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Characterization and Reporting Protocols for Structural Power Composites: A Perspective

Emile S. Greenhalgh,* Sang Nguyen,* Leif E. Asp, Alfredo Bici, Alexander Bismarck, Derrick Fam, Mats Johansson, Göran Lindbergh, Jodie L. Lutkenhaus, Milo S. P. Shaffer, Natasha Shirshova, Madhavi Srinivasan, Johanna Xu, and Dan Zenkert

Structural power composites, multifunctional materials that can withstand mechanical loads while storing/delivering electrical energy, are gaining significant interest. However, a consequence of melding disparate structural and electrochemical technologies is that there are no common characterization and reporting protocols, undermining the advancement of this emerging field. This Perspective paper sets out the challenges and resulting issues in the literature and recommends best practices and requirements for future protocols for reporting multifunctional performance. A key recommendation is that a “universal coupon” should be developed to be used for both mechanical and electrochemical characterization of cells, and hence credibly declare multifunctional performance. Ultimately, such a universal coupon can simultaneously characterize both functions, so as to glean electrochemical–mechanical coupling phenomena. This article recommends reporting guidelines so as to avoid the current ambiguities associated with normalization and permit robust comparison across the literature. The aspiration is that the guidelines and framework outlined in this paper lay the groundwork for formal standard methods to be developed and agreed upon. Establishing robust characterization and clearer reporting permits researchers and industry to take an informed view of the literature and provides a better grounding for the adoption of this technology, underpinning future industrialization of these emerging materials.

1. Introduction

1.1. Motivation and Aims

Engineering demands the efficient use of the constituent materials within a product, so the emergence of multifunctional composites, in which a single material simultaneously undertakes several roles, is a compelling prospect. This approach mirrors nature: in living creatures, very few structures have just one job. If we want to do more with less—less energy, less material, and less waste—we need to learn how to do the same.^[1] This paper focuses on mechanically load-bearing fiber reinforced polymer composites imbued with the capacity to store and deliver electrical energy,^[2,3] referred to in the literature as either *structural power composites* (SPCs) or *structural energy composites*; the former term will be used throughout this paper. Such multifunctional materials provide an exciting solution to both lightweighting and

E. S. Greenhalgh, S. Nguyen
Aeronautics Department
Imperial College
London SW7 2AZ, UK
E-mail: e.greenhalgh@imperial.ac.uk; snguyen@ic.ac.uk

A. Bici, D. Zenkert
Department of Engineering Mechanics
KTH Royal Institute of Technology
Stockholm SE-100 44, Sweden

G. Lindbergh
Department of Chemical Engineering
KTH Royal Institute of Technology
Stockholm SE-100 44, Sweden

M. Johansson
Department of Fibre and Polymer Technology
KTH Royal Institute of Technology
Stockholm SE-100 44, Sweden

L. E. Asp, J. Xu
Department of Industrial and Materials Science
Chalmers University of Technology
Hörsalsvägen 7B, Göteborg 41258, Sweden

L. E. Asp, M. Srinivasan
Wallenberg Initiative Material Science for Sustainability
Department of Industrial and Materials Science
Chalmers University of Technology
Hörsalsvägen 7B, Göteborg 41258, Sweden

L. E. Asp
Visiting Professor at the Department of Aeronautics
Imperial College London
London SW7 2AZ, UK

A. Bismarck
Institute for Materials Chemistry and Research
University of Vienna
Vienna 1090, Austria

 The ORCID identification number(s) for the author(s) of this article can be found under <https://doi.org/10.1002/aenm.202404702>

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efficient energy storage: design drivers for many sectors. These disruptive materials have gained significant public, academic, and industrial attention and are anticipated to make considerable contributions to a range of sectors, including the electrification of transportation. We expect this technology will become ubiquitous (**Figure 1**), enabling phones as thin as credit cards, aircraft seats that power the cabin, and “structural battery bricks” that store household energy.

For brevity, a detailed review of structural power composites has not been included in this paper and can be found elsewhere.^[3–6] As is often the case for emerging fields, there are considerable challenges associated with characterization and the reporting of performance of structural power composites. The problems are exacerbated by the research on these composites being profoundly interdisciplinary: the conventions, nomenclature, and standards followed by electrochemical and mechanical (composite) fields are very different. So, although potential industrial adopters are eager to be engaged with this field, the growing body of literature is challenging to interpret, and cell performance is difficult to rank. There is a need for this emerging research community to “speak the same language.” The authors of this paper have recognized that a critical priority is the development of characterization and reporting protocols and best practices. Here, we draw together leading research groups on the development of structural power composites and set out the requirements for such protocols. This framework will lay the groundwork for formal standard methods to be developed and agreed upon in the future, underpinning future industrialization of these materials.

The overarching objectives of this paper are to convey the challenges in characterizing structural power composites and, hence,

propose best practices and protocols for characterizing and reporting multifunctional performance. Such a clearer understanding will permit researchers and industry to take an informed view of the literature and provide a better grounding for the adoption of this technology. Protocols will result in the generation of robust data to support predictive models and multifunctional design tools. This data will provide the means to rank different constituents, architectures, and cells, such that structural power composite development can be advanced and useful levels of multifunctional performance realized. Ultimately, this will lead to the development of electrochemical–mechanical tests to characterize coupling phenomena, such as the influence of mechanical damage on electrochemical behavior.

Given the readership is anticipated to be broad, drawn from different communities, the paper provides a brief introduction to both fiber-reinforced polymer composites and electrochemical cells, and then sets out to define multifunctionality, with particular focus on structural power composites. A brief overview of mechanical and electrochemical characterization of conventional composites and energy storage cells, respectively, is then presented, followed by a summary of the issues associated with characterization and reporting of structural power composites, specifically. Drawing on this background, best practice and protocols are proposed, with particular focus on the need for coupled electrochemical–mechanical test methods. Illustrative examples of data reporting for structural supercapacitors and structural batteries are presented in Appendix A. For brevity, some of the equations and their associated nomenclature are included in Appendix B but are referred to in the main text.

1.2. Brief Overview of Fiber Reinforced Polymer Composites

A composite material is, by definition, a mix of different materials of different phases combined in such a way as to improve mechanical and physical properties.^[7] The most common structural composites use high-performance reinforcement fibers, such as carbon, glass, or aramid fibers, to provide stiffness and strength, embedded in a polymer matrix, to transfer load and form a coherent material. Layers of unidirectional (UD) fibers or woven fabrics are typically stacked in specific orientations to create multilayer laminated composites. The interface between the reinforcement and the matrix phases is often considered a third constituent, since it has characteristic properties critical to the overall mechanical and physical performance. The material constituents of the composite, their relative proportions, and spatial arrangements can be designed to provide a wide range of specific mechanical and physical properties, opening up a huge design window, including adding other functionality.

A typical UD composite is made from 50% to 60% volume fraction of carbon fibers and has a Young's modulus of 110–170 GPa in the fiber direction, whereas the modulus in the transverse direction is only 5–10 GPa, thus creating an anisotropic material. A quasi-isotropic composite has fibers in several directions, creating a laminate with the same Young's modulus (50–75 GPa) along different directions in the plane. Since the densities of the constituents (both fiber and matrix) are low, structural composites offer very high stiffness and strength-to-weight ratios, often the prime motivation for their use.

A. Bismarck

Chemical Engineering Department
Imperial College London
London SW7 2AZ, UK

D. Fam

Institute of Materials Research and Engineering
Agency for Science Technology and Research
Singapore 138634, Singapore

D. Fam

School of Materials Science and Engineering
Nanyang Technological University
50 Nanyang Ave., Singapore 639798, Singapore

J. L. Lutkenhaus

Artie McFerrin Department of Chemical Engineering
Texas A&M University
College Station, TX 77843, USA

M. Srinivasan

Executive Director of Energy Research Institute at NTU. Energy Research
Institute (ERI@N) at NTU & School of Materials Science and
Engineering
Nanyang Technological University
50 Nanyang Ave., Singapore 639798, Singapore

M. S. P. Shaffer

Materials Department
Imperial College London
London SW7 2AZ, UK

N. Shirshova

Department of Engineering
Durham University
Durham DH1 3LE, UK



Figure 1. Artist impressions of the impact of structural power composites: a) phones as thin as credit cards; b) aircraft cabin powered by the seat material, and c) future buildings where the bricks in the walls store electrical energy.

1.3. Brief Overview of Conventional Electrochemical Cells

Conventional electrochemical energy storage cells primarily include capacitors, supercapacitors: electrochemical double layer capacitors (EDLCs), pseudocapacitors etc., hybrid ion capacitors, and batteries. The characteristics of these devices are illustrated in a Ragone plot (Figure 2), which shows the balance between specific energy and power for different cell types.^[8] By analyzing the energy and power characteristics depicted in the Ragone plot, the most suitable device can be selected to meet specific performance requirements. Gravimetric performance (i.e., energy or power per unit mass) is a critical parameter for transport applications, but an equivalent plot can also be presented in terms of volumetric performance (i.e., energy and power densities). Generally, there is a trade-off between the capacity to store electrical energy and the ability to deliver this energy quickly.

These cells store charge at negative and positive electrodes (usually termed anodes and cathodes, respectively), separated by a suitable separator, via a variety of underlying phenomena (Figure 3) that provide characteristically different current–voltage responses.^[9] As shown in Figure 2, electrostatic (or dielectric) capacitors provide high power but low energy densities, and hence are not particularly useful in the context of energy storage. Such cells work by the accumulation of electrical charge across a dielectric material, which is sandwiched between the electrodes (Figure 3a). At the other extreme are batteries (Figure 3e), which provide high specific energy but low specific power. Such devices store energy through chemical (i.e., Faradaic) processes, and hence are governed by thermodynamic, kinetic, and diffusive phenomena. Supercapacitors offer a compromise between

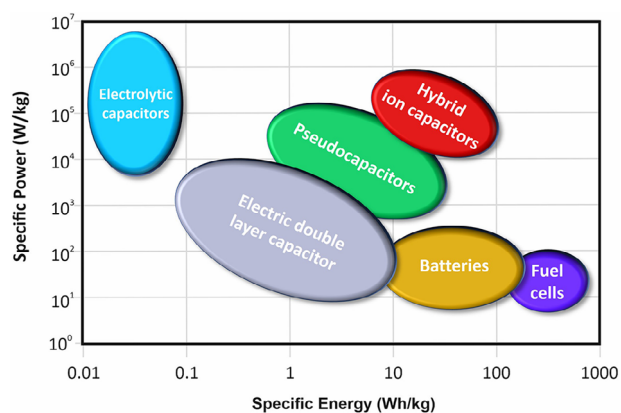


Figure 2. Ragone plot illustrating the different types of electrochemical energy storage devices.

specific energy and power.^[10–12] As illustrated in Figure 3, supercapacitors can be partitioned into different types of cells: electric double-layer capacitors (Figure 3b), pseudocapacitors (Figure 3c), and hybrid ion capacitors (Figure 3d). Other types of devices, such as fuel cells and redox flow batteries, are not considered here, as the storage capacity is determined by the volume of liquids involved, and are not well aligned to structural multifunctionality.

Conventional EDLCs (Figure 3b) consist of two high surface area electrodes which sandwich a thin, ionically conducting but electrically insulating separator, all of which is immersed within an electrolyte. Charge storage in EDLCs is considered exclusively physical in nature and occurs via electrostatic adsorption at the electrode/electrolyte interface. Pseudocapacitors (Figure 3c) enhance the energy storage capacity by utilizing some Faradaic processes at the electrode/electrolyte interface via electrosorption, redox reactions, and intercalation, while retaining a capacitor-like charge/discharge response, within a useful voltage range. Hybrid ion capacitors (Figure 3d) combine both an electrostatic electrode (i.e., Figure 3b) and an electrochemical electrode (i.e., Figure 3c) to give higher energy storage capacity than a conventional EDLC but better energy delivery than a battery.

Batteries are energy storage devices that store energy chemically.^[13,14] They are classified into primary and secondary batteries. Primary batteries are non-rechargeable and designed for single-use applications, generating electrical energy through electrochemical reactions. The continuous flow of electrons through the external circuit and ions through the electrolyte enables the primary battery to supply power until the reactants are exhausted. Secondary batteries, also known as rechargeable batteries, store energy through reversible electrochemical reactions and are designed to be recharged and used multiple times (Figure 3e). Based on the solvent used in the electrolyte, rechargeable batteries can be divided into aqueous and nonaqueous categories. The former are generally safe but have lower energy density due to the narrow stability window of the aqueous electrolyte. Nonaqueous rechargeable batteries offer higher energy density but come with potential safety concerns.^[15]

2. Definitions and Multifunctionality

2.1. Definitions

In considering the challenges, issues, and resulting protocols for structural power composites, a pragmatic approach is to partition into three levels: *Constituents*, *Cells*, and *Components* (Figure 4). The progression through increasing scale and complexity is familiar in both structural composites and electrochemical energy

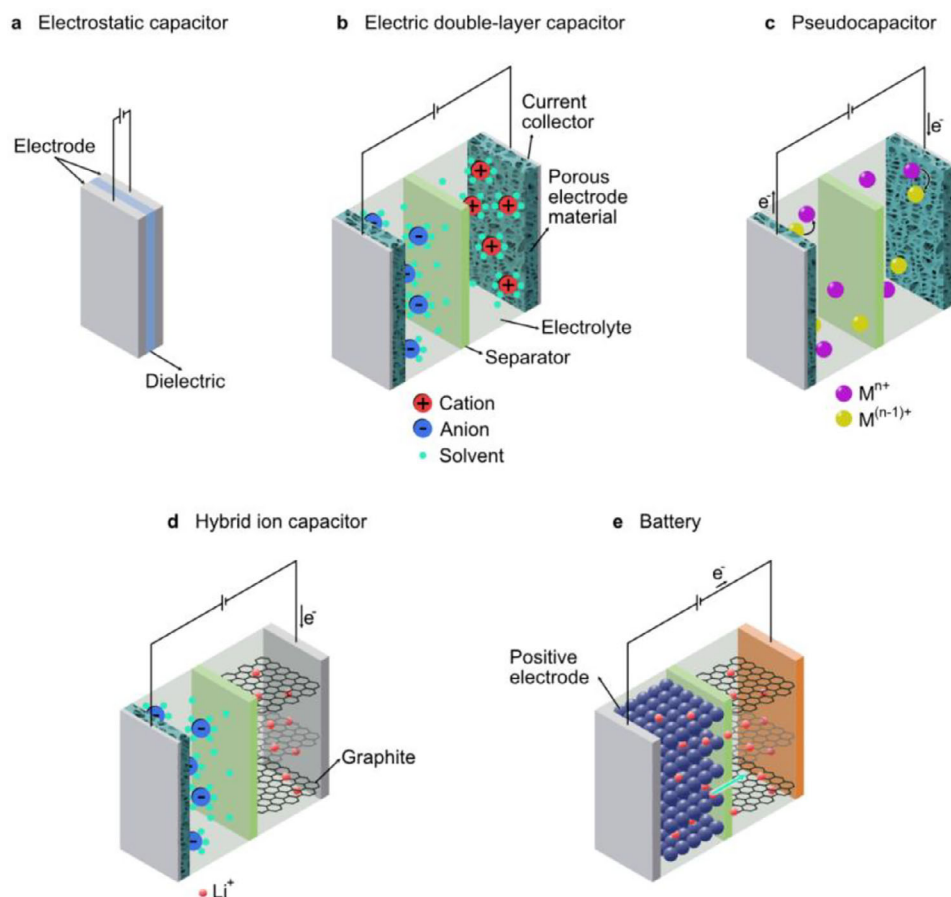


Figure 3. Schematic of a) an electrostatic capacitor, b) an electric double-layer capacitor (EDLC), c) a pseudocapacitor, d) a hybrid ion capacitor, and e) a battery.

storage; however, multifunctionality introduces additional challenges. In addition, interfacial characterization also manifests for all three levels: fiber/matrix (*Constituents*), separator/electrode (*Cells*), and between cells/encapsulation (*Component*).

First, the *Constituents* level includes the structural electrodes, structural electrolytes, separators, current collectors, and encapsulation materials. The *Constituents* level also includes the interfaces between constituents, such as the fiber/matrix interface be-

tween the structural electrode (or separator) fibers and the structural electrolyte. The *Cells* level corresponds to the assembly of the *Constituents* to deliver a structural power composite. The *Cell* should include interfaces between the constituents, such as the structural electrode/separator interface, for which resisting delamination will be an important requirement. The final level is that of *Components*, which will consist of multicell assemblies within a final product. These will probably be a hybrid of multiple

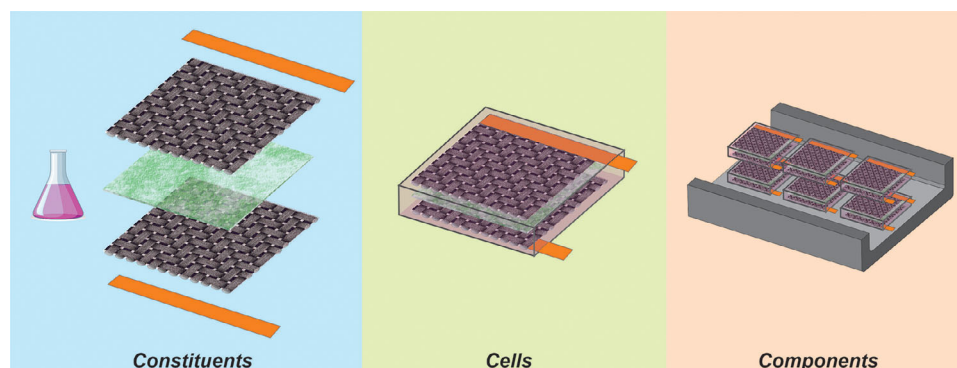


Figure 4. The *Constituents*, *Cells*, and *Components* levels.

cells, connected via bipolar plates and within structural encapsulation, all within a monofunctional (conventional) composite laminate.

As described in the following Sections, the approaches for both mechanical and electrochemical characterization and reporting differ for the different levels associated with the test pieces. Therefore, before defining multifunctionality, it is helpful to describe the nomenclature used here, as shown in **Table 1**.

2.2. Taxonomy of Multifunctionality

The term “multifunctional” has been widely used throughout materials science, although less frequently in relation to structural materials. Therefore, it is helpful to define what is pertinent to this paper and to consider the general taxonomies proposed for multifunctional materials. To maintain an appropriate context, these definitions will focus on concepts for structural power composites (i.e., combining structural and electrochemical functions). While several definitions have been proposed, perhaps the clearest is by Wetzel.^[16] This taxonomy ranked multifunctional systems in order of increasing complexity, but with increasing potential benefits (**Table 2**). *Conventional* systems (Category A) are deemed to be the current off-the-shelf (COTS) “classical” approach in which a system consists of an assembly of monofunctional parts that may be physically isolated from each other. Category B is *Embedded* systems in which COTS monofunctional parts are integrated within each other, thus providing mass and/or volume savings through efficient packaging. This approach of “a battery in a structure” is relatively mature in achieving “multifunctional structures” and is often referred to as “smart structures.”^[17] Beyond this class lie *Conformable* systems (Category C) in which the cell geometry and flexibility are such that they can fit into the component. Finally, *Structural* (Category D) refers to a material that is intrinsically multifunctional, undertaking two roles without any monofunctional constituents.

Others have adapted and expanded this taxonomy, such as Adam et al., for structural power composites, partitioning Category D in **Table 2** into different scales: “meso”, at which the lamina correspond to the electrodes, and “micro”, where tows (or even fibers) correspond to individual cells (e.g., concentric electrodes).^[18] Similarly, Ferreira et al. defined Multifunctional Structures (Categories B and C in **Table 2**), Multifunctional Composite (Category D: meso in **Table 2**), and Multifunctional Material (Category D: micro in **Table 2**).^[19] Finally, Hopkins defined “uncoupled” (Categories B and C in **Table 2**) and coupled (Category D in **Table 2**) multifunctional materials.^[20] This paper will focus on multifunctional materials that correspond to Category D in **Table 2**, i.e., the material is intrinsically multifunctional such that some or all of the constituents are multifunctional, and are assembled into a multifunctional device.

2.3. Structural Power Composite Embodiments

Structural composites strongly resemble electrochemical energy storage devices in that both are formed from layers of different materials; the structural composite lamina can become the elec-

trodes and separators of the device. As with structural composites, the properties of electrochemical cells are governed by the materials and volume fractions used in the electrodes and the electrolyte. Moreover, carbon is a common constituent of electrode materials and structural composites. Such parallels have stimulated the melding of structural composites with electrochemical energy storage. Structural power composite devices mirror the architecture and charge storage mechanisms of conventional supercapacitors and batteries (see Sections 4.2 and 4.3) but use constituents, or combinations of constituents, that can carry mechanical loads (i.e., structural electrodes, structural electrolytes, etc.). While layered architectures are common, fiber geometries have also been explored (**Figure 4**).

Drawing on Wetzel’s taxonomy, several embodiments have been proposed for structural power devices,^[21,22] which fall into categories A–C in **Table 1**. *Embedded devices* (**Figure 5a**) entail conventional cells embedded within composite laminates. *Conformable devices* (**Figure 5b,c**) can be flexed and are robust, usually employing nanocarbon electrodes in a soft or gel-like electrolyte. They can tolerate high bending deformations (although not necessarily high strains) and are durable enough to cope with environmental factors and repeated deformation. However, they do not offer high stiffness or strength under direct loading, so such embodiments are difficult to deem as mechanically load-bearing. Such *Conformable devices* can be partitioned into two architectures: *Laminated* (**Figure 5b**), in which the electrodes are non-aligned veils, and *Fiber* (**Figure 5c**), in which the individual electrodes are fibers or tows that are paired as a yarn. It is feasible that *Fiber* embodiments could be subsequently woven into lamina if the yarns are robust enough to tolerate the weaving conditions.

A particular focus of this paper, structural supercapacitors and structural batteries (i.e., Category D in **Table 1**), can sustain significant direct mechanical load (**Figure 5d,e**). One approach is to take structural electrodes (and separators) and infuse with a conventional liquid or gel electrolyte, to produce an “uncoupled”, or sometimes referred to as a “semi-structural”, device^[20,21,23] Such devices present an electrochemical performance approaching that of a conventional device but cannot resist bending, shear or compressive loading. Moyer et al. were the first to demonstrate such a structural battery composite, with graphite and lithium iron phosphate coated carbon fiber weaves as negative and positive electrodes, respectively.^[24] On the other hand, “coupled”^[20] *structural supercapacitors* or *structural batteries* utilize structural electrodes embedded within a structural electrolyte: the electrodes usually use carbon fibers as the structural backbone with high surface area or active materials added to enhance electrochemical performance.^[3,23] For structural batteries, the carbon fibers are used as the anode, but the cathode material is coated onto the carbon fibers in the positive electrode. A semisolid ionically conductive matrix surrounds the fibers (**Figure 5d**).^[25,26] Recent studies have demonstrated a “coupled”^[20] all-carbon fiber full cell in a structural electrolyte.^[27,28] As with *Conformable* devices, structural supercapacitors or structural batteries can potentially be partitioned into two architectures: *Laminated* (**Figure 5d**) in which the electrodes are unidirectional tape or woven fabrics and *Fiber* (**Figure 5e**) in which the individual electrodes are fibers or tows which are paired in a yarn (which could be then woven into a lamina). Fiber batteries could be envisaged in which

Table 1. Definitions: (a) constituents, (b) half-cell, (c) active cell, (d) full cell, (e) SPC device, (f) SPC module, and (g) SPC component (or product). Colors correspond to level (Constituent, Cell, or Component).

Level	Definition	Test intends to determine	Normalize by
Constituent			
Half-cell	Assembly of a single working structural electrode, with a suitable counter electrode, separator, and electrolyte, often characterized in dedicated glassware, Swagelok assembly, coin cell, or pouch cell, optionally with a reference electrode.	The maximum intrinsic performance of active material in a single electrode, usually compared to theoretical capacity.	Usual to normalize by the electrochemically active material. However, for structural electrodes, it is helpful to also normalize by the entire electrode (i.e., active material, binder, plus structural scaffold).
Active cell	Assembly of a single pair of balanced, complementary structural electrodes (anode/cathode), separator, and structural electrolyte, making a working full electrochemical cell, often characterized in a Swagelok assembly, coin cell, or laminated pouch cell. Depending on the test configuration and electrode conductivity, there may or may not be an explicit current collector present.	The maximum intrinsic performance (mechanical and/or electrochemical) of the full structural battery or structural supercapacitor, per unit area, volume or mass.	Electrodes, separator and electrolyte contained within these active layers. The mass of parts associated with the test (e.g., Swagelok parts, coin self-assembly, pouch), together with any additional current collectors, or excess electrolyte that are beyond the bounds of the active area, are ignored.
Full cell	Free-standing assembly of a single pair of balanced, complementary structural electrodes (anode/cathode), separator, and structural electrolyte, which makes a working full electrochemical cell, often characterized in a coin cell or laminated pouch cell.	A reliable benchmark for the electrochemical energy storage performance of structural power composites at a specific scale.	All constituents within the boundary of the assembly (including encapsulation, current collectors, and excess electrolyte).
SPC device	At least one SPC cell assembled into a multifunctional format, consistent with both mechanical and electrochemical characterization, including appropriate encapsulation, current collection, and tabs/connectors. Devices may consist of an electrochemical single cell or a multicell battery of cells connected in series using bipolar electrodes or plates.		All constituents in the device.
SPC module	An assembly of SPC cells or devices arranged in a specific configuration to provide a unified power source. Each individual device may be interconnected in series or in parallel, and optionally addressed by a battery management system, ensuring safe and efficient operation.		All constituents in the module.
SPC component	A hybrid assembly consisting of monofunctional structural materials and multifunctional devices/modules, and potentially conventional cells, to form a final product.		All constituents in the component.

Table 2. Definitions of multifunctionality as described by Wetzel.^[16]

System	Active part	Mechanical part	Benefit
(A) Conventional	Current off-the-shelf	None	Standard practice
(B) Embedded	Current off-the-shelf	Packaging	Mass/volume saving through efficient packaging
(C) Conformable	Custom	None	Efficient volume use and form factors
(D) Structural	Custom	Custom	Huge mass/volume saving possible



individual carbon fibers are coated by an electrically insulating, ionically conductive solid polymer electrolyte, in a lithium iron phosphate-doped matrix.^[29]

2.4. Multifunctional Design

Multifunctional design considers how best to exploit the benefits of multifunctionality and aims to compare performance objectively to conventional systems. One method for quantifying mul-

tifunctional performance is a multifunctional efficiency index, η_{mf} (Equation 1), calculated as the sum of structural and energy efficiency, where each performance is normalized by the corresponding conventional sub-system performance.^[30]

$$\eta_{mf} = \bar{E}_{mf} / \bar{E} + \bar{\Gamma}_{mf} / \bar{\Gamma} \quad (1)$$

where \bar{E}_{mf} and \bar{E} are the specific structural performance of the multifunctional and conventional systems, respectively, and $\bar{\Gamma}_{mf}$

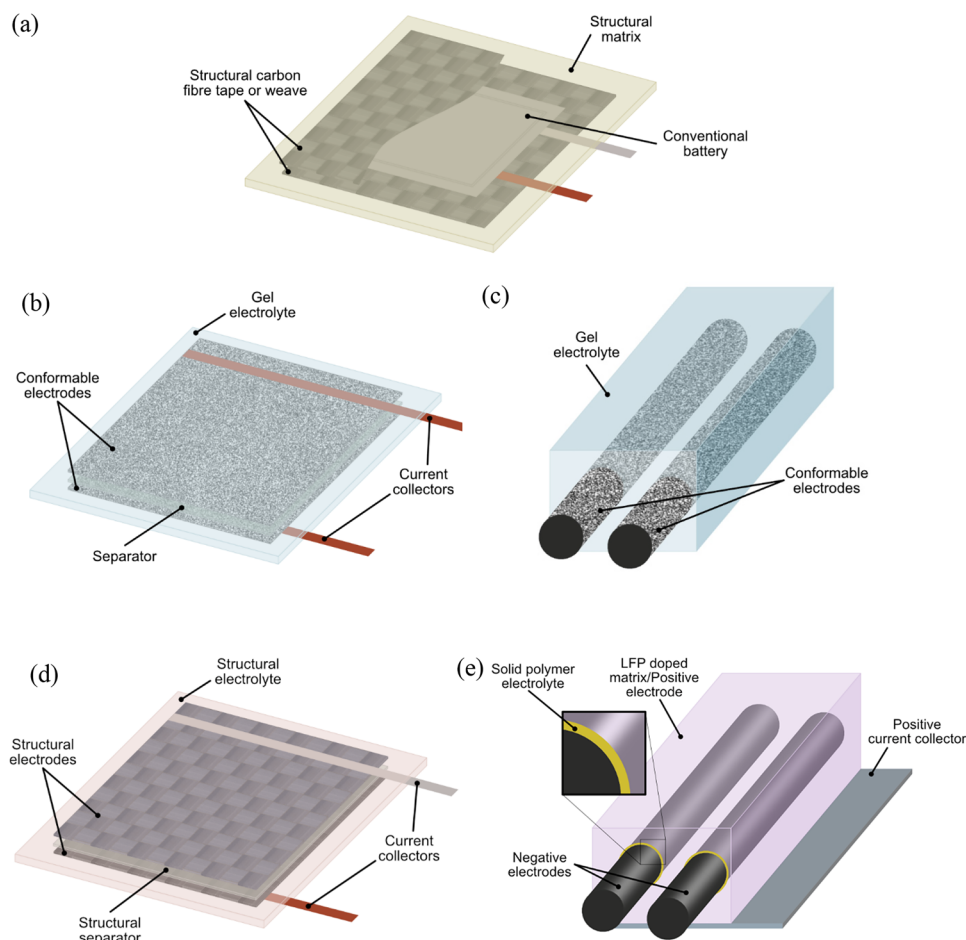


Figure 5. Different multifunctional supercapacitor and battery embodiments: a) embedded cells; b) conformable laminated cell; c) conformable fiber cell; d) structural laminated cell; e) structural fiber cell.

and $\bar{\Gamma}$ are the specific electrochemical performances of the multifunctional and conventional systems, respectively. The analysis considers the specific structural performance parameter that governs the design of the structure for the anticipated loading cases. For example, if the structural design is limited exclusively by the flexural stiffness of the structure, the analysis would consider the specific flexural modulus. If the overall multifunctional efficiency exceeds unity, the multifunctional component will provide a weight and/or volume saving over the monofunctional assembly. Several authors have used this metric, or a development of it, to describe multifunctional performance.^[4,21,30–35]

An alternative methodology has assessed the “residual” specific electrochemical performance^[36]

$$\bar{w}_{\text{res}} = w / (m_{\text{mf}} - m_2) \quad (2)$$

where w is the electrochemical performance, such as total energy, m_{mf} is the multifunctional system mass, and m_2 is the mass of a conventional structural laminate having equivalent structural performance to that of the multifunctional laminate. Here, the term “equivalent structural performance” means the *absolute* load-carrying capability (such as the displacement for a given load), not the specific or normalized material properties. The mass of the conventional structural laminate is calculated using the specific material properties for a state-of-the-art conventional material. The residual performance metric then assigns the “residual” difference in mass between the multifunctional and conventional laminates to the electrochemical function. If the “residual” mass is lower than that of an equivalent conventional device, then the multifunctional material provides a weight saving. Quantitative examples of the residual specific performance metrics for structural supercapacitors and structural batteries are provided in ref. [36]. All the methods to calculate multifunctional performance metrics rely on credible, consistent, and robust characterization data to support multifunctional design.

The multifunctional metrics mentioned above have limitations in resolving whether a multifunctional component offers a saving over the conventional assembly of monofunctional energy storage devices and structures. Very often, the introduction of multifunctional composites has wider implications for the overall system design, offering other savings, for example, in reduced wiring. There have been studies to develop the field of multifunctional design to account for such implications by studying the feasibility, performance targets, integration strategies, and benefits/challenges for specific applications. The approach has been to audit the structural mass and the energy and power demands, and to then assess what specific energy and specific power the multifunctional material would need to provide to offer a saving over conventional systems. This methodology has been used to evaluate the implications for small electric aircraft, regional aircraft cabins, future (hybrid) electric aircraft, and air taxis.^[37–39] The motivation for such studies is to permit assessment of the suitability of adopting multifunctional materials and provide target mechanical and electrochemical properties so that multifunctional materials can be tailored for specific applications, e.g., the proportion of solid polymer versus liquid electrolyte in a multifunctional matrix. The multifunctional design methodology proposed in these studies entails the main stages

outlined in **Figure 6**, which partitions the design phases into structural, functional, and multifunctional domains and shows their boundaries.

3. Overview and Comments on Mechanical Characterization

3.1. General Issues

Mechanical characterization of laminated composites has been comprehensively standardized through bodies such as ASTM and ISO.^[40,41] These methods have been validated via extensive round-robin studies and are well established despite the rapid development of new constituents, composite architectures, and fabrication processes. A fundamental requirement has been to define appropriate small “coupons” that are representative of the behavior of the material when within a larger component. Standard coupons are usually of a simple geometry such that the gauge section is under a controlled (usually uniform) stress state. The standards prescribe acceptable ranges for the coupon dimensions, geometries, and ply orientations, again to ensure well-defined loading conditions. Coupon design is driven to negate confounding factors such as edge effects, but strives to utilize the minimum amount of material to characterize the mechanical performance properly.

Because laminated composites are anisotropic, detailed mechanical test methods, including end-tabling, optimized specimen gripping, and alignment, are key to ensuring controlled load introduction and hence valid test conditions. Instrumentation, such as (optical) strain gauges, is prescribed to determine properties such as modulus. Since composites tend to have high stiffness, test machine compliance can become an important factor.

Coupons have been developed at several scales, ranging from constituents (fibers, matrices, and the interfaces between them), single lamina to multi-ply laminates. Regarding the former, statistical approaches and/or small-scale (micromechanical) tests are used for fiber or tow characterization due to the inherent brittleness of this constituent. On the other hand, matrix characterization draws on standard tests for bulk polymers. Finally, fiber/matrix interface behavior plays an important role in composite performance. Several micromechanical test methods have been developed, but may best be considered as providing a ranking tool.^[42]

For lamina characterization, a minimum thickness is usually prescribed by the standards. Lamina test methods can be partitioned into translaminar, interlaminar, and intralaminar loading modes.^[43] These properties can be characterized by direct loading onto lamina (or unidirectional laminates), although gripping and end-tabling conditions can become critical in some cases. An alternative approach is to apply bending loads, but this condition is very sensitive to coupon thickness, width, and surface conditions, and can suffer from the local contact forces at the load application points. Knowledge of the constituent performance and the proportions thereof (i.e., volume fractions) can be used to predict lamina-level behavior, particularly in the elastic regime. However, for characterizing strength (Section 3.3) and toughness (Section 3.4), it is generally necessary to test coupons at this scale.

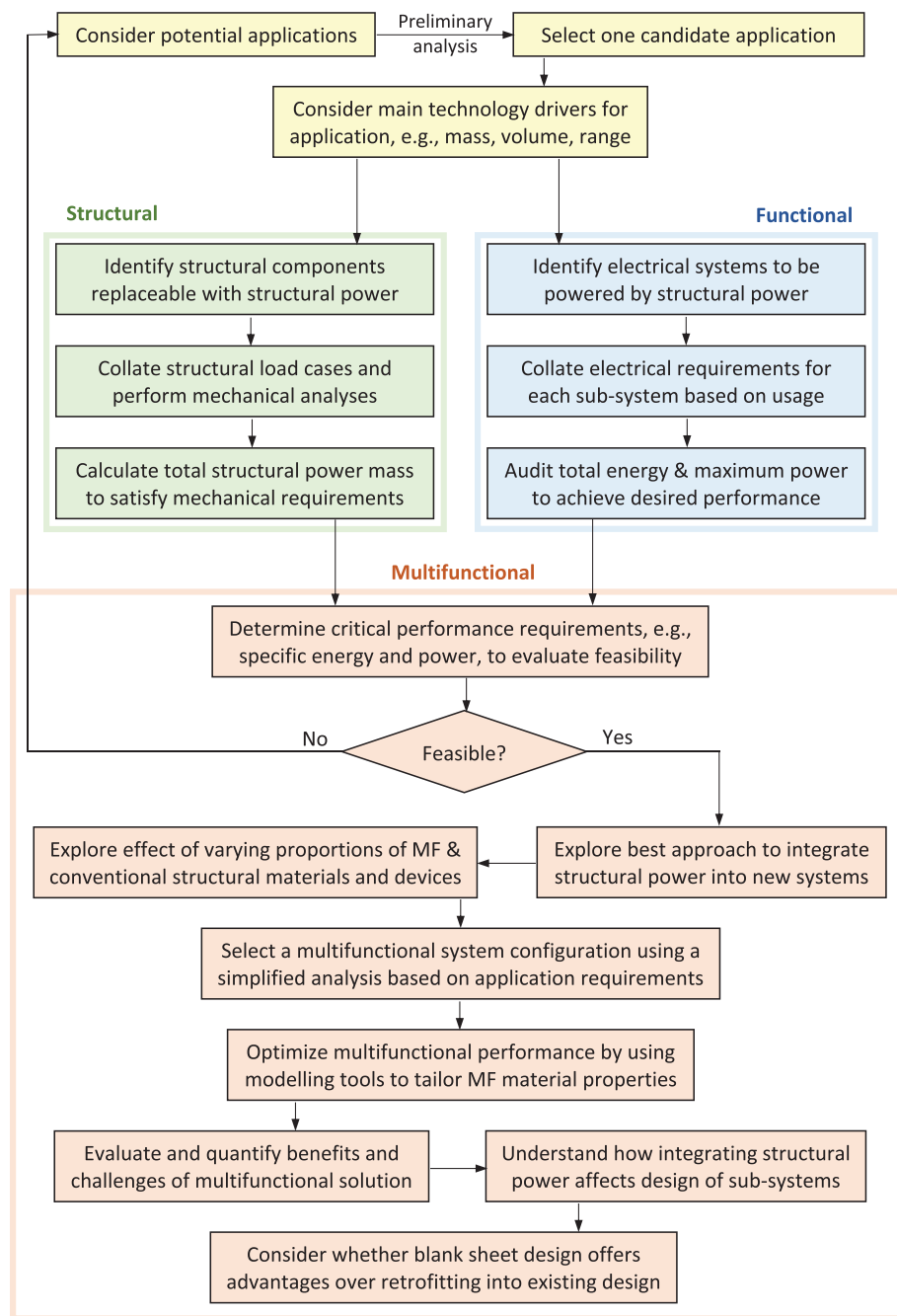


Figure 6. Flow chart of the main stages of the proposed multifunctional design methodology.

3.2. Modulus

The modulus is a measure of the elastic deformation of a material under an applied stress. Most engineering structures are driven by minimum allowable stiffness requirements, such that the structure can sustain various load cases without excessive deformation.^[44] The stiffness of the structure is directly linked to the modulus of the material used in the structure. The modulus is less sensitive to scale and preparation than other properties, such as strength. However, because composites can have a very

high modulus in the fiber direction, the gauge region may need to be very remote from the load application points to ensure uniform loading. Tensile loading provides little insight into matrix or interfacial issues since tensile modulus is dominated simply by the fiber volume fraction.

3.3. Strength

Strength is a measure of the maximum stress that can be applied to the material in its pristine state (i.e., without stress

concentrators present). Strength, particularly compressive strength, can become important for applications such as crash-worthiness, etc., or when the composite structure is thick enough such that it is not limited by buckling performance. The strength measured in a test piece is highly sensitive to its preparation. Unlike tension or compression, under in-plane shear loading, there is no sudden load drop or peak load, and therefore the in-plane shear strength is typically defined as the shear stress at a given value of engineering shear strain, typically 1% and 5%.

3.4. Toughness

The toughness, the resistance to crack growth, is perhaps more important than strength for the application of polymer composites. It provides a measure of the material's robustness and dictates residual strength after in-service damage or failure from manufacturing defects. For translaminal fracture toughness, translaminal tension and compression tests have been developed, which are based on the compact tension test for isotropic materials.^[45] Similarly, for intralaminar fracture toughness, a 90° compact tension test has been developed.^[46]

Most of the focus on composite toughness has been on interlaminar fracture (i.e., delamination), for which standard tests have been developed. These methods rely on bending tests in which the laminate planes are driven apart to form a delamination.^[47] For such standard tests, sufficient coupon thickness is essential to avoid excessive deformations and nonlinearities that will invalidate the test. Challenges arise when considering thin laminates, so solutions such as using "doubblers" to stiffen the coupon arms become necessary.^[48] Although well established, these delamination tests have only been standardized for identical substrates and encounter difficulties when characterizing dissimilar substrates at the critical interface.

3.5. Engineering Performance

Beyond the lamina level standards, there is a set of test methods that use more realistic laminate assemblies. These typically use multidirectional laminates, with more structural features and/or more complicated loading conditions. These tests are recognized as stepping stones to achieving qualification of new composites for particular applications. The simplest engineering tests are multidirectional tension or compression, in which plain laminates (with a defined stacking sequence) are loaded to failure under uniaxial load. These tests can be extended to include notches (under tension or compression) or in-service damage, such as impact. During material development, the initial engineering tests are undertaken under quasi-static loading conditions, but to advance the material development, dynamic, creep, and fatigue loading are required to characterize the material under loads representative of those it may be exposed to in-service. Finally, larger-scale element, sub-component, and component tests are necessary to fully characterize the material when within a structure. Such tests often focus on particular features pertinent to the final component design.

4. Overview and Comments on Electrochemical Characterization

4.1. General Issues

Electrochemical characterization techniques assess the performance of both materials and devices for energy storage, whether they are supercapacitors or batteries. These techniques determine critical electrochemical performance parameters such as specific capacitance, specific energy, specific power, working potential window, and stability under cycling. As for mechanical coupon tests, these metrics can be used to extrapolate the performance of a larger electrochemical system, in advance of later engineering systems testing. In addition, electrochemical characterization techniques can elucidate fundamental mechanisms of ion and electron transport, and Faradaic and chemo-mechanical reactions, which are vital for understanding deviations from theoretical performance values. Understanding these mechanisms and reactions is also crucial to understanding and ameliorating electrochemical-related degradation mechanisms.

Electrochemical characterization techniques can be broadly categorized into potentiostatic/dynamic, galvanostatic/dynamic, or impedance measurements. Potentiostatic methods involve controlling the potential of the working electrode while measuring the resulting current. Galvanostatic methods are the opposite, controlling the current while measuring the resulting potential. Specifically, in a potentiostatic measurement, a constant polarization potential is applied and the current measured. An example of a potentiostatic measurement that is commonly used in batteries and supercapacitors would be chronoamperometry, which can be used to determine the rate performance of a battery.^[49] Potentiodynamic techniques instead vary the applied potential while monitoring the resulting current. Cyclic voltammetry (CV) is a common potentiodynamic technique where the potential of a device is varied linearly, and the resulting current is measured. CV can help determine the redox potentials of specific materials, which is critical to ascertain the nature of the energy storage mechanism (whether it is capacitive or Faradaic in nature).^[9] Lastly, impedance measurements shed light on the time-dependent mechanisms (double layer charging, ion transport, Faradaic processes) that are occurring in a cell or in a material. Typically, in impedance measurements, a potentiostatic or galvanostatic signal of a pre-defined waveform and amplitude is passed into a device, and the resultant response (either current or potential) is measured to determine the impedance. In electrochemical impedance spectroscopy (EIS), a voltage waveform with a small amplitude is applied to the material over a range of frequencies to generate a complex impedance.^[50] Plotting the out-of-phase imaginary impedance (Z'') against the real in-phase impedance (Z'), in a "Nyquist plot", helps to identify the overall and charge transfer resistance, diffusivity, and any other kinetic events within the electrolyte. EIS can also be applied to specific components, for example, to measure the ionic conductivity of an electrolyte as a function of temperature. All these tests vary with the number of electrical cycles applied, since there are both initial "formation" processes and subsequent degradation processes. It is important to report the cycling history of the test cell and its variation over time.

These electrochemical measurements may be performed with different cell configurations, such as symmetrical, half, and full cells, each serving specific purposes. Symmetrical cells are cells that have the same materials for both electrodes with the same mass. Symmetrical cells are often used in supercapacitors to ascertain the specific capacitance either via charge–discharge experiments or CV (Appendix B, Equation B3). Half-cells comprise an active material as the electrode paired with the target metal (e.g., for Li-ion batteries, Li metal) that acts both as the counter and the reference electrode. Half-cells are typically used to ascertain the electrochemical behavior of battery materials with reference to the potentials (versus the target metal) to which it is biased. In these cases, it can be viewed that the Li-metal will provide the inventory of cations. In some cases, half-cell electrochemical data can also give clues as to how the full cell performs (e.g., using the coulombic efficiency of a half-cell to predict the cycling stability of a full cell).^[51] Lastly, full cells would typically come with the prescribed active cathode, anode, and electrolyte material, and their performance should reflect the baseline performance should the cell be scaled to other form factors.

The form factor of the cells refers to the packaging form that the active constituents (anode, cathode, electrolyte, separator) take. Some common form factors would be cylindrical (as shown in Appendix C), pouch, and prismatic cells (broadly classified, without consideration of other subtypes). The form factor is an important consideration for the performance metrics reported, as the specific energy and power are normalized with respect to the weight and volume of the entirety of the cell. In addition, when it comes to solid-state cells, the pressure applied to the cells affects the eventual cell performance due to better electrode–electrolyte interfaces. For example, typically, an applied pressure of 5 MPa to more than 100 MPa is needed to achieve practical specific energies for many solid-state batteries.

Lastly, gravimetric normalization poses a significant hurdle toward consistent and reliable data reporting.^[52,53] Depending on the context, the mass to which the data is normalized must be stated clearly for benchmarking to be done fairly. There are large errors associated with instrument errors in the data, should the mass of the active material is small. Finally, many materials tested at low areal loading might give good rate performances at a coin cell level, but would not be practical for commercialization.

Many journals have adopted checklists for reporting electrochemical energy storage device data.^[54–57] Most checklists require the reporting of the mass of each constituent, the areal mass loading of the active material, a diagram of the cell's assembly, the amount of electrolyte, number of cycles tested, and a minimum of three devices tested. Information on how the capacity or capacitance, energy, and power were calculated should also be provided, as well as how the C-rate was calculated. Johansson et al. have published an insightful analysis of the potential pitfalls of characterization and reporting of electrochemical performance.^[58]

4.2. Supercapacitors

Supercapacitors are often assembled in a symmetric cell configuration, in which both electrodes have identical compositions and mass.^[59] Supercapacitor characterization centers on determining the cell's capacitance at varying current densities, voltage win-

dow, and equivalent series resistance, which are then used to calculate the cell's energy and power.

Supercapacitor cells are often first characterized using CV. For a triangular voltage, a “perfect” supercapacitor response would be a rectangular voltammogram, but the response is often distorted by internal impedance, yielding a sloping or tilted profile. If the electrode contains a pseudocapacitive material, a peak in the current in the reducing and in the oxidizing sweeps will overlay onto the rectangular response. By conducting CV at different voltage windows, the proper voltage window for operation can be identified. By changing the scan rate, one can assess the nature of the charge storage mechanism. In the simplest case, the peak current (or a current at a constant voltage in the flat region of the rectangular profile) is plotted against the scan rate in a log–log scale. A resulting slope of unity indicates a capacitive process, and a value of 0.5 indicates a diffusion-limited process. A more sophisticated analysis of cyclic voltammograms can be applied to deconvolute the contributions from Faradaic and non-Faradaic processes using Dunn's method.^[60,61] The latter method is especially useful when the supercapacitor electrode is examined in a half-cell.

Galvanostatic charge/discharge (GCD) is the accepted method for determining capacitance for commercial supercapacitors, as it correlates closely to how a supercapacitor is typically used in most applications. In GCD, a constant current is applied to charge the cell, and a constant current of equal magnitude but opposite sign is applied to discharge the cell within preset voltage limits. The “perfect” response of a supercapacitor cell in GCD is triangular, but small or minor plateaus can occur if there is a pseudocapacitive reaction. High internal impedance can result in a rapid voltage drop in the first few moments of charging or discharging.^[53] From GCD, the equivalent series resistance (ESR) is calculated from the drop in voltage when the current changes from full charge to full discharge divided by twice the current applied.^[62] From the measured data, the capacitance, energy, and power can be calculated (Appendix B, Equations B3 and B4). However, simplified equations based on a perfect triangular response should only be used when appropriate, and integration used for pseudocapacitive systems.^[62]

EIS is a tool for estimating the supercapacitor cell's ESR, capacitance, and characteristic time scale. The data are often modeled with a Randles circuit consisting of a capacitance, equivalent series resistance, and a parallel resistance.^[59] The capacitance dictates the energy storage capacity, while the ESR dictates the energy delivery.^[63] EIS can be conducted before and after extended GCD cycling to identify changes in the cell due to degradation.^[62] A supercapacitor typically has a very long cycle life, particularly if pseudocapacitive effects are avoided. Cycle life testing is typically performed by GCD or CV to characterize the capacitance/capacity retention after more than 1000 or 10 000 cycles.^[64]

4.3. Batteries

Batteries are characterized using many of the same methods described in Section 4.2, but some notable distinctions arise. First, in conducting CV, a pair of peaks attributed to the redox reactions of the electrodes is expected. Second, in conducting GCD, the discharge profile should have a flat voltage over a wide charge

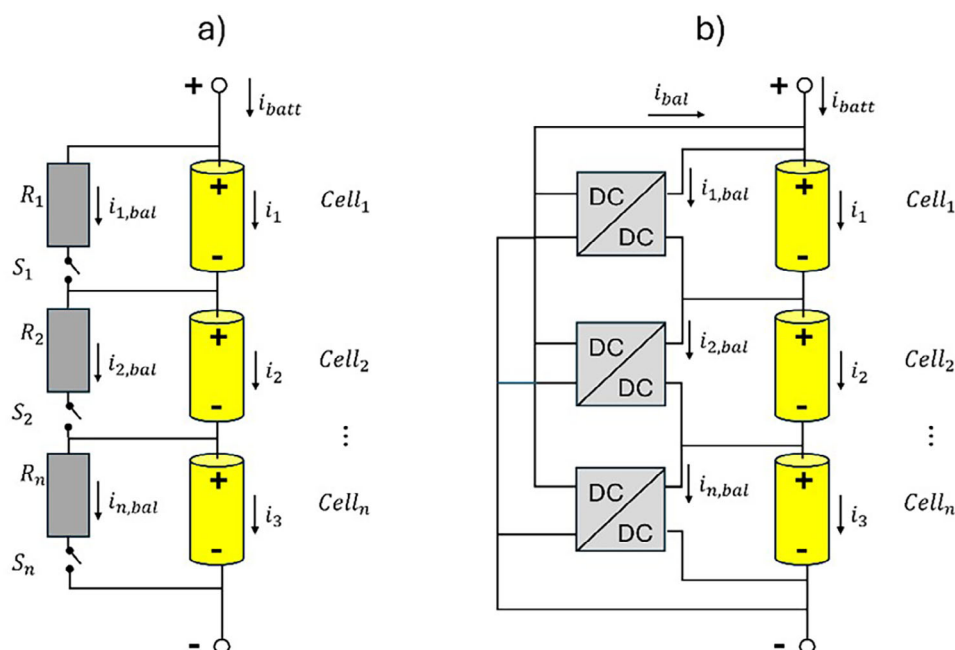


Figure 7. Example topologies for a) passive and b) active balancing circuits. The passive balancing circuit contains resistors (R_i) and switches (S_i), while active balancing is implemented through one DC-DC converter per cell.

window that coincides with the redox reaction. Finally, in EIS, the ESR can be determined as before, but a low-frequency tail will appear that is characteristic of ion diffusion limitations. Of all these methods, GCD and EIS are more commonly used for batteries, with GCD being of special use to determine both capacity and cycle life. Besides these standard techniques, other electrochemical characterizations can shed light on the battery's behavior. In a battery, the galvanostatic intermittent titration technique (GITT) is useful for determining the diffusion coefficient of Li-ions and polarization at each step.^[65,66] This method consists of a sequence of constant current pulses, followed by relaxation steps, while monitoring the open circuit potential, and fitting to a geometric diffusion model. This method is very time-consuming but provides the user with real-time information on the cell's state in a non-destructive manner.

With increasing interest in solid electrolytes for solid-state batteries, characterization of the bulk electrolyte becomes important. The main methods include EIS to obtain the ionic conductivity and certain methods to determine the electrochemical stability, including successive scanning by CV and increasingly large current pulses to monitor, for example, lithium dendrite formation.

Post-mortem analysis is especially paramount for understanding battery failure and mechanisms of degradation. Usually, a cycled cell is disassembled, visually inspected, and the electrodes are characterized using microscopy and spectroscopic methods. For example, cross-sectional Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) can reveal the presence of a SEI or CEI (solid or cathode electrolyte interphase) formation.^[67] Time of Flight Secondary Ion Mass Spectroscopy (TOF-SIMS) can reveal the composition of the SEI or CEI in a top-down surface milling approach. Similar information can be obtained from X-ray Photoelectron Spectroscopy (XPS).

These interphases protect the electrode and conduct lithium ions, but out-of-control interphase formation can lead to failure. In some cases, these processes can be observed by in situ methods.

4.4. Multicell and Engineering Performance

Multicell devices, comprising multiple cells connected in series or parallel, achieve the voltages and capacities required for specific applications. Characterization methods for single cells and multicell systems differ due to varying operational needs. Multicell systems add complexity, requiring assessments of cell balancing, thermal management, and pack integrity. Multicell testing includes thermal/electrical cycling and battery management system (BMS) evaluation for monitoring and control. Li-ion cells are connected in parallel to enhance current or in series to increase voltage. Cell variability, such as self-discharge rates and capacity differences, complicates multicell systems. In parallel, cells self-balance through conductive paths, minimizing discrepancies as long as open-circuit voltage differences remain low. In series, however, mismatches in cell parameters can cause performance degradation or hazardous conditions, like thermal runaway. Even without initial imbalances, differences between cells grow over time, reinforcing the need for cell balancing.

Balancing methods for Li-ion batteries are categorized as passive or active (Appendix B, Equations B5–B10). Passive balancing dissipates energy through resistors and controlled switches, typically used during charging to prevent overcharging or overdischarging by generating heat. Active balancing transfers energy between cells or to the pack, increasing efficiency but adding complexity (Figure 7). While passive balancing is simpler and primarily used during charging, active balancing functions

during charging, discharging, and idle periods to maintain performance and prolong battery life.^[68]

Effective thermal management is crucial, as high temperatures degrade performance and increase safety risks. Battery thermal management systems (BTMS) are essential for regulating temperature by employing cooling methods. Battery heating methods for cold climates are underdeveloped but critical for overall system performance.^[69] Performance metrics for multicell systems, such as energy density, power density, cycle life, coulombic efficiency, and safety, are tested under real-world conditions. Cycle life tests measure how many charge–discharge cycles a battery can handle before its capacity drops, while coulombic efficiency assesses the ratio of charge output to input, indicating how effectively the battery stores and delivers energy.

5. Challenges and Resulting Issues Associated with Characterization and Reporting for Structural Power Composites

5.1. Inherent Challenges

Before proposing best practices and protocols for structural power composites, it is helpful to outline the inherent challenges when attempting to characterize these multifunctional materials. Many of these inherent challenges do not apply or are less significant when characterizing structural composites or electrochemical cells in isolation. Consequently, researchers embarking on characterization and the subsequent reporting of electrochemical and structural data of structural power composites have encountered issues, which have led to invalid results and the drawing of incorrect conclusions. The inherent challenges described in this Section are partitioned into characterization and reporting challenges, although such a demarcation may not always be clear. These challenges are summarized in Table 3, and subsequently, the resulting issues that arise have been partitioned into those for constituents (Section 5.2.1), structural supercapacitors (Section 5.2.2), and structural batteries (Section 5.2.3).

5.1.1. Characterization

First, the current structural power composites (and their constituents) present atypical electrochemical and mechanical performance as compared to that of monofunctional cells and composites, respectively. For instance, the power densities of prototype structural supercapacitors are significantly inferior to those of conventional cells, and hence, the performance can be difficult to measure at typical current densities. Similarly, structural power composites present lower strength and toughness than conventional composites, leading to greater sensitivities to specimen preparation and mechanical test conditions. These atypical properties can undermine the suitability of conventional standard test configurations for characterizing multifunctional performance. However, as this technology matures toward practical levels of performance, this issue should become less significant.

Perhaps the most critical challenge is that the scale and configuration of coupons used for characterizing each function differ (Figure 8). For instance, electrochemical tests are often undertaken on coin cells 10–20 mm in diameter or length or pouch

cells, since testing on a larger scale can lead to anomalies associated with inefficient current collection. However, undertaking mechanical tests on such a small sample typically means that the results are dominated by edge effects and difficulties in ensuring controlled/uniform loading in the gauge section. This difference in “coupon” scale between mechanical and electrochemical characterization is a fundamental challenge for determining the multifunctional performance. Ideally, the same coupon should be used for both mechanical and electrochemical testing, as would be the case when such a material is used in service. Having the same coupon for both functions would also provide a much more credible and representative measure of multifunctionality. When coupon scales differ, the effects of the processing conditions introduce additional complications. Although processing influences monofunctional composites, for example, consolidation pressure dictates the fiber volume fraction, such effects are well understood. However, for structural power composites, fabrication parameters for small (electrochemical) cells may differ from those associated with larger mechanical test pieces, introducing additional discrepancies.

As with many emerging fields, the material synthesis during the early stages of development is nonoptimal, so the developed material is scarce and inherently presents high variability, imposing a constraint on achieving reproducibility (i.e., replicate tests). For mechanical characterization, it is well established that at least five replicates presenting valid failure modes are needed. Imposing such a requirement on structural power development may encounter challenges in achieving reproducible cells, and hence, the inherent scatter may be dominated by variations in cell manufacture. However, for this technology to advance, sufficient replicates for the characterization of both functions are essential.^[23,70]

From the perspective of mechanical characterization, device geometry often presents particular challenges. First, structural power composite laminates tend to be very slender, since thin electrodes minimize resistive losses associated with structural electrolytes and, in principle, allow stacking of multiple cells within a limited component thickness; the aspiration is to achieve cell thicknesses of no more than a few 100 μm . However, typical mechanical test standards define a minimum thickness of 2 mm. Conventionally, multiple (unidirectional) plies are stacked to build up this thickness, but, for structural power composites, stacking multiple cells to characterize the performance of a single cell is nonoptimal and expends considerable material. An alternative approach, manufacturing monofunctional/multifunctional hybrids, significantly complicates any data reduction. Therefore, characterization using direct loading rather than bending tests is preferable, since for the latter, the low thickness would lead to non-linearities and large deflections. For similar reasons, conventional delamination tests can only be considered by using doublers.^[48] However, further problems arise because the devices are usually hybrids: typically, carbon fiber electrodes sandwiching an insulating separator layer. The hybrid character presents difficulties during bending tests since there is a greater likelihood of delamination between plies. Similarly, delamination tests are thwarted by the asymmetric loading at the interface. For structural batteries and hybrid systems, the positive and negative electrodes will be different, which could lead to stiffness coupling during load application. This asymmetry will complicate the mechanical characterization, even when trying to deduce the elastic

Table 3. Summary of challenges associated with structural power composites.

Challenge	Details	Resulting issue
Characterization		
Atypical electrochemical and mechanical response	Structural power composites present very different properties from conventional laminates or electrochemical cells.	Adopting conventional test parameters and measurement protocols can lead to erroneous results.
Difference in scale between coupons	Electrochemical coupons are usually small, while mechanical coupons need to be large to ensure uniform stress states.	Mechanical tests from small samples are dominated by edge effects, presenting anomalous properties. Electrochemical data from large samples are dominated by current collection issues. Leads to inconsistencies in the contrasting and reporting of multifunctional performance.
Scarce material and replicates	Material is under development, so quantities are scarce and processing immature.	Mechanical tests require relatively large quantities of material, leading to high variability between replicates.
Device geometry	Devices tend to be thinner than the minimum thicknesses specified by mechanical test standards.	Mechanical tests present issues with load introduction and high deformations, leading to erroneous data.
Hybrid laminates	Devices are hybrids and may be mechanically unbalanced.	Mechanical characterization of unbalanced devices leads to stiffness coupling effects and invalid results.
Mechanical testing environment	Should devices be tested “dry” or with electrolyte present?	“Dry” devices may not reflect the actual mechanical properties of multifunctional material, while instrumentation of non-dry devices can be problematic.
Electro–mechanical coupling phenomena	In batteries, electrochemical cycling can negatively affect the mechanical properties due to constituent volume changes.	Mechanical data from electrochemically uncycled and cycled cells differ.
Function coupling phenomena	Devices can present interaction between mechanical and electrochemical functions, such as from electro–mechanical effects or damage development.	Data generated under monofunctional conditions may not be representative of that which would occur in service.
Reporting		
Normalization	No clear electrochemical protocols as to how to normalize measurements when reporting specific performance.	Ambiguities and inconsistencies in the reporting of multifunctional performance, particularly when comparing with background data.
Out-of-plane pressure	Device electrochemical performance is sensitive to applied pressure, presenting strong electro–chemo–mechanical coupling.	Discrepancies between the literature as to what defines the “device.”
Current collectors	No agreed protocol as to whether current collectors should be attached during mechanical testing.	Inconsistencies in characterization conditions leading to discrepancies in contrasting and reporting across datasets.
Encapsulation	Challenges in partitioning the test piece’s mechanical response from that of the encapsulation material.	Discrepancies in coupons used for the reporting of multifunctional data.
Multicell assemblies	There are many possible arrangements for assembling multiple cells (such as planar, tiled, stacked, offset) and many ways of electrically connecting the cells to each other (series and/or parallel) to achieve desired voltages and currents.	Discrepancies between the literature as to what defines the “device”, leading to inconsistencies when contrasting across datasets. Ambiguities in test definitions and reporting of data.

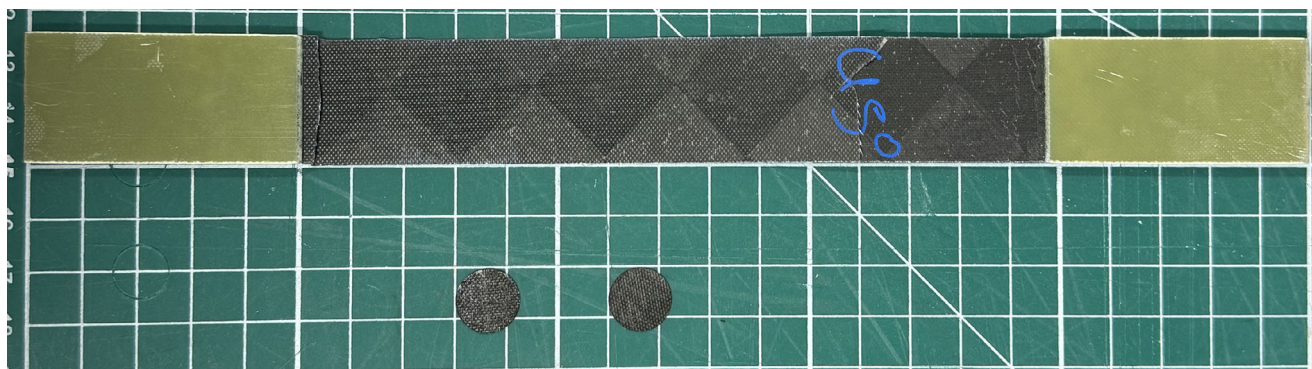


Figure 8. Comparison, to scale, of electrochemical (coin cell electrodes) and mechanical (tensile) coupons. Note the grid scale is 1 cm.

response; the problems may be exacerbated by volume changes associated with changes in the state of charge. However, there is a growing body of work on the characterization of hybrid composites, which could be drawn upon to direct the test development of structural power composites.^[71]

A particular challenge for mechanical testing of structural power composites is the need to attach strain gauges or other instrumentation to their surfaces. Structural electrolytes often contain a liquid phase such that the electrode surface “sweats” during testing. Even if noncontact methods are employed, such as optical strain gauges or digital image correlation (DIC), applying paint or patterns to the surfaces can be problematic.

A further challenge is that of the environment in which structural power composite cells can be characterized both electrochemically and mechanically. For instance, mechanical characterization of electrode tows or lamina has often been undertaken in “dry” conditions (i.e., without the electrolyte present). However, it is likely that the mechanical behavior of an electrode is influenced by the presence of the electrolyte, particularly for properties which are sensitive to interfacial effects. On the other hand, micromechanical test methods have been developed in which tows have been infused with resin; such tests could be an inspiration for characterizing multifunctional constituents.^[72] To quantify the true multifunctional performance of cells, it is imperative that mechanical and electrochemical tests are performed under the same conditions (i.e., with the electrolyte present).

A challenge yet to be fully investigated is the coupling phenomena that can occur between electrochemical and mechanical functions (Section 5.3). For example, mechanical damage introduced during loading could lead to a change in the electrochemical response (or vice versa). A further example, which has been demonstrated in structural batteries, is that even in the elastic regime, the electrochemical response is influenced by the introduction of mechanical load. In fact, this effect has been usefully exploited for sensing, actuation, and even energy harvesting.^[2] However, the volume and stiffness changes during electrochemical cycling present a complex challenge. Moreover, in structural batteries, the mechanical response is influenced by the number of electrochemical cycles the cell has experienced prior to mechanical testing. Therefore, when undertaking coupon tests, it will be important to account for such phenomena and quantify the associated coupling parameters.

5.1.2. Reporting

Now, considering the reporting of structural power composite performance, as with conventional electrochemical cells (see Section 4), perhaps the most critical challenge is that of “normalization.” The melding of mechanical and electrochemical characterization has led to difficulties in defining how multifunctional performance should be reported. This question is particularly important because, without consistently defined ranking parameters, comparison across datasets is impossible, making industrial uptake of this technology unsupportable. First, considering constituent-level tests, for electrode development, the convention is to report performance (such as charge capacity or capacitance) normalized by mass of active material. For a conventional electrode, the active material mass may be close to the total electrode mass, but for a structural electrode, which typically comprises carbon fibers decorated with electrochemically active materials, ambiguities arise. Since the carbon fibers can account for a significant mass, including them in the definition of the electrode mass will significantly depress the apparent specific electrochemical performance. But on the other hand, only considering active material mass would not provide a useful measure of the performance of the overall structural electrode. Furthermore, such an approach would make a specific performance comparison with the typical baseline (i.e., pristine carbon fibers) nonsensical. Regarding the mechanical performance of constituents, modulus, strength, and toughness are never normalized by mass (or density). Finally, for porous electrode fibers, it is important that when calculating strength and modulus, the gross (not net) cross-sectional area is used.

When considering structural power composite cells, even more ambiguities arise regarding normalization. From an end-user perspective, declaring performance normalized by electrochemically active material or electrode mass does not provide a meaningful measure. The analogy here is the specific mechanical performance of a composite laminate: properties are always defined as normalized by the entire laminate mass, rather than that of the fibers alone. Therefore, normalization by cell mass is essential but raises the question as to the definition of a cell in a multifunctional context (see Section 2.1). For instance, should normalization only include the electrodes, separator, and electrolyte (i.e., active cell in Section 2.1) or should the current collection and encapsulation mass also be considered (i.e., full cell

in Section 2.1); this issue is addressed in Section 6. Similarly, at the cell level, ambiguities in normalization arise regarding key parameters such as specific power and energy. Moreover, these parameters vary as the applied voltage squared (Appendix B, Equations B1 and B2), and since this voltage is principally dictated by the electrolyte, it will have a significant influence on the magnitude of the specific performance. Areal performance (e.g., F cm^{-2}) is perhaps a useful measure that resolves the issue of active material, electrode, or device normalization. However, this metric does not account for the mass (or volume) of a device, so it may be difficult to relate to any final application. In summary, standardization of normalization at a cell level is clearly an important challenge that needs to be addressed. It should be noted that to avoid confusion in reporting performance, it is important that consistent units are used throughout.

A further challenge is associated with the applied out-of-plane pressure exerted on a cell during electrochemical characterization. For conventional coin cells, the electrode area is relatively small, so current collection is highly efficient. Therefore, the pressure on the cell may have relatively little effect on the apparent performance and can be standardized by an internal spring. However, for structural power composites, particularly structural supercapacitors, it has been shown that the electrochemical performance is very sensitive to applied pressure.^[3,73] This sensitivity is partly attributed to pressure promoting fiber-to-fiber contact within the electrodes, increasing electrical conductivity, which results in an enhanced apparent performance. On the other hand, consolidation may displace liquid electrolyte from within the cell, depressing the electrochemical performance, or even cause shorts. While conventional cells are usually small, structural power composite cells tend to be of a larger area, so these effects become more dominant. There is clearly a need to characterize and report the pressure exerted on cells during electrochemical testing. This, of course, raises the issue as to whether mechanical tests should also be undertaken while under applied pressure. Applying out-of-plane pressure/compression while concurrently applying in-plane load to a mechanical test coupon would be extremely challenging to achieve and control.

A key requirement for electrochemical characterization of structural power composites is the need for current collectors: metallic films or tabs on the exterior of the device, which draw electrical current from carbon fiber-based electrodes. However, during mechanical characterization, the current collectors present challenges. They may inadvertently carry some of the mechanical load, leading to anomalies in the test results. They may also interfere with surface instrumentation such as strain gauges or speckle patterns for digital image correlation. These considerations would suggest that mechanical characterization would be better implemented without current collectors, although this would make electrochemical–mechanical testing of devices impossible (see Section 5.3). It should be noted that there is a lot of research on integrated sensors and devices in conventional composites; such research could provide inspiration regarding mechanical testing of multifunctional cells that have current collectors attached.^[74]

Finally, to isolate conventional cells from the surrounding environment and to ensure none of the constituents can escape, they are typically encapsulated within an inert film (e.g., a pouch). This encapsulation means the device is in a stable condition dur-

ing electrochemical testing. However, for multifunctional composites, encapsulation presents a challenge when undertaking mechanical characterization since it hinders load introduction into the coupon. Mechanical characterization of structural power devices has generally been undertaken without encapsulation. Such tests provide a direct measure of mechanical performance, but can be problematic because, without encapsulation, the environment could modify the response of the cell. For studies that have encapsulated cells in conventional composite laminates or pouch cells, it is important to partition the mechanical contribution of the cell from that of the encapsulation material. This analysis can be challenging for test methods such as flexural tests. Typically, mechanical load is introduced into the test piece through end-tabs, but the processes of attaching and machining could influence the parent material. This also raises the question as to whether or not the encapsulation material should be adhesively bonded to the test piece. Such bonding could leach out any liquid electrolyte (thus modifying the cell). If there were no bonding, such that there is no load transfer between the encapsulation material and the cell, this situation would negate the use of strain gauges on the coupon. The role of encapsulation during mechanical testing requires careful consideration when developing test protocols.

5.2. Issues Particular to Constituents and Devices

The previous Section detailed the generic issues associated with characterization and reporting of structural power composites. In this Section, the issues specific to the constituents and the different devices (i.e., structural supercapacitors and structural batteries) from the background literature are identified and discussed. A range of different methodologies have been pursued to characterize the mechanical and electrochemical performance and hence report multifunctional performance. However, many publications culminate by comparing their data against the background literature, typically using Ashby plots of, for example, specific capacitance versus strength.^[75–77] Unfortunately, difficulties arise because the background data has invariably been generated using different methods or even normalized differently to that of the foreground data. Therefore, such comparisons can be misleading and should be treated with considerable caution.

5.2.1. Constituents

First, consider electrochemical methods used to characterize constituents. Characterization of structural electrodes has often used single bundles or (spread) tows of fibers in a two or three-electrode cell filled with a liquid electrolyte.^[76,78–84] In some instances, coin cells have been used to characterize the structural electrode material.^[85,86] Some authors used a liquid electrolyte, which is consistent with the multifunctional structural electrolyte used in a cell (e.g., the same ionic liquid), hence providing a possible comparison to the cell performance. However, in instances where the electrolyte is different from that in the cell, it is unclear whether the performance at the electrode level can be usefully compared with that in the final cell.

Characterization of the structural electrolytes is usually undertaken using EIS to determine the ionic conductivity, although

sample size, dimensions, and testing conditions can vary.^[84,87,88] The electrolyte test specimens can be cut from a larger sample and polished before being placed between two electrodes, synthesized in the mould of the desired dimensions, or deposited directly onto the test electrodes.^[89–91] Some authors provide information regarding dimensions, number of replicates, as well as details of the testing conditions, i.e., frequency range, temperature, etc.^[92] while others do not.^[90] The frequency range chosen is particularly important as characterizing above 250 kHz will potentially introduce instrumentation noise, thus affecting the results.

Separators can be ranked by testing cells made using different separators while keeping all other parameters the same.^[23] The main characteristics of the separator that are likely to influence the electrochemical performance are thickness, porosity (and the related tortuosity), interface properties (such as wettability by the structural electrolyte), and how well the separator prevents shorting, while the density affects the specific performance. The separator is expected to affect the power to a greater extent than the energy storage, and so characterization of the ESR is most pertinent for evaluating separator electrochemical performance. Characterization of a separator in a cell would be particular to that combination of materials because the interfaces between the separator and the other constituents play an important role. Therefore, caution needs to be taken when trying to infer the performance of the separator if it were to be combined with other materials not tested, such as different electrodes or different structural electrolytes. Commercially available separator datasheets may provide measurement data for separator properties in common electrolytes.

The principal characterization techniques for current collectors focus on measurement of their intrinsic bulk electrical resistivity, both in-plane and through-thickness, using the four-probe technique^[73] and contact resistivity at the interface with the electrodes using the transmission line method^[93]. Electrochemical stability is another important requirement for current collectors that are susceptible to corrosion when in contact with electrolytes at varying potential. The electrochemical stability of current collectors in electrolyte has been characterized using CV of pouch cells,^[94] while EIS has been used to characterize corrosion behavior.^[95] The electrochemical performance of carbon fiber versus lithium metal half-cells with screen-printed collectors has been measured to be similar to reference half-cells using metal foil and silver adhered metal-foil collectors.^[96] Current collector foil surface morphologies have been characterized using SEM before and after tensile testing.^[97]

The encapsulation needs to insulate and provide a barrier to moisture and oxygen for chemistries that are liable to lead to degradation in ambient conditions.^[98] The water vapor transmission rate has been characterized by moisture permeability tests and diffusion models on both fresh and environmentally stressed lithium-ion pouch cells.^[99] Characterization of the electrochemical interaction between the encapsulation material and electrolyte has entailed solubility parameter analysis and gravimetric electrolyte uptake measurements of polymers cured at different temperatures.^[100] Gel fraction measurements and differential scanning calorimetry analyses have been used to assess the state of cure and optimize the cure temperature for the epoxy to achieve a highly crosslinked structure with low sol extraction.^[100]

Electrochemical tests on encapsulated cells have been used to characterize the electrochemical performance of a cell after encapsulation. For example, a carbon fiber reinforced structural battery cell encapsulated by a CFRP laminate has shown stable charge–discharge cycling performance over 50 cycles.^[24] Similarly, a structural supercapacitor using an epoxy PVA/KOH-based electrolyte encapsulated in glass fiber fabric prepreg has been demonstrated to retain 87% of its initial capacitance after seven days of cycling.^[101] However, other studies have encapsulated structural supercapacitors that contain epoxy and ionic liquid structural electrolytes using glass fiber fabric prepregs. Even after B-staging the prepreg, the encapsulation led to significant reductions in electrochemical performance (reduction in capacitance and increase in ESR) immediately after manufacturing due to the interaction of the ionic liquid with the uncured epoxy matrix.^[3]

Structural electrodes have often been mechanically characterized using established tow and bundle tests, such as ASTM C1557.^[102] These tests are usually undertaken in the “dry” condition, i.e., without the electrode having been exposed to liquid or structural electrolytes.^[70,78,85,103–108] Tension testing of electrode material has been reported using DMA, with specimen dimensions typically of the order of 10 mm × 2.5 mm. Because the structural electrodes are relatively stiff, while DMA test fixtures can be quite compliant, the reported moduli may be underestimates.^[108–110] Some studies have characterized the performance of structural electrodes, which have been infused with conventional structural matrices (such as epoxies), providing a baseline monofunctional performance.^[111] In some instances, where the electrode is a non-woven veil of nanofibers, the intrinsic modulus of the nanofibers, rather than that of the electrode itself, has been reported, which is very misleading.^[112] Similarly, some studies on porous fibers (carbon nanofibers) for electrodes have used the net, rather than the gross, sectional area of the fibers to calculate the strength, which again does not provide a reasonable parameter to compare the performance to other electrode embodiments.^[75–77]

The mechanical properties of small-scale specimens of structural electrolytes are commonly characterized in three-point bending^[81] or tension loading using dynamic mechanical thermal analysis (DMTA). Such measurements serve as ranking tools, allowing some qualitative measure of performance, but do not offer a reliable measure of the mechanical performance of structural electrolytes. Some characterization of structural electrolytes has been undertaken in compression of cylindrical specimens^[87,103,113,114] as prescribed in ASTM D695.^[115] Such a test method can provide good insights into the mechanical performance. However, the porous nature (i.e., infused with the ionic conducting phase) of many structural electrolytes can present a constitutive response very different from that of monolithic solid polymers. Finally, no interfacial testing has been reported on characterizing electrode/electrolyte interfaces.

In carbon fiber reinforced structural power composites, the carbon fibers would support most of the mechanical loads, so the interface properties between the separator and its surrounding constituents are more important than the mechanical properties of the separator itself. Therefore, as with electrochemical characterization of separators, it is more meaningful to characterize different separators within a device or at least an assembly including the structural electrolyte and one electrode. Even

parameters such as the propensity for the separator to resist short circuiting can depend on the device manufacturing process and the nature of the surrounding structural electrolyte and electrodes. Such issues make it challenging to develop a universal way to test separators alone to assess their robustness. There is a lack of comprehensive statistical studies to quantify the probability of various separators being successful in preventing short circuits. Various studies^[3,23,116] indicate that some separators (such as a thin glass fiber fabrics) are more prone to shorting than others. However, there are no universally employed test methods to check whether a separator has good resistance to shorting or good interlaminar shear strength at the interface between electrodes and structural electrolyte. More often, the approach has been to mechanically test devices and then gain qualitative insights about the mechanical performance of the separator–electrode or separator-structural electrolyte interface through microscopy and fractography.^[23,117]

The tensile properties of current collectors have been characterized using micro-tensile tests with a laser speckle extensometer as a noncontact optical strain sensor.^[118] Mechanical and electrical contact, particularly at the interface with the electrodes, is necessary to ensure integrity during electrochemical^[119] and/or mechanical cycling, as characterized using flexural tests.^[120]

Encapsulation materials for batteries have been mechanically characterized using tensile, single lap shear strength, and three-point bending tests before and after exposure by immersion in the electrolyte for up to three months.^[100] Tensile and flexural tests using three-point bending have also been used to characterize the mechanical properties of encapsulated structural supercapacitors.^[121] Overall, various test methods have been used to determine the physical, electrochemical, and mechanical properties of encapsulation materials. However, no standardized methods have been universally applied as metrics to define successful structural encapsulation that would permit like-for-like comparisons across the literature.

5.2.2. Structural Supercapacitors

Efforts to characterize the electrochemical performance of structural supercapacitors and their constituents have mainly drawn upon conventional electrochemical tests as described in Section 4.2. Electrochemical characterization of devices has used GCD,^[80,81,93,111,112,122–126] EIS,^[80,112,117,122–125,127,128] and CV,^[23,70,79,82,109,117,122,124,125,127–129] and often model the cell as a Randles circuit to deduce the critical parameters.^[111] Devices are usually free-standing or may be assembled in a coin cell to characterize the performance.^[108–110,130] Generally, the applied pressure at which the device is characterized is not reported, despite this greatly influencing its performance.^[130] Similarly, the number of electrical cycles applied prior to the reported performance is not always described, although such standards exist for conventional devices (Section 4). Finally, the relatively high ESR of structural supercapacitors means the discharge performance can be significantly lower than that during charging.

Some authors have compared the device performance to that of a “monofunctional cell”, using the same electrodes and separators but only the ionic conducting constituent of the structural electrolyte.^[23,79,117,122,127–129] It is notable that structural su-

percapacitors have inferior ion mobility to monofunctional cells comprising only liquid electrolytes, so the range of current densities over which a multifunctional cell can be characterized can differ significantly from those for monofunctional cells. This discrepancy raises a particular issue when reading across between electrode and cell characterization, complicating the interpretation (and comparison) of device performance.^[80] Researchers have reported immersing their cells (e.g., with an ionic liquid-based structural electrolyte) in very different electrolytes (e.g., aqueous electrolyte) during electrochemical device characterization.^[131–135] Such an approach does not provide a true measure of the electrochemical performance of a device while in service, and it is therefore problematic to use such an approach to glean multifunctional performance.

Across the literature, there has been no consistency in the device size used for electrochemical characterization. Tests have ranged from coin cells (with device areas of less than 1 cm²) to devices over 400 cm² in size. Device size is a particular issue for structural supercapacitors since the high current densities associated with their high power density mean in-plane current collection has a major influence on the apparent performance.^[3] The resistive losses associated with the relatively poor conductivity of the electrode material (typically carbon fibers) can mean, depending on current collector configuration, that large devices typically possess significantly inferior performance when compared to small cells. For example, while the resistivity of typical aluminum current collectors is 0.028 μΩ m, carbon fibers have a resistivity of the order of 10 μΩ m.^[136] Finally, across the literature, no common or consistent current collection configuration has been used, despite this having a strong influence on the apparent device performance.^[73]

Several studies of structural supercapacitor devices have neglected to undertake formal mechanical tests but have demonstrated a structural capacity by hanging weights from devices, etc.^[105,106,137] Such an approach is not rigorous for quantifying the performance of structural power composites. Tensile tests adopting the standard ASTM D3039^[138] have been widely used to determine the modulus and strength of structural supercapacitors.^[70,83,84,106,107,117,125,127,130–135] It should be noted that such tests deviate from the standard since it has not been defined for hybrid or thin laminates. Tensile tests are dominated by the reinforcement (i.e., structural electrode and separator constituents) and provide little insight into interfacial or structural electrolyte-dominated properties. Tensile strength, rather than modulus, is usually reported, but the latter is the more useful parameter for design. For tensile tests, it is important that direct strain measurement (i.e., gauges or noncontact devices) is used rather than crosshead displacement. Several papers have reported tensile strains for carbon fiber-based devices exceeding 5% and hence underestimated the modulus; such excessive strains are an artifact of neglecting to account for test machine compliance.^[131–135] A further test which has been widely used to characterize structural supercapacitors is the ASTM D3518^[139] ±45° in-plane shear test.^[81,87,104,111,113,117,123,128,129,131–133] This test accommodates thin test specimens with quite a limited gauge length and permits matrix and interfacial dominated properties to be characterized. Difficulties with this test method are that it requires a biaxial strain measurement, and the coupon response at large strains can be very nonlinear. One study^[140]

investigated the compression behavior of thin structural supercapacitors using the sandwich beam configuration as described in ASTM D5467.^[141] This test relies on bonding the device onto a sandwich beam and being able to attach strain gauges.

Flexural tests of structural supercapacitors, using ASTM D790,^[142] have been pursued by several researchers.^[83,86,107,130,143] Such tests are challenging because the laminates are very thin (hence large deflections) and prone to delamination. Performing flexural tests on very small specimens in a DMTA may be problematic because edge effects may dominate, and, therefore, they should only be used for qualitative ranking.^[128,129,144] As of yet, no one has investigated the delamination resistance of structural supercapacitors to determine the fracture toughness of the electrode/separator interfaces. However, some work to characterize the impact response (ASTM D5628)^[145] of structural supercapacitors has been undertaken.^[134,135]

The fiber volume fraction determines the mechanical properties of structural composites and energy storage devices, and should be reported. Mechanical properties, particularly the modulus, are very sensitive to the proportion of the reinforcement. The standard methods to determine the fiber volume fraction are detailed in ASTM D3171, suggesting acid digestion or pyrolysis of the laminate to isolate the fibers from the matrix.^[146] Several authors have attempted to characterize the volume fractions of structural supercapacitors.^[87,113,117,126,128,129] However, this methodology can be challenging because the range of constituents is greater than that in conventional composites, and not all non-fiber components may be removed as expected.

5.2.3. Structural Batteries

The most common electrochemical characterization for structural batteries has been galvanostatic (constant current) charge and discharge cycling. As for structural supercapacitors, the interpretation (and comparison) of the device's performance can be complicated since researchers use different charge/discharge currents (C-rates relating to charge/discharge times) when reporting energy density. As with structural supercapacitors, they are highly sensitive to the charging rate and current densities, with low charge rates providing much higher energy densities. EIS is often also used for determining internal resistances, but the state-of-charge in which the tests have been undertaken is often not reported.

The reported mechanical properties are mainly tensile modulus and strength in the fiber direction of structural battery laminates; properties transverse to the fiber direction are rarely reported. Although these properties are challenging to measure, they should be considered since electrochemical cycling will most likely have a greater influence on such structural electrolyte (matrix) dominated properties due to electrode expansion. To date, no standard test protocols have been followed because structural battery materials are made on a small scale with low thickness and do not comply with established test protocols. In some instances, researchers are using liquid or gel electrolytes and still report tensile modulus and strength in the fiber direction, which is viable. However, such structural batteries have no load-bearing capacity in shear, bending, or compression, making comparison very difficult with concepts using structural electrolytes.

More so than supercapacitors, batteries can be very sensitive to moisture and oxygen during operation. Therefore, mechanical tests on structural batteries usually need to be performed in dry and oxygen-free environments: inside a glovebox if tested without encapsulation. Mechanical tests must then either be done with cells enclosed in a pouch bag to prevent moisture and oxygen intrusion and electrolyte evaporation; this issue complicates the evaluation of mechanical properties, as detailed in Section 5.1.1. Another option is to remove the pouch bag, dry the cells, and test them in ambient conditions, which could also be misleading, as that is not the environment where the structural battery would be used. A further complication is the interface between the fibers and the electrolyte. It must be multifunctional, providing both mechanical load transfer and allowing for ionic conductivity. Very few studies have been reported on this parameter, although some work has been done using the microdroplet debond test.^[147]

5.3. Coupled Electrochemical–Mechanical Characterization

In the elastic range, there is no evidence that the application of mechanical strain will influence the electrochemical performance of structural supercapacitors. However, it is conceivable that as mechanically induced damage develops within the device, the electrochemical performance could change. For example, benign cracking in the electrodes may result in both increased exposed surfaces (enhancing capacitance) but increased resistivity (depressing power density). Therefore, there is a need to characterize the relationship between electrochemical and mechanical performance for structural supercapacitors. However, for such tests to be undertaken successfully and to generate useful data, it is important that the measurements of each function reflect the true material response rather than artifacts of the specimen configuration. For instance, the large coupons necessary to characterize mechanical response will present an inferior electrochemical performance to the standard coin cell or Swagelok configurations. In such instances, optimization of the current collectors provides a route to realize the intrinsic performance of the cell.^[73]

Using standard mechanical coupons, there have been efforts to characterize the influence of tensile^[137,148,149] and bending loads^[83,133,134] on the electrochemical response of structural supercapacitors. These studies demonstrated a degradation in electrochemical performance once mechanical damage developed within the cell. However, the experimental details in these studies were sparse. Potential issues that will need to be addressed for such tests include ensuring robust electrical connections, and any deformation or damage that occurs to them does not manifest as a change in the electrochemical performance. Similarly, ensuring that the device is electrically isolated from the metallic test fixtures is vital. One approach could be to encapsulate the cell, but that introduces the challenge of partitioning the response of the encapsulation from that of the cell.

In a battery, contrary to supercapacitors, the constituents undergo large volume changes during electrochemical cycling, and the elastic moduli of the active materials, e.g., carbon fiber and LFP, will change with state of lithiation. This feature will strongly affect the internal stress state, which will vary with state of charge.^[150–153] These effects stem from lithium insertion strains in the electrode materials.^[154–156] This implies that substantial

lithium insertion strains in the carbon fibers (1% in the axial and 7% in the radial direction) will be exposed to high mechanical stresses in the structural battery in service, resulting in an immediate and strong coupling of the electro-chemo-mechanical processes in a structural battery.^[2,156,157] The expansions and contractions of carbon fiber electrodes during electrochemical cycling can cause damage to the structural electrolyte. The volume changes created by the charge/discharge cycles lead to a form of mechanical fatigue, which is still not understood. These volume changes can also be exploited to realize morphing composites.^[2,158] The cell voltage of a structural battery cell will be affected by the applied mechanical stresses and strains which can be used for sensing and energy harvesting.^[2,154] It is therefore important to characterize the coupling between electrochemical and mechanical functions, and must be considered in any combined electrochemical and mechanical test.

As with structural supercapacitors it is anticipated that mechanical damage will result in changes in electrochemical performance. Developing test methods to simultaneously characterize mechanical and electrochemical properties is an important area for future development for all structural power composites. This will be necessary for identifying new phenomena, underpinning predictive modelling and certification of these materials.

5.4. Multicell Assemblies and Components

General engineering applications require large assemblies of multiple cells to achieve the required voltages for the electrical systems. The need to characterize multicell assemblies integrated into a component presents challenges beyond those for characterization and reporting for single cells. It is relatively straightforward to control the test conditions during tests on individual cells before assembly. However, after assembly and integration into a component, each cell might then encounter different conditions. For example, the through-thickness pressure or other mechanical loads on the cells might vary at different locations in a component and be difficult to control, which may affect their measured performance. There are also various ways of electrically connecting between multiple cells within a component (e.g., using integrated wiring) that would not be used when testing single cells. Therefore, the conditions for testing multiple cells inside a component may vary depending on the component.

Conventional electrochemical cells are typically small ($\approx 5\text{--}10$ cm long). It is anticipated that structural embodiments will be much larger (by about an order of magnitude) because mechanical performance, particularly stiffness, is dictated by the joints within a component. Hence minimizing the number of cells is vital to ensure efficient load transfer. Given the aspiration is to manufacture large cells, there is therefore a need to demonstrate that large cells will perform as well as small cells both mechanically and electrochemically.

6. Proposed Protocols and the Reasoning Behind Them

6.1. General Methodology

As discussed in Section 2.1 (Figure 4), we have taken the pragmatic approach to partition into three levels: *Constituents*, *Cells*,

and *Components*. First, for the *Constituents* level it is not strictly necessary to use the same geometry or coupon for both mechanical and electrochemical characterization, since the resulting data is usually used to provide constitutive behavior inputs for predictive models, engineering design, or for simple ranking of different processing parameters, etc. Hence, there is freedom to adopt conventional mechanical and electrochemical protocols for these materials.

For the *Cells* level, the conflicting requirements of the standard configurations for mechanical and electrochemical testing manifest (Figure 8). While some useful data can be obtained by subjecting the same multifunctional material to independent mechanical and electrochemical tests, multifunctionality is most credibly demonstrated by performing the tests on the same coupon (i.e., a “universal coupon”). Ideally, the data are collected simultaneously to investigate “coupled” phenomena (Section 7). Such a universal coupon will need to be large enough to negate the issues that confound small mechanical test specimens, such as edge effects, nonuniform stress and Saint-Venant’s principle. Therefore, in prescribing such a universal coupon, the electrical losses that depress the apparent electrochemical performance in large samples will need to be addressed. That is, any electrical losses related to current collection will need to be partitioned from the intrinsic “material” performance, such that an intrinsic multifunctional performance can be declared. For the final level (*Components*) the geometry will be predefined, and therefore characterization of each function in isolation, and in combination, will not be subject to the same differences in configuration seen for the other two levels.

For all these levels, the approach to data normalization will need to be considered. Since one of the principal motivations for reporting data is to compare with the background literature, it is important that all the data is declared. With the definitions outlined in Section 2.1 in mind, it would be reasonable to explicitly report normalization by mass, volume and area of a *Half-cell*, *Active cell*, *Full Cell*, and *SPC device* (Figure 9). These are discussed further in Section 6.3.

An approach which has been pursued by several researchers is to compare the performance of structural power composites against that of “monofunctional” equivalents (Figure 10).^[79,111,117] Such an approach mechanically characterizes a *Monofunctional Laminate* using identical reinforcements to that of the multifunctional cell (i.e., electrodes and separator) but with a structural matrix. Similarly, a *Monofunctional Device*, again consisting of identical reinforcements to the multifunctional device, but with a conventional (liquid) electrolyte is electrochemically characterized. If the structural electrolyte in the multifunctional device is a blend of a structural matrix and an ionic conductor (e.g., ionic liquid), it would be reasonable to make the monofunctional equivalents using these materials, assuming processing conditions permit. Such an approach provides insights into how multifunctionality has impacted on electrochemical and mechanical performance of the monofunctional equivalents. A further extension of this principle is to establish the influence of the reinforcements (electrodes and separator) by comparison with conventional baselines: COTS laminates and cells. As shown in Figure 9, this could be achieved by characterizing a *Baseline Laminate*, which uses as-received fibers and comparing to the performance of the *Monofunctional Laminate*. Similarly, a *Baseline Device*, which uses

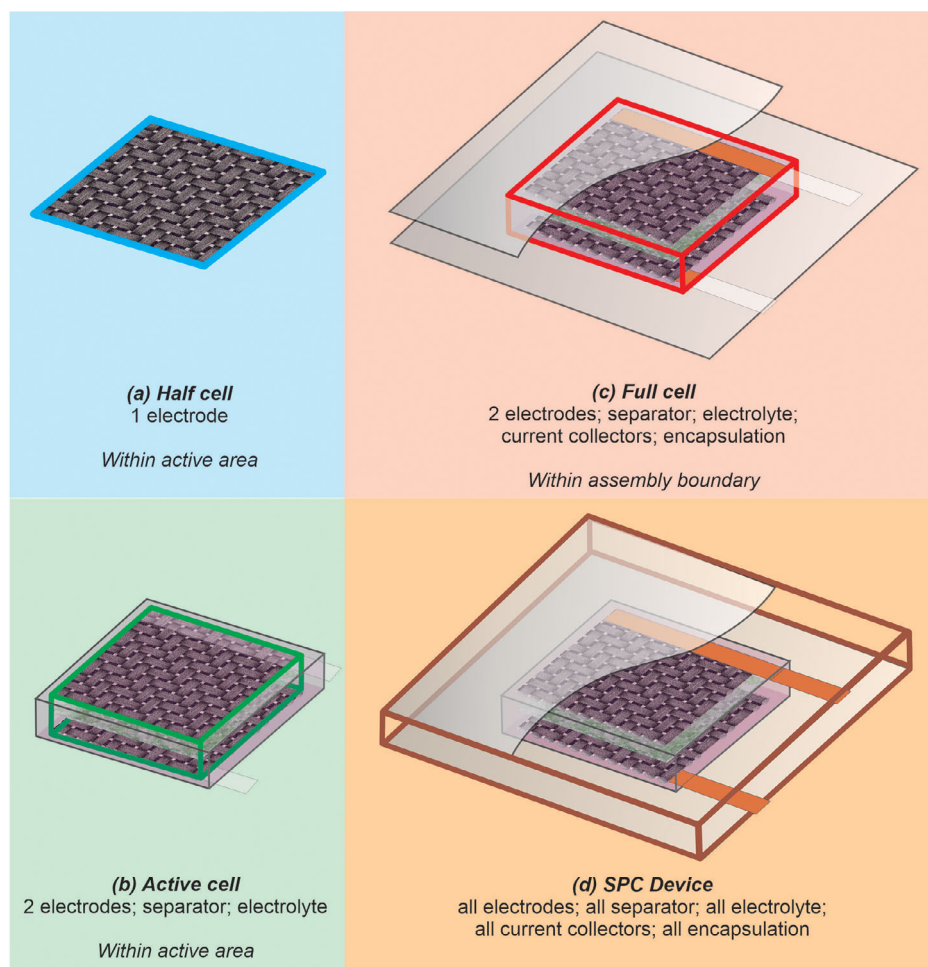


Figure 9. Boxed regions presenting suggested definitions for a) *Half-cell* (blue); b) *Active cell* (green), c) *Full cell* (red) and d) *SPC device* (brown) for reporting of normalized performance.

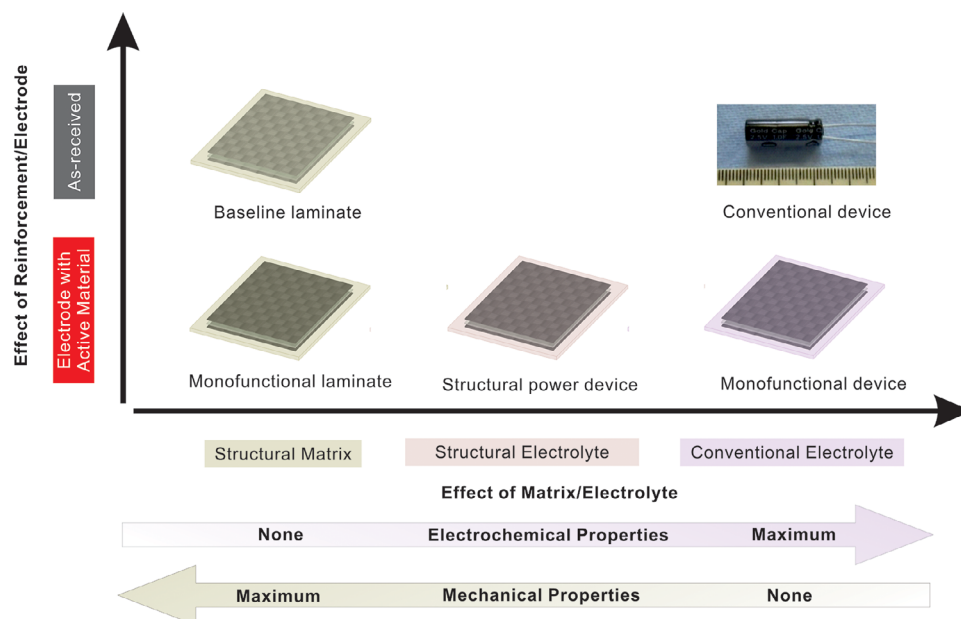


Figure 10. Proposed strategy to quantify influence of multifunctionality on each function.

conventional electrochemical materials, could be compared to the *Monofunctional Device*.

A good approach to improving the veracity and robustness of the published data on structural power composites would be to adopt guidelines and reporting checklists which are emerging for conventional devices. Good practice guidelines,^[64,159] common pitfalls,^[9,58] suggested standards,^[160,161] protocols,^[162] recommended metrics^[163] and reporting checklists^[54–57] emphasize the importance of reporting the appropriate masses and, preferably, also volumes of the constituents. Data reporting checklists for batteries^[55] and supercapacitors^[57] specify first including information about the constituents, assembly and setup, such as whether the configuration is a half or full cell and details for the counter, reference and working electrodes. A second section recommends reporting information about the measurement conditions and calculations, such as test temperature, number of cycles, rate and pressure on the cell during cycling, if applied.^[56] Some checklists include a third section to indicate which characterization techniques have been used, such as CV, electron microscopy and EIS. Authors are recommended to complete the appropriate checklist prior to submission of a manuscript and are encouraged to include the checklist in the supporting information for publication. If important information is not included, authors are requested to state if the information is not applicable for their study or provide acceptable reasons via additional comments.

6.2. Protocols for Constituents

6.2.1. General Considerations

The objectives for undertaking constituent tests are usually for two reasons: either the discovery, development and optimization of constituents, or to generate constituent data as inputs into predictive models. It is rare that constituents would be used to rank multifunctional performance, since this is only reasonable in the context of a cell. Therefore, the protocols for constituents could be drawn from those for conventional structural composites and conventional electrochemical materials.

6.2.2. Structural Electrodes

Electrochemical characterization of structural electrodes for both structural supercapacitors and structural batteries may be performed at the fiber, tow or lamina level, typically in a pouch or coin half-cell with a structural electrolyte infused into the electrode. The pouch cell configuration is recommended to permit subsequent or simultaneous mechanical testing. Any characterization using a liquid electrolyte would be to ascertain the maximum achievable electrochemical performance. In this instance, it is recommended to use an electrolyte that is consistent with the ionically conducting phase of the structural electrolyte in a SPC device. Doing so enables comparison with the performance of a corresponding SPC device made using a structural electrolyte where the operating voltage windows are the same. To mitigate for the inherent variability in structural electrode characterization tests that involve small volumes of material, it is suggested

that a minimum of five replicates are tested. The number of replicates, the mean and the standard deviation or coefficient of variation should be reported.

When reporting the gravimetric electrode performance, the performance should be normalized by the total mass of the electrode as well as relative to the electrochemically active material only. Doing so allows comparison between electrodes as well as between electrode conceptual designs. It should be made clear that the performance is for a half-cell and not a full cell, along with what constituents are included in the normalization; furthermore, it is meaningless to quote the energy and power of a single electrode.^[64]

For structural supercapacitor electrodes, it is recommended to characterize the capacity/capacitance, ESR and coulombic efficiency, preferably using GCD at several different current densities. Note that under galvanostatic measurement, the coulombic efficiency (ratio of time to discharge against time to charge) will tend to be high but the energy efficiency (ratio of energy during discharge against energy during charge) will be lower, so care should be taken not to confuse these two efficiencies.

Characterization using CV at several scan rates is also recommended to provide another measure of capacity/capacitance, as well as to identify contributions from Faradaic and non-Faradaic processes. The scan rates and voltage window used should be reported. For both GCD and CV, several cycles (typically five) should be performed and the number of cycles reported. The performance values should be calculated from the last cycle where the response shows negligible difference in response from that of the previous cycle. It is recommended to use EIS to obtain another measure of the ESR, preferably before and after GCD and CV cycling tests to identify changes in the electrode due to degradation. The frequency range and sinusoidal voltage amplitude should be reported and if the EIS data is used to fit an equivalent circuit, the circuit model parameters should be reported. For all the characterization methods, corresponding plots should be included. Nyquist plots should have equal scales for both axes and show the data points at measured frequencies as well as a curve through the points. For comparisons between supercapacitor electrodes, the specific capacity/capacitance and ESR are recommended as the preferred performance metrics rather than specific energy and power, since the former metrics are independent of the operating voltage window of any electrolyte used.

For structural battery electrodes, the active materials, e.g., carbon fibers as the anode or lithium iron phosphate (LiFePO₄) coated carbon fibers as the cathode, must be tested in a structural electrolyte.^[153,164] The structural electrode is tested in a half-cell set up versus a suitable counter electrode such as metallic lithium. The electrochemical stability and reversibility of Faradaic redox reactions of the electrodes, e.g. LiFePO₄ particles deposited on carbon fibers should be tested by CV, reflecting the inter-face performance of the structural electrode. Furthermore, the internal resistance across various frequencies in the frequency domain should be analyzed using EIS, while the cell performance should be characterized by GCD. Keeping track of the discharge capacity over a multitude of cycles is important to assess several aspects of a structural electrode's performance: the rate at which the electrode degrades, its operational efficiency, and its overall health and longevity. The discharge specific capacity of all the tested specimens should be recorded over at least five cycles at

each C-rate. The capacity will fade with increased C-rate; however, this process should be reversible, and it is to be shown that the initial capacity is restored when it cycles back at the initial slow charge rate. Following the capacity retention test, long-term cycling at a reasonable C-rate for up to 1000 cycles should be performed, and the coulombic efficiency reported.

For both structural supercapacitors and structural batteries, the approach to mechanical characterization of the electrodes will depend on the device architecture (i.e., tow, unidirectional tape, or woven). But in all instances, the choice as to whether the electrode is characterized in the as-received state (i.e., devoid of matrix), with a structural matrix or with a structural electrolyte is critical to the veracity of the results. It should be noted that extraction of the ionically conducting phase from the structural electrolyte prior to mechanical characterization is not appropriate since the washing process may damage the structural electrode or may modify the fiber/matrix interface.

For fiber testing, there is a well-established standard for characterizing the tensile modulus and strength of conventional fibers when devoid of matrix.^[102] Such tests can provide an insight into the intrinsic mechanical performance of the fibers, but they can be sensitive to handling and specimen preparation, leading to high variability. Moreover, for structural electrodes, testing under dry conditions could lead to active materials on the fibers being shed, thus influencing the measurements and leading to anomalies in the test results. Therefore testing of fibers or tows following infusion with a structural electrolyte provides the most representative measure of the electrode behavior, and would ensure realistic load transfer between the fibers.^[72] However, it is important to ensure the testing environment is controlled and compatible with the structural electrolyte: for most systems this would entail testing in an environment devoid of moisture. Any absorbed moisture may influence the mechanical response and in particular the fiber/matrix interface (and hence load transfer between the fibers). Therefore, depending on the sensitivity to moisture of the active materials and structural electrolyte, it may be necessary to test within a dry room or even a glovebox. Alternatively, mechanical testing could be undertaken within an encapsulating pouch bag: this may complicate the data capture, but there are strategies to overcome these.^[2] Because of the inherent scatter in fiber or tow level tests, and the small volume of material being characterized, it is suggested that a minimum of five replicates are tested to generate robust data. Finally, when characterizing the modulus it is important to use local strains, rather than crosshead displacement, or account for the test machine compliance in the data reduction. With this in mind, mechanical testing of fibers or tows within a DMA can be problematic because the test fixtures are very compliant, so such testing should only be used for ranking.

As with fibers or tows, it is recommended that UD tape or woven structural electrodes should be tested following infusion with the structural electrolyte, with the same caveats discussed in the preceding paragraph regarding moisture control. For these architectures, using a structural electrolyte will not only permit axial tension characterization, but also off-axis testing (such as in-plane shear or transverse tension). Although it is feasible to test unidirectional tapes as a single ply, depending on the weave style, woven lamina may need to be stacked into a two-ply laminate (mirrored at the midplane) to ensure it is balanced and sym-

metrical. Although the standards prescribe a minimum coupon thickness, testing of a multi-ply stack of structural electrodes to achieve this may not be representative of the mechanical behavior of a single electrode. Therefore, although dimensions may depend on the cell design, for UD tape and woven electrodes, it is suggested to use a test configuration inspired by ASTM D3039 to characterize fiber-dominated behavior.^[138] To characterize matrix and fiber/matrix dominated behavior, the test configurations inspired by the ASTM standards for transverse tension (UD tape) and $\pm 45^\circ$ in-plane shear (woven) materials should be considered.^[138,139] The transverse tension test can have very low failure loads, so an alternative method to measure matrix-dominated behavior in UD tape electrodes is the rail-shear test (in-plane shear) ASTM D4255, although using this test on structural electrodes is yet to be reported.^[165] For all these tests, at least five replicates that present valid failures should be characterized.

End-tabling is required for all these test methods, so caution should be followed to ensure that bonding the end-tabs does not leach out the ionic conducting phase from the coupon gauge section. For compression behavior of the structural electrodes, the thin-skin sandwich beam configuration described in ASTM D5467 is appropriate.^[141] However, this test method requires bonding of the structural electrode onto a core material, so again, care should be taken to ensure structural electrolyte is not leached from the composite.

As with tow level tests, it is important to account for test machine compliance for lamina tests. Furthermore, the requirement to ensure the structural electrolyte is not exposed to moisture can present challenges with instrumentation, such as attaching strain gauges or applying a speckle pattern for DIC. Finally, in reporting the performance, the standard methods to determine the fiber volume fraction are detailed in ASTM D3171, suggesting acid digestion or pyrolysis of the laminate to isolate the fibers from the matrix.^[146]

6.2.3. Structural Electrolytes

Bicontinuous (dual phase, solid/liquid) electrolytes have evolved as promising candidates for both structural battery and supercapacitor applications where structural integrity and electrochemical performance are simultaneously required. Several varieties within this concept have evolved over the last ten years and the properties have been reported in different ways. The properties that should be reported range from ionic conductivity to processability of the structural electrolyte, but they could differ between cell types, for example batteries versus supercapacitors.

It is essential to determine the intrinsic properties of the structural electrolyte, such as ionic conductivity; mechanical properties (modulus and strength, and elongation at break for structural battery electrolytes); and microstructure. We define a structural electrolyte as an electrolyte with Young's modulus exceeding 100 MPa and ionic conductivity of over 10^{-2} mS cm⁻¹. The former is to ensure an acceptable degree of mechanical load transfer between the reinforcement phases, while the latter is to ensure there is a reasonable level of electrochemical performance. The implication of these requirements is that only solid and bicontinuous (dual-phase) electrolytes can be considered as structural electrolytes, while gel and liquid electrolytes do not

meet the mechanical requirements stipulated. The following proposes that a series of standardized measurements under specific conditions should be reported for new systems as a baseline in characterization. It is suggested that, if possible, samples for mechanical performance are the same as those for ionic conductivity. Note that data should always be reported at room temperature, although it is reasonable that additional measurements can be made at other temperatures.

Regarding the electrochemical properties, ionic conductivity is perhaps the most important, and this should be determined using EIS. This should be undertaken at room temperature, and over a frequency of between 1 Hz to 100 kHz with a step voltage of 5 to 10 mV. The electrochemical performance of the structural electrolyte in a half-cell at room temperature using an electrode of choice should be undertaken to identify the stability of the structural electrolyte. It is essential to provide detailed information not only about the test conditions, i.e., step voltage, frequency range, etc., but also the sample shape and dimensions, the type of the electrode used and the number of replicates, as well as whether any additional liquid electrolyte was added prior to characterization.

For the mechanical properties, as described in Section 5.2, several test methods have been used to characterize the performance. But for the initial studies or screening, where only small quantities of structural electrolytes are available, DMA testing (in any mode at 1 Hz and room temperature) is appropriate. It should be noted that this should only be used as a screening or ranking tool, since the values obtained may not reflect the true mechanical performance of the structural electrolyte. To determine the mechanical properties, at least five replicates which present valid failure modes, should be presented and for all values the standard error should be given. The following paragraph suggests some standards which could be adopted, but any modification of the standard should be clearly stated, and the reasoning provided.

For uniaxial tension, to identify modulus, strength and elongation at break, ASTM D882 should be used.^[166] This is a well-established test method for polymeric materials and permits relatively small quantities of materials to be characterized. For compression, ASTM D695^[115] is appropriate, although for biphasic structural electrolytes there is a tendency for the coupon to “sweat” under mechanical load, leading to instrumentation issues. Finally, for characterizing the flexural modulus, three-point bending using ASTM D790 is recommended.^[142] Finally, it is recommended that the microstructure of the structural electrolyte is characterized with SEM, TEM or other techniques if applicable. Furthermore, their processability, such as flow properties, rheology and stability (processing window) should be characterized.

6.2.4. Structural Separators

A key constituent for structural power laminates is the separator. It is conceivable that if the consolidation can be controlled, and the structural electrolyte is stiff enough, cells could be assembled without a separator. However, from a pragmatic perspective, to achieve a reasonable reinforcement volume fraction, the separator is necessary to negate shorting of the electrodes. Guidelines for conventional cells recommend reporting physicochem-

ical characterization measurements such as electrolyte uptake, thermal analysis, shut-down tests, shrinkage, tensile strength, MacMullin number and electrochemical compatibility tests with electrodes.^[64] Unlike in a conventional cell, the separator in a structural power composite is likely to have been subject to significant through-thickness and possibly in-plane mechanical loads during the manufacturing process, leading to stresses and deformations that may be retained in the final state. In particular, the structural electrolyte infused through the reinforcing fibers or polymers in the separator would be critical in determining how the separator performs. Electrochemical testing on free-standing, isolated separators must accurately capture the electrochemical or mechanical performance of the separator in the structural power laminate. Hence, as with the structural electrodes, it is recommended that characterization of the structural separator should include the structural electrolyte and the processing method to be used in the final cell. When reporting the electrochemical performance of cells with the aim of comparing different separators, it is important to normalize by the mass of the full cell to capture the mass of the separator, as well as the mass of the structural electrolyte that would partly be determined by the pore volume in the separator. For mechanical testing on separators, it is not recommended to perform mechanical tests on the separator alone or on the separator with the matrix introduced in a way that is different to the final cell manufacturing procedure. It would be better to test the combination of the separator together with the structural electrolyte having been introduced and processed in the same way as that used for manufacturing the final device.

6.2.5. Current Collection and Bipolar Plates

The protocols discussed here refer principally to current collection but can also apply to bipolar plates. The principal design driver for the current collectors is their electrical resistivity, which is associated with the intrinsic conductivity of the current collector material and the contact resistance associated with the current collector/electrode interface.^[73] If the current collector is not isotropic, it may be necessary to characterize the longitudinal, transverse, and through-thickness conductivities of the material, the latter being particularly relevant for bipolar plates. The use of four-probe measurements to account for contact resistances at the electrical connection between the instrumentation clips and the current collector or bipolar plate tabs is recommended. It has been identified that the current collection can be very sensitive to the pressure applied, so this parameter should be controlled, measured, and reported when undertaking characterization.^[100]

6.2.6. Encapsulation

Structural encapsulation of structural power devices is an area that has not been widely considered but has an important role in realizing structural power products. Methods to check that the encapsulation material does not chemically interact with the electrolyte could include immersion of samples of encapsulation in the electrolyte and periodically measuring gravimetric uptake of the electrolyte over an extended period of time.^[100]

Any encapsulation material should prevent moisture ingress into the structural power composites. A simple method to investigate moisture (or organic vapor) uptake is dynamic vapor sorption, which also enables the determination of diffusion rates—and if equipped with a Paynen-cell, also the permeability of the vapor of interest—within the encapsulation material. Regarding mechanical performance, methods to characterize the mechanical bond strength between the encapsulation and device could include lap shear tests after the interface has been exposed to the electrolyte and typical operating environment for a suitable, measured length of time.^[100]

6.2.7. Interfacial Characterization

Interfaces, both at the fiber/matrix and structural electrode/separator scales, play an important role in defining the performance of structural power composites. Regarding the former (fiber/matrix scale), several test methods, such as the microdroplet debond test, single fiber pullout, pushout, and fiber fragmentation, have been developed to characterize fiber/matrix adhesion.^[147,168] These tests could be adopted to characterize structural fiber electrode/structural electrolyte interfaces. At the larger scale, interlaminar test methods, such as short-beam shear, could provide inspiration for characterizing the mechanical strength of structural electrode/separator interfaces.^[169] However, the low cell thickness could present challenges when adopting this test method. Similarly, standard test methods for characterizing delamination resistance have been widely used in structural composites, but would present substantial difficulties in characterizing such thin laminates with differing substrates. Therefore, new concepts are needed to develop mechanical test methods for characterizing electrode/separator interfaces. Finally, from an electrochemical perspective, the standard two and three-electrode cells, as described in Section 4, could be utilized to characterize the electrochemical properties of both fiber/matrix and separator/electrode interfaces, which, in conjunction with the mechanical interfacial properties, will provide insights into the multifunctionality of structural power composite interfaces.

When characterizing interfaces, such as that between the separator and the electrodes, the through-thickness pressure applied during consolidation to manufacture the sample should be controlled, measured, and reported. This is because the applied pressure during specimen preparation governs the final thicknesses, fiber volume fractions, mechanical interlocking between the plies, contact resistances, tortuosity of the structural electrolyte, and probability of short circuits. As with the other constituent tests, it may be necessary to undertake interfacial tests in conditions under which the ambient moisture is controlled or eliminated.

6.3. Protocols for Cells

6.3.1. General Considerations

There are several objectives for undertaking structural power cell tests. As with constituents, these objectives can be to rank and

optimize the cell configuration (different constituents, architectures, processing routes, etc.) and to provide data as inputs for or validation of predictive models. However, perhaps the overarching motivation is to quantify the multifunctional performance of a particular cell configuration. The determined ranking parameters can be compared against the background literature and also provide an input into multifunctional design calculations (Section 2.2), to provide a comparison against COTS assemblies. Therefore, to credibly quantify multifunctional performance of a structural power laminate, the coupon used to determine both electrochemical and mechanical functions should be the same: a “universal” coupon. As with any conventional coupon, this universal coupon should provide a measure of performance, which is representative of that of the material when within a larger component. As discussed in Section 7, such a universal coupon would also be used for characterizing electrochemical–mechanical coupling phenomena.

For both supercapacitors and batteries, conventional electrochemical tests (Section 4) often utilize small samples. However, mechanical characterization on such a scale will be invalid due to the dominance of edge effects, stress nonuniformities, and Saint-Venant’s principle. Therefore, this would suggest that a universal coupon would need to be inspired by a conventional mechanical test coupon. It is recommended that the standard methodologies used for electrochemical and mechanical testing should be followed as closely as practically possible. Although the universal coupon geometry will be predefined, standard guidelines for instrumentation, electrochemical testing, mechanical test machine set-up (including test machine compliance calibration), data reduction, and reporting should be adhered to if practicable. It is recommended that the number of test coupons should allow for at least five valid tests. However, it is recognized that such tests are much more challenging than conventional tests in terms of coupon manufacture and test procedure; hence, three coupons would be acceptable if such difficulties justify a reduced number. Finally, to characterize the impact of multifunctionality on the monofunctional performance, as conveyed in Figure 10, it is suggested that *Monofunctional Laminate* mechanical tests and *Monofunctional Device* electrochemical tests are undertaken and reported in parallel with the SPC characterization.

On the premise that a universal coupon should be used for characterizing both functions, quandaries associated with the confounding factors in the measurement of each function arise. An important overarching issue is the need to test in a moisture-controlled environment: for most cell chemistries, this means a moisture-free environment. If exposed to moisture during characterization, the structural electrolyte and any active materials may swell (inducing residual stresses) and interact with moisture (changing its electrochemical properties). From the perspective of electrochemical testing, this is well understood, and strategies have been established to address this issue. This includes encapsulation in a pouch prior to testing or undertaking electrochemical characterization in a dry room or glove box. However, from the perspective of mechanical characterization, a moisture-controlled environment presents difficulties if it is impossible to control humidity in the test room or to miniaturize the test so that they can be conducted in a glovebox. Strategies to partition the response of the encapsulation from that of the coupon will need to be developed. This could include mechanical testing of

the encapsulation in isolation, such that its elastic response could be accounted for in the data reduction. Encapsulation may also present difficulties for instrumentation, such as when attaching strain gauges or speckle patterns for digital image correlation (DIC). However, for membrane type tests (e.g., tension or in-plane shear) prior to damage development, the surface strains (on the encapsulation) will be representative of those within the coupon. Note this scenario would not be the case for bending or flexural tests. The alternative is to mechanically test in a moisture-controlled environment. Many mechanical tests are relatively quick (the order of a few minutes), so it may be sufficient to keep the coupon isolated from the ambient conditions until it is mounted in the test machine. Prior to mechanical testing, it is recommended that weight uptake studies are undertaken on traveler coupons, using the standard for moisture uptake measurement of polymer composites (ASTM D5229) as inspiration.^[170] This will help to establish the significance of any moisture uptake on the mechanical performance. For more stringent controls, it may be necessary to mechanically test in a dry room (typically a dew point of -30°C) or even within a glovebox (typically a dew point of -65°C).^[171] Unfortunately, mechanical test facilities with such moisture controls are rare, so it is suggested that efforts to minimize the effect of exposure of coupons to moisture during mechanical testing are pursued. Finally, it should be noted that coupons that have a biphasic structural electrolyte (i.e., containing a liquid electrolyte phase) could shed or leak corrosive materials during mechanical testing. Therefore, care should be taken to ensure that any instrumentation and test fixtures are not exposed to these materials. Overall, there is clearly a need to formalize the testing environment for structural power composites and establish how to balance the practical issues of mechanically testing moisture-sensitive materials against the need for reliable data. This challenging issue needs to be investigated and addressed in developing standard protocols.

It is necessary to attach current collectors, and the associated wiring, to the cell to facilitate electrochemical testing. If the cell is large, the resistive losses associated with the current collection could make measuring the intrinsic performance problematic. From the perspective of mechanical testing, current collectors may interfere with the strain measurements or obscure the coupon surface when undertaking noncontact measurements, such as DIC. Moreover, there is a risk that the current collector would interfere with the stress state within the coupon and hence, modify the mechanical response. Therefore, if it can be demonstrated that they can be removed (or not attached at all during fabrication) without influencing the intrinsic multifunctional performance, it is not necessary to use current collectors when undertaking purely mechanical testing.

A further issue that needs to be addressed is that of the applied pressure on the cell during characterization. Results to date have shown that cell performance can be very sensitive to this parameter, and reproducibility is improved if the applied pressure is controlled. Free-standing structural supercapacitors can present high variability in their electrochemical performance, but the performance tends to improve as the pressure is applied, until a threshold is reached, beyond which the device will short-circuit.^[3] For measuring electrochemical performance, it is recommended that the cells be characterized over a quantified range of applied pressures, using clamping plates or weights to apply the pressure, and

ensuring uniformity by using load-distribution pads, etc. Reporting of the applied pressure (in MPa) is preferred. As a minimum, whether or not pressure was applied should be reported; if applied, the method (e.g., weights, spring, screw, clips, etc.) should be conveyed. Unfortunately, applying pressure on the cell during mechanical loading will fundamentally violate the uniaxial stress state required. Therefore, it will only be practical to mechanically test a universal coupon in its free-standing state. There is certainly a need to understand and research the role of applied pressure on the intrinsic multifunctional performance of structural power composites, and the development of protocols will need to address this issue.

The appropriate electrochemical tests to use for characterizing structural supercapacitors and structural batteries are described in Sections 6.2.2 and 6.2.3, respectively. However, there are some generic issues associated with the electrochemical characterization that should be considered. It is recommended that electrochemical characterization of structural power composites be undertaken in pouch cells. Reporting of the data should be comprehensive: at the very least, conveying gravimetric electrochemical data normalized by half-cell (both active material and electrode mass), active cell, full cell, and SPC device masses, as defined in Figure 9. In addition, areal performance should also be reported. Energy and power densities should not be extrapolated but should be calculated using a voltage corresponding to the operating potential window of the structural electrolyte used during the characterization. If Ragone or multifunctional efficiency plots are presented, they should only compare the performance characterized using the same normalization method.^[64]

Although individual cells may be no more than 1 mm thick, the mechanical test standards usually prescribe a minimum coupon thickness of 2 mm or more. However, assembling a multiple-stack of cells to meet this requirement would be resource-intensive and would not present performance representative of a single cell. Therefore, it is reasonable to recommend deviating from the standard thicknesses for mechanical testing of cells as long as the failure mode can be shown to still be valid. Therefore, although dimensions may depend on the cell design, for devices using UD tape or woven structural electrodes, it is suggested to use a test configuration inspired by the standard ASTM D3039 to characterize fiber-dominated behavior.^[138] To characterize matrix and fiber/matrix dominated behavior, the test configurations inspired by ASTM transverse tension (UD tape), $\pm 45^{\circ}$ in-plane shear (woven), and rail-shear test methods should be considered.^[138,139,165] End-tapping is required for all these test methods, so caution should be followed to ensure the end-tab does not leach out the ionic conducting phase from the coupon gauge section. For compression behavior, the thin-skin sandwich beam configuration described in ASTM D5467 is appropriate.^[140] This test method requires bonding of the structural electrode onto a core material, so again, care should be taken to ensure structural electrolyte is not leached from the composite.

Given the relative immaturity of this technology, it is perhaps more appropriate to focus on the elastic performance (i.e., Young's modulus) than strength or toughness. In such emerging materials, parameters such as strength and toughness are dominated by processing issues and defects rather than providing a true measure of the intrinsic mechanical performance. Ultimately, there will be a need to characterize the delamination

resistance of structural power laminates.^[47] However, in the near term, if the issue of the low laminate thickness could be resolved, short-beam shear (ASTM D2344) could provide a means to rank the structural electrode/separator interfaces.^[169] Finally, in reporting the performance, the standard methods to determine the fiber volume fraction, detailed in ASTM D3171, should be adopted or modified depending on the particular constituents in the device.^[146]

6.3.2. Structural Supercapacitors

Electrochemical characterization of structural supercapacitance is dominated by power, rather than energy storage, measurement. This means current collection, and any associated resistive losses can have a profound effect on the apparent performance. Hence, test sample size often has a strong influence on the measurements. GCD over a range of different current densities is the preferred choice for structural supercapacitors^[172] (rather than CV or EIS) to evaluate performance under practical conditions.^[62] The performance values should be calculated from the last cycle where the response shows negligible difference in response from that of the previous cycle. The approach has often been to fit the data to a Randles equivalent circuit, characterizing the capacity/capacitance, ESR, and coulombic efficiency.^[59] When calculating capacitance from GCD datasets, the voltage range used to calculate the discharge slope should be reported, and the Ohmic voltage drop should be excluded.^[62] Care should be taken in interpreting the discharge slope, since this may present a nonlinear response due to pseudo-capacitive or Faradaic effects. Instead, in these cases, the energy stored should be calculated from the product of the current and the integrated voltage over time, then used to calculate an effective capacitance.^[9]

Characterization using CV at several scan rates will also provide another measure of capacity/capacitance, as well as identify contributions from non-EDLC processes. The scan rates and voltage window used should be reported. Finally, EIS provides a further measure of the ESR. The frequency range and sinusoidal voltage amplitude should be reported, and if the EIS data is used to fit an equivalent circuit, the model parameters should be reported. For all the characterization methods, corresponding plots should be included, and Nyquist plots should have equal scales for both axes and show the data points at measured frequencies as well as a curve through the points. Finally, the recommended equation for calculating the power is to use $P = V^2/4R$ (Appendix B, Equation B2) when possible, and to avoid using $P = E/\Delta t$, because the current applied for the latter calculation could exceed the maximum possible power output of the structural supercapacitor.^[160]

6.3.3. Structural Batteries

Electrochemical characterization of structural battery full cells concerns several types of tests, similar to those for the electrodes. These should be performed on the structural full cells, meaning that the stacked electrodes on each side of the electrically insulating separator must be in a structural electrolyte.^[27,164,173,174] These tests should be performed on prismatic cells in pouch bags and

not coin cells, thus permitting subsequent or combined mechanical testing.

As for the structural battery electrodes (Section 6.2) the electrochemical stability and reversibility of Faradaic redox reactions at the electrode interfaces is to be tested by CV. Furthermore, the internal resistance across various frequencies in the frequency domain should be analyzed using EIS which the cell performance should be characterized using GCD, recording voltage profiles during charge and discharge. Keeping track of the discharge capacity over a multitude of cycles is important to assess several aspects of structural battery performance. This characterization includes the rate at which the battery degrades, its operational efficiency, and the overall health and longevity of the structural battery. The discharge specific capacity of all the samples is to be recorded for at least five cycles at several C-rates. It is recommended to cycle first at a slow C-rate, say C/20, and increase C-rate in steps, e.g., C/10, C/2, 1C, 2C, and a final step at C/20. The capacity will fade with increased C-rate; however, this process should be reversible, and it is to be shown that the initial capacity is restored when it cycles back at the initial slow charge rate. Following the capacity retention test, long-term cycling at a reasonable C-rate, e.g., 1C, for up to 1000 cycles should be performed, and coulombic efficiency reported. Energy and power density reporting should be normalized by the active cell, i.e., electrodes, separator, and structural electrolyte, as well as by the active materials, e.g., carbon fiber and lithium iron phosphate (LFP). Reporting energy and power density with respect to the active material permits comparison against theoretical or conventional cell data, whereas normalization with respect to the active cell permits comparison between structural battery conceptual designs.

6.4. Protocols for Multicell Assemblies and Components

To date, there have been very few studies addressing multicell assemblies of SPCs in components.^[174–176] However, it is becoming increasingly important to demonstrate and assess multicell solutions for industrial applications. There are several reasons for undertaking multicell component tests. First, multicell components are required for the assessment of a multifunctional design. That is, the application in mind must be considered for an objective and relevant assessment of energy, structural, and multifunctional efficiency metrics. Thus, a multifunctional design protocol will permit direct and fair comparison with the COTS assembly, allowing stakeholders to readily assess potential benefits of employing structural power composites in their applications. Second, multicell demonstrators are needed for generating validation data for predictive models of components. Multicell assemblies also provide valuable insights on cell assembly and stacking strategies, encapsulation strategies, and battery management system requirements. Recent structural power multicell demonstrators are depicted in Figure 11. These demonstrators have been characterized electrochemically by performing tests on the individual cells and groups of cells connected either in series and/or in parallel. Their mechanical performance has not been characterized experimentally since the purpose of these demonstrators was typically to communicate the concept to stakeholders via prototypes, and structural encapsulation had not yet been resolved. However, laminate theory has been employed to predict the mechanical performance of multicell laminates.^[176]

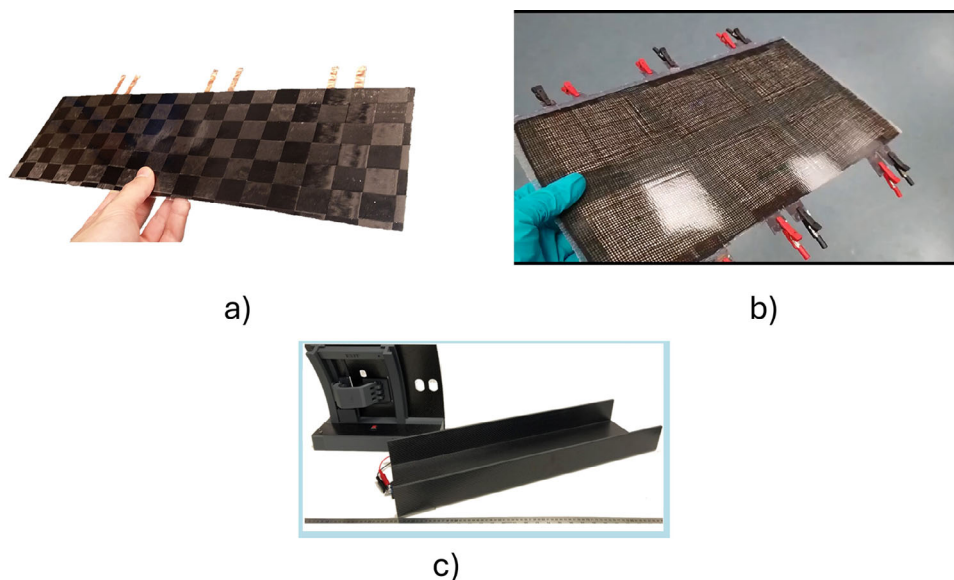


Figure 11. Structural power composite multicell demonstrators: a) a demonstrator laminate with three structural battery cells connected in series;^[176] b) six structural battery cells in a natural fiber composite multicell demonstrator laminate;^[174] and c) structural supercapacitor multicell door frame demonstrator integrated in an aircraft fuselage longeron.^[175]

The multifunctional performance assessment of multicell demonstrators is based on standard electrochemical tests used for conventional battery packs, such as constant current constant voltage (CCCV) charge/discharge of the structural battery pack and EIS. Also, conventional mechanical tests are used and chosen to reflect the loads acting in the intended applications. For example, for an aircraft fuselage frame, compression loads are of concern and should be employed for testing. For automotive applications, e.g. a boot lid, torsional loads must be considered. Mechanical testing of components with multiple cells will be highly dependent on the stacking arrangement or layout of the cells, which is an area still to be developed.

To enable a fair comparison with conventional solutions, such as a battery pack plus conventional structure, the performance metrics characterized from the multicell component tests should be normalized by including the same constituents that are used for normalization of the conventional systems. This normalization should be chosen to provide the most useful information to the end-user to evaluate the relative overall performance of the multifunctional and conventional systems. If resources permit, it would also be valuable to manufacture an equivalent monofunctional structural composite component that is mechanically tested in the same way as the multifunctional component. It is also valuable to characterize and report the electrochemical performance of cells before and after integration into a component, to improve our understanding of whether the performance of single cells characterized under well-controlled conditions changes once the cells are assembled and surrounded by structural material. This information would enable end-users to assess how well the performance data obtained from cell and coupon tests scales up to the component level. Since multicell component tests are highly resource-intensive, it is not anticipated that several replicate tests would be performed at this scale. What is important for the final application is to quantify the variations in electro-

chemical performance between the individual cells in a multicell assembly, such that if needed, appropriate power management systems can be developed to appropriately distribute the electrical demands on the cells.

Finally, there has been no work on engineering tests, such as open hole compression and compression after impact on multicell assemblies. Such tests will be necessary to advance this technology and are also necessary to mature these materials for final component applications and support certification. Perhaps a good strategy here will be to hybridize devices within conventional composite laminates and then employ such engineering tests to infer the device performance.

7. Coupled Electrochemical–Mechanical Characterization

7.1. General Considerations

In setting out the requirements for characterization and reporting of cells, the culminating aspiration is the characterization of both mechanical and electrochemical functions simultaneously. Such conditions would truly reflect how structural power composites would be used in service: providing electrical energy while carrying mechanical loads (**Figure 12**). This would also provide an insight into potential coupling between the electrochemical and mechanical functions. Therefore, developing a universal coupon, in which both functions could be characterized simultaneously and hence coupling phenomena can be assessed, will be central to future standardization efforts for this emerging technology. As conveyed in the following paragraphs, development of such a universal coupon presents several significant challenges that are yet to be resolved. Therefore, this paper sets out those challenges and outlines potential candidate test methods which could be modified to produce a universal multifunctional coupon.

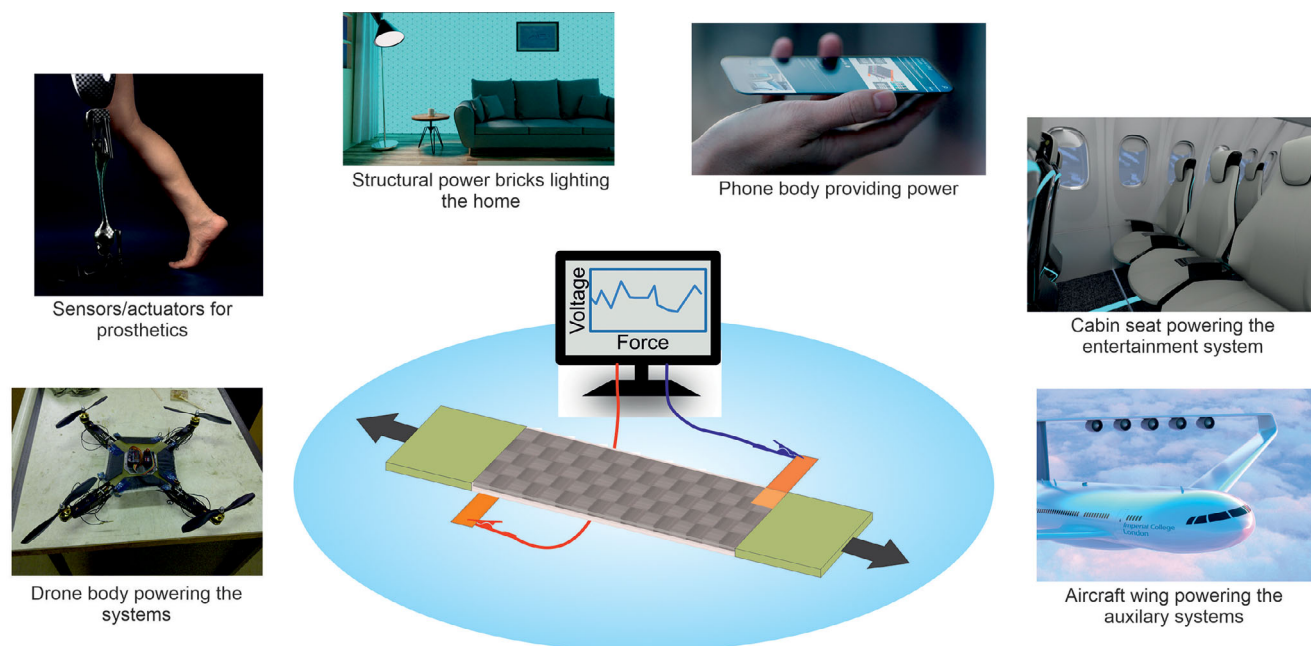


Figure 12. Motivation for universal coupon to characterize coupled electrochemical–mechanical performance.

As with any standard coupon, such a multifunctional coupon would need to replicate the conditions to which structural power composites would be exposed while within a component. This will become particularly relevant during “virtual certification” of components containing structural power composites. It is envisaged that during service, such multifunctional components will be undertaking both functions simultaneously, and could be modelling using coupling data from universal coupons as an input.

There is no evidence that structural supercapacitors present coupling between functions in the mechanical elastic range. However, as discussed in Section 5.3, the electro–chemo–mechanical processes exhibited by batteries are particularly important in structural batteries. Mechanical stress affects the chemical potential and hence the cell potential. Therefore, such tests to identify phenomena associated with the electrochemical–mechanical effects are needed: to generate validation data for predictive models of structural power devices in service; to rank coupling phenomena in different device configurations; and to evaluate the potential for using the SPC device also for sensing, actuation, or energy harvesting. For both structural supercapacitors and structural batteries, understanding of any coupled electro–chemo–mechanical processes will be vital for certification of multifunctional aircraft and automotive structures.

In the mechanical nonlinear range, such as during the formation and growth of damage, it is anticipated that there will be a change in the electrochemical performance for both structural supercapacitors and structural batteries. Such effects may be complicated: the formation of microcracks may expose and enhance direct access to the active materials by the ions in the electrolyte, enhancing energy and power densities, but may also elevate the electrical resistivity of the electrodes. Such mechanisms are starting to be understood in conventional electrochemical cells, and hence this knowledge could be exploited

in the development of suitable test methods for structural power composites.^[177] A corollary is that electrochemical cycling could influence mechanical performance, since it will result in swelling and relaxation of the structural electrolyte, and promote fiber/matrix debonding. In the longer term, damage tolerance is a key consideration. For instance, characterization of the effect of impact damage and/or penetration on the electrochemical performance of structural power composites needs to be addressed. Characterization of such effects will be vital for future certification of products using structural power composites.

An important issue particular to characterization of electrochemical–mechanical coupling is the test duration, since this will be dictated by the longest timescale of the two functions: this will tend to be the electrochemical measurement. Since it will not be possible to undertake electrochemical measurements instantaneously, it is anticipated that the mechanical test will entail stepwise loading, such that electrochemical measurements are undertaken while the applied loading is constant (dwell). Such a strategy would only be practical in the elastic testing regime, but may still result in a slight load drop-off and relaxation during the dwell. Fortunately, from an end-user perspective, characterization of coupling phenomena within the mechanical elastic regime will be of most interest. Beyond the elastic regime (i.e., upon the development of damage), it may be necessary to undertake interrupted tests (i.e., unload the coupon) to take electrochemical measurements. A potential risk is that upon removing the load, any damage may close or be modified, thus influencing the electrochemical response of the coupon. There is potentially an opportunity to learn from developments with structural health monitoring, in which electrical measurements are taken while a component is under load. An aspiration for a future protocol to characterize coupling phenomena would be to define standard mechanical loading spectra and dwell times to run in conjunction with the

electrochemical characterization. Establishing such standards would permit comparison between cells being reported across the literature. As with the single-function tests discussed in Section 6, it is recommended that five replicates be tested, if possible, although fewer replicate tests (at least three) could be justified.

Extending the points raised in Section 6.3, a key issue with simultaneous mechanical and electrochemical characterization will be the need to test in a moisture-controlled environment. However, coupling tests will have longer durations than tests characterizing a single function, further complicating the moisture control. The two strategies would be to either undertake coupled tests within an encapsulating pouch or to test within a moisture-controlled environment. For the former, as well as partitioning the pouch response from that of the coupon, further complications will arise from the need to have current collectors to simultaneously register the electrochemical response. Therefore, it is recommended that a membrane-type test (e.g., tension or in-plane shear) be pursued to ensure that any surface strain measurements are representative of those within the coupon (at least in the elastic range). The alternative would be to test in a moisture-controlled environment, but given the longer test duration, exposing the cell to ambient conditions during the actual test would result in unacceptable exposure. Hence, it will be necessary to undertake coupled tests in environments such as a dry room or a glovebox. Such mechanical test facilities are rare, which presents a challenge for the development of coupling tests for structural power composites.

A key difference between purely mechanical testing and electrochemical–mechanical coupled tests is that the latter will need current collectors. Where possible, the coupons should have current collectors and encapsulation attached in the same configuration as would be used if the material were to be characterized to determine just the electrochemical performance (Section 6). It is recognized that the current collectors and encapsulation could hinder the monitoring of the mechanical test parameters, such as the strains; therefore, tests may need to be performed with modified current collector and encapsulation configurations, and such changes should be reported.

Applied pressure presents an issue when undertaking coupling tests, since electrochemical performance is sensitive to this parameter, but mechanical testing can only be realistically undertaken on cells in their free-standing state. Moreover, any applied pressure will influence the electrical conductivity of the electrodes through mechanisms such as increased fiber-to-fiber contact. The development of a universal coupon to characterize coupling behavior will require stable electrochemical characterization of the free-standing, but mechanically loaded, cell. There is clearly a strong need to address the issue of how to consider applied pressure in the development of standard protocols for structural power composites.

For coupling tests that investigate fiber dominated behavior, axial tension (ASTM D3039) could be a good starting point for a universal coupon.^[138] To investigate matrix or fiber/matrix dominated coupling behavior, for devices with unidirectional electrodes, transverse tension (ASTM D3039) or the rail-shear test (in-plane shear) ASTM D4255.^[138,165] could be a good starting point. For cells with woven electrodes, the $\pm 45^\circ$ in-plane shear (ASTM D3518) test method could provide inspiration for a uni-

versal coupon.^[139] Points particular to structural supercapacitors and batteries are conveyed in the following Sections.

7.2. Structural Supercapacitors

Since structural supercapacitors store energy via the electrochemical double layer on high surface area active materials and not via intercalation into carbon fibers, they do not exhibit significant electrochemically induced strains upon charge and discharge, as observed for structural batteries. It is more likely that any coupling between the electrochemical and mechanical functions would occur at the interfaces between the reinforcements and the structural electrolyte. Therefore, tests that characterize the properties of the interfaces, such as in-plane shear, interlaminar shear, and toughness tests, are expected to generate the most insightful information. The time required for loading (and unloading) mechanical test coupons is of the order of several minutes, although in-plane shear tests can take longer due to the larger strains measured. This timescale is akin to that for charging and discharging conventional supercapacitors, but structural supercapacitors may have lower ionic conductivities and therefore require longer times to charge and discharge. Investigating the long-term electrochemical cycling effects on the mechanical performance may be worth investigating. The high currents delivered by supercapacitors could lead to localized joule heating, which may influence the mechanical behavior.

7.3. Structural Batteries

The effects of electrochemical cycling on the mechanical performance and vice versa may be substantial and must not be overlooked. The most important thing is that the test setup allows for applying mechanical loads at the same time as electrochemical cycling, or other electrical measurements can be performed. Thus, electrodes must be connected via a potentiostat or similar while mechanical loads are applied by a testing machine.

As discussed above, there is a concern that, e.g., lithium insertion in carbon fibers will reduce their tensile strength, but the reduction depends on state-of-charge. These effects can best be studied on the constituent level using the tensile test of charged carbon fibers. Tests on carbon fiber tows in liquid electrolyte versus metallic lithium found a moderate, permanent reduction in fiber strength from electrochemical cycling.^[178] The specific capacity of the carbon fibers was not affected by tensile loading during electrochemical cycling. Later tests showed a 12% reduction in longitudinal modulus and a 207% increase in transverse modulus from lithium insertion in the same fiber.^[151] The former, however, is due to the radial expansion of the fiber, implying that the stiffness of the fiber remains unchanged. Such large volume expansions may lead to cracking and delamination after long-term cycling. Thus, testing mechanical properties after long-term cycling (100–1000 cycles) is important to perform and report. This could be done by either tensile tests or bending tests. However, it is important to test both in the fiber direction and in the transverse direction, as the latter is most probably more affected by cracking and delaminations caused by electrochemical cycling. Although bending tests provide a less accurate

strain measurement, due to the strain variation over thickness, it has the advantage of also making it possible to test the electrodes under compressive loads. This is relevant since the compressive strength is usually more affected by damage in the structural electrolyte than the tensile strength. It will also be important to include long-term cycling in numerical models used to design structural battery composites.

Piezo-electrochemical effects can be measured on the constituent level in half-cells.^[154,155,179] It can be done, such as tests performed on an actuator cell using two identical carbon fiber electrodes.^[154] Tests have also been performed on full cells, demonstrating that applied tensile load inflicts an immediate, linear drop in cell potential in the mV range with applied strain.^[180] This can be measured and quantified on either half-cells or preferably full cells using either tensile tests^[155] or bending tests.^[154] The cell voltage change is, however, small and does not influence the total capacity to a large extent. As above, tensile tests only provide the coupling effect in tension, whereas bending tests must be used to quantify the response in bending.

It is further noted that the applied clamping force strongly affects the open cell potential. Effects of mechanical loading on the electrochemical capacity of full cells have been performed on an “uncoupled” structural battery cell.^[24] They demonstrated significant effects of high tensile loads on discharge capacity and energy density of the cell—where the discharge capacity at a tensile load close to the failure load of the laminate (200 MPa) was only approximately a third of that of the unloaded cell. Tests have also studied the effect of intermittent tensile loading on the discharge capacity and energy density of a “coupled” structural battery cell.^[176] This structural battery cell maintained its multifunctional properties also after being exposed to realistic load levels (0.4% tensile strain along the fiber direction). At higher strain levels, a significant drop in discharge capacity was observed. These examples illustrate the need for coupled electro-chemo-mechanical tests on structural battery composite cells. Any coupled electrochemical-mechanical tests must take this capacity drop into account and report the effects. Again, these effects are equally important to include in numerical models as they affect the multifunctional response of the device. Again, testing could be done either using tensile or bending tests, and preferably both in the fiber direction and the transverse direction.

Finally, large electrochemical strains and the transverse modulus increase for the carbon fibers may cause damage to the structural electrolyte.^[152] It is therefore important to characterize the matrix-dominated properties of structural battery composites and look for any indication of damage formation and its effect on the material’s multifunctionality. Recent studies have reported a decrease in transverse modulus for an all-fiber structural battery after electrochemical cycling.^[27] This observation may suggest damage has occurred, reducing the load transfer capability of the matrix. Similarly, recent studies have measured an increase in ionic conductivity for delithiated negative electrodes compared to pristine.^[181] This result suggests damage occurred to the polymer phase of the bicontinuous structural electrolyte, which again reinforces the need to test mechanical performance after long-term electrochemical cycling. From this discussion, it is evident that we need to formulate the damage tolerance of multifunctional materials based on their ability to perform their multiple tasks in the presence of damage. Since these are effects mainly affecting

the structural electrolyte, it is important to test the half- or full cells in the direction transverse to the fibers.

8. Summary of Best Practice

This paper has considered the most critical issue for advancing structural power composites: characterization and reporting protocols. With the rapidly growing body of publications in this emerging field, and the consequent interest from potential end-users and industry, there is an urgent need for this emerging research community to be “speaking the same language.” Since this technology melds structural composites and electrochemical energy storage, the profound differences in the conventions, nomenclature, and standards between these disparate fields are undermining the advancement of this exciting technology. Here we have set out the challenges for characterizing structural power composites and illustrated how these challenges have resulted in issues arising in the literature. We have then recommended important requirements for future protocols and set out what we consider to be best practice for characterizing these materials and reporting their multifunctional performance. These recommendations for best practice are summarized in **Table 4**.

9. Concluding Remarks

To conclude, we suggest partitioning the characterization and reporting into three levels: *Constituents*, *Cells* and *Components*. Many of the challenges associated with characterization and reporting manifest at the *Cell* level. The most important characterization challenge is the difference in scale between the coupons used for mechanical and electrochemical characterization. The overarching reporting challenge has been normalization of the measurements. To address these challenges, our suggested best practice and proposed protocols are briefly detailed in **Table 4**. Our aspiration is that the guidelines in this paper will lay the groundwork for formal standard protocols to be developed and agreed. Such protocols will permit the generation of reliable data to support predictive models and multifunctional design tools. They will provide the means to rank different constituents, architectures and cells, such that structural power composite development can be advanced and useful levels of multifunctional performance realized and accurately quantified. Establishing robust characterization and clearer reporting will permit researchers and industry to take an informed view of the literature and provide a better grounding for adoption of this technology, underpinning future industrialization of these exciting materials.

Appendix A Illustrative Examples

This section provides a list of parameters that it would be valuable to measure and report throughout the whole characterization process. Data from the literature on structural supercapacitors and structural batteries has been included solely to illustrate how such data may be reported and not to present new data. The parameters shown could also be characterized and reported not only for structural supercapacitors/batteries, but also for their monofunctional equivalents, i.e., structural baseline laminates, monofunctional supercapacitors comprising purely liquid electrolytes instead of structural electrolytes, etc.

Table 4. Summary of best practice and proposed protocols at *Constituent*, *Cell*, and *Component* levels.

Electrochemical	Mechanical
Structural electrodes	
<ul style="list-style-type: none"> The pouch cell configuration is recommended to permit subsequent/simultaneous mechanical tests. A minimum of five replicates is suggested. Report the number of replicates, mean, and standard deviation. Normalize by both active material only and full electrode, including structural scaffold, binder, etc. Report specific capacity/capacitance, cycling performance, and coulombic efficiency, preferably via GCD at various currents or C-rates. Report ESR from EIS. 	<ul style="list-style-type: none"> Test structural electrodes infused with structural electrolyte with appropriate moisture control. For fiber-dominated behavior, adopt or modify ASTM D3039.^[138] For matrix and fiber/matrix dominated behavior, adopt or modify ASTM D3039 for transverse tension on UD tape or ASTM D3518 $\pm 45^\circ$ in-plane shear for woven materials.^[138,139] For compression, consider ASTM D5467.^[141]
Structural electrolytes and structural separators	
<ul style="list-style-type: none"> Determine ionic conductivity using EIS at room temperature, from 1 Hz to 100 kHz using 5 to 10 mV. Report sample shape, dimensions, and the number of replicates. Report whether any additional liquid electrolyte is added prior to characterization. Report separator characterization such as electrolyte uptake, shut-down tests, MacMullin number, and electrochemical compatibility tests with electrodes.^[64] 	<ul style="list-style-type: none"> If possible, mechanically test the same samples as those used for electrochemical tests. For uniaxial tension, use ASTM D882.^[166] For compression, use ASTM D695.^[115] For flexural modulus, use three-point bending as defined in ASTM D790.^[142] For separators, include the structural electrolyte via the processing method used for the cell. Consider tests such as thermal analysis, shrinkage and tensile strength.^[64]
Current collection, bipolar plates, and encapsulation	
<ul style="list-style-type: none"> Measure conductivities using four-probe measurements to account for contact resistances at external connections. To check for chemical interaction with the electrolyte, immerse encapsulation samples in the electrolyte for a suitable, measured duration and periodically measure gravimetric uptake of the electrolyte.^[100] 	<ul style="list-style-type: none"> Control, measure and report pressure applied to the current collection materials during characterization.^[167] The interfacial strength between the encapsulation and device may be characterized using lap shear tests after the interface has been exposed to the electrolyte and typical operating environments.^[100]
Cells	
<i>Multifunctionality should be demonstrated by performing both electrochemical and mechanical tests on a "universal" coupon.</i>	
<ul style="list-style-type: none"> Electrochemical characterization of structural power composites should be undertaken in pouch cells. If possible, perform five valid tests. However, three coupons would be acceptable if difficulties justify. Report whether pressure was applied during electrochemical testing. If applied, report the method used to apply the pressure and the value. Record discharge specific capacity/capacitance over at least five cycles at each C-rate or current. Energy and power densities should be calculated using a voltage corresponding to the operating potential window of the structural electrolyte used during the characterization. If Ragone or multifunctional efficiency plots are presented, they should only compare the performance characterized using the same normalization method.^[64] Use an appropriate data reporting checklist.^[54–57] If important information is not included, state if the information is not applicable or provide acceptable reasons. 	<ul style="list-style-type: none"> Mechanical strength is very sensitive to scale and processing, and therefore is not an important parameter for ranking. Reporting should be focused on declaring the Youngs modulus. For matrix and fiber/matrix dominated behavior of UD tape, adopt or modify ASTM D3039; for woven materials, adopt or modify ASTM D3518 or ASTM D4255.^[138,139,165] For compression, adopt or modify ASTM D5467.^[140] For fiber volume fraction, adopt/modify ASTM D3171.^[146] Prior to mechanical testing, consider weight uptake studies on travelers, in ASTM D5229.^[170] The cell will need to be stacked such as to be mirrored at the midplane to ensure the laminate is balanced and symmetrical. If possible, perform five valid tests. However, three tests are acceptable if difficulties justify. When characterizing the modulus use local strains, rather than crosshead displacement, or account for the test machine compliance in the data reduction. It may be necessary to test within a sealed pouch, a dry room or even within a glovebox.
Components	
<ul style="list-style-type: none"> To fairly compare with conventional systems, normalize by including the same constituents used for normalization of the conventional systems. It is recommended to characterize and report the electrochemical performance of cells before and after integration into a component. Quantify variations in electrochemical performance between individual cells in a multicell assembly. 	<ul style="list-style-type: none"> Since multicell component tests are resource-intensive, a single test may be sufficient. Choose mechanical tests to reflect the loads expected in the intended applications. If resources permit, it is recommended to manufacture and test an equivalent monofunctional structural composite component that is mechanically tested in the same way as the multifunctional component.

A.1. Structural Supercapacitors

The parameters for structural supercapacitors include those recommended in the supercapacitor reporting checklist^[57] plus further parameters associated with mechanical performance. Providing information about the areal weights and densities of each of the constituents is helpful to allow calculation of the masses of devices of different dimensions from those tested, and therefore to enable estimates to be made of the effect of scaling on the performance. Providing information about the test conditions, such as temperature and applied pressure, is important since the performance characteristics can be very sensitive to these conditions.

Structural supercapacitor materials data

Parameter	Material/notes	L [cm]	W [cm]	T [μ m]	Areal wt [gsm]
Electrode	Spread tow CF-CAG	28	11	110	62
Separator	Polyester + ceramic	30	13	23	33
Current collector	Würth Elektronik Al tape	28	1	70	142
Encapsulation	PET-Al-PE laminate	32	15	75	150
Full cell	Including all layers	32	15	533	

Parameter	Value	Units	Material/notes
Mass of electrodes	3.81	g	For both electrodes
Separator mass	1.29	g	Including excess at edges
Current collector mass	3.97	g	Including protruding tabs
Epoxy mass	0.77	g	Includes BADGE + IPDA hardener
Ionic liquid mass	2.64	g	EMIM TFSI filling pore volume
Full cell mass	12.48	g	Electrodes, separator, epoxy, IL, CC
Active material mass loading	160	mg g ⁻¹	Carbon aerogel, capacitive
Active material areal loading	3.73	mg cm ⁻²	Carbon aerogel, capacitive
Electrode-specific pore volume	0.35	mL g ⁻¹	Spread tow CF-CAG
Separator porosity	56	%	Polyester + ceramic
Electrode pore volume	1.34	mL	Within spread tow CF-CAG plies
Separator pore volume	0.40	mL	Within PET + ceramic separator
Total pore volume	1.74	mL	In all plies
Excess electrolyte	0.17	mL	10% excess to ensure wetting
Ionic liquid density	1.52	g mL ⁻¹	EMIM TFSI
Epoxy density	1.18	g mL ⁻¹	BADGE + IPDA hardener
Multifunctional matrix density	1.32	g mL ⁻¹	40 vol % IL in multifunctional matrix
Multifunctional matrix film thickness	56	μ m	To fill all pores

Electrochemical performance

Parameter	Value	Units	Notes
Operating potential range	2	V	Determined by electrolyte
Pressure applied during test	1.3	kPa	Using weights, Al plates and Pacopad
Testing temperature	21	°C	Ambient temperature in laboratory
Supercapacitor type	Symmetric		Double-layer
Experimental setup	Full	cell	Pouch cell
Number of cycles tested	100	cycles	GCD at 1 mA cm ⁻² , 0–2 V
Full cell specific capacitance	0.35	F g ⁻¹	GCD, 1 mA cm ⁻² , 0–2 V, fifth cycle, integration
Full cell areal capacitance	14.1	mF cm ⁻²	By area of electrodes
Equivalent series resistance	125	Ω	From EIS, 1 mHz–20 kHz, 10 mV amplitude

Mechanical performance^[117]

Does the coupon include current collection?	No
Does the coupon include encapsulation?	No
Tensile specimen length \times width \times thickness (all in mm)	200 \times 20 \times 0.67 ^{a)}
In-plane shear specimen length \times width \times thickness (all in mm)	150 \times 25 \times 0.67 ^{a)}

Strain measurement method

Parameter	Value	St Dev	Units	# of specimens tested	Standard used
Tensile modulus	32.9	0.9	GPa	5	ASTM D3039
Tensile strength	110	3.3	MPa		
In-plane shear modulus	1.7	0.1	GPa	5	ASTM D3518
In-plane shear strength	13.7	0.3	MPa		
Carbon fiber vol fraction	40.7		%	5	ASTM D3171
Separator vol fraction	4.8		%		

^{a)} The specimen thickness was that of a single cell.

A.2. Structural Batteries

This section provides an example of data reported in a recent publication on a structural battery concept.^[27] The data is from various different battery cells with different mass loadings of cathode material (LFP), leading to a range of reported data points not only because of the different configurations but also due to scatter from manufacturing. Electrochemical testing was conducted using GCD using different currents, leading to different charge/discharge times (C-rates). Mechanical tests were performed on the same samples that were used for electrochemical testing, implying that standards for composite mechanical testing were not applicable. Tests were performed on both uncycled

battery cells and on cells that had been cycled, and the testing method is clearly described.

Structural battery materials data			
Parameter	Value	Units	Material/notes
Cell length	22–25	mm	
Cell width	17	mm	
Negative electrode thickness	≈50	μm	Spread tow CF 12K
Positive electrode thickness	≈90	μm	Spread tow CF 24 K + LFP coating
Separator thickness	80 + 23	μm	One-layer glass veil 6 gsm + PET-ceramic
Total thickness	230–250	μm	
Mass of active materials	0.027	g	CF anode and LFP in cathode
Total active cell mass	0.154	g	Active materials + CF cathode + separator + SBE

Electrochemical performance			
Parameter	Value	Units	Notes
Operating potential range	2–3.6	V	
Pressure applied during tests	0	kPa	
Testing temperature	25	°C	Controlled temperature laboratory
Cell-specific energy at ≈ C/50 ^{a)}	25–37	Wh kg ^{−1}	From GCD
Cell-specific energy at ≈ C/20 ^{a)}	23–33	Wh kg ^{−1}	From GCD
Cell-specific power at ≈ C/14 ^{a)}	21–30	W kg ^{−1}	From GCD
Cell-specific power at ≈ C/7 ^{a)}	18–20	W kg ^{−1}	From GCD
Cell-specific power at ≈ C/0.7 ^{a)}	7–13	W kg ^{−1}	From GCD
Cell-specific power at ≈ C/0.7 ^{a)}	8–10	W kg ^{−1}	From GCD
Electrolyte resistance	20	Ω	From EIS
Charge transfer resistance	80	Ω	From EIS

^{a)} C/X relates to C-rate where X is the charge/discharge time in hours.

Mechanical performance			
Parameter	Value	Units	Standard used
<i>Before cycling</i>			
Tensile modulus fiber direction	38	GPa	n.a.
Tensile modulus transverse direction	1	GPa	n.a.
Tensile strength fiber direction	234	MPa	n.a.
Tensile strength transverse direction	10	MPa	n.a.
<i>After 150 electrochemical cycles</i>			
Tensile modulus fiber direction	34	GPa	n.a.
Tensile modulus transverse direction	0.38 – 0.57	GPa	n.a.
Tensile strength fiber direction	252 – 261	MPa	n.a.
Tensile strength transverse direction	9	MPa	n.a.

Appendix B Nomenclature and Formulae

B.1 Electrochemical Characterization

Equations to calculate the maximum energy and power are^[160]

$$E = \frac{1}{2} CV^2 \quad (B1)$$

$$P = \frac{V^2}{4R} \quad (B2)$$

where E is the maximum energy, C is the capacitance, V is the applied voltage, P is the maximum power, and R is the equivalent series resistance. Equation B1 is only valid for capacitive (non-Faradaic) and not battery-type (Faradaic) charge storage materials.^[9]

For capacitive materials, a cell's capacitance, C , can be determined from GCD using^[172]

$$C = I / (dV/dt) \quad (B3)$$

where I is the discharge current and dV/dt is calculated from the slope of the discharge curve using two data points from the discharge curve with

$$dV/dt = \left(V_{\max} - \frac{1}{2} V_{\max} \right) / (t_2 - t_1) \quad (B4)$$

where V_{\max} to $\frac{1}{2} V_{\max}$ is the operating range of most supercapacitors, and t_2 and t_1 are the corresponding times.

To calculate capacitance from cyclic voltammetry (CV), Equation (B3) can be used where I is the average current during discharge (from V_{\max} to 0 V) and dV/dt is the scan rate.^[172]

Extrapolating the curve on a Nyquist plot, Z'' versus Z' , where Z is the complex impedance, to intersect the x -axis yields values that correspond reasonably well to ESR values calculated from GCD.^[172]

B.2 Passive Balancing

The balancing current, $i_{bal,i}$ for cell i , when the switch S_i is closed and the resistor R_i is active, is

$$i_{bal,i} = \frac{V_i - V_{ref}}{R_i} \quad (B5)$$

where V_i is the voltage of the i -th cell, V_{ref} is the target balanced voltage, and R_i is the resistor connected to the cell.

The power dissipated by the resistor during balancing is

$$P_i = \frac{(V_i - V_{ref})^2}{R_i} \quad (B6)$$

The total power dissipation for the entire system is the sum of individual power dissipations,

$$P_{total} = \sum_{i=1}^n \frac{(V_i - V_{ref})^2}{R_i} \quad (B7)$$

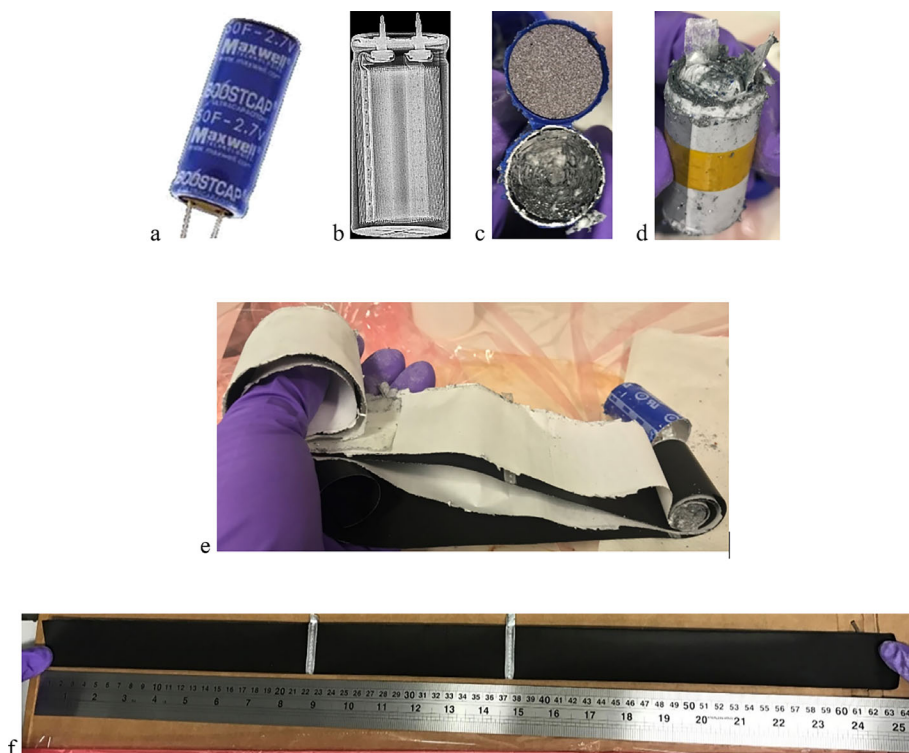


Figure C1. Maxwell 150 F supercapacitor a) before characterization, b) X-ray showing internal metallic constituents, c) end cover removed d) internal assembly removed from outer casing, e) internal assembly unraveled, f) electrode fully unrolled.

B.3 Active Balancing

For active balancing, the balancing current between a higher-voltage cell i and a lower-voltage cell j , controlled by the DC-DC converter, is

$$i_{\text{bal},ij} = \frac{V_i - V_j}{Z_{\text{DC-DC}}}$$

where V_i and V_j are the voltages of the cells, and $Z_{\text{DC-DC}}$ is the impedance of the DC-DC converter.

The power transferred between two cells is

$$P_{\text{transfer},ij} = \frac{(V_i - V_j)^2}{Z_{\text{DC-DC}}} \quad (\text{B9})$$

Considering the efficiency η of the DC-DC converter, the total power transferred for the entire system is

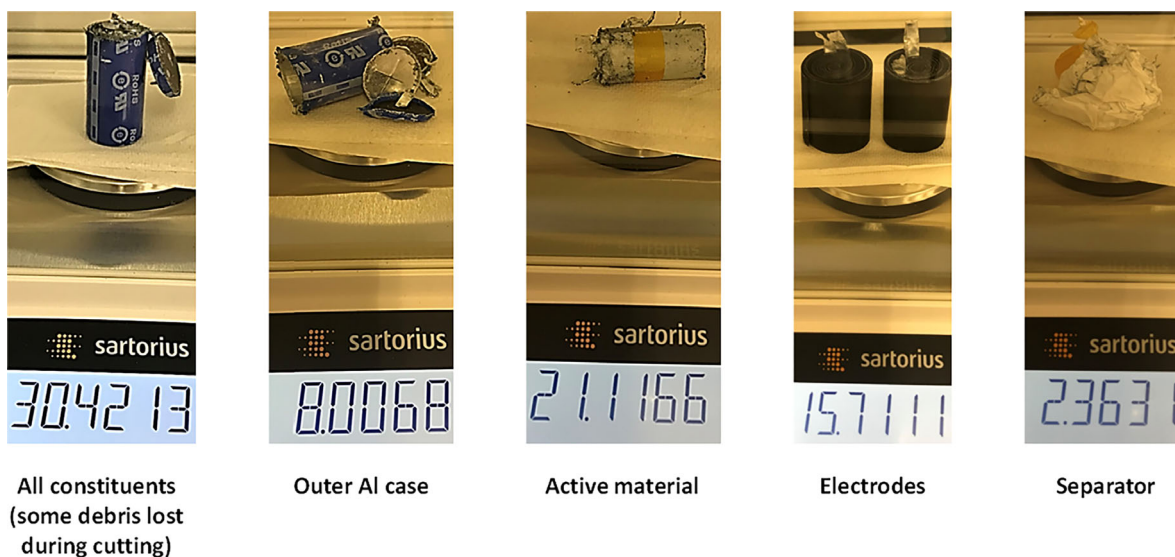


Figure C2. Masses (g) of all constituents in a conventional Maxwell 150 F supercapacitor.

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$$P_{\text{total}} = \eta \sum_{i,j} \frac{(v_i - v_j)}{Z_{DC-DC}} \quad (\text{B10})$$

B.4 Standard Composite Test Methods

<https://www.astm.org/products-services/standards-and-publications/standards/composite-standards.html#section2>

Appendix C Physical Characterization of a Supercapacitor

To characterize the relative weights and volumes of the constituents in typical conventional electrochemical devices, a cylindrical Maxwell 150 F supercapacitor (Figure C1a) was disassembled after first capturing X-ray images to show the internal metallic constituents (Figure C1b). The end furthest from the tabs was cut off (Figure C1c) such that the wound assembly (Figure C1d) could be removed while minimizing any material loss. The constituents in the unrolled assembly (Figure C1e) were separated to measure their individual masses (Figure C2) and laid flat to measure their dimensions (Figure C1f).

The two electrodes, together with the current collectors, represented approximately half the total mass and the casing was approximately a quarter of the total mass. The following dimensions were measured: electrode length = 63 cm, electrode width = 3.9 cm, electrode area = 246 cm², electrode thickness = 233 μm, separator thickness = 43 μm.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

electrochemical, mechanical, multifunctional, polymer composites, reporting, testing protocols

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Emile S. Greenhalgh is the Royal Academy of Engineering Chair in Emerging Technologies, Professor of Composite Materials in Aeronautics at Imperial College, and co-head of The Composite Centre at Imperial College. He has over 37 years of experience in polymer composites research and teaching. He began his career working on damage and failure in polymer composites in the Composites Structures Group at Farnborough and has written the seminal text on polymer composite fractography. He moved into academia in 2003 and has pioneered the development of structural power composites, now leading the Group at Imperial College London.



Sang Nguyen is a researcher in Aeronautics at Imperial College London. He has broad experience in engineering design, analysis, manufacturing, and mechanical characterization of composites through modelling and experimental research in multifunctional materials and consultancy projects with aerospace and automotive industry partners. His current research focuses on multifunctional design, development, and adoption of structural power composites through conceptual design, feasibility studies, system integration, scale-up and technology demonstration.



Leif E. Asp holds the chair as professor in lightweight composite materials and structures at the Division of Material and Computational Mechanics at the Department of Industrial and Materials Science at Chalmers University of Technology in Gothenburg, Sweden. Dr Asp's research group performs research on multifunctional composites, with a focus on structural battery composites, a material that can simultaneously store electrical energy and carry mechanical loads. The work comprises material development, modelling, and characterization, where the coupled electro-chemo-mechanical processes in structural battery cells are studied.



Alfredo Bici is a Ph.D. student under the supervision of Prof. Dan Zenkert at the Department of Engineering Mechanics at KTH Royal Institute of Technology in Stockholm, Sweden. He received his Bachelor's degree in Aerospace Engineering from Politecnico di Torino in 2021 and his Master's degree in the same field from KTH Royal Institute of Technology in 2023. His research interest focuses on the design and manufacture of multifunctional carbon fiber composites.



Alexander Bismarck is Professor of Materials Chemistry, Head of the Institute of Materials Chemistry of the Faculty of Chemistry, University of Vienna, Austria, and has been a WISE visiting Professor at Luleå Technical University since 2023. He has been working on multifunctional carbon fiber composites since 2004, focusing on structural supercapacitors and composite batteries.



Derrick Fam is the Deputy Director of the Singapore Battery Consortium and a senior scientist in the Institute of Materials Research and Engineering, A*STAR, Singapore. He has 10 years of experience in energy storage devices, including structural power composites. He is also an assistant professor (adj) at the School of Materials Science and Engineering, Nanyang Technological University. His work focuses on solid electrolytes for solid-state batteries and high-throughput experimentation for materials discovery.



Mats Johansson has been a full Professor in Polymer Technology at KTH Royal Institute of Technology, Stockholm, Sweden since 2006. Professor Johansson has more than 35 years of experience in research in the field of polymer technology with a strong focus on synthesis and characterization of thermosets in applications ranging from organic coatings to batteries. Prof Johansson has published more than 170 peer-reviewed original research scientific publications and more than 100 conference contributions. Mats Johansson is Editor in Chief for Progress in Organic Coatings.



Göran Lindbergh is a professor in Applied Electrochemistry at the Department of Chemical Engineering at KTH Royal Institute of Technology in Stockholm, Sweden. His work focuses on electrochemical power sources and electrolytic processes within the field of electrochemical engineering. Common themes in the ongoing research projects are the mathematical modelling and electrochemical characterization of batteries, fuel cells, and electrolyzers, where multifunctional carbon fiber composites are a current application.



Jodie L. Lutkenhaus is the Axalta Chair and Associate Department Head in the Artie McFerrin Department of Chemical Engineering at Texas A&M University. Lutkenhaus received her B.S. in Chemical Engineering in 2002 from The University of Texas at Austin and her Ph.D in Chemical Engineering in 2007 from Massachusetts Institute of Technology. Current research areas include polyelectrolytes, redox-active polymers, energy storage, and composites. She has received recognitions including the World Economic Forum Young Scientist and AFOSR Young Investigator. Lutkenhaus is the Deputy Editor of ACS Applied Polymer Materials and a member of the U.S. National Academies Board of Chemical Sciences & Technology.



Milo S. P. Shaffer is Professor of Materials Chemistry at Imperial College and was co-Director of the London Centre for Nanotechnology (2010-2020). He is interested in nanomaterials synthesis, modification, and hierarchical assembly, for applications including composites and electrochemical devices.



Natasha Shirshova is an Associate Professor in the Department of Engineering at Durham University, which she joined in 2014 as an Assistant Professor before promotion in 2022 to her current position. Prior to this, she held a Royal Society/NATO Postdoctoral Fellowship and a subsequent postdoctoral position at Imperial College London. Dr Shirshova has a strong background in polymer chemistry, with research interests spanning polymer synthesis and characterization to polymer composites. In addition, she has considerable experience working on multidisciplinary projects with a focus on functional polymers and composites, including multifunctional materials, such as structural electrolytes for multifunctional energy supercapacitors.



Madhavi Srinivasan is a Professor in the School of Materials Science and Engineering at Nanyang Technological University, Singapore. She is the Executive Director, ERI@N, Energy Research Institute at NTU. Her research focuses on synthesis, fabrication, and application of nanoscale materials/architectures in improving the performance of electrochemical energy storage devices such as advanced lithium-ion batteries, structural batteries, sodium ion batteries, and multivalent aqueous batteries. She also works on the circular economy with an emphasis on novel energy storage solutions and recycling of e-waste and lithium-ion batteries. Her research papers rank in the top 1% by citations in the field by Clarivate in Web of Science.



Johanna Xu is a researcher specializing in structural batteries, focusing on sustainable energy storage and multifunctional materials. She received her PhD from Luleå University of Technology in 2019. Her current research centers on the development and experimental characterization of structural battery composites, including mechanical and electrochemical assessments at the constituent, cell, and multicell levels.



Dan Zenkert is a professor in Lightweight Structures at the Department of Engineering Mechanics at KTH Royal Institute of Technology in Stockholm, Sweden. He received his Ph.D. from the same institution in 1990 and did a post-doc at the University of Auckland, New Zealand, 1991–1992. He has been working on multifunctional carbon fiber composites since around 2010, with a special focus on structural battery composites, but lately also on adding other functions, such as sensing, shape-morphing, and energy harvesting through the coupling of mechanics and electrochemistry.