An experimental investigation of the capabilities of LIBS for the elemental analysis of simulated Corroded Magnox Sludge (CMS) samples

A Nuclear Decommissioning Authority (NDA) funded programme has been sanctioned to address the *in-situ* / less intrusive analysis of intermediate level waste sludges and associated liquors. Following an extensive review of UK Nuclear Licensed Site Waste Streams, along with techniques capable of analysing particular elements, Laser-Induced Breakdown Spectroscopy (LIBS) has been highlighted as a preferred method.

This work was conducted by APL on behalf of Nexia Solutions Ltd on establishing the typical analytical measurement performance of LIBS for the elemental analysis of simulated Corroded Magnox Sludge (CMS) samples. It was established that LIBS could detect the majority of elements of interest within the CMS samples and that calibration could be achieved to allow semi-quantitative measurements.

The LIBS apparatus used during this work is illustrated in figures 1 to 4. The apparatus is based on a proprietary design of APL. The main components of the apparatus are as follows:

- A Q-switched Nd:YAG laser (model Brilliant, Quantel SA) operating at its fundamental wavelength of 1064 nm
- The LIBS head incorporating a laser beam delivery optical system combined with a dual-channel plasma light collection optical system (on-axis and off-axis, as illustrated in figure 4)
- A support frame and rotating mount for the LIBS head which allows the laser beam to directed at any angle between vertical and horizontal
- Optical spectrograph #1 - a 0.5 m focal length, triple-grating spectrometer (model SP500, Acton Research Inc.) incorporating an Intensified Photo-Diode Array (IPDA) detector (model IPDA-1024, Princeton Instruments Inc.)
- Optical spectrograph #2 - a 0.75 m focal length, triple-grating spectrometer (model SP750, Acton Research Inc.) incorporating an Intensified Photo-Diode Array (IPDA) detector (model IPDA-1024, Princeton Instruments Inc.)
- Two personal computers running WinSpec 32 (ver. 2.5.1) software for controlling the two spectrographs
- A fumehood (model AirOne FC-640, Safelab Systems Ltd) equipped with HEPA and activated charcoal filters
- A sample holder integrated with a 2-axis linear translation stage (model Nanostep, Melles Griot)

The laser beam pulse emitted from the output aperture of the LIBS head can be varied between 0 mJ and approximately 200 mJ. The pulse length is fixed at approximately 4 ns while the pulse repetition rate may be varied up to 20 Hz. The pulse energy incident on the sample was measured using a calibrated energy meter (model LPM 230-2, Litron Lasers Ltd). The angle of incidence of the laser beam with respect to the sample surface was set to approximately 50 degrees (i.e. 50 degrees from the horizontal). The samples were located inside the fumehood using a sample holder attached to the 2-axis linear translation stage (with the translation stage outside of the fumehood as illustrated in figures 1 to 3). The focal length of the laser beam emitted from the output aperture of the LIBS head was set to approximately 430 mm. The surface of the sample was located as illustrated schematically in figure 5. The spot size at the sample was estimated (using burn paper imprints) to be 0.7 x 1.0 mm which equates to an area of approximately 0.007 cm^2 (estimated uncertainty of ±20%).

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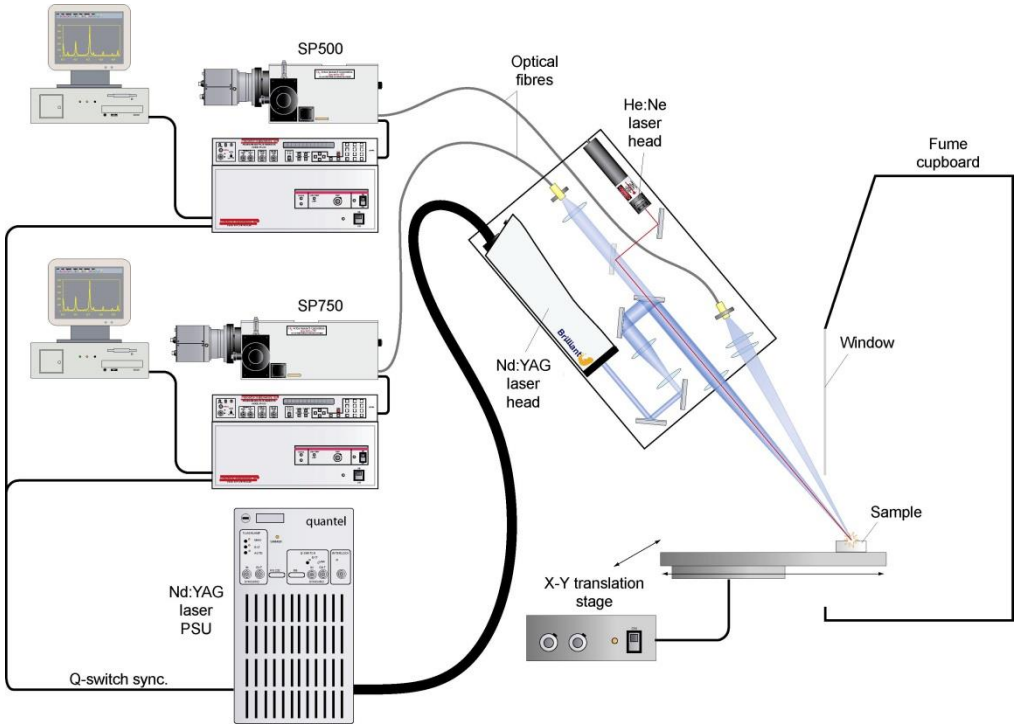
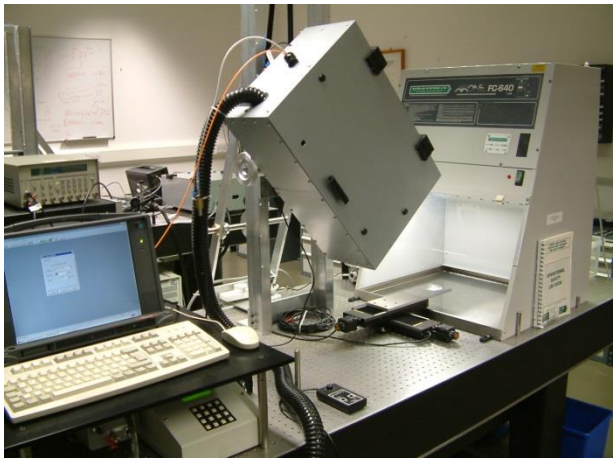


Schematic of experimental apparatus



LIBS apparatus used to analyse CMS samples

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