

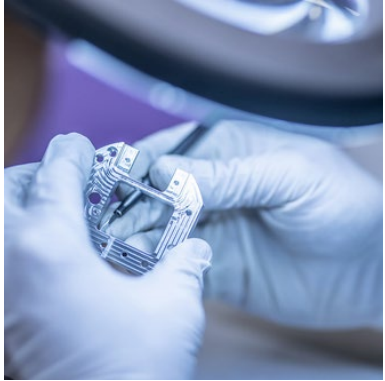
Laserinduziertes Mikroplasma in reaktiven Gasen - Ein Tool zum präzisen Ätzen von Materialien -

Klaus Zimmer, Afaque Hossain, Robert Heinke,
Pierre Lorenz, Martin Ehrhardt

Leibniz Institute of Surface Engineering (IOM);
Leipzig, Permoserstr. 15, Germany

klaus.zimmer@iom-leipzig.de

Outline

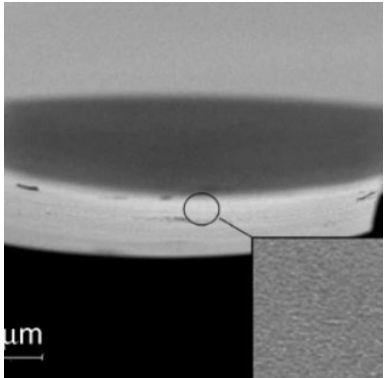


Requirements of UPSM

- lasers for UPSM
- status of different approaches for UPSM with lasers

Towards UPSM with laser radiation

- Laser-induced plasmas (LIP) an optically pumped plasma in gas
- Impact of selected parameters
- Surface characteristics



Summary/conclusions/future

- summarizing conclusions
- future developments

Ultraprecise surface machining (UPSM)

Integrity of Surface: “Inherent or enhanced condition of a surface produced by machining processes or other surface generation operations.”

application requirements



determining/measurement



machining process

Process demands

- atomic level material removal
- digital surface machining
- near-surface limited interaction process
- fast convergence of the machining
- specificity of the machining process
 - high material selectivity
 - homogeneous nonselective process
- surface binding energy management

ultra precise
surface machining

Surface requirements

- Atomic level smoothness
- Angstrom depth precision
- low/no surface defects
- across all scales; also small areas

Tool requirements

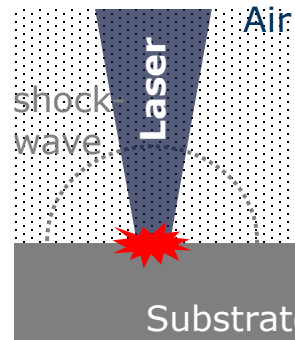
- capabilities to fulfil basic requirements of UPSM
- variable tool in size/shape/strength
- no momentum transfer to the surface
- low contamination / inert tool

Specific approaches for laser-based surface machining with view to UPSM

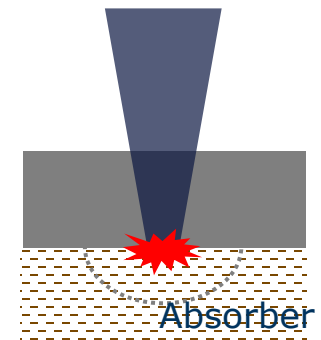
Lasers provide excellent preconditions for ultra precise surface machining (UPSM) as the tool

- is not in mechanical contact with the surface
- well controllable in space and time
- enables in-process measurement and process control
- ...

but the machining mechanism determines the surface quality.

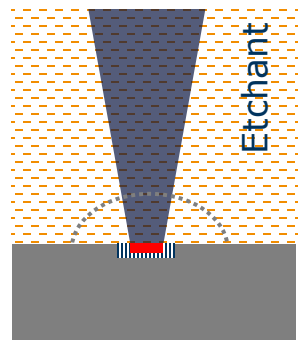


Ablation



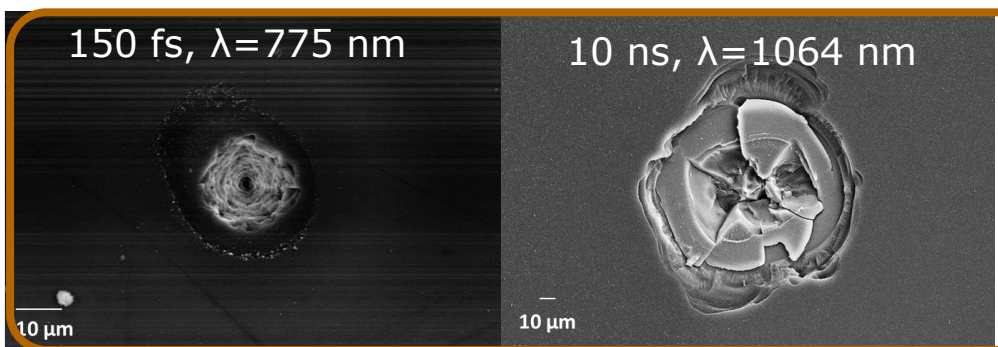
Heating, Decomposition
Ablation

Laser-induced backside etching



Etching, Heating

Laser-enhanced chemical etching



Laser ablation

Fundamental/experimental limit of precision for different laser-based techniques



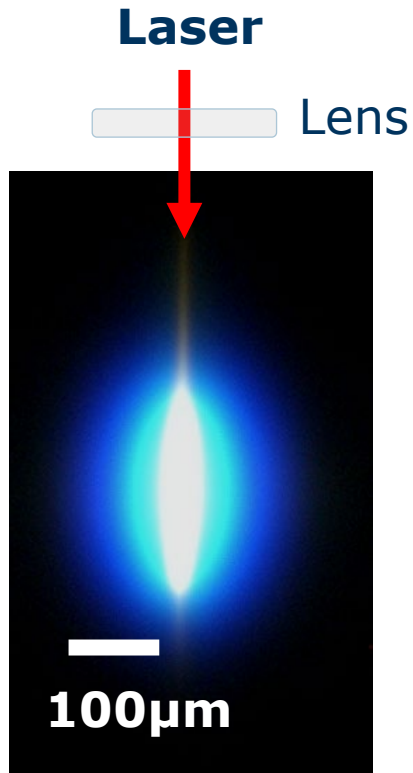
Process	Laser	Material removal rate in average	Material
PW laser ablation	Pulse	>150 nm	
USPL ablation	Pulse	50 to 150 nm	SiO ₂
LIBWE	Pulse	10 to 20 nm	SiO ₂
LESAL	Pulse	1 to 2 nm	SiO ₂
LIBDE	Pulse	80nm	SiO ₂
LIPhotoE etching	cw, min	1 to 2μm	SiO ₂
laser-induced plasma dry etching	Pulse	0.02nm to 0.05nm	SiO ₂

Characteristics of laser beams: λ , t_p , f , P_L , E_p , $2\omega_0$, v_s ...

Introduction to LIP

Electric field discharge: Focused lasers can produce high electric fields of the order of 10^5 V/cm.

→ Focusing laser pulses with high intensity results in an optical breakdown



Typical optical breakdown threshold in air:

- USP laser peak power density: $>10^{14}$ W/cm²
- SP laser peak power density: $>10^{12}$ W/cm²

Electrical and optical breakdown (USP)

$\sim 3 \cdot 10^6$ V/m

$> 10^7$ V/m

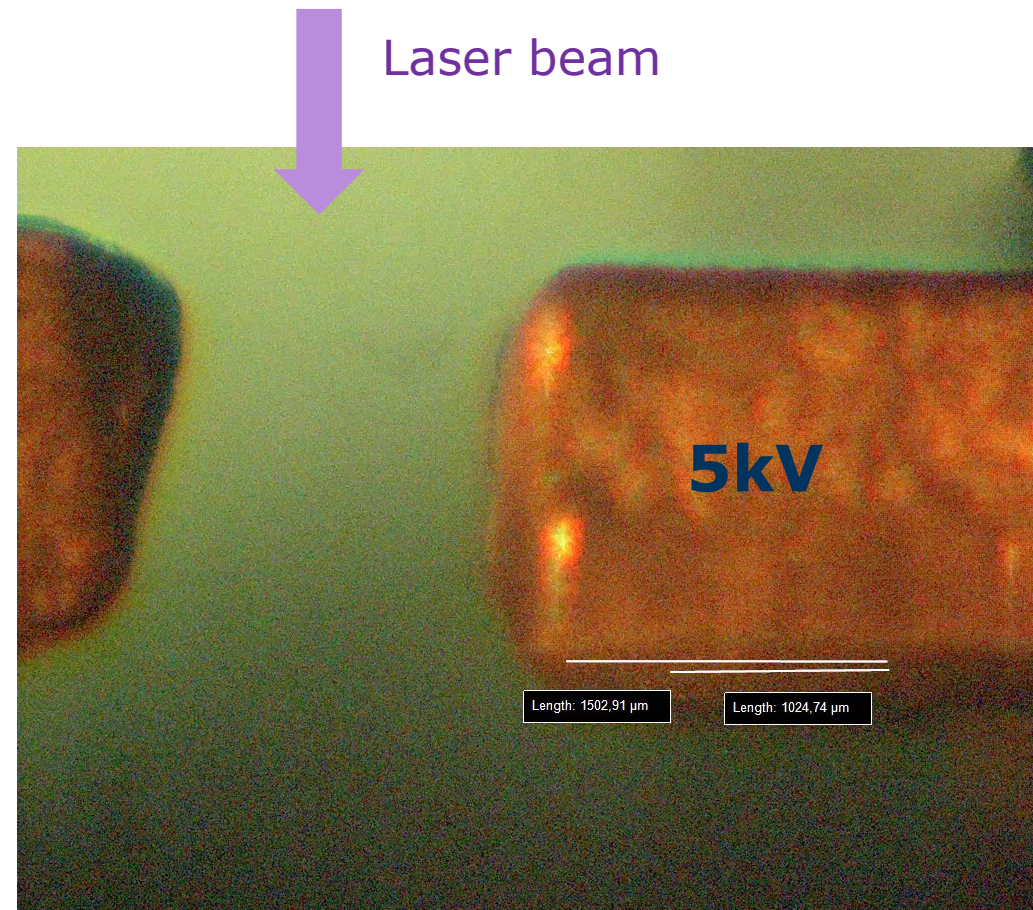
→ different mechanism of break down?

Parameters:
 $t_p = 150 \cdot 10^{-15}$ s
 $E_p = 0,5$ mJ

Electron generation and ionisation

Examples of laser-induced plasmas

- Laser ablation
- Pulsed laser deposition (PLD)
- Laser shock peening (LSP)
- Laser-induced breakdown spectroscopy
- Laser plasma for inducing/guiding electrical discharge

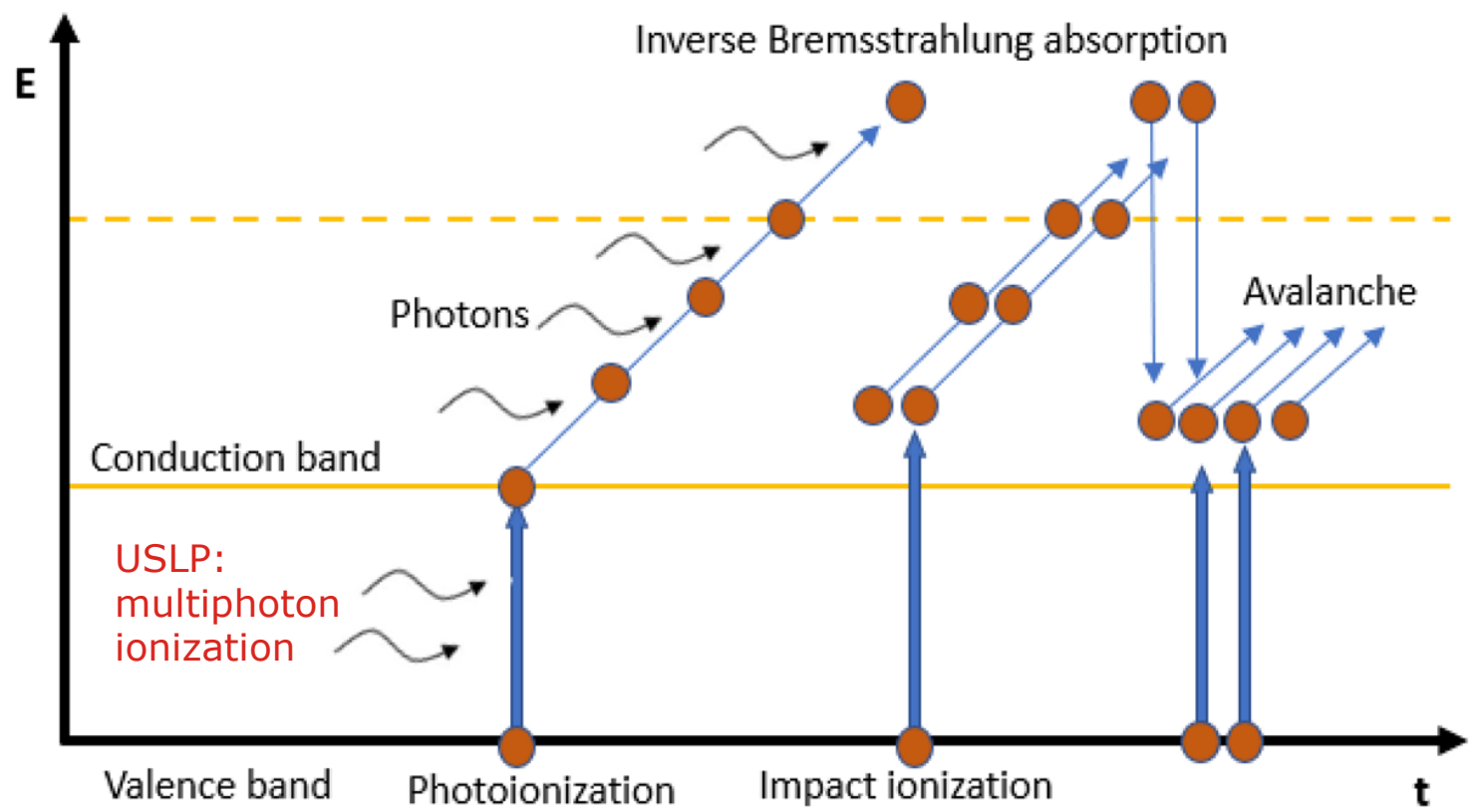
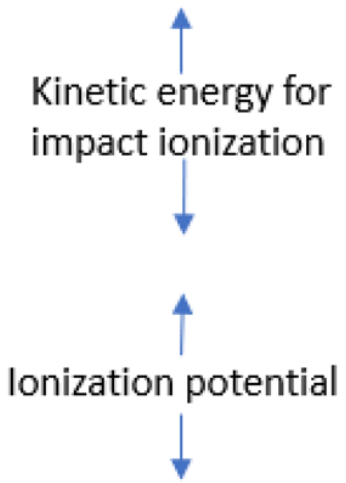


Laser-induced plasma etching (LIPE)

Electron generation and ionisation

Various processes

- thermal
- energetic photons
- high electric fields
- particles impact
- charge transfer
- energetic rays
- ...



$t_p \sim 10^{-13} \text{s}$

pulse length

$< 10^{-13} \text{s}$

10^{-11}s

10^{-9}s

frequency

$> 10 \text{THz}$

100GHz

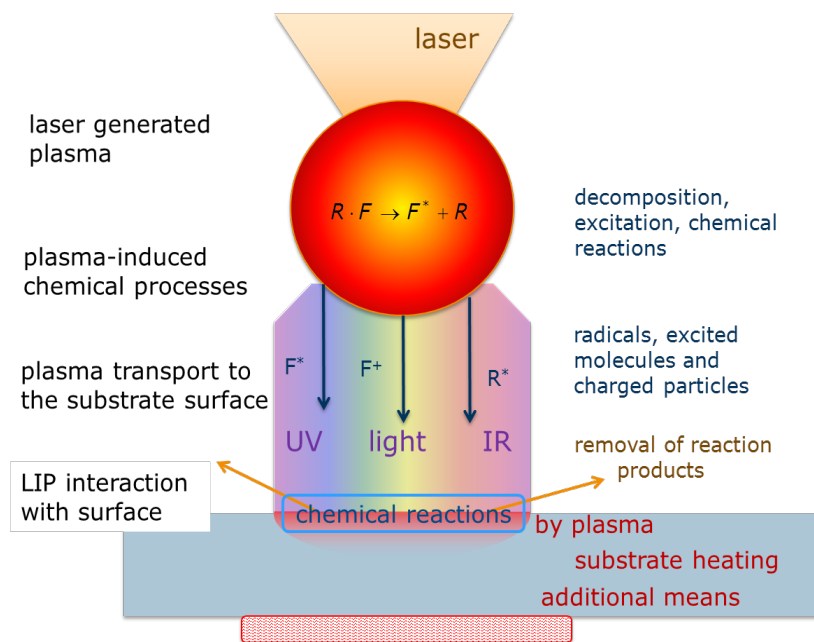
1GHz



Schema of the expected process of surface processing with LIP

Lasers Induced Plasma etching (LIPE)

- Ignition of a plasma in a gas by laser induced optical break down with USP laser



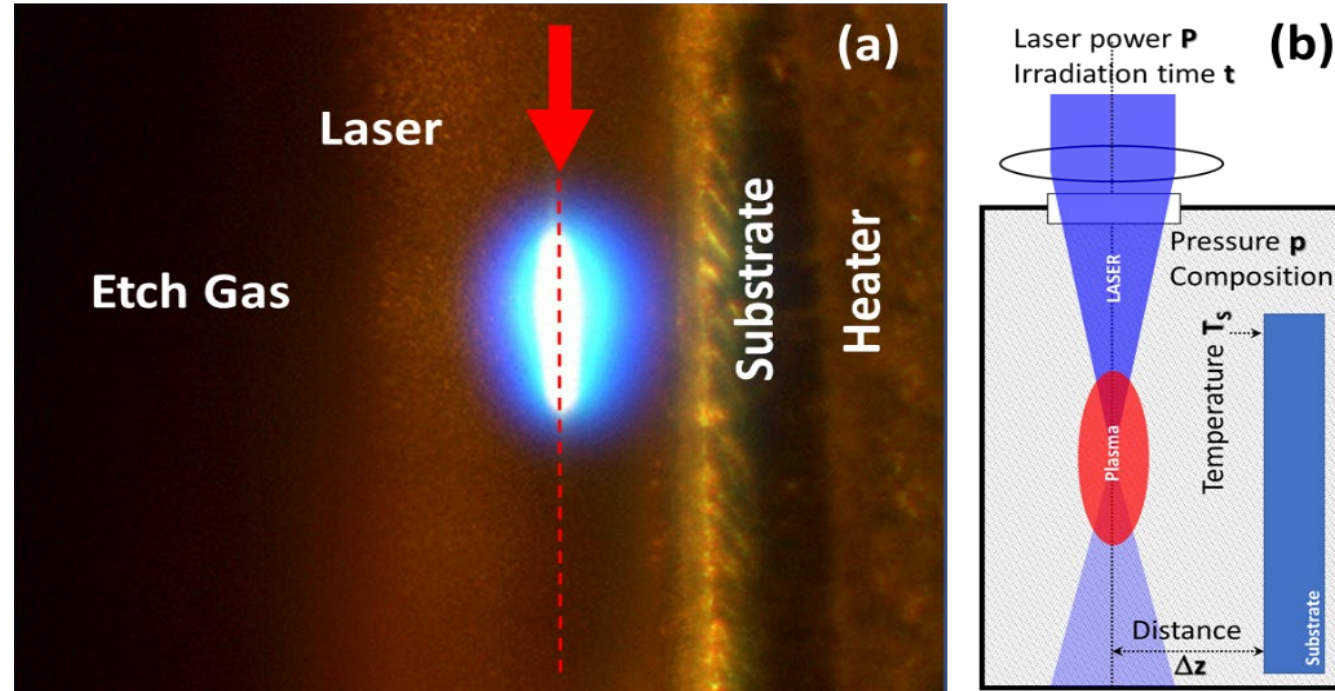
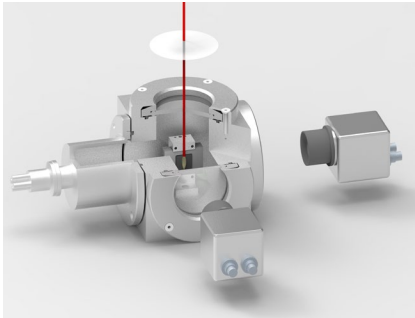
Characteristic laser induced plasma in gas:

- Small size plasma
- Contact free → "Contamination free"
- Atmospheric pressure plasma
- Generated and manipulated with optical methods

➔ Generating reactive species by decomposition/excitation of the gas

Laser-induced plasma etching

- Glancing angle of incidence in order to avoid direct irradiation the substrate



- Gas: CF_4/O_2 ; Pressure absolute: 0.3 bar to 2 bar; Temperature: RT to 500 °C
- Material: Fused Silica; Silicon

Experimental Setup

Using ultrashort laser pulses for ignition of the plasma

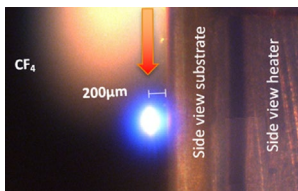
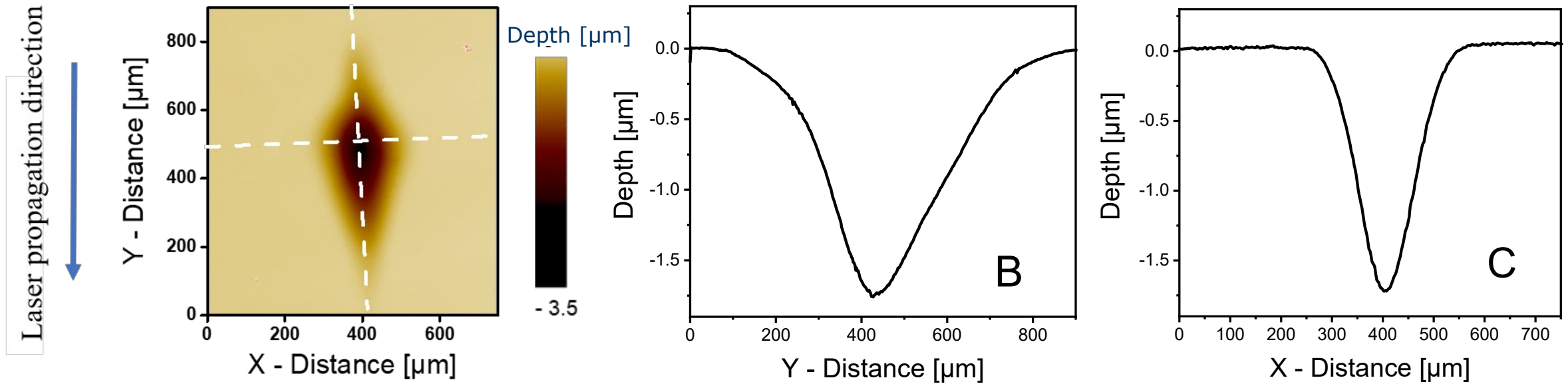
- Pulse duration = 150 fs
 - Rep. Rate = 1 kHz
 - $E_{\max} = 800 \mu\text{J}$
 - Wavelength = 775 nm
-
- Low pulse energy (but high \hat{P})
 - Low thermal/mechanical effects e.g., shockwaves
-
- High pulse repetition rate (MHz)
 - Small plasma size (spot size)

Etching gas

- **CF₄**
- O₂
- CF₄/O₂
- Air
- SF₆

Results

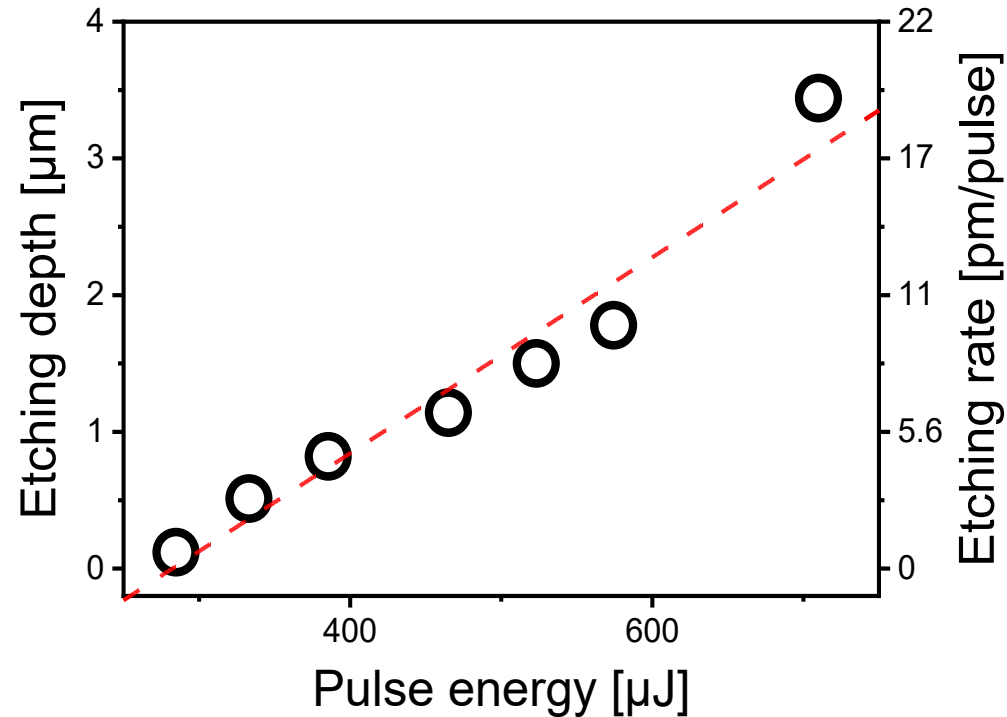
Etching of SiO_2 with laser-induced plasma ignited in a CF_4 gas mixture



Material: SiO_2 ; Gas: CF_4 ; Absolute pressure: 0.85 bar; Temperature: 450°C; Etching time: 3 min

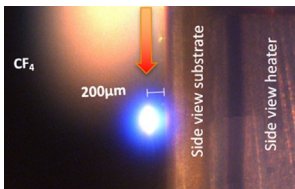
Results

Dependency of etching depth on laser pulse energy



Etching rate
in the pm range!

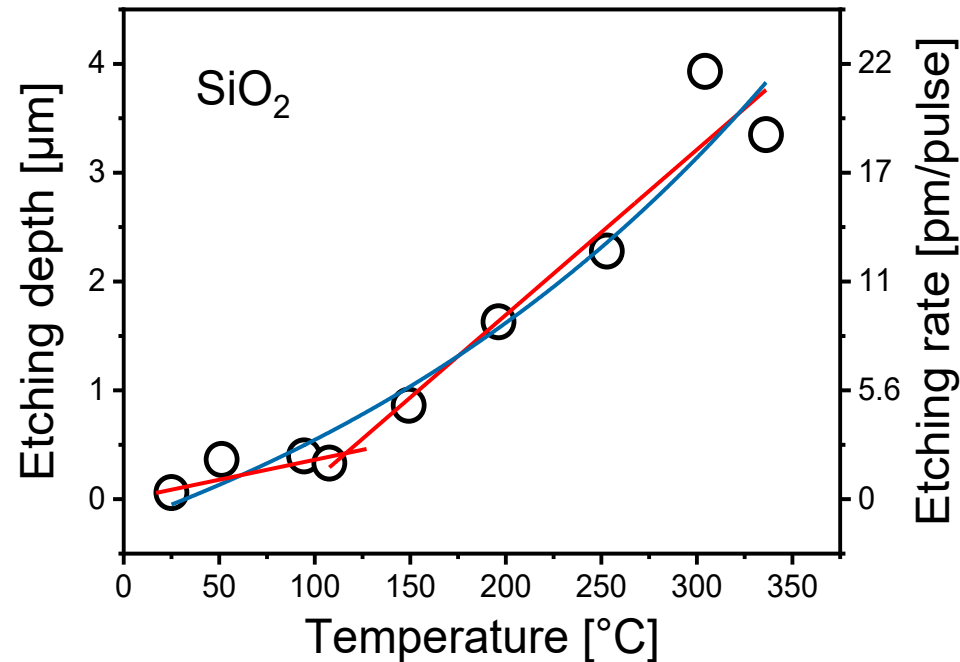
Material: SiO_2 ; Gas: CF_4 ; Absolute pressure: 0.85 bar; Temperature: 450 °C; Etching time: 3 min; Distance: 100 μm



- Linear increase of etching depth with increasing laser pulse energy
- Threshold for etching at $\sim 250 \mu\text{J}$

Results

Dependency of etching depth on the substrate temperature



Arrhenius equation

$$K = A \exp\left(-\frac{E_a}{RT}\right)$$

E_a Activation energy

T Temperature

K Reaction rate

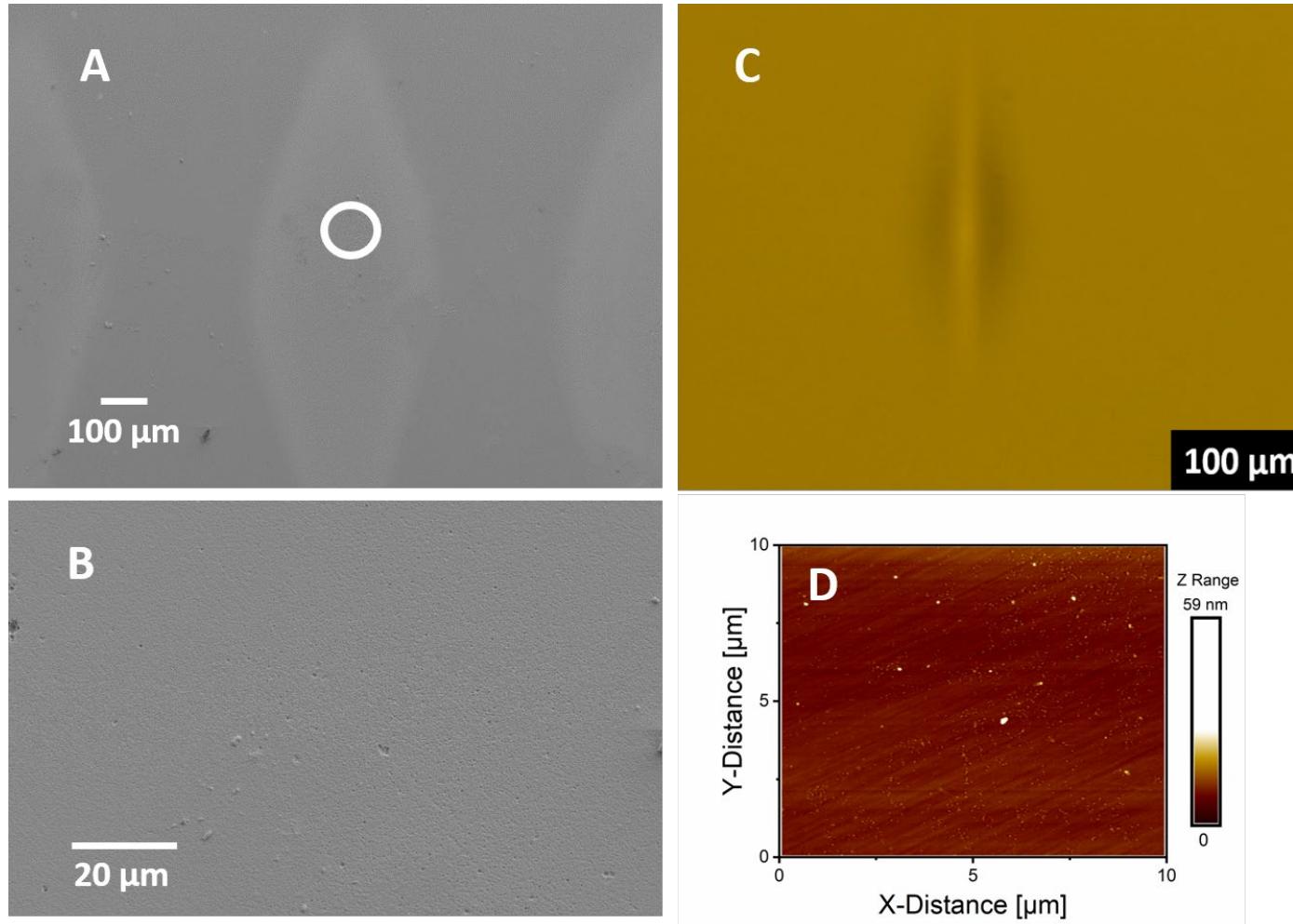
→ E_a of 4.3 kcal/mol

Material: SiO₂, Gas: CF₄; Absolute Pressure: 0.85 bar;
Etching time: 3 min, Distance: 100 μm

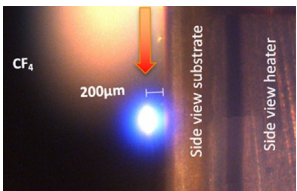
- For the simplest case of heterogeneous chemical reactions at the substrate the reaction rate K is usually described by the Arrhenius equation

Results

Surface roughness of laser-plasma etched surface



→ Surface roughness
1-2 nm RMS!



SEM, AFM and optical images of LIP-etched area SiO_2

Surface fidelity

Cross-section of laser ablated and LIP etched silicon surface

Laser ablation

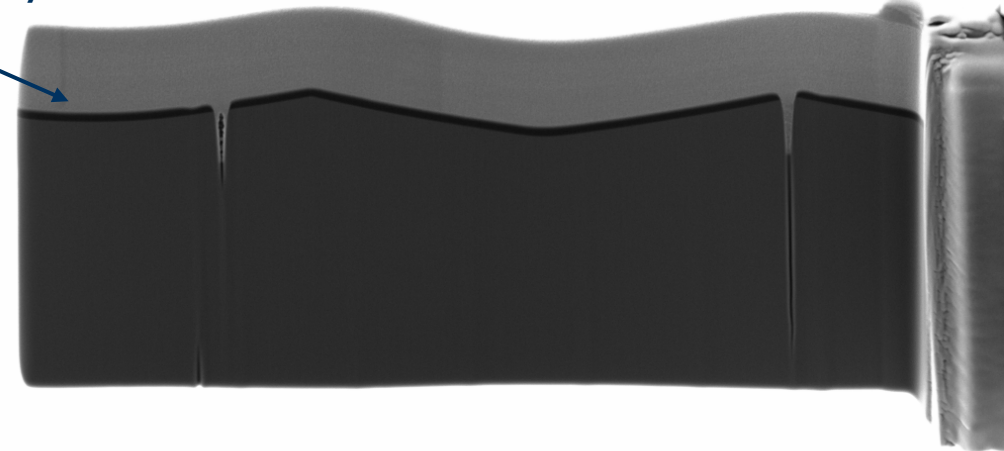


2 μm
EHT = 2.00 kV Aperture Size = 120.0 μm Stage at T = 55.4 °
WD = 4.9 mm FIB Imaging = SEM Tilt Corr. = On Stage at X = 50.028 mm Date : 7 Jul 2021
Mag = 7.26 K X Signal A = SESI Tilt Angle = 36.0 ° Stage at Y = 51.269 mm File Name = S135-1_71.tif

IOM

Protection layers

LIP etching



1 μm
EHT = 2.00 kV Aperture Size = 120.0 μm Stage at T = 55.4 °
WD = 4.9 mm FIB Imaging = SEM Tilt Corr. = On Stage at X = 49.754 mm Date : 7 Jul 2021
Mag = 7.27 K X Signal A = SESI Tilt Angle = 36.0 ° Stage at Y = 50.770 mm File Name = S135-1_76.tif

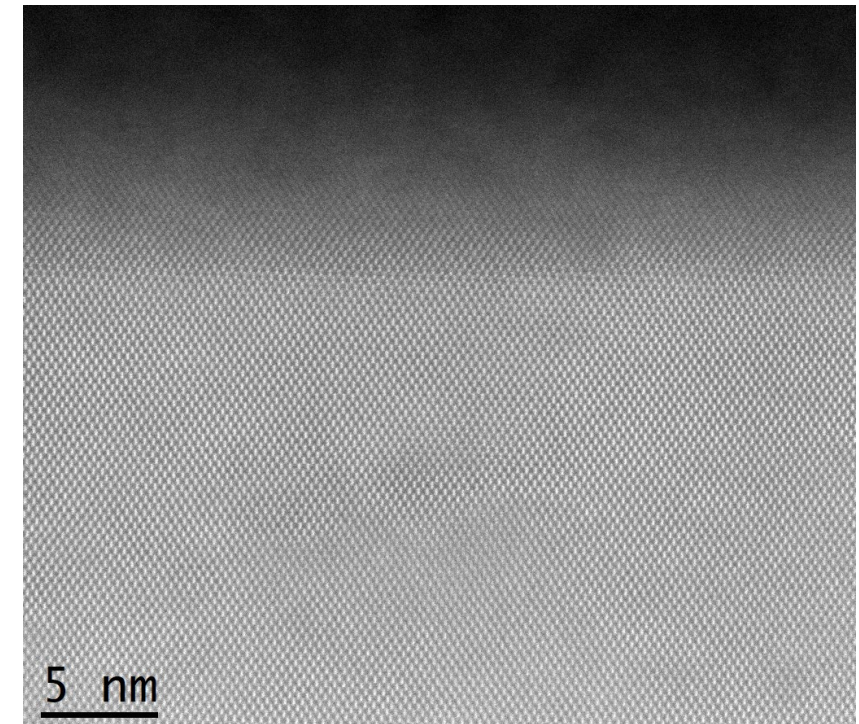
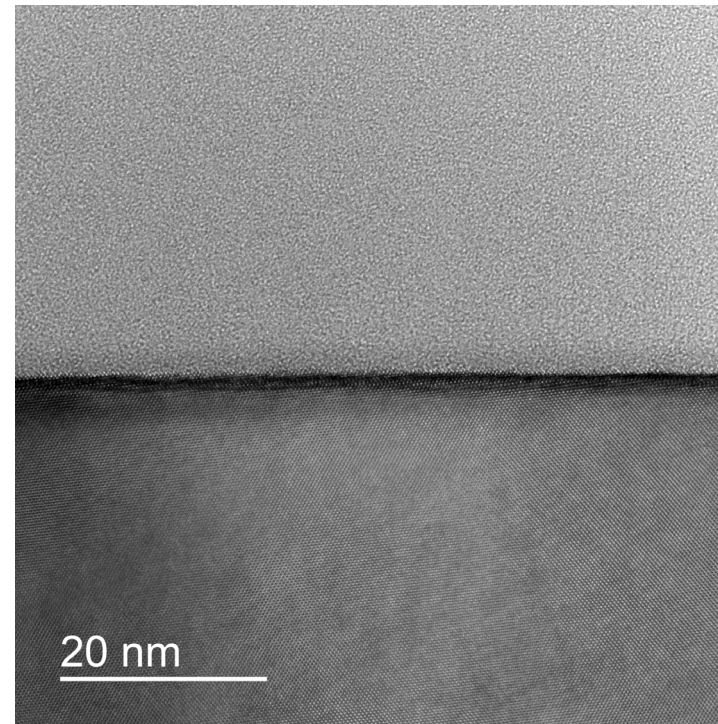
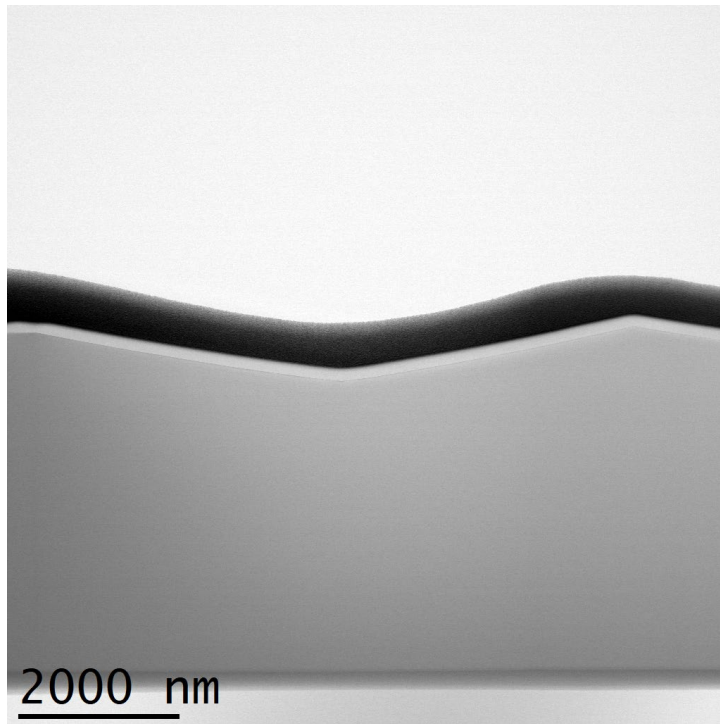
IOM

Strong (sub) surface damage is visible in the SEM.
TEM confirm:
melting, cracks, stress, and amorphization



Results

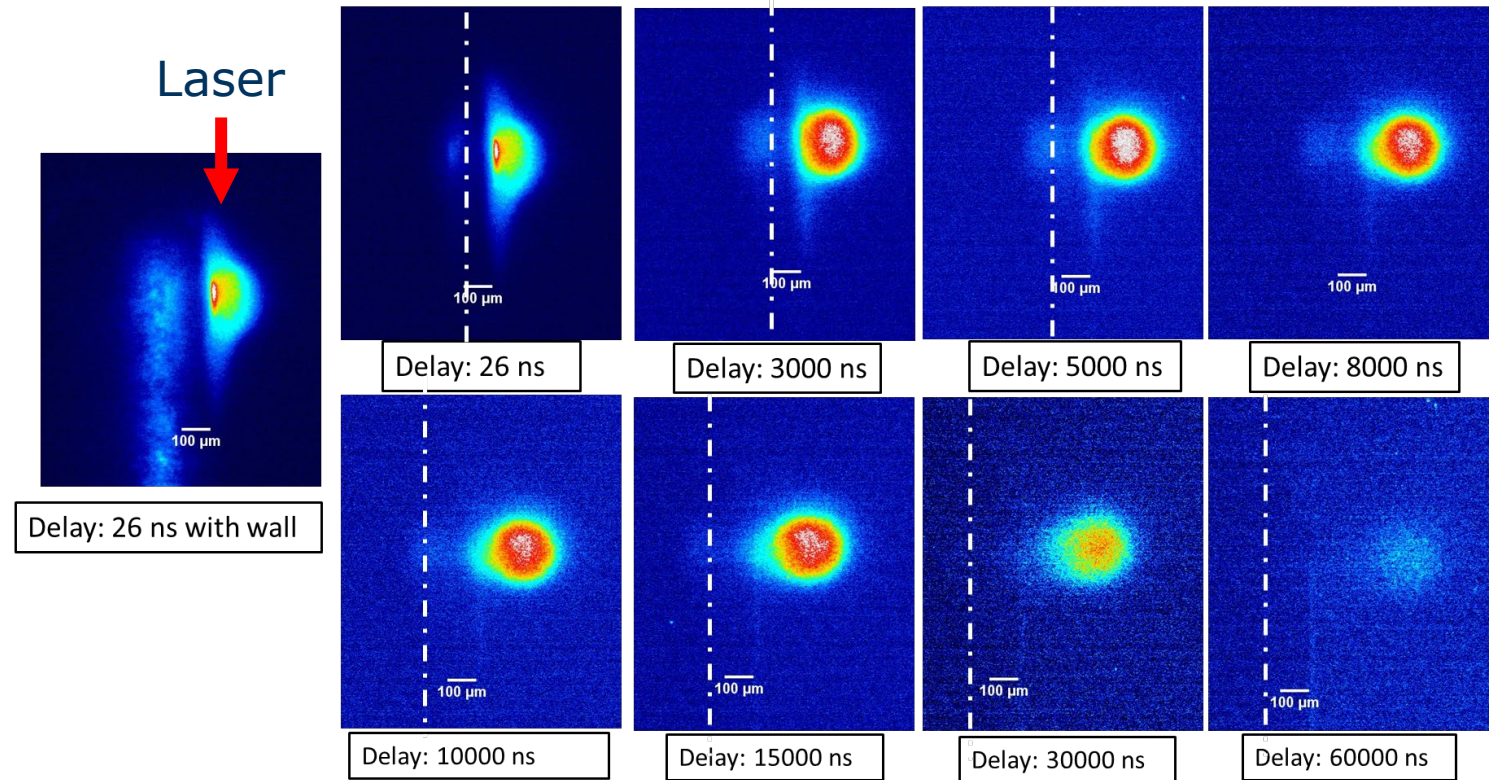
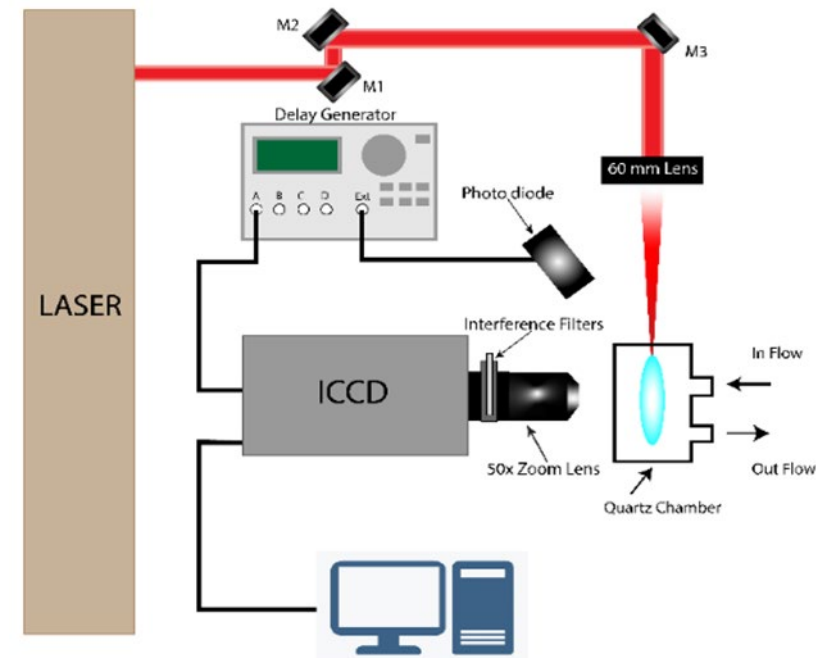
TEM image of LIP etched crystalline silicon.



➔ Almost no (sub-)surface damage!

Results

Example of optical diagnostics of LIP



Emission of a laser-induced plasma near a substrate surface

Summary/conclusion

Lasers-induced plasma as a new tool for UPSM.

- atmospheric pressure process conditions
- sub-mm size dimensions of the tool
- Extreme low etching rates → pm-range
- smooth etching enabled → nm rms
- almost no (sub)surface damage
- strong impact of the material to the etching (masking ...)

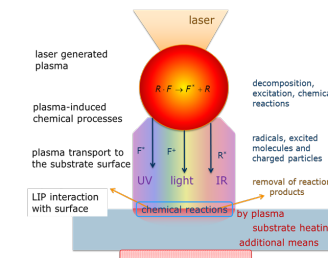
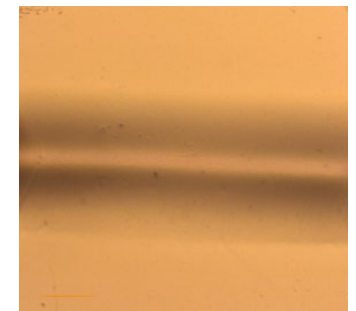
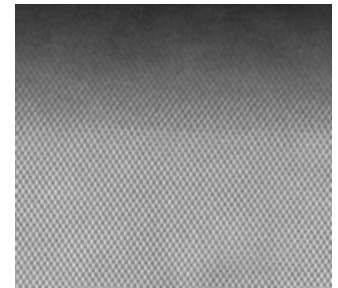
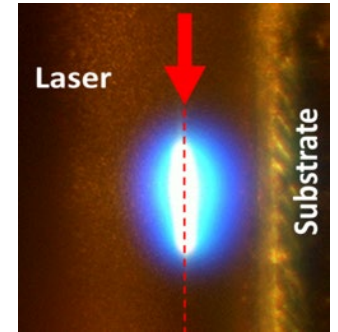
Understanding of LIPE mechanism

- LIPE mechanism
- governing processes
- physical-chemical model and the mathematical formulation simulation of the LIPE → good to have collaboration

Development of applicative cases for

- UPSM for optics, precision mechanics, etc.
- localized plasma-based processing

Improvements of the mechanism



Thank you for your attention!

Questions?

Klaus Zimmer
Leibniz Institute of Surface Engineering (IOM)
Leipzig, Permoserstr. 15, Germany

klaus.zimmer@iom-leipzig.de

Further reading

- [1] M. Ehrhardt, K. Zimmer, P. Lorenz et al., Germany Patent No. B23K 26/362 (04.09.2019 2019).
- [2] M. Ehrhardt, P. Lorenz, B. Han et al., Applied Physics a-Materials Science & Processing 126 (2020) 9.
- [3] R. Heinke, M. Ehrhardt, P. Lorenz et al., Applied Surface Science Advances 6 (2021) 100169.
- [4] A. M. Hossain, M. Ehrhardt, M. Rudolph et al., J. Phys. D 55 (2021) 125204.
- [5] M. Ehrhardt, P. Lorenz, K. Zimmer, in Ultrafast Laser Nanostructuring, in The Pursuit of Extreme Scales, edited by Razvan Stoian and Jörn Bonse (Springer Cham, 2022).
- [6] R. Heinke, M. Ehrhardt, J. Bauer et al., Appl. Surf. Sci. 597 (2022) 153712.
- [7] L. Streisel, M. Ehrhardt, P. Lorenz et al., in JLMN-Journal of Laser Micro/Nanoengineering, (2022), Vol. 1.
- [8] K. Zimmer, M. Ehrhardt, P. Lorenz et al., Ceram. Int. 48 (2022) 90.
- [9] A. M. Hossain, M. Ehrhardt, M. Rudolph et al., ACS Photonics (2023).

Acknowledgement

Help and Discussions by colleagues
of IOM and NJUST

Founding

DFG: Zi 660/17-1