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Room-temperature oxygen sensitization in highly textured, nanocrystalline PbTe films: A mechanistic study

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In this paper, we report large mid-wave infrared photoconductivity in highly textured, nanocrystalline PbTe films thermally evaporated on Si at room temperature. Responsivity as high as 25 V/W is measured at the 3.5 μm wavelength. The large photoconductivity is attributed to the oxygen incorporation in the films by diffusion. Carrier concentration as low as 1017 cm−3 is identified to be the consequence of Fermi level pinning induced by the diffused oxygen. The successful demonstration of IR-sensitive PbTe films without the need for high-temperature processing presents an important step toward monolithic integration of mid-wave PbTe infrared detectors on Si read-out integrated circuits (ROICs). © 2011 American Institute of Physics. [doi:10.1063/1.3653832]

I. INTRODUCTION

Lead chalcogenides (PbS, PbSe, and PbTe) are promising material candidates for mid-wave infrared (IR) detection (2–5 μm wavelength) because of their superior chemical and mechanical stability over HgCdTe alloys. Their detection wavelength range can be further extended into the far-wave infrared by capitalizing on their bandgap tunability through bromide ion (Br−) doping.4–6 In addition, while the HgCdTe system is highly sensitive to crystal defects and thus only single-crystalline HgCdTe materials qualify for IR detection, it has been found that polycrystalline lead chalcogenides can exhibit high infrared detection sensitivity comparable to their single-crystalline counterparts;15–17 and (5) solution-deposited PbSe films used in commercial IR detectors often exhibit high sheet resistance (107−109 Ω), but reports on the actual carrier concentrations in these films are scarce.

Polycrystalline lead chalcogenide films with high photoresponse have been reported to have large grain sizes, i.e., 0.4–0.7 μm for PbSe (Refs. 25 and 26) and 0.9 μm for PbS.27 The films achieve high photoresponse through oxidation annealing process with iodine (I2) to facilitate the film recrystallization process and incorporation of oxygen,25,26 or through bromide ion (Br−) to control film morphology and grain size.27 As a comparison, nanocrystalline PbTe films with 50–75 nm grain size are found to be more easily incorporated by oxygen through diffusion and show good photoresponse.19,28–30 Furthermore, based on the available resistance values for commercial PbSe IR detectors, we estimate a room temperature carrier concentration of 1016−1017 cm−3 depending on the film thickness and carrier mobility, which is consistent with our observation in this work. However, literatures on PbTe typically report carrier concentrations of ~1018 cm−3 in polycrystalline films. 10 times higher than our results, probably due to insufficient Fermi-level pinning (due to formation of trap states near Fermi level) as we will explain later. In this paper, we report significant photoconductivity in nanocrystalline PbTe films without high temperature sensitization (grain size 25 nm). The scope of this paper further includes study of the oxygen sensitization mechanism and the electronic structure of oxygen sensitized PbTe films.

II. EXPERIMENT

PbTe films are prepared using single-source thermal evaporation from stoichiometric PbTe bulk of 99.999%...
purity. Oxide coated Si wafers (6 in. Si wafers with 3 μm thermal oxide from Silicon Quest International) are used as starting substrates. The thermal evaporation runs are carried out at a base pressure lower than 5 × 10⁻⁷ Torr. The substrates are held on a rotating substrate holder maintained at room temperature throughout the deposition. Film deposition rate is monitored in situ with a quartz crystal sensor and is maintained at 8–10 Å/s. The thermal evaporation technique allows deposition of films with high uniformity across an entire large-area substrate, with improved throughput and much lower cost compared to molecular beam epitaxy (MBE) or metalorganic chemical vapor deposition (MOCVD). Film thickness is measured using a KLA Tencor P-16 surface profilometer, and we confirm excellent thickness uniformity across an entire substrate with thickness variations < 3%. Films with different thickness values from 100 to 1000 nm are deposited under identical conditions to study the microstructural evolution.

To quantitatively evaluate the impact of oxygen on electrical properties of PbTe films, two types of samples are prepared. In one case a thin layer of thermally evaporated Ge₂₃Sb₇S₇₀ glass film is evaporated onto as-deposited PbTe in the same deposition chamber without breaking vacuum (denoted as “low oxygen concentration”); and in the other the as-deposited PbTe films are allowed to be directly exposed to air after deposition to enable oxygen diffusion into the films (denoted as “high oxygen concentration”). The Ge₂₃Sb₇S₇₀ glass has been proven to be impermeable to oxygen up to 200 °C and is chemically stable. Tin metal contacts are pre-deposited through a shadow mask onto the substrate for electrical measurements. The Ohmic nature of electrical contact is confirmed by I–V measurements in the temperature range of 80–340 K. No significant PbTe electrical property variation is observed for films with Ge₂₃Sb₇S₇₀ capping layer and of different thickness values. Therefore, we conclude that the electrical properties we measured are dominated by the intrinsic properties of PbTe films and the PbTe-Ge₂₃Sb₇S₇₀ interface has little impact on the measured film properties.

Secondary ion mass spectrometry (SIMS) is employed to determine the oxygen concentration and “through-thickness” distribution in the PbTe films. Oxygen atomic detection limit of the SIMS technique is approximately 1 × 10¹⁸ atoms/cm³. Film phase composition and microstructure are evaluated using X-ray diffraction on a PANalytical X’Pert Pro diffractometer. Grain size and surface roughness of the films are measured using a Digital Instruments Nanoscope IIIa AFM. Cross-sectional TEM images are taken using a JEOL 200CX General Purpose TEM. The chemical states of the elements in the PbTe films are identified using X-ray photoelectron spectroscopy (XPS) on a Kratos AXIS Ultra Imaging XPS system. During the XPS analysis, we use the C 1s peak with a binding energy of 284.448 eV for energy calibration.

Hall experiments are performed using a van der Pauw technique at a magnetic field of 4000 G. IR light from a blackbody is monochromatized through a sodium chloride prism and modulated at 10.3 Hz for the photoconductivity measurement. An SP2402 thermoelectric cooler (TEC) from Marlow Industries Inc. is used for cooling samples down to −60 °C during the photoconductivity characterization. A bias current of 0.1 mA is held constant during the measurement. The incident IR light power density on the samples is calibrated using a thermopile reference.

III. RESULTS AND DISCUSSION

A. Microstructure analysis

XRD spectra in Fig. 1(a) confirm that PbTe films of all thicknesses are polycrystalline and contain a single face-centered-cubic (fcc) crystalline phase with a rock salt structure. Detailed peak-by-peak analysis indicates all the films have a lattice parameter of 6.459 ± 0.001 Å, very close to the reported value of 6.454 Å in the standard powder diffraction file (PDF) card. However, the diffraction peak intensity values in the measured spectra differ vastly from those quoted from the standard PDF card in Fig. 1(a), indicating strong

![Figure 1(a)](https://example.com/figure1a.png)

**FIG. 1.** (Color online) (a) XRD spectra of thermally evaporated PbTe films on oxide-coated Si substrates. The film is a polycrystalline single fcc phase with strong (200) texture. Standard XRD data from PDF card # 03-065-0137 is also shown at the bottom for comparison. (b) Thickness dependence of peak intensity ratio, i.e., I(200)/I(220), showing the degree of texture increases sharply with decreasing film thickness. The ratio for the standard randomly oriented PbTe sample of PDF card # 03-065-0137 is 1.45.
(200) texture in the films deposited on an amorphous substrate. The degree of texture can be quantified using the peak intensity ratio between the (200) peak and the (220) peak as shown in Fig. 1(b). In randomly oriented polycrystalline PbTe, the ratio is 1.45. In contrast, our films show strong (200) texture and the degree of texture increases prominently with decreasing film thickness. For the 100 nm thick film, an intensity ratio up to 180 is observed. Notably, this degree of texture is almost 50 times stronger compared to PbTe films deposited on soda-lime glass substrates in our previous study. 29

The evolution of texture as a function of film thickness and substrate type can be explained based on a two-step nucleation and growth model shown in Fig. 2. Figure 2(a) illustrates the kinetics of early stage nucleation and film growth which are primarily determined by interface energy between PbTe and SiO₂/Si substrate. In this stage, [200] becomes the preferred film growth direction due to the lower interface energy between (200) planes and the substrate, and thus strong (200) texture is expected. In thick films shown in Fig. 2(b), the growth of (200) grains creates new nucleation sites in the grain boundary regions, where nucleation of grains with random orientation occurs. As a consequence, the degree of texture decreases in thick films. We shall note that the transition between the two growth phases during film growth is a gradual process. This is evident in Figs. 3(a)–3(e), which show the progressive increase in density as well as size of randomly oriented grains as film growth proceeds.

This two-step growth model is further verified by AFM surface morphology studies. As shown in Figs. 3(a)–3(e), the average grain size monotonically increases with increasing film thickness from 100 nm to 1000 nm. Indicated by arrows in Figs. 3(c) and 3(d), non-equiaxial grains evolve in thick films, consistent with our hypothesis of two-stage nucleation and growth model. Figure 3(f) plots the thickness dependence of average grain size and rms surface roughness. As film thickness decreases, average grain size decreases from 50 nm for 500 nm thick film to 25 nm for 100 nm thick film, accompanied by an rms surface roughness decrease. The cross-sectional TEM image in Fig. 4 shows the columnar structure of a 500 nm thick film. The “through-thickness” grain boundaries are clearly seen as indicated by the white arrows.

### B. Oxygen concentration depth profiling and chemical states

Oxygen concentration depth profiles of the “high oxygen concentration” and “low oxygen concentration” films are shown in Fig. 5 and the average oxygen concentration values are listed in Table I. The two orders of magnitude concentration difference in the samples with and without capping layers suggests that most oxygen in the “high oxygen concentration” sample comes from oxygen in-diffusion when the films are exposed to air. Residual oxygen in the “low oxygen concentration” samples most probably originates from the bulk evaporation source materials.

SIMS measurements are made on films after deposition and exposure to air for a known time period; the corresponding diffusion coefficient fitted from the concentration profile thus may be calculated by fitting the concentration profile to the diffusion equation. The fitted diffusion coefficient is in the order of 10⁻¹⁶ cm²/s at room temperature. This high diffusion coefficient suggests that the major contribution is from “short-cut” diffusion paths, in particular grain
boundary. This is consistent with our micro-structural analysis, the abundance of vertically aligned, “through-thickness” grain boundaries provides numerous diffusion short-cuts, which enables efficient oxygen diffusion without the need for high-temperature processing. The fine grain structure and the columnar grain structure of the nanocrystalline films are thus ideal for enhanced oxygen diffusion and low-temperature sensitization process. As we will discuss in greater detail in the subsequent sections, this unique nanocrystalline columnar microstructure is critical in shaping electrical properties of the films and achieving high photoconductivity.

Using XPS, we have identified two chemical states for both Pb and Te, one is due to normal Pb-Te bonds in PbTe, and the other resulting from oxidation of Pb and Te. Figures 6(a) and 6(b) show the XPS spectra of Pb 4f and Te 3d lines in the “high oxygen concentration” sample, respectively. The black solid curves in Figs. 6(a) and 6(b) correspond to XPS spectra collected on the surface of the films, and the red dotted curves are spectra taken on films after removal of the surface layer using Ar ion milling. Clearly, the surface of the films becomes heavily oxidized forming both Pb-O and Te-O bonds. Oxygen concentration is significantly reduced beneath the surface layer, which is consistent with the SIMS measurement and confirms that the oxygen incorporation occurs through diffusion from the ambient rather than in situ during deposition. To identify the chemical binding states of diffused-in oxygen, Fig. 6(c) shows the XPS spectra of oxygen in the sample after removal of the surface layer. A sputtered TeO2 film is used as the reference sample to unequivocally identify the peak corresponding to Te-O bonding in Fig. 6(c). From the measurement we calculate the ratio of oxygen bonded to Pb and Te to be about 8:1. Thus even though both Pb-O and Te-O bonds are formed in the PbTe films, oxygen preferentially bonds with Pb. This result therefore formally confirms the long-standing postulate of the formation of (PbO)2 complex in lead chalcogenides, which serves as electron traps and leads to p-type conduction.

C. Hall experiment

Hall experiment and temperature dependence of resistivity are studied to further ascertain the electronic structure of oxygen sensitized PbTe films. Room temperature carrier concentration values of the PbTe samples with low and high oxygen concentrations are listed in Table I. Both types of samples exhibit p-type conductivity. Furthermore, for films of different thicknesses exposed to air directly after deposition and thus with high oxygen concentrations, we confirm that they are all p-type. As shown in Figs. 7(a)–7(b), repeated Hall experiments performed over a period of 4 months after film deposition do not show any change within the accuracy of the experiment, and no significant thickness dependence of either carrier concentration or Hall mobility has been observed. These findings provide further evidence that oxygen diffusion in the nanocrystalline PbTe films is a very rapid process which quickly reaches saturation over time. The room temperature carrier concentration is (1.2 ± 0.3) × 1017 cm−3, which is among the lowest reported values for PbTe [single crystalline PbTe (Ref. 32) and polycrystalline PbTe (Ref. 33), the calculated intrinsic value is 0.8 × 1016 cm−3] and is more than an order of magnitude lower than previously reported values in polycrystalline PbTe. Hall mobility of the films is (81 ± 13) cm2/V·s, which is about one order of magnitude lower than the values in single crystals and epitaxial layers.

Since we target at TEC-cooled short-wave and mid-wave IR photodetector applications (1–5 μm wavelength) and most commercially available TECs can cool...
photodetectors down to $-60 \, ^\circ C$ (213 K), we perform Hall experiment in the temperature range of 213–300 K in order to study two separate components’ contribution in film resistivity, i.e., carrier concentration and mobility. The former could help understanding the band structure, and the latter could facilitate identifying the scattering process in electrical conduction. All the films in the studied temperature range are p-type, and films of all thicknesses yield similar results and key parameters are summarized in Table II. Figure 8 shows typical temperature dependence of carrier concentration and Hall mobility of a 200 nm thick film. Figure 8(a) suggests thermally activated process exists in the polycrystalline PbTe films and the activation energy is fitted to be 0.111 eV. The activation energy fitted for films of other thicknesses are listed in Table II, and all values are around 0.11 eV.

**TABLE II.** Activation energy and power law’s exponent fitted from temperature dependence of carrier concentration and Hall mobility for polycrystalline PbTe films of different thicknesses.

<table>
<thead>
<tr>
<th>Film thickness (nm)</th>
<th>Activation energy (eV)</th>
<th>Exponent</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.108</td>
<td>1.50</td>
</tr>
<tr>
<td>200</td>
<td>0.111</td>
<td>1.25</td>
</tr>
<tr>
<td>300</td>
<td>0.112</td>
<td>0.85</td>
</tr>
<tr>
<td>500</td>
<td>0.106</td>
<td>0.65</td>
</tr>
<tr>
<td>1000</td>
<td>0.109</td>
<td>1.86</td>
</tr>
</tbody>
</table>

FIG. 6. (Color online) (a) XPS spectra of lead 4f peaks; (b) XPS spectra of tellurium 3d peaks. The black solid curves in (a) and (b) correspond to XPS spectra collected on the surface of the films, and the red dotted curves are spectra taken on films after removal of the surface layer using Ar ion milling. The change of Pb-O and Te-O peak intensity after surface removal suggests the presence of a surface oxide layer in the sample. (c) XPS spectrum of oxygen 1s peaks for the PbTe film after removal of a surface oxidation layer by Ar ion milling. Two chemical binding states due to oxidation of Pb and Te are identified and the ratio of oxygen bonded to Pb and Te is calculated to be about 8:1, indicating oxygen preferentially bonds with Pb.
In polycrystalline materials, due to the permittivity difference between the grains and grain boundaries, a quasicon- tinuous energy spectrum of grain surface states should be expected. However, according to our XPS results the grain boundary areas could include high permittivity compounds such as PbO, thus the width of the energy spectrum of the grain surface states may be found to be less than kT and we can assume the existence of a monoenergetic level of the surface states.36,37 Therefore, a schematic of the band diagram near a grain boundary could be employed as in Fig. 4 in Ref. 29. According to this model, Hall carrier concentration is thermally activated and can be described by an activation energy $E_a = (E_F - E_V) - E_S$, where $(E_F - E_V)$ is the energy separation between valence band edge and Fermi level in crystalline PbTe grains and $E_S$ corresponds to the band bending. As demonstrated before, diffused oxygen leads to formation of acceptor states in grain boundaries, and these acceptor states induce band bending on the surface of PbTe crystalline grains, forming p-type conduction channels on the grain surfaces. Neustroev and Osipov have analyzed the case of high grain surface states densities. The Fermi level on grain surfaces will reach the minimum energy of the surface states, $E_S$ becomes independent on surface states density, and the Fermi level position becomes stabilized.36 This is consistent with our experimental result, i.e., almost identical activation energy $E_a$ for films of different thicknesses. For these films with high oxygen concentration, the grain surface states density is high enough to "pin" the Fermi level. Therefore $(E_F - E_V)$ and $E_S$, and thus $E_a$ should be independent on film thickness even though the microstructure of these films changes gradually with thickness.

Another consequence of high density of the grain surface states and Fermi level pinning is that the temperature dependence of Hall mobility should be described by a power law instead of a thermally activated behavior.36 Mobility is usually a strong function of material impurities and temperature. An approximation of the mobility function can be written as a combination of influences from lattice vibrations (phonons) and from impurities,

$$\frac{1}{\mu} = \frac{1}{\mu_{\text{lattice}}} + \frac{1}{\mu_{\text{impurities}}},$$

where $\mu_{\text{lattice}} \propto T^{-(3/2)}$ and $\mu_{\text{impurities}} \propto T^{3/2}$. Fitting temperature dependence of mobility gives a power law with an exponent of 1.25 as shown in Fig. 8(b) for 200 nm thick film. The fitted exponent numbers for other thicknesses are summarized in Table II. The result indicates that ionized defect scattering...
dominates the electrical conduction in PbTe films, which could be expected given the polycrystalline nature of the films. Following the discussion on p-type inversion channel conduction in our previous work, and the microstructure study of the polycrystalline PbTe films in this paper, we conclude the defects mainly exist in the grain boundary regions and they result from the diffused oxygen induced acceptors.

D. Electrical property and photoconductivity

Temperature dependent resistivity of the two types of PbTe samples with low and high oxygen concentrations are shown in Figs. 9(a) and 9(b) and both are 1000 nm in thickness. Figure 9(a) shows an experimental result from 80 K to 340 K, and Fig. 9(b) is the enlarged portion of interest from 240 K to 340 K with fitted data. In the low temperature range (< 240 K) we can see clear deviation from the thermal activation model with weaker temperature dependence. Based on impedance spectroscopy measurement results, Komissarova et al. inferred that tunneling transport through grain boundary barriers dictates the electrical properties of In-doped polycrystalline PbTe films at low temperature. Alternatively, hopping conduction between localized states in the mobility gap also exhibit weak temperature dependence characterized by the famous $T^{-1/2}$ law, where $x$ is a constant greater than one depending on the system dimensionality and the form of density of states function near Fermi level. Lastly, it is well known that bandgap energy of PbTe changes significantly from room temperature (0.31 eV) to 77 K (0.20 eV), and thus large deviation of band structure and electronic transport behavior could be expected in the low temperature range. More experimental efforts will be necessary to confirm the low temperature conduction mechanism in the films.

In the high temperature range as shown in Fig. 9(b), we observe exponential dependence of resistivity on temperature. According to Figs. 8(a) and 8(b) and Table II, the Hall experiment performed at different temperatures shows that the hole concentration obeys exponential dependence while mobility follows the power law dependence on temperature. This suggests that the exponential dependence of resistivity on temperature is mainly due to the hole concentration. Therefore the fitted activation energy should be almost identical to the energy fitted from temperature dependence of hole concentration, i.e., $E_a = (E_F - E_V) - E_S$. Since it has been established that oxygen is concentrated on the surfaces of films and in the grain boundary regions instead of penetrating into the interior of the grains, we assume $(E_F - E_V)$ is the same for high and low oxygen concentration films. Additionally, we note that oxygen forms acceptor states in grain boundary regions and introduces band bending, thus in the high oxygen concentration samples the band bending $E_S$ should be larger than in the low oxygen concentration sample, resulting in smaller activation energy $E_a$. This is consistent with the fitted $E_a$ values for high (0.109 eV) and low (0.125 eV) oxygen concentration samples listed in Table I. Figure 10 schematically illustrates the band structure near grain boundary region of the two types of PbTe samples.

FIG. 10. Schematic band structure of PbTe films near grain boundary region: (a) "low oxygen concentration" sample and (b) "high oxygen concentration" sample. The relevant energy points are $E_F$, conduction band edge, $E_V$, valence band edge, $E_a$ Fermi level, $E_b$ bend banding on the surface of a grain, and $E_s$ activation energy of electrical conduction according to the grain boundary conduction channel model. In the "high oxygen concentration" sample the Fermi level becomes pinned resulting in larger band bending and smaller activation energy.

We measure excellent photoconductive signal in the wavelength range of 0.8–5 μm in the nanocrystalline PbTe films of all thicknesses. The responsivity spectrum of a 100 nm thick film is shown in Fig. 11 as an example. Up to 300 Hz, the maximum modulation frequency of our photoconductivity measurement equipment, no slow relaxation or residual conductivity has been observed which is evidenced by the anomalously long relaxation times (on the order of minutes, hours, or longer) of the photoresponse at the beginning or end of illumination. To explain the mechanism underlying photoconductivity in PbTe, here we refer to the theory described by Neustroev and Osipov for the other two similar lead chalcogenides, i.e., PbS (Ref. 37) and PbSe. In our polycrystalline PbTe films, the existence of grain surface states due to the diffused oxygen leads to p-type inversion channels between grains. Incident light with photon energy larger than the bandgap of PbTe generates electrons and holes in the bulk of grains, and these photo-generated carriers could diffuse to the grain surfaces and get spatially separated. Electrons are collected at the interface of the quasineutral bulk of grains and the surface space charge region, while holes are collected on grain surfaces (Fig. 10). Thus the band bending on the grain surfaces and the width of the surface space charge region could also be reduced. The

FIG. 11. Responsivity spectrum of 100 nm thick nanocrystalline PbTe film under 0.1 mA bias current and at −60 °C cooled by a TEC. Film shows excellent photoconductive signal in the wavelength range of 0.8–5 μm, and responsivity as high as 25 V/W is measured at 3.5 μm wavelength.
motion of the nonequilibrium holes along inversion channels on grain surfaces gives rise to the observed photoconductivity.

Finally, to further identify which contribution is dominant in the intrinsic physical process of photoconductivity in our PbTe films, i.e., illumination-induced change in the effective Hall mobility or hole concentration, the activation energies of four parameters should be determined at the same time: hole concentration, the electrical conductivity, the photoconductivity, and the decay time of the photoconductivity. In this paper the first two activation energies are mainly discussed and more work is underway focusing on the last two activation energies.

IV. CONCLUSIONS

To sum up, we demonstrate high photoconductivity in thermally evaporated PbTe films without high temperature oxygen sensitization. Studies on the microstructure and electronic structure of oxygen sensitized nanocrystalline PbTe films further reveal the photoconductivity mechanism. PbTe films prepared by single source thermal evaporation exhibit strong (200) texture with columnar grain microstructures, which is dictated by preferential nucleation orientations. The film grain size decreases with decreasing film thickness and reaches an average grain size down to 25 nm for the 100 nm thick film. Further, the vertically aligned grain boundaries can serve as diffusion “short-cuts” for oxygen, which are essential for oxygen diffusion and sensitization processes at room temperature. The large amount of oxygen incorporated results in band bending on grain surfaces, Fermi level pinning, and p-type inversion channels, leading to high infrared photoconductivity in the PbTe films. Peak photo-responsivity up to 25 V/W is attained.

Unlike their single crystalline counterpart, nanocrystalline PbTe films can be deposited on substrates without lattice-matching constraints, and thus facilitates integration of thin PbTe layers with photonic crystal structures for resonant-cavity-enhanced (RCE) infrared photodetectors, and multi-color detection. Our results also provide a method to achieve sensitization and high infrared response in lead chalcogenide films without traditional high-temperature processing, and open up an avenue toward the Holy Grail: RCE multispectral infrared photodetectors and focal plane arrays monolithically integrated on silicon ROICs.

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