Application Note

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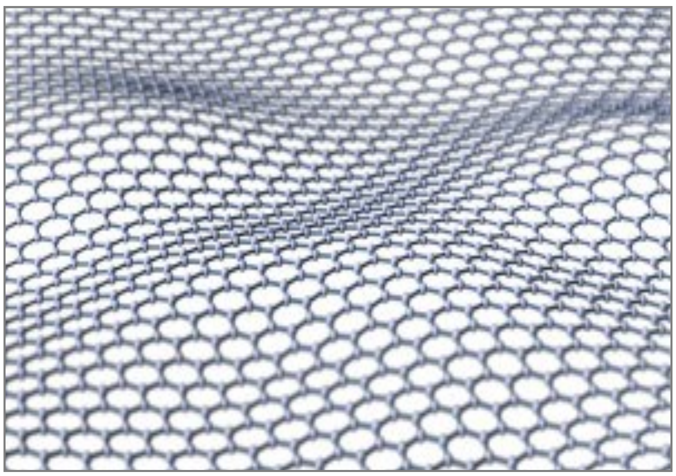
Use of Microfluidizer™ technology for graphene exfoliation

This Application Note gives an overview of the properties and applications of graphene. It also discusses preparation techniques and a case study executed by collaboration with the University of Cambridge to produce graphene for use as conductive inks. [1]

What is graphene?

Graphene is a material comprised of a sheet of carbon just one atom thick. See structure below:



Graphene has many useful properties:

- It is ultra-light yet immensely tough.
- It is 200 times stronger than steel, but it is incredibly flexible.
- It is the thinnest material possible as well as being transparent.
- It is a superb conductor.
- It can act as a perfect barrier - not even helium can pass through it.

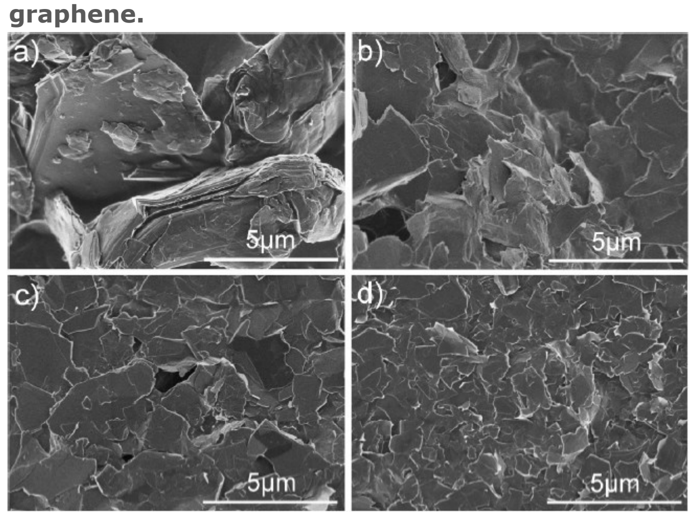
These properties lead to advances in many potential applications such as:

- Membranes to clean drinking water
- High strength composites and coatings
- High quality batteries.
- Micrometer-sized sensors
- Many electronic applications

There have been many different methods investigated for production of graphene. The first method used was the use of adhesive tape to exfoliate graphene from graphite. There have been many other techniques discovered including chemical vapor deposition (CVD), CO_2 reduction, spin coating and other complex processes. These processes have all been developed on the research scale and would be difficult/expensive to use for bulk production.

- Batch sonication or probe sonication can only achieve concentration of up to ~0.2 g/L with yield of only 1-2%.
- High speed rotor-stator mixer gave even worse results of concentration <0.1 g/L and yield <0.2%.
- With Microfluidizer, a concentration of ~100 g/L with 100% yield was achieved. This is 500-1000 times higher in terms of product concentration and 50-500 times higher in terms of yield.

The Microfluidizer processor can generate shear forces of 10^8 s^{-1} which literally rip the graphene sheets from ordinary graphite (see image below). This process is encouraging because Microfluidizer technology is linearly scalable to hundreds of liters per hour meaning that it can be used for large scale manufacturing of graphene.



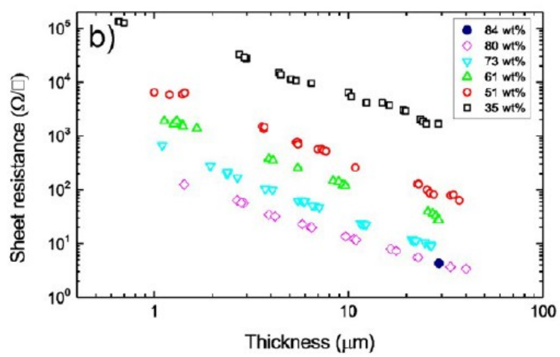
SEM images taken from coatings comprising (a) starting graphite, (b) after 1 cycle, (c) after 5 cycles, and (d) after 100 cycles through the Microfluidizer processor at 30,000 psi. The scale bar is 5 µm.

Case Study: Use of Exfoliated Graphene to Create a Printed Electronic Circuit

The researchers at the University of Cambridge performed process and formulation development to determine the impact of variables such as concentration of graphite, type of surfactant and number of passes through the Microfluidizer to determine the impact on the particle size, rheological properties and conductivity/resistivity of the inks formed.

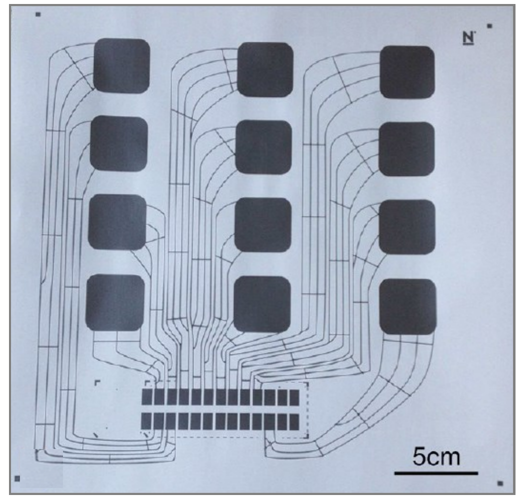
The researchers first evaluated the impact of the number of passes on the structural quality of the flakes. They found that the structural integrity was maintained up to 70 cycles, but above this, the material became distorted.

The researchers then evaluated the impact of concentration of the graphene as a function of thickness of the film on shear resistance.

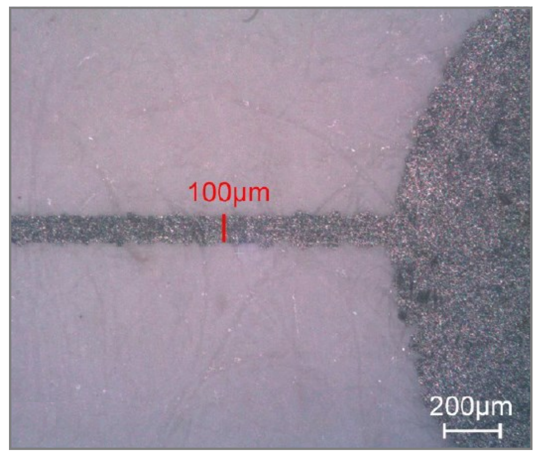


Other factors included the annealing temperature (they found that a temp of 300ºC produced much higher conductivity) and surfactant type (they found that sodium deoxycholate resisted decomposition at high temperatures much better than carboxy methyl cellulose.

The inks were printed using a semiautomatic flatbed screen printer into the pattern of a capacitive touch pad.



These lines were capable of being printed with a resolution of 100 µm.



The researchers found that they were able to generate highly conductive (with conductivity as high as 2 x 10^4 S/m) inks with adjustable viscosity. These inks could be robustly printed onto paper and exhibit excellent flexibility. Since the Microfluidizer is linearly scaleable from the lab scale to hundreds of liters per hour, they concluded that it is suitable for mass production of chemically unmodified graphene for use in a wide range of applications.

Work Cited:

[1] Panagiotis G. Karagiannidis, et. al. "Microfluidization of Graphite and Formulation of Graphene-Based Conductive Inks" *ACS Nano.* January 19, 2017. www.acsnano.org

Thanks to the researchers at the University of Cambridge for their collaboration

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