

THERMAL TREATMENT-DRIVEN MODULATION OF OPTICAL PROPERTIES IN ANTIMONY-BASED CHALCOGENIDE GLASSES

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ABSTRACT

Antimony-containing chalcogenide glasses sit at an awkward and productive boundary. They are stable enough to be shaped into films, fibres and waveguides, yet their metastable networks respond strongly to heat. This review examines how thermal treatment modulates the optical properties of Sb-based chalcogenide glasses and thin films, drawing on work published mainly between 1990 and 2025 on Sb–S, Sb–Se, Ge–Sb–Se, Ge–Sb–S and related systems. A structured literature search, explicit inclusion criteria and directional vote-counting were used to combine results that differ in composition, film thickness, annealing atmosphere and measurement technique. The synthesis shows a consistent pattern. Annealing below the glass transition relaxes the network, removes homopolar bonds and usually raises the refractive index while shifting the absorption edge to lower energy. Annealing near or above crystallization onset produces a larger, step-like change in both quantities as nanocrystalline Sb₂S₃ or Sb₂Se₃ domains nucleate, accompanied by scattering losses and a wider Urbach tail. The size of the shift scales with the Sb fraction, the distance of the as-prepared state from equilibrium, and the average coordination number. Reported index contrasts between amorphous and crystalline states reach values useful for phase-change photonics, but the evidence base is uneven. Annealing times, ramp rates and atmospheres are rarely controlled or reported in a comparable way, and band gaps are extracted with differing Tauc conventions. We identify these gaps, propose a minimum reporting standard, and outline directions for composition-aware thermal engineering of optical function.

Keywords: *Antimony chalcogenide glasses¹; Thermal annealing²; Optical band gap³; Refractive index⁴; Structural relaxation⁵; Crystallization⁶; Phase-change photonics⁷*

1. INTRODUCTION

1.1 Antimony in the Chalcogenide Glass Family

Chalcogenide glasses, built on sulfur, selenium or tellurium, transmit well beyond the range where oxide glasses fail. They have been developed for infrared lenses, fibres, waveguides and nonlinear devices for several decades [1]–[3]. Arsenic- and germanium-based compositions dominated early practice, partly because arsenic sulfide and selenide form glass readily and have been characterised exhaustively [4], [5]. Antimony has a different history. Sb_2S_3 and Sb_2Se_3 are poor glass formers on their own and usually need a network former such as Ge or As, or an excess of chalcogen, to avoid crystallising during quenching [6]. Yet replacing As with Sb often gives a higher refractive index and a smaller band gap, and it removes a toxic element from the batch. Those traits explain the steady interest in Ge–Sb–Se and Ge–Sb–S glasses for mid-infrared optics, where fibre and planar-waveguide demonstrations are well documented [7], [8]. There is a second reason antimony matters. The binary compounds are useful in their own right, as thin-film absorbers and as low-loss phase-change media. In both roles the amorphous-to-crystalline boundary is the point of interest. The response to heat is not a nuisance here. It is the property being designed.

1.2 Why Thermal History Controls Optical Response

A glass is a frozen snapshot, and its optical constants record the conditions under which the snapshot was taken. Zachariasen's random-network picture [9] explains why such a solid can exist without long-range order. Constraint-counting ideas due to Phillips and Thorpe [10], [11] explain why its rigidity, and therefore its stability, changes with mean coordination number. Films made by evaporation or sputtering are quenched far faster than any melt, so they retain homopolar bonds, undercoordinated atoms and excess free volume. Charged defect pairs in lone-pair semiconductors, described by the valence-alternation model [12], add localised states near the band edges. Heating lets this structure move. Below the glass transition the changes are gradual relaxation and bond rearrangement, in the family of metastable effects reviewed by Shimakawa and co-workers [13]. Close to the transition the kinetics become strongly dependent on heating rate [14]. Each structural change has an optical signature in the band gap, the refractive index, the sub-gap absorption tail and the dispersion. Heat treatment is therefore a post-deposition tool, and sometimes a hazard.

1.3 Scope and Objectives of This Review

Interest in phase-change materials for data storage [15], [16] and, more recently, for non-volatile photonics [17], [18] has pushed Sb-based chalcogenides into the spotlight. Interest from thin-film photovoltaics [19]–[21] has added a parallel body of work in which annealing is used to grow crystalline absorbers. The two communities rarely cite each other, and measurements are rarely comparable. This review gathers that scattered evidence under one question: how, and by how much, does thermal treatment change the optical properties of Sb-based chalcogenide glasses? The objectives are fourfold. We collate reported trends across compositions and annealing regimes. We estimate the direction and approximate size of the effects. We assess the methodological quality of the underlying studies. And we propose a reporting standard that would make future results

combinable. The review is organised as a survey of past work, a description of the synthesis method, a critical analysis of that work, a discussion of implications, and a conclusion.

2. SURVEY OF PAST WORK

The glass-forming ability of antimony compounds is the natural starting point. Ternary Ge–Sb–Se and Ge–Sb–S systems form glass over broad chalcogen-rich regions, but the region narrows as the Sb fraction rises, and the binary Sb–chalcogen compounds crystallise too readily to be quenched into bulk glass [6]. The glass transition temperature in these systems tends to rise with the average coordination number, which fits constraint theory [10], [11]. Four-fold Ge strengthens the network more than three-fold Sb, so Sb-for-Ge substitution generally softens it. Across reported compositions, transition temperatures fall roughly between 400 and 600 K, and the observed value shifts upward with heating rate in the way described by the classic analysis of Moynihan and colleagues [14]. Crystallization kinetics are usually extracted from the shift of the exothermic peak with heating rate using the Kissinger approach [28], while the gap between crystallization onset and glass transition serves as a crude index of stability against devitrification [29]. Such numbers decide how wide the processing window is for any annealing step.

The as-prepared state matters as much as composition. Melt-quenched bulk glass has already been through a slow, controlled cooling and sits relatively close to equilibrium. Thermally evaporated or sputtered films have not. They contain molecular fragments, chain-like selenium or sulfur segments, Sb–Sb and chalcogen–chalcogen homopolar bonds, and voids, all consistent with the defect picture of lone-pair semiconductors [3], [5], [12]. As a result, films respond to heat far more visibly than bulk samples of the same nominal composition, and many of the strongest effects in the literature come from films a few hundred nanometres thick.

How the optical response is measured shapes what is reported. Most studies infer an optical gap from transmission or absorption spectra using the Tauc construction [22] or the closely related Davis–Mott treatment [23], plotting a power of the product of absorption coefficient and photon energy against photon energy. The choice of exponent, and of the fitting window, changes the extracted gap by several hundredths of an electron-volt, a difference as large as many annealing effects. The sub-gap tail is quantified by an Urbach energy [24], interpreted as a measure of structural and thermal disorder. Refractive index and thickness are commonly obtained from interference fringes by the envelope method of Swanepoel [25], which works well for uniform films with clear fringes but degrades for thin, rough or scattering samples. Spectroscopic ellipsometry avoids some of these limits and is increasingly used, particularly in the phase-change photonics literature.

When films are annealed below the glass transition, the reported trend is consistent across many Ge–Sb–Se studies. The film shrinks slightly, indicating densification. Homopolar bonds convert into heteropolar ones, the refractive index rises, the optical gap narrows modestly, and the Urbach energy falls as the network settles. These changes follow the general behaviour of metastable structural change in amorphous chalcogenides [13]. Exceptions exist. Several sulfur-rich films show a widening gap after air annealing, which the authors attribute to surface oxidation or loss of volatile species, not to network relaxation. The distinction between intrinsic and extrinsic effects is rarely tested by repeating the anneal in vacuum or inert gas.

At or beyond the crystallization onset, the response is of a different order. Amorphous Sb_2S_3 has a gap near 2 eV, and the crystalline form sits closer to 1.7 eV, with a substantial rise in refractive index across the near-infrared [18]. Delaney and colleagues showed that both Sb_2S_3 and Sb_2Se_3 combine a large index contrast with very low loss at telecom wavelengths, which is exactly the property that Ge–Sb–Te alloys lack [17]. The older phase-change literature on Ge–Sb–Te established the logic of optical contrast between structural states but accepted high absorption as the price [15], [16]. Sb_2Se_3 research from the photovoltaic side adds another layer. The one-dimensional ribbon structure of the crystal makes grain orientation critical to carrier transport [19], and vapour transport deposition produced devices with efficiencies around 7.6% [20]. Reviews of the field treat annealing as the step that sets crystal quality [21]. Together these studies show that crystallization is a controllable process, but also that grain size, orientation and residual amorphous fraction all enter the optical response.

Relationships between optical quantities are frequently used to interpret the data. The single-oscillator model of Wemple and DiDomenico links refractive-index dispersion to an oscillator energy and a dispersion energy [26], and empirical rules in the lineage of Moss relate refractive index to the band gap [27]. In annealed Sb-bearing films, the index typically rises as the gap narrows, which is qualitatively consistent with such rules, though the quantitative fit is often poor. Density change and polarisability change act on the index alongside the gap, so the gap alone cannot predict the index.

Application studies close the loop. Annealing is used to trim the effective index of waveguides after fabrication, to stabilise films against further drift, and to switch between states in reconfigurable photonic circuits built on broadband low-loss phase-change materials [30]. In each case a reliable calibration curve of optical constants against annealing temperature and time is the real requirement. This is where the surveyed literature is thinnest, and it motivates the critical analysis that follows.

3. METHODOLOGY

The review followed a structured narrative approach with elements of systematic review. Searches covered Scopus, Web of Science, IEEE Xplore, ScienceDirect and Google Scholar for studies published from 1990 to 2025. Search strings combined antimony-containing system names (Sb–S, Sb–Se, Ge–Sb–S, Ge–Sb–Se, As–Sb–Se) with terms for annealing, heat treatment, crystallization, refractive index, optical band gap and Urbach energy. Backward and forward citation chasing from key papers and reviews supplemented the database searches. Studies were retained if they dealt with bulk glass or thin films containing antimony as a principal constituent, reported at least one optical quantity before and after a defined thermal treatment, and stated the annealing temperature. Conference abstracts without data, studies on doped systems where Sb was a minor additive, and papers in which the optical effect could not be separated from irradiation were excluded.

For every included study, the following variables were extracted: composition and sample form, deposition method, film thickness, annealing temperature, duration, ramp rate and atmosphere, the glass transition and crystallization temperatures where given, the optical gap and the method used to obtain it, refractive index and its wavelength, Urbach energy, and any structural evidence from X-ray diffraction or Raman spectroscopy. To compare studies with different compositions, annealing temperatures were normalised by the glass transition

temperature of the same material (reduced temperature), and optical changes were expressed as relative differences from the as-prepared value. Each result was assigned to one of three regimes, namely sub-transition relaxation, supercooled-liquid region, and post-crystallization, according to the reported thermal and structural data.

Because most studies do not report standard errors or sample sizes, a conventional random-effects meta-analysis of effect sizes was not defensible. Synthesis therefore relied on directional vote counting within each regime, supplemented by comparison of approximate magnitudes of relative change, with attention to how the figures moved with Sb fraction and mean coordination number. Heterogeneity was assessed qualitatively by examining differences in method and sample form. A simple quality checklist scored each study on whether annealing atmosphere, duration and ramp rate were reported, whether the gap-extraction method was specified, whether replicate samples were mentioned, and whether structural evidence supported the interpretation. Sensitivity checks asked whether conclusions changed when low-scoring studies were removed, and whether trends differed between films and bulk glass.

4. CRITICAL ANALYSIS OF PAST WORK

The first and most basic weakness is that annealing protocols are underspecified. Many papers state a temperature and a duration but omit the heating and cooling rates, which matter because the structure frozen in on cooling depends on cooling rate. Atmosphere is often unspecified. This matters because antimony chalcogenides can oxidise at moderate temperatures, producing antimony oxide at the surface that changes transmission and apparent band gap. Without an inert-atmosphere control, a widening gap in a sulfur-rich film cannot be assigned confidently to intrinsic structural change.

Second, the extraction of optical gaps is inconsistent. The Tauc exponent is chosen to give the best linear region, not on independent grounds, and the fitting window is rarely stated. Because annealing effects on the gap in the relaxation regime are often only a few hundredths of an electron-volt, the choice of exponent can be as large as the effect under study. The same applies to refractive index from the envelope method, which assumes a uniform, non-scattering film. After partial crystallization, scattering contributes to the apparent absorption and distorts fringe amplitudes, so values computed in that regime carry more uncertainty than their tidy tables suggest. Studies that combine ellipsometry with transmission are better placed here, and they remain a minority.

Third, replicate measurements and error estimates are scarce. A trend in a single film is a trend in one deposition run, and film-to-film variation in thickness, composition and residual stress is not captured. Because compositional drift during evaporation is a known problem for multicomponent chalcogenides, a change attributed to annealing may partly reflect a difference between samples. Few papers verify composition before and after the anneal, even though Se or S loss at higher temperatures is plausible.

Fourth, the interpretation of structural change is often qualitative. Raman and X-ray diffraction are used to show that crystallinity appears or that homopolar bonds decrease, but quantitative links between a specific structural metric and an optical quantity are seldom attempted. When correlations are shown, they typically have too few points to separate competing explanations, such as relaxation versus density change versus phase separation.

Fifth, time dependence is neglected. Relaxation is a kinetic process, and a single two-hour anneal at one temperature says nothing about whether the property has reached a steady value. Treating time and temperature as interchangeable, as some studies implicitly do, is not justified for systems as far from equilibrium as evaporated films. Closely related is the lack of ageing data. Whether an annealed state remains stable over months at room temperature or under operating light flux is crucial for devices and is almost never tested.

Sixth, the glass transition temperature used for normalisation is generally measured on bulk powder, while the optical measurements are made on films, whose effective transition can differ because of substrate constraint, thickness and different thermal history. Using bulk values as a stand-in for film behaviour introduces a systematic error into any comparison based on reduced temperature, including the one used in this review.

Seventh, the literature is skewed. Selenides and Ge-containing compositions are far better represented than sulfides or tellurides, and positive, dramatic results, such as large index contrasts on crystallization, are more likely to be published and highlighted than small relaxation effects. Phase-change research focuses on the binary compounds and the photovoltaic work on crystalline Sb_2Se_3 , so the amorphous ternary glasses between these communities receive the least systematic attention. A conclusion drawn from this literature about the sub-transition regime therefore rests on a thinner evidence base than one about crystallization.

Finally, the review itself shares some of these limits. Vote counting discards magnitude information, normalisation by a bulk transition temperature is approximate, and the search was restricted to English-language publications. These constraints argue for treating the quantitative statements as indicative ranges and not as fitted parameters.

5. DISCUSSION

Read together, the studies support a three-regime picture. Below the glass transition, the dominant process is relaxation. Densification, conversion of homopolar to heteropolar bonds and narrowing of the tail states move the refractive index up and the band gap down by modest amounts, and the Urbach energy falls. In the supercooled-liquid region between the glass transition and the crystallization onset, the same processes accelerate and phase separation may begin, so the changes grow and become more sensitive to time. Past the crystallization onset the picture changes qualitatively. Nanocrystalline Sb_2S_3 or Sb_2Se_3 domains form, the index rises sharply, the gap drops toward the crystalline value, and scattering and tail broadening raise the loss. The practical consequence is that the useful annealing window for fine, reversible-in-direction tuning is below the crystallization onset, whereas large, switch-like contrast requires crossing it.

The trade-off between contrast and loss is the central design tension. For passive photonic trimming, a few percent change in index with negligible added loss is ideal, and the sub-transition regime delivers it. For reconfigurable photonics, crystallization offers a far larger contrast, and wide-gap Sb_2S_3 and Sb_2Se_3 keep the loss low enough in the near-infrared to be useful [17], [18], [30]. For photovoltaic absorbers, the goal is the opposite, namely complete, well-oriented crystallization, and the optical constants are a means to that end [19]–[21]. The same material and the same heat treatment can therefore be a defect or a feature depending on the application.

The relationships between optical quantities deserve caution. Refractive index and band gap move in the expected opposite directions on annealing, but empirical gap–index rules [27] capture only part of the change, and the single-oscillator parameters [26] shift as well. This indicates that density and polarisability changes contribute independently of the gap. Conclusions that treat the gap as a proxy for index will therefore fail when the film densifies or when partial crystallization adds scattering.

Several conditions would overturn these conclusions. If the oxidation of Sb-bearing films in air is more rapid than assumed, some reported trends are artefacts. If bulk and film transition temperatures differ substantially, reduced-temperature comparisons shift. If time dependence is strong and unreported, apparent differences between studies may reflect annealing duration alone. The proposed minimum reporting standard addresses these directly. Studies should state atmosphere, heating and cooling rate, hold time, composition before and after annealing, film-specific transition temperature where possible, the Tauc exponent and fitting range, and the number of independent samples.

Open questions remain. What is the time–temperature equivalence for relaxation in Sb-containing films, and does it follow the same activation energy as the glass transition? Can composition be tuned so that the sub-transition response is large but the crystallization onset is far above operating temperature? And how stable are annealed states under optical flux in devices? Answering these needs systematic experiments on controlled compositions, not further single-condition reports.

6. CONCLUSION

Thermal treatment modulates the optical properties of antimony-based chalcogenide glasses in a pattern that is consistent enough to be useful and incomplete enough to be frustrating. Relaxation below the glass transition shifts the absorption edge to lower energy, raises the refractive index modestly and narrows the tail states. Crossing into the crystallization regime produces a much larger change, with a drop in gap and a rise in index toward crystalline Sb_2S_3 or Sb_2Se_3 values, at the cost of scattering and wider tails. The size of these effects depends on the Sb fraction, the coordination number and how far from equilibrium the as-prepared film sits.

The principal limitation of the current literature is comparability. Missing heating rates, unspecified atmospheres, inconsistent Tauc fits, scarce replicates and absent time-dependence data prevent a quantitative meta-analysis, and force reliance on directional evidence. A minimum reporting standard, together with in-situ ellipsometry, composition checks before and after annealing, and time-resolved relaxation studies, would turn scattered observations into predictive calibration curves. Such curves are what waveguide trimming, reconfigurable photonics and thin-film absorber processing actually need. Until they exist, annealing of Sb-based chalcogenides will remain more craft than engineering.

7. REFERENCES

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