

OBSCURATION OF OPTICAL TRANSMISSION DUE TO LASER DEPOSITED MATERIAL DURING MICRO-LIBS INTERROGATION OF A ROCK SURFACE. Christopher. B. Dreyer¹ and Greg S. Mungas²,
¹Colorado School of Mines, Center for Space Resources, Golden, CO 80401, cdreyer@mines.edu, ²Jet Propulsion Laboratory, California Institute of Technology (M/S 306-336 4800 Oak Grove Dr., Pasadena, California 91109).

Introduction: Detailed investigation of prepared and unprepared surfaces at the microscopic scale has been used extensively to study the origin and history of geologic samples in terrestrial labs [1] and on Mars [2]. We are investigating the use of Laser Induced Breakdown Spectroscopy (LIBS) focused to microscopic scale (micro-LIBS) as an analytical device for Mars exploration. Microscopic interrogation of a surface requires that the instrument be placed in close proximity to the rock surface. In LIBS, material is destructively removed (ablated) from the rock surface and is heated to form a plasma. Elemental emission from the hot plasma is collected, spectrally dispersed, and analyzed to determine elemental composition of the ablated material. The ablated material is propelled from the surface and could deposit on the outer optical elements of the LIBS device.

We have been investigating LIBS on rock samples at the microscopic (<20 μm spot diameter) scale and with micro-joule laser pulse energy (<200 μJ). In this work we present analysis and plans for experimental measurements to determine the risk of obscuration of optical transmission due to material deposited on the fore-optical element in a breadboard micro-LIBS device.

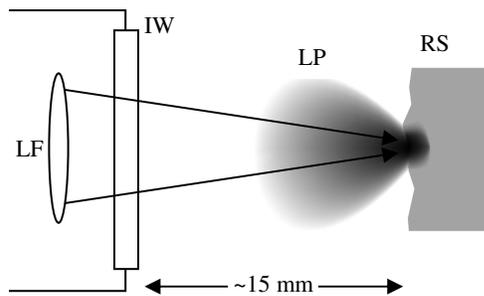


Figure 1: Schematic of arrangement of micro-LIBS optics, rock sample, and LIBS plasma/ablation plume. LF: Laser focusing optics, IW: instrument window, LP: LIBS plume, RS: rock sample

A simple schematic of the arrangement of a micro-LIBS instrument, a rock sample, and LIBS plasma/ablation plume is shown in Figure 1. The LIBS plasma/ablation plume size is dependent on gas pressure and composition [3]. The plume is larger at low pressure, however ablated material volume is independent of pressure. The mean direction of ablation material is in the direction of the local surface normal.

On a prepared surface it may be possible to direct the plume toward or away from the instrument window by placement of the instrument. On an unprepared surface it may not be possible to determine the likely plume direction.

Successful operation of micro-LIBS requires precise positioning of the instrument with respect to the surface. LIBS requires laser fluence $>1\text{GW}/\text{cm}^2$ for consistent plasma generation and 10 or more times greater fluence is preferred. For microscopic interrogation of a surface the laser is at the minimum beam waist over a small depth range. The well know Rayleigh range of a TEM00 order beam is

$$z_R = \frac{\pi d_o^2}{4 \lambda}$$

For 532 nm light, and minimum beam waist diameter, d_o , of 10 μm , the Rayleigh range is 147 μm . Over the Rayleigh range the diameter increase by $\sqrt{2}$, and the area doubles.

At the minimum beam waist the laser fluence and the plasma temperature is maximized. Hotter plasmas should result in significantly more signal strength given the strong dependence of LIBS emission signal with plasma temperature [4]. As the instrument placement is displaced from optimum, the fluence falls, and LIBS signal decreases, however for some amount of off-optimum depth positioning a usable LIBS plasma may still be obtainable. Beyond a certain amount of positioning error the laser will ablate the surface without producing a usable LIBS plasma.

Two deposition regimes can be defined: 1) plasma regime, and 2) ablation regime. In the plasma regime the majority of the material removed from the surface is heated sufficiently to produce a plasma. Subsequently the material cools to a gaseous, solid, or liquid state. In the ablation regime, the material removed is not heated sufficiently to form a plasma. The majority of the material remains as a solid or is heated to a liquid melt.

These two regimes are defined to elucidate the nature of potential material deposition on a micro-LIBS optic. In the plasma regime a greater fraction of material is gaseous, which may not deposit as readily on the instrument window as the solid and liquid material in the ablation regime. Solids and liquids may deposit in the plasma regime, however, the expected deposition rate is lower than in the ablation regime.

Material deposition on the instrument window will attenuate light transmission primarily by scattering and absorption. Solid material that could be deposited in the plasma regime is likely to be small particles, while solids and liquids deposited in the ablation regime are likely to be larger. Since scattering efficiency scales with particle size we theorize that the potential for obscuration is a greater in the ablation regime.

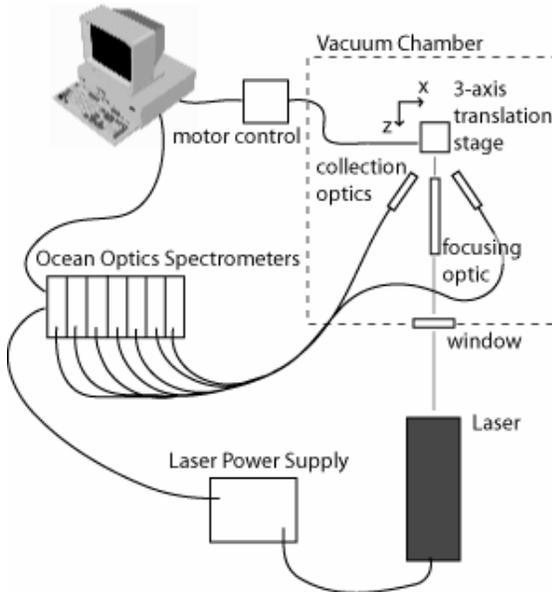


Figure 2: Schematic of micro-LIBS experimental setup.

Experimental: LIBS experiments are carried out in a low pressure simulated martian atmosphere. A schematic diagram of the experimental apparatus used for LIBS is shown in Figure 2. The laser is focused onto a sample by a custom three element focusing optic with 17 mm working distance and $f/2.8$. Microscopic images of craters remaining after LIBS plasma generation on hematite samples with 10-20 pulses per spot showed a 15-18 μm diameter. The sample, translation stage, laser delivery optic, and LIBS collection optic are housed in a low pressure chamber. Pressure can be varied from 1 torr to 100 torr. The 7 spectrometers span the spectral range of 195 to 970 nm and are from an Ocean Optics LIBS 2000+ system. Additional experimental details can be found in [5,6].

LIBS plasma emission is collected via seven optical fibers each with a single element optic of 12 mm focal length in 1:1 imaging configuration, arranged in a mount that enables alignment of the optics to view the same spatial location, custom designed and constructed by Firestar Engineering. The laser focusing optic can be translated axially relative to the collection optics and each LIBS collection fiber optical assembly can be translated axially to optimize laser delivery and collection alignment.

A diode pumped, actively q-switched, intracavity doubled, Nd:YLF laser is used in this study, produced by Crystallaser, model QG-523-800, of Reno, NV. Output is at 523nm. Below 1kHz the laser produces approximately 170 μJ per pulse with 10 ns pulse length. Peak pulse power is about 15kW at pulse repetition rates below 1kHz, sufficient to produce $>1\text{GW}/\text{cm}^2$ for spots below 50 μm diameter. Peak power decreases to 14 W at the maximum repetition rate of 100kHz [5].

An investigation is underway to examine the deposition characteristics in the plasma and ablation regimes identified. The rock surface will be positioned to produce LIBS plasma (the plasma regime) in one test and the surface will be positioned closer to the instrument where LIBS plasma is not observed (the ablation regime) in a second test. For each test the surface will be continuously translated to make fresh surface continuously available. A fresh instrument window will be used for each test. Laser transmitted power through the window will be measured before and after each test. The window will be examined under microscope, SEM, optical profilometry to estimate the volume of material deposited and mean particle size.

Conclusions: The deposition of material on the instrument window of a micro-LIBS instrument is a necessary step in determining the viability of the micro-LIBS for space exploration. The potential of detrimental obscuration of optical transmission through the micro-LIBS instrument window deserves careful examination.

Acknowledgements: This work supported under NASA grant # NNG04GL90G, ASTID, Dr. Michael New, Science Mission Directorate, Chris Dreyer PI, Colorado School of Mines, Greg Mungas IPI, Firestar Engineering, LLC.

References: [1] R. Hochleitner, N. Tarcea, G. Simon, W. Kiefer, and J. Popp, J. (2004) *Raman Spec.*, 35, 515-518. [2] S. W. Squyres *et al.*, (2006) *Science*, 313, 1403 - 1407, DOI: 10.1126/science.1130890. [3] Y.I. Lee, *et al.*, (1992) *Appl. Spectrosc.*, 46(11), 1597. [4] G.S. Mungas, (2007) *Applied Optics* In Press. [5] C. B. Dreyer, G. S. Mungas, P. Thanh, and J. G. Radziszewski (2007), *Spectrochim. Acta A*, submitted. [6] C. B. Dreyer and G. S. Mungas, LPSC 2007, abstract 2307.