Temperature Programmed Desorption Secondary Ion Mass Spectrometer (TPD-SIMS)

Nikki Cain, Colin Evans, Kirsten Larson, Dalton Stone

Faculty Advisor: Michael V. Lee

Sponsor: Sandia National Laboratories

Sandia National Laboratories

SAND2018-4579 PE

Project Background

- Sandia's Z Machine uses high magnetic fields associated with high electrical currents to produce:
  - High temperatures
  - High pressures
  - Powerful X-rays
- Sandia wants to increase power in Z Machine
  - Reduce power flowing through gases desorbed from the sample
- Sandia's Goal for this Capstone:
  - Conduct small scale experiments

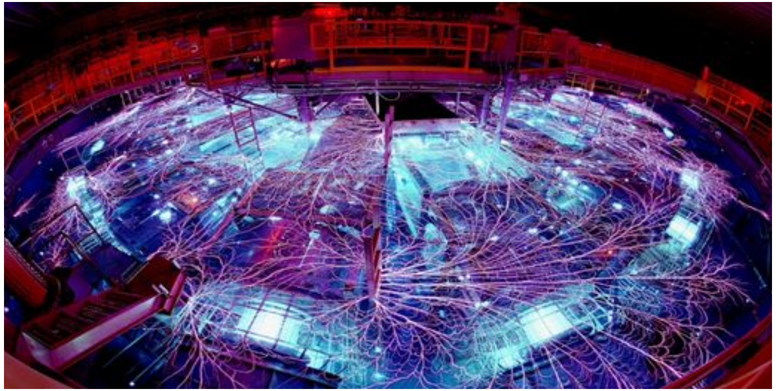

Figure 1: Sandia's Z Machine [1]

Terminology

TPD: Temperature programmed desorption
- Observing breakup of molecules from a surface as the temperature increases [2]

TOF: Time of Flight
- Time measurement of an ion's mass to charge ratio through mass spectrometry [3]

SIMS: Secondary ion mass spectrometer
- Collect and analyze secondary ions ejected from the sample due to a primary ion beam [4]

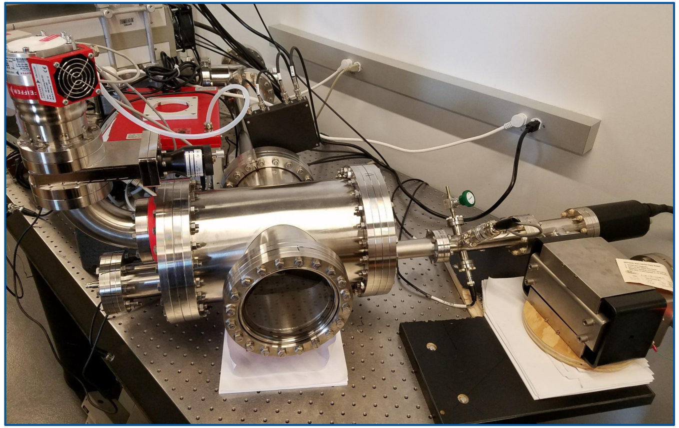

Figure 2: Improved TPD TOF-SIMS Setup

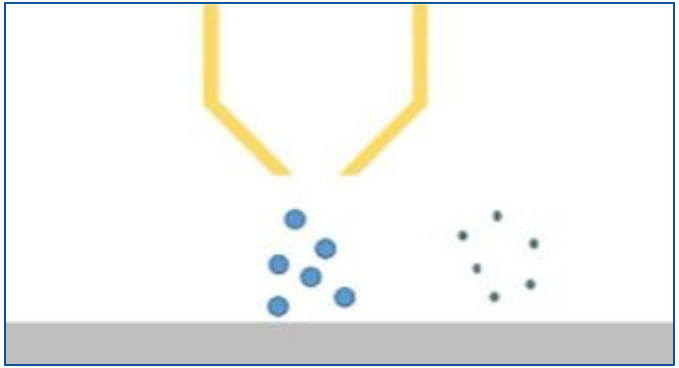

Figure 3: Particle Beam Interaction using TOF-SIMS

Project Goals

Project Goal:
- Modify the pre-existing TOF-SIMS chamber
- Design and manufacture a new sample holder

Updates to sample holder:
- Vacuum compatible
- TPD compatible
- Variable angle for the sample
- Thermally and electrically insulated

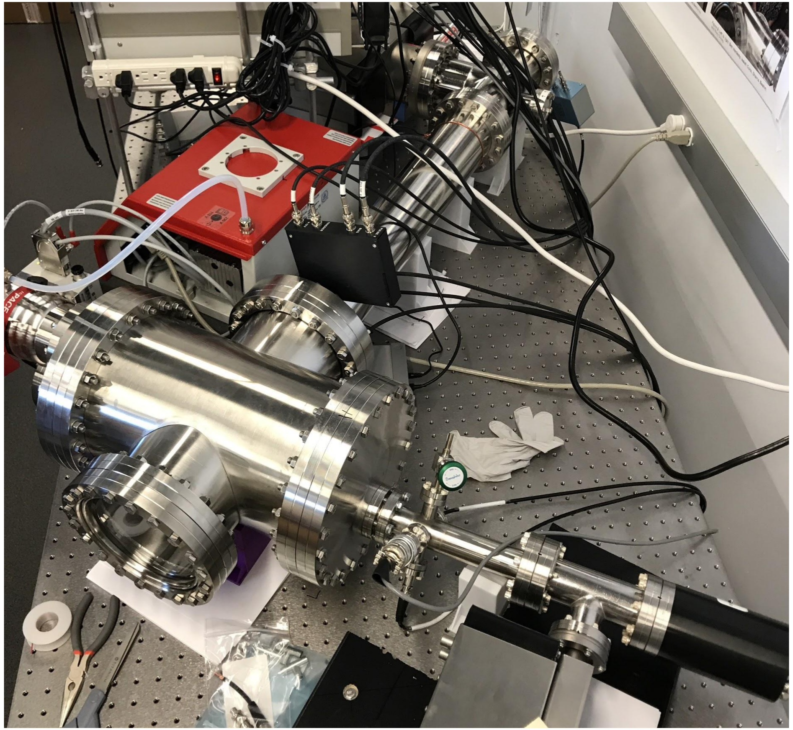

Figure 4: 2016-2017 TOF-SIMS capstone project; vacuum chamber, ion gun, 2 ion pumps, turbo pump, TOF-SIMS [5]

Design Approach - TOF-SIMS

Requirements:

- Record the spectrum of chemical compositions
  - Flood Gun and TOF
- Measure temperatures up to 1000°C
  - K-Type Thermocouple
- Keep TOF at pressures ≤ 10^{-7} torr
  - Use materials with low outgassing
  - Design to reduce trapping particles

Figure 5: Electron Flood Gun [6]

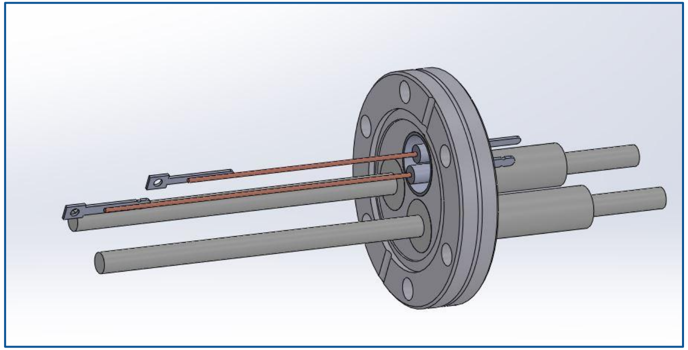

Figure 6: Thermocouple-Power Feedthrough [7]

Design Approach - Sample Holder

Requirements:

- Heat to 1000 ºC at a rate of 1 ºC/s
  - Using Joule Heating
- Temperature of TOF-SIMS chamber < 40 ºC
  - Thermal Insulator
- No electrical potential on chamber
  - Electrical Insulator

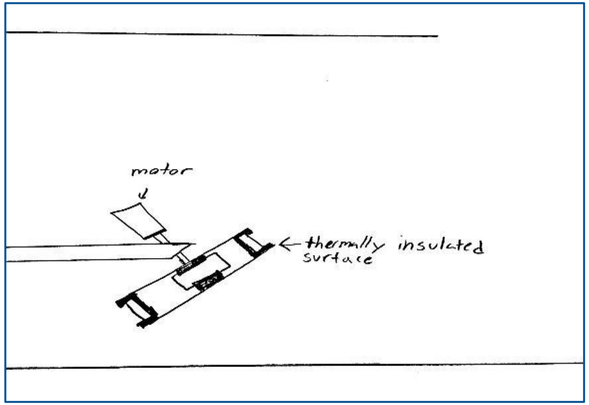

Figure 7: Concept Design for Sample Holder

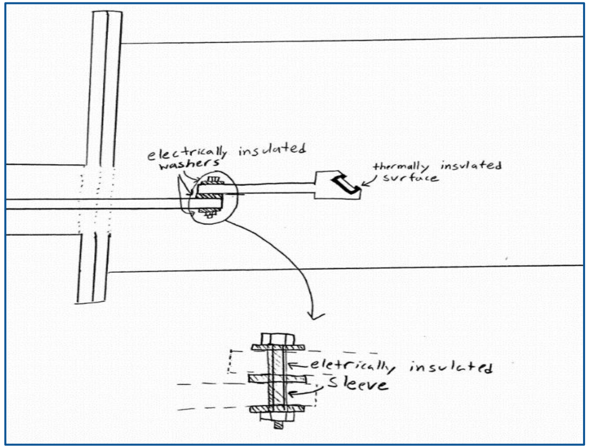

Figure 8: Concept Design for Sample Holder

Design Revision

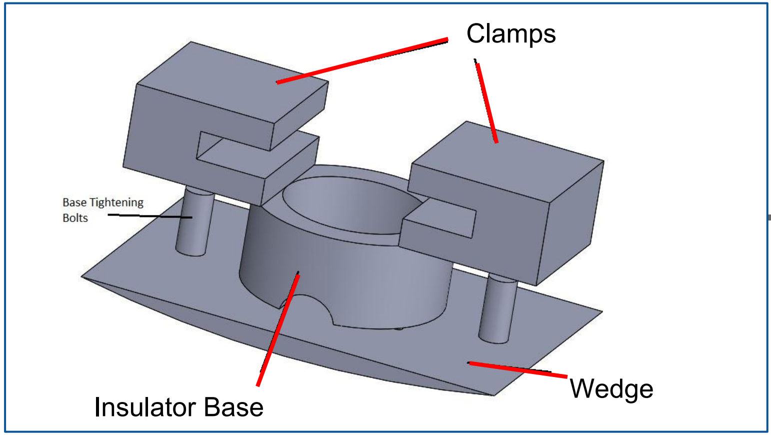

Figure 9: Conceptual Design

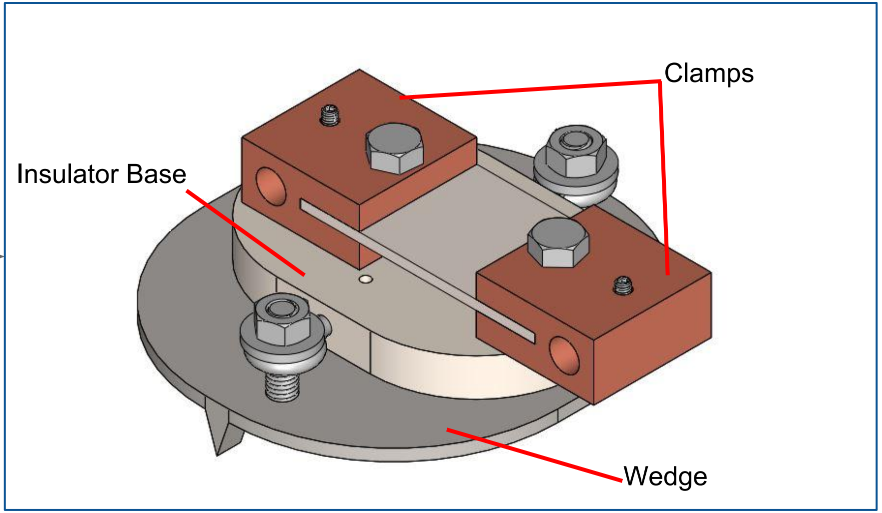

Figure 10: Final Design

Material Selection

Macor glass ceramic base for thermal/electrical insulation

Stainless steel wedge to level the sample holder in chamber

Copper clamps conducting joule heating power to the sample

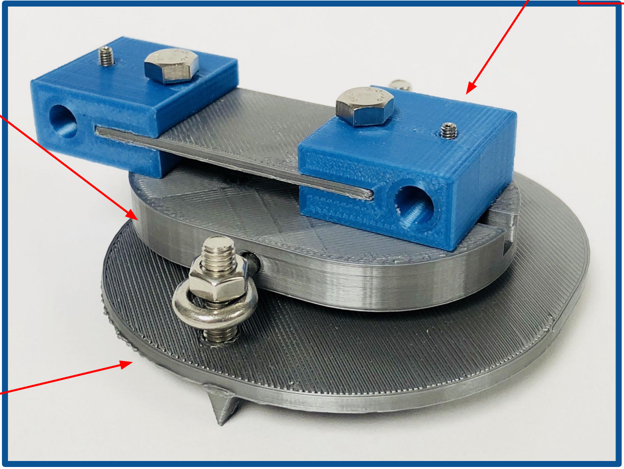

Figure 11: Prototype Design

Alignment system to make experiments repeatable

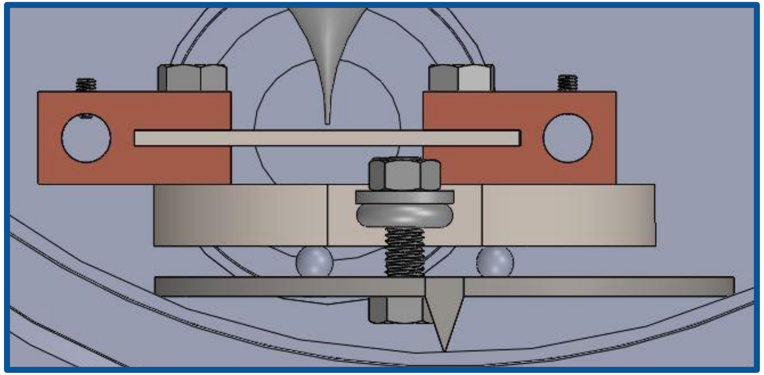

Figure 12: CAD of Final Design in Vacuum Chamber

Manufacturing

- Steel wedge and copper clamps manufactured by AC Manufacturing on a CNC machine
    - Holes drilled and tapped to correct thread
    - Teeth on the steel wedge were filed down to correct fit once in hand

- Macor sheet purchased from McMaster and machined by the engineering machine shop at NAU
    - Glued to an alternate plate in order to vice the part appropriately with no damage
    - CNC machined to correct shape and thickness
    - Holes were drilled and tapped

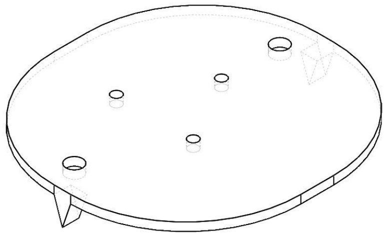

Figure 13: Final Design of Stainless Steel Wedge

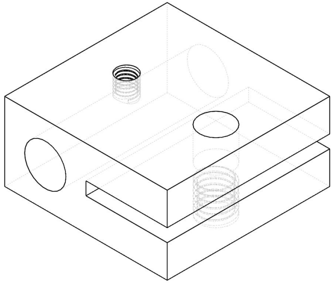

Figure 14: Final Design of Copper Clamp

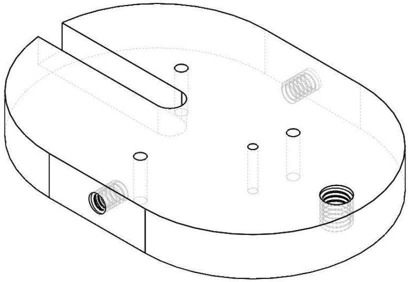

Figure 15: Final Design of Insulator

Assembly: Final Design

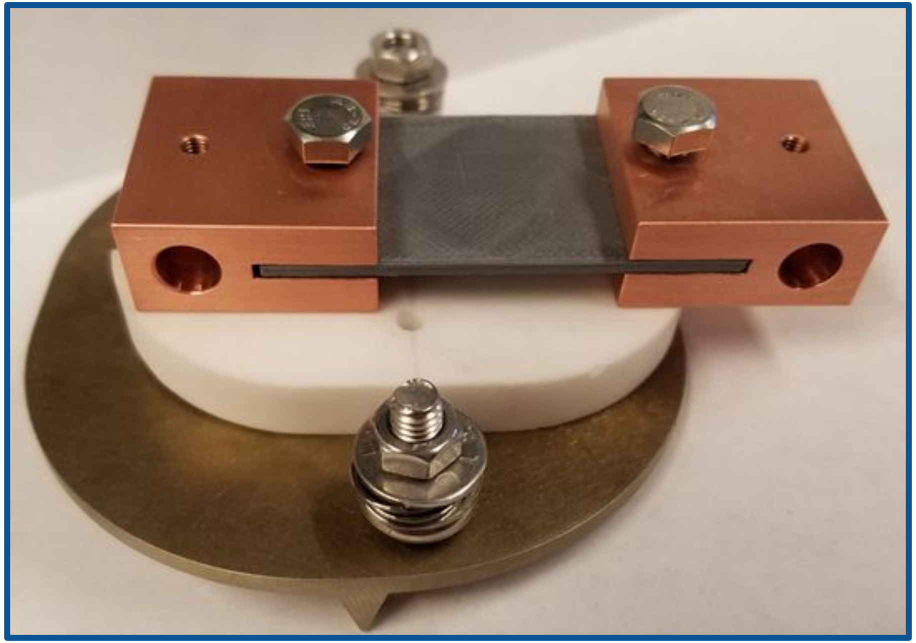

Figure 16: Final Manufactured Design

Outside Testing

Testing Completed:

- Disassemble and reassemble chamber with gate valve, elbow flange, and thermocouple power feedthrough
- Outgas TOF-SIMS chamber
  - Tested to reach below 10^{-7} torr
- Test joule heating and thermocouple
  - Outside vacuum chamber

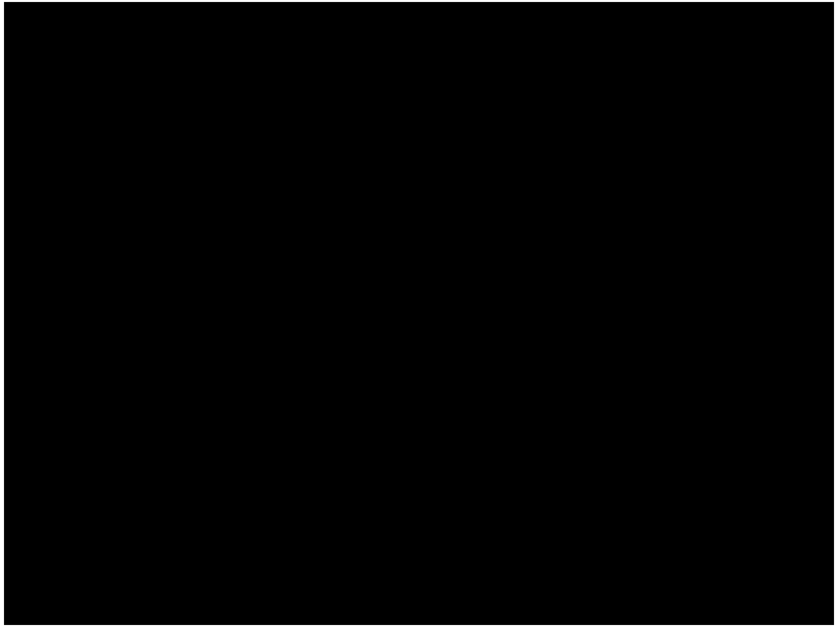

Figure 17: Outside Chamber Copper Clamp Test

Figure 18: Control Panel of Ion Pumps

Inside Testing

Testing Completed:

- Sample holder fits in chamber
- Test joule heating and thermocouple within vacuum chamber

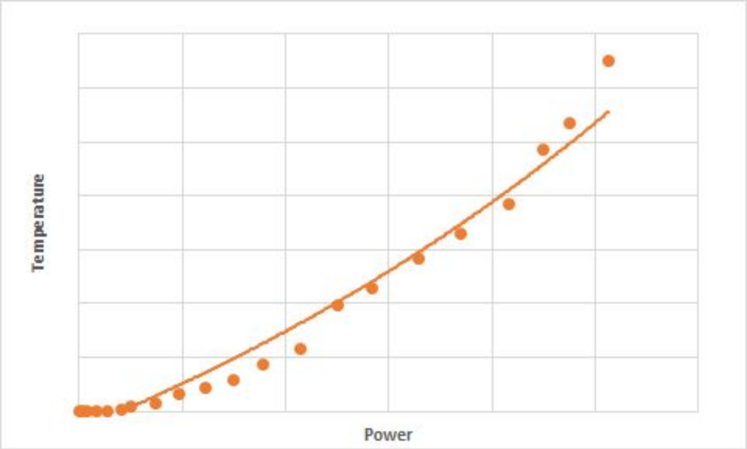

Figure 19: Data Collected From Testing Done Inside the Chamber

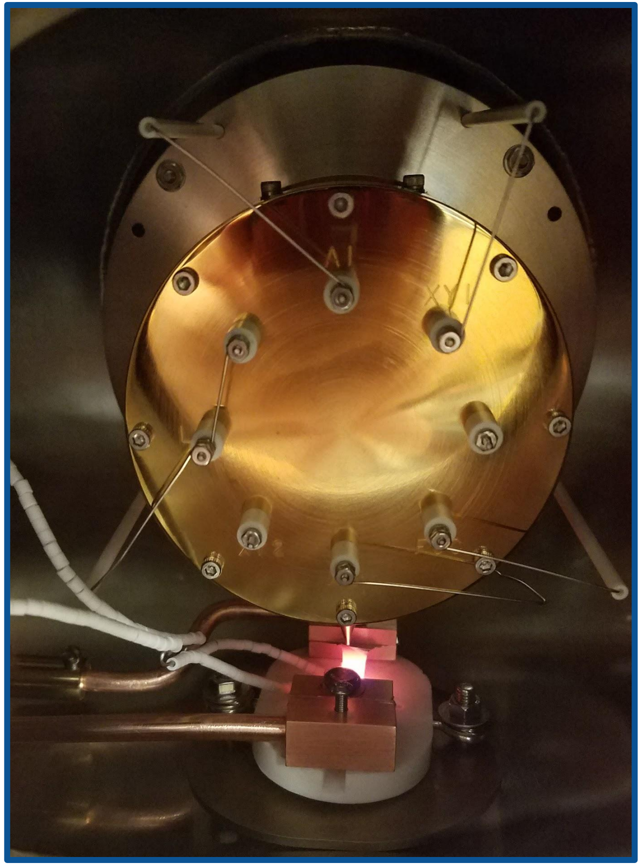
Figure 20: Joule Heating Test of Sample in the Chamber

Conclusion

- Sample can be heated to the temperatures required
- Calibration of the computer and thermocouple is completed
- Thermocouple accurately measures the temperature of the sample
- Sandia will use the testing to improve performance of their Z machine

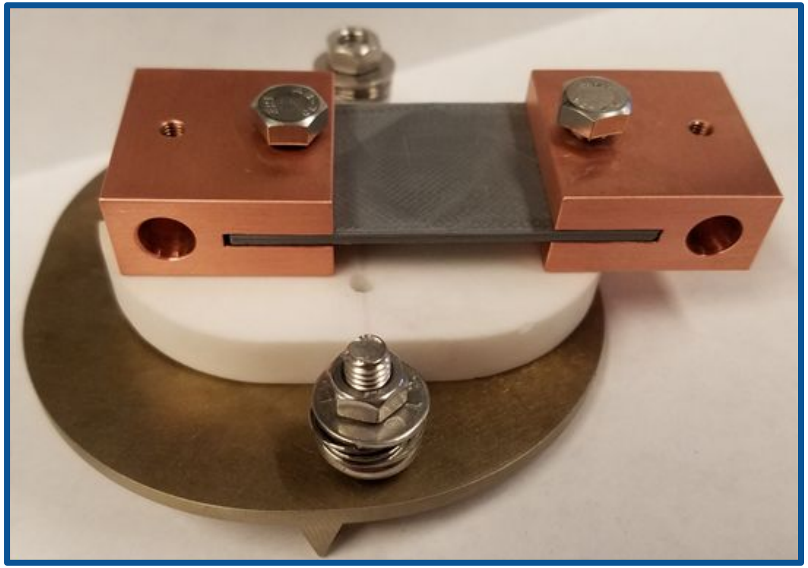

Figure 21: Final Manufactured Design

Future Work

Continue Testing with New Flood Gun May 2018

Communicate more with Sandia to further the project

Capstone 2019: Redesign entire chamber to heat sample faster

References

[1]"Sandia National Laboratories: Exceptional Service in the National Interest", Sandia.gov, 2018. [Online]. Available: http://www.sandia.gov/. [Accessed: 11- Apr- 2018].

[2] M. Komatsu and H. Hashimoto, "Time-of-flight secondary ion mass spectrometer", US7714280 B2, 2010.

[3]"TOF-SIMS Surface Analysis Technique | Physical Electronics (PHI)", Phi.com, 2017. [Online]. Available: https://www.phi.com/surface-analysis-techniques/tof-sims.html. [Accessed: 07- Oct- 2017].

[4] White, J. (1986). Measuring surface reaction rates using SIMS and TPD: An overview. Applied Surface Science, 26(4), pp.392-407.

[5] S. Beahm, I. Cons, R. Ducey and B. Hardesty, "Secondary Ion Mass Spectrometer Capstone Team 2017", *SecondaryIonMassSpec*, 2017. [Online]. Available: https://www.cefns.nau.edu/capstone/projects/ME/2017/SecondaryIonMassSpec/. [Accessed: 19- Apr- 2018].

[6]"Flood Gun FS 100", Omnivac.de, 2017. [Online]. http://www.omnivac.de/components/electron-sources/flood-source/index.html. [Accessed: 07- Oct- 2017].

[7]"Type K Mini Plug Plus Power - CF Flange, Single-Ended", Lesker.com, 2017. [Online]. Available: http://www.lesker.com/newweb/feedthroughs/thermocouple_feedthroughs_typek_miniplugpower_singleend.cfm?pgid=cf. [Accessed: 04- Nov- 2017].

Acknowledgments

Sandia National Laboratories

Dr. Michael V. Lee, NAU Chemistry Professor
Dr. Sarah Oman, NAU ME Professor

McMASTER-CARR

AC MANUFACTURING

Kurt J. Lesker Company

OmniVac
Surface Analysis and Vacuum Technology

DUNIWAY STOCKROOM CORP.

Questions?

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.