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Superconductivity at the highest transition temperature of 8.1 K in a simple cubic Au$_x$Sb$_{1-x-y}$Te$_y$ alloy system synthesized under high pressure

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Abstract
We have investigated superconductivity and structure in a gold–antimony–tellurium ternary alloy system (Au$_x$Sb$_{1-x-y}$Te$_y$) synthesized under high pressure. We found that Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ and AuSb$_2$Te when crystallized in simple cubic structures ($\alpha$-Po type), which are reported to be semiconductors above 20 K, are superconductors with a transition temperature ($T_c$) of 6.7 K. The structure and $T_c$ are mapped on an Au–Sb–Te triangular diagram. A $\beta$-Po-type rhombohedral structure appears before the crystal structure changes from As type (Sb) to $\alpha$-Po type. Superconductivity is observed in the $\beta$-Po-type structure as well as in the $\alpha$-Po-type structure. $T_c$ increases with decreasing Te concentration toward the Te-free Au–Sb end of the diagram. A $T_c$ of 8.1 K was achieved for the Au–Sb alloy with a typical composition of Au$_{0.15}$Sb$_{0.85}$. This $T_c$ value is the highest among materials with the $\alpha$-Po-type structure under ambient pressure. Our Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ sample exhibits a weak metallic behavior in resistivity. The discrepancy in the normal state resistivity behaviors between the previous study and ours must originate from a difference in the number of lattice defects in the samples.

Keywords: superconductivity, simple cubic structure ($\alpha$-Po type), gold–antimony–tellurium ternary system, antimony, phase diagram, high-pressure synthesis, $T_c$ variation

(Some figures may appear in colour only in the online journal)

1. Introduction

Superconductivity in alloys was intensively investigated in the middle of the 20th century. As a consequence, there exists an extensive database of physical properties for a variety of superconducting alloys. Most of the superconducting alloys are composed of transition elements such as Nb–Ti or Bi–Pb systems. They usually have the same crystal structures (body-centered cubic, face-centered cubic, etc) as the constituent elements. On the other hand, although they are not well understood, there exist superconducting alloys with simple (primitive) cubic structures (one atom per unit
cell). They belong to a type of rare crystal structure into which \( \alpha \)-polonium (\( \alpha \)-Po) is the only element crystallizing at ambient pressure.

Metastable alloys with the \( \alpha \)-Po-type simple cubic structure were first synthesized in 1962 by rapidly cooling Au–Te or Ag–Te systems from the melt [1]. In 1963, superconductivity at 2.18 K was found in a simple cubic In–Te alloy [2]. So far, about ten superconducting simple cubic alloys have been discovered [3–7]. Most of them are synthesized by combining Sb or Te with a transition element. The highest superconducting transition temperature (\( T_c \)) of 6.7 K is attained in Au\( _{0.25} \)Sb\( _{0.75} \) [4]. After the 1980s, simple cubic alloys of AgTe\(_3\), Au–Sb, AuSb\(_2\)Te, etc were successfully synthesized by quenching samples under a pressure of several gigapascals [8–10]. The alloy AuSb\(_2\)Te (hereafter referred to as Au\(_{0.125}\)Sb\(_{0.75}\)Te\(_{0.125}\)) is reported to be a semiconductor with a thermal energy gap of \( \Delta_E = 0.026 \) eV, based on its measured resistivity above 20 K [9].

However, we noticed that Au–Sb–Te ternary alloys synthesized under high pressure exhibited superconductivity when we were searching for a new superconductor containing valence skip elements such as Sb, Bi, Tl, etc, based on theoretical and experimental treatises [11–14]. In this paper, we report the superconductivity and structure in the Au–Sb–Te system synthesized under high pressure. \( T_c \) of up to 8.1 K was achieved in the Au–Sb system. This \( T_c \) value is the highest in substances with the simple cubic structure under ambient pressure.

2. Experimental details

Samples were synthesized using a cubic-anvil-type high-pressure apparatus [15]. Starting materials were powders of Sb (4N), Te (5N), and AuSb\(_2\). AuSb\(_2\) was the only compound in an Au–Sb binary system under ambient pressure that was synthesized by melting Au and Sb in a quartz tube. The starting materials were weighed with a nominal composition of Au\(_{x}\)Sb\(_{1-x}\)Te\(_y\), where the Au concentration \( x \) and Te concentration \( y \) were changed independently (\( x = 0.0–0.33, y = 0.0–0.25 \)). The starting materials were ground using an agate mortar and pressed into a pellet. The pellet and a graphite heater etc. were assembled in a pyroferrite cube. The sample was separated by a boron nitride (BN) vessel from the heater. After the pressure was elevated to 3.4 GPa, the temperature of the sample was raised to 800°C for 25 min. Our synthesis temperature was between the values reported in two previous studies (700 and 1000°C), while the synthesis pressure was almost the same (4.0 GPa) [9, 10]. The temperature was kept at 800°C for 10 min, after which the electric power was switched off. The temperature of the sample dropped below 200°C within 10 s.

Powder x-ray diffraction (XRD) patterns were measured using Cu K\(_\alpha\) radiation. The magnetic susceptibility measurements were performed in a magnetic field of 10 Oe using a SQUID magnetometer (Quantum Design MPMS). These measurements were carried out on warming after zero-field cooling (ZFC) and then on cooling in field (FC). The resistivity was measured by the four-probe method. Upper critical fields were determined by the two-coil mutual inductance technique operating at 60 kHz [16–18].

3. Results and discussion

3.1. Crystal structure

Powder XRD patterns are shown in figure 1 for Au\(_{x}\)Sb\(_{1-x}\)Te\(_y\) samples. The values of \( x = y = 0, 0.05, 0.075, 0.10, \) and 0.125 are selected from samples with various combinations of \( x \) and \( y \) to show the systematic change in crystal structure. The XRD pattern of an Sb sample (\( x = y = 0 \)), which was also heated under the same conditions as other samples, was almost the same as that of the starting Sb. Sb has an As-type structure (trigonal crystal system), which is schematically illustrated in figure 2(a), with lattice parameters of \( a = 4.3078 \) Å and \( c = 11.267 \) Å. The Au\(_{0.05}\)Sb\(_{0.95}\)Te\(_{0.05}\) sample retained the As-type structure even though the lattice parameters decreased to \( a = 4.260 \) Å and \( c = 11.008 \) Å. A peak corresponding to (003), which was located at about 2\( \theta \) = 23.7° in the XRD pattern of Sb, disappeared from the pattern for Au\(_{0.075}\)Sb\(_{0.85}\)Te\(_{0.125}\), indicating that the crystal structure changed from the As type to \( \beta \)-Po type (rhombohedral lattice system), as shown in figure 2(b). The lattice parameters were calculated to be \( a = 3.047 \) Å and \( \alpha = 88.60° \) in the rhombohedral lattice or \( a = 4.256 \) Å and \( c = 5.405 \) Å in the trigonal lattice, the \( \beta \)-Po-type structure can be regarded as the transitional configuration half way between the As- and \( \alpha \)-Po-type structures.

One can see that the number of diffraction peaks even decreased with further Au substitution. The diffraction peaks could be indexed as an \( \alpha \)-Po-type simple cubic structure, as shown in figure 2(c), with \( a = 3.027 \) Å and 3.016 Å for Au\(_{0.10}\)Sb\(_{0.80}\)Te\(_{0.10}\) and Au\(_{0.125}\)Sb\(_{0.75}\)Te\(_{0.125}\), respectively. The widths of their diffraction peaks were comparable to that of Sb, which represented high crystallinity of the samples. The lattice parameter of our Au\(_{0.125}\)Sb\(_{0.75}\)Te\(_{0.125}\) sample agreed well with the previously reported value of \( a = 3.018 \) Å. The Au\(_{0.15}\)Sb\(_{0.7}\)Te\(_{0.13}\) and Au\(_{0.175}\)Sb\(_{0.65}\)Te\(_{0.175}\) samples also had \( \alpha \)-Po-type structures with \( a = 3.003 \) Å and 3.001 Å, respectively (their XRD patterns are not shown in figure 1).

The change in crystal structure in the binary Au\(_{x}\)Sb\(_{1-x}\) alloys was similar to that observed in the ternary Au–Sb–Te ones. Namely, as the Au concentration \( x \) increased, the crystal structure changed from the As type (\( x = 0 \) and 0.05) to the \( \alpha \)-Po type (\( x = 0.12 \) and 0.15) by way of the \( \beta \)-Po type (\( x = 0.09 \)). The diffraction peaks of Au–Sb alloys were broad in comparison to those of the Au–Te–Sb alloys.

The dependence of the \( a \)-axis lattice parameter on the Au concentration \( x \) is shown in figure 3 for the single-phased \( \alpha \)-Po-structured samples, together with data from [4, 10] as a function of \( y \). The \( a \)-axis lengths, which hardly depended on \( y \), were located along the line connecting the data for Sb and Au\(_{0.25}\)Sb\(_{0.85}\) following Vegard’s law. Thus, the shrinkage of the lattice was mainly due to the substitution of Au for Sb. A single-phased sample could be prepared up to \( x = 0.175 \) when the sample was synthesized under a pressure of 3.4 GPa. A pure sample of \( x = 0.18 \) was obtained under a pressure of
Figure 1. Powder x-ray diffraction patterns for Au$_x$Sb$_{1-x-y}$Te$_y$ ($x = y = 0.0$ (Sb), 0.05, 0.075, 0.10 and 0.125). The peaks were indexed based on an As-type structure for $x = y = 0.0$ and 0.05, a $\beta$-Po-type structure for $x = y = 0.075$, and an $\alpha$-Po structure for $x = y = 0.10$ and 0.125.

Figure 2. Schematic illustrations of (a) an As-type structure (trigonal crystal system), (b) a $\beta$-Po-type structure (rhombohedral lattice system or trigonal crystal system), and (c) an $\alpha$-Po-type structure (simple cubic system). The figures are drawn in almost the same magnification ratios. The narrow solid lines in (a) and (b) indicate unit cells of the trigonal crystal system. The shortest bonds in each structure are shown by shaded bands.

4.5 GPa, which was the maximum pressure of our apparatus. The Au concentration $x$ could be further increased by applying higher synthesis pressure.

3.2. Superconductivity

Figures 4(a) and (b) show the temperature dependence of magnetic susceptibility for Au$_x$Sb$_{1-x-y}$Te$_y$ ($x = y = 0.075, 0.10, 0.125, 0.15$ and $0.175$) and Au$_x$Sb$_{1-x}$ ($x = 0.09, 0.12, 0.15, 0.18$ and $0.33$), respectively. Large diamagnetic transitions due to occurrences of superconductivity were observed for the susceptibility curves in the ZFC process due to a shielding effect of the superconductor. Shielding volume fractions calculated using the ZFC susceptibility values at 5 K without demagnetization correction exceeded 100% for all the samples. On the other hand, the Au$_x$Sb$_{1-x-y}$Te$_y$ ($x = y = 0.075$) and all the Au$_x$Sb$_{1-x}$ samples showed much smaller diamagnetic transitions in the FC process. This must be due to a strong magnetic flux pinning force of the samples, i.e. the expulsion of the magnetic flux from the samples by the Meissner effect is small on cooling in field. Such a large difference in the magnitude of diamagnetic susceptibility between ZFC and FC processes is widely observed for superconductors having strong flux pinning forces.

The Au$_x$Sb$_{1-x-y}$Te$_y$ samples indicated the highest $T_c$ of 6.8 K at $x = y = 0.15$ (Au$_{0.15}$Sb$_{0.75}$Te$_{0.15}$). Note that superconductivity also occurred in the $\beta$-Po-type structure (Au$_{0.075}$Sb$_{0.85}$Te$_{0.075}$), while its $T_c$ appeared to be lower than that for the $\alpha$-Po-type structure. No superconducting transitions were observed above 2 K for samples of Au$_{0.05}$Sb$_{0.75}$Te$_{0.05}$ and Sb with the As-type structures. Superconducting transitions of Au$_x$Sb$_{1-x}$ samples were
and field-cooled (FC) processes.

(b) Au

nominal composition of Au

Au concentration, described as follows. The sample with a

and the previous value was the result of the difference in

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Figure 3. Dependence of the a-axis lattice parameter on the Au

concentration x for single-phased samples with α-Po-type structures

as a function of Te concentration y. Data for Sb and Au$_{0.25}$Sb$_{0.85}$ are

also shown. The a-axis parameter of Sb was calculated regarding its

structure as simple cubic.

observed at higher temperatures than the samples including Te. The highest $T_c$ occurred at the same Au concentration ($x = 0.15$) as the Au$_x$Sb$_{1-y}$Te$_y$ ($x = y$) samples. The value for $T_c$ obtained for Au$_{0.15}$Sb$_{0.85}$ was as high as 8.1 K, above the value (6.7 K) for Au$_{0.25}$Sb$_{0.85}$ previously reported by Tsuei et al in [4]. The difference in $T_c$ between our samples and the previous value was the result of the difference in Au concentration, described as follows. The sample with a nominal composition of Au$_{0.33}$Sb$_{0.67}$, which included AuSb$_2$

as an impurity phase, had a $T_c$ of 6.4 K, as shown in figure 4(a), and its lattice parameter was $a = 2.945$ Å. The $T_c$ and lattice parameter were close to the reported values of Au$_{0.25}$Sb$_{0.85}$ ($T_c = 6.7$ K and $a = 2.93$ Å). The value of $T_c$ decreased significantly at an Au concentration of $x \sim 0.25$ while it was almost constant for $x = 0.12$–0.18. The broad and two-step transition in the Au$_{0.09}$Sb$_{0.91}$ sample can be explained by the location of the sample near the boundary between the α-Po-type structure and the β-Po-type structure with lower $T_c$.

Figure 5 shows the temperature dependence of resistivity for Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ and Au$_{0.15}$Sb$_{0.85}$ together with Sb. Compared to Sb, the metallicities of the Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ and Au$_{0.15}$Sb$_{0.85}$ samples were largely suppressed probably owing to disorder induced by the Te or Au substitution (or both) in the Sb site. The Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ and Au$_{0.15}$Sb$_{0.85}$ samples, however, still showed weak metallic temperature dependence with residual resistivity ratios of 1.16 and 1.30, respectively. The Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ and Au$_{0.15}$Sb$_{0.85}$ samples exhibited sharp superconducting transitions at 6.60 K and 8.07 K, respectively, which agreed well with the onset transition temperatures in magnetic susceptibility. As mentioned above, AuSb$_2$Te (Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$) is reported to behave like a semiconductor with a resistivity of 0.41 mΩ cm at room temperature, which is higher than that of our sample (0.27 mΩ cm). The semiconducting sample was synthesized at a temperature of 1000°C, that was 200°C higher than that used in this study. Our current sample was believed to have a smaller number of lattice defects than the previously reported one, which can explain the discrepancy in the temperature dependence of the normal state resistivity and the difference in the absolute value of the resistivity.

The temperature dependence of magnetic susceptibility was measured in an external magnetic field to estimate the upper critical fields ($H_{c2}$). The temperature dependence

Figure 4. Temperature dependence of magnetic susceptibility for (a) Au$_x$Sb$_{1-x}$Te$_y$ ($x = y = 0.075, 0.10, 0.125, 0.15$ and 0.175) and (b) Au$_x$Sb$_{1-x}$ ($x = 0.09, 0.12, 0.15, 0.18$ and 0.33). The measurements were made in an applied field of 10 Oe with zero-field-cooled (ZFC) and field-cooled (FC) processes.

Figure 3. Dependence of the a-axis lattice parameter on the Au concentration x for single-phased samples with α-Po-type structures as a function of Te concentration y. Data for Sb and Au$_{0.25}$Sb$_{0.85}$ are also shown. The a-axis parameter of Sb was calculated regarding its structure as simple cubic.
The values of $T_c$ determined by the onset of the magnetic transitions are plotted on the Au–Sb–Te triangular diagram for samples with various combinations of $x$, $y$, and $z$. The inset shows an enlargement near the transition temperatures. As the concentration of Sb or Te increases, the $T_c$ values increase. For example, the $T_c$ value for Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ is 8.1 K, while for Au$_{0.15}$Sb$_{0.85}$, it is 6.1 K. This indicates that the $T_c$ values tend to increase with increasing Sb or Te content.

3.3. Phase diagram

The values of $T_c$ determined by the onset of the magnetic transitions are shown in the Au–Sb–Te triangular diagram of figure 7. The diagram shows that the $T_c$ values are determined by the onset of magnetic transitions. The diagram is constructed based on the samples synthesized at 800 °C under a pressure of 3.4 GPa. The $T_c$ values are shown in red and blue for the $\alpha$-Po- and $\beta$-Po-type structures, respectively. The $T_c$ values for multi-phased samples are shown within brackets.

$T_c$ values are shown in red numerical values in the diagram, is located in the region of $0.10 \leq x \leq 0.175$ and $0 \leq y \leq 0.20$. The $\beta$-Po-type structure, whose $T_c$ values are shown in blue, appears over a small $x$ and $y$ region ($x = 0.05-0.075$ and $y = 0.0-0.15$). Shanov et al. reported that the homogeneity range of AuSb$_0$Te prepared at 40 kbar (4 GPa) and 1000 °C extended from AuSb$_{0.5}$Te$_{1.5}$ ($x = 0.125$ and $y = 0.1875$) to AuSb$_{0.5}$Te$_{0.5}$ ($x = 0.125$ and $y = 0.0625$). They seemed to change only with Te concentration based on AuSb$_7$. We have demonstrated that the $\alpha$-Po-type structure can be formed for wide ranges of Au and Te concentrations. If the starting value of $x$ or $y$ is too large, as in the case of Au$_{0.25}$Sb$_{0.75}$Te$_{0.05}$ or Au$_{0.15}$Sb$_{0.6}$Te$_{0.25}$, impurity phases of AuSb$_2$ or Sb$_3$Te$_3$ are formed.

$T_c$ tends to increase with decreasing Te concentration toward the Te-free Au–Sb end of the diagram. The highest $T_c$ of 8.1 K was achieved for Au$_{0.15}$Sb$_{0.85}$. This $T_c$ value was the highest in materials with an $\alpha$-Po-type structure under ambient pressure and was comparable to that of the well known typical element-based alloy of Bi–Pb ($T_c = 8.8$ K). It should be noted that $T_c$ of Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ was found to decrease from 6.7 to 6.1 K over two months, accompanied by slight expansion of the lattice parameter (from 3.016 to 3.018 Å). In the case of Au$_{0.075}$Sb$_{0.85}$Te$_{0.075}$, the crystal structure appeared to change from the $\beta$-Po type to the $\alpha$-type over 45 days as $T_c$ decreased from 4.5 to 3.0 K. These observations indicated that the samples were really metastable.

$T_c$ and the $c$ lattice parameters are summarized for the main superconductors with the simple cubic structures reported so far in table 1. Most materials have not been investigated in detail so far. There will be room to increase $T_c$ in the simple cubic system even for the known materials.
by investigating its precise composition dependence and to find new superconductors by testing various combinations of elements.

4. Conclusions

We have investigated the superconductivity and structure in the Au$_x$Sb$_{1-x}$Te$_y$ alloy system synthesized under high pressure and constructed the structure and $T_c$ map on a Au–Sb–Te triangular diagram. Single-phase samples were obtained for a wide composition range ($0.05 \leq x \leq 0.175, 0 \leq y \leq 0.2$). Superconductivity was observed in the $\beta$-Po-type structure as well as in the $\alpha$-Po-type structure. $T_c$ increased with decreasing Te concentration and the highest $T_c$ of 8.1 K was attained for the simple cubic Au–Sb alloy with a typical composition of Au$_{0.15}$Sb$_{0.85}$. The Au$_{0.15}$Sb$_{0.85}$ and Au$_{0.15}$Sb$_{0.85}$Te$_{0.125}$ showed metallic behavior in resistivity, though they had the large residual resistivity ratios. Samples were metastable, so their $T_c$ gradually decreased over a few months. The $H_{c2}$ (0) values were estimated to be 1.17 and 1.36 T for Au$_{0.125}$Sb$_{0.75}$Te$_{0.125}$ and Au$_{0.15}$Sb$_{0.85}$ alloys, respectively.

Acknowledgments

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Table 1. $T_c$ and $a$ lattice parameters for the main superconductors with the simple cubic structures reported so far.

<table>
<thead>
<tr>
<th>Materials</th>
<th>$T_c$ (K)</th>
<th>$a$ lattice parameter (Å)</th>
<th>$H_{c2}$ at 0 K (T)</th>
<th>Published year</th>
<th>Reference No</th>
</tr>
</thead>
<tbody>
<tr>
<td>In$<em>{0.5}$Te$</em>{0.5}$</td>
<td>2.18</td>
<td>3.07</td>
<td>0.08</td>
<td>1963</td>
<td>[2]</td>
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<td>Au$<em>{0.15}$Te$</em>{0.75}$</td>
<td>1.6–3.0</td>
<td>2.93–3.10</td>
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<td>1964</td>
<td>[3]</td>
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<tr>
<td>Au$<em>{0.25}$Te$</em>{0.75}$</td>
<td>2.6</td>
<td>3.06</td>
<td>—</td>
<td>1964</td>
<td>[3]</td>
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<tr>
<td>In$<em>{0.5}$Sb$</em>{0.5}$</td>
<td>4.1</td>
<td>3.18</td>
<td>—</td>
<td>1971</td>
<td>[4]</td>
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<tr>
<td>Pd$<em>{0.25}$Sb$</em>{0.835}$</td>
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<td>3.00</td>
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<td>1971</td>
<td>[4]</td>
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<tr>
<td>Au$<em>{0.15}$Sb$</em>{0.75}$</td>
<td>4.6</td>
<td>2.93</td>
<td>—</td>
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<td>[4]</td>
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<td>Au$<em>{0.25}$Sb$</em>{0.75}$</td>
<td>6.7</td>
<td>2.93</td>
<td>—</td>
<td>1971</td>
<td>[4]</td>
</tr>
<tr>
<td>Au$<em>{0.15}$Sb$</em>{0.85}$</td>
<td>8.1</td>
<td>3.00</td>
<td>1.36</td>
<td>2013</td>
<td>This paper</td>
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<tr>
<td>Sn$<em>{0.35}$Te$</em>{0.75}$</td>
<td>1.9</td>
<td>3.16</td>
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<td>1974</td>
<td>[6]</td>
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<tr>
<td>Sb$<em>{0.75}$Sb$</em>{0.3}$</td>
<td>2.85</td>
<td>3.07</td>
<td>—</td>
<td>1974</td>
<td>[6]</td>
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<td>Ge$<em>{0.35}$Sb$</em>{0.65}$</td>
<td>3.3</td>
<td>3.05</td>
<td>—</td>
<td>1974</td>
<td>[6]</td>
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<tr>
<td>Ni$<em>{0.12}$Sb$</em>{0.88}$</td>
<td>1.5</td>
<td>3.03</td>
<td>—</td>
<td>1974</td>
<td>[6]</td>
</tr>
<tr>
<td>Au$<em>{0.125}$Sb$</em>{0.75}$Te$<em>{0.125}$ (AuSb$</em>{c}$Te)</td>
<td>Semiconductor</td>
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<td>—</td>
<td>1984</td>
<td>[9]</td>
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<td>Au$<em>{0.125}$Sb$</em>{0.75}$Te$_{0.125}$</td>
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<td>1.17</td>
<td>2013</td>
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