Response of a Superconducting Transition-Edge Sensor Microcalorimeter with a Mushroom-shaped Absorber to L X-rays Emitted by Transuranium Elements

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SUMMARY A four-pixel-array superconducting transition-edge sensor (TES) microcalorimeter with a mushroom-shaped absorber was fabricated for the energy dispersive spectroscopy performed on a transmission electron microscope. The TES consists of a bilayer of Au/Ti with either a 50-nm or 120-nm thickness. The absorber of 5.0-µm thick is made from a Au layer and its stem is deposited in the center of the TES surface. A Ta2O5 insulating layer of 100-nm thickness is inserted between the overhang region of the absorber and the TES surface. A selected pixel of the TES microcalorimeter was operated for the detection of Np L X-rays emitted from an 241Am source. A response of the TES microcalorimeter to L X-rays was obtained by analyzing detection signal pulses with using the optimal filter method. An energy resolution was obtained to be 33 eV full width at half maximum value at 17.751 keV of Np LxII considering its natural width of 13.4 eV. Response to L X-rays emitted from a mixture source of 238Pu, 239Pu, and 241Am was obtained by operating the selected pixel of the TES microcalorimeter. Major L X-rays peaks of progeny elements of 239Pu and Am isotopes were clearly identified in the obtained energy spectrum. The experimental results demonstrated the separation of 241Am and plutonium isotopes by L X-ray spectroscopy.

key words: TES microcalorimeter, Mushroom shaped absorber, X-ray response, Energy Resolution, L X-ray spectroscopy

1. Introduction

Plutonium isotopes are handled with special care in nuclear fuel cycle facilities such as reprocessing plants and mixed oxide fuel fabrication facilities. Most plutonium isotopes decay into uranium progenies by emitting mono-energy α rays. The isotope of 241Pu decays to 241Am isotope by emitting a β particle. Plutonium isotopes are usually analyzed by α ray spectrometry after a complicated chemical process for separation from the matrix material and removing 241Am isotope which is progeny nuclide of 241Pu. Following the α decay of most plutonium isotopes, the uranium progenies emit γ-rays and X-rays. The emission of X-rays results from internal conversion in the uranium progenies. Following the α decay of the 241Am isotope, γ-rays and X-rays are emitted by 237Np progeny in the same manner as γ-rays and X-rays are emitted by uranium progenies created by the α decay of most plutonium isotopes. Typical values of energy and emission probability of γ-ray and/or X-ray photons are presented in Table 1 for the α decay of 239Pu and 241Am isotopes. As presented in Table 1, the values of the emission probability of L X-rays is much larger than those of γ-rays and L X-rays in the α decay of plutonium isotopes. Since most transuranium (TRU) elements emit L X-rays with the energy range from 10 to 22 keV following α decay, spectroscopic measurements of L X-rays are expected to be useful for nondestructive TRU monitors. We carried out preliminary spectroscopic measurements of L X-rays emitted from 241Am and 239Pu sources with using a CdTe semiconductor detector. Figure 1(a) and (b) show an experimental energy spectrum of L X-rays emitted by 237Np progeny resulting from the α decay of 241Am and that by 235U progeny resulting from the α decay of 239Pu, respectively by dashed lines. Solid lines indicate theoretical emission probability of corresponding L X-ray lines [2], [3]. As shown in Fig. 1, the energy resolution of the semiconductor detector is insufficient to identify all L X-ray peaks in a mixture of 241Am and 239Pu.

Various microcalorimeters with a superconducting
troscopic measurements of L X-rays emitted from $^{241}\text{Am}$, $^{239}\text{Pu}$, and $^{238}\text{Pu}$ in the energy spectra of L X-rays emitted by TRU elements. We operated the four-pixel-array TES microcalorimeter with the Au mushroom-shaped absorber of 5.0 $\mu$m thick for detecting L X-ray photons emitted from $^{238}\text{Pu}$, $^{239}\text{Pu}$, and $^{241}\text{Am}$ sources. A dry-$^3\text{He}$–$^4\text{He}$ dilution refrigerator was used to maintain the operating temperature of the TES microcalorimeter without disturbing L X-ray detection over a period of data accumulation for sufficient statistics. A response of the TES microcalorimeter to L X-rays was obtained by analyzing detection signal pulses.

2. 4-pixel-array TES Microcalorimeter with a Mushroom Shaped Absorber

A TES microcalorimeter is a detector that measures the energy of an incident X-ray photon by the temperature rise, and consists of an energy absorber and a TES [9]. The energy of the incident X-ray photon is converted into a temperature rise in the absorber. The TES is a thermosensor utilizing the strong temperature dependence of the electric resistance of a superconducting thin film in the phase transition region for a precise measurement of the temperature rise. The sensitivity of the TES is given by $\alpha = d(\ln R_{TES})/d(\ln T)$, where $R_{TES}$ and $T$ are the electric resistance of the TES and the temperature, respectively. As shown in Fig. 2, TES is electrically connected to an input coil of a superconducting quantum interference device (SQUID) array amplifier, and thermally connected to a heat bath of temperature $T_b$ through a heat link of conductance $G$. The input coil $L$ is connected in series with the TES, while a shunt resistor $R_S$ is connected in parallel with the TES–$L$ line and a constant direct-current bias current $I_b$ supplied to the TES electric circuit is divided into the TES current $I_{TES}$ and the shunt current $I_S$. The TES current $I_{TES}$ converted into the magnetic flux $\phi$ by the input coil $L$ and the SQUID array amplifier outputs the voltage $V_{out}$ proportional to the TES current $I_{TES}$. With electrothermal feedback, the theoretical energy resolution is given by

$$\Delta E = 2.35 \sqrt{k_BCT^2 \sqrt{8n/\alpha}}$$  \quad (1)
where $n$ is typically between three and five, where $k_B$ and $C$ are the Boltzmann constant, the effective heat capacity of the TES microcalorimeter, respectively [10]. Several types of mushroom-shaped absorber have been developed in an attempt to increase the sensitive area of the TES microcalorimeter [11]–[13]. The operational performance of the microcalorimeter is affected by the deformation of the overhanging structure. In the four-pixel-array TES microcalorimeter developed for the EDS on the TEM, the overhanging structure of the mushroom-shaped absorber was supported by inserting a thin insulating layer between the overhang layer of the absorber and the TES surface [8]. In this work, we fabricated the four-pixel-array TES microcalorimeter with a same pixel structure described in reference [8]. The Au absorber thickness was selected to be 5.0 μm for detecting X-rays with absorption efficiency of 50% at the energy of 20 keV.

Figure 3 shows a schematic structure of the TES microcalorimeter pixel with a mushroom-shaped absorber with supported overhanging structure. The thin-film thermometer of the TES consists of a Ti/Au bilayer formed on a SiNx membrane. The mushroom-shaped absorber is made from an Au layer and the square-shaped absorber stem is deposited in the center of the TES surface. A 0.1-μm-thick Ta2O5 insulating layer is inserted between the overhang layer of the absorber and the TES surface to support the overhanging structure. The geometrical dimensions of the elements of the TES microcalorimeter pixel are listed in Table 2.

The TES microcalorimeter and the SQUID array amplifier chips were glued on a sample holder with a GE7031 varnish. Figure 4 shows a photograph of a fabricated four-pixel-array TES microcalorimeter chip glued on the sample holder. Each pixel of the TES microcalorimeter is labeled as px1–px4. The sample holder with the TES microcalorimeter and the SQUID array amplifier chips was cooled by a compact dry-3He–4He dilution refrigerator pre-cooled by the Gifford–McMahon (GM) cooler. Precise operation of the TES microcalorimeter had been disturbed by severe mechanical vibrations accompanied with reciprocating motion of the GM cooler [14]. For suppressing mechanical vibrations the GM cooler was installed in the vacuum chamber of the 3He–4He dilution refrigerator with employing a vibration-proofing structure. After cooled below 200 mK, the electric resistance of the TES of each pixel in the TES microcalorimeter chip was measured by applying a constant bias current $I_0 = 10 \mu A$ with changing temperatures. In the circuit diagram Fig. 2, the electric resistance of the TES $R_{TES}$ is given by

![Fig. 2](image-url) Electric circuit of the TES microcalorimeter and the SQUID array amplifier.

![Fig. 3](image-url) Structure of a TES microcalorimeter pixel with a mushroom-shaped absorber. (A) Top view, (B) Cross-sectional view.

![Fig. 4](image-url) Photograph of a fabricated 4-pixel-array TES microcalorimeter with a mushroom-shaped absorber.

![Fig. 5](image-url) Relationship between the electric resistance of the TES and the temperature of px1–px3.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Dimensions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au absorber thickness</td>
<td>5 μm</td>
</tr>
<tr>
<td>Au absorber surface area</td>
<td>160 μm × 160 μm</td>
</tr>
<tr>
<td>Au absorber stem area</td>
<td>100 μm × 100 μm</td>
</tr>
<tr>
<td>Ta2O5 layer thickness</td>
<td>100 nm</td>
</tr>
<tr>
<td>Ta2O5 layer perimeter</td>
<td>180 μm × 180 μm</td>
</tr>
<tr>
<td>TES bilayer thickness</td>
<td>120 nm (Ti)/50 nm (Au)</td>
</tr>
<tr>
<td>TES surface area</td>
<td>200 μm × 200 μm</td>
</tr>
<tr>
<td>SiNx membrane thickness</td>
<td>1 μm</td>
</tr>
<tr>
<td>SiNx membrane surface area</td>
<td>400 μm × 400 μm</td>
</tr>
</tbody>
</table>

![Table 2](image-url) Geometrical dimensions of TES microcalorimeter pixel.
where \( R_S = 7 \, \text{m\Omega} \) and the value of \( \eta \) is evaluated for a superconducting state of the TES, \( R_{TES} = 0 \). Figure 5 shows measured temperature dependence of the electric resistance of the TES \( R_{TES} \) of px1–px3. The px4 could not be measured due to accidental damage in a setup preparation. Table 3 lists measured values of the transition temperature \( T_c \), the phase transition temperature width \( \Delta T_{trn} \), the normal conducting resistance \( R_N \) and the sensitivity \( \alpha \) of the TES of px1–px3.

For operating the TES microcalorimeter with the high energy resolution, the bath temperature \( T_b \) and the bias current \( I_0 \) are determined with taking account of the relation-

\[
R_{TES} = \left( \frac{I_0}{\eta V_{out}} - 1 \right) R_S
\]  

(2)

ship between the applied bias current \( I_0 \) and the electric resistance of the TES \( R_{TES} \) at temperatures below \( T_c \). Figure 6 shows an example of obtained relationship between the applied bias current \( I_0 \) and output voltage of the SQUID array amplifier \( V_{out} \) of px3 at a bath temperature of 80 mK. The superconducting state of the TES is indicated by the straight line in Fig. 6 for \( I_0 \) below 120 \( \mu \text{A} \). The temperature of the TES microcalorimeter pixel increases with Joule heat generation caused by \( R_{TES} \) with applying bias current \( I_0 \) above 120 \( \mu \text{A} \). In the range of \( I_0 \) between 120 and 300 \( \mu \text{A} \), the TES is in the phase transition region owing to the electrothermal feedback mechanism [10]. The normal conducting state of the TES is indicated by the straight line in Fig. 6 for \( I_0 \) above 300 \( \mu \text{A} \). By using Eq. (2) relationship between \( I_0 \) and \( V_{out} \) is converted to that between \( I_0 \) and \( R_{TES} \). Figure 7 shows relationship between \( I_0 \) and \( R_{TES}/R_N \) for px1–px3 at a bath temperature of 80 mK. As shown in Table 3 and Fig. 7, px1, px2 and px3 in the TES microcalorimeter chip have almost the same characteristics within a tolerance for X-ray detection operation.

3. Response to L X-rays Emitted from \( ^{241}\text{Am} \) Source

The four-pixel-array TES microcalorimeter was cooled down to 80 mK and irradiated with L X-rays emitted from a sealed \( ^{241}\text{Am} \) source of 3.7 MBq. The \( ^{241}\text{Am} \) source was wrapped with a polyimide tape for stopping \( \alpha \)-rays and attenuating intensity of L X-rays and placed inside the refrigerator. Each pixel of the TES microcalorimeter was operated individually with using a single channel electronics consisting of the TES bias current source and the readout of the SQUID array amplifier. Counting rate of detection signal pulses was found to be 1 count per second. First the px3 was selected to operate for L X-ray detection because of the highest value of the sensitivity \( \alpha \) listed in Table 3. L X-ray detection signal pulses were accumulated in operating with setting the TES resistance \( R_{TES} \) to 20, 30, 40, 50 and 60\% of \( R_N \). The TES resistance \( R_{TES} \) was set by applying the bias current \( I_0 \) with using relationship between the applied bias current \( I_0 \) and the electric resistance of TES \( R_{TES}/R_N \) shown in Fig. 7. Detection signal pulses were digitized with \( 2.5 \times 10^5 \) samples per second and accumulated in a personal computer for response analysis. As shown in Fig. 8 two types of signal pulses were observed with different shape in X-ray detection. Slow signals consist of single rise and decay components with time constants of 16 and 395 \( \mu \text{s} \), respectively, while fast signals consist of a rise component with a time constant of 6.4 \( \mu \text{s} \) and two decay components with time constants of 16 and 395 \( \mu \text{s} \). Slow signal pulses corresponded to X-rays detection events in the absorber, whereas X-rays absorbed in the TES generated fast signal pulses. The number of slow signal pulses were found to be 92\% of all detection events.

The full width at half maximum (FWHM) value of the energy resolution is expected by using the noise equivalent power \( \text{NEP}(f) \) [15]. By using the noise power spectrum \( |N(f)|^2 \), \( \text{NEP}(f) \) is expressed as

\[
\text{NEP}(f) = \frac{4}{(2\pi)^2} \frac{|N(f)|^2}{f^2}
\]
where $E_0$ is the L X-ray energy, $|M(f)|^2$ is obtained by applying the Fourier transform of the averaged voltage signal pulse. Figure 9 (a) and (b) show the Fourier transform of the averaged voltage signal pulse of 17.751 keV X-ray detection $|M(f)|^2$ and the noise power spectrum $|N(f)|^2$ with a cut-off frequency of 125 kHz. Expected FWHM values of the energy resolution were calculated for 17.751 keV X-ray detection with different operation setting of $R_{TES}/R_N$ by using the equation

$$\Delta E_{ev} = \frac{2.35E_0}{\sqrt{2\int_0^{f_{\text{max}}} |M(f)|^2 |N(f)|^2 df}},$$

(4)

where $f_{\text{max}}$ was taken to be 125 kHz. Figure 10 shows relationship between operation setting $R_{TES}/R_N$ and the expected value of the energy resolution $\Delta E_{ev}$ for 17.751 keV X-ray detection. The minimum value of $\Delta E_{ev}$ was obtained to be 34.9 eV by operation setting $R_{TES}/R_N$ of 0.3. Then the px1 and px2 were operated for L X-ray detection with applying the bias current $I_0$ for setting $R_{TES}/R_N$ to 0.3 and 0.4. Values of $\Delta E_{ev}$ for the px1 and px2 were calculated with using Eq. (4) for 17.751 keV X-ray detection with operation setting $R_{TES}/R_N$ of 0.3 and 0.4. Calculated results are plotted in Fig. 10. The smallest value of $\Delta E_{ev}$ was obtained to be 33.3 eV in operating the px2 with setting $R_{TES}/R_N$ of 0.3.

The pulse height distribution for operation of the px2 with setting $R_{TES}/R_N$ of 0.3 was obtained by analyzing accumulated detection signals with using the optimal filter method [16]. In operating the TES microcalorimeter, a fluctuation of a bath temperature was observed to be $10\,\mu K$ at 80 mK. The effect of the bath-temperature fluctuation was taken into account in the pulse height analysis. Since the signal pulses corresponding to 59.5 keV $\gamma$-ray detection were found in the pulse height analysis, the saturation energy of px2 would be higher than 59.5 keV. The obtained pulse height distribution was converted to response of the px2 by assigning peaks to energy of L X-rays emitted by $^{237}$Np progeny resulting from the $\alpha$ decay of $^{241}$Am. Figure 10 shows obtained response of the px2 to L X-rays emitted from $^{241}$Am source. Major peaks in the response in Fig. 11 are labeled with corresponding L-lines of Np X-ray. Since the expected FWHM value of the energy resolution of the px2 was evaluated to be 33.3 eV for 17.751 keV X-ray detection, the natural line width of L X-ray was taken into account in fitting peaks in the response. In this work, response of the microcalorimeter to monoenergetic X-rays and the intensity distribution of individual L X-ray with the natural line width were assumed to have a Gaussian and Lorentzian shape, respectively, and individual peak in the response was fitted by using the Voigt function, which is the convolution of the Gaussian and the Lorentzian. Values of energy and natural width of L line X-ray were cited from the literature [17]. Fitted response to L lines of Np X-ray is shown by solid line in Fig. 12. Dotted line in Fig. 12 indicates the intensity distribution of L lines of Np X-ray.
Response of the px2 to L X-rays emitted from $^{241}$Am source with setting $R_{\text{TES}}/R_N$ of 0.3 at a bath temperature of 80 mK.

The FWHM value was obtained to be 33.4 eV at the Np L$_{\gamma 1}$ peak of 17.751 keV, which agrees with the expected value. Table 4 summarizes fitting results of the FWHM value of the energy resolution with corresponding values of line-energy and the natural width.

<table>
<thead>
<tr>
<th>Line</th>
<th>$E$ (keV)</th>
<th>Natural width (eV)</th>
<th>$\Delta E$ (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Np L$_{\gamma 1}$</td>
<td>13.946</td>
<td>11.8</td>
<td>33.9 ± 1.4</td>
</tr>
<tr>
<td>Np L$_{\gamma 1}$</td>
<td>17.751</td>
<td>13.4</td>
<td>33.4 ± 1.2</td>
</tr>
<tr>
<td>Np L$_{\gamma 1}$</td>
<td>20.784</td>
<td>15.9</td>
<td>30.4 ± 2.9</td>
</tr>
</tbody>
</table>

The FWHM value was obtained to be 33.4 eV at the Np L$_{\gamma 1}$ peak of 17.751 keV, which agrees with the expected value. Table 4 summarizes fitting results of the FWHM value of the energy resolution with corresponding values of line-energy and the natural width.

4. Response to L X-rays Emitted from Mixture Source of $^{238}$Pu, $^{239}$Pu and $^{241}$Am

Since the handling of plutonium isotopes is strictly controlled under various safety regulations, the spectroscopic measurement of L X-rays emitted from plutonium isotopes was carried out by using checking sources of plutonium isotopes for calibration of the lung counter at the Nuclear Fuel Cycle Engineering Laboratories of the Japan Atomic Energy Agency. Checking sources of plutonium isotopes consist of three lymph node blocks containing $^{238}$Pu with a total intensity of 111.33 kBq and three lymph node blocks containing $^{239}$Pu with total intensity of 184.63 kBq. The 4-pixel array TES microcalorimeter and the SQUID array amplifiers were cooled by a compact dry $^3$He–$^4$He dilution refrigerator with pre-cooled by a remote helium cooling loop [18]. For confirming the performance of X-ray detection, the 4-pixel ar-

Table 5 Line energy, the natural width and fitting results of the FWHM value of the energy resolution for response of the px2 to L X-rays emitted from mixture source of $^{238}$Pu, $^{239}$Pu and $^{241}$Am.

<table>
<thead>
<tr>
<th>Line</th>
<th>$E$ (keV)</th>
<th>Natural width (eV)</th>
<th>$\Delta E$ (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>U L$_{\gamma 1}$</td>
<td>13.618</td>
<td>11.7</td>
<td>31.5 ± 2.3</td>
</tr>
<tr>
<td>U L$_{\gamma 1}$</td>
<td>17.222</td>
<td>13.5</td>
<td>39.2 ± 1.3</td>
</tr>
<tr>
<td>U L$_{\gamma 1}$</td>
<td>20.169</td>
<td>14.5</td>
<td>33.5 ± 2.6</td>
</tr>
<tr>
<td>Np L$_{\gamma 1}$</td>
<td>13.946</td>
<td>11.8</td>
<td>31.5 ± 2.3</td>
</tr>
<tr>
<td>Np L$_{\gamma 1}$</td>
<td>17.751</td>
<td>13.4</td>
<td>39.2 ± 1.3</td>
</tr>
<tr>
<td>Np L$_{\gamma 1}$</td>
<td>20.784</td>
<td>15.9</td>
<td>33.5 ± 2.6</td>
</tr>
</tbody>
</table>
ray TES microcalorimeter was irradiated with L X-ray emitted from a sealed $^{241}$Am source at a bath temperature of 80 mK. The sealed $^{241}$Am source was placed in front of the X-ray window of the refrigerator. The X-ray window was made of a disk shaped beryllium plate of 25 mm in diameter and 1 mm thick. The px2 was operated for L X-ray detection with setting $R_{TES}/R_{N}$ of 0.3. Experimental FWHM value of the energy resolution was obtained to be 34.8 eV at the Np L$_{\beta}$ peak of 17.751 keV. Although an electrical noise in the laboratory caused a degradation of the energy resolution, the energy resolution was found to be superior to that obtained in last experiments [7]. Mixture source of $^{238}$Pu, $^{239}$Pu and $^{241}$Am was placed in front of the X-ray window of the refrigerator, and the px2 was operated with setting $R_{TES}/R_{N}$ of 0.3. Figure 13 shows obtained response of the px3 to L X-rays emitted from mixture source of $^{238}$Pu, $^{239}$Pu and $^{241}$Am. In obtained response, major peaks with high intensities can be clearly distinguished. Major peaks in the response in Fig. 13 are labeled with corresponding L-lines of U and Np X-ray. The Voigt function was used for fitting individual peak in the response as mentioned above. Fitted response to L$_{\beta}$ lines of U and Np X-ray is shown by solid line in Fig. 14. Dotted line in Fig. 14 indicates the intensity distribution of L$_{\beta}$ lines of U and Np X-ray. Table 5 summarizes fitting results of the FWHM value of the energy resolution with corresponding values of line-energy and the natural width.

5. Conclusions

The four-pixel-array TES microcalorimeter was irradiated with L X-rays emitted from a seeded $^{241}$Am source at a bath temperature of 80 mK. The selected pixel of the TES microcalorimeter was operated for the detection of L X-rays. Detection signal pulses were converted to a response of the TES microcalorimeter to L X-rays with using the optimal filter method. An energy resolution was obtained to be 33 eV of FWHM value at 17.751 keV. Response of L X-rays emitted from a mixture of $^{238}$Pu, $^{239}$Pu and $^{241}$Am sources was obtained by operating the selected pixel of the TES microcalorimeter. Major L X-ray peaks of progeny elements of $\alpha$ decay of Pu and Am isotopes were clearly identified in the obtained energy spectrum. The experimental results demonstrated the separation of $^{241}$Am and plutonium isotopes by L X-ray spectroscopy.

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References


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