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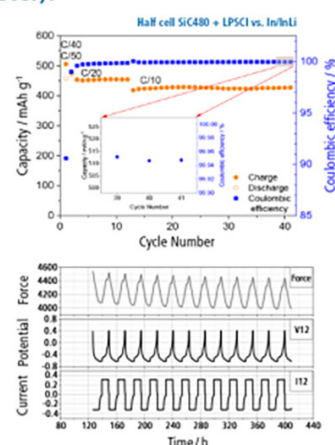
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# Large-Scale Electrosynthesis of Carbon Nano-Onions from CO<sub>2</sub> as a Potential Replacement for Carbon Black

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Graphite and carbon black are major global commodities whose production generates substantial CO<sub>2</sub> emissions. Carbon nano-onions (CNOs), consisting of concentric graphene spheres with enhanced properties due to their unique morphology, represent a promising next-generation material. This study presents a scalable, low-cost, carbon-negative electrosynthesis of CNOs directly from greenhouse CO<sub>2</sub>. Electrolysis is performed in molten lithium and lithium/strontium carbonate mixtures near their melting points, which suppresses transition metal-catalyzed Carbon NanoTube (CNT) formation, favoring uniform CNO growth. The method offers an efficient pathway for sustainable carbon nanomaterial production and CO<sub>2</sub> utilization.

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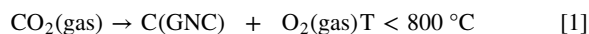
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Carbon Black (CB), a fine black carbon powder, and graphite are major global commodities with estimated values of \$29 billion and \$16 billion annually, respectively.<sup>1,2</sup> Their production contributes significantly to greenhouse gas emissions—approximately 44 million tonnes CO<sub>2</sub> for CB and at least 12 million tonnes for graphite annually.<sup>3,4</sup> CB is typically produced via partial thermal decomposition of hydrocarbons such as petroleum, coal tar, or natural gas generating soot and NO<sub>x</sub> in addition to CO<sub>2</sub>, and yielding a fine powder of unburned carbon.<sup>5</sup> Graphite is 60%–80% synthetic, derived from coke, with the remainder mined.<sup>6</sup> CB constitutes 20%–40% of vehicle tires,<sup>7</sup> primarily used in natural and synthetic rubbers, with additional roles as a black pigment<sup>8,9</sup> and as a conductive additive—comprising 5%–8% of Li-ion battery mass.<sup>10,11</sup>

Carbon black particles often exhibit a spheroidal morphology, as observed in Fig. 1A by SEM of CDH N-375 (CDH Laboratory Grade, 99.5% purity), which has a median particle size of 30 μm.<sup>12</sup> CB is used in both low-end applications, such as tires, and high-end uses, including Li-ion batteries.

Graphite morphology varies from thin flakes to nearly spherical particles. Figure 1B shows an SEM image of a spherical graphite particle (CDH Laboratory Grade, 99.5% purity) with a median size of 31 μm.<sup>12</sup> Major applications include refractories, pigments, batteries, foundries, and lubricants.<sup>15–20</sup> In Li-ion batteries, graphite, primarily used for lithium intercalation, accounts for ~28% of cell mass, comprising 95% of the anode material.<sup>13</sup>

Isotopic labeling with <sup>13</sup>CO<sub>2</sub> in 2015–16 enabled tracking of carbon transformation into CNTs via an unexpected transition metal nucleation pathway, during electrolysis in molten carbonate, termed C2CNT. CNTs, the first nanocarbon product observed, are 1D structures of cylindrical graphene walls (see Supplementary Information, SI).<sup>14,21,22</sup> Reported C2CNT CNT variants include magnetic,<sup>23</sup> long,<sup>24</sup> thin,<sup>25</sup> doped,<sup>26</sup> nano-bamboo, nano-pearl,<sup>27</sup> and macro-assemblies.<sup>22</sup> Adjusting electrolysis conditions has yielded additional 0D, 2D, and 3D Graphene-based NanoCarbon (GNC) allotropes (SI).<sup>27–30</sup> The process has been scaled for decarbonization, enabling both Direct Air Capture (DAC) and Carbon Capture and Utilization (CCU).<sup>31,32</sup> CO<sub>2</sub> electroreduction in molten Li<sub>2</sub>CO<sub>3</sub> occurs at 0.8–1.4 V.<sup>33–36</sup> The overall conversion is:



A distinct graphene-based nanocarbon (GNC) is the 0D allotrope carbon nano-onion (CNO), composed of concentric graphene shells, typically synthesized via nanodiamond pyrolysis or other methods.<sup>37</sup> CNOs have also been produced through the C2CNT process,<sup>30,31,38</sup> by inhibiting transition metal nucleation to suppress CNT formation, and were extensively analyzed. As shown in Fig. 1 (panels C1, C2) and previously reported,<sup>30,31,38</sup> C2CNT-derived CNOs are uniform spheroidal aggregates, 0.5–2 μm in diameter, each comprising clustered 30–50 nm CNOs (panels C3, C4). Panels C5–C7 show CNO TEM contrast, low-defect Raman spectra (I<sub>D</sub>/I<sub>G</sub>), and a high-crystallinity 23° XRD peak. The original study examined small-scale CNO electrosynthesis: after 5 min, ill-defined spheroids form; by 30 min, ~30 nm radius, CNOs form comprised of concentric 0.35 nm spaced graphene shells (C4) appear, loosely packed; at 90 min, the CNOs agglomerate into densely packed CNO clusters.<sup>30</sup>

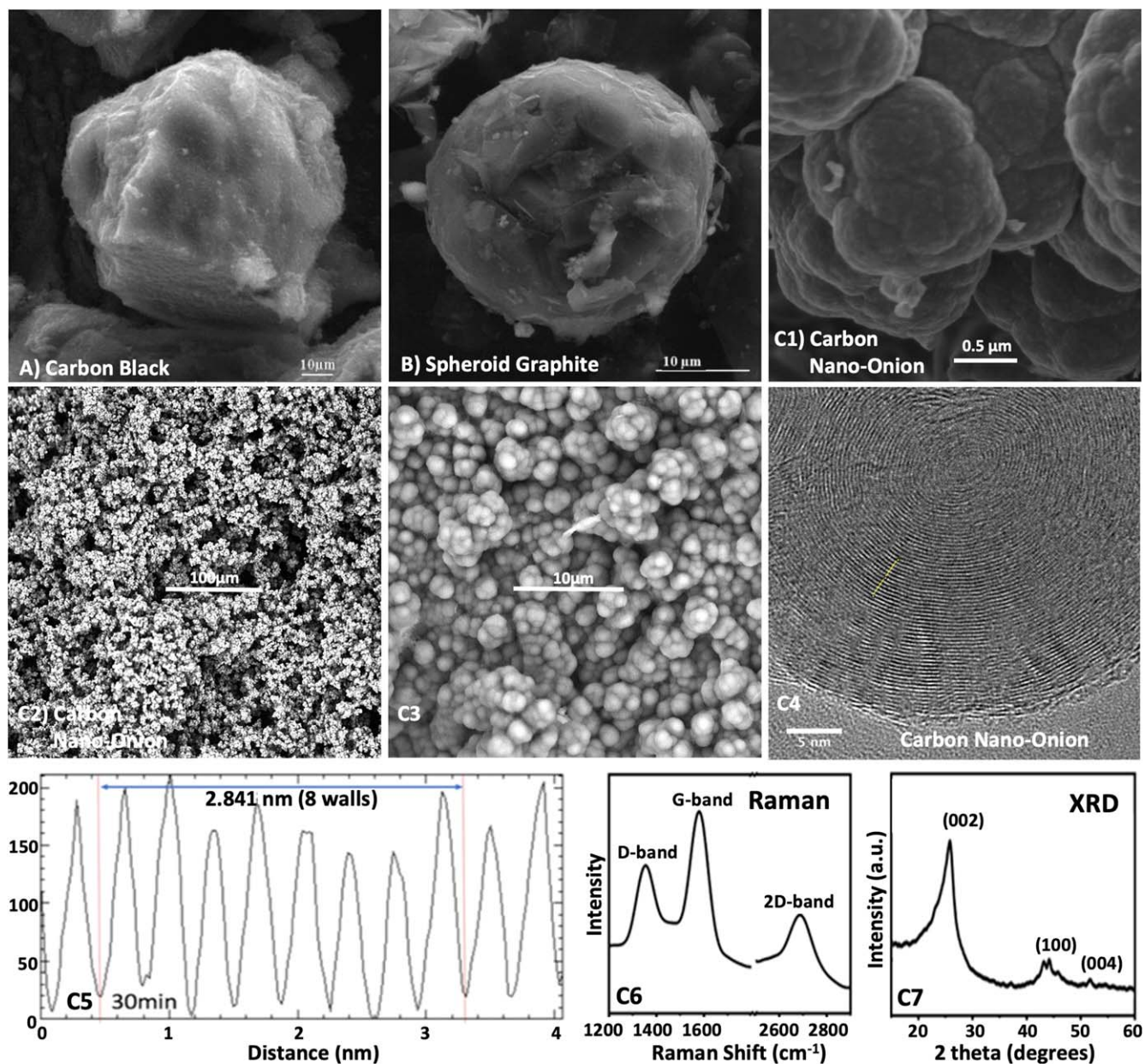
Earlier C2CNT-based CNO synthesis was achieved by limiting transition metal availability, either through oxide additions that reduce metal oxide solubility in molten carbonate, or by using noble metal anodes to prevent metal release. This study introduces a simpler approach: using standard electrolytes and stainless-steel anodes from large-scale CNT production, with electrolysis conducted near the electrolyte melting point to promote CNO formation.

## Experimental

Electrolyses were performed in lithium or lithium/strontium carbonate electrolytes using vertical planar Muntz brass cathodes and 304 stainless steel (304SS) anodes, which form the inner walls of the carbon pot. The full-scale molten carbonate electrolysis kiln (Fig. 2), operating on 5% CO<sub>2</sub> flue gas from the Shepard Energy Centre (Calgary, Canada), converts 100 tonnes of CO<sub>2</sub> per year into 25 tonnes of graphene products. Details of the large-scale synthesis are delineated in references.<sup>38–40</sup> Although typically used for CNT production, a simple adjustment of electrolytic conditions enables CNO formation.

Li<sub>2</sub>CO<sub>3</sub> (>99.5%, mp 723 °C; Shanghai Seasongreen Chemical Co., purity 99.8%) was used as received. SrCO<sub>3</sub> (99.4%, Shendong Zhi Chemical Co.) and SrO (99%, Chemsavers) were also used as electrolyte components. A lower-purity SrCO<sub>3</sub> (98.6%, Hengshui Haoye Co.), containing 0.8% BaCO<sub>3</sub> and 0.2% CaCO<sub>3</sub>, exhibited comparable solubility. Muntz brass (60% Cu, 40% Zn; 280 brass) was sourced from onlinemetals.com and Marmetal Industries. Scanning electron microscopy (SEM) was performed using a PHENOM Pro-X microscope; Transmission Electron Microscopy

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**Figure 1.** SEM of (A) Carbon Black and (B) Spheroid Graphite from Ref. 12. (C1 to C4) SEM of Carbon Nano-Onions from Ref. 14. The CNOs are synthesized by molten carbonate electrolytic splitting of  $\text{CO}_2$ . (C5-C6) CNO TEM intensity, Raman and XRD.

(TEM) with an FEI Teneo Talos F200X TEM; with an X-ray Diffraction (XRD): Rigaku D=Max 2200 XRD diffractometer, and Thermogravimetric Analysis (TGA): were assessed under air using a Perkin Elmer STA6000 heating from room temperature to  $900\text{ }^\circ\text{C}$  at  $10\text{ }^\circ\text{C min}^{-1}$ .

## Results and Discussion

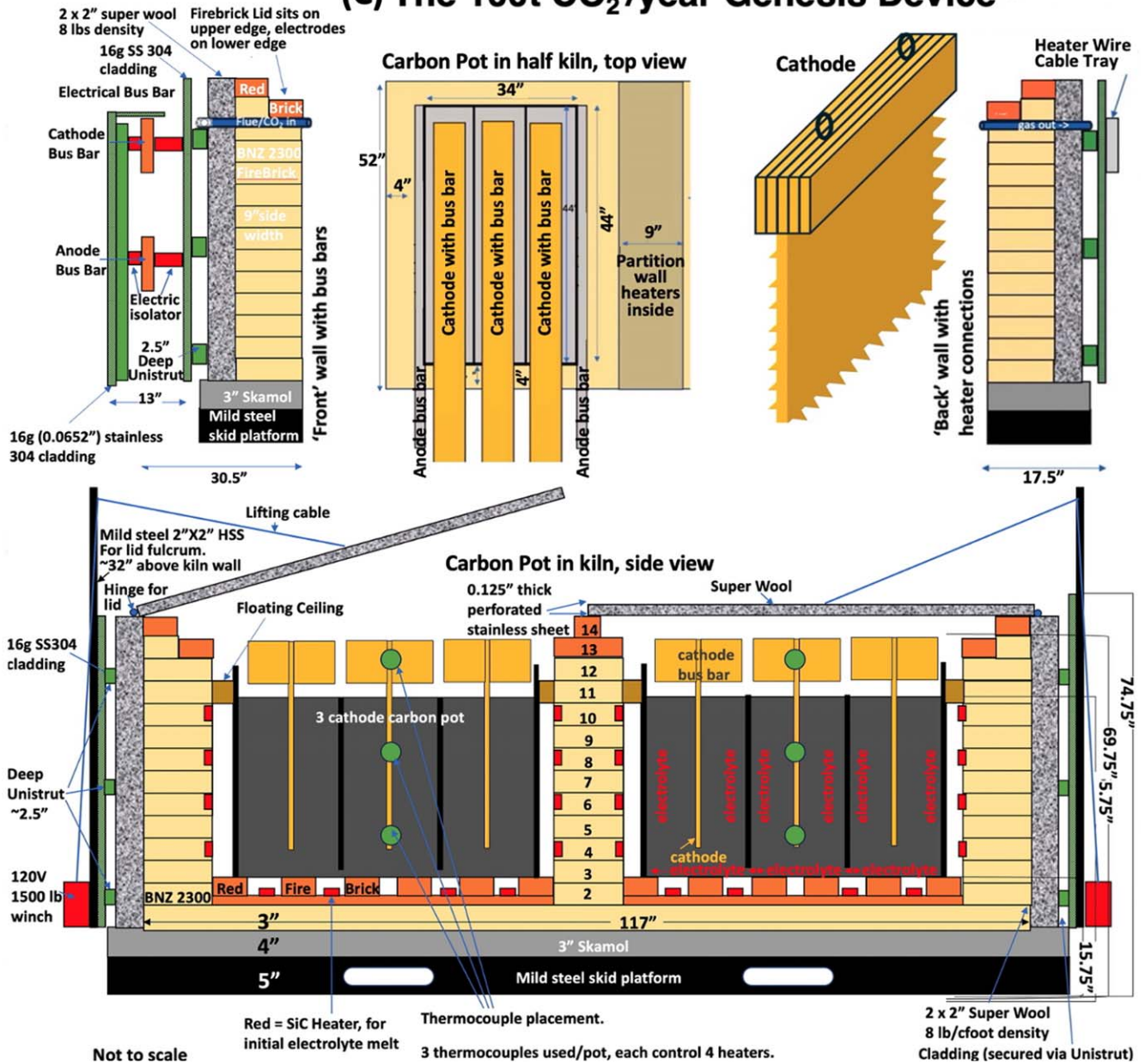
In molten carbonate CNT electrosynthesis, transition metals such as Fe and Ni typically originate from low-level dissolution of anodes like Inconel, Nichrome, or stainless steel, or added related electrolyte salts. These metal cations are reduced at the cathode, forming nucleation sites. We previously showed that suppressing metal availability, using noble metal anodes or adding alkali/alkaline earth oxides, favors CNO over CNT formation, and extensively characterized the CNOs.<sup>30,31,38</sup> The bulk, as characterized by TEM, XRD, Raman, and TEM spatial intensities are 30–50 nm CNOs aggregated into tightly packed 0.5–2  $\mu\text{m}$  multi-CNO clusters. The

CNOs are low defect concentric 0.35 nm spaced graphene spheres. Extended duration produces more, tightly packed CNOs, rather than significantly larger CNOs.<sup>30</sup> Here, we form the same CNOs through a simpler, large-scale compatible C2CNT electrosynthesis.

We hypothesize that reducing the electrolyte temperature near its melting point can similarly limit the availability of transition metal cations and thereby promote the synthesis of carbon nano-onions (CNOs). Specifically, lowering the temperature decreases the solubility of transition metals and, near the freezing point, significantly increases the viscosity of the electrolyte.<sup>41–43</sup> Both effects contribute to reduced activity and mobility of transition metal species. This is expected to lower the rate of transition metal cation reduction at the cathode relative to  $\text{CO}_2$  reduction.  $\text{CO}_2$  reduction is also less constrained by mass transport under these conditions. The latter process is in equilibrium with the abundant carbonate ion in the system, as described by the reaction:  $\text{CO}_2 + \text{O}^{2-} \rightleftharpoons \text{CO}_3^{2-}$ , followed by  $\text{CO}_3^{2-} \rightarrow \text{C}_{\text{GNC}} + \text{O}_2$ . The results of this study support the hypothesis, demonstrating that CNOs can be electrosynthesized



(C) The 100t CO<sub>2</sub>/year Genesis Device<sup>®</sup>



**Figure 2.** Industrial carbon capture via molten carbonate electrolysis was demonstrated using 5% CO<sub>2</sub> flue gas from the Shepard Natural Gas Power Plant in Calgary, Canada. (A) The Genesis Device<sup>®</sup> kiln conducts large-scale molten carbonate electrolysis modules designed to convert 100t CO<sub>2</sub>/year. (B1–B2) Cathode during removal from the electrolyte and after cooling. (C) Front, side, and back views of a 100t CO<sub>2</sub>/year Genesis Device<sup>®</sup> module, the carbon pot and saw-tooth shaped cathode, which ensures uniform, edge-growth deposition.

from CO<sub>2</sub> near the electrolyte's melting point without the need for noble metal anodes, using simple and non-corrosive electrolytes.

Figure 3A illustrates the high solubility and melting point depression of SrCO<sub>3</sub> (mp 1494 °C) in molten Li<sub>2</sub>CO<sub>3</sub>. The melting point decreases from 723 °C (pure Li<sub>2</sub>CO<sub>3</sub>) to 690 °C at the eutectic (40 wt% SrCO<sub>3</sub>), then rises to 800 °C at 65 wt%. Electrolysis above 800 °C becomes less efficient due to increased CO evolution, which dominates by 950 °C.<sup>35</sup> Mixed (Sr/Li<sub>2</sub>)CO<sub>3</sub> melts are effective for CO<sub>2</sub> electrolysis to CNTs and offer cost advantages, as SrCO<sub>3</sub> is significantly less expensive than Li<sub>2</sub>CO<sub>3</sub>.<sup>38</sup> Figures 3B–3C show a >1 m<sup>2</sup> Muntz Brass cathode coated with carbanogel (GNC/electrolyte mixture). Post-electrolysis, hot-pressing removes excess electrolyte,<sup>40</sup> followed by GNC washing.<sup>38</sup>

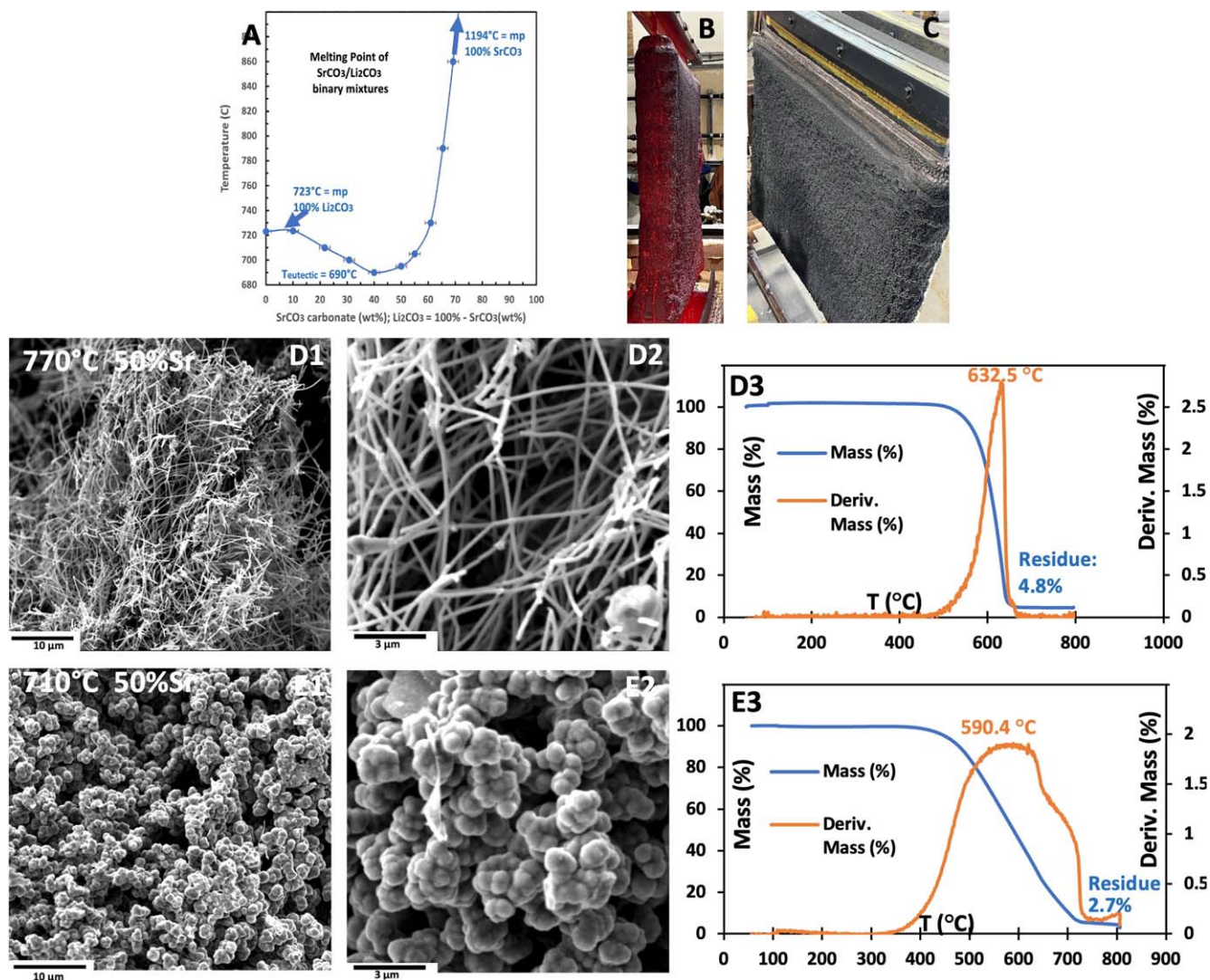
Figures SEM 3D1–2 shows CNT formation at 770 °C in 50/50 wt% (Sr/Li<sub>2</sub>)CO<sub>3</sub>. Lowering the temperature to 710 °C yields uniform 0.5–2 μm clusters composed of 30–50 nm carbon nano-onions (CNOs), SEM 3E1-E2, with high purity (>>95%), with minor platelet impurities observed at higher magnification (D2). TGAs are compared of the CNT (D3) and CNO (E3) product.

As shown in Fig. 4, lowering the electrolyte temperature near the melting point promotes CNO over CNT formation for binary Sr/Li electrolytes and pure Li<sub>2</sub>CO<sub>3</sub>. 770 °C pure Li<sub>2</sub>CO<sub>3</sub> electrolysis yields conventional CNTs (SEMs A1,A2). However, at 730 °C just above the melting point, produces high-purity 0.5–3 μm clusters of

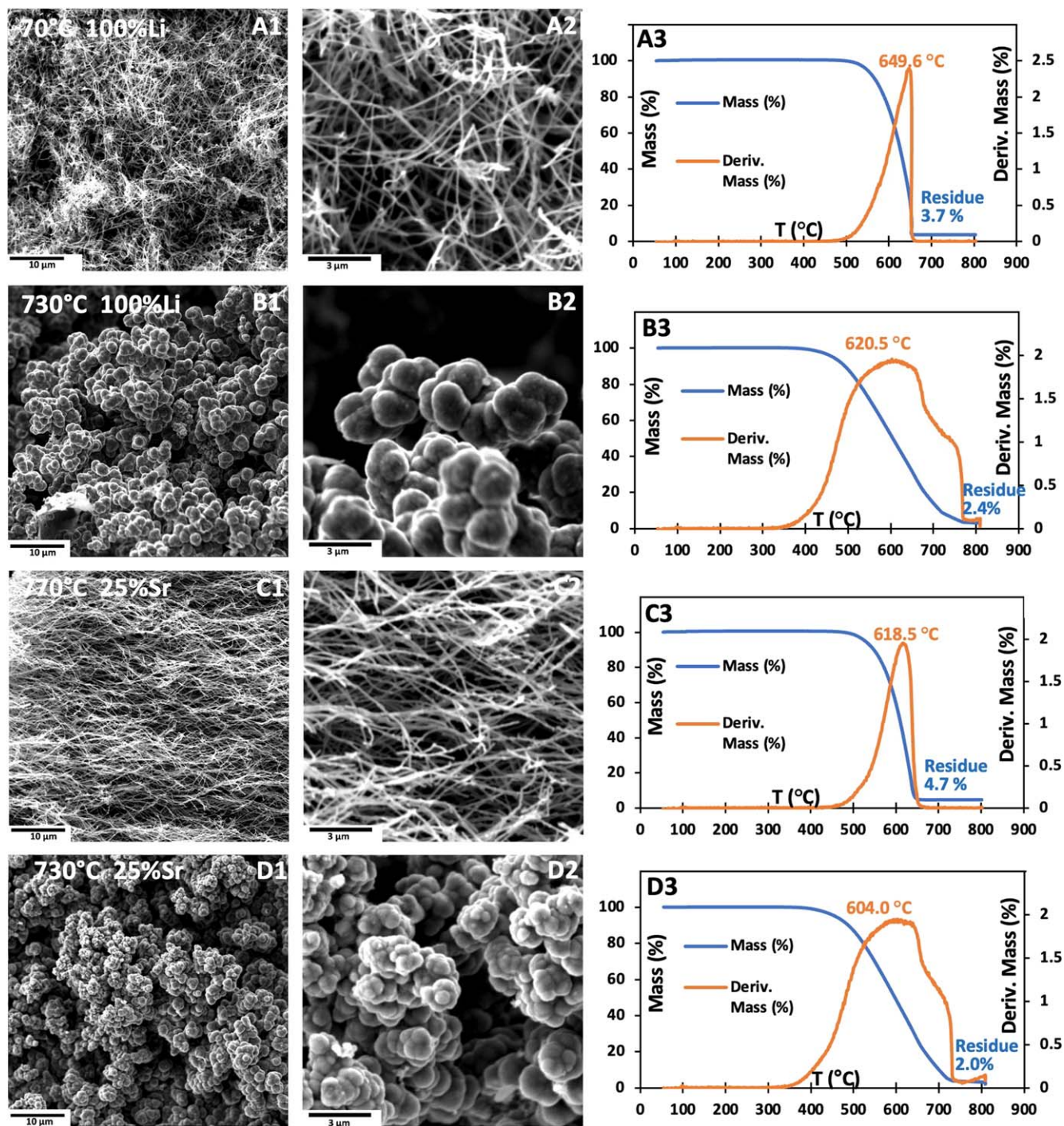
0.03–0.05 μm CNOs (SEMs B1, B2). A similar, product transition occurs to CNOs occurs in 25/75% (Sr/Li<sub>2</sub>)CO<sub>3</sub>, when the temperature is lowered from 770 to 730 °C (SEMs C1,C2 compared to D1 and D2).

Comparison of figure panels 3E3, 4B3, 43, with 3D3, 4A3, 4C3 shows that CNOs exhibit distinctive, broad high-temperature shoulder peaks, in contrast to the sharp TGA peaks of CNTs. We hypothesize that CNTs oxidize at a single, uniform rate, while CNO aggregates exhibit two distinct oxidation rates: an intra-particle rate for individual CNOs and an inter-particle rate influenced by solid-solid interface (SSI) defects and graphene layer discontinuities between packed CNOs.

GNCs, valued for their superior strength, durability, and conductivity, have been explored as substitutes for CB in tires and graphite in refractories.<sup>44–46</sup> Commercial adoption of CNOs has been limited by the high cost of traditional synthesis methods.<sup>46–48</sup> Molten carbonate synthesis from CO<sub>2</sub> yields GNCs at lower cost than graphite or premium CBs.<sup>38</sup> As shown in Fig. 1, CNOs resemble CB and spheroidal graphite, comprising concentric graphene shells that enhance graphene's properties, optimizing strength, tribology, surface area, and durability.<sup>49–54</sup> Boron doping via borate addition to the C2CNT electrolyte increases GNC, such as CNT, conductivity by an order of magnitude,<sup>26</sup> and application of other GNCs, such as CNTs in composites and buckypapers have also been explored.<sup>55–57</sup>



**Figure 3.** (A) Sr/Li<sub>2</sub>CO<sub>3</sub> mixtures melting points; (B & C) 11,000 cm<sup>2</sup> Muntz Brass electrolysis product on the cathode, upon lifting and after cooling. SEM of washed product of (D) 770 °C or (E) 710 °C 50/50 wt% Sr/Li<sub>2</sub>CO<sub>3</sub> electrolysis at  $J = 0.6 \text{ A cm}^{-2}$ . SEM magnifications are D1 & E1: 5000x, D2 & E4: 20,000x, E2 & E3 6200x.



**Figure 4.** SEM of washed product of (A,C) 770 °C or (B,D) 730 °C 50/50 wt% Sr/Li<sub>2</sub>CO<sub>3</sub> electrolysis at  $J = 0.6 \text{ A cm}^{-2}$  in either (A,B) 100 Li<sub>2</sub>CO<sub>3</sub> or (C,D) 25/75 wt% Sr/Li<sub>2</sub>CO<sub>3</sub> electrolyte. SEM magnifications are A1, B1, C1 & D1: 5000x, A2, B2, C2, D2: 20,000x.

High surface area is favored for CB applications. As shown in Figs. 1, 3, and 4, CNO clusters are 10 times smaller than the  $\sim 30 \mu\text{m}$  graphite and carbon black shown in Fig. 1, and individual CNOs are 1000 times smaller, potentially yielding a high specific surface area. Surface area characterization is warranted and may be improved through cluster deagglomeration and chemical or mechanical treatments to promote surface roughening and cavitation. A key aspect of many applications is particle dispersion. CNOs have been reported as effectively dispersed in aqueous media,<sup>58</sup> by acid treatment, and in non-aqueous polar solvents by alkylation.<sup>59</sup> Individual, not densely packed CNOs may be grown by short term (e.g. <30 min), low current density ( $\leq 0.2 \text{ A cm}^{-2}$ ) C2CNT electrolysis.<sup>30</sup> Disaggregating

clusters into single CNOs could further enhance surface area and also warrants investigation. Methods that have significantly separated or purified similar CNT particles include ball milling, thermal, microwave, acid, and KOH treatments, as well as surface modification, sonication, and exfoliation.<sup>60–62</sup>

## Conclusions

This study demonstrates a scalable, low-cost method for synthesizing CNOs directly from CO<sub>2</sub> via molten carbonate electrolysis. Lowering the electrolysis temperature near the electrolyte's melting point enables the production of high-purity CNOs without noble

metal anodes or complex electrolytes. CNOs exhibit broad high-temperature TGA peaks, unlike the sharp peaks of CNTs, suggesting two oxidation rates: intra-particle oxidation of individual CNOs and inter-cluster oxidation driven by solid-solid CNO interface defects and graphene discontinuities.

Due to their favorable morphology, durability, tribology, and conductivity, CNOs offer a sustainable alternative to conventional carbon black and graphite in applications such as batteries, tires, and refractories, potentially reducing global CO<sub>2</sub> emissions while meeting high-performance material demands.

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