Nanostructured Pd-ANF Composites for Controlled LENR Exploitation

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Public Release Slide

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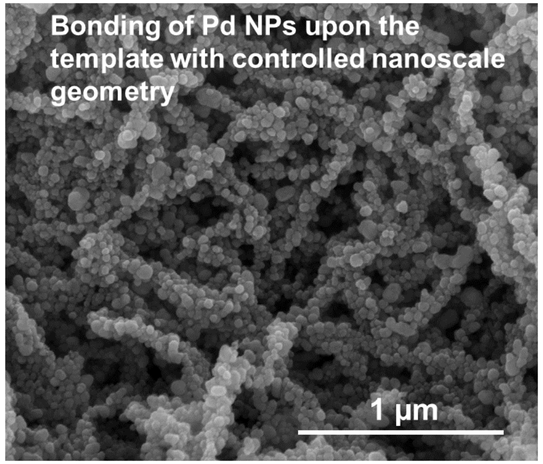
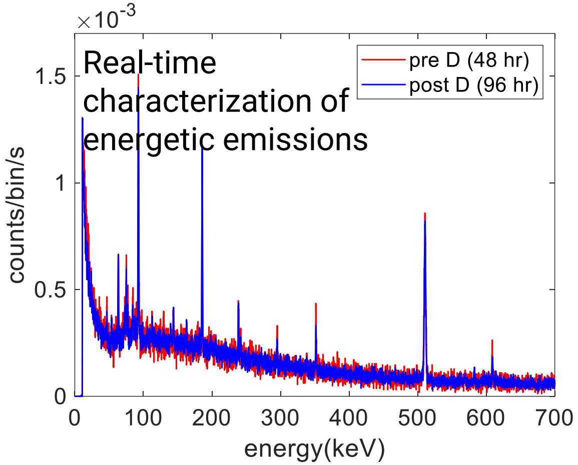
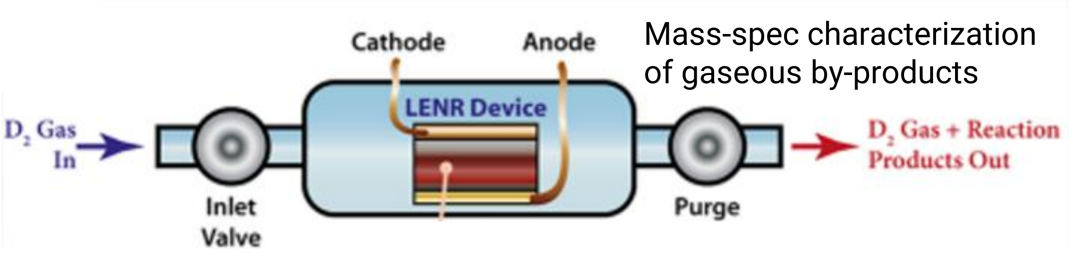




Project Title:

Nanostructured Pd-ANF Composites for Controlled LENR Exploitation



PI:

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Project Outcomes:

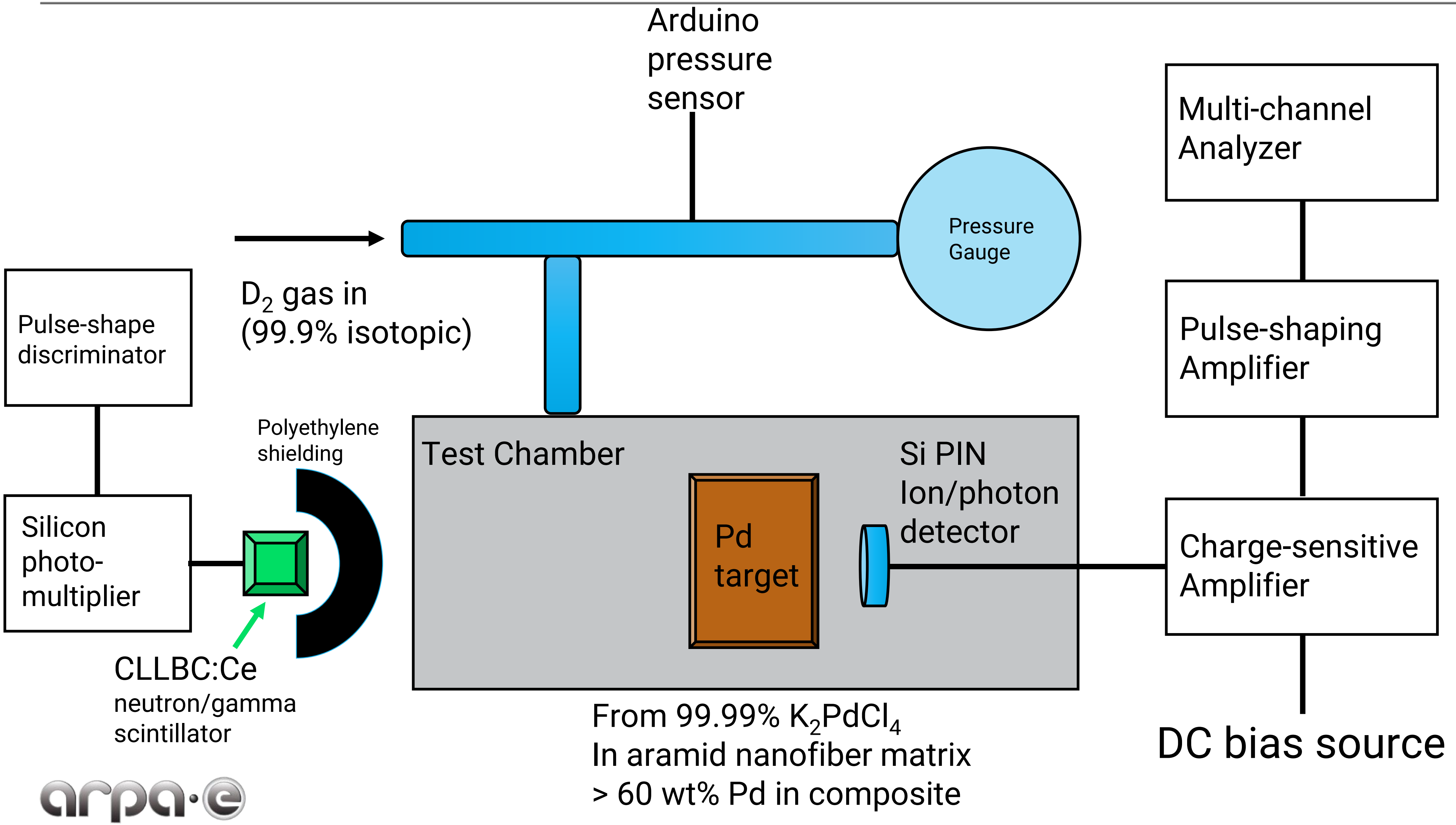
Define the nuclear processes at the core of potential low-energy nuclear reactions from D-loaded Pd

Key takeaway: Pd nanocomposites with in-situ radiation detectors enable real-time characterization of the energetic emission of any present mass-energy conversions

Hypothesis

- Utilizing a variety of ion/photon/neutron detectors, *in-situ* real-time measurements of any energetic emission occurring from D-loaded Pd
- Composites formed of nanoparticles of Pd loaded within an aramid nanofiber (ANF) matrix maximizes nanocracks where LENR is claimed to occur
- Measurements:
  - Pre-experimental measurements
    - Ion/radiation background, Pd-ANF resistance, Pd-ANF thermogravmetric data
  - Active-experimental measurements
    - *In-situ* ion/photon detection, external neutron detection, pressure
  - Post-experimental measurements
    - PdD_x-ANF resistance
- Detectors
  - Silicon-PIN (550μm): ions, photon (optical, x-rays, gamma-rays)
    - 1.3% resolution at 356 keV
  - High Purity Germanium (HPGe, 1” x 3”): gamma-rays
    - < 1% resolution at 662 keV
  - $Cs_2LiLaBr_{4.8}Cl_{1.2}$:Ce (CLLBC:Ce, 1”x1”): neutrons, gamma-rays
    - 3.2% resolution at 662 keV

Experimental Setup



Data Acquisition

Measurement	Recording Method	Settings	Latency	Storage Media
High-energy Ions	1) Adjacent Si-PIN 2) *in situ* PbTe NPs	(Si) 1 cm^2 x 500 μm V_{bias} = 70 V, shaping time 500 ns, G = 20 (PbTe) V_{bias} = 300 V, shaping time 500 ns, G = 500	< μs	Hard drive (Non-volative memory (NVM)) and cloud
Gamma-rays	1) Adjacent Si PIN 2) in situ PbTe NPs, 3) HPGe, 4) CLLBC scintillator (1" x 1"), 5) GAGG(Ce) Imager (2" x 2" x 2 cm pixelated array)	(HPGe) 3" x 3" CLLBC coupled to SiPM (~20 V bias) GAGG(Ce) coupled to SiPM (~32 V bias)	< μs	NVM and cloud
Neutrons	1) CLLBC scintillator 1" x 1", 2) GAGG(Ce) Imager (1" x 1" x 2 cm pixelated array)	CLLBC coupled to CAEN digitizer with PSD	< 10 μs	NVM and cloud
Hydrogen Loading	1) Resistance of Pd-composite, 2) Pressure of test chamber.	Resistance bridge connected to Arduino. Pressure sensor coupled to Arduino	1 s	NVM and cloud
Temperature of sample	1) Thermocouple and thermistors (connected to thermal heating element)	Heater/temperature controller (TC300 from Thorlabs) coupled to resistive cartridge heater and thermocouple)	1s	NVM and cloud

CHANGING WHAT'S POSSIBLE

Modeling

- Monte-Carlo Neutral-Particle transport code (MCNP)
  - MC gamma-ray and neutron transport and spectroscopic detection
- CASINO
  - QMC electron transport
- Penetration and ENErgy LOss of Positrons and Electrons (PENELOPE)
  - MC electron-photon transport and attenuation
- Stopping and Range of Ions in Matter (SRIM)
  - MC charged particle transport and attenuation

Initial Test Plan

- Develop Pd nanoparticle recipe book, with control over particle size
- Successfully incorporate Pd NPs to aramid nanofiber matrix, forming composite with large Pd surface area and high density of nanocracks, believed to form the NAE
- Construct the detector setup, and validate with known particle sources
  - ^{241}Am, ^{133}Ba, ^{137}Cs
- Collect extensive radiation background with no Pd target in vacuum, no Pd target in D_2, Pd-ANF in air and H_2
- Collect radiation spectral data of Pd-ANF loaded with D_2
  - Measure the resistivity of pre- and post- D_2 loaded Pd-ANF

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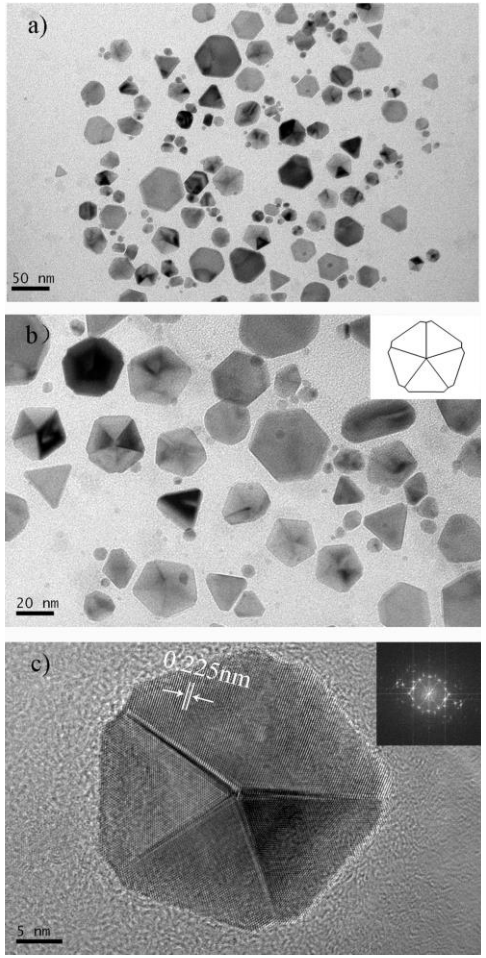
Initial Test Plan: Develop Pd NP Recipe Book

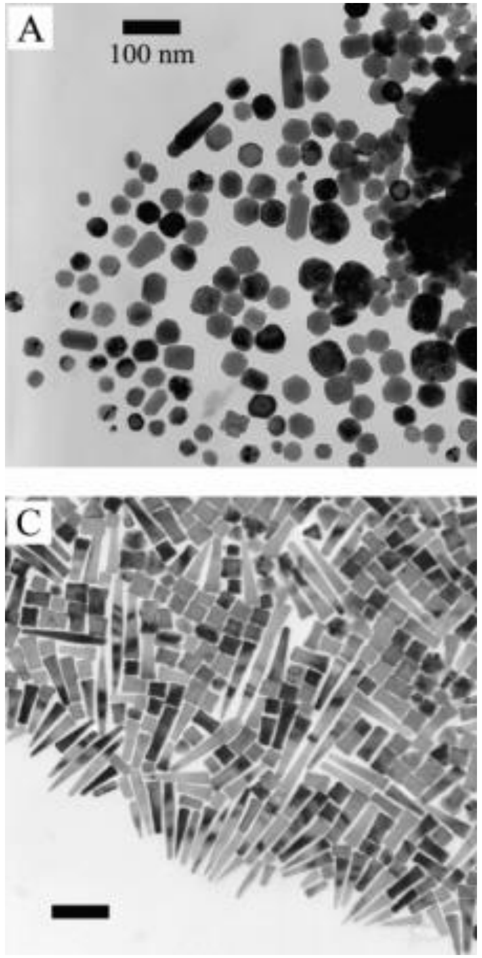
- Size and shape variation of Pd NPs largely influenced by the surface ligand choice
- Concentrations, pH, reaction time also factors to consider
- Past success incorporating NPs into ANF using Cit. with Au and Ag, PVP with Ag

PVP



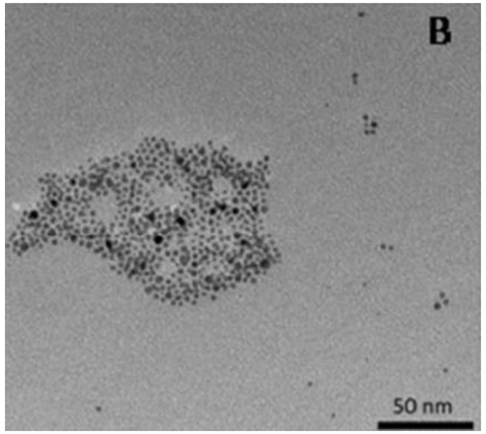
Ji, Wenhai, et al. "Synthesis of Marks-Decahedral Pd Nanoparticles in Aqueous Solutions." *Particle & Particle Systems Characterization* 31.8 (2014): 851-856.

CTAB

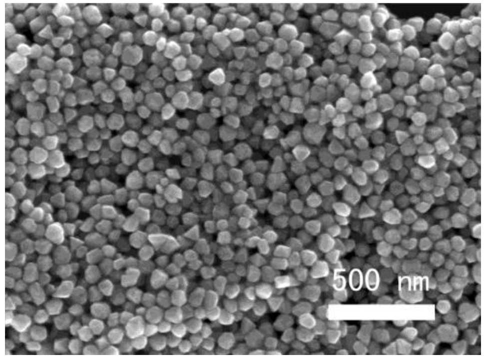


Sun, Yuan, et al. "Seedless and templateless synthesis of rectangular palladium nanoparticles." *Chemistry of Materials* 19.8 (2007): 2065-2070.

Citrate



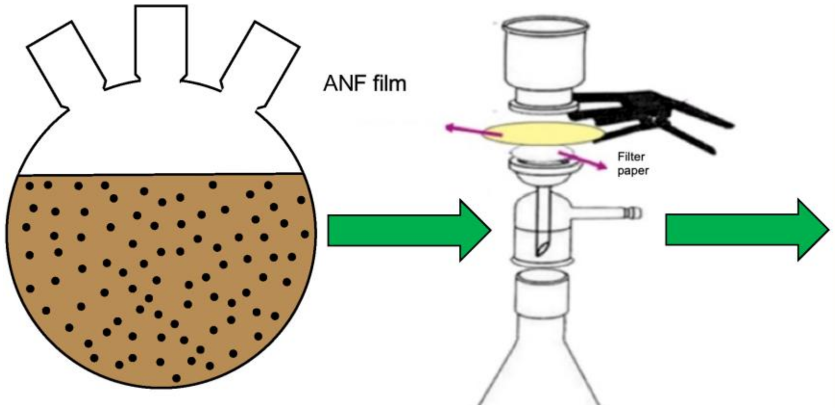
Wen, Dan, et al. "Controlling the growth of palladium aerogels with high-performance toward bioelectrocatalytic oxidation of glucose." *Journal of the American Chemical Society* 136.7 (2014): 2727-2730.

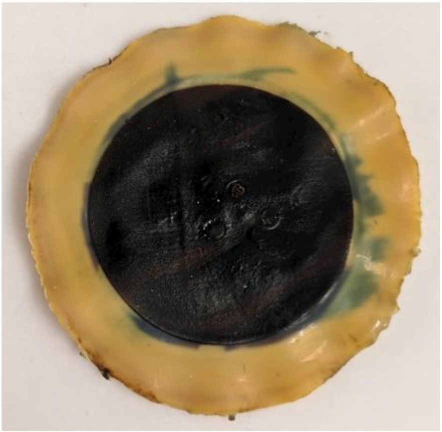


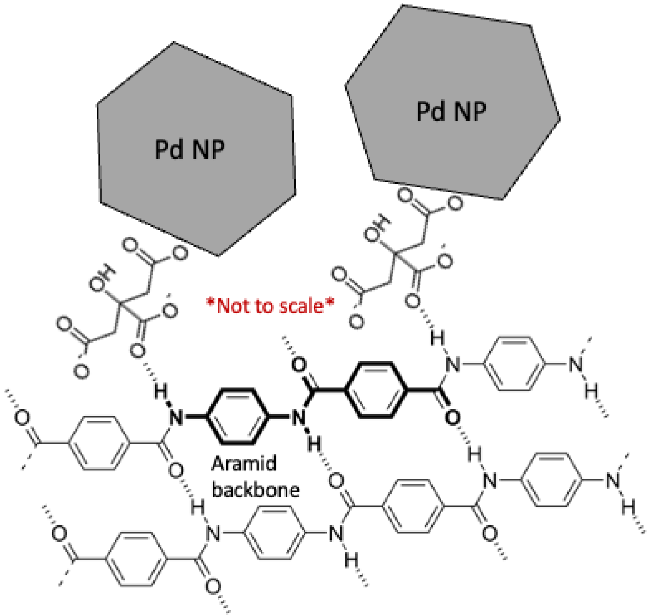
Chen, Hongjun, et al. "Synthesis of palladium nanoparticles and their applications for surface-enhanced Raman scattering and electrocatalysis." *The Journal of Physical Chemistry C* 114.50 (2010): 21976-21981.

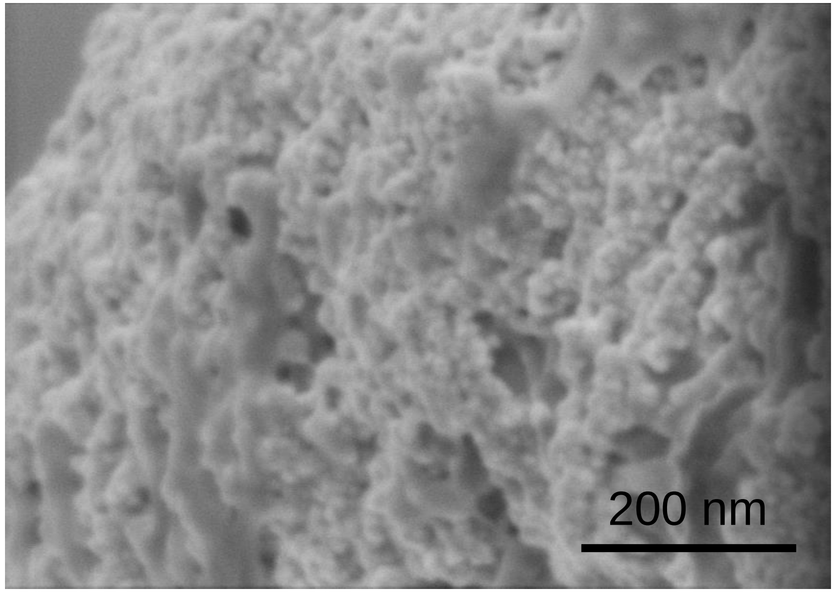
Initial Test Plan: Incorporate Pd NPs into ANF

Schematics for NP/ANF incorporation



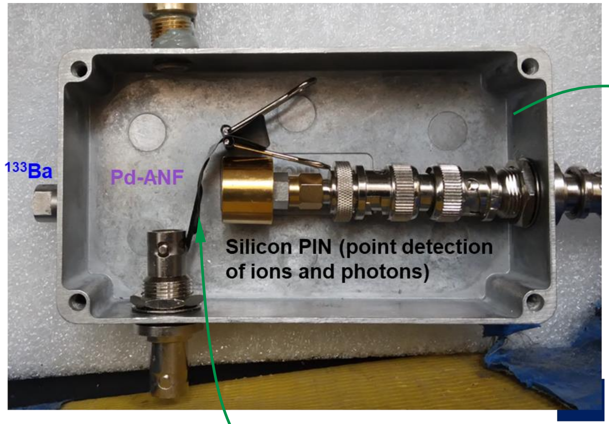


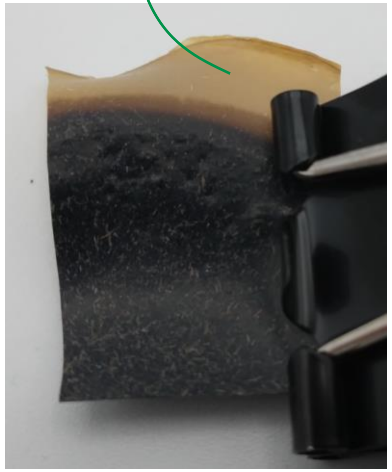


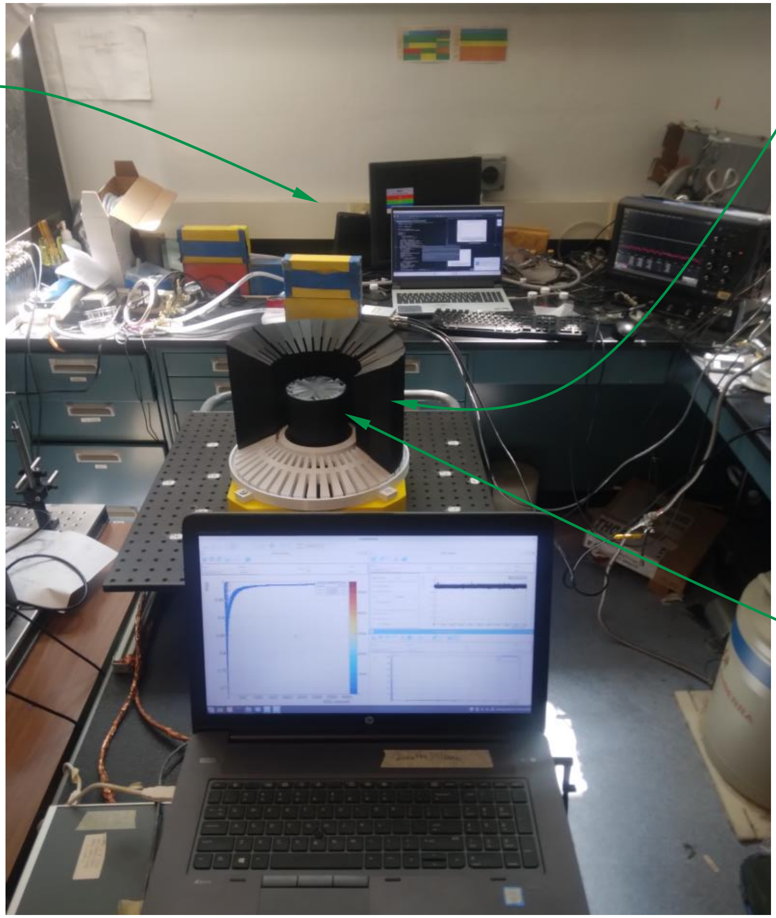


- Pd/ANF nanocomposites prepared via vacuum filtration, forcefully infiltrating NPs into the ANF film which acts as a filter
- Variations in NP diameter and geometry will vary the nanoscale “cracks” within the composite, though smaller NPs anticipated to lead to smaller “cracks” where the NAE is expected to exist

Initial Test Plan: Construct and Validate Detector Setup

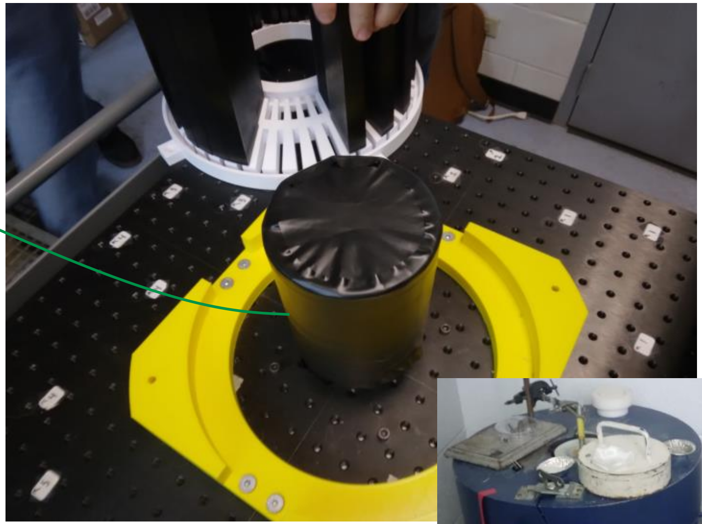


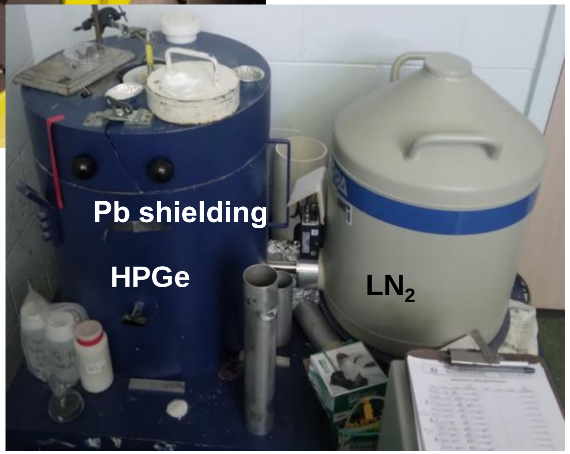




Polyethylene to thermalize the neutron flux (react with Lithium in scintillator)

1” x 1” CLLBC dual-mode scintillator
(Cesium Lanthanum Lithium BromoChloride ($Cs_2LiLaBr_{4.8}Cl_{1.2}$:Ce)





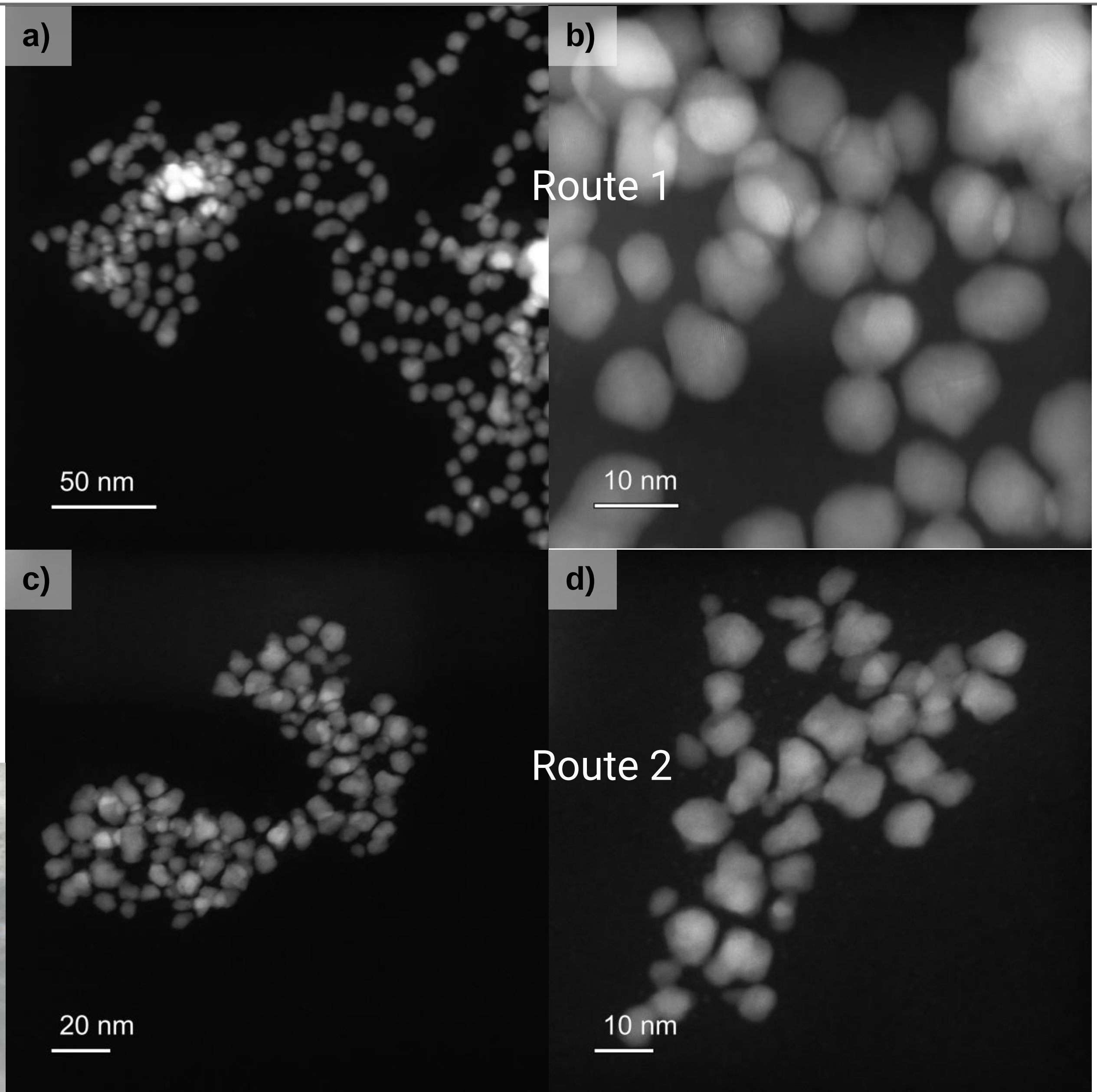
- Can use *EPSILON-G* imaging system based on GAGG:Ce (~6 - 7 % resolution) or *EPSILON-H* system based on stilbene (E_R = 10.9 %), but using CLLBC for inorganic dual-mode detection with 3 % resolution.
- Also have HPGe detector if greater photon energy resolution needed

CHANGING WHAT'S POSSIBLE

Initial Results: Aqueous Pd Nanoparticle Synthesis

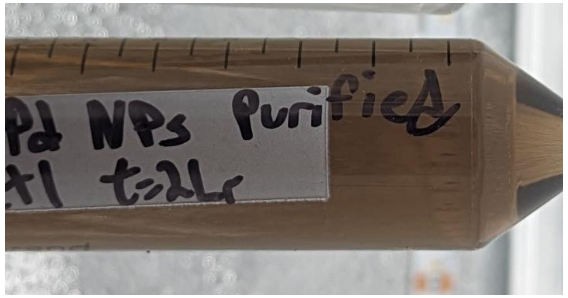
- Citrate-stabilized Pd nanoparticles successfully produced in both $NaBH_4$ (Rt.1) and tannic acid (Rt.2) reduction routes
- Autoclave preparation (Rt.3) has not succeeded in initial tests
- NPs from Rt.1 appear more spherical, while NPs from Rt.2 appear more polygonal
- Measured Pd particle size after purification is 9.6 ± 2.0 nm (N=100)
  - PDI = 0.044 (goal: PDI < 0.1)

Purified by centrifugal filtration
5000x g for 5 minutes
With 100 kDa filter

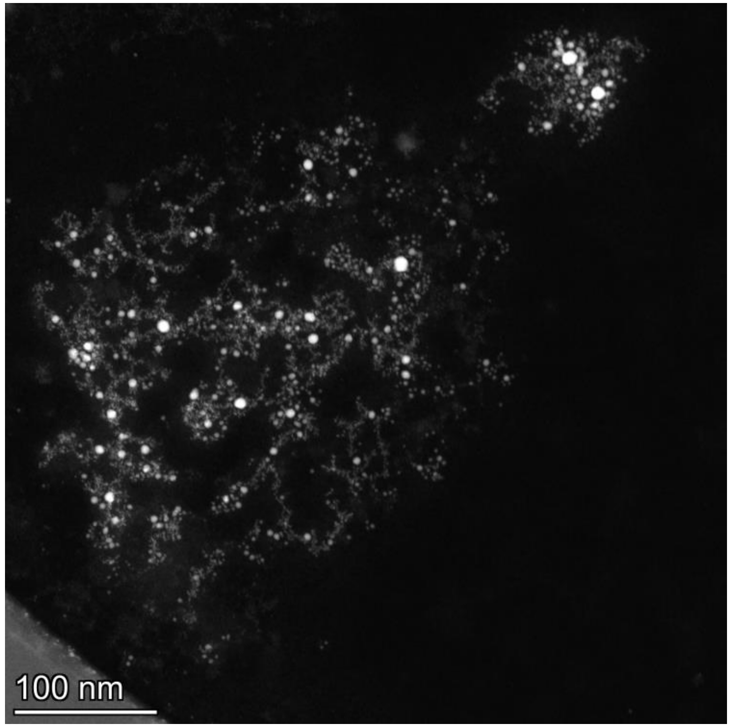


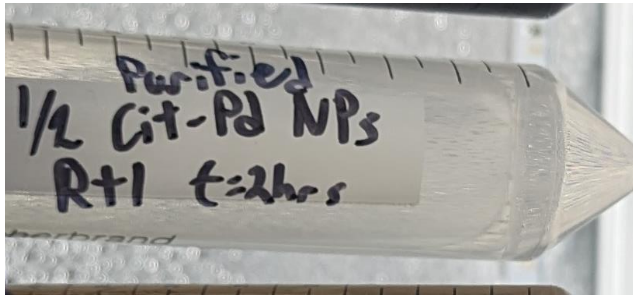
CHANGING WHAT'S POSSIBLE

Initial Results: Routes for size tunability

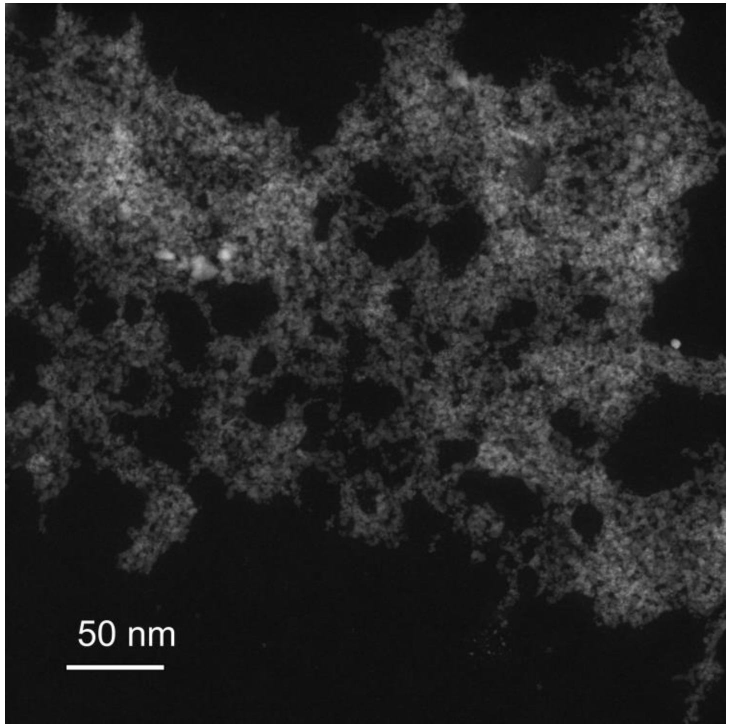


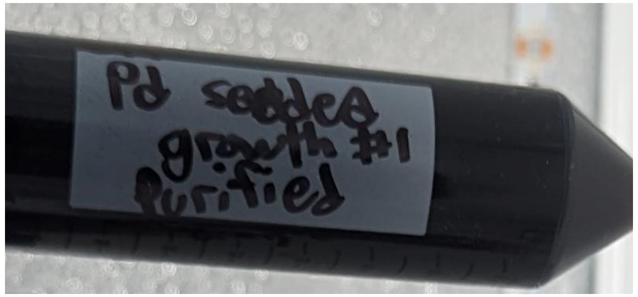
PVP-stabilized Pd



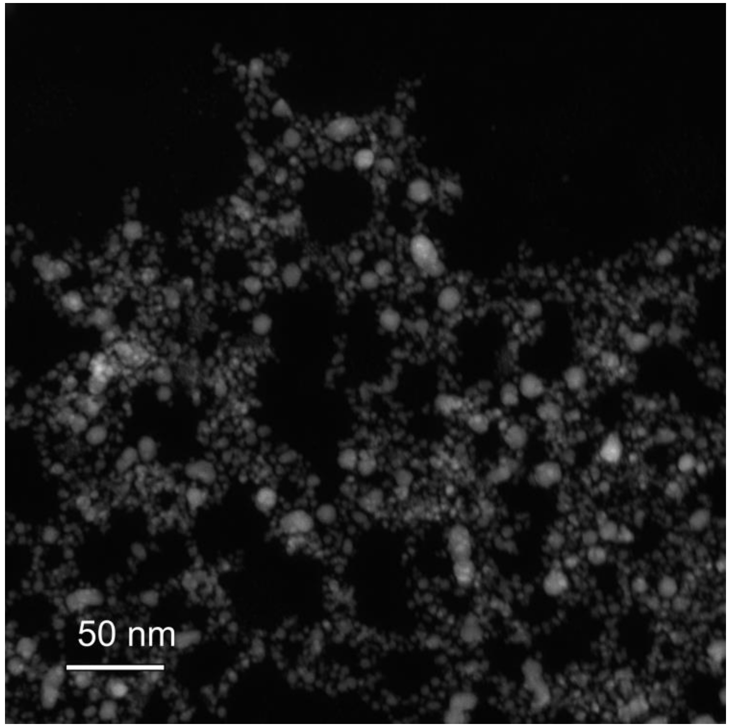


Reduced citrate ligand



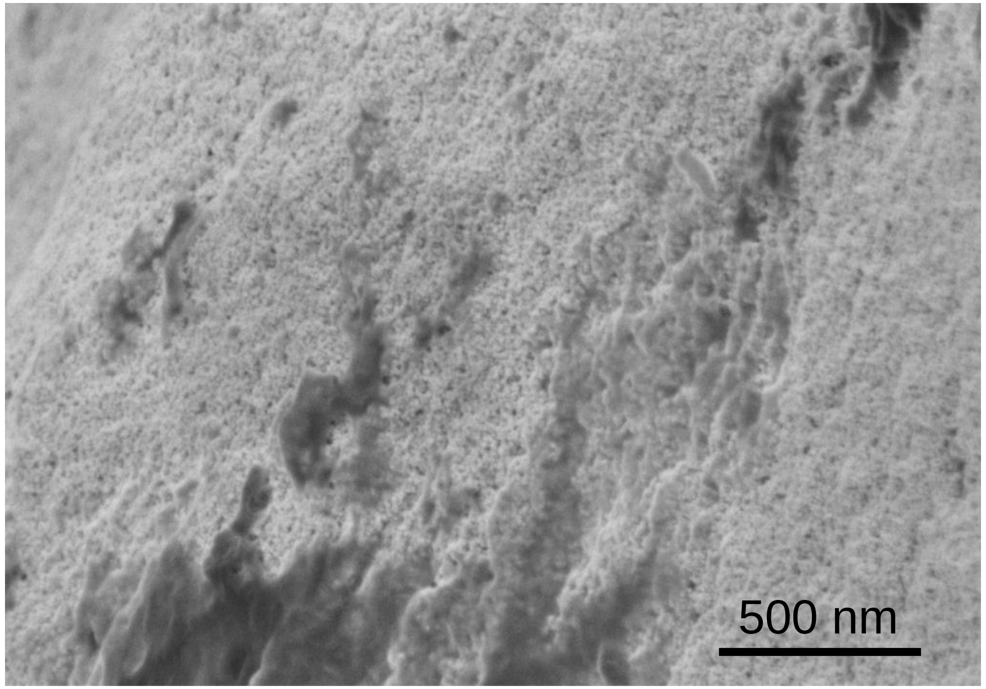


Attempt at seeded growth

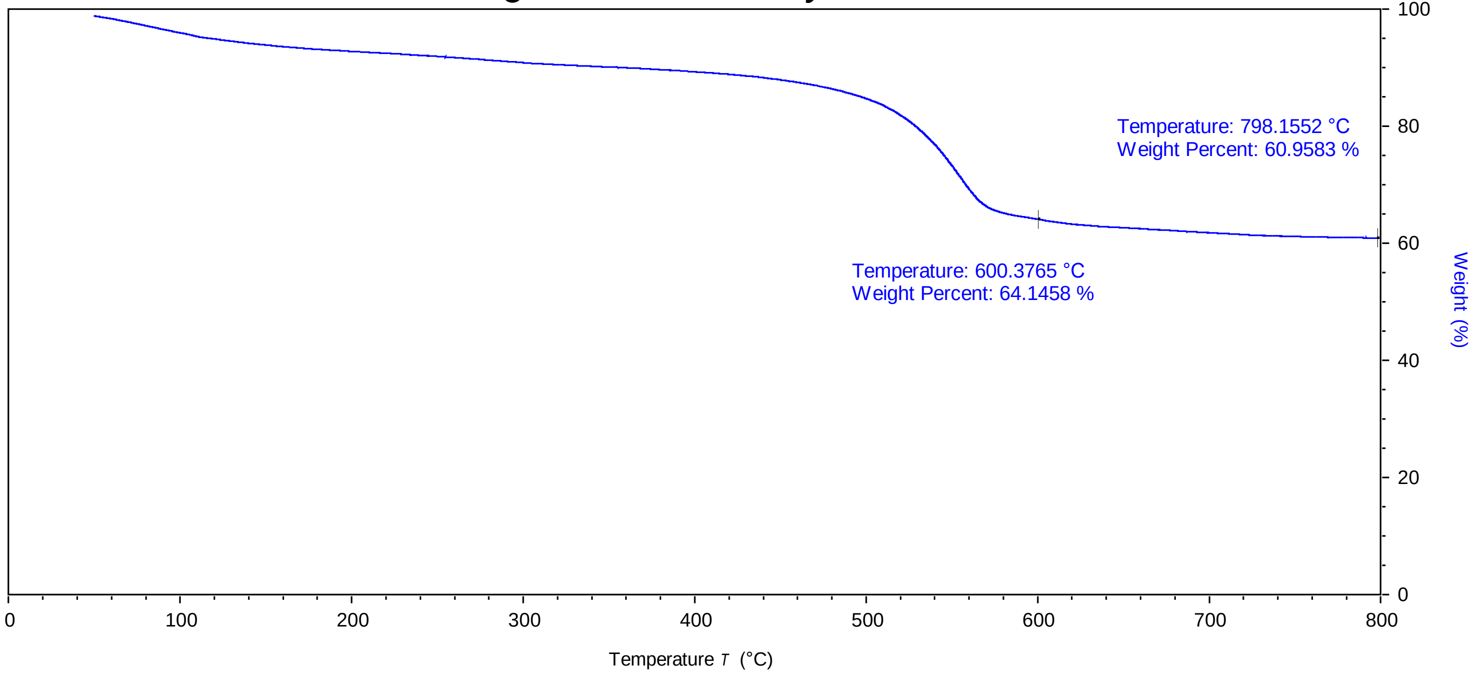


Initial Results: Pd-ANF preparation

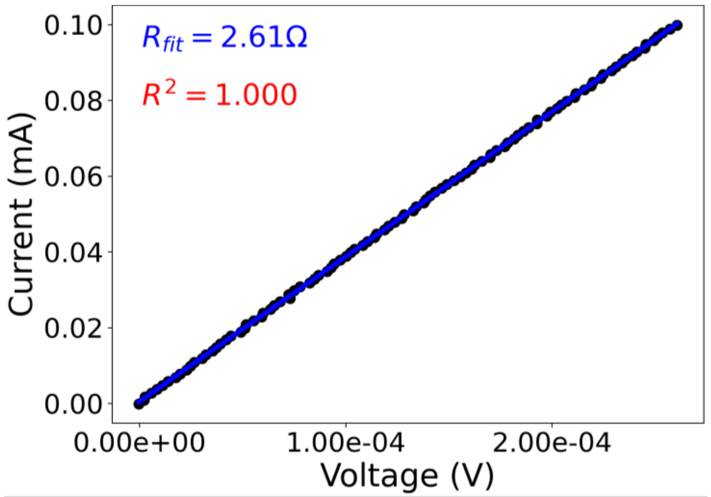
- Can successfully load with > 60 wt% Pd as measured with thermogravimetric analysis



Thermogravimetric Analysis of Pd-ANF



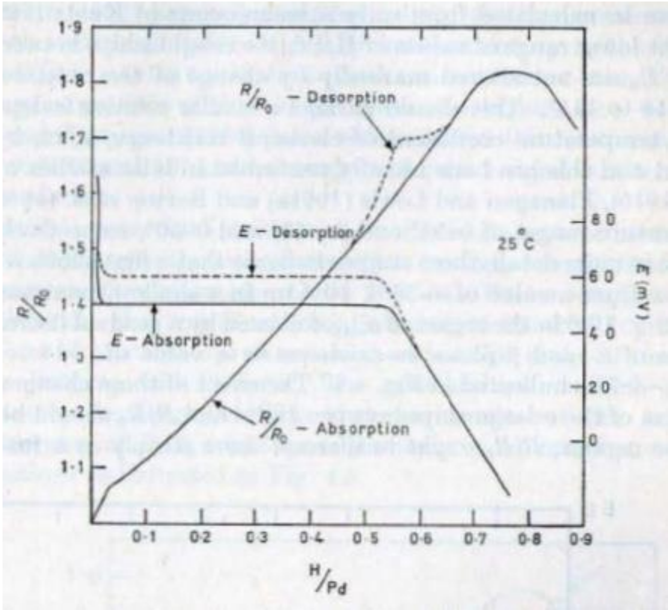
Initial Results: H-Loading via Resistance Variation



	R (Ω) Pd-ANF Glossy		
	2.5	2.5	
3.2	2.8	2.5	2.4
3.2	2.6	5.2	2.4
3.1	3.2	4.5	1.9
2.8	-	14.2	2.6
2.5	12.6	-	2.6
2.6	13.8	25.6	3.6

	R (Ω) Pd-ANF Matte		
2.31E+07			3.20E+07
2.41E+07			1.18E+07

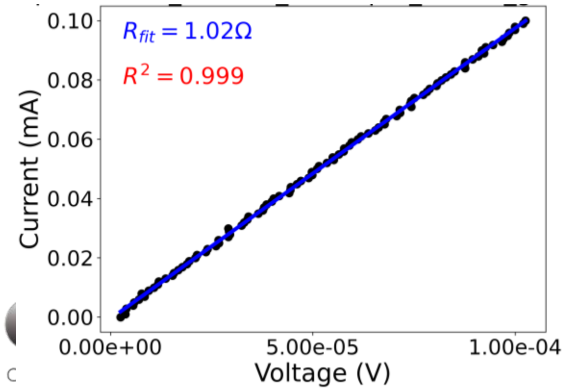
- Must load enough that sample is conductive.

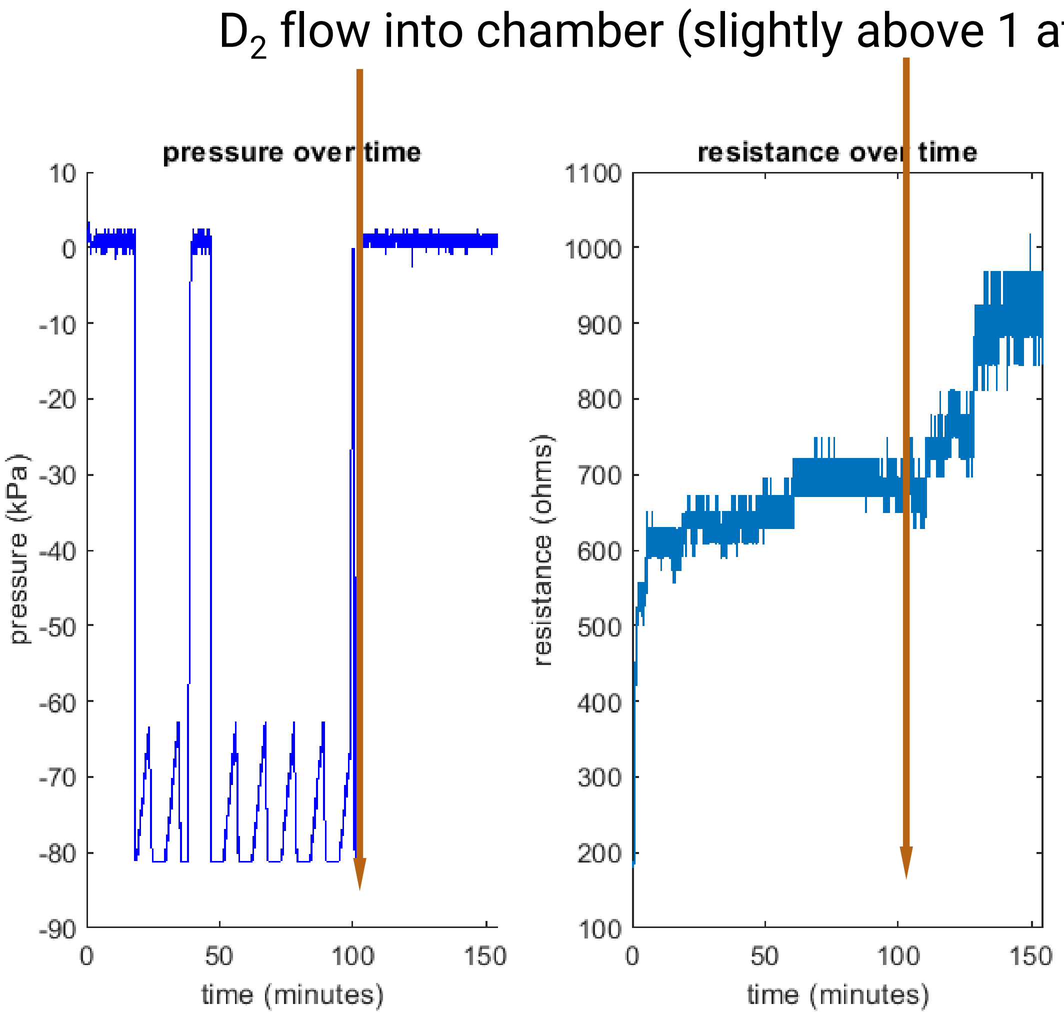


- Non-uniform loading throughout thickness but OK if surface loaded because of limited penetration depth of ions targeted (163 μm).

Initial Results: Hydrogen-Loading via Resistance Variation

	R (Ω) Pd-ANF Glossy			
	4.78	3.8	3.26	
5.44	5.6	3.89	2.82	3.92
3.75	2.73	2.28	3.2	4.04
2.52	2.12	2.52	4.66	2.08
3.36	3.95	2.64	2.6	2.38
5.31	2.1	3.05	1.51	2.12
4.9	7.57	2.48	1.49	1.02



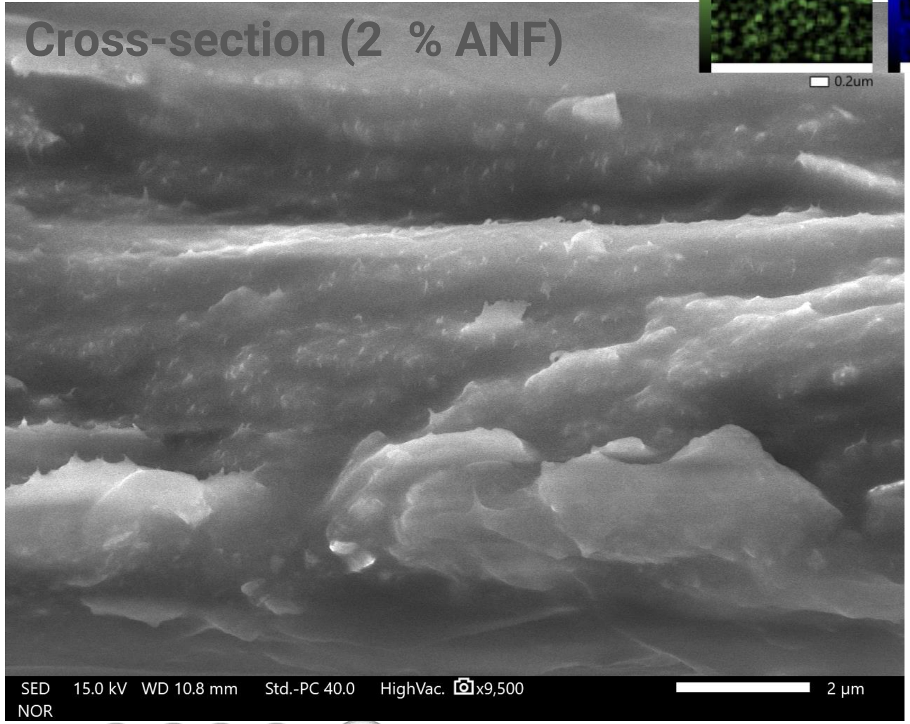


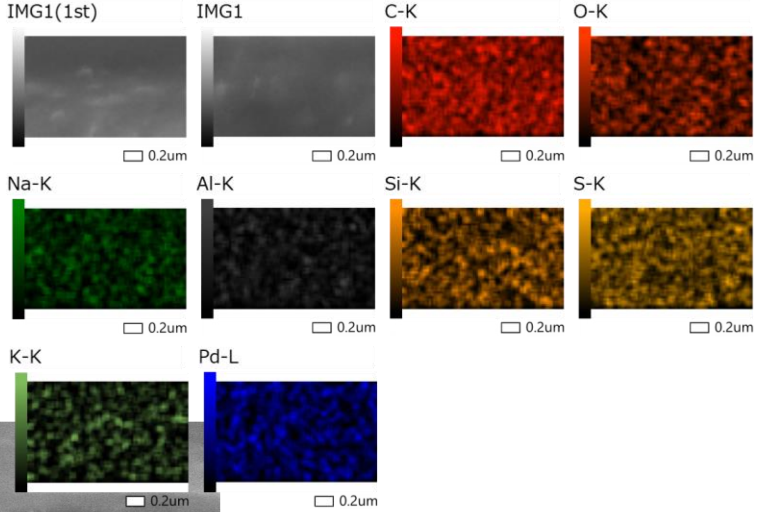
Measure pressure, temperature, and resistance with three of the analog channels of an Arduino.

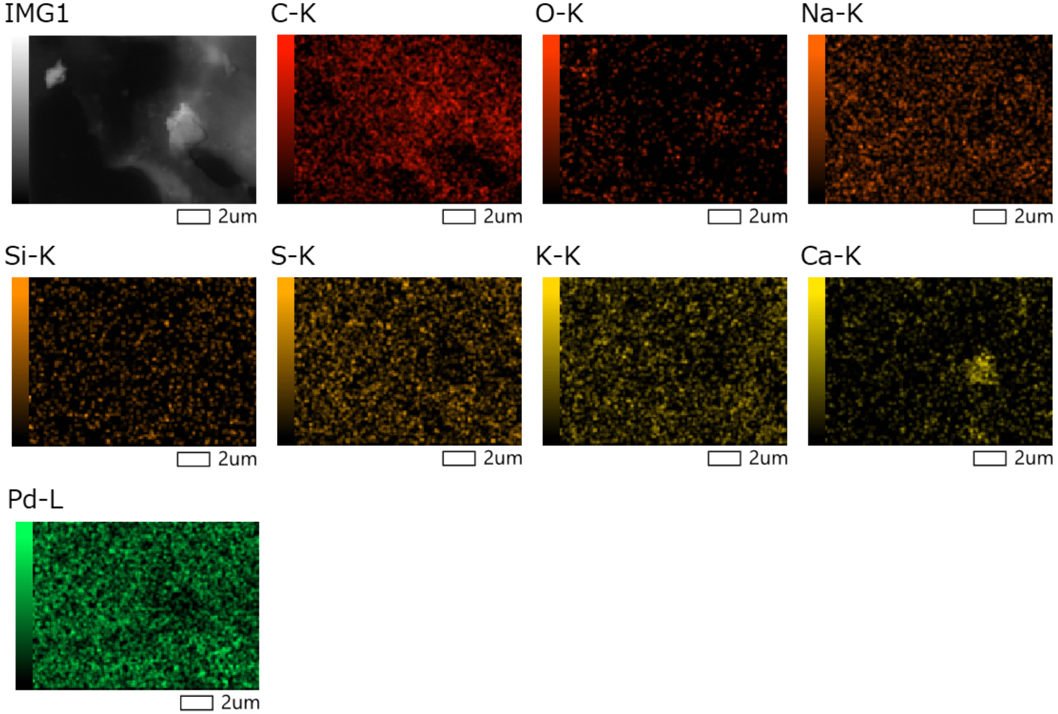
Pd-ANF is one component of resistive bridge circuit.

Resistance increases from 710 W to 940 W (R/R_o = 33 % before saturation
=> ~33 % D loaded into composite before saturation.

Initial Results: Pre- and Post-D SEM Analysis

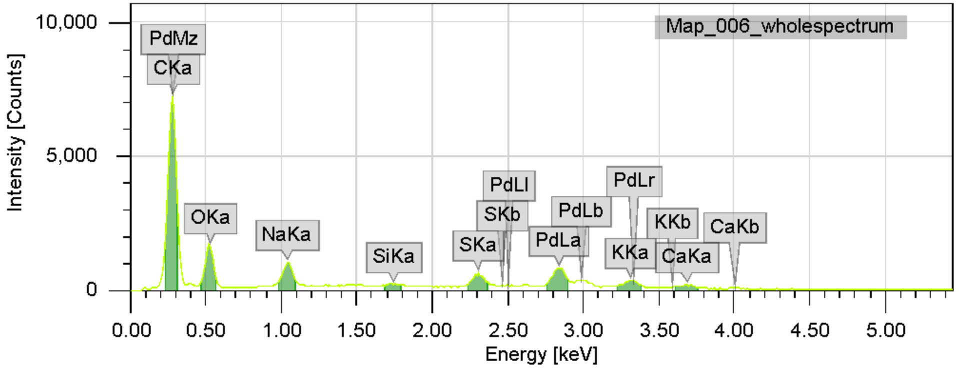






- No obvious differences observed.

Currently: Mounting Pd solid on SEM post during exposure so that we can definitely image the same spot pre- and post-D_2 exposure.
Perhaps utilize Texas Tech SEM scanner.



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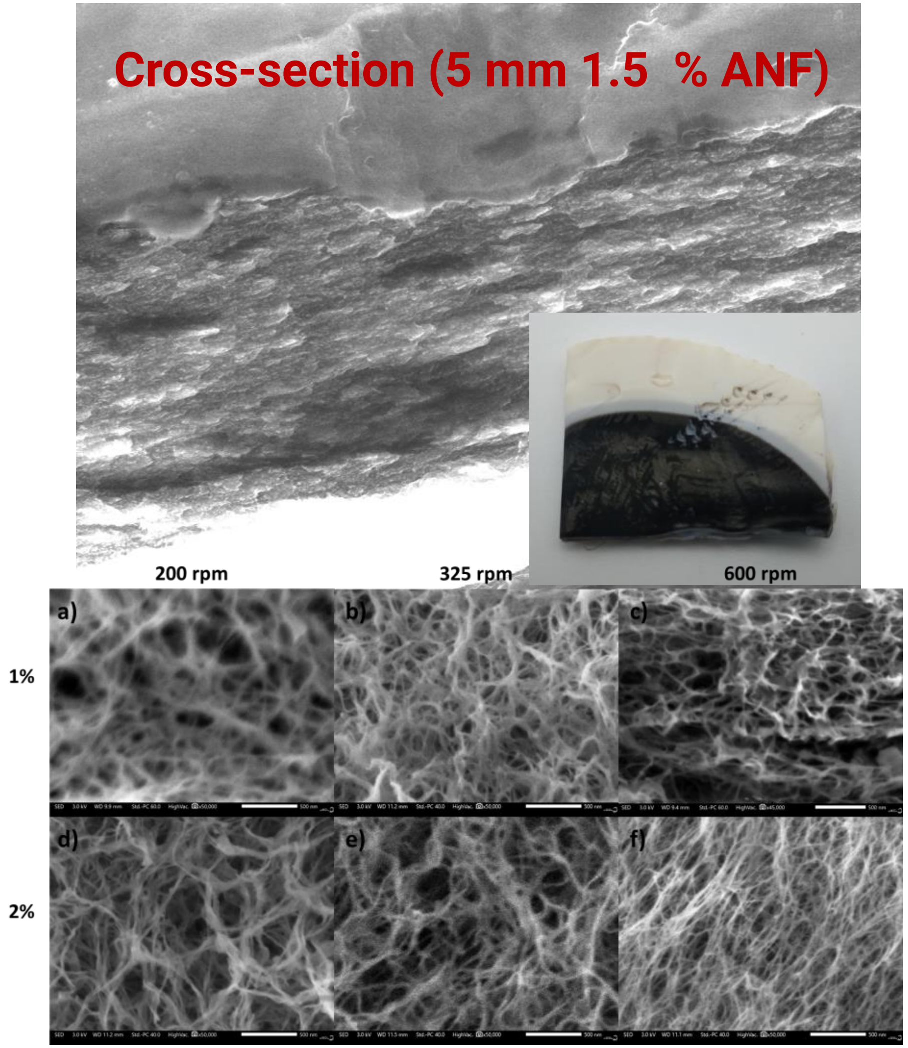
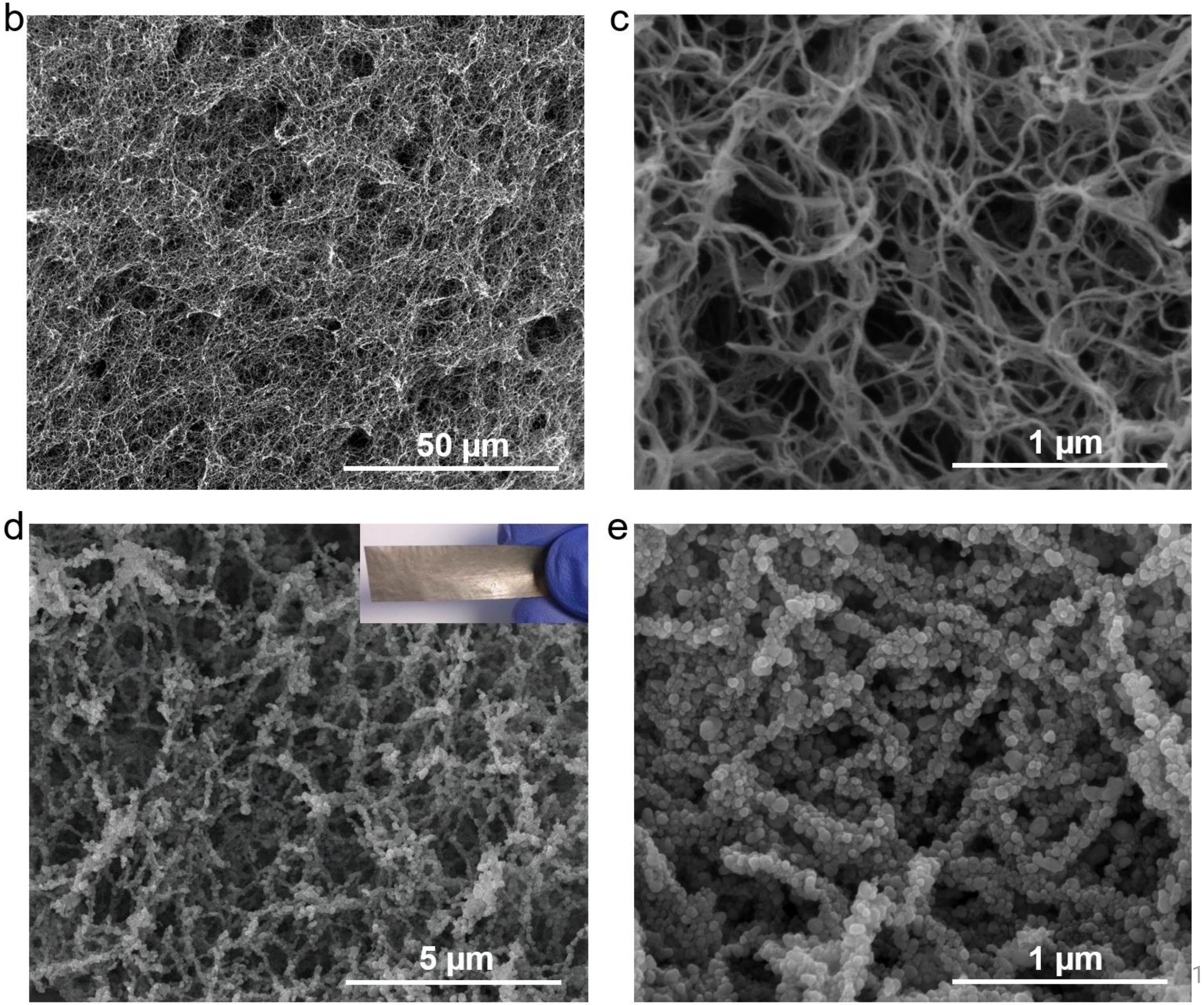
CHANGING WHAT'S POSSIBLE

Initial Results: Can change openness of microstructure

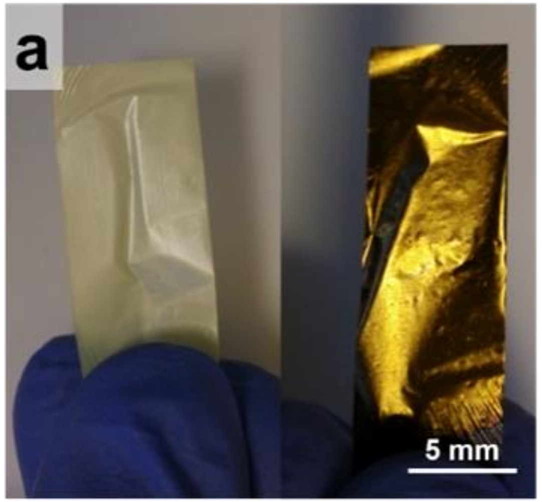
Difficult to image individual NPs and nanostructure for these air-dried samples (template collapses under surface tension).

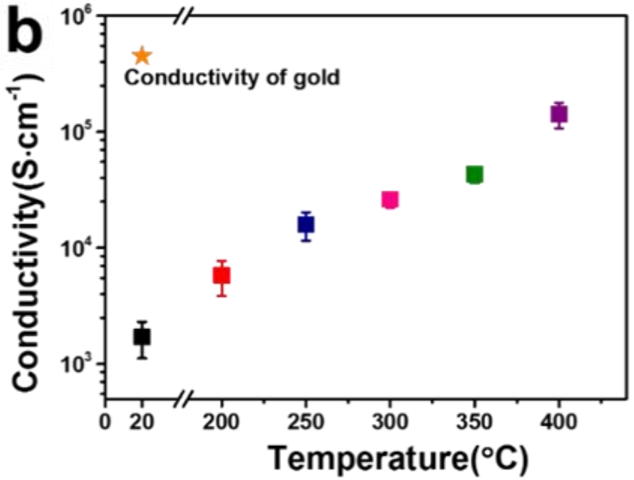


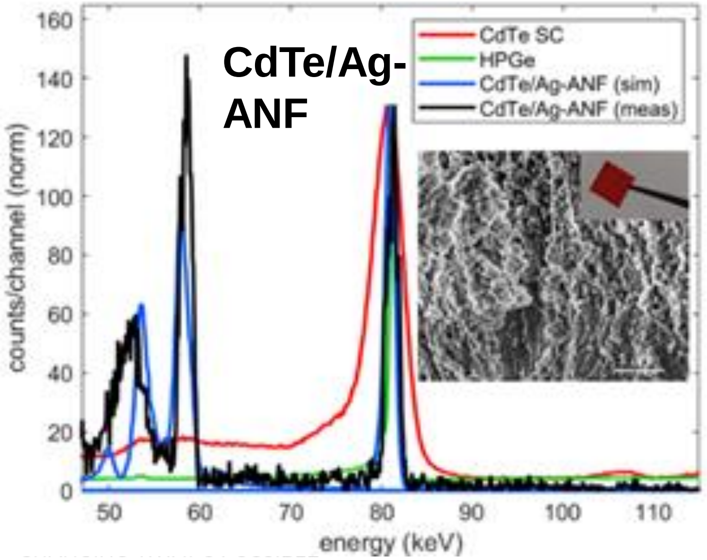


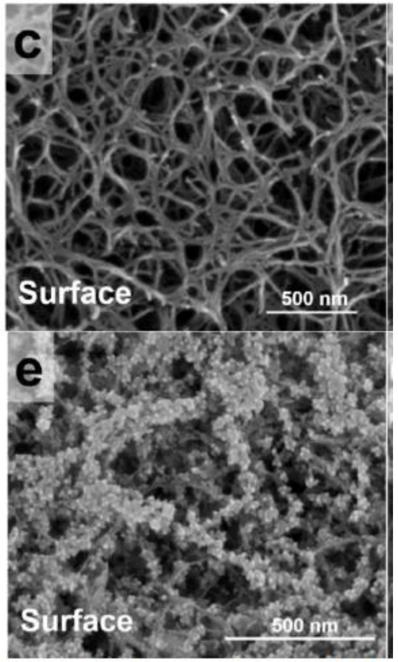
Initial Results: Can Co-locate the Pd with the semiconductor NPs (PbTe)

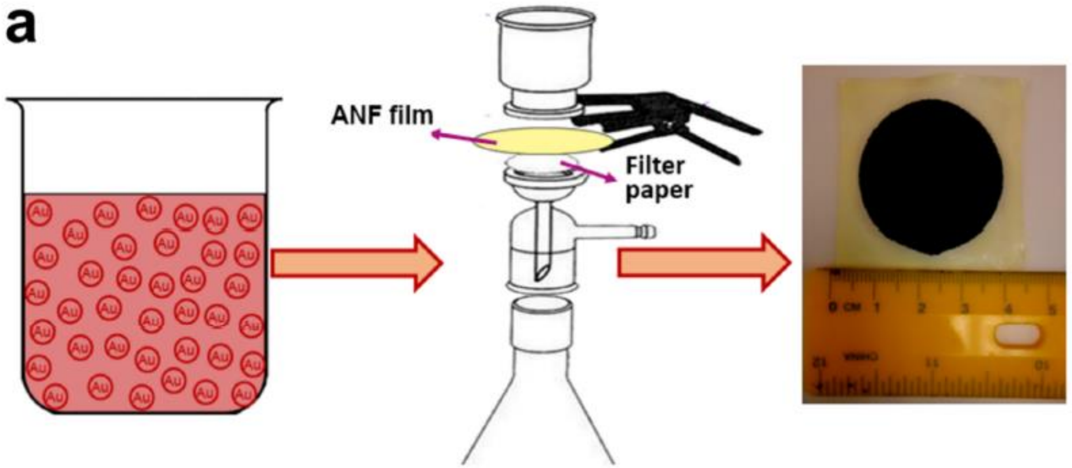
- Dot-to-dot coupling (overlap of orbitals) essential to material functionality.
- Performance of various nanostructured materials suggest this is the case (high conductivity in metallic NP / ANF, high-resolution detection for semicond. composites).



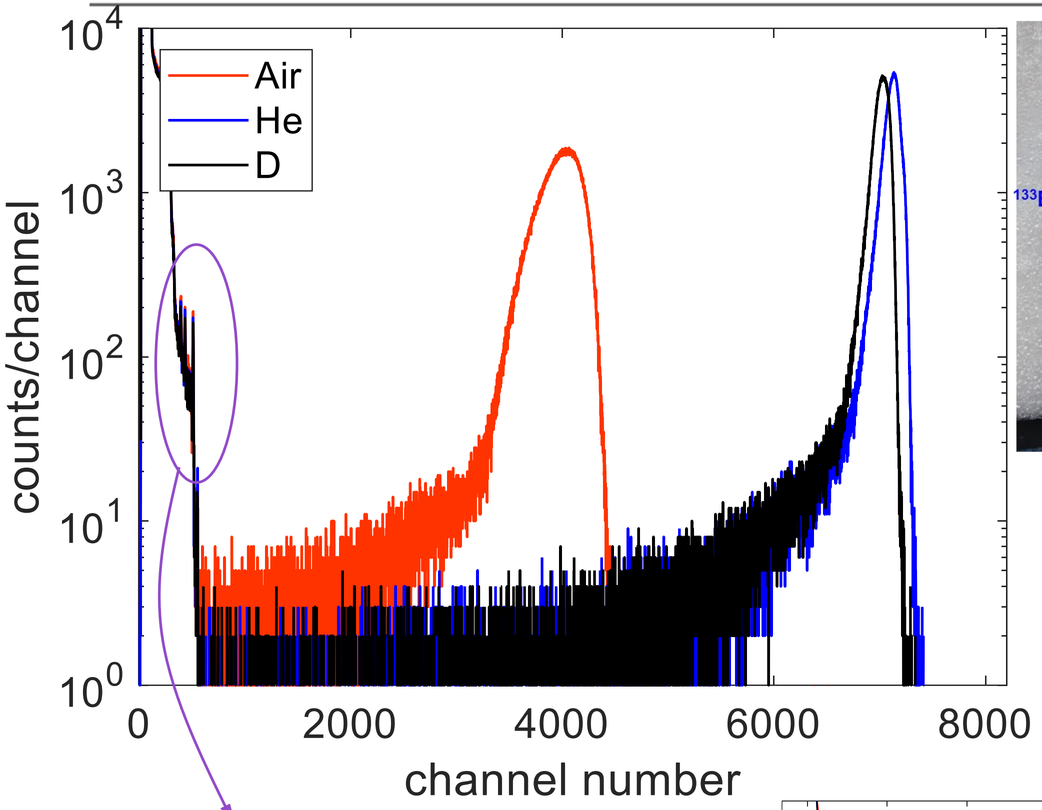


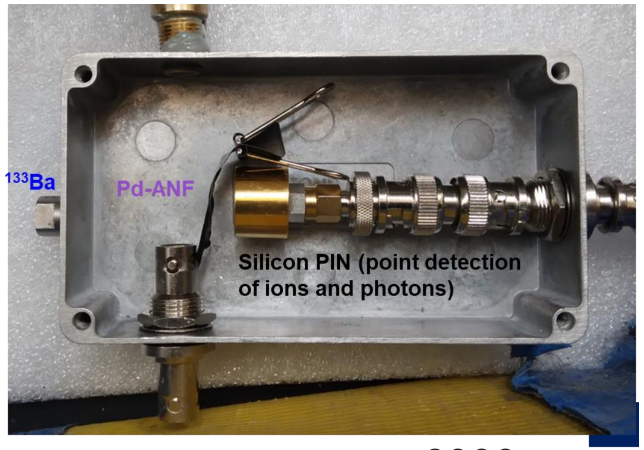




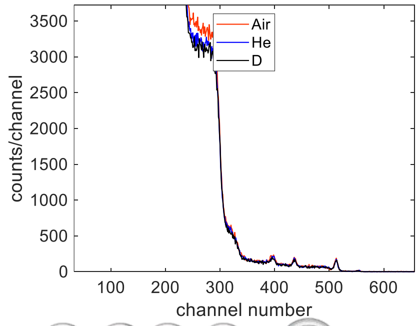


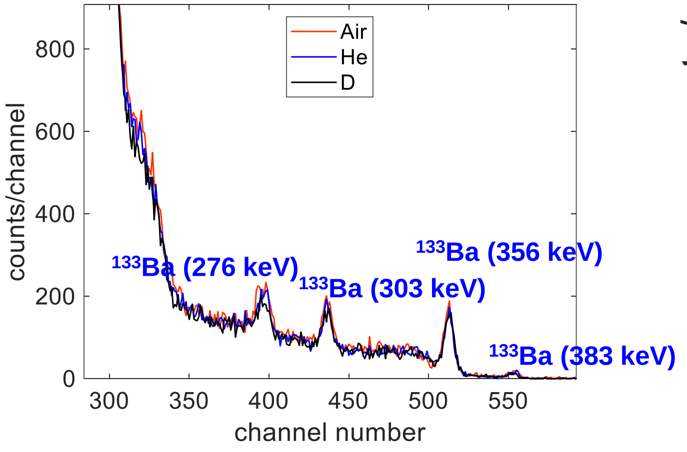
Initial Results: Radiation detection (Ions, Photons) via Si PIN

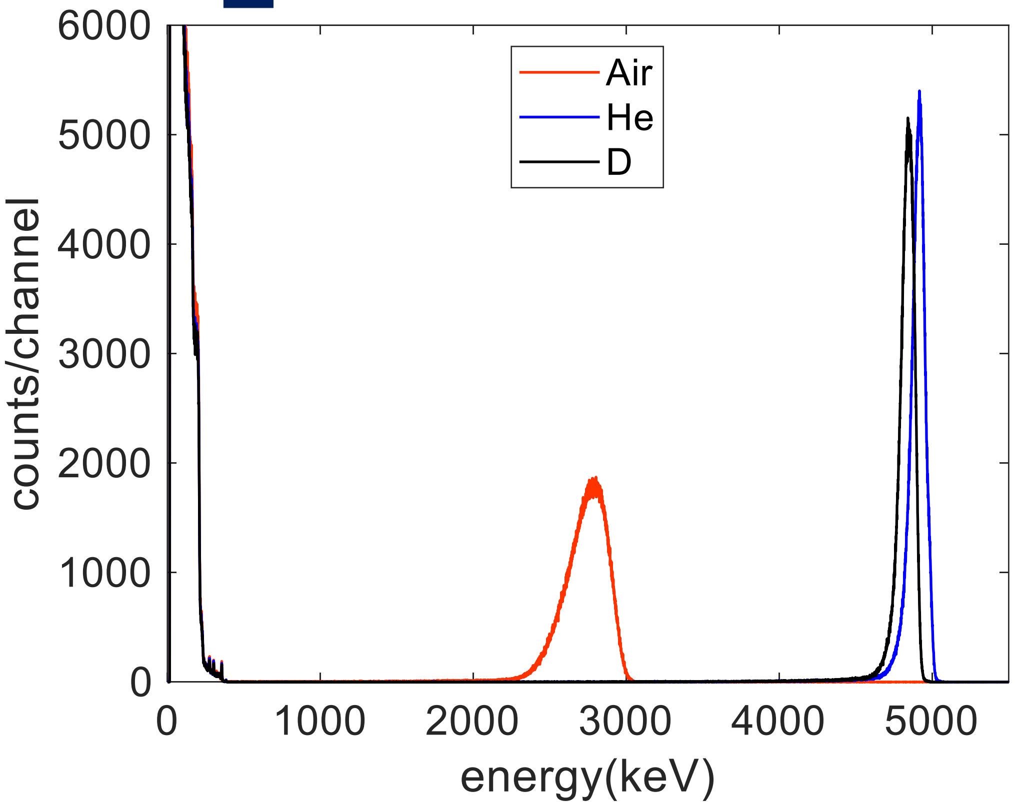




- External gamma-ray sources (^{133}Ba in this case), and internal alpha source (^{241}Am) used to calibrate the Si PIN detector.
- No Pd is present. (Control System)
- This is with commercial Si PIN detector.







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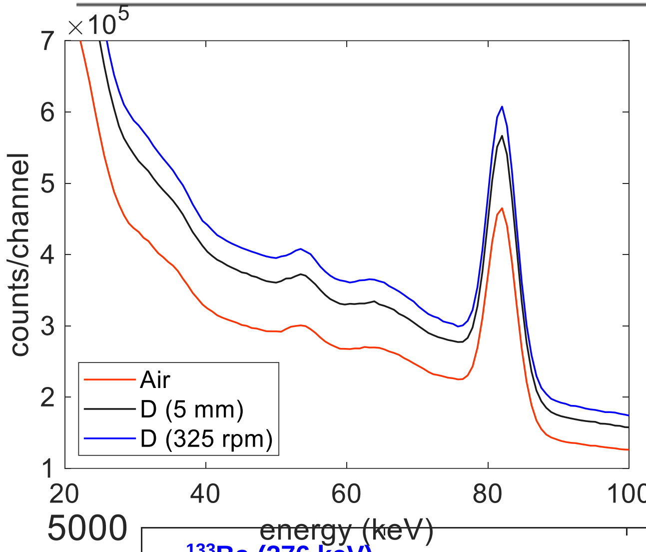
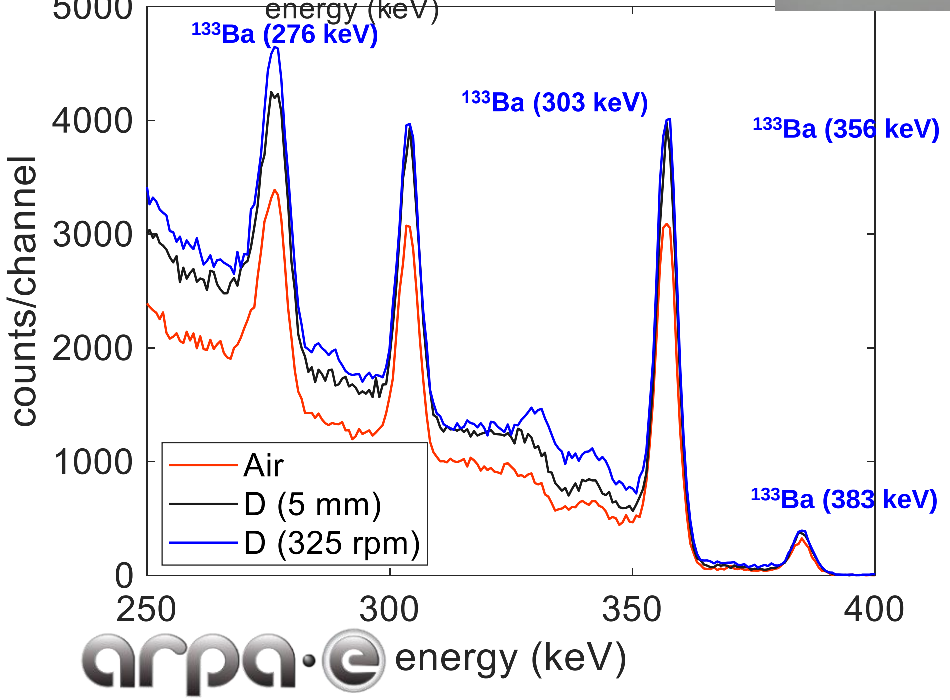
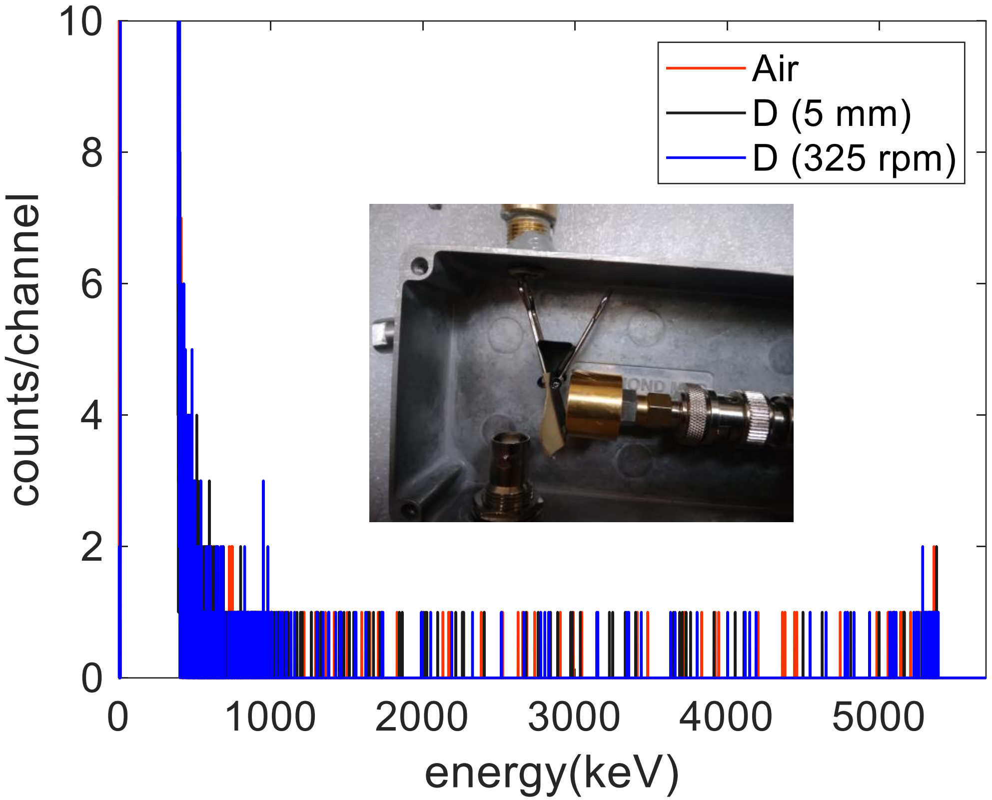
CHANGING WHAT'S POSSIBLE

Initial Results: Pd-ANF (5 mm and 325 rpm 2 %) with external ^{133}Ba source



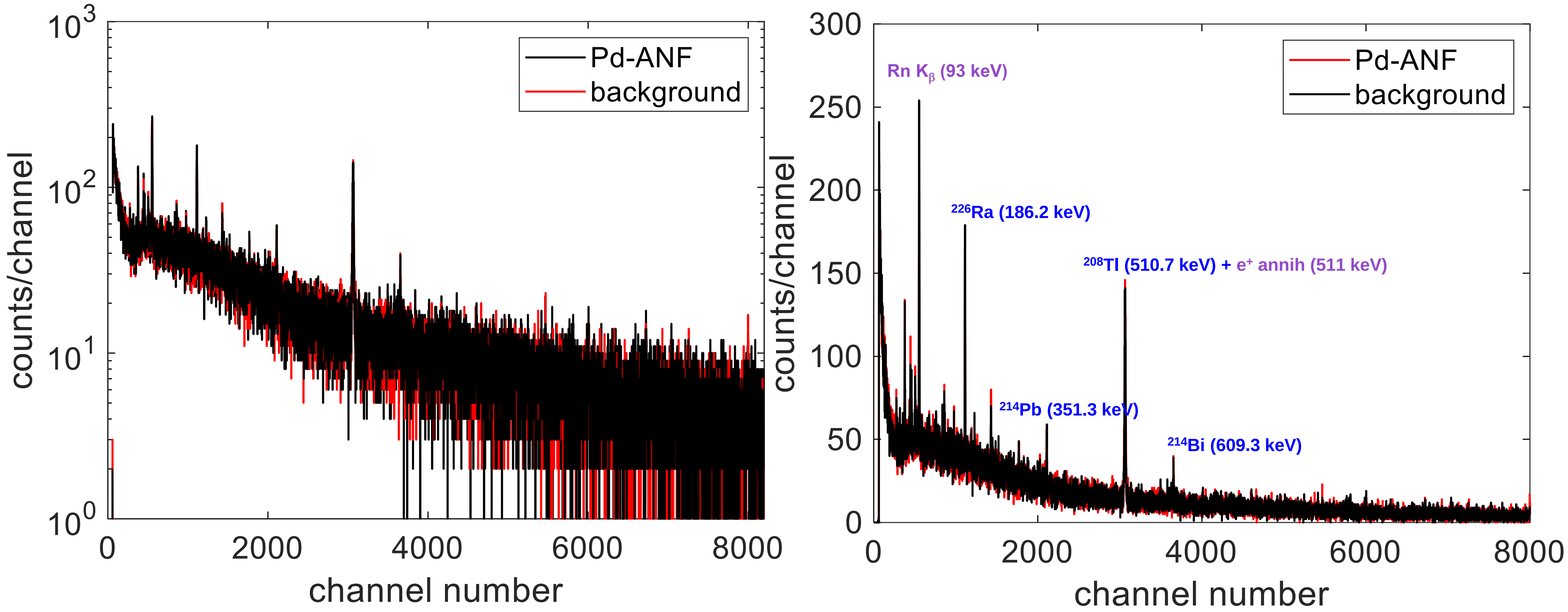




- Compare with more heavily Pd loaded, but much thinner sample.
- Note that attenuation induced by the spin-cast Pd-ANF is smaller than when mold-cast sample is present.
- ***Result:*** No evidence of MeV-scale ion creation.

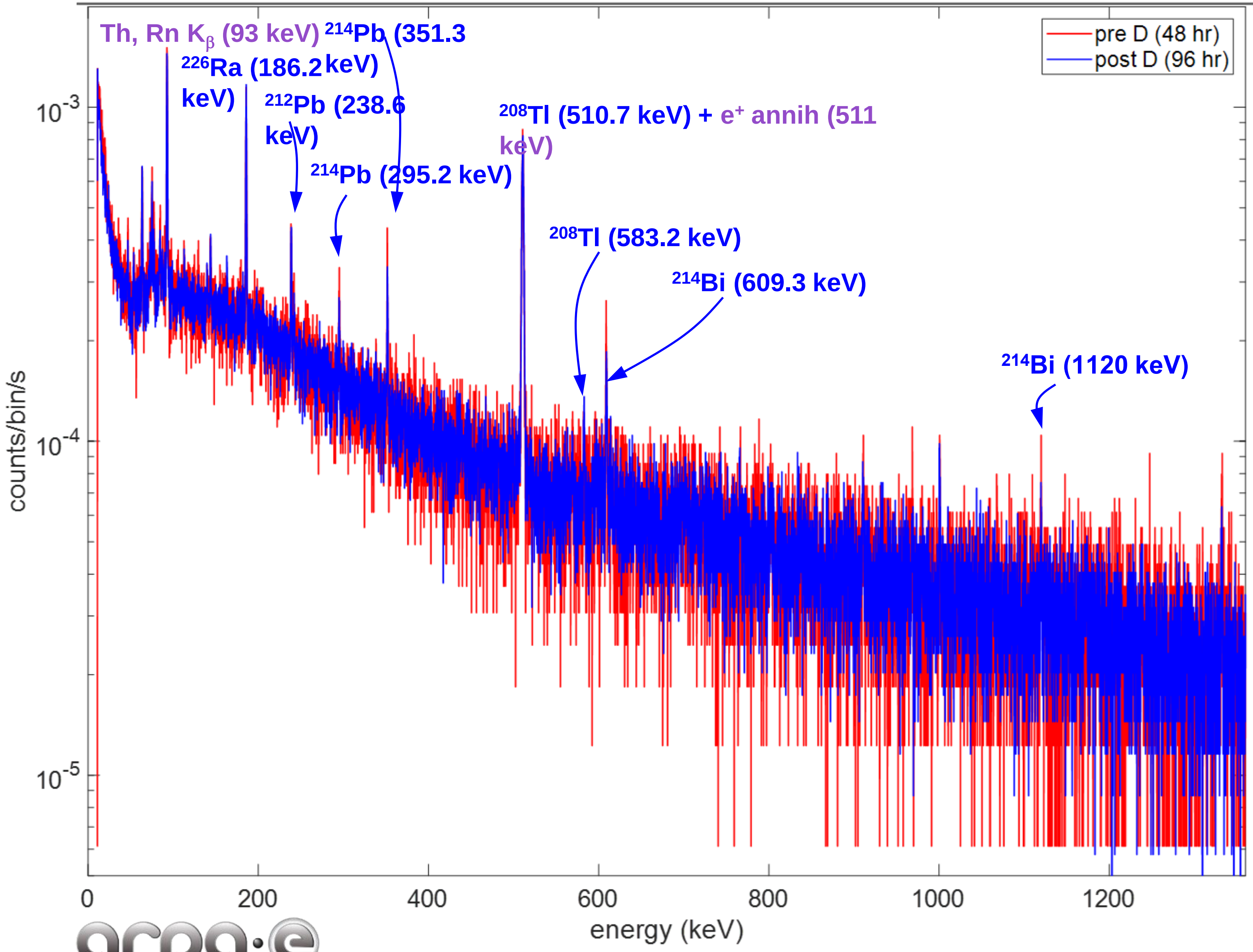
(Need to start modulating the D loading via pressure (using different chamber))

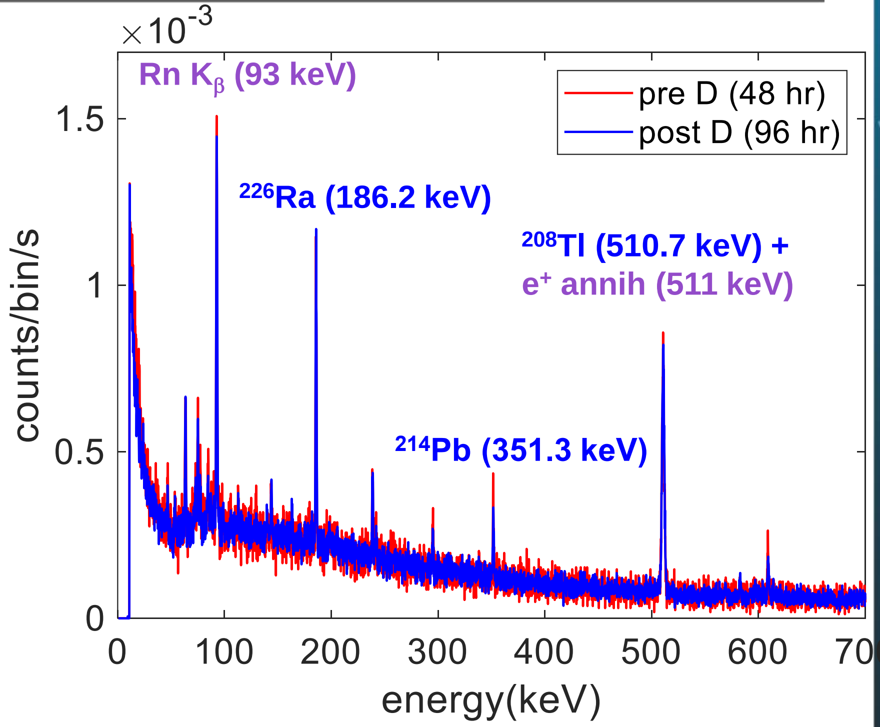
Initial Results: Initial Radioactivity of Pd-ANF (5 mm mold-cast sample)



- Assess the natural background within the Cu-lined Pb cylinder.
- Long-accumulations needed to observe lower intensity peaks in background (composed principally of uranium and thorium decay species).
- ***Result:*** *Pd nanostructured solids are not gamma-ray emitters (as expected)*

Initial Results: Pd-ANF 2 day and 4 day counts (pre D and post D measurements)





- The 4-day measurement (96 h) is smoother than the 2 day, but not too different => stick with 2 day for now.
- ***Result:*** *No induced gamma-ray radioactivity indicated.*

- What are the potential radioactive reaction products?

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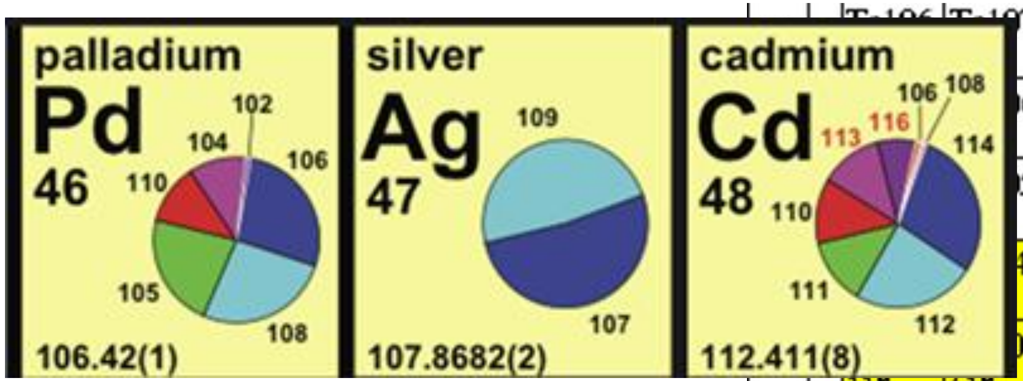
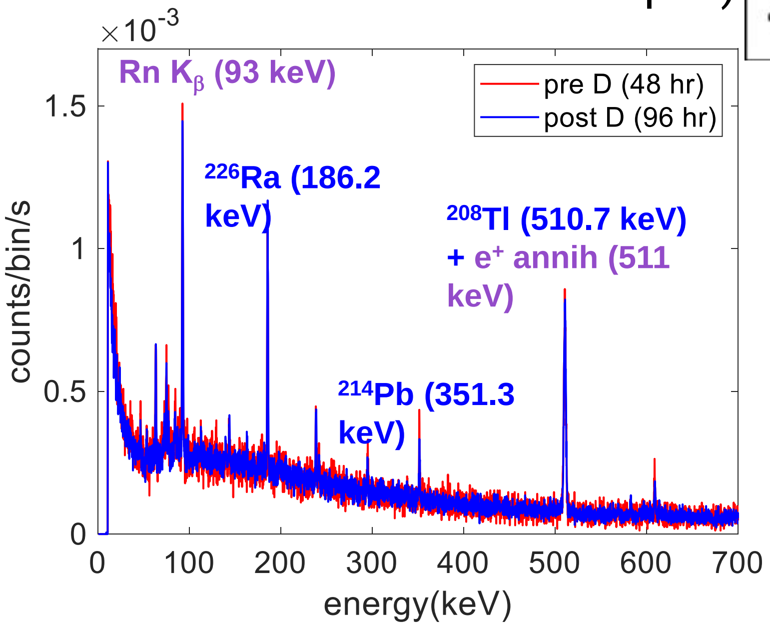
CHANGING WHAT'S POSSIBLE

Initial Results: Potential gamma-ray emitters if Pd fuses with D (or DD or DDDD)



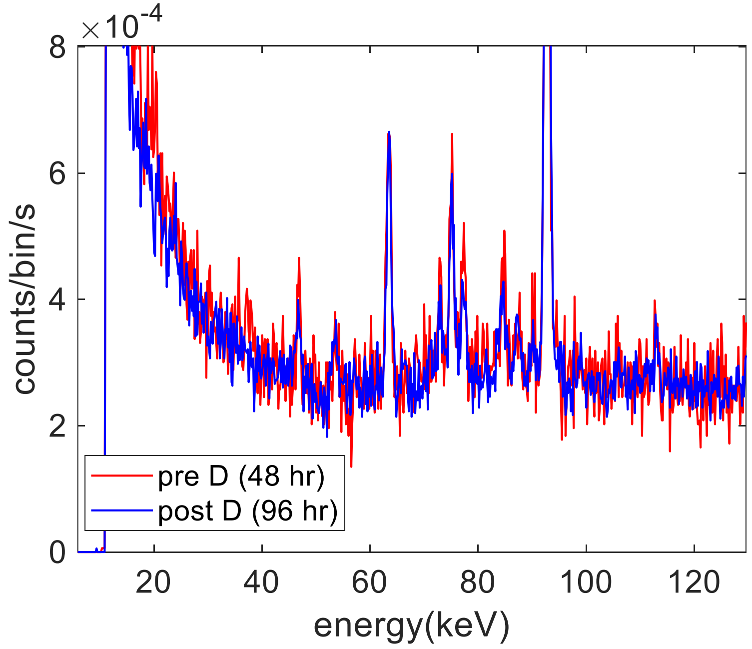
	Te106	Te107	Te108 2.1 S	Te109 4.6 S	Te110 18.6 S	Te111 19.3 S	Te112 2.0 M	Te113 1.7 M	Te114 15.2 M	Te115 5.8 M	Te116 2.49 H	Te117 62 M	Te118 6.00 D	Te119 16.03 H	Te120 0.09	Te121 19.16 D
			Sb107 4.6 S	Sb108 7.4 S	Sb109 17.0 S	Sb110 23.0 S	Sb111 75 S	Sb112 51.4 S	Sb113 6.67 M	Sb114 3.49 M	Sb115 32.1 M	Sb116 15.8 M	Sb117 2.80 H	Sb118 3.6 M	Sb119 38.19 H	Sb120 15.89 M
			Sn106 115 S	Sn107 2.90 M	Sn108 10.30 M	Sn109 18.0 M	Sn110 4.11 H	Sn111 35.3 M	Sn112 0.97	Sn113 115.09 D	Sn114 0.66	Sn115 0.34	Sn116 14.54	Sn117 7.68	Sn118 24.22	Sn119 8.59
			In105 5.07 M	In106 6.2 M	In107 32.4 M	In108 58.0 M	In109 4.2 H	In110 4.9 H	In111 2.8047 D	In112 14.97 M	In113 4.29	In114 71.9 S	In115 95.71	In116 14.10 S	In117 43.2 M	In118 5.0 S
			Cd104 57.7 M	Cd105 55.5 M	Cd106 1.25	Cd107 6.50 H	Cd108 0.89	Cd109 461.4 D	Cd110 12.49	Cd111 12.80	Cd112 24.13	Cd113 12.22	Cd114 28.73	Cd115 53.46 H	Cd116 7.49	Cd117 2.49 H
47	Ag101 11.1 M	Ag102 12.9 M	Ag103 65.7 M	Ag104 69.2 M	Ag105 41.29 D	Ag106 23.96 M	Ag107 51.839	Ag108 2.37 M	Ag109 48.161	Ag110 24.6 S	Ag111 7.45 D	Ag112 3.130 H	Ag113 5.37 H	Ag114 4.6 S	Ag115 20.0 M	Ag116 2.68 M
46	Pd100 3.63 D	Pd101 8.47 H	Pd102 1.02	Pd103 16.991 D	Pd104 11.14	Pd105 22.33	Pd106 27.33	Pd107 6500000 Y	Pd108 26.46	Pd109 13.7012 H	Pd110 11.72	Pd111 23.4 M	Pd112 21.03 H	Pd113 93 S	Pd114 2.42 M	Pd115 25 S

Pd is not isotope pure. (blue boxes are various *stable* isotopes)

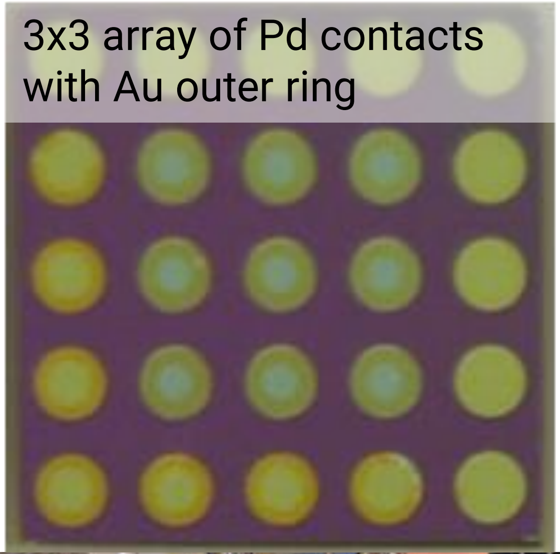


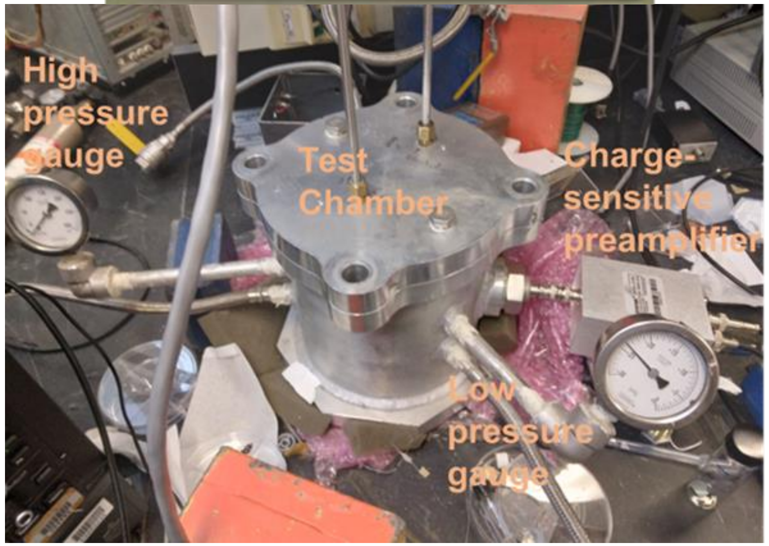
^{109}Cd 88.0 keV ($t_{1/2}$= 461 days)
^{110}Ag 657.76 keV ($t_{1/2}$= 24.56 s)
^{111}Ag 342.13 keV ($t_{1/2}$= 7.45 days)
^{111}In 171.28, 45.35 keV ($t_{1/2}$= 2.80 days)
^{113}Sn 391.69 keV ($t_{1/2}$= 115 days)
daughter x-rays (e.g. 23 – 24 keV)

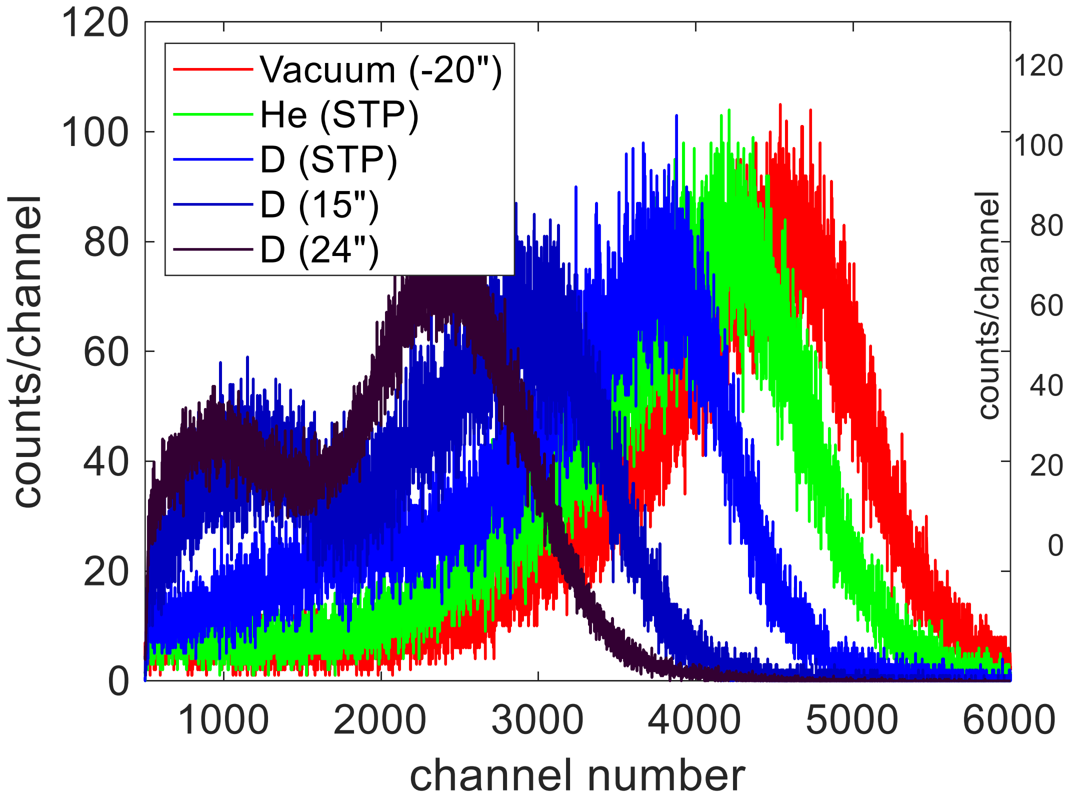
Conclusion: There are possible product isotopes that have suitable half-lives (not millions of years or microseconds) that they can be measured.

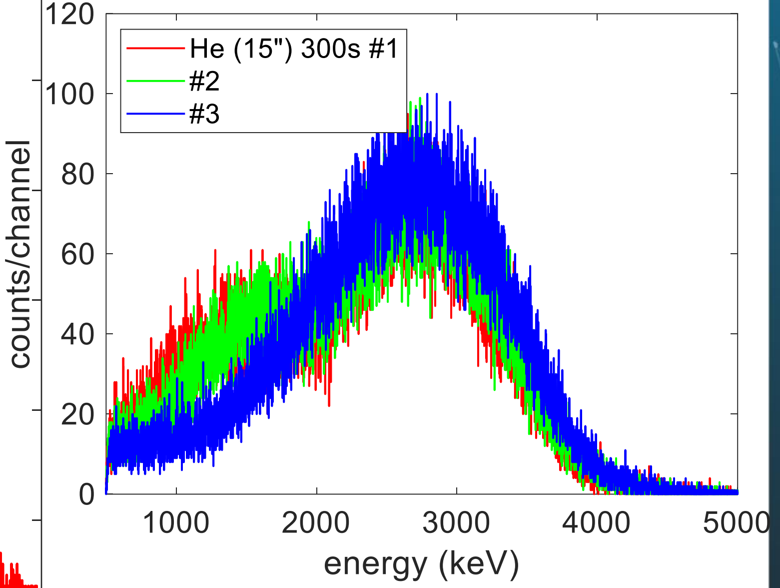


Initial Results: Evaporated Pd contact on Si PIN detector, ^{241}Am









- ***Result:***

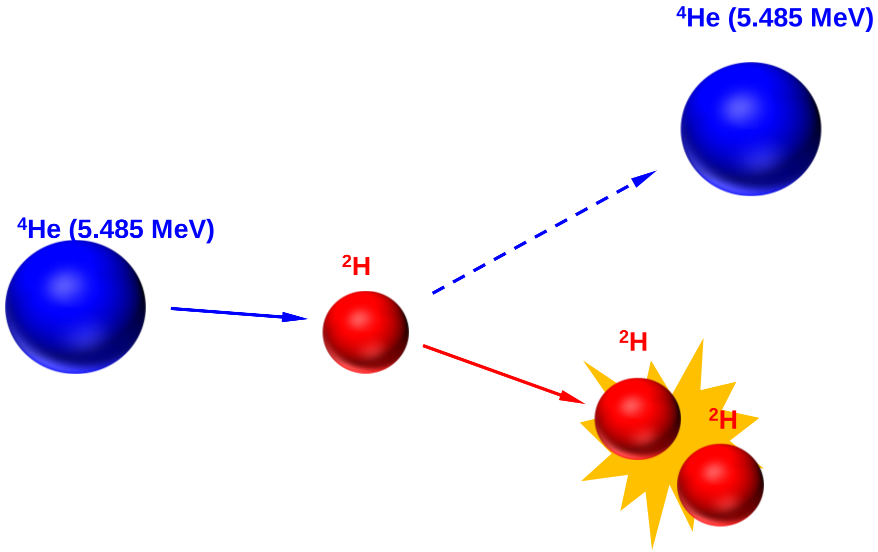
  1) Secondary MeV-scale ion creation only in the presence of D, but the position does change with the fill gas pressure.

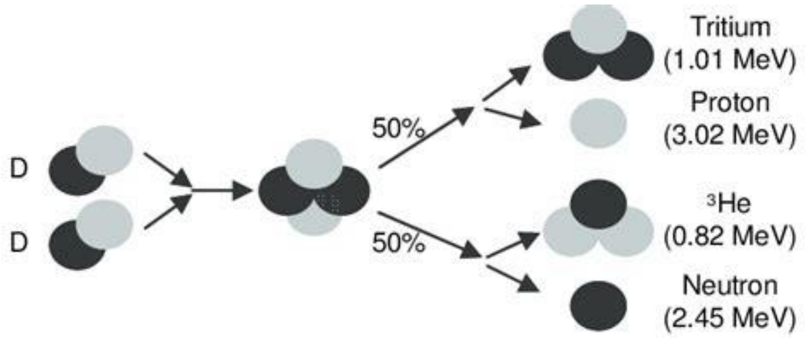
  2) Interestingly, the feature does slowly disappear after D replaced with He (D outgasing from Pd?)

  => alpha-induced DD fusion is present.

  (so we can identify DD fusion…. Just have to get rid of the alpha source)

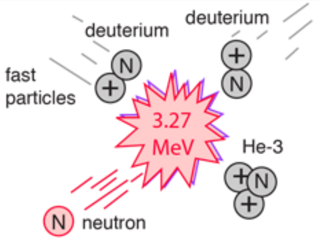
Initial Results: Alpha-induced DD fusion



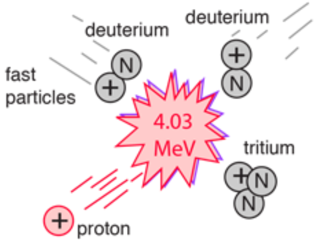


$${}_1^2H + {}_1^2H \rightarrow {}_2^3He + {}_0^1n + 3.27\,MeV$$

Deuterium-deuterium Fusion

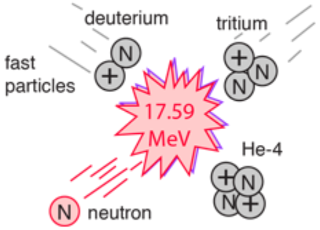


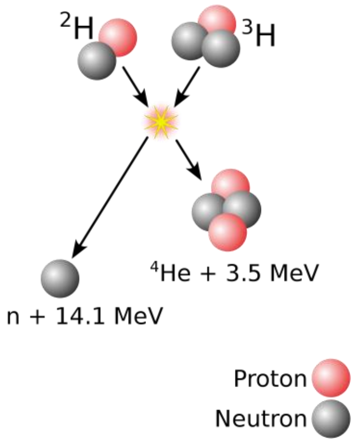
$${}_1^2H + {}_1^2H \rightarrow {}_1^3H + {}_1^1H + 4.03\,MeV$$



$${}_1^2H + {}_1^3H \rightarrow {}_2^4He + {}_0^1n + 17.59\,MeV$$

Deuterium-tritium Fusion





Observed feature is near 1 MeV and 3 MeV.

~3 MeV can be caused by: (a) ^{1}H from DD fusion (3.02 MeV) or
(b) ^{4}He from DT fusion (3.5 MeV)

~1 MeV can be caused by: (a) ^{3}H from DD fusion (1.01 MeV) or
(b) ^{3}He from DD fusion (0.82 MeV)

Neutron will generally escape the gas chamber.

=> Calibration of chamber before the experiment with ^{133}Ba, ^{60}Co, ^{137}Cs

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CHANGING WHAT'S POSSIBLE

Plans for Next Quarter

- Assembly of new 4” and 6” diameter cylindrical testing chamber, with feed-throughs to add thermal sensors, 4-point probe resistance measurement, pressure sensors, radiation sensors, and connections to collect post-reaction gas for measurement by mass spectroscopy
- Full validation of new detector setup, with temporal and energy characteristics established
- Prepare Pd-ANF with varying NP size and geometry, and test with D_2 loading
- Investigate preparing core/shell Pd/Cu NPs
- Investigating electrolytic D_2O loading using Pd-ANF as cathode
- Continue to search for indications of LENR