

L.2: Development of laser induced breakdown spectroscopy setup

Laser-induced breakdown spectroscopy (LIBS) is a versatile element analysis technique, which allows simultaneous multi-element analysis of solid, liquid or gaseous samples in an ambient atmosphere without any prior sample preparation, analysis of low Z elements where x-ray based techniques do not work, and also for analysis of samples placed several meters away. Due to these capabilities, this technique has potential applications in areas such as industrial process monitoring, contamination analysis, geochemical exploration, hazardous material identification, etc.

A LIBS setup has been developed using a Q-switched Nd:YAG laser (6 ns, 10 Hz, 450 mJ) to generate plasma and a Czerny-Turner spectrometer-CCD to detect plasma emission. The spectrometer covered a spectral range of 200-815 nm with a resolution varying from 0.1 nm to 0.18 nm. The spectrometer signal acquisition is triggered with some delay with respect to laser pulse to allow the plasma continuum emission to decay and characteristic line emission to dominate. Two types of setups have been developed. In one setup (Proximity LIBS, Figure L.2.1(a)), the sample is placed close (<100 mm) to the laser focusing optics as well as the signal collecting optics, and in the another setup (Remote LIBS, Figure L.2.1(b)), the sample is placed at ~3 m away from the laser focusing optics and ~6 m away from the signal collecting optics. For the Proximity LIBS setup, a 100 mm lens was used to focus the laser beam on the sample. Optical fiber placed at 45° with respect to the laser direction at a distance of ~10 mm from the sample, collected and coupled the LIBS signal into the spectrometer. For the Remote LIBS setup, the laser beam is expanded and focused using a combination of -25 mm and 100 mm lenses and the LIBS signal is collected by using a 4” aperture Cassegrain telescope and focused on an optical fiber.

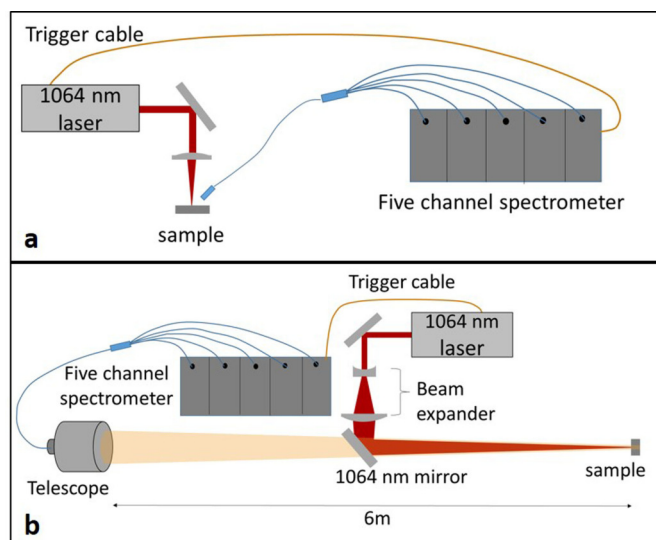


Fig. L.2.1: Schematic of experimental configurations for (a) Proximity LIBS setup, and (b) Remote LIBS setup.

Using these LIBS setups, analysis of alloys such as stainless steel, Cu-Zn alloy, Ti alloy, and rock, soil and water samples has been performed. AISI 304 and AISI 316 grades of stainless steel could be distinguished on the basis of presence or absence of Mo peak at 553.3 nm (Figure L.2.2(a)). It was also possible to distinguish between different rock samples on the basis of relative concentration of different elements present in them using a multivariate element analysis technique i.e., principal component analysis (PCA) (Figure L.2.2(b)). The Proximity LIBS setup could also be used to quantitatively analyse the elements present in the water samples. For this, cellulose papers were soaked in different water samples viz., underground water, Narmada water and lab tap water, and subjected to LIBS analysis when dried. Underground water was found to consist of the highest value of common mineral elements such as Ca, Mg, K and Na along with Sr. At present, the concentration of Sr was estimated (Figure L.2.2(c)) using calibration curve method, and found to be ~23 ppm in the underground water sample, which was more than five times the reference level of 4 ppm advised by US, EPA for drinking water.

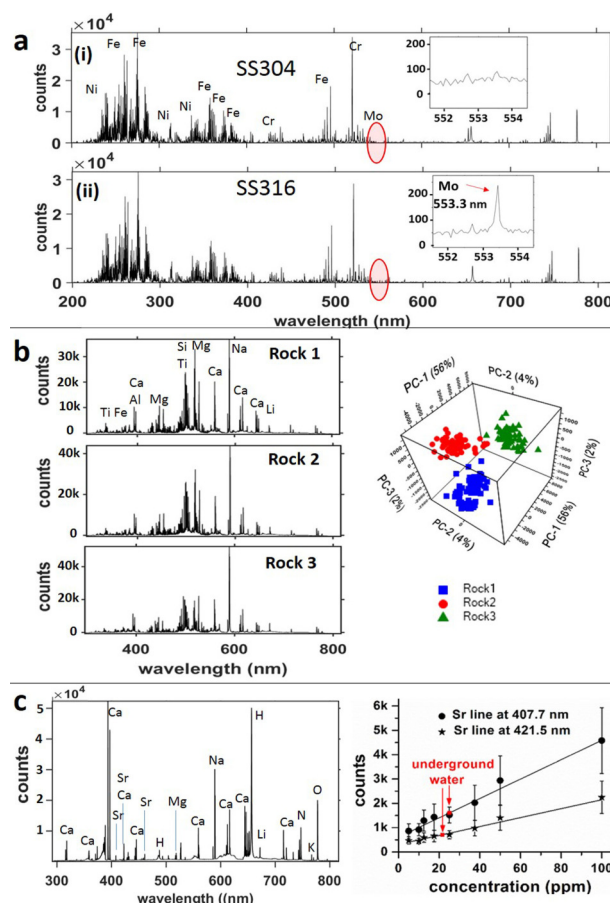


Fig. L.2.2: (a) Proximity LIBS spectra of SS304 and SS316 and inset shows the presence or absence of Mo peak, (b) Remote LIBS spectra of different rock samples and their PCA analysis, (c) Proximity LIBS spectrum of underground water and Sr concentration analysis.

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