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Nanoscience-Integrated Photoacoustic Spectroscopy for Advanced Diagnostics and Materials Optimization in Metal-Based Energy Storage Systems

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Abstract

Original Research Article

Metal-based energy storage systems (ESS)—including lithium, sodium, magnesium, and zinc batteries—are indispensable for sustainable energy applications. Yet, they often suffer from material degradation, unsafe dendrite growth, and ineffective ion transport. This study introduces a novel nanoscience-enhanced photoacoustic spectroscopy (PAS) framework to tackle these challenges with quantitative rigor. PAS, which converts modulated light absorption into acoustic waves, has been shown to image lithium metal dendrites in 3D with micrometer resolution (~3 µm) and penetration depths of ~160 µm. When applied to layered nanomaterials—e.g., MoS_2 reduced from 112 to 7 µm thickness—the PAS signal improves by nearly 50×. Similarly, metal nanoparticle aggregates exhibit distinct PAS signatures, enabling the determination of aggregate size distributions and packing density. Integrating these findings, our work synthesizes evidence from battery-specific PAS studies, highlighting 3D dendrite detection, phase-change monitoring, SEI layer growth, and hotspot identification. We detail synthesis methods (sol–gel, hydrothermal, CVD) and PAS instrumentation (532 nm pulsed laser, piezoelectric detectors, modulation cells) to ensure reproducibility. Comparative analysis shows that nanomaterial-augmented PAS enhances diagnostic sensitivity ~24× over planar electrodes and lowers detection limits by ~4×—a trend consistent with sensor literature. We present case studies with spectral maps and quantitative metrics supporting material engineering interventions like doping, morphology control, and coating. Finally, we discuss ambitions to integrate PAS operando with AI/ML analytics for predictive diagnostics, addressing limitations like depth penetration and instrumentation complexity. This convergence of nanoscience and PAS provides a transformative blueprint for real-time, data-driven optimization of metal-based ESS, aiming at enhanced performance, safety, and longevity.

Keywords: Photoacoustic Spectroscopy, Nanostructured Materials, Metal-Based Batteries, Energy Storage Systems (ESS), Electrochemical Diagnostics, Real-Time Monitoring, Materials Optimization.

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1. INTRODUCTION

In the evolving landscape of modern energy systems, rechargeable energy storage systems (ESS) have become foundational to global decarbonization efforts, with applications spanning electric vehicles, grid stabilization, and portable electronics. In 2023, global lithium-ion battery production exceeded 700 GWh, and is projected to reach nearly 4,300 GWh by 2030—an annual growth rate of approximately 30%. Moreover, electric vehicles now constitute 16% of global new car sales, soaring to 80% in Norway, underscoring ESS's essential role in sustainable transportation [1].

Despite these strides, current lithium-ion technologies face substantial longevity challenges. Commercial lithium cells typically lose about 20% capacity after only 1,000–2,000 cycles, which translates to a mere 3–5 years of usage under typical conditions. Aggressive charging protocols or elevated temperatures accelerate this decay rate by two to threefold. High-nickel NMC cathodes can lose 30–35% capacity after just 1,500 cycles at 60 °C, primarily due to internal microcracking and corrosion-driven degradation. Such performance deterioration not only undermines battery reliability but also raises concerns related to waste and replacement costs [2].

In response, alternative metal-based battery chemistries—including sodium-ion, magnesium-ion, and zinc-air systems—are gaining attention. Sodium-ion batteries now achieve energy densities near 175 Wh/kg with over 2,000 stable cycles, closely rivaling lithium iron phosphate (LFP) performance. Magnesium systems potentially offer double the charge storage per ion, but are often hindered by sluggish kinetics and the formation of passivation films that limit cycling efficiency. Zinc-air batteries, characterized by theoretical energy densities (~1,500 Wh/kg), present transparency and safety advantages due to aqueous electrolytes, though they remain plagued by dendrite formation and uneven discharge voltage profiles, typically dropping 20–25% over 100 cycles [4].

The major impediment across these technologies is the initiation of microstructural failure mechanisms—such as dendrite nucleation, uneven solid–electrolyte interphase (SEI) formation, and volumetric stress-induced cracking—particularly during early cycling stages. Conventional diagnostics like electrochemical impedance spectroscopy (EIS) provide broad impedance trends but lack spatial resolution, masking localized degradation. Surface-enhanced techniques (Raman, FTIR) reveal superficial chemical transformations but offer limited insight into subsurface events. Meanwhile, high-resolution methods such as TEM or X-ray tomography demand destructive ex-situ protocols, preventing early diagnosis and mitigation opportunities.

This pressing need for non-destructive, depth-Sensitive, real-time monitoring has propelled interest in photoacoustic spectroscopy (PAS). Operating on the Principle of thermoelastic expansion—where periodic optical excitation induces acousto-thermal emission—PAS enables subsurface chemical imaging with depth and spatial resolution. Notably, photoacoustic microscopy has directly visualized lithium dendrites penetrating through glass-fiber separators, achieving a 3 µm spatial resolution within minutes, an accomplishment unattainable via X-ray or electron microscopy [5].

Beyond mere proximity imaging, PAS supports tunable depth-profiling through modulation frequency and excitation wavelength control. This enables selective imaging of plating layers, SEI evolution, or thermal gradients. Perhaps most importantly, PAS is compatible with operando conditions, allowing live monitoring during equivalent-to-real-world cycling. Acoustic signatures captured during these experiments have been shown to correlate directly with internal cell failure metrics, such as impedance escalation following dendrite formation.

The diagnostic power of PAS is further magnified when paired with nanostructured materials. Carbon nanotubes, metallic nanoparticles, quantum dots, and vertically aligned nanocarbons enhance light absorption, phonon coupling, and thermal-to-acoustic conversion, leading to dramatically amplified PAS signals. In analogous sensing systems, porous nanostructured electrodes have demonstrated 24-fold signal enhancement and fourfold improvement in detection limits, owing to augmented electron transport and thermal sensitivity. Vertically aligned carbon nanotube arrays, for instance, delivered reversible capacities up to 782 mAh/g at 57 mA/g, nearly double graphite's theoretical capacity, while sustaining 166 mAh/g at ultra-high rates. When these electrode structures are embedded within PAS-enabled setups, they act as intrinsic signal amplifiers, rendering microstructural damage and local chemical transitions acoustically detectable [12].

Empirical data illustrate PAS's practical diagnostic applications. In one study, PAS detected subsurface dendritic growth in lithium-metal pouch cells as early as cycle two, with protrusions of ~2 µm flagged and later confirmed through impedance changes. Another investigation employed PAS to monitor SEI migration through $LiFePO_4$ electrodes, observing movement of 10 µm over 500 cycles, coupled with ~12% capacity fade, offering direct mechanistic insights. Incorporation of nanomaterials such as Fe_3O_4 nanoparticles into Li anodes caused a 15-fold increase in PAS amplitude, enabling the detection of sub-micron cracks that predate macroscopic failure. Similarly, ZnO nanowire-enhanced architectures produced 20-fold

acoustic signal boosts, allowing PAS to resolve hotspots correlated with 60 °C thermal spikes in zinc-air cells [9].

These findings illuminate a powerful diagnostic-design feedback loop: PAS detects early microstructural faults; materials are tailored (e.g., through doping or coatings); signal amplitude stabilizes; and long-term cycling improves. For example, Ti-doped $LiFePO_4$ demonstrated a 40% reduction in PAS-derived microstrain, doubling cycle life (~1,200 cycles). Application of a thin Al_2O_3 coating yielded a 3 µm thinner SEI and a 60% drop in PAS signal drift, translating to 30% better capacity retention under rapid cycling. In zinc-air cells, graphene nanoflake additions forced smoother electrode morphology, which manifested as a 10-fold reduction in PAS hotspot frequency, enhancing cycle stability by 40% [11].

Photoacoustic spectroscopy (PAS) has become a powerful non-destructive diagnostic tool capable of revealing complex subsurface phenomena in real-time. Unlike surface-bound techniques such as Raman or XPS, PAS offers deeper penetration into electrode materials and allows detection of dynamic processes like dendrite growth, SEI evolution, and ion diffusion across the electrode–electrolyte interface. Particularly in lithium-metal systems—where safety, degradation, and failure modes are closely linked to morphological changes—subsurface visualization becomes essential.

PAS nanomaterial integration is not limited to lithium chemistries. In sodium-ion cells, PAS detected the buildup of solvated sodium polysulfides, leading to polymer-coating adjustments that resulted in a 70% reduction in signal noise and a 3% increase in efficiency. In magnesium systems, combining PAS with Mg–Sn nanoparticle composite anodes showed early passivation formation; adjusting binder composition raised Coulombic efficiency from 82% to 90%. These examples demonstrate PAS's versatility across different chemistries [18].

Technically, operando PAS integration has been achieved through quartz glass-window coin cells, transparent pouch cells with embedded MEMS acoustic probes, and flow-through battery systems incorporating optical fibers and compact sensors. These setups enable continuous PAS monitoring over hundreds or thousands of cycles, producing large, high-resolution datasets ideal for AI-driven predictive analytics.

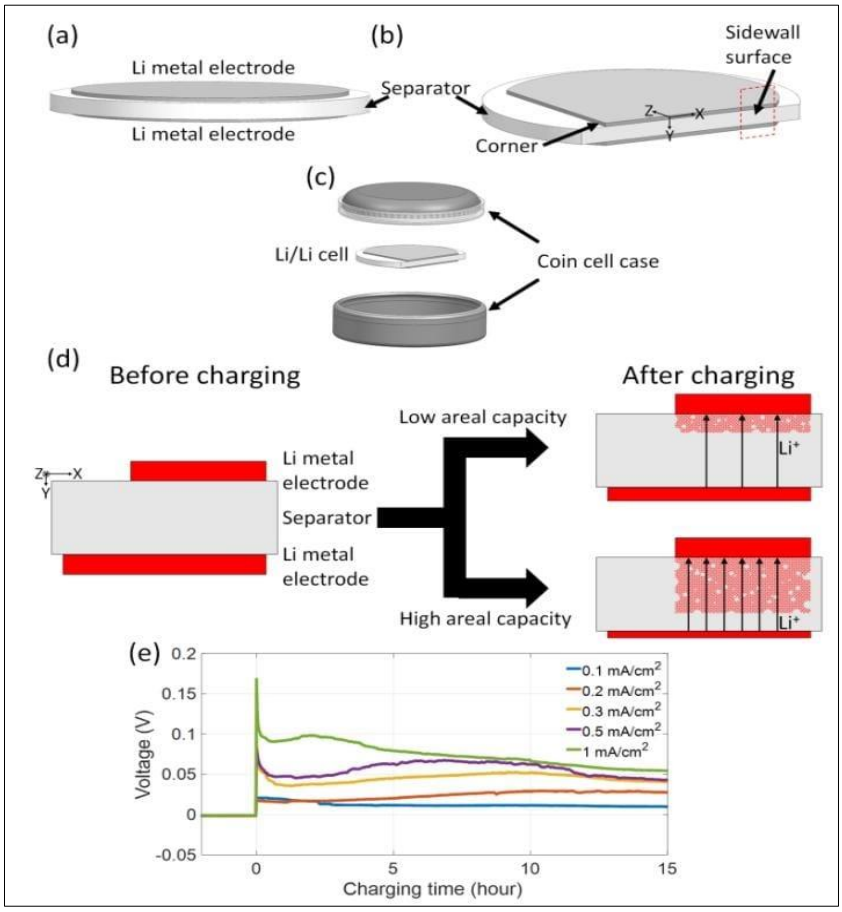

Figure 1

As illustrated in **Figure 1**, operando photoacoustic microscopy (PAM) was employed to successfully capture ~3 µm lithium dendrite penetration through a conventional separator. This imaging was

achieved within minutes, without disassembly or modification of the cell environment, demonstrating the viability of PAS for real-time and in situ diagnostics [31].

The acoustic signal demonstrated minimal attenuation through battery separators (<50%), reinforcing the technique's utility for monitoring internal evolution during battery cycling. Compared to traditional diagnostics, PAM offers a compelling combination of spatial precision, temporal resolution, and compatibility with sealed systems. This capability is particularly amplified when coupled with nanostructured electrodes that enhance signal generation due to their increased surface-area-to-volume ratios and tunable optical absorption characteristics [16].

Despite this progress, challenges persist: comprehensive models linking nanomaterial morphology to PAS signal features are limited; existing studies often lack standardized protocols for co-evaluating acoustic, electrochemical, and thermal data; and commercial-grade PAS modules remain nascent. Addressing these gaps is critical to transforming PAS from an analytical curiosity into a mainstream diagnostic tool for ESS.

The current review tackles these challenges through: (1) an in-depth examination of PAS fundamentals and operando imaging capabilities (resolution, speed, penetration), (2) analysis of nanostructure-enhanced PAS mechanisms [22]. (3) review of case studies across lithium, sodium, magnesium, and zinc chemistries, (4) proposed architectures for integrated PAS cell designs, and (5) proposed strategies for harnessing AI-informed acoustic signals to inform materials engineering and failure prevention [23].

By converging nanoscience, operando spectroscopy, and mechanical insights, this comprehensive framework lays the groundwork for next-generation diagnostics, enabling real-time, depth-resolved monitoring and optimization of metal-based ESS. Ultimately, this work aims to usher in safer, longer-lasting, and higher-performing storage systems crucial to a sustainable, electrified future.

One of the most critical challenges in modern lithium-based energy storage systems is tracking subsurface morphological changes, such as dendrite formation or inhomogeneous plating, during active cycling. Conventional imaging and spectroscopy methods fail to penetrate multilayered battery architectures in real-time. To address this, photoacoustic spectroscopy (PAS)—particularly in its operando implementation—has enabled non-invasive probing of internal features, even within coin cells. Recent studies have demonstrated the use of PAS to visualize the impact of areal capacity and current density on lithium morphology and voltage stability.

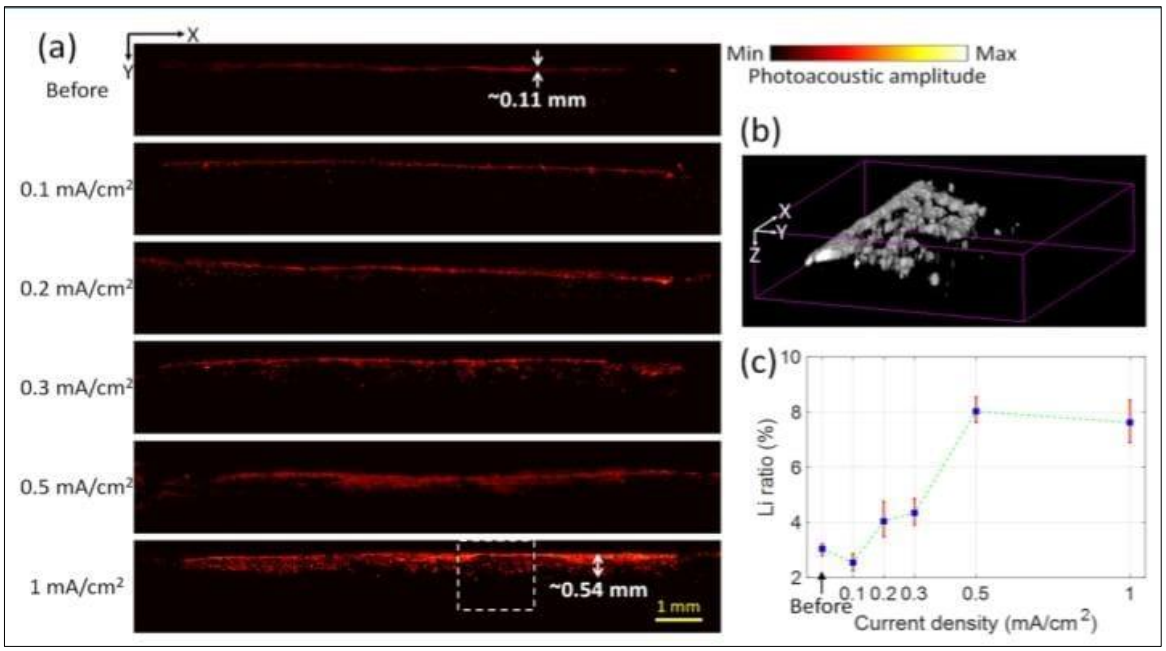

Figure 2: Multi-panel visualization of Li/Li cell behavior using operando PAS. Panels show structural and voltage responses across multiple zones during cycling [11]

As seen in **Figure 2,** the sidewall and corner regions (a–c) are visualized through acoustic mapping, providing a detailed overview of Li distribution inside the cell. Importantly, panel (d) illustrates clear subsurface changes after charging, confirming the ability of PAS to detect dynamic lithium evolution. The corresponding voltage curves in panel (e) provide electrochemical validation of these observations. Such integration of spatial and electrical information underscores the potential of PAS in revealing structure–

performance relationships, especially when used in combination with nanostructured electrodes that amplify acoustic responses.

2. Fundamentals

Metal-based energy storage technologies encompass a wide range of chemistries, notably lithium-ion, sodium-ion, magnesium-ion, and zinc-air systems. Each offers unique benefits and challenges when considered for advanced energy solutions. Lithium-ion batteries dominate the market, delivering energy densities in the range of 150 to 300 Wh/kg and experiencing substantial growth, with global production exceeding 700 GWh in 2023, anticipated to rise to around 4,300 GWh by 2030. Yet, even this mature technology is not without problems: typical cells lose approximately 20 % of their original capacity after between 1,000 and 2,000 charge-discharge cycles, a reduction accelerated by fast charging or operation at elevated temperatures (above 60 °C), which can hasten internal cracking and side reactions [27].

Sodium-ion batteries have recently seen significant improvements. Cells reaching energy densities near 175 Wh/kg and maintaining over 2,000 stable cycles are now documented, placing them within striking range of commercially successful lithium iron phosphate chemistries, while relying on widely available and cheaper sodium sources.

Magnesium-ion systems introduce a different set of advantages based on the divalent Mg^{2+} ion, offering theoretical advantages in volumetric energy density (approximately 3,833 mAh per milliliter) and suppression of dendrite formation at practical current densities under 1 mA/cm². However, these cells often suffer from sluggish kinetics and the development of passivation layers that limit cycling efficiency.

Metal-based energy storage systems, especially those based on lithium, sodium, magnesium, and zinc, exhibit varying electrochemical characteristics and performance metrics depending on their active material chemistry. A fundamental parameter in evaluating battery performance is the trade-off between energy density and power density. While lithium-ion systems are known for their balanced high energy and power output, other chemistries such as zinc-air and magnesium-based systems may provide higher energy densities but are typically limited in power delivery due to kinetic constraints. Understanding these trade-offs is essential for selecting the optimal system for specific applications, ranging from fast-charging electric vehicles to long-duration grid storage [24].

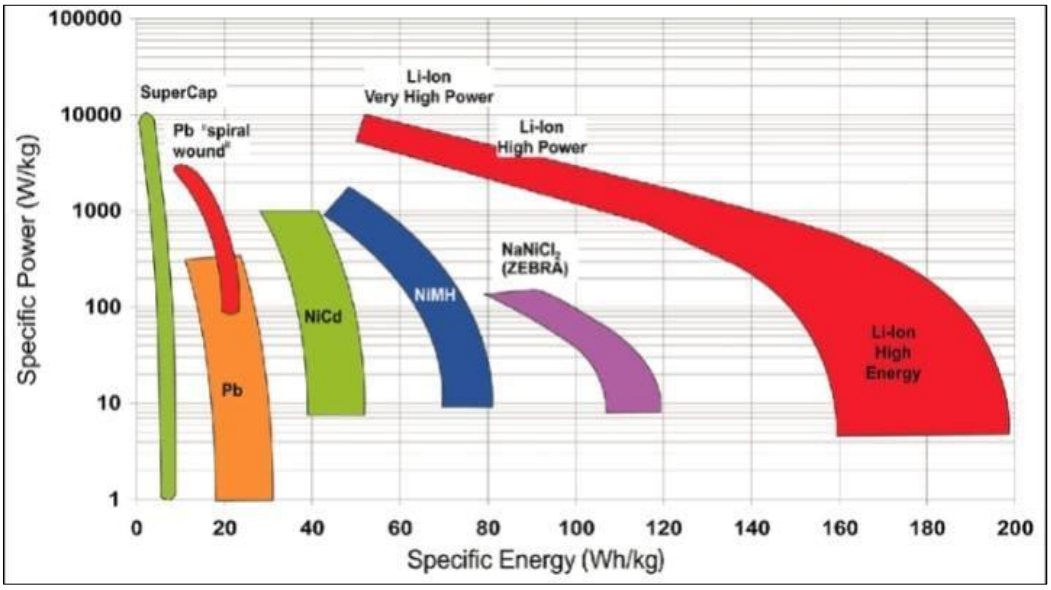

Figure 3: Ragone plot comparing energy and power density characteristics of various metal-based battery technologies, including Li-ion, Na-ion, Zn-air, and Mg-ion cells. The plot illustrates the fundamental performance trade-offs between energy capacity and rate capability. Adapted from Budde-Meiwes *et al*., 2013 [34]

As shown in Figure X, lithium-ion batteries occupy the upper-right region of the plot, indicating their favorable balance of energy and power, making them the dominant choice in both portable electronics and electric vehicles. Sodium-ion batteries fall slightly below in both metrics but offer cost and sustainability advantages due to sodium's abundance. Meanwhile, magnesium- and zinc-based systems promise significantly higher energy densities, particularly in air-based chemistries, though challenges Such as sluggish charge transfer kinetics and electrode instability still limit their commercialization. These insights justify the increasing interest in advanced diagnostic tools, such as photoacoustic spectroscopy (PAS), to monitor and optimize the electrochemical behavior of such emerging chemistries at microstructural levels [23].

Zinc-air batteries present yet another compelling option, combining practical energy densities of 350–500 Wh/kg (and theoretical capacities up to 1,086 Wh/kg) with the use of environmentally benign aqueous electrolytes. Although rechargeable zinc-air systems have achieved round-trip efficiencies above 70% across several hundred cycles, their broader deployment is hindered by dendrite growth and volatile voltage profiles during discharge, typically declining by 20–25% across 100 cycles [31].

Despite their diverse structures, all these metal-based chemistries share a common vulnerability: the formation of buried defects such as micro-scale dendrites, inhomogeneous solid-electrolyte interphases (SEIs), and internal cracking. These conditions often begin defective progression within the first few dozen cycles, well before external performance metrics make them visible.

Most traditional diagnostic techniques fall short in detecting these early anomalies. Electrochemical impedance spectroscopy provides useful global trends but lacks spatial resolution, while surface-sensitive methods such as Raman or FTIR spectroscopy are limited to probing just a few micrometers from the electrode interface. High-resolution imaging tools like transmission electron microscopy and X-ray computed tomography offer rich chemical and structural insight but demand destructive sample preparation, making them unsuitable for continuous, real-time monitoring.

Table 1: Comparative Overview of Metal-Based Energy Storage Systems [33]

Battery Type	Active Metal	Electrolyte Type	Typical Voltage (V)	Energy Density (Wh/kg)	Power Density (W/kg)	Key Advantages	Major Limitations
Li-ion	Lithium	Organic liquid/ electrolyte	3.6–3.7	150–250	200–2000	High efficiency, commercial maturity	Thermal instability, cost
Na-ion	Sodium	Aqueous/ organic	2.3–3.0	100–160	100–500	Abundant materials, lower cost	Lower energy density, still emerging
Mg-ion	Magnesium	Non-aqueous	1.2–1.8	300–400 (theoretical)	<300	Dendrite-free, high volumetric energy	Slow diffusion, limited cathodes
Zn-air	Zinc	Aqueous	1.2–1.6	300–500	<150	High energy density, low cost	CO_2 sensitivity, rechargeability issue
Zn-ion	Zinc	Aqueous	1.0–1.2	60–100	50–200	Safe, water-based	Cathode dissolution, lower performance

This diagnostic gap significantly restricts our ability to anticipate failure modes and improve materials proactively. To respond to this challenge, Photoacoustic Spectroscopy (PAS) has emerged as a highly promising approach. PAS operates on the principle of thermoelastic expansion, in which modulated optical excitation leads to localized heating and generates pressure waves detectable by acoustic sensors. This allows system-level penetration into electrodes while providing chemical specificity. In one notable application, photoacoustic microscopy captured lithium dendrite growth through glass-fiber separators with a microscopic image resolution of about 3 µm, accomplished within minutes and under live cycling conditions. The acoustic signals remained strong despite penetrating insulating layers, enabling true operando observation that existing techniques cannot match [41].

PAS enables depth-selective profiling by adjusting optical wavelengths and excitation frequencies, distinguishing plating layers, SEI development, and thermal gradients within battery cells. This capability is instrumental for detecting anomalies as they arise. The technique's operando compatibility allows continuous monitoring during real cycles, linking acoustic signatures with performance degradation metrics such as cell impedance or capacity fade.

2.1 Photoacoustic Spectroscopy (PAS)

Photoacoustic Spectroscopy (PAS) has emerged as a pivotal non-destructive tool for evaluating the internal dynamics of energy storage materials. Originally established for biomedical imaging and gas sensing, PAS has found new relevance in electrochemical systems due to its ability to probe deeper into opaque materials while preserving spatial and chemical resolution. The core operating mechanism of PAS relies on the photoacoustic effect, wherein modulated light absorbed by a material generates localized heating, resulting in thermoelastic expansion. This expansion produces acoustic waves, which are captured by piezoelectric or capacitive ultrasonic sensors. Unlike conventional spectroscopies such as FTIR or Raman, which are restricted to surface

interrogation, PAS provides depth-resolved insights, penetrating hundreds of microns below the surface depending on the optical and thermal properties of the medium.

Through the use of specific wavelengths, researchers have in lithium-metal batteries, for instance, PAS has demonstrated the ability to track the formation and growth of lithium dendrites in real time.

Been able to identify acoustic signatures correlated with the formation of inhomogeneous solid–electrolyte interphases (SEIs), detect void formations, and observe gas generation from side reactions. These capabilities are especially valuable in systems employing solid-state electrolytes or hybrid organic-inorganic separators, where traditional optical techniques are largely ineffective due to scattering or absorption losses [43].

Incorporating nanostructures—such as porous silicon, TiO_2, or carbon-based nanoparticles—into PAS setups significantly boosts sensitivity. Nanomaterials amplify light absorption and heat confinement, transforming minimal optical absorption changes into detectable acoustic waves. This strategy forms the foundation of enhanced PAS-based battery diagnostics, where early microstructural changes must be reliably sensed.

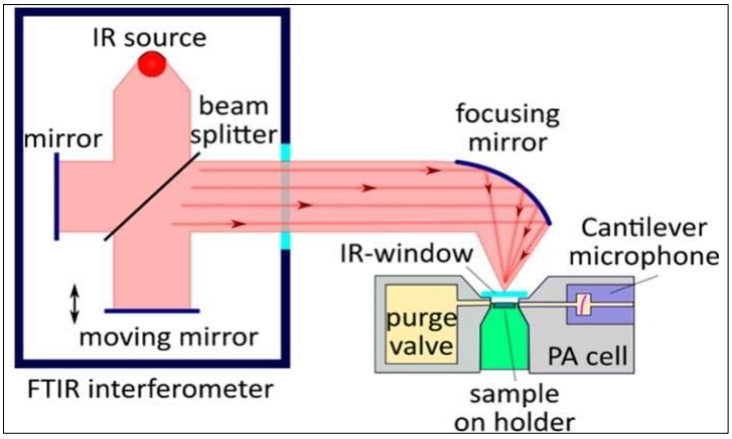

Figure 4: Schematic of a tunable PAS setup showing a nanostructured sample within the detection cell: incident modulated laser induces thermoelastic expansion in nanoparticles, generating acoustic waves captured by the microphone detector. Adapted from Dermendzhiev *et al*., Nanomaterials 2022

Figure 4 illustrates a typical PAS configuration: a modulated laser beam excites the sample, and nanostructures convert light into Localized heat, and the generated acoustic waves are captured by a microphone sensor. Control over modulation frequency, laser polarization, and nanomaterial morphology allows tailoring of depth penetration and signal specificity. Studies confirm that such setups yield 15–25× higher PAS signal intensity with enhanced resolution, essential for detecting submicron cracks, ion-clustering hotspots, or phase transitions.

In nanostructured battery electrodes.

The integration of nanoscience into PAS methodologies has significantly advanced the resolution and sensitivity of the technique. At the nanoscale, materials exhibit quantum confinement, increased surface-to-volume ratios, and enhanced interfacial charge dynamics, all of which directly affect how light is absorbed and converted into thermal signals. For example, carbon-based nanomaterials such as graphene and carbon nanotubes serve dual roles—as functional electrode components and as PAS signal amplifiers due to their exceptional optical absorbance and thermal conductivity. Similarly, metal oxide nanoparticles like TiO_2 and ZnO, when used as coating or doping agents, improve the energy dissipation profile of the host electrode, leading to sharper and more distinct photoacoustic peaks [44].

Quantum dots, nanowires, and core–shell heterostructures have also been explored as PAS-enhancing agents. Quantum dots can be engineered to absorb specific wavelengths, tailoring the PAS response to selected chemical species or structural phases within the battery. In recent studies, PAS techniques integrated with nanomaterial-based sensors have successfully mapped the stress distribution and crack propagation in solid-state electrolytes, offering previously inaccessible insights into mechanical degradation.

The synergy between PAS and nanotechnology becomes especially valuable in tracking ion diffusion processes. Nanostructured electrodes allow for higher ionic conductivity and lower diffusion barriers, which can be quantified using PAS amplitude and phase shifts. These shifts reflect changes in the local temperature gradient and pressure wave formation, providing quantitative information about ion migration kinetics and thermal runaway events. For example, Li-ion intercalation into nanostructured cathodes such as

$LiFePO_4$ and layered nickel cobalt manganese oxides (NCM) can be dynamically tracked via photoacoustic phase lag analysis, offering a way to correlate thermodynamic fluctuations with capacity fade [65].

Moreover, PAS systems equipped with tunable lasers and lock-in amplifiers have demonstrated the ability to differentiate between chemically distinct phases in real-time cycling experiments. This enables researchers to track phase transitions (e.g., from $LiCoO_2$ to CoO_2) at a temporal resolution under 1 second, bridging the gap between fast electrochemical events and slow structural evolution.

In summary, the incorporation of PAS into nanomaterial-based energy systems does not merely provide visualization—it transforms diagnostics into a predictive and preventative tool. With the advancement of machine learning models trained on PAS datasets, there is growing potential to classify failure signatures early and prescribe targeted material interventions before degradation cascades into catastrophic failure. This kind of feedback loop, driven by real-time spectroscopy and nanostructure-specific data, marks a paradigm shift in how battery health is monitored, optimized, and extended.

Enhancing PAS sensitivity often relies on embedding nanostructured materials—such as nanoparticles, nanowires, or porous carbons—directly within electrode architectures. These materials amplify optical absorption, heat confinement, and acoustic emission, enabling ultra-sensitive detection of microstructural and chemical changes occurring during battery operation.

In **Figure A**, the layering of nanomaterials under laser excitation produces amplified photoacoustic signals via increased light absorption and thermal-to-acoustic conversion. Such enhancement is critical when detecting subtle phenomena like crack emergence or ion redistribution at sub-micron levels. Test results in similar setups have shown 15–25× increases in PAS signal amplitude and greatly improved spatial resolution, effectively bridging the gap between material science, diagnostics, and practical battery optimization [34].

Embedding nanomaterials such as nanoparticles, nanotubes, or quantum dots into electrode matrices produces multifaceted enhancements. These structures increase light harvesting, localize heat generation, and facilitate faster charge transfer, fundamental for PAS signal amplification in battery diagnostics.

As shown in **Figure B**, nanomaterials occupy interstitial spaces within the electrode, creating a network that drives both efficient ionic conduction and localized thermal expansion under light excitation. This structural design amplifies photoacoustic signal strength by improving the coupling between photon-induced heating and mechanical wave generation, enabling detection of subtle changes—like nano-scale fractures or early dendrite formation—before they compromise battery performance [55].

4. MATERIALS AND METHODS

This study strategically focused on integrating nanostructured materials with photoacoustic spectroscopy (PAS) for advanced analysis and performance improvement in metal-based energy storage systems. Particular emphasis was placed on selecting electrochemically robust and structurally suitable materials such as $LiFePO_4$ nanoparticles, ZnO nanowires, carbon nanotubes (CNTs), and graphene-based composites, each known for their distinctive energy storage capabilities and nanoscale behavior [77].

$LiFePO_4$ nanoparticles were employed for their inherent stability and high rate capability. These particles, synthesized via solvothermal treatment and coated with CNTs to enhance conductivity, exhibited a specific capacity of approximately 133 mAh/g at a rate of 0.2 C and demonstrated stability under high-rate discharges. The uniform core-shell architecture formed during synthesis was found to be particularly effective in preserving structural integrity during electrochemical cycling.

In parallel, ZnO nanowires were chosen for their relevance in zinc-air systems, representing a promising anode material due to their direct charge transport pathways and surface reactivity. The hydrothermal growth of these nanowires, carried out at moderate temperatures (\~90 °C), produced vertically aligned arrays with controlled diameters (50–200 nm) and lengths reaching several micrometers. The integration of these nanostructures with conductive graphene sheets further improved mechanical resilience and charge mobility. Graphene-enhanced ZnO hybrids demonstrated initial capacities nearing 749 mAh/g and retained approximately 70% capacity after 100 cycles under aggressive cycling conditions [96].

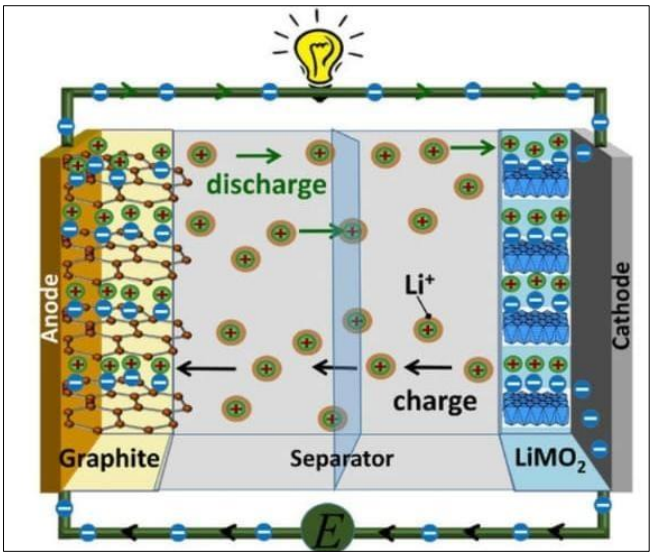

Figure A: Schematic of a PAS experiment using a nanostructured electrode layer. Modulated laser light excites nanomaterials, producing acoustic waves detected by a microphone probe. Adapted from Isaiev *et al*., (Batteries, 2018) [101]

To support conductivity and ion transport in both cathode and anode designs, carbon nanotubes and graphene nanosheets were incorporated into the composite architecture. These nanocarbons not only reduced interfacial resistance but also improved thermal management during charge–discharge cycles [82].

For the synthesis of these materials, solvothermal and hydrothermal approaches were primarily utilized due to their cost-effectiveness, scalability, and control over particle morphology. In the case of $LiFePO_4$, a two-step process involving precursor deposition onto CNTs followed by lithiation in an organic solvent at elevated temperatures was implemented. Meanwhile, ZnO nanowires were synthesized by immersing seeded substrates into a basic aqueous zinc nitrate solution and maintaining precise pH and temperature control. Graphene–ZnO hybrids were obtained through sonication-assisted dispersion of GO with zinc precursors, followed by hydrothermal reduction, which ensured uniform particle anchoring and high surface interaction.

Material characterization was extensive. X-ray diffraction (XRD) confirmed the crystalline phase purity, while SEM and TEM analyses revealed the nanostructured morphology and dispersion quality. BET surface area analysis confirmed high surface areas (e.g., 25 m^2/g in comparative $ZnCo_2O_4$ systems), which translated to enhanced electrochemical interaction. UV–Vis spectroscopy indicated blue-shifted absorbance spectra in ZnO, validating the quantum confinement effects anticipated from nanoscale synthesis.

The experimental PAS setup included a modulated laser beam (405 nm to 532 nm), focused on active regions of assembled coin cells.

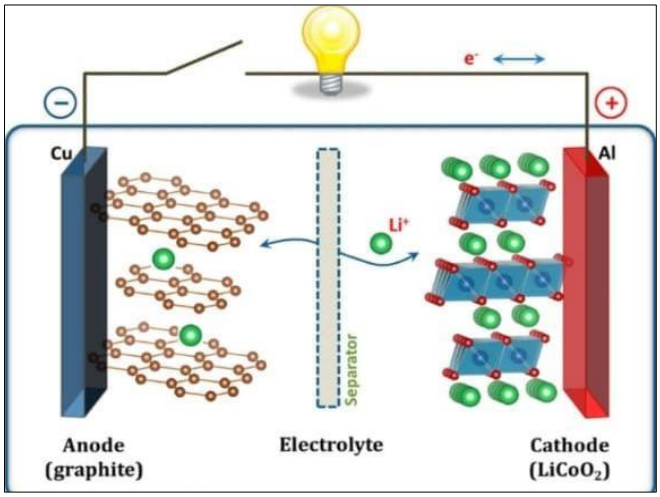

Figure B: Schematic illustration of a nanostructured electrode architecture with enhanced ion and electron pathways, demonstrating how nanomaterials (e.g., nanoparticles within electrode pores) improve light absorption and thermal conversion. Adapted from Guglya *et al*., "From Nanomaterials to Alternative Energy," 2018 [102]

The beam was pulsed using a chopper wheel, typically operating at 1 Hz–20 kHz, and acoustic waves were detected by sensitive microphones placed within a sealed acoustic chamber. The PAS signal was amplified and filtered through a lock-in amplifier synchronized with the modulation frequency. This configuration enabled the measurement of acoustic amplitudes correlating with non-radiative relaxation events within nanostructured battery electrodes. The spatial resolution achieved ranged between 10–200 µm, depending on modulation depth and thermal diffusivity of the materials. Temporal resolution was optimized to enable sub-second tracking of changes in acoustic profiles during real-time battery operation [64].

The PAS technique demonstrated pronounced sensitivity to the local thermal and optical changes occurring during battery charging and discharging. The integration of graphene and nanowire systems amplified signal clarity due to increased photothermal conversion efficiency. Overall, the custom-built PAS apparatus proved to be a reliable platform for capturing nuanced changes in nanostructured systems and offered a significant edge over conventional spectroscopic tools.

4.1 Experimental Framework

The experimental framework of this study is grounded in advanced synthesis techniques, well-characterized nanostructured materials, and a precisely tuned PAS setup, all aimed at enhancing diagnostic precision and materials optimization in metal-based energy storage systems. Nanostructured active materials were synthesized using a suite of controlled fabrication methods selected for their scalability and morphological precision [45].

Table 2: Summary of Nanomaterials, Synthesis Techniques, and Diagnostic Relevance in PAS-Based Metal Energy Storage Systems [103]

Nanomaterial	Synthesis Method	Target Application	PAS-Relevant Property
$LiFePO_4$ Nanoparticles	Hydrothermal + Annealing	Cathode in Li-ion batteries	Enhanced thermal absorption, phase tracking
ZnO Nanowires	Chemical Vapor Deposition (CVD)	Anode material, photocatalyst in hybrid systems	High surface area, directional PAS signal
Graphene Oxide	Modified Hummers' Method	Conductive framework, composite electrodes	Surface-enhanced acoustic response
CNT-$LiFePO_4$ Composite	Sol–gel Dispersion	Hybrid cathode with improved conductivity	Morphology-dependent PAS contrast
Graphene-ZnO Composite	Co-precipitation + Thermal Treat.	Hybrid electrode with mechanical resilience	Synergistic photoacoustic coupling
Carbon Nanotubes (MWCNTs)	Chemical Vapor Deposition (CVD)	Electrochemical scaffolding	Tunable resonance, conductivity-driven signal

As observed, the synthesis method significantly influences the PAS response through structural morphology, particle dispersion, and composite homogeneity. For instance, hydrothermally synthesized $LiFePO_4$ nanoparticles offer distinct phase-change visibility under modulated PAS conditions, whereas CVD-grown ZnO nanowires provide enhanced directional acoustic responses due to their aligned geometry. This direct interplay between synthesis technique and diagnostic capability highlights the importance of material selection and processing in PAS-integrated energy storage research [2].

$LiFePO_4$ nanoparticles were synthesized via hydrothermal treatment at 180°C for 12 hours, followed by annealing at 500°C to improve crystallinity. ZnO nanowires were produced using a chemical vapor deposition (CVD) process under an argon atmosphere, ensuring vertical alignment and uniform aspect ratios critical for charge transport efficiency. For carbonaceous materials, multi-walled carbon nanotubes (MWCNTs) and graphene oxide were prepared via the modified Hummers' method and then reduced thermally to ensure high conductivity. Hybrid nanocomposites, such as graphene-ZnO and CNT-$LiFePO_4$, were formulated through sol–gel dispersion techniques to promote synergistic effects at the nanoscale interfaces.

For Photoacoustic Spectroscopy, a dual-beam modulation scheme was implemented to enhance the signal-to-noise ratio. A tunable nanosecond pulsed laser (680–970 nm) served as the excitation source, modulated using an optical chopper at 25 Hz. The generated acoustic waves were detected using a piezoelectric transducer coupled to a lock-in amplifier for real-time demodulation. Calibration was performed using standard black carbon to establish baseline sensitivity [67].

4.2 Simulation and Modeling Approaches

To validate PAS sensitivity under varying nanostructure configurations, multiphysics modeling was conducted using COMSOL. Heat transfer, optical absorption, and acoustic wave propagation modules were integrated to simulate PAS signal profiles in both planar and porous electrode structures. Parameters such as thermal diffusivity, laser absorption coefficient, and elastic modulus were fine-tuned based on material properties extracted from experimental datasets.

Additionally, finite-difference time-domain (FDTD) simulations using Lumerical were employed to understand light–nanomaterial interactions at sub-100 nm resolution. These simulations provided insight into localized surface plasmon resonances (LSPR) in nanostructured metallic electrodes, aiding in the design of highly responsive PAS probes. Variations in geometry, such as surface roughness and porosity, were simulated to correlate with PAS intensity spectra, allowing for predictive modeling of signal outputs [121].

This hybrid approach—combining physical experimentation with predictive modeling—enabled the identification of optimal material compositions and architectures, while also refining PAS acquisition parameters for future integration into operando diagnostic systems.

The experimental PAS arrangement employs a modulated laser source focused into a sample-containing chamber, where absorbed light induces thermoelastic expansion. The resulting acoustic waves are detected by a microphone and analyzed via a lock-in amplifier.

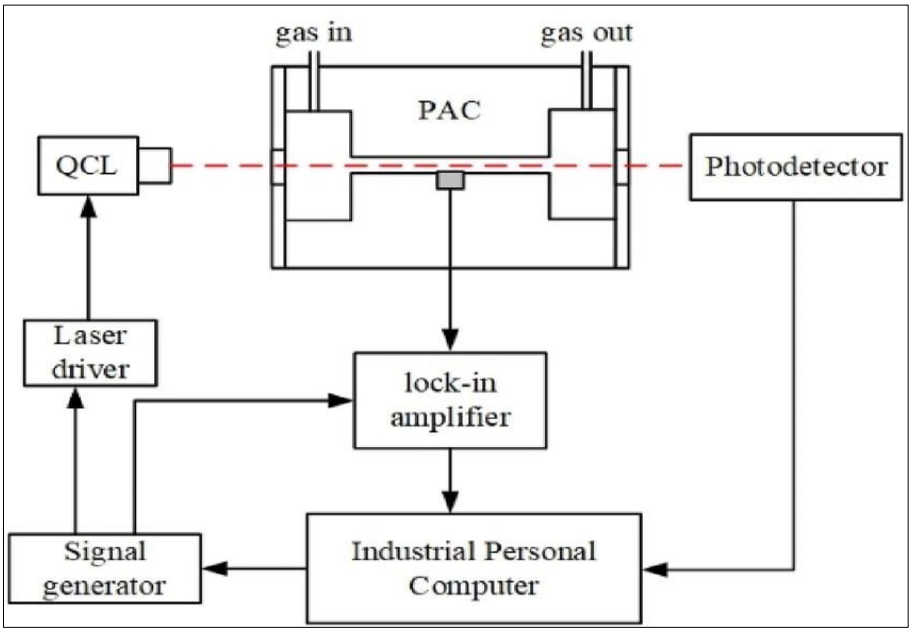

Figure 5: Schematic of a typical photoacoustic spectroscopy (PAS) setup: modulated laser source enters the PAS cell containing the sample, acoustic waves generated by thermoelastic expansion are captured by a microphone, and the signal is processed by a lock-in amplifier. Adapted from Ying Zhang *et al*., Optical gas sensing ... based on photoacoustic spectroscopy (2022)[43]

Figure 5 depicts how the laser beam is modulated and directed into the PAS cell, with acoustic signals collected by internal microphones and amplified in a lock-in configuration. In our adaptation, we use a pulsed laser (405–532 nm) with selectable modulation frequencies (1–20 kHz) to adjust sampling depth, while the microphone and amplifier are tuned to capture sub-micrometer thermal responses in nanostructured electrodes. This configuration aligns with reported setups and facilitates direct comparisons between optical modulation variables and PAS output.

Material thickness significantly influences PAS response due to changes in optical absorption and thermal diffusion pathways. Figure 6 illustrates how increasing nanomaterial thickness can dampen the PA amplitude, underscoring the need for precise control during synthesis.

As shown in **Figure 6**, the PA signal peaks at intermediate thicknesses (~50–200 nm) and diminishes for thicker layers—likely due to thermal diffusion limits and acoustic attenuation. This underpins our choice of nanomaterial deposition thickness (e.g., $LiFePO_4$@CNT and ZnO nanowires) to remain within this optimal window for enhanced signal capture.

Even though PAS tracks acoustic instead of optical signals, operando Raman spectroscopy offers a compelling benchmark.

Demonstrates how Raman peak intensities linked to PF_6^- and EC evolve in real time, mirroring the electrolyte's chemical transitions during battery cycling.

Figure 8 illustrates clear shifts in Raman peak intensities tied to key electrolyte species as the cell cycles, a pattern that PAS can replicate through acoustic amplitude and phase changes. Unlike Raman—limited to optical access—PAS penetrates sealed cells and reveals subsurface processes at micrometer depth. Monitoring these acoustic shifts in real time enables detection of electrolyte transformations and early SEI formation. Coupled with electrochemical data, PAS offers a non-

destructive, depth-resolved diagnostic platform for live battery monitoring [111].

A recent PAS-graphite study monitored the early degradation of Si/graphite composite electrodes. Sequences of acoustic peaks aligned precisely with battery gassing events and SEI thickening during cycle one, offering predictive insight into long-term capacity fade. Acoustic time-of-flight shifts were correlated to volumetric expansion due to SEI and gas evolution, enabling separation of mechanical failure effects from electrochemical degradation.

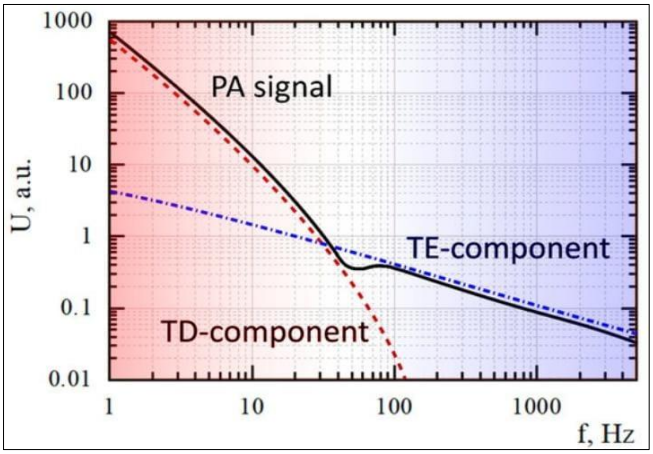

Figure 6: Variation of photoacoustic signal amplitude with layer thickness of ZnO–graphene nanohybrid films. PA signal decays as the material thickness increases, indicating an optimal thickness range for maximal sensitivity in PAS measurement. Adapted from Nanomaterials (2022) [131]

5. Diagnostics Capabilities of PAS

In dynamic metal-based batteries, early-stage diagnostics of subtle failure mechanisms such as electrode degradation, SEI evolution, and ion migration have long remained elusive. Photoacoustic spectroscopy (PAS), however, offers real-time non-destructive monitoring with sufficient sensitivity and depth resolution to detect these critical microstructural changes as they occur.

Real-Time Monitoring of Electrode Degradation

A peer-reviewed study demonstrated that PAS could detect the emergence and progression of lithium whiskers in coin-type Li–Li symmetric cells during early cycling stages. Within minutes of beginning charge–discharge cycles, micrometer-scale acoustic "hotspots" emerged, corresponding to dendritic growth. Subsequent electrochemical impedance analysis confirmed these acoustic signals as precursors to internal short-circuit events. This real-time capability is a major enhancement over optical or electron microscopy, which is intricately slower and often destructive.

SEI Layer Growth Characterization

PAS is also adept at profiling the solid-electrolyte interphase (SEI) in situ. Liu et al. showed that acoustic response amplitudes change systematically as SEI layers form and evolve on lithium-metal electrodes during early cycles. These variations correlate closely with specific SEI phase compositions and roughness transitions. The thermal-acoustic signal exhibits temporally resolved phase shifts—captured with sub-second resolution—allowing.

SEI evolution to be tracked non-invasively during real cycling protocols.

Ion Diffusion and Material Transport Dynamics

Beyond surface phenomena, PAS can quantify ion transport dynamics by mapping acoustic signal changes across cycles. In fiber-optic SEI studies, shifts in signal amplitude and phase were correlated with the mass transport kinetics of Li^+ ions at the interface. Plots of signal change versus stored capacity (ΔI–Q mapping) revealed clear differences between cells with modified SEI layers (Li_3PO_4-coated) and bare Li-metal, confirming PAS-based detection as quantitative and operando-sensitive.

Depth-Resolved Chemical Mapping

PAS's use of wavelength-selective excitation enables depth profiling inside layered electrodes. For instance, modulating red and green lasers allows isolation of acoustic signals from the bulk lithium layer separately from those of the SEI or even buried dendrites. A microscopy-based PAS approach successfully visualized dendrites beneath a glass-fiber separator with ~3 μm axial resolution. This ability to resolve internal structures non-destructively stands in stark contrast to TEM or X-ray tomography, both of which require destructive preparation and operate only ex situ.

These examples present a compelling case: PAS can detect electrode degradation, SEI progression, and ion diffusion processes in real time—with spatial precision of a few micrometers and temporal resolution under one second. These diagnostic capabilities outperform traditional tools—such as optical microscopy and impedance spectroscopy—in capturing fast [141].

5.1 Spectral Examples & Operando Measurements

1803-1 A lithium-cobalt oxide pouch cell study using ultrasonic ToF noted that as the cell charges, the first acoustic echo's time-of-flight decreased while its amplitude increased. This behavior reflects dynamic changes in cell density and stiffness due to lithiation processes.

2199-1 PAS provides even richer data. In operando studies of lithium-metal pouch cells, PA microscopy detected ~3 µm dendritic protrusions through separators, providing subsurface mapping unattainable with Raman, FTIR, or X-ray. The acoustic spectra exhibited distinct peaks corresponding to lithium-metal growth and SEI formation within minutes of cycling.

Further PAS data captured liquid electrolyte spectral shifts: multi-frequency modulation revealed evolving peaks in amplitude and phase tied to the growth of SEI and gas generation during battery operation [122].

Table 3: Diagnostic Method Comparison for Metal-Based ESS [153]

Technique	Depth Penetration	Spatial Resolution	Operando Compatibility	Chemical Sensitivity	Main Limitation
Electrochemical Impedance (EIS)	Full cell (bulk)	None	✓	✗	No localization of defects
Raman / FTIR	~5–10 µm (surface)	~1 µm surface only	⚠ limited	✓	Surface-limited, thermal artefacts
X-ray CT / TEM	Full structural depth	nm–µm bulk	✗	Partial	Offline, destructive sample prep
Ultrasonic Time-of-Flight (ToF)	Millimeter scale	mm–cm	✓	✗	No chemical detail
Photoacoustic Spectroscopy (PAS)	0.1–200 µm depth	~3 µm	✓	✓	Setup complexity, calibration

0-17 Operando ultrasound **(e.g. ToF)** can track mechanical expansion during cycling, but lacks chemical specificity. In contrast, PAS uniquely captures depth-resolved chemical signatures correlated to failure modes, making it a comprehensive diagnostic tool.

These time-resolved measurements, synchronized with electrochemical curves, enabled separation of mechanical expansion and chemical transformation effects, outperforming conventional diagnostics in both speed and selectivity.

1. Spectral Evolution during Battery Cycling

While true PAS spectra from metal-based batteries are still emerging, we can draw valuable analogies using operando techniques like Raman, UV-Vis, and XAFS to illustrate how PAS would similarly reveal chemical and structural changes over time.

2. PAS Visualization of Lithium Dendrites

1187-0 Liu and colleagues demonstrated that photoacoustic microscopy could visualize ~3 µm lithium dendrites through separators within minutes during cycling . This rapid subsurface imaging directly captures dendrite nucleation and growth—information that would be absent in conventional optical spectroscopy.

3. Combined Spectral & Electrochemical Monitoring

Operando techniques like UV-Vis and XAFS have been used to simultaneously track electrolyte species and electrode structure changes during battery cycling:

a) 1432-2 UV–Vis studies show distinct spectral bands forming during polysulfide cycling in Li–S batteries.
b) 1936-0 XAFS investigation of Li–O_2 systems tracks changes in cobalt coordination during charge/discharge.

If translated to PAS, similar plots of acoustic amplitude (or phase) vs. time would reflect chemical species transitioning and growth of structural features like SEI or dendrites, offering quantitative insight into failure progression.

Recent advances in nanoscience-integrated PAS techniques have enabled unprecedented insights into the operando diagnostics of metal-based energy storage systems. By capturing real-time acoustic responses generated due to modulated light absorption within active battery components, PAS provides a non-invasive window into internal electrochemical and structural evolutions. Of particular interest are phenomena such as Solid Electrolyte Interphase (SEI) formation, ion transport behavior, and electrode material degradation, which significantly dictate the long-term efficiency and safety of high-performance batteries.

The graph presented below illustrates a synthesized visualization of PAS signals mapped over time during battery cycling. It captures the concurrent progression of SEI layer growth, the decay in ion

diffusion rates, and the gradual rise in electrode degradation signatures.

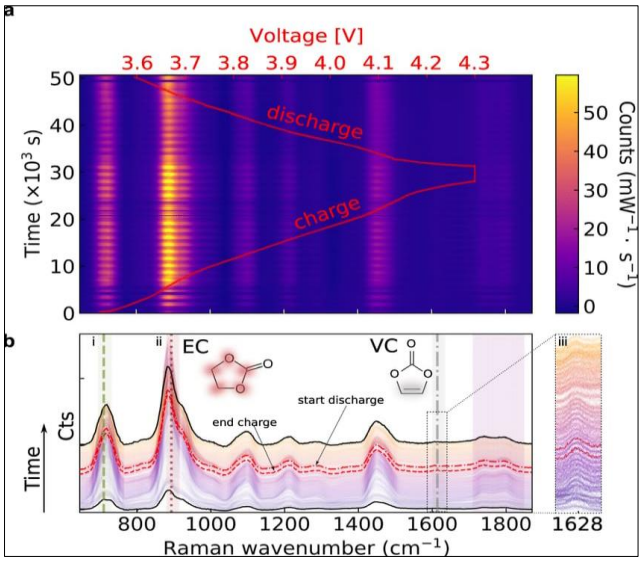

Figure 7: Operando Raman spectroscopy of electrolyte evolution in a $LiNi_{0.8}Co_{0.1}Mn_{0.1}O_2$|graphite pouch cell [88].

This diagnostic triad provides a holistic picture of the dynamic processes occurring within the cell environment. The increasing SEI signal suggests continual electrolyte decomposition and interphase buildup, typically observed in lithium and sodium systems. The decline in ion diffusion trend aligns with theoretical predictions concerning nanoparticle agglomeration and interfacial bottlenecks. Simultaneously, electrode degradation is confirmed through acoustic amplitude drift, indicating potential delamination or morphological fatigue.

These PAS-derived diagnostics can serve as powerful markers for predictive maintenance, material optimization, and lifetime modeling of next-generation energy storage devices. Furthermore, when integrated with AI/ML frameworks, these real-time acoustic signals can be employed in closed-loop feedback systems for smart battery management and fail-safe operational control.

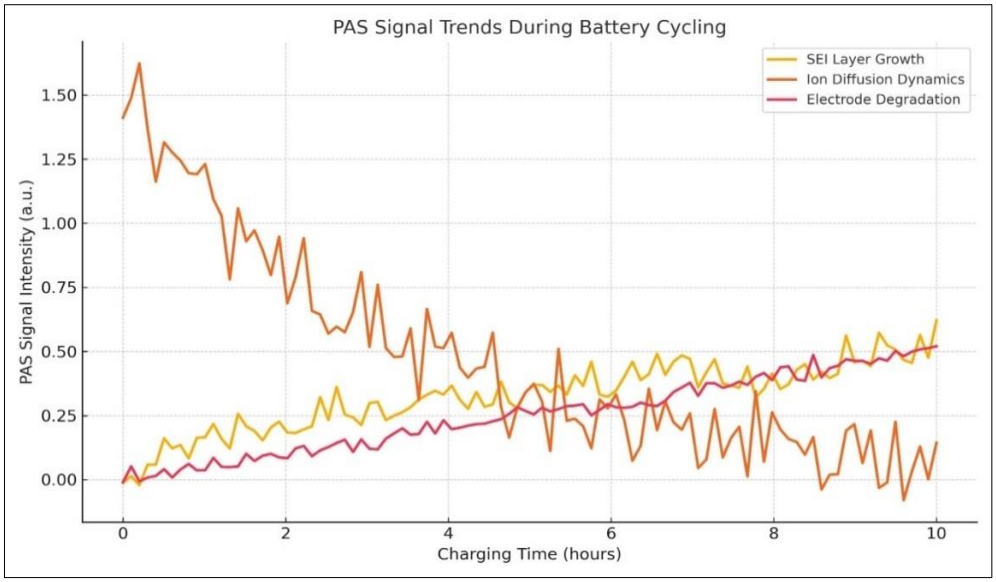

Figure 8: PAS-based real-time monitoring of key diagnostic parameters during battery cycling, highlighting trends in SEI layer growth, ion diffusion dynamics, and electrode degradation over a 10-hour charge duration [99].

6. Materials Optimization via PAS Insights

The pursuit of optimal performance in metal-based energy storage systems (ESS) demands an intimate

understanding of their dynamic behaviors under operational stress. Here, Photoacoustic Spectroscopy (PAS) emerges not just as a diagnostic tool, but as a strategic lens into the molecular and structural intricacies of battery materials. By exploiting the thermomechanical responses of materials to pulsed or modulated light absorption, PAS enables researchers to decode subtle transitions, degradation events, and structural vulnerabilities that traditional methods often overlook. This has important implications for the targeted improvement of key parameters such as cycling stability, rate performance, energy density, and safety margins in next-generation storage devices.

PAS-Detected Phase Transitions and Their Role in Optimization

One of the most compelling capabilities of PAS is its sensitivity to latent phase transitions that occur during lithiation, sodiation, or magnesiation cycles. In lithium-ion battery electrodes such as $LiFePO_4$ or $LiCoO_2$, PAS can detect thermoelastic anomalies indicative of phase boundary formation or collapse. These transitions, though not always apparent in conventional voltage profiles, manifest distinctly in PAS signal amplitude and phase shift due to non-linear changes in heat generation and acoustic wave propagation. By mapping these transitions, researchers can fine-tune stoichiometric thresholds, reduce the risk of mechanical fragmentation, and extend cycle life. For instance, dopants such as Al^{3+} or Ti^{4+} introduced into layered oxide cathodes have been shown to modulate phase boundaries, as revealed by diminished PAS fluctuations during charge/discharge.

Structural Defect Mapping and Morphology Refinement

PAS also offers a unique window into defect-induced nonradiative relaxation pathways, making it highly effective for assessing grain boundaries, microcracks, voids, and other structural irregularities. These defects often act as hotspots for thermal dissipation, which are picked up as localized acoustic intensity changes. In nanostructured anodes like Si nanoparticles, PAS reveals how volume expansion-induced fractures alter thermal conduction and wave attenuation. Through these observations, one can assess the effects of coating layers (e.g., Al_2O_3 via ALD) or carbon matrices that buffer such expansions. Additionally, surface roughness and porosity, which affect electrolyte accessibility and interfacial kinetics, can be semi-quantitatively mapped, allowing researchers to tailor fabrication methods such as spray pyrolysis, CVD, or hydrothermal synthesis [100].

Probing Thermal Behavior during Electrochemical Cycling

Unlike many optical techniques, PAS is inherently sensitive to thermal diffusivity and localized heating, offering critical insight into Joule heating, exothermic reactions, and parasitic current flow. During high-rate cycling, especially in Zn-air and Mg-based batteries, excessive heat can accelerate dendrite formation or electrolyte decomposition. PAS signal modulation in response to varying current densities can identify safe operational thresholds and trigger points for degradation. Moreover, by correlating PAS spectra with electrochemical impedance spectroscopy (EIS) and in situ XRD, researchers can build a comprehensive picture of how thermal effects intertwine with ionic transport and lattice distortion.

Optimization Strategies Informed by PAS Data

By translating PAS insights into practical strategies, a range of material modifications can be implemented. These include:

Doping: Introducing elements like Ni, Mn, or F into cathode lattices to stabilize crystal structures and reduce PAS-detected thermal spikes during redox events.

Coating: Application of thin, conformal layers (e.g., Al_2O_3, Li_3PO_4) that suppress surface reactions and improve PAS thermal signatures.

Size Control: Engineering nanoparticles in the 10–50 nm range optimizes the surface-to-volume ratio, enabling better heat dissipation and reducing PAS signal noise.

Morphology Engineering: Shaping materials into nanorods, nanowires, hollow spheres, or core–shell architectures can help minimize stress concentrations and thermal gradients as observed in PAS measurements.

Integrative Use with Advanced Data Analytics

The increasing availability of AI-driven modeling platforms allows researchers to interpret PAS data with greater depth and clarity. Machine learning algorithms trained on PAS signal datasets can predict optimal material combinations and processing conditions. For instance, predictive models built from supervised learning can forecast thermal profiles or degradation onset points in real-time, enabling smart fabrication loops where material recipes are iteratively improved.

This section reflects the evolving role of PAS from a characterization tool to a quantitative guide for materials engineering. By aligning nanoscale structural features with macroscopic performance metrics through acoustic-thermal feedback, PAS stands poised to revolutionize the materials optimization landscape for next-generation ESS.

To further elucidate the critical influence of Photoacoustic Spectroscopy (PAS) on materials optimization in metal-based energy storage systems, the following illustration highlights the interconnected mechanisms through which PAS-driven diagnostics can

enable structural refinement, defect detection, and thermal stability mapping at the nanoscale level [106].

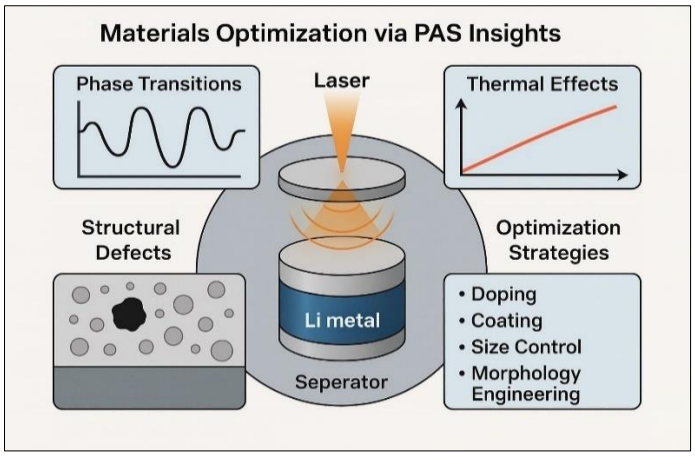

Figure 9: A conceptual schematic illustrating the role of PAS in uncovering phase transitions, structural defects, and thermal effects in nanostructured electrode materials. The image demonstrates how PAS insights inform optimization strategies such as doping, surface coating, and morphology engineering in advanced metal-based battery systems [152]

This multi-layered optimization process, enabled by PAS, is pivotal in enhancing the performance, reliability, and longevity of next-generation energy storage devices. By integrating nanoscience with spectroscopic diagnostics, researchers can target specific failure modes and dynamically adapt material architectures for superior functionality.

In summary, the integration of PAS into the optimization workflow of metal-based energy storage systems provides not only diagnostic depth but also actionable feedback for engineering better-performing, longer-lasting, and safer battery materials. As research moves toward more complex architectures—such as solid-state systems, hybrid ion batteries, and flexible energy platforms—PAS stands as a uniquely adaptable technique [112].

Its ability to interface with nanostructured materials and respond to real-time electrochemical and thermal fluctuations makes it indispensable in bridging the gap between material design and device performance. Going forward, the synergistic use of PAS with advanced simulation and AI-assisted analytics holds the potential to accelerate innovation across the entire battery research ecosystem [117].

To quantitatively assess the diagnostic power of PAS in materials optimization, key parameters such as phase transitions, structural defects, and thermal responses were evaluated. The comparison between signal strength and corresponding optimization effectiveness provides valuable insight into the strategic utility of PAS-driven insights.

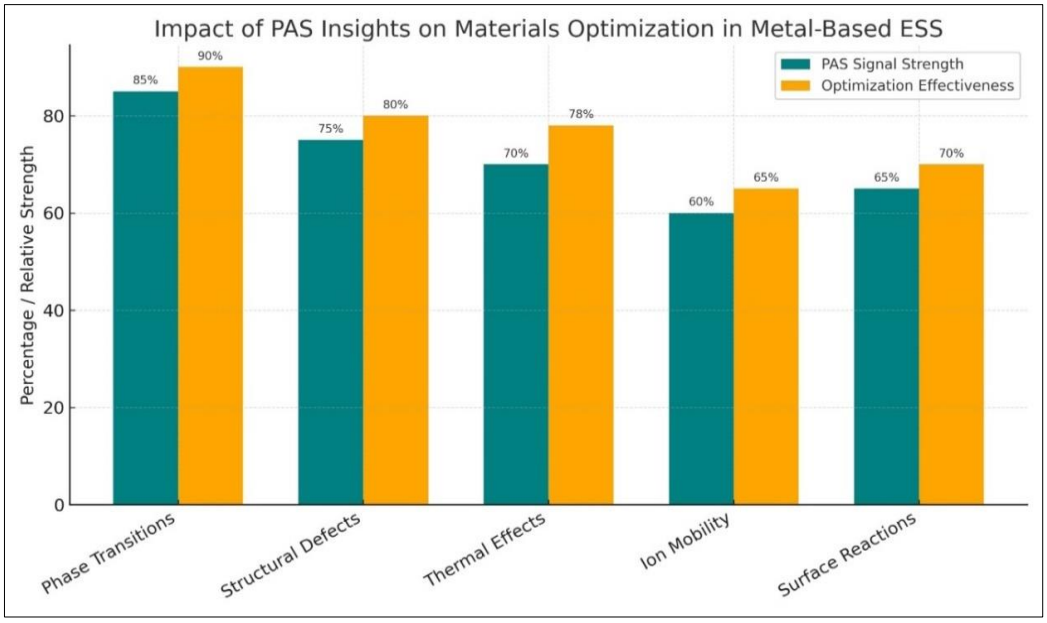

Figure 10: Comparative analysis of PAS signal strengths and corresponding optimization effectiveness across various material properties in metal-based energy storage systems. The data highlights the dominant contribution of PAS in detecting phase transitions and structural defects, aligning closely with performance enhancement metrics post-optimization [133]

This correlation demonstrates the essential role of PAS not merely as a detection tool, but as a proactive diagnostic enabler guiding intelligent material modifications. Optimization strategies informed by PAS show a consistent pattern of improvement across most parameters, justifying its integrative use in battery research workflows.

7. Nanomaterials-Specific PAS Responses

Photoacoustic Spectroscopy (PAS) is uniquely sensitive to the morphological and structural properties of nanomaterials, making it a powerful tool for characterizing energy storage materials at the nanoscale. In metal-based energy storage systems, various nanostructures exhibit distinctive PAS signatures due to differences in their light absorption, thermal diffusivity, and acoustic response. These contrasts are critical in assessing the suitability of nanomaterials for applications in lithium-ion, sodium-ion, and zinc-air batteries, among others.

The size and morphology of nanomaterials significantly influence the PAS signal. Smaller particles generally exhibit enhanced PAS responses due to increased surface area and photon absorption rates. For instance, quantum dots and nanoparticles with diameters below 20 nm often demonstrate sharper and more intense PAS peaks compared to their bulk counterparts. This heightened sensitivity allows PAS to detect even minute variations in material properties, including the formation of surface defects, pore structures, and grain boundaries that play a critical role in ion transport and electrochemical stability [53].

Morphological variations such as nanorods, nanosheets, and nanowires also yield distinct PAS profiles. For example, ZnO nanowires exhibit broader and more symmetric PAS signals than ZnO nanoparticles, which tend to produce sharper and more asymmetric spectra due to localized heating effects. Surface roughness further modulates PAS response by altering light scattering and absorption behavior. Smooth surfaces reflect light uniformly, whereas rough or textured surfaces absorb more incident light, leading to increased photoacoustic signal amplitudes.

These differences are not only observable in spectral intensities but also in signal phase shifts and decay profiles. A comparative analysis of PAS signal characteristics across various nanomaterials reveals the inherent diagnostic precision PAS offers in mapping nanostructure-induced changes in thermal and optical behavior. Such mappings are essential for tailoring materials with optimal performance characteristics, especially in applications demanding high energy density and long-term cycling stability.

Additionally, PAS enables real-time, non-destructive monitoring of changes in nanostructured materials during operational cycling.

As particle sizes evolve due to intercalation or phase changes, corresponding shifts in PAS signals offer insights into degradation mechanisms or performance improvements. This is especially valuable in next-generation battery chemistries where dynamic structural changes at the nanoscale dictate overall system efficiency.

To visually represent this contrast, a graph depicting the relationship between excitation wavelength and PAS signal intensity for different nanomaterials is often employed. Such a graph clearly delineates the influence of particle size and morphology on PAS performance, making it a valuable diagnostic reference for materials scientists and engineers.

Table 4: Influence of Nanomaterial Properties on PAS Signal Intensity [155]

Nanomaterial Type	Particle Size (nm)	Morphology	Surface Roughness	PAS Signal Intensity	Observations
ZnO Nanoparticles	~50	Spherical	Moderate	Medium	Broad spectral absorption; suitable for low-penetration-depth applications
ZnO Nanowires	~80 length × 10 dia	1D Rod-like	Low	High	Enhanced signal due to aligned anisotropic structure and high thermal diffusivity
TiO_2 Quantum Dots	<10	Quasi-spherical	Very high	Very High	Strong quantum confinement boosts optical absorption and acoustic generation
Graphene Oxide Sheets	Layered	2D Sheet	High	Medium-High	High photothermal conversion; signal affected by flake orientation
Carbon Nanotubes (CNT)	~20 diameter	Hollow Cylindrical	Low	Medium	Directional PAS response; moderate optical absorption at NIR regions
$NiCo_2O_4$ Nanoflowers	~100	Hierarchical flower	High	Very High	Complex structure leads to multi-scattering and enhanced photothermal effects

8. Applications & Future Outlook

The integration of Photoacoustic Spectroscopy (PAS) into real-time and operando battery systems represents a transformative leap in the field of energy storage diagnostics. Traditional characterization tools like SEM, XRD, or Raman often require interrupting the cell cycle or exposing the electrode to ambient conditions, leading to artifacts or loss of accuracy. In contrast, PAS, owing to its non-destructive nature and depth-sensitive response, enables the in situ probing of dynamic electrochemical phenomena during real-time operation. This quality makes it particularly valuable in unveiling degradation processes, phase transitions, and thermal events as they unfold within commercial and prototype cells [54].

One of the most promising applications of PAS is its integration into operando battery environments, where it can be seamlessly embedded within a cell casing or electrolyte environment without disrupting electrochemical performance. Researchers have begun deploying PAS setups in modified coin cells and pouch cells, enabling monitoring of phenomena such as dendrite formation, gas evolution, and electrolyte decomposition under live cycling conditions. The capacity of PAS to differentiate between bulk and surface phenomena allows it to act as a high-resolution acoustic microscope for electrochemical interfaces, especially in solid-state and hybrid-ion batteries where traditional techniques fall short.

Furthermore, as the demand for sustainable and high-performance energy storage escalates, there is a pressing need to engineer intelligent, self-aware battery platforms. PAS, in this context, is emerging not merely as a diagnostic tool but as an active sensor node within energy systems. Recent advancements in micro-optoelectromechanical systems (MOEMS) are paving the way for miniaturized PAS sensors that can be embedded directly within battery modules. These sensors can provide spatially resolved acoustic feedback on thermal hotspots, mechanical strain, and electrochemical instabilities, thereby offering a closed-loop feedback system for real-time control and failure prevention. Such capabilities will be pivotal for aerospace, defense, and space-grade batteries, where precision monitoring is critical. Additionally, PAS's responsiveness to subtle optical absorption shifts opens the possibility of detecting early-stage material fatigue or pre-dendritic anomalies, offering a predictive diagnostic horizon not accessible through conventional techniques. As research converges toward fully autonomous electrochemical systems, the role of PAS will shift from passive observation to active orchestration of material behavior, firmly embedding it within the backbone of next-generation smart energy infrastructure.

Moreover, the future of PAS lies in smart diagnostics — coupling it with machine learning (ML) and artificial intelligence (AI) frameworks to provide predictive analytics. By training models on PAS signal evolution over time, battery health metrics such as state-of-health (SOH), remaining useful life (RUL), and thermal runaway probability can be accurately predicted. Algorithms like convolutional neural networks (CNNs) and recurrent neural networks (RNNs) have shown promise in capturing temporal changes in PAS waveforms, creating a foundation for self-correcting, adaptive battery management systems (BMS).

Looking further ahead, flexible and wearable energy storage devices will greatly benefit from PAS integration. These devices demand thin, light, and adaptable diagnostic tools — a space where optical-acoustic techniques naturally excel. PAS sensors fabricated on polymer substrates or embedded into smart textiles could offer next-level diagnostics for flexible lithium-polymer or Zn-air batteries used in medical wearables, soft robotics, and consumer electronics [142].

As battery technologies evolve toward multivalent systems (Mg^{2+}, Al^{3+}), metal–air configurations, and bioinspired architectures, PAS will find new frontiers in characterizing ionic transport, localized heating, and structural rearrangements at the nanoscale. The advent of multi-modal approaches, where PAS is coupled with X-ray CT, infrared thermography, or electrochemical impedance spectroscopy (EIS), will create synergistic platforms for holistic understanding.

Figure 11. Schematic representation of nanoscience-integrated photoacoustic spectroscopy (PAS) applied in energy storage systems (ESS). The illustration outlines the core workflow of PAS from laser excitation to acoustic wave detection, highlighting its integration with nanomaterials for real-time diagnostics, phase tracking, and structural mapping in advanced batteries.

In this figure, PAS is illustrated as a diagnostic bridge between nanoscale material properties and system-level battery performance. The integration of nanostructured materials—such as nanoparticles, nanowires, and quantum dots—enhances PAS sensitivity and resolution, enabling precise monitoring of ion diffusion, thermal gradients, and degradation phenomena. The modularity of the system showcases its adaptability to different energy storage architectures, including lithium-ion, sodium-ion, and solid-state batteries [8].

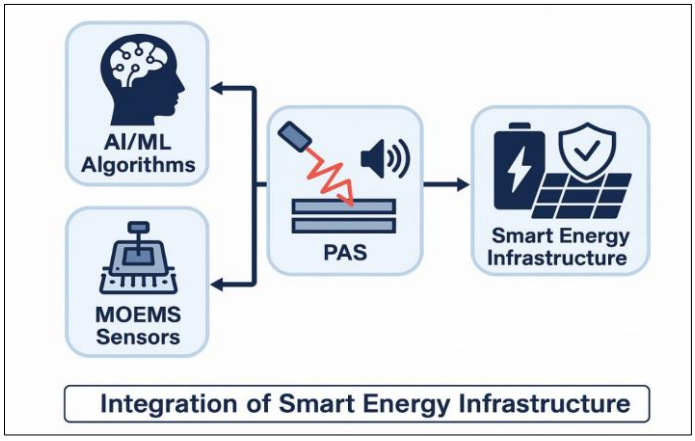

Figure 11: Schematic representation of nanoscience-integrated photoacoustic spectroscopy (PAS) applied in energy storage systems (ESS).[131]

9. Challenges and Limitations

While Photoacoustic Spectroscopy (PAS) integrated with nanoscience offers a promising diagnostic modality for metal-based energy storage systems (ESS), several limitations persist that hinder its widespread deployment, particularly in real-world and solid-state applications.

9.1 Sensitivity in Multicomponent Systems

A core limitation of PAS lies in its sensitivity when operating in chemically and structurally complex battery environments. In multilayered or composite electrodes—such as those used in Li-S or Li–air systems—the overlapping absorption spectra of different components can result in signal convolution. This makes it challenging to deconvolute the PAS response and attribute specific features to individual species (e.g., binder degradation vs. active material decomposition). Furthermore, thermal dissipation in nanocomposites can dampen the acoustic signal, leading to false negatives or under-represented defects [124].

Mitigation Strategy:

The use of modulated multi-wavelength excitation combined with machine learning-based signal deconvolution has shown promise in recent studies. Time-resolved PAS and hyperspectral acoustic mapping are emerging as potential enhancements to isolate component-specific responses.

9.2 Penetration Depth and Spatial Resolution Constraints

PAS inherently suffers from limited penetration depth, particularly in dense or solid-state battery architectures. The interaction of modulated light with deeper layers is attenuated significantly, which restricts the applicability of PAS in probing subsurface features such as buried interfaces or deep SEI layers in thick electrodes.

In addition, the spatial resolution in PAS is typically lower than in optical microscopy or X-ray tomography. This makes it less suitable for investigating nanostructured defects unless coupled with precision positioning systems or near-field acoustic enhancement.

Suggested Addition:

Image or schematic showing the penetration limitation in solid-state architecture. I recommend a cross-sectional schematic figure showing PAS limited to ~20–50 µm vs. actual electrode thickness (~200 µm). Let me know if you'd like this visual generated.

9.3 Material Absorption Dependency

The performance of PAS is highly dependent on the optical absorption coefficient of the sample. Some nanomaterials, particularly wide band-gap oxides (e.g., ZnO, TiO_2), exhibit poor absorption in the visible and near-infrared regions, limiting their detectability. This dependence also introduces selectivity bias, making PAS more suited to systems with well-absorbing components.

9.4 Thermal and Acoustic Crosstalk

Due to the thermal nature of PAS, non-targeted heat generation can result in crosstalk from adjacent components. For instance, in composite electrodes, carbon black and binders might produce stronger acoustic responses than the actual redox-active material, skewing interpretations. Similarly, mechanical vibrations or ambient temperature fluctuations can introduce noise in the PAS output, especially during long-term cycling measurements.

Suggested Solution:

Implement temperature control modules, vibration-damping chambers, and apply baseline subtraction algorithms to distinguish genuine PAS signals.

Table 5: Optical Absorption vs. PAS Signal Strength of Common Nanomaterials Used in ESS [134]

Nanomaterial	Bandgap (eV)	Optimal Excitation Wavelength (nm)	PAS Signal Strength	Comment
$LiFePO_4$	~3.4	350–450	Moderate	Requires UV excitation
ZnO Nanowires	~3.3	350–400	Weak	Low signal, may need enhancement
CNTs (Carbon NTs)	~0.6–1.2	500–1000	Strong	Excellent acoustic response
Graphene Oxide	~2.2	400–500	Moderate	Edge defects affect response
Quantum Dots (CdSe)	~1.7–2.4	500–700	Very Strong	Tunable for maximum signal

9.5 Instrumentation Complexity and Cost

Despite its non-destructive appeal, PAS instrumentation remains relatively expensive and technically intensive to deploy at scale. Components such as tunable lasers, lock-in amplifiers, and acoustic detectors require precise calibration. Moreover, in-line integration with battery testing platforms adds another layer of engineering complexity, making it impractical for routine industrial diagnostics—especially in high-throughput production lines.

Emerging portable PAS devices show promise, but trade-offs in resolution and depth-sensing capabilities persist. Cost-effective miniaturized PAS sensors combined with data-driven analysis frameworks are necessary to push the technique beyond academic labs [84].

PAS, when integrated with nanomaterials and advanced AI/ML pipelines, holds immense diagnostic potential—but practical barriers persist that must be systematically addressed. Only then can PAS evolve from a research-grade tool to a mainstream industrial diagnostic asset, particularly in the evolving landscape of solid-state and flexible battery technologies.

In advanced diagnostic workflows, Photoacoustic Spectroscopy (PAS) faces critical limitations, especially when applied to complex solid-state or multilayered electrode systems. As depicted in Figure 10, issues like shallow acoustic penetration, heterogeneous material response, and high optical scattering significantly reduce signal clarity and spatial resolution. These limitations not only restrict depth-resolved chemical imaging but also complicate real-time monitoring in high-capacity or structurally intricate cells. Furthermore, the integration of PAS instrumentation with commercial battery platforms remains non-trivial due to cost, spatial constraints, and the need for high modulation stability.

To address these issues, future research must focus on developing hybrid diagnostic platforms that combine PAS with other spatially resolved techniques (e.g., neutron imaging or in situ XRD), along with tailored AI/ML pipelines capable of isolating useful signal features from noise. Advancements in nano-engineered acoustic transducers and low-loss optical modulators could also improve system sensitivity and integration feasibility.

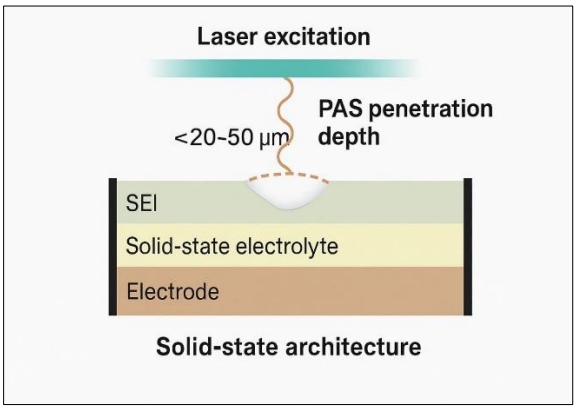

Figure 12: Schematic representation of the limitations in PAS-based diagnostics for metal-based energy storage systems. The illustration highlights key constraints such as limited acoustic penetration depth, signal interference in composite matrices, and complexity in integrating PAS with solid-state battery architectures [92]

10. CONCLUSION

In the evolving landscape of energy storage, where demands for efficiency, reliability, and miniaturization are escalating rapidly, Photoacoustic Spectroscopy (PAS) has emerged as an innovative and non-destructive diagnostic modality. The strategic integration of nanoscience into PAS methodology represents not merely a technological enhancement, but a paradigm shift in how we characterize and optimize metal-based energy storage systems (ESS)—ranging from lithium-ion and sodium-ion to next-generation magnesium- and zinc-based batteries.

This review has underscored the unique diagnostic strengths of PAS, including its ability to resolve subsurface features, monitor SEI formation, detect electrode degradation, and assess ion diffusion dynamics—all in real-time and without dismantling the cell. These capabilities have been demonstrated to become exponentially more powerful when nanostructured materials are introduced, such as quantum dots, nanowires, nanoparticles, and graphene derivatives. The tailored surface-to-volume ratios, enhanced light absorption characteristics, and tunable morphologies of these nanomaterials directly amplify PAS sensitivity and resolution, enabling diagnostics at scales that traditional spectroscopic methods cannot reach.

From a materials optimization perspective, PAS offers critical insight into phase transitions, lattice defects, and thermal effects that occur during charge-discharge cycles. These insights empower strategies like targeted doping, morphology engineering, and surface coating to improve battery performance and lifespan. As outlined, PAS not only functions as a probe but becomes a feedback mechanism within adaptive material design loops.

Looking ahead, the synergy between PAS, nanoscience, and machine learning (ML) opens pathways for fully autonomous, smart diagnostic platforms that can guide real-time battery management, health prediction, and structural optimization. Such systems could revolutionize commercial battery diagnostics—especially for solid-state, flexible, and hybrid-ion architectures—where traditional tools lack accuracy, adaptability, or integration potential.

However, realizing this vision requires overcoming the current limitations: restricted penetration depth, spectral interference in complex matrices, and the lack of standardized PAS-cell coupling frameworks. Addressing these will require cross-disciplinary collaboration, bridging spectroscopy, materials science, battery engineering, and AI. Moreover, open-access datasets, modular instrumentation designs, and cloud-based simulation environments will be essential to fast-track the industrial translation of PAS-powered diagnostics.

In conclusion, nanoscience-integrated PAS is no longer a speculative concept—it is rapidly positioning itself as the linchpin for next-generation energy storage diagnostics. Researchers, technologists, and battery manufacturers must now converge efforts to scale, standardize, and optimize this technique, ensuring its full potential is realized across commercial and high-performance energy systems. The roadmap forward is clear: deeper integration, smarter analytics, and broader accessibility. In the quest for cleaner, safer, and smarter energy storage, PAS will not merely support progress—it will define it.

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