Aluminium thin films depth profiling using LIBS

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Abstract. Laser Induced Breakdown Spectroscopy (LIBS) is an analytical technique used to classify and potentially quantify elements in complex hosts (or matrices) [1,2]. In this study, silicon based aluminium thin films were developed to study the depth profile and ablation rate of the material. Five films with different thicknesses from 1mm to 1.5 micron were used. The experimental setup consisted of a single pulse system with a Nd:YAG laser (1064 nm, up to 450 mJ, pulse duration 6 ns) used to irradiate the samples, an optic fibre spectrometer was used to detect the spectrum. The results show low ablation rate with time integrated method.

1. Introduction
Depth Profiling is a process where the element or chemical content of a sample is measured as a function of depth [3]. There are various techniques used for thin films depth profiling which include Secondary Ion Mass Spectroscopy (SIMS), Auger Electron Spectroscopy (AES), Glow Discharge Optical Emission Spectroscopy (GD-OES) and Laser Induced Breakdown Spectroscopy (LIBS) [3, 4,5]. These techniques have been used for many applications including solar cell analysis, electronic parts analysis and nanostructures analysis [6,7,8] In this research Time Integrated LIBS is used at a power density of 1.6x10⁹ Wcm⁻² to determine the ablation rate of the silicone based aluminium thin films.

2. Experimental Setup
The experiments on depth profiling of metal targets were performed using a simple UV LIBS system. A commercially available Nd:YAG Laser Surelite Model III-10 (pulse width 6.0 nanosecond, wavelength 1064 nm, and energy 730 mJ) was used to irradiate thin aluminium metallic films with focused laser pulses in air. Figure 1 represents the schematic of the experimental setup for the UV LIBS system. The laser pulses were focused on to a 40 µm spot on the metal target, using a specially designed optical system. This optical system was composed of two focusing mirrors, half-wave plates, polarizers, and a focusing lens. The first mirror M1 at an angle of 45 degrees was used to transmit the laser beam to the half-wave plate HWP1 and polarizer P1, this combination was used to maximize the energy at 700 mJ to get maximum polarization and a good profile. The second mirror M2 was again angled at 45 degrees to transmit the light to a second half wave plate HWP2 and polarizer P2, this combination was used to control the energy at the 1 mJ level. A plano-convex lens with a focal length 150 mm was used to focus down the spot size to 40 µm diameter. A power density level of 1.6x 10⁹ Wcm⁻² was used which was achieved by manipulating the optical system and by the setting of half waveplate. The system was used in the spatially-resolved, time-integrated (over each laser shot) mode.

The laser was used at a repetition frequency of 10 Hz. The laser pulses irradiate the aluminium thin film metallic target surface which resulted in the formation of plasma. The plasma radiation formed at
the target were collected by using two 5 cm diameter biconvex lenses (L2 and L3 as shown in figure 1) and then delivered to an ANDOR Technology ICCD detector through a Shamrock SR-163 spectrometer based on a Czerny-Turner optical layout. The spectrometer had a focal length of 163 mm and a f/3.6 numerical aperture. This configuration enabled higher throughput with better resolution. The data from the spectrometer were transferred to a PC for analysis with the commercially available software, MATLAB.

![LIBS experimental setup](image)

**Figure 1.** LIBS experimental setup.

Five metallic thin films of different thicknesses, 1.5 µm, 15µm, 30µm, 45µm and 1 mm, were used. The aluminium thin films were based on a silicon substrate. The laser was shot on all five samples. The ablation count was taken as the total number of lasers shots required to burn through the film onto the silicon substrate, as evidenced by the disappearance of aluminium lines from the spectrum. The ablation rate was calculated for each sample.

### 3. Results and Discussion

The experiments were performed using a laser power density of $1.6 \times 10^9$ Wcm$^{-2}$. Figure 2 (A) shows the Al intensities for Al thin films with different thicknesses. As the experiments were conducted with a low power density, the 1 mm film did not show complete ablation even after 160 shots, whereas for the 45 µm thick film, after 74 to 77 laser shots the aluminium intensity had dropped down to near to zero. The 30 µm thin film aluminium was completely ablated after 50 to 59 laser shots. For the 15 µm thick film, the aluminium intensity was significantly dropped after only 22 laser shots. Finally, for the 1.5 µm thin film, the aluminium was ablated completely after 3 shots.

![Graphs showing Al and Si intensities](image)

**Figure 2.** The intensity of Aluminium (A) and Silicon (B) emission using time integrated UV LIBS.

Due to low laser power density, the silicon intensity was difficult to be acquired adequately. Figure 2 (B) shows the recorded intensity response of the silicon substrates.
Figure 3 shows the aluminium ablation count as a function of film thickness. The ablation rate calculated for this low power density using the results obtained from the experiments was found to be 0.51-0.66 µm/shot.

![Ablation Rate](image)

**Figure 3.** Ablation count (number of laser shots required for complete ablation of aluminium thin films from silicon substrates) as a function of film thickness.

4. **Conclusions**
We have used a time integrated, single-shot technique to determine the ablation rate of thin films, for applications of LIBS in depth profiling of aluminium. At a power density 1.6x10^9 Wcm^-2 the ablation rate was found to be 0.51-0.66 µm/shot. It was also observed that the usual Al spectrum, with a high signal to noise ratio, was typically only recorded for the first 10 to 24 shots. Initial results are very encouraging for further investigation of the LIBS geometry and also to understand the plasma properties, laser material coupling and LIBS emission.

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**References**

[1] Jiang X, Hayden P, Laasch R, Costello JT and Kennedy ET 2013 *Spectrochim. Acta B.* **86**, 66
[2] Noll R 2012 *Laser Induced Breakdown Spectroscopy Fundamentals and Applications*, Springer
[3] Dudragne L, Adam Ph and Amouroux J 1998 *Appl. Spectrosc.* **52**, 132
[4] Messaoud Aberkane S, Abdelhamid M, Yahiaoui K, Mahieddoune C, Abdelli-Messaci S and Harith MA 2018 *Thin Solid Films*, **653**, 293
[5] Payling R, Aeberhard M and Delfosse D 2001 *J. Anal. At. Spectrom.* **16**, 50
[6] Aragon C, Madurga V and Aguilera J 2002 *Appl. Surf. Sci.* **197**, 217
[7] Novotný K, Vaculovič T, Galiová M, Otruba V, Kanický V, Kaiser J, Liška M, Samek O, Malina R and Páleníková K 2007 *Appl. Surf. Sci.* **253**, 3834
[8] Popescu A, Beldjilali S, Socol G, Craciun V, Mihailescu I and Hermann J 2011 *J. Appl. Phys.* **110**, 083