# Laser Induced Breakdown Spectroscopy as an in-space sample return canister sterilization method and instrument


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## Introduction

We are investigating the use of focused laser beams to ablate the surface of spacecraft surfaces and contaminating materials. The method can be applied such that the ablated material is heated to a plasma, making it a laser induced breakdown spectroscopy (LIBS) approach (Figure 1).

Potential benefits include:

- Sterilization in space or on a planetary surface.
- Direct characterization of ablated material.
- Complete vaporization of surface material.

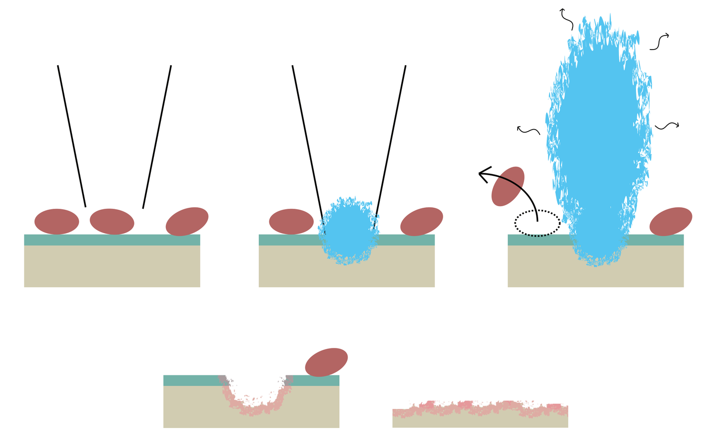

**Figure 1:** Ablation sequence in which a focused pulsed laser generates a plasma. The laser is scanned to remove a surface layer. The extent to which foreign material is transported away from the plasma is being investigated.

Low irradiance lasers (~1 W/cm²) have been used to kill microbes [1][2]. Irradiance >10 MW/cm² [3] will ablate most materials, and for LIBS ~10 GW/cm² [4] is needed. Sub-ablation irradiance can weaken cell membranes causing loss of membrane integrity and produce oxidative species that destroy enzymes and DNA.

## Conceptual Design

In practice the concept may use a robotic arm to scan the laser (Figure 1). Emission from a plasma is collected by a spectrometer. The particular means of implementing the method will depend on the object and mission objectives.

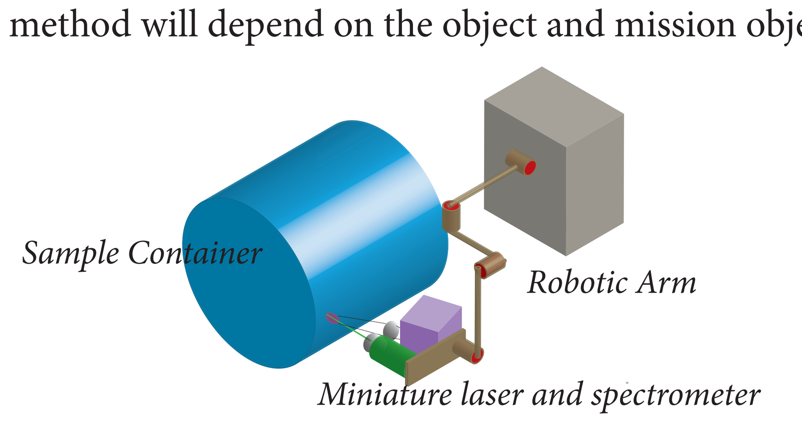


**Figure 2:** Conceptual layout of a device for sterilizing an object.

The laser irradiance has important implications for setting the laser power consumption, laser spot size, scanning rate, and time for sterilization.

The time to fully treat a surface of area ($A$) is: $\tau_s = \frac{A}{a_l f}$
Where $a_l$ is the area treated by the laser per laser pulse and $f$ is the laser repetition rate. To ensure complete sterilization several passes may be necessary.

**Table 1:** Relationship between spot size and laser repetition rate at 10W laser (25% efficiency), 10 GW/cm² irradiance 2 ns pulse, and sterilization area of 1m². $\tau_s$ = 22.2 hours per 1m².

| Spot diameter (μm) | Pulse Energy (mJ) | Repetition Rate (Hz) | |
|---|---|---|---|
| 1000 | 157.1 | 15.9 | |
| 500 | 39.3 | 63.7 | Reasonable for spaceflight |
| 250 | 9.8 | 398.9 | |
| 100 | 1.57 | 1591.5 | |
| 50 | 0.393 | 6366.2 | |

A system using a 10 mJ/pulse laser, a ChemCam-like optical system, and scanning with a robotic arm, would use ~76W. Time for one sterilization pass of a sample return canister 9 cm diameter by 10 cm length (0.028 m²) is 37 min. Sterilization requires ~50 W-hr and 888,000 laser shots per pass.

## Proof of Concept Work

Proof of concept tests have been conducted to show the ability to remove surface contaminantes while simultaneously identify contaminates via LIBS. Materials studied to date are listed in Table 2.

**Table 2:** Materials and contaminates proof of concept tests.

| *Base Material* | *Contaminant* | *Form* |
|---|---|---|
| 316 SS | bare | polished finish |
| 316 SS | biofilm | growth, natural spring |
| 316 SS | JSC-1a <100 μm | thick layer + adhesive |
| 6061 Al | bare | polished finish |
| 6061 Al | Lipopolysaccharide | thin even layer 4.5ng/ml |
| 6061 Al | JSC-1a <100 μm | thin dusting |

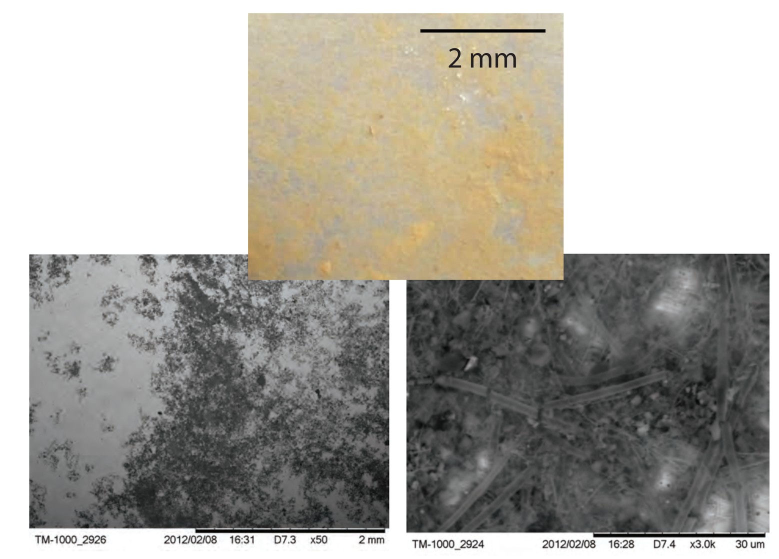


**Figure 3:** Biofilm on 316 stainless steel, photo (top), SEM (bottom).

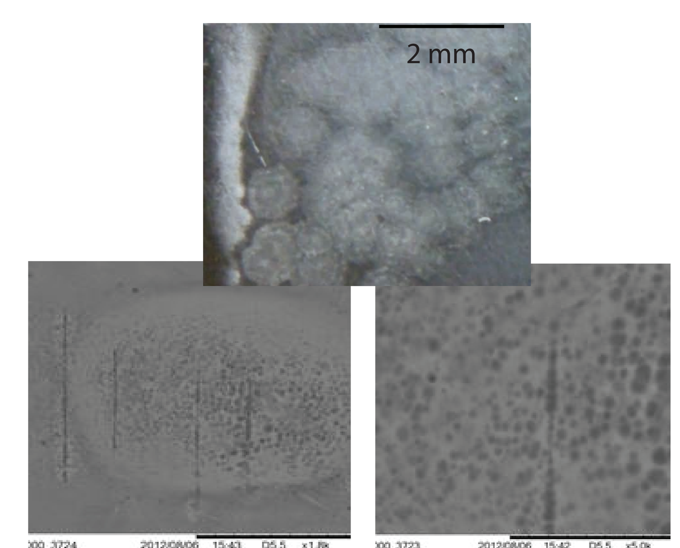


**Figure 4:** Lipopolysaccharide layer on 6061 Aluminum, photo (top), SEM (bottom).

The 1064nm output of a pulsed Nd:Yag laser, 8 ns duration, at 4 Hz, was focused to an elliptical focus of approx. 80 μm width and 4 mm height. The beam was focused into a vacuum chamber containing a 3-axis sample translation stage (Figure 5). Plasma emission was delivered to Ocean Optics miniature spectrometers via optical fibers. Tests were at 7 to 9 torr in a $CO_2$ atmosphere (SS) and air (aluminum).

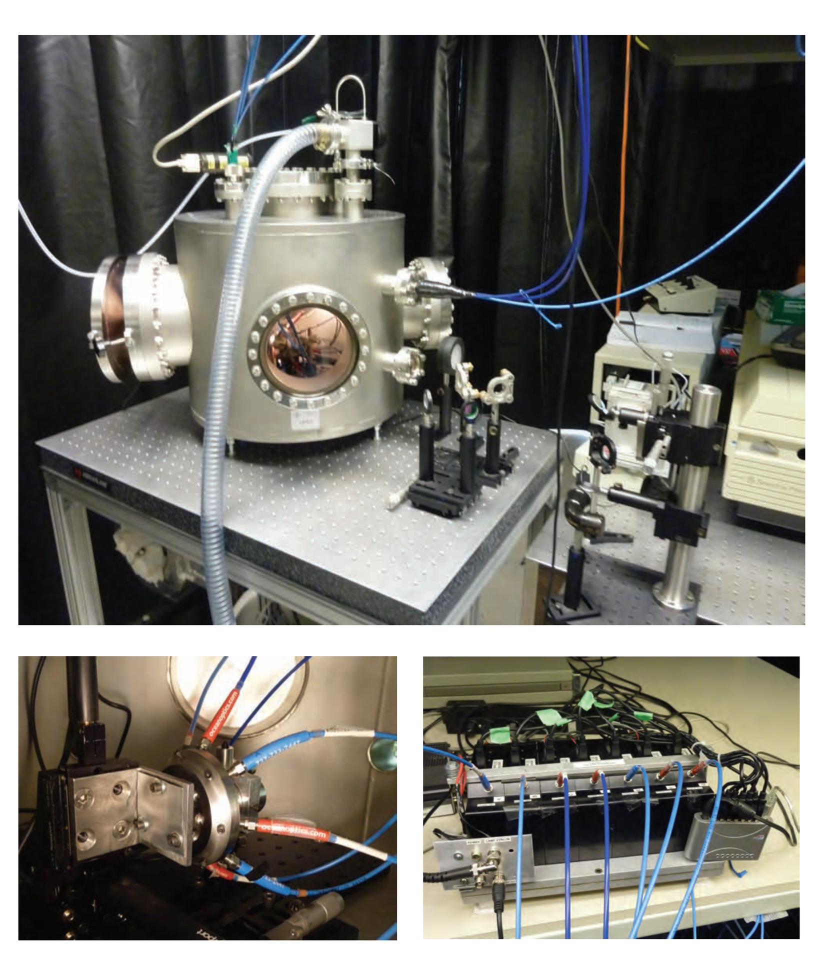

**Figure 5:** CSM LIBS vacuum chamber. Top: overview. Bottom left: chamber interior with sample holder and collection fibers. Bottom right: Ocean Optics spectrometers.

## Results: Ablation

Laser irradiance was set at approx. 10 GW/cm². The sample was translated horizontally, causing the focused beam to sweep out 6 mm in 45 seconds. Images of ablated contaminates are shown in Figure 6, 7, and 8.


**Figure 6:** Laser ablation cleaning of a biofilm from 316 SS. A 4x6 mm area of biofilm was removed, scanning left to right. Left, visible image. Right, SEM image of removed edge.

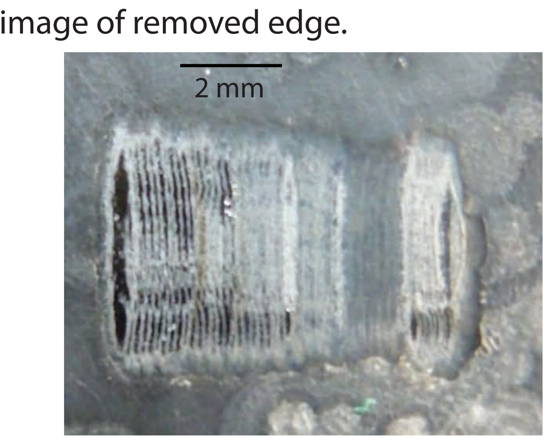


**Figure 7:** Laser ablation of lipopolysaccharide (LPS) on 6061 Al.

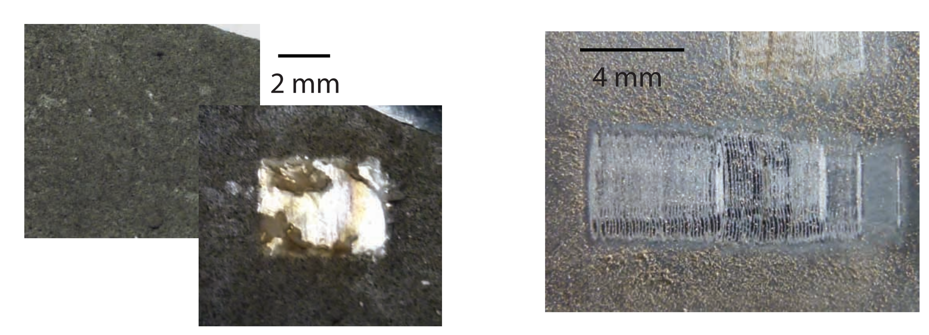


**Figure 8:** JSC-1a, a lunar mare simulant composed of crushed basalt [5], on stainless steel (left side) before and after ablation, held on with spray adhesive. Right side, dusting of JSC-1a on 6061 aluminum after ablation.

## Results: LIBS

The LIBS spectrum contains hundreds of emission lines emanating from neutral and ionized species in the hot plasma. Figure 9 shows the spectra from 200 to 510 nm. Most are from Fe in the stainless steel and JSC-1a.

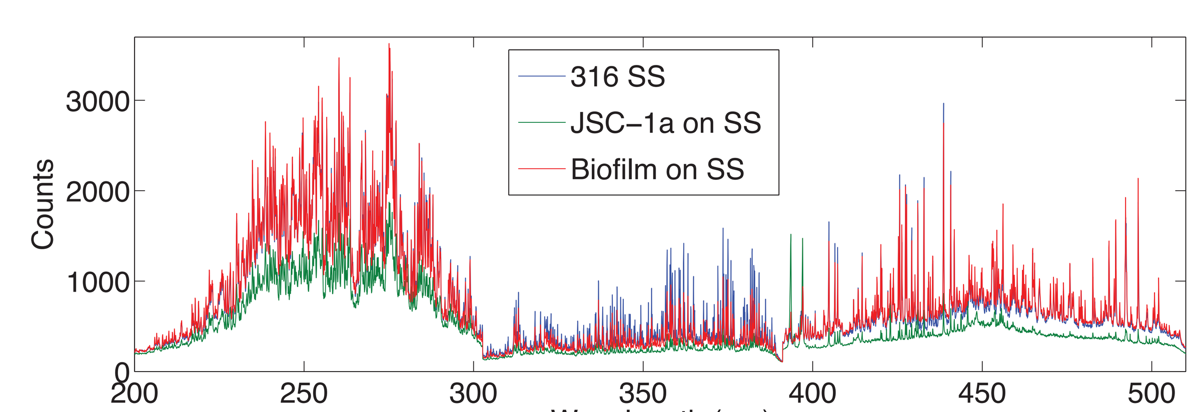


**Figure 9:** UV and portion of visible spectra from the plasma recorded by three Ocean Optics spectrometers.

LIBS can be used to identify the type of surface contaminant. A simple comparison of Fe emission near 392 nm to Ca emission near 393.5 nm (Figure 10) reveal that a biofilm ladened surface can be differentiated from the bare surface and a geological contaminant.

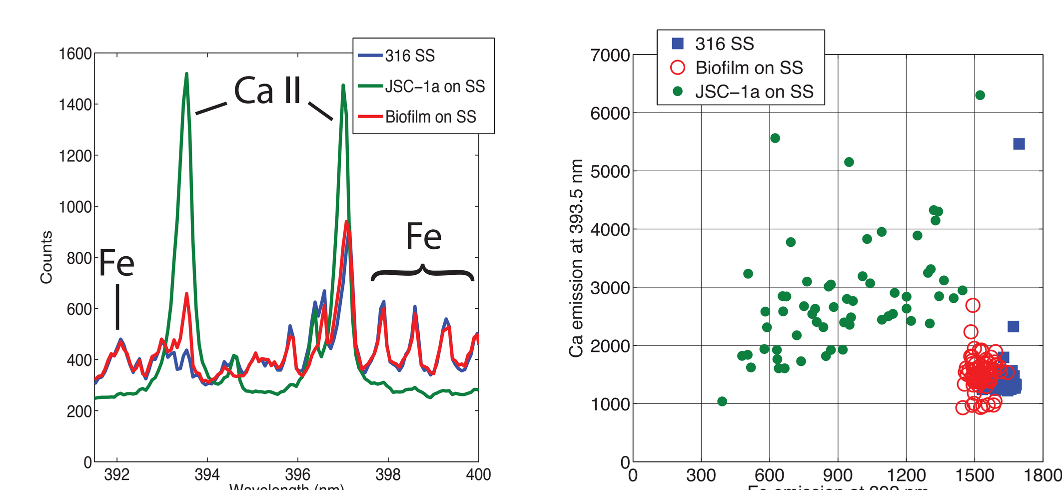


**Figure 10:** Cluster analysis of LIBS data identifies the different samples.

## Results: Aluminum samples

Spectra from 316 SS was dominated by Fe emisison, making identification of emission from other elements difficult. With 6061 aluminum the base metal is less dominate. Evenly distributed bio-layers and thin geological layers produce more consistent pulse-to-pulse spectra (Figure 11).

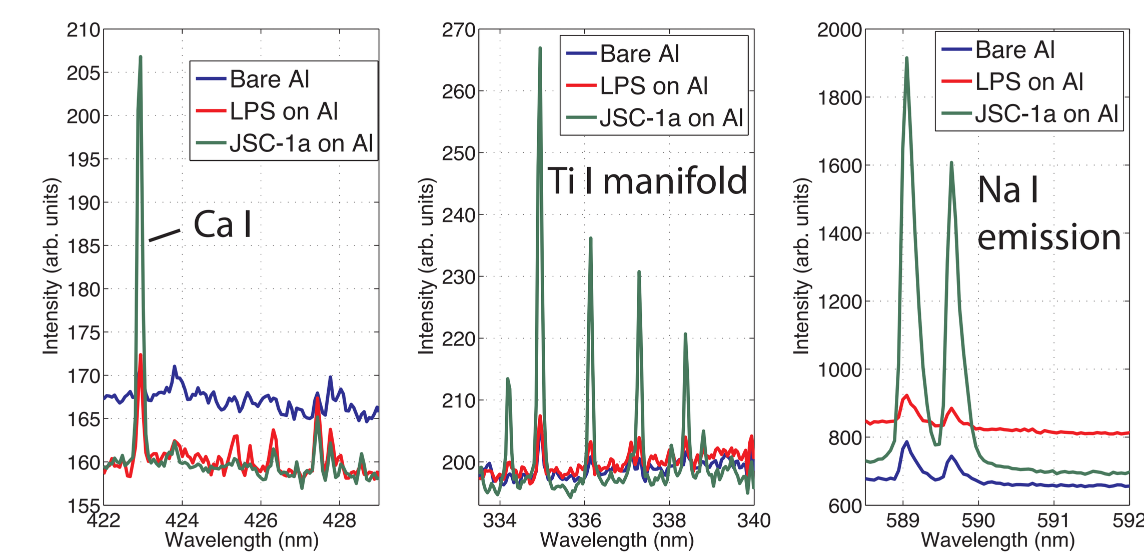


**Figure 11:** LIBS spectra from contaminantes on 6061 aluminum.

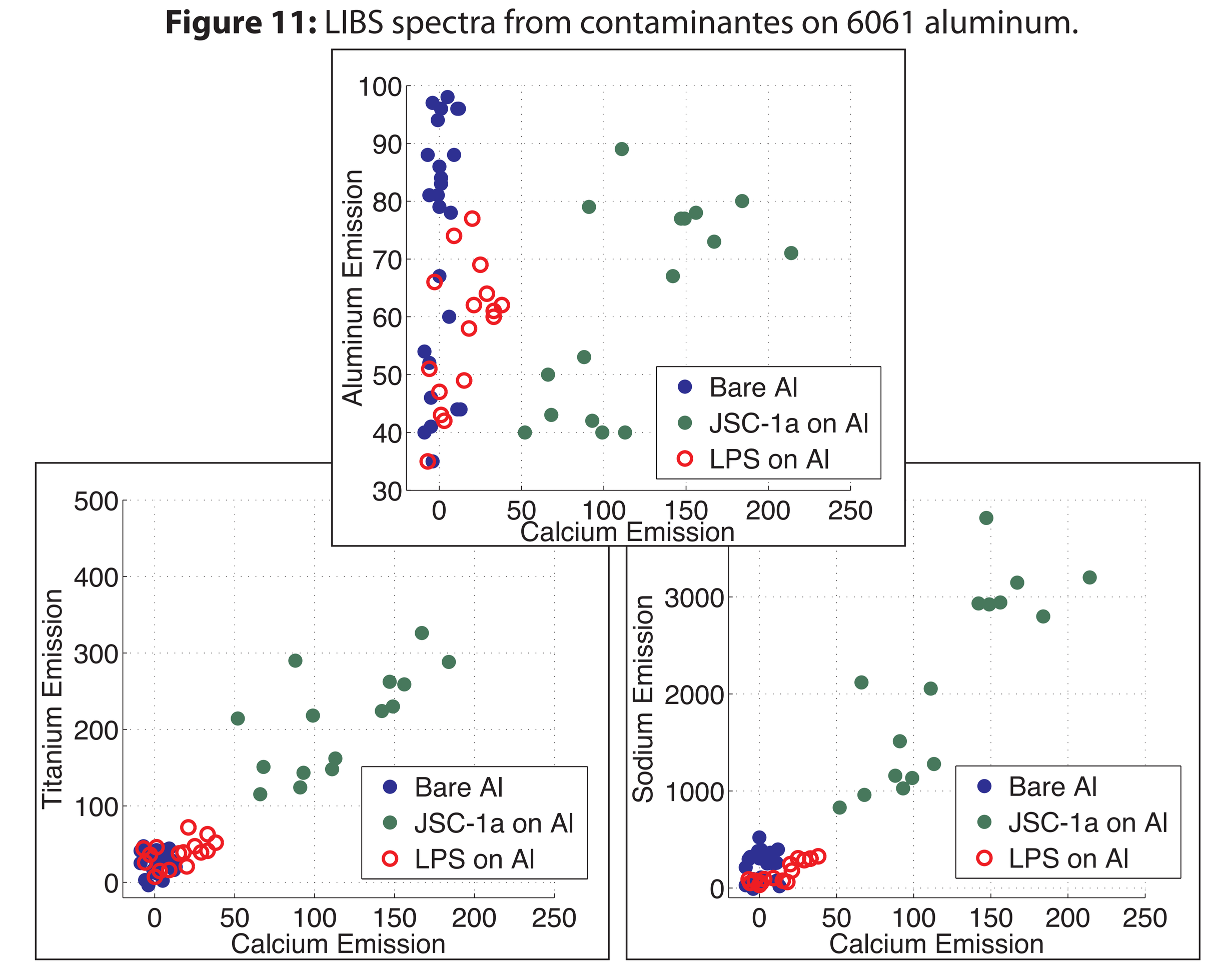


**Figure 12:** Cluster analysis of LIBS spectra of contaminantes on 6061 Al.

Cluster analysis of aluminum samples using Ti, Ca, and Na emission shows that the three groups can be identified (Figure 12). More work is needed to eliminated collection misalignment, improve sample cleanliness, and spectrometer timing need to be optimized.

## Conclusions

Contaminantes can be removed from a surface via laser ablation with simultaneous identification of the ablated material via LIBS. Areas of future work are:

- Test with a greater variety of substrates and microbes.
- Determine microbe viability after full sterilization of a surface.
- Examine microbe transport during ablation.
- Test with a tightly focused and low pulse energy beam, which is more likely for a space flight application, and optimize analysis.
- Tests of LIBS and ablation at very low pressure (more similar to an in-space application) which is known to dramatically decrease emission intensity [6][7].

## Literature Cited


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