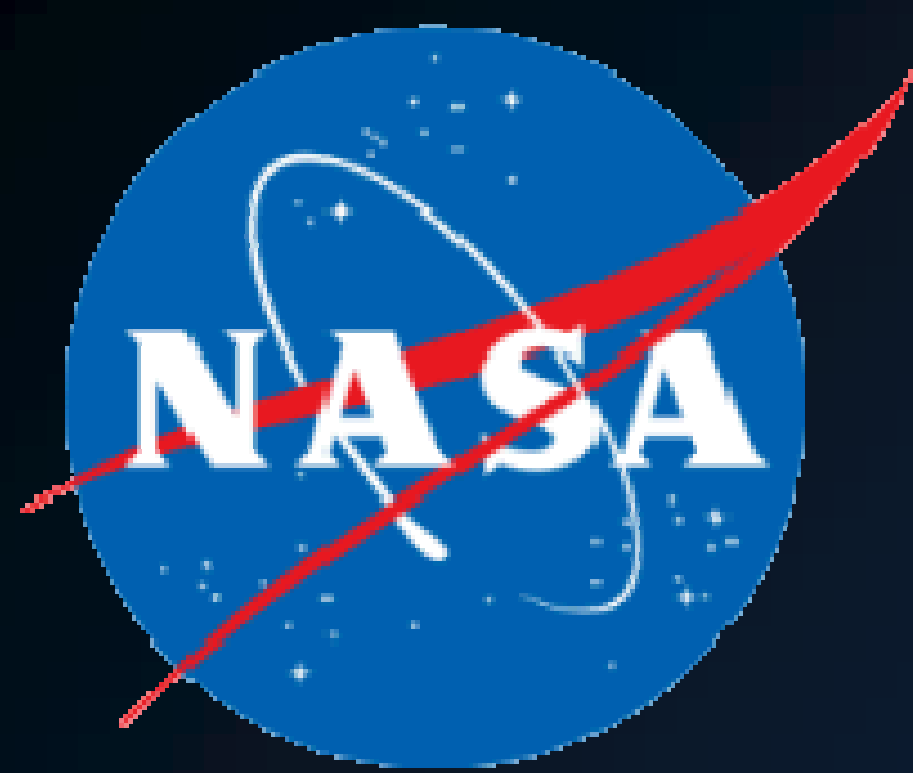




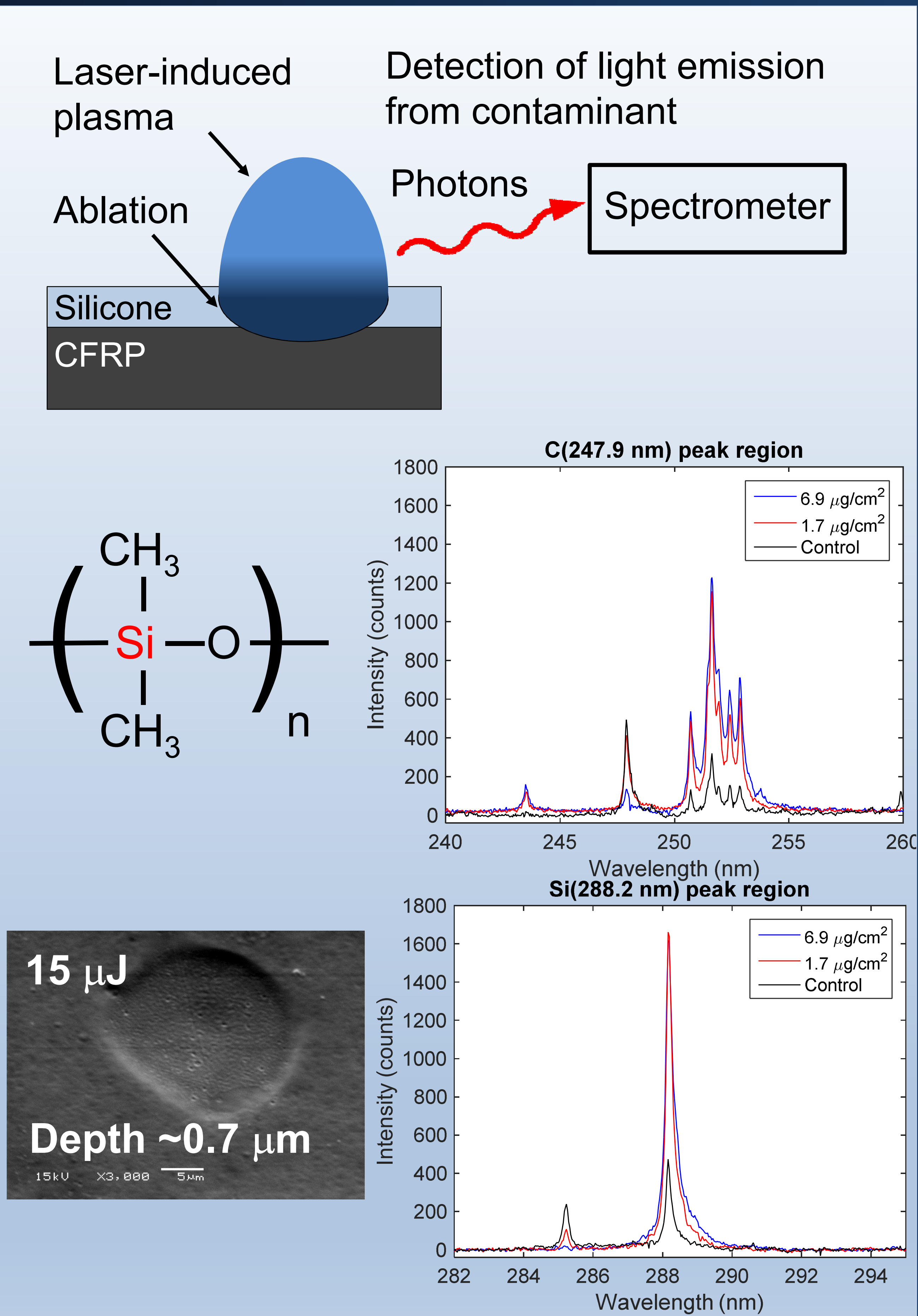
Detection of Trace Silicone Contamination on Carbon Fiber Reinforced Composite (CFRP) Surfaces by Picosecond Laser Induced Breakdown Spectroscopy (LIBS)

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To advance the use of adhesive bonding in primary structural applications on commercial transports, the development of high fidelity surface treatment and analysis methodologies has been under investigation in the Advanced Composites Project (ACP). Contamination of composite surfaces by even trace amounts of silicones is known to be a serious threat to achieving reliable adhesive bonds. The use of laser surface treatment has shown significant promise in removing silicone contaminants, and laser induced breakdown spectroscopy has been shown to be a rapid and sensitive method to detect very minute concentrations of silicone contaminants, is not size limited, and requires no sample preparation.

Laser Induced Breakdown Spectroscopy

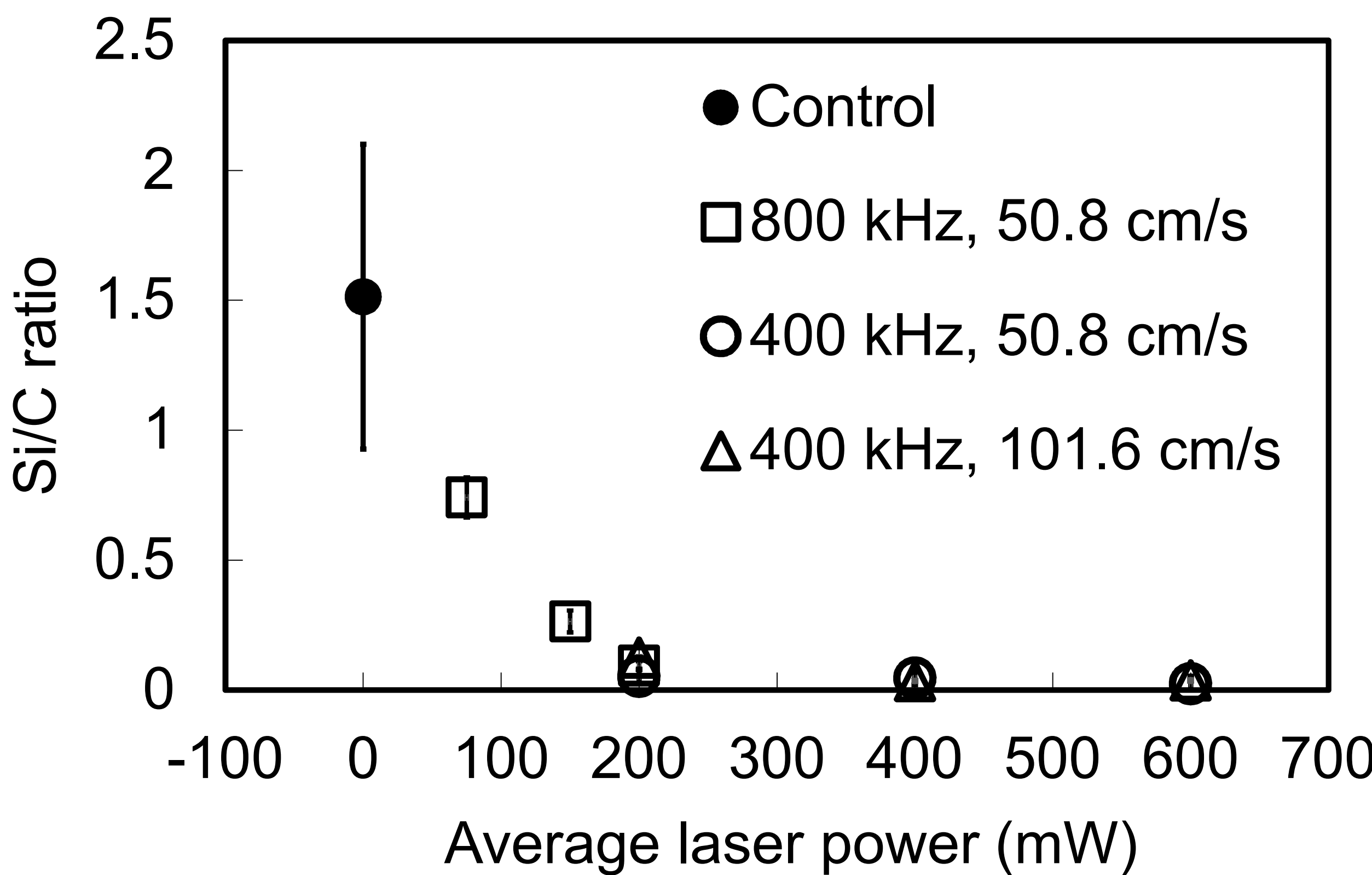


- LIBS detection using single laser pulses
- Higher sensitivity to surface contaminants
- Rapid monitoring of CFRP surface conditions

Laser Ablation Conditions

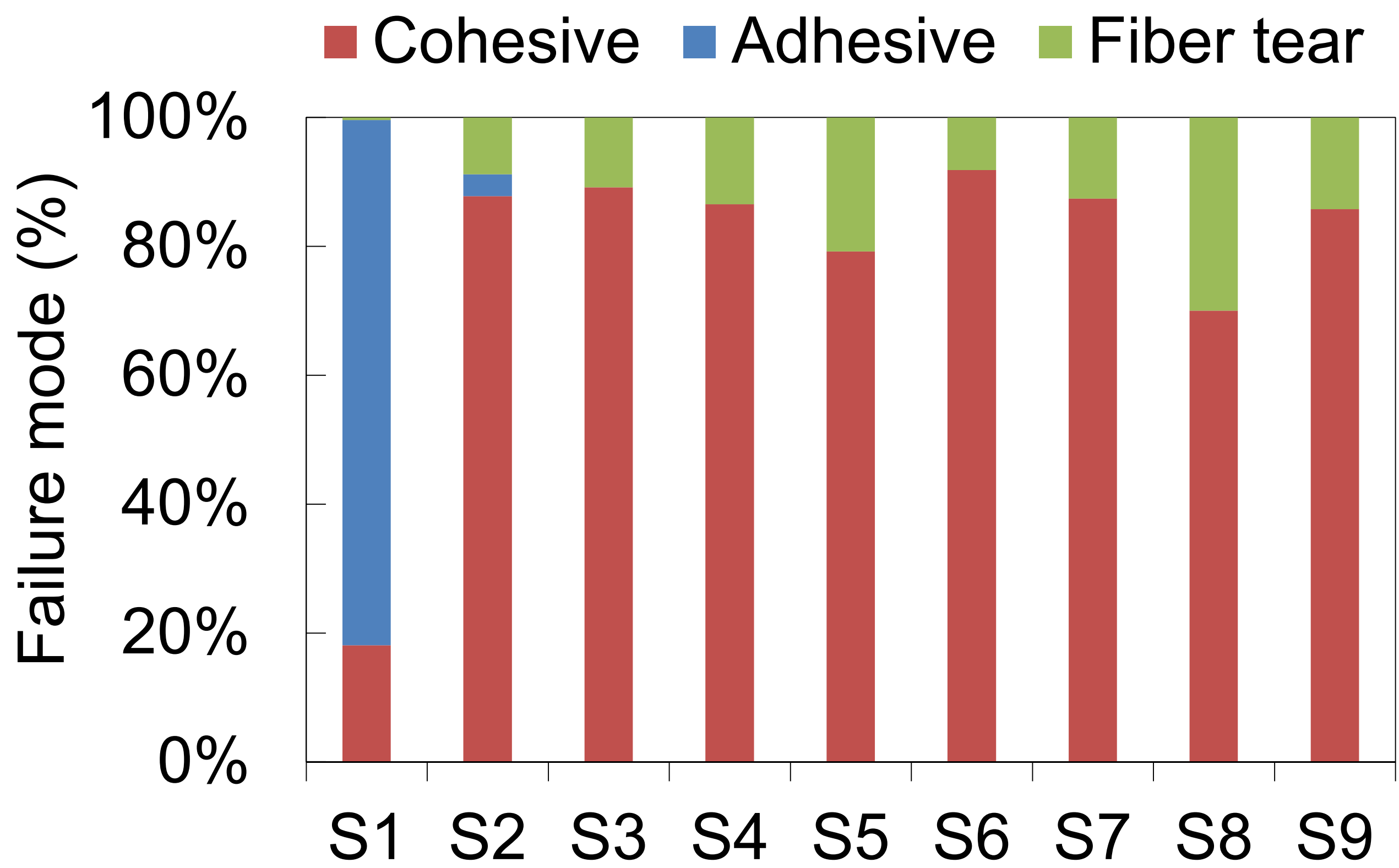
Sample	Laser power (mW)	Frequency (kHz)	Scan speed (cm/s)
Control	0	-	-
S1	75	800	50.8
S2	150		
S3	200		
S4	200		
S5	400	400	50.8
S6	600		101.6
S7	200		
S8	400		
S9	600		

Laser Parameters Analysis



- Above 200 mW, Si/C decreased and had similar values.

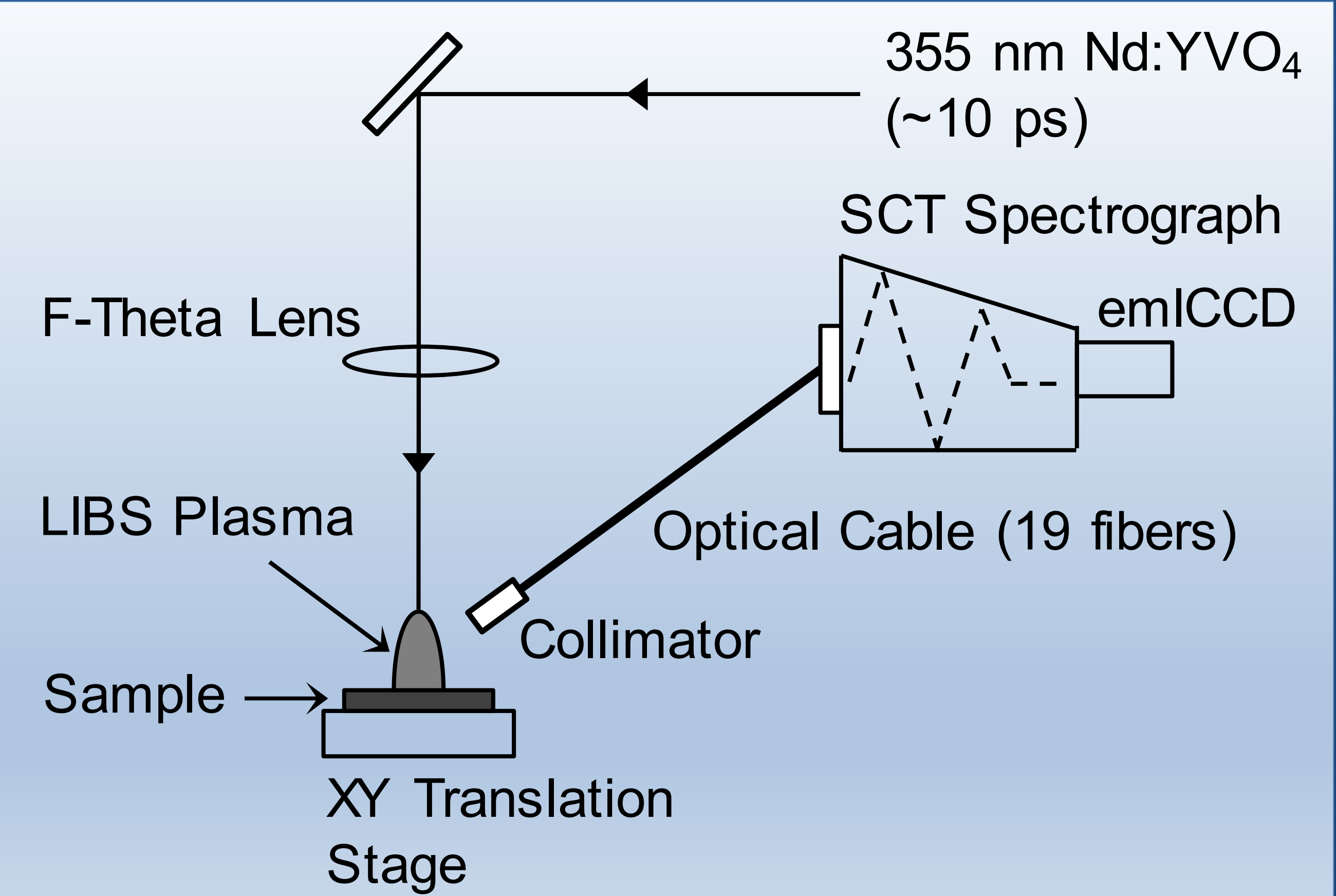
Failure Mode Analysis



Sample	Si/C	Cohesive	Adhesive	Fiber tear
		(%)	(%)	(%)
S1	0.74 ± 0.08	18	82	0
S2	0.26 ± 0.04	88	3	9
S7	0.13 ± 0.05	87	0	13
S3	0.09 ± 0.02	89	0	11
S4	0.05 ± 0.03	87	0	13
S5	0.05 ± 0.03	79	0	21
S9	0.04 ± 0.01	86	0	14
S8	0.04 ± 0.01	70	0	30
S6	0.03 ± 0.03	92	0	8

- Below the Si/C ratio of 0.26 ± 0.4 , high cohesive failure mode and no adhesive failure mode were observed.

LIBS Detection System



➤ Si relative content:

- White text = before laser treatment
- Red text = after laser treatment

➤ Laser treatment dramatically reduces Si concentration

Panel treated at 200 mW and 355 nm



Conclusions

- LIBS was used to characterize CFRP surface conditions before and after surface treatment.
- LIBS was able to detect trace silicone even after laser surface ablation.
- Minimal ablation required for LIBS inspection.
- Laser power has a significant influence on the Si/C ratio.
- Below the Si/C of 0.26 ± 0.4 , high percentage of cohesive failure mode (~100%).
- Measurement of silicone contamination prior to surface treatment is critical to adjusting the laser parameters to ensure sufficient silicone removal prior to bonding.
- LIBS provides real-time data, is automatable and requires no sample preparation or special atmosphere.
- Limit of detection of Si comparable to x-ray photoelectron spectroscopy (XPS).