



NEBRASKA CENTER FOR ENERGY SCIENCES RESEARCH

Surface Area and Porosimetry Measurements on Femtosecond Laser Surface Processed Copper Foils

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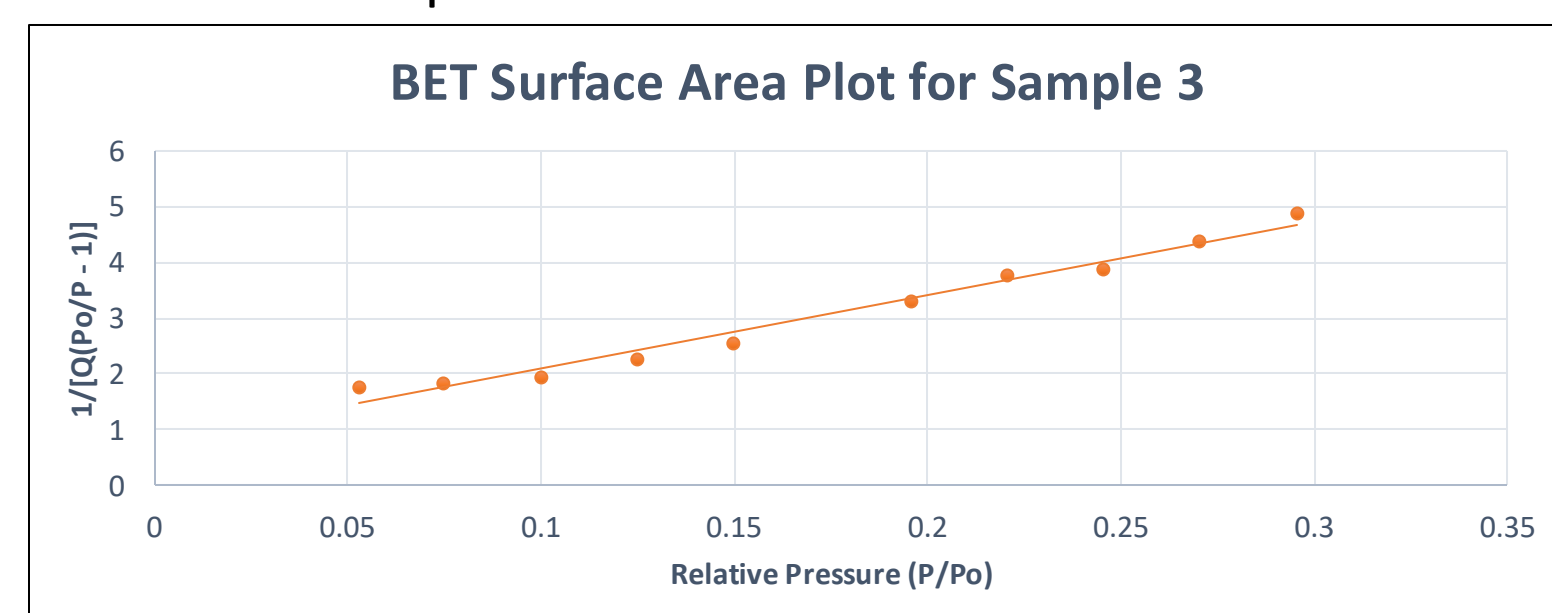
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Introduction

In this project, the Advanced Surface Area and Porosimetry Analyzer (ASAP 2460) and the confocal laser scanning microscope are compared as methods of measuring surface area on thin femtosecond laser surface processed (FLSP) samples. The ASAP 2460 machine was predicted to take better measurement of subsurface porosity and better detect micro- and nano-scale features because it measures using gas adsorption techniques. The opportunity to better characterize heat transfer in femtosecond laser surface processed copper using porosity measurements was why the project was started, because studying hydrogen annealed copper as a boiling surface in two phase immersion heating is useful in energy management applications.

The ASAP 2460

The Advanced Surface Area and Porosimetry Analyzer (ASAP 2460) uses **the Brunauer, Emmett, and Teller (BET) technique** to measure surface area and pore characteristics of a solid or powder. The BET technique is a calculation of surface area using the gas molecules absorbed as relative air pressure (P/P_0) increases. Degas conditions must be customized to the specific sample, and choosing the wrong conditions can provide incorrect results.



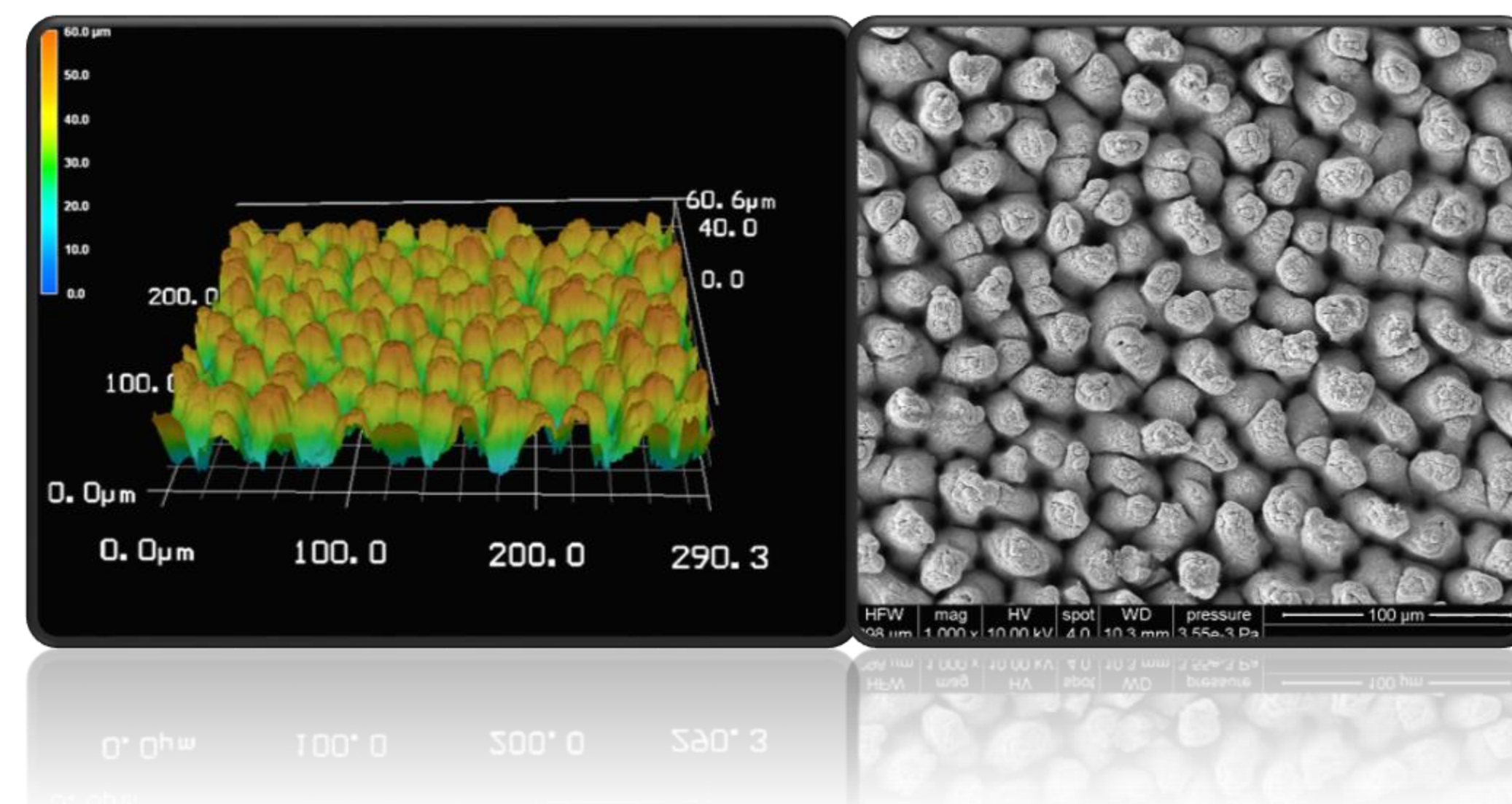
$$\frac{1}{X[(P_0/P) - 1]} = \frac{1}{X_m C} + \frac{C - 1}{X_m C} \left(\frac{P}{P_0} \right)$$

Thermogravimetric Analysis

If degas conditions are unknown, and there is concern about changes to the sample due to degas temperatures, thermogravimetric analysis can be performed using **the TGA 109 F1 Libra** or similar machines. In experiments on FLSP samples, this may not be useful unless there are special coatings, since the miniscule amounts of air or moisture released will not show up well. If the breakdown point of the sample is unknown, TGA could be used in that case as well.

Procedure

An FLSP copper foil is processed on both sides and cut into strips (about 1 cm by 2 cm) that can be loaded into the ASAP 2460 sample tubes. Foils are selected over thicker samples to minimize the unprocessed area being measured and increase surface area to mass ratio. The samples are examined under the Keyence confocal laser scanning microscope to measure surface area and provide visuals of the surface.



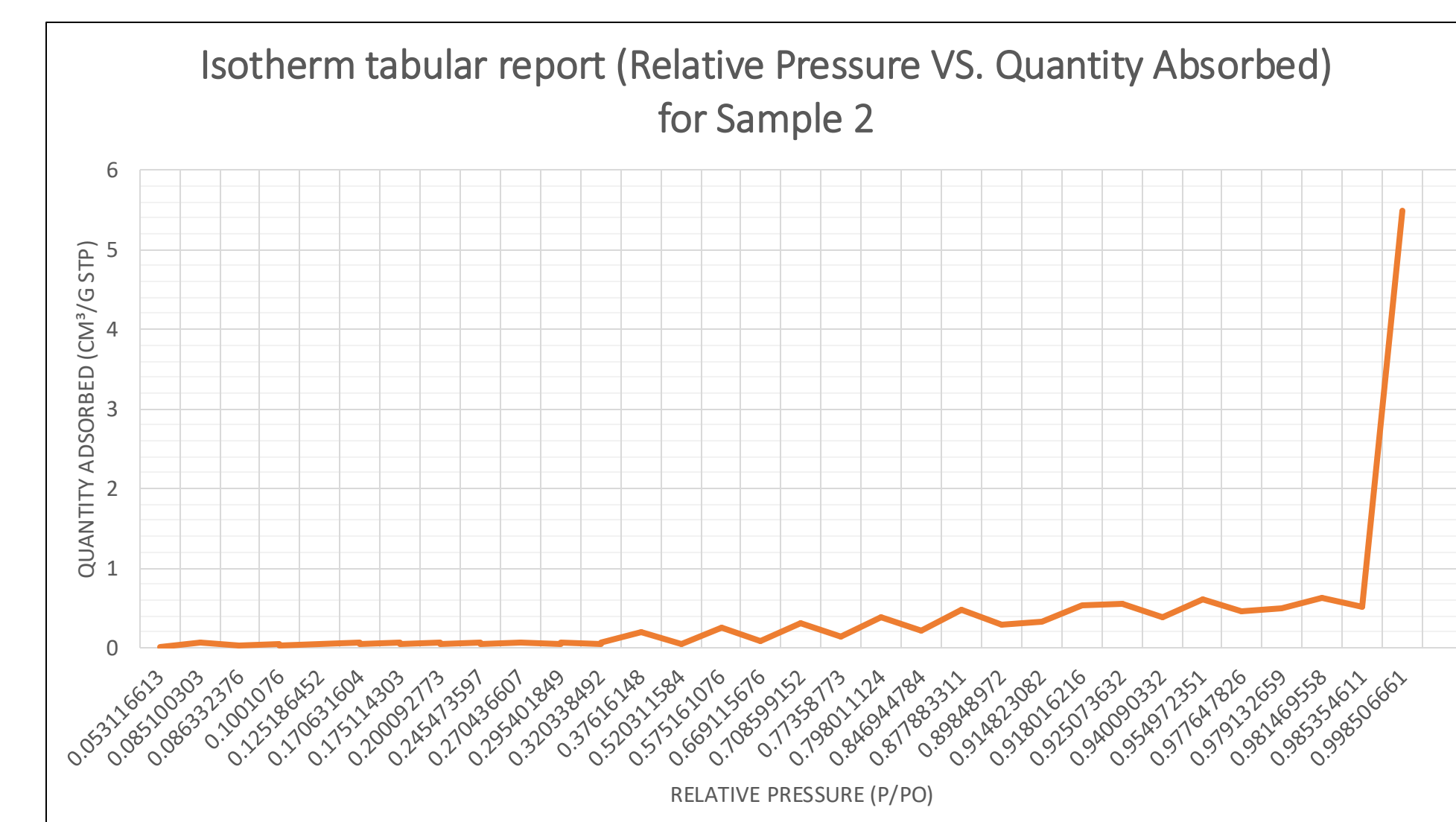
In this first experiment, the sample masses are at 2 grams or above per sample tube, but in subsequent experiments an occasional low signal issue is resolved by bringing samples of 5 grams or above. The following steps are completed to get the measurements:

1. Degas for 4 hours at 120 °C at an evacuation rate of 25 mmHg/s to a vacuum level of 0.04 mmHg. All other conditions are set based on the standard procedures.
2. Leave the samples overnight at 50 °C.
3. Measure with liquid nitrogen using high throughput analysis.



Identifying when results are inaccurate and how to fix the issue was an important part of the process. When an isotherm graph has **no negative absorption values** and the BET graph is **positively linear**, the results are likely to be accurate. Inaccurate results indicate that the degas conditions should be adjusted, or there is a leak. If data is missing from the report completely, this is likely from a bad signal, **meaning the sample does not have enough mass**.

Results



ASAP 2460 Results for a 2.1 cm x 0.7 cm Sample:

Average BET Surface Area Per Gram: 0.36 m²/g

Average Pore Diameter (BET): 5.7 nm

Total Surface Area: 0.093 m²

Confocal Laser Scanning Microscope Results

for a 2.1 cm X 0.7 cm Sample:

Surface Area Per Area: 6.1586

Total Surface Area: 0.0018 m²

The ASAP 2460 measured a higher surface area. This supports predictions about the ASAP 2460's abilities to measure subsurface porosity and small structures.

The higher surface area measurements, linear BET plots, and positive isotherm graphs all indicate that measuring solid FLSP surfaces with the ASAP 2460 can produce proper results.

ASAP 2460:

- Better accuracy—a measurement is taken of the whole sample instead of one small part
- Measurements take hours to days and require more expertise on operation of the equipment.
- The user must learn the degas conditions, but there is not much literature on solid FLSP samples on ASAP machines.

Confocal Laser Scanning Microscope:

- A single detailed measurement can take about 5-10 minutes
- Can only measure a tiny amount of the surface at a time, so the total surface area is a rough estimate at best.
- There's a very significant difference in the amount of surface area it picks up

ACKNOWLEDGMENTS AND REFERENCES

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"BET Theory." *Anton Paar Wiki*, Anton Paar. <https://wiki.anton-paar.com/en/bet-theory/>, accessed 7 July 2025.