



Ultra-low radioactivity flexible printed cables

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Abstract

Flexible printed cables and circuitry based on copper-polyimide materials are widely used in experiments looking for rare events due to their unique electrical and mechanical characteristics. However, past studies have found copper-polyimide flexible cables to contain 400–4700 pg ²³⁸U/g, 16–3700 pg ²³²Th/g, and 170–2100 ng ^{nat}K/g, which can be a significant source of radioactive background for many current and next-generation ultralow background detectors. This study presents a comprehensive investigation into the fabrication process of copper-polyimide flexible cables and the development of custom low radioactivity cables for use in rare-event physics applications. A methodical step-by-step approach was developed and informed by ultrasensitive assay to determine the radiopurity in the starting materials and identify the contaminating production steps in the cable fabrication process. Radiopure material alternatives were identified, and cleaner production processes and treatments were developed to significantly reduce the imparted contamination. Through the newly developed radiopure fabrication process, fully-functioning cables were produced with radiocontaminant concentrations of 20–31 pg ²³⁸U/g, 12–13 pg ²³²Th/g, and 40–550 ng ^{nat}K/g, which is significantly cleaner than cables from previous work and sufficiently radiopure for current and next-generation detectors. This approach, employing witness samples to investigate each step of the fabrication process, can hopefully serve as a template for investigating radiocontaminants in other material production processes.

Keywords: Flexible cables; Polyimide; Radioactivity; Low background experiments

1 Introduction

Cabling associated with signal sensors and readout electronics are often a significant contributor to the radioactive background budget of physics experiments looking for rare events, such as searches for neutrinoless double beta decay or the direct detection of dark matter [1–8]. Circuitry and cables are composed of two major components—the conductor (typically copper) and insulator. Polyimides are widely used as an insulating substrate in the electronics industry due to their unique properties of high resistivity, high dielectric strength, and flexibility. Polyimides are also stable across a wide range of temperatures, have good thermal conductivity, a thermal expansion coefficient that is close to copper, and a low outgassing rate, which make them a favorable material for use in the ultra-high

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vacuum and cryogenic environments that are commonly found in low background experiments.

However, commercial flexible printed cables composed of laminates of copper and polyimide layers are not very radiopure, with typical contamination levels of the primordial radioactive nuclides ^{238}U and ^{232}Th measured at a few thousand and a few hundred parts-per-trillion by mass (ppt or pg/g), respectively. These values often exceed the stringent radioactivity requirements of rare event searches and experiments therefore have to either limit the amount of cabling used to the absolute minimum necessary (as in nEXO [9], OSCURA [2]), or use other materials [10, 11] that, while more radiopure, do not have all the advantageous properties of polyimide-based flexible cables, such as ease of clean and reliable installation. Additionally, since polyimide-based cables are an industry standard for electronics, the use of alternative material often requires custom-made components, which can increase cost, risk, and production time.

Efforts have previously been made to reduce the contamination in copper-polyimide cables used for rare event searches. The EXO-200 collaboration invested significant effort in selecting amongst different commercial copper-polyimide laminates, measuring contamination levels during the photolithography process, investigating alternative chemicals, as well as working with vendors to reduce contamination during handling and packaging [12, 13]. The EDELWEISS collaboration also surveyed different commercial laminates and adhesives to identify low radioactivity materials and used a custom fabrication process [7, 14]. Even with these efforts, the final cables used in the experiment contained ^{238}U and ^{232}Th contamination at the level of hundreds to thousands of ppt.

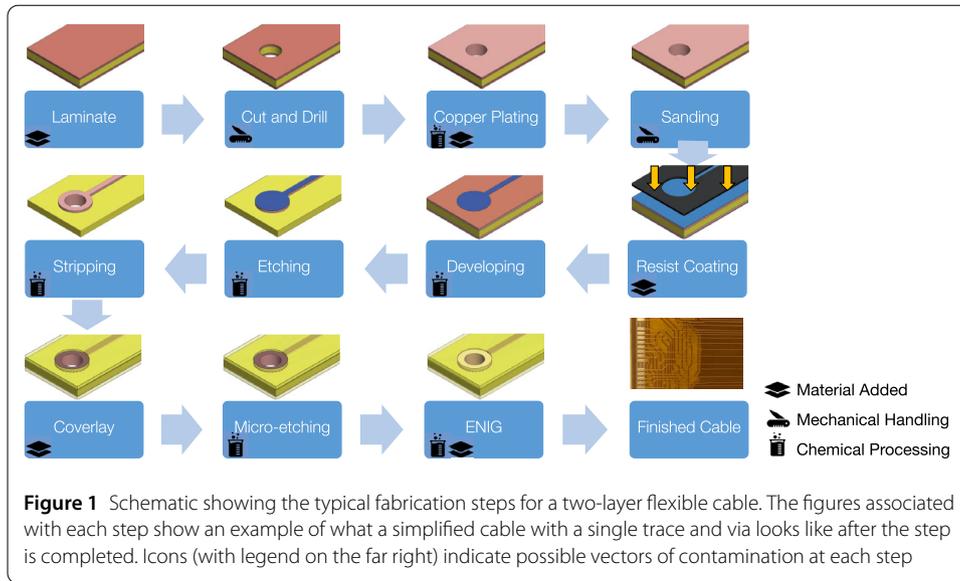
Some of the authors of this paper previously worked to identify clean copper-polyimide laminates, in collaboration with DuPont, leading to the production of custom material that had ^{238}U contamination levels <10 ppt, roughly $10\times$ cleaner than commercial options [15]. However, as described in this paper, even when starting with radiopure laminates the fabrication of the cables introduces large amounts of contamination, leading to roughly the same levels found in commercial cables, i.e., a few thousand ppt ^{238}U and a few hundred ppt ^{232}Th .

In this paper we systematically investigate sources of contamination in the existing cable production process and study alternative strategies—such as modifying processes, developing new cleaning methods, and changing sources of raw materials—to minimize radioactivity in the end product cable.

2 Cable fabrication process and research approach

All cable production work was spearheaded by Q-Flex Inc. who specialize in the fabrication of custom long flexible cables. The production of flexible cables involves a number of fabrication steps that start from a simple laminate of copper and polyimide and then adds vias, patterns of traces and interconnections, insulating coverlays, and metallized pads for external connections. The typical commercial photolithographic process for producing a two-layer flexible cable is shown in Fig. 1. As part of the fabrication process, the starting laminate is subjected to various chemicals, addition of additional copper and polyimide layers, and mechanical manipulation (drilling, cutting, lamination, etc.). Each one of these steps is a potential source of radioactive contamination.

To investigate the contamination levels at each step of the fabrication process, we produced panels of small detachable “coupons” (Fig. 2). The coupons act as surrogates for



real cables—they contain all the relevant features such as traces, vias, coverlays, and go through the same production steps as regular cables. The advantage of this approach is that individual coupons can be easily detached after each step in the process and measured for radioactive contaminants. By comparing the contamination levels in each successive coupon one can assess the level of contamination imparted from each step towards production of the final cable. Where a spray or chemical bath was used for a particular production step, aliquots of the chemicals were also collected for assay along with the coupon for that step.

Inductively coupled plasma mass spectrometry (ICP-MS) was chosen as the analysis method for measuring the radioactive contamination in each coupon. ICP-MS allows for ultrasensitive assays of materials, processes, and reagents, and the ICP-MS capability at Pacific Northwest National Laboratory (PNNL) for supporting ultralow background physics has developed a variety of methods to reach sub-ppt sensitivities for ^{238}U , ^{232}Th , and $^{\text{nat}}\text{K}$ in a variety of materials [15–18]. ICP-MS also has the distinct advantage of being able to investigate small samples (<1 gram) with sufficient sensitivity and having quick turnarounds of sample batches, allowing for detailed, systematic investigations of a large

number of samples. A detailed description of the ICP-MS capability and method is given later in this paper. Because ICP-MS testing is destructive, the technique cannot be used to measure the actual final cables that are deployed. By adding these small detachable surrogate coupons to the same panel as the cable, we can sample the contamination during the production of the final cables—enabling verification that the cables meet radiopurity requirements.

ICP-MS directly detects the atoms (ions) within a sample and is therefore most sensitive to those radioisotopes with long half-lives (e.g., ^{238}U and ^{232}Th). In order to measure contamination levels of isotopes further down in the chain, one could use gamma counting of the radioactive decays. However, the sensitivity is typically limited to specific activities at the level of mBq/kg (equivalent to 81 ppt ^{238}U , 246 ppt ^{232}Th), and it requires kilograms of material and counting times on the order of a month. It should be noted that comparison of ICP-MS measurements of the ^{238}U levels and gamma counting of ^{226}Ra for cables used in the DAMIC experiment indicated secular disequilibrium, i.e., the activity of ^{226}Ra in the lower half of the chain was found to be significantly lower than the ^{238}U activity at the top of the chain [4]. We do intend to eventually measure our final cables through gamma counting but the limited sensitivity, large sample mass and long counting times needed, led to us not pursuing it at the R&D stage.

3 Analysis methods

All analyses of the materials reported in this work were performed at PNNL. Analytical work was performed in a Class 10,000 cleanroom, and a laminar flow hood providing a Class 10 environment was used for sample preparations. Details on the chemical reagents used and the preparation of all labware prior to sample handling are given in Appendix A.

3.1 Sample digestion

Subsamples of ~50 mg were cut from original sample with clean stainless steel scissors, collected, and weighed in validated perfluoroalkoxy alkane (PFA) screw cap vials from Savillex (Eden Prairie, MN). Unless explicitly stated, only central portions of the coupons/cables were used for subsampling, excluding any regions with vias, pads, or ENIG processing. Low background clean plastic tongs were used to transfer subsamples to validated polytetrafluoroethylene PTFE iPrepTM vessels for microwave-assisted digestion in a Mars 6 system (CEM Corporation, Matthews, NC). All samples and process blanks were spiked with a known amount (~100-200 fg) of gravimetrically measured ^{229}Th and ^{233}U radio-tracers (Oak Ridge National Laboratory, Oak Ridge, TN) before digestion. Triplicates of each sample and three process blanks were prepared for each batch of analyses. Digestion was performed in 5 mL of concentrated Optima grade HNO_3 at 250 °C. After complete digestion, sample solutions were transferred to cleaned and validated PFA Savillex vials, and properly diluted for ICP-MS analysis.

3.2 ICP-MS analysis

Determinations of Th and U were performed using either an Agilent 8800 or 8900 ICP-MS (Agilent Technologies, Santa Clara, CA), each equipped with an integrated autosampler, a microflow PFA nebulizer and a quartz double pass spray chamber. Plasma, ion optics and mass analyzer parameters were tuned based on the instrumental response of a tuning standard solution from Agilent Technologies containing 0.1 ng/g Li, Co, Y, Ce, Tl. In

order to maximize the signal-to-noise in the high m/z range for Th and U analysis, the instrumental response for Tl, the element in the standard with m/z and first ionization potential closest to those of Th and U, was used as a reference signal for instrumental parameter optimization. Oxides were monitored and kept below 2% based on the $m/z = 156$ and $m/z = 140$ ratio from Ce (CeO^+/Ce^+) in the tuning standard solution. An acquisition method of three replicates and ten sweeps per replicate was used for each reading. Acquisition times for monitoring m/z of interest (*e.g.*, tracers and analytes) were set based on expected signals, in order to maximize instrumental precision by improving counting statistics.

Quantitation of ^{232}Th and ^{238}U was performed by isotope dilution methods, using the equation:

$$\text{Concentration} = \frac{A_{\text{analyte}} \cdot C_{\text{tracer}}}{A_{\text{tracer}}}, \tag{1}$$

where A_{analyte} is the instrument response for the analyte, A_{tracer} is the instrument response for the tracer and C_{tracer} is the concentration of the tracer in the sample. Isotope dilution is the most precise and accurate method for quantitation for ICP-MS analysis, allowing the verification of sample preparation efficiency, and accounting for analyte losses and/or matrix effects. Absolute detection limits on the order of a few femtograms were attained for both ^{238}U and ^{232}Th . Detection limits normalized to sample mass were on the order of single digit pg/g for both Th and U, corresponding to $\mu Bq/kg$ in terms of radioactivity.

Determinations of natural potassium contamination levels were performed in cool plasma with NH_3 reaction mode. Instrumental parameters were optimized based on the instrumental response from a solution containing $\sim 1 \text{ ng/g } ^{nat}K$ with natural isotopic composition, diluted in-house from standard solutions. Quantitations of potassium were performed using an external calibration curve, generated using in-house diluted standard solutions with natural isotopic composition. Relatively high potassium backgrounds from the microwave digestion PTFE iPrepTM vessels (see Sect. 3.1) limited detection limits on potassium to the ng/g range. Due to the extensive preparation required to minimize these backgrounds, potassium determinations were only performed on a limited number of samples.

All reported central values are the average value of multiple replicates (typically $n = 3$). Uncertainties for individual replicates were determined from the propagated uncertainties of the instrumental precision. The overall uncertainty on the central value was taken to be the larger of the uncertainty from the instrumental precision and the standard deviation of the replicates. Samples for which the analyte concentration was measured below the detection limit are reported as an upper limit. Values are reported in pg/g of sample for ^{238}U and ^{232}Th , and in ng/g of sample for ^{nat}K . Corresponding activities in $\mu Bq/kg$ of sample can be obtained based on the specific activity and isotopic abundance of the radionuclides. For reference, 1 pg $^{238}U/g$ of sample corresponds to 12.4 $\mu Bq/kg$ of sample, 1 pg $^{232}Th/g$ corresponds to 4.06 $\mu Bq/kg$, and 1 ng $^{nat}K/g$ corresponds to 30.5 $\mu Bq/kg$.

4 Contaminating steps

The measured contamination of ^{238}U and ^{232}Th in coupons at each step of the fabrication process, along with measurements of any materials and solutions involved in those steps, is shown graphically in Fig. 3 with the detailed results and uncertainties presented

in Table B.1. As can be seen, despite starting the process with clean material (few ppt ^{238}U and ^{232}Th), the final cable has contamination levels of ~ 7000 ppt ^{238}U and ~ 700 ppt ^{232}Th . These values are similar to the previous findings of the EXO-200 [12] and EDEL-WEISS [14] collaborations. By studying the evolution of the cumulative contamination in the coupons after each step, we were able to target specific production steps that were the dominant source of contamination. In the following subsections we describe each of the key contaminating steps (not necessarily in order) and the approach used to reduce the contamination introduced during that step.

4.1 Starting laminate

The starting material for the production of flexible cables is a laminate, a foil consisting of an insulating flexible polymer, typically polyimide, layered with a conductive metal, usually copper. Since this material forms the bulk of the mass of the finished cable, it is critical that it be as low in radioactivity as possible.

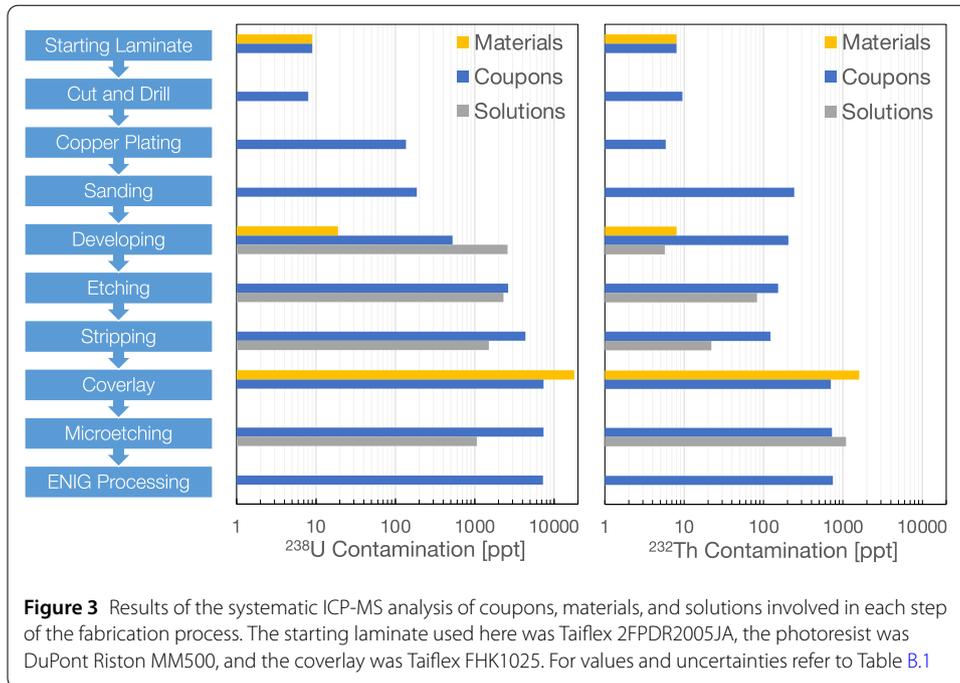
In previous work [15], we have demonstrated the possibility to obtain low-background laminates with single-digit ppt levels of ^{232}Th and ^{238}U impurities ($\sim \mu\text{Bq/kg}$ range). As shown in Table 1, this is significantly cleaner than other laminates we surveyed as part of that study as well as laminates measured by other low background experiments. The specialized Kapton laminate produced by the DuPont R&D division [15] was only available in short individual sheets, limiting the ability to make long cables. Measurements conducted prior to the rest of the work described in this paper, found that a copper-polyimide laminate from Taiflex [19] has similar radiopurity to that of the Dupont R&D material (see Table 1). The Taiflex material is two-sided copper laminated to a central polyimide layer with a thermoplastic polyimide. The central polyimide layer is $50 \mu\text{m}$ and each copper layer is $18 \mu\text{m}$ thick. This commercial material is available in rolls, making it suitable for manufacturing long cables, as is often needed in low background experiments. All samples made for testing subsequent steps in the process were made starting from this Taiflex laminate.

4.2 Photolithography and cleaning

Photolithography [21] is the patterning of electrical traces and other features on the copper surface of the laminate. A photo-sensitive material (photoresist), in our case DuPont Riston MM500, is applied to the laminate to mask sections of the copper and then hardened (developed) by exposure to ultraviolet light. The exposed copper is then removed

Table 1 Comparison of the radiopurity of copper-polyimide laminates. The (xN) factor in the fourth row indicates whether the copper was on one or both sides of the laminate

Type	Vendor	Polyimide thickness [μm]	Copper thickness [μm]	^{238}U [pg/g]	^{232}Th [pg/g]	$^{\text{nat}}\text{K}$ [ng/g]
Taiflex 2FPDR2005JA	Taiflex	50.8	18 (x2)	8 ± 6	9 ± 4	<100
300ELJ+Cu laminate [15]	DuPont R&D	76.2	18 (x2)	8.6 ± 3.6	20 ± 14	164 ± 82
Pyralux AP8535R [15]	DuPont	76.2	18 (x2)	158.0 ± 6.1	24.1 ± 0.9	<210
Novaclad 146319-009 [15]	Sheldahl	50.8	5.0 (x1)	283 ± 21	50.1 ± 3.9	<210
Cirlex [15]	Fralock	228.6	34 (x2)	413 ± 45	71.4 ± 2.1	<210
Espanex MC15-40-00VEG [20]	Nippon Steel	40	15 (x1)	121 ± 32	<250	880 ± 120
Espanex MC18-25-00CEM [20]	Nippon Steel	25	18 (x1)	<46	<260	<146
Espanex SB [14]	Nippon Steel	25	18 (x2)	<284	<812	<1777



(etched) and finally the resist is removed (stripped) and dried. This process involves immersing the coupons in a sequence of different chemical solutions to perform these steps. As can be seen in Fig. 3, the photolithography steps (“Developing”, “Etching”, and “Stripping”) were found to be a major source of contamination with 1000’s of ppt ^{238}U and 100’s of ppt ^{232}Th measured after the stripping step. We assayed the chemical solutions used in the developing, etching and stripping steps: anhydrous Na_2CO_3 diluted to 1%, 0.5 M HCl, and a KOH based solution diluted to 20%, respectively, and found significant levels of contamination (shown in Fig. 3) indicating that the solutions were the likely source of contamination. The chemicals used in this process have been carefully chosen and balanced for cable production and changing the chemicals (e.g. purifying or replacing with alternatives) was determined to require significant amount of R&D and to likely be impractically expensive. Instead, we pursued the development of a post-cleaning method to try and reduce the contamination after lithography.

The lithographic process for flexible cable manufacturing is almost entirely a “subtractive” procedure: the electrical traces and connections are produced through removal of copper from the outer layers of the starting copper-polyimide laminate with no additional material added (the photoresist is removed at the end)—see Fig. 1. With this in mind, it was believed that contaminant species from the processing should be at or near the surface and be removable through well-tailored post-processing cleaning.

Several different cleaning methods were tested. A partially manufactured test cable, processed up to and including the stripping step in Fig. 3 but skipping the “Copper plating” step, was used for this dedicated study. This test cable had a contamination level of ~ 4000 ppt ^{238}U and ~ 100 ppt ^{232}Th . The cleaning consisted of submerging subsamples of the test coupon in the chosen cleaning solution for a fixed length of time while agitating, followed by rinsing in deionized water and air drying in a class 10 laminar flowhood. Subsamples of the same size as those used for ICP-MS assay (~ 50 mg, ~ 1 cm² surface) were cleaned and assayed before and after cleaning to assess the efficacy of the method.

Initial attempts tested combinations of various diluted acid solutions (e.g., nitric acid, sulfuric acid) and durations (30 sec–15 min) to determine the optimal procedure. Although some of these cleaning methods provided a very significant reduction of contaminants in the cable ($\sim 75\times$ and $\sim 2\times$ in ^{238}U and ^{232}Th respectively) the residual contamination was still above our target radiopurity levels.

Surveying the literature, we learnt that treatments of polyimide films with alkaline solutions, e.g., ethylenediamine, resulted in modifications to the chemical structure of the polyimide surface, increasing adhesion [22]. Based on this finding, a cleaning procedure involving the use of an alkaline solution was developed, targeting a more efficient removal of surface contaminants, in particular from the polyimide surface. The cleaning consisted of submerging the samples in 5% v/v tetramethylammonium hydroxide (TMAH) for 15 min, rinsing in deionized water (DIW), submerging the samples in a 2% nitric acid solution for ~ 1 min, followed by rinsing in DIW and air drying in a class 10 laminar flow hood. A nylon brush with soft polyester bristles was used for scrubbing the samples while they were submerged in each cleaning solution. The duration of the submersion in nitric acid was set based on previous test results, while the duration of submersion in TMAH was set based on a study of cleaning efficacy and visual inspection of the cables after treatment. The efficiency of the cleaning improved with longer duration, but long exposures to TMAH eventually resulted in visible modification of the copper surface. It was found that repeating the entire cleaning cycle thrice was more effective at reducing the contamination than a single cycle. This cleaning recipe reduced the ^{238}U and ^{232}Th contamination in the test coupon to ~ 20 and ~ 10 ppt, respectively.

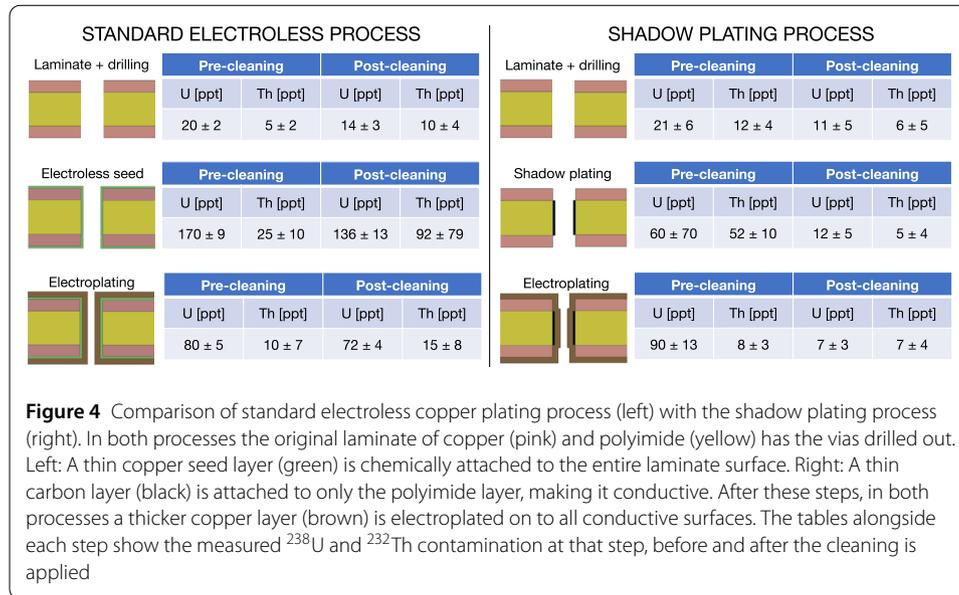
Optical inspection of the cable (at 100, 200, and 500 \times magnification) after cleaning did not show any visible structural changes of the copper traces. For the cleaning of the final cables, described later in Sect. 5, electrical tests were performed after cleaning to ensure the cable still functioned electrically.

4.3 Copper plating

For cables that require interconnections between the conductive copper layers, vias are used. A hole is drilled and then plated with copper to form the electrical connection between the outer two copper layers, across the polyimide insulation (Copper plating step in Fig. 3). Because copper cannot be directly electroplated onto polyimide, a first “seeding” step is used to chemically attach a thin conductive layer to the entire laminate surface. Once the conductive seed layer is added, copper is deposited through electroplating (see the left side of Fig. 4 for a schematic overview).

Despite several attempts, it was found that the surface cleaning methods we developed (described in Sect. 4.2) were not effective in removing the contamination introduced during electroplating—see the tables on the left side of Fig. 4 for the results of the measurements before and after cleaning. The electroless seeding process is not selective and deposits the conductive layer over the entire surface of the cable. The subsequent copper electroplating then also deposits a layer of copper over the entire cable. Thus contamination introduced in these steps can be trapped between the base laminate, seed, and electroplating layers, preventing removal through surface cleaning.

To investigate whether the contamination was being trapped between the seed and electroplated layers, we made coupons using a “pad plating” process [23]. In this method, after the seed layer is applied, photoresist is applied to mask the areas of the cable that do not



need an additional copper layer (i.e. everything except the vias). The electroplating then only deposits copper in the via regions and the photoresist is removed after the plating. This is a standard industrial process used to avoid the additional plated copper from covering the entire surface area and stiffening the cable. A systematic analysis of coupons after each step of the process, found similar contamination levels with and without pad plating, indicating that the dominant contamination was not trapped between the seed and electroplated layers, but likely between the base laminate and seed layer.

The standard electroless seeding process involves several chemical treatment steps and typically requires a catalyst [24]. A newer alternative process to the electroless Cu step is the “shadow” process [25, 26]. In this approach, a thin layer of carbon is laid down on the cable. An etch is then performed so that carbon only adheres to the polyimide and so does not cover the copper surface [27] (see the right side of Fig. 4 for a schematic overview). Unlike the standard electroless seed process, the shadow process uses fewer chemicals and also deposits less material over much smaller area than the standard approach. The shadow process therefore has the potential to be intrinsically less dirty than the standard electroless seed method.

We investigated the contamination levels in coupons before and after the shadow process as well as after the subsequent electroplating (see tables on right side of Fig. 4). We found significant contamination after both seeding and plating steps, however, unlike in the case of the electroless seeding, the contamination could be removed by our cleaning method. In fact, the resulting coupons had contamination levels consistent with the base laminate level, roughly 9 times cleaner in ²³⁸U than the electroless seed process. Given these promising results, we elected to use the shadow plating process for copper plating vias in our final cables.

4.4 Sanding

Prior to the application of photoresist, the copper surface of the cable is prepared for optimal dry film adhesion and subsequent clean release. Surface preparation processes typically aim to remove any surface imperfections after drilling and electroplating and

roughen the surface to increase film contact area. At Q-Flex, this is done by mechanical scrubbing of the surface with abrasive pads. The scrubbing process was found to increase ²³²Th contamination, presumably due to the implantation of small amounts of the abrasive material into the laminate surface. With this in mind, we worked to remove the abrasive material from the surface by sonication in 18.2 MΩ·cm water. The results before and after sonication were within error of each other, indicating that this method did not remove the contamination. The acid-base cleaning method described in Sect. 4.2 was also applied at this step but did not significantly reduce the contamination.

Since our attempts to remove any embedded material from the surface through sonication and cleaning proved ineffective, we reviewed the available commercial options for scouring pads. We decided to exclusively use commercial Scotch Brite™ pads made from SiC [28], known to generally be a radiopure material, rather than previously used pads that used aluminum oxide, titanium dioxide, and other fillers and pigments [29]. The change in abrasive material led to roughly a 10x reduction in ²³²Th at this step.

4.5 Coverlay

A coverlay is an insulating layer that is applied over the outer surfaces of a cable to prevent oxidation and shorting of the exposed copper traces. Commercial coverlays are typically composed of two materials—the polyimide that produces the electrical insulation and an adhesive that bonds the polyimide to the surface of the cable. In addition to reviewing previous measurements of coverlays in the literature [3], we surveyed several different commercially available coverlays, as shown in Table 2.

As can be seen from the results, the contamination levels of ²³⁸U and ²³²Th can vary by several orders of magnitude amongst commercial coverlays. Although Taiflex copper-polyimide laminates were found to be extremely radiopure, the epoxy-based adhesives they utilize have extremely high levels of radiocontaminants. Our measurements indicate that coverlays based on acrylic adhesives have the lowest ²³⁸U and ²³²Th contamination. We also measured the ^{nat}K contamination levels in these acrylic coverlays and found high contamination levels (> ppm) with significant variations (see final column of Table 2). Overall, the MCL coverlay was the best choice, with contaminations of ²³⁸U and ²³²Th lower by factors of roughly 200 and 30, respectively, compared to the Taiflex coverlay. Upilex C120 should also be considered for experiments that are significantly more

Table 2 Radiopurity results from survey of commercially available coverlay materials, including results from previous measurements in the literature [3]. The second column indicates the polyimide (PI) thickness and the third column the adhesive thickness. The materials in the last two rows are adhesives only

Sample	PI thick. [mil]	Adh. thick. [mil]	Notes	²³⁸ U [pg/g]	²³² Th [pg/g]	^{nat} K [ng/g]
Taiflex FHK1025	1	1	Epoxy adhesive	18,000 ± 2000	1600 ± 140	
ShinEtsu CA 333 [3]	1	1		5179 ± 424	<242	
ShinEtsu CA 335 [3]	1	1.4		12,020 ± 390	9370 ± 340	
Dupont LF0110	1	1	Acrylic adhesive	314 ± 13	49 ± 8	4000 ± 2000
Upilex C120	2	1		30 ± 2	280 ± 20	21,300 ± 300
MCL Plus 110	1	1		78 ± 4	45 ± 7	5030 ± 140
Dupont FR 70001 [3]	0.5	0.5		<1065	<473	
Dupont FR 0110 [3]	1	1		<818	<273	
Dupont LF0100	0	1	LF0110 adhesive	16 ± 4	39 ± 11	
Imitex MI-100	0	1	Adhesive	9 ± 5	<14	

sensitive to backgrounds from ^{238}U than ^{232}Th or ^{40}K . We also separately measured acrylic adhesives and found them to be cleaner than the combined acrylic + polyimide coverlays (last two rows of Table 2). For future work we intend to investigate coverlays made from custom lamination of radiopure acrylic and polyimide as well as all-acrylic coverlays.

5 Production of an ultra-low background cable

Having identified the key contaminating steps in flexible cable fabrication and developed methods and alternatives to reduce the contamination in each individual step, we produced two sets of fully-functional flexible cables by combining all the modifications described in the previous section into a single low-background fabrication process. We chose two different flexible cable designs that are being considered for on-going and future nuclear and particle physics experiments. The first design is under development for the readout of silicon photomultipliers (SiPMs) in the nEXO neutrinoless double beta decay experiment [9]. Based on findings from previous investigations into radiopure cable conducted for the EXO-200 cable [3, 12], the cable design was simplified to keep contamination levels as low as possible. The cable (shown on the left in Fig. 5) consists of traces on only one side of the cable and a continuous ground plane on the other. As all the traces are on one side of the cable with no interconnections between the two layers, no vias or copper plating is required. Additionally, while it would be advantageous, the design does not include a coverlay or surface metallization of contacts in order to keep contamination levels to a minimum.

The second cable design was developed for the readout of charged couple devices (CCDs) for the DAMIC-M [30] dark matter experiment. The CCD cable (shown on the right in Fig. 5) consists of multiple traces and interconnections between the two copper layers that require copper-plated vias. The cable also includes coverlays on both sides of the cable and ENIG metallization on the pads.



Table 3 Measurements of the ^{238}U , ^{232}Th and $^{\text{nat}}\text{K}$ concentrations in the final SiPM and CCD custom cables. Each row within the cable type lists the values for a given subsample (identified by the repetition number in the second column) with the uncertainties indicating the instrumental uncertainty. *Averages are reported as the average and standard deviation of the central values of all replicates for that isotope (upper limits excluded). Where all replicates are upper limits, the largest upper limit is reported

Cable	Rep.	^{238}U [pg/g]	^{232}Th [pg/g]	$^{\text{nat}}\text{K}$ [ng/g]
SiPM Cable (Custom)	1	20 ± 2	<9.8	<38
	2	21 ± 2	<9.4	<37
	3	18 ± 2	<8.6	<34
	4	20.9 ± 1.2	<10.4	47 ± 6
	5	19 ± 2	<10.3	32 ± 8
	6	18.8 ± 1.2	<12.3	<20
	7	19.6 ± 1.5	<12.0	52 ± 7
	8	19 ± 3	<12.0	28 ± 7
	Avg.*	20 ± 2	<12.3	40 ± 12
CCD Cable (Custom)	1	32 ± 2	12 ± 3	559 ± 13
	2	31 ± 4	11 ± 3	529 ± 12
	3	29 ± 2	<8.9	572 ± 12
	4	32 ± 3	16 ± 4	569 ± 13
	5	31 ± 2	<11.7	558 ± 12
	6	30 ± 2	<10.9	546 ± 9
	7	30 ± 2	<11.1	515 ± 9
	Avg.*	31 ± 2	13 ± 3	550 ± 20

The SiPM and CCD cable designs were chosen because they represent two ends of the spectrum in terms of complexity of two-layer flexible cables. Figure 5 shows the process steps used to fabricate these two cables, highlighting the modifications made to the standard process. As before, panels consisting of the cables along with several detachable coupons were used and coupons were removed after several key steps so that the contamination levels could be tracked, if needed. The cleaning process described in Sect. 4.2 was applied at key steps in the fabrication process where it would not interfere with the production and would remove surface contamination before material was permanently added to the cable. Since the cleaning recipe required the use of relatively hazardous chemicals and precise cleaning procedures, at intermediate steps (indicated in green in Fig. 5) the partially-processed panels were shipped from Q-Flex to PNNL, cleaned, and then shipped back. The shipping was done in sealed plastic bags and the cleaning was done as promptly as possible to avoid oxidation of the copper layers between steps. Once the fabrication steps were complete, the cable was shipped to PNNL for a final cleaning and subsequent measurement. For the measurements of the contamination levels in the final cables, we sub-sampled the cables themselves (not witness coupons) and used 7-8 sub-samples, rather than the standard 3, to increase our confidence that the average of our measurements were representative of the “true” contamination in the cable overall.

The results are shown in Table 3. The levels of contamination measured in the final SiPM cable were extremely low, with roughly 20 ppt of ^{238}U , ^{232}Th levels below our sensitivity of ~10 ppt, and $^{\text{nat}}\text{K}$ at roughly 36 ppt. The CCD cable, which included additional copper plating of the vias, coverlays on both sides, and ENIG metallization of the contacts, was only slightly higher in ^{238}U and ^{232}Th , with roughly 31 and 12 ppt respectively. The $^{\text{nat}}\text{K}$ levels were significantly higher, consistent with the intrinsic contamination levels measured in the MCL Plus 110 coverlay used and the relative contribution of the coverlay

to the overall mass of the cable. It is worth remarking that while commercial cables can sometimes have values that vary significantly from subsample to subsample, all subsample replicates measured in the custom final cable were consistent with each other, indicating that the cleaning procedure (applied at key steps) likely removed any non-uniform surface contamination.

5.1 ENIG metallization

While measurements of the center of the cable (where all samples were taken from for the results in Table 3) represent the contamination levels in the vast majority of the cable material, the copper contacts at the ends of the cable typically have an additional metallization layer to avoid oxidation and improve solderability [31]. This metallization, typically added through electroless deposition in a chemical bath with the addition of a catalyst, can add additional contamination, and though the total mass is often small the contacts at one end of the cable can be located very close to the sensitive volume. The CCD cables were metallized with electroless nickel immersion gold (ENIG) using 3.0-5.6 μm (118-220 microinch) of nickel and 0.076-0.127 μm (3-5 microinch) of gold. In order to measure the contamination levels of the ENIG layer, subsamples were cut from the ends of the cable to include the metallized pads. Since the size of the ENIG region varied by subsample, for each subsample measurement we estimated the ENIG-covered area and subtracted out the average contamination of the rest of the cable layers (estimated from the measurements at the center). The residual contamination was attributed to the application of the ENIG layer. We find contamination levels per unit area of ENIG at roughly 0.1 pg/mm^2 for ^{238}U , 0.01-0.1 pg/mm^2 for ^{232}Th , and 10 ng/mm^2 for $^{\text{nat}}\text{K}$. These results can be used to estimate the backgrounds for specific cable layouts with ENIG metallization layers.

6 Summary and discussion

As a direct point of comparison, we also measured commercial cables with the same design as our SiPM and CCD custom cables. For the SiPM cable we measured a cable that was produced with the same low-background starting laminate but using the original standard Q-Flex fabrication process that did not include any of the modifications described in this work. Cables from different batches showed large variations in the ^{238}U and ^{232}Th contamination levels and so we quote a range of values (see Table 4). For the CCD cable we obtained a cable from the DAMIC collaboration of the same design, fabricated by a different commercial vendor (PCB Universe [32]) with no special instructions on materials to use or process modifications. It can be seen from comparing the results in Table 4 that, compared to our custom low radioactivity cables, ^{238}U and ^{232}Th levels in the commercial cables are roughly $100\times$ and $10\times$ larger, respectively. Interestingly, the potassium contamination levels in the commercial CCD cable were roughly $2\times$ lower than the levels measured in our custom cable, likely due to the choice of a different coverlay material.

It is also worthwhile to compare our results to those obtained by other experiments that use flat flexible cables in rare event searches, though the cable designs vary significantly. As noted in the Introduction, the EXO-200 collaboration, which searched for the neutrinoless double beta decay of ^{136}Xe , invested significant effort to reduce the contamination in their detector cables [12, 13]. They used single-layer cables and worked with the vendor to use fresh chemicals and containers and add isopropanol rinses during the production process. After fabrication, all small cables were subjected to a post-production plasma etch

Table 4 Comparison of the final radiopurity levels achieved in custom cables produced as part of this work (highlighted in bold) with other commercial and custom cables used in low background rare event experiments. The commercial (abbreviated as Com.) cables listed near the top of the table are the same design as our custom cables and can be compared directly. The cables listed at the bottom of the table have different construction and fabrication options—see text for details

Cable	Copper layers [μm]	Polyimide layers [μm]	Coverlay	Surface finish	²³⁸ U [pg/g]	²³² Th [pg/g]	^{nat} K [ng/g]
nEXO SiPM	18 (x2)	50.8 (x1)	No	No	20 ± 2	<12.3	40 ± 12
nEXO SiPM [Com.]	18 (x2)	50.8 (x1)	No	No	1300-6200	16-63	
DAMIC-M CCD	18 (x2)	50.8 (x1)	x2	ENIG	31 ± 2	13 ± 3	550 ± 20
DAMIC-M CCD [Com.]	18 (x2)	50.8 (x1)	x2	ENIG	2600 ± 40	261 ± 12	170 ± 50
EXO-200 [3, 12]	18 (x1)	25.4 (x1)	No	No	412 ± 47	<117	
EDELWEISS III [7, 14]	18 (x4)	25/125 (x3/x4)	No	No	650 ± 490	3700 ± 2500	2100 ± 840
DAMIC at SNOLAB [4]	18 (x5)	25.4 (x4)	x2	ENIG	4700 ± 400	790 ± 120	940 ± 60

followed by a cleaning with acetone and ethanol. To reduce contamination, no metallization of the contacts was applied and no coverlay was used within the detector cryostat. The copper and polyimide layers of the cable were separately assayed for radiocontamination using ICP-MS [20]. Taking the best obtained values for the copper (Entry 261 in Ref. [20]) and polyimide (Entry 262 in Ref. [20]) we have combined them by the mass ratio to calculate the final value shown in Table 4. The EDELWEISS collaboration, searching for dark matter, surveyed several different materials and selected specific materials for their copper clad polyimide laminates [14]. Though they only used four conducting layers, the cable had additional polyimide layers of varying thickness in between to reduce the capacitance. They also identified the photolithography process as an additional source of contamination and used a custom fabrication procedure at the Oxford Photofabrication Unit [14]. The contamination levels in the final cables used in the EDELWEISS III experiment [7] are listed in Table 4. The DAMIC collaboration, searching for dark matter, used a 5-layer cable to read out their CCDs installed at SNOLAB [4]. These were commercial cables with no effort made to reduce the contamination. The contamination levels, measured through ICP-MS, are listed in Table 4. It can be seen that the radiopurity levels achieved in the cables produced as part of this work, compared to the ones used in previous experiments, are at least an order of magnitude better in ²³⁸U and ²³²Th, and lower in ^{nat}K as well.

This work was motivated by the specific need for low radioactivity cables in either planned upgrades of currently running experiments such as DAMIC-M [30] and SuperCDMS [33] or in future proposed experiments such as nEXO [9] and Oscura [2]. We have demonstrated that simple cable designs can be produced with contamination levels at 20 ppt ²³⁸U, <12 ppt ²³²Th, and 40 ppb ^{nat}K, nearly at the levels of the raw materials. We believe these levels to be sufficiently radiopure for all the experiments listed above.

We have also shown that cable designs that require additional features such as interconnected layers, coverlays, and metallization can also be made with only a relatively small increase in the ²³⁸U contamination, though there is a significant increase in ^{nat}K due to the coverlay material. Additional investigations into multi-layer cables, higher radiopurity coverlays, and alternatives to ENIG metallization are planned in the near future. It should be noted that low radioactivity cables can increase the sensitivity of these experiments by not only reducing the background budget but also by allowing for the use of additional sensors and simpler designs of the sensor readout within the same background budget.

Finally, while we have focused this work on radiopure cables, the same low-radioactivity investigative technique could be applied to flexible printed circuits and related applications. For example, in future work we will use a similar approach with the goal of developing low radioactivity superconducting cables with possible applications to cryogenic rare-event searches [11, 34], and to develop low radioactivity printed circuit boards, which have been predicted to be the dominant material within dilution refrigerators that impact decoherence from background ionizing radiation [35].

Appendix A: Reagents and labware

Electronic grade tetramethylammonium hydroxide (TMAH), 100% pure ethanol, and Optima grade nitric, hydrochloric, and sulfuric acids (Fisher Scientific, Pittsburg, PA, USA) were used for sample cleaning and preparation. 18.2 MΩ·cm water from a MilliQ system (Merk Millipore GmbH, Burlington, MA) was used for sample rinsing and in the preparation of reagent solutions. Ultralow background perfluoroalkoxy alkane (PFA) screw cap vials from Savillex (Eden Prairie, MN) were used as sample containers, to collect solutions from microwave digestion vessels and as ICP-MS autosampler vials.

All labware involved in sample handling and analysis (vials, microwave vessels, tongs, pipette tips) were cleaned with 2% v/v Micro-90 detergent® detergent (Cole-Parmer, Vernon Hills, IL), triply rinsed with MilliQ water, and leached in Optima grade 25% v/v HCl and 6M HNO₃ solutions. Following leaching, all labware underwent validation to ensure cleanliness. The validation step consisted of pipetting a small volume of 5% v/v HNO₃ into each container: 1.5 mL in the PFA vials and 5 mL in the iPrep™ microwave vessels. Vials were closed, shaken, and kept at 80 °C for at least 12 hours. Microwave vessels underwent a microwave digestion run at 220 °C. Tongs and pipette tips were soaked into a 5% v/v HNO₃ leaching solution (ca. 1.5 mL) for 5-10 minutes. The leachate from all labware was then analyzed via ICP-MS. The validation was performed to assure sufficiently low background for Th and U. Only labware for which Th and U signals were at reagent background levels passed validation. Labware failing validation underwent additional cycles of leaching and validation tests until background requirements were met.

Table B.1 ²³⁸U and ²³²Th concentrations at each cable fabrication step for raw materials, photolithography solutions, and representative coupons. Measurements of the materials and solutions are normalized to their individual component masses, while the values listed for the coupons are normalized to the mass of the coupon, including all layers present, at that step. *Laminate and coverlay from Taiflex (2FPDR2005JA and FHK1025, respectively). †Photoresist from DuPont (Riston MM500)

Fabrication step	Materials		Solutions		Coupons	
	²³⁸ U [pg/g]	²³² Th [pg/g]	²³⁸ U [pg/g]	²³² Th [pg/g]	²³⁸ U [pg/g]	²³² Th [pg/g]
1 Laminate*	9 ± 4	8 ± 6				
2 Cut and Drill					< 16	< 20
3 Copper Plating					140 ± 10	< 20
4 Sanding					190 ± 20	240 ± 110
5 Developing	19 ± 13 [†]	8 ± 6 [†]	2630 ± 110	6 ± 2	530 ± 40	210 ± 10
6 Etching			2300 ± 200	80 ± 10	2600 ± 600	150 ± 40
7 Stripping			1510 ± 70	22 ± 2	4400 ± 500	120 ± 30
8 Coverlay*	18,000 ± 2000	1600 ± 140			7400 ± 200	710 ± 20
9 Microetching			1060 ± 60	1090 ± 60	7400 ± 400	730 ± 50
10 ENIG Processing					7300 ± 200	750 ± 40

Appendix B: Contamination by fabrication step

The results of the systematic ICP-MS analysis of coupons, materials, and solutions involved in each step of the original Q-Flex fabrication process, illustrated in Fig. 3, are given in Table B.1.

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Availability of data and materials

The data generated and analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

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Author contributions

IA: Conceptualization, Methodology, Validation, Investigation, Writing—Review & Editing, Funding acquisition. MLdV: Methodology, Validation, Formal analysis, Investigation, Data Curation, Writing—Review & Editing. NR: Methodology, Formal analysis, Investigation, Data Curation, Writing—Review & Editing. RS: Conceptualization, Methodology, Writing—Original Draft, Writing—Review & Editing, Visualization, Supervision, Project administration, Funding acquisition. TS: Formal analysis, Investigation. RP: Methodology, Project administration. JP: Methodology, Software, Validation. MP: Methodology, Investigation, Validation. HU: Methodology, Supervision, Funding acquisition. All authors read and approved the final manuscript.

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