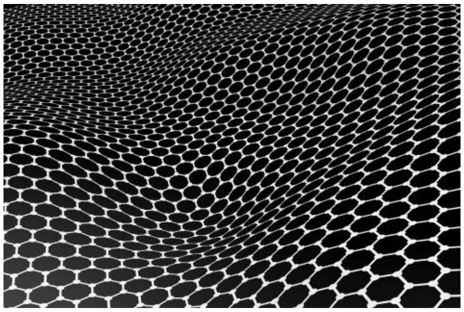
Microfluidics™

Application Note

Microfluidizer® Technology for the exfoliation of graphene & other 2D materials



INTRODUCTION

Two-dimensional (2D) topological materials, sometimes referred to as single layer materials, are currently one of the most active areas of advanced material research. Amongst these, graphene was the first one discovered and since has become the most researched 2D material. Following the isolation of graphene, other elementary 2D materials have emerged during the past decade such as boron nitride (BN) & transition metal dichalcogenides (TMDC) - MoS_2, WS_2, $MoSe_2$, WSe_2 etc.

The unique structures of these materials enable their many remarkable properties. Graphene has recently been used as an ideal material in some innovative biosensors for fast and reliable detection of Covid-19 in fighting against the current global pandemic.

This Application Note gives an overview of the properties and applications of graphene or few layer graphene (FLG) and discusses the preparation techniques. Included is a case study on producing graphene based conductive inks. [1]

Exfoliation of graphene & other 2D materials

WHAT IS GRAPHENE?

Graphene is a material comprised of a sheet of carbon that is just one atom thick. It has many useful properties including being:

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

FLG (few layer graphene) has similar properties to single layer graphene in many applications.

These properties have led to advances in many potential applications such as:

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

METHODS OF PRODUCTION

The first successful isolation of graphene from graphite was simply done with adhesive tape. This is probably the simplest method for obtaining graphene, but is unfortunately the least cost-effective and non-scalable process.

Methods for producing graphene and FLG fall into two categories. Top-down approaches to produce powdered or liquid graphene flakes via cleavage or exfoliation and then bottom-up approaches to produce graphene sheets through epitaxial growth or chemical vapor deposition (CVD).

Many methods, especially chemical or electrochemical based methods, are complex processes involving the use of high temperature and environmentally unfriendly gasses or chemicals which are often toxic. Therefore, mechanical exfoliation methods are good alternative methods. Among them liquid phase exfoliation is considered the most simplistic method and Microfluidizer® technology falls into this group.

However, most of these processes have been developed on the research scale but are difficult or expensive to use for bulk production.

- Batch sonication or probe sonication only achieves a concentration of up to ~0.2 g/L with a yield of only 1-2%.
- High speed rotor-stator mixers give even worse results of concentration at <0.1 g/L and a yield of <0.2%.
- Microfluidizer® technology is proven to be able to create 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 generates uniform shear forces up to 10^8 s^{-1} which efficiently peels off graphene sheets from ordinary graphite[1-4] or separate other 2D materials[5]. The technology is proven to be able to create a concentration of ~100 g/L with 100% yield[1]. This is 500-1000 times higher in terms of product concentration and 50-500 times higher in terms of yield. The technology is linearly scalable to hundreds of liters per hour meaning that it can be used for large scale manufacturing of graphene.

Exfoliation of graphene & other 2D materials

CASE STUDY: USING EXFOLIATED GRAPHENE TO CREATE A PRINTED ELECTRONIC CIRCUIT

University of Cambridge researchers developed a nonsolvent based liquid exfoliation process with a Microfluidizer® processor to create FLG[1]. The process was optimized after defining the impact of variables such as concentration of graphite, type of surfactant and processing conditions of the Microfluidizer® processor on the particle size, rheological properties, conductivity & resistivity of the inks formed.

After processing, the flakes were effectively exfoliated as shown in Figure 1, with the majority having a height of around 7 nm. The flake aspect-ratio continued to increase with subsequent processing cycles. No undesired chemical functionalization was reported that might have affected the processed samples, even after 100 passes. 100% of the produced FLG could be used to form the conductive ink.

The impact of concentration of the graphene & processing conditions on the ink formulations were evaluated and compared by blade coating. Favorable correlations were found between the ink and the resulting film thickness, sheet resistance and corresponding conductivity. Resistance as low as 2Ω/sq, corresponding conductivity as high as 2×10^4S/m, at 25 µm film thickness was achieved, making it suitable for printed electronics applications (Fig 2).

The printability of selected ink formulations was demonstrated with screen printing. Results proved the ink can be robustly printed onto paper to form the pattern of a capacitive touch pad (Fig 3) with line resolutions of 100µm (Fig 4).

Microfluidizer® technology is linearly scalable from the lab to hundreds of liters per hour, ideal for mass-production of chemically unmodified graphene.

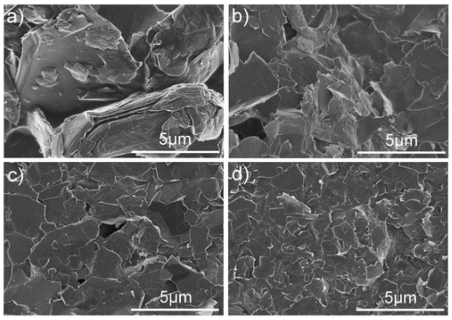


Figure 1 – SEM images taken from coatings comprising (a) starting graphite, (b) after 1 cycle, (c) after 5 cycles, (d) after 100 cycles through the Microfluidizer processor at 30,000 psi. The scale bar is 5 µM[1]

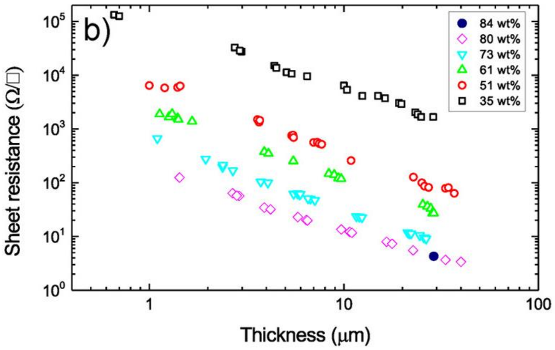


Figure 2 – Sheet resistance of coated graphene ink films as a function of film height[1]

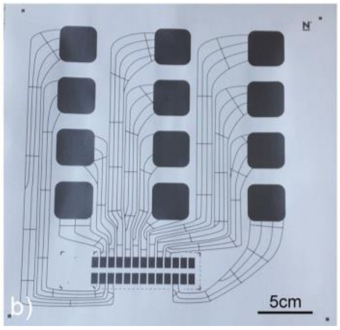


Figure 3 – Example of capacitive touchpad design printed on paper via screen printing of the graphene based conductive ink[1]

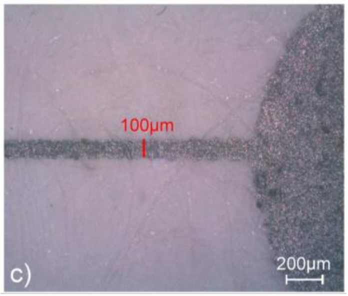


Figure 4 – a close-up on the printed line to show line resolution[1]

References:

1. P.G. Karagiannidis, et. al., ACS Nano. 2017; 11(3).
2. K.R. Paton, et al., Mater. Res. Express, 2017; 4(2).
3. M. Buzoglo, et al., Chem. Mater., 2016; 28(1).
4. S. Baskut, et al., J. Eur. Ceram., 2018; 38(11).
5. A.T. Seyhan, et al., J. Solid State Chem., 2017; 249

©2020 Microfluidics Int'l Corp