# Characterization of Low Density Materials and Polymer Capsules Using X-rays

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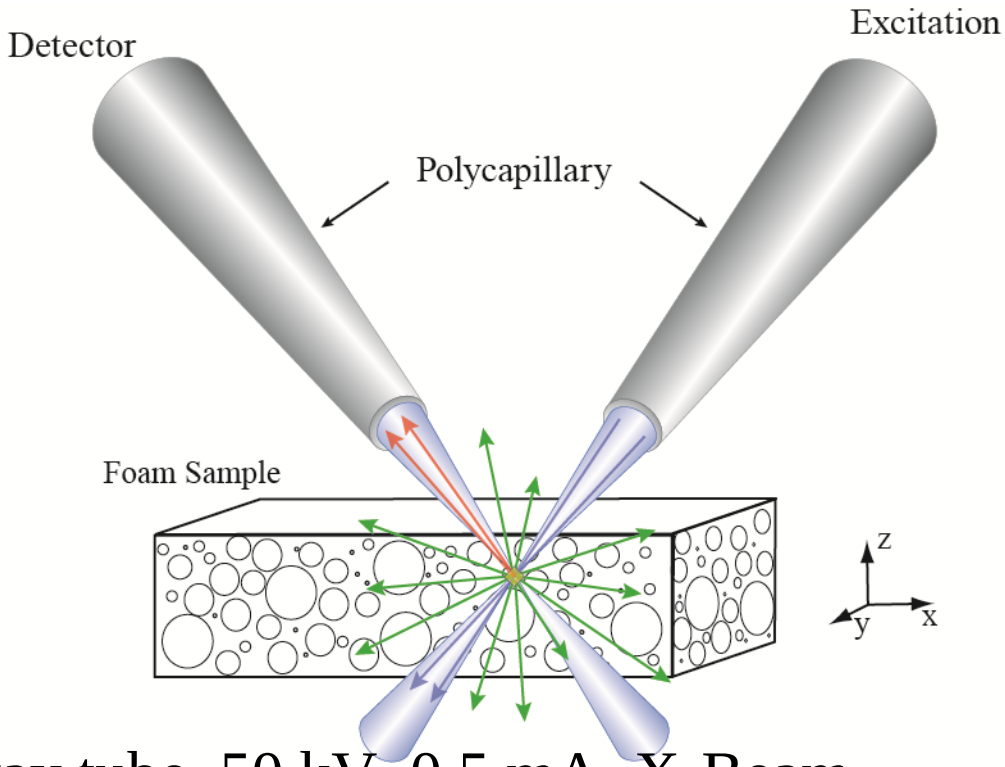
# Overview

- Introduction
  - ➔Confocal MXRF
  - ➔X-ray Tomography
- Applications to Density Measurements
  - ➔Polymer Foams and Aerogels
    - ✓ 2D and 3D imaging
- Applications to Polymer Capsules

# Confocal MXRF Instrument Geometry




- Ag X-ray tube, 50 kV, 0.5 mA, X-Beam
- Excitation and detection optics are ~30 micrometer focal spots with ~10 mm working distance

# Low Density Standards

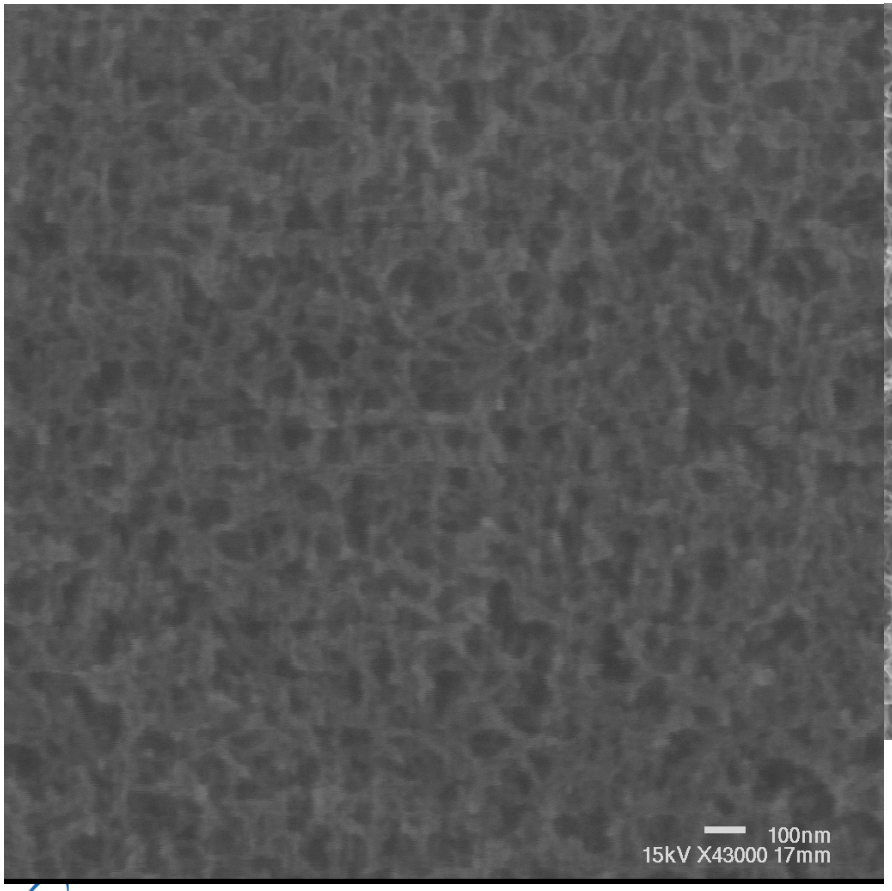
- Polymer Foams
  - ➔CH, poly(styrene); ~28-160 mg/cm$^3$
  - ➔$CH_2$, poly(methylpentene); ~11-70 mg/cm$^3$

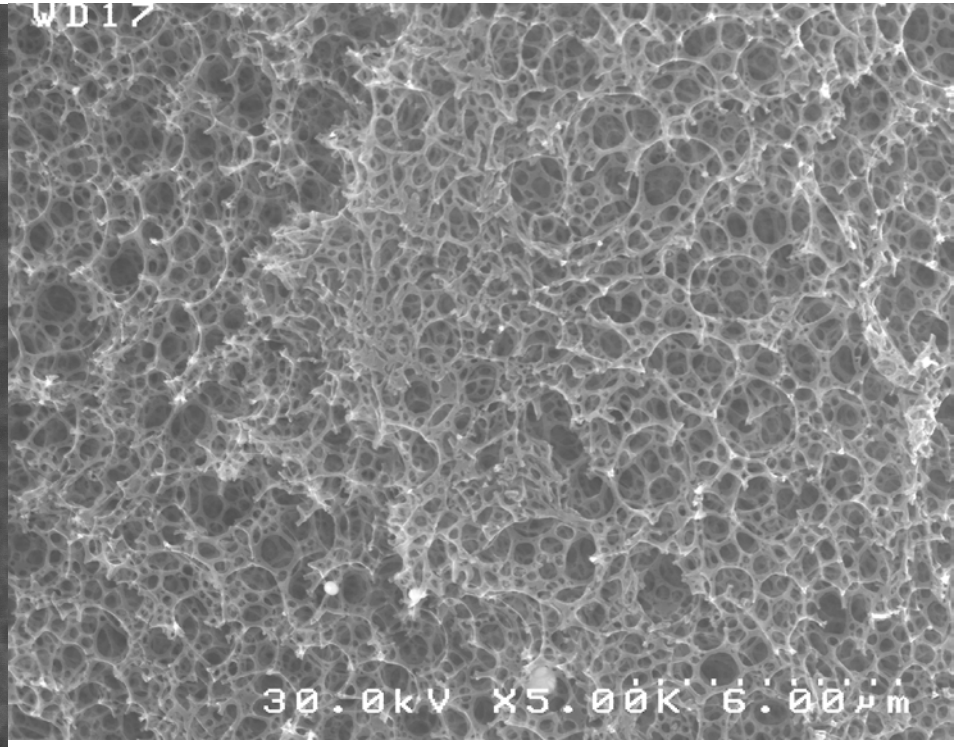
- Two formulations of aerogels
  - ➔TMOS (~30-120 mg/cm$^3$) and CS (~5-200 mg/cm$^3$) aerogels
  - ➔Cast and Machined



Patterson, B. M., et. al; X-ray Spectrometry, 2012, Accepted

# SEM Images of Aerogel (l), PS (r)



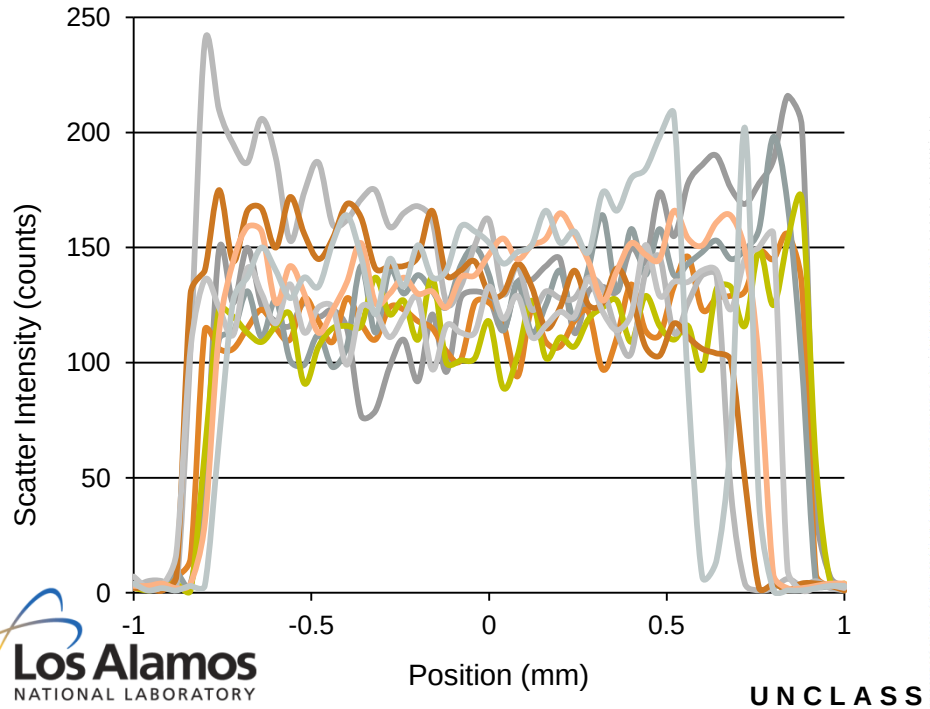





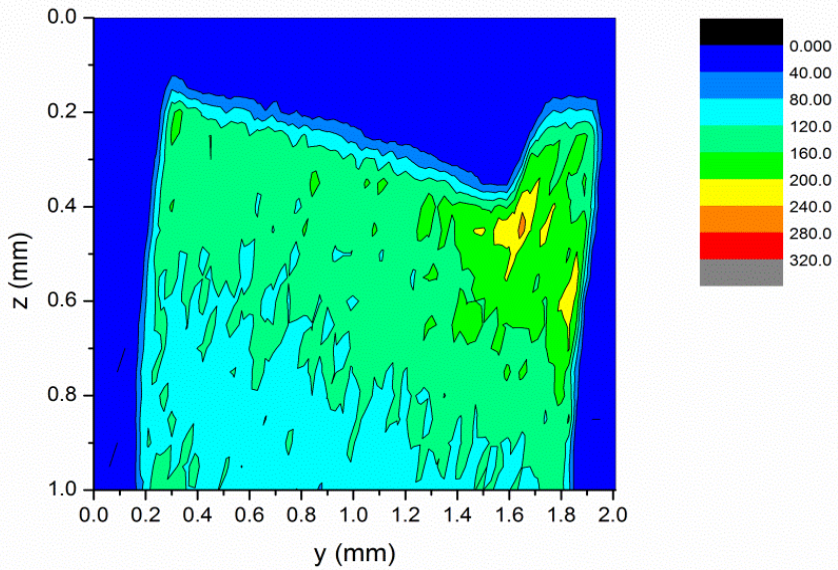

Patterson, B. M., et. al; X-ray Spectrometry, 2012, Accepted

# Repeatability of 30 mg/cm$^3$ aerogel

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Average | 127.3 | 117.2 | 130.9 | 113.6 | 146.0 | 137.0 | 122.2 | 141.0 | 147.3 | | 131.4 |
| std dev | 25.6 | 11.1 | 17.5 | 12.1 | 22.2 | 18.4 | 11.4 | 13.0 | 34.4 | | 12.3 |
| | | | | | | | | | | | |
| %RELSTD | 20.1 | 9.5 | 13.4 | 10.7 | 15.2 | 13.4 | 9.4 | 9.2 | 23.4 | | 9.4 |

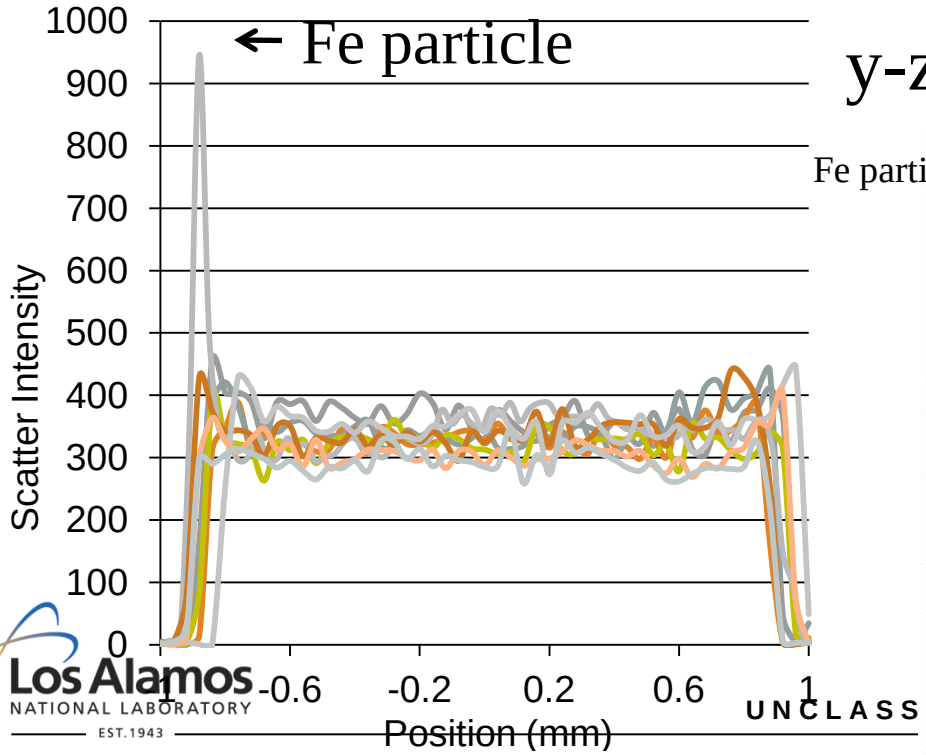



## y-z profile of 30 mg/cm$^3$

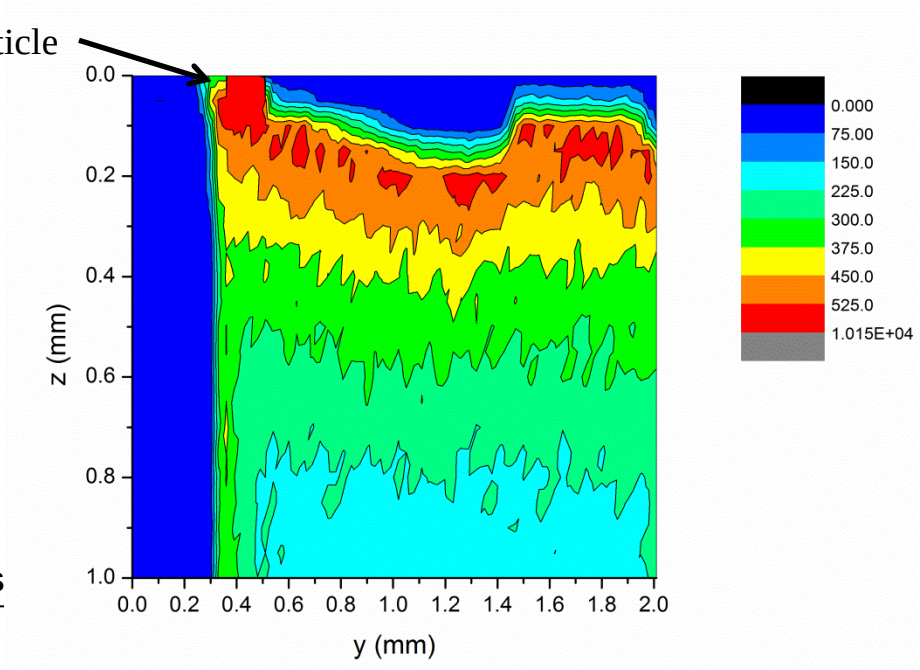


# Repeatability of 30 mg/cm$^3$ aerogel

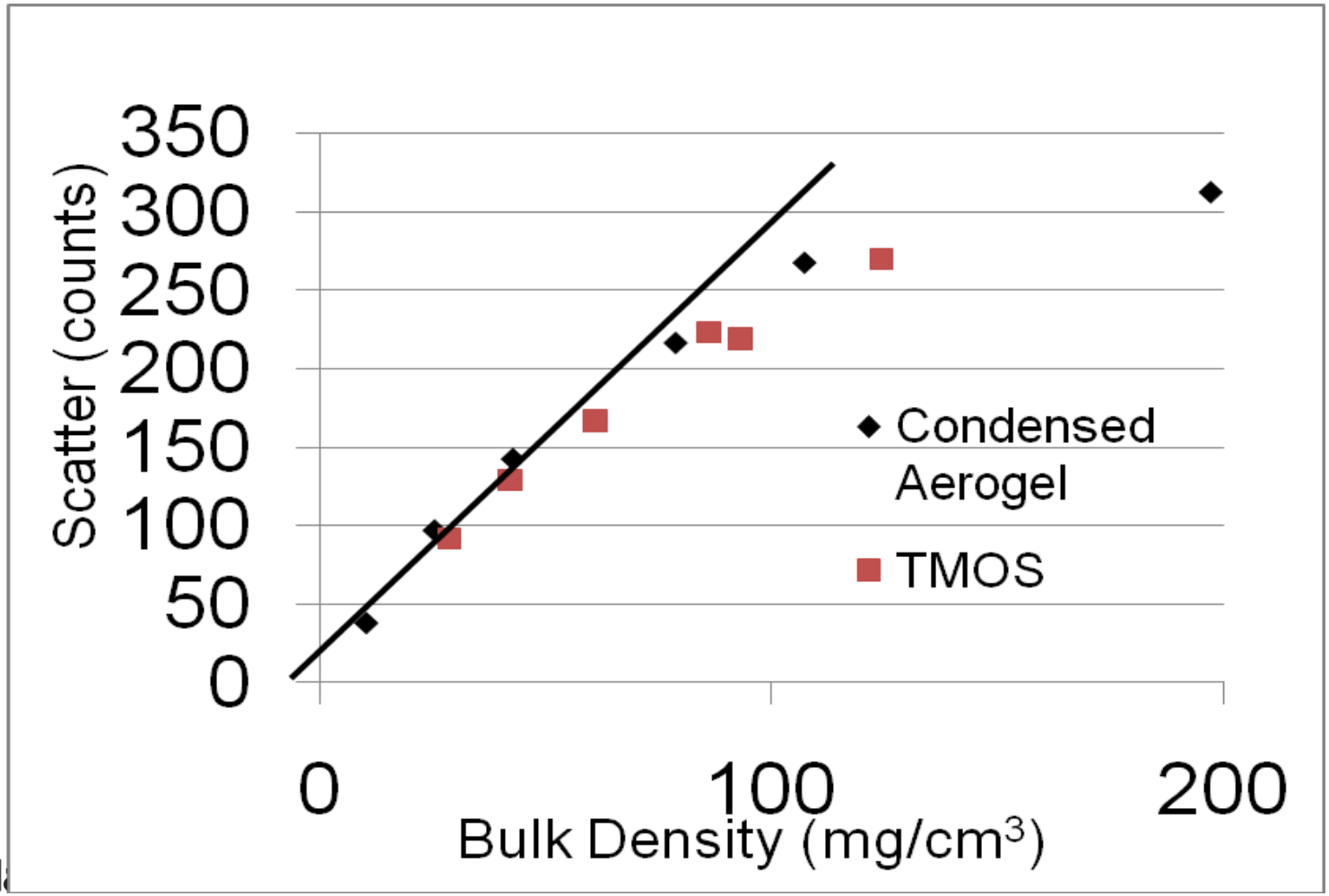
| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Average | 345.3 | 336.4 | 327.9 | 347.1 | 338.3 | 361.4 | 303.2 | 294.3 | 322.5 | | 330.7 |
| SD | 26.9 | 19.2 | 15.8 | 20.0 | 20.1 | 22.1 | 13.6 | 20.3 | 17.9 | | 21.4 |
| | | | | | | | | | | | |
| %RELSTD | 7.8 | 5.7 | 4.8 | 5.8 | 5.9 | 6.1 | 4.5 | 6.9 | 5.5 | | 6.5 |




y-z profile of 120 mg/cm$^3$ foam



# Scatter vs. Bulk Density (aerogel, 0.5 mm)



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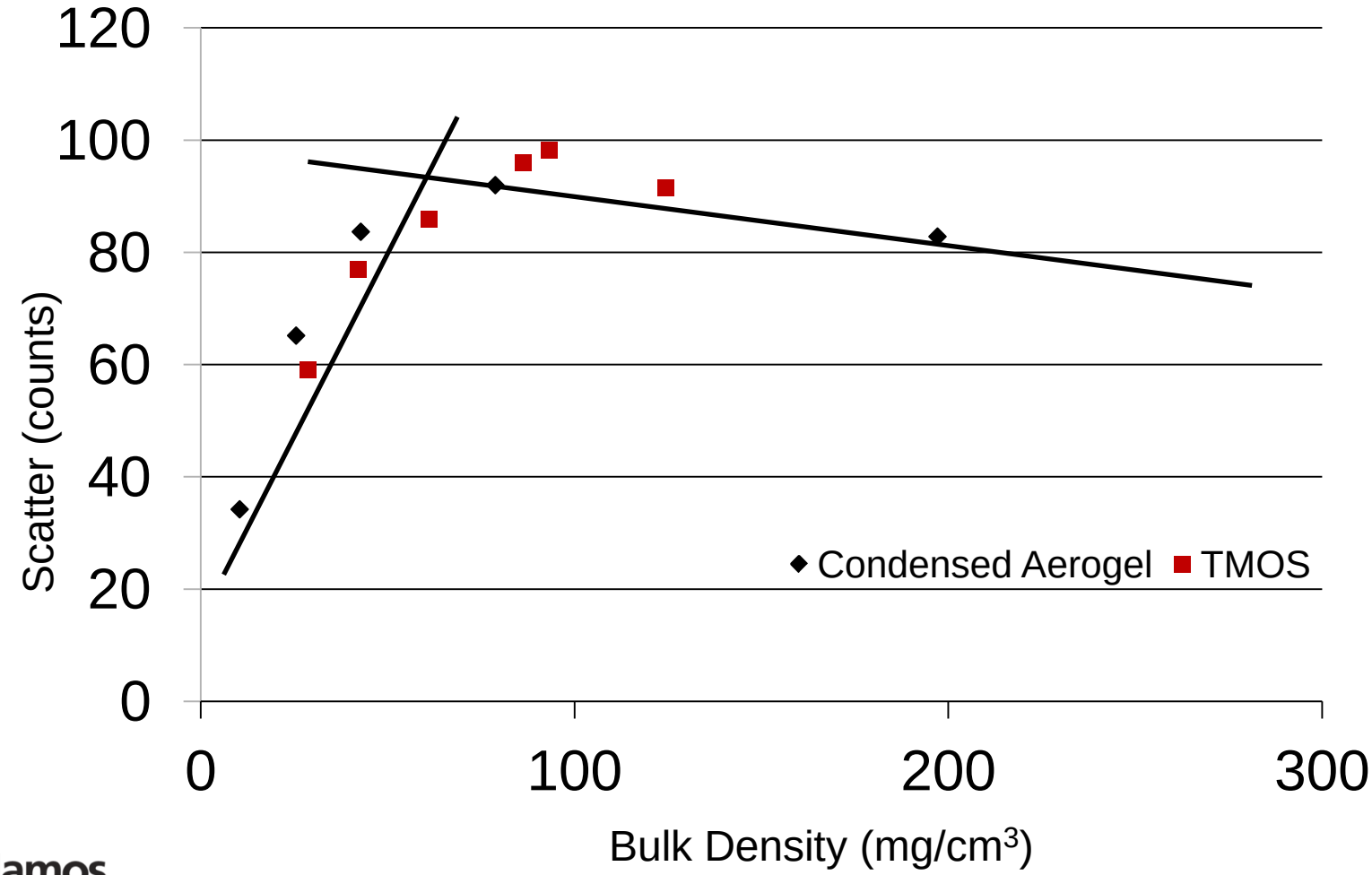
# Scatter vs. Bulk Density (aerogel, 2mm)



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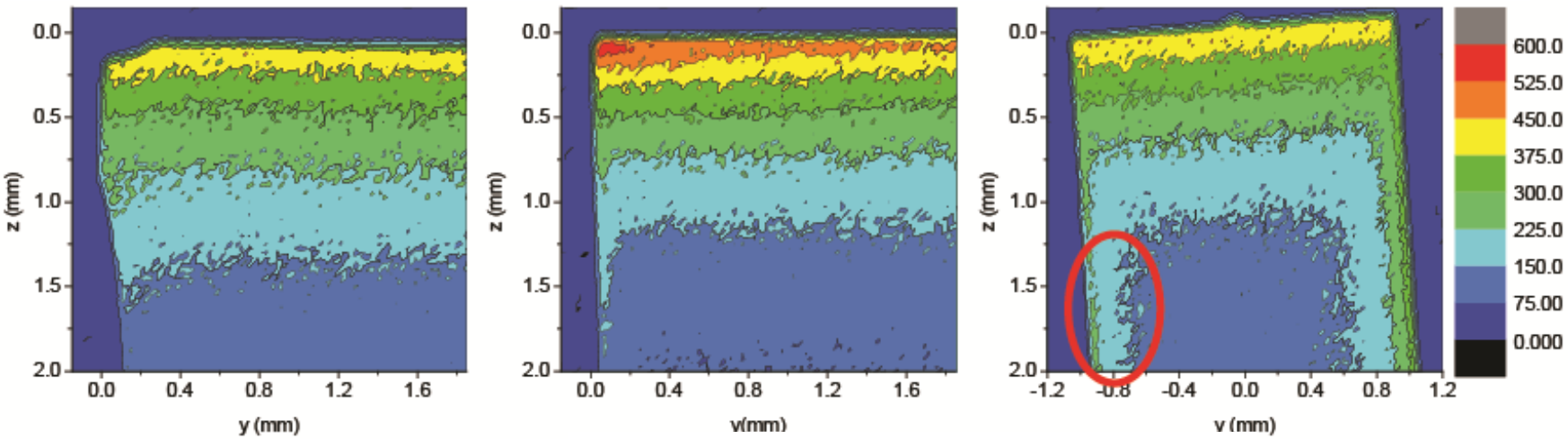
# y-z Slices Through Aerogels

Cast Ungreased

Machined




Cast Greased

Vertical changes in scatter intensity expected and due to absorption of the signal from the lower layers by the upper layers.

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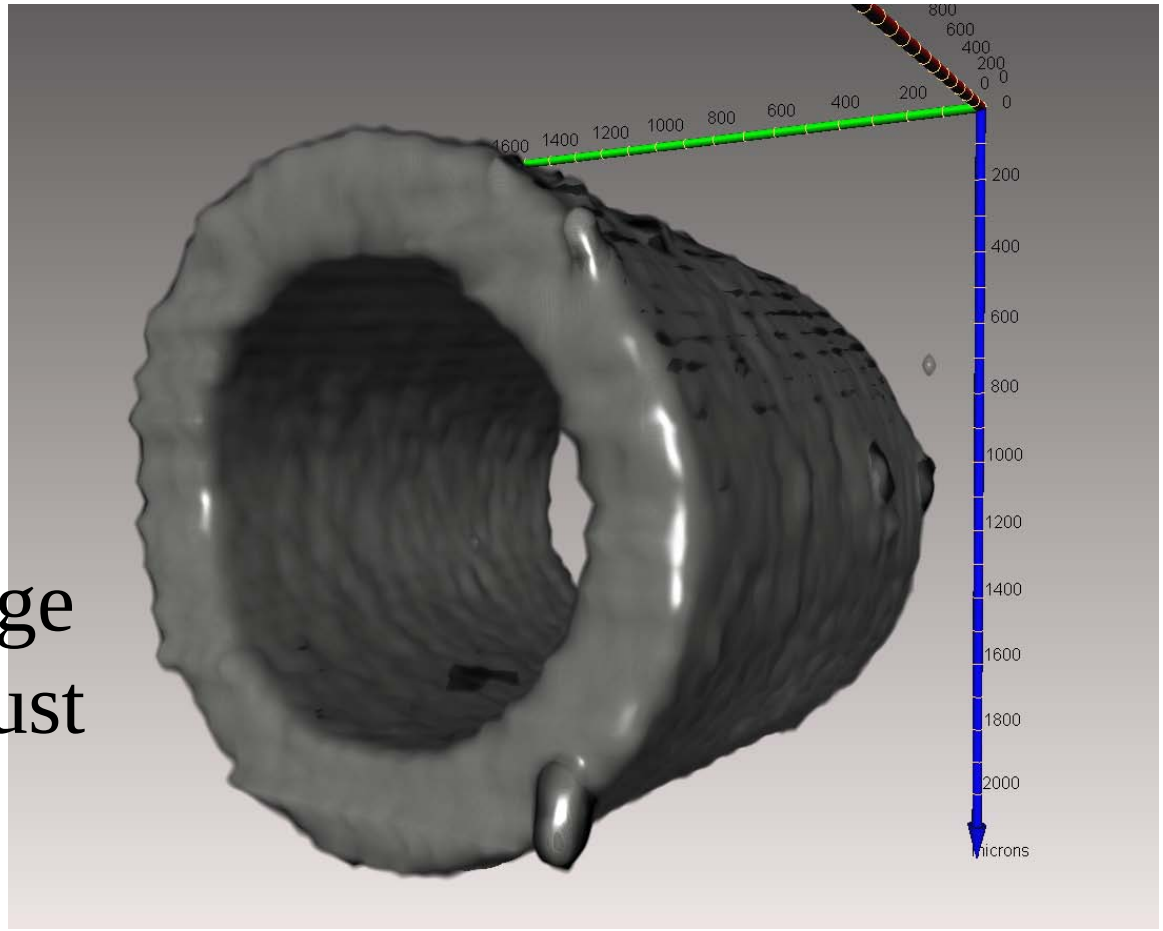
# 3D Image of Aerogel




- 80 mg/cm$^3$
-4 weeks to image
-‘bubbles’ are dust on surface



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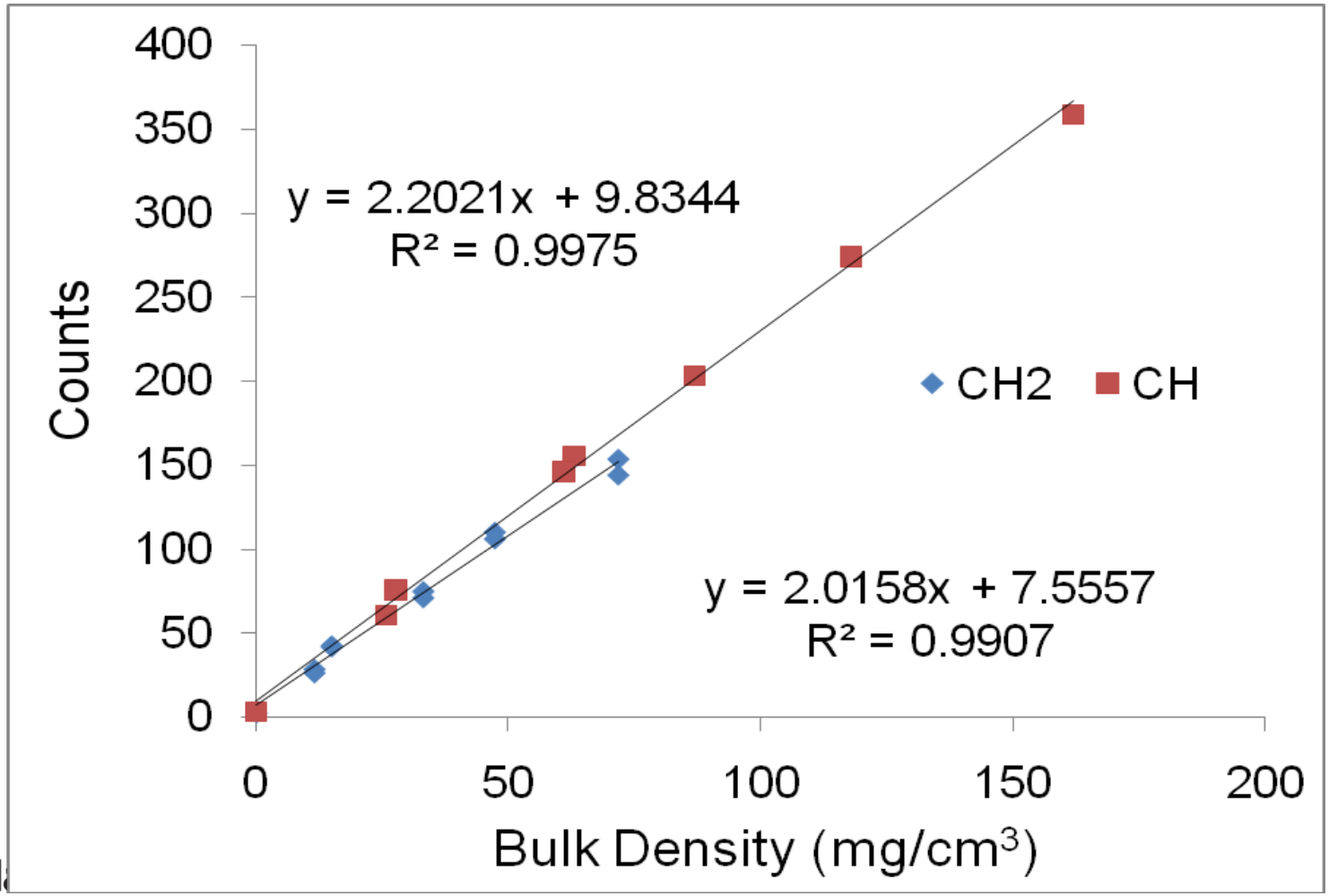
# Scatter vs. Bulk Density (CH and $CH_2$)



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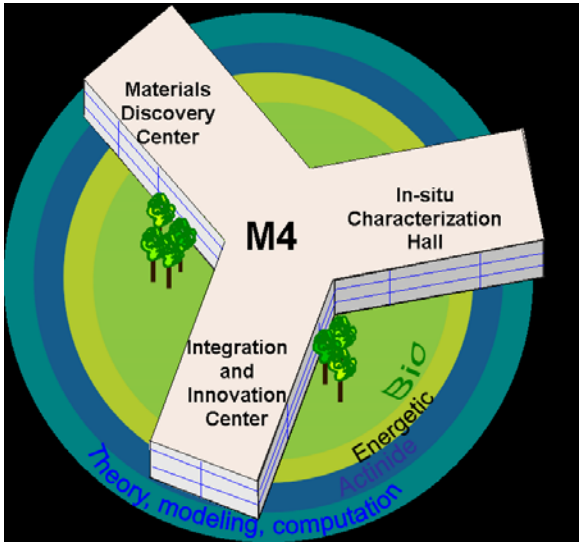
# 3D Imaging National Security Materials




50 nm resolution in 3D

3 µm resolution in 3D

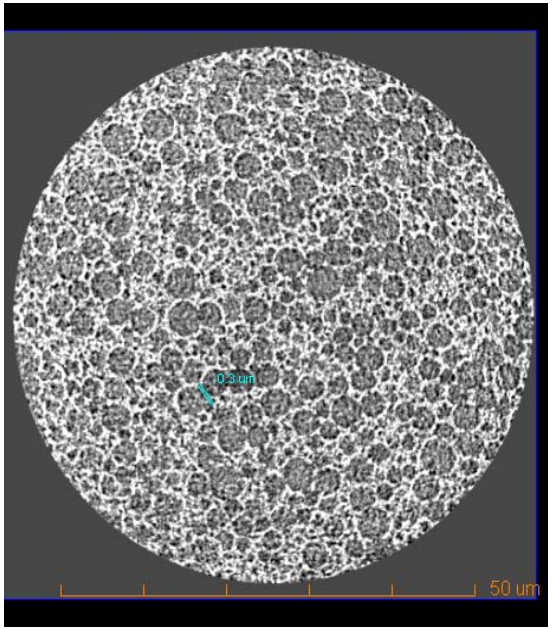
Nano-scale imaging in 3D

Imaging during experiment

Imaging either before or after experiment

Current research experience in processing 3D images gained on the micro-scale can be used on the nano-scale. Experience in 3D imaging gained on the nano-scale in the laboratory directly feeds into the requirements for MaRIE when processing dynamic 3D image sets.

# 3D image of Foam Using UltraXRM




Reconstructed slice through foam showing ligaments

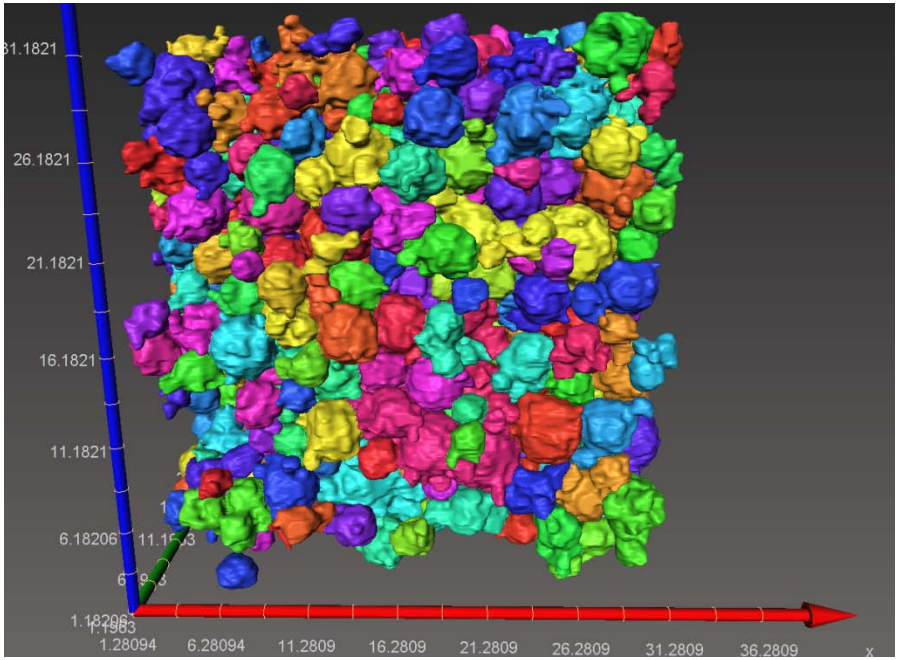


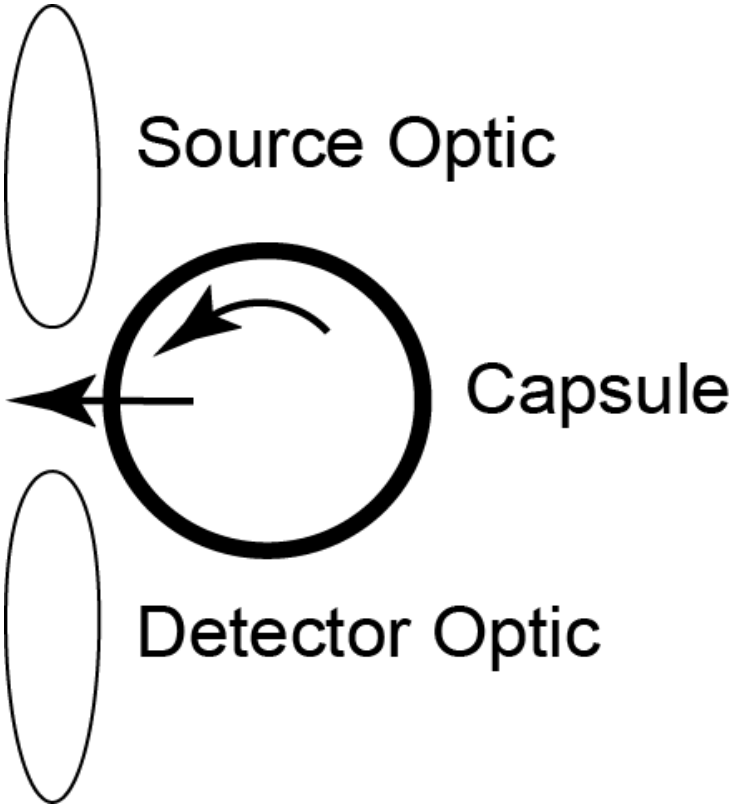

Image of labeled voids. This information can be used to gather statistics of the voids to compare lot-to-lot and experiment-to-experiment variations. Axis labels are in μm.

# Rigorous Meas. of Ge polymer Capsules

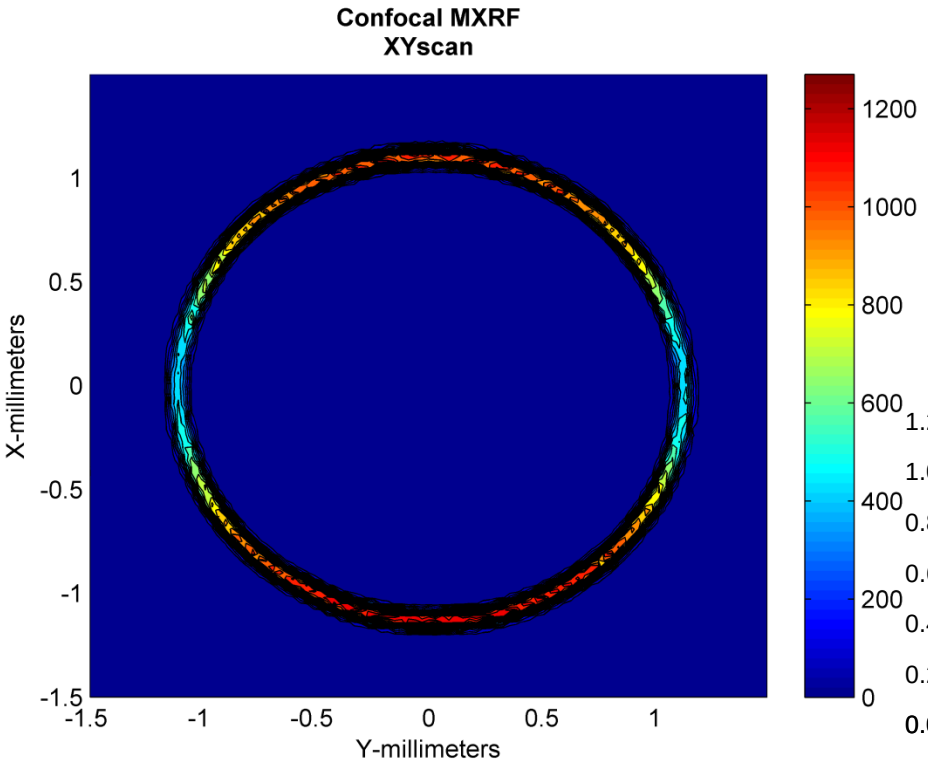
- Four capsules measured using confocal MXRF.
- Examine the ability of confocal MXRF to quantify Ge dopant; determine if measurement affects polymer.

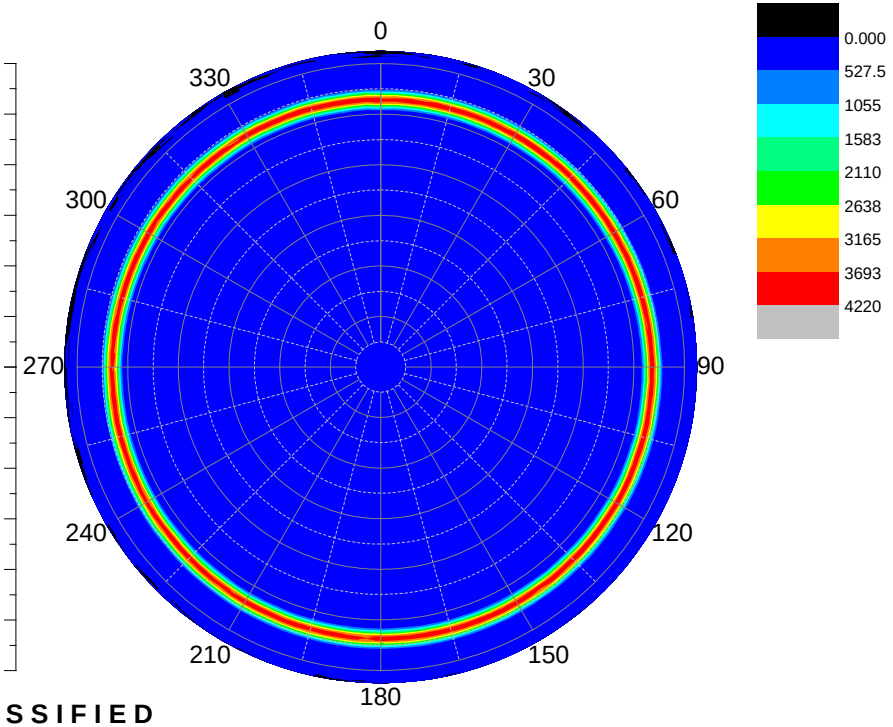
*"Further Characterizations of Sputtered Copper Beryllium Capsules using Confocal Micro X-ray Fluorescence"*, B. M. Patterson, et. al.; Fusion Science and Technology, (2011), 59(1), 121-125.

# Geometry of Capsule Measurement

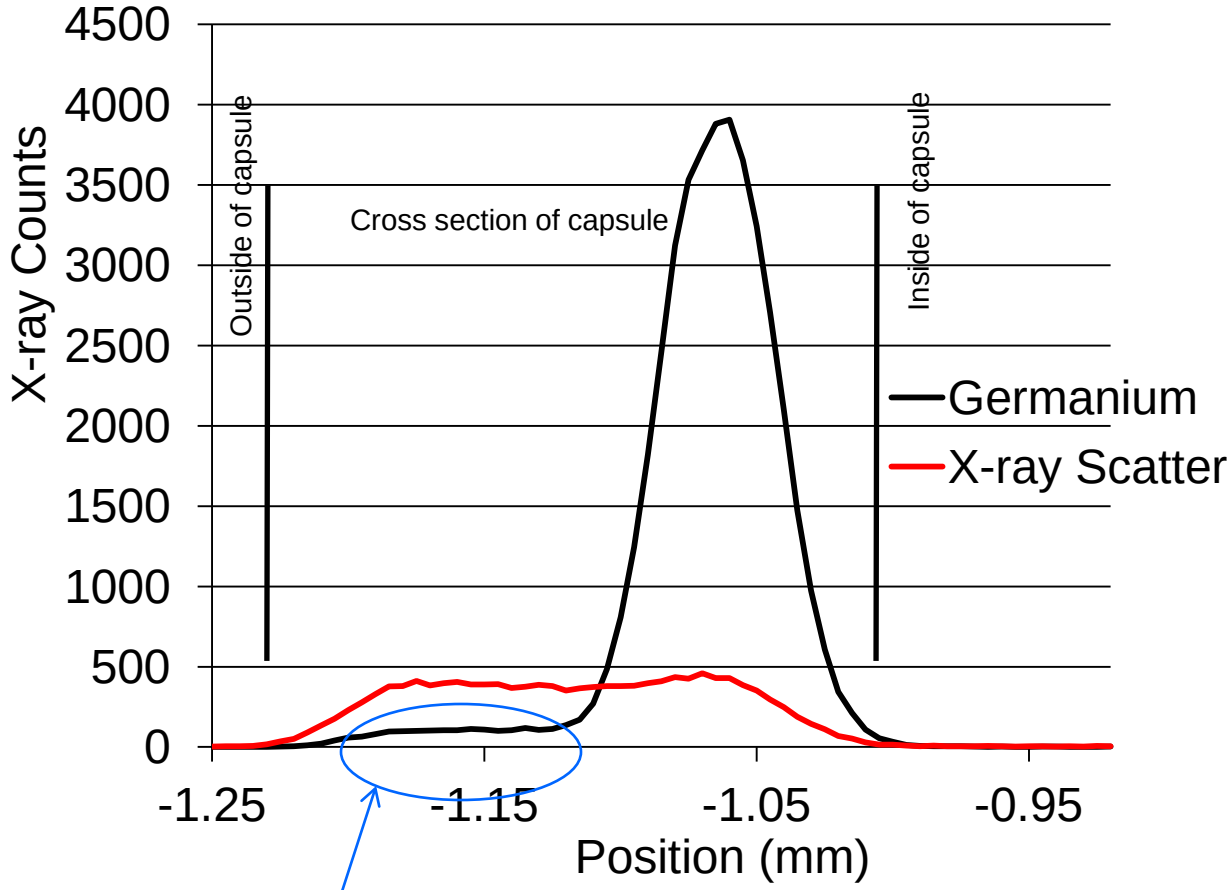


# Beam Geometry Effects



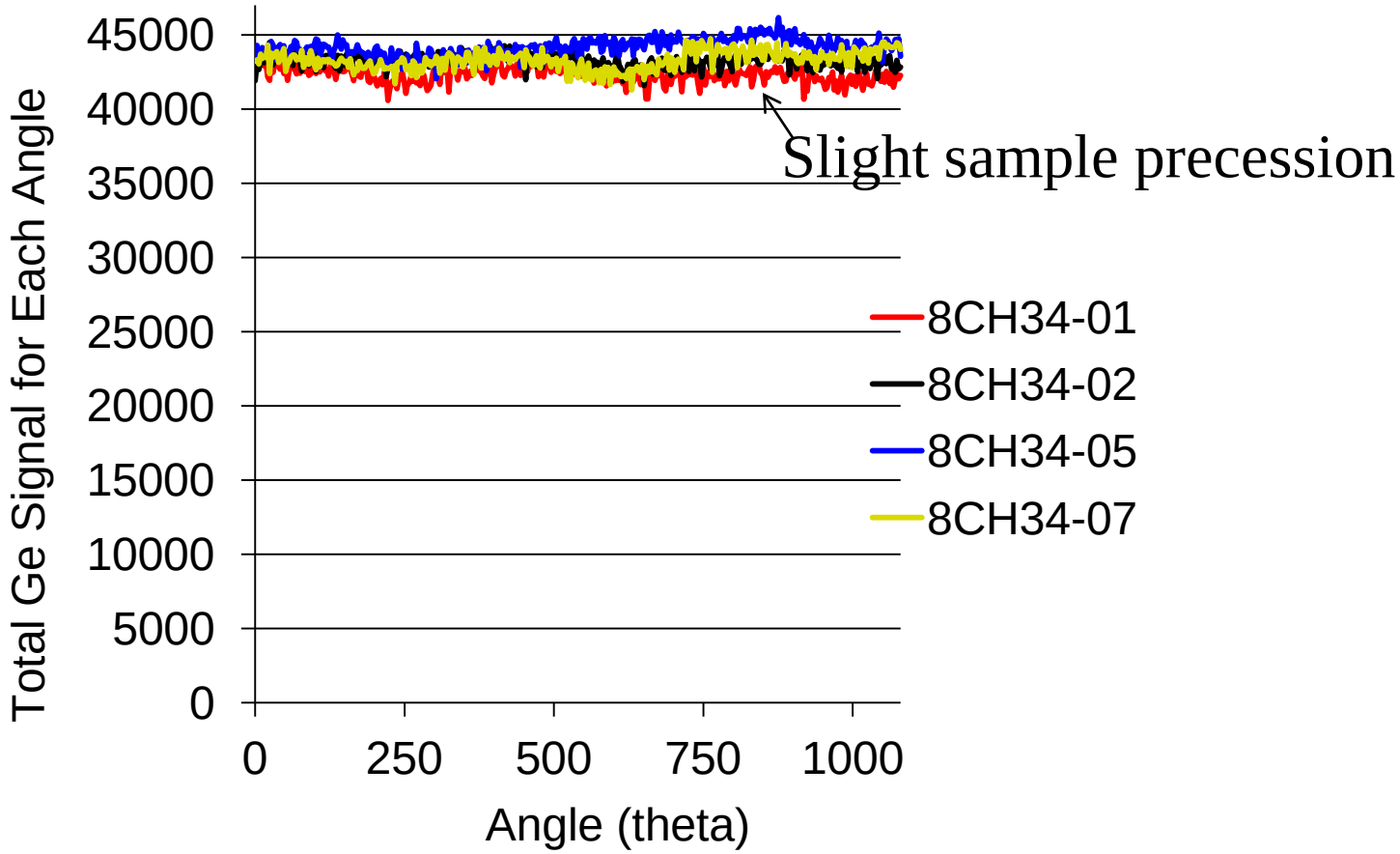



# Cross Sectional Profile

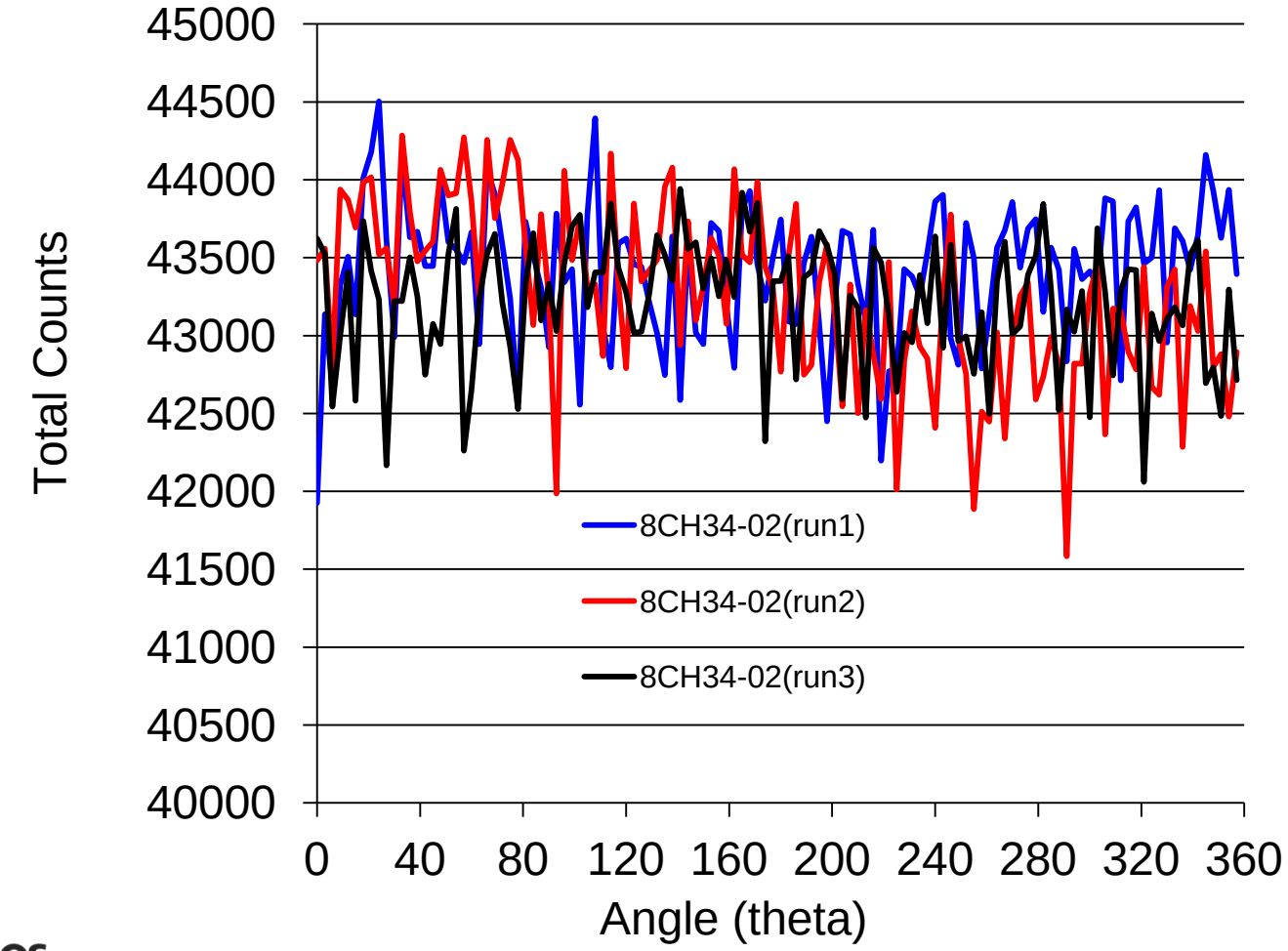



No baseline correction in elemental signal.

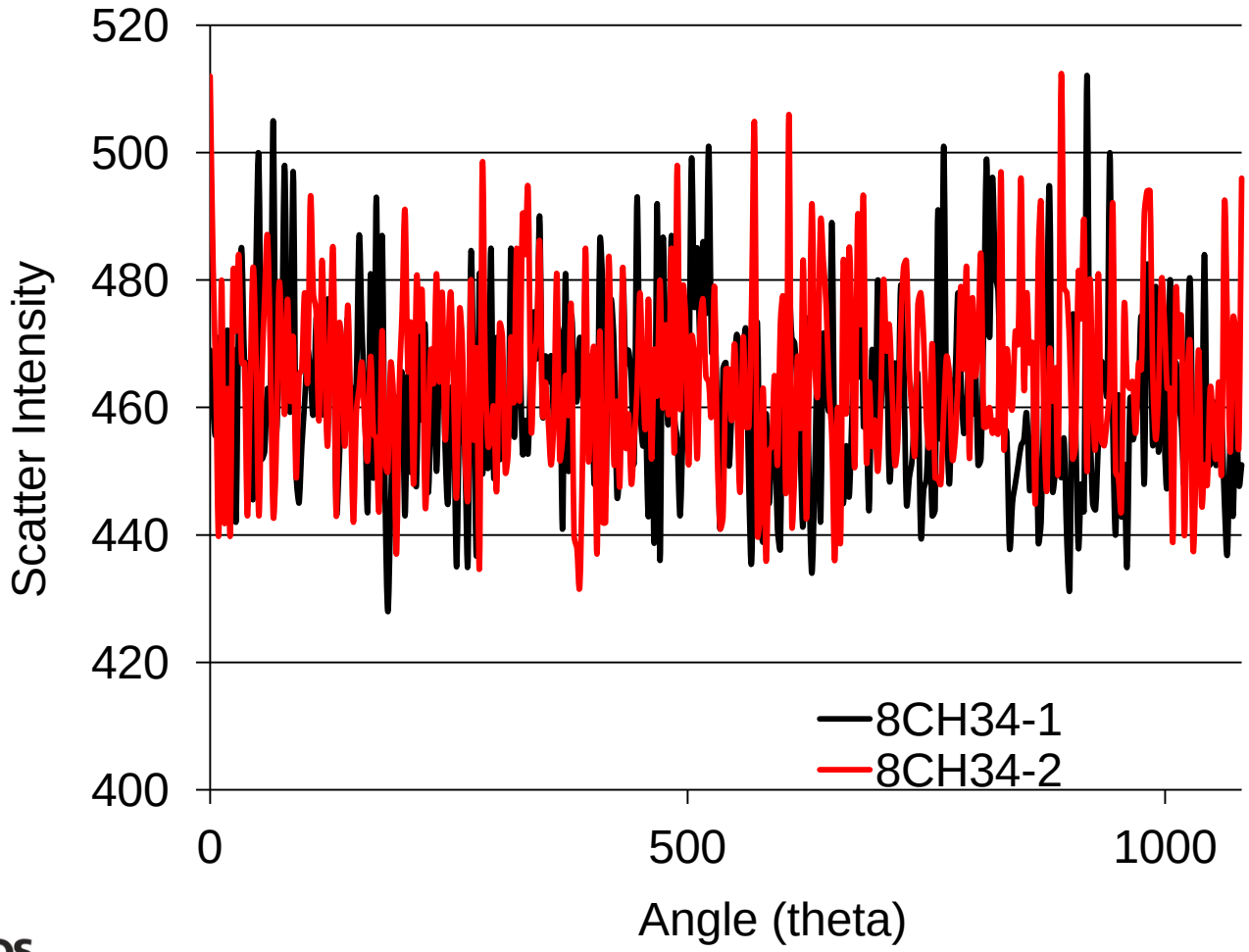
# Ge Signal f(theta)



# Repeatability of Ge Signal



# Scatter Intensity f(theta)



# Comparison of Capsules

| | 8CH34-1 | 8CH34-2 | 8CH34-5 | 8CH34-7 | Between |
|---|---|---|---|---|---|
| Ge Max | 14801.5 | 14875.1 | 15477.0 | 15216.1 | 15092.4 |
| StdDEV | 272.5 | 205.9 | 155.5 | 125.3 | 313.6 |
| %RelSTD | 1.8 | 1.4 | 1.0 | 0.8 | 2.1 |
| Ge Sum | 157677.0 | 160035.3 | 164024.8 | 162058.6 | 160948.9 |
| StdDev | 3471.2 | 2403.9 | 3009.4 | 2307.7 | 2722.3 |
| %RelStdDev | 2.2 | 1.5 | 1.8 | 1.4 | 1.7 |

# Ge doped capsule conclusion

- Some precession seen in the data.
- Confocal MXRF instrument very stable and repeatable over days and between shutdowns.
- X-ray scatter intensity is not affected by exposure.

# Conclusions

- Confocal MXRF gives us clues to the bulk density differences; future work with ‘nano-CT will image variations in void structure to better understand collapse.
- Future instrument based upon confocal MXRF should allow for quantification of dopant levels without damaging polymer.

# Acknowledgments


- Doug Hatch and Derek Schmidt for providing the machined aerogel.
- Derek Schmidt and Jim Williams for upgraded instrument software.
- Funding (LANL)
  - ICF Program



*"Three-Dimensional Density Measurements of Ultra Low Density Materials by X-ray Scatter Using Confocal Micro X-ray Fluorescence Spectroscopy"* Patterson, B. M., et. al; X-ray Spectrometry, 2012, accepted.

*"Further Characterizations of Sputtered Copper Beryllium Capsules using Confocal Micro X-ray Fluorescence"*, B. M. Patterson, et. al.; Fusion Science and Technology, (2011), 59(1), 121-125.

# Questions?