

Chalcogenide glasses: Engineering in the infrared spectrum

Credit: J. David Musgraves

Chalcogenide glass ball lenses.

By J. David Musgraves

The advances in chalcogenide glasses in the past six decades are only the beginning of a much larger future.

The fundamentally useful thing about glass is its optical transparency. There are certainly stronger building materials and other equally inert container materials, but the fact we can see through glass makes it very special.

The glass that we experience every day in windows, bottles, and screens for electronic devices is typically different types of silicate glass. Silicate glasses transmit more than 90% of the light that humans can see, i.e., in the wavelength range of 400–800 nm.

However, visible light is only a small slice of the electromagnetic spectrum. The same glass that transmits light with wavelengths from 400–800 nm does not fare nearly as well if we want to view light with wavelengths from 4,000–8,000 nm. As such, other types of glass are needed for these applications.

Devices that transmit light in the infrared region of the electromagnetic spectrum are all around us in the modern world, from contactless thermometers to the lasers used to repair our vision. These applications, discussed in more detail

later, require windows, lenses, and fiber optics that can transmit wavelengths anywhere between about 1 and 15 microns (1,000–15,000 nm).

Creating a glass that maximizes transparency in these long wavelengths requires different starting materials and approaches to melting and forming the glass than is used in silicate glasses. Fortunately, chalcogenide glasses stand ready to fulfill this role.

Chalcogenide glass compositions

The compositional families of glasses studied by materials scientists divide into two broad categories: oxide and nonoxide glasses.¹ The silicate glasses that surround us in everyday life fall into the first category. These glasses are usually soda-lime silicate glasses, meaning they are formed from a backbone of silicon dioxide (SiO₂) with the addition of the oxides of sodium, calcium, magnesium, and aluminum.

Chalcogenide glasses, in contrast, are one of only several types of nonoxide glasses, a category which also includes halide and metallic glasses. They are named for, and formed of, three of the chalcogen elements in Group 16 on the periodic table: sulfur, selenium, and tellurium (Figure 1). These three elements can be made into amorphous solids without the addition of any other elements, though only in very small pieces or thin films.

Oxygen is also in the chalcogen group, but it cannot form a glass by itself. Polonium and livermorium, the final two chalcogen elements, are rare and acutely hazardous. Polonium is produced as the byproduct of nuclear reactors, while livermorium is produced in a beam line and has a half-life of about 60 seconds. So far, no one has attempted to synthesis these elements in a glassy form.

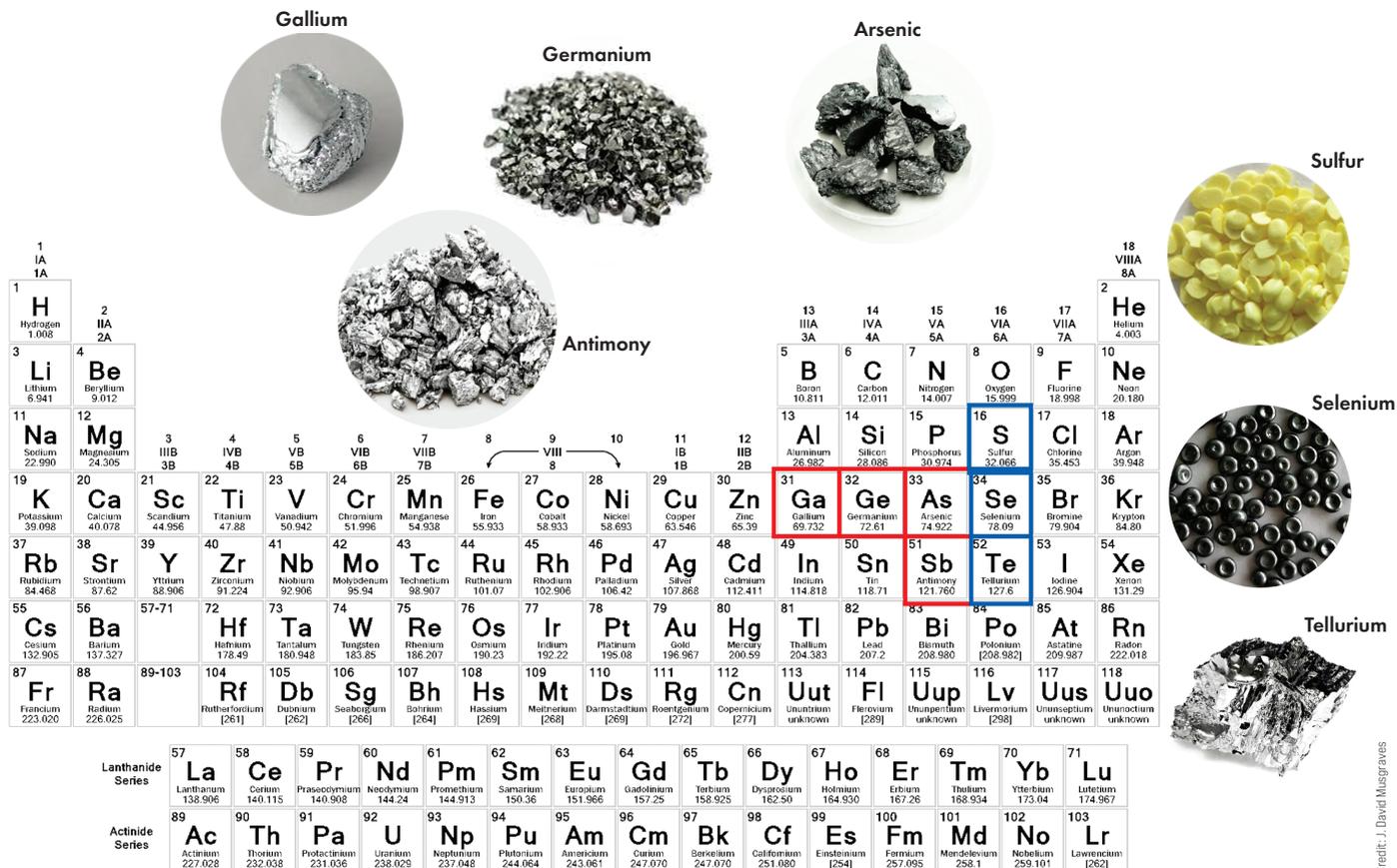


Figure 1. The chalcogen elements used to create chalcogenide glasses (blue outline) and main additives used in these glasses (red outline).

While films and small pieces of elemental sulfur, selenium, and tellurium can be quenched into a glassy form, in commercial applications, they are always modified with some combination of germanium, arsenic, gallium, and/or antimony to improve the thermal and optical properties of the resulting glass. For specialized applications, chalcogenide glasses can also be doped with several different elements, including the rare earths, which make them useful for active infrared optical systems.

The GeAsS and GeAsSe glass families both exhibit a wide glass-forming region, as seen in Figure 2. The glass-forming boundaries of the two glass families is shown in the ternary diagram, with the forming window extending from the line to the 100% chalcogen (sulfur or selenium) in the lower left corner. It is interesting to note that very heavy germanium concentrations can be vitrified in the selenium-based glasses, and that especially in the sulfur-based glass family, small melts of extremely chalcogen-deficient glasses are still achievable.

The changes in transmission window, refractive index, and glass transition temperature of chalcogenides compared to commercial silicate glasses can best be understood by comparing the atomic level structures of the two glass types. The silicate glass structure, shown in Figure 3a, is dominated by the backbone of Si-O bonds in the shape of SiO₄ tetrahedra connected at the corner oxygens. This bond arrangement gives these glasses an inherently 3D interconnected network structure.

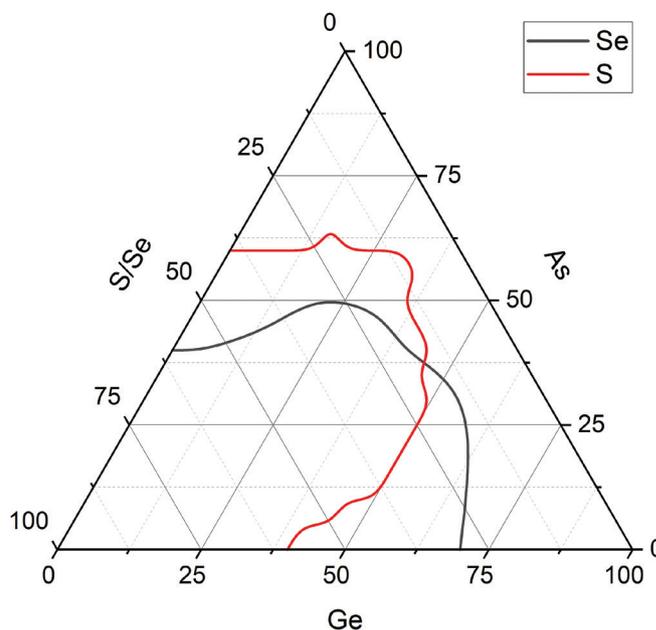


Figure 2. Glass-forming regions of the Ge-As-(S/Se) families.

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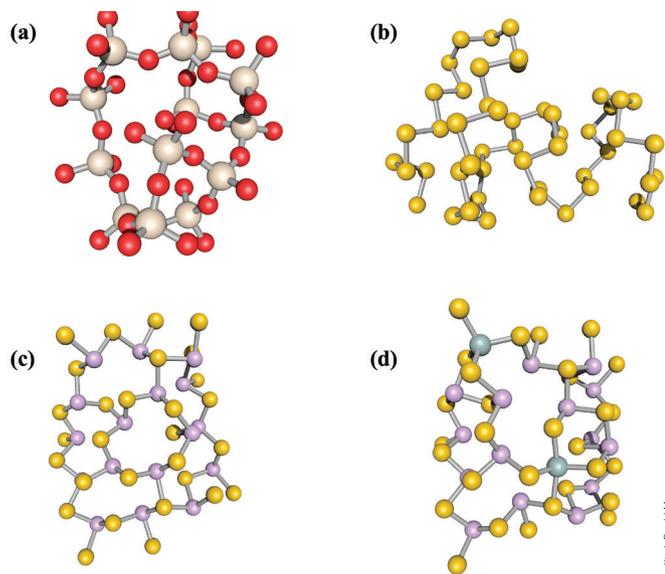


Figure 3. Network structures of glassy (a) silica, (b) selenium, (c) $As_{40}Se_{60}$ and (d) $Ge_{10}As_{30}Se_{60}$.

Compare this structure to the very basic selenium glass in Figure 3b, which is inherently a one-dimensional chain due to selenium’s 2-fold coordination. When arsenic is added to selenium, as in the workhorse commercial chalcogenide composition $As_{40}Se_{60}$ (Figure 3c), for example, the 3-fold coordination of arsenic creates inherently 2D sheet-like structures. The 4-fold coordination of germanium is used to crosslink the chalcogenide network as in Figure 3d. Antimony is 3-coordinated, like arsenic, and can be used in commercial spaces where avoiding arsenic contamination is critically important.

The inherently 3D crosslinked structure of silica means it takes more thermal energy to break the network apart; this fact is reflected in the much lower glass transition and melting temperatures of chalcogenide glasses compared to silicates. Amorphous selenium has a glass transition temperature near room temperature and is completely molten by 221°C. The Si-O bond strength also contributes strongly to the elevated glass transition temperature of silicates compared to chalcogenide glasses.

Manufacturing chalcogenide glasses

One of the most difficult engineering constraints on the manufacturing of chalcogenide glasses is that they cannot contain oxygen. Any oxygen in the glass causes it to begin absorbing infrared light, just like silicate glasses do. It is the presence of the heavy glass-forming elements and lack of oxygen that give chalcogenide glasses their infrared transparency.

Synthesis of chalcogenide glasses begins with securing elemental sulfur, selenium, tellurium, germanium, arsenic, and antimony of high purity (>99.999%). These raw materials must be stored and shipped under inert atmospheres. Once received, the elements are weighed into a container, typically fused silica, inside a glove box environment. The container is then transferred from the glove box to a vacuum chamber using a specialized vacuum fitting that keeps the fused silica container sealed to the atmosphere.

Once under vacuum, the silica container is sealed with a torch to form an ampoule containing only the raw elements under vacuum. The ampoule is then placed into a furnace and slowly raised to the melting temperature. (See the next section on “Properties of chalcogenide glasses” for more information regarding the thermal properties of chalcogenides.)

Fabricating chalcogenide glasses that are free of bubbles and striae (internal ripples in the density of the glass) requires additional steps. The melt is typically rocked inside the furnace at the melt temperature for roughly 12 hours to ensure complete dissolution of the elements and melt homogeneity. Following this period, the ampoule is slowly quenched down from the melt temperature over a period up to six hours. Such slow annealing has several benefits:

1. Removes bubbles from the viscous melt.
2. Gives the slowly stiffening melt sufficient time in a viscoelastic state to remove the stress that causes striae in the subsequent glass.
3. Prevents cracking of the glass billet from thermal shock as it comes to room temperature.

This process, shown in Figure 4, is much more akin to what would be called pot melting in the oxide glasses. It is difficult to imagine ever scaling chalcogenide glass production sizes to anything comparable to today’s window glass manufacturing scale.

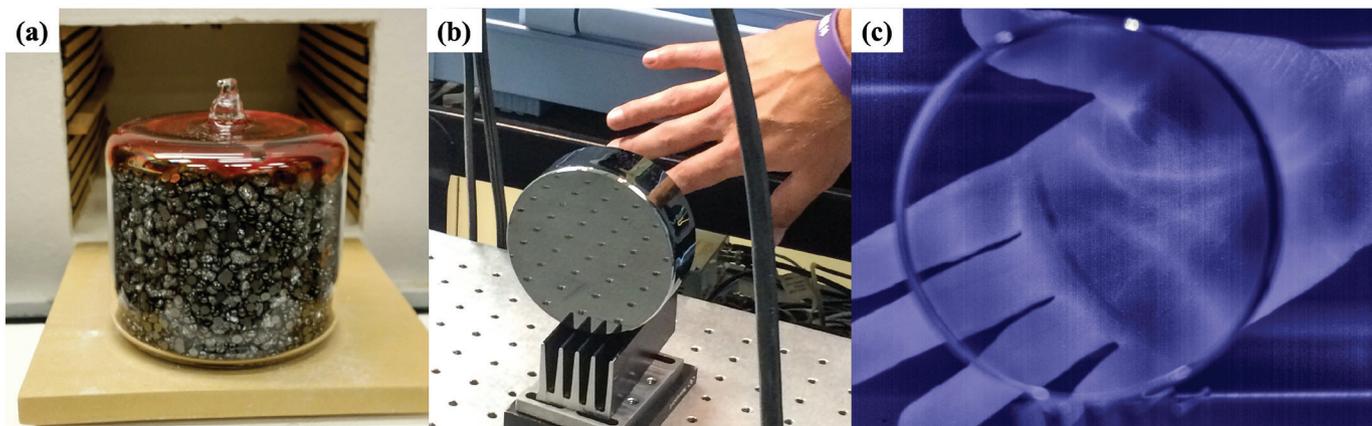


Figure 4. (a) Unreacted batched elements. (b) Polished billet from the melt. (c) Infrared image through the billet.

Properties of chalcogenide glasses

Optical properties

Infrared transmission window

The infrared region of the electromagnetic spectrum is typically divided into four ranges: the near-infrared (NIR) is 0.75–1.4 μm , the short-wavelength infrared (SWIR) band is 1.4–3 μm , the mid-wavelength infrared (MWIR) is 3–8 μm , and the long-wavelength infrared (LWIR) is 8–15 μm .

Different applications operate in different segments of the infrared region. The NIR is used in a wide variety of biomedical imaging applications, the SWIR is home to almost all of the world's telecommunication signals, the MWIR is where many important molecular detection fingerprints lie, and the LWIR is useful for thermal imaging of people and houses. Depending on the application requirements, a chalcogenide glass for a lens or fiber may need to operate at a single wavelength or across several of the infrared bands.

Pure fused silica transmits the furthest into the infrared of all the oxide glasses, but it experiences a cutoff at about 2.3 μm . This cutoff makes it the perfect material for long-haul telecommunication fiber optics, which operate at 1.55 μm , but makes it ineligible for any of the MWIR and LWIR applications.

Sulfide glasses have the valuable property of being transparent in a small part of the visible band in addition to the NIR, SWIR, and MWIR bands. The small addition of visible transparency (the $\text{As}_{40}\text{S}_{60}$ glasses are a deep ruby color to the eye) means that complex optical systems that use a visible guide laser can be aligned in the visible but will be guaranteed to work in the MWIR.

The selenides are the most widely used chalcogenide glasses. Except for the $\text{As}_{40}\text{S}_{60}$ glass, all currently available commercial chalcogenides are selenide glasses. The selenides transmit all the way from the SWIR through the LWIR.

Certain specialized applications may require operation in the far-infrared (15–1,000 μm). In that case, the telluride family of chalcogenide glass is the only candidate material class.

High refractive index

As can be seen indirectly in Figure 5, the chalcogenide glasses have much higher refractive indexes than the oxide glasses. The baseline transmission of the chalcogenides is much lower than silica because more of the light is lost due to the Fresnel reflection at the high index surfaces.

At a wavelength of 4 μm , $\text{As}_{40}\text{S}_{60}$ has a refractive index of 2.417, and the baseline transmission is only 70%. Meanwhile, $\text{As}_{40}\text{Se}_{60}$ has a refractive index of 2.795 and a baseline transmission of about 60%. The tellurides can lose up to 50% of the incident light due to surface reflection. This transmission loss highlights the need for good antireflective coatings on chalcogenide optics, which can be challenging if the application demands a very wide wavelength range.

However, the high refractive index of the chalcogenides lets infrared lens manufacturers achieve designs that are not possible in silica systems. The high refractive index permits much more bending of the light rays at the glass surface (due to

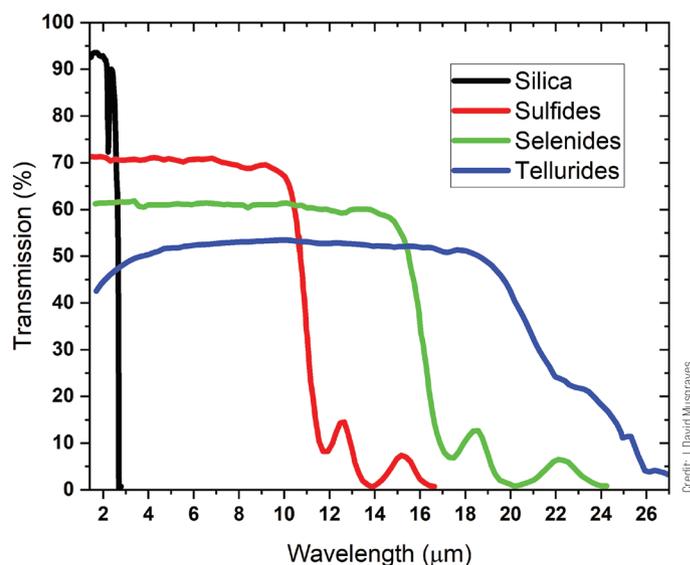


Figure 5. Infrared transmission of several families of glass.

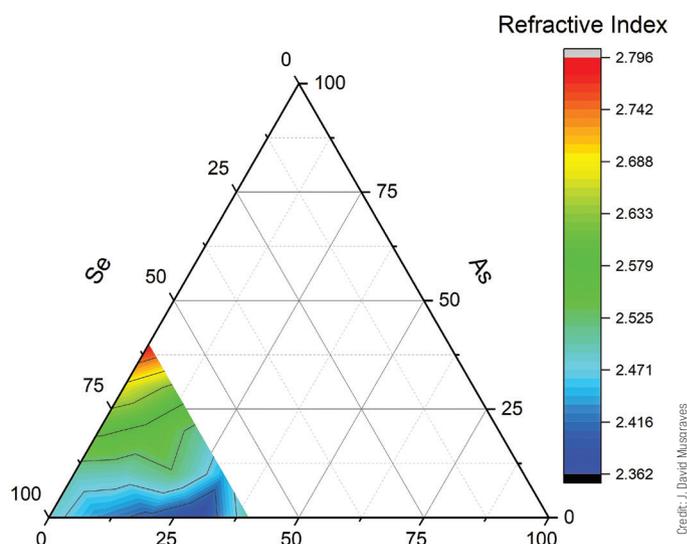


Figure 6. Variation of refractive index at 4 μm for some GeAsSe glasses.

Snell's Law), which allows production of much smaller lenses and the ability to achieve a very high numerical aperture.

Another benefit of the chalcogenide glasses is that their refractive indexes can be tuned to a required value (within a range) by simply altering the ratio of the constituent elements, as shown in Figure 6. In the photonic and fiber optic applications discussed in the next section, a change of 0.01 in the refractive index is enough to provide waveguiding behavior, so the ability to tune the refractive index all the way from 2.362 to 2.796 is a powerful tool for the optical designer. The refractive index of a variety of compositions at a variety of wavelengths can be found in References 2–4.

High n_2 and rare earth solubility

In addition to the high refractive index, chalcogenides also exhibit both a high nonlinear refractive index (n_2) and high rare earth solubility, both of which make these glasses excellent

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materials for active optical elements. The high n_2 of chalcogenide glasses, about 1,000x that of silica, is used in applications such as all-optical switching, optical limiting and modulation, and infrared supercontinuum generation.⁵ The incorporation of rare earth elements mean chalcogenide glasses can be made into infrared lasers, especially when drawn into fiber form. More details on these applications are included in the next section.

Thermal properties

The exact definition of “glass” has varied over the years, but a simplistic definition is that glasses are solids at room temperature but with the atomic structure of a liquid; the material converts to a liquid directly upon heating without undergoing a first-order phase change, such as melting.⁶ Instead, glasses have a glass transition temperature (T_g), which represents the transition point between a glassy solid and a supercooled liquid as the material is heated. Increasing from this temperature range, which can be detected using a calorimeter, causes the glass to soften to a water-like fluid according to its viscosity curve.

Low T_g

Soda-lime glass has a glass transition temperature of anywhere from 520 to 600°C; fused silica, the pure SiO_2 backbone of the soda-lime silicates (shown in Figure 3a) has a T_g around 1,200°C. In contrast, the T_g of commercial chalcogenide glasses ranges from 185–368°C, with 185°C being the T_g of the most common commercial composition, $\text{As}_{40}\text{Se}_{60}$.

For hot forming applications, such as precision glass molding of lenses or drawing of fiber optics, the lower T_g can be very beneficial from the perspective of cost savings: the energy required to hot form chalcogenides is somewhere between a half and a third that of silicate glasses.

On the other hand, the low T_g of chalcogenides can eliminate their consideration in some very high-temperature applications where the only viable option may be crystalline silicon, depending on the required wavelength range. Crystalline germanium also transmits well in the infrared, but its optical properties change dramatically at high temperatures, often eliminating it from consideration for a different reason than the chalcogenides.

Steep viscosity curve

Once the temperature of any glass is raised higher than its T_g , its viscosity decreases monotonically with temperature until reaching that of water. The viscosity of a solid is 10^{12} Pa•s and the viscosity of water is 10^{-3} Pa•s, meaning a change of 18 orders of magnitude between a glass's T_g and what could be called its “melting point.” The temperature window between these two points is critical during any hot forming operations.

Chalcogenides exhibit a relatively abrupt change in viscosity near their T_g , which provides a narrower temperature range within which the glass must be processed. This steep change from a brittle state to a malleable one allows for more precise control in molding and fiber drawing processes. The viscosity of chalcogenide glasses drops rapidly over a small temperature range, reducing the risk of crystallization and allowing for the formation of complex shapes with smoother surfaces and finer details.

In contrast, silicate glasses require a broader temperature range to achieve a workable viscosity, which can often lead to a more energy-intensive process due to the need for higher temperatures to maintain the material in a plastic state suitable for forming. This broader temperature range for silicate glasses can lead to increased structural relaxation and potential for devitrification, which can adversely affect the optical quality of the final product.

The temperature window between the T_g and the point at which the glass behaves like a liquid is referred to as the “working point.” The narrow working point of chalcogenides is particularly beneficial in precision glass molding operations, where it enables rapid thermal cycling and increased production rates. For fiber drawing, the advantage of chalcogenide glasses lies in their ability to be drawn at lower temperatures with high precision, leading to uniform fiber diameters and exceptional optical properties.

Table 1 lists the commercially available chalcogenide glasses along with their density, coefficient of thermal expansion, and some thermal and optical properties.

Chalcogenides in application

Molded infrared optics (thermal imaging)

The largest commercial application (by volume of glass) for chalcogenide glasses is in thermal imaging systems in the form of molded lenses. The thermal properties of chalcogenide glasses discussed above make them perfect candidate materials for precision glass molding (PGM).

PGM is a replicative process, where a glass preform is heated above its T_g , pressed into the lens shape between two precision mold surfaces, and then cooled back to room temperature.⁷ This lens-making process can be quite cost effective because the precision mold surfaces can be made large enough in diameter to have multiple lens cavities, thus allowing for the molding of 2–10 lenses per heating/cooling cycle. PGM is also capable of easily and repeatably molding aspheric surfaces, which cannot be obtained by standard grinding and polishing operations; instead, it requires diamond turning, which is a very serialized and time-consuming process.

Chalcogenide glasses, with their unique optical properties, have found a significant niche in the production of optical components for spectroscopic applications. These glasses are particularly suitable for fabricating diffraction gratings and prisms, which are essential in spectroscopy for dispersing light into its component wavelengths. The process, akin to PGM, involves imprinting the delicate structures necessary for spectral analysis onto the glass, leveraging its softening point characteristics for precise moldability.⁸ This manufacturing technique is highly efficient because it allows for the production of multiple optical components with intricate patterns in a single cycle, significantly reducing the cost and time associated with traditional methods.

The high refractive index and broad transmission spectrum of chalcogenide glasses enhance the performance of spectroscopic instruments, enabling the accurate detection and

Table 1. Commercially available chalcogenide glasses and their properties.

*Vendors: 1) CDGM Glass Company Ltd. (Central Islip, N.Y.), 2) LightPath Technologies (Orlando, Fla.), 3) Rochester Precision Optics (West Henrietta, N.Y.), 4) Schott AG (Mainz, Germany), 5) Umicore SA (Brussels, Belgium), and 6) Vitron Spezialwerkstoffe GmbH (Jena, Germany).

Composition	Density (g/cm ³)	CTE (ppm/°C)	T _g (°C)	Refractive index at 5 μm	Thermal change at 5 μm (ppm/°C)	Vendors*
As ₄₀ S ₆₀	3.20	22.5	197	2.4129	-3.2	1, 3, 4
As ₄₀ Se ₆₀	4.63	21.4	185	2.7909	33.4	1-6
Ge ₁₀ As ₄₀ Se ₆₀	4.47	20.0	225	2.6192	21.15	1, 3, 4, 6
Ge ₃₃ As ₁₂ Se ₅₅	4.41	12.5	368	2.1404	67.7	1, 3, 4, 6
Ge ₂₈ Sb ₁₂ Se ₆₀	4.66	14.0	285	2.6183	62.0	1-4, 6
Ge ₃₀ As ₁₃ Se ₃₂ Te ₂₅	4.84	13.4	275	2.7993	103.8	1, 3, 4, 6

analysis of chemical, biological, and environmental samples across a wide range of industries. These applications take full advantage of the glasses' properties to provide high-resolution spectral data, which is crucial for analytical techniques such as gas chromatography and mass spectrometry.

The adaptability of chalcogenide glasses to these demanding applications further solidifies their role as a material of choice in the advancement of thermal imaging devices.

Photonics

Chalcogenide glasses are extremely interesting materials in the developing photonics ecosystem. One chalcogenide photonic application is already widely deployed: rewritable memory. The composition Ge_{22.2}Sb_{22.2}Te_{55.6}, referred to as GST or GST225, is initially deposited on the rewritable disk substrate via sputtering and deposits in a glassy form. During operation of the rewritable memory disk, lasers are then used to write and rewrite bits of memory into this chalcogenide layer.⁹

During the write phase, a laser is used to warm the glassy area until it is just able to crystallize. The crystalline form of GST has a much higher refractive index than the glassy form, so during the read step, the high index will be read as 1 as opposed to 0 for the glassy form. During the rewrite step, the crystalline bits are flash melted by a second laser and quenched back into the glassy state.

Academic researchers have harnessed all the optical properties of chalcogenide glasses to form a wide array of waveguides, resonators, modulators, and more. Because of their large refractive index, chalcogenides can reduce the diameter of optical ring resonators in sensing systems, and some researchers have even selectively tuned the refractive index post-deposition by taking advantage of the photosensitivity of the glass. Chalcogenide glasses have been fabricated into photonic ring resonators with a Q-factor of 10 million.¹⁰

Specifically for ring resonators, which are commonly used in photonics for applications such as filtering, modulating, and sensing, the Q-factor measures the resonator's ability to confine light for extended periods, thereby enhancing light-matter interactions within the resonator. The higher the Q-factor, the lower the energy loss and the longer the photon lifetime within the resonator, which corresponds to a narrower resonance linewidth. High-Q ring resonators are sought for their ability to enhance light-matter interactions, making them invaluable in applications that require high sensitivity

or selectivity, such as narrowband filters, optical sensors, and nonlinear optical devices.¹¹

Chalcogenide glasses are rapidly emerging as a pivotal material in on-chip photonic applications due to their remarkable optical properties. Their mid-infrared transparency makes them ideal for the fabrication of on-chip photonic circuits, which are essential for next-generation environmental sensing and health diagnostics. These materials are being integrated into on-chip lasers that rely on their ability to be doped with rare earth elements, offering a spectrum of laser wavelengths tailored for specific chemical detection and analysis. The high nonlinearity of chalcogenide glasses also makes them suitable for on-chip nonlinear optical devices, which are critical for frequency conversion and the generation of entangled photon pairs in quantum circuits.

Moreover, the thermal stability and moldability of chalcogenide glasses facilitate the creation of complex photonic structures on-chip, including high-quality microresonators and integrated optical isolators. Such components are key in controlling light propagation and interaction in photonic integrated circuits, paving the way for advancements in optical computing and signal processing.

The versatility and high performance of chalcogenide glasses in these applications highlight their transformative potential in the realm of on-chip photonics.

Fibers

Optical fibers made from chalcogenide glasses are commercially available through several sources. The main use of these fibers is the transmission of infrared laser light, acting just like their silicate glass counterparts but operating in the SWIR to LWIR range. CO₂ lasers, which can be used for everything from eye surgery to antimissile countermeasures, operate principally at 10.6 μm, and delivery of this laser power to a manipulable point (e.g., an optical scalpel) requires transmission through a fiber delivery mechanism.^{12,13}

Unlike their silicate fiber optic counterparts, which are drawn from a single doped preform rod, chalcogenide glass fibers are often drawn using the double crucible method.¹⁴ As implied by the name, this technique relies on a nested pair of crucibles containing the glass of the core and cladding separately. During the draw process, the core fiber is drawn out through the nozzle of the cladding glass, which encases the emerging core as it solidifies.

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In addition to pure laser power delivery, many applications use the broad infrared transparency window to perform spectroscopy because many important chemicals have characteristic infrared-absorption features. Many drugs, explosives, and contaminants can be identified through these “molecular fingerprints,” which makes infrared spectroscopy useful in everything from water monitoring to pharmaceutical quality control to TSA security inspections.

Challenges and perspectives

There has been an explosion in technologies and applications for chalcogenide glasses since the first commercial chalcogenide glass was developed at Texas Instruments in 1962. Although their original TI1173 glass composition still lives on under a variety of trade names, the number of commercial compositions has increased to six (Table 1), with another 10 under final development.

Academic researchers continue to explore even more varieties of chalcogenide glass compositions, optimizing their materials on a laboratory scale to achieve perfect combinations of refractive index, thermal expansion, and many other properties necessary in the development of tomorrow’s technologies.

Future research and development of chalcogenide glasses face many challenges. In the realm of infrared lenses, crystalline germanium continues to be the gold standard material, though chalcogenides are appearing on more and more customer drawings. Crystalline germanium is a much tougher material than chalcogenide glasses, which tend to be both brittle and comparatively soft, so many U.S. Department of Defense or space customers still require germanium. There is not a clear materials science path to increasing the toughness of glasses to that of germanium, but many optical coating companies have developed diamond-like-carbon coatings that make the scratch resistance of the glasses similar to germanium.

In terms of commercial adoption, there are always fears about the arsenic content of the common chalcogenide glasses. The European Registration

Evaluation Authorization of Chemicals (REACH) Regulation went into effect in 2015, effectively banning the use of arsenic in the glass industry in the bloc. Increasingly, chalcogenide glass manufacturers are being asked to supply glass containing antimony rather than arsenic while maintaining the same thermal and optical performance.

In the booming photonics industry, chalcogenide glasses face the challenge of gaining acceptance into a large fabrication environment. In such tightly environmentally controlled spaces, fears of cross contamination from arsenic and selenium make it a slow process to adopt the glasses. In smaller companies and in academic research laboratories, there is less of a barrier to entry, and chalcogenide photonic devices are already being widely commercialized.

As glass manufacturers begin to offer more and more chalcogenide glass compositions, infrared optical designers and engineers will have a much bigger toolbox with which to work. The advances in chalcogenide glass since that first TI1173 have been amazing, but they are only the beginning of a much larger future.

About the author

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