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Research Article

Synthesis and Study of Electrical Properties of SbTeI

Harish K. Dubey,[1] L. P. Deshmukh,[2] D. E. Kshirsagar,[1] Madhuri Sharon,[3] and Maheshwar Sharon[3]

[1] *Physics Department, Birla College, Kalyan 421301, India*
[2] *Department of Physics, Solapur University, Solapur 413255, India*
[3] *N.S.N. Research Center for Nanotechnology & Bionanotechnology, Jambhul Phata, Ambernath (W) 421505, India*

Correspondence should be addressed to Maheshwar Sharon; sharonmaheshwar@gmail.com

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Needle shaped SbTeI crystals were obtained by solid state reaction. Electrical resistance was measured in the temperature range of 4 K to 550 K. SbTeI shows a metallic behavior from 4 K to 300 K, and at higher temperature (>300 K), it shows semiconducting behavior. Unlike SbSI, this material shows almost zero resistance around 550 K. It shows a piezoelectric behavior with a capacitance of 717 pF and its carrier density and nobilities are found to be 2.12×10^{16} cm^{-3} and 1.01 cm^2/(V·s), respectively. Crystals of SbTeI are characterized by XRD, SEM, and Raman analysis. Electrical activation energy is found to be 0.52 eV. It is suggested that this material may be studied for its application as a superconductor with Tc higher than room temperature.

1. Introduction

Many ternary chalcogenide compounds with the combination of Sb, S, Te, Bi, Cl, Br, Se, and I have been reported in the recent years and various attempts have been made to study their applications, like the electrostrictive constant, electroceramics property, semiconducting properties [1, 2], and so forth. SbSI is one of the most intensively studied compounds of this family [3–6]. Nitsche and Merz [7] have synthesized materials like SbSI, SbSBr, SbSeBr, SbSeI, SbTeI, BiSCl, BiSBr, BiSI, BiSeCl, and BiSeBr and studied their photoconducting properties. Papazoglou and Rentzeperis [8] have examined in detail the crystal structure of SbTeI and also reported that SbSI has metallic and semiconducting behavior. Kichambare et al. [9–11] prepared SbTeI at different temperatures and calculated the values of lattice constants, ionization potentials, and activation energies. Later, they doped SbTeI with Bi and studied its impact on the values of lattice constant, ionization potential, and activation energy.

Since in-depth studies on the electrical properties of SbTeI are not made [3–6, 8–12], it was thought to be desirable to study the electrical properties of SbTeI to find out the carrier concentration, mobility, piezoelectric property, capacitance, and so forth. In this paper, we report synthesis of SbTeI using solid state reaction technique and electrical properties studies using van der Pauw and two probe techniques in the temperature range of 4 K–550 K.

2. Experimental

2.1. Synthesis of SbTeI by Solid State Thermal Reaction. SbTeI was synthesized by keeping equal weighed quantity of the constituents (Se, Te and I) in a quartz tube of 24 cm length and 1.0 cm diameter (Figure 1(a)). The quantities of Sb, Te, and I were taken in the ratio 1 : 1 : 1, that is, 2 gm each. The tube was sealed under vacuum and then kept in a PID controlled tubular furnace heated to a set temperature (Figure 1(b)). Based on several experiments, finally two temperatures 400°C and 600°C were selected for the synthesis of SbTeI. After the end of thermal treatment the furnace was cooled down slowly at room temperature. One end of the tube was broken to take out the material. Crystals were deposited on the walls of the quartz tube. Needle shaped crystals were formed (Figure 1(c)). The reaction involved in the process is

$$\mathrm{Sb} + \mathrm{Te} + \mathrm{I} = \mathrm{SbTeI}. \quad (1)$$

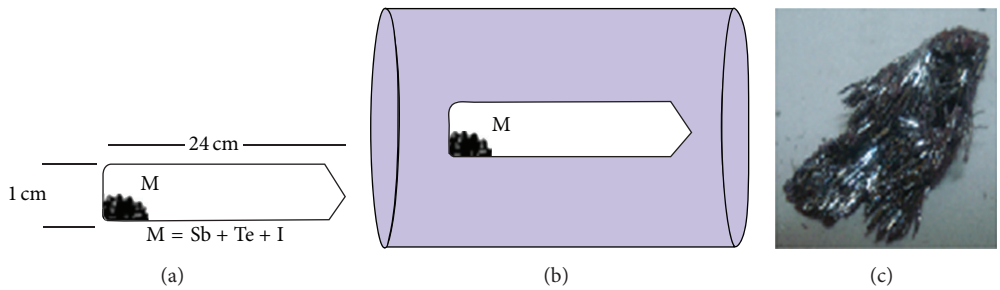


Figure 1: (a) Quartz tube of length 24 cm and diam. 1cm containing mixture of Sb, Te, and I in equal portion. (b) Quartz tube (M) kept in a tubular furnace and (c) photograph of needle shaped SbTeI.

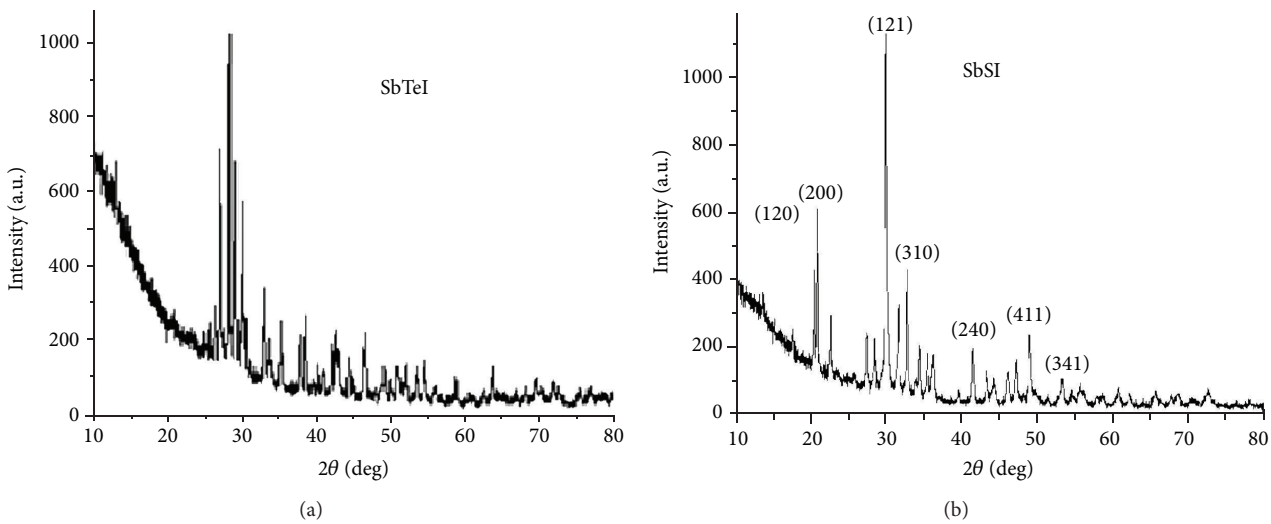


Figure 2: X-ray diffractogram of a (a) SbTeI and (b) SbSI.

2.2. The X-Ray Diffraction (XRD) Study of SbTeI. The XRD pattern was recorded by a Phillips PW 3710 X-ray diffractometer using CuKα radiation. The scanning angle (θ) was set between 10° and 80°. The XRD patterns of the powdered samples were taken (Figure 2).

2.3. The Scanning Electron Microscopy (SEM) of SbTeI. SEM images of two typical samples of SbTeI were taken using JEOL-6360 (LA) EDAX cum SEM. Probe current of 1 nA was set in the energy range of 0–20 KV for scanning the samples (Figure 3).

2.4. Micro-Raman Spectroscopy of SbTeI-HORIBA. Jobin-Yvon Lab Raman spectrophotometer was utilized to obtain Raman spectra. Based on the Helium-Neon laser source (632.88 nm), laser pulses were generated with the resolution <1.5 cm^{-1}. All measurements were performed using a 50X objective lens and D 0.3 filter randomly at 10 different positions (Figure 4).

2.5. Electrical Resistivity of SbTeI. Since the crystals were not of very large size, the crystals were powdered with a hand grinder and pressed with 140 Kg/cm^3 pressure with a 10 ton hydraulic press to make a pellet. The pellets were circular with 1 cm diameter and 0.5 to 1.5 mm thickness. Pellets were made without the use of any binder. To make the pellets strong, they were sintered for 90 min at 200°C. Pellets of SbTeI were used to study the electrical (Figure 5) and dielectric properties. Many pellets were used for these studies to confirm their reproducibility.

2.6. Charge Carrier Density Determination of SbTeI. The charge carrier density was determined by Hall Effect measurement with the Polytronics Hall apparatus set-up. At constant magnetic field of 2790 Gauss, applied current was varied and the corresponding voltages were measured to calculate the Hall Coefficients. Average of all values was used to calculate donor density.

2.7. Dielectric Constant of SbTeI. The dielectric constant was calculated by sandwiching pellet of SbTeI between two parallel plates of PCB sheet of size 1 × 1.2 cm. The surface of copper coating was exposed to the material. Two copper leads were soldered to these plates. LCR meter (Aplab India) was used for this purpose. The capacitance was measured at 1 KHz and 10 KHz frequencies. It was found that the capacitance in both the frequencies was almost the same. Therefore,

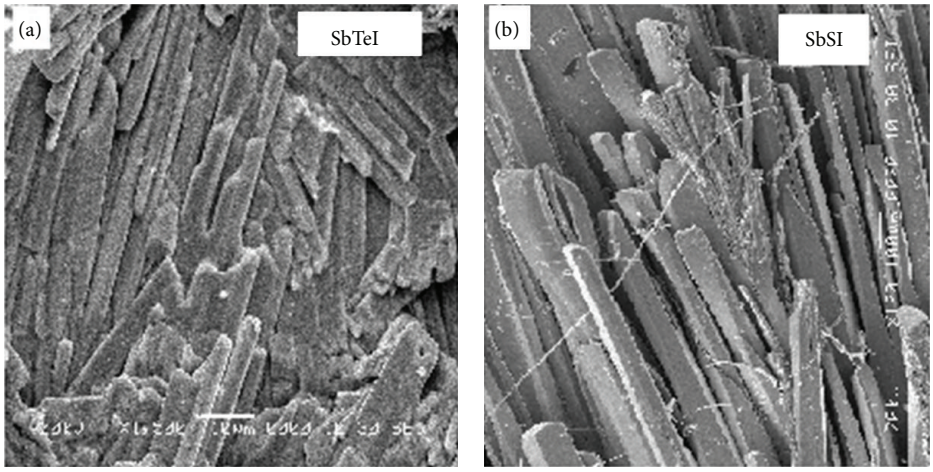


Figure 3: SEM micrographs (a) SbTeI and (b) SbSI.

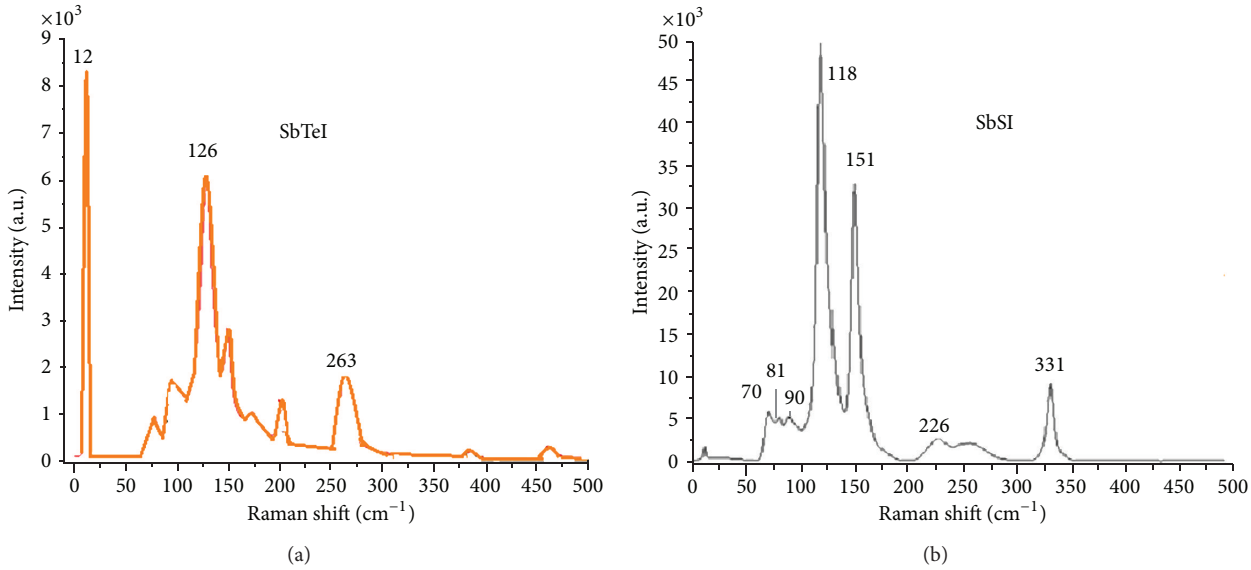


Figure 4: Micro-Raman plot for (a) SbTeI-2 and (b) SbSI.

the calculations were done on the basis of the capacitance observed for 1 KHz.

2.8. Piezoelectric Behavior of SbTeI. The system used for measuring the dielectric constant was also used to study the ohmic behavior of SbTeI.

3. Results and Discussions

3.1. XRD Analysis. XRD peaks obtained with SbTeI are shown in Figure 2(a). Due to nonavailability of JCPDS data, indexing of SbTeI was not possible. However, for comparison, the XRD of SbSI is shown in Figure 2(b) [13]. It is observed that two peaks around 20 degree observed with SbSI are absent with SbTeI. In addition, several closely packed peaks at an angle <30 degree were observed with SbTeI. This is in contrast to peak observed with SbSI. Though they are similar, they are well separated at >30 degree (Figure 2(b)) [13]. Apart from these variations, other low intensities peaks at angle greater than 40 degree are also observed which are of similar nature for both materials.

3.2. SEM Image Observations. Figure 3(a) shows the SEM images of SbTeI and Figure 3(b) shows the SEM of SbSI [13]. It is observed that though both show the rod type structures, SbTeI shows very compact branched type rods whereas rods of SbSI are separated [13]. In Table 1, SEM micrographs of two selected samples of SbTeI are shown. Enlarged micrographs show the rod to be hollow. SEM micrographs suggest that long duration (480 h) of thermal treatments at 400°C produces better crystal structure than sample treated for shorter duration (4 h) at higher temperature (600°C). From the SEM images of the samples it is confirmed that SbTeI grew in rod shaped orthorhombic crystals. This is also in agreement with the previously reported results [14–16].

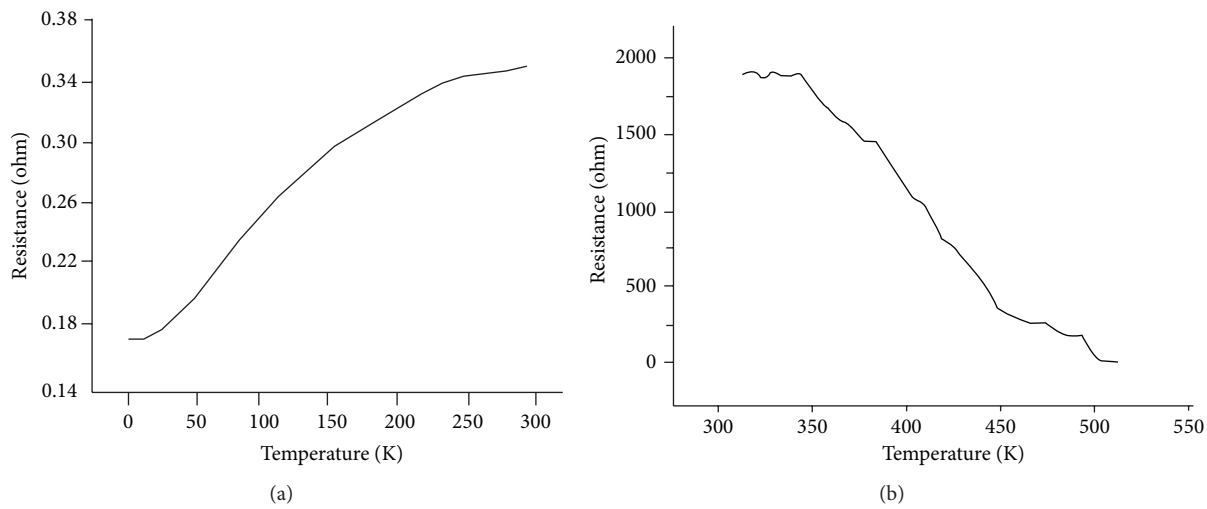


Figure 5: (a) Graph showing the variation in resistance versus temperature of SbTeI sample in the temperature range of 4 K to 300 K and (b) variation in resistance versus temperature of SbTeI in the temperature range 313 K to 523 K.

Table 1: SEM morphologies of compounds prepared under different conditions.

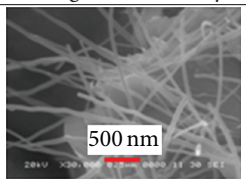
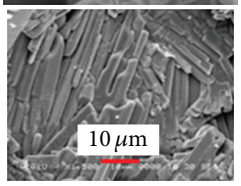
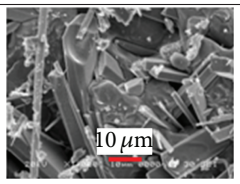
Sample	SbTeI Growth conditions	SEM images of SbTeI Crystals	Crystal details
SbTeI-1	Thermal treatment at 400°C for 480 hrs	 	Size 2–8 μm. Smooth rods stacked in random fashion. Enlarged micrograph shows hollow rods
SbTeI-2	Thermal treatment at 600°C for 4 hrs		Size 5–20 μm. Mixture of rods, rectangular plates and, big plates

Table 2: Stoichiometric composition of SbTeI calculated from EDAX results.

Sample and synthesis conditions	% elemental composition
SbTeI-1	Sb = 37.57
Time—480 hrs	Te = 34.11
Temp—400°C	I = 27.41
SbTeI-2	Sb = 31.63
Time—4 hrs	Te = 27.57
Temp—600°C	I = 40.80

3.3. Micro-Raman Assessment of SbTeI. The peaks observed in Raman spectrum of SbTeI is shown in Figure 4(a). For comparison, Raman spectrum of SbSI is shown in Figure 4(b) [13]. It is noticed that most of peaks obtained with SbTeI appear at different Raman shift as compared to Raman spectrum of SbSI except one peak at 12 cm^{-1}, which is absent in SbSI.

3.4. EDAX Analysis. Stoichiomentric composition of SbTeI was calculated from the EDAX attached to the SEM unit. Composition of two samples of SbTeI is presented in Table 2. It is noticed that SbTeI synthesized at 400°C for 480 h shows deficiency in iodine while sample prepared at 600°C for 4 h shows deficiency in Te and excess in iodine, assuming the composition to be (1 : 1 : 1). It can also be concluded from these results that SbTeI prepared at 400°C for 480 h contains

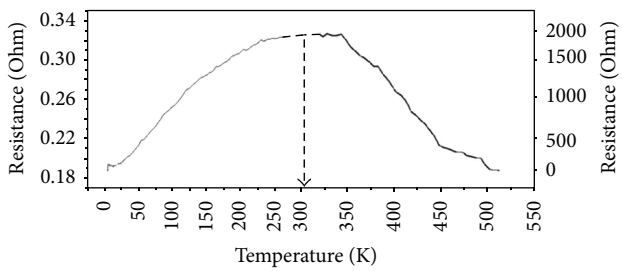


Figure 6: Graphs shown in Figures 5(a) and 5(b) for SbTeI are combined to examine the variation in resistance from 4 K to 550 K.

composition nearer to 1 : 1 : 1 as compared to sample prepared at 600°C.

3.5. Electrical Resistivity Measurements. Resistance of SbTeI was measured by four probe method in temperature range 4 K to 300 K. Variation of voltage of SbTeI sample at different temperatures was recorded at constant current of 100 mA. Resistance was calculated for each of measurements. The variation in resistance with temperature is plotted (Figure 5(a)). The trend in the resistance behavior at different temperatures confirms SbTeI to behave like metal.

By using two probe technique, resistance of SbTeI was measured in the temperature range of 300 K to 550 K. Results are plotted in Figure 5(b). This graph suggests that SbTeI behaves like a semiconductor from 300 K to higher temperature. In order to observe the transition temperature when SbTeI shifts its behavior from metallic to semiconducting, both of these graphs were joined and plotted (Figure 6).

It can be noticed from Figure 6 that the resistance increased with increase of temperature from 4 K to 250 K and from around 300 K, it starts decreasing linearly with increase of temperature. It is also interesting to note that, while resistance of SbSI was observed to be in Mega ohm [13] even at around 500 K, resistance of SbTeI tends towards zero resistance at higher temperature. This is a unique property of SbTeI and needs to carry out research to confirm whether it can show a superconductivity behavior at such a high temperature. If this is possible, it would be a result of great interest.

3.6. Activation Energy for the Conduction. The resistivity (ρ) of SbTeI sample was calculated at different temperature in the range of 313 K to 523 K. Finally, a graph of $\ln(\rho)$ versus $(1/T)10^3$ was plotted (Figure 7).

From the graph (Figure 7) of $\ln(\rho)$ versus $(1/T)10^{-3}$, the activation energy for the conduction was calculated by measuring the slope of the linear portion of the plot in the higher temperature region. The slope was found to be 3.055 $\times 10^{-3}$ K from which the activation energy was found to be 0.52 eV.

3.7. Charge Carrier Density. Carrier density was calculated by Hall measurement technique. The result of Hall voltage

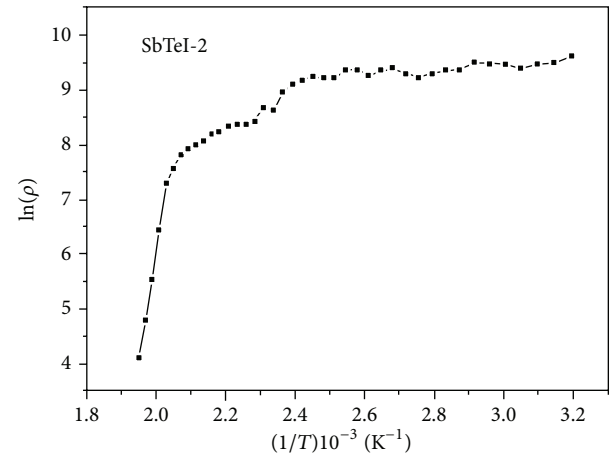


Figure 7: $\ln(\rho)$ versus $(1/T)10^{-3}$ K graph of SbTeI sample.

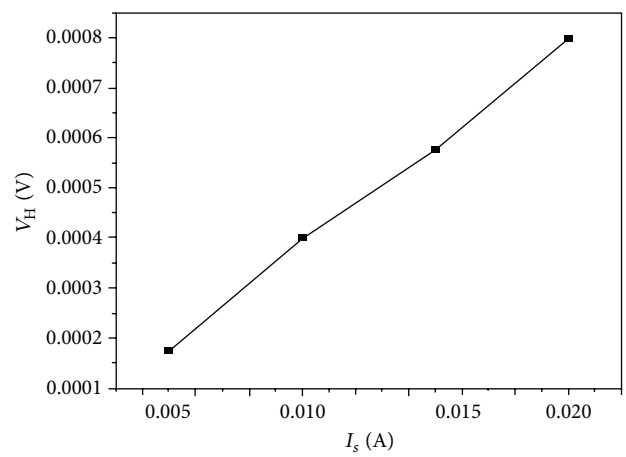


Figure 8: Graph of V_H versus I_s for SbTeI sample.

(V_H) versus current (I_s) was plotted (Figure 8) which gives a linear graph. From the slope of the graph, Hall coefficient R_H was calculated 293.90. Using this value, the carrier density and mobility of carriers were calculated 2.12×10^{16} cm^{-3} and 1.01 cm^2(V·s), respectively.

3.8. Dielectric Constant. To avoid the impact of the stray capacitance, the capacitance of the empty capacitor with the spacing between the plates equal to the thickness of the sample was subtracted from the capacitance with the sample. The net capacitance of SbSI sample was found to be 717 pF.

Using the relation $k = (C \times d)/(A \times 8.854 \times 10^{-12})$, the value of k is found to be 10112.

3.9. Piezoelectric Behavior of SbTeI. The SbTeI sample was found to be ohmic (Figure 9) in nature. It was interesting to observe that SbTeI pellet shows significant decrease in the resistance when pressed between two plates; that is, resistance was found to decreased from 100's of ohms to as low as 10 ohms.

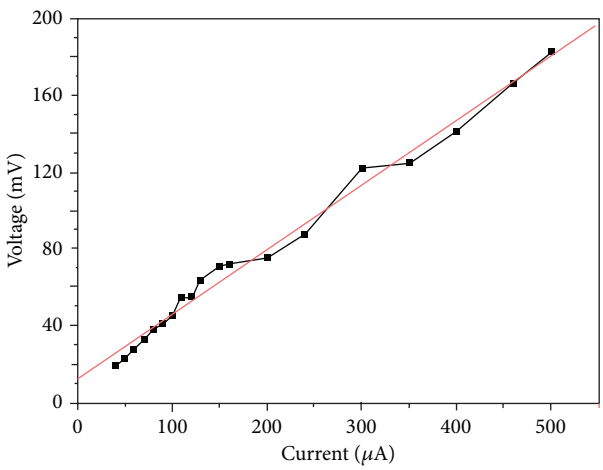


Figure 9: *I-V* graph of SbTeI at room temperature when pressed between two plates.

This indicates the piezoelectric property of SbTeI. As the material belongs to SbSI family and SbSI is already confirmed to be a piezoelectric material [8], there is a strong possibility that SbTeI is also a piezoelectric material.

4. Conclusions

SbTeI was synthesized by solid state reaction. SEM micrographs show that SbTeI crystals are rod shaped. SbTeI shows metallic conductivity in the temperature range 4 K to ~300 K and semiconducting bahaviour in temperature higher than 300 K. The resistance of SbTeI tends to almost zero at around 550 K. Its mobility and carrier density are calculated to be 2.12 $\times 10^{16}\,\mathrm{cm}^{-3}$ and $1.01\,\mathrm{cm}^2/(\mathrm{V{\cdot}s})$, respectively. It is proposed that SbTeI is a piezoelectric material. The activation energy obtained by electrical measurement in the range of 300 K to 550 K is 0.52 eV. The electrical properties of SbTeI are reported for the first time. SbTeI is characterized by XRD, SEM, and Raman analysis. More study is needed to confirm whether this material can be used as superconductor.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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