

Superconducting Transition Edge Sensor for Gamma-Ray Spectroscopy

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SUMMARY Superconducting Transition edge sensor (TES) coupled with a heavy metal absorber is a promising microcalorimeter for Gamma-ray (γ -ray) spectroscopy with ultra-high energy resolution and high detection efficiency. It is very useful for the non-destructed inspection of the nuclide materials. High resolving power of γ -ray peaks can precisely identify multiple nuclides such as Plutonium (Pu) and Actinides with high efficiency and safety. For this purpose, we have developed the TES coupled with a tin absorber. We suggest the new device structure using the gold bump post which connects a tin absorber to the thermometer of the superconducting Ir/Au bilayer. High thermal conductivity of the gold bump post realized strong thermal coupling between the thermometer and the γ -ray absorber, and it brought the benefit of large pulse height and fast decay time. Our TES achieved the good energy resolution of 84 eV FWHM at 59.5 keV. Using this TES device, we also succeeded to demonstrate the nuclear material measurements. In the measurement of a Pu sample, we detected the sharp γ -ray peaks from ²³⁹Pu and ²⁴⁰Pu, and of a Fission Products (FP) sample, we observed fluorescence X-ray peaks emitted by the elements contained in FP. The TES could resolve the fine structures of each fluorescence X-ray line like $K_{\alpha 1}$ and $K_{\alpha 2}$. In addition to that, we developed the TES coupled with tantalum absorber, which is expected to have higher absorption efficiency for γ -rays. This device reported the best energy resolution of 465 eV at 662 keV.

key words: transition edge sensor, gamma-ray, nuclide analysis

1. Introduction

The nuclide analysis methods are broadly separated into two categories. One is the non-destructed inspection based on the measurement of Gamma-ray (γ -ray) or neutron emitted by the nuclide samples, and another is the destructed inspection based on chemical analysis. It is notable that Actinides are considered as most hazardous radionuclides due to their high radiological and chemical toxicities. Separation

of Actinide elements applying the solvent extraction and the collection of the chemical precipitate is allowed to analyze the samples accurately, however, it is extremely inefficient because it requires many complicated and dangerous work for many hours. On the other hand, the non-destructed inspection by applying the γ -ray spectroscopy allows a rapid analyzation and also it is superior in the safety and the efficiency than the destructed inspection. However, it is quite difficult to precisely identify the nuclides because the energy peaks derived from the nuclide elements such as Pu (Plutonium) and Am (Americium) overlap each other on the γ -ray energy spectra due to the low energy resolution of the conventional γ -ray detectors, such as the scintillation detectors or the High-purity Germanium (HPGe) detectors. In order to maintain effective nuclear safeguards, it is necessary to establish the non-destructive inspection of the nuclide elements like the spent nuclear fuel, with high efficiency, safety and more accuracy. These requirements lead us to the precise γ -ray spectroscopy using the ultra-high energy resolution superconducting transition edge sensor (TES) [1], [2].

2. Transition Edge Sensor for Gamma-Ray Spectroscopy

2.1 Principle of the Sensor Operation

The TES is able to precisely detect the energy of an incident photon, such as γ -ray, by detecting the temperature increase derived from the absorption of the photon. It is a very sensitive thermometer, utilizing the superconducting phase transition between the superconducting state and the normal state. The operating principle of the TES microcalorimeter is shown in Fig. 1. The sensor consists of the radiation absorber, the thermometer and the thermal link. The thermometer is made by a superconducting thin film which is biased in a constant voltage. Now we assume that the temperature of the thermometer is in the region of the superconducting phase transition, such as the superconducting film is heated by the Joule heating. When a γ -ray photon is incident on the TES, temperature of the superconducting film is increased and its resistance is also increased. Therefore Joule heating is decreased because the Joule heating is inversely proportional to R . The TES is cooled down quickly by this negative electrothermal feedback (ETF). Then the deposited energy is removed by the reduc-

Manuscript received July 11, 2016.

Manuscript revised November 28, 2016.

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DOI: 10.1587/transele.E100.C.283

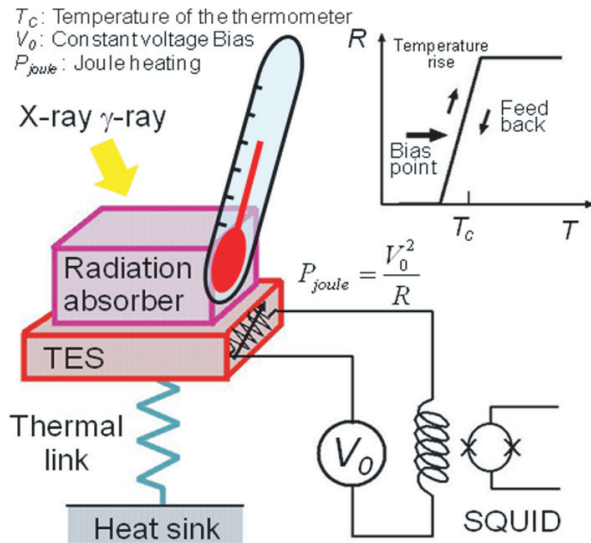


Fig. 1 The operating principle of the ETF-TES.

tion of the Joule heating. A constant heat flows from the sensor to the heat bath through the weak thermal link, and the power reduction of the Joule heating compensates the energy of the deposited γ -ray photon. Applying this negative feedback mechanism, the constant voltage bias stabilizes the operating point on the transition curve, where heat flow to the heat bath is equal to the Joule heating, and very high count rate is allowed [3].

2.2 γ -Ray Absorber

To realize a higher detection efficiency for γ -rays, a heavy metal bulk is the ideal material for the γ -ray absorber due to its high stopping power for γ -rays [1]. We considered three kinds of heavy metals, Sn (tin), Ta (tantalum) and Pb (lead). The heat capacity at 100 mK and absorption efficiency for γ -rays of each material is shown in Table 1. These values are calculated for each absorber size of 0.5 mm \times 0.5 mm \times 0.3 mm-thick. Figure 2 shows the calculated absorption efficiency of each material. Because all of these materials have relatively higher superconducting transition temperatures, the electronic specific heat drops below the normal electronic specific heat at about $T \sim T_c/2$ and then it drops to zero quickly as the temperature decreases. Therefore each heat capacity is quite suppressed at the sensor operating temperature of around 100 mK. However, especially the heat capacities of Sn and Ta are much more smaller than that of lead at 100 mK. Furthermore, the stopping power for γ -rays of Ta is superior to that of Sn. Especially, for the γ -ray of 600 keV, the absorption efficiency of Ta is about 4 times larger than that of Sn. Considering these qualities, Ta is looked like the best candidate as the material of γ -ray absorber. However, in our research works, it becomes clear that the Ta has a variation of the heat capacity. We consider that this variation is brought by the molecular adsorption characteristics on the Ta surface [1].

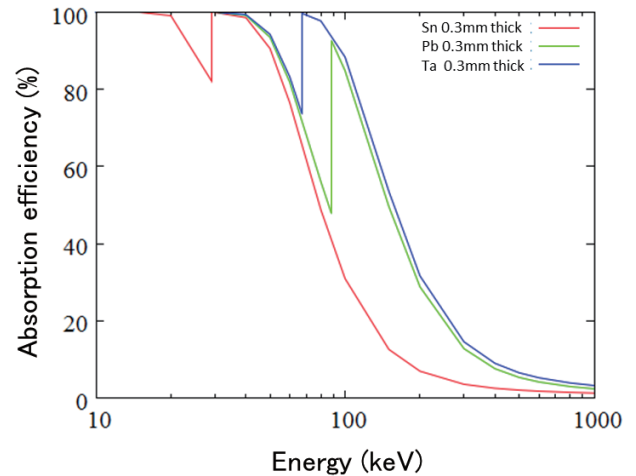


Fig. 2 Absorption efficiency for γ -ray of Sn, Pb, Ta.

Table 1 Properties of Gamma-ray absorber material.

Material	T_c (K)	Heat capacity @100 mK (keV / mK)	Absorption Efficiency @ 100 keV (%)	Absorption Efficiency @ 600 keV (%)
Sn	3.72	5.96	30.9	1.39
Ta	4.5	4.87	89.8	5.23
Pb	7.2	43.0	84.9	4.15

Calculated value for each materials for 0.5mm \times 0.5mm \times 0.3mm size.

3. γ -Ray TES Coupled with Tin Absorber

3.1 The Structure of the Sensor and the Sensor Fabrication

In the γ -ray energy spectrum obtained from the nuclear sample of the spent nuclear fuel, many of the energy peaks derived from the nuclide elements such as Pu, U (uranium), Am, and Np (neptunium), are concentrated in the energy range from 30 keV to 150 keV. In order to achieve the precision γ -ray spectroscopy for this energy region, we have developed the TES coupled with a Sn absorber. We consider that the strong thermal coupling between the thin superconducting film and the Sn absorber provides the integrally operation with the γ -ray absorber and the thermometer, although only the superconducting film is heated directly by Joule heating, and suppression of the thermal fluctuation noise which depends on the thermal conductance between the γ -ray absorber and the thermometer. To realize the strong thermal coupling between the thermometer and the γ -ray absorber, we have introduced the gold bump post which connects the tin absorber to the superconducting film.

We used an Ir (Iridium)/Au (gold) bilayer superconducting film as the thermometer [4]. Figure 3 shows our Ir/Au -TES device of 0.25 mm \times 0.25 mm in size coupled with a Sn absorber of 0.5 mm \times 0.5 mm \times 0.3 mm in size.

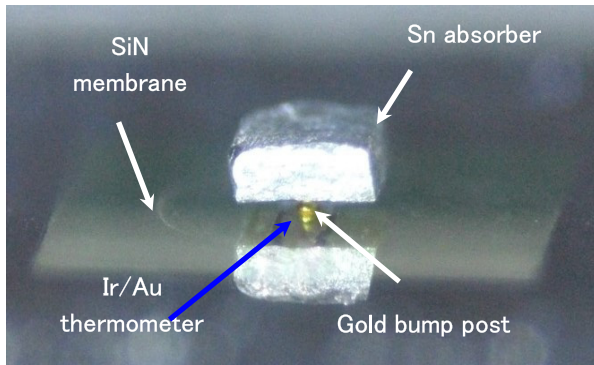


Fig. 3 The TES with a tin absorber for Gamma-ray detection.

The Ir/Au TES film was deposited on a $1\ \mu\text{m}$ thick silicon nitride membrane. This thin silicon nitride membrane is used as the weak thermal link which has very low thermal conductivity, which restricted the heat flow from the TES to the heat bath. Ir/Au film was fabricated on the center of a $1.2\ \text{mm} \times 1.2\ \text{mm}$ silicon nitride membrane. The silicon nitride was deposited on both sides of silicon wafer by LPCVD. For the silicon etching, windows were opened in the silicon nitride film on the back surface of the wafer. The Ir/Au bilayer film was deposited on the front side by a magnetron sputtering method while heating the substrate to 600 K and it was etched by a reactive ion etching method using BCl_3 reactive gas. The thickness of the Ir and the Au are 100 nm and 15 nm respectively and this Ir/Au film showed the reproducible sharp superconducting transition at 145 mK with a transition width of less than 1 mK. Nb (Niobium) electrode was deposited by the sputtering method and patterned using a lift-off process. Before silicon etching from back surface, the gold bump post was made on the center of the Ir/Au bilayer film. Then silicon was wet etched in a hydrazine mono-hydrate solution from the back surface of the wafer. In the final process the Tin absorber was mounted on the gold bump post using a tiny amount of epoxy glue [5], [6].

3.2 Performance of Gamma-Ray Detection

The detector and the current read out circuit were mounted on a cold stage in a dilution refrigerator with pulse-tube pre-cooling head. Our experiments of γ -ray detection indicated that the mechanical vibration derived from the pulse-tube had great effect on the base line noise of the output current from the TES detector coupled with the heavy metal absorber. Therefore to achieve the good energy resolution, propagation of the mechanical vibration from the pulse-tube unit to the cold stage in the dilution refrigerator should be suppressed as small as possible. Figure 4 shows the photograph of our dilution refrigerator system which is used for the γ -ray TES operation. The pulse-tube unit, which was suspended from a top board through several vibration dampers, is separated from the dilution Dewar vessel, and they are connected with welding bellows. Current through the TES was measured with a one-stage superconducting

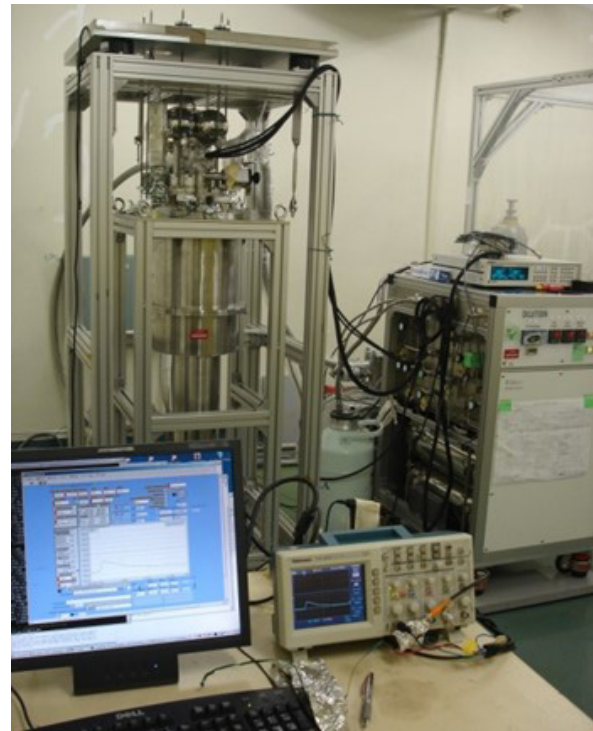


Fig. 4 The dilution refrigerator with pulse-tube pre-cooling head.

quantum interference device (SQUID) current amplifier that consists of 100 series-array dc-SQUIDs. The TES is connected in series to the SQUID input coils and in parallel to the shunt resistor, in which resistance is much smaller than the TES resistance, such as a constant voltage bias is applied to the TES. As this device showed a normal resistance of $850\ \text{m}\Omega$ at 1 K, we choose a shunt resistor of $20\ \text{m}\Omega$.

The TES was cooled to around 100 mK, and it was biased at the resistance of $85\ \text{m}\Omega$ (10% of normal resistance) in the transition edge. Then the γ -rays were incident on the TES from a γ -ray source at the room temperature through a beryllium window of the dilution Dewar vessel. Figure 5 shows the measured current pulse of ^{241}Am (59.5 keV) γ -ray event. In this figure the pulse shape of the new device using the gold bump post is compared with the pulse shape of the previous device using the epoxy post (Stycast 2850FT). The pulse height of the signal of the Au bump post TES was larger than that of the epoxy post TES by more than 6 times. The inset of Fig. 5 shows the normalized plot of these pulse shapes with respect to the maximum pulse height of the Fig. 5. In the decay curve of each plot we can observe a slow component following the fast component. However the difference between the slow component and the fast component of the epoxy post TES seems to be larger than that of the gold bump post TES. The time constant of slow decay component improved from 24 ms of the epoxy post TES to less than 5 ms of the gold bump post TES. Although the origin of these two time components in the decay curve is still not clearly understood, we consider that the strong thermal coupling between the Ir/Au film and the Sn absorber, which

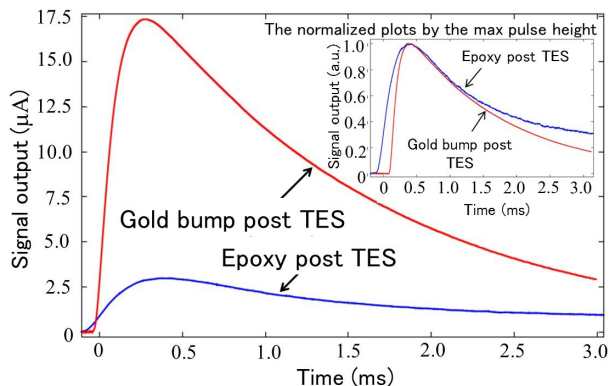


Fig. 5 The signal current pulse of ^{241}Am (59.54 keV) γ -ray event, measured by the TES with a gold bump post comparing with that of the TES with the epoxy post, and the normalized plots by the max pulse height (inset).

is brought by the high thermal conductivity of the gold bump post, may suppress the decay time constant of the slow decay component [7], [8].

γ -ray measurements taken by putting two γ -ray sources of ^{241}Am and ^{57}Co together in front of the beryllium window, obtained the energy spectrum as shown in Fig. 6. We observed the photopeak of ^{241}Am at 59.54 keV, and two escape peaks at 31.07 keV and 34.35 keV, which correspond to the escape energy loss of the Sn $K\beta$ and $K\alpha$ X-rays, respectively. And also, we observed the photopeak of ^{57}Co at 122.06 keV and the small peaks at around 95 keV, which arise from the escape events of Sn $K\beta$ and $K\alpha$ X-rays. In the analysis of γ -ray spectrum obtained by the TES detector, we should pay attention to these escape events and need to clean up these artifacts. These escape events complicate identification of the nuclide much more in heavy metal materials used for γ -ray absorber than in Si (Silicon) or Ge (Germanium), due to the much higher energies of the characteristic X-rays. In this spectrum as shown in Fig. 5, the energy resolution of 59.54 keV peak is estimated to 84 eV FWHM. This resolution is more than 4 times better than the energy resolution of HPGe whose theoretical limit is 400 eV at 60 keV by the Fano statistics of electrons and holes, produced by the incident photons. However, our energy resolution is still limited by the low frequency noise and the mechanical vibration of the pulse-tube pre-cooling head of our dilution refrigerator system dominates it. Figure 7 shows the base line noise power spectrum of this TES detector. We observed the very large noise peaks, at 873 Hz, 1.77 kHz and 2.87 kHz which is derived from the vibrations of the refrigerator, because we confirmed that these noise peaks were disappeared by temporary stopping of the pulse-tube unit. These effects of the mechanical vibration on the frequency band around these noise peaks might contribute considerably to degrade the energy resolution. We consider that vibration level is much higher because our refrigerator system uses the valve motor integrated pulse-tube unit. Therefore now we are replacing it to a pulse-tube unit, which is separated from the valve motor unit to achieve a better energy resolution.

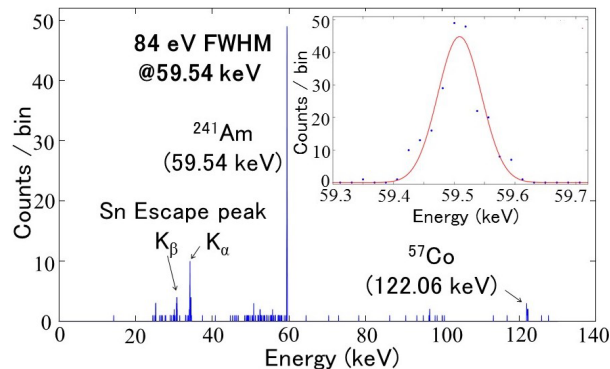


Fig. 6 The energy spectrum of γ -ray from ^{241}Am source and ^{57}Co source, measured by the TES with a tin absorber.

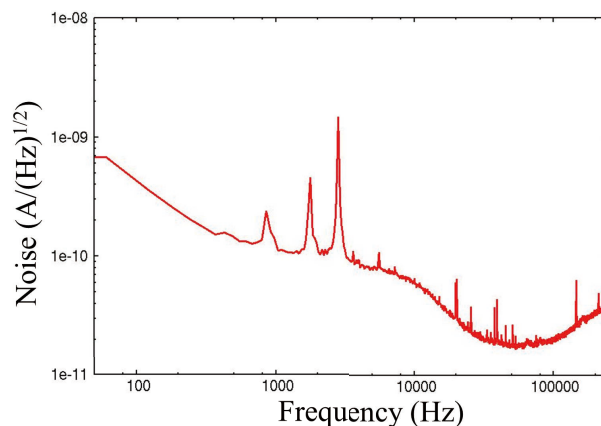


Fig. 7 The base line noise power spectrum of TES coupled with tin absorber, which cooled by the dilution refrigerator with pulse-tube pre-cooling unit.

4. Nuclear Material Measurement

To demonstrate the precision measurements of nuclear material samples by the TES coupled with a Sn absorber, we moved the dilution refrigerator integrated TES detector and the signal read out system described above, to the facilities of Japan Atomic Energy Agency (JAEA).

First, γ -ray measurements from a Pu nitrate sample were performed. Also, for reference of the energy spectrum, the same Pu sample was measured by the HPGe detector. The energy spectrum of the TES detector comparing with that of the HPGe detector is shown in Fig. 8. In the TES spectrum, we can identify the sharp energy peaks derived from the ^{239}Pu at 38.66 keV, 51.62 keV and 129.30 keV among many γ -ray peaks. And, at 45.24 keV the energy peak derived from ^{240}Pu is also identified. On the other hand, these energy peaks from Pu nuclides cannot be confirmed or can be hardly confirmed in HPGe spectrum. Therefore, HPGe detector cannot evaluate precisely the concentration ratio of these Pu nuclides in this spectrum. From these results, it is notable that the TES detector has the ability to identify the isotope ratio of Pu nuclides, and we expect

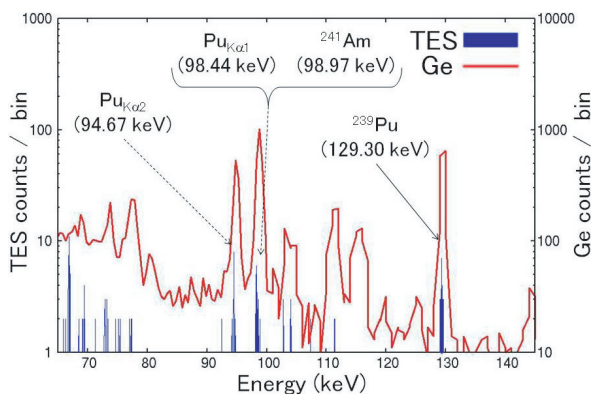
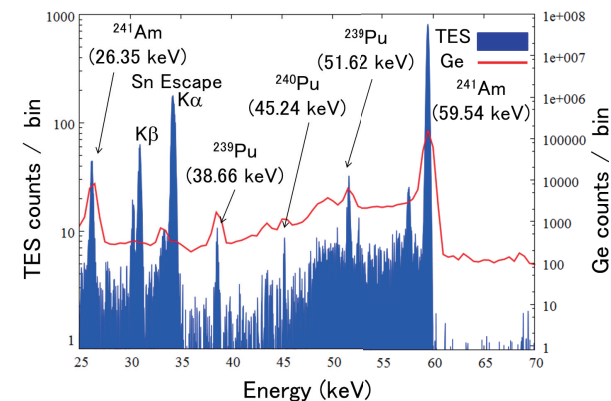


Fig. 8 The energy spectrum of γ -ray from Pu sample, measured by the TES comparing with that of the HPGe detector. (upper: 25 ~ 70 keV, lower: 70 ~ 150 keV)

that TES detector become powerful tool of the advanced nuclear material analysis of the next generation nuclear safeguards. The energy peaks of characteristic X-rays emitted by Pu are also founded. We estimate the peak at 94.70 keV as Pu $K\alpha_1$, and however the peak of Pu $K\alpha_2$ at 98.44 keV are not completely separated from γ -ray peak of ^{241}Am at 98.97 keV due to the statistical dispersion.

Next, we tried to measure γ -rays from nitrite solutions which contains Fission Products (FP) derived from the spent nuclear fuel. Although the TES detector was exposed by many Compton scattered photons from a ^{137}Cs , we succeeded to demonstrate the identification of the nuclides which was contained in FP sample. Figure 9 shows γ -ray energy spectrum of FP sample measured by the TES detector, and also represents the energy spectrum measured by HPGe for comparison. In this spectrum we observed several energy peaks of characteristic X-rays emitted by the elements of Cs (Ba), and Eu (Gd), contained in FP, and γ -ray peaks of ^{241}Am , ^{154}Eu , and ^{155}Eu . It is remarkable that, in each energy peak, TES can resolve the fine structures of the fluorescence X-ray line like $K\alpha_1$ and $K\alpha_2$, which cannot be resolved by HPGe detector. We expect that this strong resolving power of γ -ray energy peaks might contribute to improve the detection sensitivity of nuclides under the high background by Compton scattered events of high energy γ -rays in the assumption of the inspection of the spent nuclear

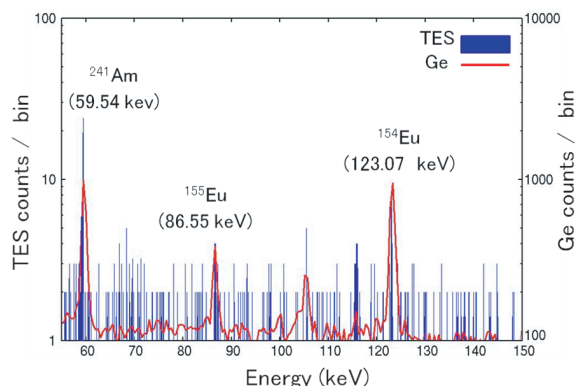
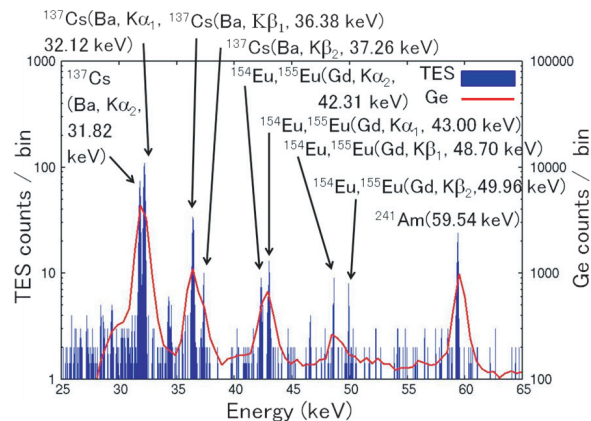


Fig. 9 The energy spectrum of γ -ray from nitrite solutions which contains FP derived from the spent nuclear fuel, measured by the TES comparing with that of the HPGe detector. (upper: 25 ~ 65 keV, lower: 58 ~ 150 keV)

fuel.

5. TES Coupled with Tantalum Absorber

Improvement of the detection efficiency of γ -rays around 100 keV is expected for the nuclide analysis of Pu and actinide, therefore we focus on the property of Ta, which features small heat capacity and large stopping power for γ -ray as shown in Table 1. Further, Ta has measurable absorption efficiency for γ -ray energies from 500 keV to 1 MeV. If the TES detector realizes the high energy resolution spectroscopy in this energy range, it might be useful for Coincidence Doppler Broadening (CDB) Positron Annihilation Spectroscopy (PAS) studies, which gives information on the electron momentum distribution in the sample with the irradiation damage or the lattice defect [9]–[12].

Our TES coupled with a Ta absorber was fabricated in a similar way as the TES coupled with Sn absorber [13]. Figure 10 (upper photograph) shows the TES with Ta absorber of 0.5 mm \times 0.5 mm \times 0.3 mm in size. The gold bump post connects Ta absorber to the superconducting Ir/Au bilayer film which deposited on the silicon nitride membrane of 1 μm thick. We cooled this TES sensor using the dilution refrigerator as same as before. The cold stage of the refrigerator was maintained at 100 mK, and the measurement of

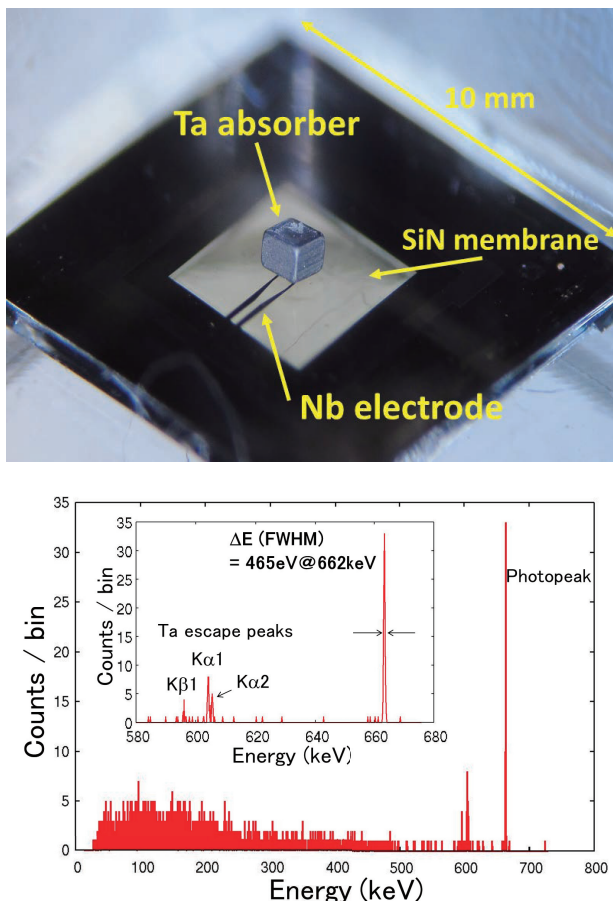


Fig. 10 The TES with a Ta absorber for Gamma-ray detection and the energy spectrum of γ -ray from ^{137}Cs source.

γ -ray from a ^{137}Cs source was performed. We found out that the real heat capacity of this device that was calculated from the decay time constant of γ -ray incident signal was much larger than the theoretical heat capacity. This increase of the heat capacity may be brought by the hydrogen absorption on the Ta surface as other research group pointed out [1]. In addition to that, because Ta absorber is heavier than the Sn absorber, the vibration of the refrigerator results a larger noise level on the TES detector and it degraded the energy resolution. The γ -ray energy spectrum measured by the TES with Ta absorber is shown in Fig. 10 (lower). We can observe a very sharp photopeak at 662 keV and the escape peaks at 596.8 keV, 604.5 keV and 605.8 keV, which correspond to the escape energy loss of the Ta $K\beta$, $K\alpha_1$ and $K\alpha_2$ X-rays, respectively. Although the energy resolution is limited by the noise derived from the vibration of the refrigerator and by the increase of the heat capacity, we achieved a good energy resolution of 465 eV FWHM at 662 keV. This value is the best energy resolution in this energy range, while HPGe detector is still limited to 1.5 keV at 662 keV. Further, due to this good energy resolution and high absorption efficiency, peak-to Compton ratio reaches 30 in this spectrum, while the ratio of typical Ge detector is around 50.

6. Conclusion

We have developed a TES coupled with a bulk heavy metal absorber for γ -ray spectroscopy with ultra-high energy resolution. To realize the strong thermal coupling between the γ -ray absorber and the thermometer, we introduced the gold bump post which coupled the heavy metal absorber to the superconducting Ir/Au bilayer film, and we succeeded to get a large pulse height and a fast decay time. Our TES with Sn absorber achieved the good energy resolution of 84 eV at 60 keV. And also our TES with Ta absorber achieved the best energy resolution of 465 eV at 662 keV. These resolution is much better than that of HPGe detector, although it is still now limited by the noise derived from the vibration of the dilution refrigerator system. Further, we performed the nuclear material measurements by using TES with Sn absorber. In the measurement of Pu nitrate sample, we succeeded to identify the sharp energy peaks derived from the ^{239}Pu and ^{240}Pu , in which HPGe detector could not identify clearly. In the measurement of FP sample, we succeeded to detect several energy peaks of characteristic X-rays emitted by the elements contained in FP and γ -ray peaks of ^{241}Am , ^{154}Eu , and ^{155}Eu with much better energy resolution than that of HPGe detector. Based on these results, we consider that γ -ray spectrometer using a TES detector can be applied for the advanced non-destructed inspection of the nuclide material.

Acknowledgments

This work was financially supported by “The Center of World Intelligence Project for Nuclear S&T and Human Resource Development”, Ministry of Education, Culture, Sports, Science and Technology (MEXT), and SENTAN, Japan Science and Technology agency (JST). And also this was supported by JSPS KAKENHI Grant Number 15H02341 and 26630480.

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