

Article

Post-Annealing Effect on the Physicochemical Properties of Sn-Te-O Thin Films

Hee-Seung Yoon, Jihyeon Lee, Juyun Park and Yong-Cheol Kang * 

BB21 Plus Program, Department of Chemistry, Pukyong National University, 45-Yongso-ro, Namgu, Busan 48513, Republic of Korea; yhs1565@pukyong.ac.kr (H.-S.Y.); ljh8195896@naver.com (J.L.); jypark@pknu.ac.kr (J.P.)

* Correspondence: yckang@pknu.ac.kr; Tel.: +82-51-629-5585

Abstract

This study explores how post-deposition thermal annealing alters the structural, morphological, and electronic properties of Sn-Te-O thin films grown by radio-frequency magnetron co-sputtering. Thin films were annealed at temperatures ranging from 298 K to 873 K and analyzed using a suite of techniques, including X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and atomic force microscopy (AFM). Annealing at 473 K resulted in increased surface roughness (R_q) in Te-rich films, while higher annealing temperatures promoted a chemical shift in tin oxidation states from Sn^{2+} to Sn^{4+} . XRD patterns of films annealed at 473 K revealed the emergence of cubic-phase SnTe reflections not prominent in unannealed samples. Contact angle measurements indicated enhanced wettability in high-Te films after annealing, and work function analysis via Kelvin probe showed a trend of decreasing surface potential with lower Te content. These results provide insight into the thermal oxidation behavior and surface evolution of SnTe films, relevant for thermoelectric and topological applications.

Keywords: thin films; annealing; tin telluride; RF magnetron co-sputtering; X-ray photoelectron spectroscopy



Academic Editor: Aleksey Yerokhin

Received: 13 October 2025

Revised: 16 November 2025

Accepted: 18 November 2025

Published: 21 November 2025

Citation: Yoon, H.-S.; Lee, J.; Park, J.; Kang, Y.-C. Post-Annealing Effect on the Physicochemical Properties of Sn-Te-O Thin Films. *Surfaces* **2025**, *8*, 83. <https://doi.org/10.3390/surfaces8040083>

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Tin telluride (SnTe) is a narrow-bandgap (~0.18 eV) p-type IV–VI semiconductor that has garnered increasing attention due to its promising electrical and thermoelectric performance [1]. In addition to its excellent electronic transport properties, Sn is relatively non-toxic, earth-abundant, and compatible with green chemistry principles, making SnTe-based materials attractive for next-generation sustainable technologies [2–5]. As a result, SnTe has found application in diverse areas, including thermoelectric devices, microelectronics, superconductors, energy storage systems, and photovoltaics [6–10].

While most prior studies have focused on the intrinsic electronic and thermoelectric properties of crystalline SnTe, recent work has revealed that surface oxidation and stoichiometric variations play a decisive role in determining real-world performance. Consequently, films in this system are better described as Sn-Te-O thin films, which encompass both oxide and telluride domains that evolve with temperature and ambient exposure. Many researchers have studied the annealing effect of SnTe thin films [11–13]. The properties of the nanomaterials depend heavily on morphology and crystallinity, and temperature is one of the most important factors in controlling the morphology of such materials [14–16]. S. Xu et al. reported that the electrical conductivity of an SnTe thin film annealed at 400 °C was the highest (6.5×10^5 S/m), and this result was attributed to high-temperature

annealing reducing crystal defects at grain boundaries [11]. While several studies have examined annealing effects in SnTe films [11–13], there is a notable lack of systematic analysis addressing how annealing behavior varies across different compositional ratios of Sn and Te: a parameter known to influence both oxidation tendencies and carrier transport.

SnTe/Sn–Te–O thin films can be synthesized through various deposition methods, including chemical vapor deposition [17], thermal evaporation [18], atomic layer deposition [19], molecular beam epitaxy [20], electrodeposition [21], chemical bath deposition [22], and radio-frequency (RF) magnetron co-sputtering [23]. Among these, RF magnetron co-sputtering offers key advantages such as high deposition rates, uniform film coverage, strong film–substrate adhesion, and fine-tuned compositional control through independent power modulation of the targets [24–28]. However, films deposited at room temperature by this method are often amorphous or poorly crystalline [29], necessitating thermal post-treatment to achieve phase formation and desirable film texture. Post-deposition thermal annealing plays a critical role in tailoring the structural and electronic properties of IV–VI semiconductor thin films. For Sn–Te–O systems in particular, annealing can promote atomic diffusion and recrystallization, leading to improved crystallinity and oxidation layer evolution. It also influences stoichiometry by driving Te volatilization and Sn oxidation: processes that strongly impact carrier concentration and mobility. Previous studies have shown that controlled annealing can enhance grain growth, relieve residual strain, and modify surface electronic states, all of which are essential for optimizing film performance in thermoelectric and topological applications [30–33]. Therefore, a systematic investigation of how annealing temperature affects the composition, morphology, and electronic structure of Sn–Te–O thin films is crucial to understanding their thermal stability and functional tunability.

In this study, we fabricated Sn–Te–O thin films with controlled Sn-to-Te ratios using RF magnetron co-sputtering at room temperature, followed by systematic annealing across a range of temperatures. In contrast to earlier reports that examined SnTe oxidation at a single composition or under specific conditions, this study establishes a comprehensive composition–temperature grid (Te = 0–100%, T = 298–873 K). This systematic approach enables a direct comparison of oxidation, phase transformation, and surface-chemical evolution across the Sn–Te–O system [34–36]. The effects of both compositional ratio and thermal treatment on film properties were investigated using a comprehensive suite of surface, structural, and electronic characterization techniques. These include scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), contact angle analysis and Kelvin probe measurements, which have been used to successfully study other systems [37–40]. Through this multifaceted approach, we aim to provide a deeper understanding of the thermal evolution of Sn–Te–O thin films and offer insight into optimizing their functional properties for future device applications.

2. Materials and Methods

Sn–Te–O thin films with varying compositional ratios of Sn to Te were fabricated at 298 K using a homemade RF magnetron co-sputtering system, where this advanced instrument is previously described in detail elsewhere [41,42]. The system's base pressure was maintained at 2.4×10^{-2} Pa using a combination of two rotary vane pumps and a turbo molecular pump. High-purity Sn and Te targets (99.99%, Vacuum Thin Film Materials, Incheon, Republic of Korea) were used for deposition onto p-type Si (100) wafers, which served as the substrates. Prior to deposition, the substrates were cleaned with acetone and dried with nitrogen gas. During deposition, target temperatures were stabilized at 9 °C using a water-cooled chiller, while the substrate temperature was maintained at 25 °C.

Argon (99.999%, DONGIL, Busan, Republic of Korea) was introduced into the chamber at a controlled flow rate of 5 sccm using a mass flow controller. A pre-sputtering step was carried out for 10 min to stabilize the plasma and remove surface contamination from the targets. During this step, RF powers of 50 W and 25 W were applied to the Sn and Te targets, respectively, under a pressure of 5.33 Pa. Co-sputtering was subsequently performed at a working pressure of 1.33 Pa, with the Si substrate rotating at 5 rpm to ensure uniform film deposition.

The Sn-to-Te ratio in the films was tuned by adjusting the co-sputtering duration and applied RF powers, as detailed in Table 1. Film thicknesses, measured by a surface profiler (Alpha-step 500, Tencor, Milpitas, CA, USA), averaged 200.0 ± 4.0 nm with a uniformity variation of 5%. Post-deposition, the films were annealed in a tube furnace (DTF-40300-12T, Daeheung Scientific Co., Incheon, Republic of Korea) at 473 K, 673 K, and 873 K for 3 h. Distinct films were prepared and annealed separately at each temperature (298, 473, 673, and 873 K); no step-annealing of a single specimen was performed. The tube furnace was selected to ensure a uniform and stable temperature profile across all samples, enabling consistent annealing and reproducible thermal exposure. All annealing treatments were carried out under ambient air without external gas flow, allowing a controlled level of oxygen exposure during heating. This condition was intentionally chosen to study the oxidation behavior and thermal stability of Sn–Te–O system, as oxygen incorporation plays a key role in phase evolution and surface electronic modification. In this study, we denote the samples using the format “XSTY,” where X represents the annealing temperature (in K), “ST” stands for the Sn–Te–O system, and Y indicates the relative atomic percentage of Te, as determined by XPS and EDX analyses. Samples labeled ST10 to ST40 are classified as Sn-rich, whereas ST70 to ST80 are considered Te-rich. The surface morphology and elemental composition were analyzed using a field-emission scanning electron microscope (SEM, MIRA3, TESCAN, Brno, Czech Republic) equipped with an energy-dispersive X-ray spectroscopy detector (EDX, X-maxN 50, Oxford Instruments, Abingdon, UK). SEM micrographs were acquired at $150,000\times$ magnification and 10.0 kV accelerating voltage. Surface topography and roughness were characterized using an atomic force microscope (AFM, Park NX10, Park Systems, Suwon, Republic of Korea).

Table 1. The applied RF sputtering power on targets and co-sputtering time for fabricating SnTe/Sn–Te–O thin films.

Sample		ST0	ST10	ST20	ST40	ST70	ST80	ST100
RF sputtering power [W]	Sn	30	46	35	29	17	14	NA
	Te	NA	8	12	16	20	20	20
Co-sputtering time [s]		1152	510	532	510	600	615	900

NA stands for not available.

XPS measurements were performed using a monochromatic Al K α X-ray source ($h\nu = 1486.6$ eV) with the ESCALab MKII system (V.G. Scientific Co., West Sussex, UK). The analysis chamber was maintained at a base pressure of 2.4×10^{-6} Pa, supported by a rotary vane pump, turbo molecular pump, and ion pump. Survey spectra were acquired at a pass energy (PE) of 100 eV with an energy step size of 0.5 eV to determine overall composition, while high-resolution spectra of Sn 3d, Te 3d, O 1s, and C 1s were recorded at PE = 50 eV with 0.05 eV steps for chemical-state analysis. All samples were transferred from the sputtering chamber to the XPS analysis system under ambient laboratory air. Because no load-lock was available on the instrument, each specimen experienced approximately 1–2 h of unavoidable air exposure during handling and instrument preparation. The significant O 1s signal observed in the room-temperature samples therefore reflects native

surface oxidation formed during this air-transfer period rather than intentional oxygen incorporation during deposition. Each high-resolution spectrum represents the average of nine scans to improve the signal-to-noise ratio. The take-off angle was fixed at 45° , and data fitting was carried out using XPSPeak 4.1 software. Spectra were deconvoluted using mixed Gaussian–Lorentzian GL(30) line shapes with Shirley background subtraction. The instrumental energy resolution was verified from the Ag Fermi-edge width (0.55 eV at 50 eV PE). For core-level fitting, metallic Sn^0 and Te^0 peaks were slightly narrower FWHM (2.29–2.33 eV) than oxides (2.33–2.38 eV), and all spectra were modeled using a GL(30) mixed Gaussian–Lorentzian line shape; mild asymmetry for the metallic components did not significantly improve residuals [43]. $\text{Sn } 3d_{5/2}$ and $\text{Te } 3d_{5/2}$ were fitted as doublets with fixed 3:2 area ratios to avoid complexity, as the spin–orbit coupling is very large and separations of 8.45 ± 0.05 eV and 10.40 ± 0.05 eV, respectively. O 1s has no spin–orbit partner and was fitted as a single core level, and the full width at half maximum (FWHM) can be found in Table S1. Relative sensitivity factors (RSFs) of Sn 4.3, Te 5.4, and O 0.66 were used for quantification (Please see Table S2). All spectra were referenced to the C 1s = 284.6 eV adventitious carbon peak. Charge neutralization was achieved using a low-energy flood gun. The fit residuals ($\chi^2 \approx 2\text{--}3$) verified excellent agreement between experimental and modeled spectra. Survey spectra and representative C 1s regions are provided in Figures S1–S3. Crystallinity was examined via X-ray diffraction (XRD, Ultima IV, Rigaku, Tokyo, Japan) using a Cu K α source (40 kV, 30 mA). Contact angle measurements were conducted using a custom-built system with distilled water (DW) and ethylene glycol (EG) as probe liquids to estimate the surface free energy. All quoted uncertainties ($\pm$ eV) for XPS binding energies and FWHM values represent the fitting standard error obtained from the nonlinear least-squares peak-fitting procedure.

Kelvin probe (KP6500, McAllister Technical Services, Coeur d’Alene, ID, USA) measurements were conducted in ambient air using a vibrating capacitive probe. The probe work function (ϕ_{tip}) was calibrated before each session against Au using $\phi_{\text{tip}} = \phi_{\text{Au}} + \text{CPD}_{\text{measured}}$. The sample work function was then calculated as $\phi_{\text{sample}} = \phi_{\text{tip}} - \text{CPD}_{\text{sample}}$. The tip–sample spacing was maintained at ~ 1 mm using a manufacturer-supplied spacer. Each contact potential difference (CPD) value represents an average of 5 measurements per sample. The absolute uncertainty of KP measurements was $\pm 0.10\text{--}0.20$ eV, with intra-session repeatability of $\pm 0.02\text{--}0.03$ eV. Electrical properties were measured using a 4-point probe system (Loresta-GP MCP-T600, Mitsubishi Chemical, Kanagawa, Japan). The sheet resistance values were calculated from the measured voltage–current characteristics, with an estimated film thickness of 200 ± 4 nm. Each data point represents the average of five measurements taken at different surface locations, and the error bars denote one standard deviation (Figure S5).

3. Results and Discussion

Table 2 summarizes the elemental composition (Sn, Te, and O) of the XSTY thin films, revealing how thermal treatment influences the chemical makeup of the samples. Variations in stoichiometry across annealing temperatures are attributed to temperature-dependent chemical reactions. At 473 K, the STY thin films formed a stable cubic Sn–Te crystalline structure, as previously reported [30]. Up to this temperature, the Sn and Te components retained near-stoichiometric balance. However, at 673 K, approaching Te’s melting point, the Te content in ST80 films declined markedly from 65.2% at 473 K to just 18.0% [31]. This drop indicates significant loss of Te, likely due to volatilization. Although the absolute Te abundance differs among compositions, the overall temperature-driven Te depletion is reproducible across independently prepared samples. Simultaneously, the oxygen content increased at 673 K, consistent with the oxidative transformation of SnTe into SnO_2 and TeO_2 .

via the reaction: $\text{SnTe} + 2\text{O}_2 \rightarrow \text{SnO}_2 + \text{TeO}_2$ [32]. At the highest annealing temperature (873 K), the depletion of Te became more pronounced, likely due to gas-phase evaporation of both elemental Te and TeO_2 [32]. Because annealing was conducted in ambient air, a progressive rise in oxygen incorporation was observed with increasing temperature. This behavior aligns with thermodynamic expectations, given the more negative standard enthalpy of formation for SnO_2 (−577.6 kJ/mol) [33] compared to TeO_2 (−322.6 kJ/mol) [44], suggesting that Sn has a greater tendency to form stable oxides than Te. Accordingly, the observed compositional shifts can be attributed to the preferential binding of oxygen to Sn at elevated temperatures, effectively displacing Te from the films.

Table 2. The relative atomic percentages of Sn, Te, and O in the XSTY thin films that were obtained by EDX.

Annealing Temperature [K]	Constituent Element	Relative Atomic Percentage [%]						
		Sample						
		ST0	ST10	ST20	ST40	ST70	ST80	ST100
298	Sn	41.4	43.3	39.6	24.2	11.7	11.9	0.0
	Te	0.0	4.0	9.6	18.6	33.9	35.5	60.6
	O	13.7	9.3	8.3	11.4	6.2	4.2	1.4
	Si	44.9	43.5	42.5	45.8	48.2	48.3	38.0
473	Sn	37.7	45.1	37.6	24.7	9.0	11.7	0.0
	Te	0.0	2.4	8.9	17.2	26.8	38.5	56.4
	O	12.1	18.1	10.9	13.4	9.0	6.8	2.7
	Si	50.2	34.4	42.6	44.7	55.2	43.0	40.9
673	Sn	18.1	19.8	24.1	18.8	5.7	5.0	0.0
	Te	0.0	1.4	4.7	13.6	0.6	4.1	0.8
	O	28.9	28.7	36.5	16.6	14.5	11.9	3.0
	Si	53.0	50.0	34.7	51.0	79.3	79.0	96.3
873	Sn	18.1	20.0	19.5	13.3	5.0	4.2	0.0
	Te	0.0	NA	0.1	0.2	0.2	0.2	NA
	O	40.1	46.1	45.0	32.7	13.2	10.8	1.7
	Si	41.8	33.9	35.5	53.9	81.6	84.8	98.3

(NA stands for not available).

Although the films were deposited in an Ar atmosphere without intentional oxygen introduction, a significant O 1s signal was detected in the as-deposited (298 K) samples. This oxygen presence can be either attributed to trace amounts of O_2 and H_2O remaining in the sputtering chamber at its base pressure ($\sim 10^{-2}$ Pa) or from native/air oxidation during the transfer process. Given the strong oxygen affinity of both Sn and Te, partial oxidation can occur even under these low-pressure conditions. Additionally, brief exposure to ambient air during sample transfer may have further contributed to the formation of a thin native oxide layer. Since XPS probes only a few nanometers into the film surface, this surface oxidation is readily detected despite the bulk film remaining largely stoichiometric. Such surface oxidation phenomena are widely observed in Sn–Te–O thin films and do not affect the overall interpretation of the compositional or electronic results.

Each panel includes the corresponding Z-range (± 10 to ± 200 nm) to allow direct comparison without normalizing contrast. The surface roughness was derived from height histograms, and the instrument's vertical resolution (< 0.05 nm) was used to estimate uncertainty. The roughness progressively increases with temperature, reflecting the transition from dense to granular oxide textures. Complementing these morphological observations, Figure 1 displays the root mean square (R_q) roughness values derived from AFM measurements. At 298 K, R_q values rose in tandem with increasing Sn concentration, reinforcing

the correlation between Sn content and surface texturing. In contrast, among the films annealed at 473 K, the highest R_q values were observed in Te-rich compositions. For Sn-rich and pure Sn films, grain coarsening and surface roughening became more pronounced as the annealing temperature increased from 298 K to 873 K, an effect attributed to the thermal-driven agglomeration of Sn nanoparticles [45]. Meanwhile, in Te-rich thin films, surface roughness initially increased between 298 K and 473 K. However, beyond this range, as the temperature rose to 873 K, a decline in R_q was observed, likely due to the melting of Te and subsequent surface smoothening. Interestingly, pure Te films exhibited a distinct trend: R_q increased from 298 K to 673 K, consistent with melting and coalescence of Te into larger grains. Upon annealing at 873 K, however, the roughness decreased, presumably due to the partial or complete evaporation of Te under high-temperature conditions.

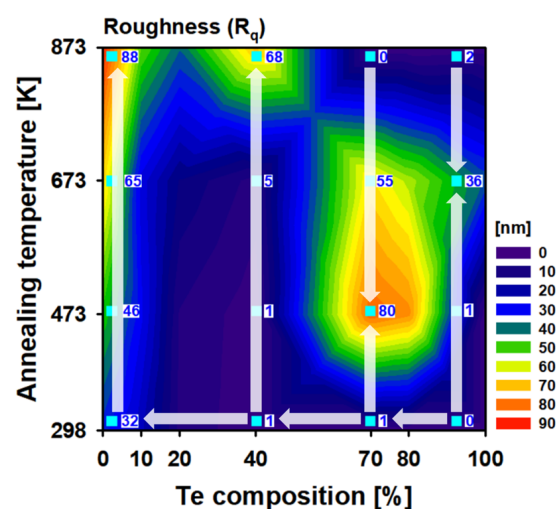


Figure 1. The contour plot displaying the surface roughness of the SnTe/Sn–Te–O thin films at various annealing temperatures. (The direction of arrow indicates the direction of increasing value).

The observed morphological evolution can also be partially attributed to strain and stress relaxation processes occurring during annealing. Thin films of Sn–Te–O experience differential thermal expansion relative to the SiO_2/Si substrate, resulting in interfacial strain and stress accumulation during heating. These stresses promote grain boundary migration and coalescence, consistent with the increased grain size and surface roughness observed in Figures 1 and 2. At moderate annealing temperatures (≈ 473 K), such strain relaxation aids in recrystallization and the formation of the cubic SnTe phase, as supported by the XRD patterns. However, at higher temperatures, excess strain energy may be relieved through Te volatilization and oxidation, producing voids or stress-induced delamination that contribute to the observed roughness decline above 673 K. Similar strain-driven morphological transitions have been reported in other IV–VI chalcogenide thin films, where lattice mismatch and thermal expansion gradients govern film texture and stability [46–48]. These considerations suggest that the interplay between thermal strain relaxation, compositional redistribution, and interfacial stress significantly influences the structural and electronic evolution of the XSTY thin films during annealing.

SEM micrographs of the 873ST70 and 873ST80 thin films revealed distinct contrast variations: bright and dark regions suggestive of compositional inhomogeneity. EDX analyses of these regions, presented in Figure 3, confirmed that the brighter areas were rich in Sn, while the darker areas were predominantly composed of Si. These findings underscore Sn’s strong propensity to aggregate under elevated annealing conditions. To further probe the chemical states of the constituent elements, high-resolution XPS was conducted. The resulting spectra for Sn $3d_{5/2}$, Te $3d_{5/2}$, and O $1s$ are shown in

Figure 4a–e, 4f–j, and 4k–o, respectively. Notably, the large spin–orbit splitting values of Sn 3d (8.50 eV) and Te 3d (10.34 eV) [49,50] necessitated deconvolution focusing solely on their 3d_{5/2} components to simplify peak assignment. The binding energy peak positions and oxidation states derived from this analysis are summarized in Table 3.

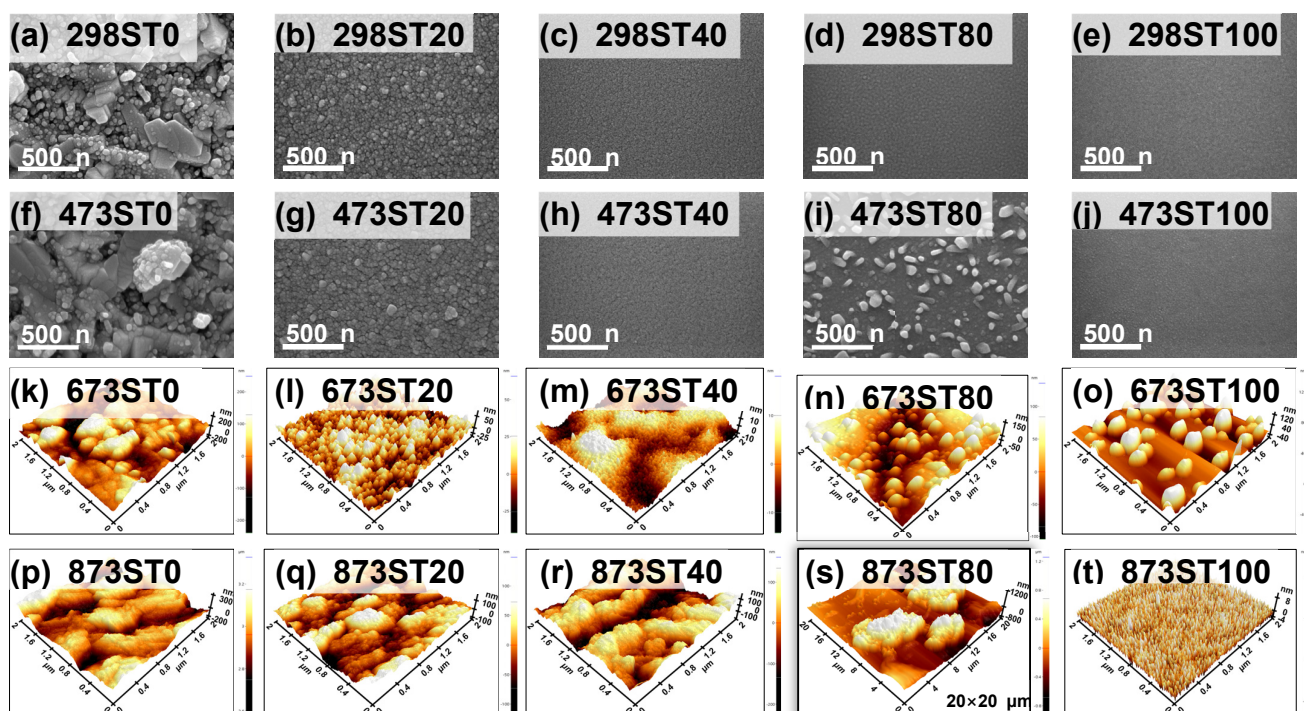


Figure 2. SEM micrographs ($\times 150$ k) of XSTY thin films: (a–e) for 298 STY [44] and (f–j) for 473 STY. AFM micrographs of XSTY thin films: (k–o) for 673 STY and (p–t) for 873 STY. 298 K data are used as a reference [51].

Figure 2 presents the surface morphologies of STY thin films subjected to various annealing temperatures. Notably, films deposited at 298 K displayed an increase in grain size with higher Sn content, suggesting that Sn significantly influences grain formation more so than Te [51]. SEM micrographs show that film morphology remains compact up to 673 K but becomes non-uniform and partially discontinuous at 873 K, consistent with enhanced oxidation and island formation. AFM micrographs were analyzed using the manufacturer’s tip (checked before and after use).

For Sn 3d_{5/2}, three chemical states were resolved: Sn⁴⁺ at 487.1 ± 0.3 eV, Sn²⁺ at 486.2 ± 0.3 eV, and metallic Sn⁰ at 485.1 ± 0.3 eV [52,53]. A clear thermal evolution was observed: Sn in the XSTY thin films transitioned from primarily Sn²⁺ (SnO) at lower temperatures to predominantly Sn⁴⁺ (SnO₂) upon annealing at 873 K. Interestingly, metallic Sn⁰ species vanished at 673 K in Sn-rich films, while in Te-rich compositions, this transition occurred earlier, disappearing by 473 K. This accelerated oxidation behavior in Te-rich films may stem from the higher electronegativity of Te (2.1) compared to Sn (1.96), which facilitates oxidation of Sn in such environments. Similarly, deconvolution of the Te 3d_{5/2} spectra revealed four distinct species: Te⁶⁺ at 577.3 ± 0.2 eV, Te⁴⁺ at 576.4 ± 0.2 eV, elemental Te⁰ at 573.5 ± 0.2 eV, and Te^{2−} at 572.5 ± 0.2 eV [54,55]. Between 298 K and 473 K, Te exhibited divergent behaviors depending on the film composition. In Sn-rich samples, the dominant Te species was Te^{2−}, consistent with its bonding to Sn in the formation of SnTe domains within the oxidized matrix.

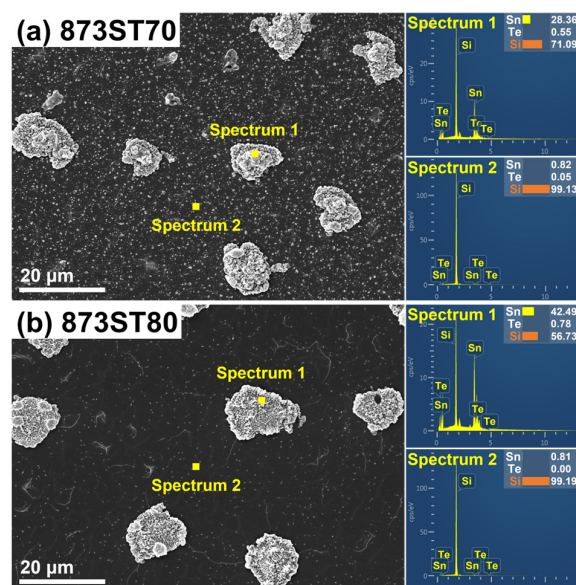


Figure 3. EDX spot profile of SnTe/Sn-Te-O thin films; (a) 873ST70 and (b) 873ST80.

In contrast, Te-rich thin films predominantly featured neutral Te (Te^0) and/or oxidized Te^{4+} species, with no detectable Te^{2-} present in pure Te samples due to the absence of Sn for compound formation. As annealing progressed, the $\text{Te } 3d_{5/2}$ peak intensity in the ST100 films declined, indicative of Te volatilization at elevated temperatures. Interestingly, the Te signal in Sn-Te binary films annealed at 873 K remained stronger than in pure Te films. This observation suggests that the presence of Sn might have mitigated Te loss through a stabilizing reaction with TeO_2 , potentially forming a ternary oxide compound such as SnTe_3O_8 via the reaction: $\text{SnO}_2 + 3\text{TeO}_2 \rightarrow \text{SnTe}_3\text{O}_8$ [32]. O 1s spectra further support this hypothesis, showing a progressive increase in the O-Sn $^{4+}$ signal as the annealing temperature rose from 298 K to 873 K, confirming the thermal oxidation of Sn. This trend aligns well with the Sn 3d spectral analysis in Figure 4. For the ST100 films, the O 1s spectrum (Figure 4o) revealed peaks associated with O-Si bonding that appeared only in samples annealed at 673 K and above. These signals point to the underlying substrate becoming more exposed as Te evaporated at higher temperatures.

At elevated annealing temperatures (≥ 773 K), the Sn-Te-O films exhibit partial discontinuity, allowing localized detection of the Si substrate. To ensure accurate compositional accounting, Si was explicitly included in both EDX and XPS analyses. In the O 1s spectra, deconvolution was performed into lattice oxygen (O-Sn/O-Te), interfacial oxygen (O-Si), and minor adsorbed species (O=C, H_2O). The emergence of the O-Si component in the 873 K sample (ST100) indicates partial film thinning and SiO_x formation at the interface. To support the O 1s deconvolution where O-C, O-Sn, and O-Te components overlap within the instrumental FWHM (≈ 2.3 eV), assignments were cross-checked against the thermal evolution of Sn and Te core-level states and valence-band trends; the O-Te component was retained only when consistent with co-evolving Te oxidation and VB spectral shape.

Figure 5 presents the relative atomic concentrations of Sn, Te, and O in XSTY thin films, as determined through XPS analysis. As a surface-sensitive technique, XPS is widely employed to probe the chemical composition of thin film surfaces [56]. The distribution of elements varied significantly with annealing temperature, highlighting changes in surface chemistry across different film compositions. In the case of pure Sn thin films, higher annealing temperatures led to a pronounced increase in both Sn and O content at the surface, suggesting progressive surface oxidation and the formation of a Sn oxide-rich layer. For Sn-Te binary films, the behavior was more dynamic: between 298 K and

473 K, a noticeable reduction in surface Te was observed, attributed to Te diffusion into the bulk. However, upon further annealing up to 873 K, Te re-emerged at the surface, likely due to outward diffusion, accompanied by an increased oxygen signal indicative of various oxide formation processes. In contrast, pure Te thin films showed a different trajectory. As the annealing temperature increased, Te gradually evaporated from the surface, leading to greater exposure of the underlying Si substrate and a corresponding decline in Te signal. This redistribution and volatilization of elements are expected to influence not only the electrical characteristics but also the overall physicochemical behavior of the XSTY thin films. Figure 6 illustrates the evolution of XRD patterns in Sn–Te–O thin films under varying annealing temperatures. Figure S6 shows XRD patterns of Sn–Te–O thin films recorded over the full 2θ range ($10\text{--}90^\circ$). The Si substrate peaks are labeled, and the region above 60° is indicated as the Si fingerprint zone, where film reflections may overlap with substrate features. To enhance data clarity, the main figure focuses on the lower-angle region ($10\text{--}60^\circ$), where the principal film reflections are observed with minimal substrate interference. The p-type Si(100) substrate has a diamond cubic structure and does not undergo any temperature-driven phase transitions within our annealing range; therefore, the strong Si(220) and Si(311) reflections near $\sim 48^\circ$ and $56\text{--}58^\circ$ 2θ originate solely from the substrate and provide no film-related structural information. Because these intense substrate peaks compress the dynamic range of the thinner Sn–Te–O reflections, they were excluded from the main-text XRD plots for improved visibility; however, full unedited scans ($10\text{--}90^\circ$ 2θ) for all samples are provided in the Supporting Information. The major diffraction peaks correspond to SnO₂ (rutile) and SnO (tetragonal), with weaker signals attributable to TeO₂ or residual Te⁰ at lower annealing temperatures. Minor reflections appearing at higher temperatures are marked as tentative because they originate from isolated weak peaks or overlap with substrate signals.

At room temperature (298 K), pure Sn and Sn-rich films exclusively exhibited tetragonal Sn phases, with diffraction peaks at 30.65° , 32.02° , 43.87° , 44.91° , and 55.35° , corresponding to Sn(200), Sn(101), Sn(220), Sn(211), and Sn(301), respectively, in agreement with JCPDS no. 86-2265. Meanwhile, pure Te films displayed peaks at 22.97° , 27.55° , 40.33° , 46.91° , and 49.56° , characteristic of a hexagonal structure (JCPDS no. 78-2312), including Te(100), Te(101), Te(200), and Te(021) reflections. Interestingly, as-deposited Sn–Te films lacked any discernible SnTe crystalline peaks. This observation aligns with previous findings by K.A. Campbell et al., who noted that IV–VI metal chalcogenides typically crystallize only above 473 K [57]. Upon annealing beyond this threshold, distinct SnTe reflections emerged, confirming the onset of crystallization, as evident in Figure 6b,c.

The XRD patterns of the films are shown in Figure 6. Upon annealing the STY thin films at 473 K, the XRD patterns began to reveal distinct SnTe crystalline phases with a cubic structure, complementing those seen at room temperature (298 K). These SnTe-specific reflections emerged exclusively in the mixed-composition samples, with characteristic diffraction peaks at 28.27° , 40.45° , and 50.10° , corresponding to the (200), (220), and (222) planes of cubic SnTe (JCPDS no. 08-0487). As the annealing temperature increased to 673 K, signs of SnTe oxidation became prominent [32]. This transition was in agreement with the earlier EDX results, which had indicated the formation of various oxide phases beginning at this temperature. Consequently, new peaks appeared in the XRD spectra, consistent with the formation of both SnO and SnO₂. Specifically, SnO phases were identified by their reflections at 18.35° , 29.94° , and 50.79° , corresponding to the (001), (101), and (112) planes (JCPDS no. 85-0712). SnO₂ phases were similarly evident at 26.56° , 33.89° , and 51.75° , indexing to the (110), (101), and (211) planes (JCPDS no. 88-0287).

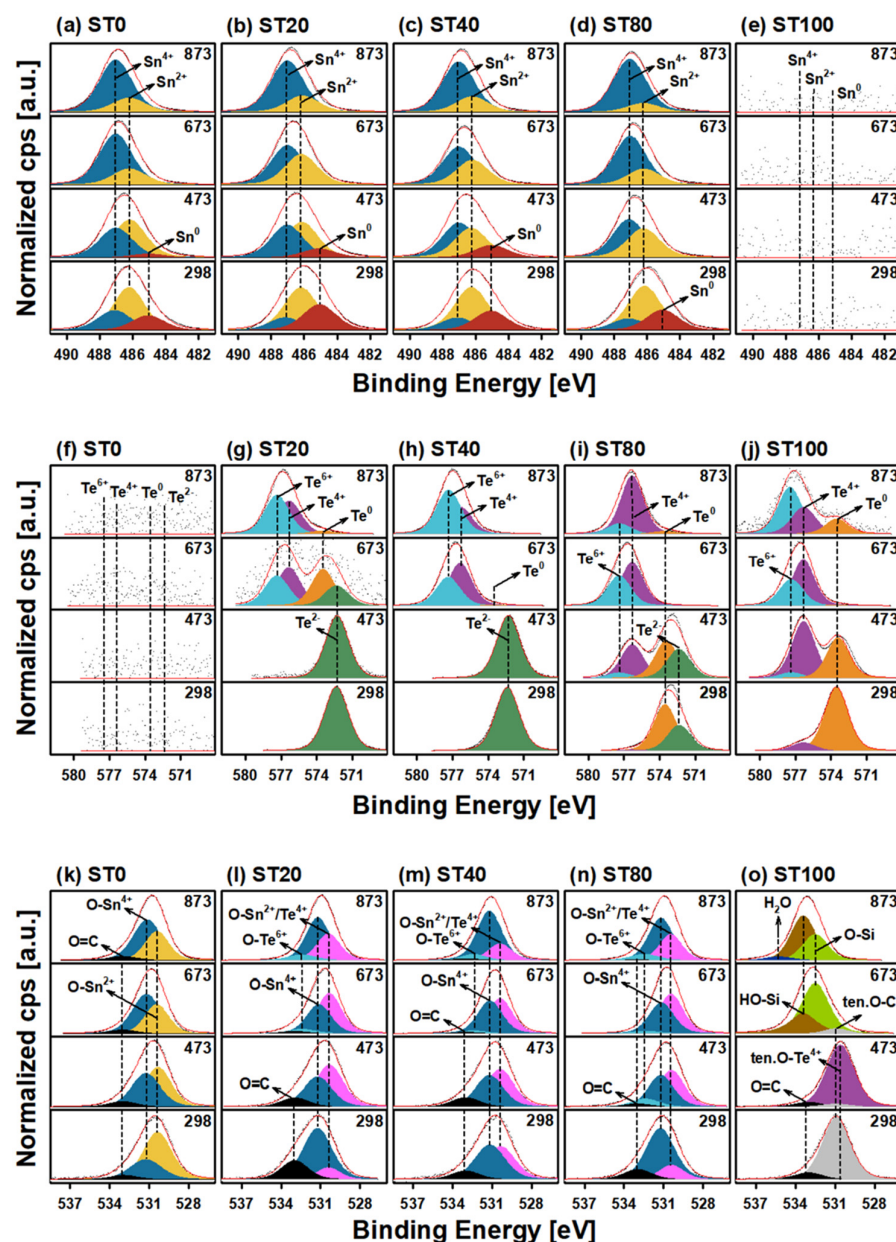


Figure 4. Deconvoluted XPS spectra: (a–e) for Sn 3d_{5/2}, (f–j) for Te 3d_{5/2}, and (k–o) for O 1s region of the SnTe/Sn–Te–O thin films. 298 K data are used as a reference [51] (ten. stands for tentative). The dots represent the raw data, color filled peaks represent deconvoluted peaks with the same binding energy, and the red line represents the sum of the deconvoluted peaks.

Table 3. The relative atomic percentages of Sn, Te, and O in the XSTY thin films that were obtained by XPS.

Relative Atomic Percentage [%]															
Sample	Sn				Sn%	Te				Te%	O				O%
	Sn ⁰	Sn ²⁺	Sn ⁴⁺	Te ²⁻		Te ⁰	Te ⁴⁺	Te ⁶⁺	O-Sn ²⁺		O-Sn ⁴⁺	O-Te ⁶⁺	O=C		
ST0	298	21	54	25	39						64	31	NA	5	61
	473	5	52	43	40						48	45	NA	7	60
	673	NA	24	76	34						36	58	NA	6	66
	873	NA	21	79	34						35	59	NA	6	66

Table 3. Cont.

Relative Atomic Percentage [%]																
Sample	Sn					Te					O					O%
	Sn ⁰	Sn ²⁺	Sn ⁴⁺	Sn%	Te ^{2−}	Te ⁰	Te ⁴⁺	Te ⁶⁺	Te%	O-Sn ²⁺	O-Sn ⁴⁺	O-Te ⁶⁺	O=C			
										O-Sn ²⁺ /Te ⁴⁺	O-Sn ⁴⁺	O-Te ⁶⁺	O=C			
ST10	298	35	44	21	43	100	NA	NA	NA	7	34	54	NA	12	50	
	473	13	60	27	38	100	NA	NA	NA	1	62	29	NA	9	61	
	673	NA	47	53	35	NA	NA	NA	NA	0	63	29	6	2	65	
	873	NA	13	87	31	NA	NA	50	50	5	51	39	10	NA	64	
ST20	298	32	54	14	50	100	NA	NA	NA	10	13	65	NA	22	40	
	473	10	46	44	37	100	NA	NA	NA	2	51	38	NA	11	61	
	673	NA	43	57	35	NA	NA	NA	NA	NA	52	37	7	4	65	
	873	NA	23	77	31	NA	NA	50	50	6	35	55	10	NA	63	
ST40	298	28	57	15	33	100	NA	NA	NA	15	42	46	NA	12	52	
	473	17	38	45	37	100	NA	NA	NA	6	47	40	NA	13	57	
	673	NA	38	62	31	NA	NA	55	45	6	49	46	3	2	63	
	873	NA	25	75	28	NA	NA	29	71	7	22	66	9	3	65	
ST70	298	27	57	16	19	41	59	NA	NA	39	45	50	NA	5	42	
	473	NA	33	67	21	18	32	45	5	22	53	31	12	4	57	
	673	NA	31	79	26	NA	NA	55	45	11	41	47	10	2	63	
	873	NA	25	75	31	NA	16	52	32	6	40	52	8	NA	63	
ST80	298	25	58	17	12	36	62	2	NA	42	17	68	NA	15	46	
	473	NA	42	58	26	31	31	31	7	13	45	39	11	5	61	
	673	NA	25	75	12	NA	NA	59	41	27	54	42	4	NA	61	
	873	NA	13	87	31	NA	NA	83	17	6	32	57	9	2	63	
ST100											O-Te ⁴⁺	O-C	O-Si	O=C	HO-Si	H ₂ O
	298					NA	88	12	NA	65	NA	91	NA	9	NA	35
	473					NA	39	55	6	49	88	4	NA	8	NA	51
	673					NA	NA	67	33	6	NA	5	64	NA	31	94
	873					NA	NA	NA	NA	NA	NA	NA	35	NA	59	100

(NA stands for not available).

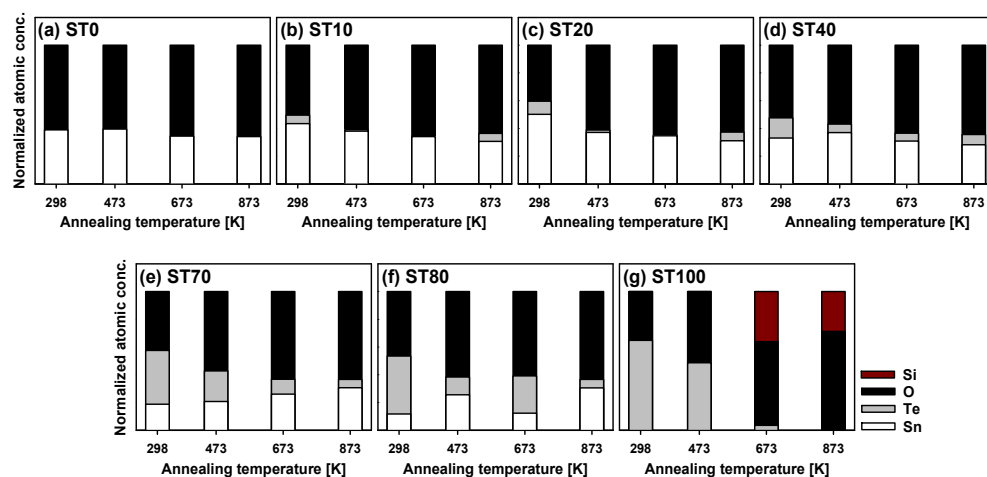


Figure 5. The relative atomic concentration of Sn, Te, and O on SnTe/Sn-Te-O thin films was obtained by XPS: (a) ST0, (b) ST10, (c) ST20, (d) ST40, (e) ST70, (f) ST80 and (g) ST100 thin films.

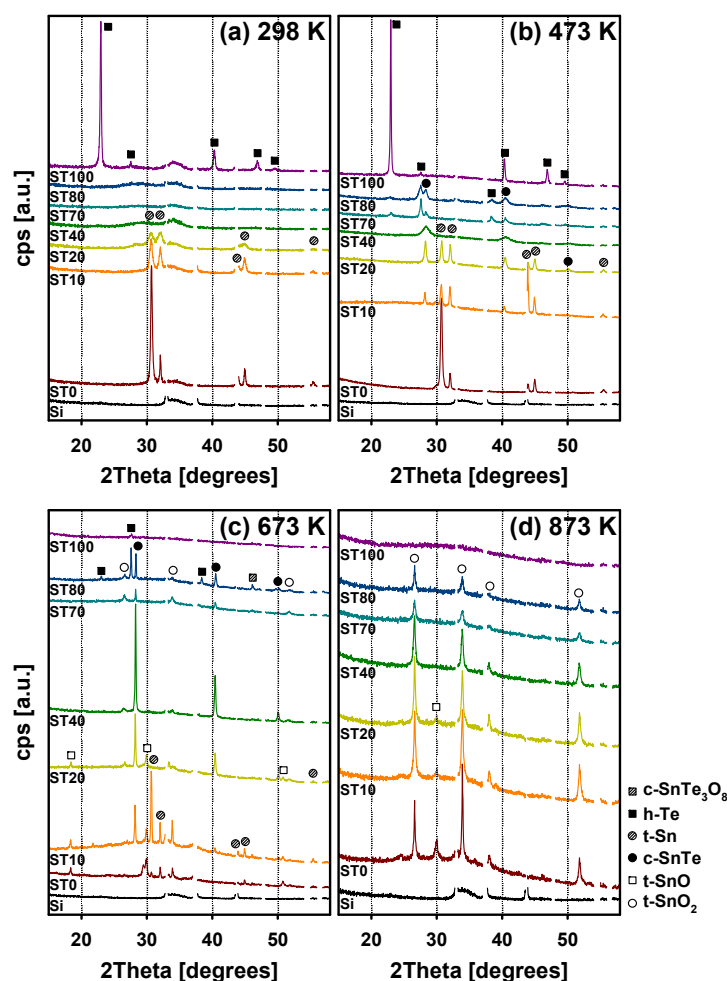


Figure 6. The XRD patterns of XSTY thin films: (a) 298 STY, (b) 473 STY, (c) 673 STY, and (d) 873 STY thin films. 298 K data are used as a reference [51].

The oxidation of SnTe under elevated thermal conditions is known to proceed via the reaction $\text{SnTe} + 2\text{O}_2 \rightarrow \text{SnO}_2 + \text{TeO}_2$ [32]. In our XPS analysis at 673 K (Figure 4, Table 3), the dominant chemical species were consistent with Sn^{4+} and Te^{4+} , corroborating the oxidation products observed in the XRD. Notably, in Te-rich films, a unique oxide phase, SnTe_3O_8 , was detected. Its cubic structure yielded a diffraction peak at 46.05° , assignable to the (440) plane (JCPDS no. 70-2440). This compound likely forms through a secondary reaction between SnO_2 and TeO_2 : $\text{SnO}_2 + 3\text{TeO}_2 \rightarrow \text{SnTe}_3\text{O}_8$ [32]. JCPDS reference patterns corresponding to Si (41-1111), Sn (86-2265), Te (78-2312), SnO (85-0712), SnO_2 (88-0287), SnTe (08-0487), SnTe_3O_8 (70-2440), and TeO_2 (84-1777) have been overlaid onto the XRD plots provided in the Supporting Information (Figure S7). The 1:3 stoichiometric requirement of this reaction aligns with the excess TeO_2 found in Te-rich films, offering a plausible explanation for the observed phase. At the highest annealing temperature of 873 K, the XRD patterns were dominated by Sn oxide peaks alone, as volatile Te species had evaporated from the surface [31]. Overall, the trend across increasing temperatures indicated a gradual decline in the intensity of elemental Sn, Te, and SnTe peaks, while various oxide phases became increasingly prominent.

Figure 7 presents the measured contact angles of deionized water (DW) and ethylene glycol (EG) droplets placed on the XSTY thin film surfaces, alongside the corresponding surface roughness data obtained from atomic force microscopy (AFM). From these contact angle measurements, the surface free energy (SFE) of each thin film was subsequently derived. Static water contact angles were measured at four distinct positions per sample,

yielding an average standard deviation of $\pm 2.43^\circ$, indicating high reproducibility. The calculation of SFE followed the approach proposed by Owens and Wendt, which refines Young's equation to account for both polar and dispersive interactions at the solid–liquid interface [58]:

$$\gamma_L(1 + \cos \theta) = 2\sqrt{\gamma_S^d \gamma_L^d} + 2\sqrt{\gamma_S^p \gamma_L^p} \quad (1)$$

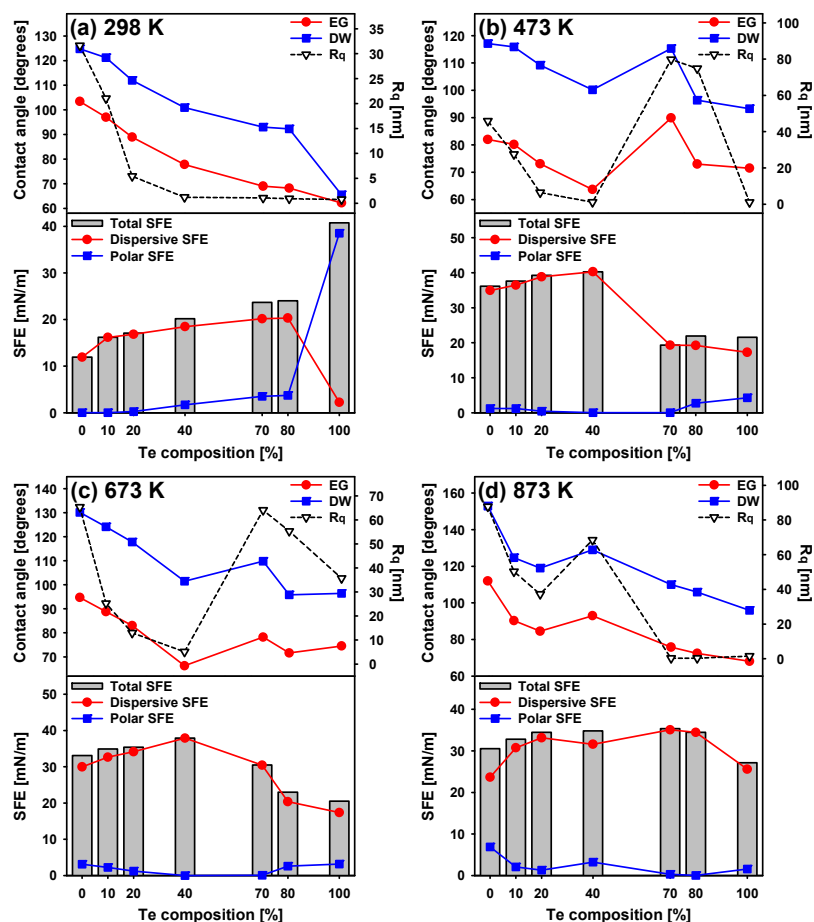


Figure 7. The correlation between contact angle and root mean square value of roughness along with the SFE of XSTY thin films: (a) 298 STY, (b) 473 STY, (c) 673 STY, (d) 873 STY thin films. 298 K data are used as a reference [51].

The surface tension of the liquid is denoted as γ_L , and the contact angle between the liquid and the surface of the solid thin film is expressed as θ . γ_S^d and γ_S^p are the dispersive SFE and polar SFE on the surface of the solid thin film. γ_L^d and γ_L^p are the dispersive SFE and polar SFE of the used liquid, respectively. Total SFE (γ_S) was calculated by adding γ_S^d and γ_S^p . At all four annealing temperatures studied, the Te composition showed a consistent trend with contact angle for both DW and EG. Additionally, contact angle variations appeared to correlate with changes in surface roughness across the films. However, these relationships differed between the as-deposited and annealed samples. For the STY thin films fabricated at 298 K, the polar component of SFE increased with rising Te content. Interestingly, the dominant contributor to the total SFE varied with composition: in the pure Sn thin film, the dispersive component prevailed, whereas in the pure Te thin film, the polar component was more significant. This shift aligns with the higher intrinsic polarity of Te compared to Sn and explains the observed decrease in contact angle with increasing Te concentration [51].

In contrast, the annealed films, processed at 473, 673, and 873 K, were governed predominantly by dispersive SFE. Notably, roughness emerged as a critical factor. It has been previously reported that nanoscale roughness can significantly increase contact angle values [59]. This suggests that in these thermally treated films, surface roughness had a stronger influence on wettability than compositional polarity. Indeed, as shown in Figure 2, annealing induced substantial morphological changes. Aggregates became prominent, particularly in the ST70 and ST80 samples, which also exhibited the highest R_q values. The resulting large contact angles can be attributed to a transition in wetting behavior: from the Wenzel regime, characterized by full contact between the liquid and surface, to the Cassie regime, where trapped air pockets reduce liquid-solid contact. This transition is known to occur on roughened surfaces and weakens surface adhesion [60,61].

Figure 8 illustrates how annealing temperature influences the work function of XSTY thin films, as determined by KP measurements, alongside the log-scale electrical conductivity data obtained via a four-point probe method. Each dataset includes the Kelvin probe calibration using a metallic Au reference, confirming measurement consistency within ± 0.1 eV. The relatively high ϕ values observed for the oxidized films (~ 6.0 eV) can be attributed to the formation of SnO_2 -rich surface layers, consistent with reported values as high as 5.9 eV for stoichiometric SnO_2 [62]. In contrast, Te-rich films exhibit markedly lower work functions of ~ 5.0 – 5.5 eV, in agreement with our Kelvin probe measurements and the lower electron affinity of elemental Te. At elevated annealing temperatures, partial Te volatilization and surface inhomogeneity further reduce Φ by exposing regions of the underlying Si substrate ($\phi_{\text{Si}} \approx 4.66$ eV). These competing effects reconcile the observed differences: SnO_2 -dominated surfaces drive the ϕ upward toward ~ 6.0 eV, whereas Te-rich or partially Te-depleted surfaces pull ϕ downward into the 5.0–5.5 eV range. This compositional sensitivity explains the non-monotonic work-function evolution observed across the annealing temperatures. XPS analysis (Figure 4k–o) verified that adventitious carbon was minimal following annealing, thus excluding C-related surface charging as a major factor. The slightly higher apparent ϕ values may also reflect upward band bending caused by oxygen-rich surface termination, as has been observed in other oxide–semiconductor systems. Since the Sn–Te–O films were deposited on p-type Si (100) substrates, the measured conductivity reflects a composite film + substrate system, particularly at higher annealing temperatures (≥ 673 K) where partial discontinuity or oxidation reduces film coverage. The influence of the substrate has therefore been explicitly acknowledged in the interpretation, and resistivity values reported here are normalized to the measured film thickness with corresponding error bars (Table S3). The reported work function values primarily reflect the composite film/substrate system. The observed trend, an initial decrease in resistance up to 473 K followed by an increase at higher temperatures, is consistent with the oxidation-induced loss of metallic Te and increased film nonuniformity (Figures 8 and S4).

A notable divergence in behavior emerged between pure-element films (Sn or Te) and alloyed Sn–Te compositions. For the ST0 film (pure Sn), the work function increased progressively with annealing. This enhancement likely stemmed from the growing presence of Sn oxides on the surface, as evidenced in Figures 4a and 5, and Table 3 [61]. In contrast, the work function of the ST100 film (pure Te) diminished as annealing temperature rose. This trend is plausibly attributed to thermal evaporation of Te at elevated temperatures, leading to exposure of the underlying Si substrate, which has a lower work function (Te: 4.95 eV, Si: 4.66 eV) [63,64]. For the intermediate ST10–ST80 compositions, the work function exhibited a two-phase response to annealing. From 298 K to 473 K, the work function declined, likely because Te atoms migrated into the bulk, enriching the surface in Sn. As the temperature increased further from 473 K to 873 K, the work function reversed course and rose. This upward trend is attributed to Te re-emerging at the surface and

concurrent oxidation processes, reflected in the rising relative concentrations of Te and O, as shown in Figure 5.

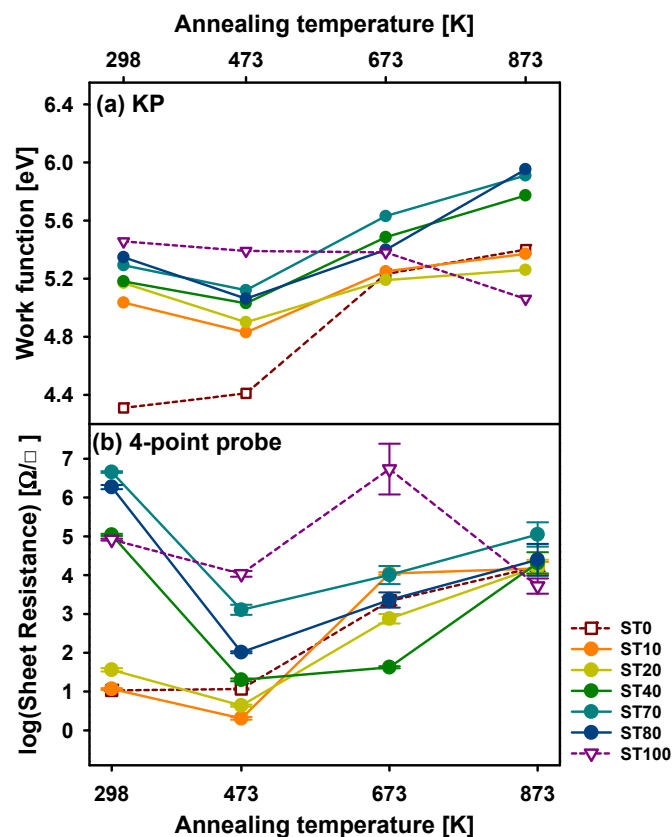


Figure 8. Work functions were measured by (a) KP, and electrical conductivity in the log scale was determined by (b) 4-point probe. Electrical conductivity values correspond to composite film + substrate behavior measured by the four-point probe method on p-Si, as no insulating-substrate control was employed. 298 K data are used as a reference [51].

Importantly, the variations in work function captured by KP were in good agreement with changes in electrical conductivity (log scale) measured using the 4-point probe, as shown in Figure 8b. This parallelism underscores the significant role of surface composition in governing the electronic properties of XSTY thin films. The observed conductivity behavior across the annealing series can be further understood by considering how stoichiometry, oxidation, and phase evolution influence carrier transport. At moderate annealing temperatures (≈ 473 K), improved crystallinity and the emergence of cubic SnTe domains within the Sn–Te–O matrix enhance charge percolation pathways and reduce defect scattering, leading to temporarily higher conductivity. However, at higher annealing temperatures (≥ 673 K), significant Te volatilization induces Sn-rich regions and facilitates oxidation of Sn to SnO_2 , as evidenced by the XPS and XRD analyses (Figures 4–6). The formation of SnO_2 and related oxide phases introduces deep trap states and widens the local valence band maximum (VBM) position, thereby reducing free carrier density and overall mobility. In addition, phase segregation observed in SEM micrographs and compositional inhomogeneity (Figure 3) create interfacial barriers between SnTe, SnO_2 , and residual Sn domains, further hindering charge transport.

Consequently, the non-monotonic conductivity trend observed with increasing annealing temperature arises from the interplay between structural improvement and oxidative degradation. While moderate annealing promotes grain coarsening and defect healing, excessive thermal exposure leads to carrier depletion and enhanced scattering at oxide

interfaces. This interpretation is consistent with the simultaneous shifts observed in the work function (Figure 8) and band structure evolution (Figure 9), collectively indicating that carrier concentration and mobility in XSTY thin films are highly sensitive to both Te stoichiometry and the progressive formation of oxide phases.

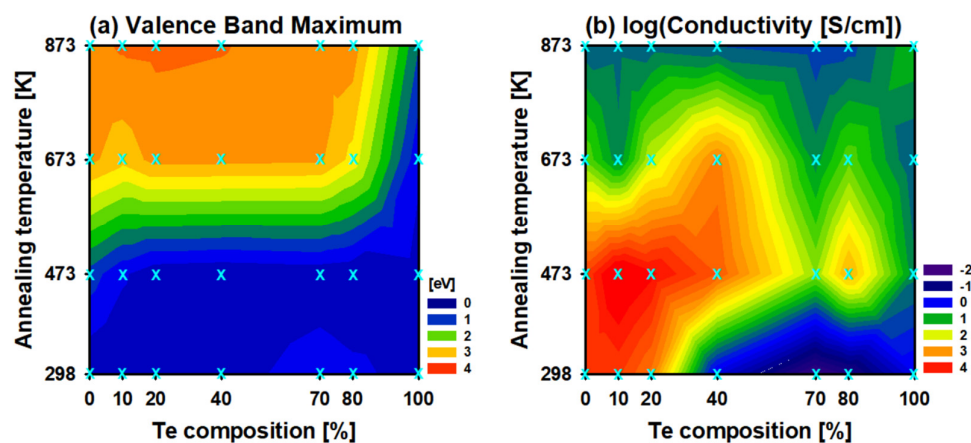


Figure 9. The contour plot of (a) VBM position energy obtained from XPS and (b) log scale value of conductivity measured by the four-point probe. Electrical conductivity values correspond to composite film + substrate behavior measured by the four-point probe method on p-Si, as no insulating-substrate control was employed. x marks show the film condition of the data obtained.

Figure 9a presents the energy band diagram of XSTY thin films, constructed using VBM values derived from high-resolution XPS measurements of the valence-band region. All spectra were energy-referenced to the adventitious carbon C 1s peak at 284.6 eV to ensure consistency across all samples. The VBM was determined by linearly extrapolating the leading edge of the valence-band density of states to the background baseline, following the methodology established in earlier works [41]. Representative valence-band spectra and corresponding fitting regions are provided in Figure S4, illustrating the extraction procedure and baseline range. All samples were internally referenced via the C 1s peak, and no additional cross-sample correction was required. The VB region exhibits two main features: a broad band centered near 7–8 eV attributed primarily to O 2p and Te 5p hybrid states, and a distinct shoulder at 3–5 eV arising from Sn 5s/5p–O 2p hybridization. The 3–5 eV Sn 5s/5p–O 2p shoulder becomes more pronounced as the Sn⁴⁺ fraction increases, consistent with reports that higher-valent Sn–O bonding enhances this feature in the VB region [65]. The relative intensity and broadening of this shoulder increase with annealing temperature, consistent with a higher proportion of Sn⁴⁺ species, in agreement with the XPS core-level analysis. Although the ST0 and other low-temperature samples exhibit a comparatively stronger 3–5 eV VB shoulder, this arises from the combination of (i) surface sensitivity of XPS, where thin native oxides dominate the VB signal at low T, and (ii) increased spectral broadening and reduced film continuity at high T (673–873 K), which suppress the apparent shoulder even as the Sn⁴⁺ fraction increases in the core levels. These effects explain the non-monotonic VB shoulder intensity without contradicting the Sn²⁺/Sn⁴⁺ evolution extracted from the high-resolution Sn 3d spectra. This correlation between VB shape and Sn oxidation states aligns with previous reports [46]. In parallel, Figure 9b displays the logarithmic electrical conductivity of the same set of thin films, as measured by the four-point probe technique. These conductivity trends closely mirrored the behavior observed in the work-function data obtained via Kelvin probe (Figure 8) and provided a complementary perspective to the XPS-derived VBM analysis. Together, the band-structure information and conductivity data reinforce the relationship between surface electronic structure and charge transport in the XSTY system.

4. Conclusions

In conclusion, this study employed complementary surface and structural characterization techniques to examine the influence of annealing temperature on the physicochemical properties of Sn–Te–O thin films. Across the dataset, reproducible temperature-dependent trends were observed. SEM and AFM analyses revealed that Te-rich films exhibited the greatest surface roughness near 473 K, suggesting enhanced morphological evolution under moderate thermal treatment. XPS measurements indicated a progressive oxidation of Sn species (from SnO to SnO₂) with increasing annealing temperature (298–873 K), consistent with a thermally driven oxidation sequence. Wettability tests showed that increasing Te content in as-deposited films (298 K) led to lower contact angles, reflecting composition-dependent changes in surface energy. Work-function measurements obtained by the Kelvin probe displayed consistent variation with annealing temperature, likely associated with the changing oxidation state and surface composition. Overall, these observations illustrate how air annealing influences oxidation, morphology, and electronic surface characteristics of Sn–Te–O thin films. Further in-depth analyses, such as angle-resolved XPS and measurements on insulating substrates, would help refine the mechanistic understanding of these transformations.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/surfaces8040083/s1>, Figure S1: XPS survey spectra of the different STX thin films; Figure S2: XPS narrow spectra of C1s before peak position correction; Figure S3: XPS narrow spectra of C1s after peak position correction; Figure S4: High-resolution valence-band spectra for all representative films, with the linear extrapolation fits used to determine VBM positions. The dominant O 2p/Te 5p manifold (~7–8 eV) and the Sn 5s/5p–O 2p hybridized shoulder (3–5 eV) are explicitly labeled. These assignments highlight how VB feature evolution correlates with changes in Sn oxidation states across the annealing series.; Figure S5: Sheet Resistance (log scale) vs. Annealing Temperature. The error bar represents the standard deviation; Figure S6: Full, unedited XRD scans (10–90° 2θ) for all XSTY thin films; Figure S7: Full, unedited XRD scans (10–90° 2θ) for all XSTY thin films. All substrate-derived Si reflections are labeled according to the ICDD/ICSD reference card (JCPDS 41-1111).; Table S1: Table outlining the FWHM values and standard deviation for each elemental state. SD values in Table S1 represent the fitting standard error for each peak component.; Table S2: Table outlining the SOS and RSF values; Table S3: Table outlining thickness-normalized resistivity values with error bars for the STY thin films. SD values in Table S3 represent the standard deviation obtained from 10 independent four-point probe measurements for each sample.

Author Contributions: Conceptualization, Y.-C.K. and J.P.; methodology, Y.-C.K. and H.-S.Y.; validation, Y.-C.K. and H.-S.Y.; formal analysis, Y.-C.K., H.-S.Y. and J.L.; investigation, Y.-C.K., H.-S.Y. and J.L.; data curation, H.-S.Y.; writing—original draft preparation, Y.-C.K.; writing—review and editing, Y.-C.K.; supervision, Y.-C.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by a Research Grant of Pukyong National University (2025).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data supporting the findings of this study, including raw XPS, XRD, AFM, and contact-angle measurements, are available from the corresponding author upon reasonable request.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Zhang, X.; Wang, Z.; Zou, B.; Brod, M.K.; Zhu, J.; Jia, T.; Tang, G.; Snyder, G.J.; Zhang, Y. Band Engineering SnTe via Trivalent Substitutions for Enhanced Thermoelectric Performance. *Chem. Mater.* **2021**, *33*, 9624–9637. [\[CrossRef\]](#)
2. Robinson, F.; Newbrook, D.W.; Curran, P.; de Groot, C.H.K.; Hardie, D.; Hector, A.L.; Huang, R.; Reid, G. Low Temperature CVD of Thermoelectric SnTe Thin Films from the Single Source Precursor, $[\text{Bu}_3\text{Sn}(\text{Te}^n\text{Bu})]$. *Dalton Trans.* **2021**, *50*, 998–1006. [\[CrossRef\]](#)
3. Fu, T.; Xin, J.; Zhu, T.; Shen, J.; Fang, T.; Zhao, X. Approaching the Minimum Lattice Thermal Conductivity of P-Type SnTe Thermoelectric Materials by Sb and Mg Alloying. *Sci. Bull.* **2019**, *64*, 1024–1030. [\[CrossRef\]](#) [\[PubMed\]](#)
4. Jiang, Q.; Hu, H.; Yang, J.; Xin, J.; Li, S.; Viola, G.; Yan, H. High Thermoelectric Performance in SnTe Nanocomposites with All-Scale Hierarchical Structures. *ACS Appl. Mater. Interfaces* **2020**, *12*, 23102–23109. [\[CrossRef\]](#) [\[PubMed\]](#)
5. Tang, J.; Gao, B.; Lin, S.; Li, J.; Chen, Z.; Xiong, F.; Li, W.; Chen, Y.; Pei, Y. Manipulation of Band Structure and Interstitial Defects for Improving Thermoelectric SnTe. *Adv. Funct. Mater.* **2018**, *28*, 1803586. [\[CrossRef\]](#)
6. Shenoy, U.S.; Bhat, D.K. Selective Co-Doping Improves the Thermoelectric Performance of SnTe: An Outcome of Electronic Structure Engineering. *J. Alloys Compd.* **2021**, *892*, 162221. [\[CrossRef\]](#)
7. Mahdy, I.A.; Mahmoud, E.A.; Mahdy, M.A. Tin Telluride Quantum Dot Thin Films: Size Dependent Structural, Optical and Electrical Properties. *Mater. Sci. Semicond. Process.* **2021**, *121*, 105398. [\[CrossRef\]](#)
8. Han, G.; Zhang, R.; Popuri, S.R.; Greer, H.F.; Reece, M.J.; Bos, J.G.; Zhou, W.; Knox, A.R.; Gregory, D.H. Large-Scale Surfactant-Free Synthesis of P-Type SnTe Nanoparticles for Thermoelectric Applications. *Materials* **2017**, *10*, 233. [\[CrossRef\]](#)
9. Giri, A.; Kumar, M.; Kim, J.; Pal, M.; Banerjee, W.; Nikam, R.D.; Kwak, J.; Kong, M.; Kim, S.H.; Thiyagarajan, K.; et al. Surface Diffusion and Epitaxial Self-Planarization for Wafer-Scale Single-Grain Metal Chalcogenide Thin Films. *Adv. Mater.* **2021**, *33*, 2102252. [\[CrossRef\]](#)
10. Li, W.; Zheng, L.; Ge, B.; Lin, S.; Zhang, X.; Chen, Z.; Chang, Y.; Pei, Y. Promoting SnTe as an Eco-Friendly Solution for p-PbTe Thermoelectric via Band Convergence and Interstitial Defects. *Adv. Mater.* **2017**, *29*, 1605887. [\[CrossRef\]](#)
11. Xu, S.; Zhu, W.; Zhang, L.; Zhang, Z.; Deng, Y. Enhanced Thermoelectric Performance of SnTe Film with Optimized Carrier Transport Induced by Facile Post-Annealing Process. *Mater. Lett.* **2018**, *221*, 12–14. [\[CrossRef\]](#)
12. Yin, D.; Dun, C.; Gao, X.; Liu, Y.; Zhang, X.; Carroll, D.L.; Swihart, M.T. Controllable Colloidal Synthesis of Tin (II) Chalcogenide Nanocrystals and Their Solution-Processed Flexible Thermoelectric Thin Films. *Small* **2018**, *14*, 1801949. [\[CrossRef\]](#)
13. Weng, Z.; Ma, S.; Zhu, H.; Ye, Z.; Shu, T.; Zhou, J.; Wu, W.; Wu, H. CdTe Thin Film Solar Cells with a SnTe Buffer Layer in Back Contact. *Sol. Energy Mater. Sol. Cells* **2018**, *179*, 276–282. [\[CrossRef\]](#)
14. de Kergommeaux, A.; Faure-Vincent, J.; Pron, A.; de Bettignies, R.; Malaman, B.; Reiss, P. Surface Oxidation of Tin Chalcogenide Nanocrystals Revealed by ^{119}Sn -Mössbauer Spectroscopy. *J. Am. Chem. Soc.* **2012**, *134*, 11659–11666. [\[CrossRef\]](#) [\[PubMed\]](#)
15. Yin, Y.; Alivisatos, A.P. Colloidal Nanocrystal Synthesis and the Organic-Inorganic Interface. *Nature* **2005**, *437*, 664–670. [\[CrossRef\]](#) [\[PubMed\]](#)
16. Meng, Z.; Stolz, R.M.; Mendecki, L.; Mirica, K.A. Electrically-Transduced Chemical Sensors Based on Two-Dimensional Nanomaterials. *Chem. Rev.* **2019**, *119*, 478–598. [\[CrossRef\]](#) [\[PubMed\]](#)
17. Ahmet, I.Y.; Hill, M.S.; Raithby, P.R.; Johnson, A.L. Tin Guanidinato Complexes: Oxidative Control of Sn, SnS, SnSe and SnTe Thin Film Deposition. *Dalton Trans.* **2018**, *47*, 5031–5048. [\[CrossRef\]](#)
18. Tanwar, P.; Panwar, A.K.; Singh, S.; Srivastava, A.K. Microstructural and Optical Properties Investigation of Variable Thickness of Tin Telluride Thin Films. *Thin Solid Films* **2020**, *693*, 137708. [\[CrossRef\]](#)
19. Lee, Y.K.; Park, E.S.; Yoo, C.; Kim, W.; Jeon, J.W.; Ha, M.; Hwang, C.S. Atomic Layer Deposition of SnTe Thin Film Using $\text{Sn}(\text{N}(\text{CH}_3)_2)_4$ and $\text{Te}(\text{Si}(\text{CH}_3)_3)_2$ with Ammonia Coinjection. *Cryst. Growth Des.* **2020**, *20*, 4649–4656. [\[CrossRef\]](#)
20. Zou, K.; Albright, S.D.; Dagdeviren, O.E.; Morales-Acosta, M.D.; Simon, G.H.; Zhou, C.; Mandal, S.; Ismail-Beigi, S.; Schwarz, U.D.; Altman, E.I.; et al. Revealing Surface-State Transport in Ultrathin Topological Crystalline Insulator SnTe Films. *APL Mater.* **2019**, *7*, 051106. [\[CrossRef\]](#)
21. Şişman, İ.; Öz, H. Preparation of SnTe Thin Films on Au(111) by Electrodeposition Route. *Electrochim. Acta* **2011**, *56*, 4889–4894. [\[CrossRef\]](#)
22. Wang, Y.Y.; Cai, K.F.; Yao, X. Facile Synthesis and Characterization of SnTe Films. *Appl. Surf. Sci.* **2011**, *258*, 919–922. [\[CrossRef\]](#)
23. Klett, R.; Schönle, J.; Becker, A.; Dyck, D.; Borisov, K.; Rott, K.; Ramermann, D.; Büker, B.; Haskenhoff, J.; Krieft, J.; et al. Proximity-Induced Superconductivity and Quantum Interference in Topological Crystalline Insulator SnTe Thin-Film Devices. *Nano Lett.* **2018**, *18*, 1264–1268. [\[CrossRef\]](#)
24. Lelis, M.; Tuckute, S.; Varnagiris, S.; Marius, U.; Giedrius, L.; Bockute, K. Tailoring of TiO_2 film microstructure by pulsed-DC and RF magnetron co-sputtering. *Surf. Coat. Technol.* **2019**, *377*, 124906. [\[CrossRef\]](#)
25. Cattaruzza, E.; Battaglin, G.; Canton, P.; de Julian Fernandez, C.; Ferroni, M.; Gonella, F.; Maurizio, C.; Riello, P.; Sada, C.; Sangregorio, C.; et al. Radiofrequency magnetron co-sputtering deposition synthesis of Co-based nanocomposite glasses for optical and magnetic applications. *Appl. Surf. Sci.* **2004**, *226*, 62–67. [\[CrossRef\]](#)

26. Borowski, P.; Mysliwiec, J. Recent advances in magnetron sputtering: From fundamentals to industrial applications. *Coatings* **2025**, *15*, 922. [\[CrossRef\]](#)
27. Lee, S.G.; Ahn, J.R.; Kim, Y.; Moon, S.H.; Lee, K.W.; Kim, I.S.; Park, Y.K. Properties of MgB₂ thin films made by radio frequency magnetron co-sputtering. *Supercond. Sci. Technol.* **2003**, *16*, 1550. [\[CrossRef\]](#)
28. Chen, S.C.; Wang, C.H.; Sun, H.; Wen, C.K.; Lu, C.F.; Tsai, C.L.; Fu, Y.K.; Chuang, T.H. Microstructures, electrical and magnetic properties of (Ga, Co)-ZnO films by radio frequency magnetron co-sputtering. *Surf. Coat. Technol.* **2016**, *303*, 203–208. [\[CrossRef\]](#)
29. Ponce-Mosso, M.; Pérez-González, M.; García-Tinoco, P.E.; Crotte-Ledesma, H.; Morales-Luna, M.; Tomás, S.A. Enhanced Photocatalytic Activity of Amorphous MoO₃ Thin Films Deposited by rf Reactive Magnetron Sputtering. *Catal. Today* **2020**, *349*, 150–158. [\[CrossRef\]](#)
30. Wang, G.; Zhang, Y.; Lotnyk, A.; Shi, H.; Chen, C. High Thermoelectric Performance in ZnSb-SnTe Pseudo-Binary Materials. *Scr. Mater.* **2021**, *194*, 113670. [\[CrossRef\]](#)
31. Saghir, M.; Walker, M.; McConville, C.F.; Balakrishnan, G. SnTe Microcrystals: Surface Cleaning of a Topological Crystalline Insulator. *Appl. Phys. Lett.* **2016**, *108*, 061602. [\[CrossRef\]](#)
32. Yang, M.; Zhang, W.; Su, D.; Wen, J.; Liu, L.; Wang, X. Flexible SnTe/Carbon Nanofiber Membrane as a Free-Standing Anode for High-Performance Lithium-Ion and Sodium-Ion Batteries. *J. Colloid Interface Sci.* **2022**, *605*, 231–240. [\[CrossRef\]](#)
33. Cox, J.D.; Wagman, D.D.; Medvedev, V.A. *CODATA Key Values for Thermodynamics*; Hemisphere Publishing Corp.: New York, NY, USA, 1989.
34. Bettini, M.; Richter, H.J. Oxidation in air and thermal desorption on PbTe, SnTe, and Pb_{0.8}Sn_{0.2}Te surfaces. *Surf. Sci.* **1979**, *80*, 334–343. [\[CrossRef\]](#)
35. Berchenko, N.; Trzyna, M.; Wojnarowska-Nowak, R.; Szczerbakow, A.; Badyła, A.; Cebulski, J.; Story, T. Surface oxidation of SnTe topological crystalline insulator. *Appl. Surf. Sci.* **2018**, *452*, 134–140. [\[CrossRef\]](#)
36. Wortman, M.; Bednarz, B.; Nezafat, N.B.; Viertel, K.; Kuschel, O.; Schmalhorst, J.; Ennen, I.; Garner, M.; Frese, N.; Jakob, G.; et al. Oxidation state depth profiling by self-consistent fitting of all emission peaks in the X-ray photoelectron spectrum of SnTe. *Appl. Surf. Sci.* **2025**, *713*, 164356. [\[CrossRef\]](#)
37. Choi, J.; Park, J.; Kang, J.; Kehayias, C.E.; Oh, J.W.; Kang, Y.C. Annealing Temperature Effect on the Surface Properties of the MoSe Thin Films. *Phys. Status Solidi A* **2023**, *220*, 2300477. [\[CrossRef\]](#)
38. Mun, J.; Han, S.; Yoon, H.S.; Kang, J.; Jonas, O.; Park, J.; Kang, Y.C. Etching and Compositional Ratio Effect on the Surface Properties of Bismuth Telluride Thin Films. *Surfaces* **2024**, *7*, 181–195. [\[CrossRef\]](#)
39. Lee, J.; Kim, D.; Park, J.; Kang, J.; Kehayias, C.E.; Johnson, A.T.C.; Kang, Y.C. Investigation on the Surface Properties of Annealed Copper Selenide Thin Films with Various Compositional Ratios. *Phys. Status Solidi A* **2025**, *222*, 2400618. [\[CrossRef\]](#)
40. Jeong, C.; Mun, J.; Park, J.; Kang, J.; Kehayias, C.E.; Johnson, A.T.C.; Kang, Y.C. Surface Properties of Copper Oxytelluride Thin Films with Different Composition Ratios Fabricated by RF Magnetron Co-Sputtering. *Physica B Condens. Matter* **2025**, *701*, 416963. [\[CrossRef\]](#)
41. Choi, S.; Kang, J.; Park, J.; Kang, Y.C. Tin Nitride Thin Films Fabricated by Reactive Radio Frequency Magnetron Sputtering at Various Nitrogen Gas Ratios. *Thin Solid Films* **2014**, *571*, 84–89. [\[CrossRef\]](#)
42. Choi, S.; Park, J.; Kang, J.; Johnson, A.T.C.; Kang, Y.C. Surface Characterization of PZT Thin Films Obtained at Various O₂ Gas Ratios. *Vacuum* **2016**, *128*, 234–239. [\[CrossRef\]](#)
43. Biesinger, M.C.; Payne, B.P.; Lau, L.W.M.; Gerson, A.; Smart, R.S.C. X-ray Photoelectron Spectroscopy Reference Values for Transition Metal Oxides and Hydroxides: Mixed Oxide Systems. *Appl. Surf. Sci.* **2011**, *257*, 2717–2730. [\[CrossRef\]](#)
44. Cordfunke, E.H.P.; Ouweltjes, W.; Prins, G. Standard Enthalpies of Formation of Tellurium Compounds I. Tellurium Dioxide. *J. Chem. Thermodyn.* **1987**, *19*, 369–375. [\[CrossRef\]](#)
45. Nguyen, T.T.N.; Sasaki, N.; Tsutsumi, T.; Ishikawa, K.; Hori, M. Formation of Spherical Sn Particles by Reducing SnO₂ film in Floating Wire-Assisted H₂/Ar Plasma at Atmospheric Pressure. *Sci. Rep.* **2020**, *10*, 17770.
46. Dziawa, P.; Kowalski, B.J.; Dybko, K.; Buczek, R.; Szczerbakow, A.; Szot, M.; Łusakowska, E.; Balasubramanian, T.; Wojek, B.M.; Berntsen, M.H.; et al. Topological crystalline insulator states in Pb_{1-x}Sn_xSe. *Nat. Mater.* **2013**, *11*, 1023–1027. [\[CrossRef\]](#)
47. Zhang, G.; Qin, G.; Zhang, F. Effects of Internal Relaxation of Biaxial Strain on Structural and Electronic Properties of In_{0.5}Al_{0.5}N Thin Film. *Coatings* **2022**, *12*, 598. [\[CrossRef\]](#)
48. Abadias, G.; Chason, E.; Keckes, J.; Sebastiani, M.; Thompson, G.B.; Barthel, E.; Doll, G.L.; Murray, C.E.; Stoessel, C.H.; Martinu, L. Stress in thin films and coatings: Current status, challenges, and prospects. *J. Vac. Sci. Technol. A* **2018**, *36*, 020801. [\[CrossRef\]](#)
49. Xu, C.; Xu, G.; Liu, Y.; Zhao, X.; Wang, G. Preparation and Characterization of SnO₂ Nanorods by Thermal Decomposition of SnC₂O₄ Precursor. *Scr. Mater.* **2002**, *46*, 789–794. [\[CrossRef\]](#)
50. Lakshminarayana, G.; Baki, S.O.; Lira, A.; Sayyed, M.I.; Kityk, I.V.; Halimah, M.K.; Mahdi, M.A. X-ray Photoelectron Spectroscopy (XPS) and Radiation Shielding Parameters Investigations for Zinc Molybdenum Borotellurite Glasses Containing Different Network Modifiers. *J. Mater. Sci.* **2017**, *52*, 7394–7414. [\[CrossRef\]](#)

51. Yoon, H.S.; Lee, J.; Park, J.; Oh, J.W.; Kang, Y.C. Physicochemical Properties of SnTe Thin Films Dependent on Compositional Ratios. *Phys. Status Solidi A* **2022**, *219*, 2200059. [\[CrossRef\]](#)
52. Quackenbush, N.F.; Allen, J.P.; Scanlon, D.O.; Sallis, S.; Hewlett, J.A.; Nandur, A.S.; Chen, B.; Smith, K.E.; Weiland, C.; Fischer, D.A.; et al. Origin of the Biopolar Doping Behavior of SnO from X-ray Spectroscopy and Density Functional Theory. *Chem. Mater.* **2013**, *25*, 3114–3123. [\[CrossRef\]](#)
53. Hanyš, P.; Janeček, P.; Matolín, V.; Korotcenkov, G.; Nehasil, V. XPS and TPD study of Rh/SnO₂ System-Reversible Process of Substrate Oxidation and Reduction. *Surf. Sci.* **2006**, *600*, 4233–4238. [\[CrossRef\]](#)
54. Neudachina, V.S.; Shatalova, T.B.; Shtanov, V.I.; Yashina, L.V.; Zyubina, T.S.; Tamm, M.E.; Kobeleva, S.P. XPS Study of SnTe(100) Oxidation by Molecular Oxygen. *Surf. Sci.* **2005**, *584*, 77–82. [\[CrossRef\]](#)
55. Sen, S.; Muthe, K.P.; Joshi, N.; Gadkari, S.C.; Gupta, S.K.; Roy, J.M.; Deshpande, S.K.; Yakhmi, J.V. Room Temperature Operating Ammonia Sensor Based on Tellurium Thin Films. *Sens. Actuators B Chem.* **2004**, *98*, 154–159. [\[CrossRef\]](#)
56. Maslakov, K.I.; Teterin, Y.A.; Popel, A.J.; Teterin, A.Y.; Ivanov, K.E.; Kalmykov, S.N.; Petrov, V.G.; Petrov, P.K.; Farnan, I. XPS Study of Ion Irradiated and Unirradiated CeO₂ Bulk and Thin Film Samples. *Appl. Surf. Sci.* **2018**, *448*, 154–162. [\[CrossRef\]](#)
57. Campbell, K.A.; Anderson, C.M. Phase-Change Memory Devices with Stacked Ge-Chalcogenide/Sn-Chalcogenide Layers. *Microelectron. J.* **2007**, *38*, 52–59. [\[CrossRef\]](#)
58. Owens, D.K.; Wendt, R.C. Estimation of the Surface Free Energy of Polymers. *J. Appl. Polym. Sci.* **1969**, *13*, 1741. [\[CrossRef\]](#)
59. Montes Ruiz-Cabello, F.J.; Bermúdez-Romero, S.; Ibáñez-Ibáñez, P.F.; Cabrerizo-Vílchez, M.A.; Rodríguez-Valverde, M.A. Freezing Delay of Sessile Drops: Probing the Impact of Contact Angle, Surface Roughness and Thermal Conductivity. *Appl. Surf. Sci.* **2021**, *537*, 147964. [\[CrossRef\]](#)
60. Bhushan, B.; Nosonovsky, M. The Rose Petal Effect and the Modes of Superhydrophobicity. *Philos. Trans. R. Soc. A* **2010**, *368*, 4713–4728. [\[CrossRef\]](#)
61. Hu, L.; Zhang, L.; Wang, D.; Lin, X.; Chen, Y. Fabrication of Biomimetic Superhydrophobic Surface Based on Nanosecond Laser-Treated Titanium Alloy Surface and Organic Polysilazane Composite Coating. *Colloid Surf. A* **2018**, *555*, 515–524. [\[CrossRef\]](#)
62. Lv, H.; Wu, C.; Tang, J.; Du, H.; Qin, F.; Peng, H.; Yan, M. Two-Dimensional SnO/SnO₂ Heterojunctions for Electromagnetic Wave Absorption. *Chem. Eng. J.* **2021**, *411*, 128445. [\[CrossRef\]](#)
63. Yuan, Z.; Zhao, Q.; Xie, C.; Liang, J.; Duan, X.; Duan, Z.; Li, S.; Jiang, Y.; Tai, H. Gold-Loaded Tellurium Nanobelts Gas Sensor for PPT-Level NO₂ Detection at Room Temperature. *Sens. Actuators B Chem.* **2022**, *355*, 131300. [\[CrossRef\]](#)
64. Dillon, J.A.; Farnsworth, H.E. Work Function and Sorption Properties of Silicon Crystals. *J. Appl. Phys.* **1958**, *28*, 1195. [\[CrossRef\]](#)
65. Berg, U.; Chassé, T.; Brümmer, O. Investigation of the XPS Valence Band Structure from Sn Chalcogenides. *Phys. Status Solidi B* **1981**, *108*, 507–510. [\[CrossRef\]](#)

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.