A Reliable Supply Platform for Graphene and other 2D Materials

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ABSTRACT

Thomas Swan Advanced Materials division is devoted to the development of high value materials for emerging technologies. In 2014, we launched a new range of high quality, few layer graphene nanoplatelet products - these materials are now available in kilogramme quantities. The proprietary liquid phase exfoliation process used is exclusive to Thomas Swan and we continue to explore the flexibility of this process to deliver new graphene products and non-carbon 2D materials.

Keywords: Thomas Swan, Elicarb®, graphene, nanoplatelet, liquid exfoliation.

1 INTRODUCTION

Thomas Swan & Co. Ltd. is an independent chemical manufacturing company with offices and warehousing in the UK, USA and China and a global network of distributors, we service the domestic and international markets and export to over 80 countries worldwide. Founded in 1926 in Consett, in the North East of England (still home to our manufacturing facilities) Thomas Swan today produces over 100 products, in kilogramme to multitonne quantities and offers an experienced and flexible manufacturing service.

At Thomas Swan we have always searched for new and exciting technologies. Building on our core competency in chemistry, over the years we have founded or invested in many different early stage companies. From our beginnings in road surfacing, we have diversified into a wide range of businesses including photonics (Cambridge Photonics Ltd.), protein separation (Bioprocessing Ltd.), flat screen displays (Screen Technology Ltd.), contract research (BioDynamics Research Ltd.), MOCVD reactors (Thomas Swan Scientific Equipment Ltd.) and hydrogen storage (Cella Energy Ltd.).

Our willingness to try something new is core to our business philosophy and we are always keen to be challenged with new ideas and technically led business opportunities. Whilst we are now a leading supplier to the international chemicals market, we have retained the flexibility and customer-focused approach of a family business which enables us to offer outstanding service and support to customers ranging from major multinationals to small startups.

2 ELICARB® CARBON NANOTUBES

Thomas Swan were early investors in carbon nanotube technologies, forming a strong collaboration with the University of Cambridge to develop a scalable process for the manufacture of carbon nanotubes. The carbon nanomaterials business was launched in 2004, and has continued to add capabilities. Today, Thomas Swan Advanced Materials can manufacture kilogramme quantities of both single-wall (SW) and multi-wall (MW) carbon nanotubes. A particular strength is our ability to manufacture high quality semi-conducting, single walled carbon nanotubes with low catalyst residues and a high surface area: our Elicarb[®] SW products.

Thomas Swan continues to build on its position as a world leading supplier of high quality single wall carbon nanotubes with the installation and commissioning of a new carbon nanotube purification plant in late 2013. The new plant has a capacity of 500 kg/yr and is used for the manufacture of Elicarb[®] SW Low Residue carbon nanotubes.

3 ELICARB® GRAPHENE

In 2012 Thomas Swan commenced work on routes to produce high quality graphene nanoplatelets, seeing this next step as a good fit with our existing carbon nanotube business. At the time, there was a clear market need for consistent graphene products to enable application development and also multiple academic initiatives developing novel routes to graphene products.

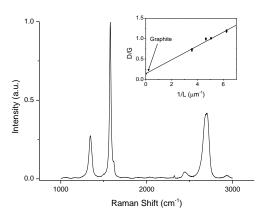


Figure 1: Raman spectrum (532 nm laser) of Elicarb® Graphene Powder produced at Thomas Swan & Co. Ltd showing a high quality sp² carbon network with a typical D/G ratio of 0.28 and D/D' \leq 5.0. Comparison of D/G ratio against flake size for a variety of samples (inset) confirms the D band is associated with edge defects only rather that in plane defects.

Graphene is the thinnest, strongest, most flexible, best barrier material, most conductive (thermal & electrical) material and most transparent material known to mankind. Since its isolation by Geim & Novoselov in 2004, intensive research efforts have been invested to realise the potential of graphene, develop manufacturing routes and identify value added applications. To date – no significant commercial applications have adopted the material.

Clearly, the commercial development of disruptive new materials faces many challenges. Based on our particular experience at Thomas Swan we identified two primary hurdles for the development of a sustainable graphene industry. These are:

- The need for a reliable source of good quality raw materials.
- The development and prototyping of applications where the new material properties add value.

By focusing on a manufacturing route which produces pristine graphene nanoplatelets and then reliably delivers good quality prototyping quantities of graphene, Thomas Swan is enabling application holders and OEMs to move forward and develop applications and components. Also, by making graphene materials commercially available at an early stage we have enabled customers access to evaluate our materials at the earliest opportunity with "no strings attached", such as non-disclosure or joint development agreements.

Back in 2011, we sought routes to produce high quality graphene nanoplatelets, targeting applications requiring high electrical and thermal conductivity. Prof. Jonathan Coleman at the CRANN Institute, Trinity College Dublin (TCD) was chosen as a suitable academic partner; he had identified a route to pristine graphene nanoplatelets *via* ultrasonic processing of graphite. We started a collaborative project in 2012 with the challenge of making the same product by a scalable process. In 2013, a scalable, patentable process was identified and an exclusive license agreed. Full details of this novel technique were published in Nature Materials in early 2014.¹ At the same time, the TCD process was effectively transferred to our Consett laboratories and our Advanced Materials Division started manufacturing and selling R&D quantities to customers in May 2014 (see Figures 1 and 2 for Raman spectrum and TEM images of first batches produced at Thomas Swan).

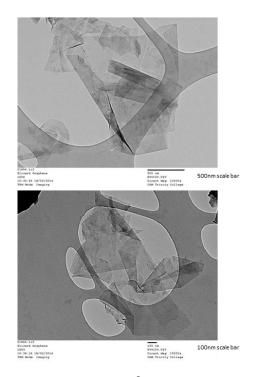


Figure 2: TEM image of Elicarb[®] Graphene Powder produced on-site at Thomas Swan & Co. Ltd.

The process from TCD was capable of yielding high quality graphene in a mixture of graphene, graphite and solvent. Thomas Swan has enabled the isolation of graphene nanoplatelets in an industrially relevant and scalable manner. The Consett based engineering team was mobilised to design, test and assemble a series of unit operations which would exfoliate graphene from graphite, separate and recycle non-exfoliated graphite, polish final graphene products and isolate graphene as Elicarb® Graphene Powder or Elicarb® Graphene Dispersion. A pilot line capable of manufacturing 1kg/day of Elicarb® Graphene was installed and commissioned by November 2014.

As of 2015, Thomas Swan has further stepped up both application and process development with Elicarb[®] Graphene. The Advanced Materials team were successful in

numerous joint bids to Innovate UK's "Realising the Graphene Revolution", a funding call for joint development proof-of-concept proposals seeking to find real-life value added applications for graphene products. The applications under investigation include energy storage, capacative touch screens and conductive inks for printed electronics. We are also driving towards an initial manufacturing capability of 10 tonnes per annum in a project supported by Horizon 2020 funding from the European Union.

Graphene was first produced at Thomas Swan in early 2014. Crucially the graphene produced at our manufacturing site was characterised in precisely the same way as that produced by our academic partners at TCD. This detailed characterisation (Figures 1, 2, 3 and 4) confirmed that our manufacturing techniques at Thomas Swan produced graphene of similar quality – lateral dimensions, platelet thickness and electrical conductivity relative to that prepared at TCD.

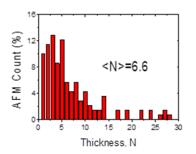


Figure 3: Number average flake thickness data of Elicarb Graphene[®] Powder by AFM statistical analysis.

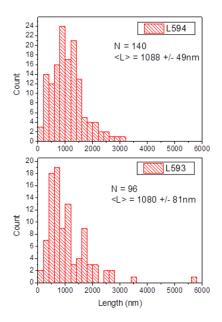


Figure 4: Number average platelet size of Elicarb® Graphene Powder by TEM image statistical analysis. Batch comparison shows excellent reproducibility.

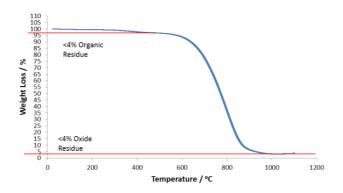


Figure 5: Thermogravimetric analysis of Elicarb® Graphene Powder.

4 CONCLUSIONS

Thomas Swan & Co. Ltd. has successfully scaled up a graphene production system from the bench scale to pilot scale in a short timescale. Today, the plant is delivering, high quality graphene nanoplatelets to customers around the world and getting excellent feedback. We are receiving repeat orders and the pilot line is enabling our customers to move forward with product development.

We are continuing to move forward with product scale-up in parallel to collaborative application development with customers in a range of markets. We continue to seek for new opportunities for graphene and non-carbon 2D materials.

REFERENCES

[1] K. Paton *et al.*, "Scalable production of large quantities of defect-free few-layer graphene by shear exfoliation in liquids," Nature Materials, 13, 624 - 630, 2014.

ACKNOWLEDGEMENTS:

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