IN-HOUSE EXPERIMENT

COMPARISON OF TERRESTRIAL AND EXTRATERRESTRIAL ROCKS USING LIBS

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BACKGROUND INFORMATION

Meteorites are extraterrestrial rock that fall to the surface of Earth, as opposed to meteors that burn up before making contact. Meteorites have played a crucial role throughout the history of our planet. Some hypothesize that the Moon formed from the remnants of a large meteorite colliding with Earth. Another hypothesis suggests that the water that covers Earth was brought here by meteorites. Perhaps most notably, it is commonly believed that the Cretaceous-Paleogene extinction event, which caused the extinction of all non-avian dinosaurs, was caused by one or several meteorite impacts. With meteorites being linked to these events, among many others, it is no surprise that they are sought-after specimens of historians, geologists, and anyone interested in what is beyond our planet. Unfortunately, determining whether a rock is a true meteorite or has been misidentified (colloquially called a "meteor-wrong") can be a difficult process. Simple indicators may be; density, magnetism, and composition: meteor-wrongs often contain quartz, which is not found in meteorites. But in cases where terrestrial rocks are generally similar to meteorites in appearance and composition, such as amorphous rocks primarily composed of iron, more targeted methods must be used to determine the origin of a rock. One such method is laser-induced breakdown spectroscopy, or LIBS.

LIBS is a type of atomic emission spectroscopy which uses a highly energetic laser pulse as the excitation source. The laser is focused to form a plasma plume, which atomizes and excites samples. Plasma formation begins when the focused laser achieves a certain threshold for optical breakdown. From the plasma plume, the spectrometer measures peaks at specific wavelengths that can be correlated to particular elements, as all elements emit light of characteristic frequencies when excited to sufficiently high temperatures. As a result of this phenomenon, LIBS is an excellent spectroscopic technique for identifying the specific composition of different types of metals and minerals.

This experiment aims to use LIBS to distinctly characterize a collection of rocks as either meteorites or terrestrial rocks. Two iron-rich meteorite samples (H5 ordinary chondrite and iron ataxite) were measured along with iron oxide (FeO2) to compare similar extraterrestrial and terrestrial rock samples. Titanium dioxide (TiO2) was also measured to provide a vastly different spectrum to compare the other samples against.

FIGURE 1 Rock and meteorite samples used for this experiment (from left to right: H5 ordinary chondrite, iron ataxite, iron oxide, and titanium dioxide)and Summer Gray).

DESCRIPTION OF SPECTROSCOPY SETUP

The setup for this experiment (Figure 2) is based on the Compact Spectrometer. In cases where size matters, the AvaSpec CompactLine family offers compact spectrometers, some of the smallest units on the market today. Their compact size enables easier integration of the spectrometers into machines and handheld devices. The ease of integration makes the CompactLine especially suited for OEM users wanting to integrate a spectrometer into their devices. The Compact Spectrometer is equipped with a 4096-pixel CMOS detector array. The CMOS array features an incredible resolution of up to 0.09 nm, and the low stray light design allows stray light levels as low as 0.1%. The fast response time boasts data transfer speeds as fast as 1.12 ms/scan and integration times ranging from 9 µs to 30 s. The Compact Spectrometer used in this experiment is optimized for the UV range at a 200-450 nm wavelength range.

The light source used for this experiment was the plasma generated from a DPSS (double pulse, single scan) Nd:YAG laser, courtesy of Lumibird. Lumibird provides a wide range of lasers, with this one in particular built for rugged applications and unpredictable environments where temperature and vibration would destroy most commercial lasers. This laser operates at eye-safe wavelengths, specifically at 1574 nm, with an energy of 4.5 mJ per pulse. Designed for battery operation in hand-held and mobile applications, this compact yet robust laser features maintenance-free operation for years of hard use.

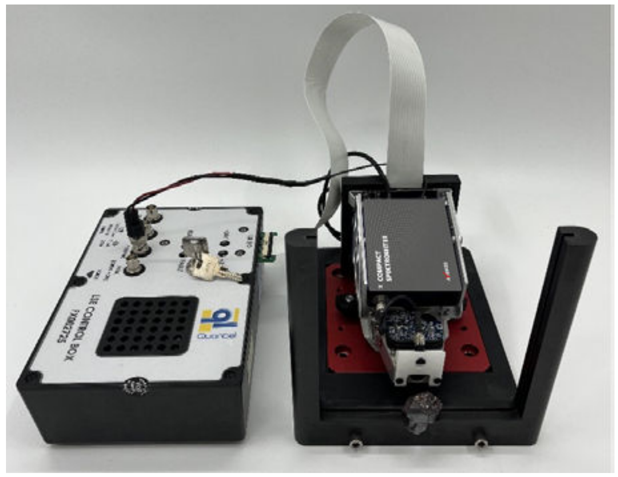

FIGURE 2 Experimental setup for LIBS measurement. The spectrometer is attached to a custom fixture and mounted directly above the laser. The unit measures the LIBS plasma plume via a mounted short fiber optic cable.

Other accessories used for this experiment included a custom short 100-micron core fiber optic cable to directly measure the plasma plume, a Quantel control box (FX002725) to power the laser and sync the laser pulses and spectrometer measurements, a custom interface cable to connect the AvaSpec-PCT4096CL to the control box, and a custom 3D-printed mount to hold the fiber optic cable at the precise angle to line up with the focal point of the laser.

DESCRIPTION OF METHODOLOGY

Two of the four total samples are terrestrial rocks (iron oxide and titanium dioxide), and two meteorite samples (H5 ordinary chondrite and iron ataxite). Each sample was placed in the firing range of the laser, and the laser was activated using serial commands. The spectrometer was synced to the laser pulses on the hardware side using the Quantel control box and on the software side through defined settings in AvaSoft, our custom software package.

For data analysis, we used the Scope-Minus-Dark mode in AvaSoft. This is a common mode for LIBS measurements, as it subtracts the dark spectrum (what the spectrometer measures with no light source) from the raw counts (i.e., scope mode) for each wavelength. This helps minimize noise in the spectrum and better isolate and identify the plasma plume peaks. We used an integration time of 10 milliseconds, which in most cases, can be adjusted to increase or decrease the amount of light being measured at one time and affects the overall magnitude of the reported spectrum. For LIBS applications, since the plasma plume is generated and decays at such short intervals, increasing the integration time will increase the portion of time where no light is measured and decrease the measurement amplitude. We set averaging to 1, meaning each measurement corresponds to one plasma plume instance.

TEST DATA AND RESULTS

Displayed below are the LIBS spectra of the samples in the scope-minus-dark mode for each sample.

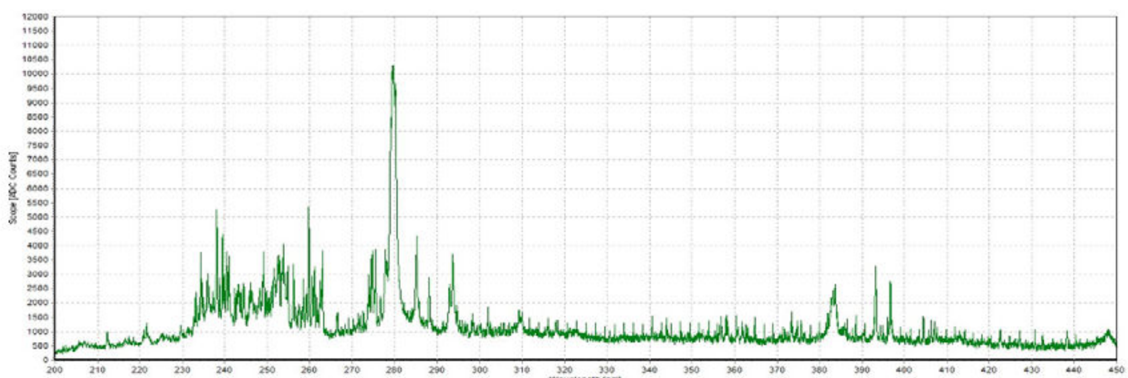

FIGURE 3: LIBS spectra of H5 ordinary chondrite sample

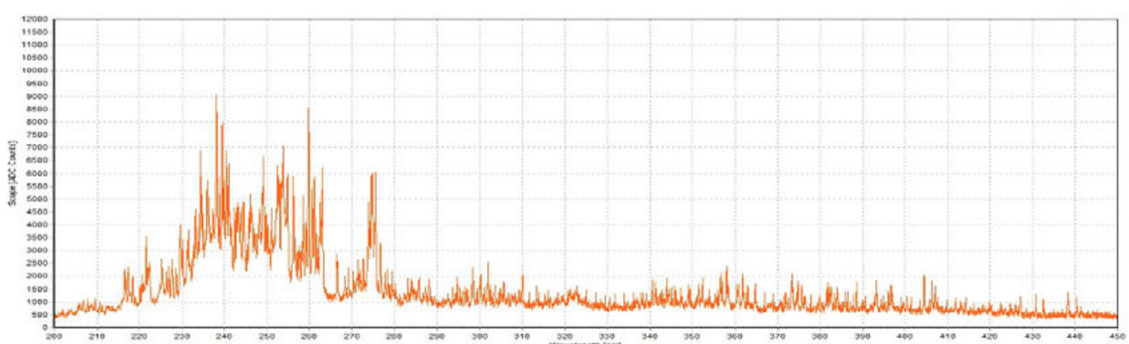

FIGURE 4: LIBS spectra of iron ataxite sample

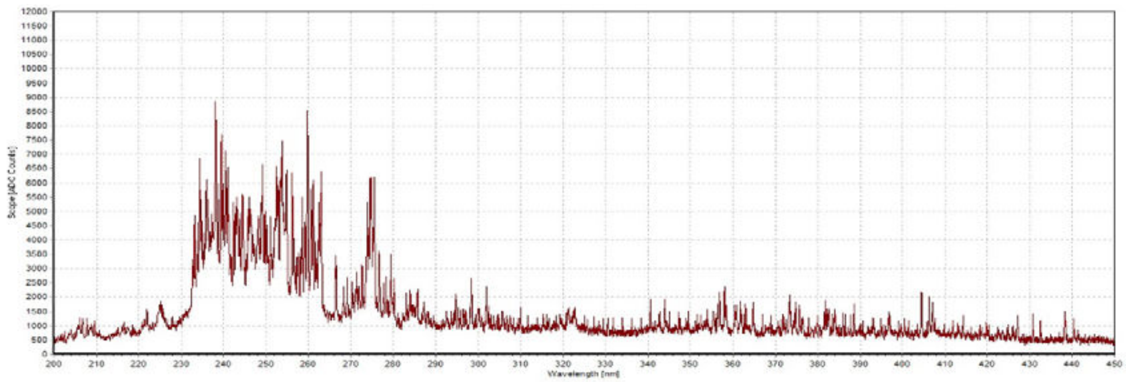

FIGURE 5: LIBS spectra of iron oxide sample

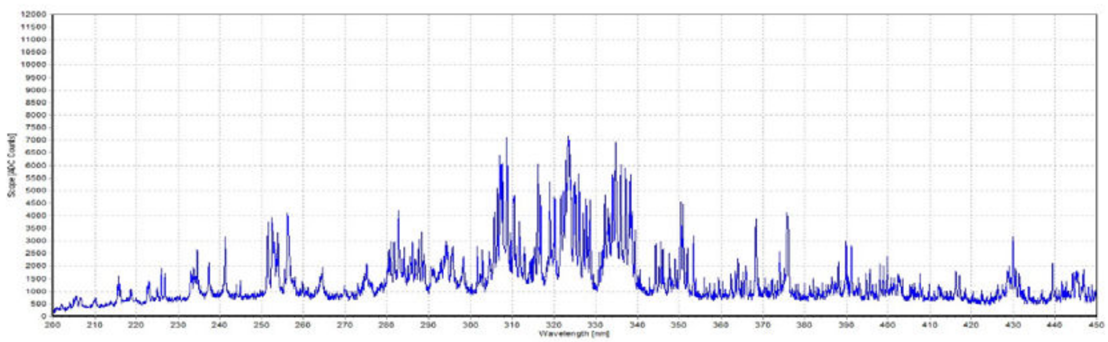

FIGURE 6: LIBS spectra of titanium dioxide sample

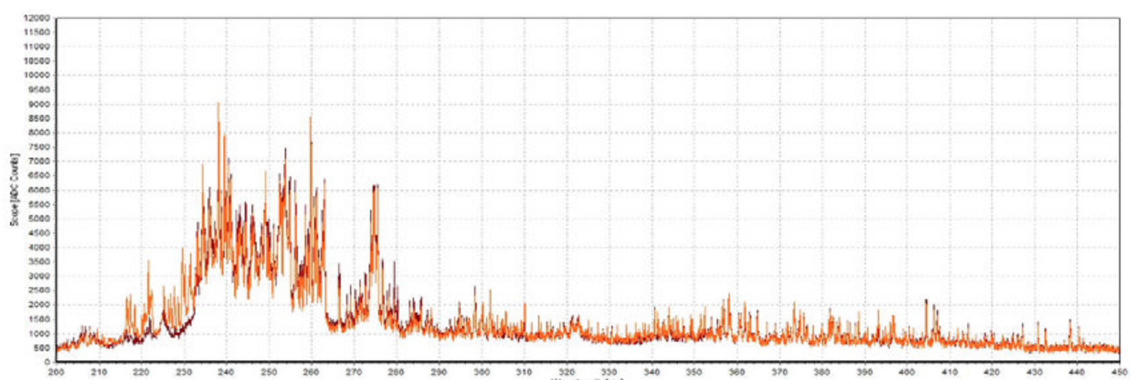

FIGURE 7: LIBS spectra of iron ataxite (orange) and iron oxide (red) samples, shown together for comparison

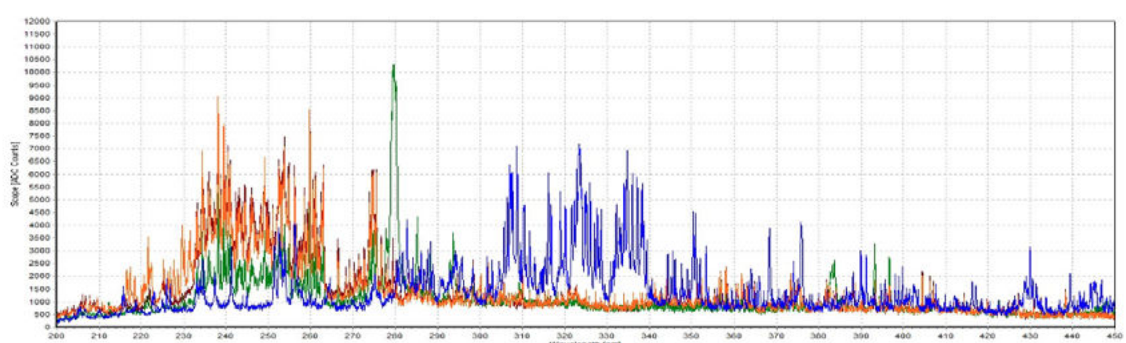

FIGURE 8: LIBS spectra of H5 ordinary chondrite (green), iron ataxite (orange), iron oxide (red), and titanium dioxide (blue) samples, shown together for comparison

ANALYSIS AND CONCLUSION

Three of the four samples measured (H5 ordinary chondrite, iron ataxite, and iron oxide) showed a cluster of peaks in the range of approximately 230-265 nm (Figures 3-5). This range indicates iron, which is an obvious conclusion from the names of the iron ataxite and iron oxide samples. The H5 designation in the chondrite sample categorizes it to have a high iron abundance, so these results align with the expected spectra. The H5 ordinary chondrite sample has lower values in this 230-265 nm range but does have a significant peak around 280 nm, where the other two iron samples do not (Figure 5). While the lower magnitude in the 230-265 nm range cannot be directly correlated to a lower iron percentage without more complex analysis, the significant peak around 280 nm would indicate the presence of magnesium. This makes sense since the most abundant minerals in H-type ordinary chondrites are bronzite and olivine, both of which are partially made up of magnesium. Silicon has a prominent peak around 288 nm, which is also seen in the H5 ordinary chondrite spectrum. Not surprisingly, silicon is also present in the minerals bronzite and olivine. Besides iron, the other elemental component of these minerals is oxygen, though its presence is not seen since its peak is in the 770-780 nm range.

The titanium dioxide sample (Figure 6) shows significantly fewer peaks in the 230-265 nm range, indicating little iron content. Instead, the main peak range is visible in the 300-340 nm range, which correlates well with titanium. Titanium also has significant peaks at higher wavelengths, which may also be indicated in the spectrum.

Comparing the iron ataxite and iron oxide samples, little difference can be seen between the two (Figure 7). While this seems to thwart our attempt to present a clear distinction between terrestrial and extraterrestrial rocks, this is consistent with the ataxite classification, which is composed mainly of meteoric iron in addition to high nickel content. A slightly higher peak around 221 nm is seen in the iron ataxite sample, which may correspond to a nickel peak, which is often seen between 200-250 nm. A graph of all the spectra together is included for comparison of all samples (Figure 8).

CONCLUSION

In conclusion, the present experiment highlights the use of the LIBS measurement technique in determining the elemental compositions of samples. While no distinct feature could be used here to determine if a rock sample was terrestrial or a meteor, the elemental compositions could be partially determined easily by the significant peaks measured from each sample. Further analysis could fully characterize the composition of each sample, which in turn could uncover specific qualities to determine the origin of rock samples. The Compact Spectrometer is an ideal solution for OEM applications or any other cases where a compact form factor is critical. The Lumibird laser pairs perfectly with the Compact Spectrometer for LIBS measurements, but there are many more suitable lasers available. Both the custom interface cable and the 3D-printed cable mount highlight the capabilities of our engineering team to provide custom assemblies and solutions for customer needs. Please get in touch with Avantes for more information on the configuration that is best suited for your data collection.

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