RheinAhrCampus Remagen ● University of Applied Sciences Koblenz ● Optics and Laser Technology

P32 LIBS – A Versatile Tool for Spectroscopically Controlled Micro-Structuring of Thin Layers

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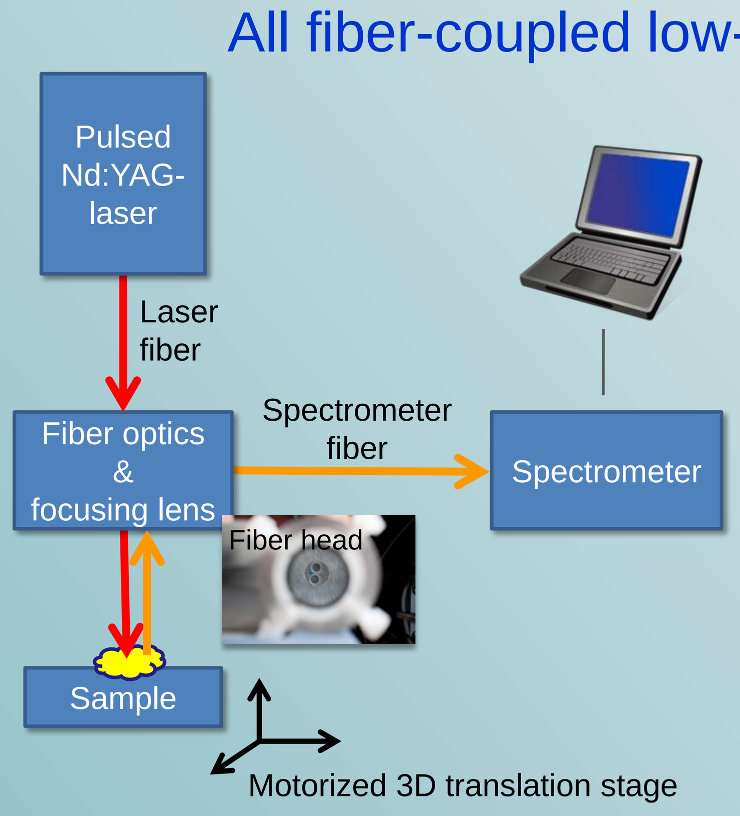
ABSTRACT

We present experimental results regarding micro-structuring of thin indium tin oxide (ITO) layers on glass substrates under LIBS process control. ITO $(In_2O_3)_x{:}(SnO_2)_{1-x}$ is an optically transparent conducting material widely used for various industrial products, such as organic solar cells and liquid crystal displays. LIBS can be used well for both the ablation process for micro-structuring and for real-time controlling the depth profiles by simultaneously analyzing the elemental composition. We studied the ablation process by irradiating different quantities of short laser pulses focused on ITO layers with a thickness of 200 nanometers. All characteristic spectral emission lines of indium tin oxide disappear if the ITO layer in the laser focus has been completely removed from the substrate. Since the ITO coated plate is made of glass calcium lines and the typical sodium doublet around 589 nm are strongly increasing. This indicates that the laser ablation process must be stopped to prevent further damages of the substrate. Similar results for spectrometrically controlled micro-ablation were obtained for silver/salt (Ag/NaCl) stacks and gold (Au) layers in their specific emission regimes. Patterned films of these materials are often used as diffraction filters in the extreme ultra violet (EUV). Images of ablation were made with a scanning electron microscope (SEM). The elemental composition of the layers could be confirmed by energy dispersive X-ray (EDX) spectroscopy measurements.

EXPERIMENTAL SETUP

All fiber-coupled low-power LIBS system



ELEMENTAL ANALYSIS PROCEDURE

Single-component analysis

- Peak detection method: Combination of 2nd derivative and embedded Gaussian line fit
- Spectral data base: *NIST Atomic Spectra Database 2011*

Multi-component analysis

- Principal Component Analysis (PCA) combined with a neuronal network

LIBS-SYSTEM

Compact all fiber-coupled system

Laser (CryLas DSS 1064-3000)

- Passively Q-switched DPSS Nd:YAG laser
- Wavelength 1064 nm, 2 mJ/pulse, 2.4 ns pulse length
- Laser irradiance 2 GW/cm², laser fluence 5 J/cm²
- Laser fiber: core diameter 400 µm, NA 0.37 (43° full angle)
- Aspheric lens focusing with focal spot size of 225 µm

Mobile Spectrometers (Ocean Optics)

- Δλ: 260 to 645 nm, δλ = 0.2 nm
- Δλ: 480 to 593 nm, δλ = 0.1 nm
- Spectrometer fiber: core diameter 910 µm

Scanning Electron Microscope

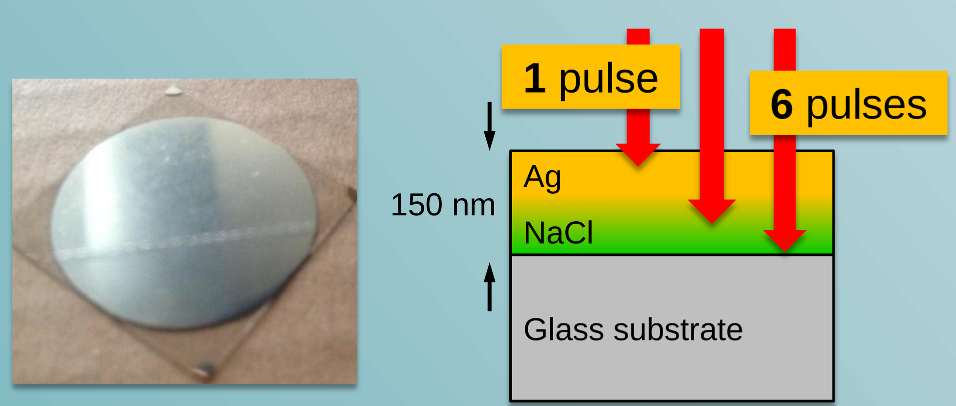
- Philips SEM 515 with EDX Oxford

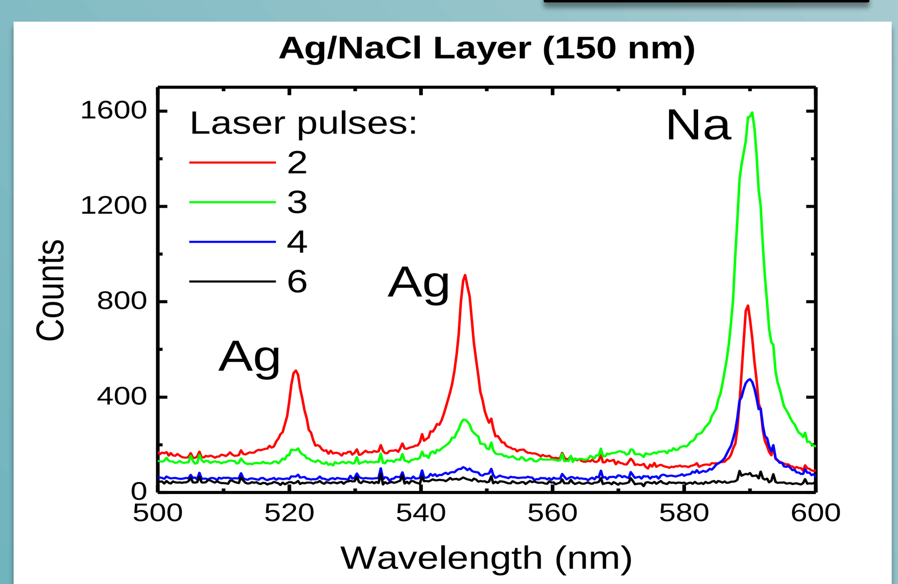
Layer Materials

- Indium tin oxide (ITO)
  - Thickness 200 nm
  - Surface resistivity R□ ≈ 20 Ω
- Silver/salt stack, thickness 150 nm
- Gold, thickness 30 nm

EXPERIMENTS AND RESULTS

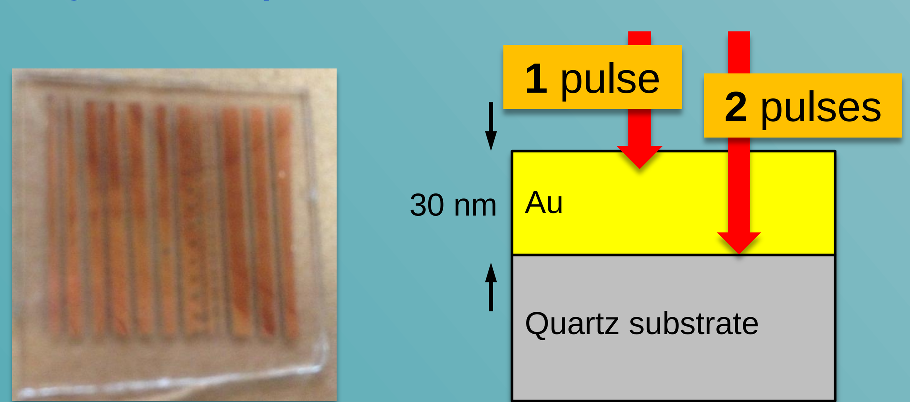
Spectroscopically controlled laser ablation of a stacked Ag/NaCl layer system on glass

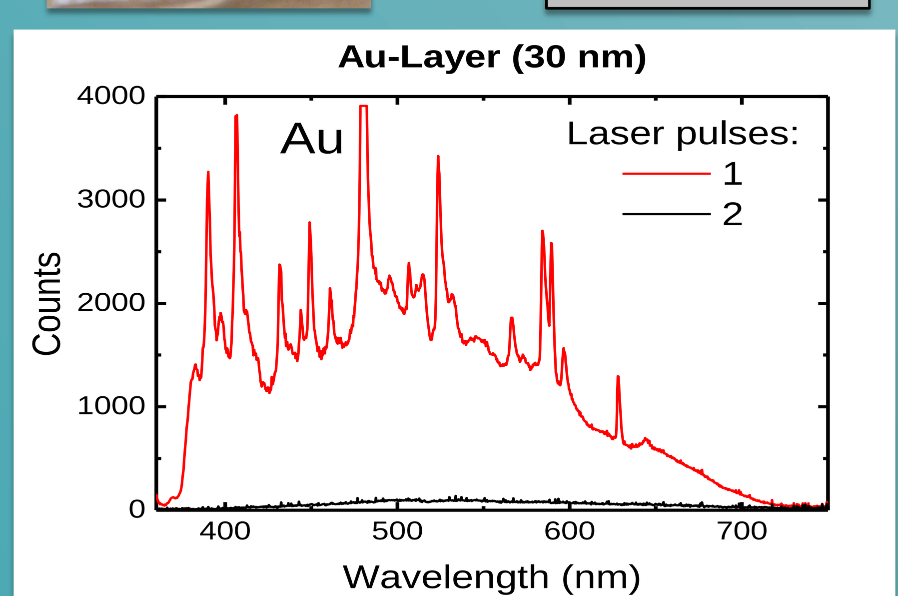




- After irradiating 6 laser pulses the Ag/NaCl stack system on glass substrate has been completely removed in laser focus. Ablation can be stopped.

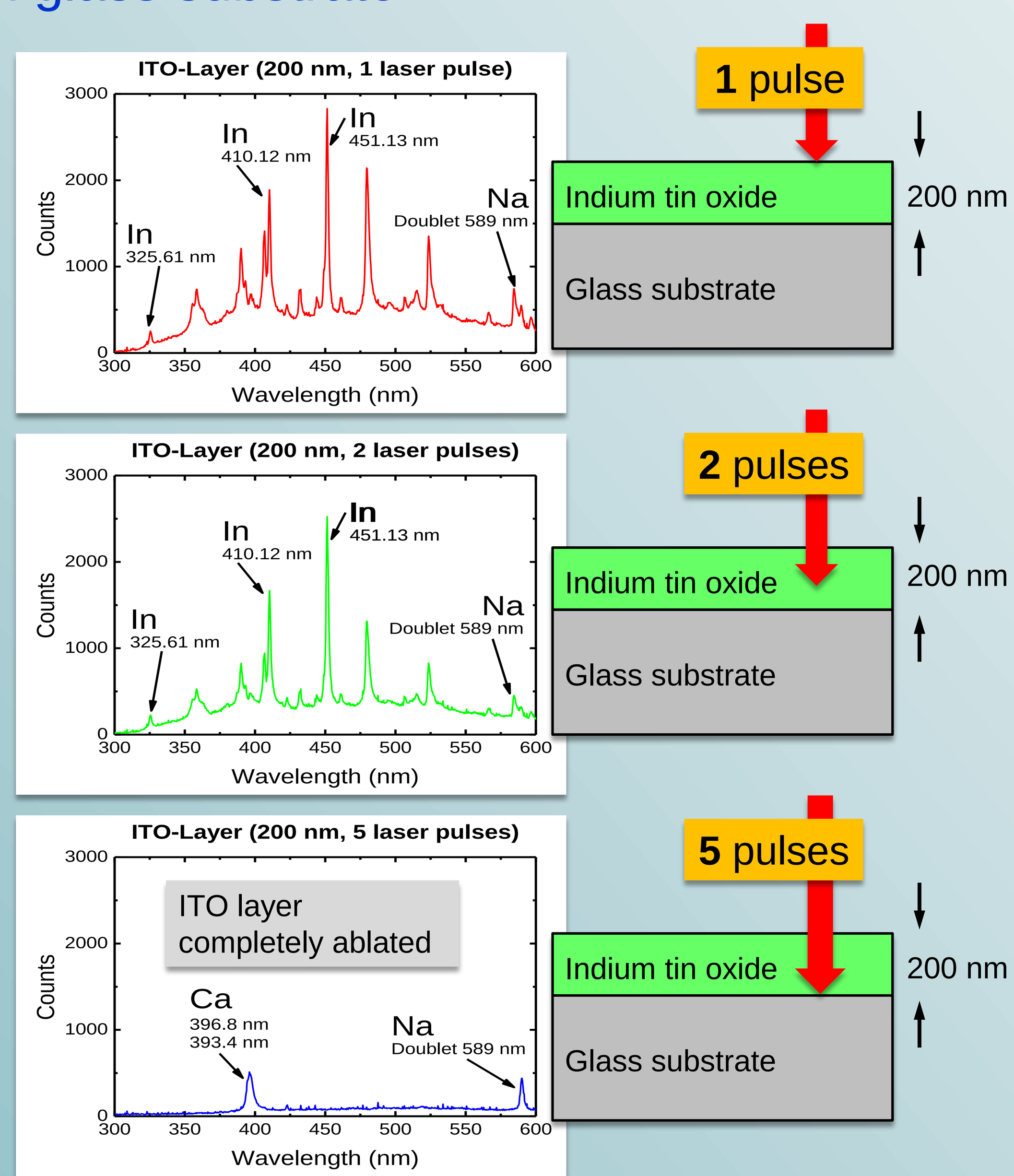
Controlled laser ablation of a single Au layer on quartz substrate



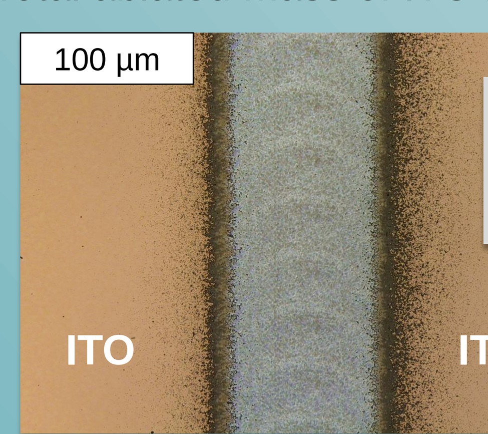


- Irradiating 2 laser pulses onto a thin 30 nm Au layer are sufficient for ablation – no measurable electrical conductivity between Au tracks.

Laser micro-structuring of ITO layers on glass substrate



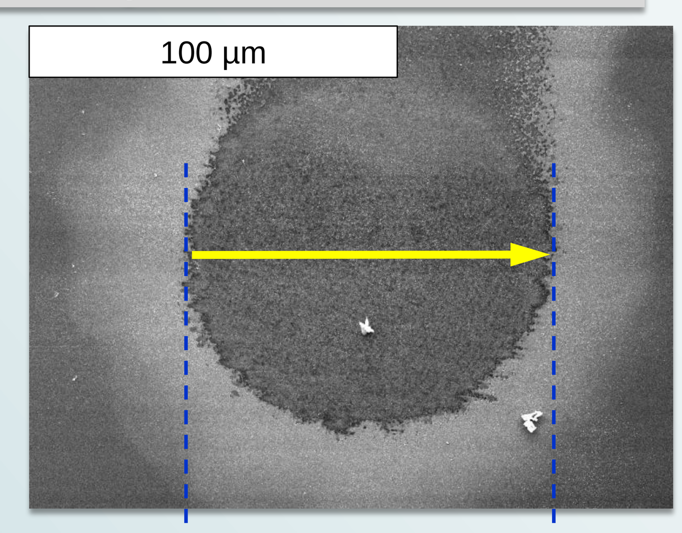
- Beginning the ablation process by irradiating a sequence of laser pulses the indium emission line at 451.13 nm declines while the sodium doublet line increases and characteristic calcium lines at 396 nm appear. Total ablated mass of ITO is about 1.6 ng.



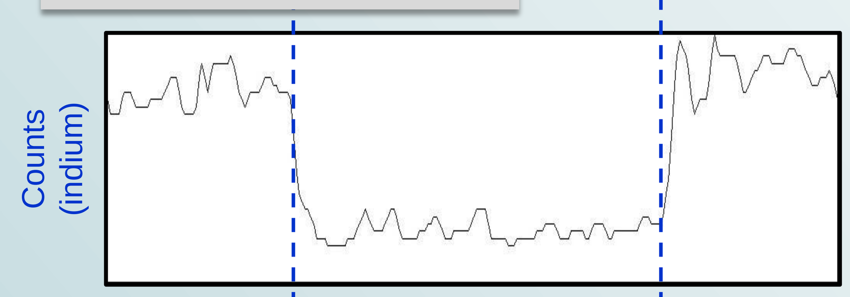
Small trench in an ITO layer ruled by spectroscopically controlled laser ablation

SEM and EDX analysis of the laser ablated zone

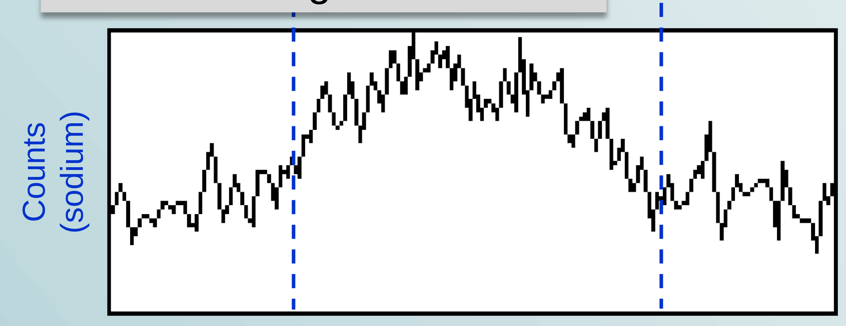
SEM image of laser ablated ITO film



EDX elemental analysis of **indium** in ITO film



EDX elemental analysis of **sodium** in glass substrate



- Images by scanning electron microscopy (SEM) demonstrate the laser ablated zone.
- An EDX analysis reveals the elemental composition on substrate surface: Decrease of indium counts, increase of sodium counts.

CONCLUSIONS

- ▶ We demonstrated qualitatively that LIBS could be a quite sufficient method for spectroscopically controlled laser micro-patterning of thin layers on substrates.
- ▶ The experiments showed that LIBS signals of selected elemental emission lines of different layer materials change with deposited laser pulse energy indicating the depth of the laser ablation process.
- ▶ The elemental depth profile of the laser-ablated ITO/glass stack could be confirmed by energy dispersive X-ray (EDX) spectroscopy.

ACKNOWLEDGMENT

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