

# Copper Electroforming at the Sanford Underground Research Facility

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South Dakota School of Mines and Technology MAJORANA DEMONSTRATOR Collaboration





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LRT, Seattle, WA

# 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 0vββ 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 <sup>76</sup>Ge crystals
- –10 kg of <sup>nat</sup>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, lowactivity 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 <sup>60</sup>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**





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- 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**



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- 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**

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Mar. 19, 2015

#### **Electroformed Parts**



- All pieces machined underground in clean machine shop
- Assigned and labeled with unique database identifier
- Stored in N<sub>2</sub> purge, etched and passivated in cleanroom wet lab



## **Material Properties**





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In the growth direction

Perpendicular to the growth front

- 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 MJD.



### **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  $\mu$ 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 µBq <sup>238</sup>U/kg
  - Th decay chain 0.1 µBq <sup>232</sup>Th/kg
- Sensitivities with current MDL for ion exchange copper sample preparation
  - U decay chain <0.131 µBq <sup>238</sup>U/kg
  - Th decay chain <0.034 µBq <sup>232</sup>Th/kg

Reportable Values														
		Th-232 (µBq/k	kg Cu)											
		Measured	+/-	Measured										
Sample Name	Stock Material	Value	1s	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



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



#### Background Rate (c/ROI-t-y)

#### **Future Plans**





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

- 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 0vββ or other low background experiments



# 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)**



