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3D microfabrication of large-scale objects in glass using an ultrafast laser

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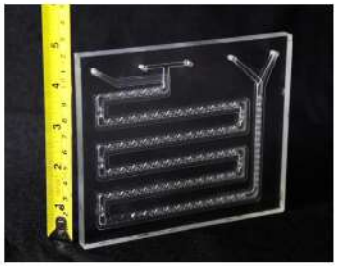
XXL - THE EXTREME OPTOELECTROMECHANIX LAB

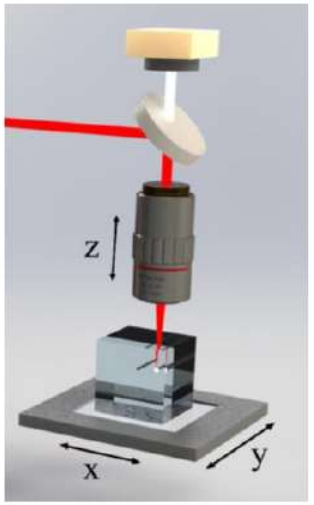
What are we going to talk about today?

- 3D printing: large scale but still of micrometer-scale resolution

- Chemical microreactor: microdevices for chemistry industrial application



- Only a laser is allowed to be used for making these structures.



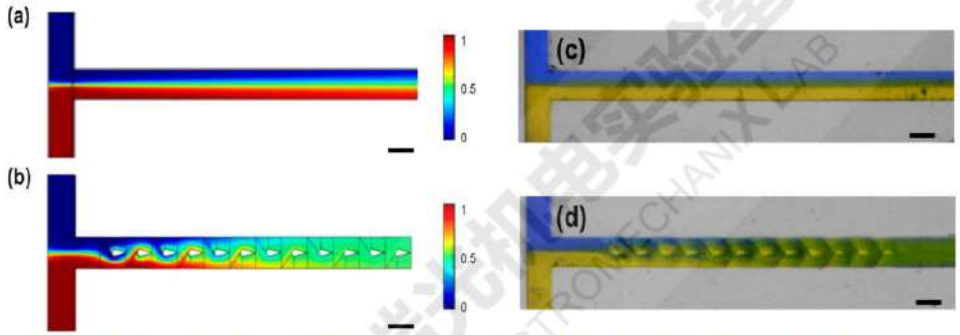
3D printing: large scale but still of high resolution

Statue of Monkey King fabricated out of glass

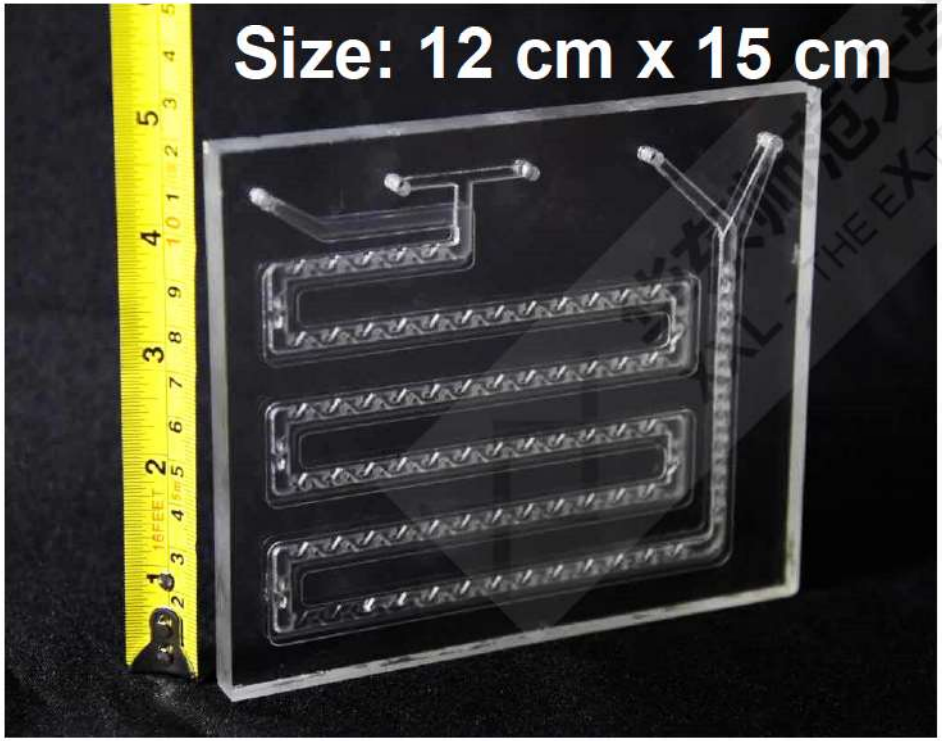
(Height: 4 cm; XYZ resolution: 15 μm)

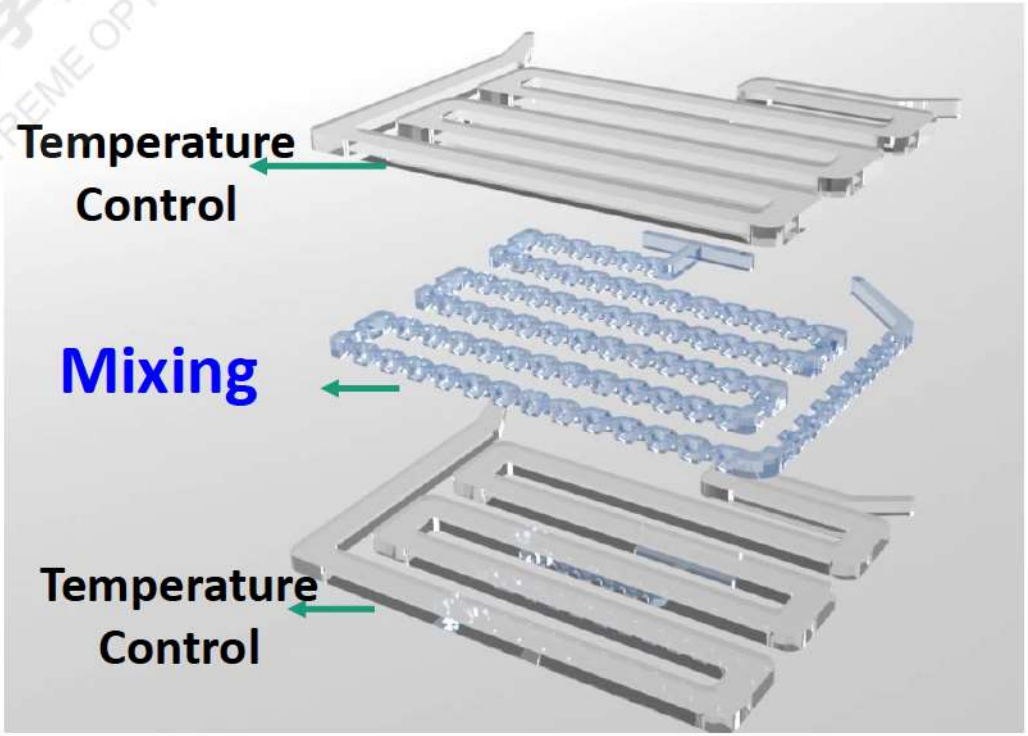
Microreactor for industrial applications

Mixing performances in the 1D and 3D channels

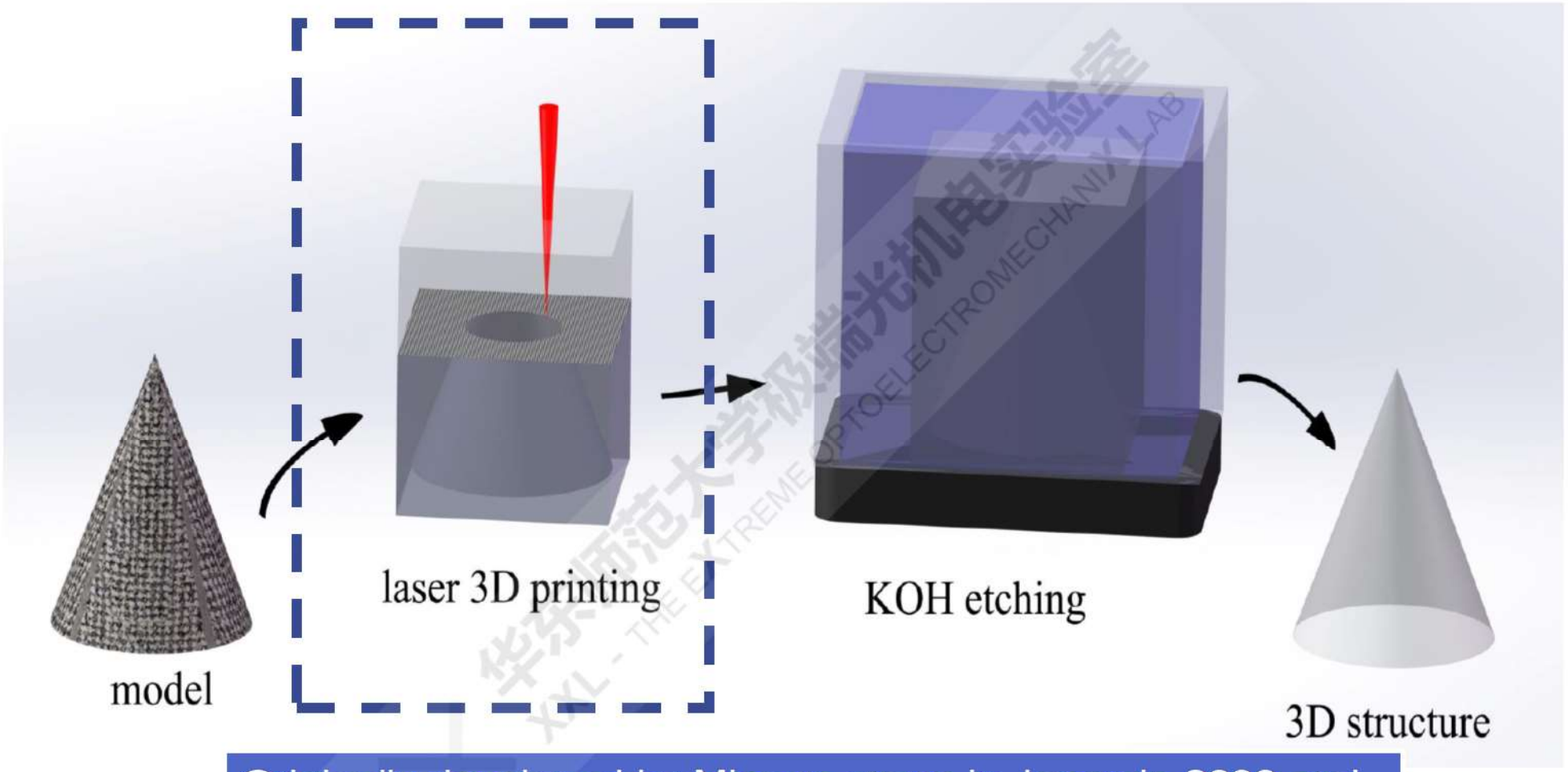


Jia Qi, et al., Micromach. **11**, 213 (2020).





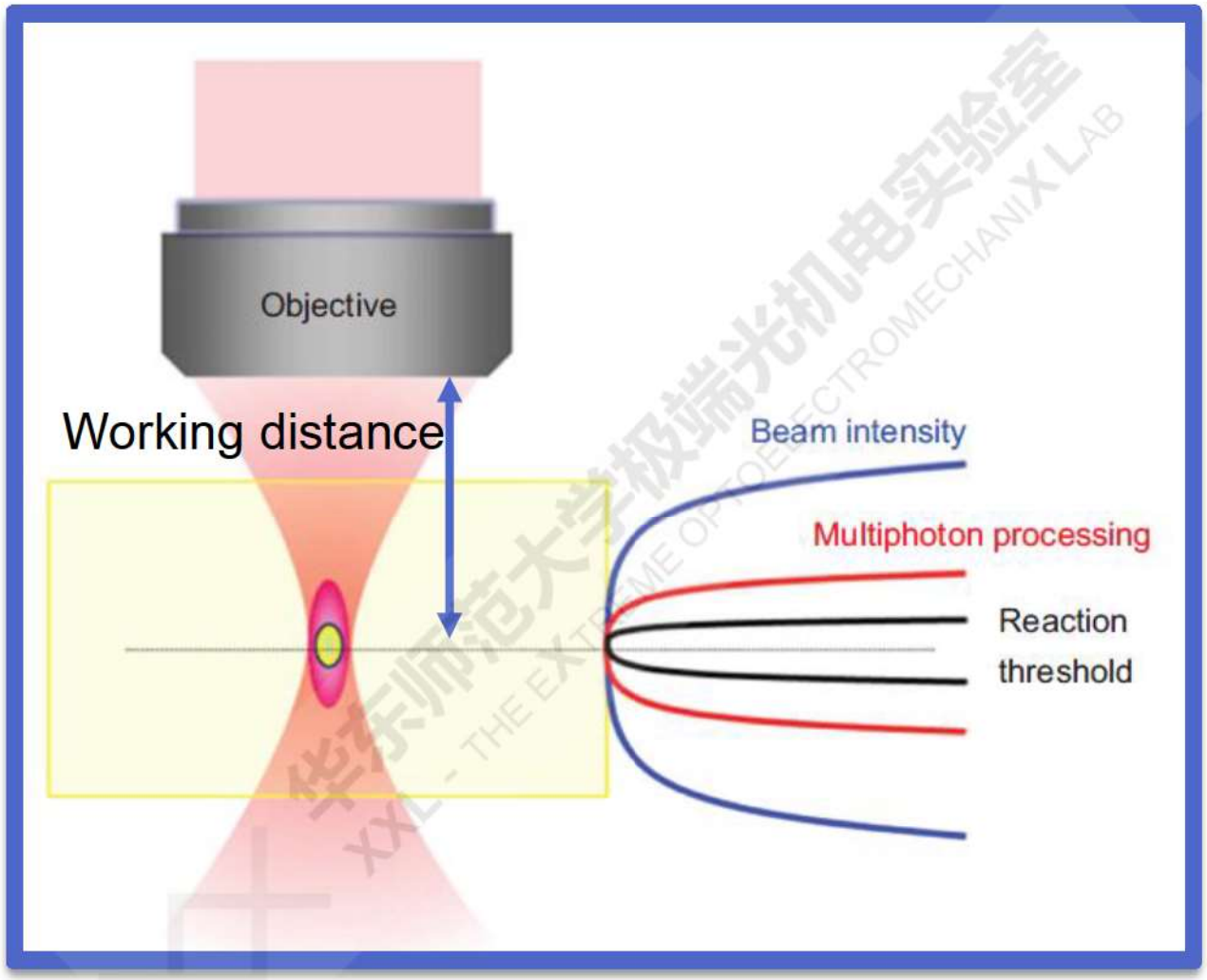
Technique: selective laser-induced etching



Originally developed by Misawa group in Japan in 2000 and then improved by many groups all around the world.

A subtractive process that can produce structures with unspoiled material properties.

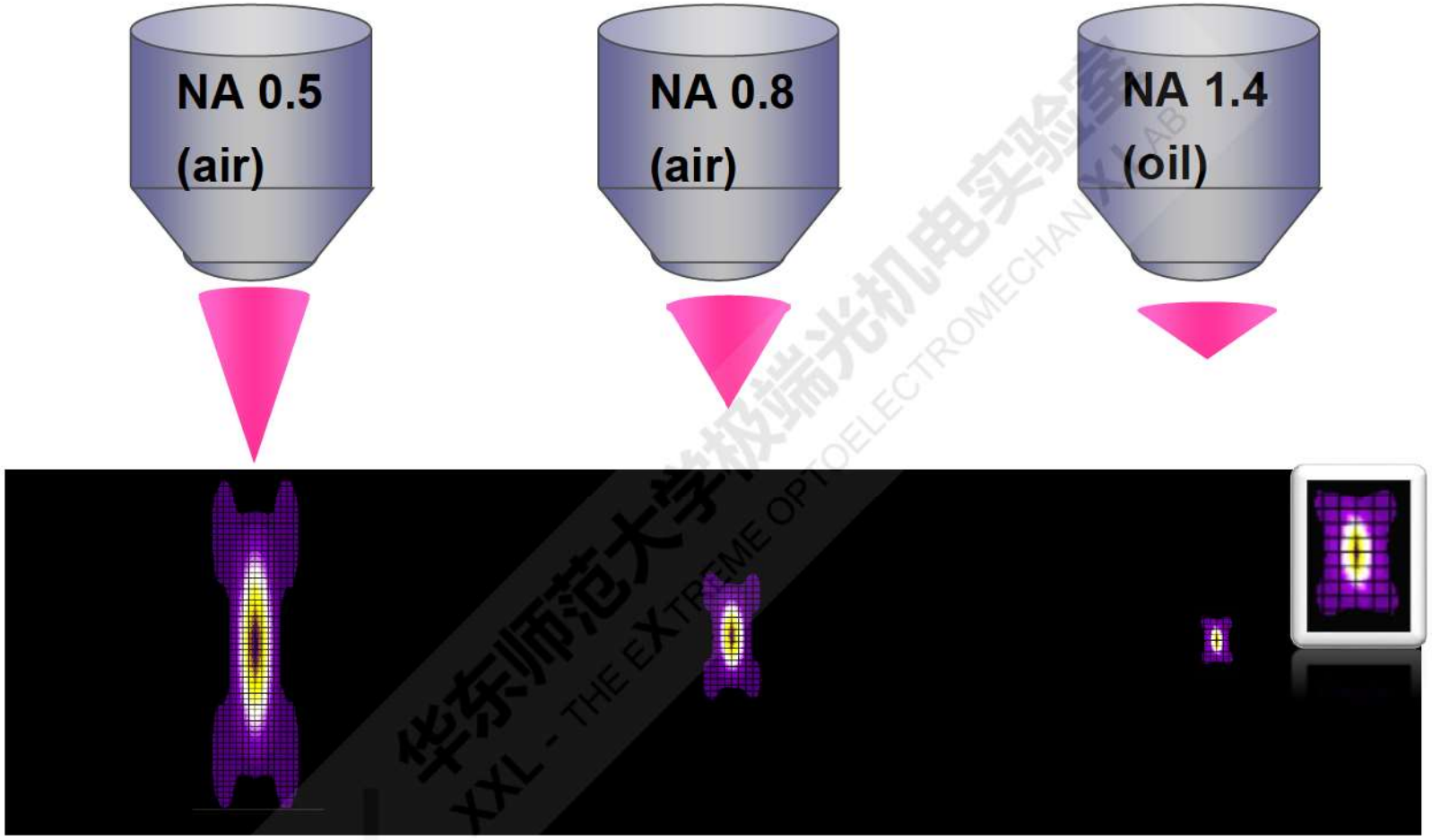
Mechanism: multiphoton absorption inducing modification



Highest achievable resolution: ~1 μm
Thickness of sample: less than 1 mm limited by lens working distance

K. Sugioka and Y. Cheng, Light: Sci. & Appl. 3, 2014.

The challenges for large scale glass printing

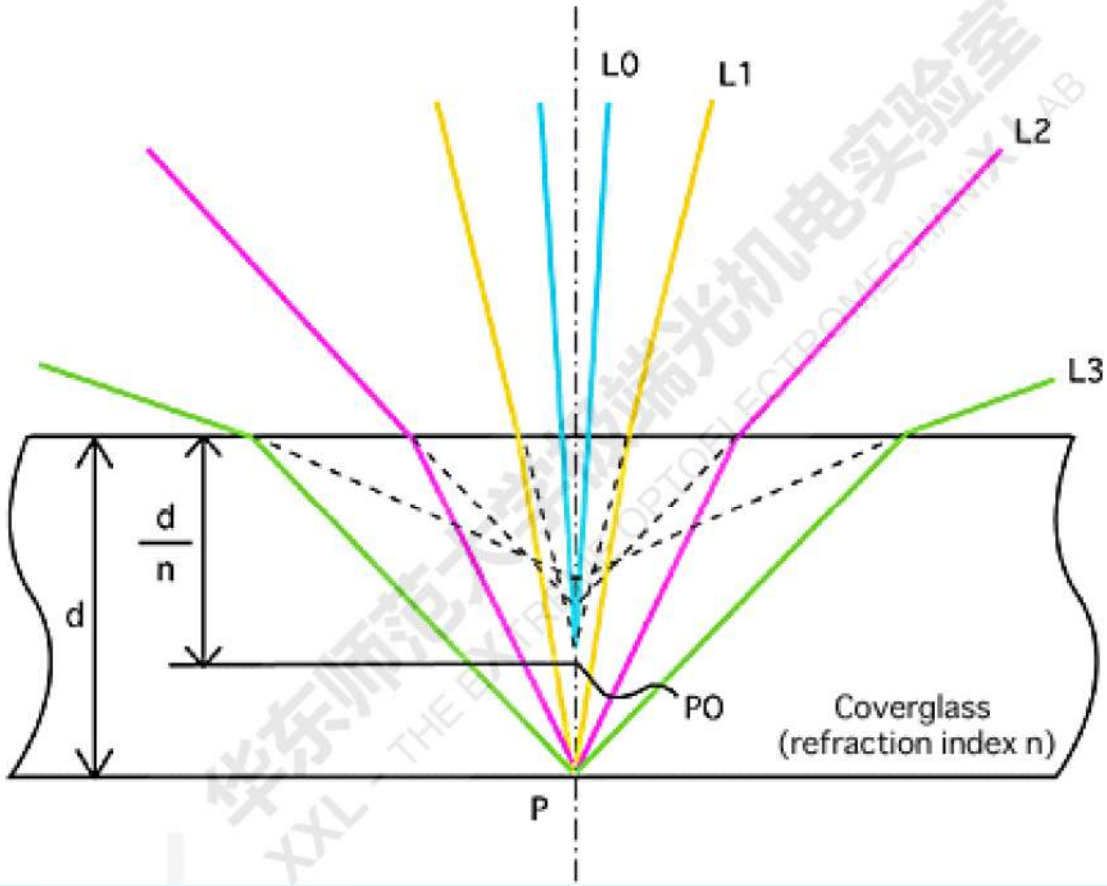


Large size
Low resolution

Small size
High resolution

1. Long working distance inherently associated with low resolution!

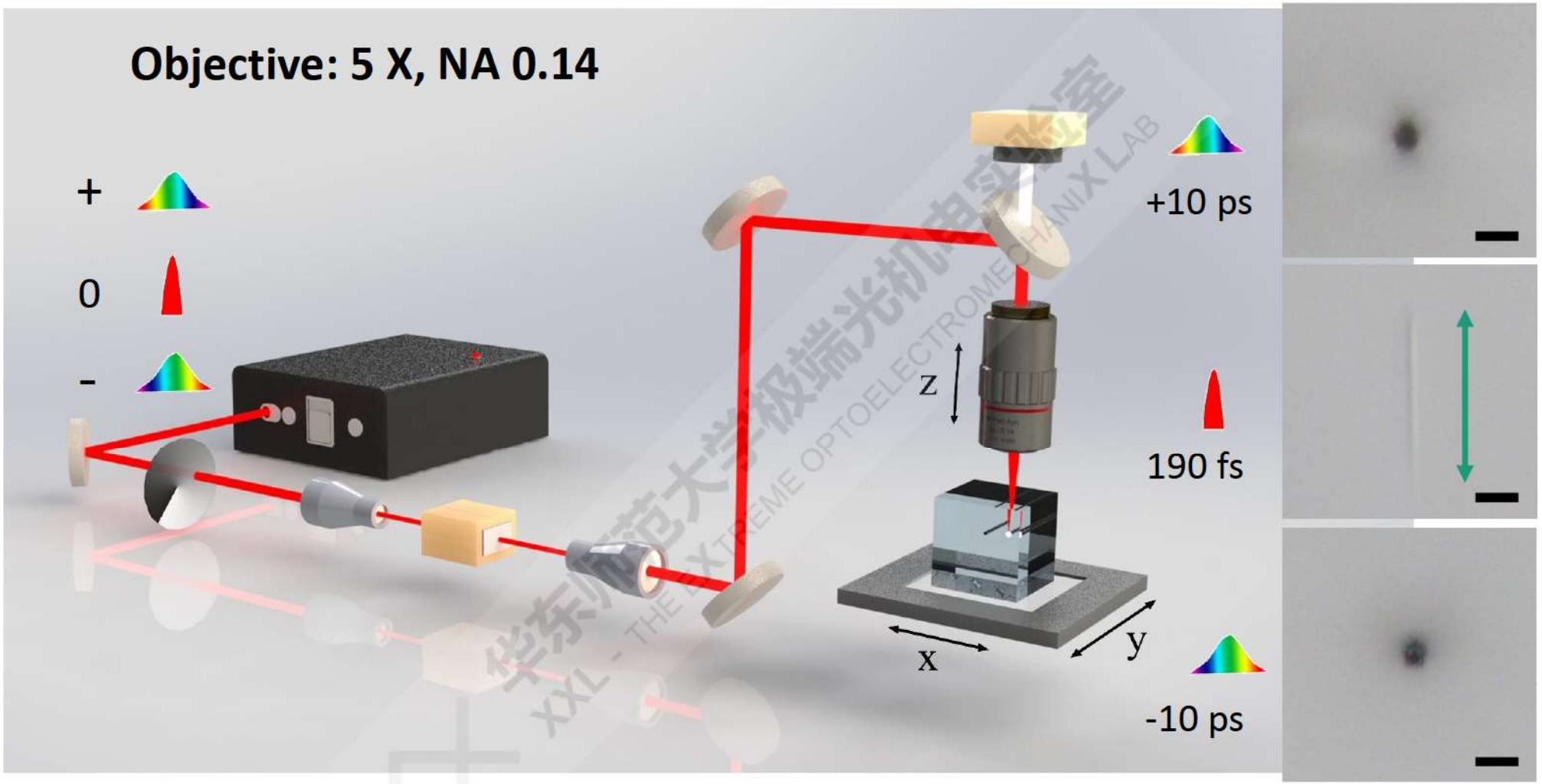
The challenges for large scale glass printing



Concept of spherical aberration

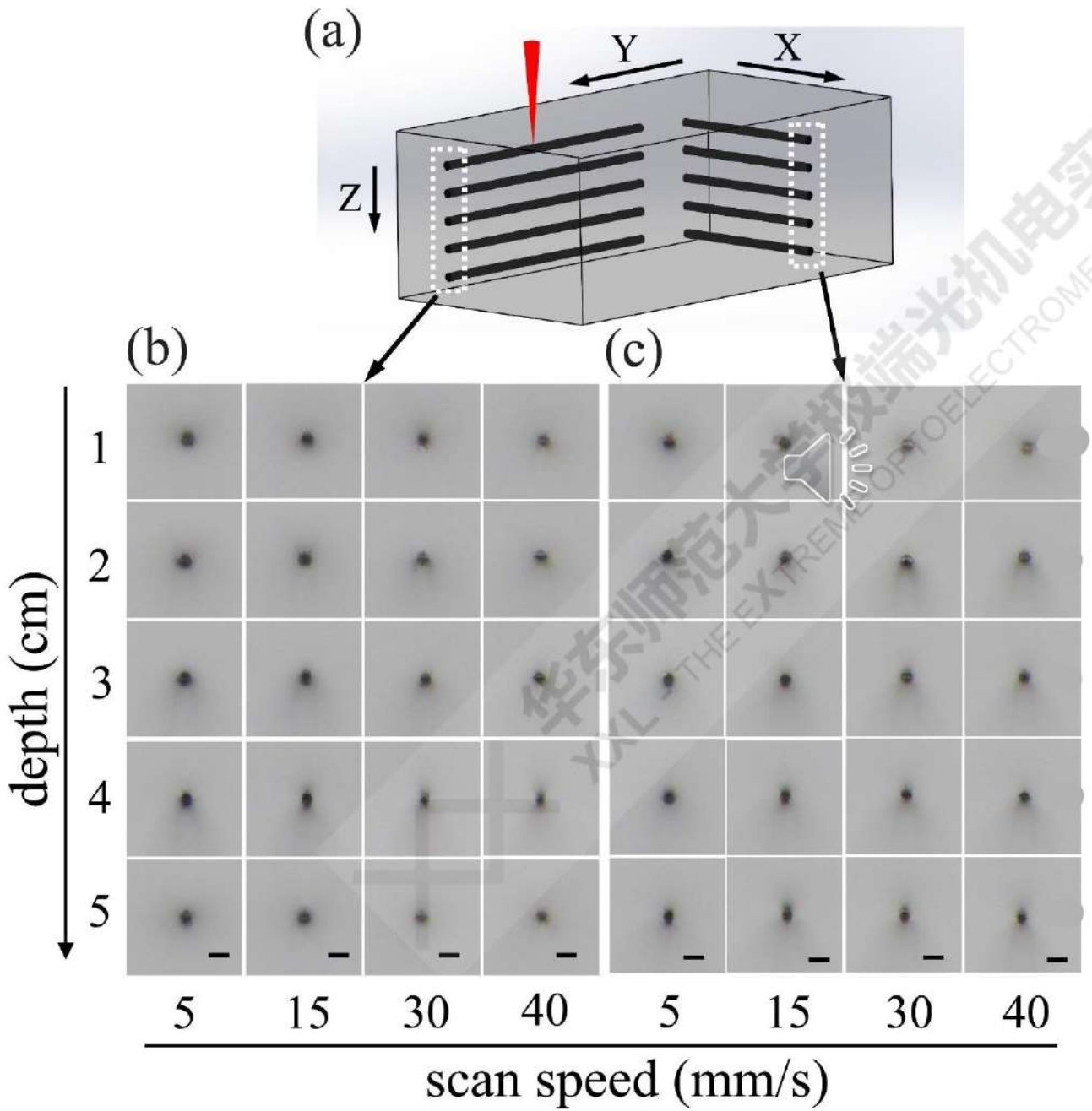
2. Focusing deeply into glass inherently leads to low axial resolution!

Our solution: chirped femtosecond laser modification



Schematic of the experimental setup. Scale bar, 25 μm.

Cross-section of lines written by 10 ps laser pulses

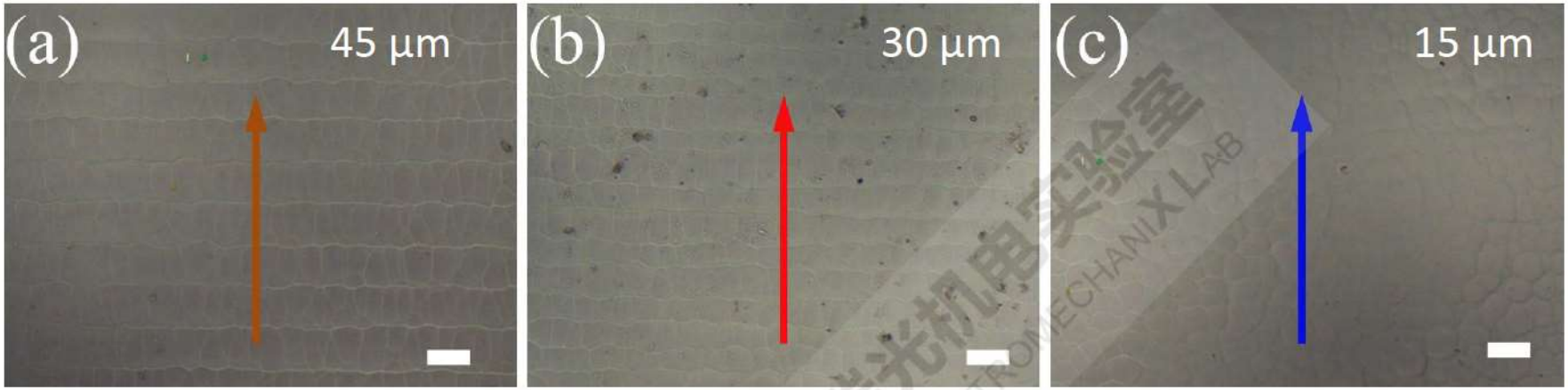


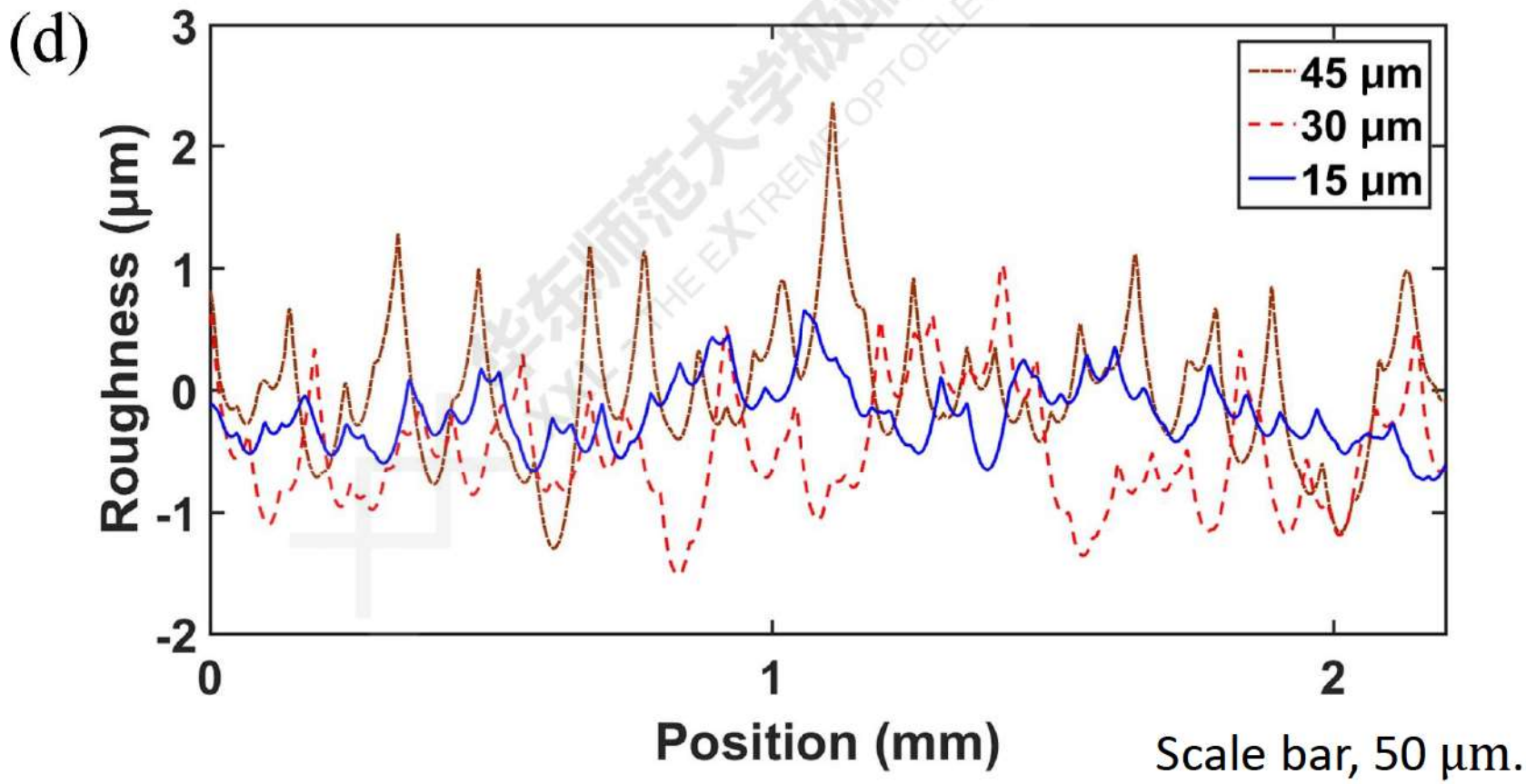
Spherical spot independent of

- depth
- direction
- speed

Scale bar, 25 μm.

Evidence showing a resolution of ~15 μm in Z direction





Scale bar, 50 μm.

Another challenge: controlling chemical etching

We can produce isotropic, aberration-free modification in glass. But this is not sufficient.

Due to the well-known nanograting effect, the chemical wet etching depends on the orientation of polarization of the writing beam. How can we remove such dependence to achieve homogeneous writing of complex 3D structures ?

Polarization sensitivity vs pulse duration

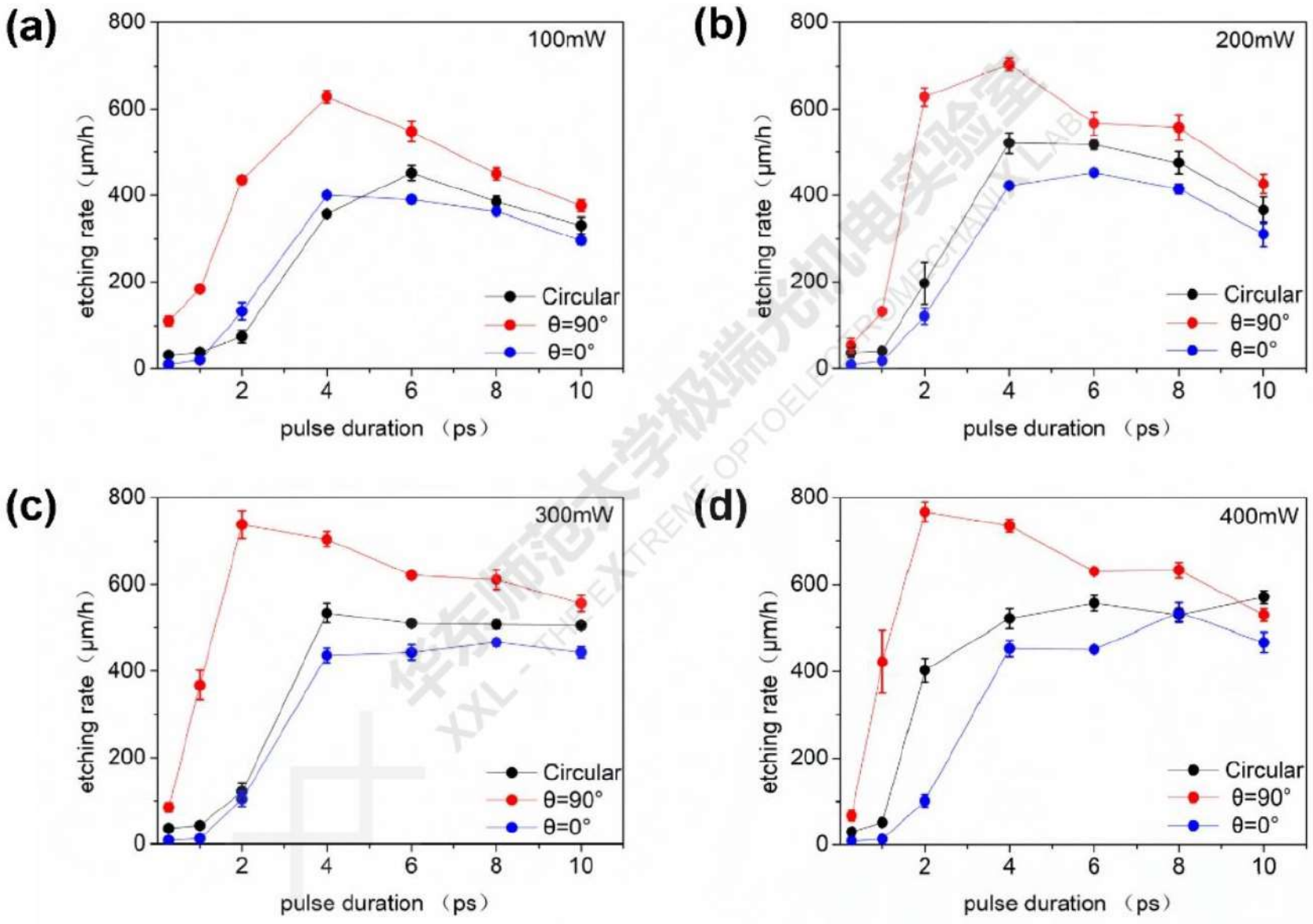
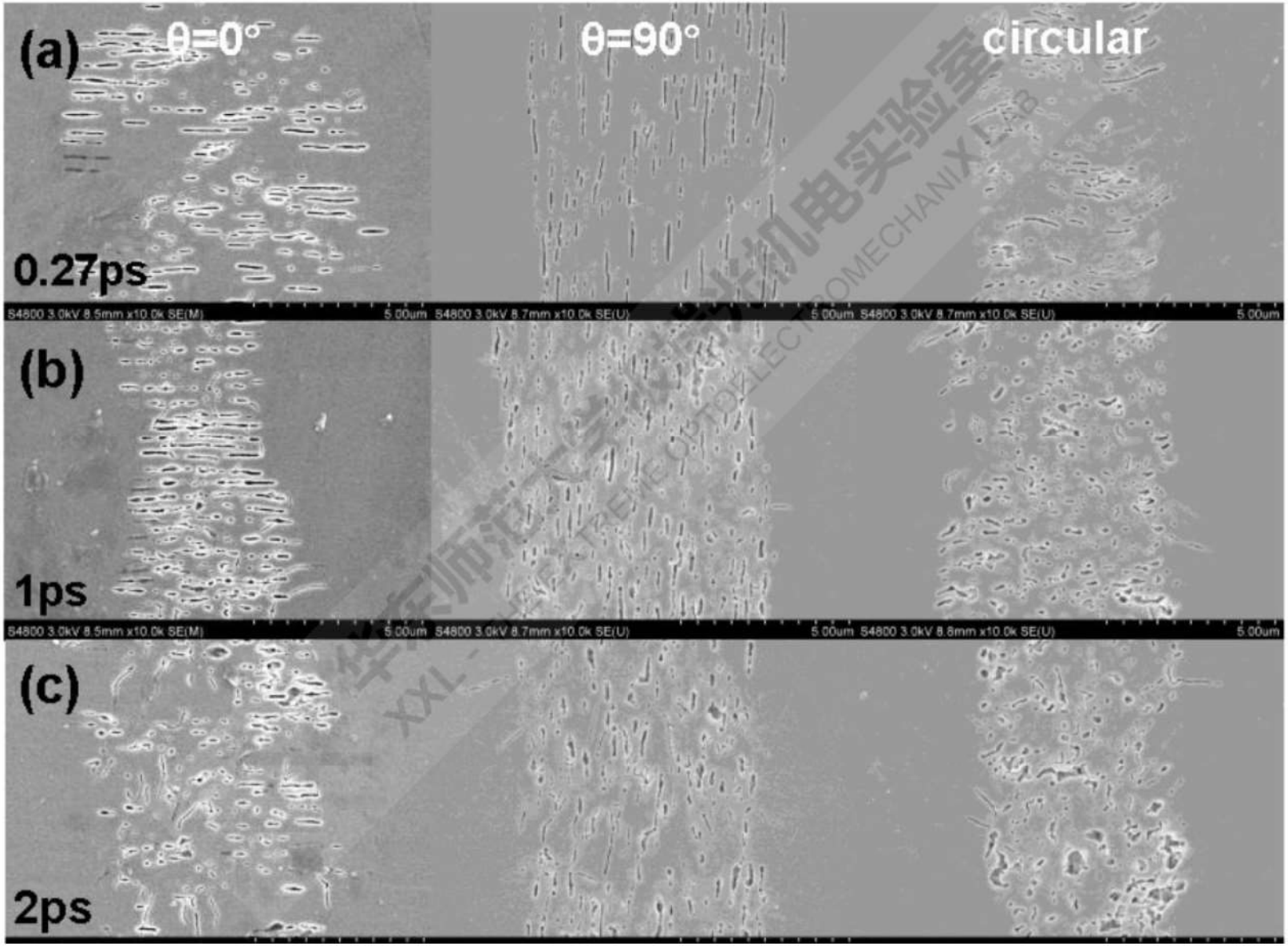


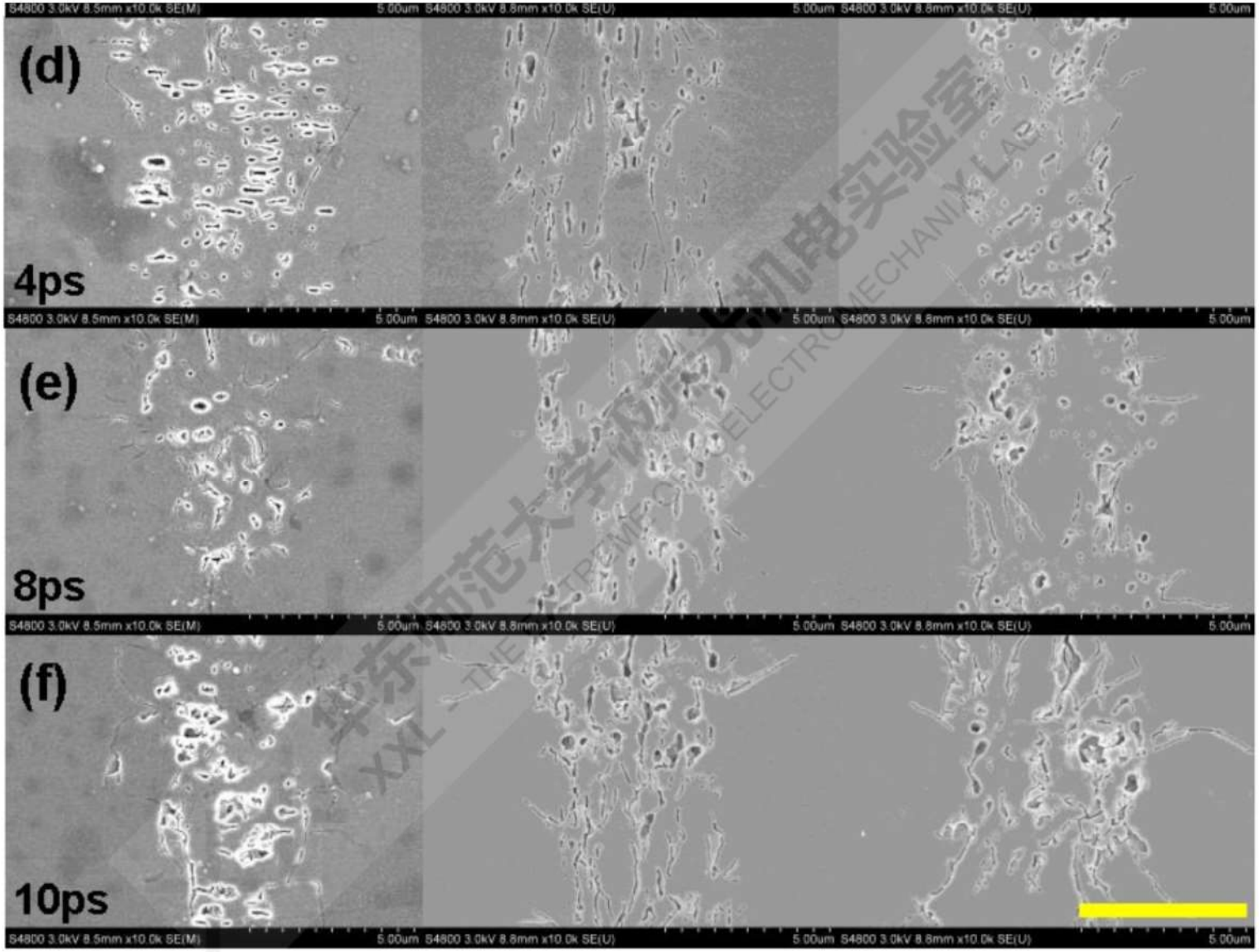
Fig. 2. Etching rates of laser modified lines in fused silica versus pulse durations at different polarization conditions and laser powers: (a) 100 mW; (b) 200 mW; (c) 300 mW; (d) 400 mW.

Shorter pulses: nanogratings



X. Li, et al, Applied Surface Science 485 (2019) 188–193

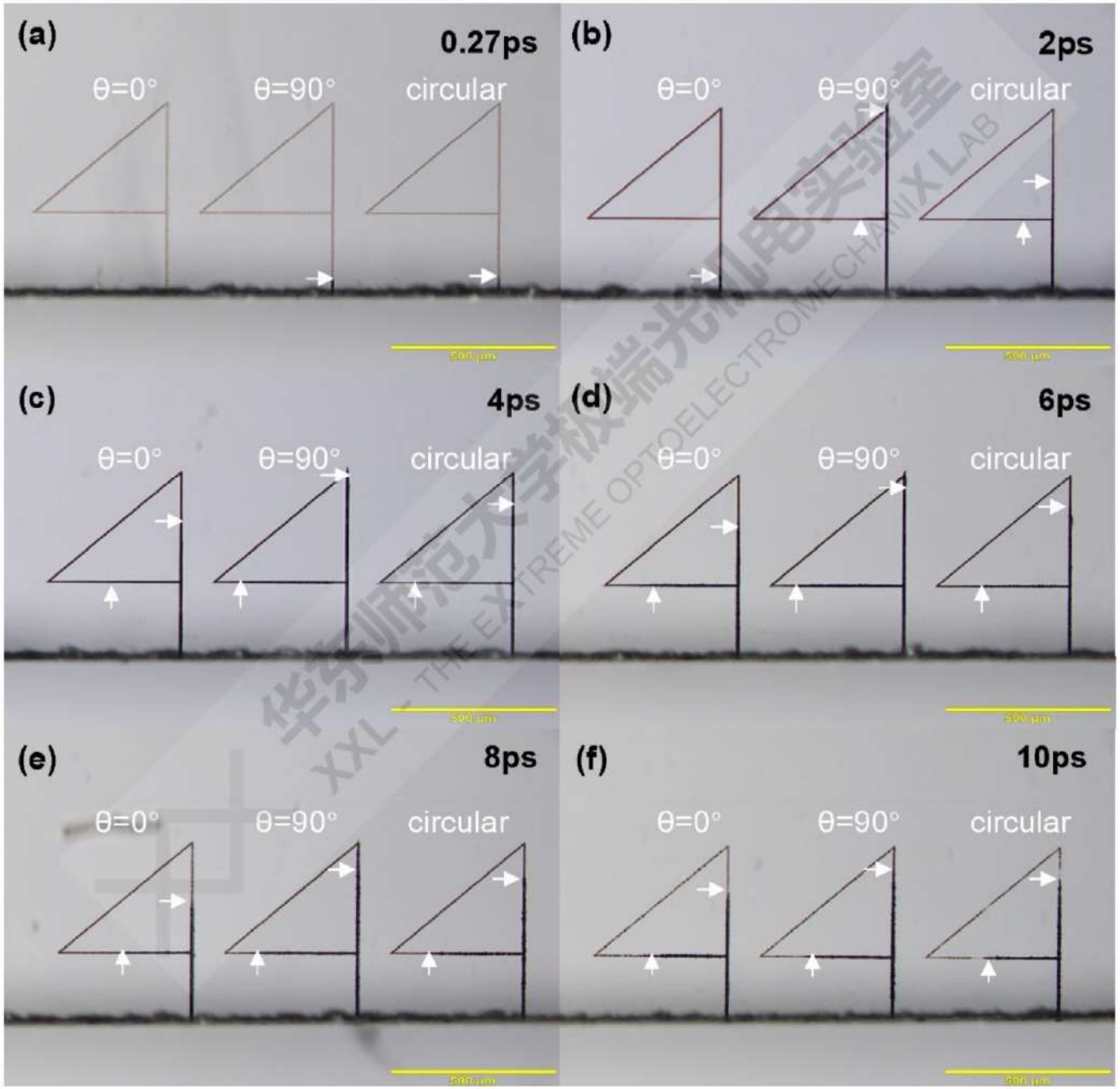
Longer pulses: stress induced cracks along scan direction



Scale bar: 5 μm

X. Li, et al, Applied Surface Science 485 (2019) 188–193

Selective etching independent of polarization

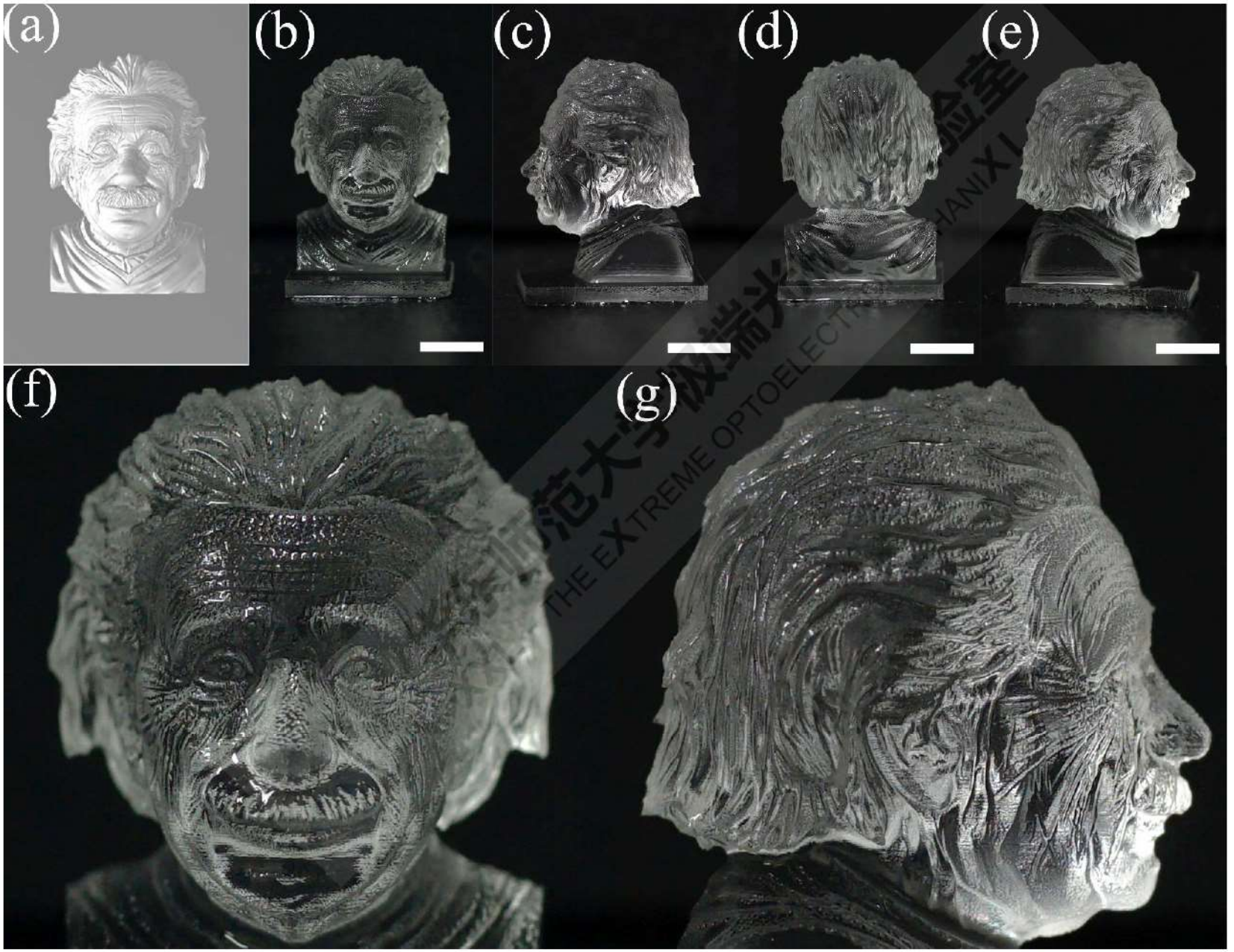


Maintain the high resolution at large and variable depth.

Produce polarization insensitive internal modification.

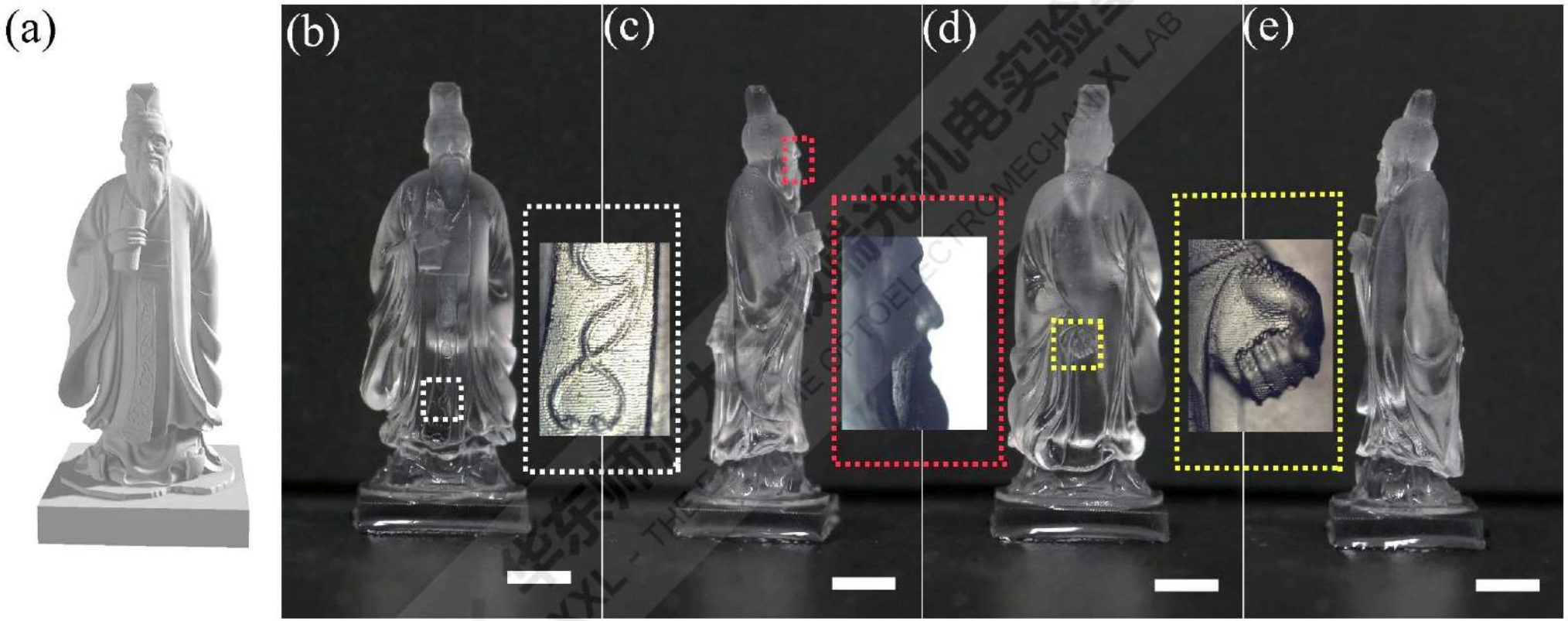
With the picosecond laser, the difficulties in maintaining the high axial resolution at large and variable depth and in achieving the polarization insensitive internal modification can be overcome at once! We can move on to practice the 3D glass printing using these unusual yet lucky findings.

Structures: Einstein of a height of 2 cm



Scale bar, 5 mm.

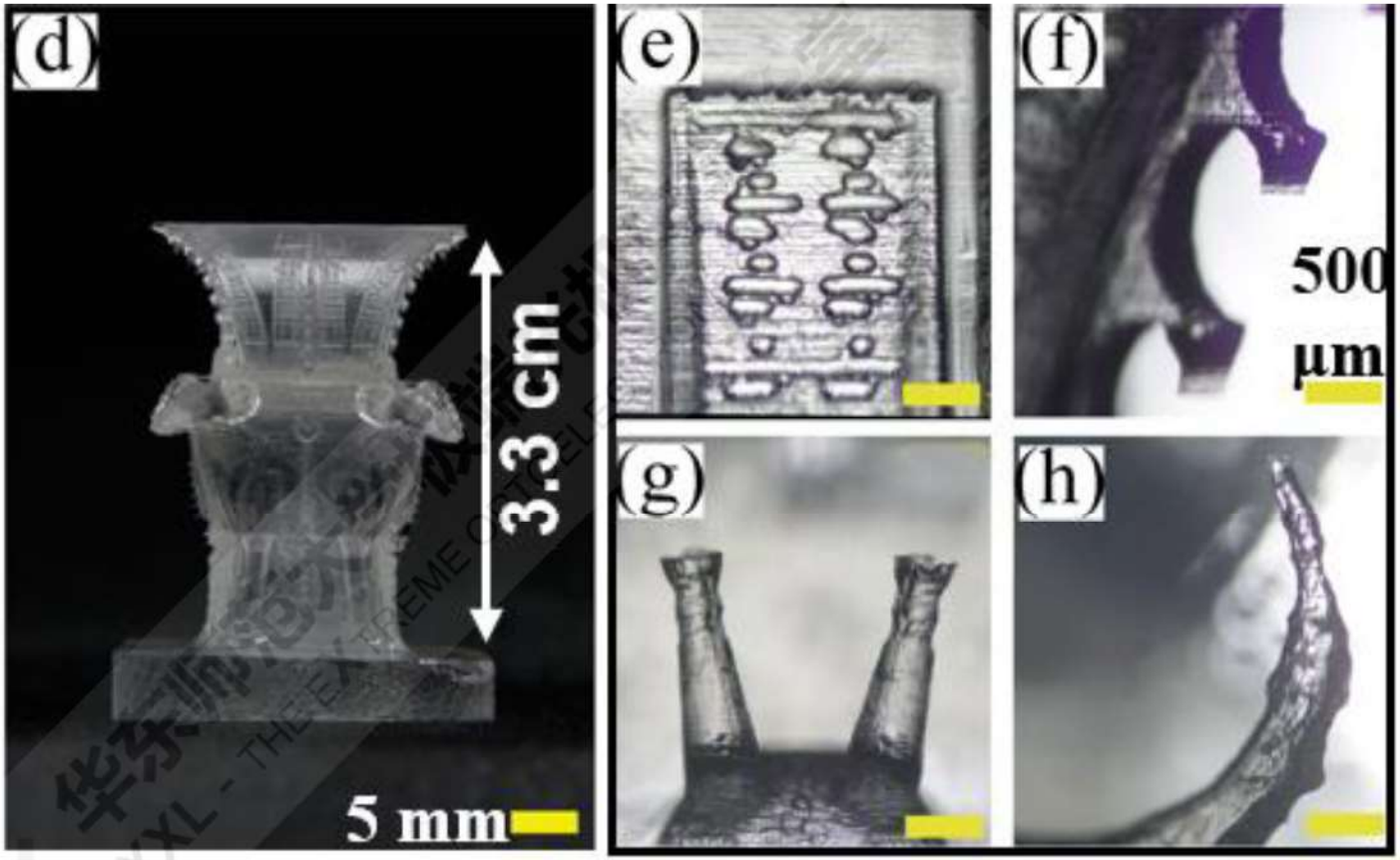
Structures: Confucius of a height of 3.8 cm



P. Wang et al, Micromachines **2019**, *10*, 565;
doi:10.3390/mi10090565

Scale bar, 5 mm.

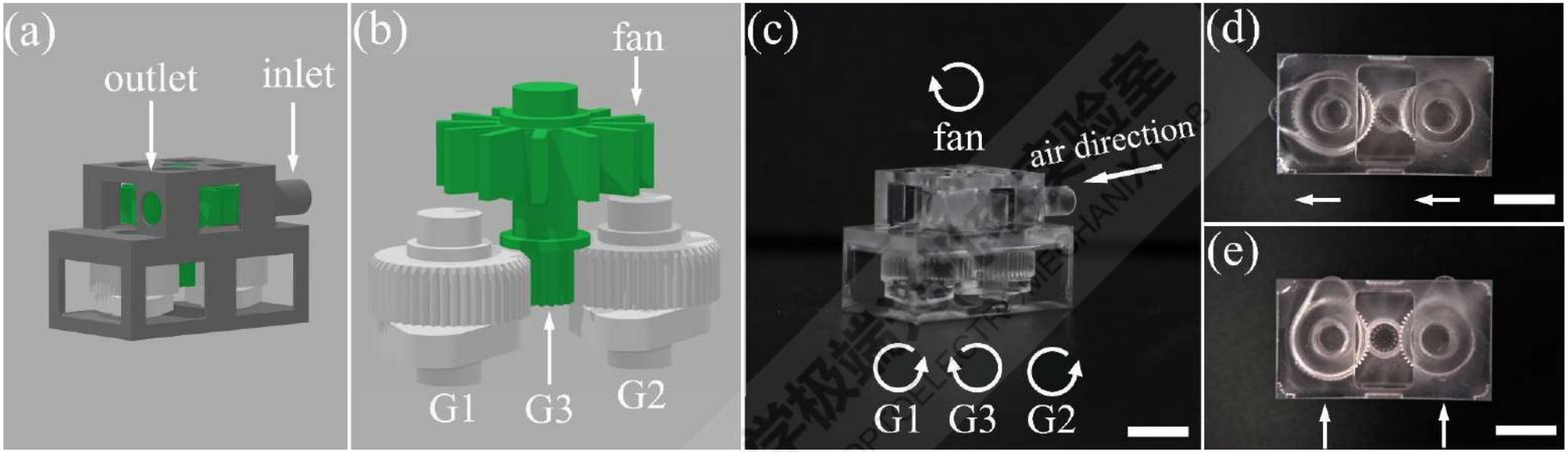
Structures: Four-ram Zun (B. C. ~1000 yrs, China)



Statue of Four-ram Zun
one of the most famous bronze ware

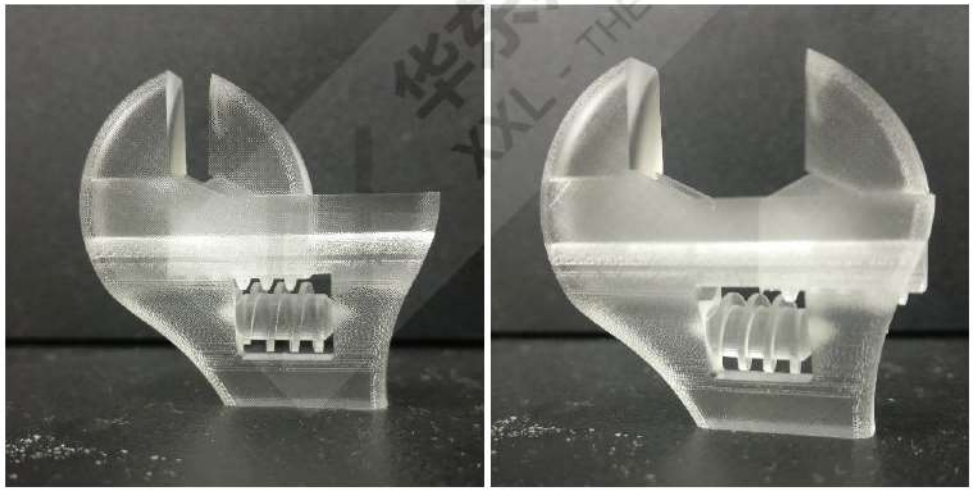
H. Zhang et al, Submitted for publication

Structures: micromachines with movable parts



Air turbine

Scale bar, 5 mm.

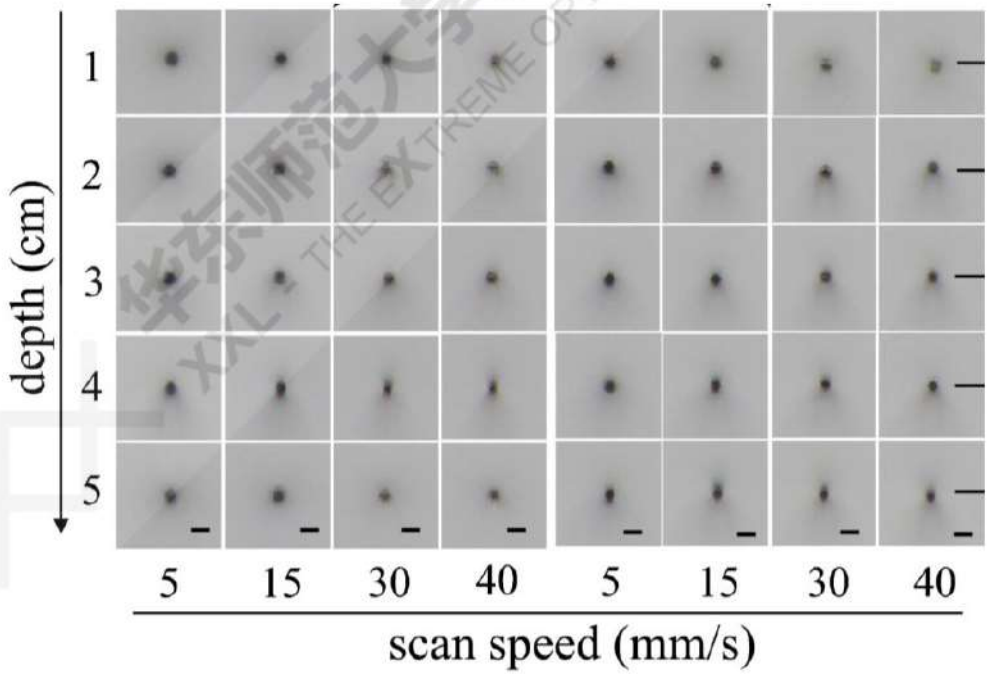


4 cm

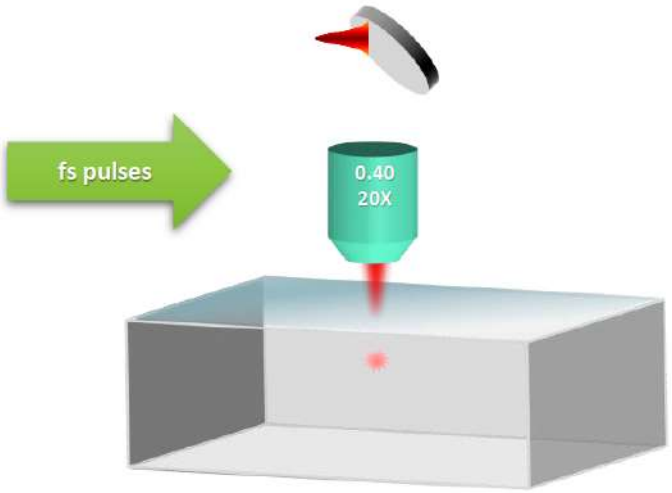
Adjustable wrench

New physics of uniform and isotropic XYZ resolution along Z axis:

Incubation induced localization

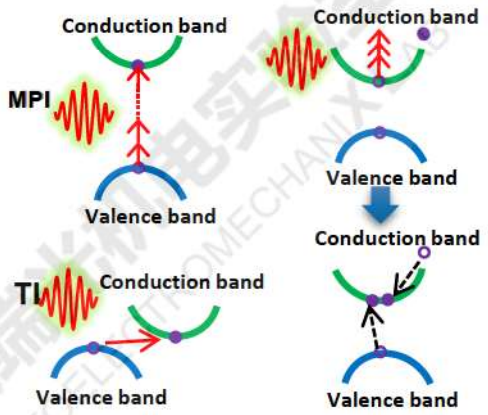


General physics of ultrashort laser-material interaction

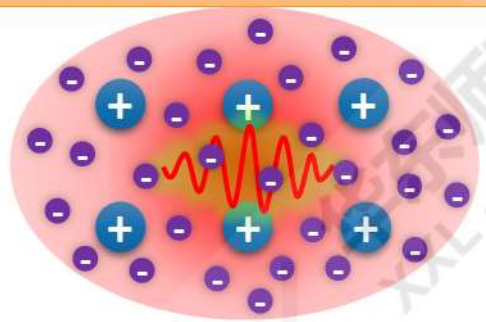


(a) Free electron generation

Photoionization & Avalanche ionization



(b) Plasma absorption

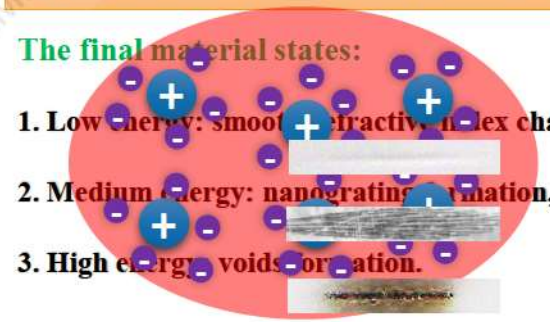


Critical plasma density:

$\rho_c = \epsilon_0 m_e \omega_0^2 / e^2$,

$\rho_c \sim 10^{21}$ cm^{-3} @ 800nm.

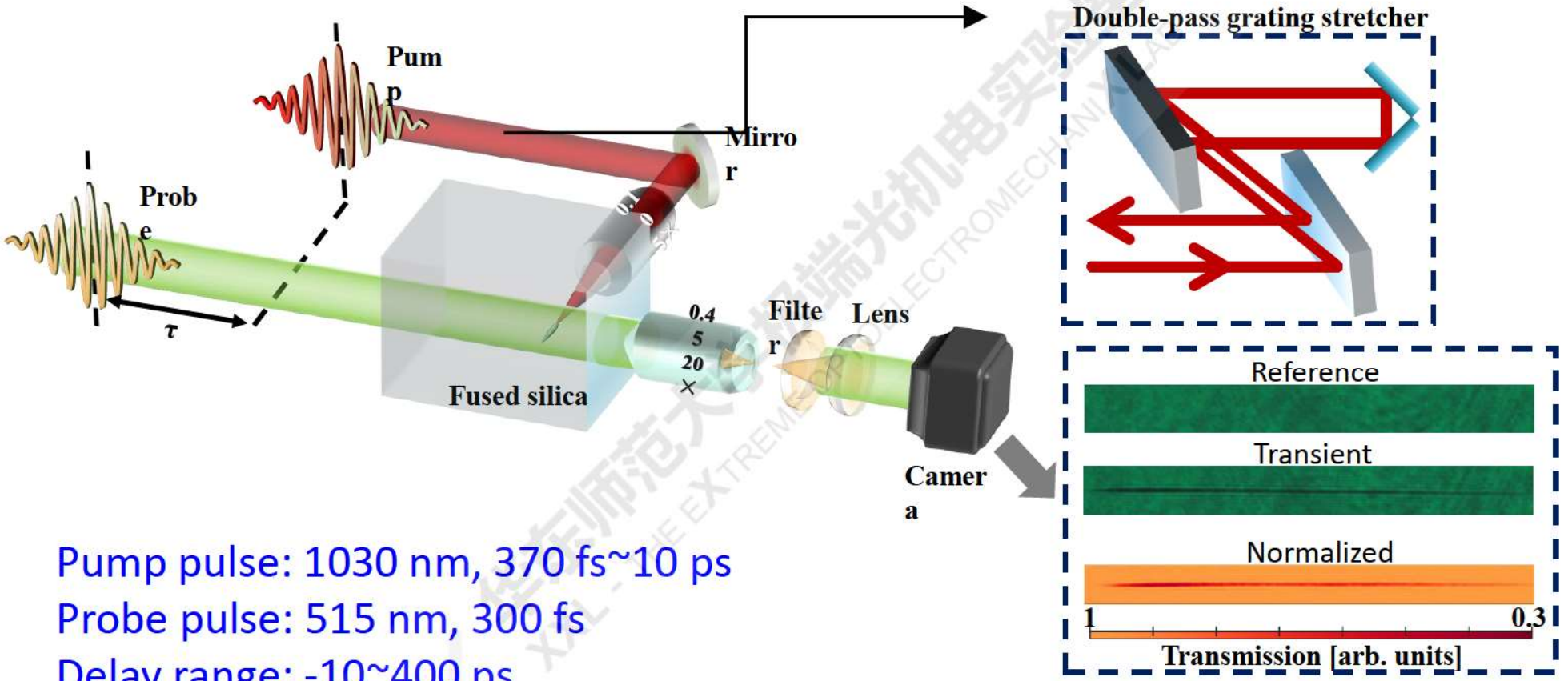
(c) Lattice heating & relaxation



Hot plasma energy transfer to the lattice (~10ps),
Shock wave generation (~100ps),
Thermal diffusion (~10ns),
Resolidification (~1μs).

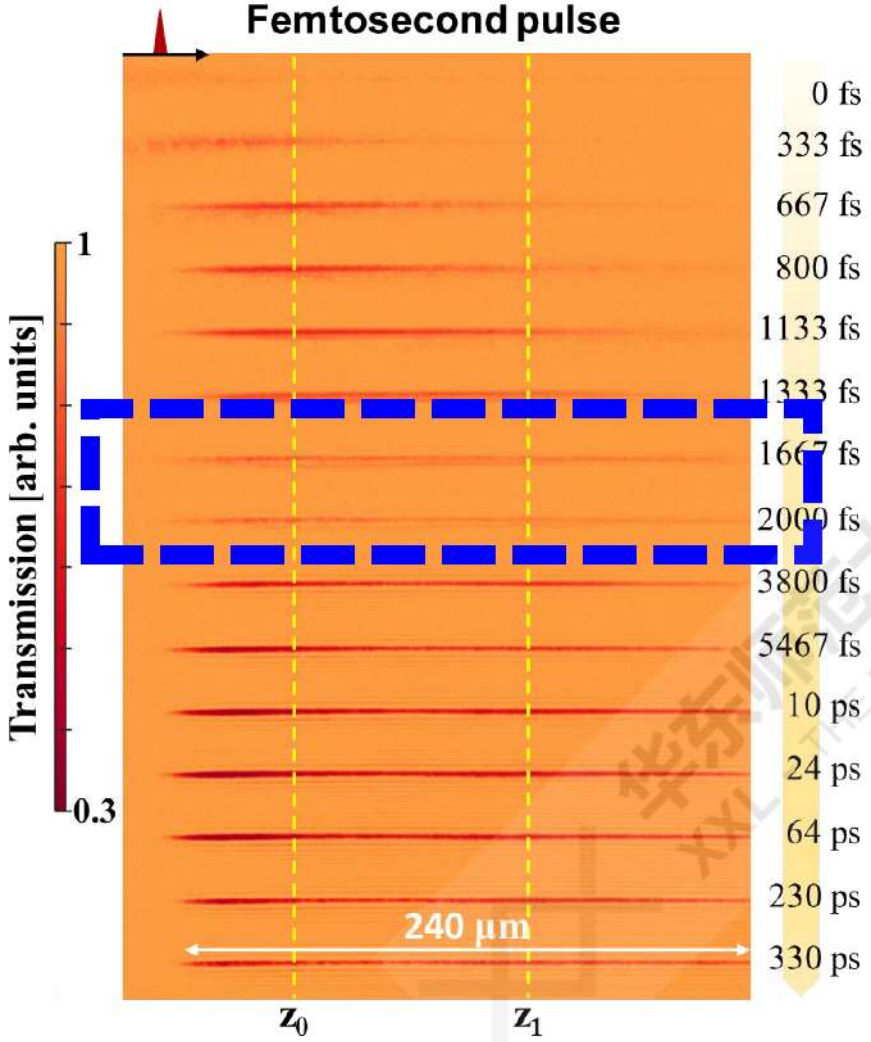
R. R. Gattass & E. Mazur, *Nat. Photonics* **2**, 219 (2008).
R. Osellame et al., *Femtosecond Laser Micromachining* (2012).
K. Sugioka & Y. Cheng, *Light: Sci. Appl.* **3**, e149 (2014).

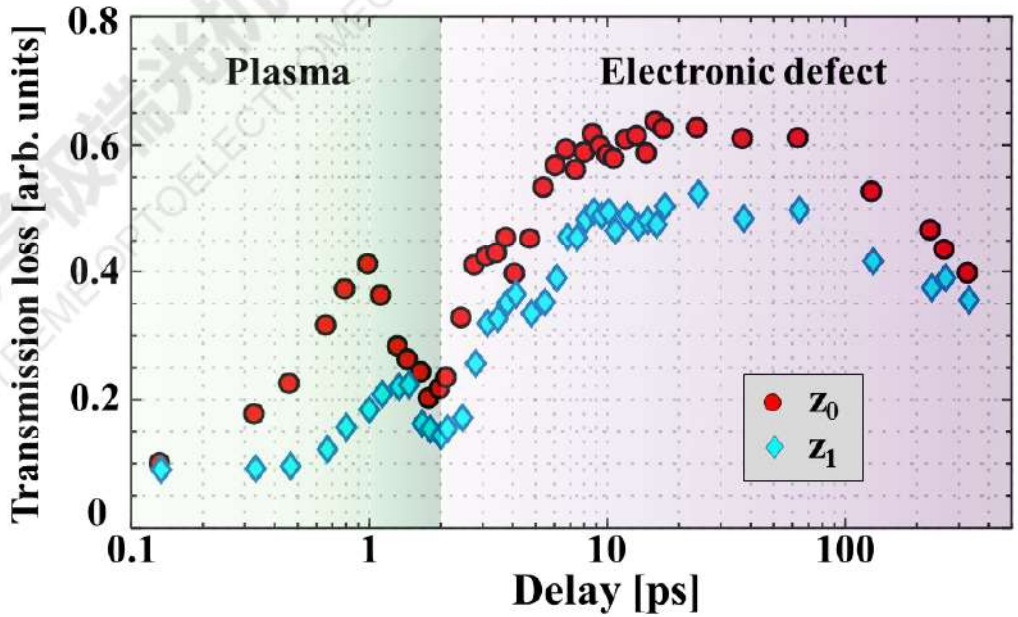
Pump-probe measurement revealing plasma dynamics



Pump pulse: 1030 nm, 370 fs~10 ps
Probe pulse: 515 nm, 300 fs
Delay range: -10~400 ps
CMOS camera: 1024x1280 pixels, RGB 8-bit/16-bit dynamic range

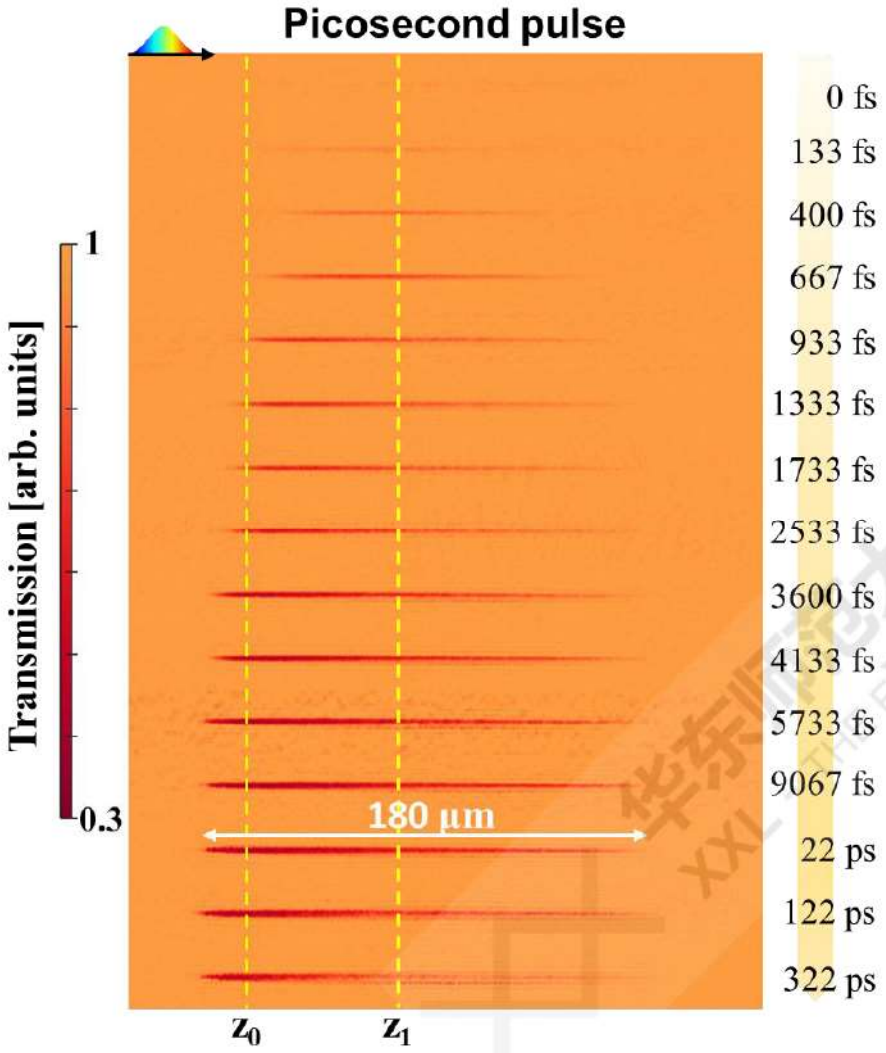
Plasma images from 0 fs to 330 ps: single fs pulses

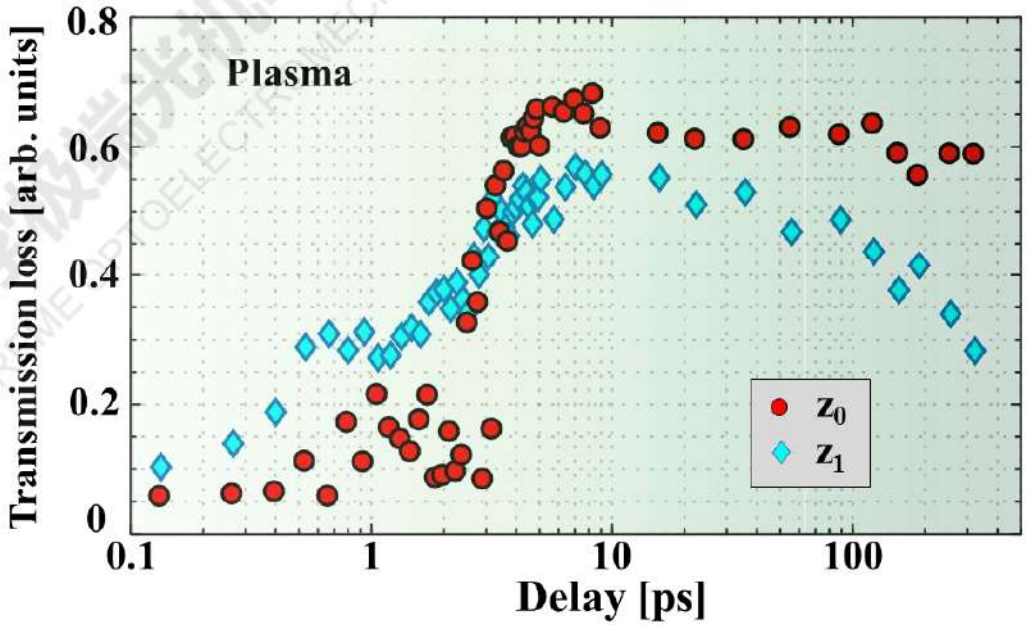




The plasma absorption becomes weaker in the blue frame and then again gets stronger. Dynamics plotted in the right panel.

Plasma images from 0 fs to 322 ps: single chirped ps pulses

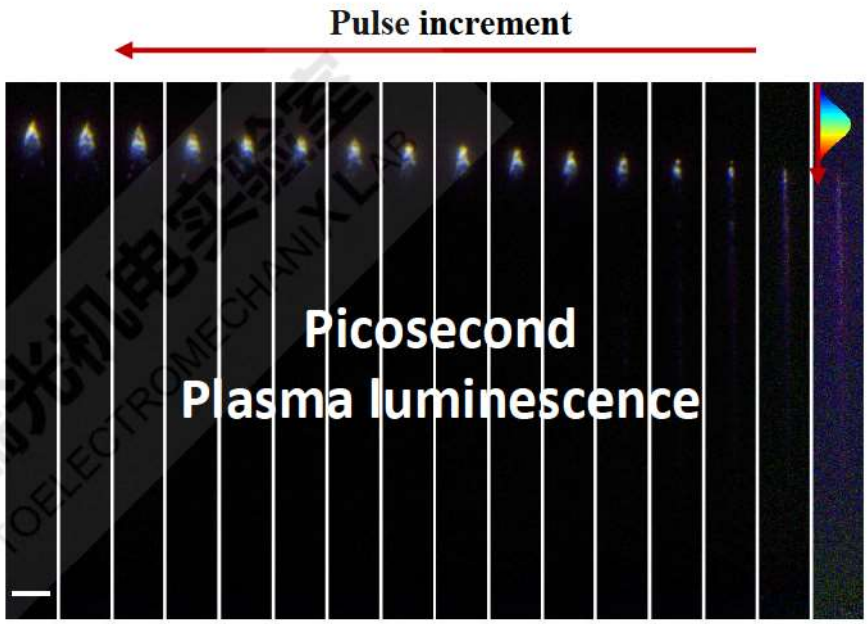


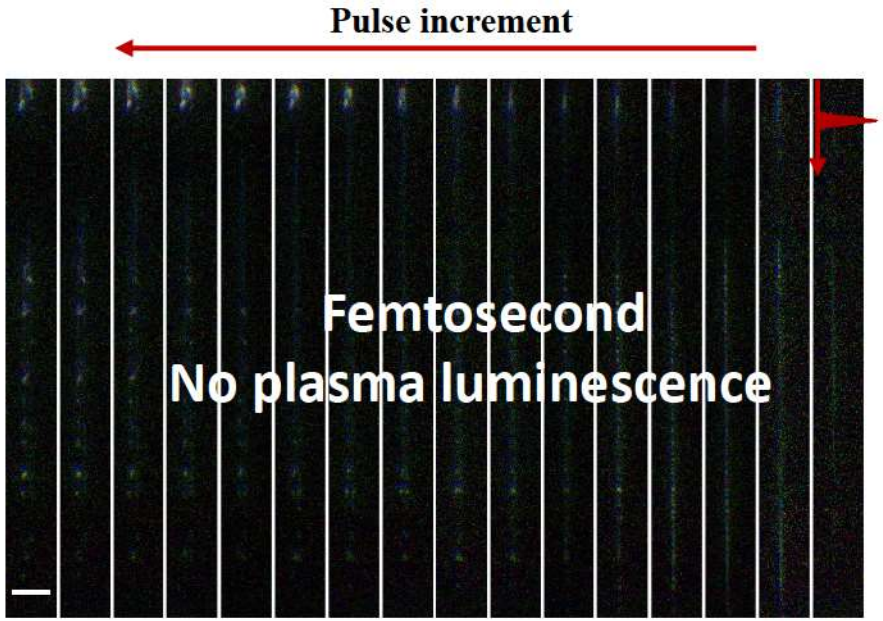


The plasma absorption keeps going stronger until saturates. Dynamics plotted in the right panel.

Multiple-pulse incubation effect

1. For picosecond pulses, incubation leads to highly localized and strong plasma luminescence !!!
2. Localization should be a result of light blockage by the high-density plasma.
3. This could be a universal effect in various transparent materials but need to be checked !





Can this technique be economically valuable for the mankind?

YES!

First example: chemistry

Challenges in traditional chemical reaction

c.1556

1956

2008

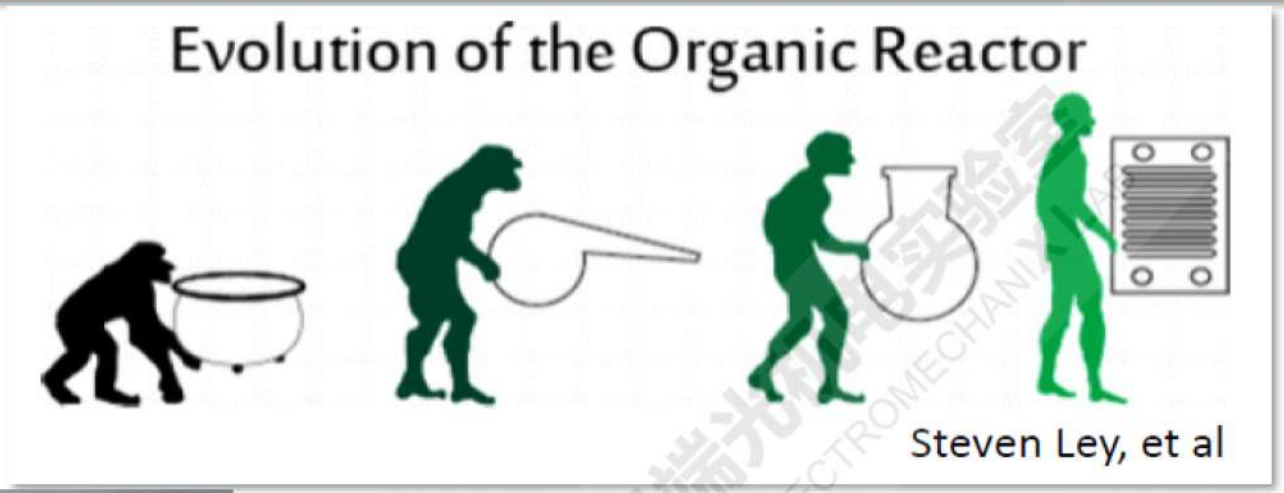
Nothing has changed in nearly 500 years

Big issues:

- Low reaction efficiency
- High reagent consumption
- Poor scalability
- Low heat diffusion

.....

New trend: miniaturization of reactor



Batch chemistry

Microreactor chips

Flow chemistry

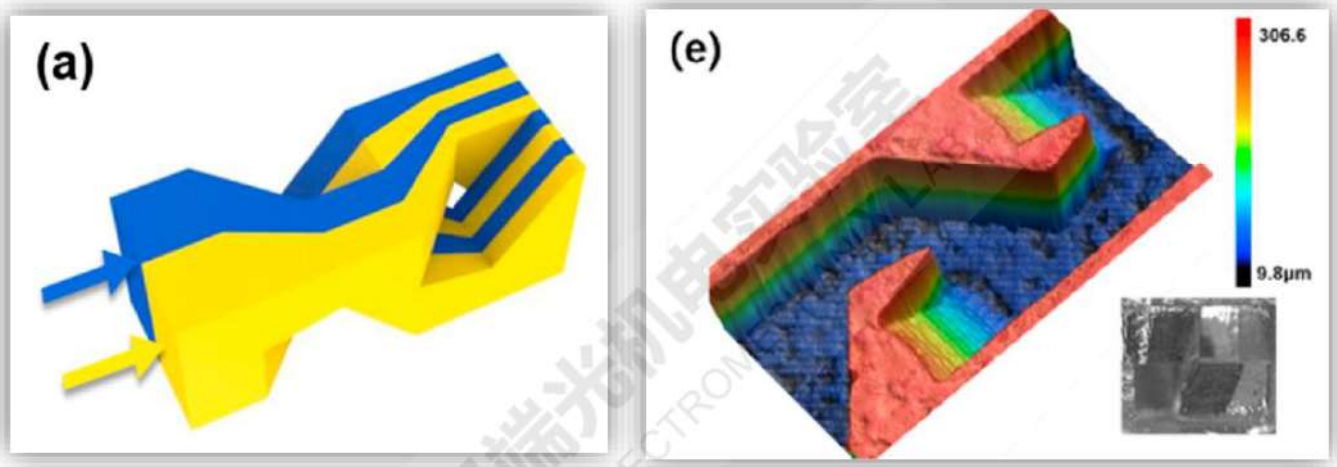
Benefits:

- **Higher** reaction rates
- **Purer** products
- **Better safety**
- **Integration of synthesis and analysis steps**
- **Rapid optimization**
- **Easy scale-up**
- **Reaction conditions not possible using traditional methods**

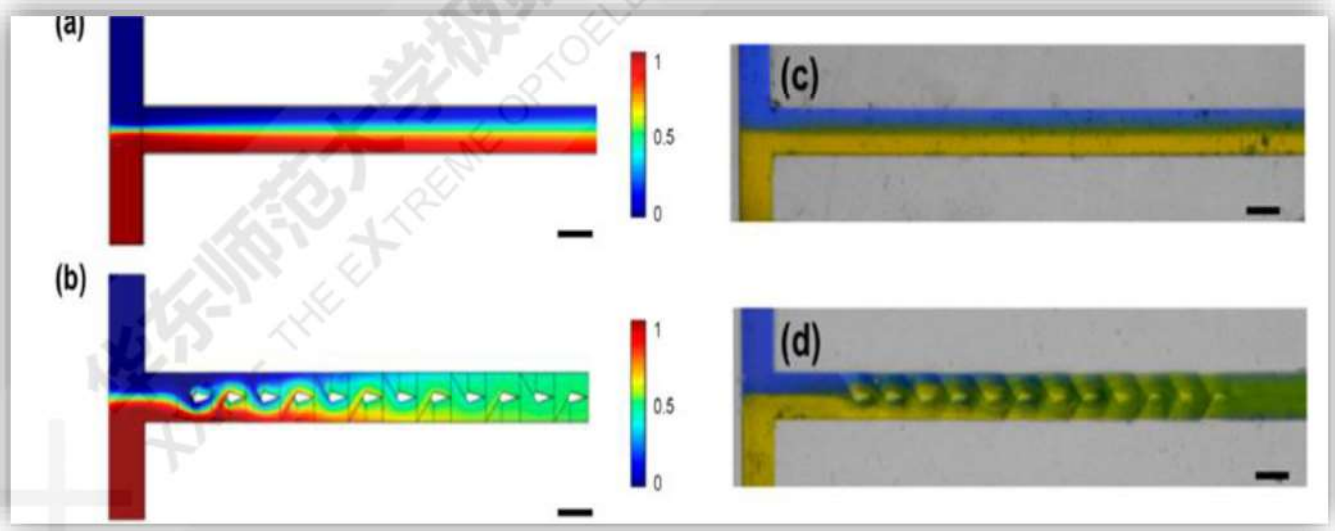
Concept of a 3D microreactor

3D mixing unit:

Schematic and internal 3D structure



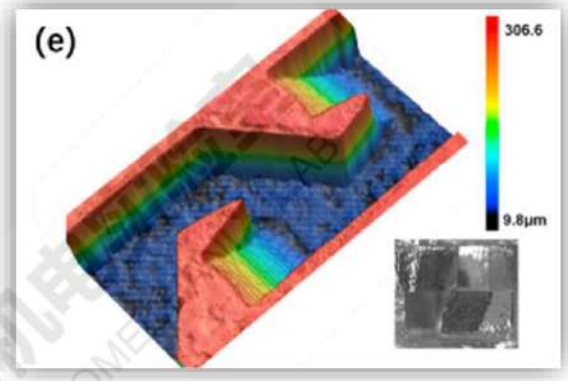
Performance comparison in 1D and 3D microchannels

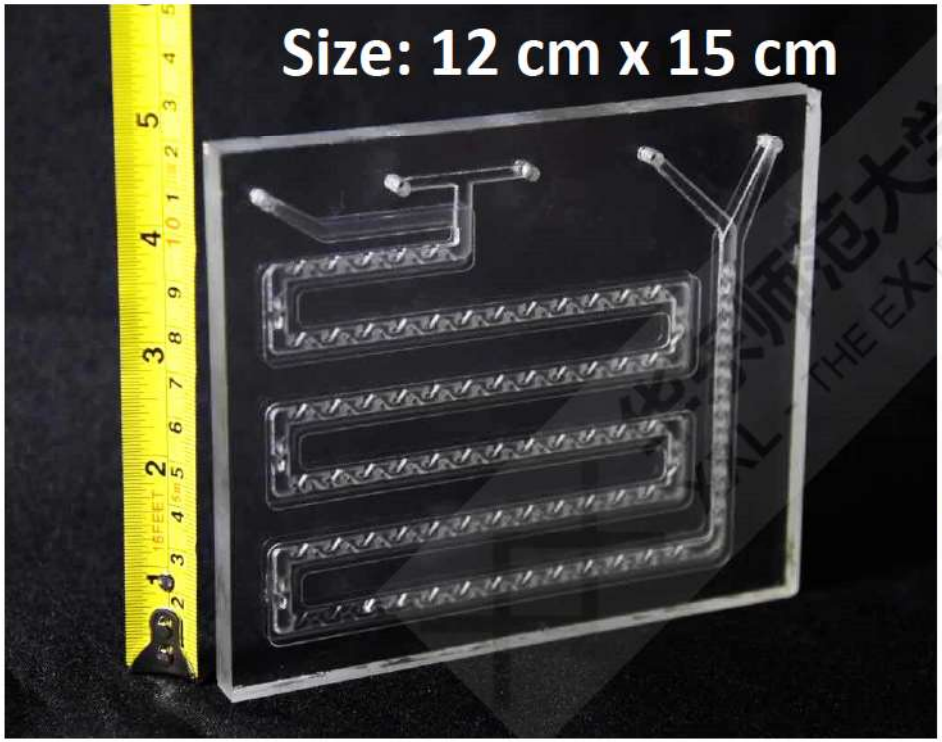


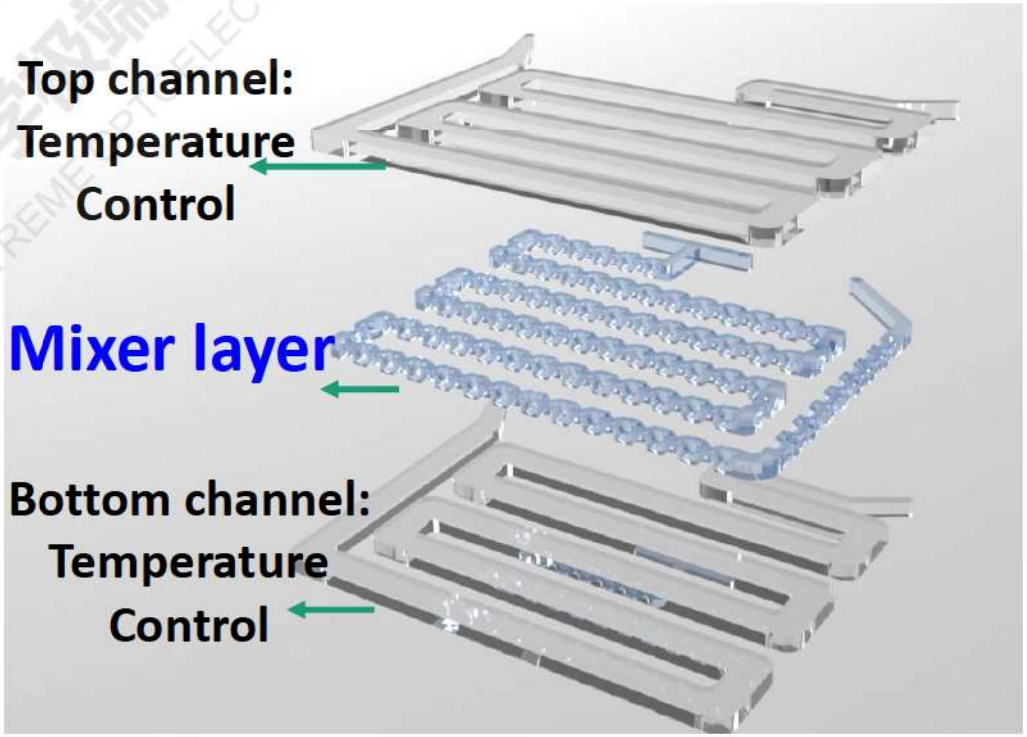
Mixing performances in 1D and 3D microchannels (Left: modeling; right: experiment)

Jia Qi, et al., Micromach. **11**, 213 (2020).

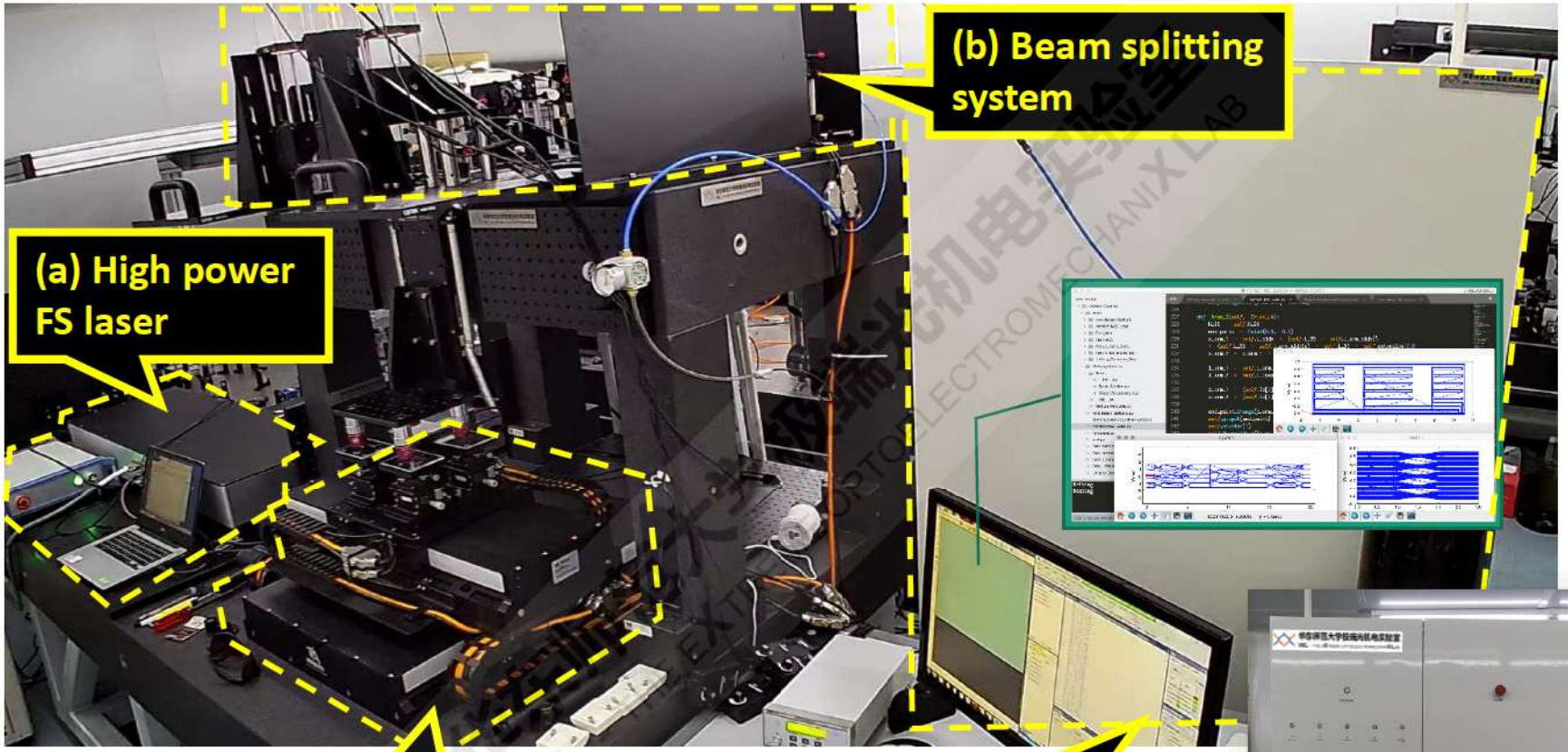
Industrial scale 3D microreactor







Increase the efficiency with the chirped pulses



Front view of the controlling case

Large focal volume combined with high axial resolution brings up the fabrication efficiency to an unprecedented level!

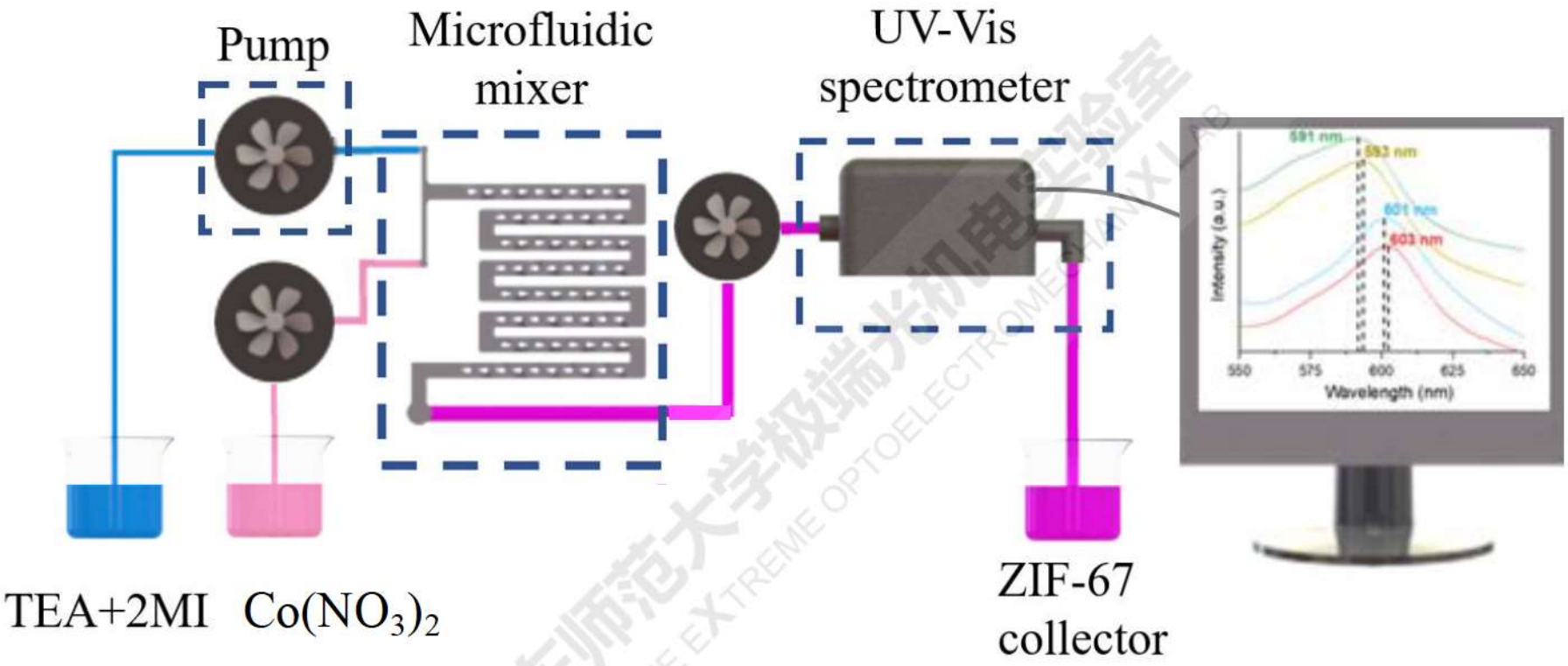
Four-foci focal system for speeding up the fabrication

Four-foci focal system for speeding up the fabrication

1. Four focal spots of individually tunable power
2. Large scale XY motion range of 30 cm by 30 cm
3. Inline real-time focus tracking system

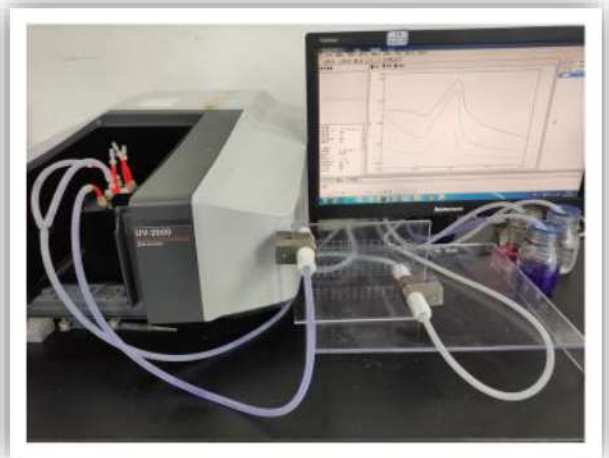
……

Size-controlled flow synthesis of porous crystals

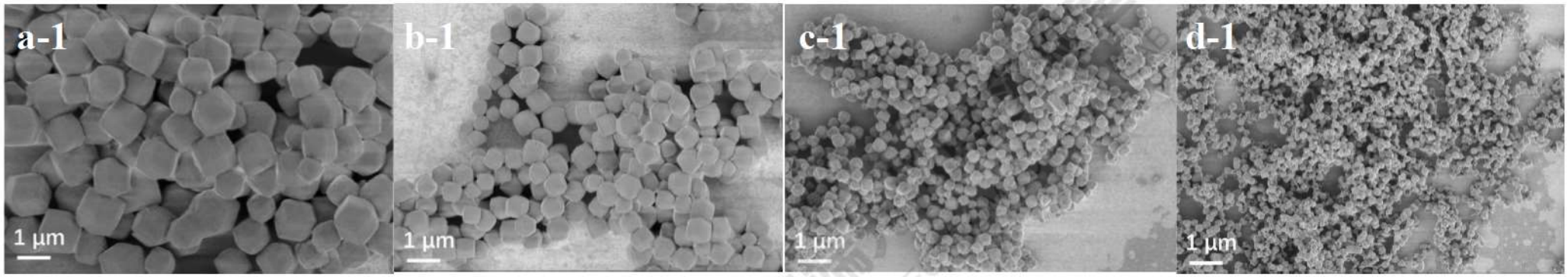


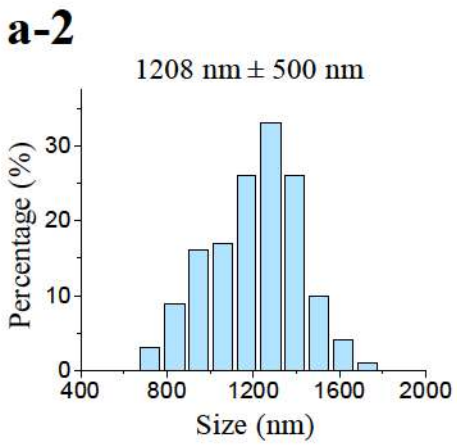
1. In-line monitoring of sizes of metal-organic frameworks (ZIF-67) crystals

2. Size-controlled flow synthesis

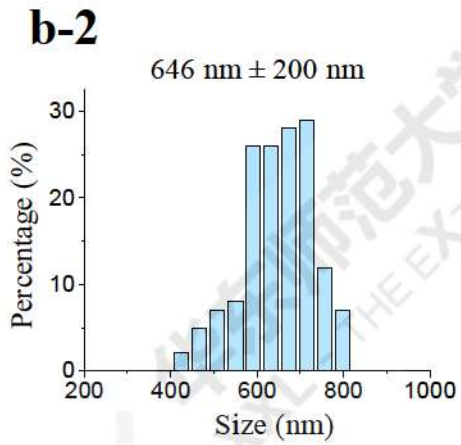


Size-controlled flow synthesis of porous crystals

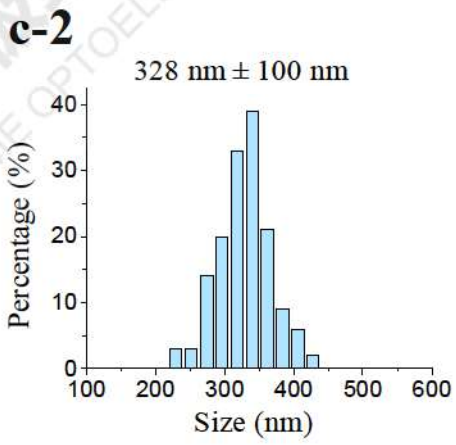




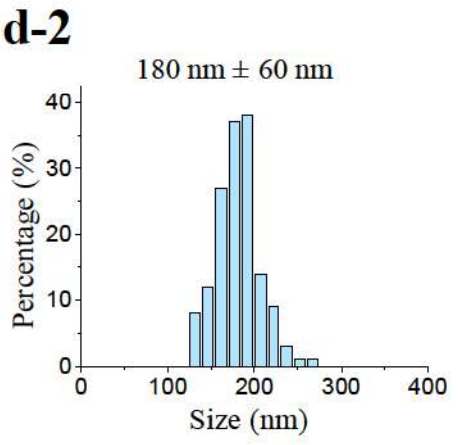
1200 nm



650 nm



300 nm



180 nm

Size decreasing with increased concentration of an additive

Just for fun: combining 3D printing and microfluidics

FULL PAPER

ADVANCED MATERIALS TECHNOLOGIES
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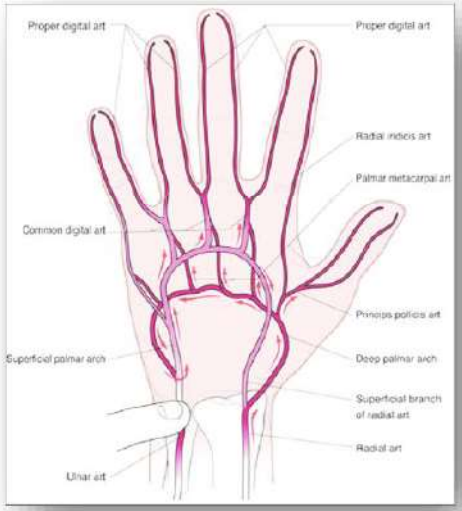
Check for updates

Freeform Microfluidic Networks Encapsulated in Laser-Printed 3D Macroscale Glass Objects

Zijie Lin, Jian Xu, Yunpeng Song, Xiaolong Li, Peng Wang, Wei Chu, Zhenhua Wang, and Ya Cheng**



Conclusions and outlook

1. We show how to make large 3D structures in glass with a micrometer scale fabrication resolution. The opportunity provided by the nonlinear interaction between loosely focused femtosecond laser pulses and transparent material is truly beyond imagination and extremely valuable for various applications!
2. We reveal the interesting new physics which exclusively exists in the multi-shot interaction regime between femtosecond laser pulses and glass.
3. We demonstrate fabrication of chemistry reactors for industrial applications taking the advantages of the findings.

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Thank you for your listening !