Commercialization of GN3 Graphene Material as an Active Electrode for High Energy Supercapacitors

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ABSTRACT

A novel preparation method of high energy density, nitrogen-doped graphene material (GN3/SC-GN3) prepared based on fluorographene chemistry was introduced during the last ESA SPCD 2022[1],[2]. This highly nitrogen-doped (~16%) graphene, with diamond-like carbon-carbon bonds and an ultra-high mass density of 2.8 g/cm^3, is an excellent host for the ions of an ionic liquid electrolyte, capable of delivering unprecedented energy densities of up to 200 Wh/L at a power of 2.6 kW/L and 143 Wh/L at 52 kW/L.

The paper provides an update on the latest achievements of the material development towards its mass manufacturing readiness and commercialization. The first GN3 wound and pouch supercapacitor prototypes are introduced, including their initial characterization, and the next step qualification evaluation test plans are also discussed.

1. INTRODUCTION

Supercapacitors (SCs) are energy storage devices with remarkable qualities including fast charging/discharging (i.e., high power) and extra-long-life cycles (i.e., they can be charged and discharged for hundreds of thousands of cycles). On the other hand, matching the energy density (i.e., the ability to store charge/energy) of the marketed batteries remains a challenging task. Commercial SCs have cell-level specific energies (and energy densities) of 10 Wh/kg (58 Wh/L). For comparison, lead-acid batteries offer 20–35 Wh/kg (40-80 Wh/L), whereas state-of-the-art Li-ion batteries achieve~250 Wh/kg (~400 Wh/L) (Figure 1.).

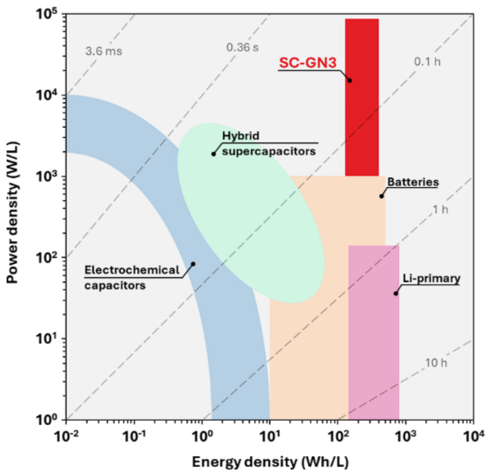

Figure 1. The Ragone plot shows that SC-GN3 supercapacitor material lays the ground for achieving energy densities like batteries while keeping the power performance of supercapacitors.

The energy density of SCs is determined by the electrochemically active electrode material and by the electrolyte used. It is necessary to develop electrode materials with significantly improved energy densities combined with long life and high power to boost and broaden the application range of SCs. Another important aspect of SCs lies in their carbon-based electrodes, which do not require critical metal elements like Co, or Li, thus shaping an energy storage technology that will be eco-friendly and sustainable.

Because the electrode material/electrolyte interface plays an important role in the charge storage mechanism, considerable effort has been invested in lightweight materials with high surface area materials, such as nitrogen-doped mesoporous carbons, carbon nanosheets, activated carbons, graphenes, and carbon nanotubes. The specific energies of these materials range from ca. 10 to 90 Wh/kg, with the highest values being reported for activated graphene, carbon nanosheets, and nanotubes in ionic liquid (IL)-based electrolytes. Unfortunately, like most commercial electrodes, these carbon materials have very low mass densities (ca. 0.3-0.7 g/cm^3). Consequently, the volumetric energy densities achieved with these materials reach 22– 45 Wh/L.

Within the ERC Consolidator grant 2D-CHEM and the subsequent ERC Proof of Concept grant UP2DCHEM, we developed, via fluorographene's chemistry, a nitrogen super-doped graphene electrode material (SC-GN3) with an unprecedented density of 2.8 g/cm3[2]. The nitrogen doping of graphene via mild and low energy processes to afford homogeneous product composition and topology with high nitrogen content (>10 at. %) remains a challenge of contemporary 2D materials chemistry.

We developed a previously unexplored route to synthesise N-doped graphene by exploring another pathway in our heavily studied area of fluorographene chemistry. We have employed the reaction of fluorographite, a widely available precursor, with a standardly used solvent dimethyl formamide and sodium azide, to prepare N-doped graphene with up to 16 at% of N in its lattice in the pyridinic, pyrrolic and graphitic configurations.[2]

The resulting high density of the material (SC-GN3) is ascribed to the presence of diamond-like carbon-carbon bonds close to the nitrogen incorporation sites in the original material.

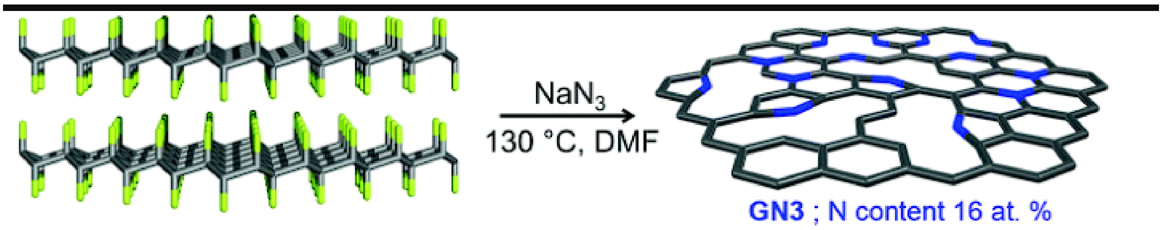

Figure 2. Tunable synthesis of N-doped graphene from fluorographene at easily affordable temperature of 130 °C.

The ultrahigh mass density of SC-GN3, combined with its polar nitrogen moieties and vacancies, is showing a promising performance in the charge storage field. Its fast charge transport allows reaching an energy density of 200 Wh/L at a power density of 2.6 kW/L and almost 150 Wh-/L at the exceptionally high-power density of 52 kW/L. Such performance, obtained from a symmetric SC full cell, corresponds to an improvement of 74% and 190% in energy and power density, respectively, compared to the previous record reported in the scientific literature. Therefore, the utilization of SC-GN3 in SCs holds great potential towards novel energy storage devices having the energy density of up to 200 Wh/L, approaching that of some rechargeable batteries, while enabling fast charging/discharging and long life-cycle stability because SC-GN3 displays 100% retention of capacitance after 10,000 cycles at 20 A/g. Demonstrating such impressive durability, cycle life, and very high-power density while maintaining high-energy density approaching that of advanced rechargeable batteries means that we here introduce a very novel and attractive new technology for energy storage and conversion.

So far, we have proposed here a new electrode material that enables energy density far beyond the competition in the SC field, even approaching that of some rechargeable batteries, along with extended cycle life and high-power density exceeding the state-of-the-art of this field. The added value of the SC-GN3 material also lies in its composition, which is mainly based on carbon and does not contain any metals, let alone critical ones, in the form of a black powder.) Other research efforts aim at SCs that may achieve relatively improved performance with respect to the state-of-the-art but involve the use of (heavy) metals and often complicated growth of the electrode materials on complex current collectors. Such processes are incompatible with roll-to-roll industrial manufacturing practices. Application of SC-GN3 in SC devices may significantly advance the field of SCs, boosting their performance and expanding their application potential.

Two main construction types of SCs dominate in the market today. The pouch (stacked) type (Figure 3.D.) brings maximum energy density at a low profile and flexible designs, while the wound (cylindrical) type (Figure 3.B.) fits better with high manufacturing volumes at a lower cost.

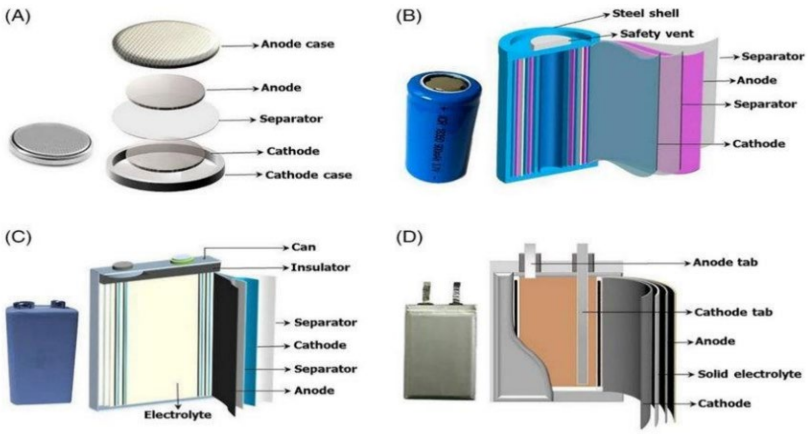

Figure 3. Construction geometries used for energy storage devices (batteries, capacitors and supercapacitors), a) coin cell, b) wound (cylinder), c) prismatic and d) pouch cell (source: Info Mat 1(1):6–32, 2019).

The specific objectives of the work presented herein are to prepare commercialization-ready functional SC-GN3 material pouch and wound supercapacitor prototypes which can address the energy, power density and efficiency challenges while maintaining the sustainability of the supercapacitors and supply chain. The properties and efficacy of the SC prototypes are to be investigated and evaluated in relevant applications/environments. Upon reaching the industry SC qualification standards, the prototype design will be frozen, and we will design manufacturing information sheets for scale-up. In parallel, we will perform an evaluation and qualification testing of the SCs according to industry standards.

The main application benefits of the SC-GN3 supercapacitors are in further downsizing of the energy storage and power supply/management modules without compromising its power delivery features. The space and weight savings may become enabling technology for new projects, especially in micro-transportation or flying hardware such as drones or the aerospace industry. Supercapacitor technology has already been on an ESA roadmap, and a further increase in energy and power density is of prime interest to enable new missions. Close cooperation with ESA may bring new qualified EU-based vendors for supercapacitors and support EU independence/competitiveness in the space environment.

2. SC-GN3 SUPERCAPACITOR CONSTRUCTION

The basic supercapacitor design, as illustrated in Figure 3. consists of two electrodes with active material deposition, a separator, an electrolyte and packaging. Manufacturing processes are then specific to different types of construction types and geometries.

Wound supercapacitor prototypes have been prepared in cooperation with ITELCOND, a capacitor manufacturing company and flat pack pouch types with Bar-Ilan University, which has wide experience in energy storage chemistry and pouch prototyping. (See Figure 4.)

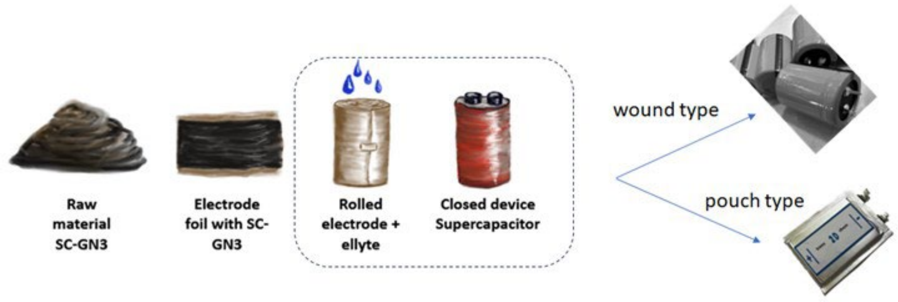

Figure 4. SC-GN3 supercapacitor key manufacturing process steps

2.1. Target Supercapacitor Parameters

The market analyses and other inputs defined target specification requirements as follows:

Tier 1. Requirements (Must Have)

Match with Industry Standard Supercapacitor Specifications:
- Energy Density exceeding 16Wh/L
- Power Density exceeding 29kW/kg
- Voltage: >2.85V
- Temperature Range:
  Wound cell: -40°C to +65°C / +85°C with derating
  Pouch cell: -40°C to +70°C
- Environmental Life Tests: as per the industry standards

Tier 2. Requirements (Nice to Have)

- Improved Temperature Range: 40°C to +85°C
- Higher Voltage: >3V
- Low Self-Discharge /w Temperature Characteristics
- Reflow Capability
- Flexible Design (Wearables)
- Bio-Waste Material Use / Sustainability even at Lower ED/PD

2.2. Commercialization-ready SC-GN3 Active Electrode Material

The first objective target of commercialization of the SC-GN3 material is validation (and any necessary modification) of the material processes that have been used for lab scale evaluation, ready and efficient also for commercial mass manufacturing production. The validation also includes verification that (i) the supercapacitor manufacturing processes (that may include mechanical and electrical loads/shocks) may not degrade the SC-GN3 material performance and from the other hand (ii) the supercapacitor manufacturing processes will be able to adopt SC-GN3 material to achieve performance benefits. The economic feasibility of the processes must be kept under consideration in addition to offering high-cost vs performance value of the final product.

<u>SC-GN3 coated aluminum film electrode preparation</u>

Aluminum electrode foils covered by SC-GN3 active materials have been prepared for supercapacitor manufacturing using Dr Blade deposition technology (Figures 5. and 6.).

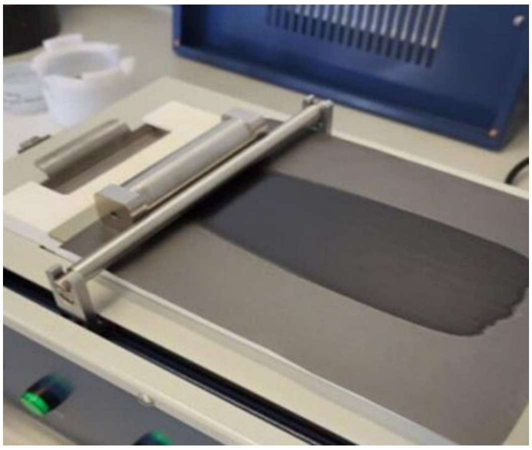

Figure 5. Laboratory Dr Blade deposition technique layer

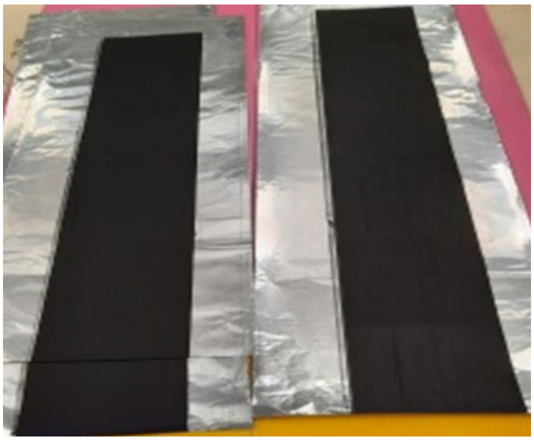

Figure 6. Electrodes with SC-GN3 deposited

2.3. Electrolyte Selection

Traditional electrolytes are based on sulfuric acid or KOH aqueous solutions. They are RoHS and REACH compliant, offer low internal resistance, but limit the voltage to roughly one volt per cell.

Organic electrolytes are generally based on acetonitrile or propylene carbonate that allow higher voltage per cell – a reason why they are currently favored but with higher internal resistance.

Ionic electrolytes are salts molten at room temperature. Compared to existing organic electrolytes, ionic liquids offer the potential for increased operating temperature, increased cycle life, improved safety, and increased energy density. By replacing conventional electrolytes with ionic liquids, the energy storage capacity of ultracapacitors could double by allowing them to be charged to higher voltages. Ionic liquids proved to be the most promising electrolyte to enhance the voltage of supercapacitors to values higher than 3.0 V. However, their relatively high viscosity and low ionic conductivity reduce, at present, the power performance of supercapacitors.

Energy content in a device is a function of voltage squared, thus, the selection of a higher voltage electrolyte may bring higher energy densities benefits, nevertheless, charge mobility within the electrolyte also defines its other features such as non-flammability, long-term thermal and chemical stability and its temperature performance and final temperature range with respect of its long-life expectations. As evident, a proper choice of the electrolyte is a complex and compromising decision in most cases.

In our case of the first prototype, we evaluated three various ionic liquid electrolyte types to meet 2.85V long-term stability at standard temperature range -40°C to +65°C / +85°C with derating. See chapter 2.5. compatibility testing of these materials. The next prototypes will target extension to >3V at the same temperature range.

2.4. Separator Selection

Selecting the appropriate separator for a supercapacitor may impact the device's efficiency and safety. The separator acts as a physical barrier between the anode and cathode, preventing short circuits while allowing ions to pass through. Ideal separator materials should possess high ionic conductivity, high electric resistivity, mechanical strength, wettability, and chemical and dimensional stability. Common materials used include glass fiber, cellulose, ceramic fibers, and polymeric films. Advances in materials science have led to the development of separators that offer improved thermal stability, specific capacity, and life of supercapacitors. For instance, plasma treatment has been used to enhance the wettability of polymer-based membranes, which are typically limited by their hydrophobic nature. Our aim was to use separators that can provide higher ionic conductivity, mechanical strength, and thermal stability at a lower manufacturing cost.

2.5. Wound Cell Construction

The most common types of wound construction capacitors are aluminium electrolytic, film and supercapacitors. Those different types of capacitors are built starting from a number of common raw materials; the major differences are concentrated in the dielectric, spacers and electrolyte. The general construction resulting in the wound cylindrical type is also similar, as one can see from Figure 7. below.

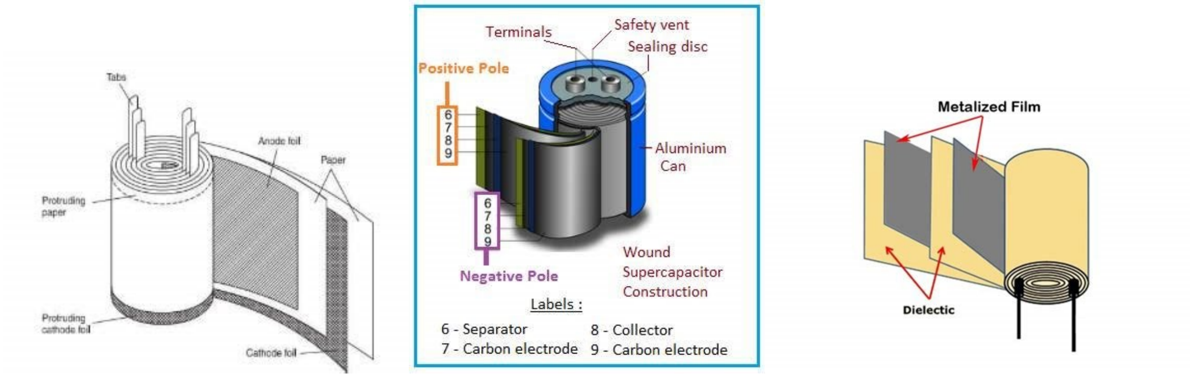

Figure 7. Wound capacitor construction

Therefore, from a general point of view, not only is the appearance similar, but also the failure mechanism is so. Additionally, some fail mechanisms are common, and the general knowledge of the technology of the less recent capacitor, namely aluminium electrolytic, can be successfully transferred to the most recent types of supercapacitors.

There are several issues to consider when scaling up the results of pure research into industrial practice to make a reliable, repeatable, sustainable and economically viable production.

For all types of capacitors, one of the early failure mechanisms is the loss of electrical parameters caused by corrosion, especially in harsh environmental conditions (which may vary depending on the specific type of item) due to various factors, such as high humidity, high temperature, and exposure to corrosive substances. This may cause the electrolyte to leak or degrade, leading to reduced capacitance, increased ESR, increased power consumption, and overheating.

From the materials point of view, a supercapacitor is built using:

- Aluminium foil, coated
- Activated material
- Separators
- Electrolyte
- Can
- Cover and rheophores
- Rubber sealing

One of the main challenges for electrochemical and thermal stabilities are the **compatibility of electrolytes with the electrode material and all capacitor components**, the stability of the potential window at higher voltages, and the standardization of the characterization methods.

Apart from the failure caused by overstress in the application and the raw material's choice, one of the weakest points in reliability is the long-term stability of aluminium foil with coated active material. Thus, part of the construction reliability-by-design is testing of material compatibility between the capacitor components and the selected electrolyte type.

The testing of the interactions of the electrolyte also includes cover and rubber sealing to avoid performance issues such as short-time corrosion caused by the release of components into the supercapacitor electrolyte and medium to long-term reliability preventing electrolyte evaporation, increasing water content or pollution.

The individual parts of commercial capacitors were cut into small pieces and immersed into three different electrolyte types for 14 days in the atmosphere of an argon glovebox. Few analytical techniques were chosen to investigate samples. Scanning electron microscopy (SEM) with Energy Dispersive X-Ray (EDS) mapping and Fourier transform infrared microscopy (FTIR). All samples were weighed before and after the treatment with electrolytes, the masses are reported in Table 1.

samples	Electrolyte I.			Electrolyte II.			Electrolyte III.		
	mass before [g]	mass after [g]	Change [%]	mass before [g]	mass after [g]	Change [%]	mass before [g]	mass after [g]	Change [%]
Coated Foil	0.51905	0.51541	-0.7	0.50012	0.49777	-0.5	0.37324	0.37213	-0.3
Can	0.17496	0.17332	-0.9	0.13025	0.12918	-0.8	0.12890	0.13035	1.1
Rubber	0.11279	0.11223	-0.5	0.09261	0.09207	-0.6	0.09338	0.09239	-1.1
Separator	0.37999	0.38004	0.0	0.37922	0.37923	0.0	0.38061	0.38071	0.0

Table 1. Table with masses of samples before and after treatment in organic solvents. Highlighted in yellow are increases in mass after the test, and highlighted in red are decreases in mass after the test.

The compatibility analysis shows the materials in direct contact with electrolytes, show a relatively good variation of the characteristics in terms of mass changes below 1% for all three electrolyte types considered. Other analytical techniques show minor differences between the samples. In conclusion, all the proposed electrolyte types seem fit for use in commercial supercapacitors.

The SC-GN3 wound SC cell manufacturing process considers using the well-proved typical industrial methods of manufacturing, testing storage and packaging. This is useful to prevent non-forecasted behaviour.

Upon the compatibility check, the supercapacitors have been wound using the SC-GN3-covered aluminium foil electrodes with leading termination tabs and separator. The sample electrode termination tab connection is shown in Figure 8.

The wounded stack was then assembled into an aluminium can using standard capacitor assembly techniques and the first (zero prototype) of SC-GN3 supercapacitors was manufactured (Figure 9.). The wound stack inside the can has dimensions of 76 mm in length and 45 mm in diameter, featuring a capacitance of 1500 F and a rated voltage of 2.85 V. Capacitance values were measured by the methodology described in Chapter 3. below.

Figure 8. Sample electrode termination tab connection

Figure 9. Final SC-GN3 "zero prototype" of wound supercapacitor with 1500F / 2.85V active element inside

2.6. Pouch Cell Construction

The construction of a pouch supercapacitor consists of the same elements as wound capacitors, e.g. two electrodes, a separator immersed into an electrolyte and sealed in a package. Unlike the wound type, the pouch structure uses electrode and separator plates made by stamping tools and sealed in a usually flexible package. The Figure 10. Shows a schematic construction of a pouch supercapacitor.

The advantage of the pouch technology is its capability to produce thin, low-profile supercapacitors for flexible or space constrained applications. The main disadvantage includes limitations for high volume mass production and thus higher manufacturing cost compared to the wound types.

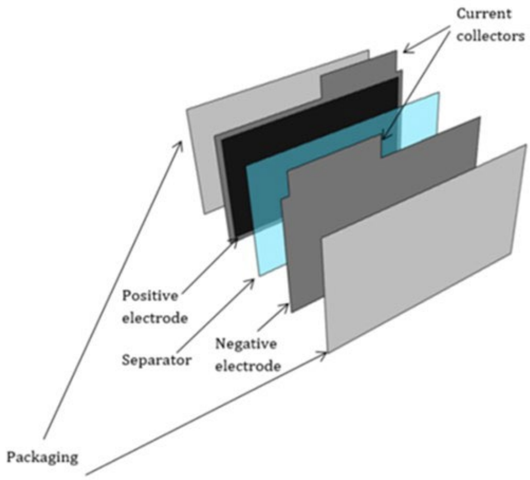

Figure 10. Pouch "flat pack" supercapacitor construction

The SC-GN3 pouch cell is using the same active electrode foils covered by SC-GN3 active material. In difference to the wound types, the electrode is not wounded into a roll, but it is cut into individual square elements (Figure 11.) and the final pouch capacitor is created by stacking those elements together (Figure 12.).

Each pouch cell is composed of 20 positive and 20 negative electrodes in our case, in the dimension of 57x41 mm, reaching 48 F capacitance. The initial voltage rating is 2.85 V, but the projection is to increase the rated voltage up to 3.7 V level in the later prototype stage.

Figure 11. Pouch SC-GN3 cell electrode stamping tool

Figure 12. Pouch SC-GN3 manufactured prototype cells

In addition, a high energy module has been prepared for testing purposes (Figure 13.) with a total capacitance value of 500 F at 2.85 V.

Figure 13. 500 F 2.8 5V pouch supercapacitor module

3. FIRST PROTOTYPE CHARACTERIZATION AND BENCHMARK

The first SC-GN3 wound, and pouch cell supercapacitor prototypes described in Chapter 2. were characterized by basic electric parameters:

Capacitance: is measured during discharge of the ultracapacitor with a constant current source from its rated voltage to half its rated voltage. Referring to the Figure 14., capacitance is calculated from the following:

$$C = \frac{I_d \bullet t_d}{V_w - V_f}$$

ESR value: Referring to the Figure 15. the DC resistance, or ESR, is calculated from the following:

$$ESR = \frac{V_f - V_{\min}}{I_d}$$

The resistance measurement considers all resistive components over approximately five-time RC constants of the capacitor and is inclusive of all resistive elements. The actual resistance measured would be lower if measured over a shorter duration than 5 seconds.

DCL leakage current: Current measured after 72 hours of constant voltage hold at VR and 25 °C. The initial leakage current can be higher.

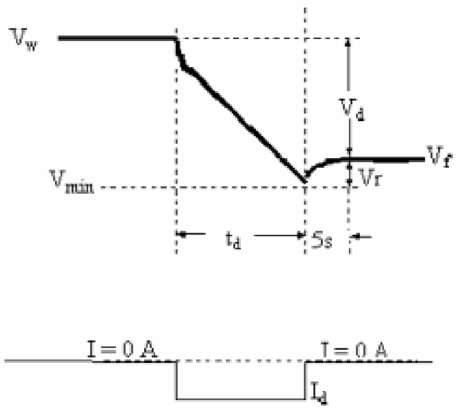

Figure 14. Supercapacitor voltage and current time base chart for Capacitance, ESR and DCL measurement reference

The measured Capacitance and ESR values were used to calculate the samples' energy density, power densities and compared with the existing supercapacitors on the market. See results in the following chapters 3.1. and 3.2 for wound and pouch type supercapacitors, respectively.

3.1. Wound geometry SC benchmark

The first made (“zero”) prototype and projected SC-GN3 characteristics were compared to the current supercapacitor features on the market. Table 2. below summarizes the key parameters of WOUND supercapacitors.

Most supercapacitor components on today’s market reach energy densities below 10 Wh/L maximum with 16 Wh/L state of the art Vendor 1. as the current state of the art.

	wound supercapacitors on the market				GN3 Our Project	
Supercapacitor Types	Vendor 1.	Vendor 2.	Vendor 3.	Vendor 4.	projection	achieved prototype 0
Capacitance F	5000	3400	3000	350	3400	1500
Voltage V	3	3	3	2,7	3	2,85
Energy Content Wh	6,25	4,25	3,75	0,35	4,25	1,69
Energy density Wh/L	16,0	10,9	9,6	6,7	**55,2**	**14,0**
Specific energy Wh/kg	11,1	8,5	7,1	5,5	**39,4**	**11,3**
Power density kW/kg	28,8	24,0	25,1	9,8	58,5	TBD
Diameter mm	60	60	60	35	35	45
Length mm	138	138	138	55,4	80	76
Volume cc	390	390	390	53	77	121
F/cc	12,8	8,7	7,7	6,6	44,2	12,4
Temperature Range	-40 to +65C	-40 - 65C/85d	-40 - 65C/85d	-40 to +65C	-40 to +65C	-40 to +65C
Weight kg	0,565	0,500	0,525	0,065	0,11	0,15
ESR mOhm	0,20	0,24	0,23	3,5	0,5	TBD

Table 2. Competitor wound supercapacitor benchmark vs the SC-GN3 projection and first prototype characterization

As seen in Table 2. above, our first wound cell prototype, even at an early stage without optimization, has already achieved an energy density level of 14Wh/L. This surpasses most SCs on the market and keeps us optimistic about achieving the projected ED level of 55 Wh/L, which is ~4x higher compared to the state-of-the-art. Similarly, our power density level exceeds the leading 10 Wh/kg values, demonstrating significant potential in this important parameter.

The SC-GN3 projected performance advantage is clearly visible compared to the Vendor 2. existing SC on the market. SC-GN3 projected can volume at the same capacitance and voltage 3400F /3V is just 77cc vs 390cc e.g. 20% of Vendor 2 can volume. The achieved results are at 121cc e.g. 30% of the vendor 2 volume.

3.2 Pouch geometry SC benchmark

The following Table 3. shows an overview of POUCH supercapacitor types on the market.

	pouch cells				GN3 Our Project	
SC Manufacturer	Vendor 1.	Vendor 2.	Vendor 3.	Vendor 4.	projection	prototype
Capacitance F	20	2	2,4	0,4	48	
Voltage V	2,5	2,7	2,75	5,5	3,7	
Energy Content Wh	0,02	0,002	0,003	0,002	0,09	
Energy density Wh/L	2,2	2,5	2,0	1,3	**10,8**	
Specific energy Wh/kg	3,47	0,81	1,01	0,88	**6,66**	
Power density kW/kg	2,50	17,50	14,52	12,25	TBD	
ESR mOhm	60	20	25	156	TBD	
Length mm	50	30	39	20	57	
Width mm	46	14	17	18	41	
Thickness mm	3,4	1,9	1,9	3,5	3,6	
Volume cc	8	1	1	1	8	
F/cc	2,6	2,5	1,9	0,3	5,7	
Temperature Range	-40 to 65C/85d	-40 to +70C	-40 to +70C	-40 to 85C	-40 to 65C/85d	
Weight kg	0,0050	0,0	0,0	0,0019	0,0137	
ESR mOhm	40,0	30,0	30,0	156,0	40,0	

Table 3. Competitor POUCH supercapacitor benchmark vs the SC-GN3 projection

Projection based on realistic estimated values of the final pouch supercapacitor is ~4-5x times higher compared to similar products on the market.

Two Tables 2. and 3. highlight the benefits of the SC-GN3 technology as a gate opener for discussion with potential stakeholders and customers.

4. EVALUATION AND QUALIFICATIONS TEST PLAN

Evaluation and qualification tests were proposed based on the common industry standards as shown in Table 5. One of the key tests applicable to supercapacitors is life cycle assessment 0.5/1.0Vr 100A cycles in 5RC duty cycle in the duration of 500 000 cycles to prove the stability of capacitance under high charge / discharge continuous load.

Environmental tests include dry heat at top temperature ratings and 6 months of shelf life. Mechanical robustness is tested by vibration resistance. In addition, a low pressure/vacuum test was added to check the capability of the construction to withstand flying hardware conditions.

The test is to be initiated in late Autumn 2024, and the results data package will be available around May 2025.

Description of the Tests and Characterization Conditions

Charge / Discharge Test: The charge / discharge test consists of 500 000 cycles 0.5Vr to 1.0Vr under a maximum current of 100A at 5RC duty cycle at room temperature and no forced cooling. 15-second rest is allowed between each c/disc cycle.

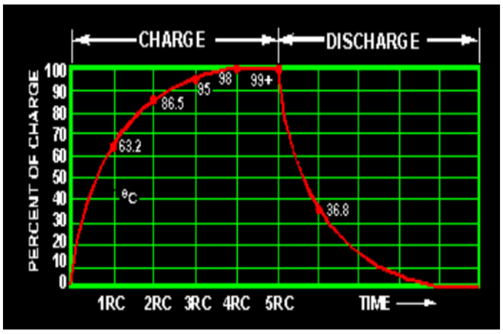

Figure 15. Charge / discharge chart illustration

BOL and EOL Criteria:

BOL (Beginning of Life): Rated/Initial product performance at 25C:

Capacitance: minimum rated value.
ESR: maximum rated value.
DCL Leakage Current: maximum rated value.

EOL (End of Life):
Capacitance Change: < 20% of BOL
ESR: 100% of max. BOL
DCL: Initial BOL
Appearance: No remarkable defects

Ref.	TEST	TEST METHOD	REF STANDARD	SAMPLES	PARAMETER	FREQUENCY	LIMITS
1.	Life Cycle	Cycle: 0.5Vr / 1Vr at 5RC duty time		5	CAP, ESR	Init, every 100k	EOL
		Current: 100A			DCL Leakage		
		No. of cycles: 500,000			Appearance		
		Temp: +25C					
		no forced cooling, 15 second rest is allowed between each c/disc cycle					
2.	High Temperature Life dry heat	Temperature: +65C	MIL STD 202	10	CAP, ESR	Init, every 250h	EOL
		Voltage: Vr	Method 108		DCL Leakage		
		Test Duration: 1,000hrs	life		Appearance		
3.	Shelf Life	Temperature: +25C	MIL STD 202	10	CAP, ESR	Init, every month	EOL
		Voltage: no load	Method 108		DCL Leakage		
		Test Duration: 6 month	life		Appearance		
4.	Vibration Resistance (mechanical robustness)	Temperature: +25C	MIL STD 202	5	CAP, ESR	Init, each axe	EOL
		Amplitude: 1.5mm	Method 201		DCL Leakage		
		Frequency: 10~55 Hz	vibration		Appearance		
5.	External Humidity	Temperature: +65C	MIL STD 202	10	CAP, ESR	Init, final	EOL
		RH: 90%	Method 103		DCL Leakage		
		Voltage: Vr	humidity		Appearance		
		Test Duration: 1,000hrs	MIL STD 202				
6.	Vacuum/Low Pressure	Temperature: 55C	Method 105	5	CAP, ESR	Init, every 250h	EOL
		Pressure: <50mBar	barometric pr.		DCL Leakage		
		Voltage: 0.85Vr	ESCC 226300		Appearance		
		Test Duration: 1,000hrs	7.2.4.4.Vacuum				

Table 4. SC-GN3 supercapacitor evaluation and qualification test proposal

5. NEXT STEPS

Optimizing electrode designs

The “zero” prototype results presented in this work at the time of writing this paper, are based on simple laboratory electrode foil coating equipment with limited active material mass loading capability. The active loading is thus below the capability of the SC-GN3 material. Advanced prototyping equipment has been ordered to enable higher active loading of the material; therefore, further significant improvements of energy densities are foreseen with the next generation of electrodes and prototypes.

Figure 16. A representation of a roadmap of next generation electrodes and prototypes.

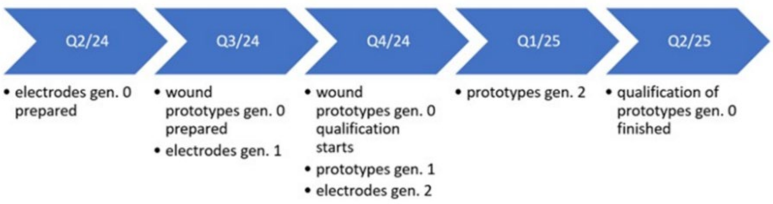

Figure 16. SC-GN3 coated electrode and prototype introduction roadmap

Sustainability of SC-GN3 material supply chain

Sustainability of the supply chain is a key consideration to avoid geopolitical issues, conflict materials and disruption in mass SC-GN3 production. The further focus is to ensure a stable and economic supply of fluorographene precursor from local sources as the core SC-GN3 synthesis element.

6. CONCLUSION

The advancements in SC-GN3 nitrogen-doped graphene materials are a significant milestone in the field of energy storage technologies. The ability of SC-GN3 to enhance supercapacitor energy and power densities while using sustainable and readily available materials marks a leap forward in electrode material science.

The first unoptimized “zero” prototypes already reached market state-of-the-art energy density values of 14Wh/L with a realistic projection to reach the record-high level of 55Wh/L at a high-power density of over 30kW/kg in next prototypes. This may significantly downsize the existing SC can size weight and volume by up to ~80% or boost the same case size energy content to record high level.

The next prototypes will be introduced by the end of the year 2024 with basic characterization. Follow-up reliability testing will start in a duration of 6 months. Thus, a reliable data package is expected to be available in mid-2025.

7. REFERENCES

[1] Zednicek.T.at col.; Novel Graphene Material for High Energy Storage Supercapacitors; ESA SPCD 2022 ESA ESTEC, https://epci.eu/novel-graphene-material-for-high-energy-storage-supercapacitors/
[2] Šedajová.V. at col.; Nitrogen-doped graphene with diamond-like bonds achieves unprecedented energy density at high power in a symmetric sustainable supercapacitor; Energy Environ. Sci. 2022, 15 (2), 740-748, doi 10.1039/d1ee02234b

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Further information can be found at the project website: https://trans2dchem.com/