Surface Evolution of Femtosecond Laser Processed Copper Subject to Hydrogen Annealing

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Abstract

The escalating power densities of modern electronic components demand increasingly effective thermal management solutions. This is a critical issue, particularly for high-performance computing and AI data centers, which are projected to consume 134 TWh annually by 2027. Current air-cooling methods are inherently inefficient, accounting for 30-40% of data centers' energy usage, necessitating more efficient alternatives like two-phase liquid cooling.

Femtosecond Laser Surface Processing (FLSP) allows for the creation of micro- and nano-scale features on materials, enhancing heat transfer efficiency and critical heat flux (CHF) in two-phase liquid cooling setups. FLSP-treated surfaces exhibit remarkable wicking capabilities, rapidly rewetting after bubble departure and enabling continuous heat release.

While copper (Cu) is an ideal material due to its superior thermal conductivity, FLSP creates an oxide layer on its surface, which greatly reduces its effectiveness. Our research focuses on Hydrogen Gas Annealing (H_2A), an effective method for reducing this surface oxide layer while preserving the beneficial micro- and nano-structures, thereby paving the way for significantly more efficient cooling solutions.

For this research, nine samples of copper were separated into three groups of three. A specific FLSP parameter combination was assigned to each group. Scanning electron microscope (SEM) and laser scanning microscope (LSCM) images and measurements were taken before annealing. Subsequently one sample from each group was annealed at a specific temperature (300 °C, 500 °C, and 675 °C). Post-annealing, SEM and LSCM analyses were repeated, and the results were compared to the pre-annealing data.

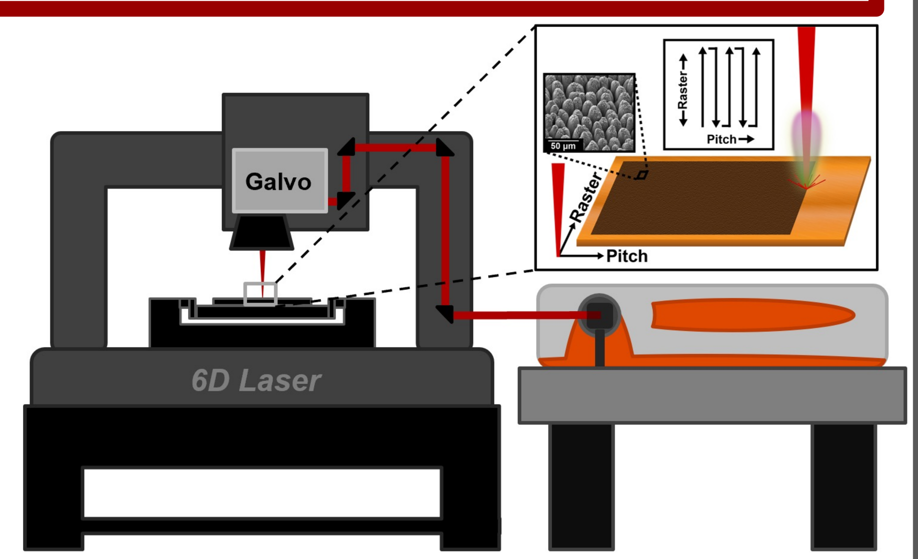
Femtosecond Laser Surface Processing

Process: FLSP was applied to Cu substrates by raster scanning a focused femtosecond laser beam, making long passes in the direction of raster that were separated by short steps in the direction of pitch

Material: Cu 101 (99.99% Pure)

Parameters: Three combinations of laser pulse energy density (fluence) and number of pulses applied to any given spot on the Cu surface (pulse count) were controlled for:

- 1.8 J/cm^2, 4700 PC
- 3.6 J/cm^2, 2400 PC
- 5.4 J/cm^2, 1600 PC

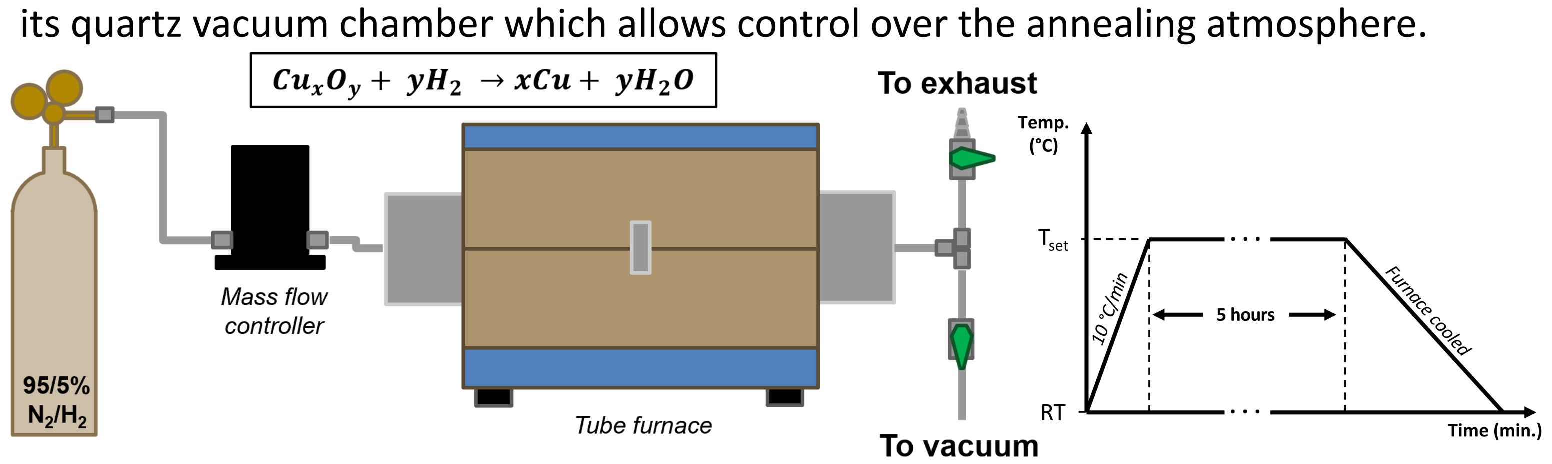


Equipment: Amplitude Tangor 300, 5-axis laser CNC
Pulse Length: 470 fs
Wavelength: 1030 nm
Average Power: 300 W
Repetition Rate: single pulse - 40 MHz
Pulse Energy: 3 mJ (at 100 kHz)

Hydrogen Gas Annealing

Equipment: An MTI OTF-1200x tube furnace was chosen for this experiment due to its quartz vacuum chamber which allows control over the annealing atmosphere.

$$Cu_xO_y + yH_2 \rightarrow xCu + yH_2O$$



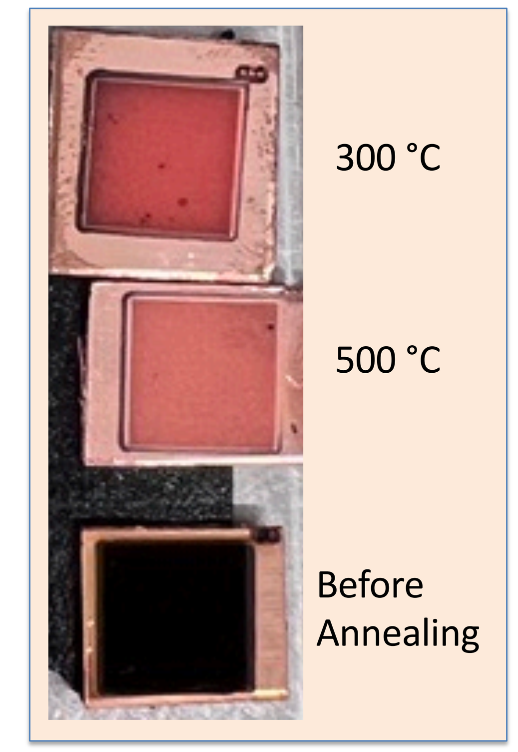
- Samples were annealed in a 95% Nitrogen / 5% Hydrogen atmosphere for five hours, then gradually cooled to ≤30°C before re-exposure to air.
- In the absence of O_2, H_2 reacted with surface oxides, forming H_2O and reducing the oxide layer. Nitrogen served as an inert diluent for safety
- After H_2A, the surface chemistry of the Cu FLSP was metallic in nature

Characterization

Imaging: FEI Quanta 200 Environmental Scanning Electron Microscope (SEM)
Quantitative Analysis: Keyence VK-X200K Laser Scanning Confocal Microscope (LSCM)

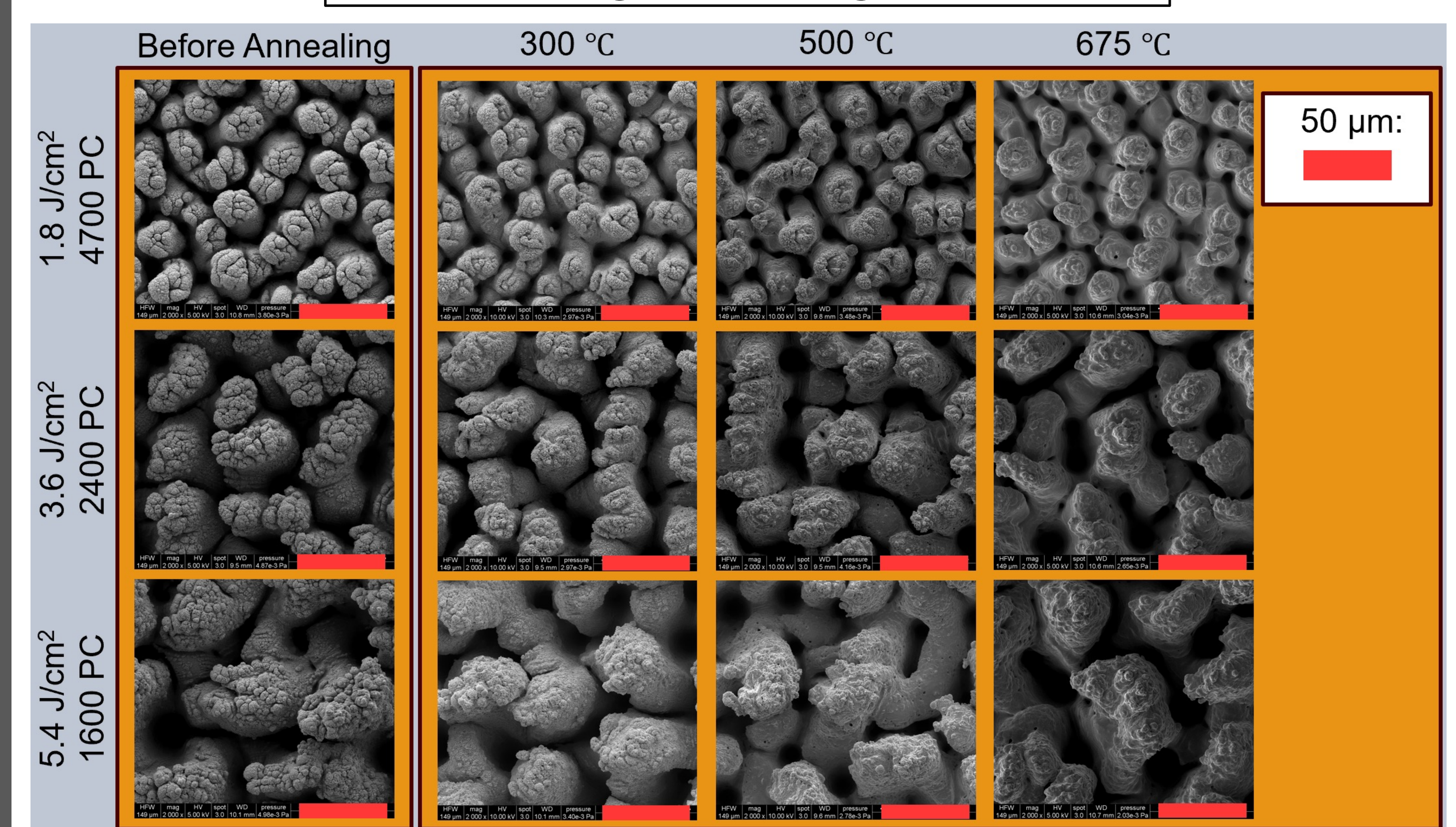
Results

A notable visible change in the color of the samples was observed. Before annealing, the samples appeared black due to the presence of extreme nanoscale roughness and the laser-induced oxide layer. Following H_2A, the samples turned a salmon color, indicating the successful reduction of the Cu oxide to a metallic chemical state.



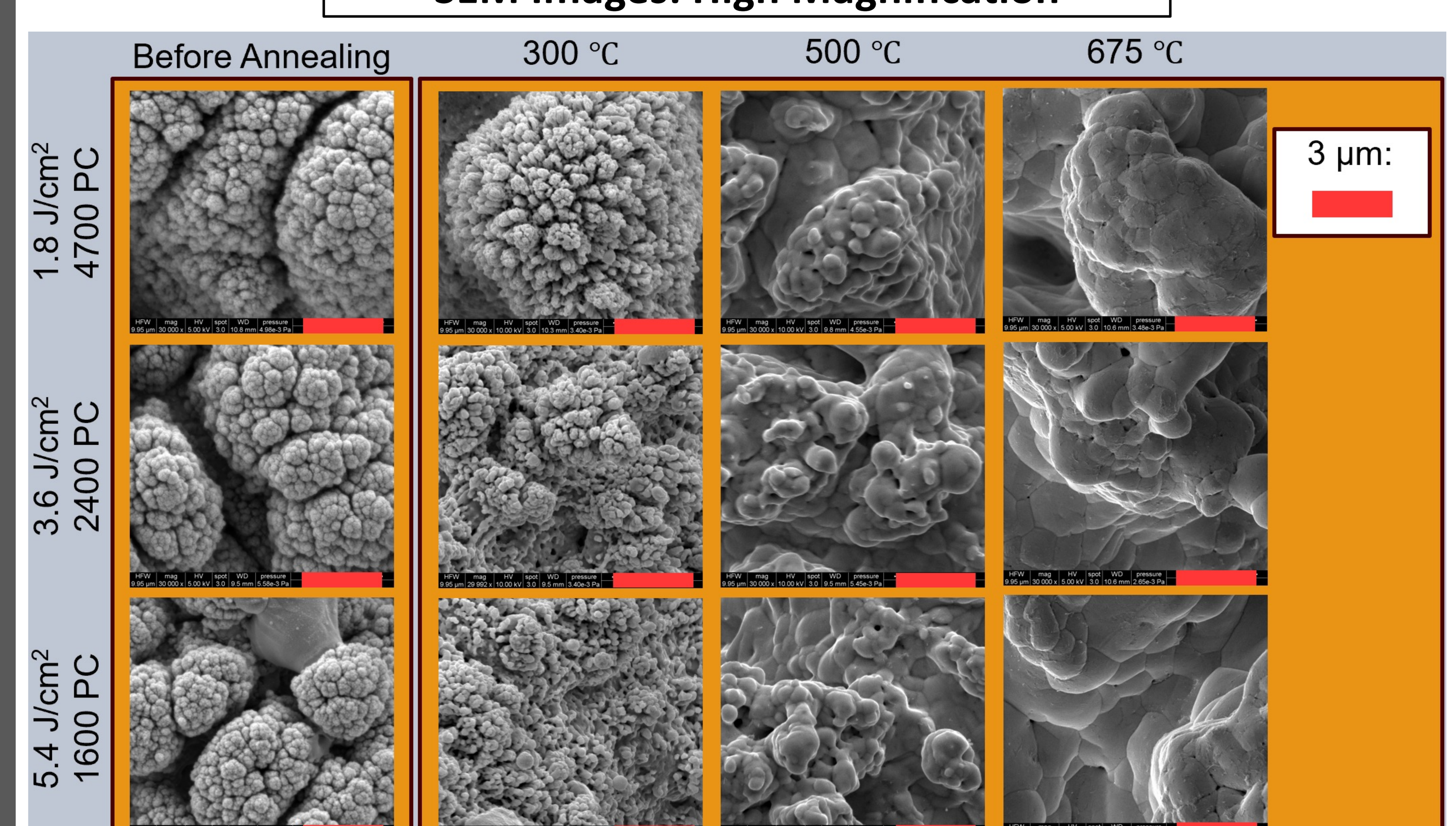
The subtle color difference between samples annealed at 300 °C and 500 °C can be attributed to the nanoparticles becoming coarser in size at higher annealing temperatures. Before annealing and at lower annealing temperatures, there are many small nanoparticles which effectively scatter light, giving it more chances to be absorbed into the material. It is unlikely that there is a significant difference in the thickness of the oxide layer between both H_2A temperatures.

SEM Images: Low Magnification



At this magnification, the overall shapes and layouts of the microstructures are largely maintained across temperature changes, though variations in nanoparticle density become visible. This indicates that H_2A effectively reduces oxide buildup while preserving the fundamental microstructures created by FLSP.

SEM Images: High Magnification



At the nanoscale, H_2A significantly impacts surface morphology. As annealing temperature increases, the surface becomes progressively smoother with fewer but larger nanoscale particles, likely due to increased melting and coalescing of fine nanoparticles as the annealing environment increases in temperature. This demonstrates H_2A's ability to control FLSP nanostructures based on application needs.

Surface Roughness Analysis

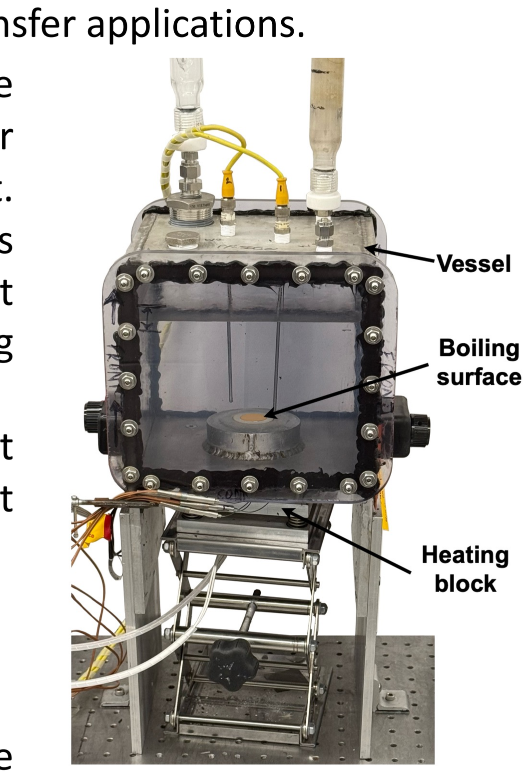
Fluence (J/cm^2)	Pulse Count	Temperature (°C)	R_z Before (µm)	R_z After (µm)
1.8	4700	300	63.1 ± 4.1	53.0 ± 2.2
		500	56.6 ± 5.6	51.5 ± 7.1
		675	61.9 ± 8.9	54.6 ± 4.0
3.6	2400	300	118.9 ± 20.2	93.3 ± 7.6
		500	112.7 ± 10.6	96.0 ± 7.8
		675	113.4 ± 16.4	99.3 ± 6.1
5.4	1600	300	150.3 ± 12.2	135.1 ± 16.0
		500	143.8 ± 12.8	137.2 ± 11.4
		675	147.9 ± 13.3	143.1 ± 14.9

A consistent reduction in peak-to-valley roughness (R_z) is observed across all fluence and temperature combinations, with an average decrease of 11.8 µm. However, increased reflectivity after annealing (due to a thinner oxide layer and microstructure smoothing) contributed to noisier measurements, requiring cautious interpretation.

Discussion & Future Work

These findings demonstrate that Hydrogen Gas Annealing (H_2A) is an effective method for reducing the surface oxide layer on FLSP-treated Cu, while largely preserving the desired micro- and nano-scale features. Crucially, while FLSP provides precise control over the initial microstructure of the copper surface, the subsequent annealing process allows for fine-tuning and control over the nanoscale features in addition to the reduction of the oxide layer. The ability to independently control both micro- and nanostructures through these processes provides a powerful toolset for optimizing functionalized surfaces for specific heat transfer applications.

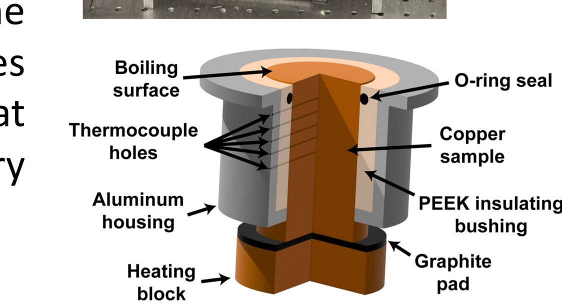
The next steps involve correlating these material analysis results with pool boiling heat transfer performance using the vessel pictured to the right. This will allow us to identify specific surface features that yield optimal heat transfer, guiding subsequent rounds of material fabrication and further enhancing two-phase heat transfer of copper.



The success of this research holds significant promise for various industries requiring efficient cooling of high-power electronics including:

- Artificial intelligence data centers
- National defense systems
- Advanced computing hardware

Furthermore, this work contributes to the development of scalable functionalization techniques for oxide-free Cu, extending its impact beyond heat transfer to applications such as catalysis and battery electrodes.



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