



# Ultra-pure Nickel for Structural Components of Low-Radioactivity Instruments

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On behalf of the nEXO Collaboration



PNNL is operated by Battelle for the U.S. Department of Energy

## Motivation

- Candidate material for inner and outer vessels of the cryostats for nEXO.
  - Possible replacement carbon fiber/resin.
  - For components that require strength and high degree of radiopurity.
- Broadly of interest as a low background material.
  - For potential use in future  $0\nu\beta\beta$  and dark matter experiments.

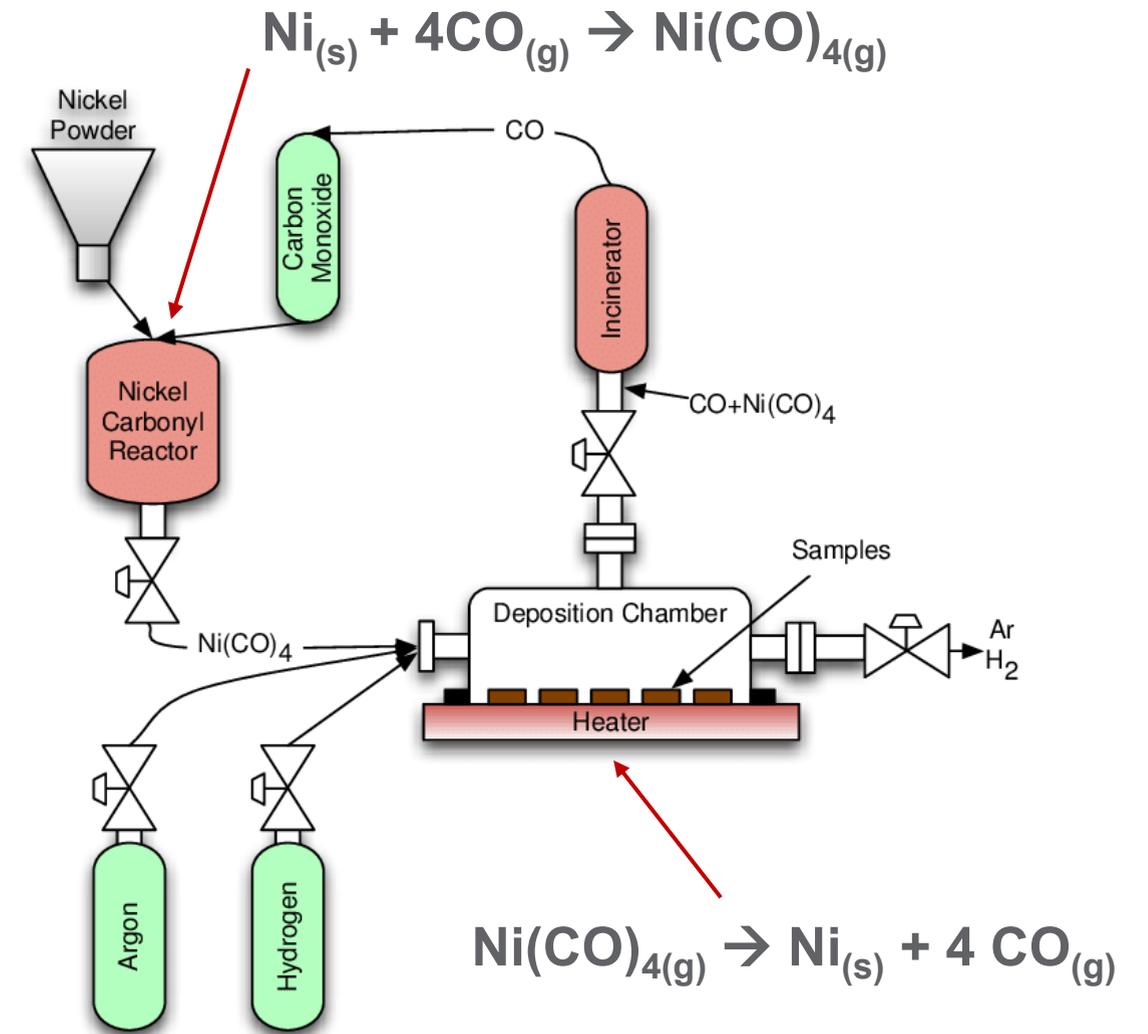
# CVD Nickel Production

- **Chemical Vapor Deposition**

- Solids deposited from vapor by chemical reaction at or near heated substrate.
- Under pressure @ 50°C, Ni powder reacts with CO to form Ni carbonyl.
- At 200°C, Ni carbonyl decomposes back to Ni and CO.

- **Production Details**

- Growth rate: 0.75 mm/hr
- Max thickness: >5 cm
- Generally deposited on Al



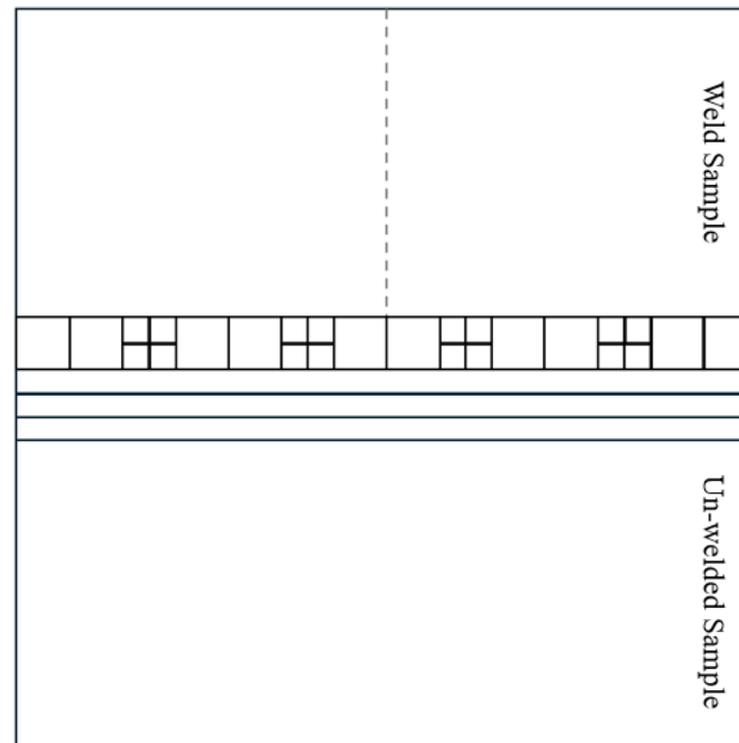
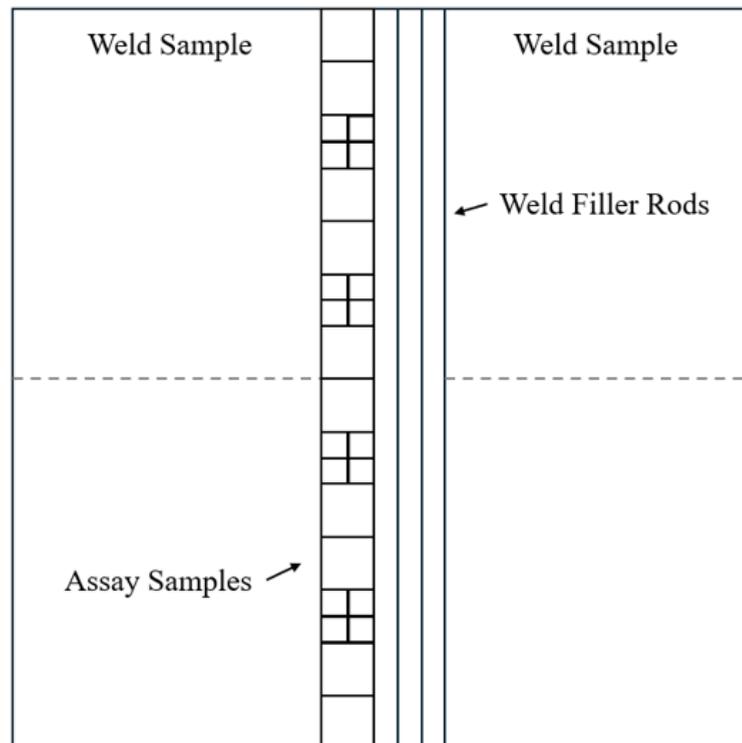
Generalized schematic of Mond, or carbonyl, process used by CVMR (Muralidharan et al., 2011)

# Goals

1. Evaluate Mechanical Properties – Tensile Strength and Welding Tests
  - Determine raw strength of CVD Ni
  - Assess a ‘joining method’ (TIG welding)
  
2. Evaluate Material Radiopurity – Material Assay
  - Determine bulk level of impurities [U, Th, and K]
  - Assess surface contamination

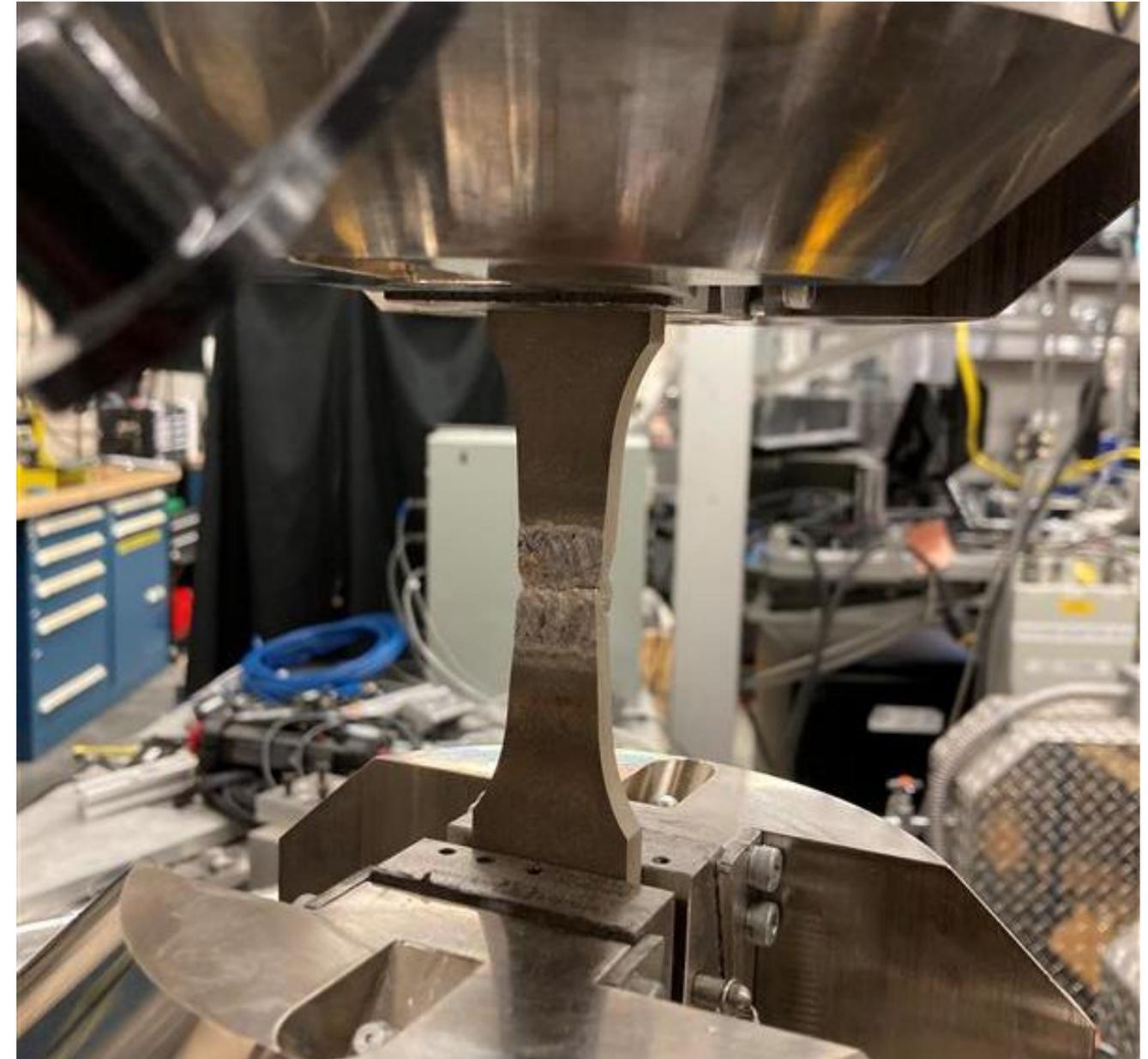
# Samples

- Produced by CVMR (Toronto, Canada)
- Nominally 155 mm square
- 6.3-7.3 mm thick
- Deposited on Al



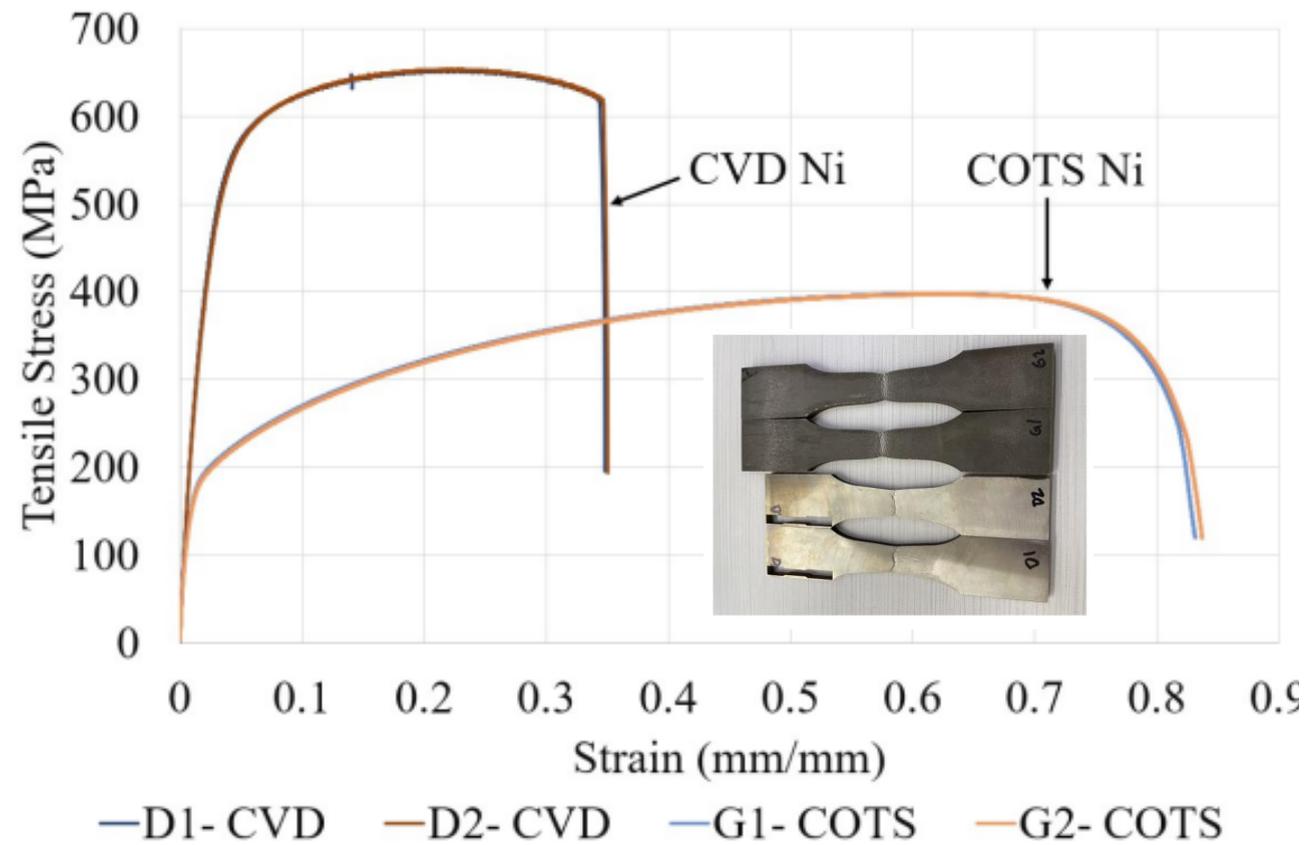
# Mechanical Testing

- PNNL Mechanical Testing Lab
- 8 CVD Nickel Samples
  - 6 Welded
  - 2 Unwelded
- 6 COTS Nickel Samples
  - 4 Welded
  - 2 Unwelded

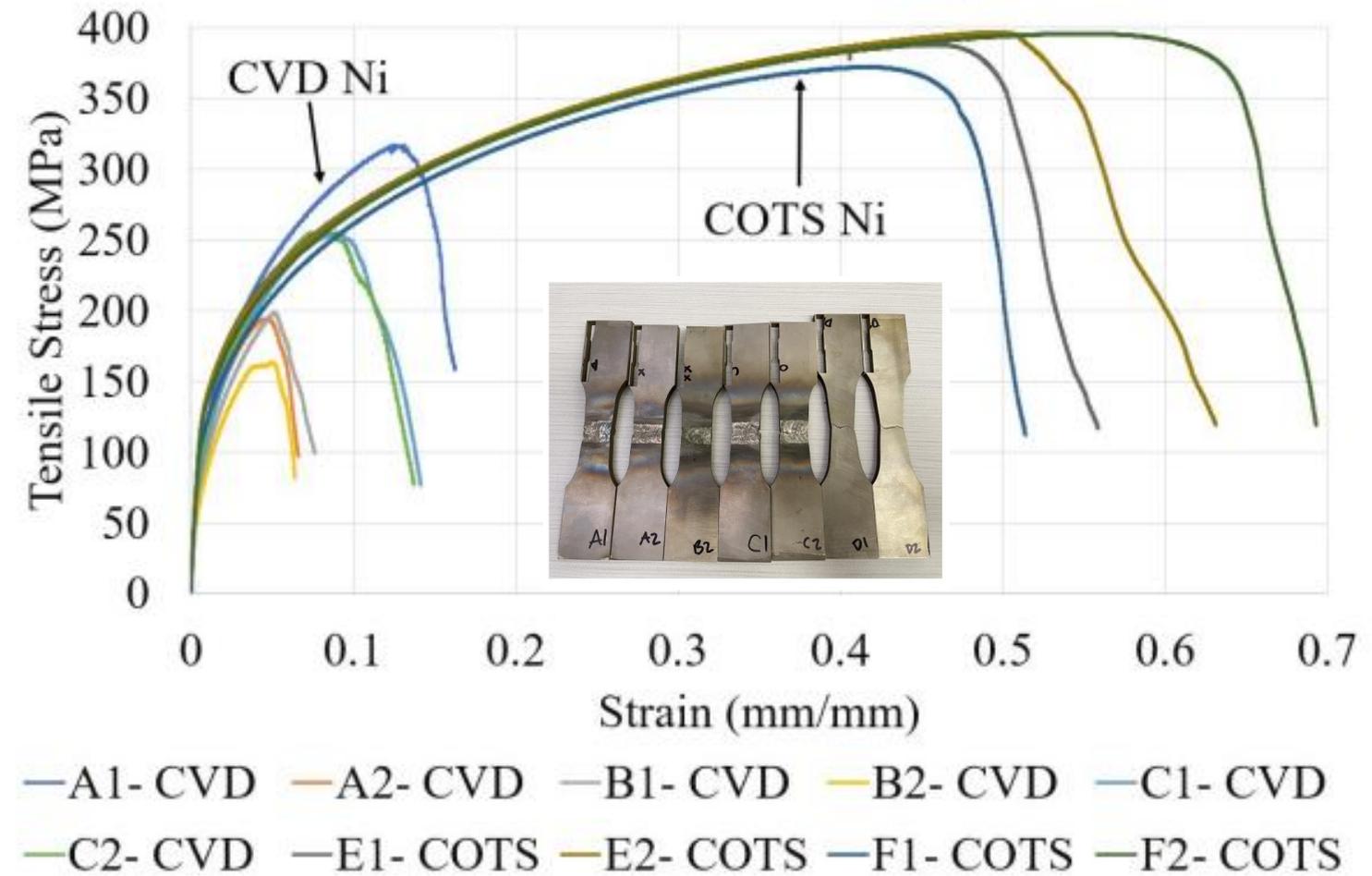


# Tensile Strength Testing

Tensile Test of CVD and COTS Nickel

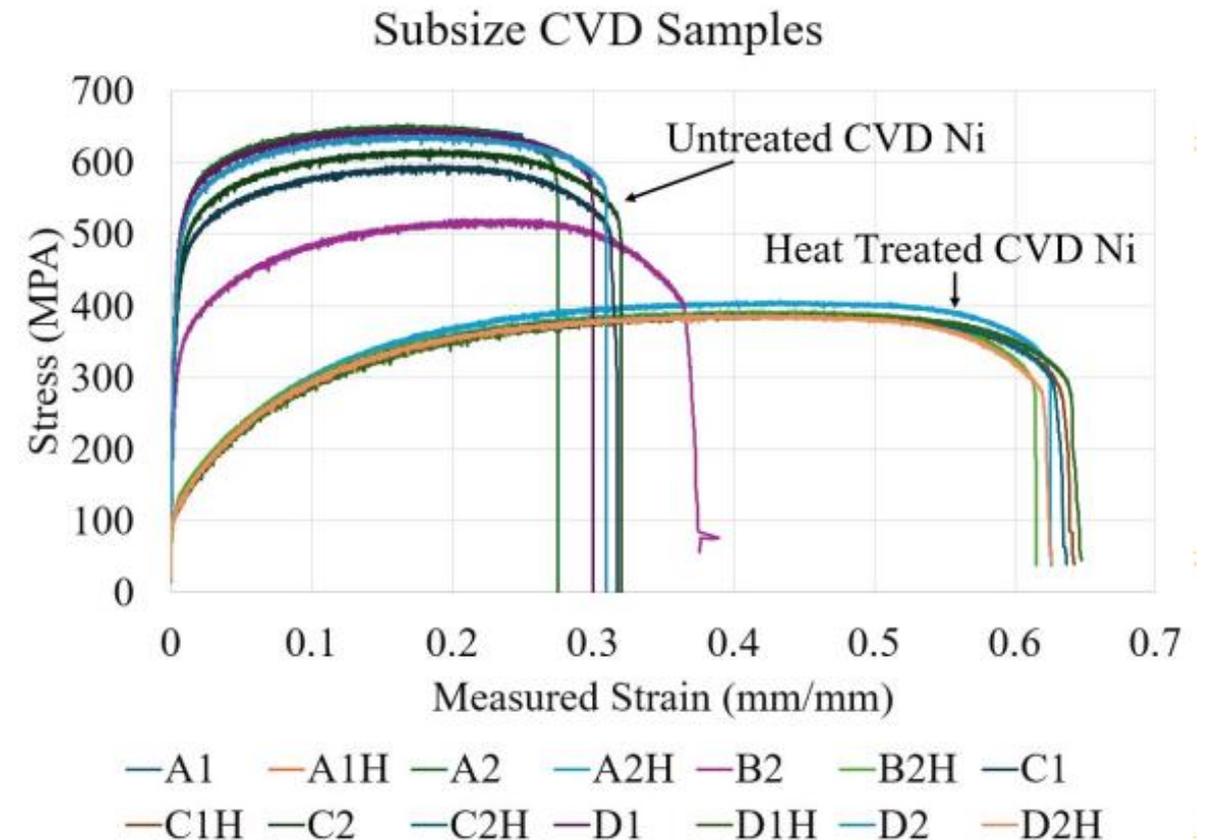
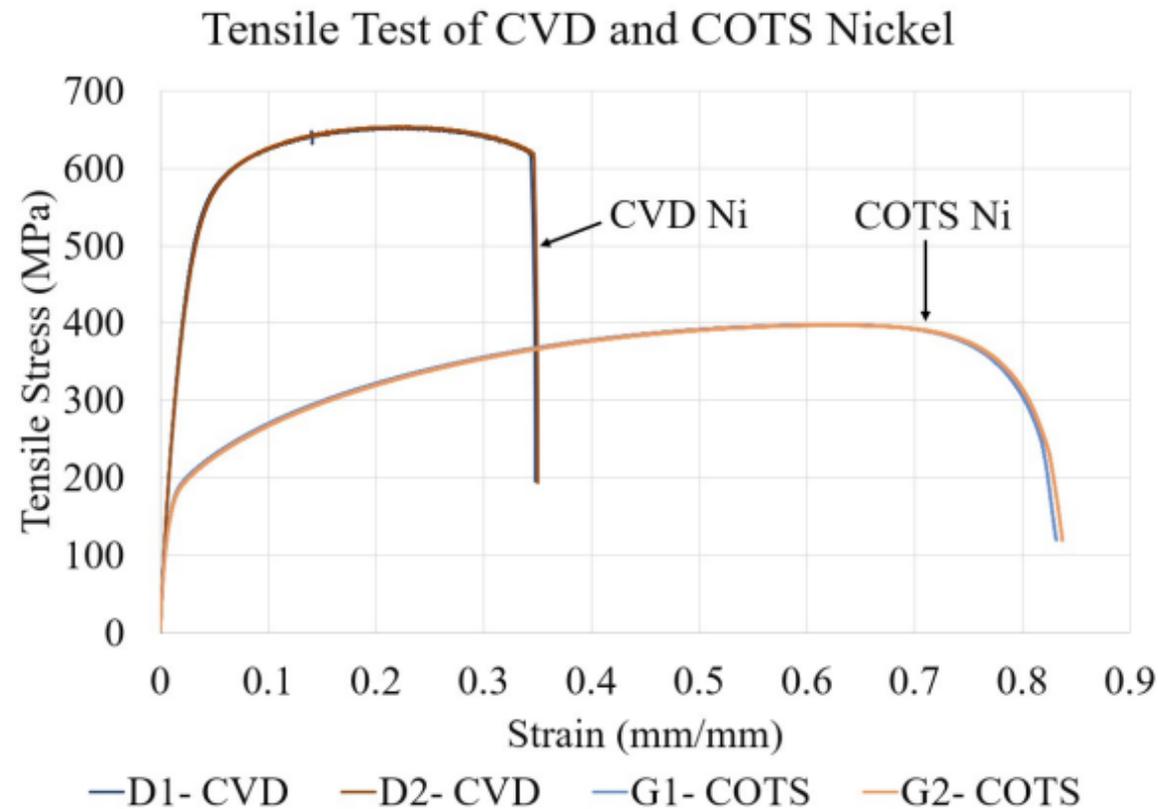


Tensile Test of Welded Samples



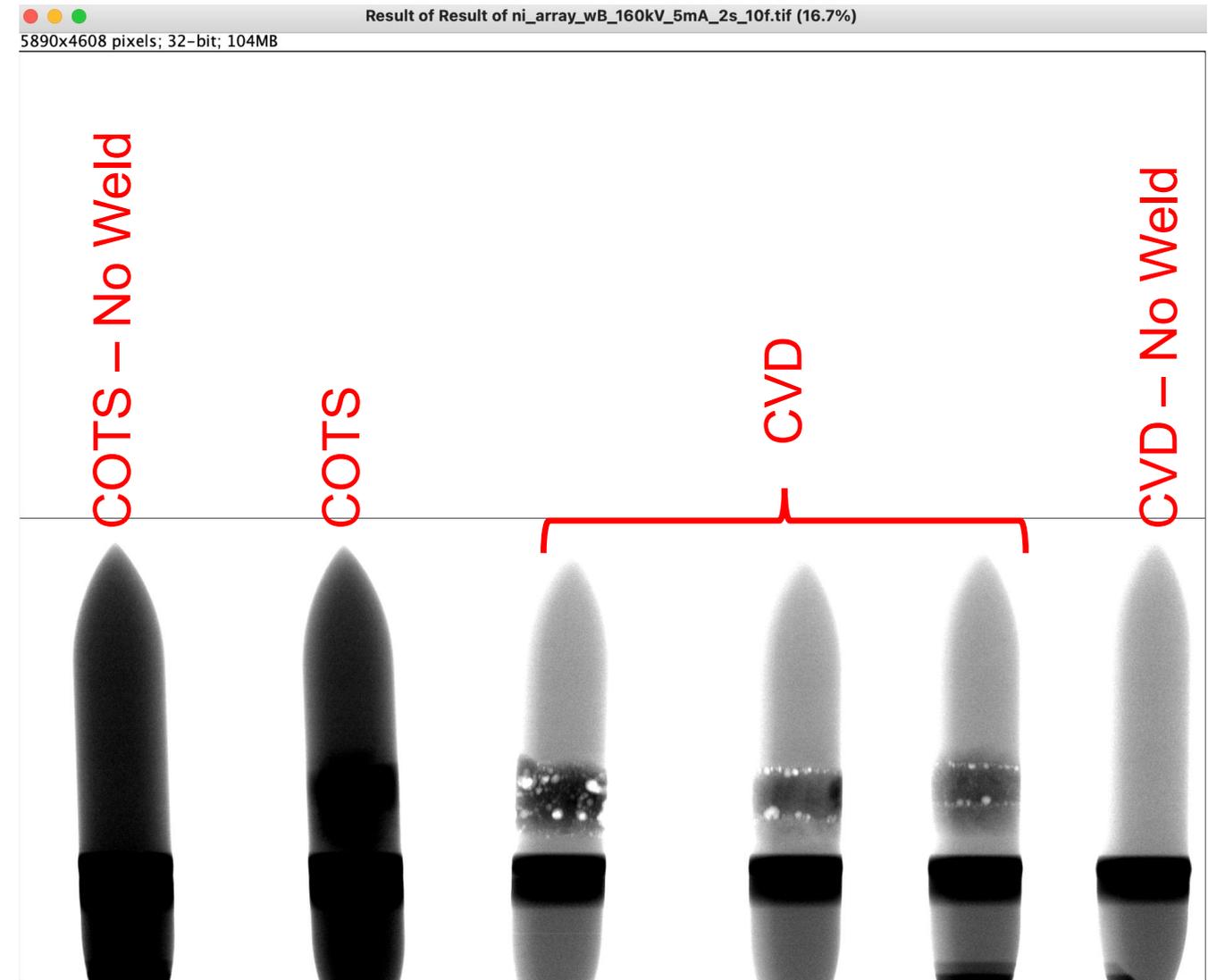
# Thermal Annealing of CVD Ni

- Heat treat in Argon atmosphere up to 1000° C to simulate welding conditions
  - 1000°C is within annealing range for standard Ni
- Allowed to cool naturally as with welds



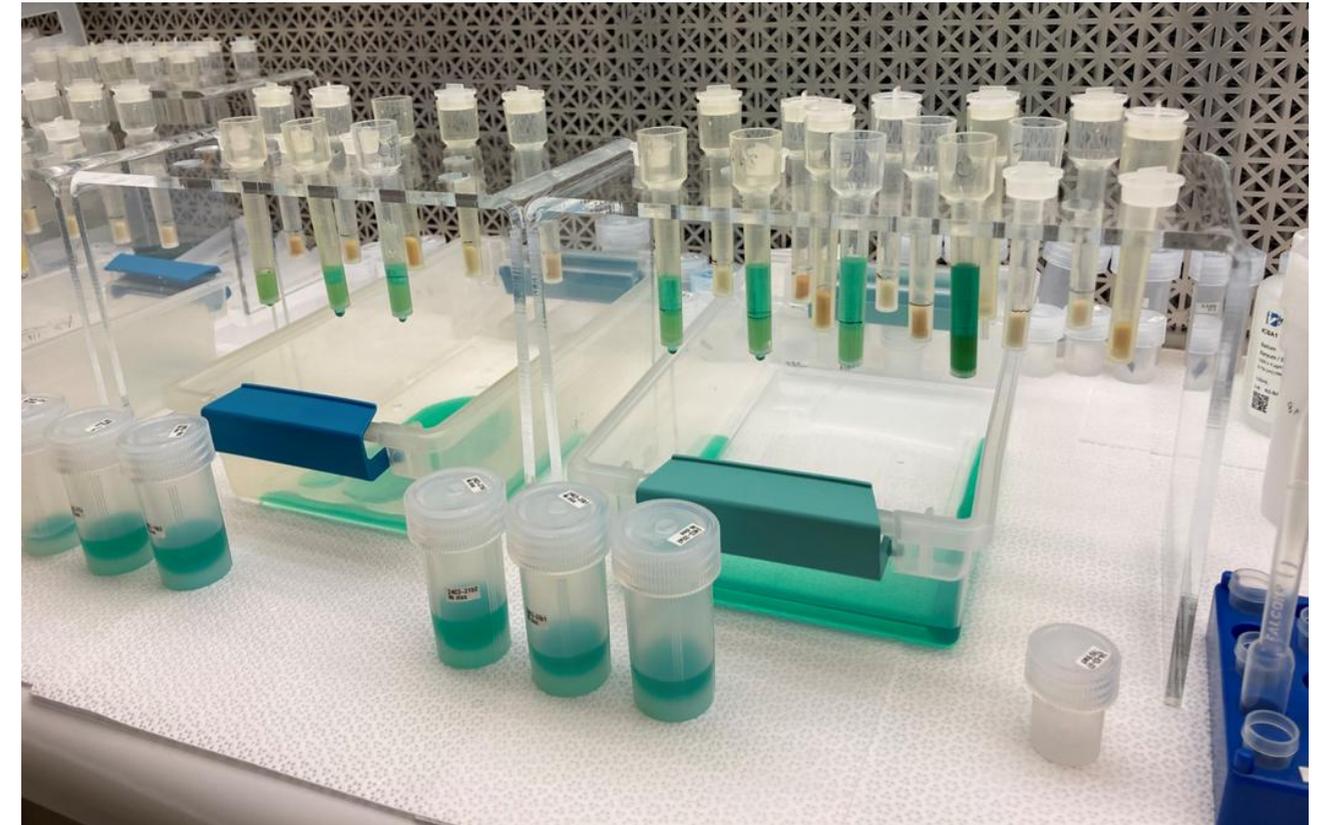
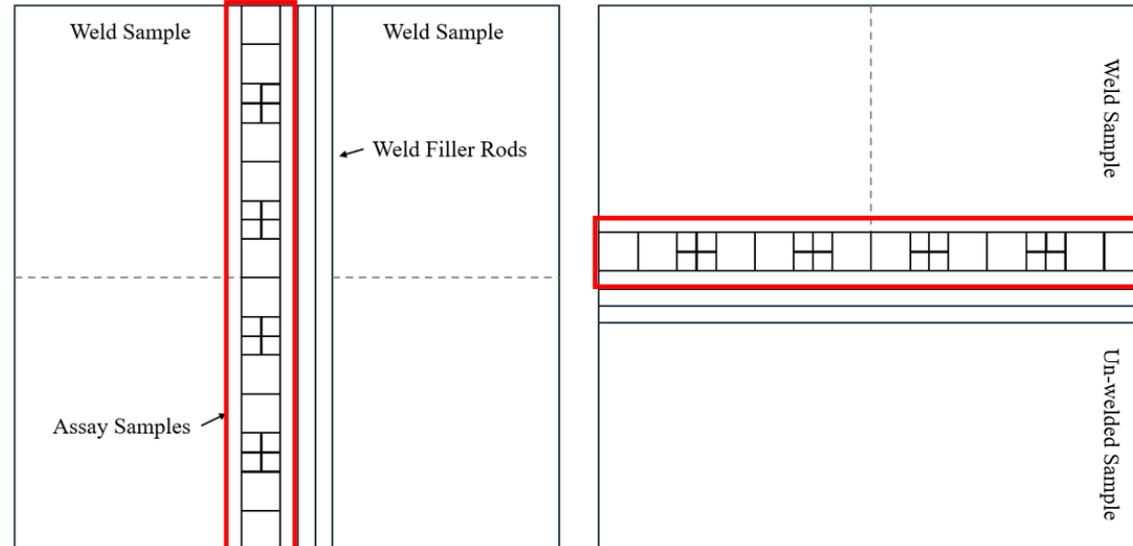
# Summary of Mechanic Testing

- Raw CVD Ni exhibits higher strength than COTS Ni.
- Welded CVD Ni displays comparable strength to COTS Ni.
  - Due to thermal annealing.
- Welding techniques need improvement.

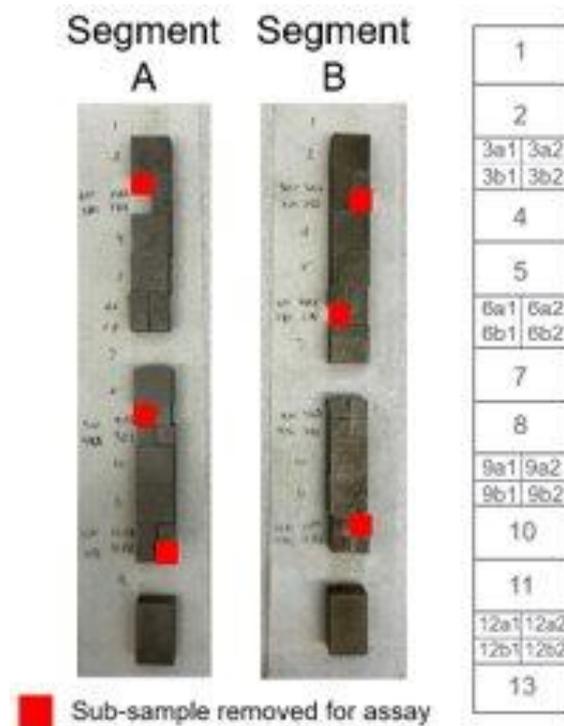


[ArXiv:2508.08230](https://arxiv.org/abs/2508.08230)

# Initial Bulk Assay



*Ni separation via ion chromatography.*



1. Samples cleaned and etched to remove >30% of total mass
2. Dissolution in 4M HNO<sub>3</sub>
3. Matrix removal and analyte preconcentration
4. Analysis by ICP-MS

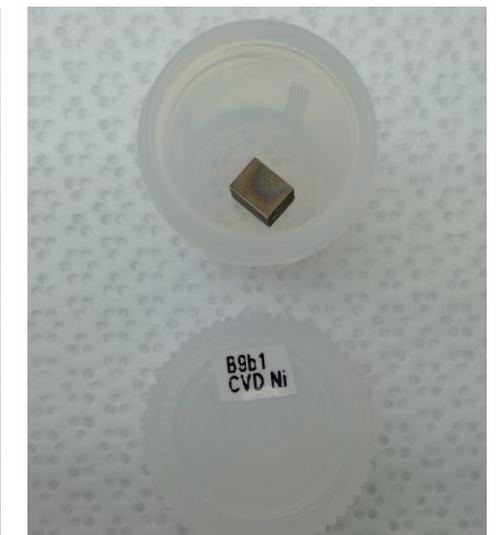
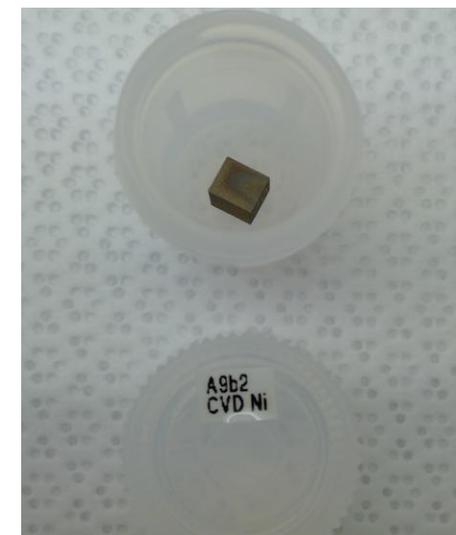
# Bulk Assay Results

- Results:  $\sim 1000$  pg/g  $^{nat}\text{K}$ ,  $<100$  fg/g  $^{232}\text{Th}$  and  $^{238}\text{U}$

Ni segment	sample mass [g]	$^{nat}\text{K}$		$^{232}\text{Th}$		$^{238}\text{U}$	
		ppt	$\pm (1\sigma)$	ppq	$\pm (1\sigma)$	ppq	$\pm (1\sigma)$
A	0.8270	820	70	<19	--	<98	--
A	0.7930	980	70	23	6	<71	--
A	0.8673	1000	100	90	6	<64	--
B	0.8540	1000	200	88	7	<65	--
B	0.8250	850	100	48	4	<68	--
B	0.7659	800	100	74	7	<73	--

# Radiocontamination distribution

- Goal: understand distribution of radio-contaminants in CVD Ni
  - How much material needs to be removed to reach low-levels of bulk Ni?
- Approach: serial etch Ni
  - Measure contamination ( $^{232}\text{Th}$ ,  $^{238}\text{U}$ ,  $^{\text{nat}}\text{K}$ , Al) in etchant
  - Construct depth profile of impurities into Ni
  - Assay remaining bulk Ni for comparison to bulk assay results

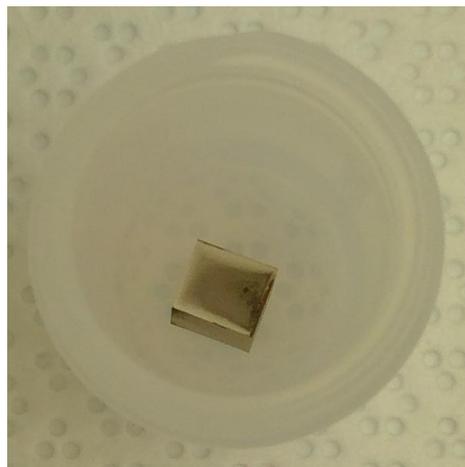


*Ni samples during etching process.*

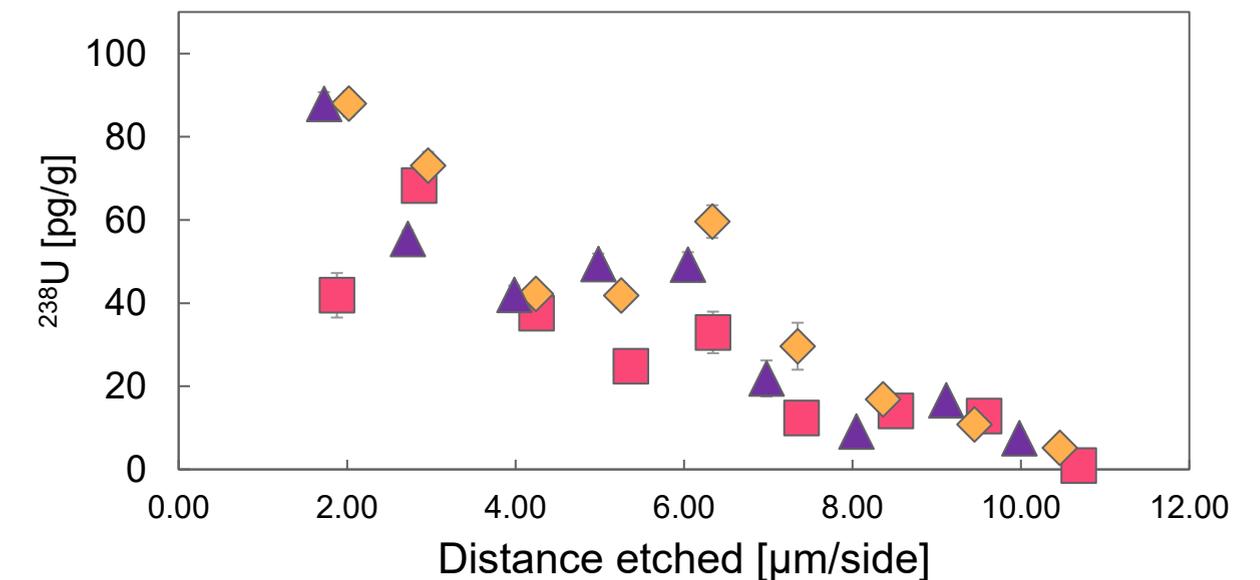
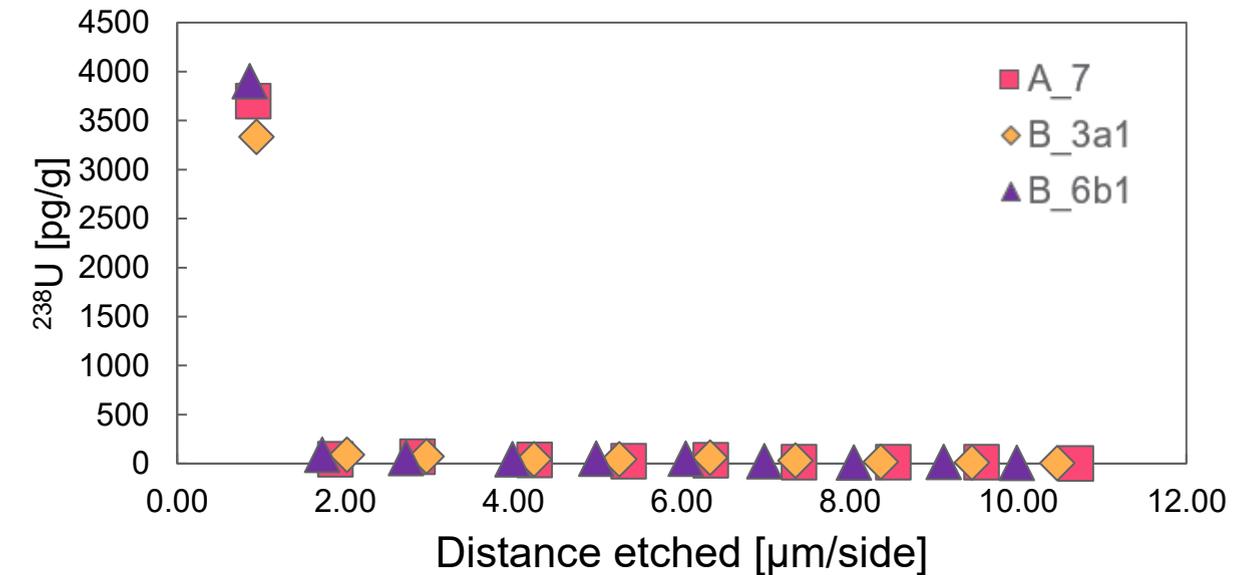
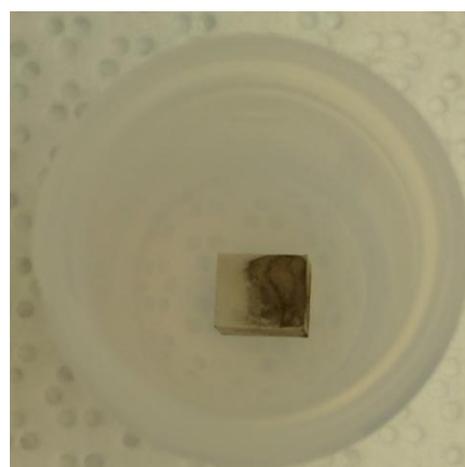
# Surface Etching

- Ten stoichiometric etches with 4M HNO<sub>3</sub>
  - Removed 1 μm Ni/side [mass equivalent]
- 100x reduction after first 1μm equivalent etched
  - Progressive >5x reduction from etch 2-10
- Do not reach bulk cleanliness
  - Likely from non-uniform removal of outermost oxidation layer – outermost oxide layer still present after 10 etching cycles
  - Can be tested in future (e.g., thermal pretreatment)

A7

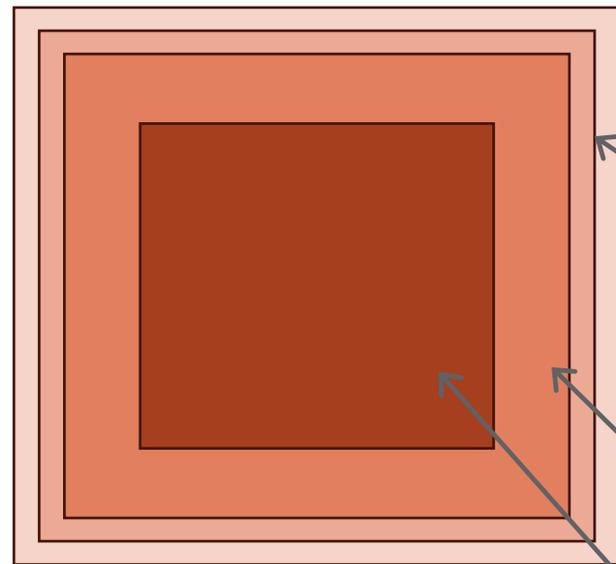


B\_3a1



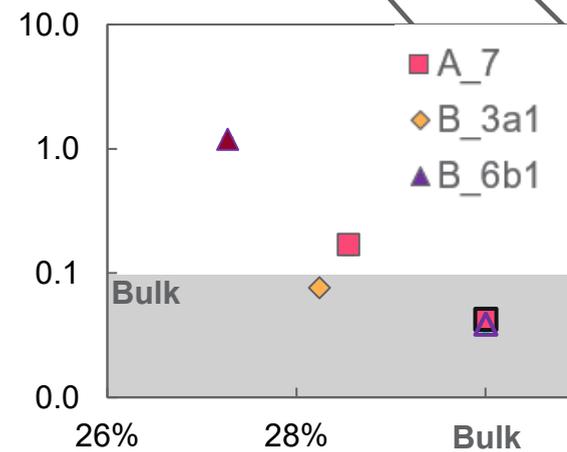
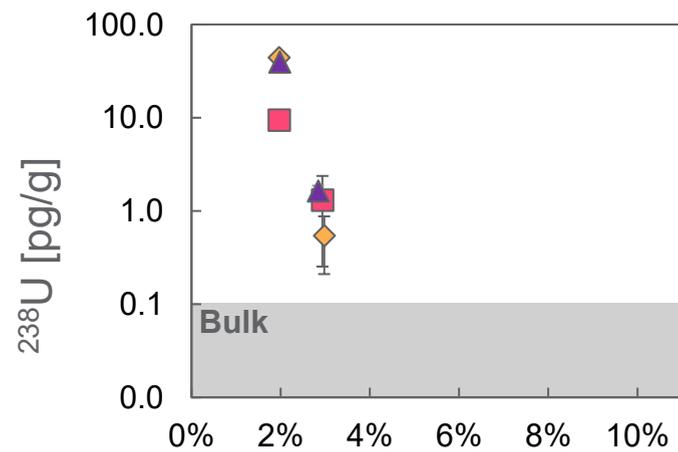
# Remaining Ni etch and bulk assay

(Sample after ten 1  $\mu\text{m}$  stoichiometric etches – or ~1% by mass)



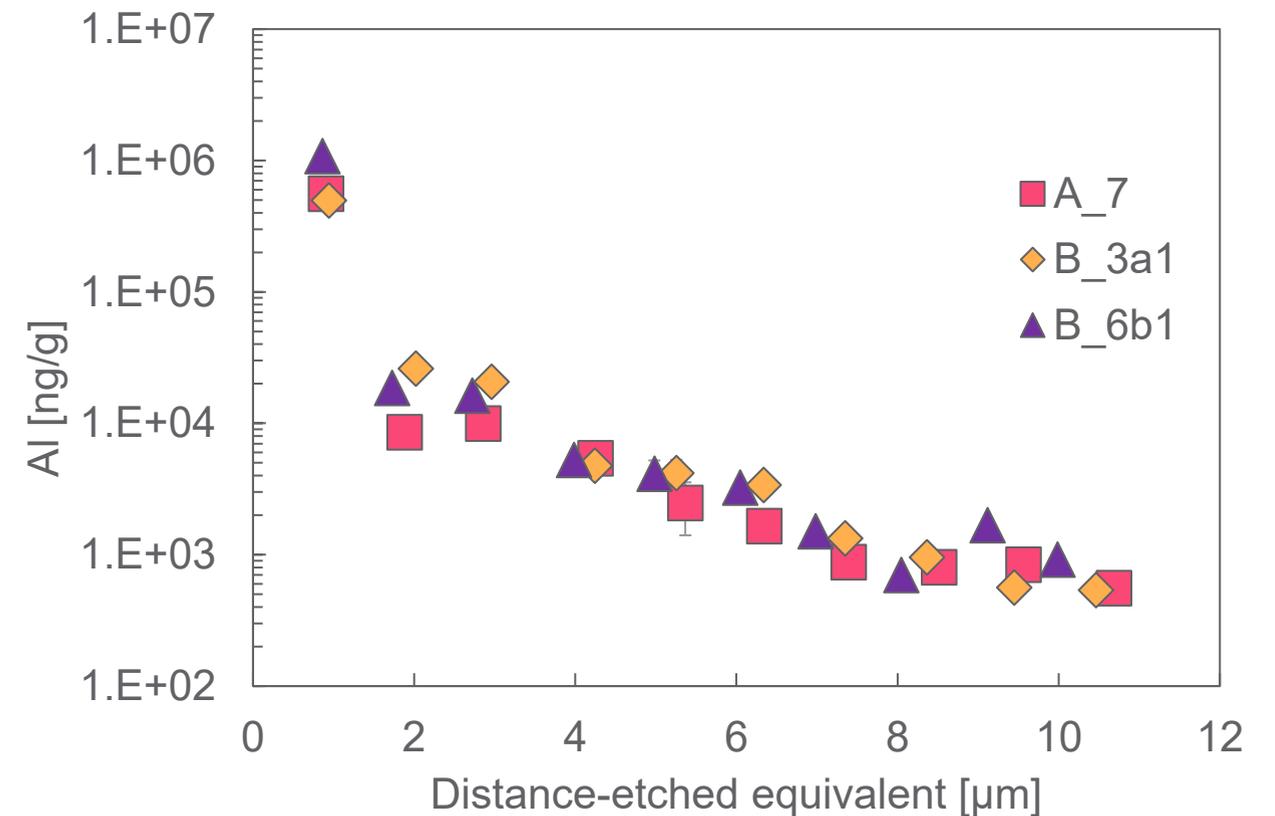
Ni portion	Ni Segment	$^{232}\text{Th}$		$^{238}\text{U}$	
		ppt	$\pm$	ppt	$\pm$
1% etch (first)	A7	42	5	9.5	0.8
	B3a1	77	6	44	2
	B6b1	63	3	40	3
1% etch (second)	A7	7.0	0.6	1.3	1.1
	B3a1	7.2	0.5	0.5	0.3
	B6b1	16	3	1.6	0.2

Ni portion	Ni Segment	$^{232}\text{Th}$		$^{238}\text{U}$	
		ppq	$\pm$	ppq	$\pm$
25% etch	A7	1060	40	170	30
	B3a1	710	40	<76	--
	B6b1	650	40	1190	40
Bulk	A7	25	7	<43	--
	B3a1	56	12	<40	--
	B6b1	54	10	<39	--



# Radiopurity Summary

- Bulk radiopurity of CVD Ni
  - ~1000 pg/g <sup>nat</sup>K
  - <100 fg/g <sup>232</sup>Th and <sup>238</sup>U
- Majority of impurities are present at Ni surface
  - >97% mass = <5 ng/g <sup>nat</sup>K, ≤1 pg/g <sup>232</sup>Th and <sup>238</sup>U
  - Non-uniform etching of outer oxide layer may obfuscate true distribution of contaminants with depth
  - Several avenues to explore in greater detail exist (e.g., reduce Ni prior to etching)
- Source of impurities may be Al substrate used during CVD process
  - Al contamination is present in CVD Ni and decreases with depth into segment
  - Testing purity of Al substrate may be helpful
  - Can selectively test Ni surface in contact with Al substrate



# Summary and Next Steps

- Raw CVD Ni is strong compared to COTS Ni
  - Welded CVD is comparable in strength to COTS
  - Likely due to thermal annealing
- Bulk CVD Ni is radiopure
  - Majority of contamination is present at material surface
  - Contamination may be associated with Al substrate
- Next Steps
  - Refine welding techniques
  - Better understand source of surface contamination
  - Investigate alternative substrates for deposition
- **Key takeaway: CVD Ni is a promising material for future  $0\nu\beta\beta$  and dark matter experiments.**

- Details available on arXiv

[ArXiv:2508.08230](https://arxiv.org/abs/2508.08230)

Ultra-pure Nickel for Structural Components of Low-Radioactivity Instruments

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ALSO: [High Purity CVD Tungsten!!](#)  
di Vacri et al. (2025) NIM A

# Thank you

## Acknowledgements:

- nEXO Collaboration
- DOE Office of Nuclear Physics
- PNNL team: Roosendaal, T.J., Overman, C.T., Ortega, G.S., Rocco, N.D., Horkley, L.K.S., Hobbs, K.P., Orrell, J.L., di Vacri, M.L., Arnquist, I.J.

## Void Spaces in CVD Ni welds

- Dog bones were cut out using EDM
- After cutting samples lots of different sized voids in the weld path were noticed
- Some of the voids were pinhole size while others were a couple mm
- Voids seen in all welded CVD Ni samples. No voids seen in unwelded CVD Ni
- Large void spaces not seen in COTS Ni, although there was a thin line void where the weld was stopped and then finished days later.



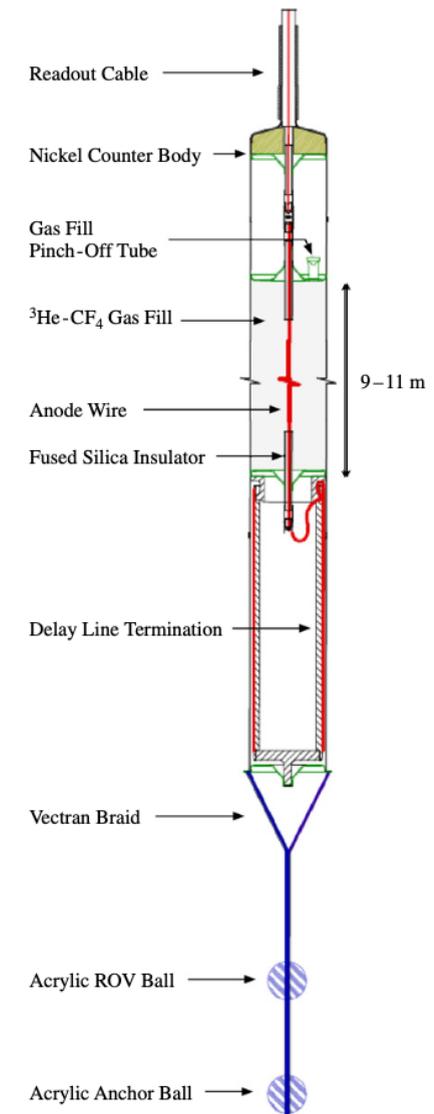
# SNO Neutral Current Detectors (NCDs)

Sudbury Neutrino Observatory (SNO)  
used CVD Ni

- CVD Ni used for tube body of Neutral Current Detectors (NCDs)
- Strict radiopurity was required

SNO NCDs are thin-walled tubes

- Future use would expand to *structural* uses
- Radiopurity and surface cleanliness were and remain key considerations



# Tensile Strength Comparison

## Tensile strength

- Report provided by company →
  - ✓ SNO did not make measurements
- Our results for ultimate tensile
  - ✓ ~600 MPa as provided
  - ✓ ~200 MPa after annealing

## Welding impact

- SNO used laser weld & learned to avoid over heating
- We used TIG, finding porosity and strength loss
- Clear opportunities for improved weld processing

## NIM A 579 (2007) 1-54-1080

Table 2  
Properties of CVD nickel (courtesy Mirotech, Inc.)

Specific gravity	8.871
Yield strength $\parallel$	440 MPa
Yield strength $\perp$	600 MPa
Ultimate tensile strength	~640 MPa
Elongation	~25%
Modulus of elasticity	178 GPa
Residual stress (surface)	30–60 MPa tensile
Coefficient of thermal expansion	$13.1 \times 10^{-6} \text{ K}^{-1}$
Thermal conductivity	$88 \text{ W m}^{-1} \text{ K}^{-1}$

# Material Assay Comparison

Bulk U & Th:

- SNO 1993 measurement ~ **10 ppt**
- SNO 2008 thesis recommends ~ **2-3 ppt**
  - ✓ ... in ~370  $\mu\text{m}$  thick material
- We measured ~**0.05 ppt**
  - ... in ~5 mm thick material

Profile of impurities:

- SNO identified an Al impurity profile  $\rightarrow$
- Our impurity profiles are consistent...

None of these are true apples-to-apples!

(2000 – SNO-STR-2000-005)

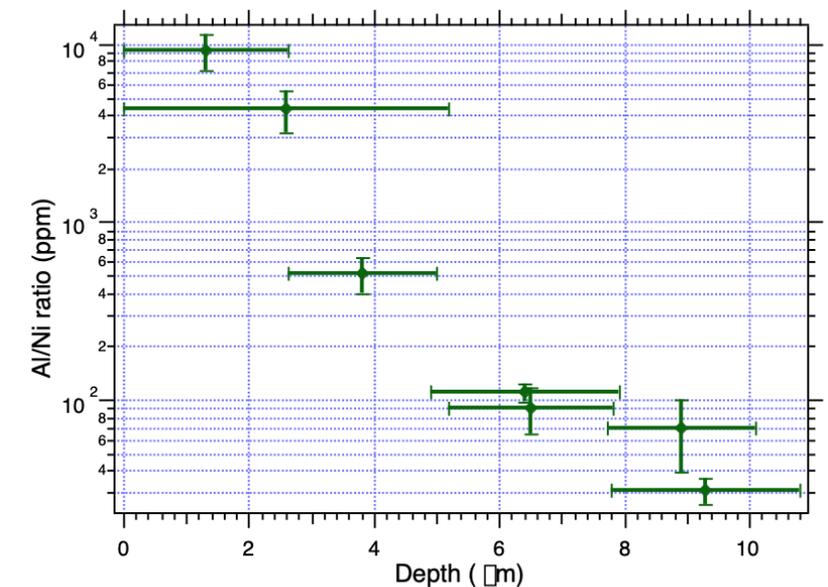


Figure 7: Aluminum contamination as a function of depth