Development of Laser Induced Breakdown Spectroscopy at ACRHEM for Applications Relevant to High Energy Materials

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ABSTRACT

In this paper the results of Laser-Induced Breakdown Spectroscopy (LIBS) on ambient air, Boron Potassium Nitrate (BPN) and cyclotrimethylenetrinitramine (RDX) studied using 532 nm, 7 ns and 800 nm, ~40 fs laser pulses are presented. The LIBS spectra of RDX demonstrate emission corresponding to CN and C_2 bands. The LIBS spectra of BPN illustrate strong emission corresponding to K.

1. INTRODUCTION

The potential of LIBS for stand-off detection is attractive particularly for High Energy materials [1–2]. The ability to detect potential hazardous and explosive materials from a distance can not only save precious time at places like airports but also can save precious lives in battle like situations. LIBS is an analytical technique which involves the interaction of a target with an intense laser pulse followed by emission of light [3–5]. This technique has applications in diverse fields including combustion [4] and detection of High Energy Materials (HEMs) [1]. LIBS has a number of advantages that makes it attractive for the detection of high energy materials, including stand-off detection capability, the need for small amount of the material, and fast detection speed. LIBS studies on number of explosives including TNT, RDX, HMX, and PETN have been reported [6–7]. Most of these materials are composed of hydrogen, carbon, oxygen, and nitrogen enabling the task of identifying one material from the other a complicated issue. Moreover, contribution from the ambient air to the signal at same wavelength(s) makes the task still more difficult. It has been shown that it is possible to reduce the affects of ambient air by using dual pulse technique [8].

The ultimate challenge and dream of any spectroscopist would be to achieve discrimination between given HEMs. A detailed statistical analysis involving ratios of various peaks combined with different chemometric techniques and possibly the time evolution of the ratios could perform this kind of discrimination. The main endeavor of our research group at ACRHEM is to develop the LIBS technique to probe various HEMs, including their detection and combustion studies, using nanosecond and femtosecond laser pulses.

2. EXPERIMENTAL

In our experiments we have used two different lasers (a) Second harmonic of a Nd :YAG laser at 532 nm, 7 ns, 10 Hz with 10 mm spot size and (b) 800 nm, 40 fs, 1 KHz with 10 mm spot size. In the former case the maximum energy was 40 mJ/pulse while in the latter case it was 2.5 mJ/pulse. The laser was focused using an 80 mm lens for ns pulses and 25 cm lens for fs pulses. At sufficient laser energies we can observe light and sound coming from the focal region where the sample is kept on a translation statge. The LIBS signal is collected using a lens placed 30° to the propagation direction, which in turn is connected to a fiber and a spectrometer (USB 4000 from Ocean Optics). Schematic of typical LIBS experimental set up is shown in Figure 1.

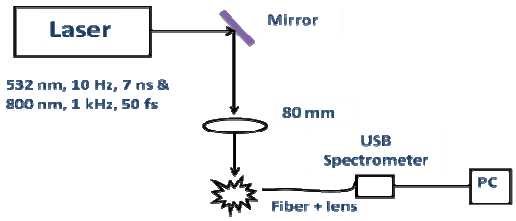


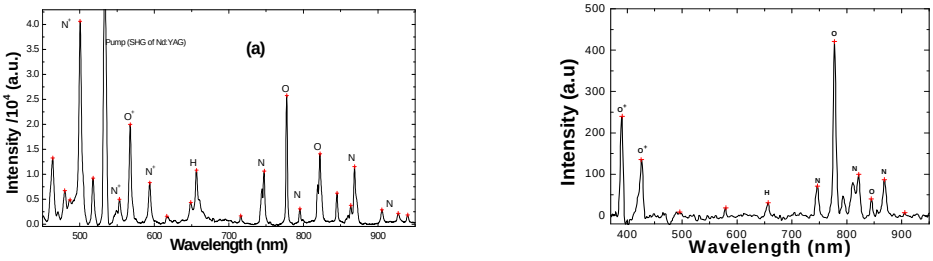
Fig. 1: Schematic of the Experimental Setup Used for LIBS

3. RESULTS AND DISCUSSION

The LIBS spectrum of the ambient is recorded since its contribution to the signal cannot be completely ruled out in the applications related to HEMs. Figure 2 shows the LIBS spectrum of ambient air. The background correction was performed using Matlab 7. The background was less prominent with femtosecond pulses. The prominent lines are at 777 nm from oxygen, 747 nm and 822 nm from nitrogen, 656 nm from Hydrogen.

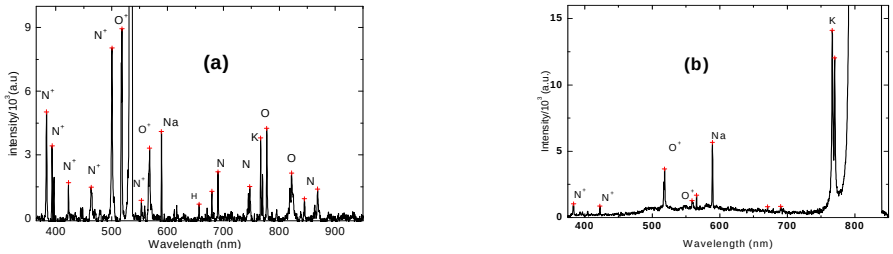
It is important to record the air spectrum each time LIBS spectrum is recorded as various factors may affect the constituents, like humidity will affect the presence/absence of the hydrogen line. Though it is not risky working with small amounts of HEMs, one needs to exercise caution while working with them as they are sensitive to friction and impact. In our experiment, only a very small area of the sample of the dimensions of the few tens of microns containing micrograms of HEM is exposed to the laser pulses. Figure 3 shows the LIBS spectrum of bare BPN. BPN belongs to the class of primary explosives and is used as an initiator in the ignition of rocket motors, air bags and decoy flares. The potassium

lines can be seen at 770.05 nm and 766.65 nm. The nitrogen lines are seen at 822.1 nm, 868.61 nm and Oxygen at 777.57 nm and 844.88 nm. We observe an increase of the strengths of these lines when compared to the ambient air alone and KBr alone.



(a) (b)

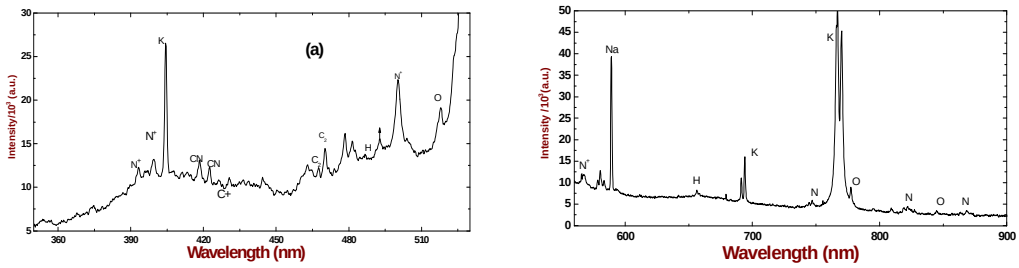
Fig. 2: LIBS Spectrum of Ambient Air with (a) Nanosecond and (b) Femtosecond Pulses



(a) (b)

Fig. 3: LIBS Spectrum of BPN Obtained Using
(a) Nanosecond and (b) Picosecond Laser Pulses

RDX is an explosive and is one of the very widely used materials both in defense and industrial applications. It is generally mixed with other explosives such as TNT and HMX. Figure 4 shows the LIBS spectrum of RDX without background correction. The weighed quantities of samples and KBr are ground in agate mortar to ensure the uniform mixing. The mixture is taken in a mould and pressed with 3 tons pressure to make pellets of 12 mm radius. We observe CN peaks near 420 nm and C_2 peaks near 470 which are attributes of nitro compounds [9]. N^+ peak at 462.33 nm was observed only in RDX sample while it was clearly absent in the KBr spectrum. Similarly, H_α (486.6 nm), N (868.43 nm) peaks were exclusively present in RDX spectrum. H (656.6 nm), O (777.38 nm, 845.6 nm), N (742.36 nm, 744.64 nm, 747.12 nm) and N (822.1 nm and 868.43 nm) Na (589.11 nm) peaks exhibited enhanced intensity in the RDX + KBr spectrum compared to pure KBr spectrum. We have recently acquired an Michelle spectrometer mounted on an ICCD (ANDOR DH-734). A stainless steel chamber is being designed where all t he future experiments will be performed.



(a) (b)

Fig. 4: LIBS Spectrum of RDX (a) 350–525 nm (b) 525–900 nm Obtained with Nanosecond Pulses

4. CONCLUSION

LIBS experimental set up to study various HEMs is set up at ACRHEM, University of Hyderabad. We have reported the LIB spectrum of two HEMs - BPN and RDX recorded with a non gated spectrometer. Less number of lines were observed when the spectra was recorded with fs pulses. A more detailed experiment with gated spectrometer will enable us to reveal more information.

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