ASX Announcement

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Strong Advances in VFD Development

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ASX Symbol
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FSE:M11

HIGHLIGHTS

- Newly developed conditioning stage improves graphene oxide surface oxidation levels.
- Improved methodology for controlling the surface oxidation to suit targeted applications.
- Work continuing to understand end-user application requirements, such as toxicity levels in biological applications, anti-fouling properties in membranes and use in water filtration applications.
- The results provide the potential of being able to provide bespoke graphene oxide for varying applications.
- Operating parameters will now be established to provide yield data for future use in scaling the system for commercial production.

Advanced materials company, First Graphene Limited ("FGR" or "the Company") (ASX: FGR) is pleased to provide an update of the work conducted in conjunction with 2D Fluidics Pty Ltd on the Vortex Fluidic Device (VFD) at the Company's facilities at the Graphene Engineering and Innovation Centre (GEIC) in Manchester, UK and Flinders University

Background Summary on Graphene Oxide

Graphene oxide (GO) is the chemically modified derivative of graphene, whereby the basal planes and edges have been functionalised with oxygen containing functional groups such as hydroxyl, epoxy and carboxyl groups. These oxygen functionalities make GO hydrophilic and therefore dispersible, forming homogenous colloidal suspensions in water and most organic solvents. This makes it ideal for use in a range of applications.

To date, the most widely used process for the synthesis of graphene oxide is Hummer's method. This typically requires strong acids and oxidants, such as potassium chlorate ($KClO_3$), nitric acid (HNO_3), concentrated sulfuric acid (H_2SO_4) and potassium permanganate ($KMnO_4$). Much work has been done to improve the synthesis methods while maintaining high surface oxidation, however these all required strong acids and oxidants.

Through its subsidiary 2D Fluidics Pty Ltd, FGR is developing a more benign processing route for oxidised graphene. The objective is to provide controlled levels of surface oxygen functionality to give better easier compatibility in aqueous and organic systems. This will not incur the higher oxygen (and other defect) levels which result from Hummer's method and its subsequent reduction steps. It will also provide the ability to "tune or optimise" the surface oxidation level to suit respective applications.

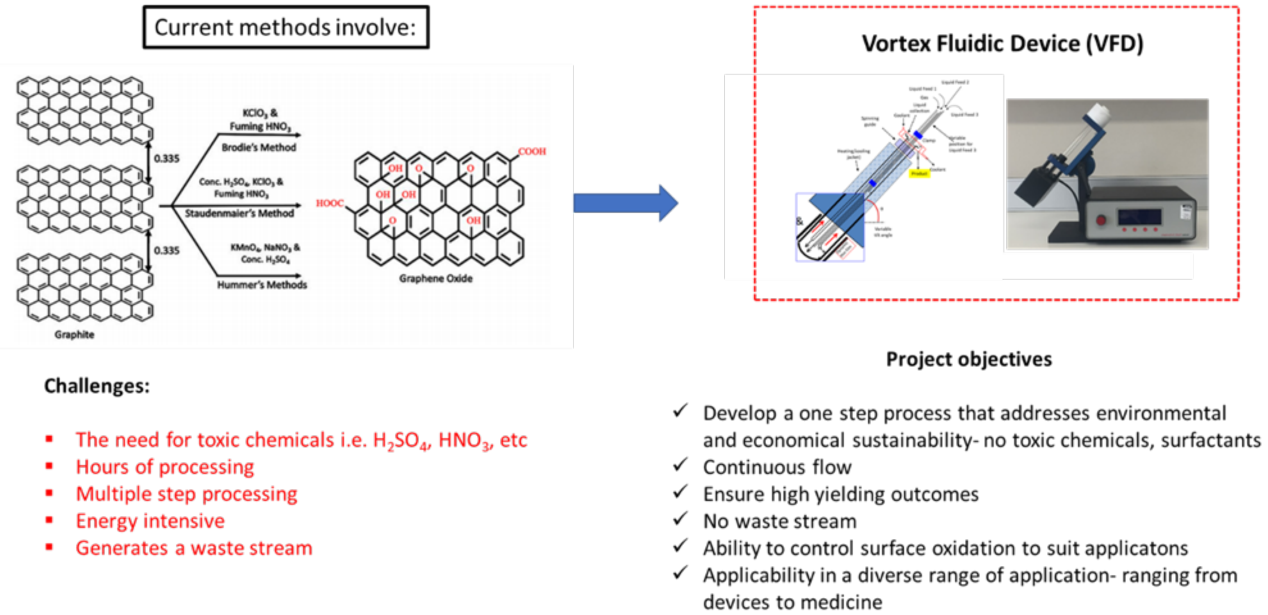

Figure 1: Synthesis of graphene oxide via the chemical synthesis route[1] vs the Vortex Fluidics Device (VFD) route

FGR's method synthesises GO directly from bulk graphite using aqueous H_2O_2 as the green oxidant. Different energy sources have been used for the conversion of H_2O_2 molecules into more active peroxidic species, such as a combination of a pulsed Nd:YAG laser and/or other light sources. The irradiation promotes the dissociation of H_2O_2 into hydroxyl radicals which then leads to surface oxidation.

The technology has been successfully transferred to the FGR laboratories at the Graphene Engineering and Innovation Centre (GEIC) in Manchester where it has undergone further development and optimisation to identify, understand and resolve future upscaling issues.

[1] Adetayo et al Open Journal for Composite Materials, 2019, 9, 207-229. Synthesis and fabrication of graphene and graphene oxide.

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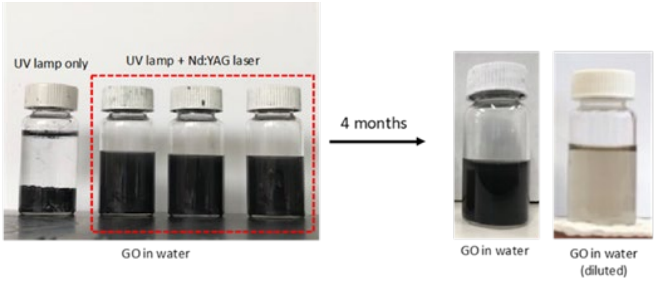
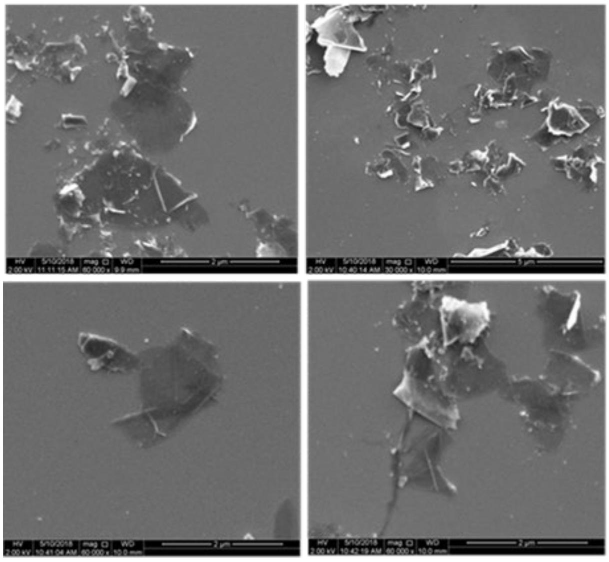
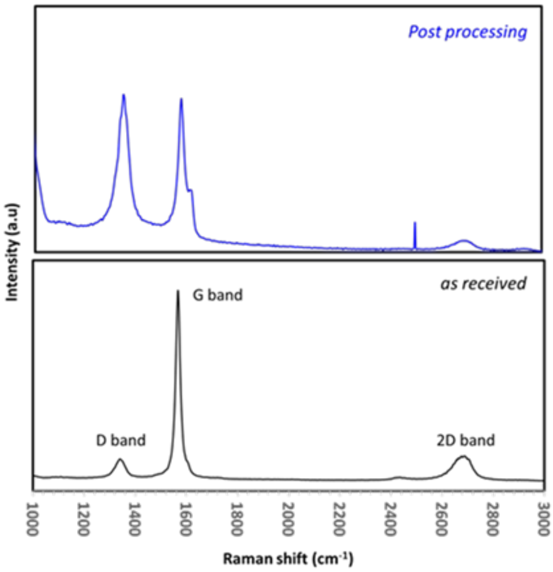
(a)  (b) 

Figure 2: Oxidised graphene produced from 2 different starting materials; (a) graphite ore and (b) PureGRAPH® graphene. Both starting materials gave oxidised graphene sheets with an enhanced level of surface oxidation





	Raman Shift Positions (1/cm)			Metrics	
Reference	D Band	G Band	2D Band	I(D) / I(G)	I(2D) / I(G)
Graphite Flakes (starting material)	1350	1578	2714	0.30	1.00
Green Graphene Oxide (Post Treatment)	1348	1586	2683	0.85	0.54
Peak Shift	1.9	-7.4	30.4		

Figure 3: SEM (above left) and Raman analysis of oxidised sheets (above right). The Raman analysis shows the changes in the peak positions and increases in peak intensity ratios, confirming the synthesis of oxidised surfaces

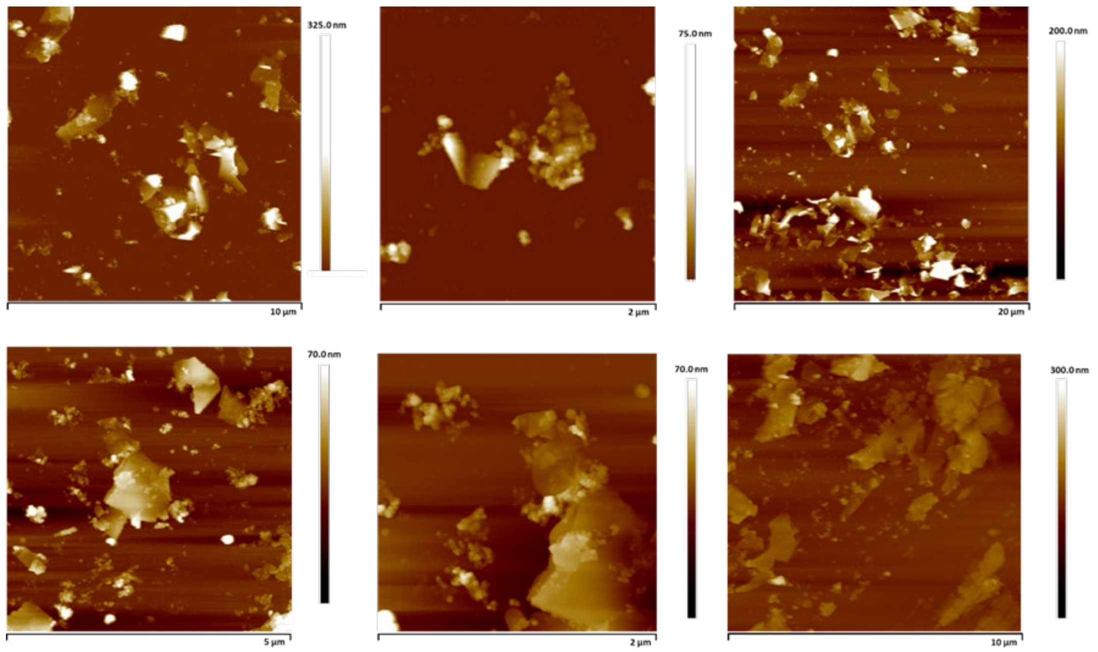
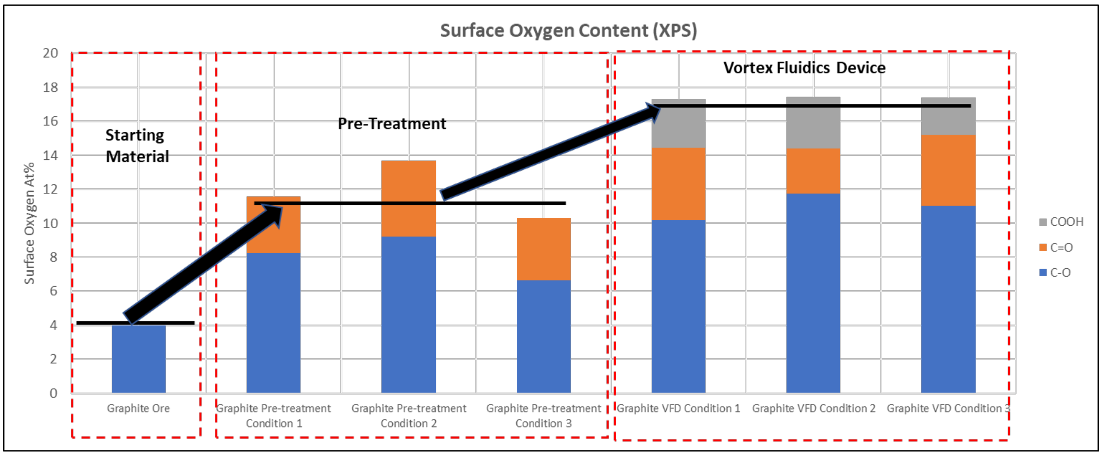


Figure 4: AFM height images of the oxidised graphene sheets

XPS analysis showed that the use of pre-treatment step in combination with the near infrared laser gave oxidised graphene sheets with an average surface oxidation of ~30-35%: this will enhance compatibility with aqueous systems.

Further trials have already demonstrated that the two-step process is reproducible and versatile, with the ability to process different starting materials of graphite. The multi-disciplinary team has identified that control of the feed rate and energy input will allow us to control the surface oxidation, providing a consistent material that can be tailored as required for a range of applications.

Figure 5 shows that increase in surface oxygen content for two starting materials: graphite ore (top) and PureGRAPH® graphene (bottom). As we go through the two-stage process, in both cases the surface oxygen functionality increases. The end-product has a range of functional groups, including C-O, C=O and COOH.



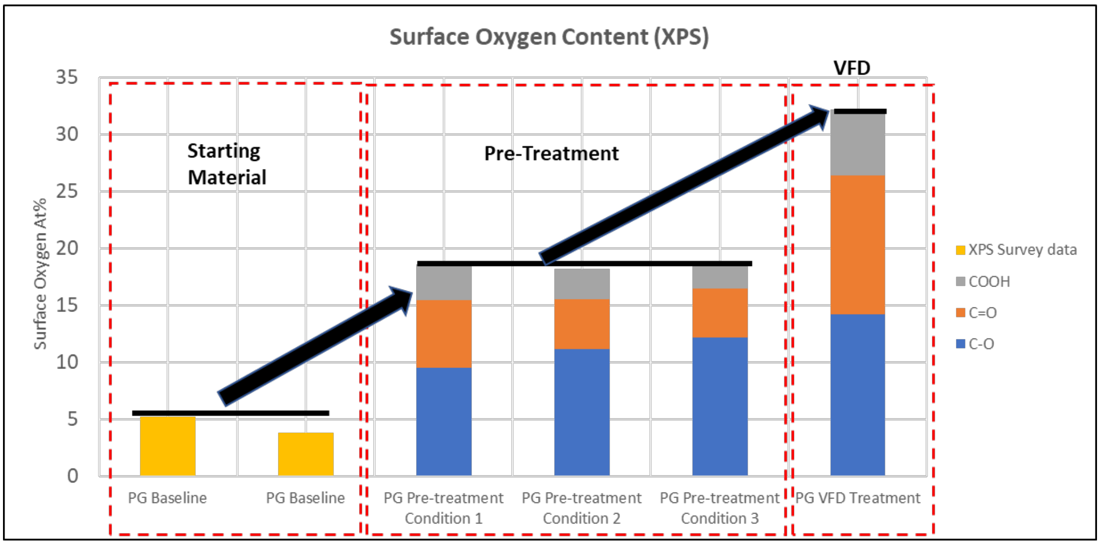


Figure 5: XPS analysis. XPS analysis shows the increase in oxidation after pre-treatment and Vortex Fluidics Device (VFD) processing. The top chart shows graphite as the starting material, the bottom chart shows PureGRAPH graphene as the starting material.

Next Steps

Operating parameters will now be established to provide yield data for future use in scaling the system for commercial production.

It will also commence examining the end applications including, but not limited to the use in electronic devices, testing levels of toxicity for biological applications, for water filtration membranes and incorporation in membranes for studying anti-fouling properties.

Craig McGuckin, Managing Director of FGR, said, *"The complementary characterisation techniques used to confirm the synthesis of oxidised graphene gives us confidence we are on the right route towards fabricating a material which is comparable to the historical GO fabricated using the conventional Hummers method. We are now reviewing end applications and thus exploring a number of avenues which include but are not limited to the use in devices, testing levels of toxicity for biological applications, for water filtration membranes and incorporation in membranes for studying anti-fouling properties."*

About First Graphene Ltd (ASX: FGR)

First Graphene Ltd. is the leading supplier of high-performing, graphene products. The company has a robust manufacturing platform based upon captive supply of high-purity raw materials and an established 100 tonne/year graphene production capacity. Commercial applications are now being progressed in composites, elastomers, fire retardancy, construction and energy storage.

First Graphene Ltd. is publicly listed in Australia (ASX:FGR) and has a primary manufacturing base in Henderson, near Perth, WA. The company was recently incorporated in the UK as First Graphene (UK) Ltd. and is a Tier 1 partner at the Graphene Engineering and Innovation Centre (GEIC), Manchester, UK.

About The University of Manchester

The University of Manchester is the home of graphene – it is where the one-atom thick material was first isolated. Today we have an unrivalled breadth of academic expertise and work in collaboration with dozens of partners. By leveraging the research power along with the vast infrastructure we have put in place we can leverage the investment in fundamental science and facilities to collaboratively generate value, IP and skills needed for the development of products and applications.

PureGRAPH® Range of Products

PureGRAPH® graphene powders are available in tonnage volumes with lateral platelet sizes of 20µm, 10µm and 5µm. The products are high performing additives, characterised by their high quality and ease of use.

With authority of the board, this announcement has been authorised for release, by
Peter R. Youd
Director, Chief Financial Officer and Company Secretary

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