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Surface-Area-Enhancing CVD Process

a subsidiary of CVD Equipment Corporation

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Challenge : implementing a multi-step scalable CVD process to create a hierarchically structured all-carbon material for electrochemical reactors/sensors

I : RAW MATERIAL / APPLICATION

CARBON IS THE MATERIAL OF REFERENCE/CHOICE FOR MANY ARCHITECTURED NANOSTRUCTURES FOR ELECTROCHEMICAL REACTORS/SENSORS

Properties:

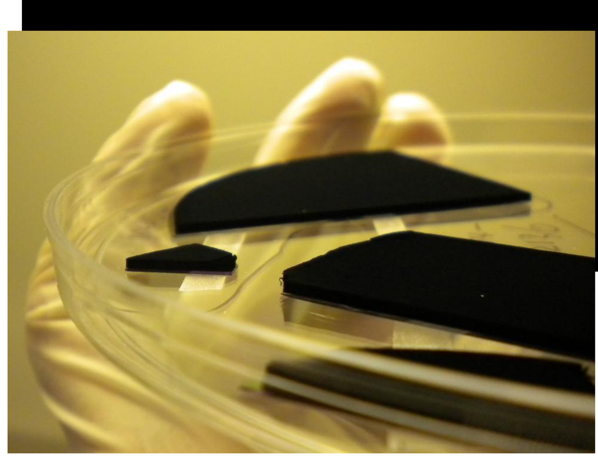
- Inert, chemically stable
- Light
- Cheap (mines, gaseous precursors, carbonization processes)

Forms:

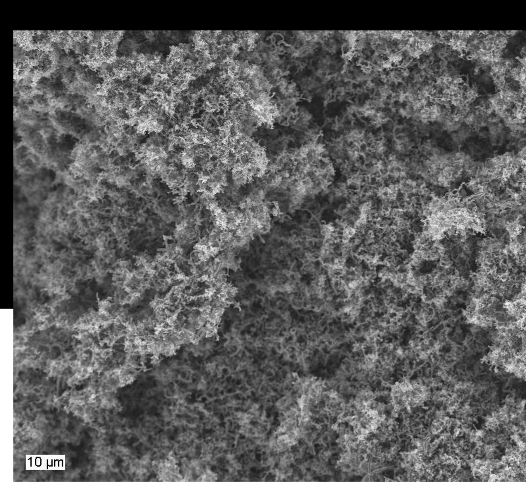
- Activated carbons
- CNTs, CNFs, C-fibers
- Carbon aerogels
- Graphene

Applications in:

- Energy Conversion and Storage, Biotechnology, Filtration, Membranes, Chemical Sensors, Polymer Nanocomposites, etc.



Vertically aligned CNTs (a few mm tall)



CNFs on Nickel Foam

CHOICE I : all carbon architecture

- ***CNTs or CNFs as "active" materials***
- ***Multi-layer Graphene/Graphitic Backbone as conductive subtrate***

II : DESIGN / PROPERTIES

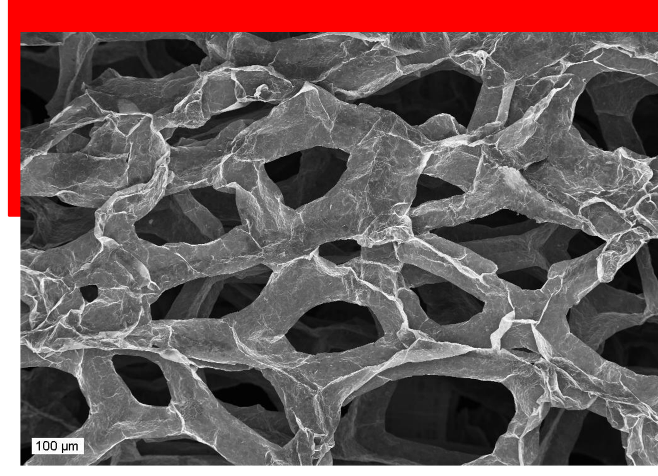
3D-STRUCTURES WITH HIGH SURFACE-AREA HIGH DENSITY ARE TARGETED TO OFFER HIGHER REACTIVITY

Known routes : random mix of carbon species (usually based on activated carbon thin films) and additional binders :

- *Loss of mechanical properties due to binders*
- *Average conductivity compared to true graphite*
- *Non-fully interconnected pristine structures (need of binders)*

Design problems:

- Porosity control : enabling gas molecules, ions or other active species motion without favoring any sealing at smallest scales.
- Density control : especially when specific volumetric/gravimetric properties are targeted depending on the application.
- Periodicity not necessarily required, as long as the phenomena characteristic lengths match the characteristic dimensions of the network.
- Continuity of substrate to insure good electrical properties without the use of binders if possible
- Mechanical properties have to be taken into account when one considers the final device.



Highly porous foam (Multi-layer Graphene/Graphitic backbone using Nickel foam template method)

CHOICE II : porous graphitic backbone

- ***Non-periodical interconnected flexible structure,***
- ***Multi-scale porosity***

III : PROCESS / TECHNOLOGY

VERSATILE, SCALABLE, LOW IMPACT PROCESS HAS TO BE IMPLEMENTED FOR LARGER PRODUCTION GOALS

Underlying conditions :

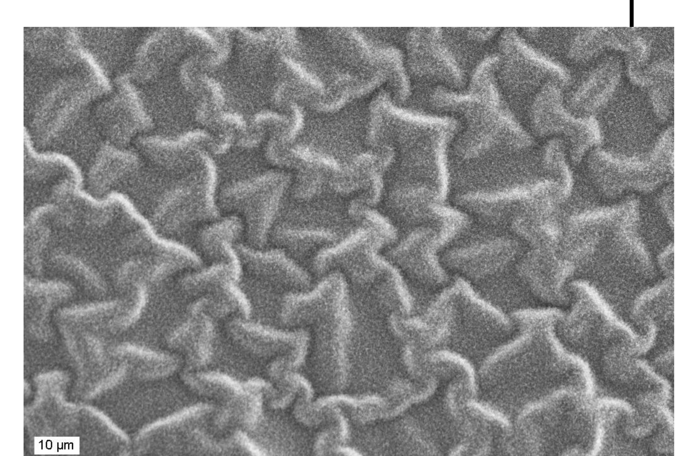
- wet chemistry processing to be avoided as much as possible

- *As less as possible chemical impact*

- lowest temperature possible route

- *Less energy consumption*
- *For less impact of thermal effects, particularly growth-induced stress effects*

Wrinkled CNFs thin films partially bonded to their SiO_2 wafer substrate as a result of growth-induced stress (expansion mismatch) .



Requested :

- Lower temperature CVD process
- Pre-treatment of substrate for covalent bonding
- Controlled infiltration of gaseous precursors

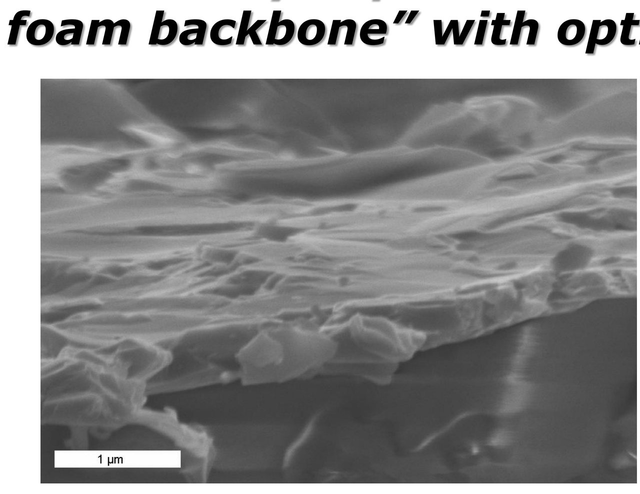
EasyTube® 3000

CHOICE III : adapted CNFs process

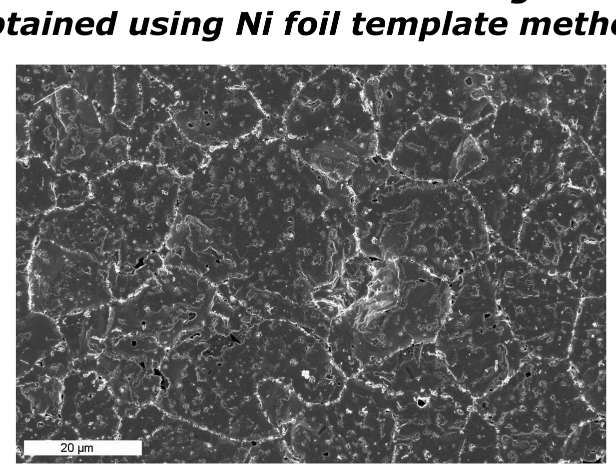
- ***Direct growth process (Ni catalyst)***
- ***Substrate oxidation pre-treatment to create roughness and higher chemical reactivity***
- ***Low temperature CNFs thermal CVD process***

Proof of concept : Chemical Vapor Deposition of CNFs on top of free-standing graphitic carbon film

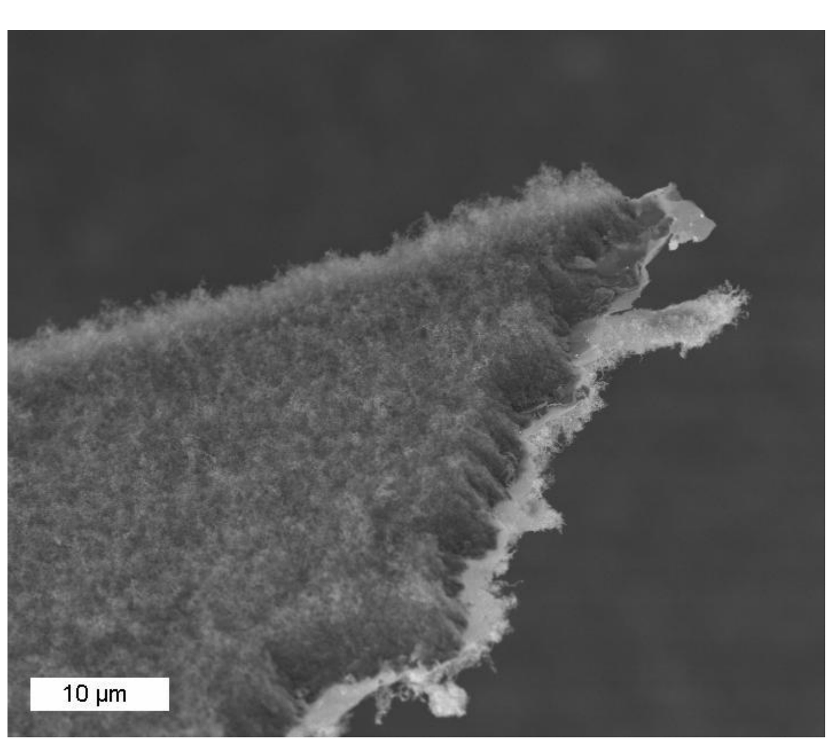
We start by experimenting on a flat graphitic film (~ 500nm thickness), keeping in mind that the final goal is to use as a support a "graphitic foam backbone" with optimized [i] surface area available for CNFs growth, [ii] porosity, [iii] mechanical behavior [iv] electrical conductivity.



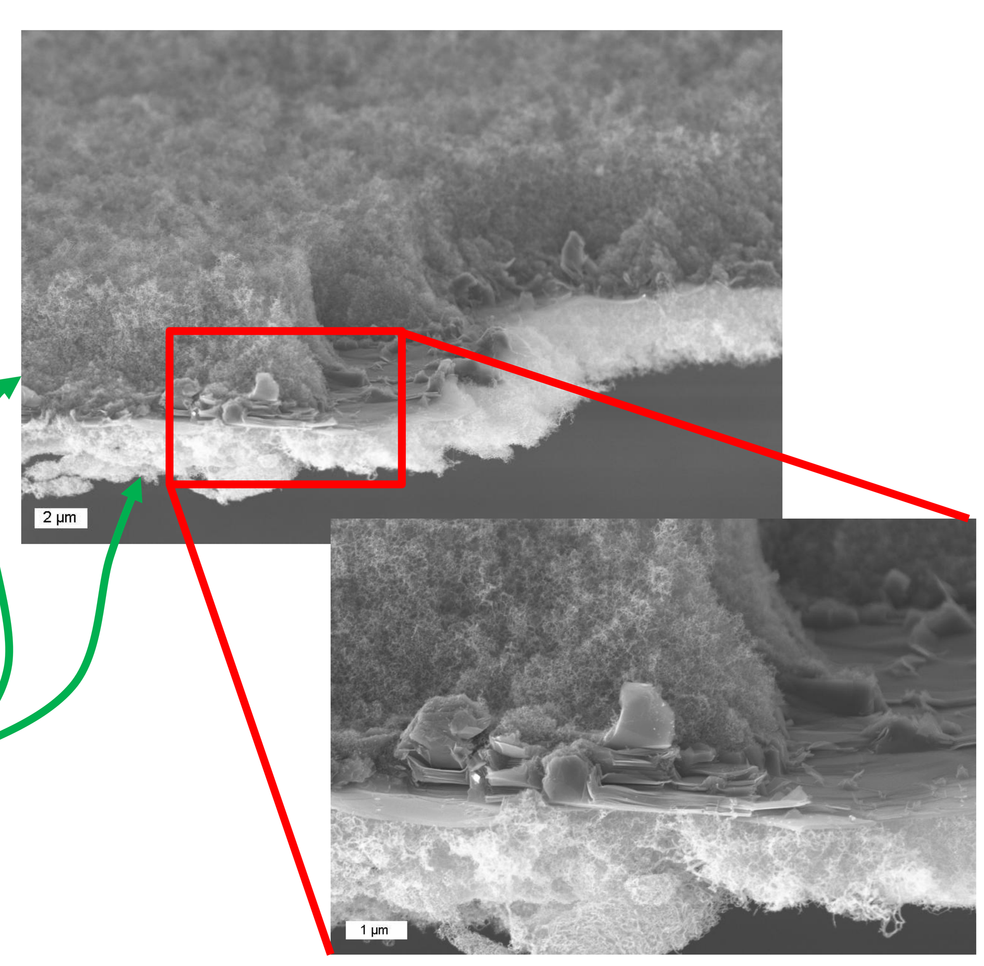
Tilted view on the flat graphitic substrate used as substrate before CVD growth (obtained using Ni foil template method)



Oxidation treatment of the graphitic film creates roughness and active sites on edges and defects for enhanced CNFs growth



Resulting free-standing all-carbon film after 5µm CNFs forests growth on both sides of the flat graphitic film



Next : test on 3D-graphitic foam

Ultra-light, high surface area, electrically and mechanically continuously connected 3D material platform for dense packing of active material

Free standing, ultra-light, 3D CVD foam with ~100nm thick graphitic walls

REFERENCES

1. Frackowiak E., Beguin F., Carbon, **239** (2001), 937-950.
2. Rolinson D.R., Long J.W., Lytle J.C., Fischer A.E., Rhodes C.P., McEvoy T.M., Bourg M.E., Lubers A.M. Chem.Soc. Rev. **38** (2009), 226-252.
3. Lytle J.C., Wallace J-M., Sassin M.B., Barrow A.J., Long J.W., Dysart J.L., Renninger C.H., Saunders M.P., Brandell N.L., Rolinson D.B., Energy Environ. Sci. **4** (2011), 1913-1925.
4. Gogotsi Y., Simon P., Science **334** (2011), 917-918.
5. Chen Z., Ren W., Gao L., Liu B., Pei S., Cheng H-M., *Nature Materials* **10** (2011), 424-428.
6. Li S., Luo Y., Yu. W., Wu, S., Hou P., Yang Q., Meng Q., Liu C., Cheng H-M., Adv. Energy Mater. **1** (2011) 486-490.

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