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Absorber Clamp for Microcalorimeter Decay Energy Spectrometry

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Abstract

Microcalorimeter Decay Energy Spectrometry (DES) is of interest to nuclear safeguards due to its ability to provide high precision isotopic compositions of nanogram- to microgram-scale samples of Pu and U and related daughter products. The DES method is able to record decay energy of each alpha-decay event in a sample that is embedded in a metal matrix (absorber) and thermally linked to a microcalorimeter detector. This work optimizes the DES technique used to thermally link the absorber and microcalorimeter detector element to allow for more rapid assembly and to increase detector performance and operating life. Optimized attachment methods are crucial for enhancing the viability of DES in high-sample-throughput facilities, such as those that support international nuclear safeguards measurements. Here, we designed and implemented a pressure-based absorber clamp and evaluated the performance of this new attachment method relative to pressed indium bond attachment. Results using the absorber clamp demonstrate a streamlined detector assembly procedure that minimizes accidental damage to detectors, as well as increasing detector pulse speeds by 57%. Spectral comparison shows the clamp preserves detector performance relative to indium attachment.

Keywords: decay energy spectrometry, microcalorimeter, transition edge sensor, plutonium, safeguards, instrumentation

1.0 Introduction

Microcalorimeter Decay Energy Spectrometry (DES) is of interest to international nuclear safeguards as a high-precision radiometric isotopic analysis method that can reduce the time, labor, and cost associated with analyzing safeguards samples while increasing the accuracy and confidence in reported results [1, 2, 3]. DES uses nanogram- to microgram-scale samples of Pu, U, or other actinides encapsulated in a microcalorimeter detector to measure the total decay energy for each alpha-decaying nuclide with nearly 100% detection efficiency [4, 5]. This unique measurement gives ultra-high energy resolution (less than 1 keV FWHM at 5 MeV) with a simplified spectrum compared to conventional alpha spectroscopy [1], with no isobaric interferences that hinder mass spectrometric methods, and no chemical separation of samples required.

The DES technique, however, is currently limited in its sample throughput efficiency. To support international nuclear safeguards measurements, hundreds of samples per year must be analyzed with the highest possible precision and accuracy to ensure small biases in the declarations of States are not masking diversions of fissile materials [6, 7]. Therefore, any emerging techniques under consideration for enhancing safeguards measurements must be able to process samples quickly and precisely. Current DES techniques and technologies are not able to support such high sample throughputs, but new instrumentation design work aims to address this problem.

Here, we present the design and evaluation of a new pressure-based absorber clamping attachment method. We cover the current procedure for absorber attachment and DES detector assembly using the indium press bond, highlighting areas for improvement. Tests using the absorber clamp evaluate detector performance metrics such as pulse rise and fall times and energy resolution relative to indium attachment. We also discuss the impact of clamp attachment on detector assembly efficiency and operating lifetime.

2.0 Background

DES measures the decay energy of each alpha-decay event in a sample that is embedded in a metal absorber and thermally linked to a superconducting Transition Edge Sensor (TES) microcalorimeter. These TESs operate in the superconducting phase transition of a superconducting

film, such that small changes in temperature from a single nuclear decay result in significant and measurable changes in resistance. This work uses TESs with a Mo-Cu bilayer with a superconducting critical temperature of around 100 mK. When operated under a voltage bias, each decay event produces a brief change in bias current through the TES, i.e. a pulse, whose amplitude is proportional to the energy deposited in the absorber [8]. Figure 1 diagrams the TES detector layout for DES-type measurements, highlighting factors to consider when assembling these devices such as heat capacity and thermal conductance. For TES microcalorimeter detectors, the decay time constant of a detector pulse is related to C/G (in the absence of electrothermal feedback from voltage bias), where C is the detector heat capacity (largely dominated by the heat capacity of the absorber) and G is the thermal conductance from the absorber to the cold bath [8]. Figure 1 shows two thermal links: G_2 between the absorber and TES, and G_1 between the TES and cold bath. G_1 is an inherent feature of the TES microcalorimeter chip that is controlled in the chip fabrication stages. This thermal conductance can be adjusted at fabrication by changing the length of the deep-etched silicon legs used for thermal isolation of the detector island [8]. G_2 can vary based on the absorber attachment method chosen during detector assembly, but, generally, G_2 should be much greater than G_1 so the thermal system behaves like a single heat capacity. In a single-body-like system with G_2 much greater than G_1, the pulse rise time is governed by G_2 and the fall time is dominated by G_1.

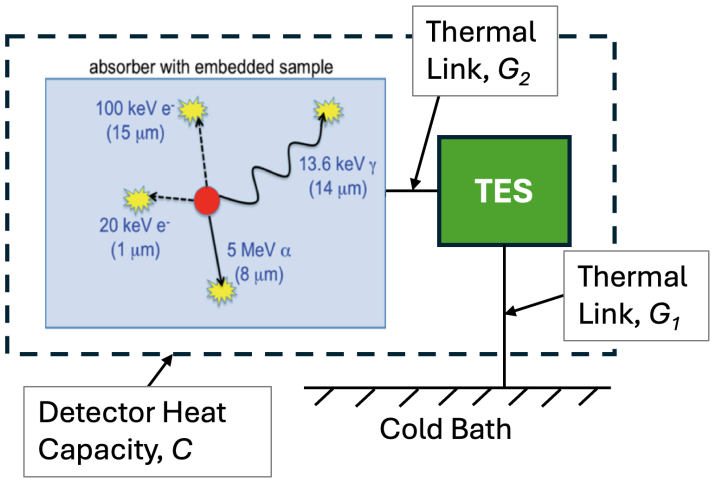
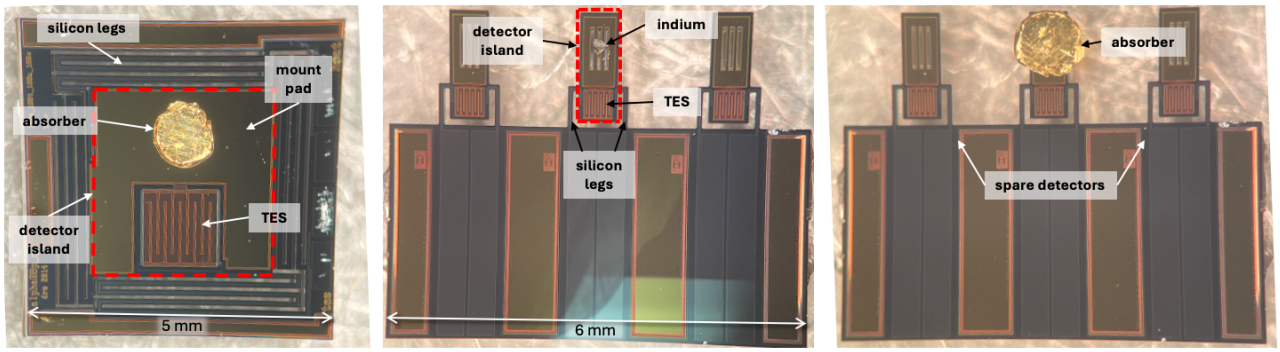


Figure 1: Conceptual detector setup for DES-type measurements. Pulse shapes are affected by the detector heat capacity (C) and the thermal conductance of the detector (G). There are two thermal links (G_1 and G_2) contributing to the overall conductance of the detector.

2.1 DES Detector Assembly

DES detector assembly is tied to radioactive sample preparation due to the integration of the sample into the absorber. The current method for sample preparation used at Los Alamos National Laboratory (LANL) is mechanical alloying, also called mechanical kneading. The procedure starts with depositing a drop of radionuclide solution onto a thin gold foil (typically 1 mm x 2 mm x 25 μm thick). Once dried, the gold foil is folded over and pressed between the jaws of pliers. This folding and pressing is repeated 100 times to ensure the dried sample deposits are broken up into very small size domains (~50 nm wide [1]) and distributed throughout the gold matrix. Though this procedure produces excellent results with very high energy resolution [1, 9, 10], it is tedious and time consuming, requiring around two to three hours per sample to complete the 100 kneading cycles. Ongoing research outside the scope of this paper aims to develop new methods to streamline DES sample preparation to further enhance sample throughput capabilities [10, 11].

After kneading, the gold absorber is attached to the microcalorimeter detector chip with a pressed indium metal bond. This is accomplished by placing a small piece of indium on the detector mount pad, then pressing the absorber into the malleable indium to form a metallic bond between the absorber and detector. During this process, the detector chip is on a clean flat surface to prevent uneven pressure from cracking the 380 μm thick silicon detector chip. Figures 2(a) and 2(c) show two styles of TESs with the same gold absorber attached via indium press bond. The square-style TES shown in Figure 2(a) uses long meandering silicon legs to isolate the detector island from the chip frame. The TESs shown in Figures 2(b) and 2(c) are a next-generation design by NIST that uses a robust all silicon detector configuration in which full wafer thickness silicon legs are used to set *G*. NIST refers to this new TES design as 'pronghorn' TESs. These pronghorn TESs, shown in Figures 2(b) and 2(c), include multiple detectors per chip, allowing for backup detectors in the event of accidental damage.



(a) Square-Style TES **(b) Pronghorn TES without absorber** **(c) Pronghorn TES with absorber**

Figure 2: Detector images. (a) Square-style TES with an absorber attached via indium press bond. Various detector components are labelled. (b) Pronghorn TES with an indium press bond before absorber attachment. (c) After absorber attachment. The same absorber is shown in both image (a) and (c).

Once the absorber is attached, the detector chip is mounted to the detector assembly which holds the circuitry for the SQUID (Superconducting Quantum Interference Device) current amplifier readout and the TES bias voltage, as shown in Figure 3. When mounting the detector, the detector island is thermally isolated so the decay heat from the sample flows directly to the TES and is not lost to the surroundings. Figures 2(a) and 2(b) highlight the detector island in red dashed lines, which consists of the absorber and the TES, and the silicon legs that isolate the detector island from the chip frame. Figure 4 shows a side view of the detector chip mounted on the detector assembly. The chip is mounted using BeCu clips that press down on two opposite corners of the detector chip frame. The clips are slowly and evenly tightened with screws, since too much pressure from either clip can cause the chip to bend and the silicon legs to snap. Once mounted, the detector chip is wire bonded with two wire bonds around a 90° angle to the bias voltage supply for the TES. When swapping samples, the wire bonds are removed, the BeCu clips are carefully loosened, and the chip is removed from the detector assembly before the current absorber can be removed and replaced. Additionally, any indium from the previous measurement is scraped off of the mount pad before a new indium press bond is placed, as excessive indium buildup can degrade detector performance by reducing the thermal conductance between the absorber and TES.

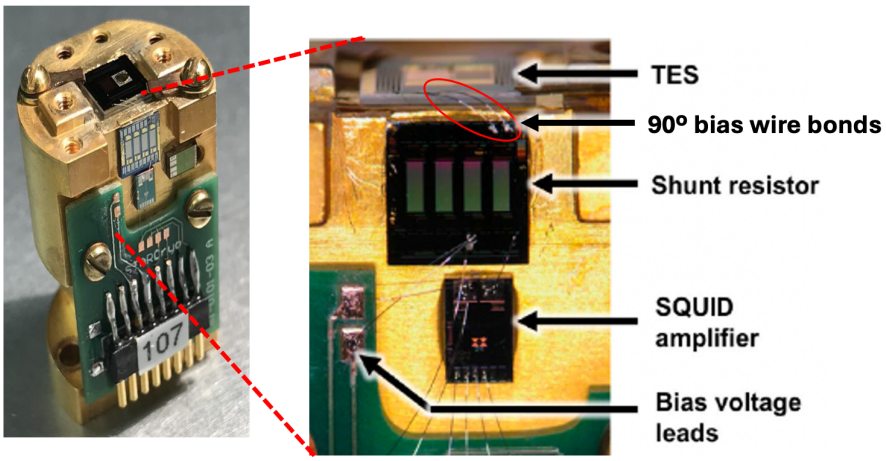


Figure 3: DES detector assembly. The 90° bias wire bond has to be removed with every sample change.

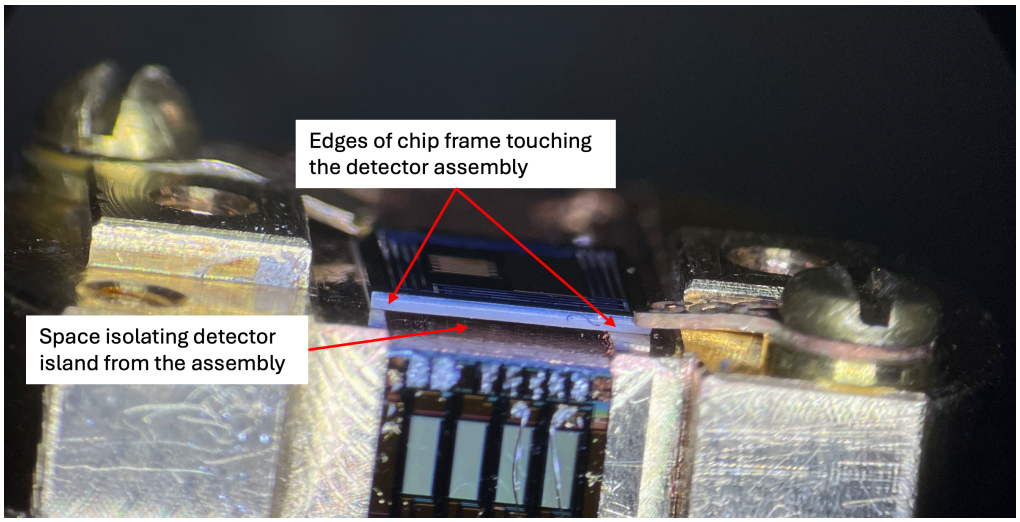


Figure 4: Side view of detector assembly. The TES detector chip is pressed onto the assembly using two copper clips. The detector assembly design allows the chip frame to be in contact with the metal tabs of the assembly while leaving space under the chip to isolate the detector island.

Overall, this process of assembling detectors becomes tedious because the detector chips are fragile, so each time the detector chip needs to be cleaned, handled, mounted, and unmounted, there is a risk of breaking the detector. Additionally, having to re-bond the 90° bias wire bonds after each sample swap is an inefficient process. For nuclear safeguards applications, high sample throughput is imperative, and an inefficient detector assembly procedure can impede throughput potential. A new method for absorber attachment that eliminates the need to remove the detector chip from the mount would streamline this assembly procedure and enable increased sample throughput.

2.2 Absorber Attachment Considerations

A recent review of DES measurements covers the various methods used to attach absorbers to sensors as a function of resolving power [9]. This work did not show a strong correlation between attachment method and resolving power, indicating investigations into more efficient attachment techniques are not likely to result in detriments to energy resolution. At LANL, early DES development used an epoxy-based attachment method that was phased out in favor of the indium

press bond which proved to be less permanent and easier to implement. Figure 5 shows the comparison of average detector pulses using epoxy and indium attachment methods, where the same absorber was used on the same square-style TES at the same detector bias voltage and measured at 80 mK bath temperature for both attachment methods. In Figure 5(a), the pulse tails were fit with an exponential decay function to determine the time constant, τ, which provides information about the pulse fall time. The exponential fit region for the epoxy pulse excludes a larger portion of the pulse due to athermal effects in epoxy that distort the pulse shape [12]. Both pulse tail fits were chosen only over the regions that appeared approximately exponential. With these fits, the indium attachment produced a time constant 1.5 times faster than epoxy. Because we do not expect a difference in G_1 between these two attachment methods, the difference in τ is likely due to an increase in heat capacity using the epoxy configuration, since epoxy has a larger heat capacity than indium at 100 mK [13]. Figure 5(b) shows a comparison of these pulse shapes with normalized pulse heights on a logarithmic scale. Figure 5(c) shows the comparison of pulse onset times. Comparing the onset times from 10% to 90% of the pulse heights, epoxy has a rise time 1.9 times faster than indium, indicating a higher G_2 thermal conductivity relative to indium. Despite epoxy's faster onset time, indium provides the better overall thermal performance by minimizing the pulse fall time, which equates to a reduced detector deadtime. However, indium has a relatively poor thermal conductivity at low temperatures, especially compared to gold [13]. With advances made in microcalorimeter design that have produced faster detectors, it is hypothesized that indium's poor thermal conductivity at low temperatures is hindering the full potential of these detectors.

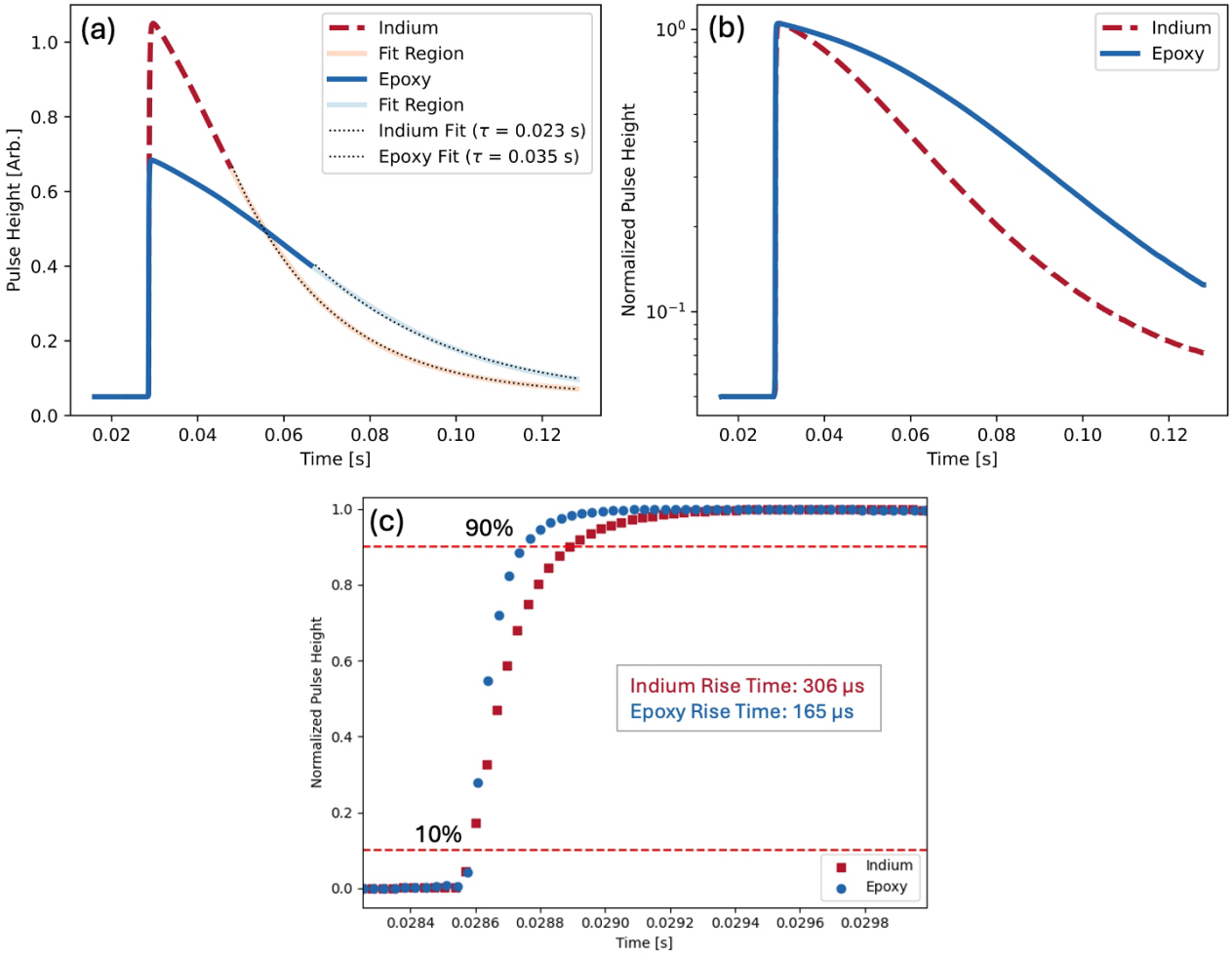


Figure 5: Pulse shape comparisons with indium and epoxy attachments. These two measurements used the same square-style TES, absorber, and measurement settings—only the attachment method differed. (a) Average detector pulse comparison between an indium attachment configuration and an epoxy attachment configuration. The tails are fit with an exponential decay function to determine the decay time constant, τ, for each method. (b) Normalized average detector pulses from indium and epoxy attachment configurations on a log scale. Comparison shows a decrease in pulse decay time (from 0.035 s to 0.023 s) using an indium attachment. (c) Average detector pulse onset time comparison between epoxy and indium attachment shows 1.9 times faster rise time using epoxy.

Safeguards applications require measurements with good statistics to be obtained quickly, so the supporting detectors need a high maximum count rate to accommodate higher activity sources. To enable count rates of higher activity sources without increasing pulse pileup, detector pulses with shorter tails are desirable. Because the decay time constant of a detector pulse is proportional to C/G, faster pulses can be accomplished by decreasing the heat capacity of the detector or increasing the thermal conductance. Heat capacity constraints are driven by the desired volume of the sample embedded in the absorber. Because our goal is to measure higher activity, higher volume sources, it is not feasible to reduce the heat capacity of our absorbers enough to achieve a faster decay time constant. Additionally, the heat capacity needs to be large enough to avoid saturating the TES detector. Therefore, this work focuses on increasing thermal conductance. Recent work by the

National Institute of Standards and Technology (NIST) has focused on creating faster TES detectors called pronghorns with enhanced G_1 (labelled in Figure 1 above) discussed in [14]. To benefit from these newer, faster pronghorn TES chips, the thermal conductance between the absorber and TES must also be increased (shown as G_2 in Figure 1).

This paper investigates the use of a new clamping method for attaching the absorber directly to the microcalorimeter chip without indium or adhesives. A clamping method must increase the thermal conductance between the absorber and TES, thus increasing the overall speed of our detectors. Eliminating the use of indium is also expected to extend the life of the detector chips by removing the need to clean and handle the delicate chips, thereby reducing the likelihood of accidental damage. Additionally, a sample swap using the clamping method would allow the detector chip to stay in place on the assembly, so this process would not involve redoing wire bonds, making the sample exchange procedure much simpler and more efficient.

To determine the effectiveness of an absorber clamp, an initial clamp design was tested using a pronghorn TES and absorber. Results using the clamp were compared to previous results using an indium press bond attachment with the same absorber and TES to assess the differences in average pulse shape. The following three results are evaluated: whether the clamping method (1) increases the speed of our detector pulses, (2) affects energy resolution (3) increases the sample swap efficiency relative to indium, and (4) has the potential to extend the lifetime of our detectors.

3.0 Absorber Clamp Design

The first step in designing an absorber clamp is choosing the material for the clamp. As discussed in Section 2.1, TES chips are designed to isolate the absorber and TES on a 'detector island'. This isolation ensures decay heat produced in the absorber from the sample flows directly to the TES. Introducing a clamp to the system breaks that isolation and risks the loss of decay heat through the clamp. Decay heat absorbed by the clamp results in partial detection of the decay heat, which will deteriorate energy resolution. To minimize energy loss through the clamp, the thermal conductivity of the clamp should be much lower than G_2. Additionally, the clamp material should not be magnetic, as the SQUID amplifier and TES are particularly sensitive to stray magnetic fields, which can degrade detector performance. With these considerations, a niobium wire was chosen to form an absorber clamp prototype. Niobium is a superconductor at low temperatures with low thermal conductivity, and it is not ferromagnetic like other spring materials [13].

For initial testing of an absorber clamp, we decided to use a very simple design concept that can be refined in the future to improve usability. This clamp design uses a piece of niobium wire (0.3 mm in diameter and 9 mm long) that is wrapped around a screw on the detector assembly and swiveled into place on top of the absorber similar to the movement of a record player arm. Figure 6 shows the design and prototype used for this experiment.

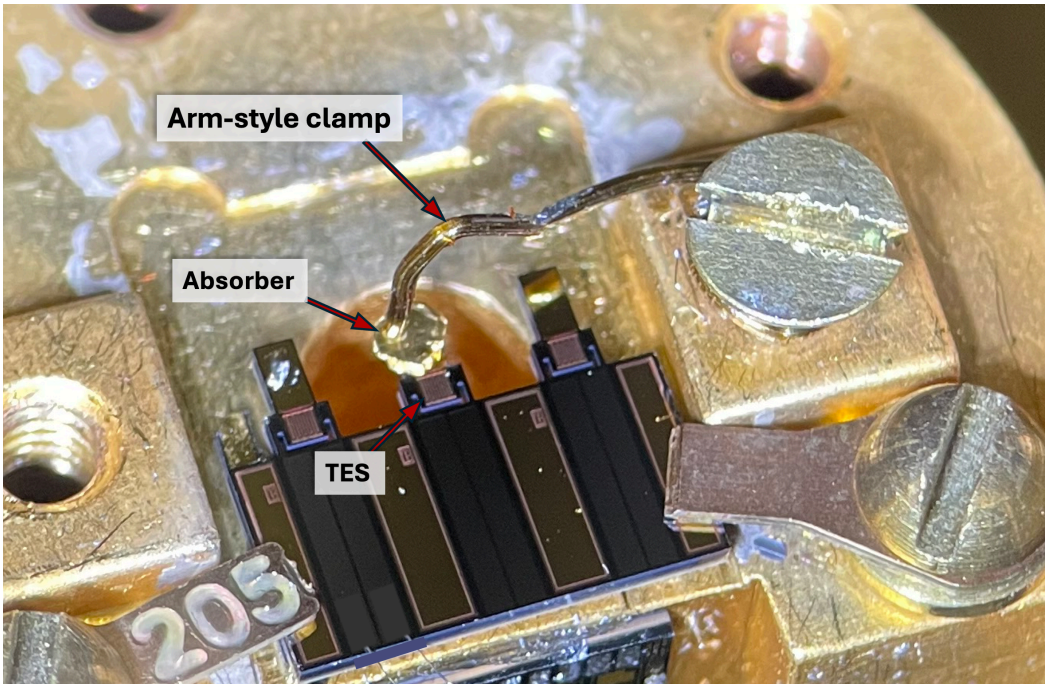


Figure 6: New clamp absorber attachment. Arm-style clamp formed from niobium wire wrapped around a screw, clamping an absorber onto a pronghorn TES.

Because the absorber is on the detector island suspended by thin silicon legs, a downward force from a clamp could cause the legs to snap and the detector to break. A pogo-pin style support on the detector assembly under the chip to counter the clamp force was proposed. However, initial testing of the arm-style clamp with the newer pronghorn TES, which uses much shorter silicon legs, has proven to be much sturdier than previous TES designs and has been capable of withstanding the force from the clamp. Solid modelling indicates that about 40 gram-force can be applied to the absorber mount pad of the pronghorn TES without exceeding the yield strength of the silicon. The clamp design used here applies approximately 10 gram-force. Future work should focus on methods of repeatedly applying a consistent force, since the thermal conductance between the absorber and TES is dependent on the magnitude of the force applied by the clamp.

Another feature of the clamp design to consider is electrical isolation. Initial testing of the clamp as described above showed an increase in noise relative to indium attachment, which was the result of a new pathway to ground through the clamp. To electrically isolate the clamp, the tip was coated in a layer of GE varnish, which is commonly used to insulate wires and as an adhesive at low temperatures [13]. The application of GE varnish successfully reduced the noise to expected levels. The results discussed in Section 4 were obtained with this addition of GE varnish to the tip of the clamp.

4.0 Results and Discussion

We have tested the arm-style clamp attachment method and the indium attachment method using the same absorber and the same pronghorn TES, with both measurements set to the same detector bias voltage and the same measurement temperature of 80 mK. Results from this test are shown in Figure 7 and Figure 8. It should be noted, these measurements comparing the indium and clamping configurations used a different absorber and a different style of TES than those shown in Figure 5.

In Figure 7, the tails of the average detector pulses are fit by exponential decay to determine the decay time constant, τ, for each method. Here, the average pulses were constructed from pulses in the energy range of 5.2 to 5.3 MeV, encompassing all pulses that contribute to the Pu-239 and Pu-240 peaks. Comparison of these two average pulse shapes shows τ is reduced by a factor of 2.3 using the clamp attachment method, indicating the increased thermal conductance provided by this attachment style can enhance our detector performance by providing faster pulses with shorter fall times. These results are consistent with an increase in G_1 due to the additional thermal pathway to the bath through the clamp. Also notable, the average pulse height in the clamp configuration increased, producing similar pulse integrals between the indium and clamp attachments with a 0.0002% difference. This suggests there was no significant loss of radionuclide decay heat through the clamp, indicating the choice of niobium wire for the clamp material was successful. Additionally, Figure 7(c) highlights the onset times of the normalized average detector pulses from clamp and indium attachment configurations. The sharp onset does not allow for adequate data points to interpolate rise times of the pulses, but we can conclude thermal performance is preserved with the new clamp attachment method.

Figure 8 shows a comparison of the spectral results of the datasets used in Figure 7, which compare the clamp and indium attachment configurations using the same absorber and same TES. This test used a Pu certified reference material, CRM137, with an activity of approximately 2 Bq. The Pu-239 and Pu-240 peaks in each spectrum were fit with a one-tailed Bortels function (i.e. a Gaussian convolved with one left-sided exponential) [15], shown in Figure 8. One- or two-tailed Bortels are often chosen for DES fits because they best describe the low energy tailing that occurs in decay energy spectra, which is best understood to be caused by energy loss due to lattice damage effects [1, 2, 16]. The choice of one- or two-tailed Bortels is empirical and is meant to best capture the counts in these low energy tails. From independent fits of the two spectra, we obtain a sigma from the Gaussian component of 0.763 keV ± 0.0102 keV and 0.450 keV ± 0.007 keV for the indium and clamped spectra, respectively. The FWHM of the Gaussian components of these peaks is therefore 1.80 keV ± 0.024 keV (indium) and 1.06 keV ± 0.015 keV (clamped). These FWHMs differ by 0.737 keV ± 0.028 keV. This difference in FWHM corresponds to a z-score of 26 (two-tailed $p < 10^{-146}$), thereby demonstrating an improvement in resolution from using a clamp style attachment over an indium attachment method. Additionally, the noise levels were comparable between the two measurements, indicating an increase in signal to noise ratio (SNR) with the increase in pulse height using the clamp, which is consistent with the improvement in energy resolution. However, the pulse height and fundamental detector noise are expected to scale by the same factor [8], suggesting the noise in our measurement is dominated by noise from the electronics, not the TES. This should not take away from the conclusion that the absorber clamp is a viable attachment method and improves detector performance relative to indium.

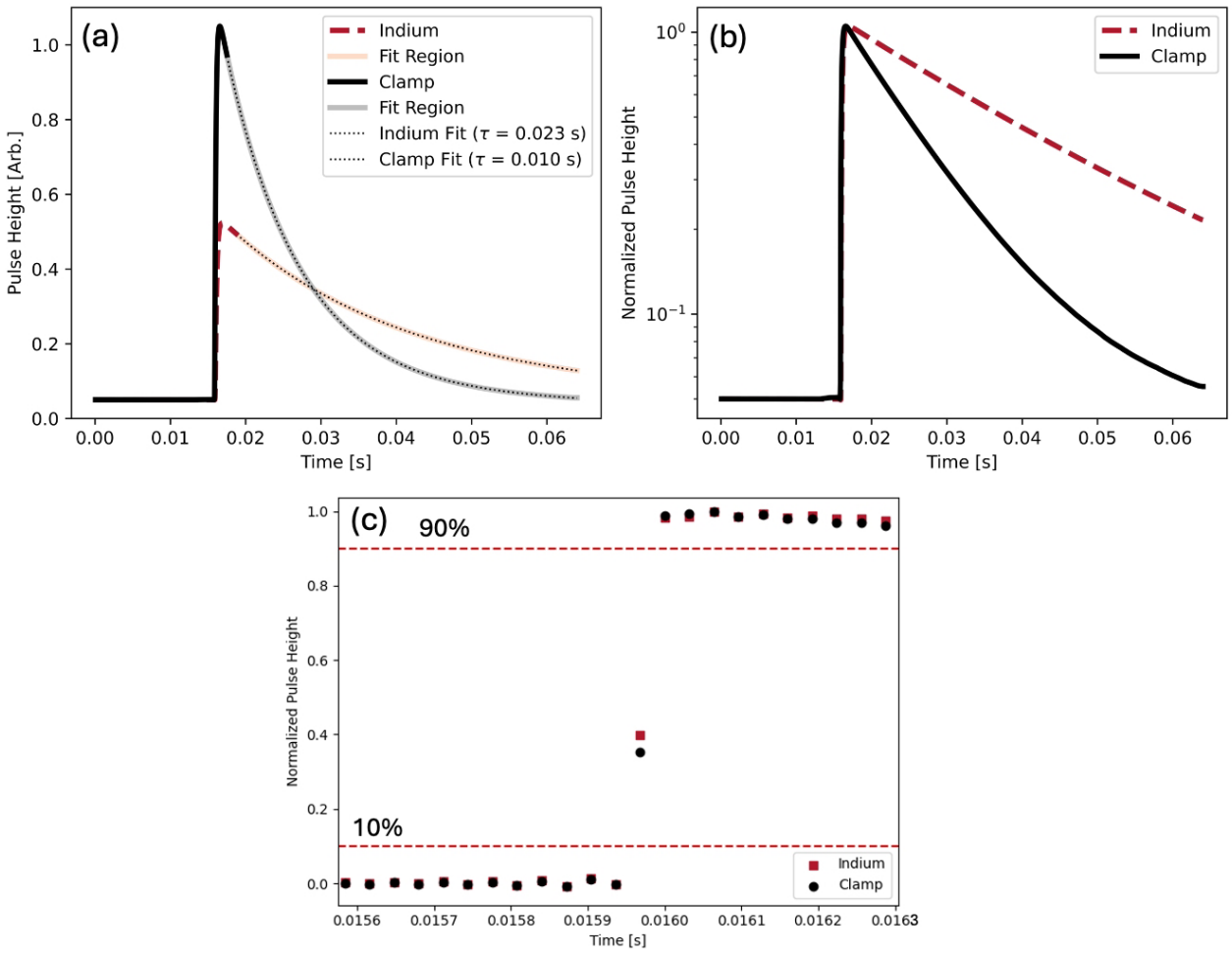


Figure 7: Pulse shape improvements with clamp attachment. These two measurements used the same pronghorn TES, absorber, and measurement settings—only the attachment method differed. (a) Average detector pulses with exponential decay fits using the indium attachment method and the clamp attachment method with the same absorber and same TES, set to the same detector bias voltage. The decay time constant, τ, is reduced by a factor of 2.3 using the clamp attachment method, indicating the increased thermal conductance provided by this attachment style can enhance detector performance. (b) Comparison of the two methods' average detector pulses with normalized pulse heights, shown on a log scale. (c) Comparison of pulse onset times using normalized pulse heights shows thermal performance is preserved with the clamp attachment. NOTE: The measurements shown in this figure did not use the same absorber or the same style of TES as the measurements shown in Figure 5.

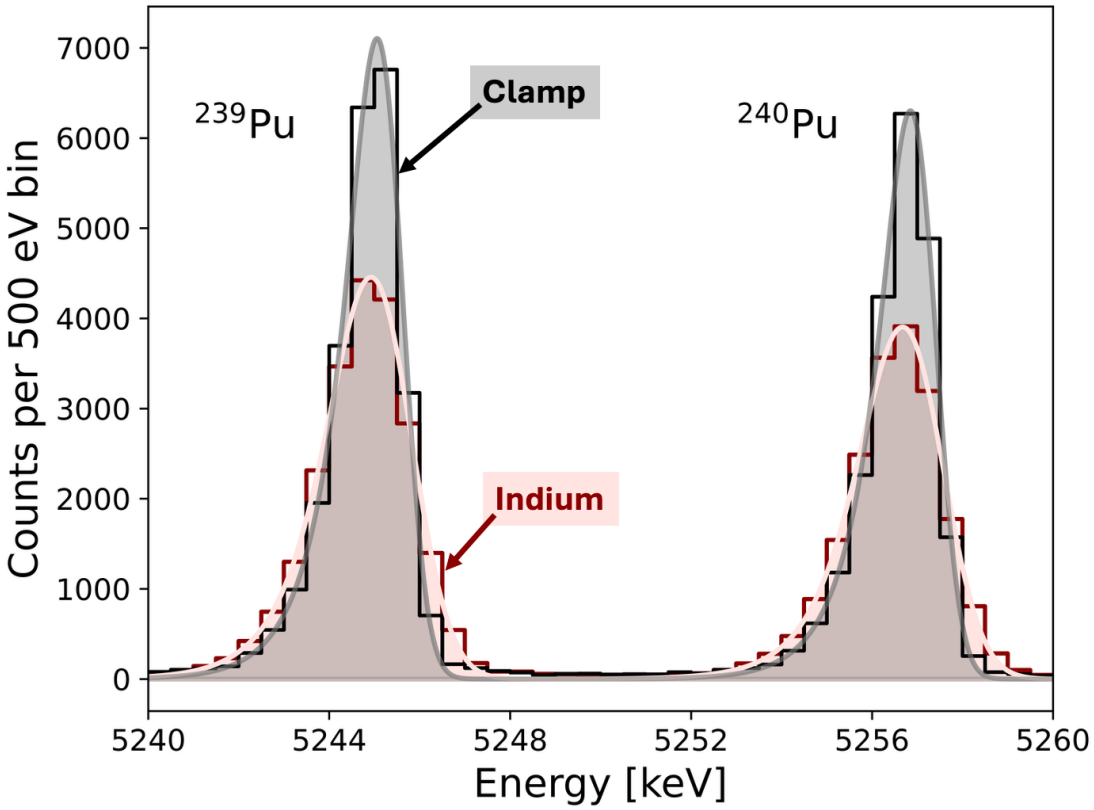


Figure 8: Spectra from two absorber attachment methods. Spectra of CRM137 demonstrate an improvement in resolution (41%) using a clamp attachment (data: black; fit: gray) over an indium attachment (data: dark red; fit: pink).

The second metric for success being evaluated is whether the clamp can streamline the detector assembly procedure. This metric is important to evaluate because we are interested in increasing our sample throughput capabilities, and a streamlined detector assembly procedure would aid in throughput. Using the clamp attachment method allows the detector chips to stay on the assembly. From initial tests using the clamping attachment method, it is clear that eliminating routine wire bonding and the need to remove detector chips from the mounts has significantly streamlined our sample swap procedure.

Lastly, we are interested in evaluating whether the clamp can extend the life of our detector chips. Because the TES chips are fragile, any handling, cleaning, mounting and unmounting increases the likelihood of accidentally damaging a functional chip. In theory, with the indium attachment method the detector chips can be re-used indefinitely. However, LANL experience indicates that chips often need replacement due to accidental damages during removal from the detector assembly, and because the absorber mount pad gets worn out over time. Because we proved a clamping method eliminates the need to remove the chip from the assembly during a sample swap, we have removed the riskiest parts of the procedure. This means our chips should last longer and need fewer replacements for damaged detectors. Also notable, this pressure-based clamping method could be similarly beneficial to DES measurements using magnetic microcalorimeters (MMCs), which typically use several gold wire bonds between the absorber and MMC to make a thermal connection [5, 11]. A clamping attachment method applied to MMCs would eliminate these gold wire bonds and could similarly extend the lifetime of the detectors.

5.0 Conclusions

This work presents our investigation into the use of a clamp as a new method for absorber attachment to increase the speed of TES detector pulses, increase the efficiency of sample swapping, and extend the lifetime of TES chips. A clamp prototype was formed from niobium wire and tested. The average detector pulse shape was compared to that of an indium attachment configuration. The decay time constant of the average detector pulse was reduced by 57% using the clamp attachment method, indicating that the increased thermal conductance provided by this attachment style can enhance our detector performance. Comparison of the resulting spectra from these two device configurations showed the clamp attachment preserves detector performance relative to indium, and the clamp may even improve energy resolution—though further testing is needed. Additionally, a streamlined sample swapping procedure was demonstrated using the clamp attachment method relative to indium, which eliminates routine wire bonding as well as the need to remove the detector chip from the detector assembly. Eliminating the need to handle and clean the detector chips also allows for a longer chip life by reducing the likelihood of accidental damage.

Overall, we have demonstrated the feasibility of an absorber clamp for DES measurements, including some clear advantages of this new attachment method. Future work will focus on refining the design of the clamp to increase usability and to ensure a consistent, repeatable force is applied. Work is already underway to incorporate the use of clamping attachment into future measurements and instrument designs.

CRediT Authorship Contributions

Sophie L. Weidenbenner: Conceptualization, Formal analysis, Investigation, Visualization, Writing – original draft
Katrina E. Koehler: Formal analysis, Resources, Visualization, Writing – reviewing & editing, Supervision
Mark P. Croce: Conceptualization, Resources, Funding acquisition, Supervision, Project administration, Writing – reviewing & editing
Matthew H. Carpenter: Supervision, Resources
Rico U. Schoenemann: Investigation, Writing – reviewing & editing, Resources
Katherine A. Schreiber: Writing – reviewing & editing, Resources
Daniel T. Becker: Resources
Jason E. Nobles: Resources
Daniel R. Schmidt: Resources
Joel N. Ullom: Resources
Camille J. Palmer: Supervision

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