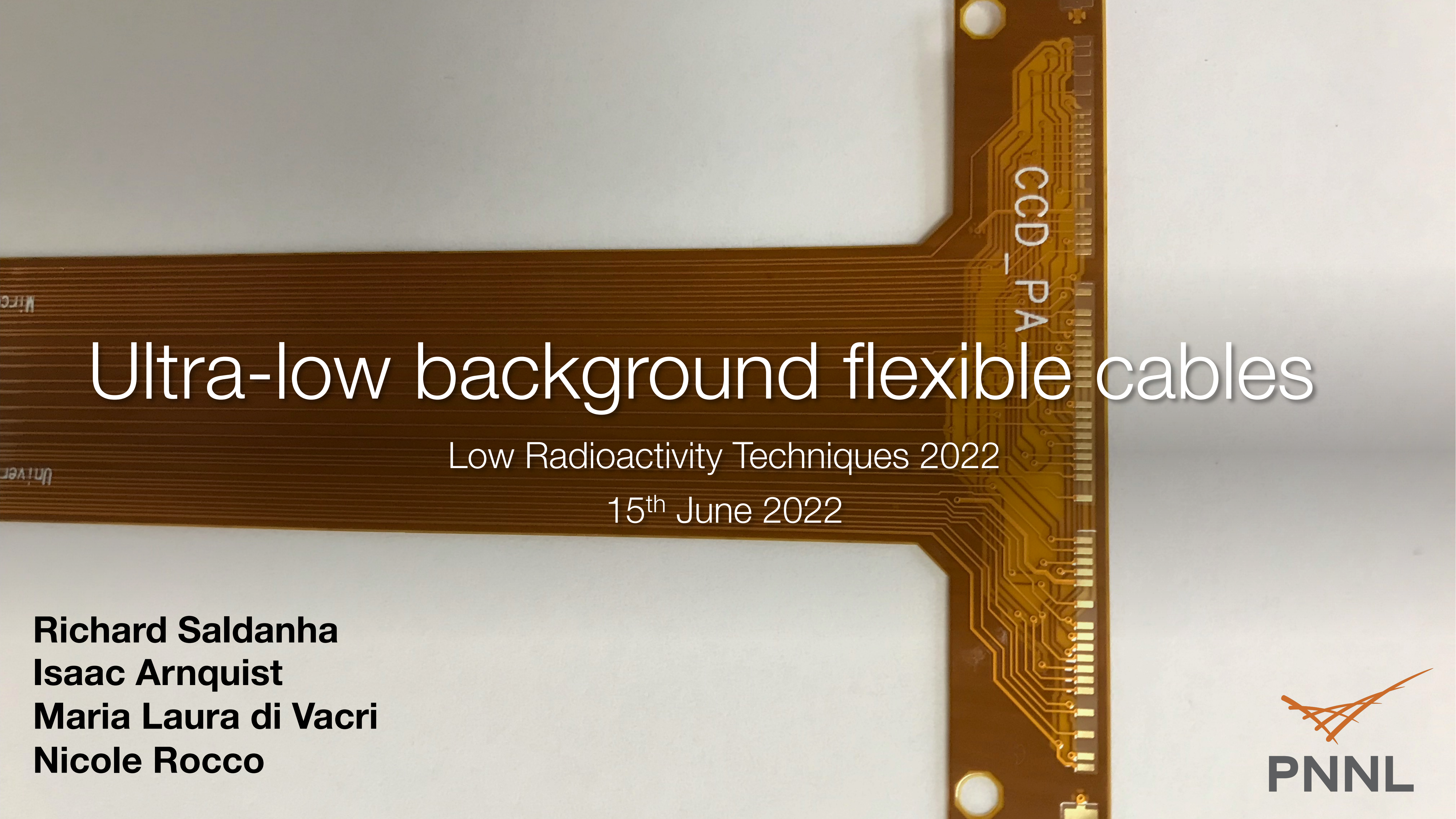

Ultra-low background flexible cables

Low Radioactivity Techniques 2022

15th June 2022

Richard Saldanha
Isaac Arnquist
Maria Laura di Vacri
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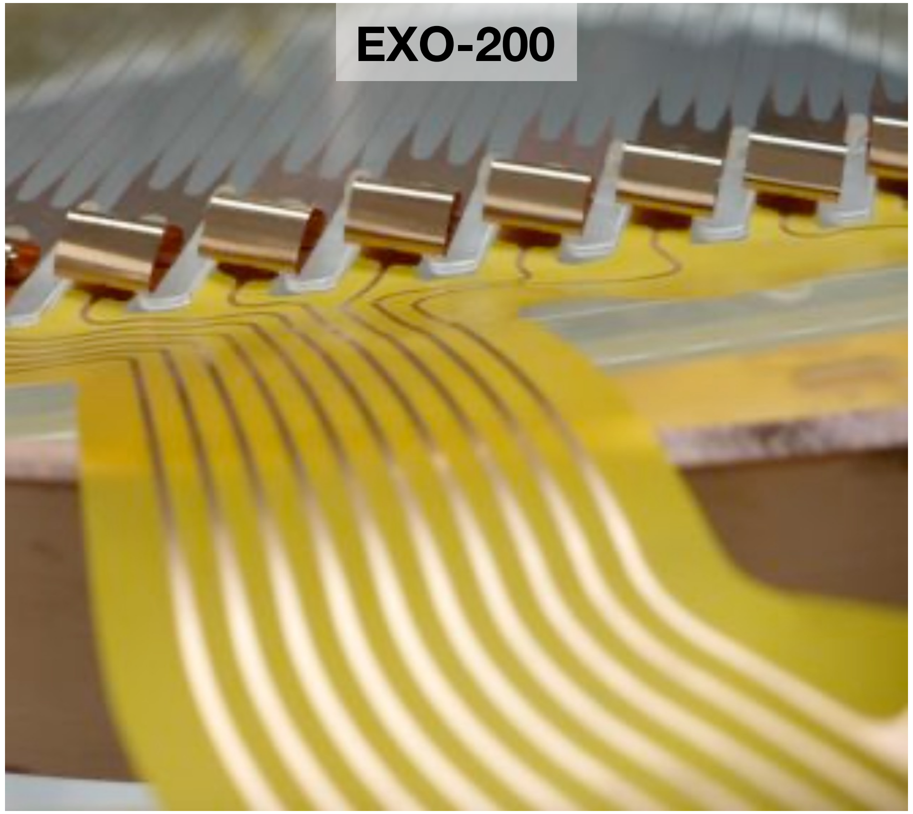
PNNL

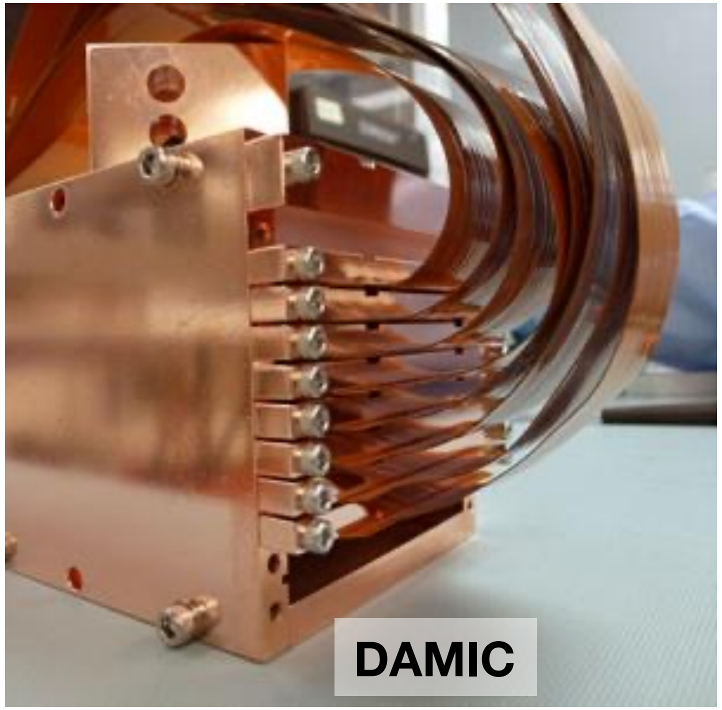
Flexible Cables and Circuitry for Rare Event Searches

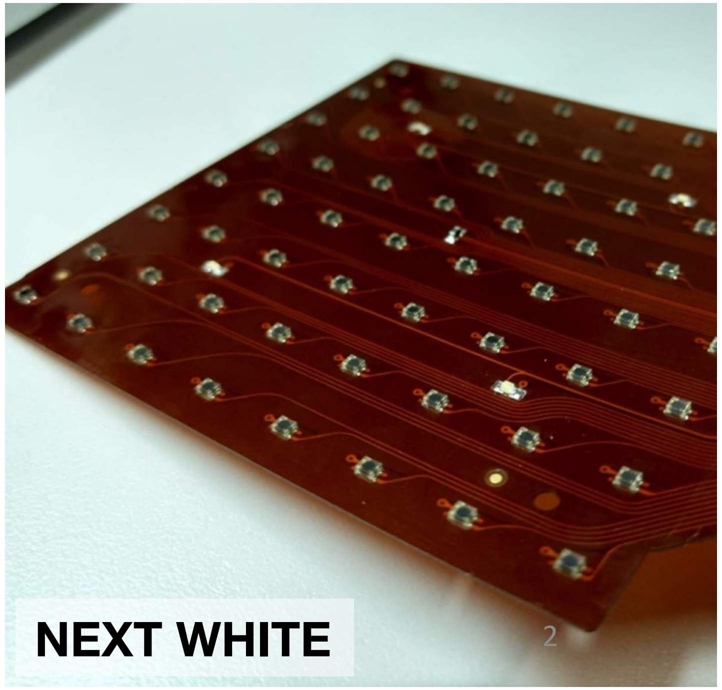
- Flat flexible polyimide-copper cables have several advantages for reading out sensors in low background experiments

- Large number of signal channels requires significant amount of cabling

- Commercial options are not very clean (~ 1000 ppt ^{238}U) leading to large contributions to the total background budget

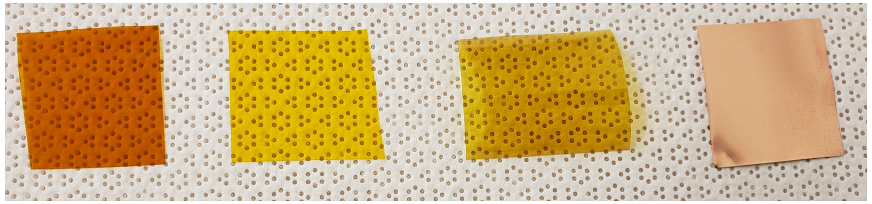






Low Background Polyimide

- Commercial Kapton is not very clean - typically 1000 ppt ^{238}U
- Collaboration with DuPont and study of the manufacturing process led us to identify the dominant source of contamination in Kapton material: DCP a slip additive used to improve processing yield
- Special production of DCP-less Kapton and Kapton-copper laminates showed significant reduction in radiocontamination
- Demonstrated ability to produce Kapton-copper laminates (starting material for all flexible cables) with ~20x lower radioactivity



	^{238}U [pg/g]	^{232}Th [pg/g]	^{40}K [ng/g]
Commercial Kapton HN	1082 +/- 43	249.6 +/- 8.5	44 +/- 18
Radiopure R&D Kapton	12.3 +/- 1.9	18.5 +/- 2.3	34 +/- 14

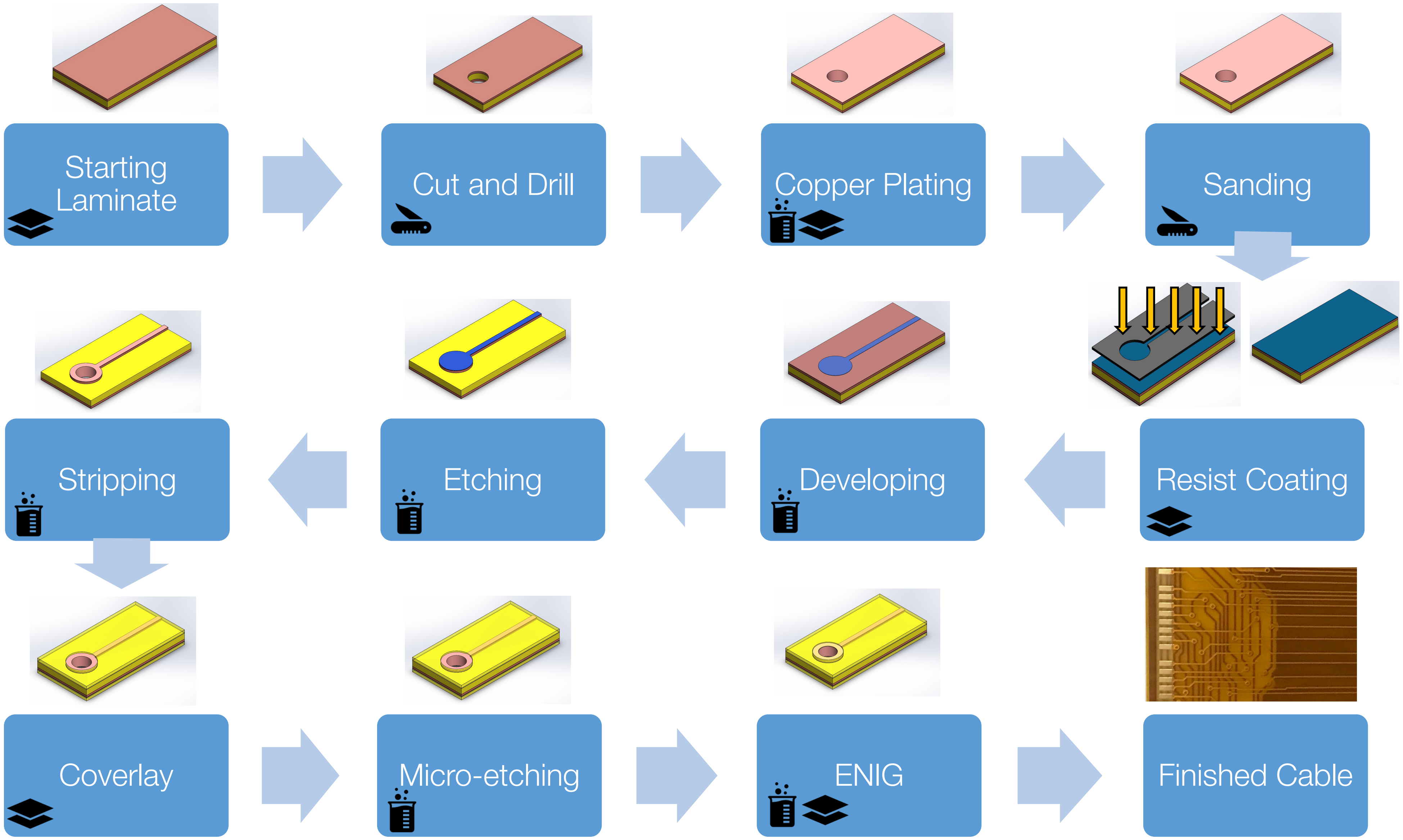
	^{238}U [pg/g]	^{232}Th [pg/g]	^{40}K [ng/g]
Commercial Kapton-Cu Laminate	158.0 +/- 6.1	24.1 +/- 0.9	< 210
Radiopure Kapton-Cu Laminate	8.6 +/- 3.6	20 +/- 14	164 +/- 82

Ultra-low radioactivity Kapton and copper-Kapton laminates

Isaac J. Arnquist *, Chelsie Beck, Maria Laura di Vacri, Khadouja Harouaka, Richard Saldanha *
Pacific Northwest National Laboratory, Richland, WA, 99352, USA

NIM A 959 (2020): 163573

Flexible Cable Fabrication



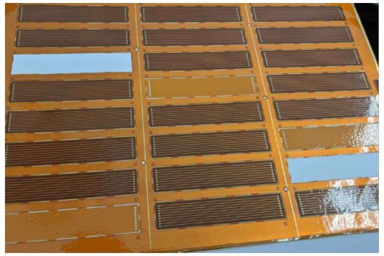
- Even when starting with clean (~ 10 ppt U/Th) materials, fabrication of cables introduces large contamination, resulting in cables that have contamination levels that are typically 1000-5000 ppt U/Th
- Process includes use of several chemicals, addition of materials, and mechanical handling
- Anyone of these steps could introduce radioactive contaminants

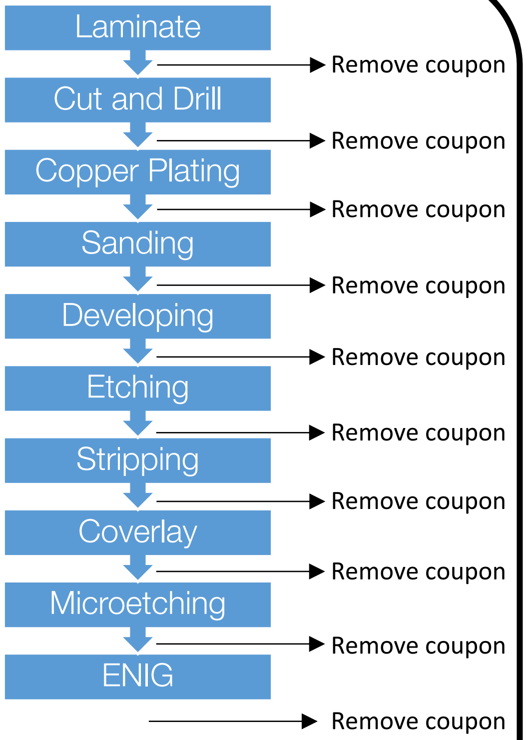
Investigative Method

Q-FLEX INC.

SBIR · STTR America's Seed Fund

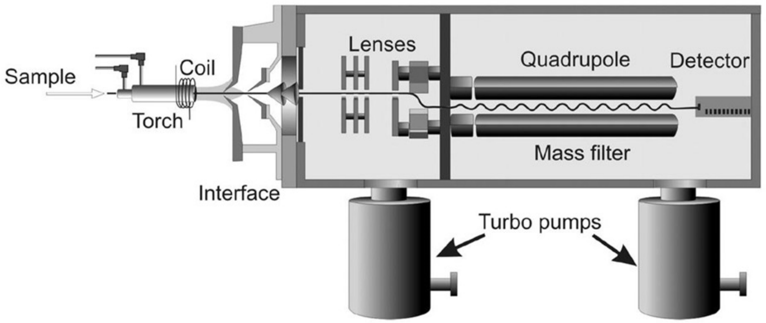
- Worked with commercial manufacturer Q-Flex Inc.
- Experts in custom flexible cables
- Received SBIR Phase 1 award to investigate feasibility of making radiopure flexible cables





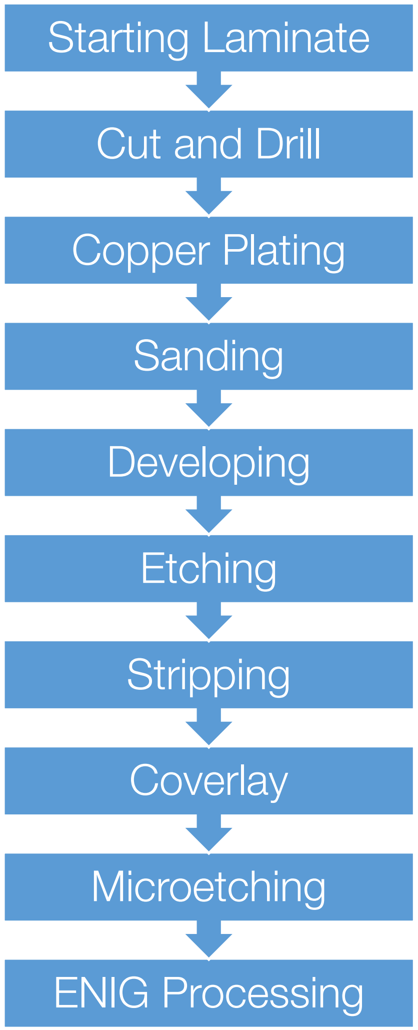
- Developed method to systematically investigate fabrication process using detachable coupons
- Allows sampling of process after each step
- Coupons can be used in parallel with cable production to act as witness sample for final cable production

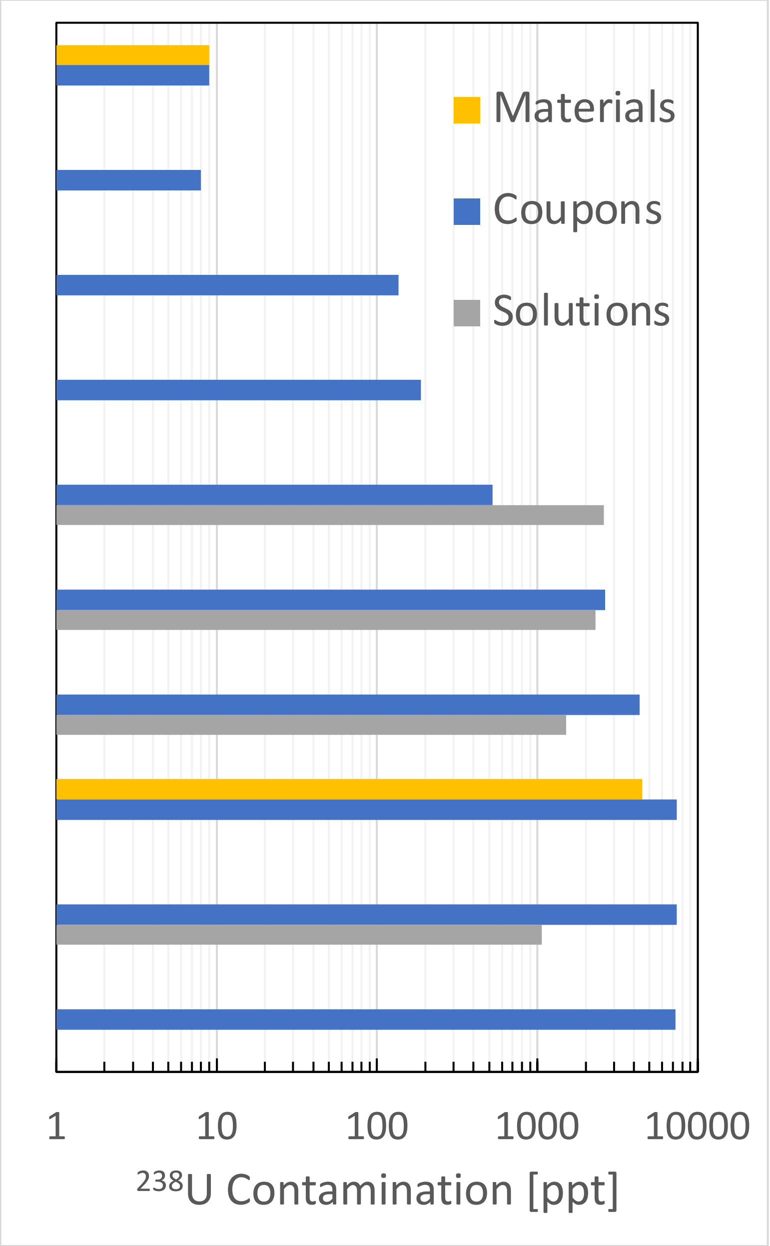
PNNL

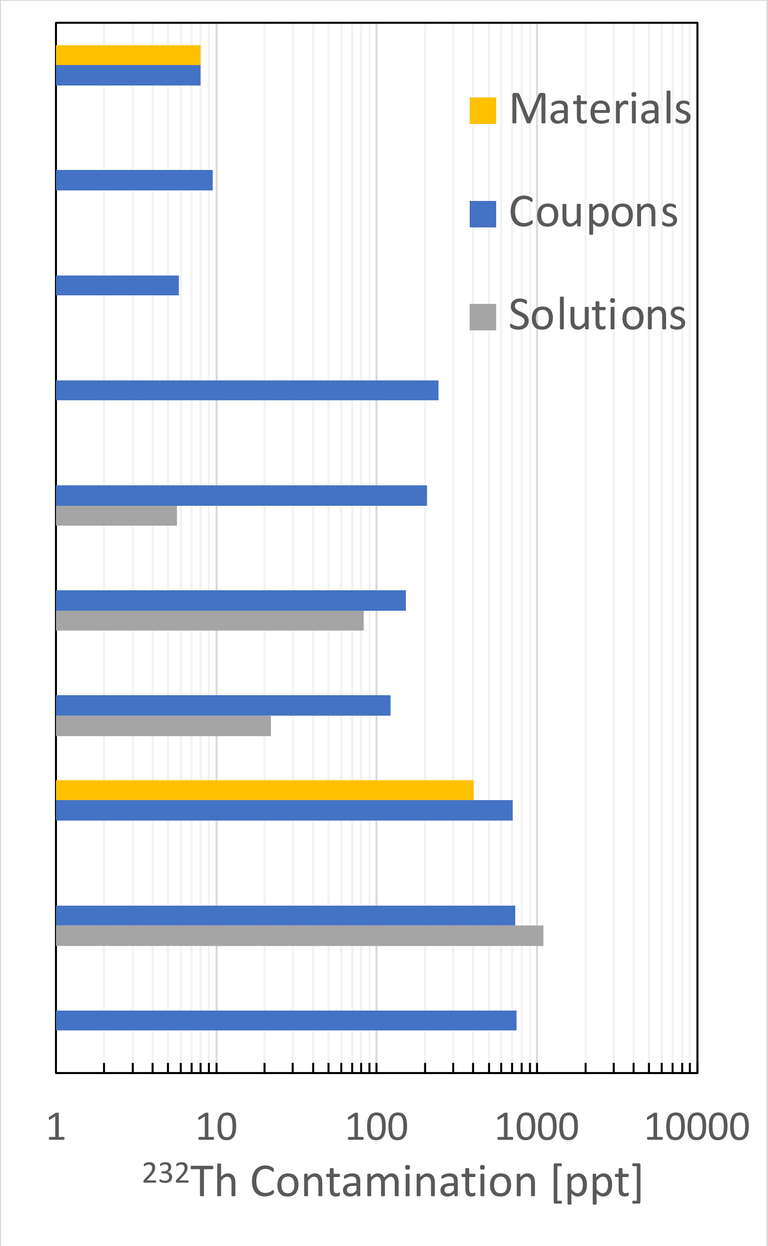


- Each coupon was analyzed using ICP-MS
- Requires small quantities and quick turnaround – ideal for investigative analysis
- PNNL chemists can obtain better than parts-per-trillion sensitivity to ^{238}U and ^{232}Th

Contamination during Fabrication



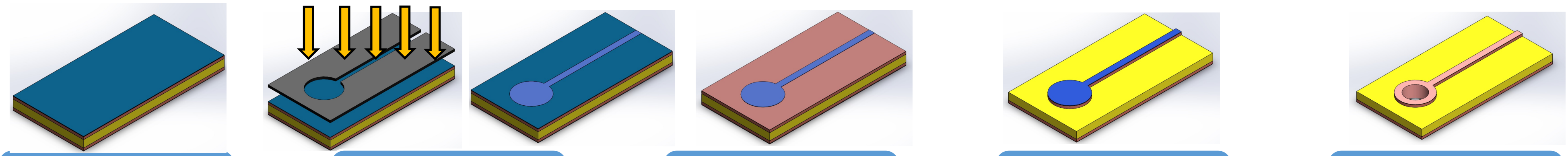




- Performed systematic assay of contamination level at each step, as well as measuring any solutions used in the process and materials added
- Final contamination levels are ~ 7000 ppt ^{238}U and ~700 ppt ^{232}Th
- Realized that there are several steps with significant increases in contamination
- Need different approach to address each issue

Photolithography

- Laminate
- Cut and Drill
- Copper Plating
- Sanding
- Developing
- Etching
- Stripping
- Coverlay
- Microetching
- ENIG



Resist Coating → Exposure → Developing → Etching → Stripping

Resist Coating: DuPont Riston MM540
Heat and Rollers

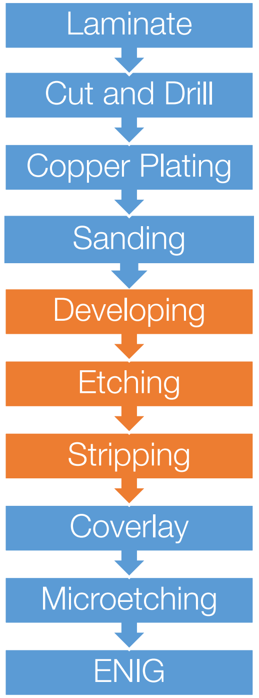
Developing: Soda Ash (Na_2CO_3)
Tap water rinse

Etching: Hydrochloric Acid (HCl)
Elevated temperature

Stripping: Rstrip 35
- Potassium Hydroxide
- Monoethanolamine
- Ethylenediamine
- Diethanolamine

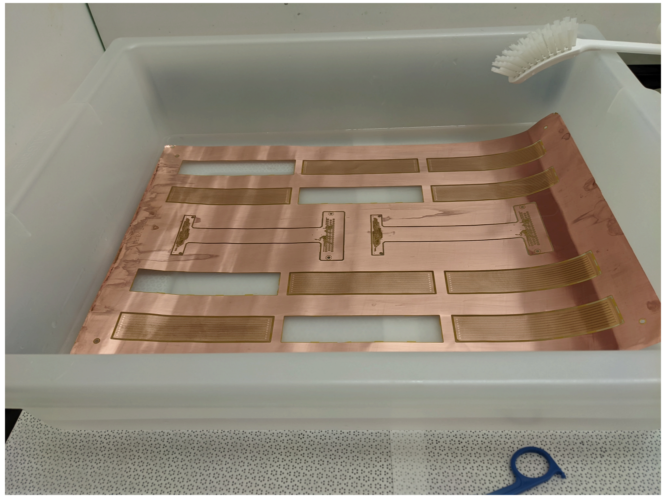
- Several chemicals involved in the photolithography
- Includes several chemicals (soda ash, potassium hydroxide) that are likely to be highly contaminated
- Crucially: the overall process is subtractive, and no surface is covered at the end of the steps

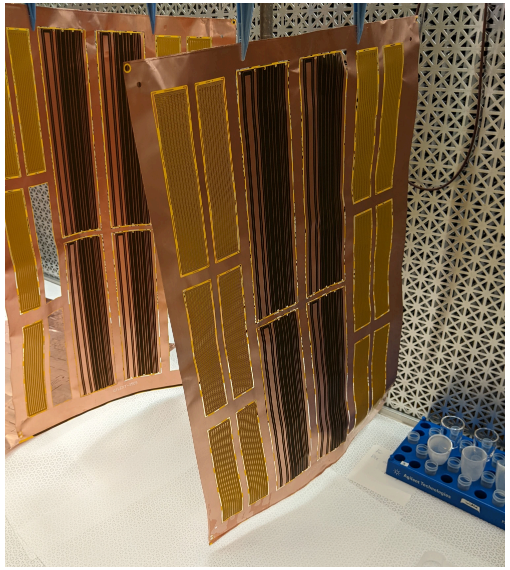
Cleaning

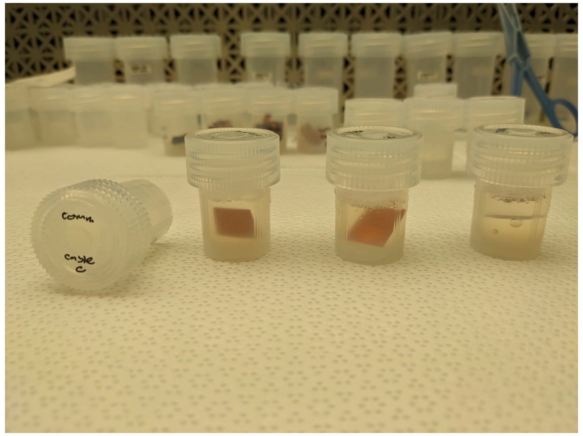


Final Cleaning Recipe (combination of basic and acidic rinse):

- Submerge in 5% v/v **TMAH** for 15 min
- Brush surface
- Submerge in DIW, rinse at sink
- Submerge in 2% **nitric acid** for 1 min
- Brush surface
- Submerge in DIW, rinse at sink
- Repeat







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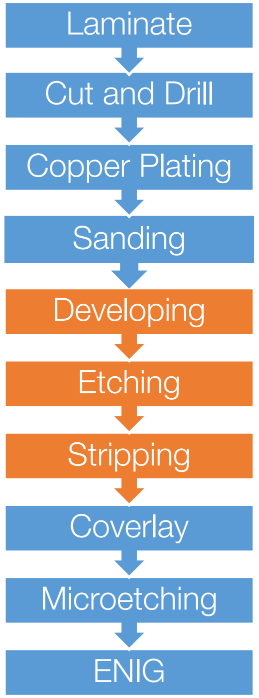
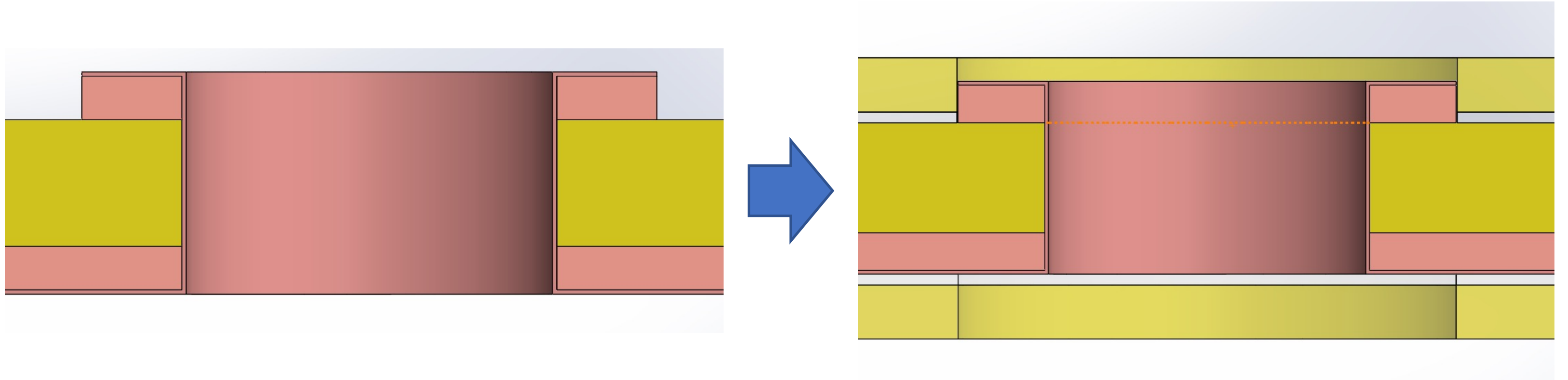
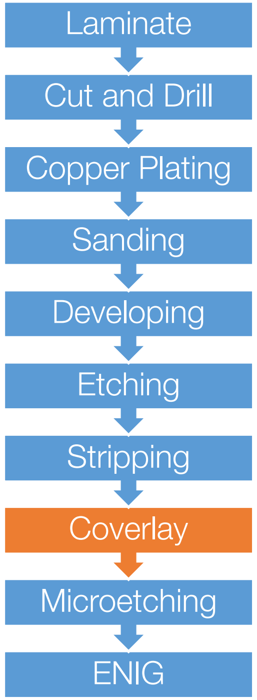


	^{238}U [ppt]	^{232}Th [ppt]
After Stripping Before Cleaning	6000 +/- 200	27 +/- 3
After Stripping After Cleaning	20 +/- 1.3	< 9.3

Cleaning brings contamination back down to the tens of ppt levels

Coverlays





A coverlay is an insulating layer that is applied over the outer surfaces of a cable to prevent oxidation and shorting of the exposed traces.

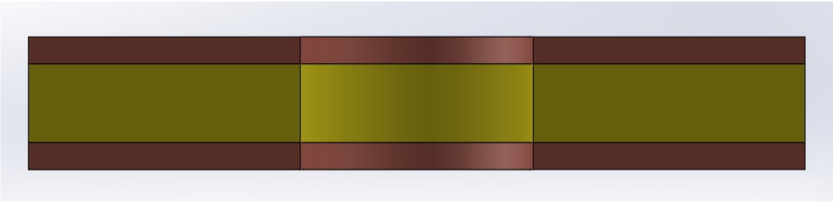
Typically consists of a polyimide and adhesive layer

We surveyed several commercially available coverlays and found a fairly large range of contamination levels (> 100x variation in ^{238}U).

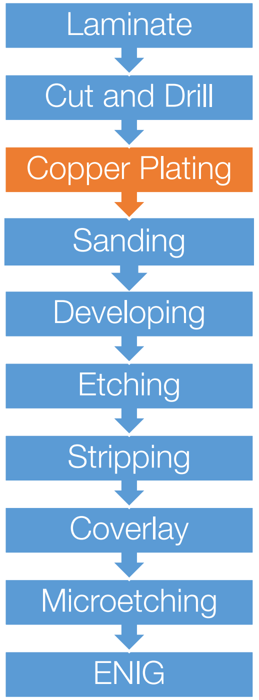
Acrylic-based adhesives were noticeably cleaner than epoxy-based adhesives.

Sample	PI Thick. [mil]	Adh. Thick. [mil]	Notes	^{238}U [ppt]	^{232}Th [ppt]
Taiflex FHK1025	1	1	PI + B-stage epoxy	$18\,000 \pm 2000$	1600 ± 140
ShinEtsu CA 333 [EXO-200]	1	1	PI + epoxy adhesive	5179 ± 424	< 242
ShinEtsu CA 335 [EXO-200]	1	1.4	PI + epoxy adhesive	$12\,020 \pm 390$	9370 ± 340
Dupont LF0110	1	1	PI + acrylic adhesive	314 ± 4	49 ± 4
Upilex C120	2	1	PI + acrylic adhesive	30.0 ± 0.4	280 ± 20
Panasonic MCL Plus 110	1	1	PI + B-stage acrylic adhesive	78 ± 2	45 ± 7
Dupont FR 70001 [EXO-200]	0.5	0.5	PI + acrylic adhesive	< 1065	< 473
Dupont FR 0110 [EXO-200]	1	1	PI + acrylic adhesive	< 818	< 273
Dupont LF0100	0	1	Acrylic adhesive used in LF0110	16 ± 4	39 ± 11
Imitex MI-100	0	1	Adhesive only	9 ± 5	< 14

Copper Plating

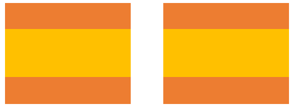


Interconnections between layers (vias) need to be plated with copper



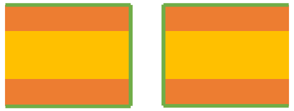
STANDARD ELECTROLESS PROCESS

Laminate + drilling + scrubbing

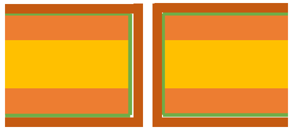


Pre-cleaning		Post-clean (PNNL)	
U [ppt]	Th [ppt]	U [ppt]	Th [ppt]
8 ± 2	9 ± 2	< 7	< 23

Electroless seed



Electroplating

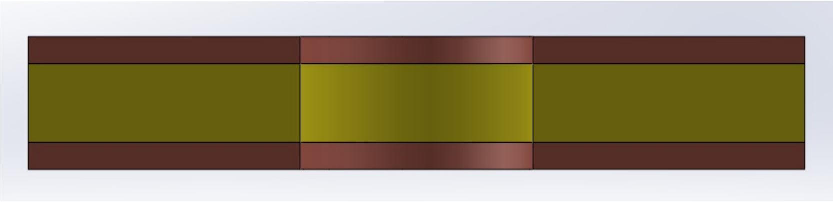


Pre-cleaning		Post-clean (PNNL)	
U [ppt]	Th [ppt]	U [ppt]	Th [ppt]
113 ± 9	< 25	45 ± 4	< 25

This process involves several solutions and catalysts, and both the seed and plating layers cover the entire copper surface, potentially trapping contamination

We were unable to reduce the contamination below ~ 50 ppt U through cleaning

Copper Plating

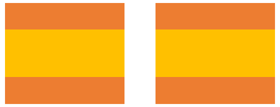


Interconnections between layers (vias) need to be plated with copper

- Laminate
- Cut and Drill
- **Copper Plating**
- Sanding
- Developing
- Etching
- Stripping
- Coverlay
- Microetching
- ENIG

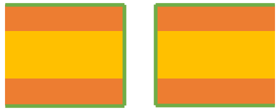
STANDARD ELECTROLESS PROCESS

Laminate + drilling + scrubbing

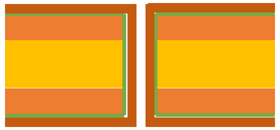


Pre-cleaning		Post-clean (PNNL)	
U [ppt]	Th [ppt]	U [ppt]	Th [ppt]
8 ± 2	9 ± 2	< 7	< 23

Electroless seed



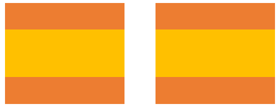
Electroplating



Pre-cleaning		Post-clean (PNNL)	
U [ppt]	Th [ppt]	U [ppt]	Th [ppt]
113 ± 9	< 25	45 ± 4	< 25

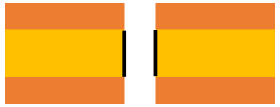
SHADOW PLATING PROCESS

Laminate + drilling + scrubbing

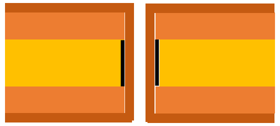


Pre-cleaning		Post-clean (PNNL)	
U [ppt]	Th [ppt]	U [ppt]	Th [ppt]
21 ± 6	12.3 ± 1.5	10.9 ± 0.1	6 ± 5

Shadow plating



Electroplating



Pre-cleaning		Post-clean (PNNL)	
U [ppt]	Th [ppt]	U [ppt]	Th [ppt]
90 ± 10	9.3 ± 1.0	7.6 ± 0.5	7 ± 3

A newer alternative process to the electroless Cu step is the “shadow” process where a thin carbon layer is added only to the polyimide region and involves fewer chemicals

The resulting coupons had contamination levels consistent with the base laminate level, **roughly 6x cleaner in ^{238}U than the electroless seed process**

Sanding

- Laminate
- Cut and Drill
- Copper Plating
- **Sanding**
- Developing
- Etching
- Stripping
- Coverlay
- Microetching
- Processing

- Prior to the application of photoresist, the cable surface is mechanically prepared for optimal film adhesion and clean release.
- The scrubbing process was found to increase ^{232}Th contamination, presumably due to the implantation of small amounts of the abrasive material into the laminate.
- Cleaning was tried but found ineffective
- Switched to only using commercial pads made from SiC, rather than previously used pads that used aluminum oxide, titanium dioxide, and other fillers and pigments.
- **This led to roughly a 10x reduction in ^{232}Th contamination after this step**

Ingredient	% by Wt
Aluminum Oxide Mineral	30-45
Nylon Fiber	20-30
Filler	5-10
Pigment	0-2.5
Titanium Dioxide	0-2.5
Silica	0-2.5
Cured Resin	20-30

Ingredient	% by Wt
Silicon Carbide Mineral	25-40
Nylon Fiber	20-35
Quartz Silica	0.05-0.2
Cured Resin	30-45
Hookit™ Backing	0-10

1. Laminate Selection
2. Cut Laminate
3. Sanding
4. Cleaning at QFlex
5. Resist Coating
6. Developing
7. Etching
8. Stripping
9. Drying
10. Cleaning at PNNL

Blue: Standard Step
Orange Outline: Modified Step
Orange: New Step
Green: Step done at PNNL

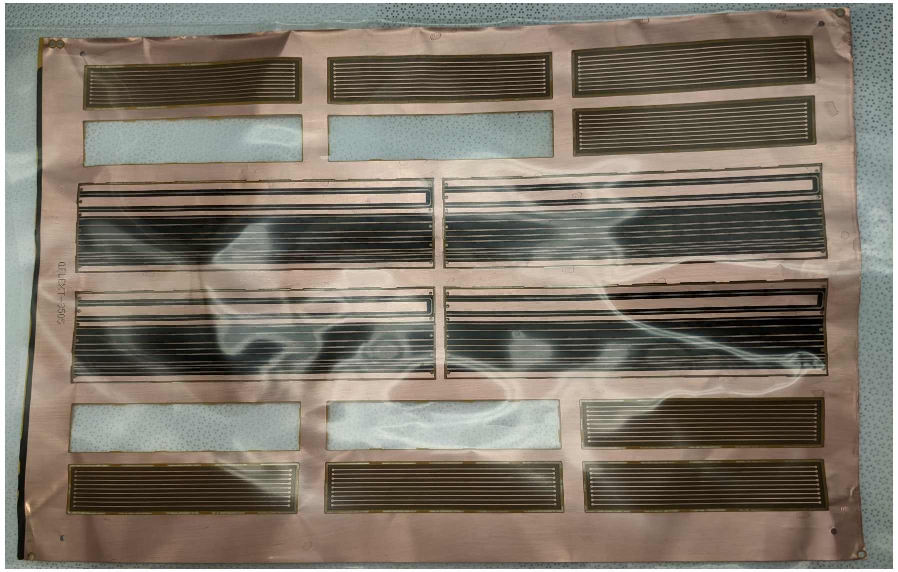
Simple 2-layer Cable

In the baseline design for nEXO, we only need a very simple 2-layer cable, with all the traces on one side and a ground plane on the other

Instead of coverlays, the baseline design has separate insulating layers mechanically placed between different cables to prevent electrical contact

These cables therefore do not need electroplated vias or coverlays, which helps reduce the radiocontamination

Combining all the improvements we have developed, we fabricated a baseline nEXO cable



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Combining all the improvements we have developed, we made a baseline nEXO cable

	^{238}U [ppt]	^{232}Th [ppt]
Starting Laminate	8 +/- 6	9 +/- 4
Standard Cable Trial 1 Standard Cable Trial 2	6200 +/- 100 1300 +/- 300	63 +/- 5 16 +/- 6
Our Final Cable	20 +/- 1	< 9.3

We have managed to reduce the ^{238}U contamination by > 65x

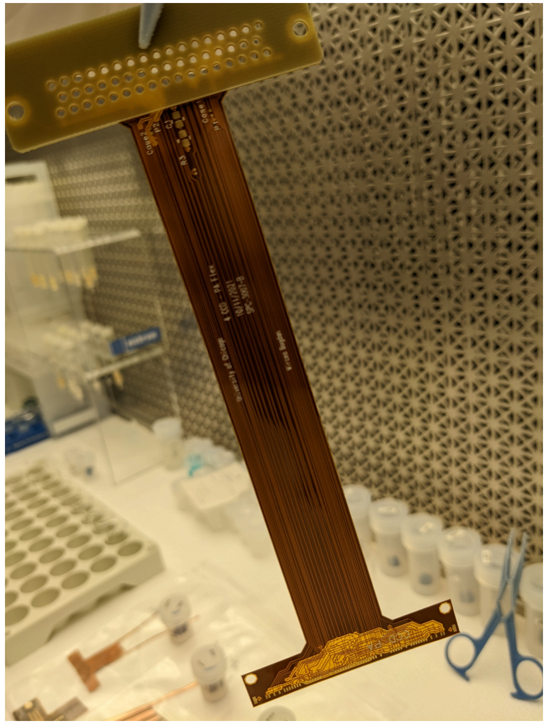
1. Laminate Selection
2. Cut and Drill Laminate
3. Cleaning at QFlex
4. Shadow Seeding
5. Electroplating
6. Sanding
7. Cleaning at PNNL
8. Resist Coating
9. Developing
10. Etching
11. Stripping
12. Drying
13. Cleaning at PNNL
14. Coverlay Application
15. Microetching
16. ENIG Processing
17. Cleaning at PNNL

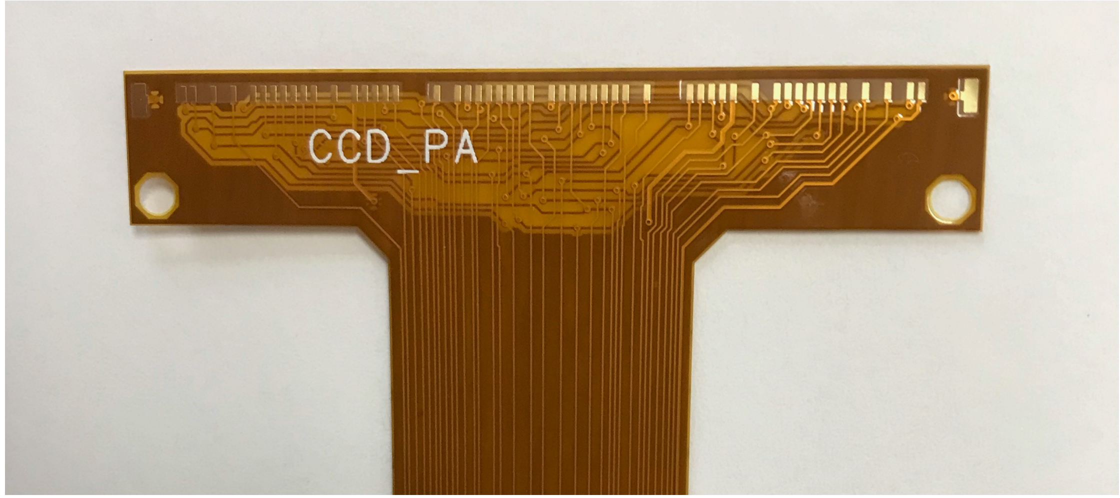
Blue: Standard Step
Orange Outline: Modified Step
Orange: New Step
Green: Step done at PNNL

Full 2-layer cable

CCD cables typically require a large number of traces, and interconnected layers

We used the design for a 2-layer DAMIC-M CCD cable with vias and coverlay to evaluate how clean we could get it compared to a commercially fabricated cable by another company with no modification to standard materials or process





DAMIC–M 2-layer CCD cable

1. Laminate Selection
2. Cut and Drill Laminate
3. Cleaning at QFlex
4. Shadow Seeding
5. Electroplating
6. Sanding
7. Cleaning at PNNL
8. Resist Coating
9. Developing
10. Etching
11. Stripping
12. Drying
13. Cleaning at PNNL
14. Coverlay Application
15. Microetching
16. ENIG Processing
17. Cleaning at PNNL

Blue: Standard Step
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	^{238}U [ppt]	^{232}Th [ppt]
Commercial Cable	2670 +/- 30	270 +/- 60
Our Cable	31 +/- 1	11 +/- 1

We have managed to reduce the ^{238}U contamination by ~100x

Even with the addition of vias, coverlays, and ENIG, the U and Th contamination levels are at ~10's of ppt

Summary

- Commercial flexible cable options are very radioactive (~1000's of ppt ^{238}U, ~100's of ppt ^{232}Th), limiting the use of cables in experiments

- By working closely with a commercial company and systematically investigating the fabrication process, we have identified the key sources of contamination

- Following a diverse approach of developing new cleaning steps, modifying fabrication processes, identifying radiopure raw material, and improving mechanical handling, we have **reduced the U and Th backgrounds to the level of ~ 10's of ppt ^{238}U and ^{232}Th**

- **We have demonstrated that coverlays, vias, and ENIG metallization can be added with only small increases to the radiopurity – possibly simplifying the design and layout of low background cables**

Next Steps

- Based on promising results and wide interest in the low background community, we have received a **2-year SBIR Phase II grant** to continue to make improvements and simplify the fabrication process with the goal of making radiopure cables commercially available
  - Test cables to make sure they still meet all electrical requirements after cleaning and modifications
  - Fine tune cleaning procedure
  - Investigate possibilities for making custom radiopure coverlays
  - Setup cleaning procedure at company facility to avoid intermediate shipping to PNNL
  - Standardize entire process to make cables commercially available

Q-FLEX INC.

SBIR · STTR
America's Seed Fund

Broader Message:

- Radiopurity of commercial materials can be improved through:
  - Working with a vendor that is willing to perform R&D
  - A systematic approach of investigating each step of the fabrication process
  - Collaboration between chemists and physicists to target and reduce the key contaminations

Acknowledgements

We are extremely grateful to our commercial partners at Q-Flex for providing all the samples used in this work and their willingness to work with us to study and adapt their fabrication process.
We would like to specifically thank
Mario Perez, Jay Patil, Pete Uka, and Raj Patel

We would like to thank Dave Moore and the entire **nEXO collaboration** for providing the design for our "simple" cables and Alvaro Chavarria and the entire **DAMIC-M collaboration** for providing the design of the "full" cables

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DOE Small Business Innovations Research (Phase I 2021, Phase II 2022-2024)