Laser Induced Breakdown Spectroscopy

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[b] School of Chemical Engineering, National Technical University of Athens, 9, Iroon, Polytechniou Str., Zografou, 15780 Athens, Greece
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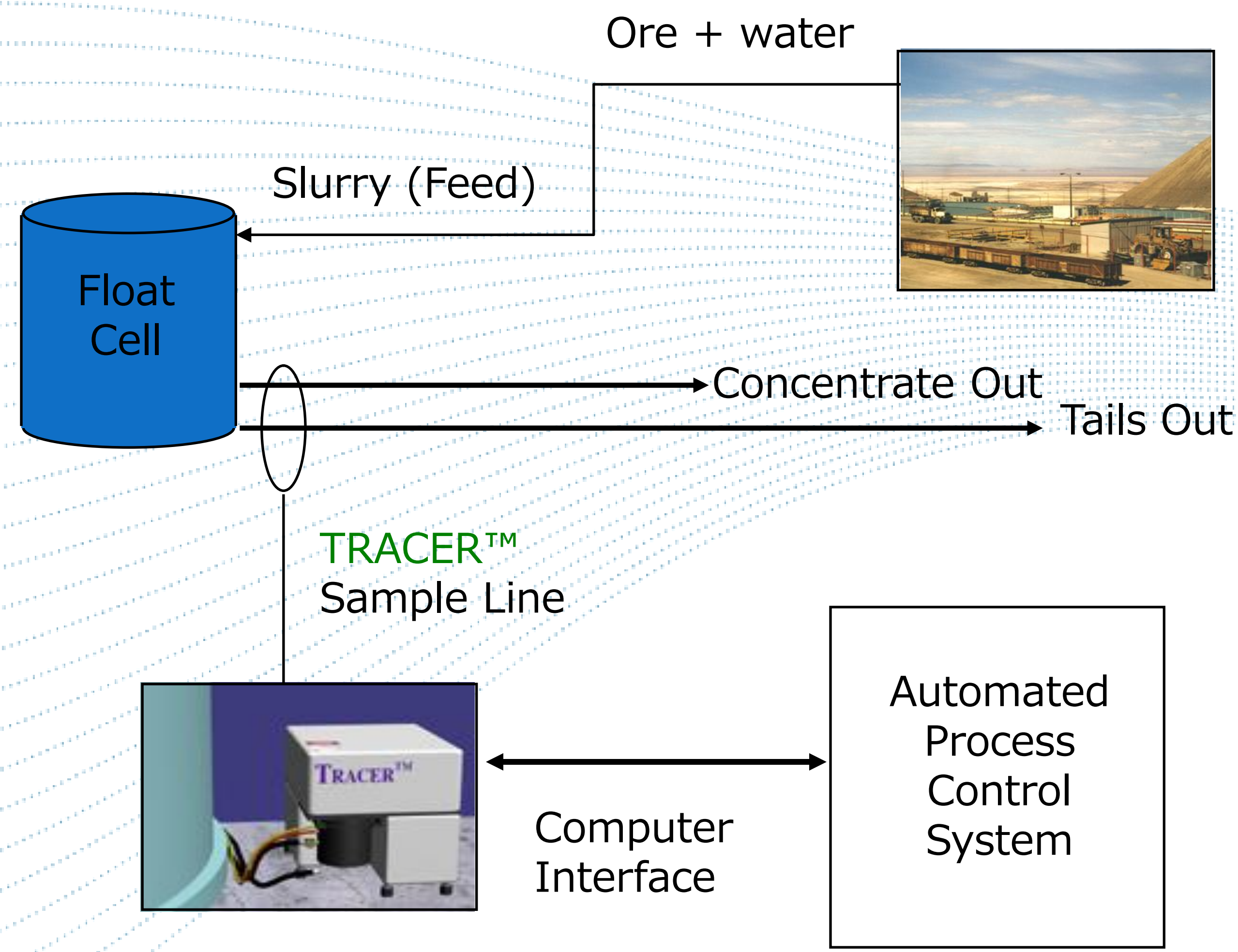
Received 26 July 2006; accepted 5 October 2006

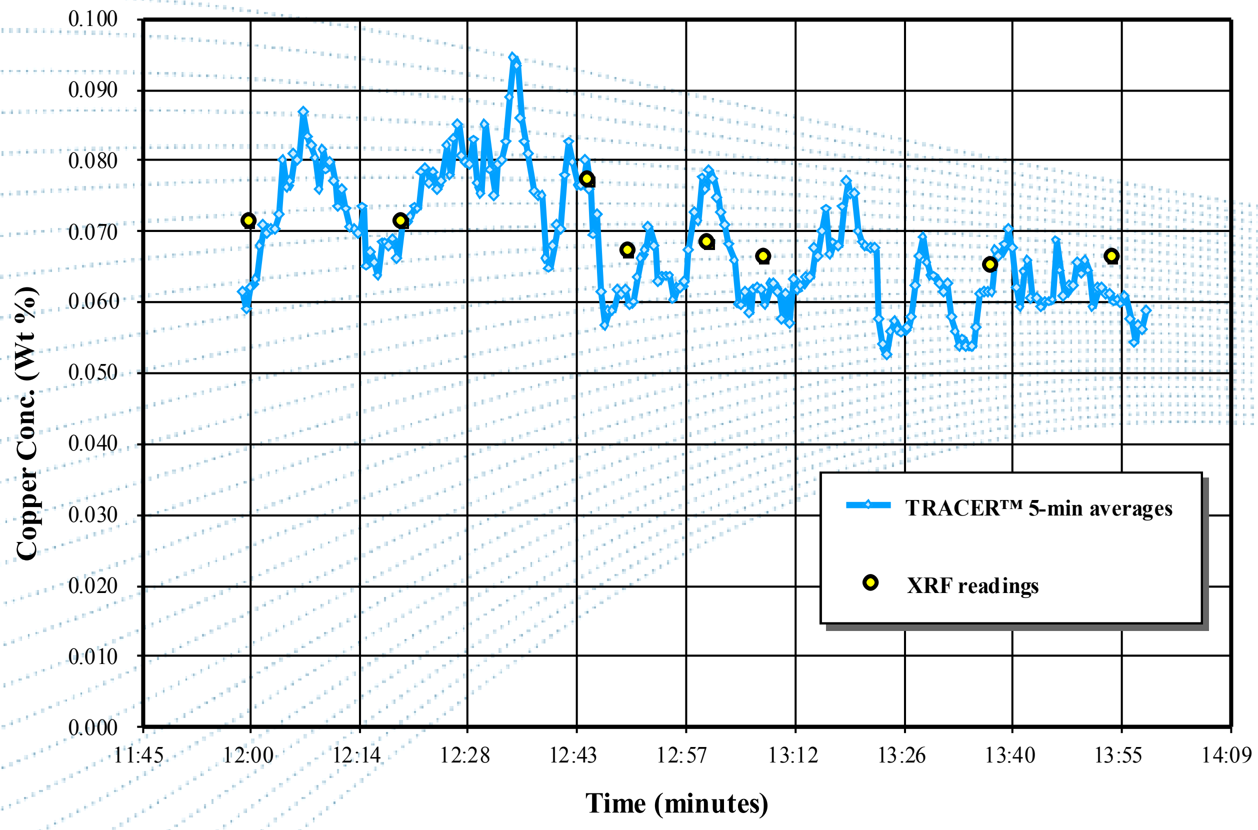
**Abstract**

We report development and application of an in-situ applicable method to determine phosphate ore rock quality based on Laser-Induced Breakdown Spectroscopy (LIBS). This is an economically viable method for real-time evaluation of ore phosphate rocks in order to separate high-silica pebbles prior to deep beneficiation. This is achieved by monitoring relative emission line intensities from key probe elements via single laser ablation shots: the ratio of the phosphorous to silica line intensities (P/Si ratio) provides a simple and reliable indicator of ore rock quality. This is a unique LIBS application where no other current analytical spectroscopic method (ICP or XRF) can be applied. Method development is discussed, and results with actual ore samples are presented.

# Online Cu Slurry Measurement

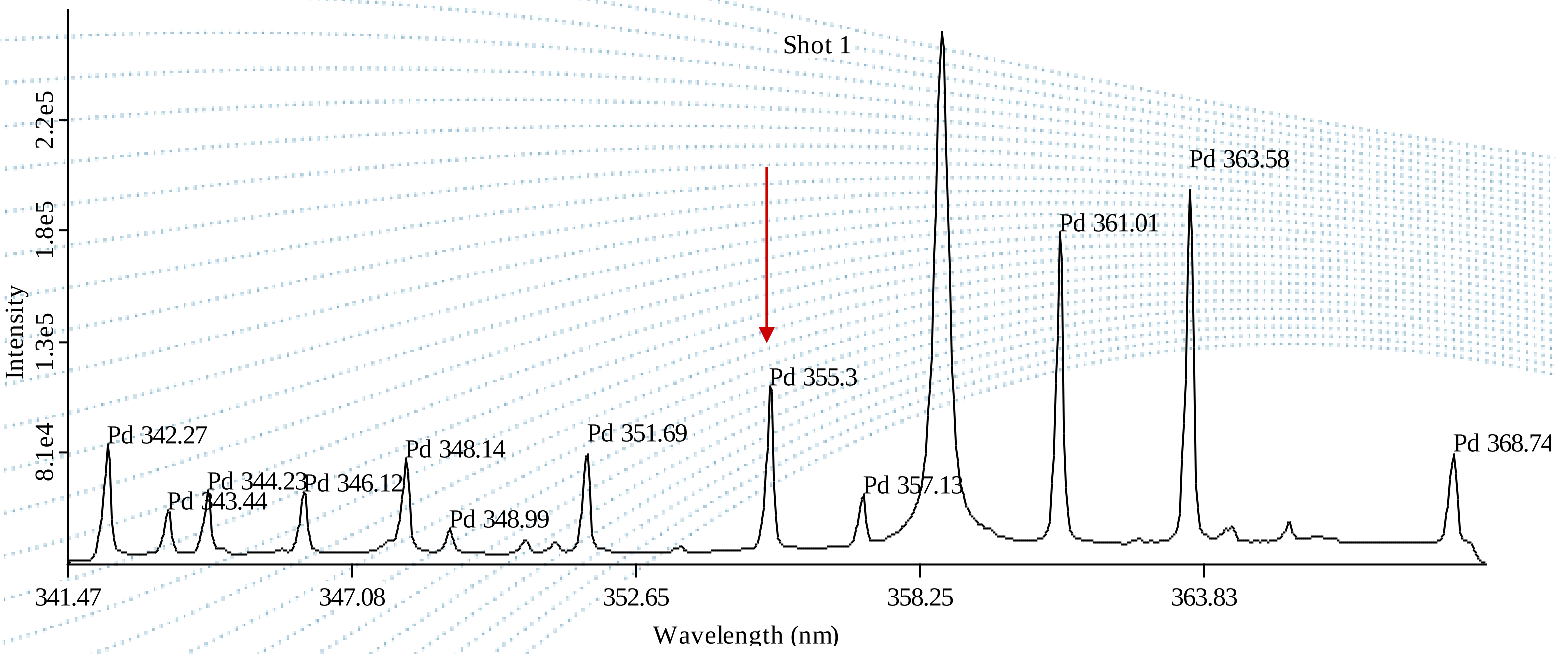


# On-line slurry Cu Slurry Analysis



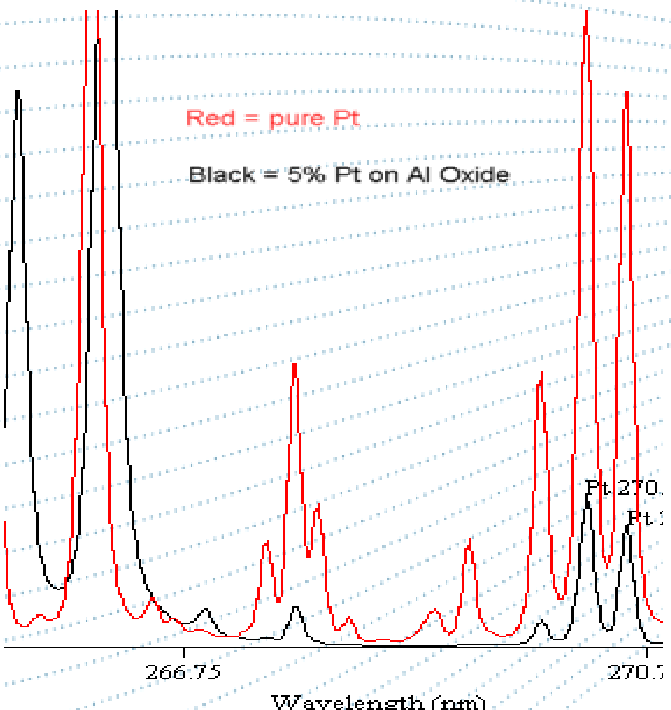

On-line data showing dynamic response of real-time LIBS data vs. the less responsive XRF analysis.

# Catalyst Coating QA Analysis – Spectral Line Window

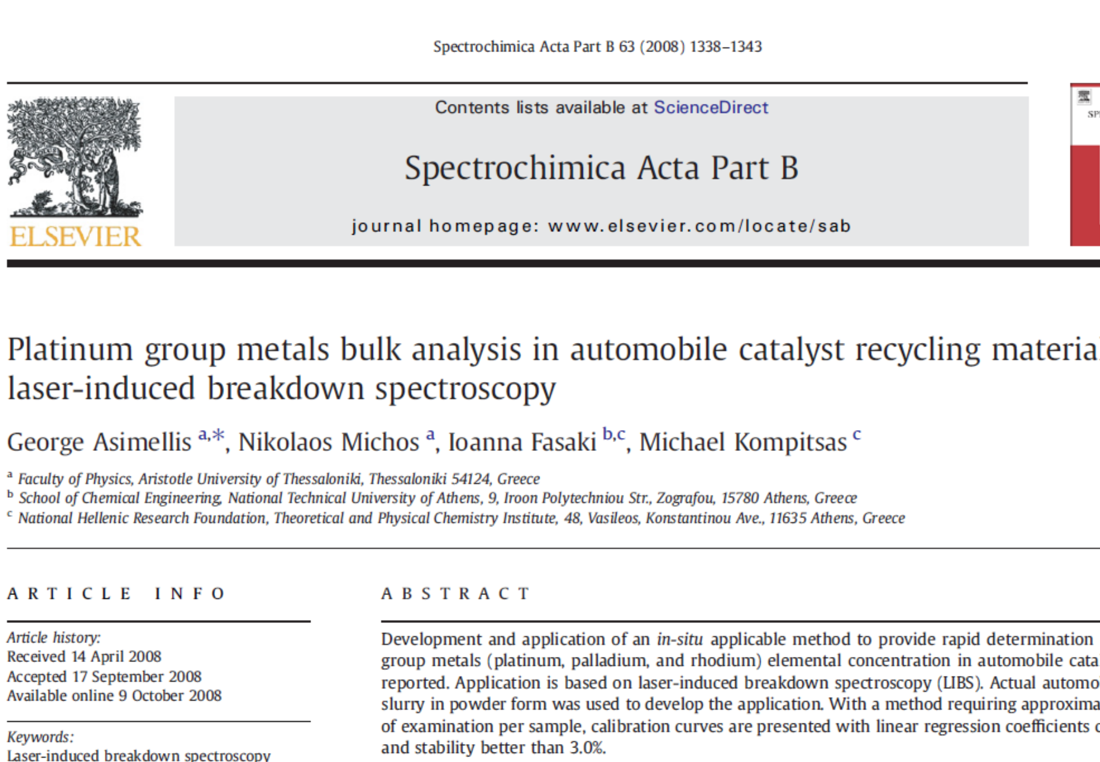



Palladium

# CATALYST COATING QA ANALYSIS – SPECTRAL LINE WINDOW




Platinum

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Platinum group metals bulk analysis in automobile catalyst recycling materia
laser-induced breakdown spectroscopy


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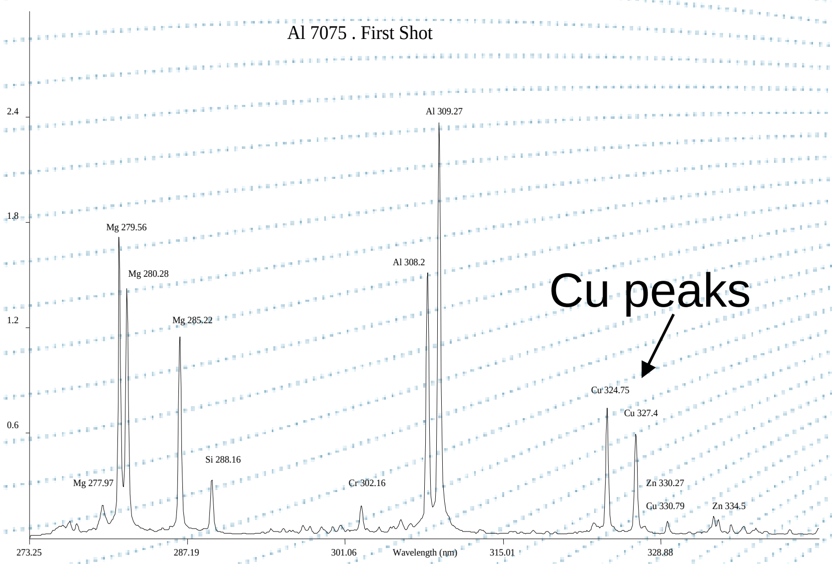
ABSTRACT

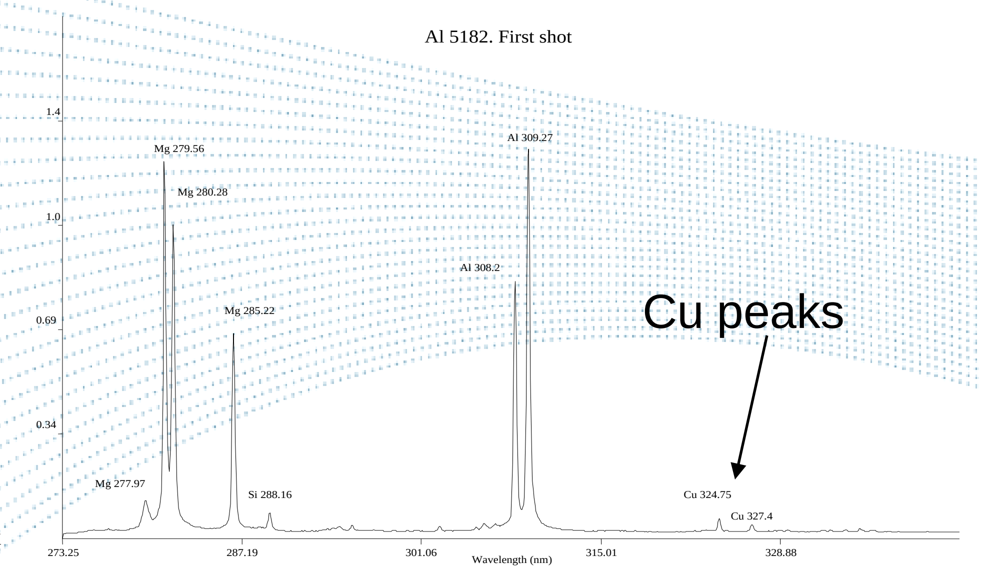
Development and application of an *in-situ* applicable method to provide rapid determination
group metals (platinum, palladium, and rhodium) elemental concentration in automobile cata
reported. Application is based on laser-induced breakdown spectroscopy (LIBS). Actual automo
slurry in powder form was used to develop the application. With a method requiring approxima
of examination per sample, calibration curves are presented with linear regression coefficients
and stability better than 3.0%.

# Rapid Metal / Alloy Sorting

7075 Aluminum



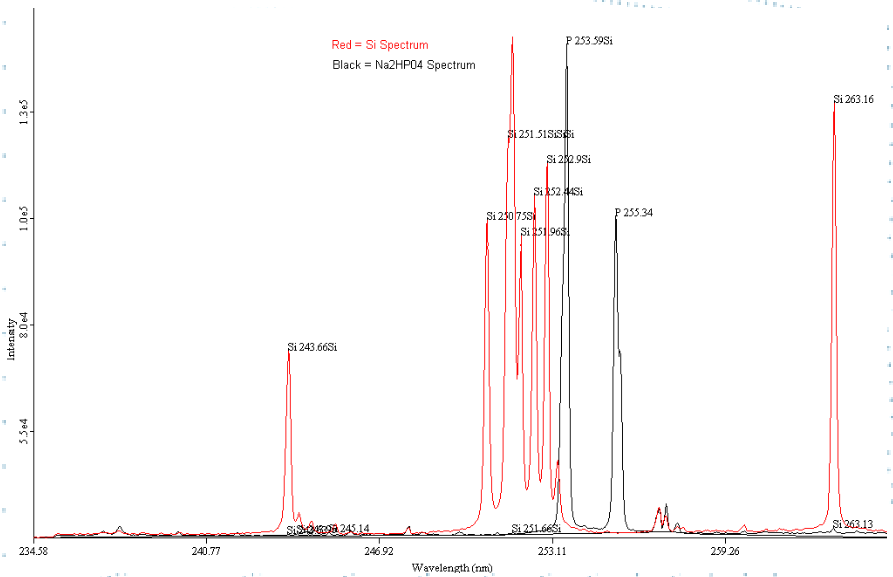

5182 Aluminum



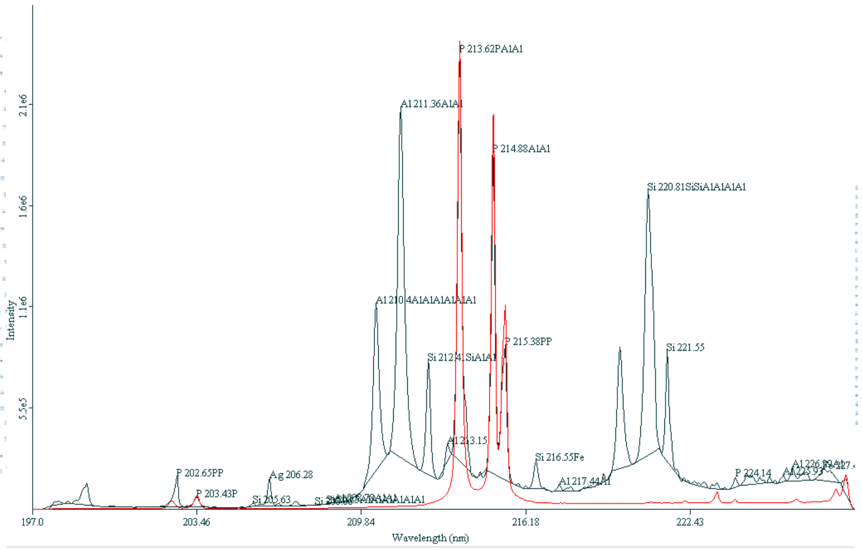

**Highly reliable and fast results**

**Spectra obtained and compared in 100 msec**

# Rapid Ore Sorting - Beneficiation



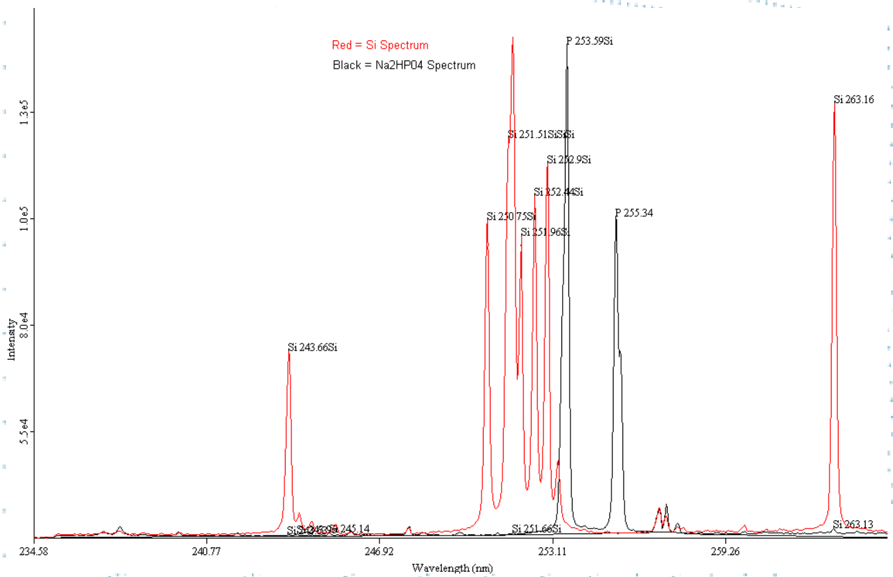

Method Development

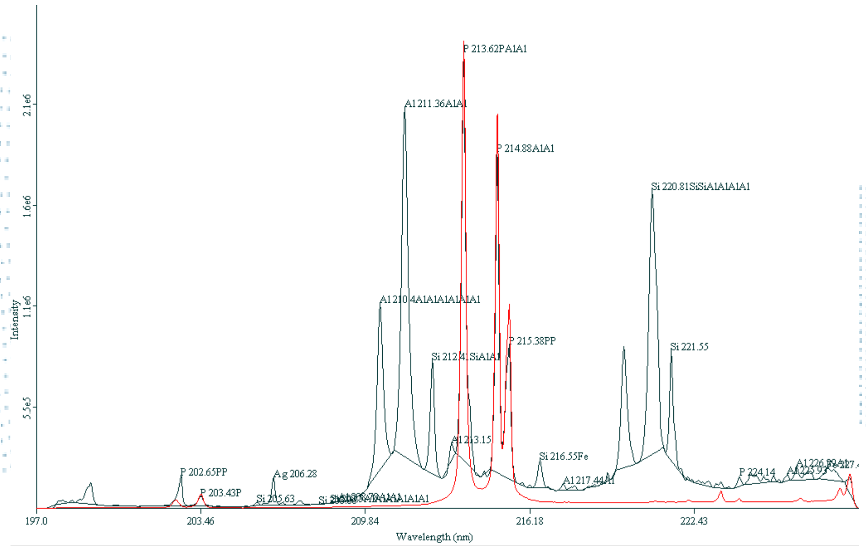



Calibration with SRM

**Spectra ratio identifies high Si content with one shot**

# Rapid Ore Sorting - Beneficiation



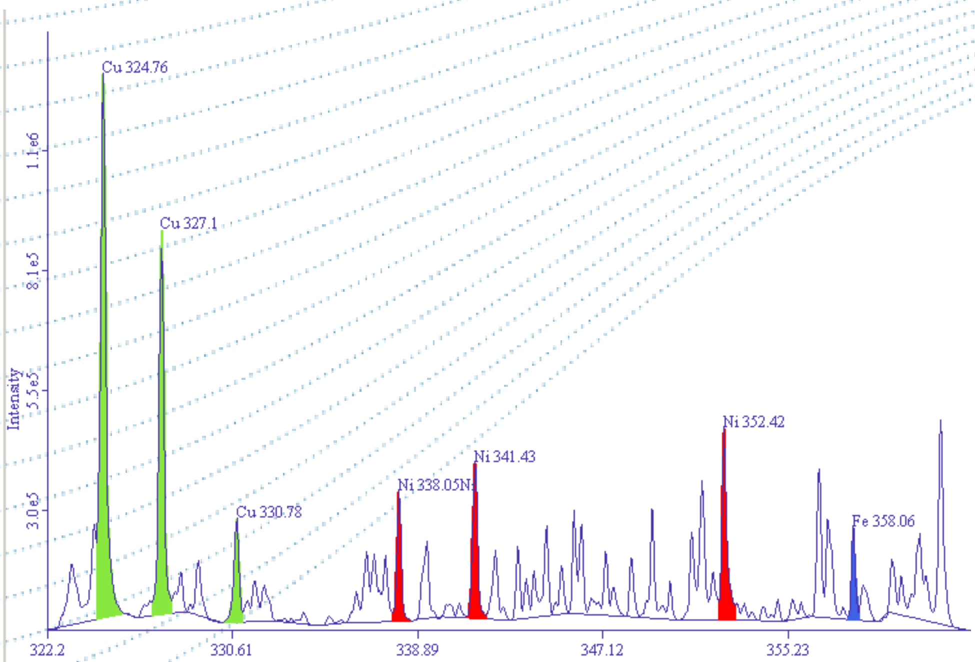

Method Development

Calibration with SRM

**Spectra ratio identifies high Si content with one shot**

# LIBS for Plated Layer Thickness Identification & Measurement

## Single spectral window (one shot) identifies all elements of interest




### Rapid, automated measurement of layer thicknesses on steel coin blanks using laser-induced-breakdown spectroscopy depth profiling


George Asimellis, Aggelos Giannoudakos, and Michael Kompitsas



We report application of a near-real-time method to determine layer thickness on electroplated coin blanks. The method was developed on a simple laser-induced-breakdown spectroscopy (LIBS) arrangement by monitoring relative emission-line intensities from key probe elements via successive laser ablation shots. This is a unique LIBS application where no other current spectroscopic method (inductively coupled plasma or x-ray fluorescence) can be applied effectively. Method development is discussed, and results with precalibrated coins are presented. © 2007 Optical Society of America

*OCIS codes:* 120.0120, 120.6200, 140.3440, 300.6360, 350.3390.


**1. Introduction**

The Royal Canadian Mint (RCM) plating facility in Winnipeg, Canada, has identified a problem with performing quality control of their electroplated coin blanks. Typically, coins, of a ferritic steel core, are electroplated with three layers, two layers of approximately 4–8 μm thick nickel, separated with a middle layer of 7–10 μm thick copper. A rapid and automated technique was sought to perform near-real-time quality assurance on production coin blanks to replace current methods of coin sectioning and subsequent optical metallographic measurements, because these mechanical methods are slow and labor intensive.

Laser-induced-breakdown spectroscopy (LIBS) enables multielemental identification and quantitative analysis requiring little or no sample preparation. A schematic of LIBS arrangement is shown in Fig. 1. A high-power laser pulse is focused just above or below the sample surface, creating a localized area of material removal (less than 1 mm wide and submicrometer deep per shot). Particles from the ablated material are subsequently ionized, and thus a hot plasma is created. Upon plasma cooling, ions recombine, and consequent excited atoms relax, radiating atomic emission lines, characteristic of the ablated sample constituent elements. By spectrally examining the optical emission, it is possible to identify the ablated area's atomic composition. This is achieved by a high-resolution spectrograph, on which the optical plasma radiation is directed, by means of fused-silica optics and steering mirrors. The spectrum is recorded on a gated, intensified CCD detector. Thus specific quantitative element analysis can be accomplished in near real time.[1–3]

This work focuses on the applicability of LIBS for rapid identification of plated layer thickness. The elemental composition of the ablated layer can be analyzed for every successive shot as the laser penetrates deeper into the material at a repeatable rate. Thus the number of shots required to penetrate a specific layer can be determined by monitoring the appearance of new probe elemental lines or the sharp decline or increase in the line intensities (photon count) of existing elemental lines. Thus LIBS can provide detailed knowledge of the layered depth profile on a very localized sample area, less than 1 mm wide.[4–13] It is this characteristic depth-profiling capability of LIBS that provided motivation for this work.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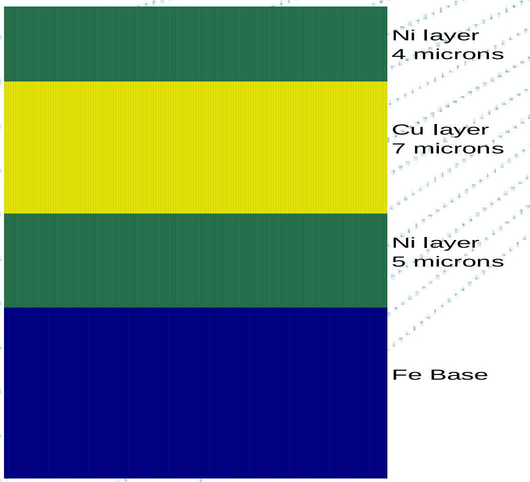
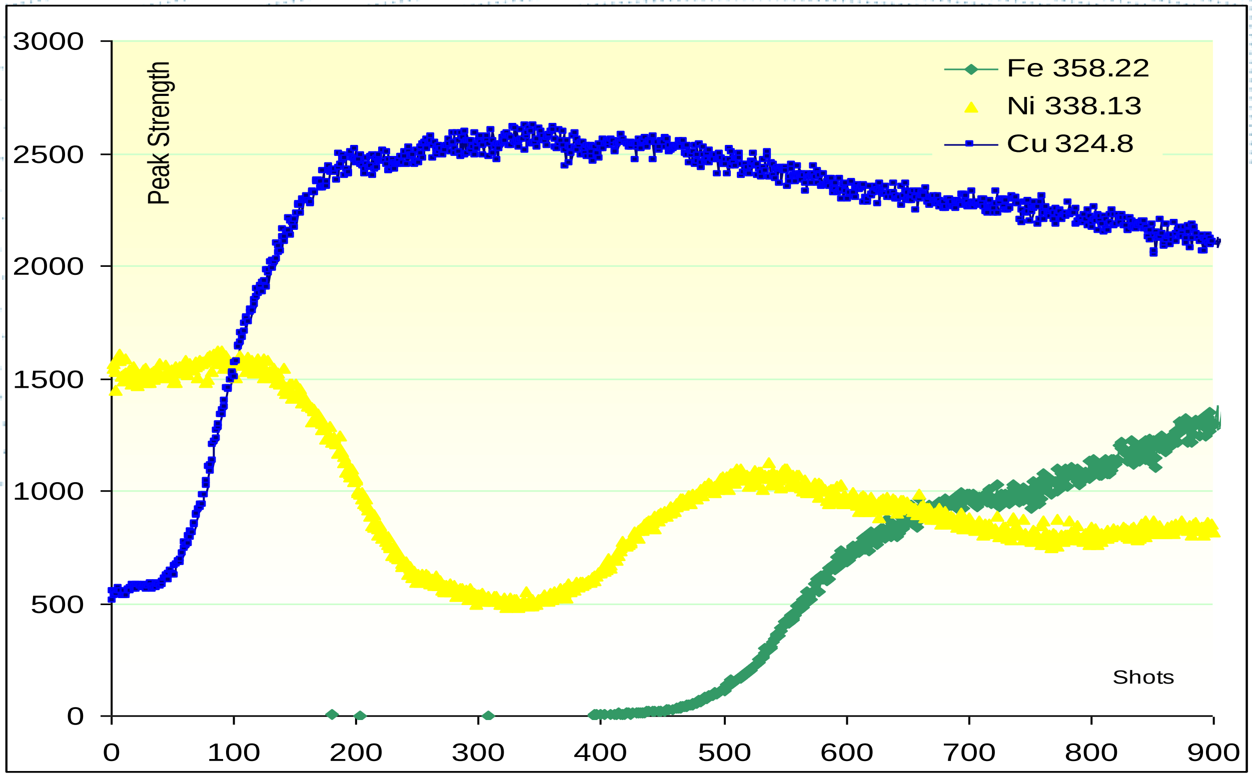
0003-6935/07/060935-08$15.00/0

# LIBS for Plated Layer Thickness Identification & Measurement

**Successive Ablation Shots Penetrate through layers.**

**When interface is reached, change in amplitude and/ or slope is observed.**



# HALOGEN DETECTION

ELSEVIER

Spectrochimica Acta Part B 60 (2005) 1132 – 1139

SPECTROCHIMICA ACTA PART B

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## Controlled inert gas environment for enhanced chlorine and fluorine detection in the visible and near-infrared by laser-induced breakdown spectroscopy☆

George Asimellis [a,*], Stephen Hamilton [b], Aggelos Giannoudakos [c], Michael Kompitsas [d]

[a] Aristotle University of Thessaloniki, Department of Physics, Solid State Section, 313-1, GR 54124 Thessaloniki, Greece
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Received 30 November 2005; accepted 30 May 2005
Available online 23 June 2005

**Abstract**

Efficient quantitative detection for halogens is necessary in a wide range of applications, ranging from pharmaceutical products to air polluting hazardous gases or organic compounds used as chemical weapons. Detection of the non-metallic elements such as fluorine (F) and chlorine (Cl) presents particular difficulty, because strong emission lines originating from their resonance states lie in the VUV spectral range (110–190 nm). In the present work we detect F and Cl in the upper visible and in the near IR (650–850 nm) under controlled inert gas ambient atmosphere. Investigation of the controlled atmosphere effects suggests that there exists an optimum pressure range that optimizes signal strength and quality. Ablation and ionization were achieved with a UV laser at 355 nm, and a gated GaAs photocathode-based detector was used for detection with quantum efficiency in the range of 20% in the wavelengths of interest. Our results indicate that our approach provides quantitative detection with linearity over at least two orders of magnitude that is achieved without the need for Internal Standardization Method, and improved limits of detection. In particular, fluorine has been detected for concentration values down to 0.03 wt.% Definite spectral assignment revealing all major emission lines centered around 837 nm for F and 687 nm for Cl has been obtained for the first time in Laser-induced breakdown spectroscopy application.


Anal Bioanal Chem (2006) 385: 333–337
DOI 10.1007/s00216-006-0345-1

SPECIAL ISSUE PAPER

George Asimellis · Aggelos Giannoudakos · Michael Kompitsas

## New near-infrared LIBS detection technique for sulfur

Received: 11 January 2006 / Revised: 3 February 2006 / Accepted: 3 February 2006 / Published online: 11 March 2006


**Abstract** Sulfur has been detected in a spectral window (around 868 nm) previously unexplored by laser-induced breakdown spectrometry (LIBS), using an ablation laser with an ultraviolet wavelength, a gated detector, and inert ambient gas at a low, controlled pressure. This spectral window enables new-generation gated iCCD cameras to be used, which have adequate quantum efficiencies up to 900 nm. Application of our technique can substantially improve signal strength and thus extends the ability of LIBS to detect many nonmetallic elements.

**Keywords** Halogens · Ambient gas · Plasma · Sulfur

### Introduction

products [10], classification of minerals and drill core samples in oil exploration [11], on-line analysis in steel processing [7], detection of air-polluting hazardous gases [12] and aqueous solutions [13], determination of organic compounds in consumer electronics [5,14] and chemical weapons in non-standard environments [8, 15, 16].

The fact that nonmetallic elements have not been thoroughly investigated by LIBS can be attributed to a number of issues related to their energy level distributions: their strongest emission lines lie in the vacuum ultraviolet (VUV) range (125–190 nm). Practical application of LIBS in this VUV range is complicated by strong absorption from atmospheric oxygen (which necessitates that the light travels through a vacuum, or at least an oxygen-free atmosphere), the lack of gated (iCCD) detectors with adequate sensitivity, and the need to use non-UV-absorbing

ELSEVIER

Spectrochimica Acta Part B 61 (2006) 1270 – 1278

SPECTROCHIMICA ACTA PART B

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## Near-IR bromine Laser Induced Breakdown Spectroscopy detection and ambient gas effects on emission line asymmetric Stark broadening and shift

George Asimellis [a,*], Aggelos Giannoudakos [b,c], Michael Kompitsas [c]

[a] Aristotle University of Thessaloniki, Faculty of Physics, Thessaloniki 54124, Greece
[b] School of Chemical Engineering, National Technical University of Athens, 9, Iroon Polytechniou Street, Zografou, 15780 Athens, Greece
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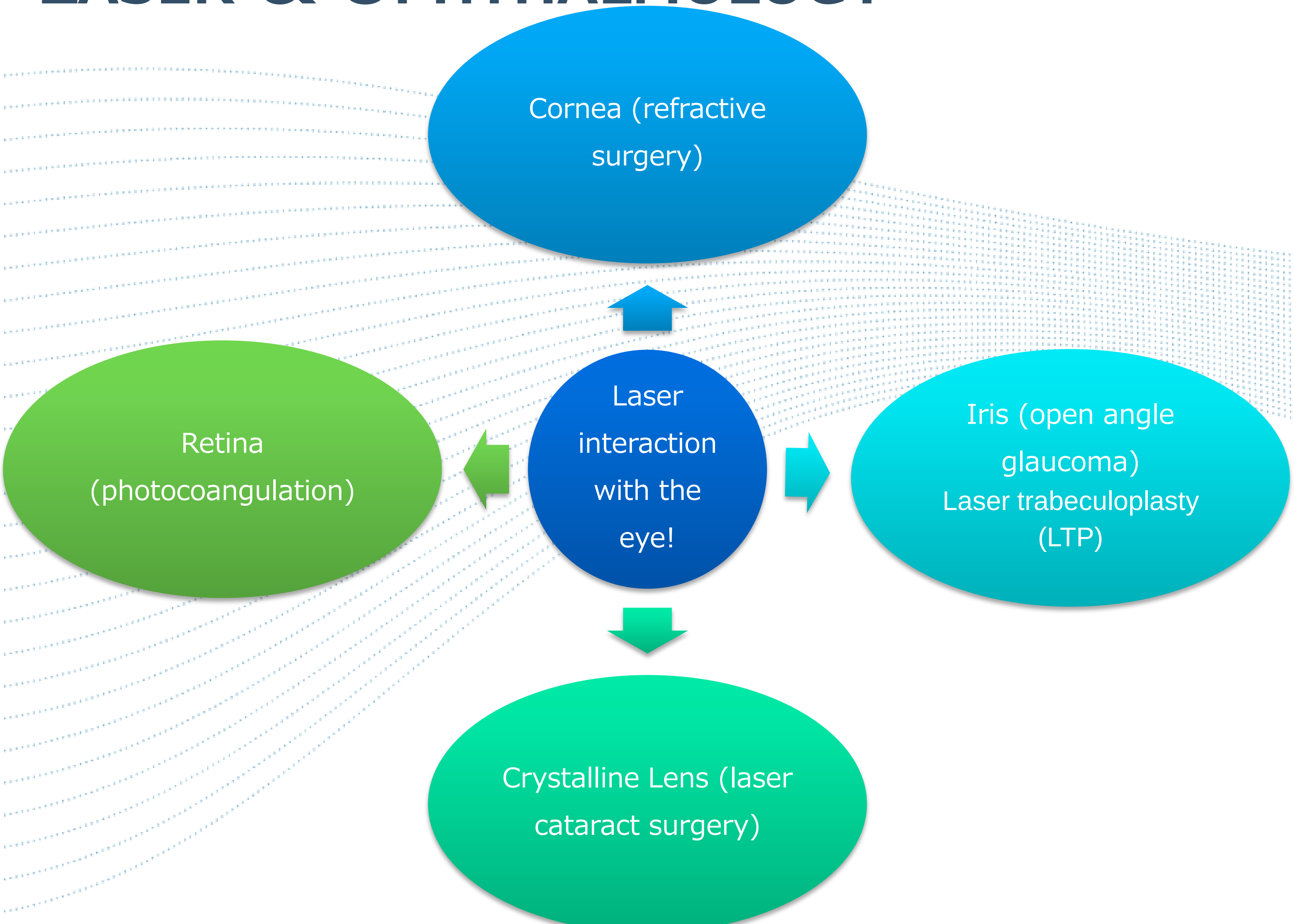
Received 14 May 2006; accepted 22 October 2006

**Abstract**

Investigation via Laser-Induced Breakdown Spectroscopy of a near-infrared bromine emission line (827.2 nm) with an UltraViolet ablation laser and a gated detector is reported. The effects of ambient pressure and gas species (air, $O_2$, $N_2$ and He) on the atomic emission line strength and spectral profile were systematically investigated. Substantially improved signal strength and reduced background radiation are demonstrated near 100 mbar ambient pressure with all gases. Optimal results were achieved when helium was used. Asymmetric broadening and shift of the 827.2 nm bromine line, attributed to pressure-dependent Stark effect has been revealed. This effect is prominent when air, oxygen or nitrogen are present and is much less manifested when helium is used. Possible interpretations of this effect are presented.


*Keywords:* Laser-induced breakdown spectroscopy; Halogen detection; Bromine content; Stark effect; Ambient gas effects

# Laser & Ophthalmology

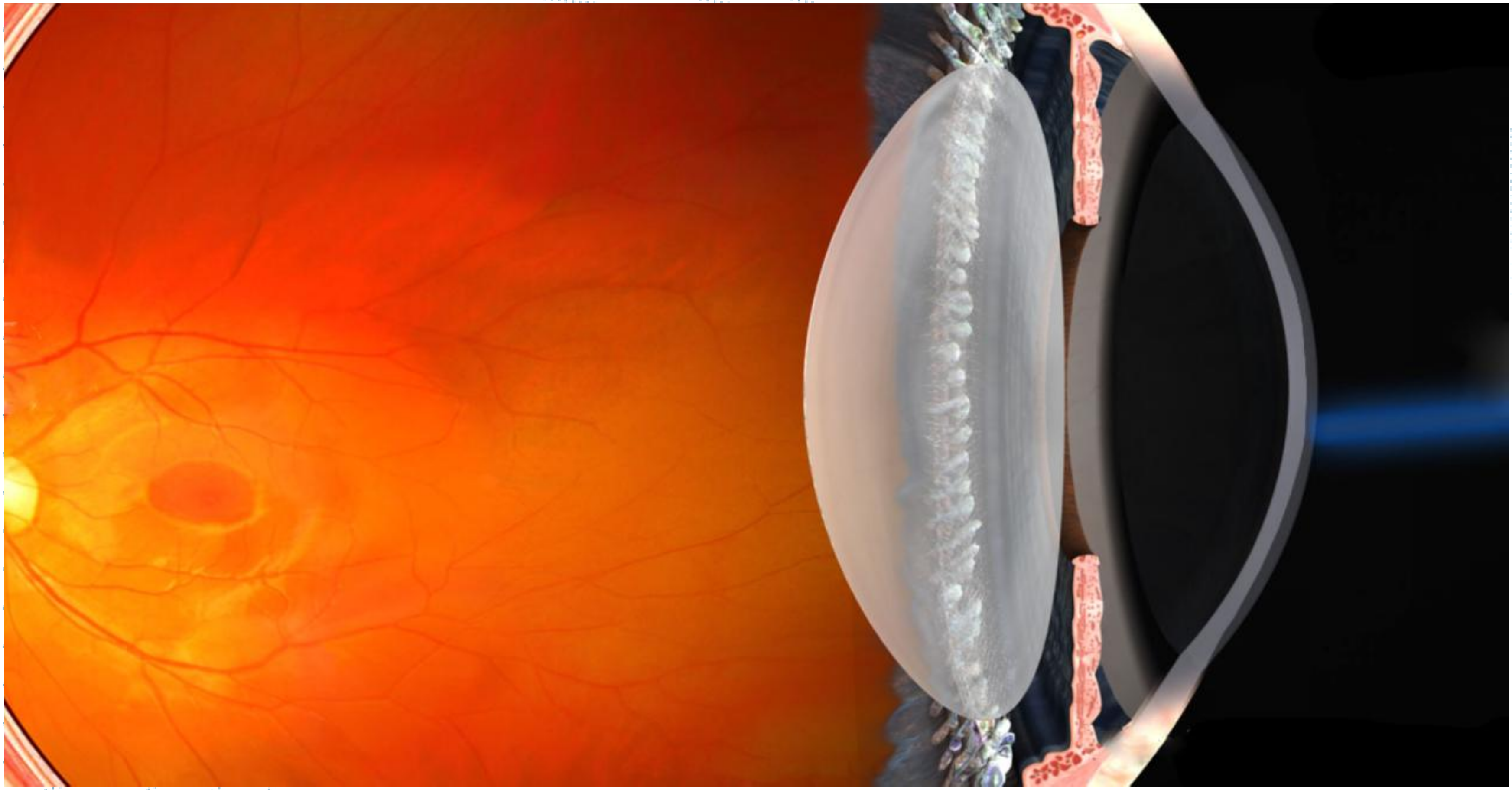


# Ophthalmic Lasers

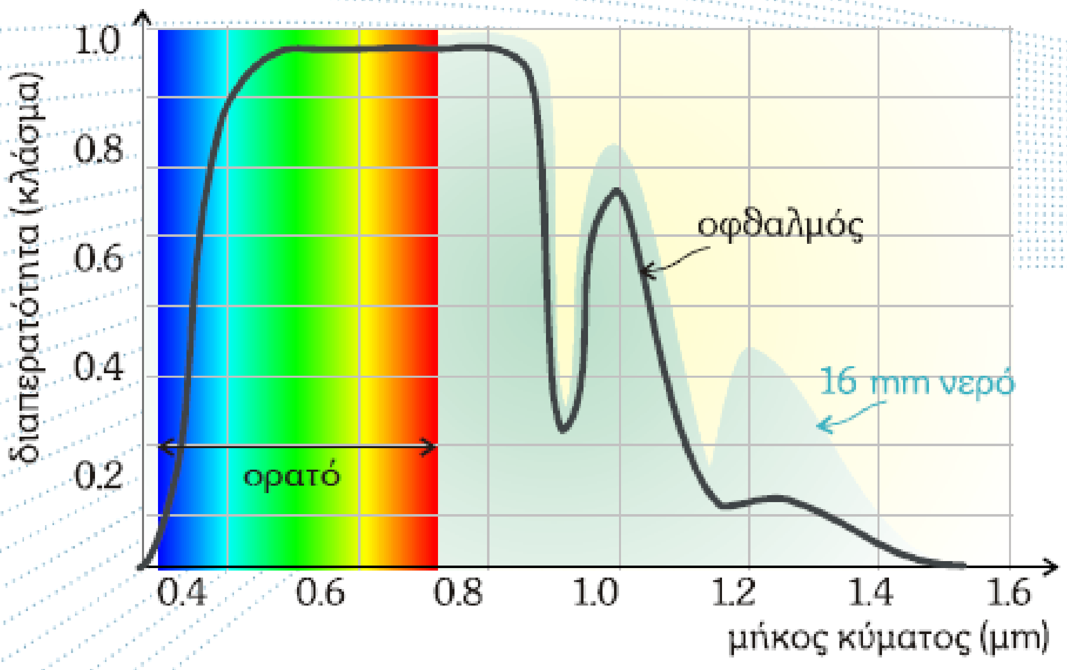
Ophthalmic lasers provide good examples of three fundamental laser applications, based on:

- **total output energy** (thermal effects, photocoagulation)
- **output power** (tissue ionization, photodisruption)
- **photon energy** (breaking molecular bonds, photoablation)
- spectral transmission properties of the eye

# Laser Within the Eye!

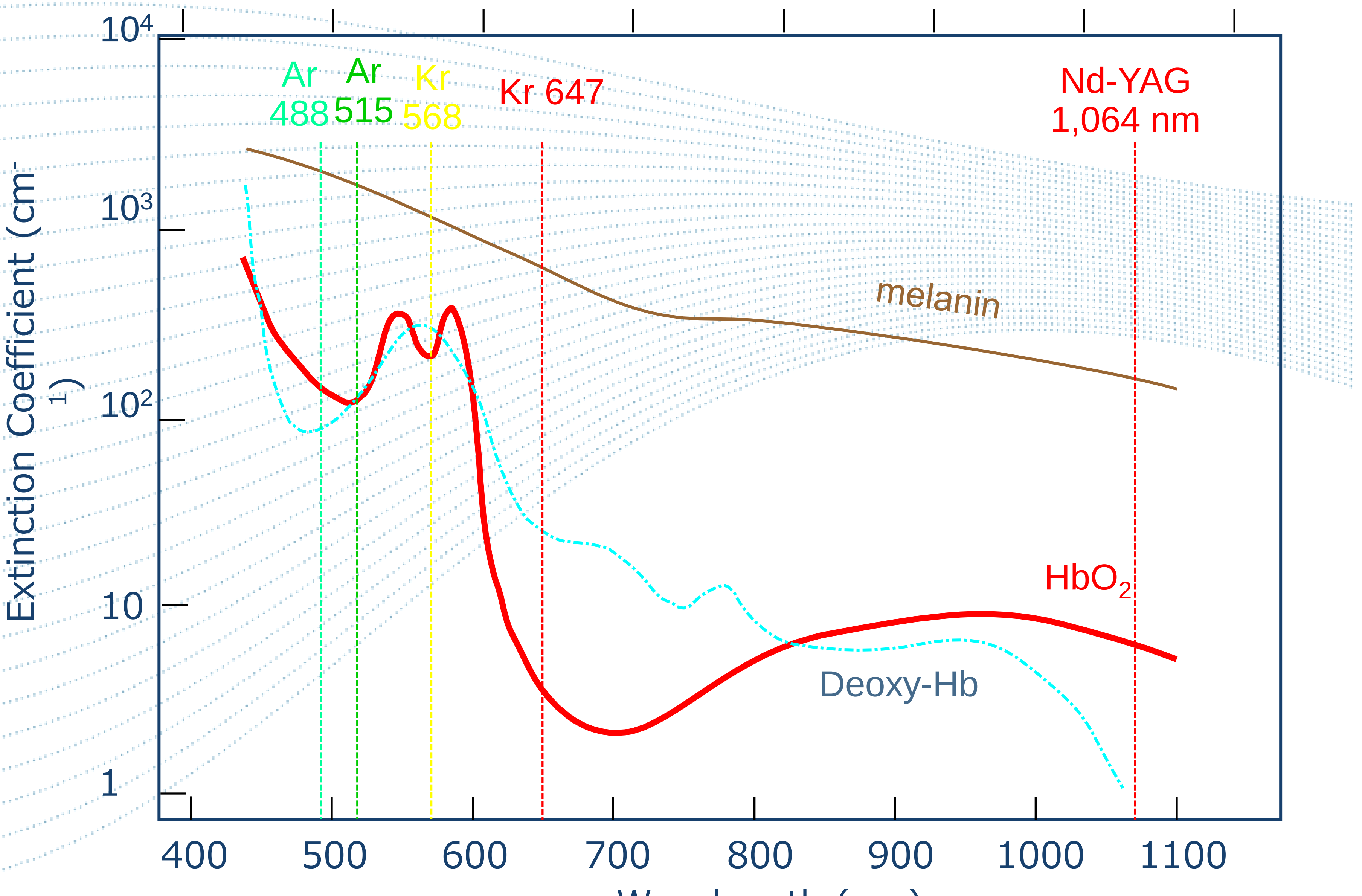


# Absorption by Ocular Media




Σχήμα 5-3-15: Φασματική διαπερατότητα του οφθαλμού.

# Absorption Spectra of Important Ocular Chromophores



# PHOTODISRUPTION

A Q-switched Nd-YAG laser often used:

causes photodisruption mainly by focal heating thermionic emission (from linearly absorbing chromophores)

Higher power mode-locked Nd-YAG laser (20-30 psec pulses):

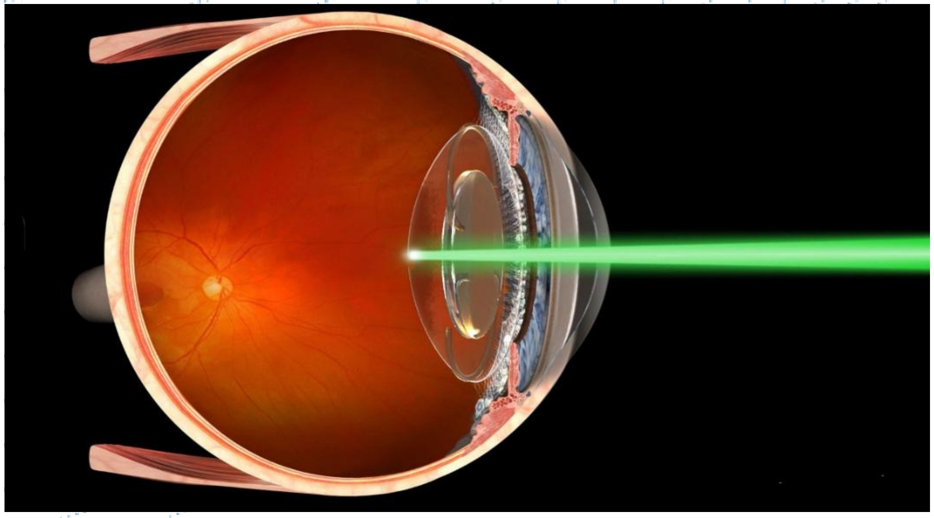
ionizes by multiphoton absorption

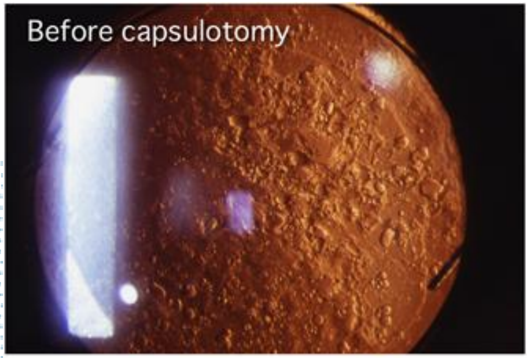
each 1,064 nm photon carries 1.17 eV of energy

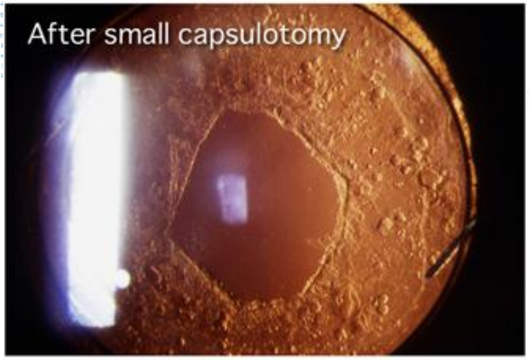
because atoms typically must absorb > 10 eV to ionize need “multi”-photon absorption for ionization

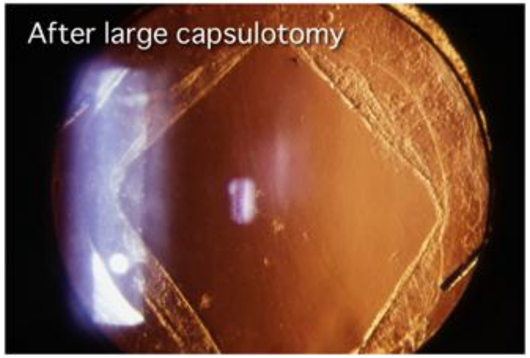
# Posterior Capsulotomy

to open up an opacified lens capsule resulting from prior extracapsular cataract surgery



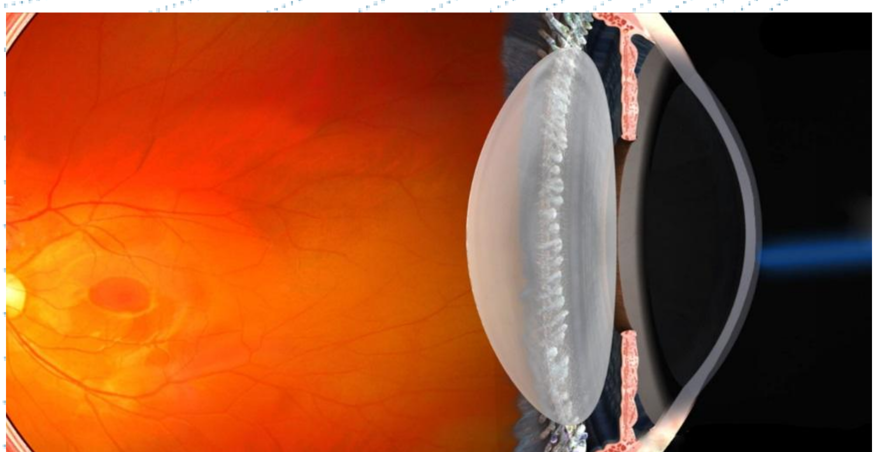



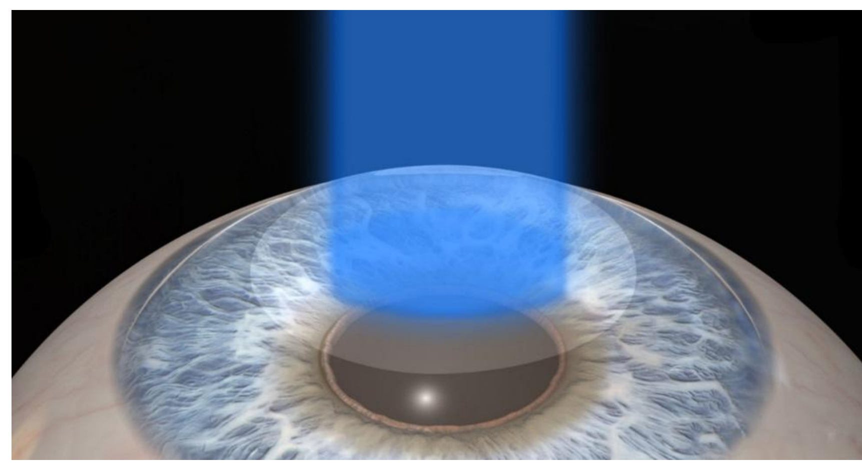
# PhotoAblation by Excimer Laser

Removal of corneal tissue by high energy photon directly breaking molecular bonds with sufficient photon energy,

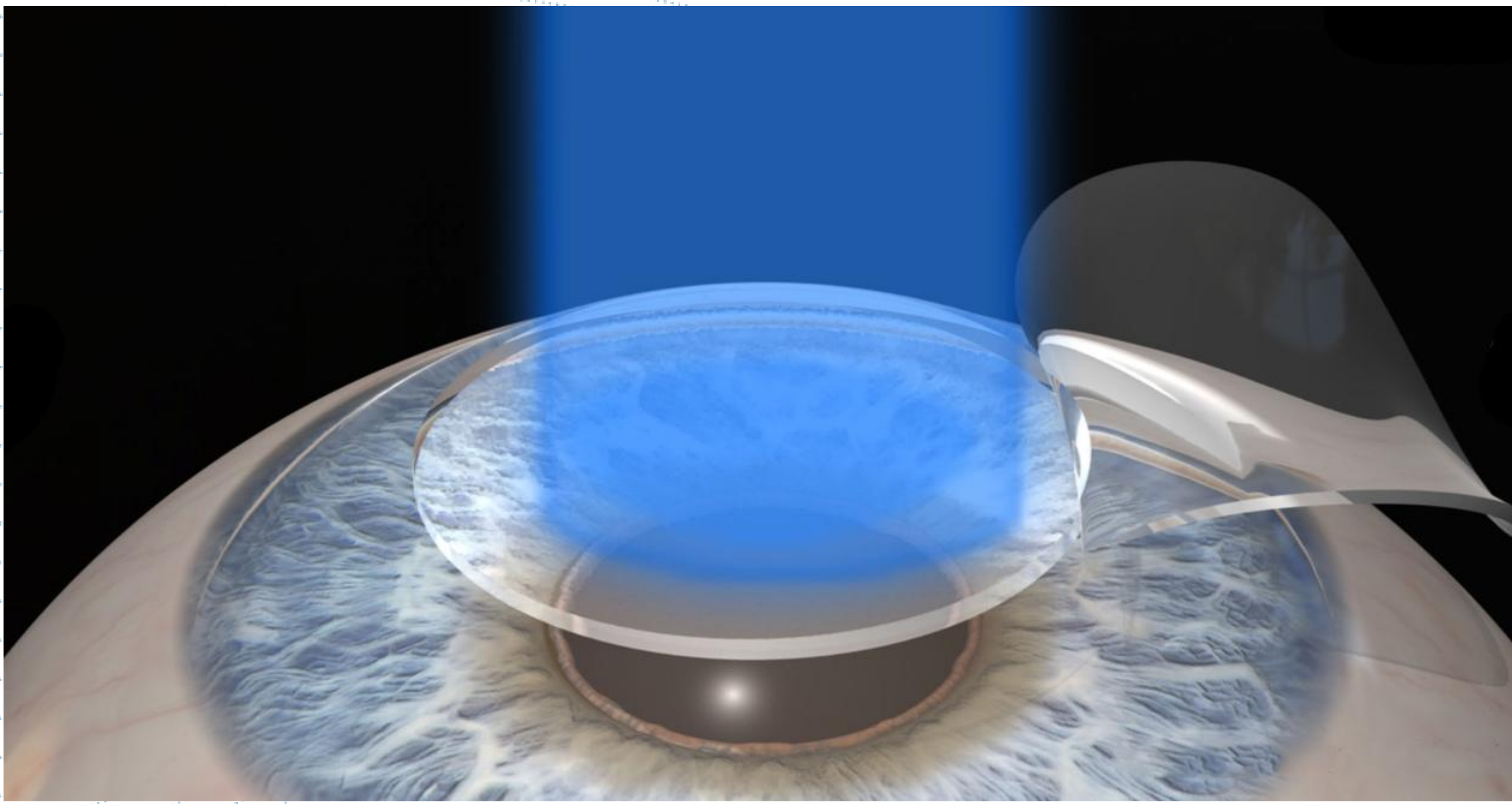
193 nm, 6.4 eV energy

Excimer laser

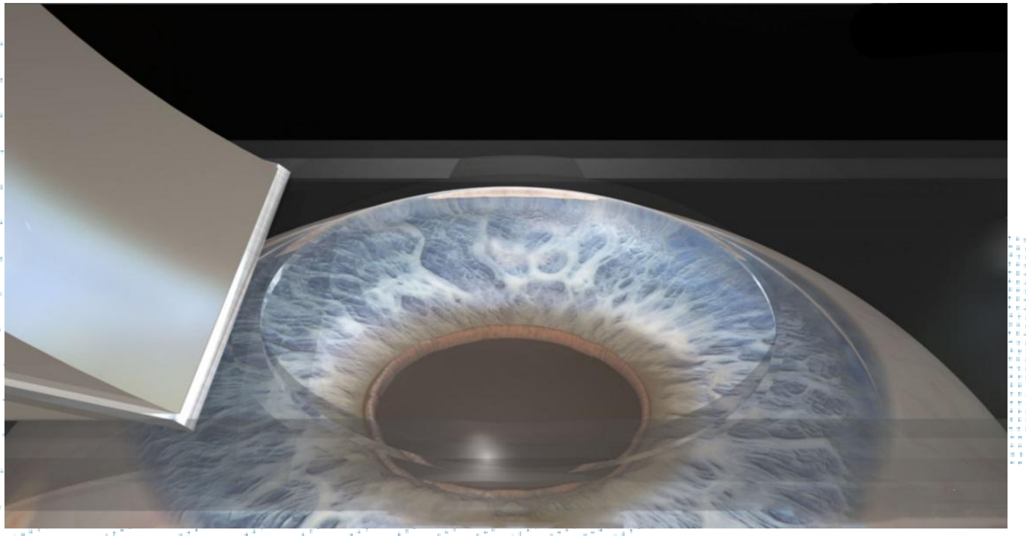


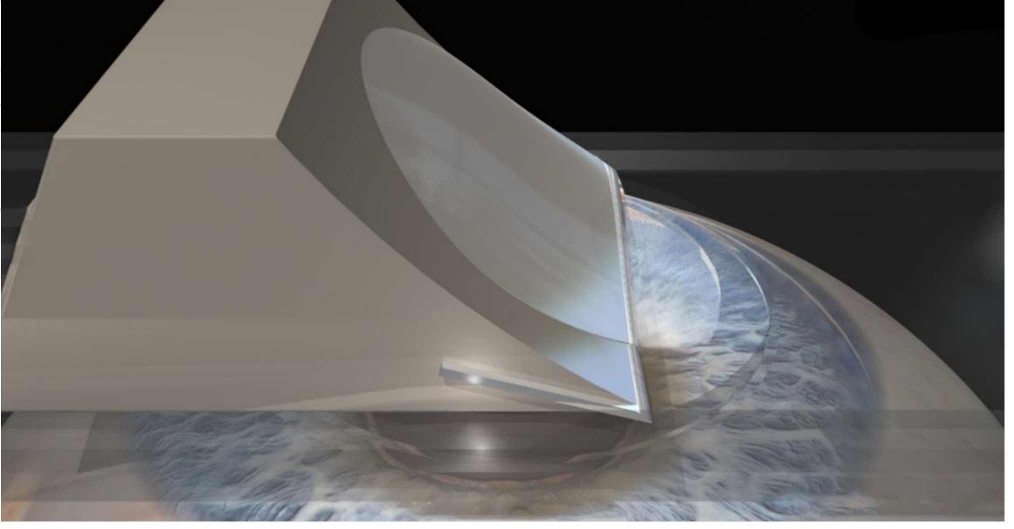


# The LASIK Procedure

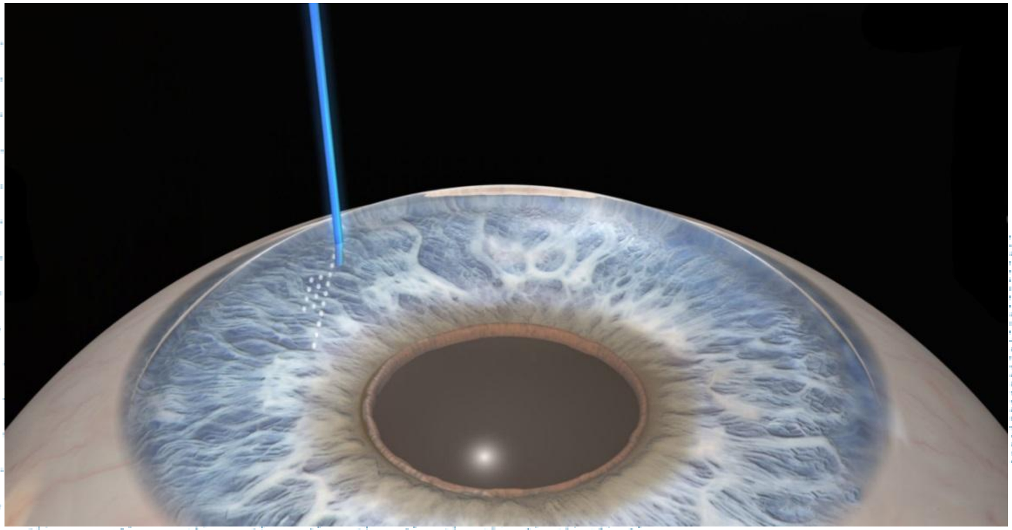


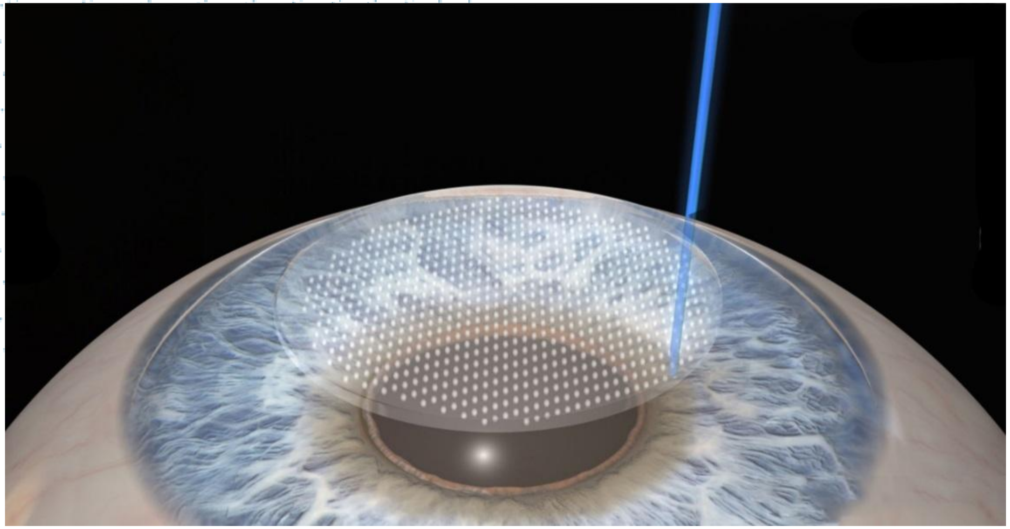
# Creating a 'Flap' Mechanically



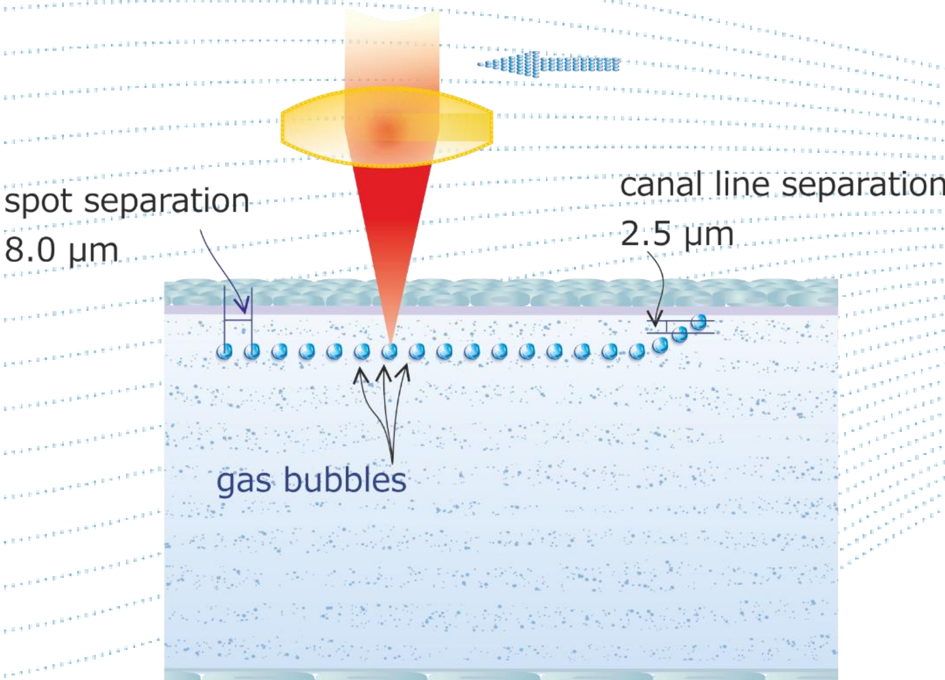


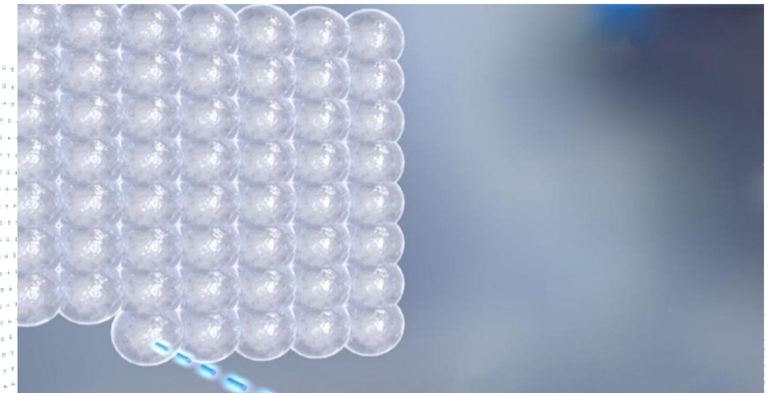
# Creating a 'Flap' with a LASER



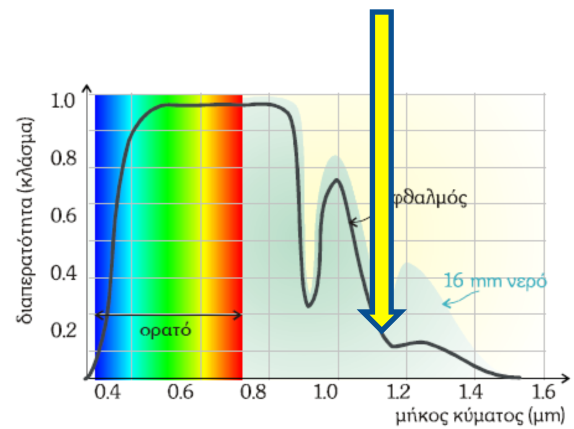


# Flap Creation by MicroAblations



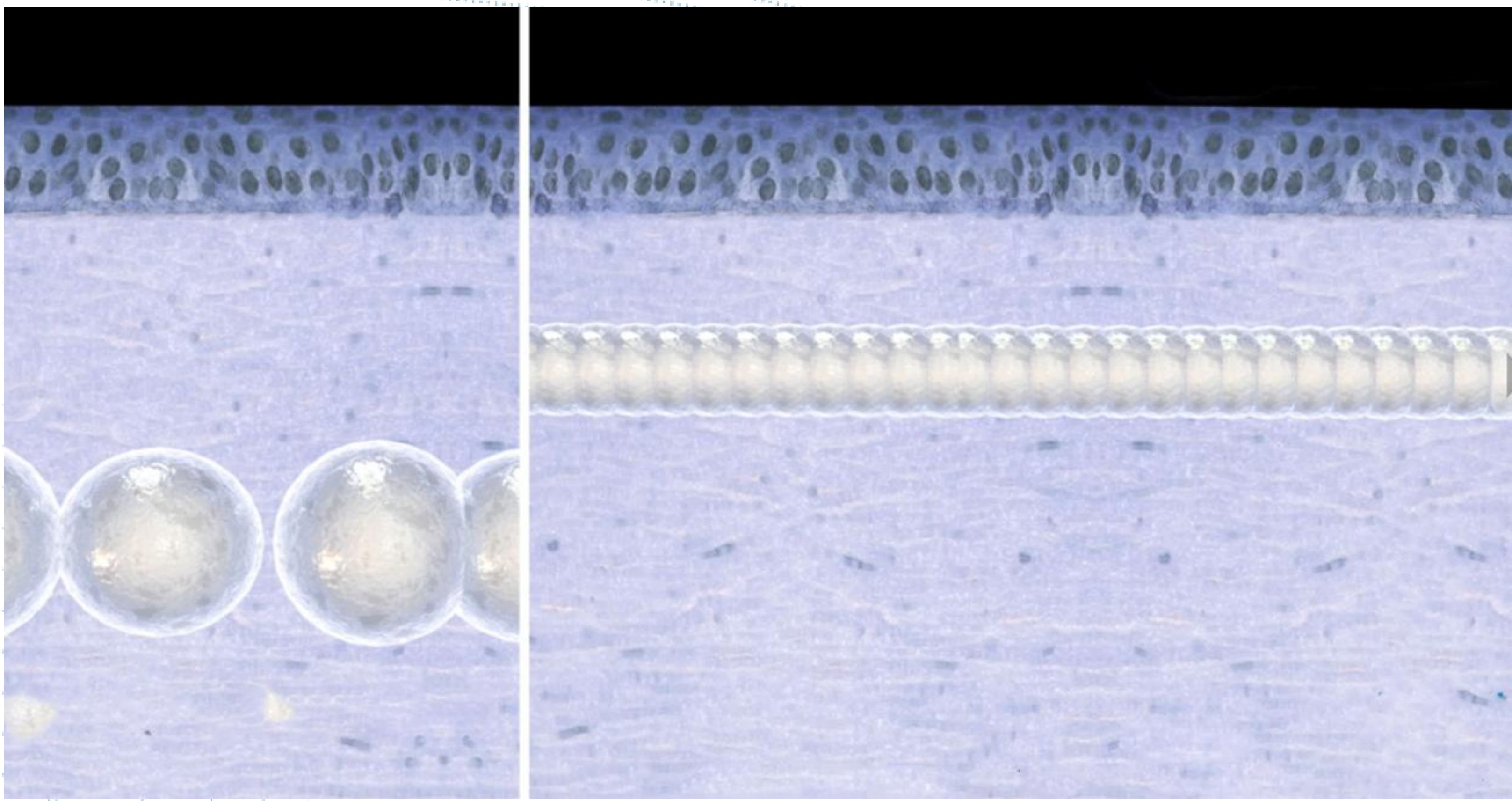
Needs different wavelength!




Σχήμα 5-3-15: Φασματική διαπερατότητα του οφθαλμού.

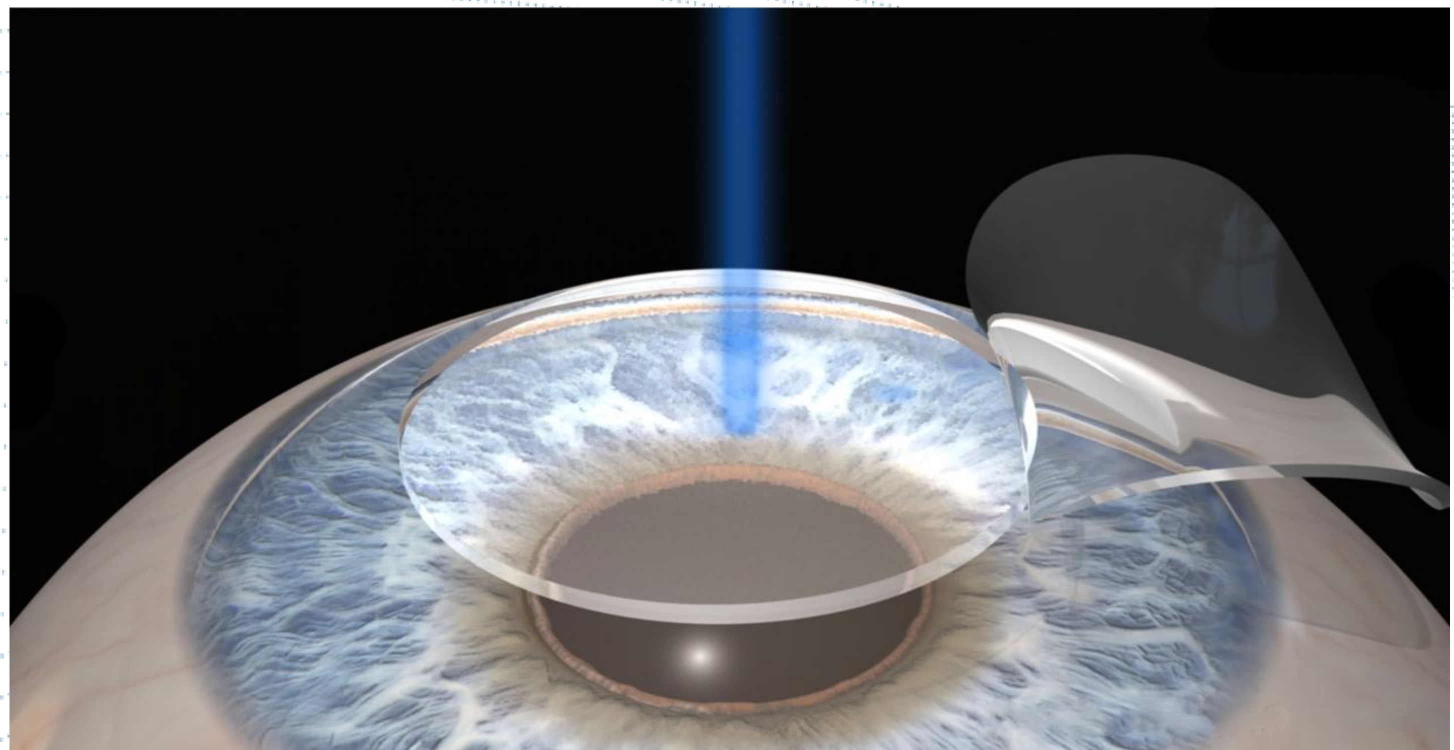
Nd:YAG pulsed laser
Pulse duration: few $10^{-15}$ sec
Femto-second laser

# Femto-Second Ablation Within the Cornea

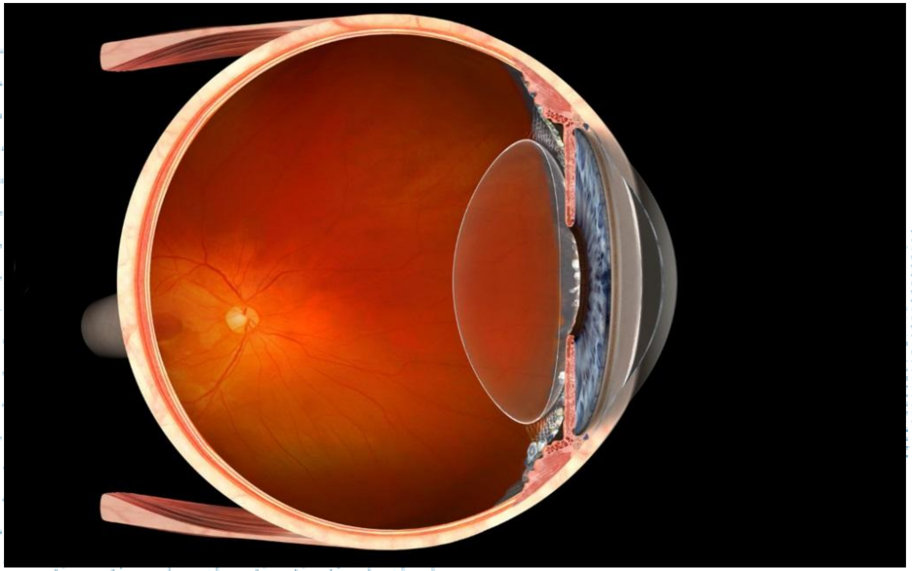


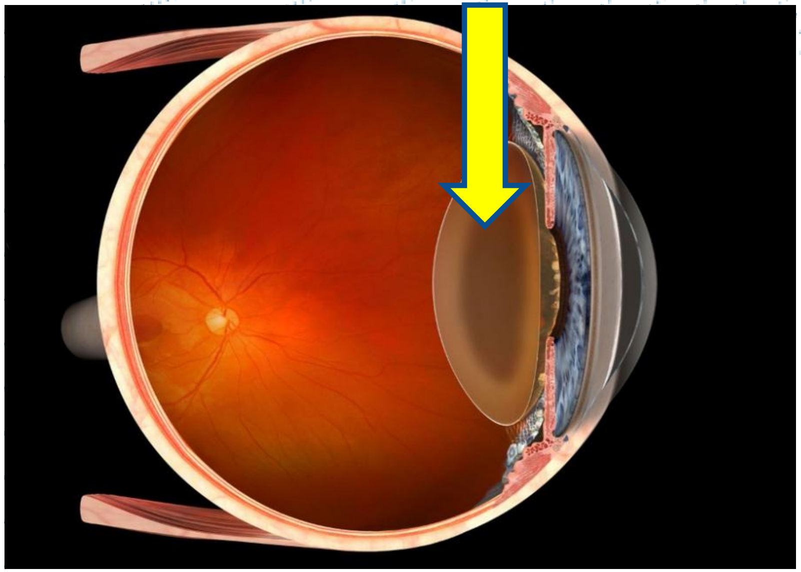
# Ablation Within the Cornea

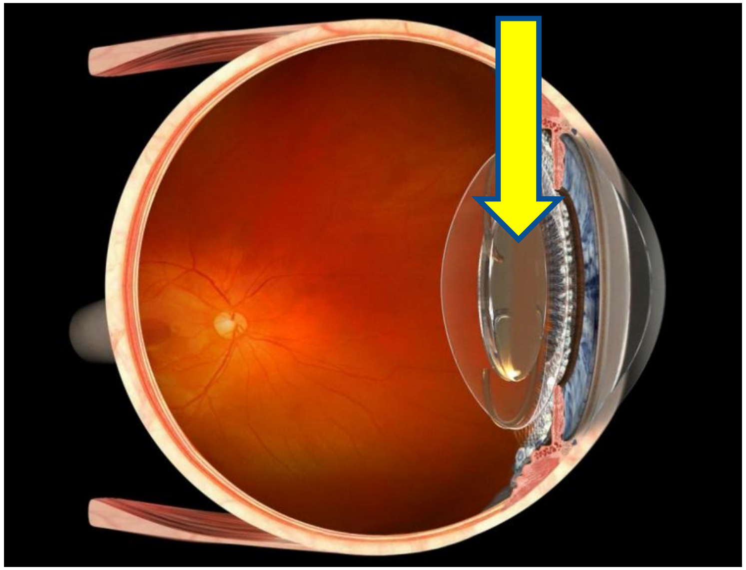
# Lifting of the Flap, Excimer Ablation



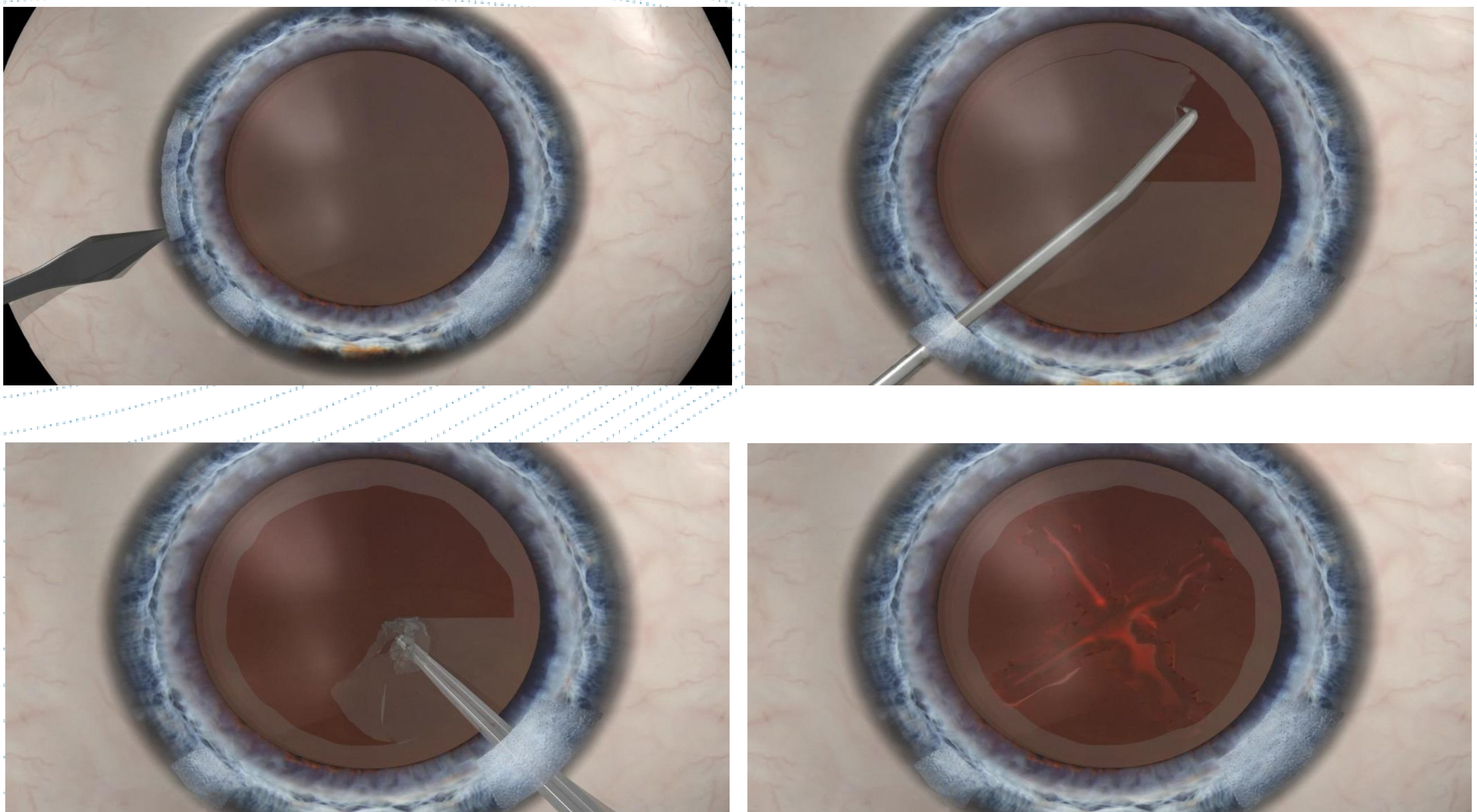
# Cataract Removal: The Most Common Ophthalmic Surgery







# Cataract Surgery



# Cataract Surgery with FemtoSecond Laser