

Supplementary Information accompanying:

Direct Air Capture (DAC): Molten carbonate direct transformation of airborne CO₂ to durable, useful carbon nanotubes and nano-onions

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Elevated levels of atmospheric CO₂ are the primary driver of global warming. Historically, atmospheric CO₂ concentration fluctuated within a range of $235 \pm \sim 50$ ppm over the past several hundred thousand years, until 1850. Currently, it stands at 426 ppm and continues to increase annually, leading to widespread climate disturbances, habitat degradation, and species extinction.¹⁻⁴ The inherent chemical stability of CO₂ poses a significant challenge to its removal by conversion into a non-greenhouse material, a subject explored in our US NSF workshop on Chemical Recycling and Utilization of CO₂.⁵ However, overcoming this stability of CO₂ and utilizing it as a carbon-negative precursor for the production of valuable products provides an incentive for the reduction of this greenhouse gas.

Greenhouse gas removal from the atmosphere is necessary to achieve net-zero emissions and limit climate change.^{6,7} Today's chemical Direct Air Capture (DAC) decarbonization technologies⁷⁻¹¹ require an active CO₂ concentration system, often on sorbents such as solvent phase or membrane fixed amine binding¹²⁻¹⁸ or based on aqueous phase or solid powder lime reactions.¹⁹⁻²⁴ The processes involved are energy-intensive and have a significant carbon footprint, and they represent just the initial step in Carbon Capture and Storage (CCS) or Carbon Capture, Utilization, and Storage (CCUS) since they focus on concentrating carbon dioxide rather than storing it. In contrast, this demonstration presents an efficient chemical Direct Air Capture (DAC) process that eliminates the need for active CO₂ concentration.

Carbon nanotubes (CNTs) possess the highest tensile strength ever recorded (93,900 MPa)^{25,26} and feature excellent thermal conductivity, high charge storage, flexibility, and catalytic properties. As described in the SI, they enhance structural materials like cement and steel,²⁷ and are used in medical²⁸ and electrochemical applications,²⁹ electronics, batteries, and

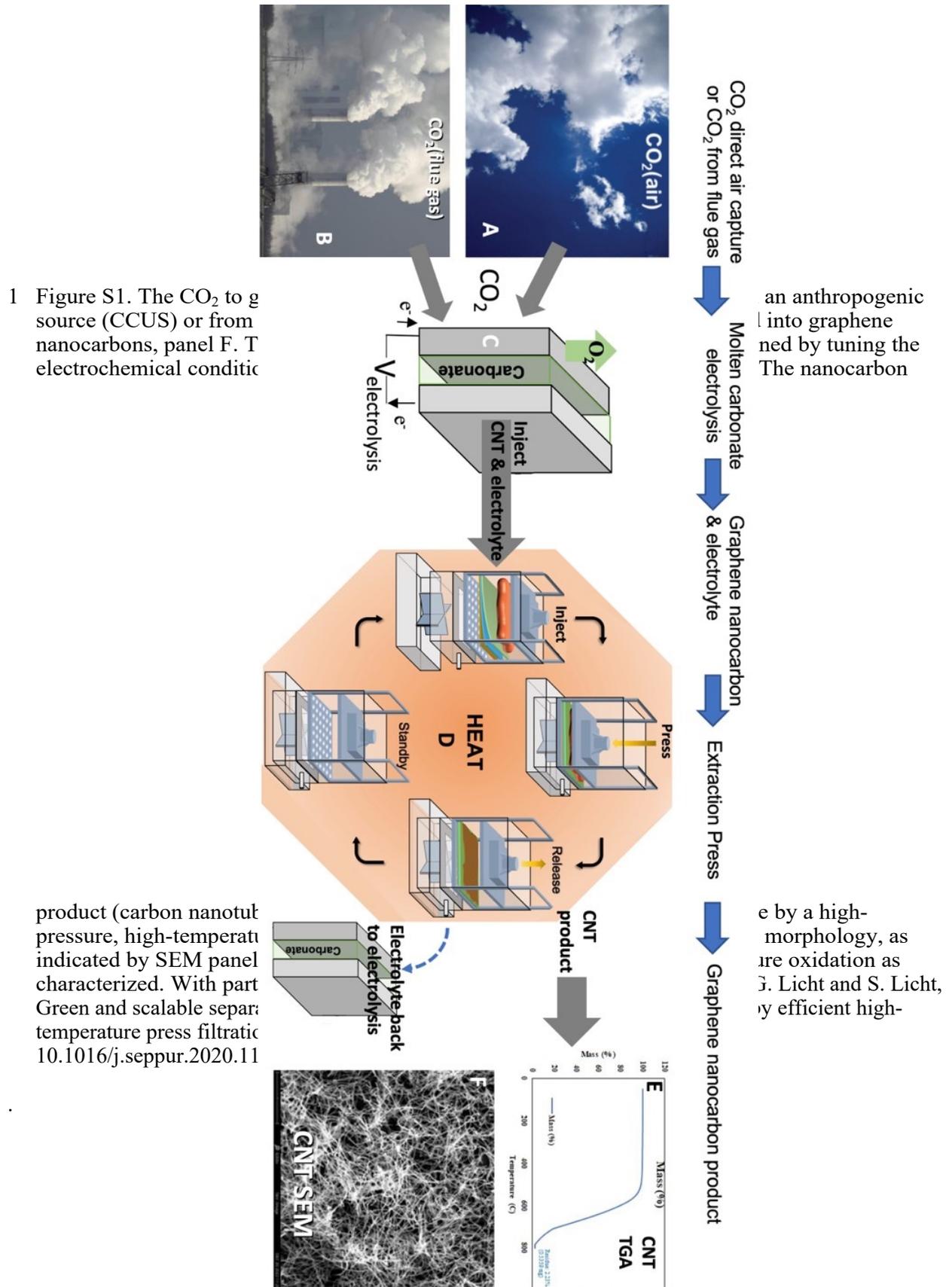
supercapacitors^{30,31} and are used for sensing,³²⁻³⁴ plastics,³⁵⁻³⁹ textiles,⁴⁰ hydrogen storage,⁴¹ and water treatment.^{42,43}

The high-temperature molten carbonate electrolytic splitting of CO₂ into carbon and oxygen as a GHG mitigation strategy was introduced in 2009-2010.^{44,45} In 2015, it was shown that the growth of transition metal nuclei during molten carbonate electrolysis process leads directly to the conversion of CO₂ into readily sequestered, stable, pure graphene nanocarbons, including carbon nanofibers and CNTs.⁴⁶⁻⁵¹ Graphite is an analogous macroscopic form of layered graphene, and as a mineral graphite has an established geologic (hundreds of millions of years) lifetime. The type of GNC produced depends on the electrochemical conditions and delineated in the SI.^{46,52} This method transforms CO₂ into valuable graphene-stabilized carbon products, offering a promising climate change mitigation approach. The CCUS molten carbonate electrolysis process for GNC production has evolved into a sophisticated technology now protected by numerous patents.⁵³ By adjusting CO₂ electrolysis conditions, specific GNCs can be produced—carbon nano-onions at high oxide conditions around 725°C and CNTs at 750-770°C.^{46,55-62}

Research has also explored alternatives, such as sodium carbonate mixtures and lower electrolysis temperatures, which can produce 3D symmetry graphene scaffolds.⁶³ Electrolysis current densities from 0.03 to 0.6 A cm⁻² affect GNC growth, with higher densities favouring helical CNTs.⁶⁴⁻⁶⁵ The energy required ranges from 0.8 to 2 volts, and using renewable energy can further reduce the carbon footprint.⁶⁶⁻⁷² Various GNC allotropes, including carbon nano-bamboo and carbon nano-pearl, can be produced by adjusting electrode and electrolyte conditions.⁷³⁻⁷⁴ GNCs form directly on the cathode and are separated from the molten electrolyte through high-temperature filtration.^{75,76}

The CO₂ to nanocarbon process, including electrolyte separation and return to the electrolysis chamber, and extraction of the pure GNC product is illustrated Figure S1. As illustrated in Figure 1, during electrolysis CO₂ either sourced directly from the air or industrial emissions are transformed to GNCs by electrolysis in molten carbonates. The CO₂ is split into C and O₂ with a GNC-electrolyte matrix growing at the electrolysis cathode. This

nanocarbon/carbonate electrolyte mix has been termed a carbanogel and is refined through the separation of the electrolyte.



1 Figure S1. The CO_2 to graphene nanocarbon source (CCUS) or from nanocarbons, panel F. The electrochemical conditions are indicated by SEM panel F.

product (carbon nanotubes) under pressure, high-temperature conditions, as indicated by SEM panel F. The product is characterized by SEM panel F. With part of the product, Green and scalable separation of the nanocarbon at high temperature press filtration (DOI: 10.1016/j.seppur.2020.11)

The high electrical conductivity character of the graphene nano-allotropes supports continuous growth during the CO₂ molten electrolysis at low electrolysis voltage. This cathode product grows as an interconnected matrix with electrolyte in the matrix pores. This matrix containing carbonate electrolyte has been termed a carbanogel. Some of the electrolyte in this matrix is rather loosely bound. For example, a post-electrolysis cathode lifted out of the molten electrolyte can release over 30% of the bound electrolyte by gravitational drip.

Figure S2 illustrates larger vertical presses that have been scaled up, which include the transfer of applied pressure to the pressing chamber using a hydraulic ram, as described previously.⁷⁶ In Figure S2A, there's a cross-sectional depiction of an intermediate scaled-up carbanogel electrolyte extraction unit, detailing the plunger, filter screen platform, and electrolyte exit chamber.⁷⁶ Figure S2B shows a larger carbanogel electrolyte extraction unit in operation, capable of pressing up to 0.25 tonnes of carbanogel. Presses in the unit with 50 kg carbanogel have already achieved over 99% electrolyte extraction efficiency.

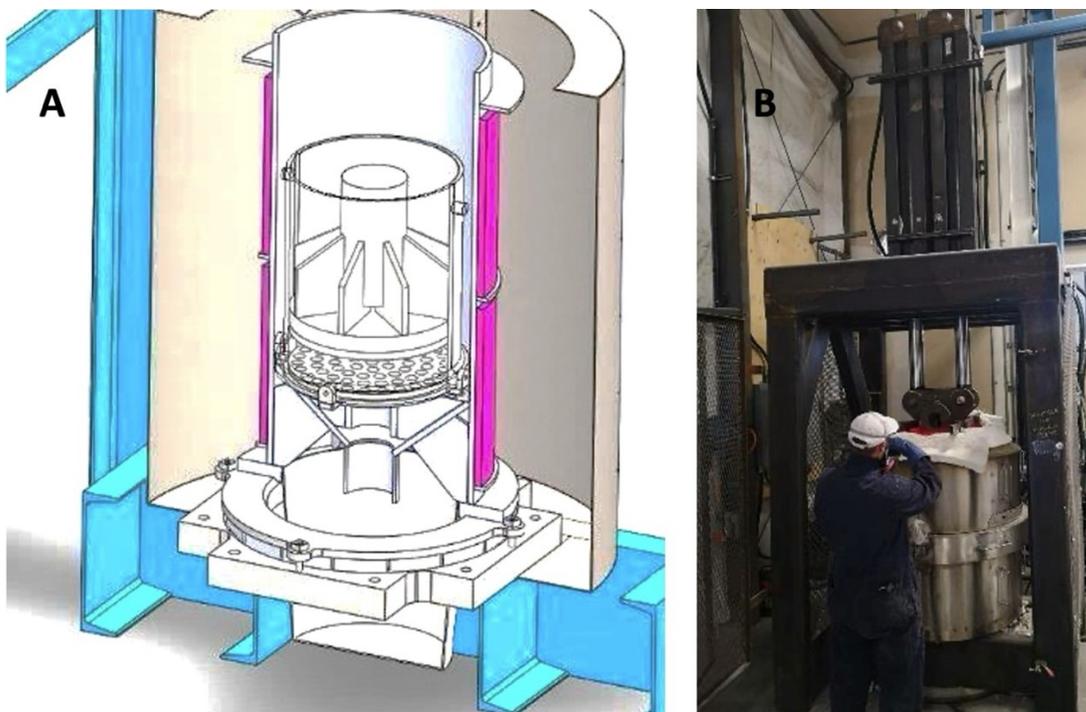


Figure S2. Scaled-up carbanogel electrolyte extraction units. A) Cross-sectional illustration of a mid-scaled extraction unit illustrating the plunger, the filter screen platform and the electrolyte exit chamber. B) Larger scaled carbanogel electrolyte extraction unit in action. From open access paper Licht, K. Hofstetter, S. Licht, Separation of molten electrolyte from the graphene nanocarbon product subsequent to electrolytic CO₂ capture. *Decarbon 4* (2024) 100044. <https://doi.org/10.1016/j.decarb.2024.100044>.

Control of the electrode and electrolyte composition, and CO₂ electrolysis splitting temperature and current density tunes the decarbonization process to form a range of high purity graphene nanocarbon products, including carbon nanotubes. Typical SEM, TEM and HAADF (High Angle Annular Dark-Field TEM) elemental analysis imaging of the CNTs are presented in Figure S3,⁵⁹ and of the carbon nano-onions in S4, and have been extensively detailed.⁵⁴

Control of the CO₂ electrolysis conditions is used to tune the specific GNC generated by control of the temperature, current density, and the composition of the electrolyte.^{59,74} For example, a lower temperature (725°C) is typically used in the electrolytic growth of carbon nano-onions, while higher temperature (750 to 770°C) is used in the electrolytic growth of carbon nanotubes. Lithium carbonate, a typical electrolyte, has a melting point of 723°C. Binary lithium carbonate mixtures have a lower melting point. A high sodium carbonate content in a mixed sodium/lithium carbonate electrolyte and a lower electrolysis temperature (670°C) drive the formation of a graphene scaffold nanocarbon product formation. Applied electrolysis current densities generally range from 0.03 to 0.6 A cm⁻². High current density (0.6 A cm⁻² or over) is

one of the principal conditions driving the formation of fascinating helical, rather than straight, carbon nanotubes.

Electrode (and electrolyte additive) composition variation has been used to grow other GNC allotropes from CO₂. These include carbon nanobamboo, carbon nanopearl, graphene from nanocarbon platelets, carbon nanofiber, carbon nanobelt, carbon nanotree, and other specific carbon allotrope morphologies. SEM of a range of these GNC products is presented in Fig. S5, and XRD and Raman spectra of the products are presented in Figs. S6 and S7 as previously detailed.⁷⁴ The solid graphene nanocarbon product from CO₂ grows as a matrix directly on the cathode. Under constant current electrolysis conditions, the product formation is continuous, and the growth occurs in the direction towards the anode.

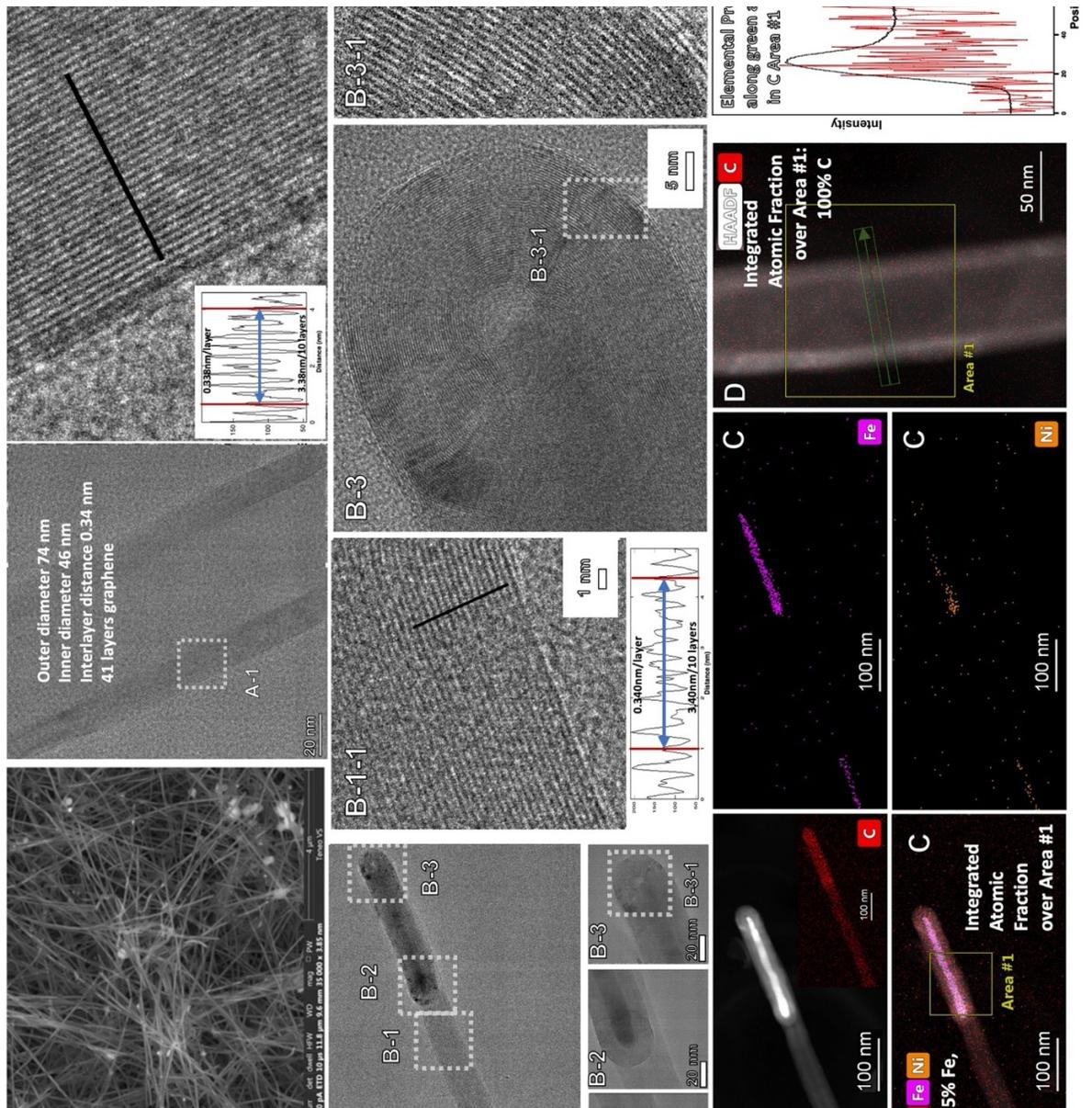


Figure S3. SEM TEM and HAADF of the synthesis product of high purity, high yield carbon nanotubes by electrolytic splitting of CO_2 in 770°C Li_2CO_3 . The SEM has a scale bar of $5\ \mu\text{m}$. Panels B are TEM with scale bars decreasing from 100, 20, 5 and 1 nm. Bottom rows panels C are HAADF elemental analyses with scale bars decreasing from 100 to 50 nm, and in the bottom right a HAADF elemental carbon profile analysis of the carbon nanotube cross section. Modified from open access paper X. Liu, G. Licht, S. Licht, Controlled Transition Metal Nucleated Growth of Carbon Nanotubes by Molten Electrolysis of CO_2 Catalysts 12 (2022) 137. <https://doi.org/10.3390/catal12020137>.

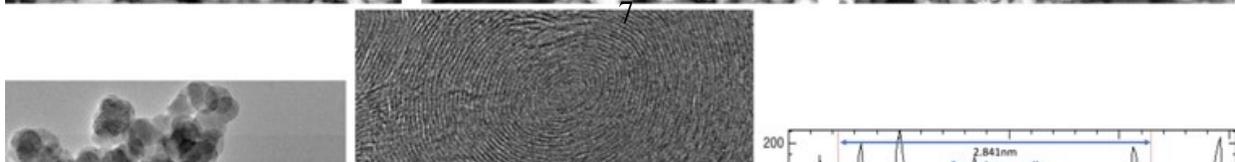
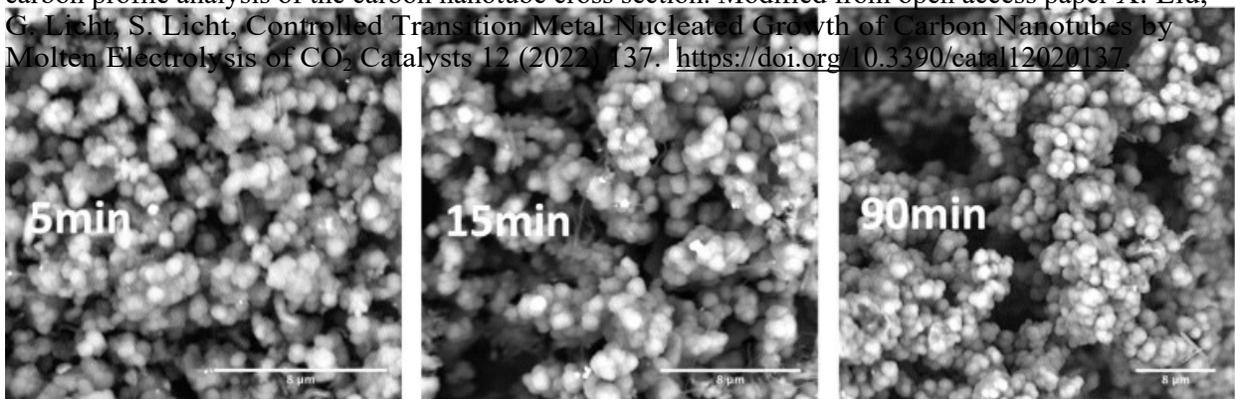


Figure S4. Top: SEM comparison of the graphene nanocarbon synthesized as a function of electrolysis duration without added nickel (compared to Fig. 3 with added nickel) in a 770°C Li_2CO_3 containing 5.9 m Li_2O . When nickel is not added, the initial spheroid product that is evident becomes a distinct carbon nano-onion morphology. Each product contains a high yield of uniform pure carbon spheroids. Bottom: TEM of CNOs obtained with 30 minutes of molten carbon carbonate electrolysis and delineating the concentric graphene sphere morphology of the CNOs..

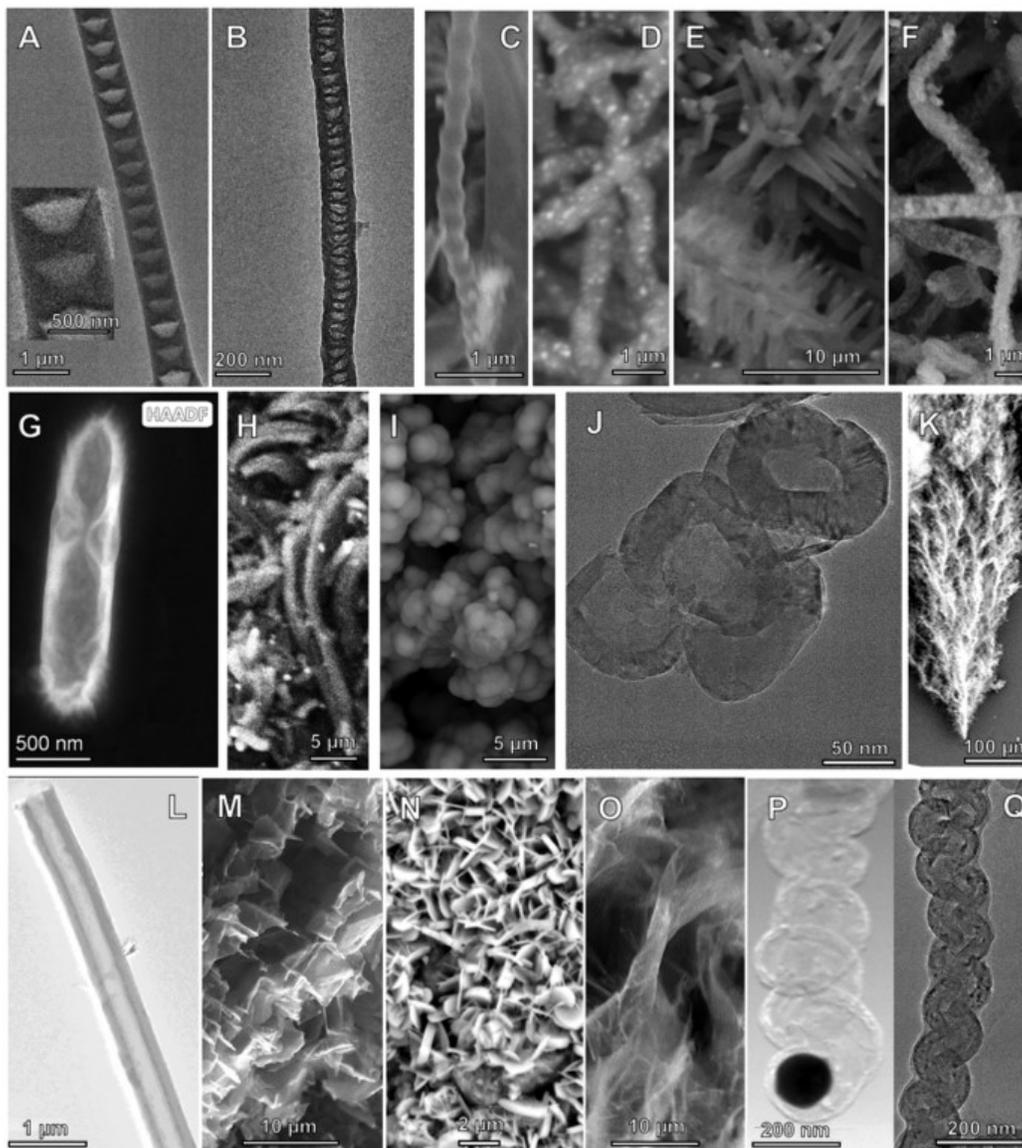


Figure S5. SEM of nanocarbon allotropes synthesized by the electrolytic splitting of CO_2 in molten carbonate. Top row (from A to F) conical CNF, nano-bamboo, nano-pearl, Ni coated CNT, nano-flower, nano-dragon. Middle row (from G to K): nano-rod, nano-belt, nano-onion, hollow nano-onion, and nano-tree. Bottom row (from L to Q) Carbon nanotube, nano-scaffold (ref. 50), nano-platelet, graphene, nanohelices. Modified from open access paper X. Liu, G. Licht, X. Wang, S. Licht, Controlled Growth of Unusual Nanocarbon Allotropes by Molten Electrolysis of CO_2 . *Catalysts* 12 (2022) 137. <https://doi.org/10.3390/catal12020125>.

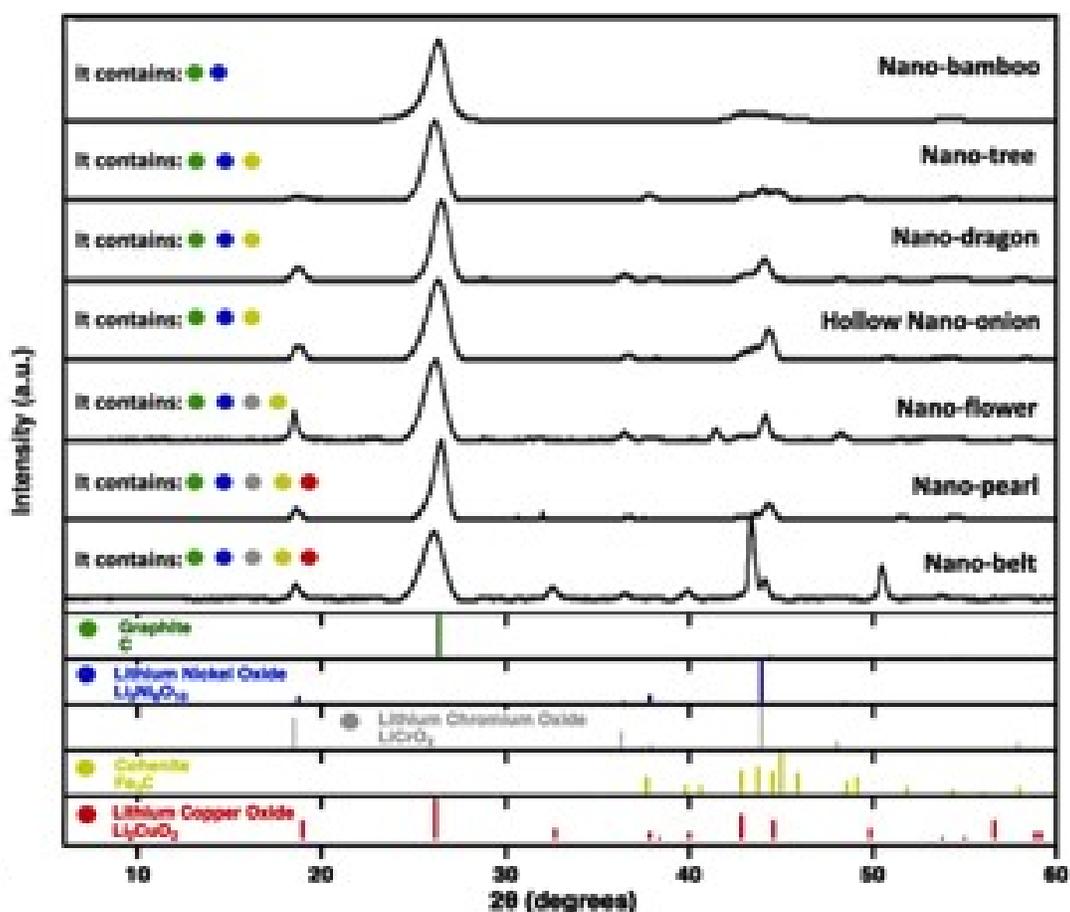
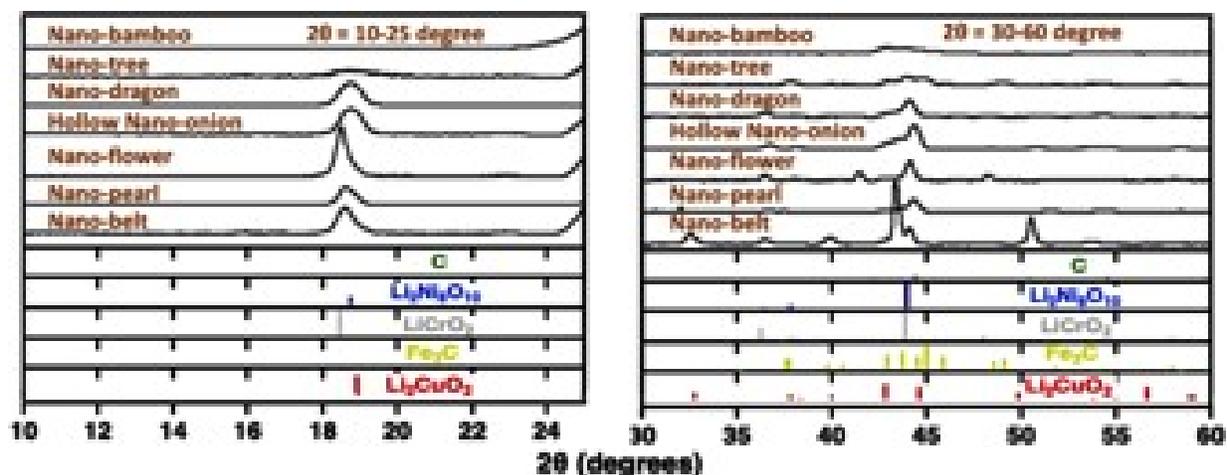
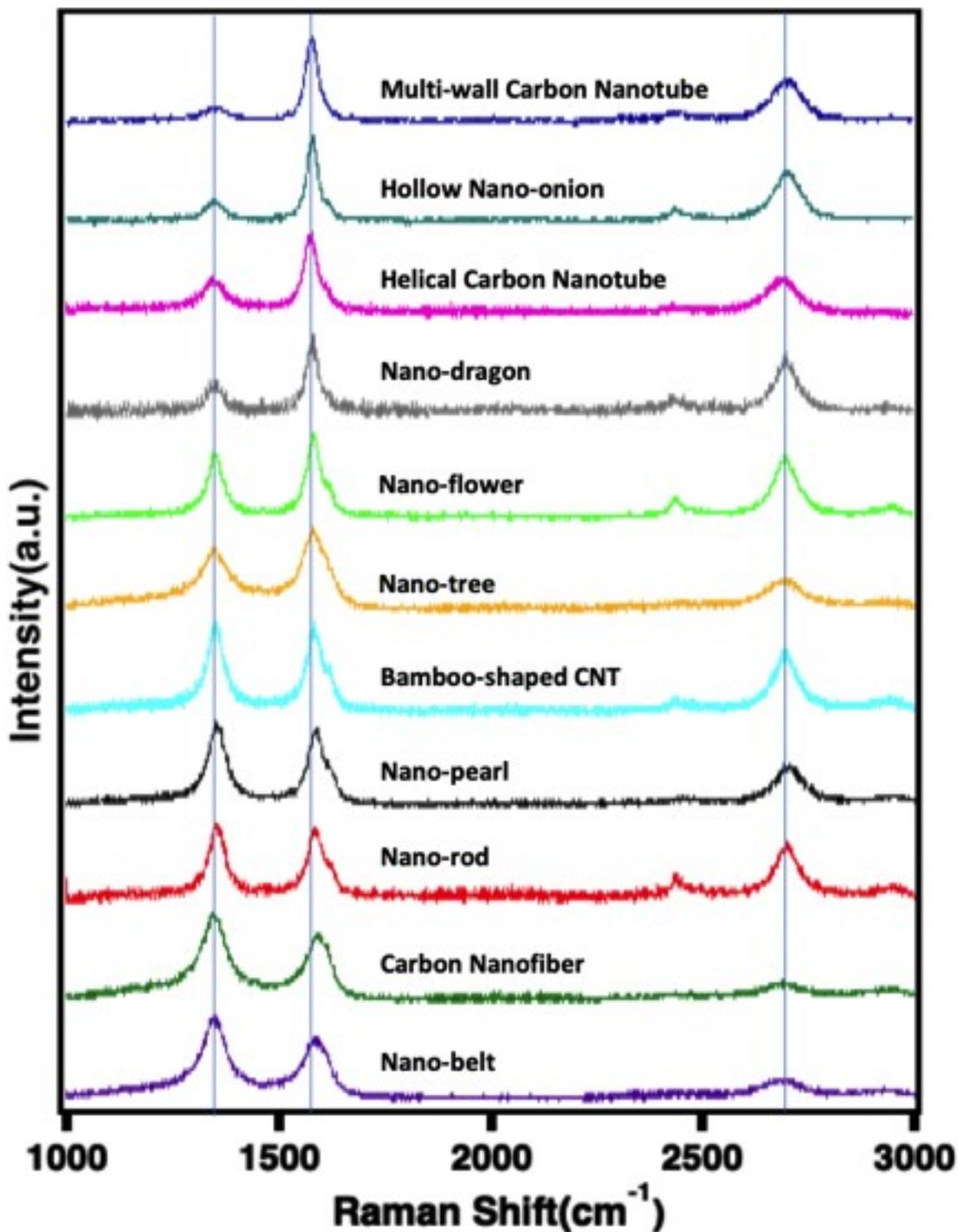


Figure S6 XRD of the synthesis product consisting of various labeled unusual nanocarbon morphologies synthesized by the electrolytic splitting of CO_2 in 770°C Li_2CO_3 with a variety of systematically varied electrochemical conditions. Modified from open access paper X. Liu, G. Licht, X. Wang, S. Licht, Controlled Growth of Unusual Nanocarbon Allotropes by Molten Electrolysis of CO_2 . *Catalysts* 12 (2022) 137. <https://doi.org/10.3390/catal12020125>.

Figure S7. Raman spectra of the synthesis products consist of various labeled GNCs and packed carbon nanotube assemblies synthesized by the electrolytic splitting of CO₂ in 770°C Li₂CO₃ with a variety of systematically varied electrochemical conditions.



Modified from open access paper X. Liu, G. Licht, X. Wang, S. Licht, Controlled Growth of Unusual Nanocarbon Allotropes by Molten Electrolysis of CO₂. Catalysts 12 (2022) 137. <https://doi.org/10.3390/catal12020125>.

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