



Copper Electroforming at the Sanford Underground Research Facility

Cabot-Ann Christofferson

South Dakota School of Mines and Technology

MAJORANA DEMONSTRATOR Collaboration



The MAJORANA Collaboration



Black Hills State University, Spearfish, SD

Kara Keeter

Duke University, Durham, North Carolina, and TUNL

Matthew Busch

Institute for Theoretical and Experimental Physics, Moscow, Russia

Alexander Barabash, Sergey Konovalov, Vladimir Yumatov

Joint Institute for Nuclear Research, Dubna, Russia

Viktor Brudanin, M. Shirchenko, Sergey Vasilyev, E. Yakushev, I. Zhitnikov

Lawrence Berkeley National Laboratory, Berkeley, California and the University of California - Berkeley

Nicolas Abgrall, Mark Amman, Paul Barton, Adam Bradley, Yuen-Dat Chan, Paul Luke, Susanne Mertens, Alan Poon, **Christopher Schmitt**, Kai Vetter, Harold Yaver

Los Alamos National Laboratory, Los Alamos, New Mexico

Pinghan Chu, Steven Elliott, Johnny Goett, Ralph Massarczyk, Keith Rielage, Larry Rodriguez, Harry Salazar, Wenqin Xu

Oak Ridge National Laboratory

Cristian Baldenegro-Barrera, Fred Bertrand, Kathy Carney, Alfredo Galindo-Uribarri, Matthew P. Green, Monty Middlebrook, David Radford, **Elisa Romero-Romero**, Robert Varner, Brandon White, Timothy Williams, Chang-Hong Yu

Osaka University, Osaka, Japan

Hiroyasu Ejiri

Pacific Northwest National Laboratory, Richland, Washington

Isaac Arnquist, Eric Hoppe, Richard T. Kouzes, Brian LaFerriere, John Orrell

South Dakota School of Mines and Technology, Rapid City, South Dakota

Adam Caldwell, Cabot-Ann Christofferson, Stanley Howard, **Anne-Marie Suriano**, Jared Thompson

Tennessee Tech University, Cookeville, Tennessee

Mary Kidd

University of North Carolina, Chapel Hill, North Carolina and TUNL

Tom Gilliss, **Graham K. Giovanetti**, Reyco Henning, **MacMullin**, **Samuel J. Meijer**, **Benjamin Shanks**, Christopher O'Shaughnessy, **Jamin Rager**, **James Trimble**, **Kris Vorren**, John F. Wilkerson

University of South Carolina, Columbia, South Carolina

Frank Avignone, Vince Guiseppe, David Tedeschi, **Clint Wiseman**

University of South Dakota, Vermillion, South Dakota

Dana Byram, **Ben Jasinski**, Ryan Martin, **Nathan Snyder**

University of Tennessee, Knoxville, Tennessee

Yuri Efremenko

University of Washington, Seattle, Washington

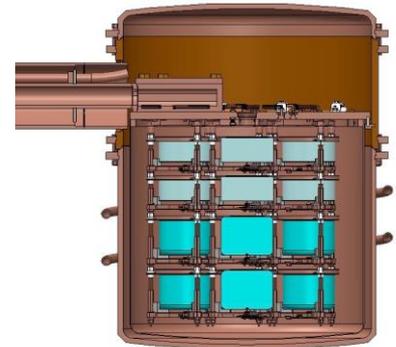
Tom Burritt, **Micah Buuck**, Clara Cuesta, Jason Detwiler, **Julieta Gruszko**, **Ian Guinn**, Greg Harper, **Jonathan Leon**, David Peterson, R. G. Hamish Robertson, Tim Van Wechel

The MAJORANA DEMONSTRATOR



Funded by DOE Office of Nuclear Physics and NSF Particle Astrophysics,
with additional contributions from international collaborators.

- Goals:**
- Demonstrate backgrounds low enough to justify building a tonne scale experiment.
 - Establish feasibility to construct & field modular arrays of Ge detectors.
 - Searches for additional physics beyond the standard model.



- **Located underground at 4850' Sanford Underground Research Facility**
- **Background Goal in the $0\nu\beta\beta$ peak region of interest (4 keV at 2039 keV)**
3 counts/ROI/t/y (after analysis cuts) **Assay U.L. currently ≤ 3.1**
scales to 1 count/ROI/t/y for a tonne experiment

- **40-kg of Ge detectors**

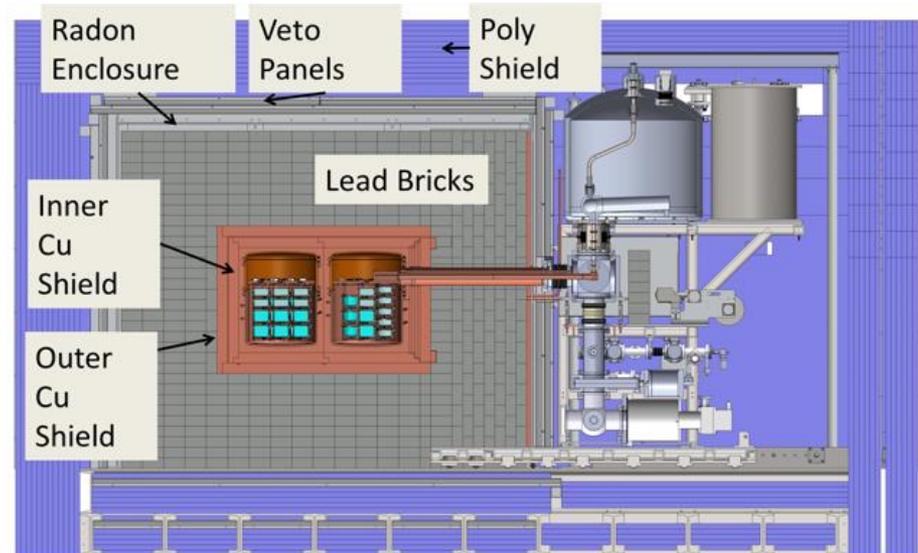
- 30 kg of 87% enriched ^{76}Ge crystals
- 10 kg of $^{\text{nat}}\text{Ge}$
- Detector Technology: P-type, point-contact.

- **2 independent cryostats**

- ultra-clean, electroformed Cu
- 20 kg of detectors per cryostat
- naturally scalable

- **Compact Shield**

- low-background passive Cu and Pb shield with active muon veto



Introduction



- Copper is the key material for MJD
 - detector mounts
 - Cryostats
 - inner shielding
- The collaboration has utilized PNNL's method of producing ultra-clean, low-activity electroformed copper
- All MJD electroformed copper machined underground at SURF

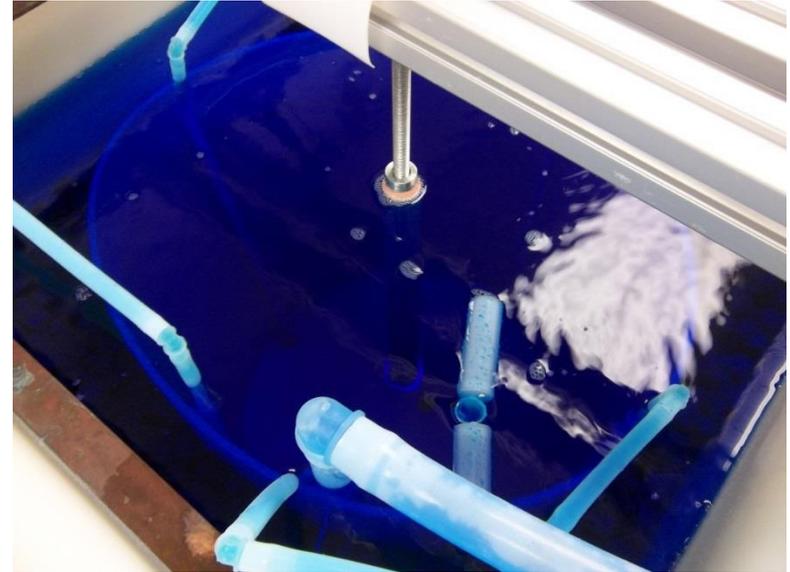


- Electroforming copper in a clean environment allows for the reduction of radioactive contaminants like U/Th while conducting the technique underground prevents cosmogenic activation forming ^{60}Co

Electroforming



- Electroplated onto 316 SS mandrels of various diameters up to 33 cm
- MJD has been operating 6 baths at the shallow UG site at PNNL and 10 at the Temporary Clean Room (TCR) facility at the 4850' of SURF
- TCR was constructed separate from main MJD laboratory (Davis Campus) at SURF allowing for 10 baths, each able to produce on the largest mandrel ~90 kg of Cu over the course of 14 months to a final thickness of 14 mm
- Average growth rate of 1 mm/month, or 0.033 mm/day



Preparation of TCR



- The exterior of the TCR was cleaned in preparation for equipment to be delivered
- This process of cleaning the drift was repeated often to reduce mine dust from entering the cleanroom area
- Before installation of flooring or any equipment the TCR was cleaned and all leaks sealed
- All surfaces were vacuumed and then scrubbed several times to remove all debris



Activities at SDSMT



- Electroforming baths were triple leached at SDSMT in a temporary cleanroom
- Modifications to the electroforming tanks and secondary containment completed before sealing/transport
- Equipment was shipped in from various vendors and DOE labs
- All equipment was triple wrapped in a cleanroom for the transition into an underground clean facility

TCR Cleanroom Preparation



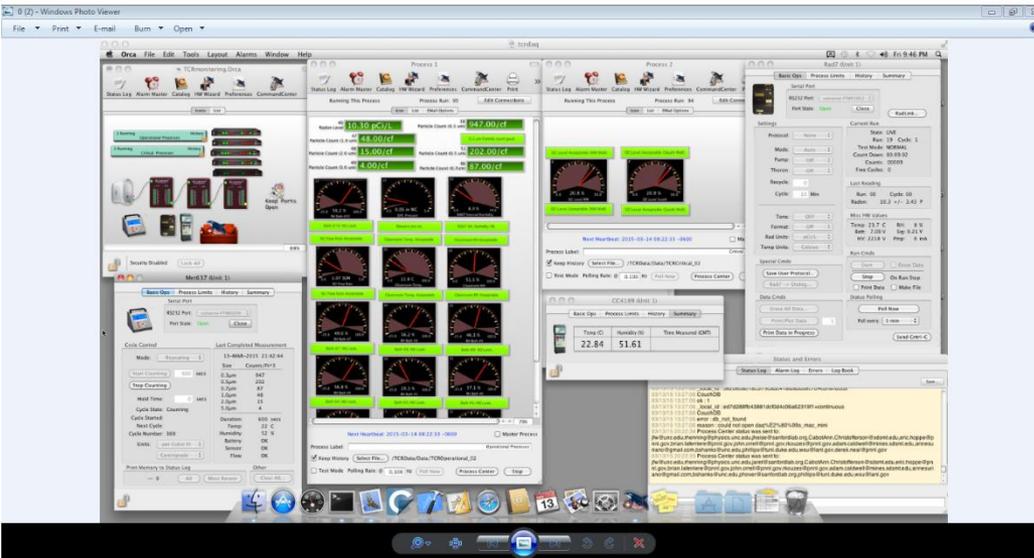
- Baths were taken to the entrance of the TCR for an assembly line process removing cover layers at each step before being placed in the cleanroom

Safety



Eyewash, spill kits, and acid cabinet across from TCR

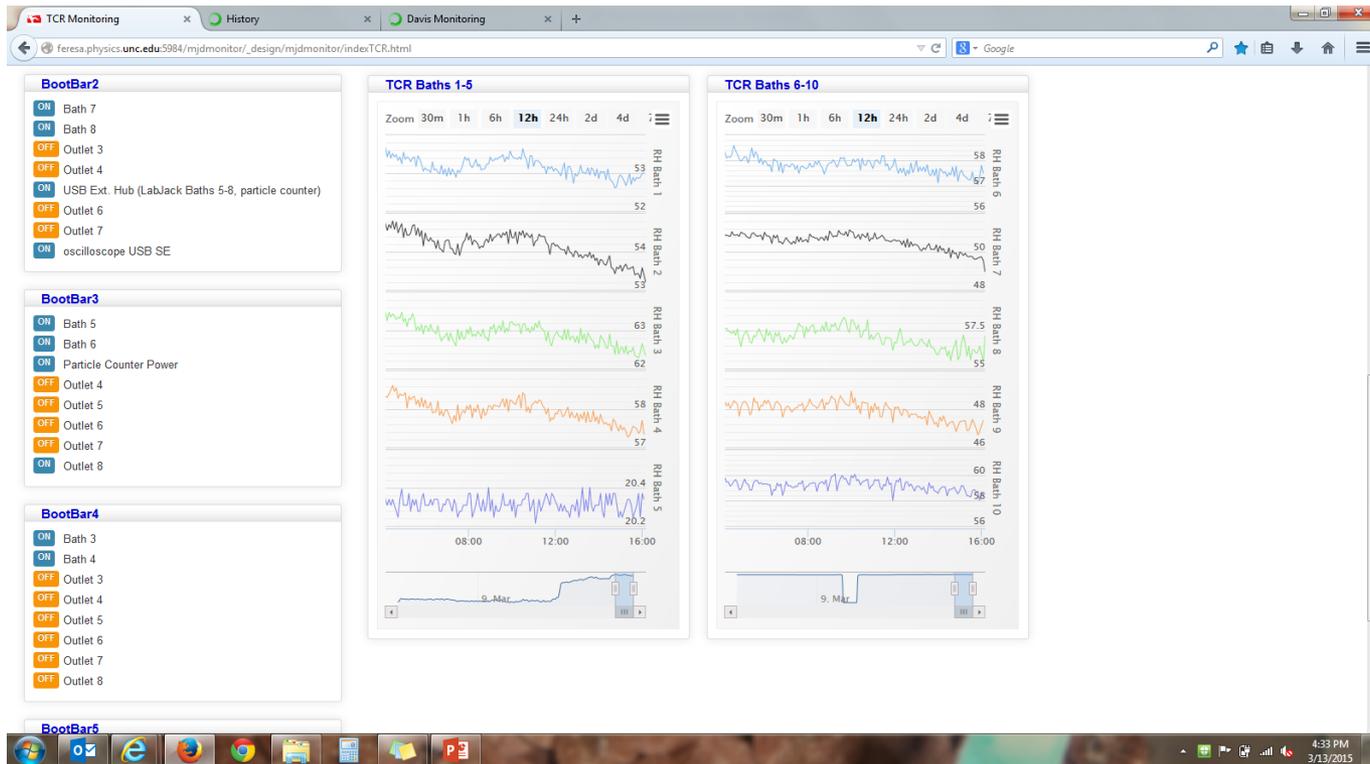
Electroforming Lab Environment



- The cleanroom is maintained as a class 1000 cleanroom using 0.3 micron HEPA filters and positive pressure
- Radon concentration is minimized with continuous airflow in the drift and cleanroom
- Air conditioners maintain temperature in the cleanroom at around 26°C
- The bath headspace is purged with LN boil off to further reduce radon and other contaminants
- All processes monitored remotely and integrated into experiment DAQ data and safety



Electroforming Lab Monitoring



- Monitoring of electroforming baths within MJD DAQ serves as a leak sensor. This allowed for automatic shut down of a bath accompanied by alarms
- Electroforming parameters kept on a separate system but still allowed for remote monitoring

Electroforming Setup



View of the partially complete cleanroom with all 10 baths in place against east wall

Electroforming Setup



- Baths reinforced with SS frames in preparation for copper loading
- OFHC anode nuggets double rinsed with DI water, ~5 M nitric acid, and DI water before being placed in baths

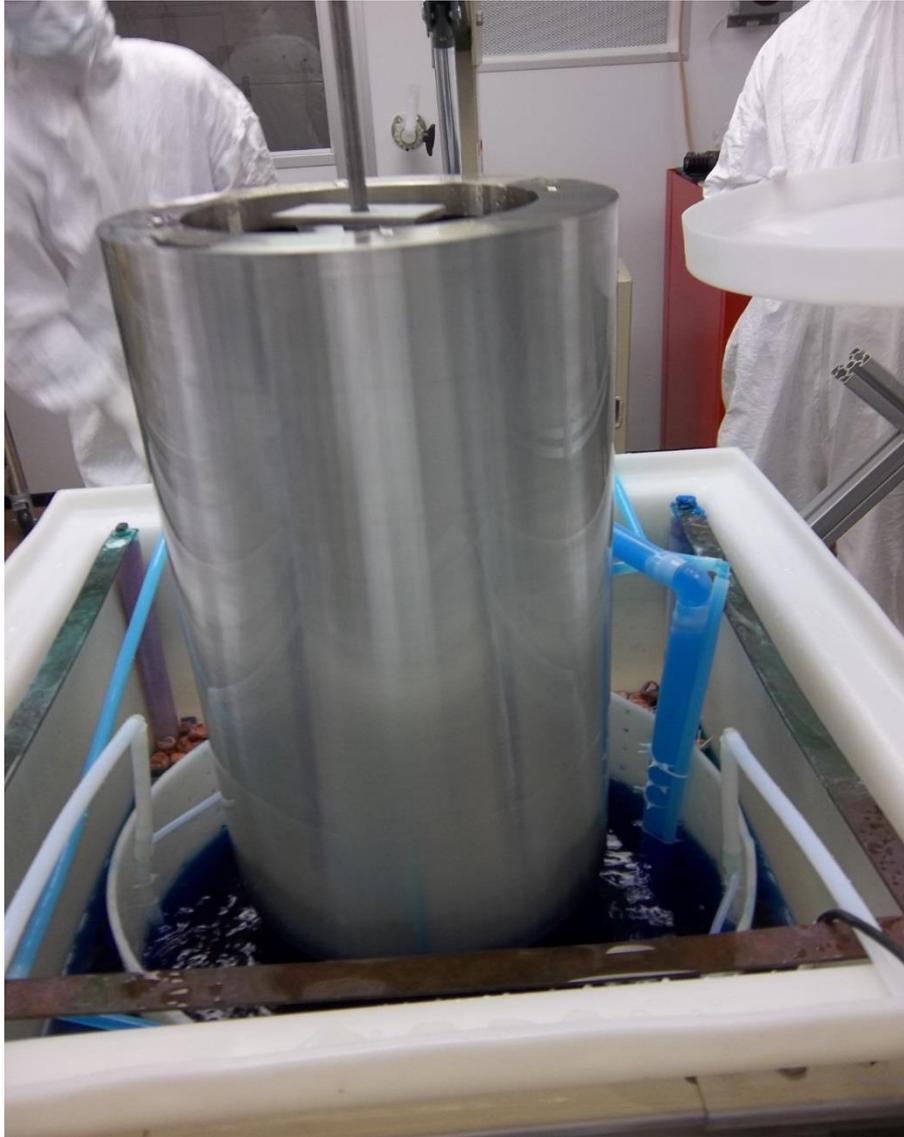
- Anode loading required several weeks to load all 10 baths with ~ 800 kg each
- Copper allowed to generate electrolyte solution over ~1 wk before plating could begin

Electroforming Setup



Electroforming room showing all equipment in place and operational

Mandrel Preparation

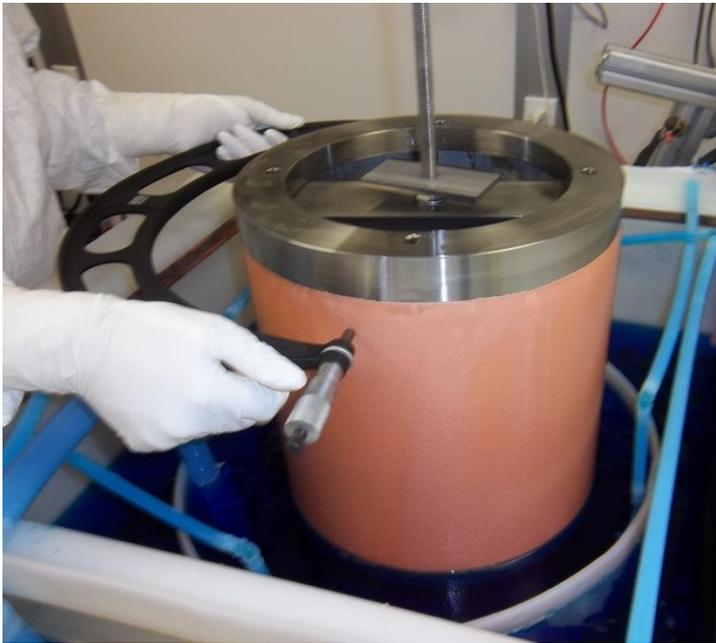
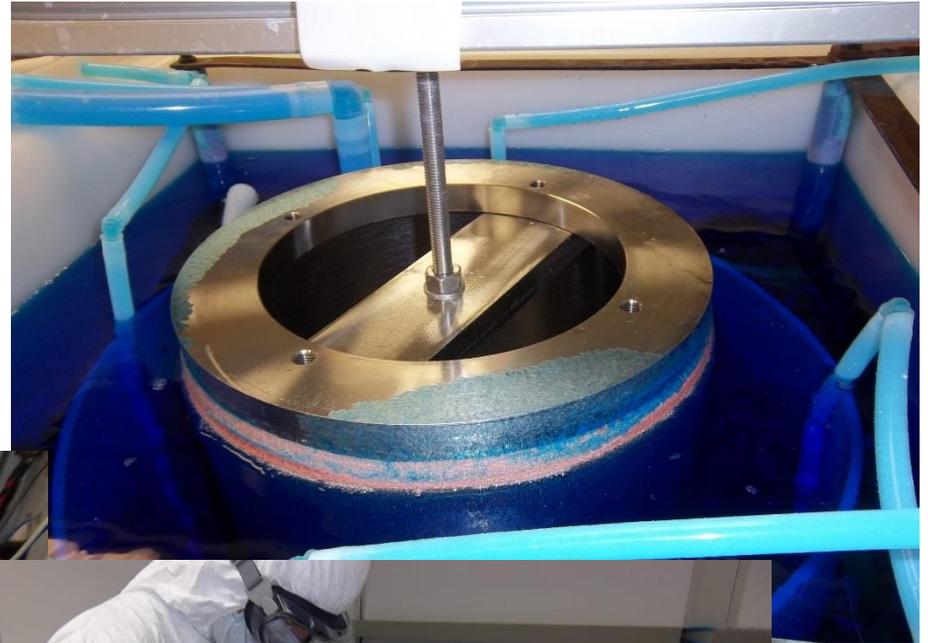


- Mandrels ranged in diameter from 1.9 cm to 33 cm, length 40 cm to 91 cm
- Once any mandrel has gone through the normal stages of cleaning to be brought into the cleanroom two more steps are taken before it is clean enough to plate copper
- Micro 90 detergent is used to clean the inside and exterior surface
- Optima grade nitric acid is used to finish the preparation process by etching and passivating the surface so it can be placed into the bath for growth

Maintenance



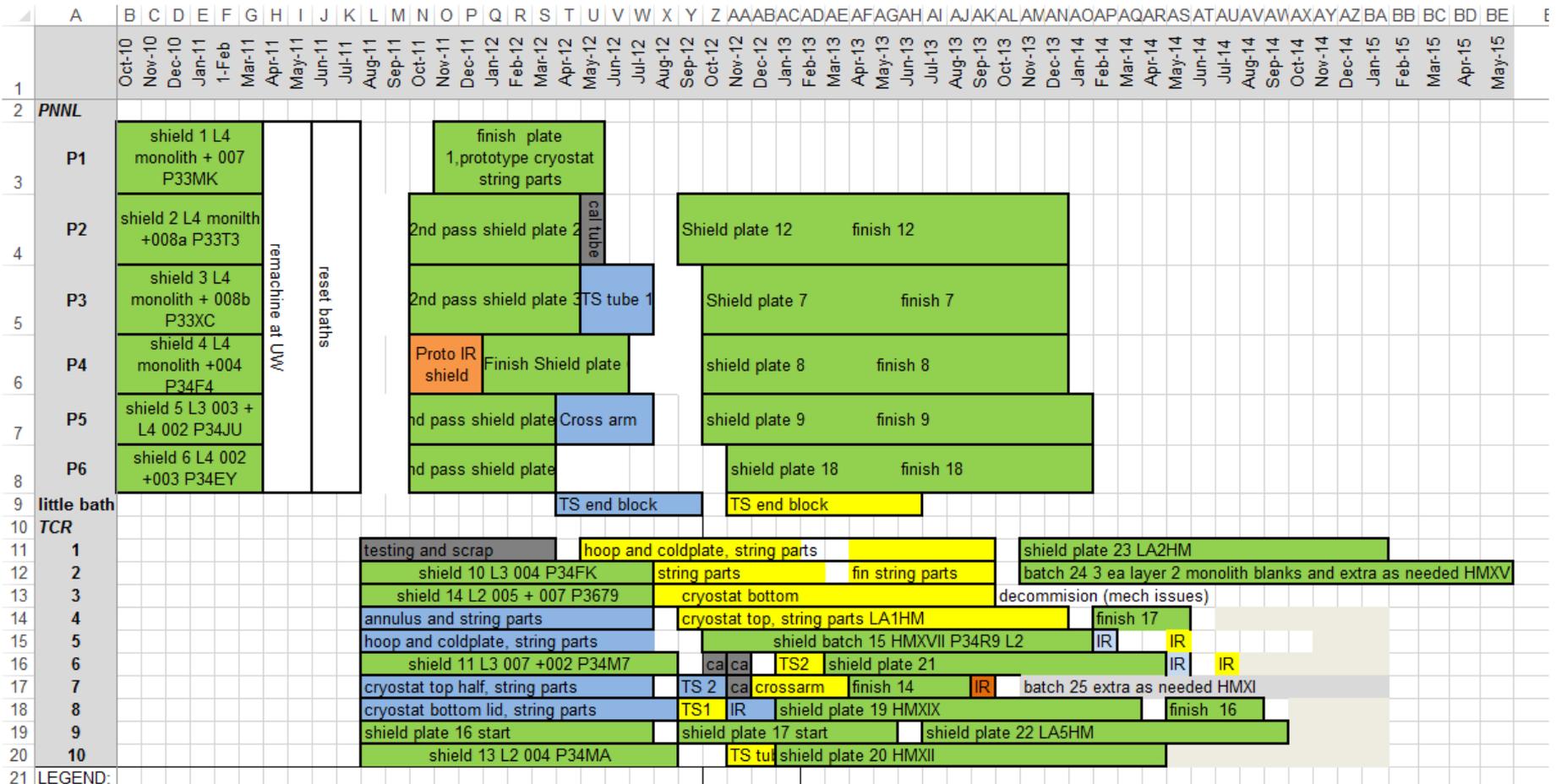
- Electroforming mandrels are rinsed and monitored for correct growth along with anode nugget additions
- General housecleaning is done on a daily basis to reduce particle count
- Remote monitoring equipment is checked to ensure proper operation



Electroforming Production



| Component group | percent eformed | kg eformed to date | kg eformed to produce final parts | kg final installed |
|----------------------------|-----------------|--------------------|-----------------------------------|--------------------|
| inner copper shield plates | 99% | 2300 | 2090 | 1100 |
| Cryo 1 copper | 100% | 382 | 382 | 96.5 |
| Cryo 2 copper | 100% | 382 | 382 | 96.5 |



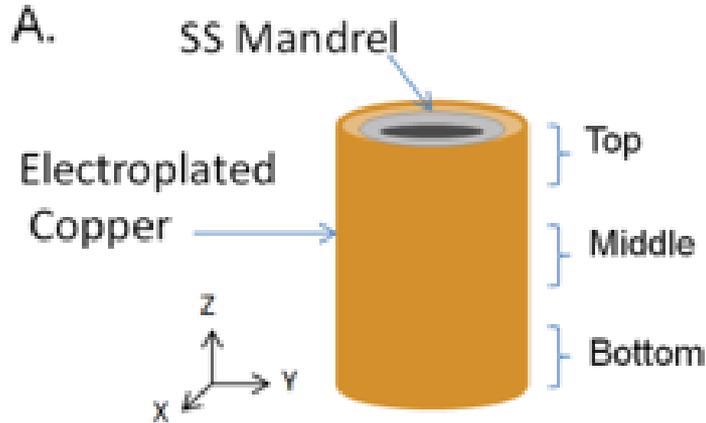
Electroformed Parts



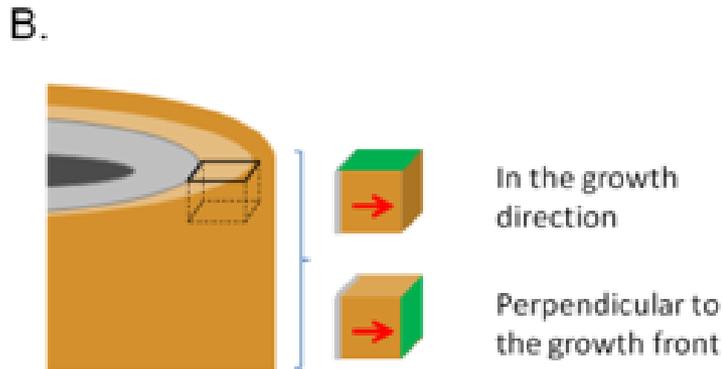
- All pieces machined underground in clean machine shop
- Assigned and labeled with unique database identifier
- Stored in N₂ purge, etched and passivated in cleanroom wet lab



Material Properties



- Each electroformed mandrel sampled in regions of Top, Middle, and Bottom (A) along with both plane orientations (B)
- Samples used in the following tests to evaluate mechanical response of Cu
 - ✓ Tensile testing
 - ✓ Optical metallography/SEM
 - ✓ Vickers hardness

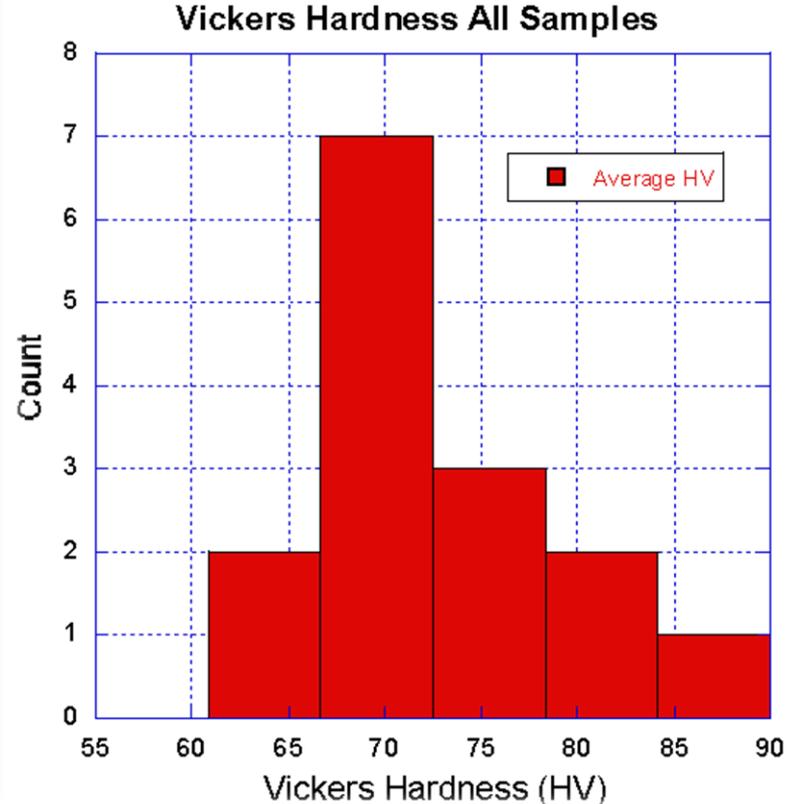
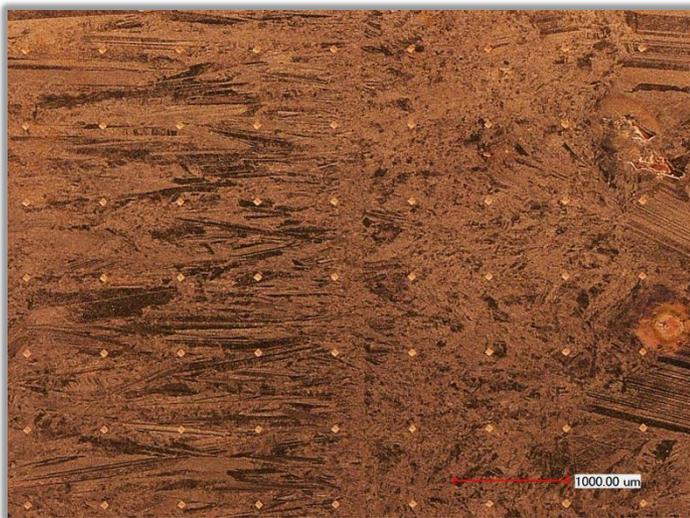


- Results show consistent mechanical response from copper grown at both TCR and PNNL facilities.
- All electroformed material exceeded the 10 ksi yield strength design specifications of MJM.

Material Properties



- A histogram of hardness results was generated to understand the degree of mandrel variation
- Samples from all the mandrels show a normal distribution in average hardness values
- Optical metallography and SEM showed polycrystalline structure with some small voids

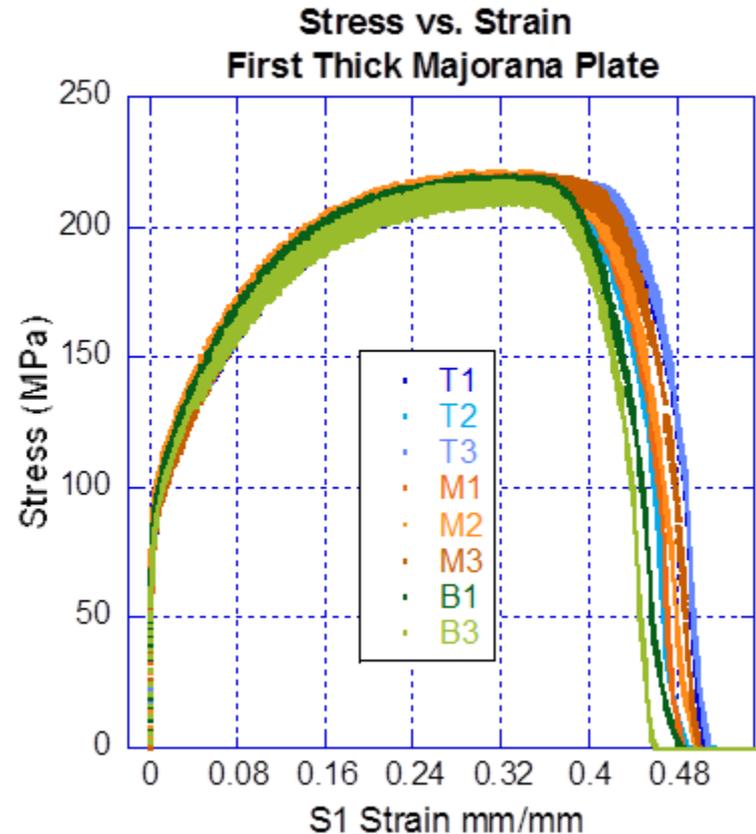


- Voids do not appear to have a significant effect on hardness results or density

Material Properties



- Results of the tensile tests were plotted to the ultimate tensile strength (UTS - maximum stress)
- Specimens with voids and interfaces generated more noise in the tensile data and resulted in reduced UTS in some cases
- Voids & interfaces did not impact the yield strength
- Both PNNL and TCR samples show a uniform material response
- Results to this point have indicated electroformed copper is capable of withstanding the engineering design requirement of 10 ksi (14 ksi average)



Assay Properties



- Assay of samples from all materials used in the DEMONSTRATOR.
 - Radiometric, NAA, & ICP-MS techniques.
- By necessity have developed world's most sensitive ICP-MS based assay techniques at PNNL for U and Th in Cu

(Original MJD Goal : <0.3 $\mu\text{Bq/kg}$ for U and Th)

- Alternate MDL (method detection limits) with iridium anode improvements (presented at MARC X by E. W. Hoppe)
 - U decay chain 0.1 $\mu\text{Bq } ^{238}\text{U/kg}$
 - Th decay chain 0.1 $\mu\text{Bq } ^{232}\text{Th/kg}$
- Sensitivities with current MDL for ion exchange copper sample preparation
 - U decay chain <0.131 $\mu\text{Bq } ^{238}\text{U/kg}$
 - Th decay chain <0.034 $\mu\text{Bq } ^{232}\text{Th/kg}$

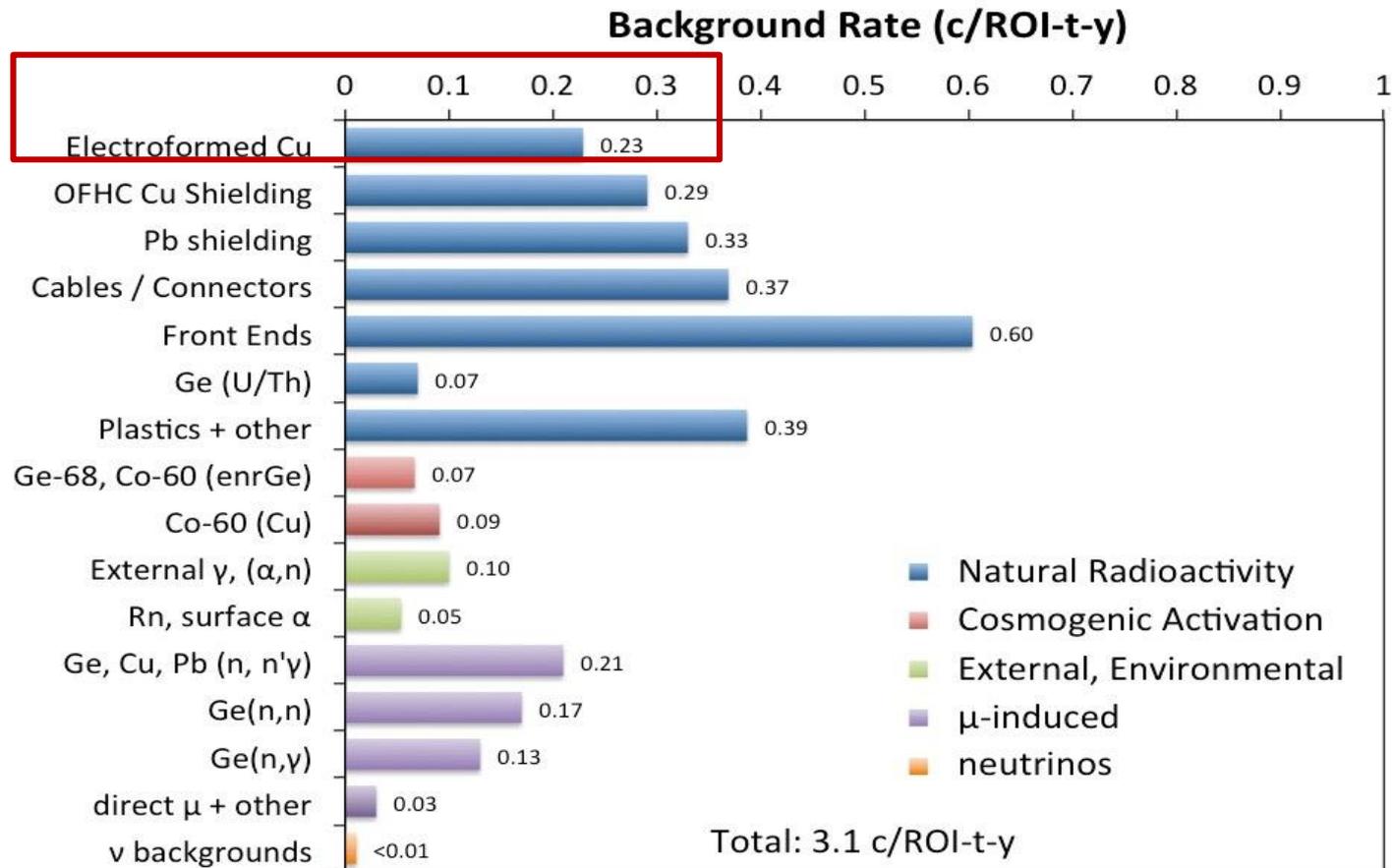
| Reportable Values | | | | | |
|-------------------|----------------|---------------------------------|--------|--------------------------------|--------|
| | | Th-232 ($\mu\text{Bq/kg Cu}$) | | U-238 ($\mu\text{Bq/kg Cu}$) | |
| Sample Name | Stock Material | Measured Value | +/- 1s | Measured Value | +/- 1s |
| P34MQ | HMXIX Run 1 | <0.118 | | 0.120 | 0.040 |
| P36CD | HMXIII Run 1 | <0.118 | | <0.104 | |
| P36CG | HMXIII Run 1 | <0.117 | | <0.104 | |
| P34N9 | HMXIX Run 1 | <0.118 | | <0.104 | |
| P3CPH-1 | LA1HM Run 2 | <0.113 | | <0.100 | |
| P3CPH-2 | LA1HM Run 2 | <0.114 | | <0.101 | |

Table Courtesy Eric Hoppe, PNNL

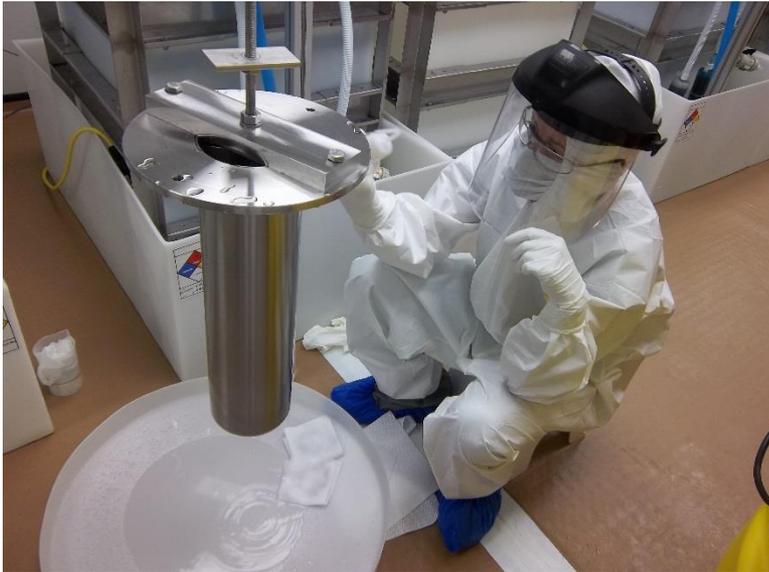
Assay Properties



- Contribution of U and Th in MJD electroformed Cu
 - Th decay chain $0.06 \pm 0.02 \mu\text{Bq/kg}$ (0.15 counts in ROI)
 - U decay chain $0.17 \pm 0.03 \mu\text{Bq/kg}$ (0.08 counts in ROI)

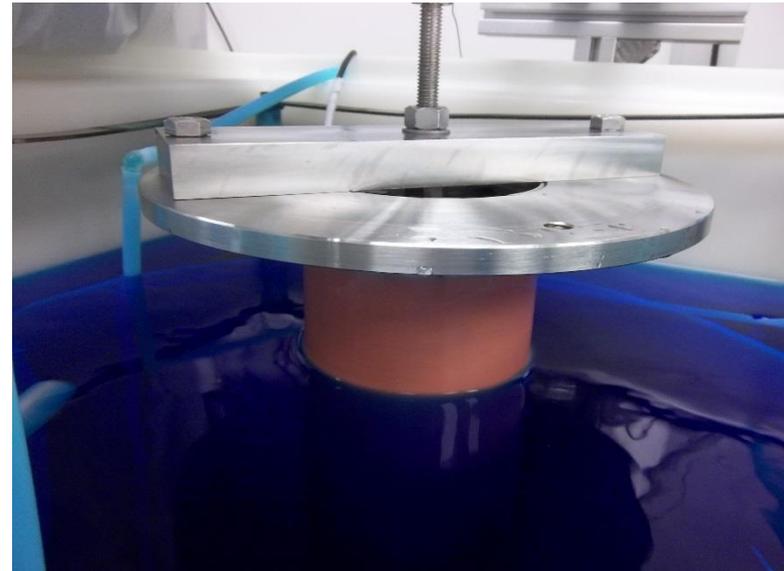


Future Plans



- Ongoing research involving electroforming alloy development of Cu at PNNL with SDSMT MES PhD. candidate Anne-Marie Suriano under a DOE Office of Science Graduate Student Research award
- Electroforming for 1-tonne $0\nu\beta\beta$ or other low background experiments

- Electroforming of small cryostat for a new ultra-low background gamma-assay detector at KURF for Dr. Reyco Henning, UNC



Electroforming Setup (Backup)



- RO Water system was attached to the fire suppression line which will produce the stream that is fed to the cleanroom
- Once inside the cleanroom the water runs through a DI system to produce the quality of water needed for the electroforming process

Electroforming Setup (Backup)

