

Laser-Induced Breakdown Spectroscopy

LIBS ANALYSIS OF METAL SURFACES

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INTRODUCTION

Two increasingly popular areas of research in environmental science are the chemistry at surfaces (surface composition, reactivity and contamination) as well as remote sensing to determine concentrations of species at a distance or in hard-to-reach or hazardous environments. Laser-Induced Breakdown Spectroscopy (LIBS) is a rapidly growing technique used in both of these areas.

LIBS uses a powerful, pulsed laser to both prepare the sample by ablation of the surface and create the plasma where analysis of the species formed occurs. The laser pulse delivers enough energy to not only vaporize a small fraction of the surface of the sample but also to induce electronic excitation of the atoms and ions in the resulting plume of rapidly expanding vaporized material. Upon relaxation of the excited electrons, energy is released in the form of electromagnetic radiation that is detected by a spectrophotometer. Each element has a unique line spectrum of electron energies that act as a “fingerprint”, allowing qualitative and quantitative determination of the elemental surface composition.

This technique has been successfully applied in studies of soil composition, aerosol detection and analysis in the atmosphere, steel and coal analysis, corrosion in nuclear reactors, surface contamination, and recently has branched into analysis of biological materials (the popular laser eye surgery procedure being an example).¹

This experiment acts as a first-hand experience with laser operation and introduces the LIBS technique in the surface analysis of several common metals. The main goals of this lab are:

- Learn about pulse lasers, plasmas, and emission spectroscopy
- Identify elements in metal samples by their emission spectra from the laser induced plasma
- Measure spectra emitted by various light sources such as lamps, light emitting diodes, TV remotes, lasers, etc.

BACKGROUND

To better appreciate the LIBS technique, there are several key concepts in which a basic understanding should be developed. The **laser** is the main component of any LIBS instrument. **Plasma** is a unique state of matter that is the key to LIBS analysis. Finally, **spectroscopy** is a scientific art of interpreting colors in terms of atoms and molecules that emit them.

I: Lasers

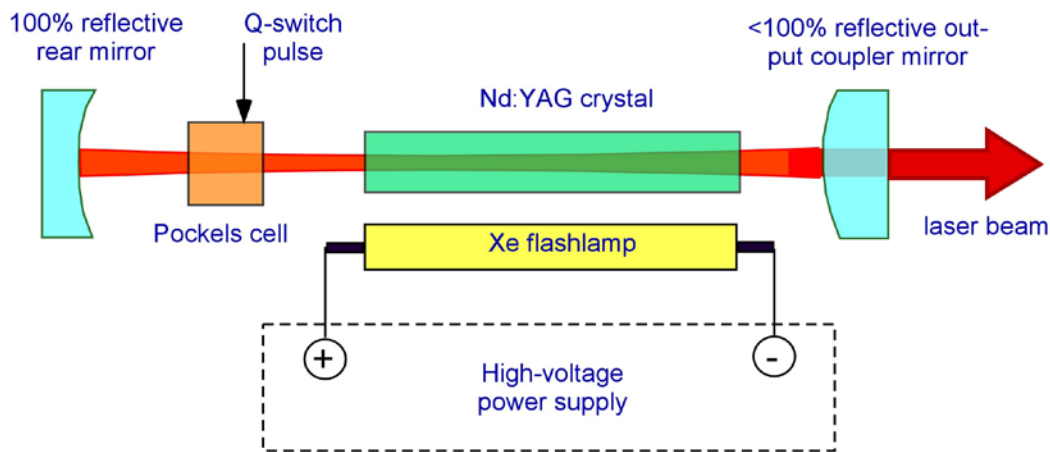
The development of lasers began in the 1960's, and has since completely revolutionized science and technology. Lasers are used in most areas of our life, from basic CD player operation to major surgical procedures. Laser is an acronym for “**L**ight **A**mplification by **S**timulated **E**mission of **R**adiation”.

Typically, a laser operates by exciting atoms in a lasing medium using a bright flash of light from a flash lamp (see Figure 1 on the next page). A *population inversion* is created in which there are more atoms in an excited higher energy state than the lower energy ground state. This

lasing medium is placed in a cavity that is capped by two aligned mirrors. The mirror on one side is 100% reflective, and the other allows some radiation to escape. When an atom loses its excess energy and falls back to the ground state, a photon of electromagnetic radiation (“light”) is emitted. This photon can stimulate emission of another photon from a neighboring excited atom, and so on, causing a “cascade” of photon emission. The light is then reflected back and forth by the mirrors through the lasing medium, forming an amplified, highly coherent (all photons are in phase) and monochromatic (all photons have the same wavelength) beam of radiation that exits the cavity as either a continuous beam or in pulses.

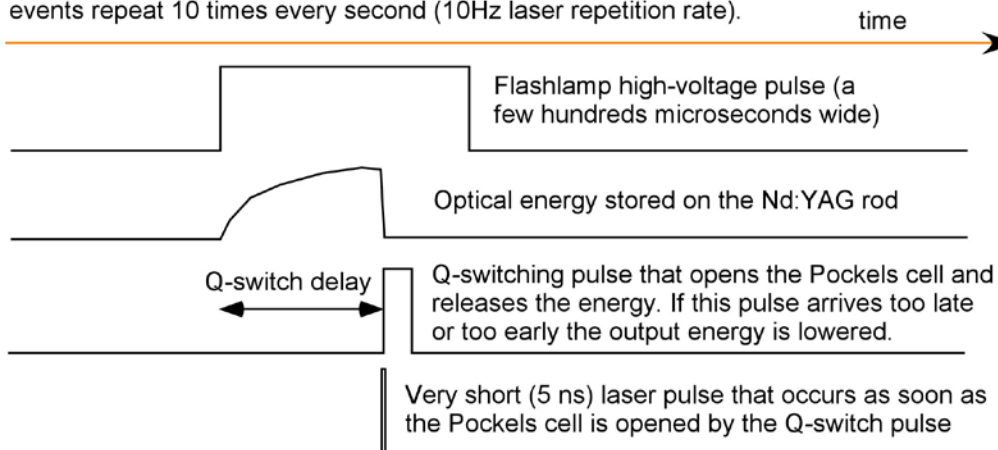
Figure 1: Basic diagram of a typical solid state Nd:YAG laser and timing sequence involved into firing of a laser pulse.

(a) Nd:YAG schematic diagram



(b) Nd:YAG timing

This timing diagram applies to a single laser pulse. All these events repeat 10 times every second (10Hz laser repetition rate).



There have been different lasing mediums used, ranging from the original Ruby crystal to gases, dye solutions, and semiconductors. The particular laser medium used in this experiment (Figure 1) is Nd:YAG crystal, which is shorthand notation for yttrium aluminum garnet doped

with neodymium. The flash lamps that deliver energy to the Nd:YAG rod are quartz tubes filled with pressurized Xenon. This laser is classified as a nanosecond (ns) pulsed laser, because it emits repetitive pulses of light, with each pulse lasting about 5 ns ($1 \text{ ns} = 1 \times 10^{-9} \text{ s}$). The laser *repetition rate*, the number of laser pulses emitted every second, is 10 Hz.

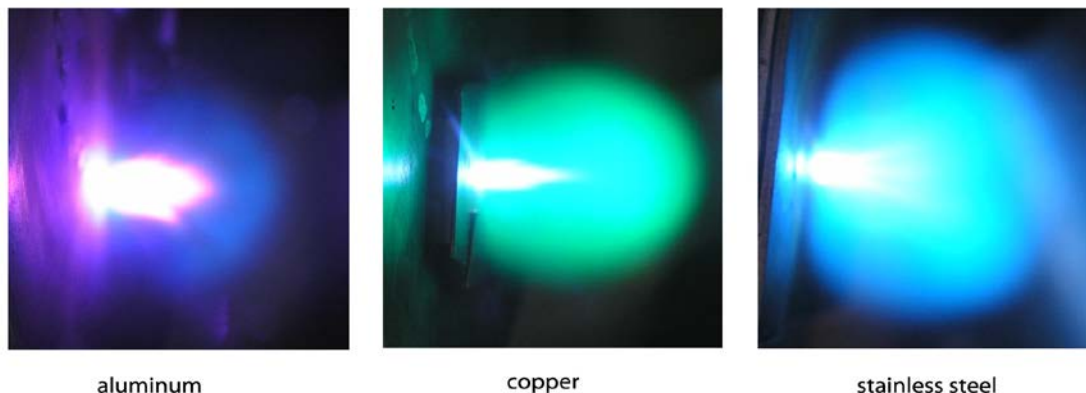
The *energy* of a pulsed laser is commonly measured in Joules (J) per pulse. The *power* is measured in Watts ($\text{W} = \text{Joule per second}$), and the *irradiance*, or the power density distributed over the laser beam area, in W/cm^2 . There are two ways to report power. The most common way is to specify the *average power*, which is total energy divided by the period of time over which the energy is delivered. Average power is calculated by averaging energy from multiple laser pulses. Sometimes it is more convenient to use *peak power*, which is the effective power of a single pulse equal to the pulse energy divided by the pulse duration.

The following example illustrates these concepts. The laser used in this experiment is capable of delivering 0.2 J per laser pulse, with a pulse duration of 5 ns. The laser emits 10 such pulses per second, thus delivering $10 \times 0.2 = 2 \text{ J}$ of energy every second. This is equivalent to average power of $2 \text{ J} / 1 \text{ s} = 2 \text{ W}$. The peak power is considerably higher because the pulse duration is so small. It is equal to pulse energy / pulse duration $= 0.2 \text{ J} / 5 \times 10^{-9} \text{ s} = 4 \times 10^7 \text{ W}$ ($= 40 \text{ megawatt}$) during the laser pulse! The laser beam is typically focused on an area that is about 0.01 cm^2 , therefore, the irradiance used in this LIBS experiments can be as high as $4 \times 10^9 \text{ W}/\text{cm}^2$, or 4 gigawatts per square centimeter! It is the large peak powers that make it possible to efficiently vaporize the sample in LIBS. Therefore, it is advantageous to use very short laser pulses to achieve high peak powers.

II: Plasmas

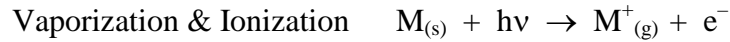
Plasma can be defined as a local assembly of atoms, positive ions, negative ions, and free electrons. Although there are charged particles present in plasma, it is normally neutral on the whole. Particles in the plasma are typically characterized by high temperature, which make the plasma glow in a color that depends on the plasma composition. Figure 2 shows typical plasmas generated in a LIBS experiment.

Figure 2: Sample plasmas induced by LIBS



The leading edge of the laser pulse rapidly heats, melts and vaporizes the surface of the solid sample into a layer just above the surface. For the irradiance values used in LIBS, the

temperatures of most plasmas created may approach 10,000 K. The vaporization and initial ionization of atoms generated can be represented by the following highly simplified equations:



where “s” stands for solid, “g” stands for gas, “M” stands for metal atom, plus denotes a positively charged atom, and e^- stands for a free electron. Further ionization can occur as the free electrons collide with other atoms in a self-accelerating process that causes gas ionization and breakdown:



The spatial and temporal characteristics of the plasma after a laser pulse can be quite complicated, especially in a vacuum environment as in our experiment. We will only qualitatively look at plasma shapes and colors.

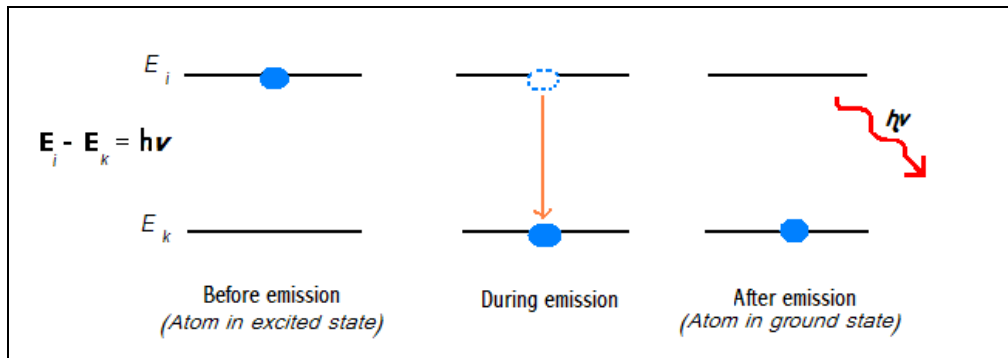
III: Spectroscopy

As stated previously, the atoms and ions of each element have unique electronic energy levels (per quantum mechanics). When atoms absorb energy that is equal to the difference between two energy levels, they become “excited” and promoted to a higher energy level. In the case of LIBS, this excitation is done by electrons, photons, and excited atoms present in the plasma.



Similar equations can be written for ions. These excited atoms or ions rapidly relax back to their lower energy states in a process called spontaneous emission, releasing energy as electromagnetic radiation of specific wavelengths (Figure 3).

Figure 3: Schematic representation of spontaneous emission: Before emission the particle is in the excited state. After relaxation of the electron, a photon is emitted and the electron returns to the ground state energy level.



A spectrophotometer measures the intensities of the photons emitted, and displays the output as an emission spectrum as a function of wavelength. The wavelength is related to the energy of electronic energy levels involved in the transition as follows (E_i and E_k are the upper and lower state energies, respectively):

$$E_i - E_k = h \nu_{ik}$$

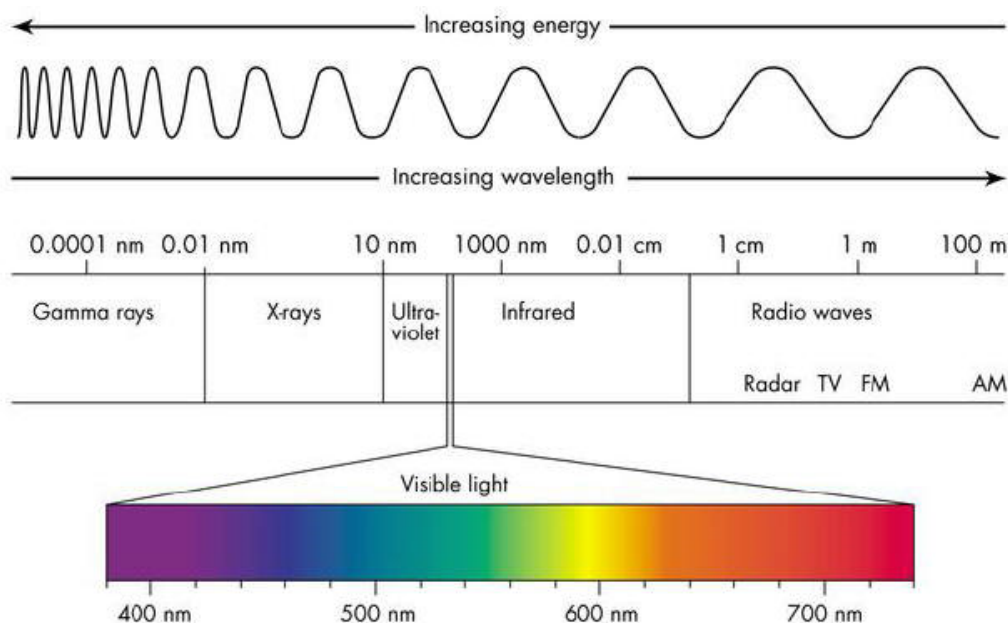
$$\lambda_{ik} = \frac{hc}{E_i - E_k}$$

With increasing energy the wavelengths get smaller and are higher in frequency. Radio waves have the largest wavelengths and are about the size of buildings. Gamma rays are the smallest and are on the order of atomic nuclei. Visible wavelengths are right in the middle and are the size of bacteria. The spectrometer will provide measurements of plasma and various light sources within the electromagnetic spectrum (Figure 4).

This spectrum can be compared to reference spectra to qualitatively identify what atoms and ions are present in the plasma. The peak intensities can also be used to quantitatively determine the amounts of each species (not done in this experiment).

With the advent of fiber optics, the spectrophotometer could be separated from the plasma via a fiber optics cable, allowing remote analysis of samples in hazardous, distant, or difficult to reach areas. A fiber optics cable will be used in this experiment to transmit light from the vacuum chamber, where plasma will be located, to a USB-powered spectrophotometer.

Figure 4: The electromagnetic spectrum. The spectrometer used in this lab is capable of resolving the colors ranging from UV (200 nm) to near-infrared (1000 nm).



SAFETY

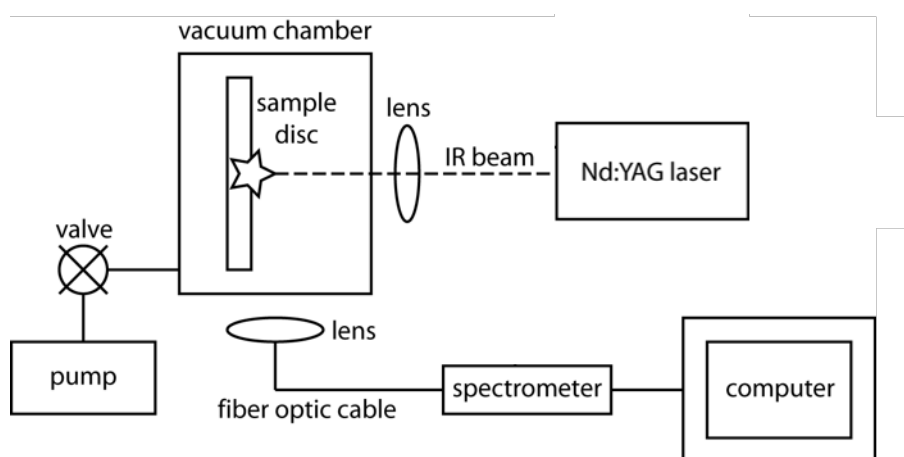
The Nd:YAG laser used in this experiment is a Class IV laser that can cause permanent vision and skin damage. **It is NOT a toy.** The infrared beam produced by the laser is **INVISIBLE**, so avoid standing in any location where you can accidentally look into the laser beam or catch the reflection of the beam off of a smooth surface. Likewise, make sure that the laser beam path is clear, and remove any reflective accessories (e.g., jewelry, watches, etc.) from your hands and arms. **Laser safety goggles should be worn when the laser is in use.** Appropriate laser goggles for this project **should absorb 1064 nm and UV, but not the visible radiation**. The green or red laser goggles next to the LIBS setup are suitable for this purpose. When you are not collecting data, reduce the laser power output by increasing the delay time between pulses or close the internal cavity shutter on the laser. Become familiar with the lab protocol and review UCI's Laser Safety Guidelines prior to starting the experiment.

EXPERIMENTAL

The experimental apparatus has three main components: the laser, the sample chamber, and the spectrometer, as shown in Figure 5. The laser generates a pulsed beam that travels through a focusing lens, strikes the sample disc in the vacuum chamber, and induces the formation of plasma. Radiation emitted by the plasma is focused onto a fiber optics cable that carries the signal to a spectrometer. The computer analyzes and displays the data collected by the spectrometer.

Note that the Nd:YAG laser used in this experiment fires in repetitive pulses. The time between pulses is fixed and equal to 0.1 seconds (inverse of the laser repetition rate). The energy of each pulse can be adjusted by changing the time delay between the flash lamps (the Q-switch), which charges the lasing medium (the longer the time delay, the lower the pulse energy). The laser cavity then electronically opens and forces the laser to emit a pulse. This delay time can be controlled by setting the *Q-Switch delay* on the laser control panel.

Figure 5: Diagram of the LIBS apparatus



Also note that this laser has the ability to create three different wavelengths by using a special attachment with a doubling and tripling crystal in it. The laser itself produces an infrared beam at 1064 nm which can be doubled with a doubling crystal to make a weaker green beam at

532 nm, or tripled with a tripling crystal to generate a UV beam around 355 nm. The rotating sample disc is centered in a vacuum chamber that is pumped by a mechanical pump ($\sim 10^{-3}$ torr pressure). Be sure that the pump is on before powering up the laser. The sample disc can be removed without turning off the pump by closing the pump valve.

The default setting for the spectrometer is to continuously record data. The spectrometer can also be configured to only collect data for a brief period of time after the laser pulse if necessary. The amount of time the spectrometer waits after the laser fires is controlled by the delay controller. The longer the delay, the longer the spectrometer waits before collecting and displaying data.

PROCEDURE

I: Laser Warm-Up and Orientation

1. Refer to the laser diagram in Figure 1. Have your TA carefully pull off the laser cover of the unused laser on the counter top and locate the lasing medium (plasma chamber) and flash lamp (together inside a water-cooled cylindrical container), internal laser cavity shutter, mirrors and optics. Do not touch anything inside. Carefully replace the laser cover. The laser being operated will not be opened.
2. Warm up the laser. This takes about 20 minutes, and during this time the orientation of the LIBS apparatus can be completed in step 3. Refer to Appendix A and complete up through step 4 to begin warming up the laser.
3. While the laser is warming up, familiarize yourself with the experimental setup by locating the following parts of the LIBS apparatus (Refer back to Figure 5):
 - a) the rotatable shutter on the top left of the laser (should be shut)
 - b) the external knob on the left side of the vacuum chamber that rotates the sample disc inside the chamber
 - c) the valve of the vacuum pump line
 - d) the on/off switch on the vacuum pump (make sure the pump is on)
 - e) the spectrometer and fiber optic cable

II: Spectra of Different Radiation Sources

The goal of this part of the lab is to observe the spectrum of various light sources and to become familiar with the spectrometer.

1. You will be using a compact Ocean Optics spectrometer, which is controlled by a computer. Locate the OOIBase32 icon on the computer and double-click it to start up the Ocean Optics spectrometer program.
2. If the spectrometer does not start measuring right away, do the following: select SPECTROMETER > CONFIGURE from the menu at the top to open the Spectrometer Configuration dialog box. Click on the A/D INTERFACE tab and check that S2000/PC2000/USB2000/HR2000 is selected in the Spectrometer. If it is,

you do not have to do anything (press Cancel). If there is no spectrometer selected ask your TA for help.

3. Before you start the LIBS project, use the spectrometer to record emission spectra of more common radiation sources (see step #6). For each light source, mount the fiber and your light source appropriately so that you can view the emission spectrum. Please DO NOT bend the fiber too much as it will damage it. DO NOT touch the end of the fiber-optics cable with your hands.
4. On the main screen of the program, locate the Integration Time box (labeled “Data Acquisition Box” when hovered over by the mouse) and select an appropriate integration time (500 milliseconds is a good starting value). This is the time the detector spends collecting photons, so the longer the integration time, the more intense the peaks should be. Locate the Average box and set the average to 1 so that spectrometer responds faster.
5. If the signal is too large, the spectrum can be “saturated” at the peak wavelengths. If this is the case, reduce the integration time, move the fiber further away from the source, or rotate the fiber away from the source slightly. Conversely, if the signal is too small do the opposite. Once you are happy with the integration time you may want to increase the averaging to 5-10 in order to improve the quality of your recorded spectra.
6. The following are specific suggestions for different sources. For each, attempt to predict the region (IR, Visible, UV, etc.) and, if possible, an estimated wavelength that should be seen (if in the Visible region). You can use the provided electromagnetic spectrum (Fig. 4) to help with your predictions. Record your observations in the spaces provided.
 - a. Incandescent lamp: Point the lamp (old nightlight) provided towards the fiber and adjust the lamp position to give an acceptable signal intensity. Avoid saturating the detector.
 - b. Fluorescent lights: Point the fiber towards the lights on the ceiling and find a position giving acceptable intensity level. Record the spectrum; you may be surprised at what you see.
 - c. Green laser pointer: This light source should be monochromatic (only one wavelength emitted). Predict this wavelength. Do NOT shoot the laser into the fiber – it may destroy the detector. Instead, scatter the laser off a white piece of paper and record the scattered light.¹ There may be more than one color available.

¹ This is a battery operated device. If the signal is too weak, take the batteries out and check their voltage with a multimeter. If the battery voltage dropped below 1.3 V, replace the batteries.

- d. Mercury vapor lamp: **Warning: wear protective eye wear.** This lamp is commonly used for wavelength calibration. The lamp should run for 30-60 seconds to warm up before you take its spectrum. The light emitted by this lamp is caused by a transition between different quantum levels in electronically excited mercury atoms. Would you expect broad peaks (polychromatic) or emission lines? Take two spectra: the first one with the strongest line not in saturation, and the second one with this line saturated so that the weaker lines are also visible.

- e. Small battery-operated UV-A lamp: **Warning: wear protective eye wear.** Do not forget to turn off the lamp when finished.¹

- f. White-light LED: Use the same lamp from part e, which has the LED built into it. Record the spectrum of this seemingly white light source.¹

- g. TV remote: Keep pressing one of the buttons of the remote and shoot directly into the fiber. What type of emission would you expect?¹

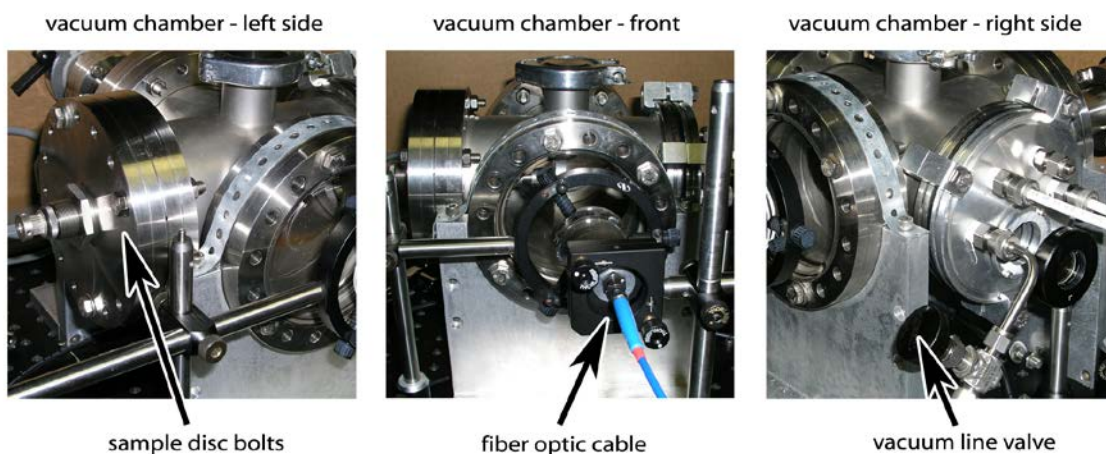
- h. RGB color matrix: Bring up a color wheel and image the colors directly off the laptop screen. By moving the spectrometer around your screen you should be able to record spectra corresponding to distinct colors (R-red; G – green; B – blue). Also record the spectrum of a WHITE portion of the computer screen. The signal is going to be **quite weak** – you may want to adjust the screen brightness to the maximum and make the fiber touch the computer screen. You may need to set averaging to a reasonably large number, 20-30.

III: Vacuum

The goal of this part of the lab is to understand the importance of vacuum to the experiment.

1. Close the shutter on top of the laser if it is open.
2. If the chamber is under vacuum, close the valve on the vacuum line and vent the chamber by unscrewing the vacuum line. You will hear a hissing sound as the chamber vents to atmospheric pressure. Reattach the vacuum line to the chamber.
3. Remove the sample disc from the vacuum chamber. First, locate the bolts holding the left side of the vacuum chamber in place (see Figure 6 below). Remove the bolts and carefully pull the entire left side of the chamber out (it may be heavier than expected). Since the sample disc is attached to the left wall of the chamber, keep the entire piece level to remove it.

Figure 6: LIBS vacuum chamber



4. Place a laser screen to block the backside of the now open chamber.
5. Make sure you are wearing laser goggles and then open the shutter.
6. Lower the Q-Delay switch value until sparking from the laser beam is clear (do not go below 280 units).
7. What do you observe? Why is the vacuum important for this experiment?
8. Once you are done, increase the Q-Delay switch value to 365 units.
9. Make sure that the shutter is closed.
10. The sample disc should still be outside of the chamber and ready to load sample on.

IV: LIBS Setup

1. Your TA will provide you with various samples without telling you what they are. Affix the samples onto the sample disc using bits of double-sided tape. The samples should be small enough to fit comfortably onto the sample disc. If needed, cut the samples down to approximately $1\text{-}2\text{ cm}^2$ in size. Be sure to record the order of your samples and describe each carefully so they can be located in the chamber. The disc can rotate, so the angular position of each sample is irrelevant. However, the center of each sample should be aligned with the circle created by the previous laser pulses (easily visible on the sample holder). Make sure the double sided tape is not sticking out from under the samples.
2. Write down the order or draw a picture of the approximate location of your samples with respect to each other in the space below. Example: beer cap: 3 o'clock; penny: 5 o'clock; white soft metal sample; 7 o'clock; etc. This will help you know which sample you are hitting with laser. For reference, the laser is aligned to hit the sample wheel below the middle.
3. Slide the sample disc back into the vacuum chamber and bolt it closed (this will require 2 people). When bolting the chamber closed, make sure the O-ring is sitting in its groove (this is the trickiest part!). If the O-ring is not properly seated, you will not get a good seal when you pump the chamber to vacuum. If you do it correctly there should be a barely visible gap between the flanges. If the gap is bigger or if you hear hissing sound when the chamber is under vacuum, the O-ring must have slipped out of place. Repeat the installation.
4. Open the vacuum line valve (two to three turns) to pump the chamber to vacuum.

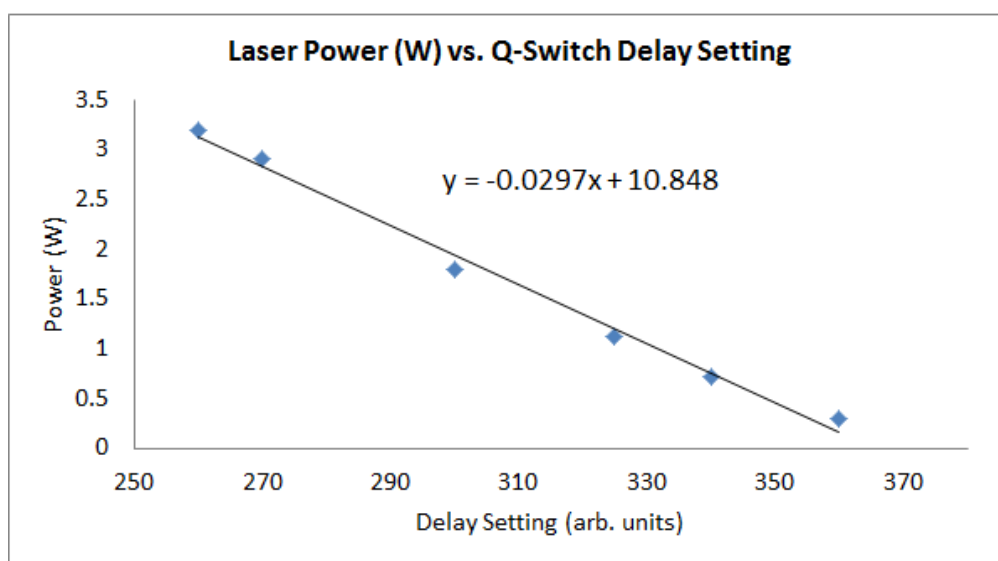
V: Plasma of Unknown Metals

The goals of this part of the lab are to observe the characteristics of plasmas induced on several different surfaces and to determine the identity of the unknown metals by using the emission spectra and comparing to the known spectra provided in Appendix B. You will be recording the descriptions, plasma colors and plasma shapes of each unknown sample provided by the TA directly onto your Postlab Worksheet.

1. The laser should be warmed up by now.
2. Make sure there are no objects obstructing the path of the laser beam. **Put on your laser safety goggles** (the green or brown ones).
3. Open the shutter on the laser cavity by rotating it.

4. Press start on the Q-Switch control section of the laser control panel
5. Optional: To test for the presence of the invisible infrared beam, take a piece of photographic paper wrapped in plastic and lower it carefully into the path of the beam. The laser should leave burn marks on the photographic paper.
6. On the laser control panel, press the **Center (symbolized by enter key) key** button to scroll through the settings using the up/down arrows and find the **Q-Switch** menu. Push **Enter** again, Scroll down to **FL-QS Dly.** Use the arrow buttons to reduce the delay time between laser pulses to 340 (or any value > 330). Make the laser hit one of your samples. What will lowering the Q-Switch delay do to the power? This will increase the laser power as shown in Figure 7.

Figure 7: Laser power calibration (as of June 2014)



7. On the computer set the spectrometer integration time to 350 milliseconds (the spectrometer will now record in 350 ms windows, which covers 3 laser pulses). Set the average to 1.
8. Adjust the position of the fiber optics cable mount to maximize the intensity of the peaks generated by the plasma. Place a piece of paper just in front of the mount holding the fiber optics cable to locate where the lens is focusing the radiation. Then, move the mount so that the opening of the fiber optics cable is where the imaged radiation is the most intense. Monitor the intensities of the peaks on the screen to determine which position is best (the higher the peaks are the better). If the signal is too strong and is saturated, reduce the integration time or reduce the laser power.
9. Now that the fiber optics cable is properly aligned, set the Average box to 10 to average 10 spectra together before displaying the data. Although there will be lag in the display of the spectra, this will considerably improve the signal-to-noise ratio.

10. Rotate the sample disc a miniscule amount (~1 mm) so that the beam is striking a fresh spot on the first sample. You can also “freeze” a spectrum by clicking on the Camera Icon on the upper left of the screen
11. Look into the sample chamber through the large viewing window (not through the small window where the laser enters!) to observe the plasma. If the laser is striking the middle of a flat sample it may be safe to briefly lift up your goggles for a better view. Warning: DO NOT lift up your goggles when the laser is striking an edge or when you are rotating the sample disc. Record the color and characteristics of the plasma generated by the laser striking your target. Your notes may look something like this: “Pale-yellow spherical puff about 1 cm in size from the soft white metal sample.”
12. Rotate the disc so that the laser strikes the next sample, and record the shape and color of the generated plasma in your lab book. You may increase the laser power (by reducing the Q-switch delay) if the plasma is too weak. However, do not reduce the delay below 260 units, the setting that corresponds to about 2 Watts of laser power according to Figure 7.
13. If a digital camera is available, take photographs of the different types of plasma. You will have to secure your camera and play with the exposure to get good results.
14. Rotate about 1 mm to a fresh portion of the sample and collect the emission spectra of the plasma as before in Step 9.
15. Collect the spectra and plasma descriptions for the rest of the samples.
16. After you are done, set the delay on the laser control box to 365 and close the shutter button on the laser to disable the laser beam.

VI: Depth Profiling

You may need to vent the chamber as done in Part III to load new samples for Parts VI and VII. Try to have different people get the practice each time.

One of the unique features of LIBS is its ability to drill into the sample and probe different depths during the analysis. When the laser beam starts to hit the sample it vaporizes elements from its surface. As the laser keeps heating the same spot, it slowly penetrates deeper into the sample, and probes the composition of the interior. The easiest way to observe this effect is to look at the spectrum of a penny or a zinc-plated or chrome-plated steel sample. To observe this effect, set averaging to 1 (to make the spectrometer respond faster), move the laser onto such a sample, and look at what happens to the spectrum as a function of time. For the first few seconds, you should see a spectrum corresponding to the outer coating. A few seconds later, the spectrum should change drastically as the laser bores into the inner material. Try to capture and save the spectra in the early and late stages of the depth profiling through zinc- or chrome-plated steel. A “capture” icon (that looks like a camera) is a convenient tool in the Ocean Optics software for freezing the spectrum at a particular time. Next, try to do the depth profiling for a pure material such as copper. Does the spectrum change as you drill through?

VII: Spectra vs. Laser Power

The goal of this section is to observe the effect of different laser powers on the plasma induced. To change the laser power you will be changing the Q-switch delay as shown in Figure 7. Spectra of plasma induced at different laser powers will be recorded and then compared to one another. Your TA will supply you with samples to try. The default set of materials is: W (tungsten), Mo (molybdenum), and Si (silicon).

1. Install the samples provided by the TA in the vacuum chamber as instructed in Parts III and IV, unless the samples are already in the chamber.
2. Set the spectrometer integration time to 350 milliseconds (the spectrometer will record in 350 ms windows, which covers 3 laser pulses). Set the average to 1.
3. Set the Q-switch delay to 340 units (or any value > 260 at which the plasma starts to be visible) and direct the laser beam onto one of your samples.
4. Adjust the position of the fiber optics cable mount to maximize the intensity of the peaks generated by the plasma. Monitor the intensities of the peaks on the screen to determine which position is best (the higher the peaks are the better). If the signal too strong and is saturated reduce the integration time or reduce the laser power.
5. Now that the fiber optics cable is properly aligned, set the Average box to 10 to average 10 spectra together before displaying the data. Although there will be lag in the display of the spectra, this will considerably improve the signal-to-noise ratio.
6. Rotate the sample disc a miniscule amount (~ 1 mm) so that the beam is striking a fresh spot on your sample.
7. Similarly, record spectra for Q-Switch delays of 320, 300, and 280. Make sure that the beam is hitting a fresh spot on the disc just prior to recording the spectrum.
8. Set the delay on the laser control box to 360 after you are done collecting spectra.

VIII: Clean Up

1. Close the shutter and power down the laser according to the instructions in Appendix A.
2. You may leave the pump running and chamber under vacuum.
3. Exit the computer software.

Appendix A

Standard Operating Procedures (SOP) for the Quantel Laser

Turning on the laser:

- 1) Turn the key switch to the “on” position (clockwise). Wait a few seconds for the laser to finish its diagnostics. The box is situated behind the optical table towards the back right corner.
- 2) Slide the shutter wheel on the laser head.
- 3) Press the **Center (symbolized by enter key) key** button to scroll through the settings using the up/down arrows and find the **Q-Switch** menu. Push **Enter** again, Scroll down to **FL-QS Dly.** The delay should read ~340 μ s, if it does not, use the +/- or Left/Right arrows to adjust it.
- 4) Press the **Start** button on the **Flashlamp** controls. The yellow LED will come on (If the laser has not been warming up then the Flashlamp may shut off until the laser is at 29°C) The laser head is now flashing at 10 Hz frequency. Let it warm up for approximately 20 minutes for thermal stabilization. You should familiarize yourself with the rest of the LIBS apparatus and experimental procedures while waiting for the laser to warm up.
- 5) Put on your laser safety goggles, which block out the specific radiation being emitted by the laser (specifically 1064 nm in the Infrared region which is not visible to the naked eye!).
- 6) Press the **Start** button on the **Q-Switch** controls. The LED will come on as laser radiation is now leaving the laser. You should hear a click sound as the laser radiation hits the bare carousel surface in the chamber (this sound will diminish as the vacuum is turned on).
- 7) Change the Q –Switch delay settings to alter the laser power as instructed in the lab procedure:

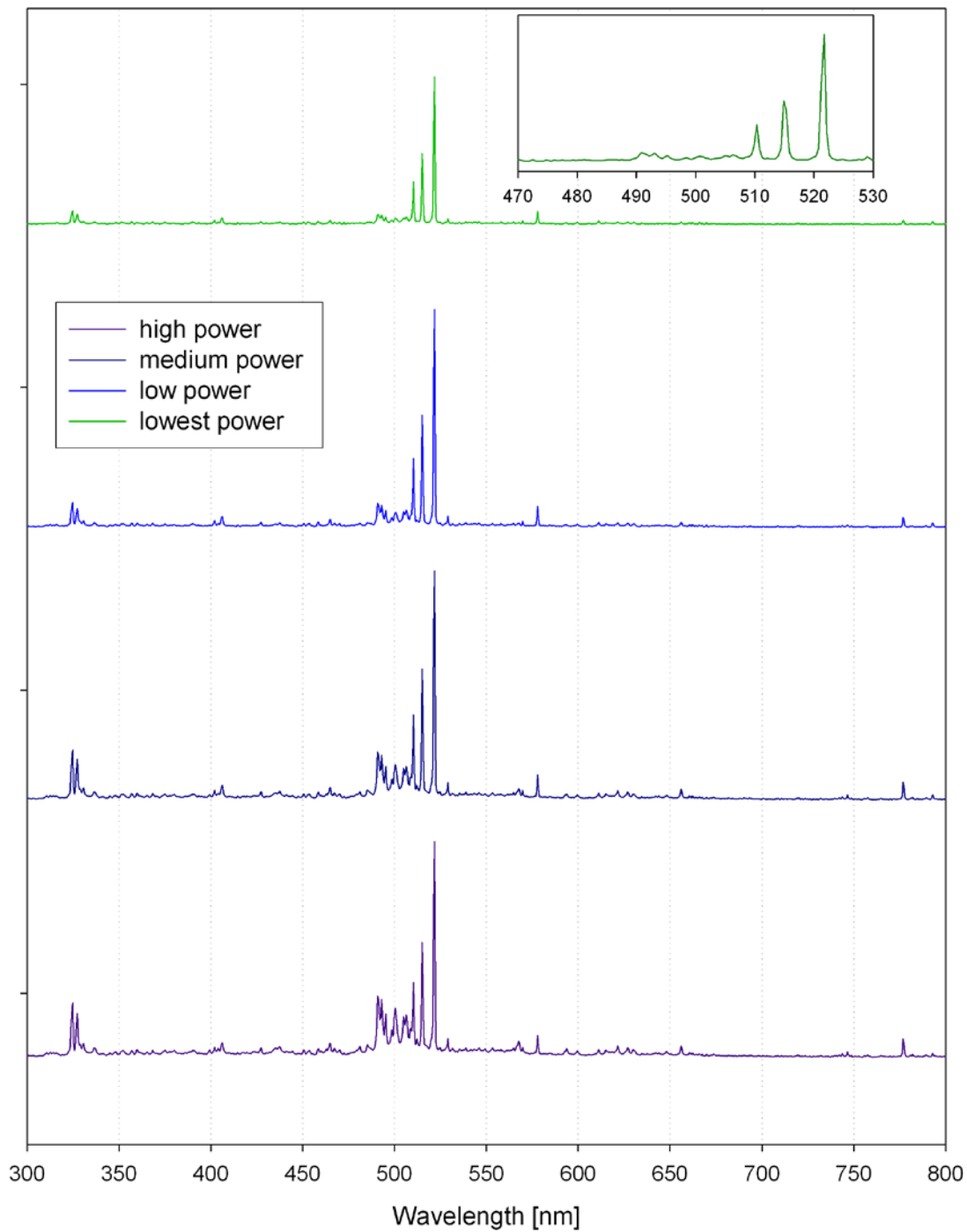
Delay Settings	Note
360-375	Low power; Good for laser alignment.
330-340	Enough power to generate a weak LIBS signal.
300-330	Enough power for a sufficiently bright LIBS plasma.
280	Maximum power allowed without instructor approval!!
180	Maximum power....very dangerous!

Turning off the laser:

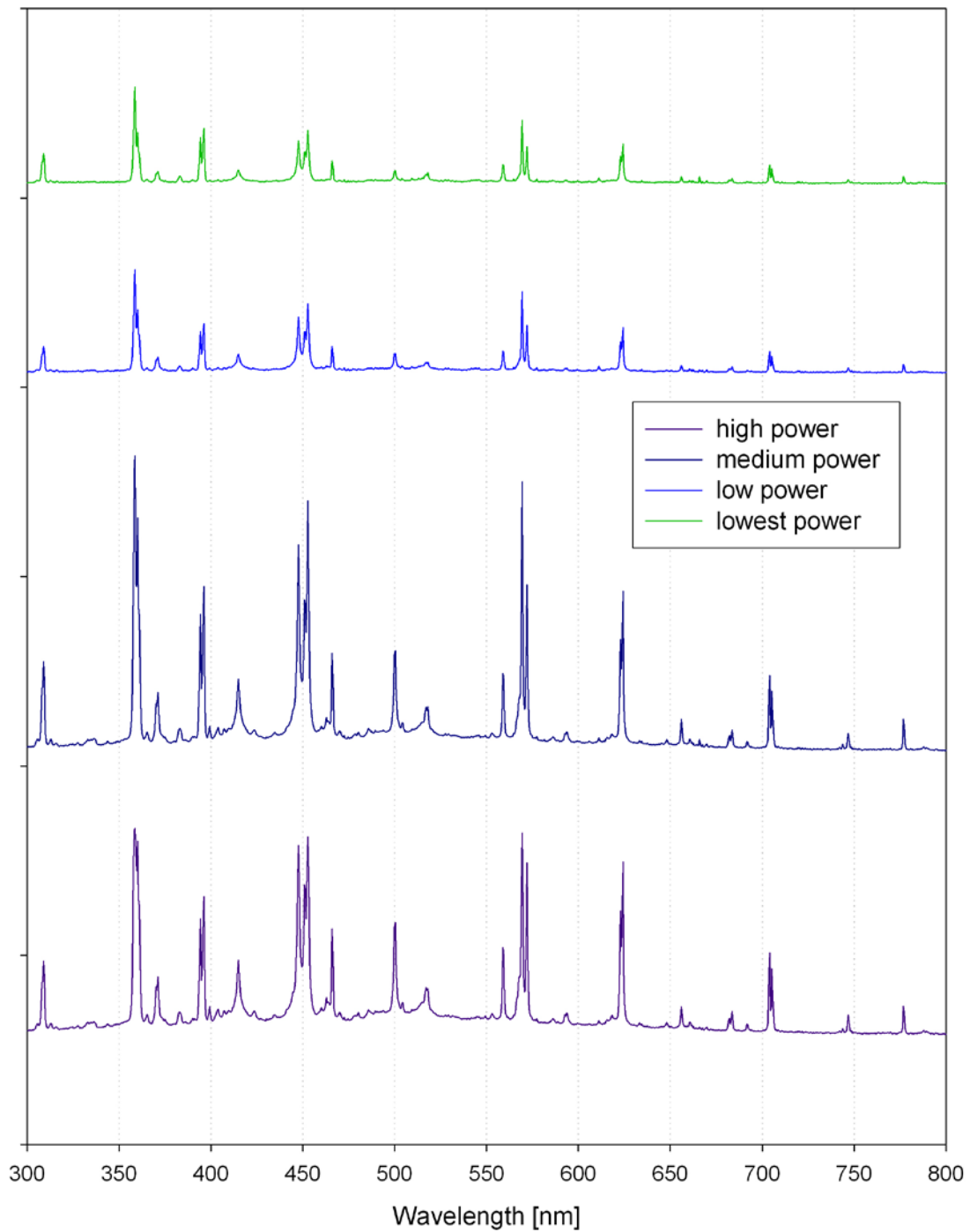
- 1) Press the **Stop** button on the **Q-Switch** and then the **Flashlamp**. The LED will go off as well as the LED on the Flashlamp button should also go off. The flash lamps should stop flashing.
- 2) Slide the shutter wheel on the front of the laser to **Closed**.
- 3) On the Laser Power Supply, turn the key counter clockwise to the **O** position.

Appendix B: Spectra of different materials you may use in this lab

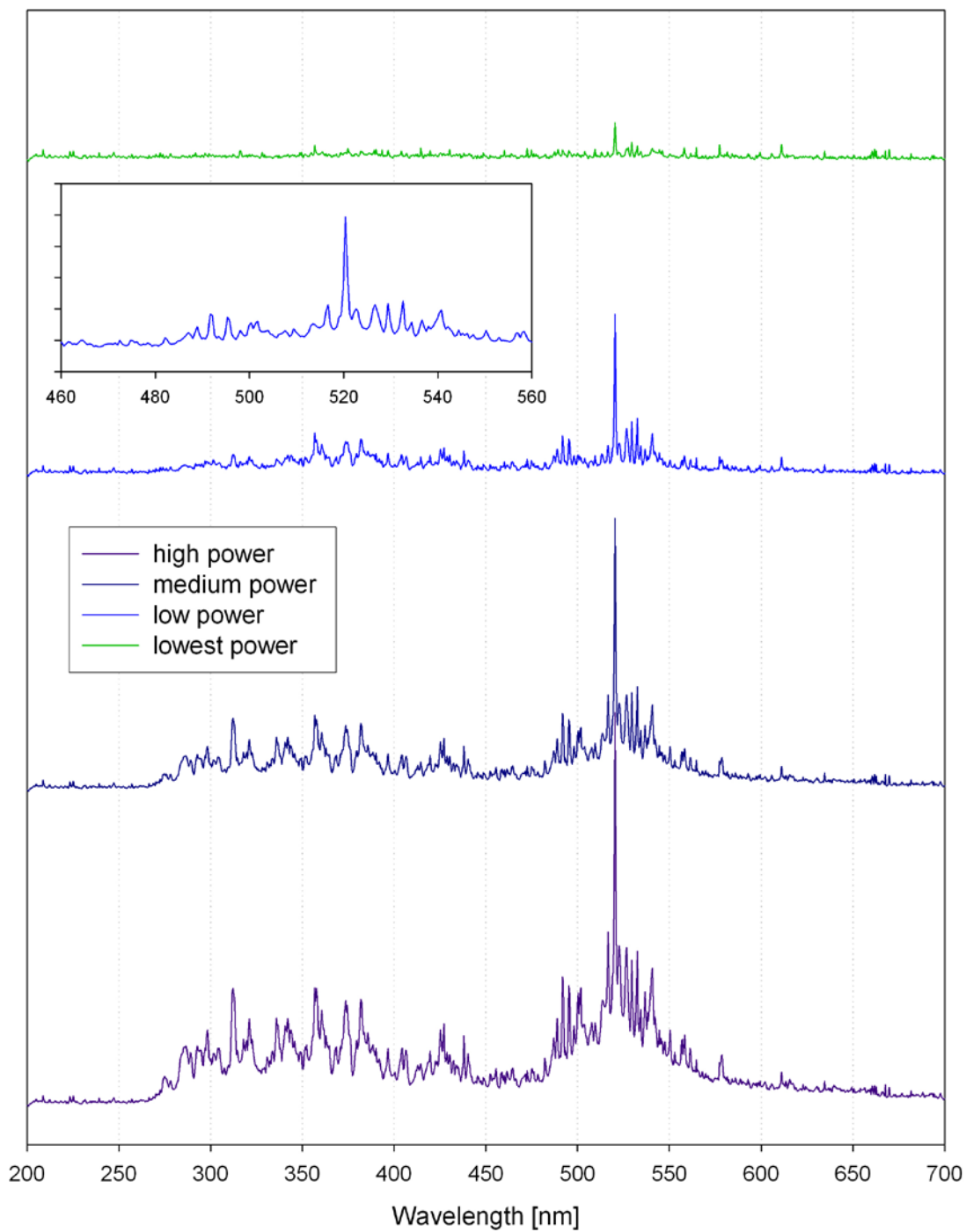
LIBS spectra of Copper (Cu) samples



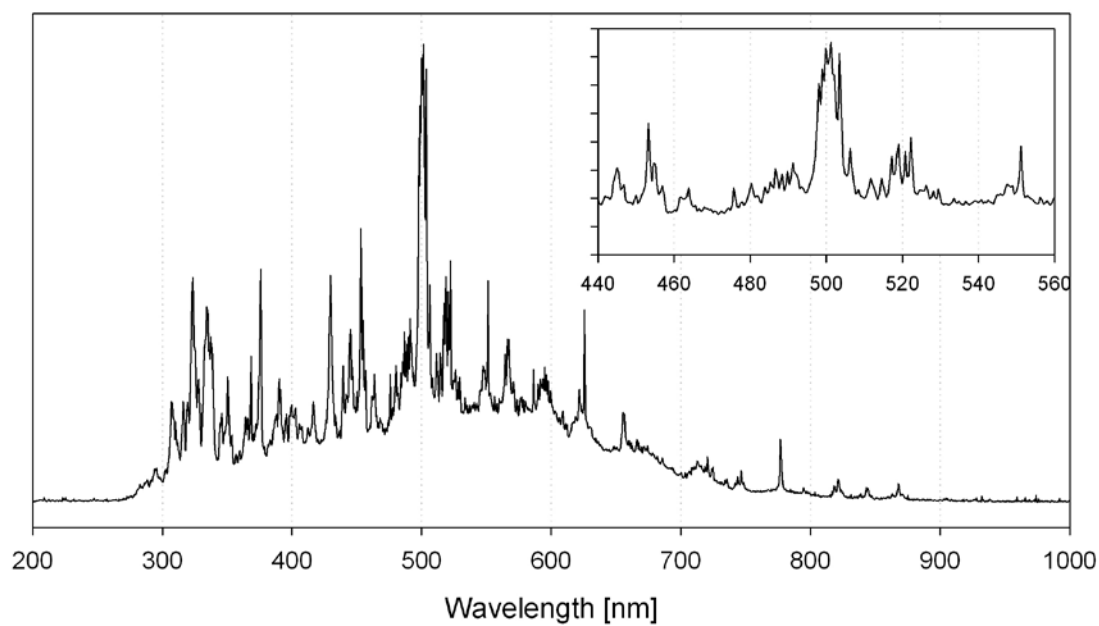
LIBS spectra of Aluminium (Al) samples



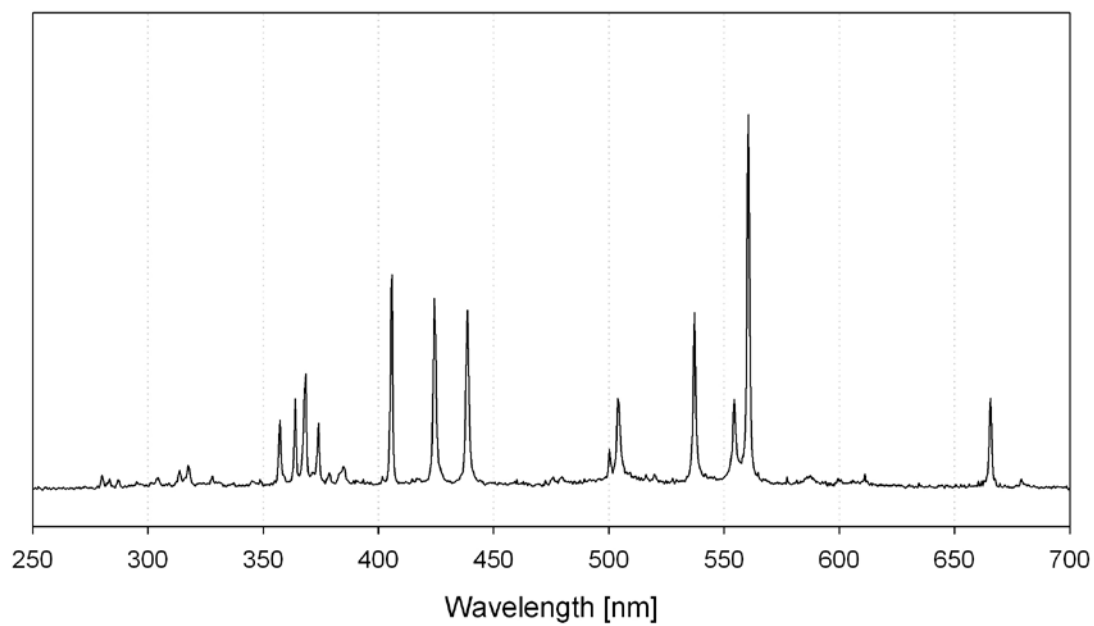
LIBS spectra of Steel samples



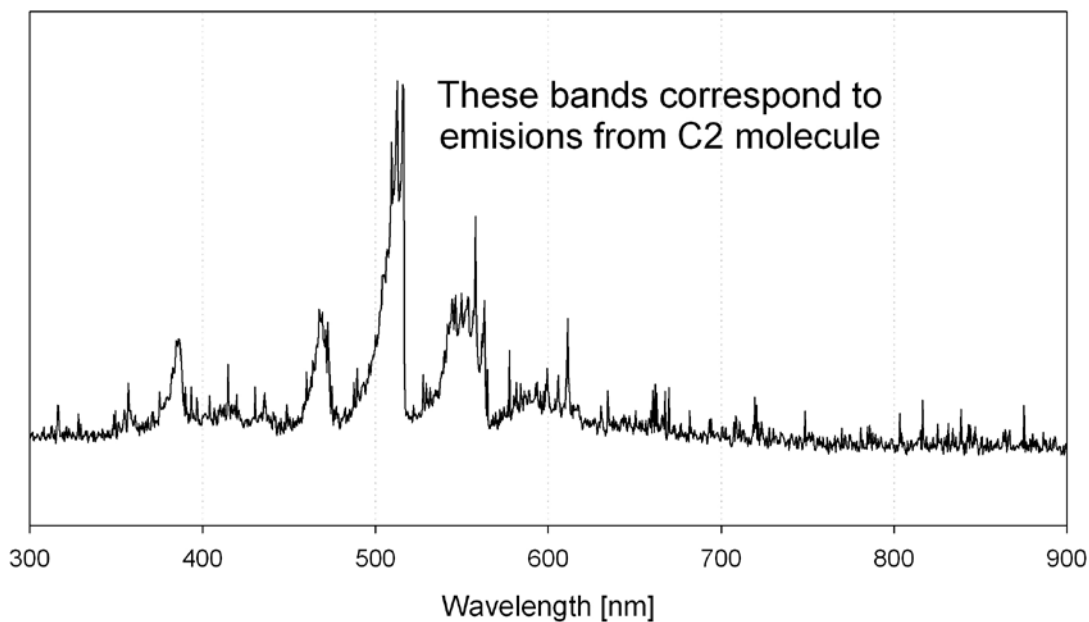
Titanium (Ti)



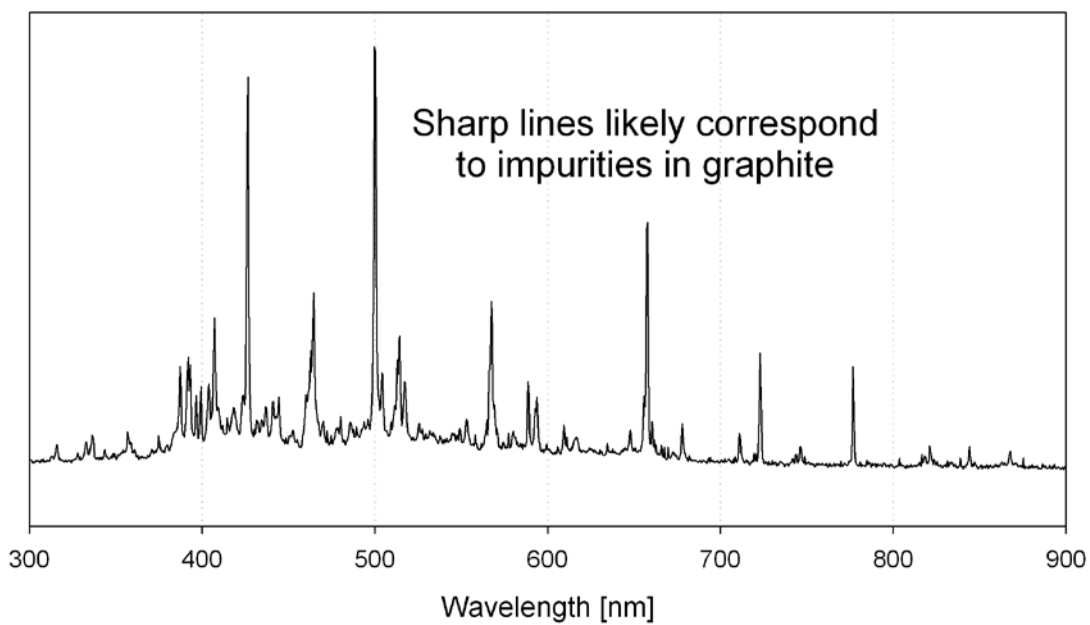
Lead (Pb)



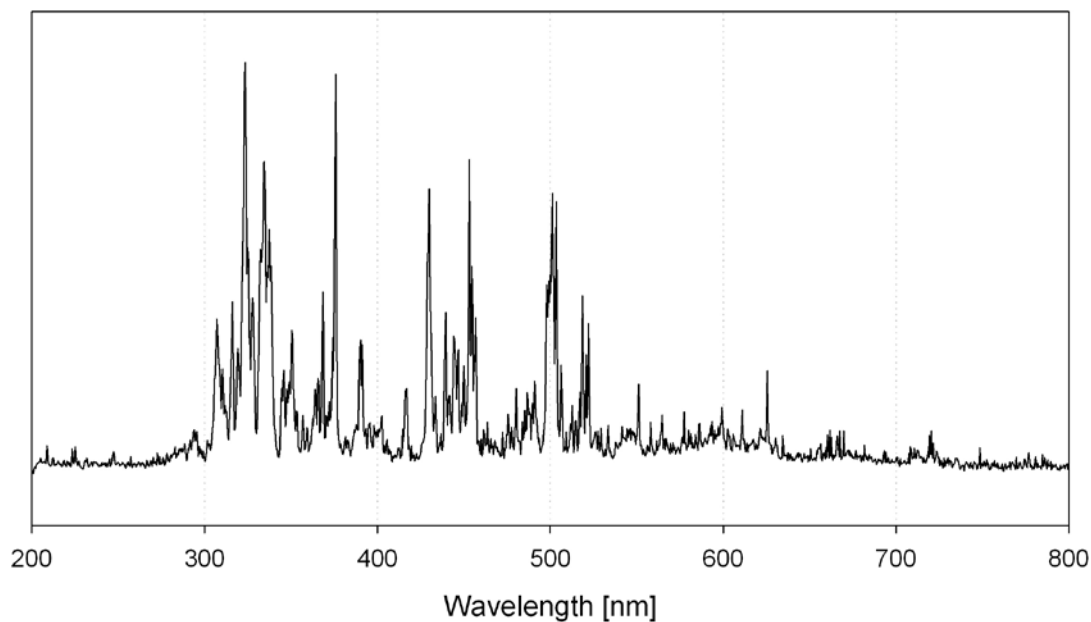
Pure HOPG (highly-oriented pyrolytic graphite, C)



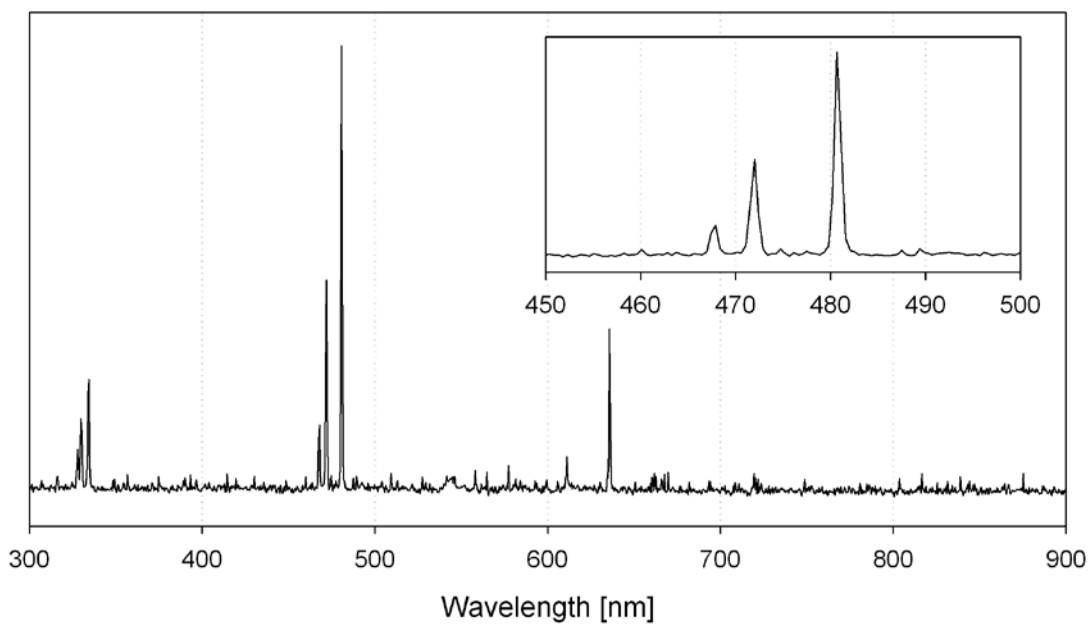
Lower-quality pyrolytic graphite



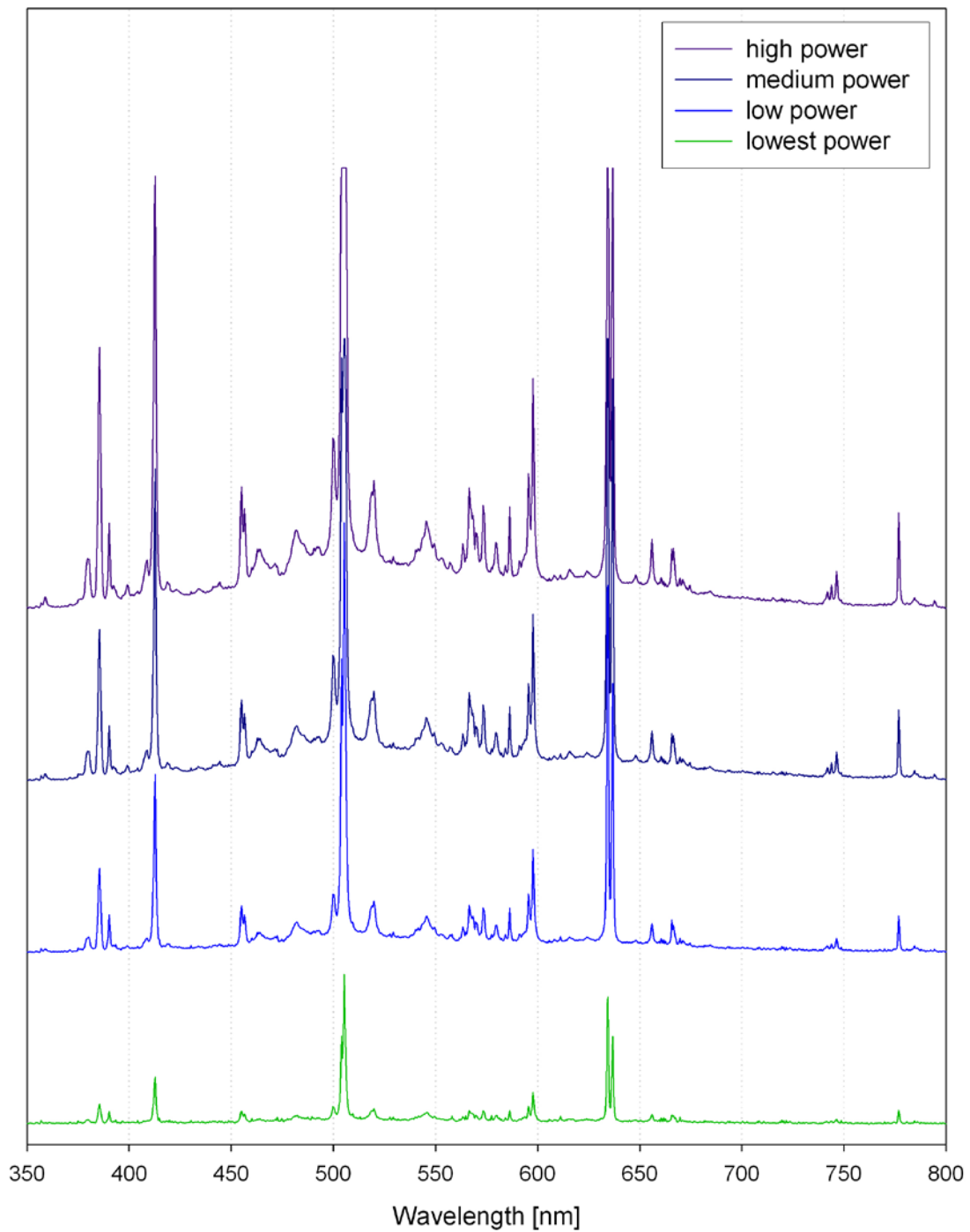
Molybdenum (Mo)



Zinc (Zn) from the inner layers of a new penny



LIBS spectra of Silicon (Si) samples



LIBS spectra of Tungsten (W) samples

