

Functionalizing metallic surfaces using femtosecond laser surface processing



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Lincoln

Fabrication and application of biomimetic micro/nano surfaces via Femtosecond Laser Surface Processing (FLSP)

Natural Functionalized Surface



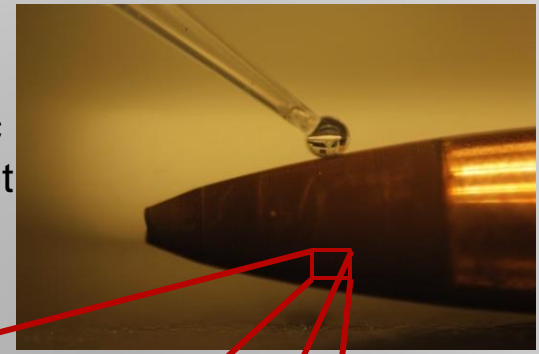
Superhydrophobic Lotus Leaf



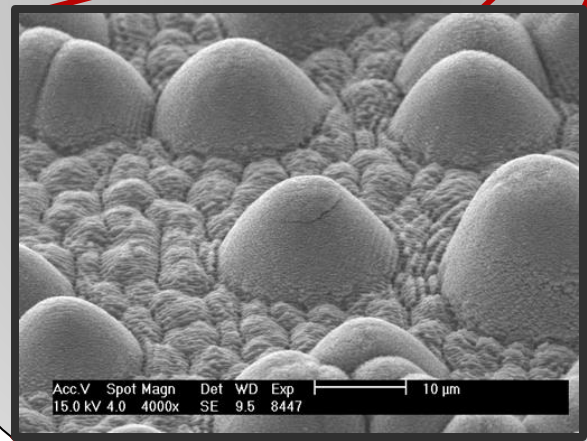
Natural Multiscale Surface Structure

K. Koch and W. Barthlott, *Philos. Trans. Royal Soc. A*, **367**, 1487–1509 (2009).

FLSP Biomimetic Surface



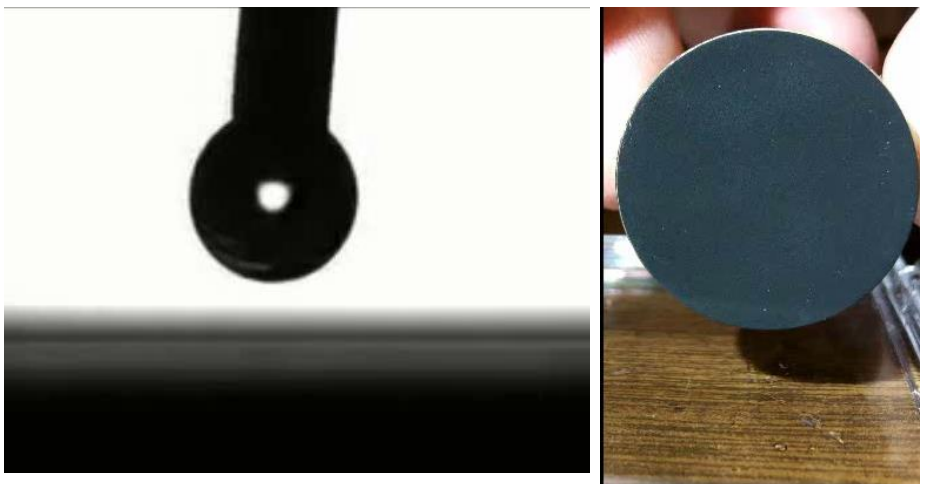
Superhydrophobic Copper Alloy Bullet



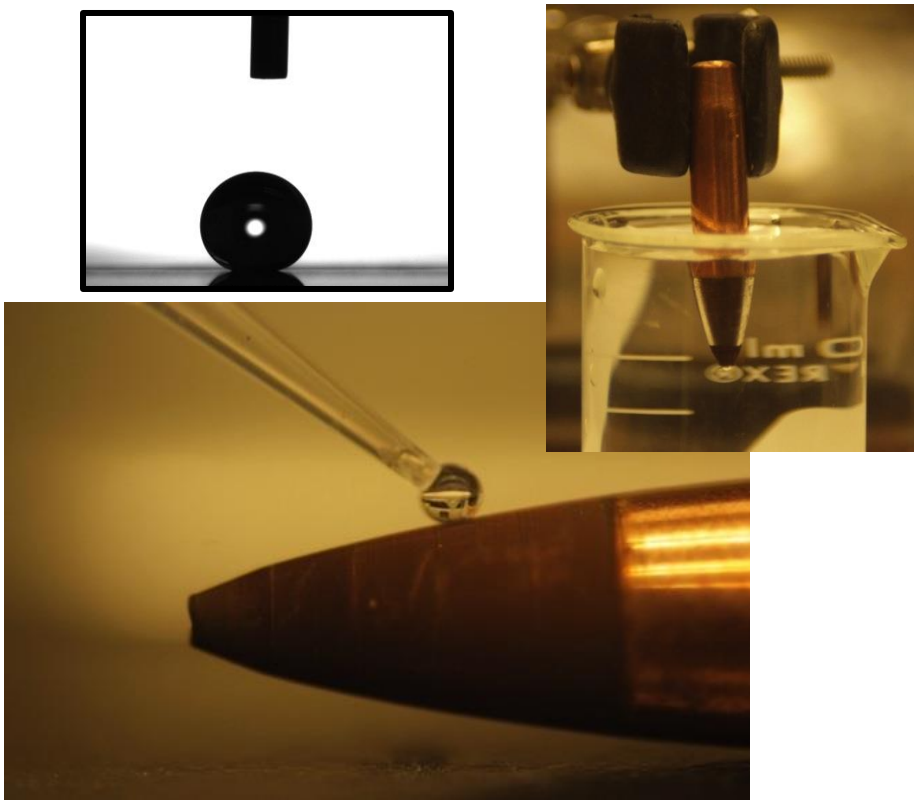
Fabricated Multiscale Surface Structure

The introduction of multiscale (micro- and nano-scale) surface features and controlled surface chemistry enables extreme control over the surface properties without using a coating

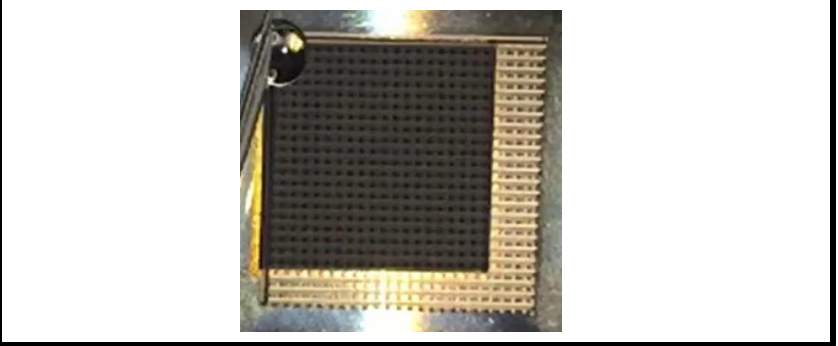
Superhydrophilic



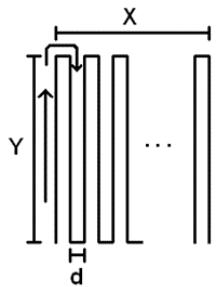
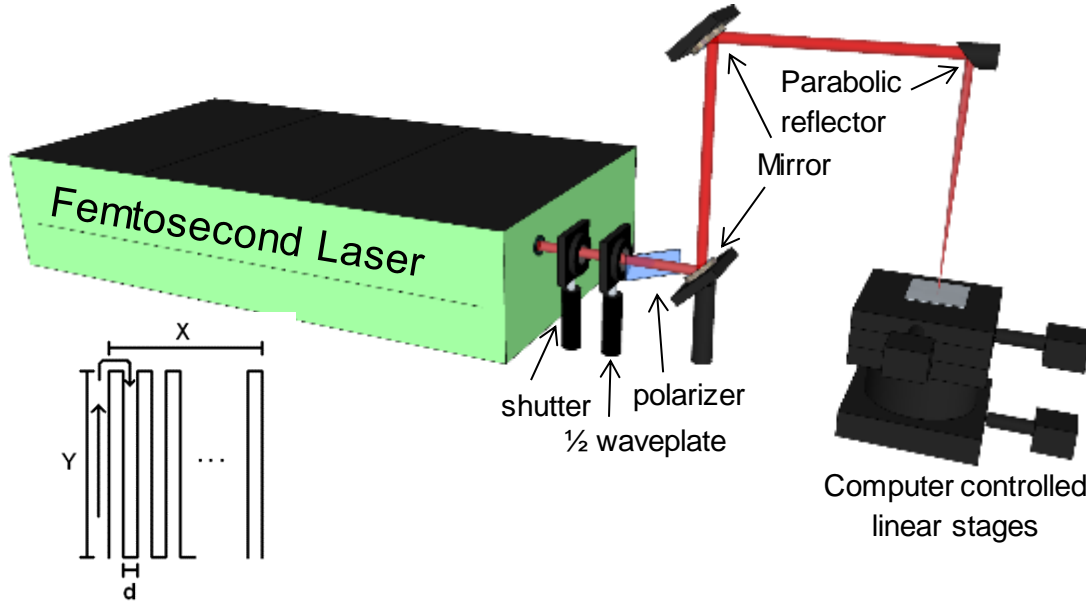
Superhydrophobic



Alternating zones of wettability



Femtosecond Laser Surface Processing (FLSP)

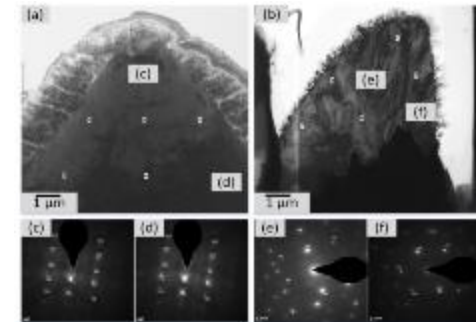
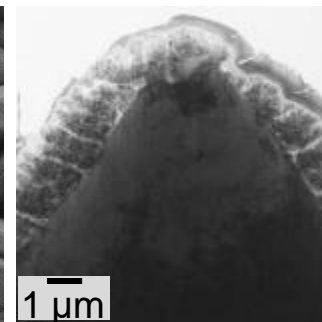
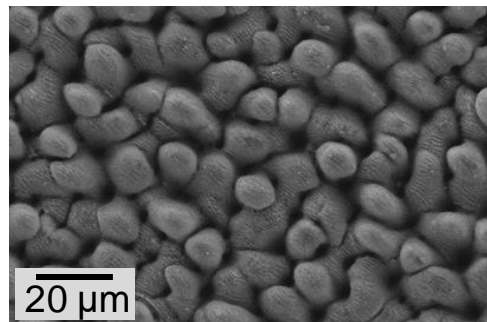
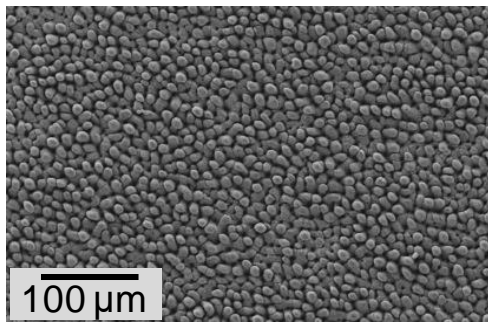


Typical programmed raster scan path

Specifications of main lasers associated with FLSP research

Laser	Astellia	DURIP laser
Pulse Energy	6 mJ	10 mJ
Center Wavelength	800 nm	1.1 μm – 15 μm ; 800 nm + harmonics
Rep Rate	1 kHz	1 kHz
Pulse Duration	35 fs	35 fs

Other available high powered lasers: Extreme Light Core Facilities (100 mJ), Diocles (30 J) (note: low repetition rate > 10 Hz)



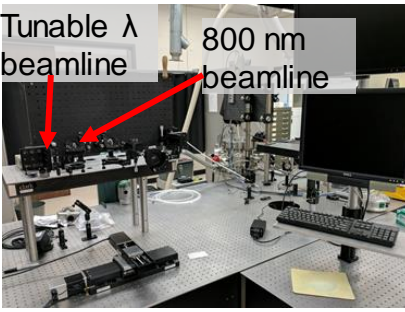
Internal crystalline structure

Typical Structure: Increasing Magnification

Developing World Class FLSP Research Facilities

DURIP 2016 funded femtosecond laser (\$488,684): 10 mJ, 35 fs laser that is tunable from 1,100 – 15,000 nm plus the fundamental 800 nm wavelength and harmonics.

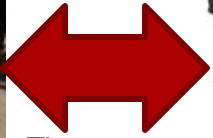
DURIP 2017 funded FLSP/material analysis chamber (\$961,830): includes an UHV chamber for carrying out FLSP in controlled environments that is connected to a material analysis UHV chamber with SEM, AES, sputtering and XPS capabilities.



FLSP open air setup

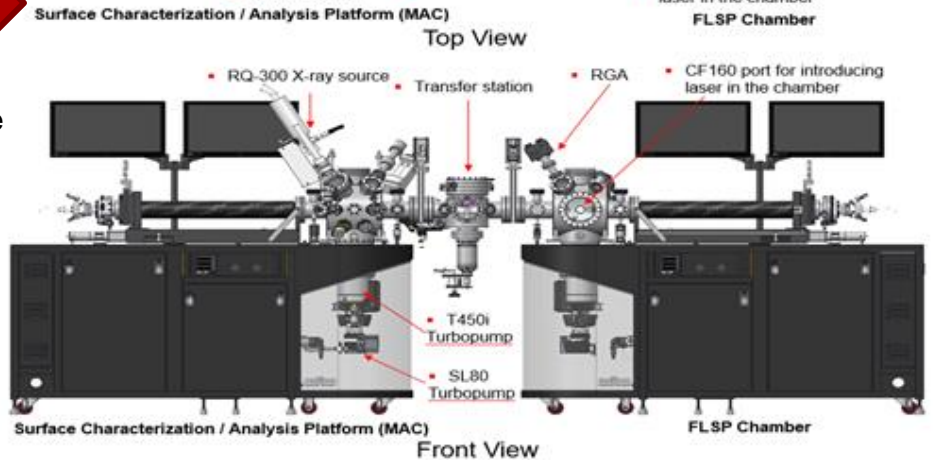
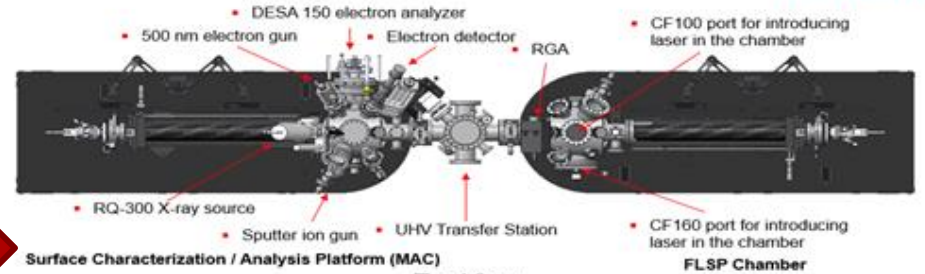


FLSP environmental chamber



These systems are being combined

UNL Configuration

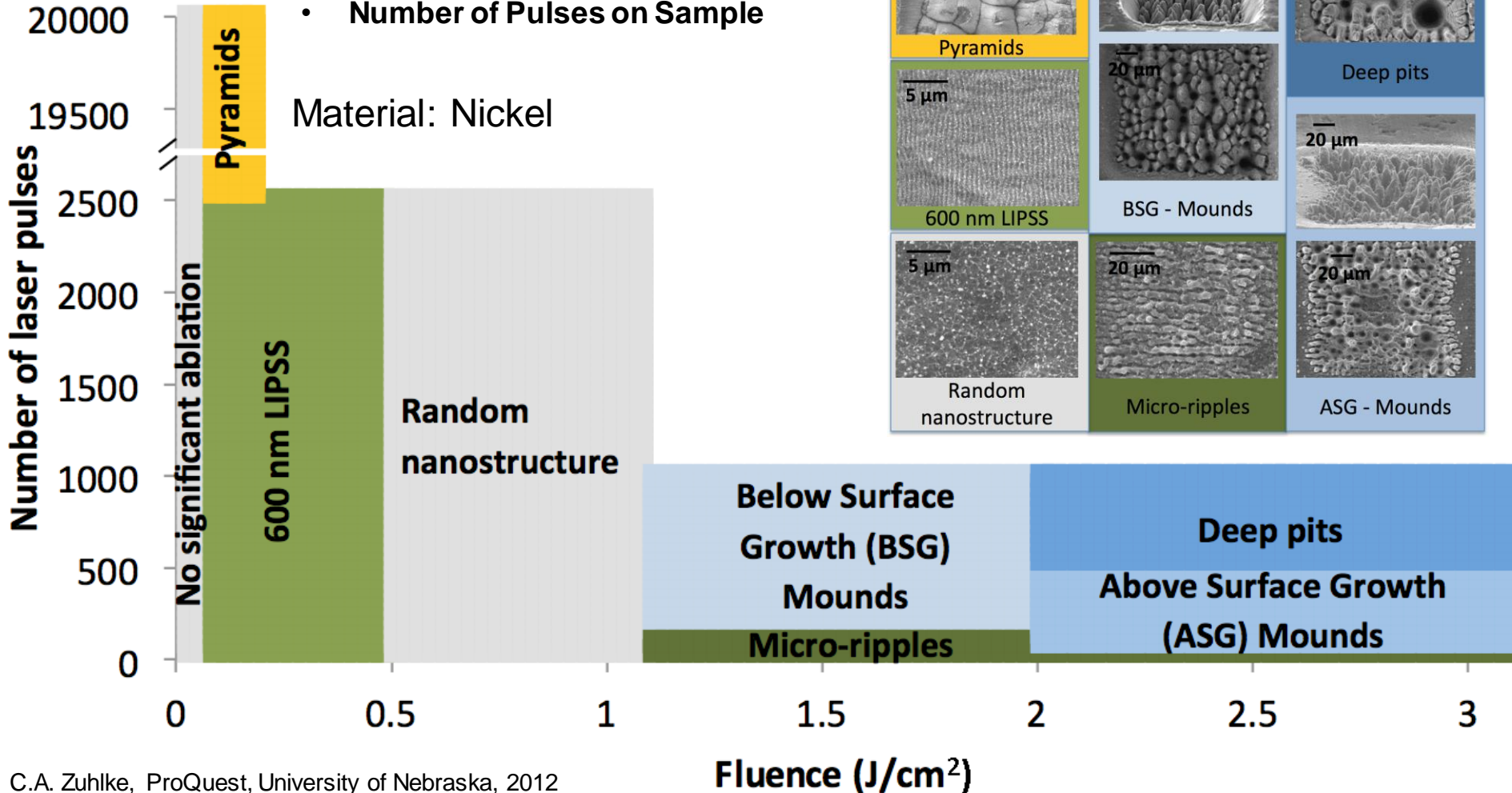


Range of Structures Produced by FLSP on Nickel

A diverse range of unique surface structures can be fabricated by varying two critical parameters:

- Laser Fluence
- Number of Pulses on Sample

Material: Nickel

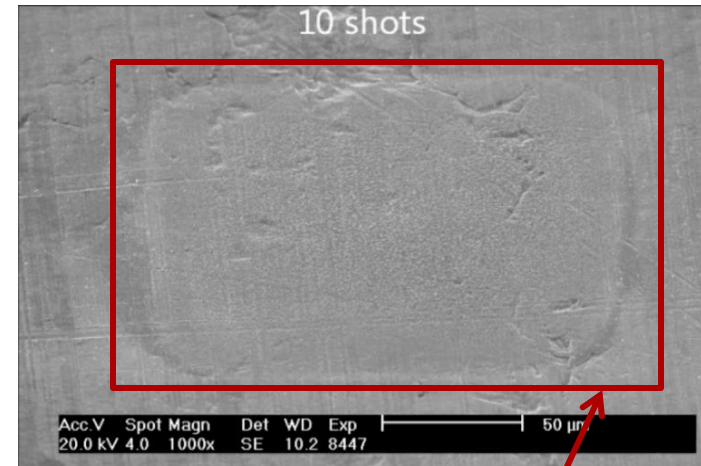


C.A. Zuhlke, ProQuest, University of Nebraska, 2012

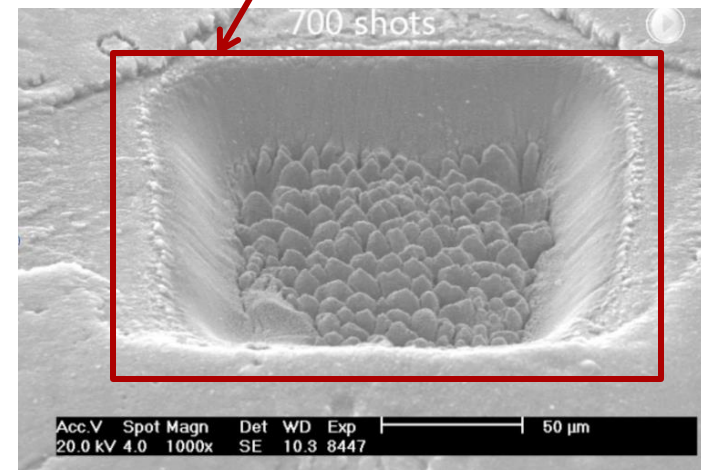
SEM Stop-Motion Imaging of the Dynamics of Self-Organized FLSP Structure Development

Analyze the shot by shot development of individual surface structures through a stop-motion technique utilizing a scanning electron microscope

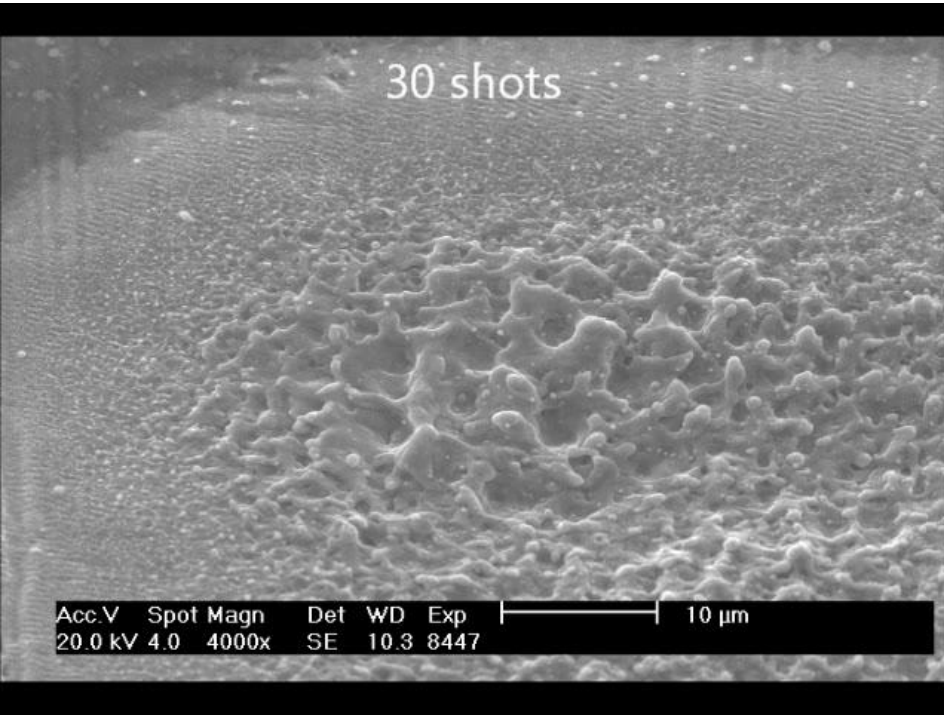
1. Image the sample
2. Precisely realign the sample
3. Irradiate the sample with 1 (or several) pulses
4. Image the sample
5. Precisely realign the sample
6. Irradiate the sample with 1 (or several) pulses
7. ...



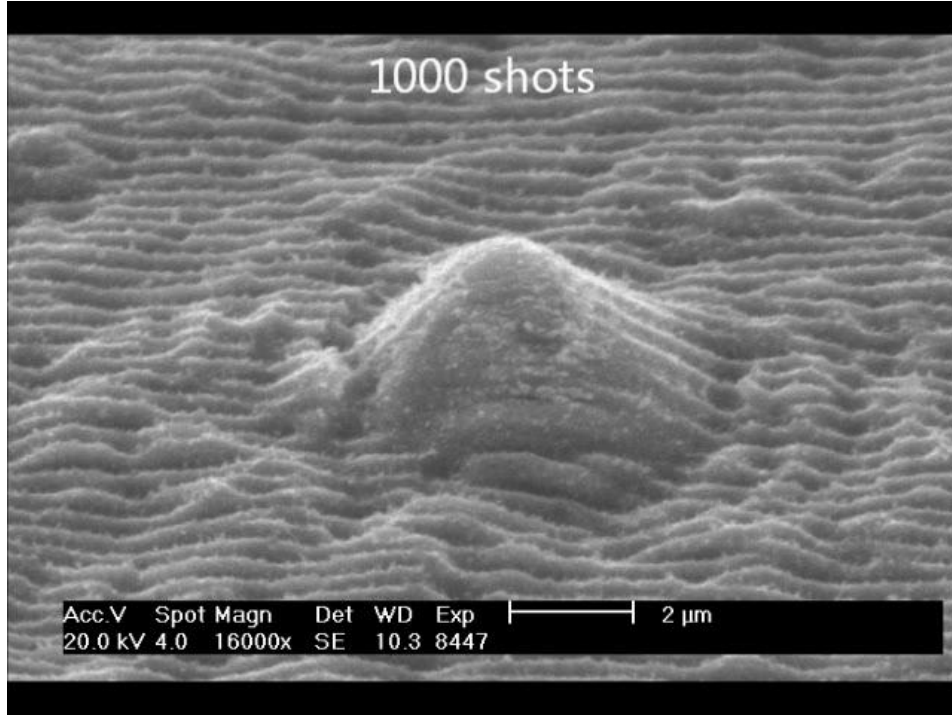
Outline of flat-top beam profile



ASG-mounds: (3.08 J/cm²)

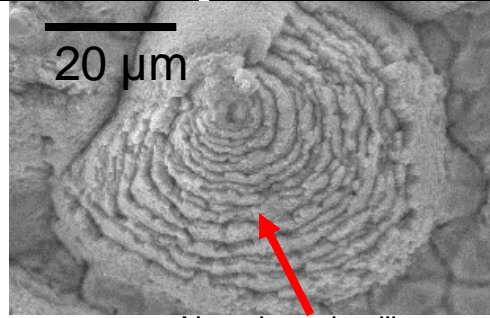


NC-pyramids: (0.12 J/cm²)



Video: clearly observable fluid flow/redeposition in structure development

Zuhlke, et. al. *Optics Express* (2013).



Note the onion-like layers of nanoparticles

Video: nanoparticles build up on the surface and preferential etching occurs simultaneously with incident pulses

Zuhlke C. A., et. al. (2013) *Applied Surface Science*, 21(7), 8460–73.

Critical Strengths of FLSP

These are all self-organized structures

Multiscale Structures in a single step

Control over micro/nanoscale features via laser processing parameters

Structure Permanency

Functionalization through shaping of substrate – retain durability of substrate

Contactless Fabrication

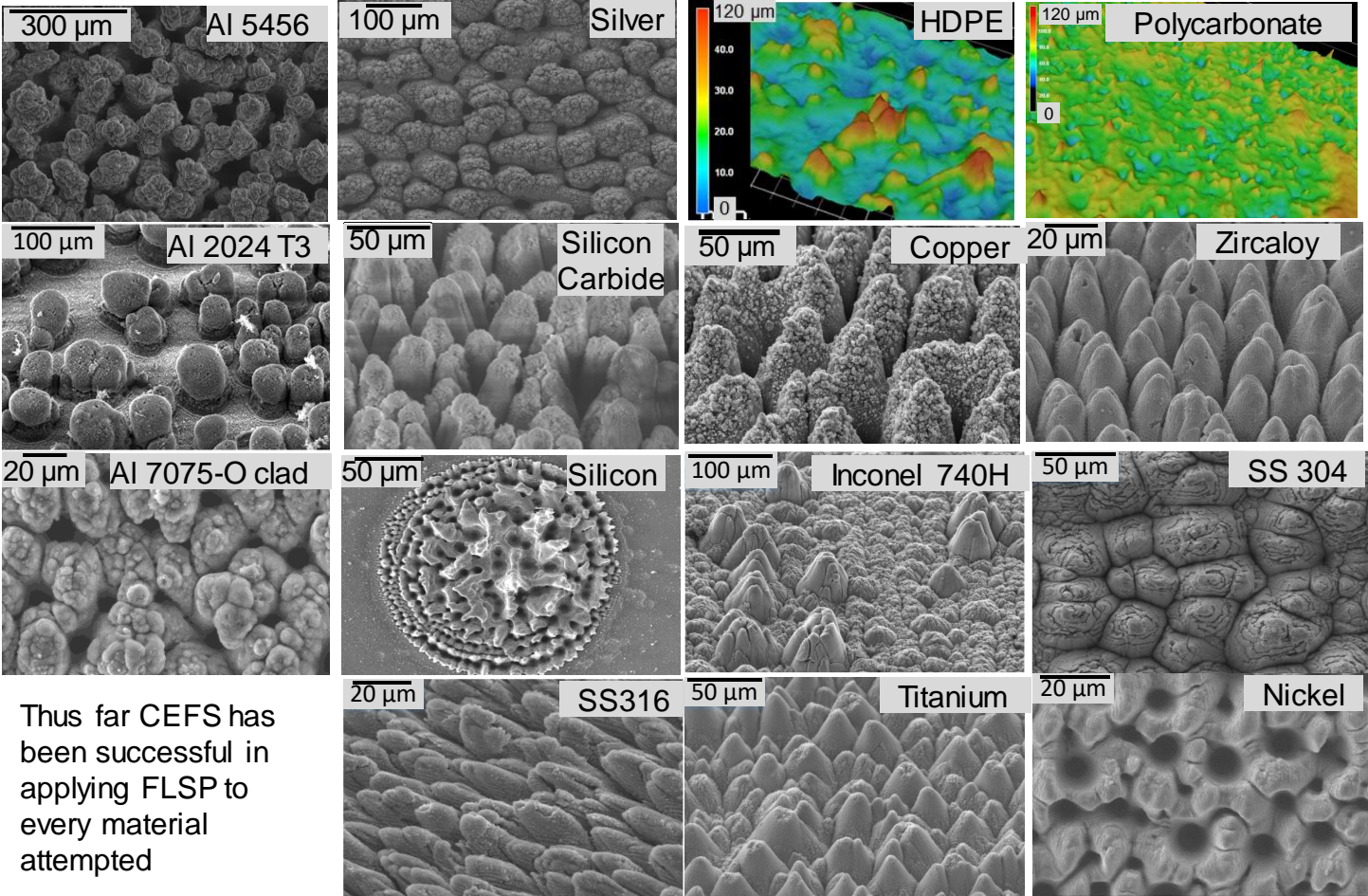
Open-air laser processing enables processing of arbitrary shaped 3D surfaces

Scalability

Large areas can be processed by scaling the laser power and repetition rate

Versatility

FLSP can be extended to a wide range of materials



Thus far CEFS has been successful in applying FLSP to every material attempted

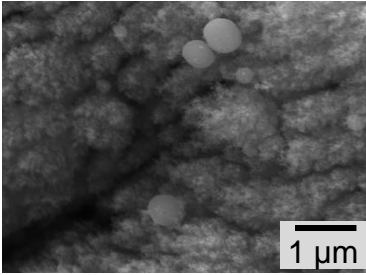
Material Characterization is Fundamental to Understanding FLSP Phenomena and Results Being Obtained in Specific Applications

FEI Helios NanoLab 660 (dual beam SEM/FIB mill)



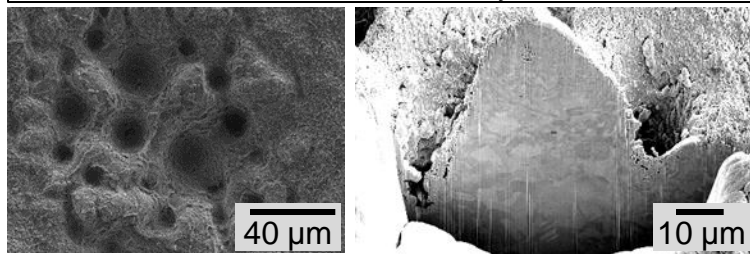
- Microstructure analysis:
- Fundamental materials science/engineering
 - Size, shape, phase of crystalline grains, material composition

Extreme high resolution (XHR) imaging (SEM – sub-nanometer capabilities)

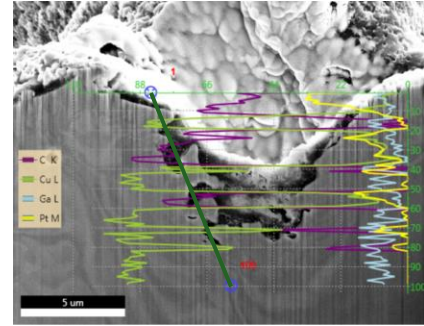


Dual-beam imaging (e- & Ga+)

Cross-sectional analysis



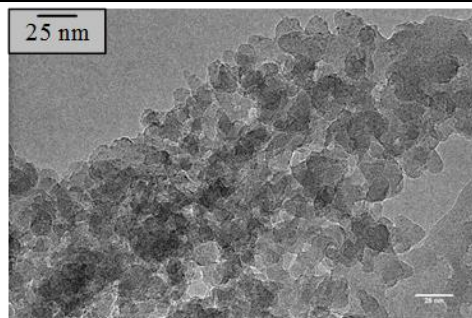
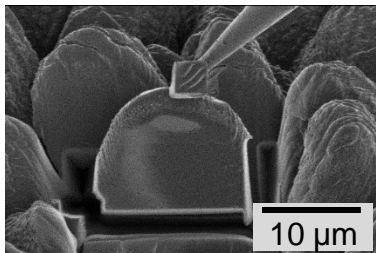
Energy-dispersive x-ray spectroscopy (EDS)



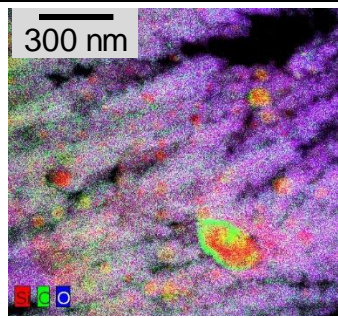
Octane Super EDS detector capable of detecting carbon

TEM analysis capabilities at UNL

Structure lift-outs for TEM analysis



Sub-nanometer imaging



Spatial composition mapping

Crystal analysis – Selected area electron diffraction (SAED)

FLSP on Ti: Crystal Structure of ASG vs BSG Mounds

BSG-mounds

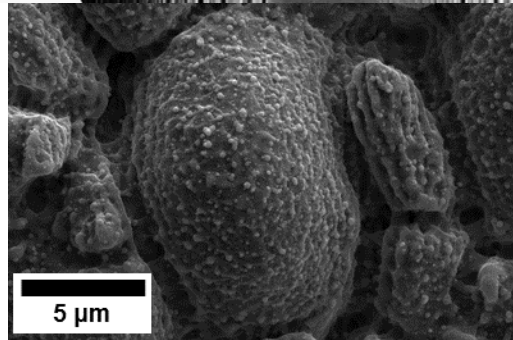
ASG-mounds

FIB mill cross-sectioned mounds

Resolidification layer

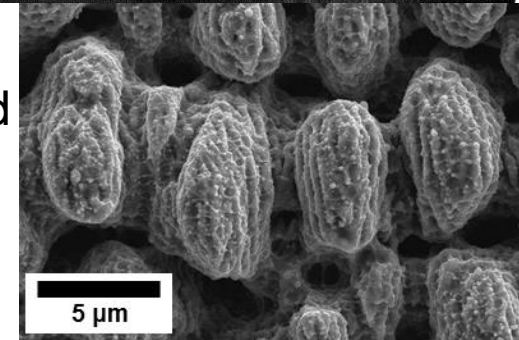
10 μm

5 μm



Microstructure analysis

- Rapidly resolidified layer composed of smaller Ti grains (e.g. 200 nm)
- Original Ti microstructure w/ large (average 10 μm) Ti grains



BSG-mound structure prior to FIB mill cross-sectioning

ASG-mound structure prior to FIB mill cross-sectioning

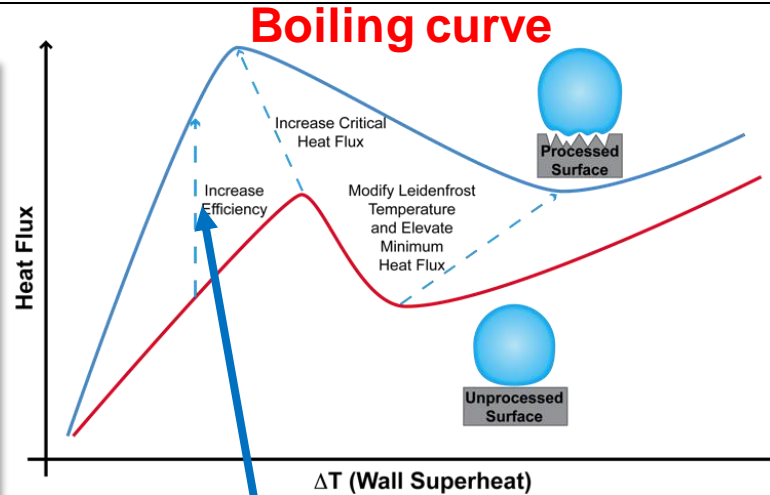
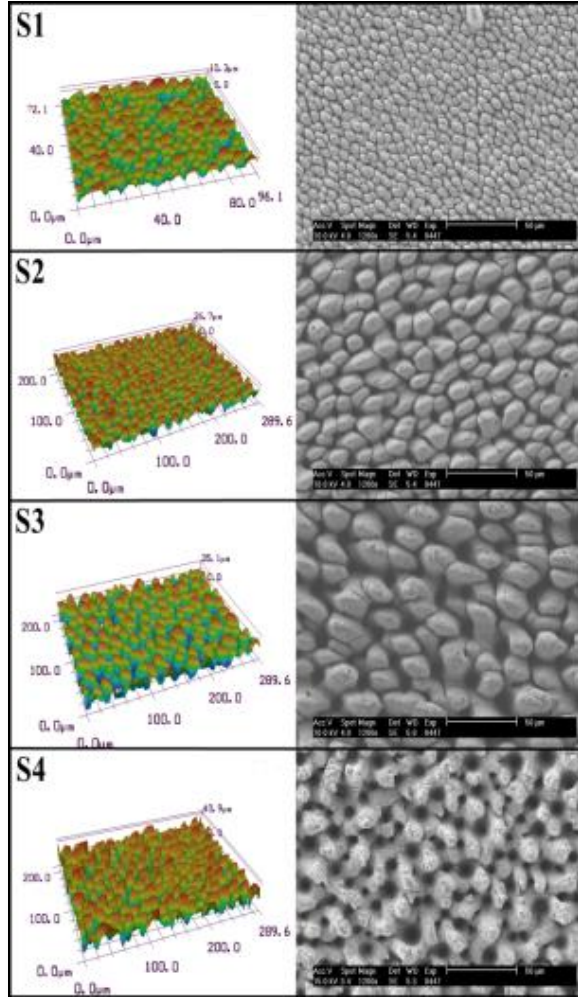
E. Peng, et. al., Appl. Phys. Lett. **108**, 31602 (2016).

E. Peng, et. al., Appl. Surf. Sci. **396**, 1170 (2017).

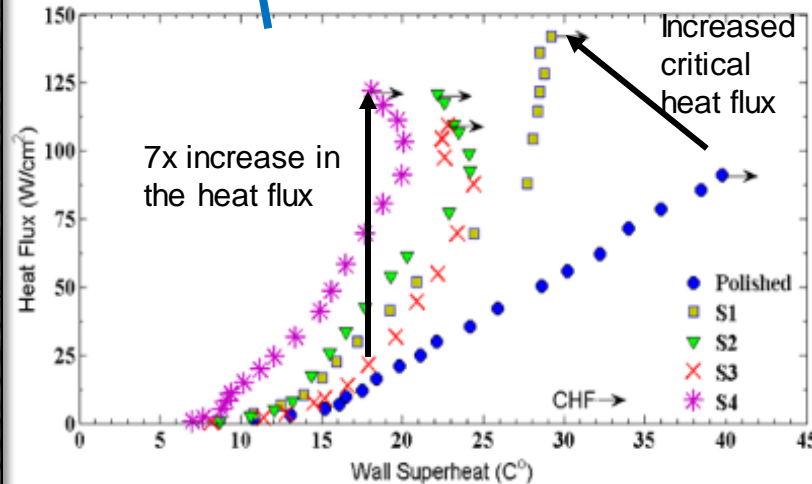
Enhanced Heat Transfer Using FLSP

FLSP results in an enhancement of the critical heat flux and heat transfer coefficient

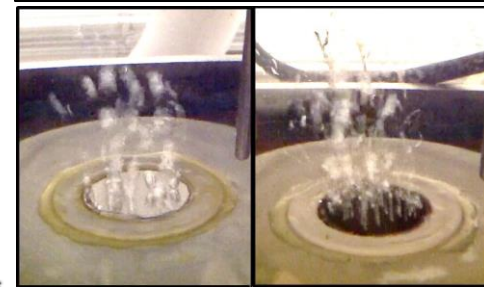
SS 304 FLSP surfaces



Video: enhanced nucleate boiling efficiency



Higher nucleation site density on FLSP enhanced surface

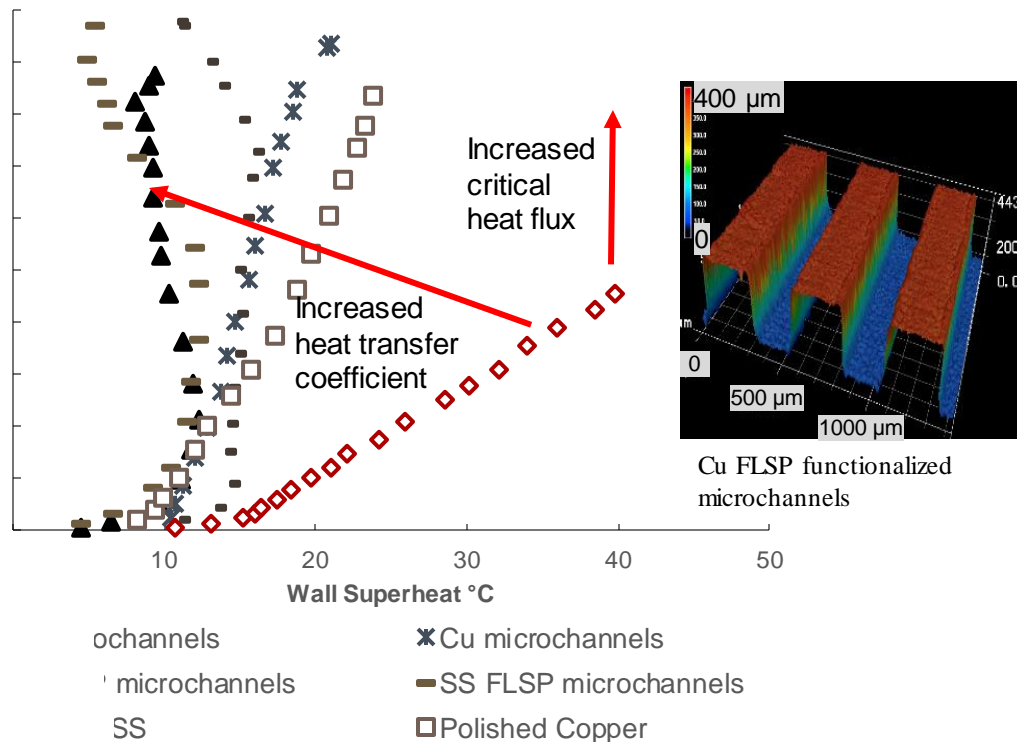


Unprocessed

FLSP enhanced

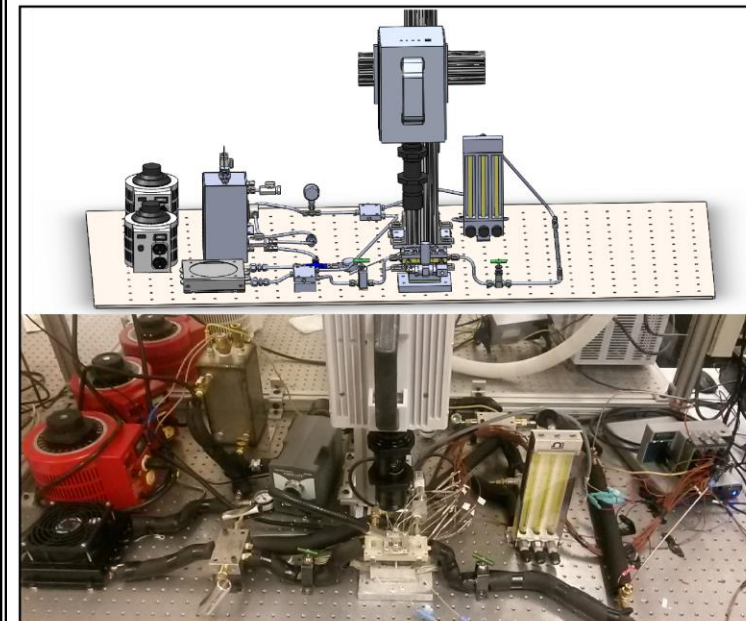
C.M. Kruse, et. al., Int. J. Heat Mass Transf. **82**, 109 (2015)
C. Kruse, et. al., Appl. Phys. Lett. **108**, 51602 (2016)
C. Kruse, et. al., Langmuir **29**, 9798 (2013)

Micro-Channel Pool Boiling Results



Pool boiling results for Cu and 304 SS surfaces comparing polished flat reference surfaces with surfaces with 400 μm deep and wide microchannels in the surface along with FLSP functionalized microchannel surfaces.

FLSP functionalized SS microchannel surface
 transfer coefficient: 355 kW/m²K
 al heat flux: 195 W/cm²



Experimental setup for measuring heat transfer performance of FLSP surfaces with two phase flow in microchannels

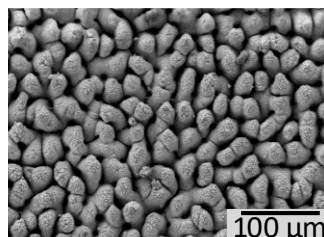
Antibacterial Work for NASA Johnson (Scott Hansen) on Next Generation Critical Heat Exchanger for ISS and Deep Space Missions



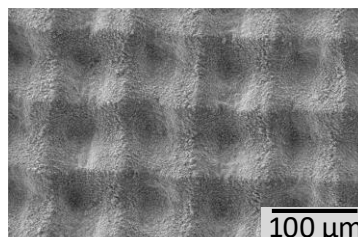
International Space Station (ISS)



ISS CHX



al pulse FLSP mounds on silver



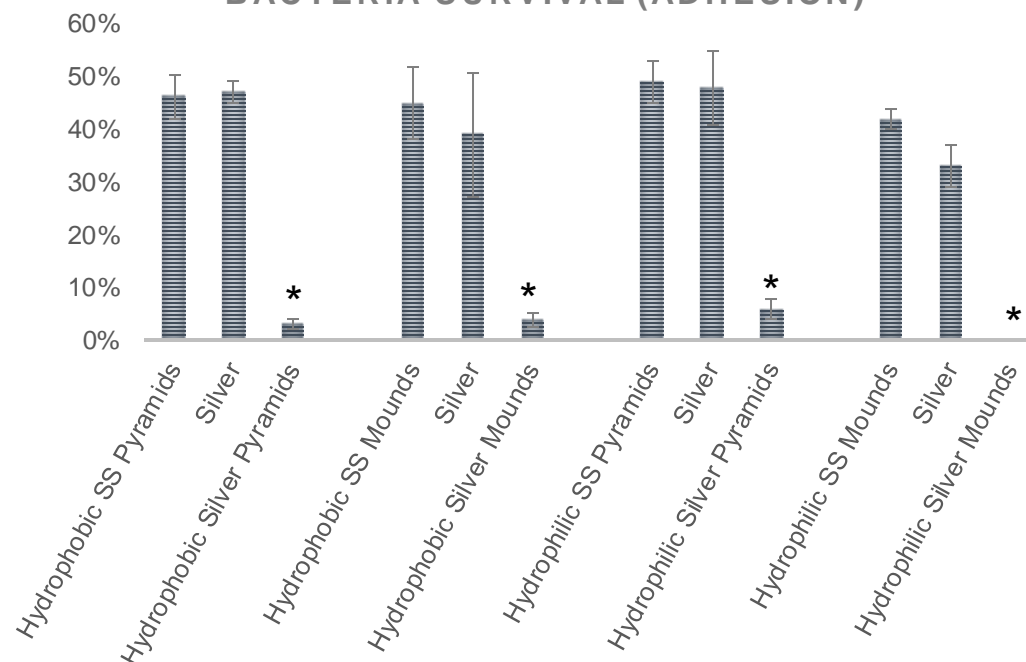
Direct write FLSP pyramids on silver

Critical need: superhydrophilic or superhydrophobic, antibacterial surface that is permanent and does not contaminate the water supply.

Bacterial consortium used in this study:

- *Bacillus megaterium*
- *Staphylococcus epidermidis*
- *Sphingomonas paucimobilis*

BACTERIA SURVIVAL (ADHESION)



* FLSP functionalized silver surfaces

For the first time we are able to functionalize silver, which has important implications for antibacterial superwicking surfaces for ISS CHX's.




Laser processed CHX fins

Anti-icing Properties of FLSP Functionalized Al 7075-O Clad Surfaces (Previously Funded by Boeing)


Supercooled droplet icing prevented through FLSP functionalization on Al 7075-O Clad surface

Unprocessed Al 7075-O Clad



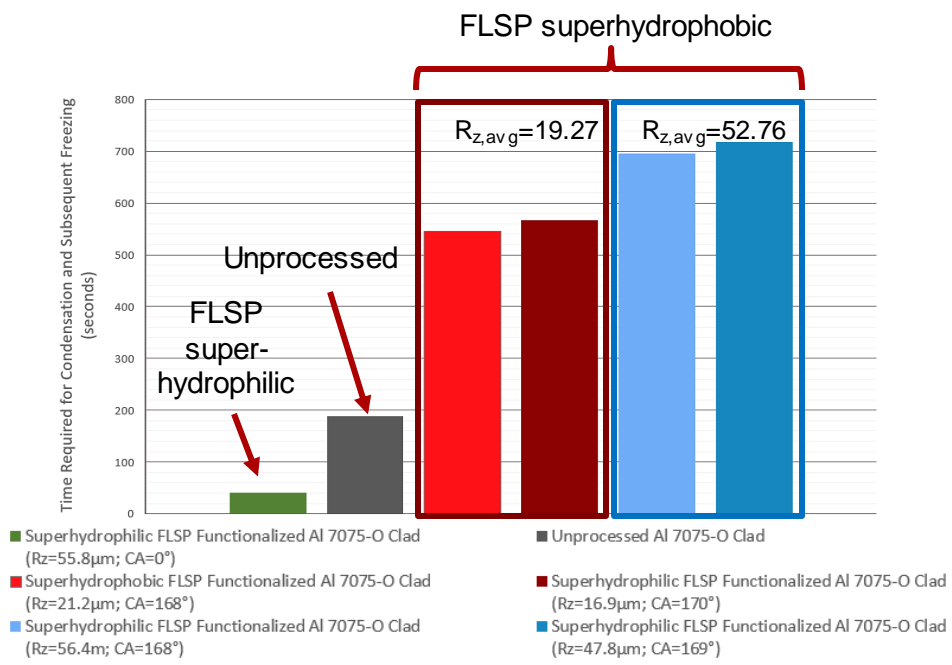
Video: supercooled droplets impact and stick (changing phase) to the unprocessed surface.

Superhydrophobic FLSP Al 7075-O Clad
Contact Angle: $168.42 \pm 1.13^\circ$

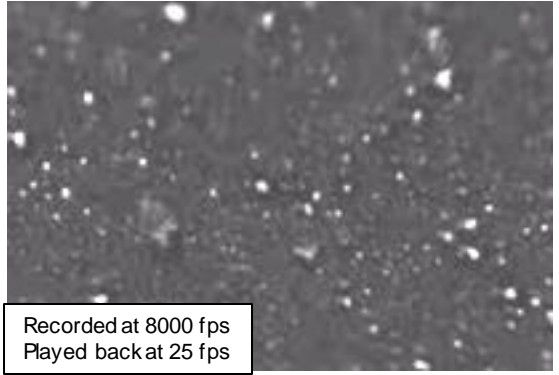


Video: falling supercooled droplets impact and bounces off the FLSP functionalized surface.

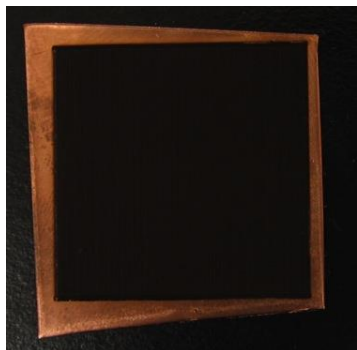
Condensation and delay in subsequent freezing on FLSP functionalized Al 7075-O Clad surfaces



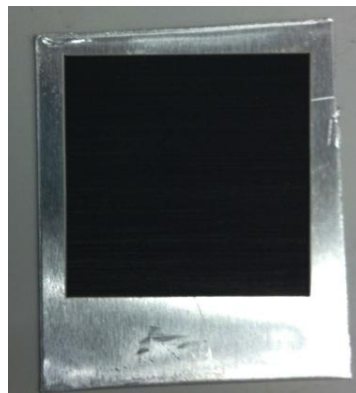
Video: condensate droplet coalescence can cause self-propelled jumping from the FLSP functionalized surface.



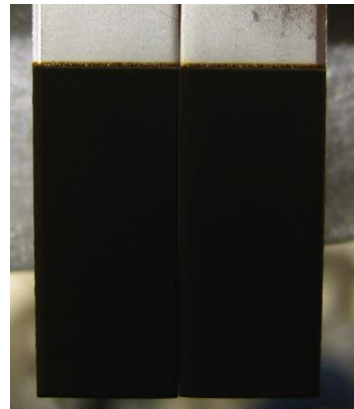
Copper



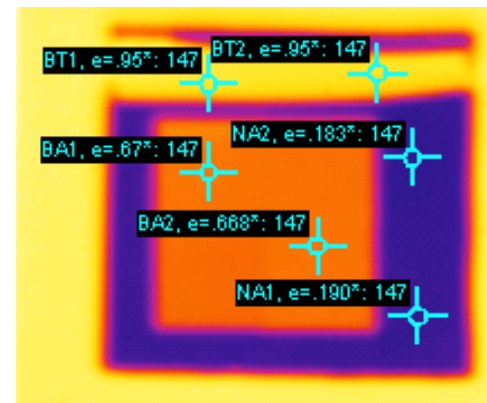
Aluminum



Stainless Steel



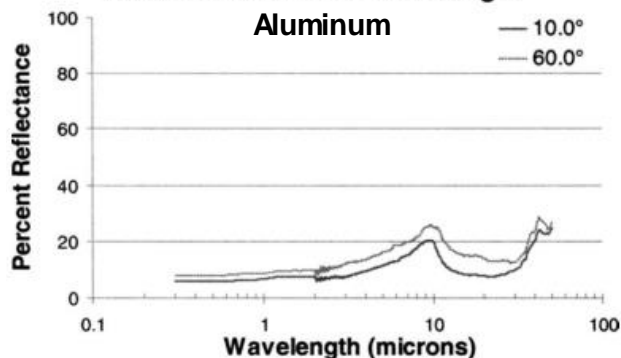
The emissivity of a metallic surface can be significantly enhanced through FSLP



Infrared image of processed (orange) and unprocessed (purple) surfaces on a computer processor

Emissivity	
Unprocessed	0.19
Processed	0.67

**Total Reflectance vs. Wavelength
Aluminum**



**Reflectance vs. Wavelength
Stainless Steel**

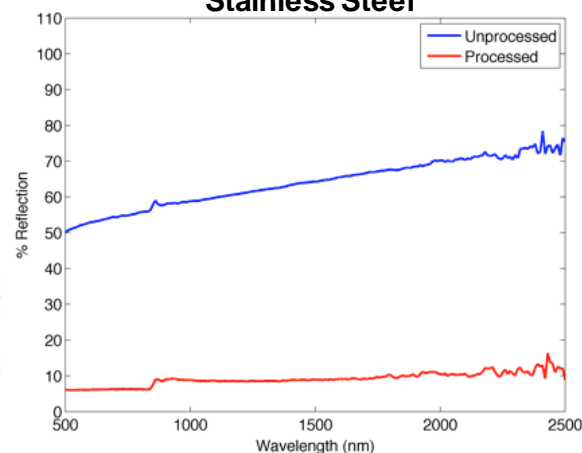
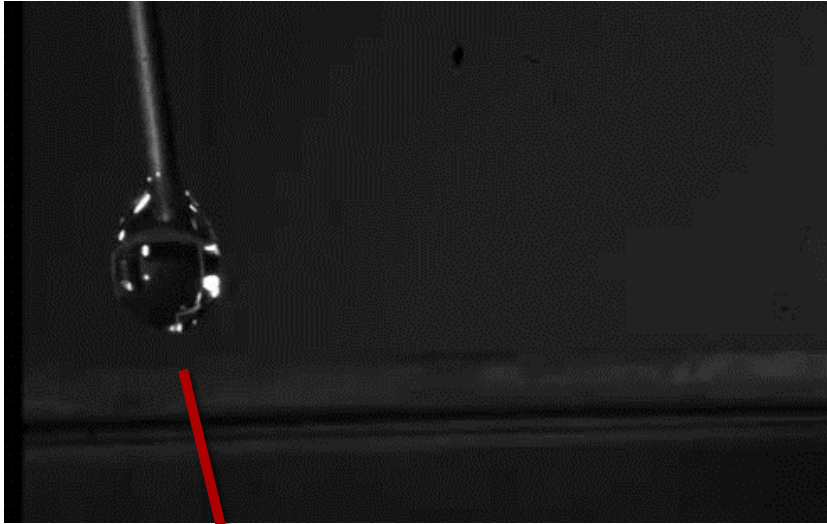


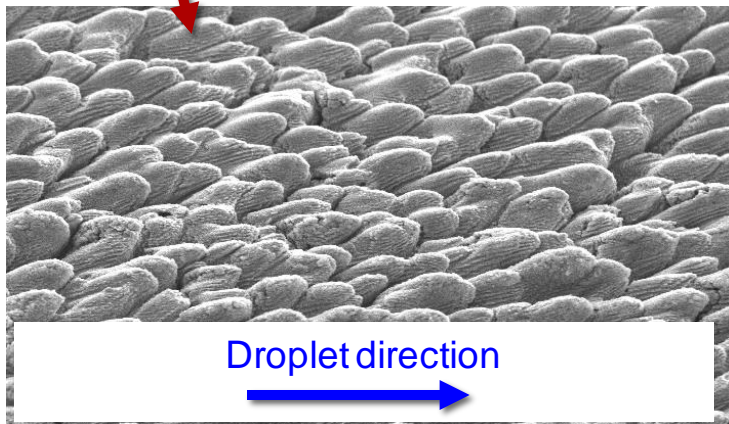
FIG. 6. Typical reflectance of Al sample ablated at fluence of 13.5 J/cm², in the wavelength range of 0.3–50 μm

N. Singh, D. R. Alexander, et. al.,
J. Laser Appl. 18, 242 (2006).

Self-Propelled Leidenfrost Droplets

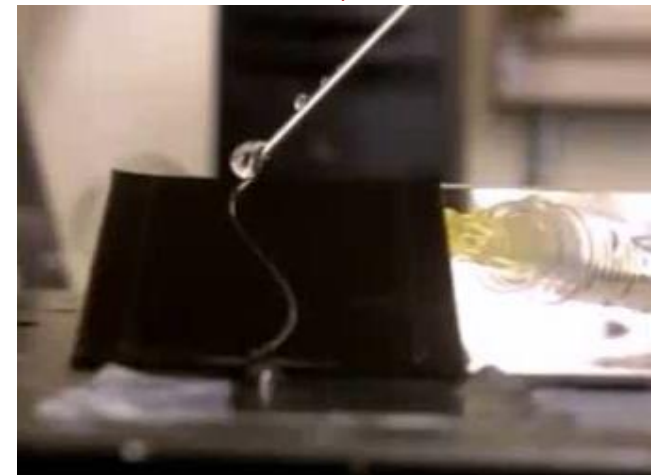
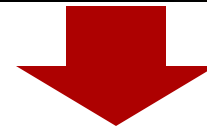
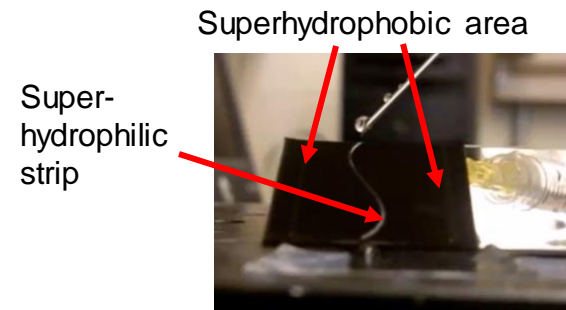


Video: Leidenfrost driven droplet motion on FLSP surface



C. Kruse, et. al., *Microfluid. Nanofluidics* **18**, 1417 (2015)

Alternating zones of wettability at room temp.



Video: Water droplet following superhydrophilic channel on superhydrophobic surface

Questions?

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Other CEFS Undergraduate students: Cole Dempsey, Joseph Sheehan, Christopher Currey, Joshua Edgar, Angoua Konan, Owen Fike



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