

Nanoscale Silicon Anodes and Solid Electrolyte Interfaces: A Materials Perspective

Sunil B. Hiwale

Department of Chemistry, Sant Ramdas Arts, Commerce & Science College, Ghansawangi, Dist. Jalna, Maharashtra, India-431209

Corresponding Author: sunilhiwale139@gmail.com

Abstract

Silicon has re-emerged as the leading candidate to succeed graphite as the commercial anode material for high-energy lithium-ion and solid-state batteries, owing to a theoretical specific capacity nearly ten times greater than graphite and a favorably low delithiation potential. Realizing this potential, however, requires resolving two intertwined materials problems: the extreme volumetric breathing (>300%) that silicon undergoes during lithiation/delithiation, and the chronic instability of the interface it forms with the surrounding electrolyte. This review synthesizes recent progress on both fronts from a nanoscale materials perspective. We first summarize the mechanistic origins of silicon's capacity fade—particle pulverization, continuous solid electrolyte interphase (SEI) regrowth, and loss of electrical percolation—before surveying the principal synthesis routes used to engineer nanostructured silicon (ball milling, magnesiothermic/aluminothermic reduction, chemical etching, CVD, and hydrothermal methods) and the architectures they enable (nanoparticles, nanowires, porous/hollow frameworks, and Si–carbon composites). We then examine how the interfacial problem evolves when silicon is paired with liquid electrolytes versus solid-state electrolytes (sulfide, oxide, and polymer classes), emphasizing the mechanochemical coupling between particle-scale stress and interphase chemistry that is unique to all-solid-state architectures. Finally, we discuss emerging interface-engineering strategies—artificial SEI layers, conformal coatings, stress-accommodating binders and electrolytes, and stack-pressure management—alongside operando characterization approaches, and outline the remaining barriers to commercial deployment of silicon-based anodes in next-generation energy storage systems.

Keywords: *silicon anode; nanoparticle synthesis; solid electrolyte interphase (SEI); all-solid-state battery; interfacial engineering; lithium-ion battery*

1. Introduction

The demand for higher-energy-density, safer, and longer-lived rechargeable batteries—driven by electric mobility, portable electronics, and grid-scale storage—has pushed research well beyond the graphite anode that has underpinned commercial lithium-ion batteries (LIBs) for three decades. Graphite offers excellent cycling stability but is capacity-limited to 372 mAh g⁻¹ by its LiC₆ intercalation stoichiometry. Silicon, by contrast, alloys with lithium to form Li₁₅Si₄ (and, at full lithiation, Li₂₂Si₅), yielding a theoretical specific capacity of approximately 3579 mAh g⁻¹, roughly an order of magnitude higher, together with a low working potential (~0.4 V vs. Li/Li⁺), natural abundance, and low cost.

This promise is offset by two coupled failure modes. First, the insertion of this much lithium causes silicon to expand by more than 300% in volume, generating mechanical stresses that fracture particles, pulverize electrodes, and disconnect active material from the conductive network. Second, every newly exposed silicon surface reacts with the electrolyte to form a solid electrolyte interphase (SEI); because the particle surface area changes continuously with each cycle, the SEI never stabilizes, consuming electrolyte and mobile lithium and steadily increasing impedance. In all-solid-state batteries (ASSBs), a third dimension of difficulty appears: the electrode–electrolyte contact is inherently solid–solid, so volume breathing can physically separate silicon from its ion-conducting pathway even before any chemical degradation occurs.

Nanostructuring has become the dominant strategy for mitigating the mechanical half of this problem. Reducing at least one particle dimension below roughly 150 nm suppresses fracture by keeping absolute (rather than fractional) strain within a tolerable range, while also shortening lithium diffusion paths and providing additional storage sites at grain boundaries. This review takes a materials-perspective approach: Section 2 outlines the fundamentals of silicon lithiation

and degradation; Section 3 surveys nanoparticle and nanostructure synthesis routes; Sections 4 and 5 examine interfacial behavior in liquid-electrolyte and solid-state systems, respectively; Section 6 discusses interface-engineering strategies; Section 7 summarizes characterization approaches; and Section 8 outlines open challenges and future directions.

2. Fundamentals of Silicon as an Anode Material

2.1 Lithiation Mechanism and Capacity

Unlike graphite's staged intercalation, silicon stores lithium through an alloying reaction. Pristine crystalline silicon lithiates via a two-phase mechanism—a sharp reaction front separates crystalline Si from an amorphous Li_xSi phase—before crystallizing into $\text{Li}_{15}\text{Si}_4$ near full lithiation at room temperature. This alloying mechanism is intrinsically more disruptive to the host lattice than intercalation, which is the root cause of the volume expansion problem.

2.2 Degradation Pathways

Three interlinked degradation mechanisms limit cycle life in silicon-based anodes:

- Mechanical pulverization: repeated volumetric breathing generates stresses that exceed the fracture toughness of silicon particles above a critical size, leading to particle cracking, loss of electrical contact, and capacity fade.
- SEI instability: because particle surfaces expand and contract each cycle, the SEI layer repeatedly cracks and reforms, irreversibly consuming active lithium and electrolyte and increasing charge-transfer resistance over time.
- Electrode-level integrity loss: at the composite-electrode scale, binder detachment, current-collector delamination, and electrode swelling compound the particle-level effects, reducing Coulombic efficiency and rate capability.

Nanostructuring, composite design (particularly silicon–carbon architectures), and interfacial engineering—the subjects of the remaining sections—each target one or more of these degradation pathways.

3. Synthesis Strategies for Nanostructured Silicon

The choice of synthesis route determines particle size, morphology (0D nanoparticles, 1D nanowires/nanotubes, or 3D porous/hollow architectures), surface chemistry, and ultimately scalability. Table 1 compares the principal methods used to produce nanostructured silicon for anode applications.

Table 1. Comparative overview of synthesis routes for nanostructured silicon anode materials.

Method	Typical Precursor	Particle Architecture	Key Advantages	Key Limitations
High-energy ball milling	Metallurgical-grade Si, ferrosilicon, PV waste	Irregular 0D nanoparticles (<150 nm)	Scalable, low cost, feedstock-flexible, industrially mature	Broad size distribution, agglomeration, surface oxidation, high defect density
Magnesiothermic / aluminothermic reduction	SiO_2 (nanoporous, diatomite, rice-husk ash, clay)	Porous 0D/3D sponge-like Si	Retains templated porosity, low-cost natural precursors, tunable pore volume	Exothermic runaway risk, HCl/HF post-etch needed, moderate yield (10–90%)
Chemical/electrochemical etching (HF-based)	Bulk Si wafers, metallurgical Si	Porous Si, mesoporous spheres	Fine control of pore size and porosity	Hazardous reagents, poor scalability, high cost

Chemical vapor deposition (CVD)	Silane (SiH ₄), disilane	Thin films, coated nanowires, core-shell Si@C	Excellent morphology and coating control, strong adhesion to current collector	High capital cost, low throughput, silane toxicity/flammability
Hydrothermal / solvothermal synthesis	Silicate precursors, organosilanes	Mesoporous nanospheres, hollow structures	Mild conditions, tunable morphology	Energy-intensive, limited scale-up, long reaction times

3.1 Zero-Dimensional (0D) Silicon Nanoparticles

High-energy ball milling remains the most industrially attractive route to 0D silicon nanoparticles because it is scalable, low-cost, and compatible with diverse and even waste-derived feedstocks such as photovoltaic-grade silicon scrap, metallurgical-grade silicon, and ferrosilicon. Mechanical impact reduces bulk silicon to particle sizes below roughly 150 nm, alleviating pulverization by lowering the absolute expansion strain during lithiation, although over-milling can promote agglomeration and requires co-processing with carbon sources to maintain dispersion and conductivity.

Magnesiothermic and aluminothermic reduction of silica precursors—ranging from engineered nanoporous SiO₂ to natural feedstocks such as rice-husk ash, diatomite, and clay minerals—offer a complementary route that inherits and preserves the porosity of the silica template. Recent work has demonstrated ball-milling-assisted magnesiothermic reduction that proceeds without external heating or clay pretreatment, improving process simplicity and scalability, while controlled-reduction approaches that regulate magnesium particle size have been used to limit the exothermic heat accumulation that otherwise collapses the porous architecture. Reported reversible capacities for porous nano-silicon produced by these routes reach approximately 1500 mAh g⁻¹ after extended cycling at moderate rates, though post-synthesis acid leaching (HCl/HF) to remove MgO/Mg₂Si by-products remains a scalability and safety bottleneck.

3.2 One-Dimensional (1D) Nanowires and Nanotubes

Silicon nanowires (SiNWs) and nanotubes (SiNTs), typically grown by CVD-based vapor–liquid–solid processes or by metal-assisted chemical etching, provide direct, unbroken conduction pathways along their length and can accommodate radial expansion more gracefully than isolated particles. Their nanoscale diameter shortens the lithium diffusion distance, improves electrolyte penetration, and reduces polarization at high rates. However, growth processes generally require complex vacuum equipment and precise catalyst control, resulting in higher production costs that continue to limit large-scale deployment relative to particulate approaches.

3.3 Porous, Hollow, and Composite (3D) Architectures

Porous silicon—produced by electrochemical/chemical etching, magnesiothermic reduction, or templating methods—introduces internal void space that can absorb volumetric expansion without transmitting stress to the outer particle envelope. Combining porous or nanoscale silicon with conductive, mechanically buffering carbon shells (Si@C, graphene-wrapped Si, or reduced-graphene-oxide-encaged architectures) further improves electronic conductivity and helps isolate the silicon surface from direct electrolyte contact, thereby limiting parasitic SEI growth. Two-step ball-milling processes that first reduce micron-silicon to sub-micron particles and then integrate alumina or graphene-oxide coatings exemplify this composite design philosophy, and hierarchical architectures that embed silicon nanoparticles within high-entropy alloy matrices or honeycomb-inspired carbon scaffolds have been reported to deliver improved initial Coulombic efficiency and capacity retention over hundreds of cycles.

3.4 Chemical Vapor Deposition and Hydrothermal Routes

CVD, typically using silane or disilane precursors, allows precise control over film thickness, coating conformality, and direct deposition onto current collectors or onto pre-formed nanostructured scaffolds, making it well suited to laboratory-scale architecture optimization and thin-film solid-state cells, though the toxicity and flammability of silane precursors and the high capital cost of vacuum deposition equipment constrain throughput. Hydrothermal and solvothermal methods, using silicate or organosilane precursors under mild aqueous conditions, provide additional morphological

control—mesoporous spheres and hollow shells in particular—at the expense of longer reaction times and modest scalability.

3.5 Toward Scalable, Sustainable Synthesis

A consistent theme across recent literature is the shift toward feedstocks that are inexpensive, abundant, or waste-derived—photovoltaic kerf waste, agricultural silica (rice husk), and natural clay minerals—processed through mechanochemical routes that minimize energy input and hazardous reagent use. Recycled or bio-derived binders, such as polyacrylamide-based binders applied to silicon kerf-waste anodes, further extend this sustainability trend into the electrode-formulation stage. Balancing scalability, cost, safety, and electrochemical performance remains the central commercialization bottleneck for nanostructured silicon, motivating continued interest in one-dimensional and hierarchical architectures that can mitigate the intrinsic limitations of simple 0D nanoparticles.

4. The Solid Electrolyte Interphase in Liquid-Electrolyte Systems

4.1 Formation and Composition

In conventional carbonate-based liquid electrolytes, the SEI that forms on silicon is compositionally similar in broad strokes to that on graphite—comprising inorganic species such as LiF and Li_2CO_3 alongside organic lithium alkyl carbonates—but is mechanically far less stable because the underlying substrate continuously changes volume. Each lithiation/delithiation cycle can crack the existing SEI, exposing fresh silicon that reacts anew with electrolyte, so the interphase thickens and its composition drifts over cycling rather than reaching the quasi-steady state observed on graphite. This continuous parasitic reaction is a principal contributor to the low initial Coulombic efficiency and progressive capacity fade characteristic of silicon anodes, and studies have shown that initial Coulombic efficiency correlates closely with a particle's specific surface area—smaller, higher-surface-area nanoparticles trade improved mechanical tolerance for larger first-cycle irreversible losses.

4.2 Mitigation Strategies

Several complementary strategies have been developed to stabilize the silicon–liquid electrolyte interface:

- Conductive/protective coatings (carbon, Al_2O_3 , conducting polymers) that physically separate silicon from direct electrolyte contact while accommodating volume change.
- Electrolyte additives (e.g., fluoroethylene carbonate, vinylene carbonate) that preferentially decompose to form thinner, more LiF-rich and more flexible SEI layers.
- Functional binders with strong, often reversible, silicon-surface adhesion (e.g., cross-linked or ion-conductive binders) that maintain electrode integrity and can facilitate more uniform prelithiation.
- Prelithiation strategies, including mixed-conducting approaches for uniform lithium pre-insertion, that compensate for first-cycle irreversible lithium loss to the SEI.

Collectively, these approaches have pushed practical silicon-dominant and Si–graphite blended anodes closer to commercial viability in liquid-electrolyte LIBs, though achieving both high mass loading and long cycle life simultaneously remains challenging at the composite-electrode level.

5. Silicon Anodes in Solid-State Battery Architectures

5.1 Why Solid-State Changes the Interfacial Problem

All-solid-state batteries (ASSBs) replace the liquid electrolyte with a solid ion conductor, promising improved safety and enabling the pairing of silicon anodes with high-capacity, high-voltage cathodes at cell-level energy densities that liquid systems struggle to match. However, the electrode–electrolyte contact in an ASSB is fundamentally solid–solid rather than solid–liquid. Because a solid electrolyte cannot flow to accommodate a breathing particle the way a liquid can, silicon's volumetric expansion in ASSBs risks not only particle fracture but outright physical separation from the

ion-transport pathway—a failure mode largely absent in liquid systems. Recent mechanistic work has shown that low expansion alone is insufficient if intimate solid–solid contact cannot be maintained under realistic operating conditions, particularly at the low stack pressures relevant to practical cell design.

5.2 Solid Electrolyte Classes and Their Interfacial Behavior

The three principal classes of solid electrolyte—sulfide, oxide, and polymer—interact with silicon in materially different ways, summarized in **Table 2**.

Table 2. Comparison of solid electrolyte classes and their interfacial behavior with silicon anodes.

Electrolyte Class	Representative Materials	Ionic Conductivity (RT)	Interfacial Behavior with Si	Primary Challenge
Sulfide-based	Li ₆ PS ₅ Cl, Li ₁₀ GeP ₂ S ₁₂ , Li ₃ PS ₄	10 ^{−3} –10 ^{−2} S cm ^{−1} (high)	Reactive; forms mixed Li ₂ S/SiS ₂ /P ₂ S ₅ -type interphase layers	Air/moisture sensitivity; interphase (de)lithiation-driven cracking
Oxide-based	LLZO (garnet), NASICON, LiPON	10 ^{−4} –10 ^{−3} S cm ^{−1}	Rigid and brittle; poor conformal contact with expanding Si	High interfacial impedance; difficulty maintaining solid–solid contact
Polymer-based	PEO, PVDF-HFP composite electrolytes	10 ^{−5} –10 ^{−4} S cm ^{−1} (RT, unmodified)	Compliant; can chemically anchor to Si surface and buffer stress	Lower intrinsic ionic conductivity; narrower electrochemical window
Composite hybrid	Polymer–ceramic, polymer–sulfide blends	Intermediate, tunable	Combines mechanical compliance with higher conductivity pathways	Interfacial compatibility between constituent phases; processing complexity

Sulfide electrolytes (e.g., Li₆PS₅Cl, Li₁₀GeP₂S₁₂) offer the highest room-temperature ionic conductivities, approaching or exceeding liquid electrolytes, but are chemically reactive toward silicon. In model Si/sulfide systems, an interfacial layer only a few nanometers to tens of nanometers thick, composed of species such as Li₂S, SiS₂, and P₂S₅-derived glasses, forms during the first cycle and functions as a Li-conductive, electronically insulating interphase—analogue in role, though distinct in chemistry, to the conventional SEI. In situ electrochemical impedance and X-ray computed tomography studies have identified multiple crack-formation mechanisms in silicon/sulfide composite electrodes, including through-thickness channel cracks and delithiation- or relithiation-induced interfacial fracture, the latter observed predominantly in thicker silicon electrodes.

Oxide electrolytes (e.g., garnet-type LLZO, NASICON-type conductors) offer excellent electrochemical and chemical stability but are mechanically rigid and brittle, making it intrinsically difficult to maintain conformal contact with an anode that swells by hundreds of percent; interfacial impedance arising from poor and evolving physical contact is consequently a dominant limitation for oxide-based Si-ASSBs.

Polymer electrolytes (e.g., PEO- and PVDF-HFP-based systems) are mechanically compliant and can chemically anchor to the silicon surface, actively regulating interphase chemistry, stress accommodation, and contact preservation rather than serving merely as a passive ion-conducting medium. Recent work combining composite polymer electrolytes with silicon nanosheet electrodes via in situ secondary coating has demonstrated lower charge-transfer resistance and more stable cycling than mechanically stacked cells, by maximizing solid–solid interfacial contact area. LiF-rich and Li₂SO_x-rich interphase chemistries, along with chemically anchored interfaces, have emerged as particularly effective at stabilizing the silicon–polymer electrolyte boundary.

5.3 Mechanical–Electrochemical Coupling

A distinguishing feature of silicon in ASSBs is the tight coupling between mechanics and electrochemistry: stack pressure, particle-level stress fields, and interphase evolution jointly determine cell performance in a way that has no direct liquid-electrolyte analogue. Multiphysics modeling of silicon/carbon composite anodes in ASSBs has identified distinct, spatially separated failure mechanisms—shear-driven failure at the anode–electrolyte interface versus tension-driven failure at particle–coating interfaces—underscoring that interfacial adhesion strategies must be tailored to the specific stress state at each length scale rather than applied uniformly across the electrode.

6. Interface Engineering Strategies

Across both liquid and solid-state architectures, interface engineering strategies for silicon anodes converge on a common set of design principles, adapted to each electrolyte class:

- Conformal protective coatings (carbon, oxide, or polymer) that decouple mechanical buffering from ionic/electronic transport requirements.
- Composite and hierarchical architectures (Si@C core–shell, graphene-wrapped particles, honeycomb or high-entropy-alloy matrices) that distribute mechanical stress and maintain percolating conductive networks.
- Artificial or electrolyte-derived interphases engineered for specific chemistries (LiF-rich, Li₂SO_x-rich) that combine ionic conductivity with mechanical resilience.
- Stress-accommodating binders and, in ASSBs, compliant polymer or composite electrolyte layers that maintain solid–solid contact under cycling-induced volume change.
- Cell-engineering measures such as stack-pressure optimization and prelithiation, which address failure modes that originate above the single-particle length scale.

No single strategy fully resolves the volume-expansion/interphase-instability problem in isolation; the strongest recent results combine nanostructuring at the particle level with tailored interphase chemistry and, in ASSBs, deliberate mechanical design of the composite electrode and stack architecture.

7. Characterization Approaches

Understanding and optimizing silicon anode interfaces increasingly relies on operando and in situ techniques capable of resolving structural and chemical evolution in real time under electrochemical operation. In situ electrochemical impedance spectroscopy (EIS) tracks the growth of interfacial resistance associated with SEI/interphase evolution and contact loss. In situ and operando X-ray computed tomography (X-ray CT) visualizes crack initiation and propagation within composite silicon electrodes at the particle and electrode scale. In situ thin-film X-ray diffraction resolves structural changes at cathode/electrolyte and anode/electrolyte interfaces in ASSBs. Complementary ex situ techniques—transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and Raman spectroscopy—remain essential for resolving interphase composition and local structure, while multiphysics and chemo-mechanical modeling (including particle-SEI stress-coupled models) provide a framework for interpreting these observations mechanistically and guiding materials design.

8. Challenges and Future Directions

Despite substantial progress, several challenges must be resolved before nanostructured silicon anodes achieve widespread commercial deployment, particularly in solid-state formats:

- Scalable, low-cost, and safe synthesis: methods that combine the performance benefits of porous or composite nanostructures with the throughput and safety profile of simple mechanical processing (e.g., ball milling of waste-derived feedstocks) require continued optimization.
- First-cycle and long-term Coulombic efficiency: the trade-off between nanoscale surface area (which improves mechanical tolerance) and irreversible interphase formation (which consumes lithium) remains only partially resolved.
- Solid–solid contact maintenance under realistic stack pressure: laboratory results obtained under high applied pressure often do not translate directly to the lower pressures achievable in practical cell and pack designs.
- Electrolyte–anode chemical compatibility: sulfide electrolytes in particular require interphases or coatings that suppress continued reactive interdiffusion without compromising ionic conductivity.
- Thick, high-mass-loading electrodes: most laboratory demonstrations use thin electrodes; translating nanostructuring and interphase-engineering benefits to the thick electrodes needed for commercially relevant energy density is a persistent scale-up gap.
- Standardized, predictive characterization and modeling: broader adoption of operando diagnostics and chemo-mechanical models, validated against a wider range of electrolyte chemistries and electrode architectures, would accelerate rational design.

Looking forward, promising directions include hierarchical architectures that integrate high-entropy alloy or carbon matrices with nanoscale silicon, polymer and composite solid electrolytes engineered to actively regulate interphase chemistry rather than serve as passive separators, machine-learning-assisted optimization of synthesis and formulation parameters, and continued valorization of waste and low-cost silicon feedstock's to align performance gains with sustainable and economically viable manufacturing.

9. Conclusion

Nanostructuring has transformed silicon from a theoretically attractive but practically unworkable anode material into a leading candidate for next-generation lithium-ion and solid-state batteries. By controlling particle size, morphology, and composite architecture, researchers have substantially mitigated the mechanical degradation caused by silicon's large volumetric expansion. Yet the interfacial problem—the instability of the SEI in liquid electrolytes and the combined chemical and mechanical fragility of the electrode–electrolyte contact in solid-state systems—remains the central obstacle to commercialization. Progress will depend on continued integration across three fronts: scalable and sustainable nanoparticle synthesis, chemically tailored and mechanically resilient interphases suited to each electrolyte class, and cell-level engineering (stack pressure, electrode architecture, prelithiation) that connects particle-scale materials design to practical device performance. The convergence of these fronts, informed by increasingly sophisticated operando characterization and multiphysics modeling, positions nanoscale silicon anodes as a realistic near-term pathway to higher-energy-density, safer batteries.

Conflict of interest

The author affirms that he has no conflicts of interest related to this work's publication. The research, analysis, findings interpretation, and paper preparation were not impacted by any financial, personal, or professional interactions.

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