

Graphite Development From a Manufacturers Perspective: Processing, Formation, and Benchmarking

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Graphite remains the most widely used anode material in lithium-ion batteries, and the initial formation of the solid electrolyte interphase (SEI) largely dictates the long-term performance of commercial cells. Developing a new graphite anode to diversify the supply chain remains challenging due to proprietary formation protocols and inconsistent test formats. In this study, we evaluate a pitch-coated synthetic graphite developed by James Durrans & Sons against a commercial benchmark using a harmonized methodology across a coin, three-electrode PAT cell, and pilot line built single-layer pouch cells. By standardizing electrode morphology, formation procedures, and testing conditions, we isolate the influence of active material properties, e.g., how the first cycle loss is linked to SEI formation and how coatings and cycling conditions such as temperature can influence it. Under “like for like” comparative studies of NMC 811/graphite pouch cell, the modified synthetic graphite developed displayed higher rate performance and increased capacity retention at 1C over 200 cycles over established commercial counterparts.

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I. INTRODUCTION

Lithium-ion batteries (LIBs) have a crucial role in the modern world, from portable electronics to electric vehicles [1]. As global energy demand increases and the shift toward electrification accelerates, the performance, safety, and sustainability of LIBs are being placed under greater scrutiny. Within LIBs, graphite is the most used active material in the anode electrode component due to its stability, safety, cost, and availability [2]. Graphite for LIBs is sourced either from naturally occurring ores (natural graphite) or from the thermal graphitization of non-graphitic or amorphous hydrocarbon materials at temperatures $>3000\text{ }^{\circ}\text{C}$ (synthetic graphite) [3]. Whilst natural graphite has the advantages of lower cost, high capacity and a smaller environmental footprint, synthetic graphite benefits from high performance characteristics such as higher purity, wider electrolyte compatibility, faster charge and discharge rates and longer cycle life [4]. Synthetic graphite can also be tailored by the manufacturer during the production process to optimize specific properties

(e.g., electrical and thermal conductivity) for potential customers [5].

Formation of the solid electrolyte interface (SEI) on the anode during battery formation is one of the most critical steps during LIB cell manufacturing [6]. The SEI layer allows lithium ion to pass through whilst preventing electrolyte decomposition and other undesirable side reactions at the interphase during cycling [7]. It also provides strong electrical insulation, high ion conductivity, mechanical and thermal stability [8]. Therefore, the formation of the SEI is crucial for both the electrochemical and safety properties of the LIB cell, as a poorly formed SEI will cause inadequate performance and premature cell failure [9]. Despite its importance, SEI formation remains one of the least understood and most variable aspects of battery production [10]. There is no designated “best formation protocol”; the procedure will often depend on cell chemistry, format, and size. Each material manufacturer will also have their own internal battery formation protocol, which are often not fully disclosed to potential customers [11]. This in turn makes the screening of new LIB materials a challenge within both academic and industrial settings. For example, differences in electrolyte formulation [12], formation temperature [13], formation voltage windows [14], current densities [15], or stepwise charging protocols [16] can lead to significantly different SEI compositions and battery-aging behavior. Understanding these differences and their impact on performance is

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therefore essential for the rational design and evaluation of novel anode materials.

In this study we have collaborated with a commercial graphite manufacturer to develop and characterize synthetic graphite derived from needle coke with (XG24C) and without (XG24) pitch coating to highlight the link between formation conditions and graphite morphology with first cycle loss (FCL). The pitch-coated graphite anode was subsequently benchmarked against a commercially available pitch-coated synthetic graphite (V–H, BTR) reference anode in a variety of cell formats. To isolate material-driven effects and better assess the scalability of different graphite types, it is essential to maintain comparable electrode morphology and processing conditions. Therefore, we aimed to make a “like-for-like” comparison under realistic, application-relevant formats, thereby ensuring that any observed differences in electrochemical performance or SEI behavior could be attributed to intrinsic material properties rather than variation in electrode architecture or fabrication parameters.

II. METHODS

A. Preparation of electrodes, electrolyte, and cells

XG24 and XG24C synthetic graphite was supplied by James Durrans & Sons Ltd (Penistone, Great Britain). V–H synthetic graphite was purchased from BTR New Materials Group Co. Ltd. (Shenzhen, China). Images and characteristics of the graphite powders can be found in Fig. S1 and Table S2 in the Supplemental Material, respectively [17]. The synthetic graphite anodes contained XG24/XG24C/V–H graphite: CMC (BVH8, Ashland): SBR (BM-451B, Zeon): C65 (Imerys) in a ratio of 95.25:1.5:2.25:1 wt % with CMC/SBR as the binder system and water as the solvent. The cathode was NMC811 (S85EFT, Ningbo Ronhaby): PVDF (Solef 5130, Solvay): C65 (Imerys) in a ratio of 96:2:2 wt % with N-methyl-2-pyrrolidone (NMP, ACROS Organics) used as the solvent. For the half-cell studies, graphite anode slurries were prepared using a THINKY ARE-250 planetary centrifugal mixer (see Table S3 in the Supplemental Material for the mixing method) [17] and coated to 100 gsm (3.3 mA h cm^{-2}) on 10- μm -thickness copper foil using an Erichsen coatmaster 510 and fixed height slurry applicator. For the full coin cell, three-electrode PAT cell and SLP studies the synthetic graphite anodes were prepared using an EIRICH EL1 lab mixer (see Table S4 in the Supplemental Material for the mixing method) [17] and coated to 120 gsm (4.0 mA h cm^{-2}) on 10- μm -thickness copper foil using a Megtec reverse comma bar coater. The cathode was prepared using an EIRICH EL1 lab mixer and coated to 172 gsm (3.5 mA h cm^{-2}) on 15- μm aluminium foil using a Megtec reverse comma bar coater. After an initial drying stage, the drawdown and scaled-up electrodes were calendared down to the target porosities of 30% (anode) and

27% (cathode), using an Innovative Machine Corporation (IMC) calendar. The separator used within all cell configurations was Celgard® H1609 (Celgard LLC, Charlotte, NC, USA). The standard electrolyte used for all cell configurations was 1 mol dm^{-3} LiPF_6 in EC: EMC = 3:7 vol, with 1 wt % VC (Solvionic, Toulouse, France).

The coin cells were constructed using Hohsen 2032 cell components and assembled within an argon-filled Mbraun glovebox (O_2 and $\text{H}_2\text{O} < 0.5 \text{ ppm}$). Anode and cathode sheets were cut into disks of 15.0-mm diameter and 14.8-mm diameter, respectively, and vacuum dried in a Buchi oven at 120 °C to remove excess moisture prior to assembly. Graphite anode half-cells were prepared with 15.0-mm diameter pre-cut disks of lithium metal as the reference and counter electrode. Anode half-cells also included a Whatman borosilicate glass fiber separator next to the lithium metal. Stainless-steel spacers were used to fill out the coin cell volume.

A “PAT” cell purchased from EL-cell with three-electrode setup was used to investigate the electrochemical behavior of each electrode individually during cycling. The 3E “PAT” cell configuration has been described previously [18]. The graphite anode and NMC 811 cathode electrodes were cut into 18 mm diameter disks and placed into an EL-cell inside an Mbraun glovebox (O_2 and $\text{H}_2\text{O} < 0.5 \text{ ppm}$). The three-electrode “PAT” cell also contained a Li ring reference electrode and Whatman borosilicate glass fiber separator.

The single-layer pouch (SLP) cells were assembled in a dry room environment (dewpoint $< -50^\circ\text{C}$) using production line equipment supplied by Sovema. SLPs were constructed following similar steps described in previous work [19]. Initially, the electrodes were punched to size; the anode active area was $70 \times 50 \text{ mm}$, and the cathode $68.5 \times 48.5 \text{ mm}$. Tags (cathode: Al, anode: Ni-coated Cu) were then welded to enable external connection before the electrodes were hand wound with H1609 separator. The stack was then placed inside laminated Al pouch material and partially heat sealed to form the pouch. 1 g of the electrolyte was then syringe added before the final vacuum sealing of the cell at 50 mbar.

B. Electrochemical characterization

All cells were subjected to an 8-h wetting period at open circuit prior to cycling. Electrochemical testing was carried out using Biologic 805/810 cyclers (coin and pouch cells) and a Biologic VMP3 cycler (three-electrode cells). For the anode half-coin cells, a formation cycle (C/20, C/20) was applied prior to lithiation rate testing (C/5 C, up to 10C D) and delithiation rate testing (up to 10C C, C/5 D). The anode half-cells were cycled between 0.005 and 1.5 V. For the three-electrode “PAT” cells and SLPs, two formation cycles (C/10, C/10) were performed, followed by electrochemical impedance spectroscopy (EIS). The SLPs

were cycled in a testing rig compressed between two Perspex glass plates to ensure homogeneous stack pressure. During EIS, a 10-mV amplitude ac voltage signal was applied over the frequency range 1 MHz to 10 mHz. Lithiation rate testing up to 3C (with C/3 charge) was then carried out, followed by long-term cycling (1C/1C with C/3 capacity checks) over 200 cycles. The full coin cells, PAT cells, and SLPs were cycled between 2.5 and 4.2 V.

C. Characterization

Cross sections of the anodes were prepared using the Hitachi IM4000plus broad beam (Ar+) ion miller. Milling was performed for 3 h at an acceleration voltage of 6 kV, a discharge voltage of 1.5 kV and a stage rocking angle of 40°. The cross sections were imaged on a ThermoFisher Scios FIB-SEM Dualbeam microscope with an acceleration voltage of 30 kV and a beam current of 0.1 nA. Images shown were collected with the Inlense detector. Imaging of the XG24C graphite powder (Fig. S1 in the Supplemental Material) [17] was done on a TESCAN Clara SEM.

III. RESULTS AND DISCUSSION

Needle coke was used by James Durrans & Sons Ltd to produce uncoated synthetic graphite XG24 and pitch-coated synthetic graphite XG24C. Figure 1 shows the electrochemical formation profiles for half-coin cells containing either uncoated XG24 graphite anode electrodes or coated XG24C graphite anode electrodes formed at either 25 °C (both) or 40 °C (XG24 only).

The plateau observed in Fig. 1(a) at 0.4 V during the electrochemical formation of the uncoated XG24 anode at 25 °C indicated exfoliation of the uncoated graphite surface by solvent co-intercalation into the graphite layers. Solvent co-intercalation during SEI formation has been observed for graphite with a low active surface area (ASA) and/or lack of oxygen-containing functional groups [20]. This process causes continuous electrolyte decomposition on the exposed graphite layers during graphite SEI formation leading to a large FCL from the resulting charge loss. This suggested that the uncoated XG24 graphite had a low ASA and/or a lack of oxygen-containing functional groups, which hindered SEI formation. Conversely, the pitch-coated XG24C graphite anode formed at 25 °C did not show the 0.4 V plateau and had a low FCL. The carbon pitch coating likely suppressed solvent co-intercalation and surface exfoliation by introducing more functional groups to the graphite surface, increasing its active surface area and influencing composition of the SEI [21]. Interestingly, SEI formation at higher temperatures can also suppress solvent co-intercalation and graphite exfoliation. Figure 1(a) shows the formation profile for a XG24 graphite half-cell formed at 40 °C instead of 25 °C. At this higher temperature, the 0.4 V exfoliation

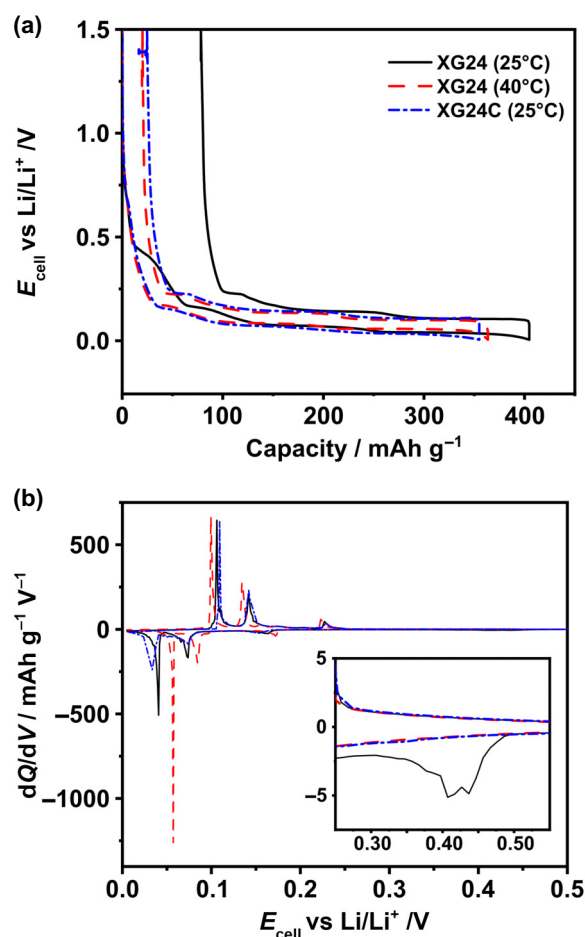


FIG. 1. (a) Half-cell formation for XG24 graphite formed at 25 °C with no pitch coating, at 40 °C with no pitch coating and at 25 °C with pitch coating (XG24C) and (b) dq/dv plots for XG24 graphite formed at 25 °C with no pitch coating, at 40 °C with no pitch coating and at 25 °C with pitch coating (XG24C).

plateau and large FCL are both absent. Higher temperatures increase the rate of lithium-ion diffusion and reduce the charge-transfer resistance of the lithium ions in the graphite and the electrolyte. The reduction potential for electrolyte decomposition becomes higher and the kinetics of desolvation improve so the SEI is formed more readily on the graphite surface, suppressing solvent co-solvation and graphite layer exfoliation [22,23]. This effect can be observed in Fig. 1(b), where the dq/dv peaks are shifted for the XG24 anode formed at 40 °C due to its enhanced reaction kinetics. The graphite exfoliation peak at 0.4 V can also be clearly seen in Fig. 1(b)'s inset graph for the XG24 graphite anode and not for the pitch-coated XG24C anode or XG24 anode formed under higher-temperature conditions. Alongside pitch coating or higher-temperature formation, electrolyte additive choice will also play a role in the suppression of solvent co-intercalation and graphite exfoliation during SEI formation. Ethylene carbonate (EC)

originally replaced poly(vinyl) carbonate (PC) in order to help form stable SEI by suppressing the continuous decomposition of electrolyte on graphite [24]. Vinylene carbonate (VC) is commonly used to minimize solvent decomposition by forming a compact and stable SEI layer on the graphite surface [25]. Additional additives in the electrolyte such as fluoroethylene carbonate (FEC) have previously been reported to suppress co-intercalation and graphite exfoliation [23]; this was also observed for uncoated XG24 graphite anodes formed at 25 °C (see Fig. S5 in the Supplemental Material) [17].

As previously discussed, each battery material manufacturer will have its own internal battery formation protocols (i.e., C-rate, temperature, electrolyte composition). These protocols are often not fully disclosed to potential customers as they are considered intellectual property of the manufacturer [11]. It is often the case that only the postformation material capacity and cycling data will be shared with potential customers and not the associated electrode formulations and/or cycling conditions. It is therefore difficult to ascertain suitable formation protocols when assessing unfamiliar or novel materials, as there is no “standard” formation protocol supplied. For that reason, “like-for-like” formation and electrochemical testing must be performed to objectively benchmark novel battery materials against comparable materials that are already commercially available.

To illustrate this point, we applied identical electrolyte composition and formation conditions to a XG24C graphite anode and an equivalent formulation graphite anode containing a commercially available pitch coated synthetic graphite V–H (purchased from BTR). The V–H graphite anode was also coated to the same coat weight and calendared to the same density as the original XG24C graphite anode. Figure 2 shows the formation profiles, lithiation capacity vs C-rate and delithiation capacity vs C-rate profiles for the compared XG24C and V–H graphite anode half cells.

This initial attempt at benchmarking the novel XG24C graphite against a comparable commercially available graphite material suggested that under these testing conditions the electrochemical performance of each graphite was near identical. However, this is only the first step of a robust benchmarking process. To be fully confident with a novel or unfamiliar battery material a “like-for-like” comparison must also be made in an industrially relevant format such as pouch cells. To that end, the XG24C graphite anode manufacture was scaled up from small-scale Thinky planetary mixing and drawdown coating to the larger scale 1-liter Eirich mixing/roll-to-roll coating on WMG’s battery scale-up (BSU) pilot line. Figure 3 compares the smaller-scale electrode manufacturing process with the larger-scale mixing and coating process on the BSU pilot line. Cross-section SEM images of calendared equivalent formulation XG24C anodes manufactured using

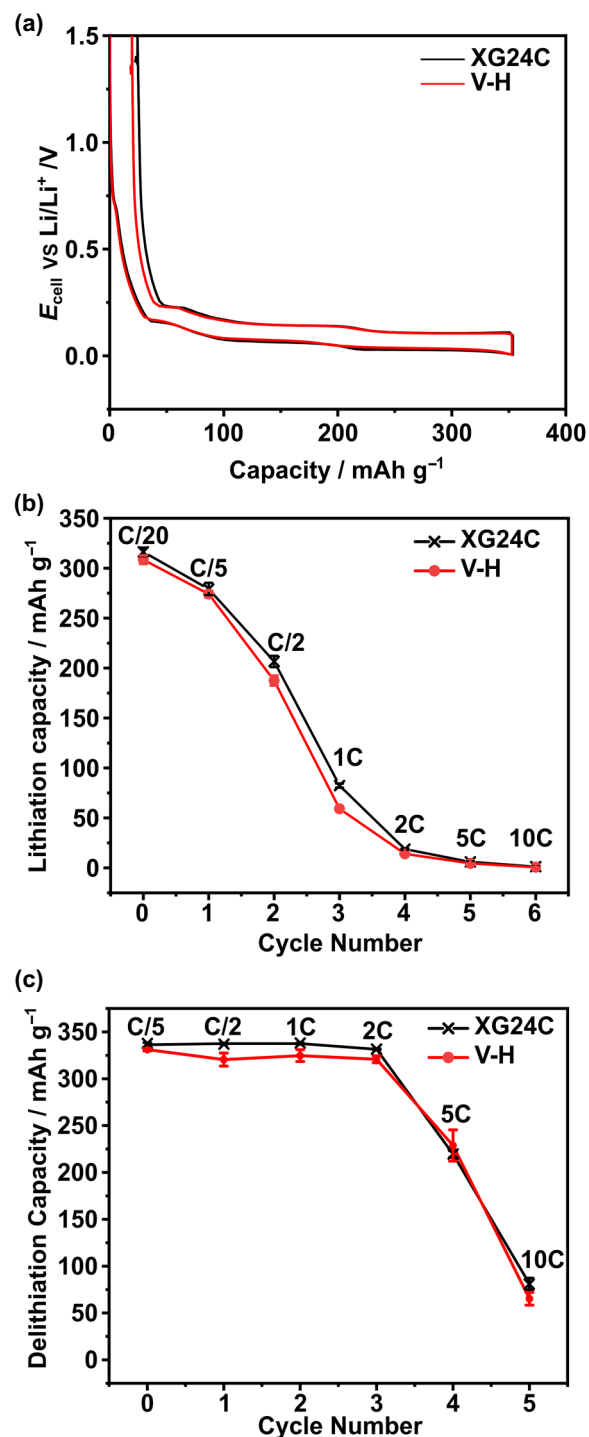


FIG. 2. (a) Half-cell formation at 25 °C for XG24C graphite vs V–H commercial graphite, (b) half-cell lithiation rate performance at 25 °C for XG24C graphite vs V–H commercial graphite and (c) half-cell delithiation rate performance at 25 °C for XG24C graphite vs V–H commercial graphite.

each method highlighted how the larger scale roll-to-roll coating, drying, and calendaring on the BSU pilot line increases the microstructure uniformity throughout the

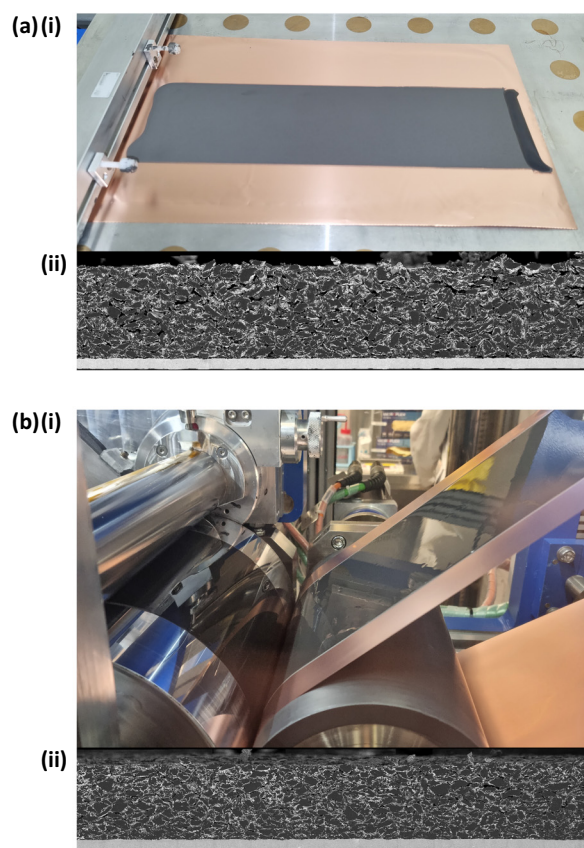


FIG. 3. (a) (i) Photograph of a drawdown coating of XG24C anode on a sheet of copper foil, (ii) SEM image of a 500- μm -wide cross section of the dried and calendared drawdown coated anode and (b) (i) photograph of the roll-to-roll scale up of the single-sided XG24C graphite anode on a Megtec pilot line coater, (ii) SEM image of a 500- μm -wide cross section of the dried and calendared XG24C anode produced on the pilot line.

electrodes, which should enhance their electrochemical cycling performance [26].

Following anode electrode manufacture and calendering, the electrochemical performance of the scaled-up XG24C anode was paired with a scaled-up NMC 811 cathode and validated in different cell configurations. Figure 4 displays the electrochemical performance validation of the scaled-up XG24C anode in full coin cells, three electrode PAT cells and single-layer pouch cells.

It is crucial to evaluate novel active materials in different cell configurations to fully comprehend cell-cycling behavior. For example, SEI formation and rate performance testing will be significantly influenced by the cell configuration. The differences in geometry, electrode stack pressure, and electrolyte distribution will directly impact ionic and electronic transport phenomena. This is illustrated in Fig. S6 of the Supplemental Material, which shows how cell impedance will differ for identical chemistries in different cell formats [17]. Additionally, each cell configuration will have its own inherent

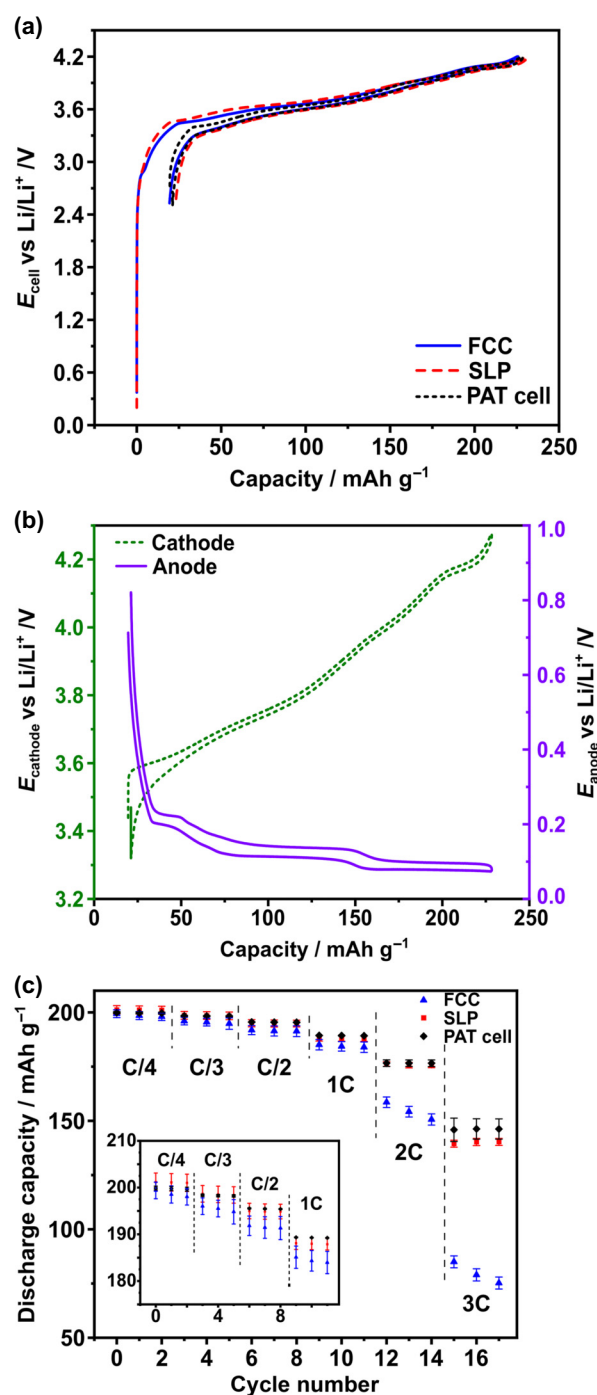


FIG. 4. Validating the performance of first formation cycle of XG24C graphite in (a) full coin cell (FCC), single-layer pouch cell (SLP), and three-electrode PAT cell, (b) separate anode and cathode from the PAT cell and (c) rate performance comparison using three different cell formats (inset – enlarged graph showing rate data from C/4 to 1C).

advantages and disadvantages that need to be considered during the material evaluation process. During the early stages of material evaluation, coin cells are highly useful as they can rapidly facilitate the initial assessment

of formation cycling performance, particularly when only a small quantity of material is available for assessment. However, their stack pressure is relatively uncontrolled and typically low, which can result in inhomogeneous electrode contact and non-uniform SEI formation [27]. This variability can amplify at high rates, as shown in Fig. 4(c), which displays severe capacity fade during the rate test at 3C for the full coin cells. This is due to the higher impedance of the coin cell, likely caused by poor contact and/or localized lithium plating. In contrast, the three-electrode PAT cells provided detailed insight into individual electrode behavior by isolating the anode and cathode potentials against a lithium reference electrode [see Fig. 4(b)], enabling the identification of polarization sources and overpotentials at each electrode. These cells also maintain a more defined electrode spacing and moderate stack pressure (approximately 150 kPa), allowing for more consistent electrolyte wetting and better interpretation of kinetic effects, especially under controlled formation protocols [28].

Single-layer pouch cells, by comparison, more closely emulate commercial cell conditions with well-defined electrode alignment, higher and more uniform stack pressure, and more realistic electrolyte volumes. These design features support more efficient SEI formation and enable better heat and mass transport at higher currents, contributing to improved rate capability and cycle stability. Moreover, the larger format reduces edge effects and provides a more representative assessment of scaling behavior, critical for translating lab-scale findings to commercial viability [29]. Therefore, discrepancies in rate performance and capacity retention between cell formats should be interpreted within the context of these structural and operational differences. While coin cells are useful for rapid material screening, PAT and pouch cells offer deeper mechanistic insight and practical performance benchmarks.

The single-layered pouch cells containing the scaled-up XG24C graphite anode were benchmarked against equivalent single-layer pouch cells containing a scaled-up V-H graphite anode. Both sets of single-layer pouch cells contained a scaled-up NMC 811 cathode. A photograph example of one of the finished single-layer pouch cell manufactured on the WMG pilot line is shown in Fig. 5(a). Figure 5(b) and 5(c) show the single-layer pouch cell formation and rate performance for NMC 811\graphite pouch cells containing either XG24C graphite anodes or commercial V-H graphite anodes.

NMC 811\graphite SLPs were also subjected to longer-term 1C/1C cycling to assess the stability of the different graphite anodes, as shown in Fig. 6.

Over 200 cycles, the SLPs containing the XG24C graphite anodes showed a higher capacity than the SLPs containing the commercial V-H graphite anodes. The XG24C graphite also showed comparable long-term

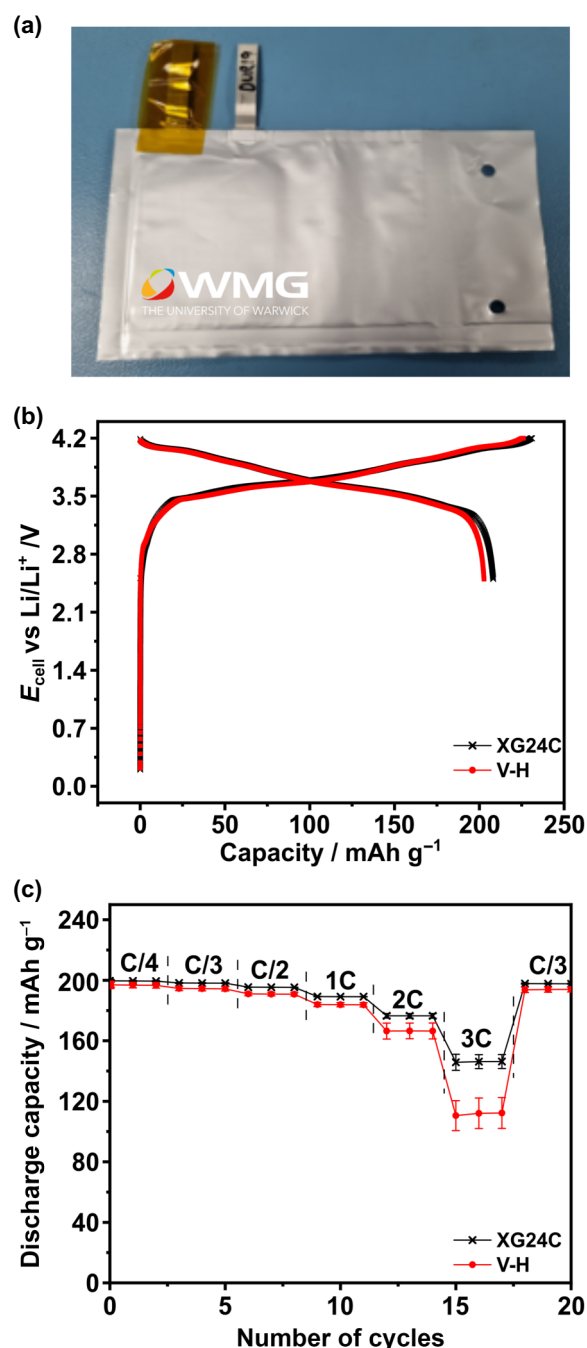


FIG. 5. (a) Photograph of a standard A7 pouch cell produced at WMG and the (b) first formation cycle and (c) discharge capacity rate performance of NMC 811\graphite single-layer pouch cells containing either XG24C graphite or V-H commercial graphite. The formation curve is representative of three pouch cells.

cycling stability to the commercially available V-H graphite. Overall, the final SLP benchmarking activity robustly demonstrated that the Durrans XG24C pitch-coated graphite was a viable candidate for further consideration, based on these like-for-like benchmark studies further enhancement is expected from more elaborate and specific cell format industry protocol alignment.

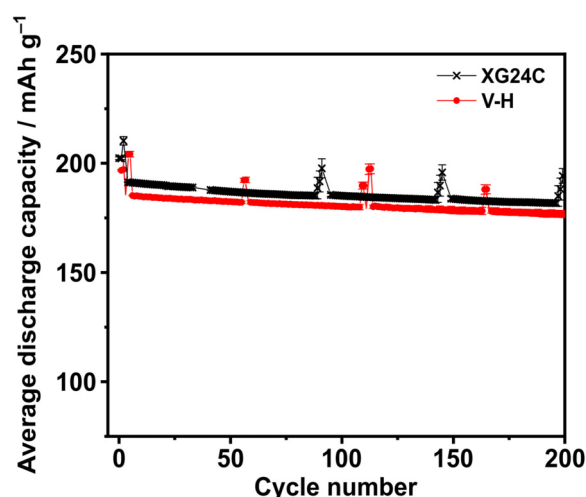


FIG. 6. Averaged 200 cycles, 1C/1C electrochemical charge/discharge cycling for SLPs containing an NMC811 cathode and XG24C (black) and V-H (red) graphite anode with regular capacity checks – indicated by the peaks and dips in the spectra.

IV. CONCLUSIONS

A deep understanding of lithium-ion cell formation is pivotal to advancing battery performance, as it governs critical interfacial dynamics and long-term electrode stability. This study highlights that subtle variations in material surface properties and formation protocols can significantly influence first-cycle loss, directly linking early-stage electrochemical behavior to both intrinsic material characteristics and processing choices. By systematically comparing a novel graphite material to a commercial benchmark across multiple cell formats—from coin cells to single-layer pouch cells—we isolated material-specific effects while maintaining consistent morphology, processing, and formation conditions. This rigorous approach establishes a robust electrochemical baseline, enabling meaningful, apples-to-apples comparisons across graphite sources. More importantly, it bridges the gap between lab-scale experimentation and commercial relevance, ensuring that promising materials are evaluated under conditions reflective of real-world manufacturing. By identifying critical electrochemical indicators early in the cell's lifecycle, our work provides a robust framework for high-throughput material screening to accelerate the translation of novel anode materials into scalable, high-performance battery systems.

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DATA AVAILABILITY

The data that support the findings of this article are not publicly available because they contain commercially sensitive information. The data are available from the authors upon reasonable request [30].

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- [30] See Figs. 1–6 of this paper.