

Figure 1. (a) Tilted focused-ion-beam SEM view of a multilayer woodpile photonic crystal (before delamination from the silicon handler substrate). Colors indicate various layers.<sup>2</sup> (b) SEM image of a chalcogenide glass waveguide integrated photodetector. (c) Measured photocurrent of the photodetector as a function of waveguided optical power at 1550-nm wavelength. Inset shows *I*-*V* characteristics of the detector in dark and at 250-μW input optical power.

# Amorphous thin films for mechanically flexible, multimaterial integrated photonics

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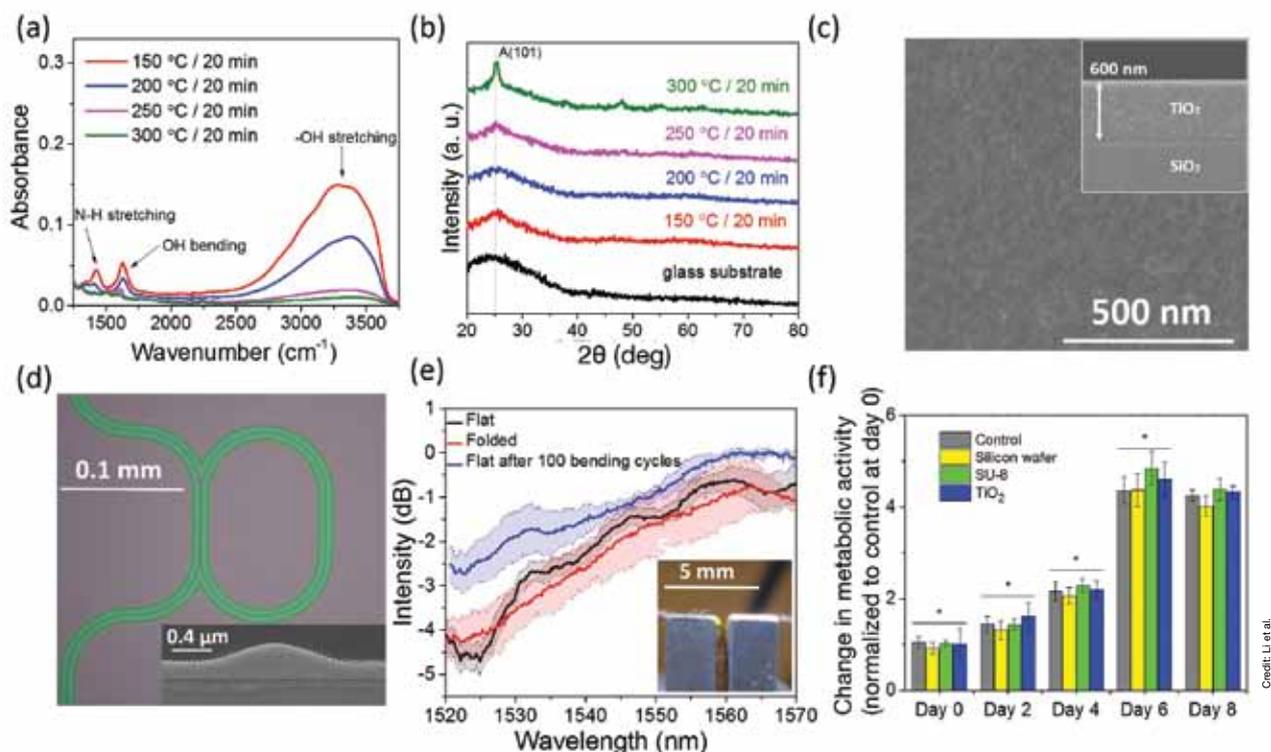
Integration of amorphous chalcogenides and TiO<sub>2</sub> on polymers can enable photonic devices with exceptional mechanical flexibility.

Flexible integrated photonics is a new technology that started to burgeon during the past few years. It opened applications from flexible optical interconnects to conformal sensors on biological tissues. Material choice is one of the most important factors dictating performance of these flexible devices.

Organic polymers generally are compatible with flexible substrates. However, low refractive indexes of polymers (compared with semiconductors) cannot provide the strong optical confinement necessary for compact photonic integration. Besides polymers, researchers are actively pursuing semiconductor nanomembranes—thin slices of single-crystal semiconductors with submicrometer thickness—for photonic device integration on flexible substrates.

Unlike their rigid bulk counterparts, nanomembranes can bend tightly without cracking, because surface strain induced by bending linearly scales with membrane thickness. Usually, we make photonic devices from nanomembrane structures that are patterned on a rigid substrate, such as silicon. We then pick up the fabricated structures using a poly(dimethylsiloxane) rubber stamp and transfer them onto final flexible substrates. This multistep hybrid method limits processing yield and throughput.

Therefore, we turned to amorphous glasses—the material of choice for optics given their exceptionally low optical attenuation. We use these noncrystalline materials in flexible photonics because they enable monolithic fabrication and can be deposited directly onto flexible substrates without resorting to epitaxial growth. Specifically, we



**Figure 2.** (a) IR spectra and (b) X-ray diffraction spectra of sol-gel  $\text{TiO}_2$  thin films annealed at various temperatures. Arrows in (a) indicate characteristic optical absorption bands of chemical residues, and diffraction peak of the anatase phase in (b) is noted by “A(101).” (c) Top-view SEM photograph of a  $\text{TiO}_2$  film annealed at 250°C. Inset represents a film cross-section. (d) Optical microscopy photograph of top-view of a  $\text{TiO}_2$  racetrack micro-resonator. Inset shows cross-sectional SEM photograph of the waveguide. (e) Normalized optical transmission spectra of a flexible  $\text{TiO}_2$  waveguide prior to and after repeated folding. Inset shows a folded  $\text{TiO}_2$  waveguide sample under test. (f) Proliferation of human mesenchymal stem cells in indirect contact with photonic materials. \*Significantly different ( $p < 0.01$ ) from days 0–6. No significant difference was observed between days 6 and 8.<sup>1</sup>

focused on chalcogenide glass materials and amorphous  $\text{TiO}_2$  because they can be deposited at relatively low temperatures ( $\leq 250^\circ\text{C}$ ) compatible with flexible substrate integration.<sup>1–4</sup>

### Chalcogenide glasses for 2.5-D photonic integration on flexible substrates

Chalcogenide glasses are amorphous semiconductors that contain one or multiple chalcogen elements, namely sulfur, selenium, and tellurium. Extraordinary infrared (IR) transparency makes these materials popular for optical components, such as IR windows, lenses, optical fibers, and coatings. Phase change memories, all-optical signal processing, chem-bio sensing, and on-chip light switching and modulation are other emerging applications where chalcogenide glasses are important.<sup>5</sup> By incorporating a multi-neutral-axis mechanical design, we demonstrated low-loss, robust photonic devices on flexible polymer substrates capable of sustaining repeated bending down to submillimeter radii,

despite intrinsic fragility of chalcogenide glass materials.<sup>2</sup>

Besides excellent optical properties, chalcogenide glasses exhibit extreme processing versatility—they can be monolithically deposited on virtually any technically important substrate and can be shaped into functional device forms via traditional lithography or a variety of soft lithographic methods, including molding, imprinting, and ink-jet printing.<sup>6</sup> Therefore, chalcogenide glasses are uniquely poised for 2.5-D photonic integration, which refers to vertical stacking of photonic devices in multiple layers.

Fink et al. showed that chalcogenide glasses readily can form planar multilayers (e.g., Bragg mirrors) via sequential thin-film deposition.<sup>7</sup> We extend the process to stacking of patterned photonic devices by introducing a planarization step between film depositions. In the process, we spin-coated a polymer layer on top of a patterned chalcogenide glass film. We then thermally annealed the polymer/glass to allow the polymer to flow and planarize the surface before

cross-linking, thereby facilitating subsequent deposition and patterning. Using these techniques, we have demonstrated an array of multilayer photonic components on flexible substrates, such as vertically stacked optical resonant filters, overpass structures for waveguide crossings, and woodpile photonic crystals (Figure 1(a)).<sup>2</sup>

Recently, we showed that the approach also is applicable to integration of active optoelectronic components with passive glass photonics. Figure 1(b) shows a top-view scanning electron microscopy (SEM) photograph of a chalcogenide glass waveguide integrated with an adhesive-bonded semiconductor nanomembrane photodetector. Figure 1(c) is a plot of the measured photocurrent as a function of guided power in waveguides. Compared with traditional photodetectors, which capture only free space illumination, the much smaller optical mode volume enabled by waveguide integration underlies a much larger—and potentially much faster—optical response in these detectors. These results

open up exciting applications in which chalcogenide glasses can seamlessly integrate with other optical materials to enable unconventional functionalities.

## Foldable and cytocompatible sol-gel TiO<sub>2</sub> photonics

We also investigated sol-gel TiO<sub>2</sub> as another amorphous material for flexible photonic integration. Besides sharing the same processing advantages—such as low-temperature deposition and ease of integration as chalcogenide glasses—TiO<sub>2</sub> is particularly attractive for biophotonic applications because it is generally considered biocompatible and has been used in dental fillers, cosmetic products, and artificial bone scaffolds.

In our work, we deposited amorphous TiO<sub>2</sub> films using an organic-free sol-gel process. The SEM photograph in Figure 2(c) indicates the uniformity and smooth surface of a sol-gel-coated TiO<sub>2</sub> thin film. Postdeposition annealing temperature is a critical parameter in determining TiO<sub>2</sub> film quality. Figures 2(a) and (b) show that increased annealing temperature contributed to removal of chemical residues and reduction of parasitic optical absorption. However, annealing at >250°C results in partial crystallization, which leads to optical scattering by crystalline grains. TiO<sub>2</sub> films annealed at 250°C feature a uniform and smooth surface (Figure 2(c)) and a relatively low optical loss of 3 dB/cm, which is suitable for photonic integration.

Using a sol-gel technique and plasma etching, we fabricated and tested TiO<sub>2</sub> optical waveguides and resonators monolithically integrated on flexible polymer substrates (Figure 2(d)). Similar to chalcogenide glass flexible photonics, the multi-neutral-axis design renders TiO<sub>2</sub> devices extremely flexible—fabricated TiO<sub>2</sub> waveguides can be repeatedly folded in half without introducing measurable optical degradation (Figure 2(e)). We further validated cytocompatibility of these TiO<sub>2</sub> devices through in-vitro cell viability tests (Figure 2(f)), which show that human mesenchymal stem cells cultured on TiO<sub>2</sub> devices exhibit the same level of metabolic activity as those grown on a reference cell culture

plate. Building on these results, we now focus our ongoing work on integrating TiO<sub>2</sub> flexible photonic sensors with biological tissue engineering platforms to enable real-time monitoring of cell growth.<sup>1</sup>

## Thin films continue to be important

Processing of amorphous thin films is far more forgiving compared with epitaxy, which is mandated for growing traditional optical crystal materials. Consequently, they can be readily mated with other functional materials to create composite structures possessing unique properties not accessible to glasses alone. Here, we demonstrated that integration of amorphous chalcogenides and TiO<sub>2</sub> on polymers can enable photonic devices with exceptional mechanical flexibility. On the other hand, integration of glasses with semiconductor nanomembranes enables full active-passive integration toward realizing standalone flexible “system-on-a-chip” photonic platforms. These are certainly two cases exemplifying the universal multimaterial photonic integration paradigm, in which we foresee that amorphous thin films will continue to play a pivotal role.

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## Editor's note

Li will present the 2016 Kreidl Award Lecture at the Glass and Optical Materials Division Annual Meeting in Madison, Wis., on May 24, 2016.

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