Application of an Echelle spectrometer

for 2D mapping of aluminum alloy surfaces using MicroLIBS

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Application Note

Introduction

Laser-induced breakdown spectroscopy (LIBS) is a material characterization technique that uses the optical emission from a laser-induced plasma [1 - 3]. A new area called µLIBS [4 – 8] uses pulse energies less than 100 µJ, which allows the probing of smaller target spots, reduction of damage to targets and the use of fiber or microchip lasers.

Single shot detection and identification of precipitates in aluminum alloys has recently been demonstrated for femtosecond pulses down to 7 µJ [9]. Below this energy, reliable signals from the minor constituents which make up precipitates are not observed.

This work investigates the application of an echelle spectrometer to 2D mapping of surfaces via the µLIBS technique. Simultaneous observation of the µLIBS plasma by a echelle spectrometer system and a Czerny-Turner spectrometer is performed, and a detailed comparison of the sensitivities, noise floors and overall performance of the two systems is performed.

Experimental Setup

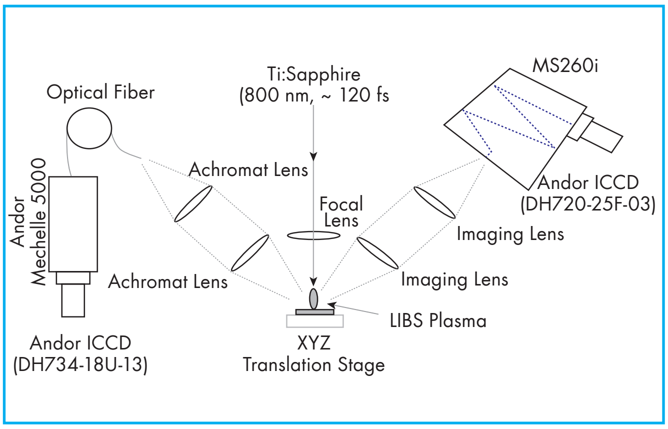

Figure 1: A schematic view of experimental setup. The plasma is observed simultaneously by both spectrometers at an angle of 45 degrees from the incident laser beam.

The experimental setup is given in Fig. 1. The µLIBS plasma is observed at 45 degrees from the laser axis for both a f/3.9 Czerny-Turner spectrometer (Oriel MS260i) and an f/7 echelle spectrometer (Andor Mechelle 5000). A Ti:Sapphire laser (Spectra Physics Hurricane) produced 800 nm, 120 fs pulses with a maximum pulse energy of ~ 80 µJ on target.

The laser is focused using a 15X Schwarzschild objective. An 50 µm diameter optical fiber (Ocean Optics Q50-2-UV/VIS) is used to couple the plasma emission into the echelle spectrometer and define the entrance aperture of the echelle.

The echelle was coupled with a ICCD (Andor iStar DH734-18U-13) with an enhanced response from 500 nm to 800 nm, while the Czerny-Turner was coupled with a ICCD (Andor iStar DH720-25F-03) with an enhanced response from 200 nm to 500 nm. The quantum efficiencies and gain characteristics for both detectors are given in Fig. 2. The gate delays used ranged from 2 to 5 ns, gate width was 1 µs and both ICCDs were operated at -10 C. The ICCD on the Czerny-Turner spectrometer was used in full vertical binning mode with a pixel readout time of 16 µs, while the ICCD on the echelle was operated in imaging mode with a pixel readout time of 1 µs.

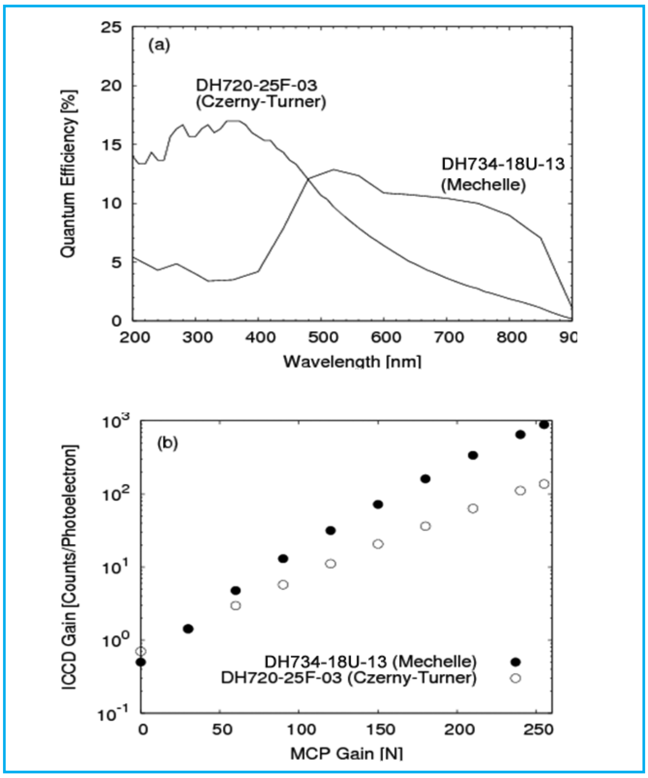

Figure 2: Quantum efficiency (a) and gain (b) for the ICCD detectors used in these experiments

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Both spectrometer/ICCD combinations were calibrated for absolute photon flux using a number of different sources. The sensitivity of both systems at maximum gain is given in terms of counts/photon in Fig. 3a. This figure includes the effects of the spectrometer and ICCD, but not the optical fiber used to couple the light into the echelle. The pixel integration time used for the echelle ICCD was 1 µs, while the Czerny-Turner ICCD was used at 16 µs. It is expected that the gain reported here for the echelle would increase by about 40% if a pixel integration time of 16 µs was used. The noise equivalent power (NEP) of the two systems was investigated by characterizing the standard deviation of spectra obtained when the entrance slit of the spectrometer was shuttered at different gains. The area observed by the echelle is well defined by the aperture of the input fiber, and the effective slit height of the Czerny-Turner system was estimated at 500 µm based on plasma imaging experiments. Solid angle and spectral width of the systems were taken into account for the NEP presented in Fig. 3b.

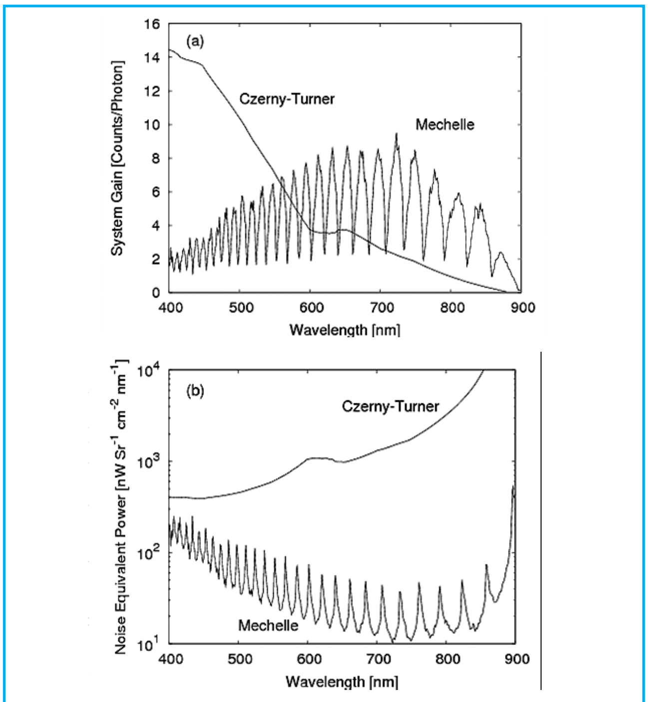

Figure 3: (a) Spectrometer/ICCD sensitivity in counts per photon and (b) NEP at maximum gain for the systems used here. A pixel readout time of 16 µs would be expected to increase Mechelle/ICCD gain about 40% for all wavelengths.

A final comparison between the two systems is presented in Fig. 4 and uses a figure of merit (FOM), defined as:

$$FOM = \frac{1}{NEP}$$

The results of the FOM comparison indicate that the Mechelle is capable of operating at performance levels at or above that of a Czerny-Turner. However, it is clear from the detector characteristics presented in Fig. 2 that the Mechelle ICCD has a significantly higher gain. In addition, the rate of cathode noise spikes is significantly lower for the ICCD used with the Mechelle. This has a large effect on the NEP and FOM values quoted as the analysis used includes all spikes in the calculations. An echelle spectrometer has the advantage of using fewer pixels for a given signal than would be the case in a Czerny-Turner spectrometer. As a result, echelle spectrometers will be less sensitive to the noise spikes that affect the Czerny-Turner NEP results.

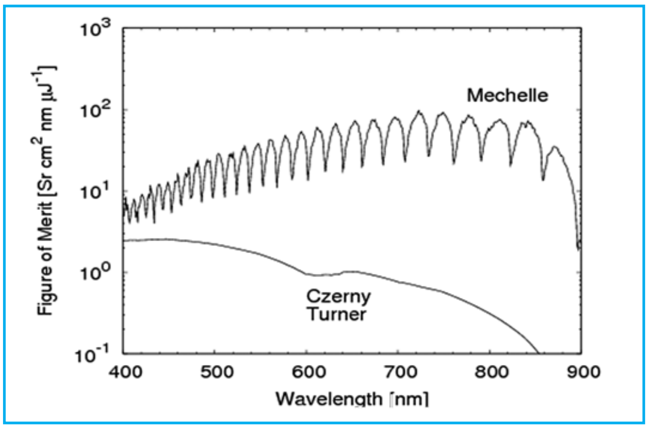

Figure 4: 'Figure of Merit' for both spectrometer/ICCD combinations.

Combining the facts of the higher gain and reduced noise of the Andor DH734-18U-13 ICCD, it is clear that the Czerny-Turner performance could be considerably improved with an improved detector. Further work will be required to eliminate these effects from this comparison.

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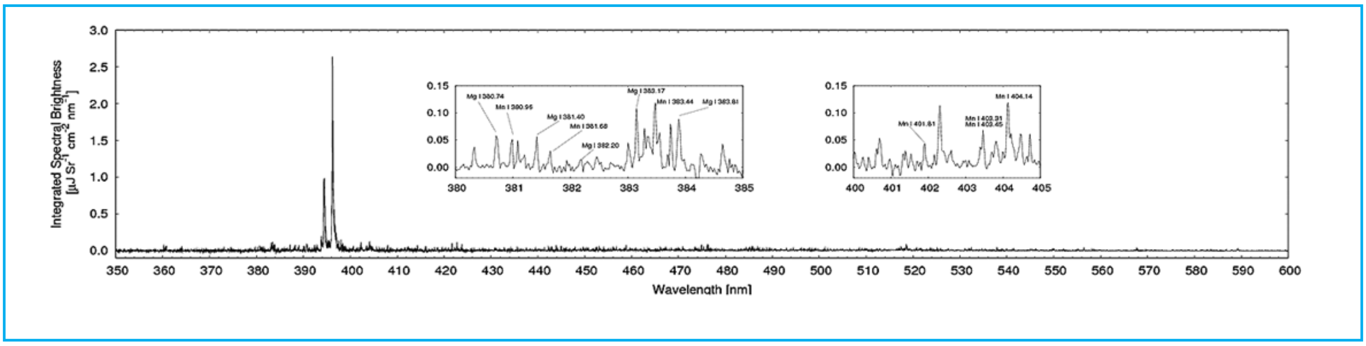

Figure 5: Mechelle spectra from Al2024 alloy, using a single 29 µJ, 800 nm pulse. Current spectral width is limited by use of achromatic imaging lenses. Insets are regions relevant to detection and identification of precipitates [8,9]. Outside these regions are many unidentified lines that aren't used in the current analysis. Much more work will be required to make full use of the information available.

Experimental Results

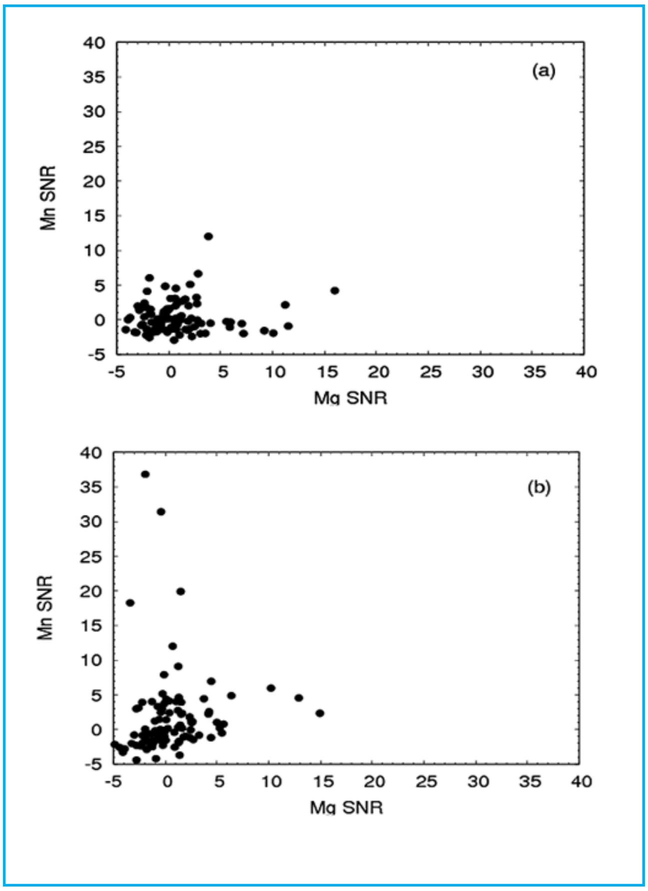

Figure 6: Correlation plot of Mg and Mn SNR from 4 µJ shots on Al2024 alloy for (a) Czerny-Turner spectrometer (2 Lines) and (b) Mechelle spectrometer (12 lines).

One of the key advantages of an echelle spectrometer is the extremely broadband spectrum that may be acquired. A sample spectra from a 29 µJ shot on a sample of Al2024 alloy is presented in Fig. 5 (top right of poster). The spectral width presented is limited by the achromatic imaging lenses used to image the plasma light onto the optical fiber. Reflective optics would allow viewing of a broader range.

The insets are regions which are relevant to the detection and identification of precipitates in aluminum alloys [8,9]. Using the Czerny-Turner spectrometer only two unresolved doublets are visible [8,9]. Following the procedure outlined in [9], the spectra were analyzed for Mg and Mn SNR. Fig. 6a gives the results of the analysis of the Czerny-Turner observations using the current setup for 4 µJ pulses on Al2024 alloy. The same analysis was performed using the spectra observed by the Mechelle for the same 4 µJ shots, but using all of the lines identified in the insets of Fig. 5. The results of this analysis is presented in Fig. 6b.

The use of the multiple lines available with the Mechelle improves the detection of Mn precipitates. Combined with the detection of Mg precipitates, this result indicates that single shot 2D mapping of Al2024 surfaces would be possible at 4 µJ. Many more lines are available for analysis, and it may be possible to improve the performance of the echelle spectrometer for 2D mapping beyond that reported here.

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Conclusions

The absolute response, NEP and overall performance of a Czerny-Turner and an echelle spectrometer have been quantified. The echelle spectrometer is competitive with the Czerny-Turner, though the performance of the Czerny-Turner is limited by the ICCD used. µLIBS has been demonstrated with an echelle spectrometer at energies less than 10 µJ. The broadband, high resolution spectra acquired with the echelle allows the use of additional lines which were not observed with the Czerny-Turner. These additional lines improved the performance the analysis technique used here to detect and identify aluminum precipitates. Many more lines are available for analysis, and it may be possible to further improve the performance of µLIBS by the use of an echelle spectrometer.

Acknowledgements

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