Haydale Limited

Graphene Stakeholders Association

Commercialisation of Graphenes

17th October 2013

haydale
PART OF THE ICL GROUP

Haydale evolution

Haydale set up 2003

- Plasma treating recycled rubber crumb and Nitrile
- Introduction of the technology to CNTs

ICL purchased Haydale in May 2010

- Invested £3.5m to date
- Process patent applications in 2009-2012. International phase started
- Added mined graphite in November 2011
  - Production of Graphene Nano Platelets ("GNPs")
  - First Conductive GNP based ink
- Growth prospects
  - The hot sector
  - In house functionalisation
  - Collaborations/licensing
- New Nano safe facility opened in May 2013- cost £1m
- First commercially available UK conductive Graphene ink launched June 2013
  - No additives, 10 ohms/sq, coverage 350 cm^2/gm
- Current functionalisation of nano particles: 1 tonne (market share <2%?)

haydale
PART OF THE ICL GROUP

Nano Processing Facility

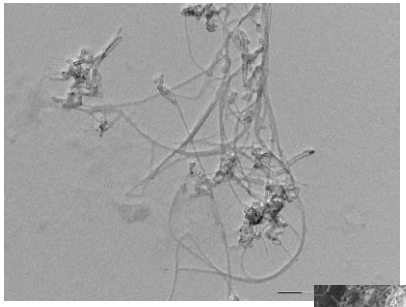
haydale
PART OF THE ICL GROUP

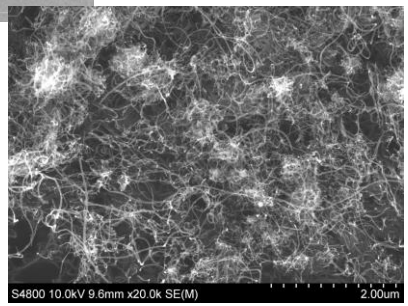
Haydale Material Focus

Functionalised Nano Materials :

Carbon Nano Tubes

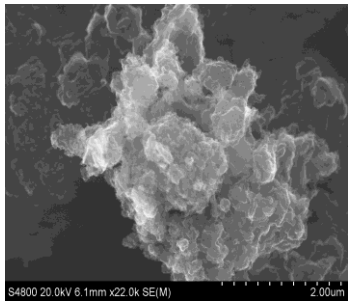
- Single/Double Wall DWCNT
- Multi Wall MWCNT

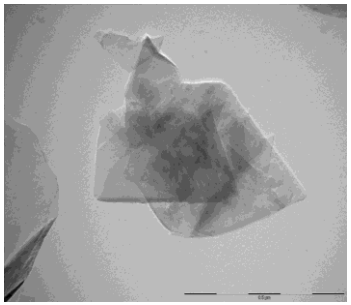




Graphenes

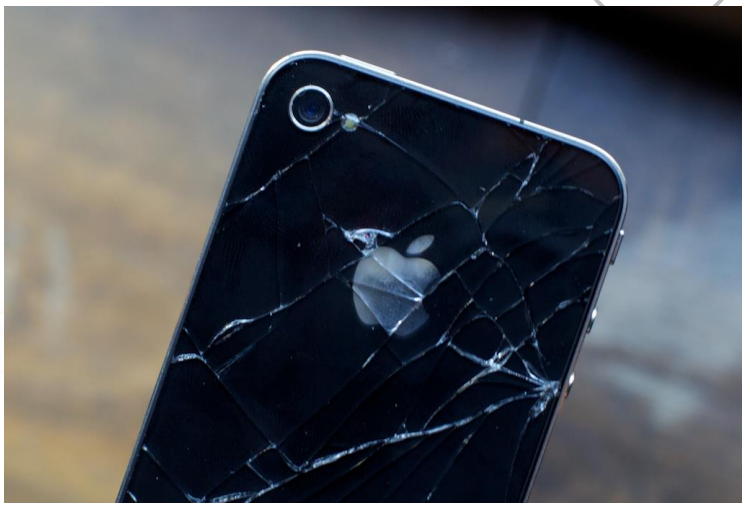
- Few Layer FLG
- Nano Particles GNP





haydale
PART OF THE ICL GROUP

Graphene: What can it do?



haydale
PART OF THE ICL GROUP

haydale
PART OF THE ICL GROUP

The mechanics of graphene nanocomposites: A review

Robert J. Young[a], Ian A. Kinloch[a], Lei Gong[a], Kostya S. Novoselov

6. Conclusions and prospects

- Graphene and GO show promise as reinforcements in high-performance nanocomposites and ……ought to have outstanding mechanical properties.

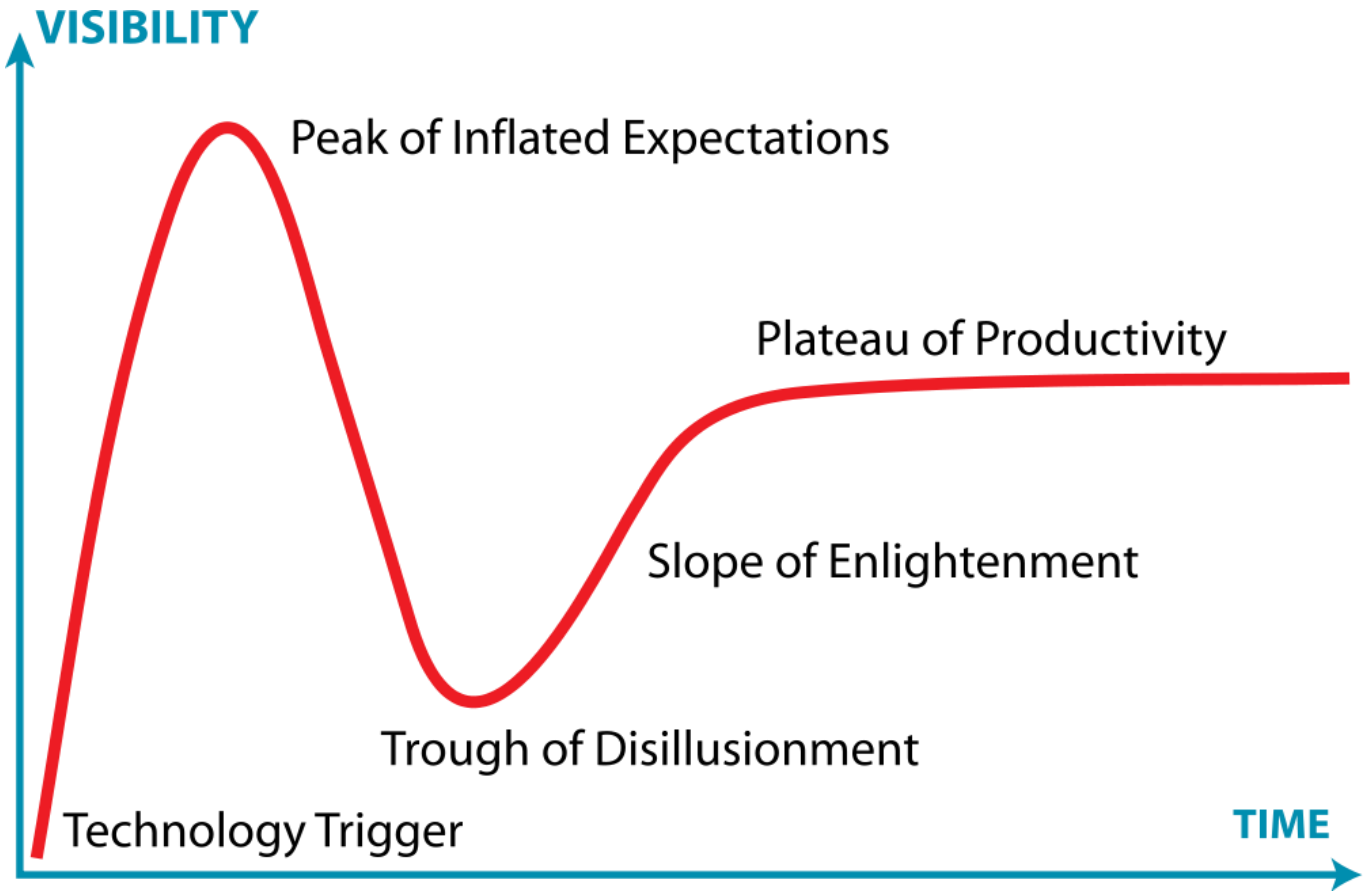
BUT there are problems

- Obtaining good dispersions
- Exfoliation of graphene into single- or few-layer material with reasonable lateral dimensions
- Producing graphene/GO without imparting significant damage upon the flakes.

It Needs

- Strong interface between the reinforcement and the polymer matrix to obtain the optimum mechanical and conductive properties.

The dangerous Hype Cycle



haydale
PART OF THE ICL GROUP

Graphene

The new wonder material

- The science is complex
- The cynics could say- FT 15th October 2013
  - "it's advanced properties give it unrivalled ability to separate investors and their money"

We need to prove them wrong!

How

- By getting the materials fit for purpose
- Delivering consistent quality in volume
- At an affordable price

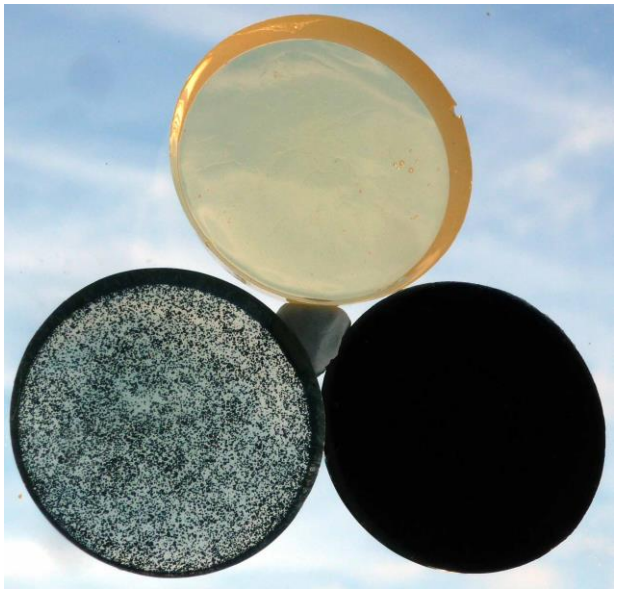
haydale
PART OF THE ICL GROUP

Removing Hype

- Standardise Definitions
  - Makes comparisons easier
  - Removes confusion and misconceptions
- Educate the customer
- Produce materials that:
  - “Does what is says on the tin”
- Dispersion is the key to commercialisation

Dispersion and covalent bonding

0.5 wt% NON Functionalised CNT in epoxy resin



Epoxy (no filler)

0.5 wt% Functionalised HDPlas CNTs in epoxy resin

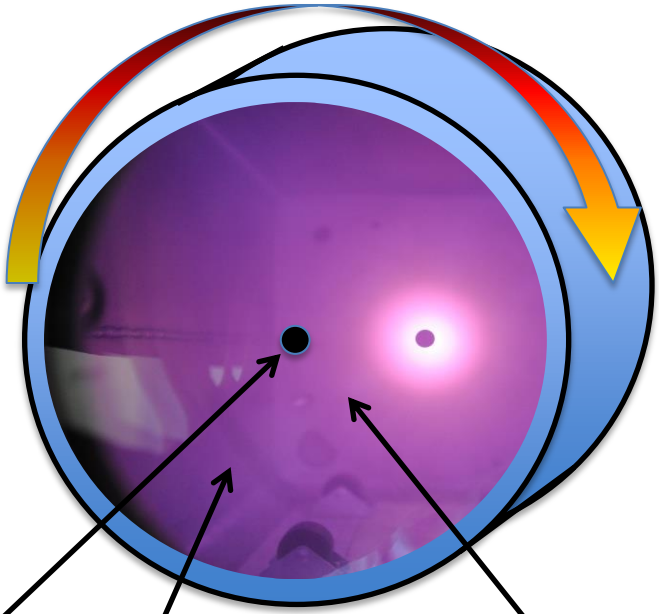
haydale
PART OF THE ICL GROUP

Haydale Core Technology

Split Plasma Processing :

- **Patented technology**
- **Low temperature**
- **Low energy**
- **Bespoke functionalisation of materials**

Haydale Process Technology

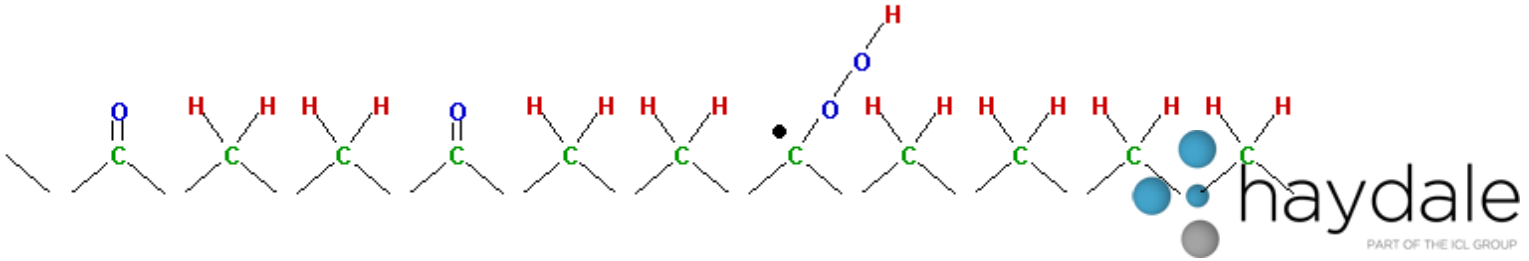


Plasma spectra influenced by the atmosphere

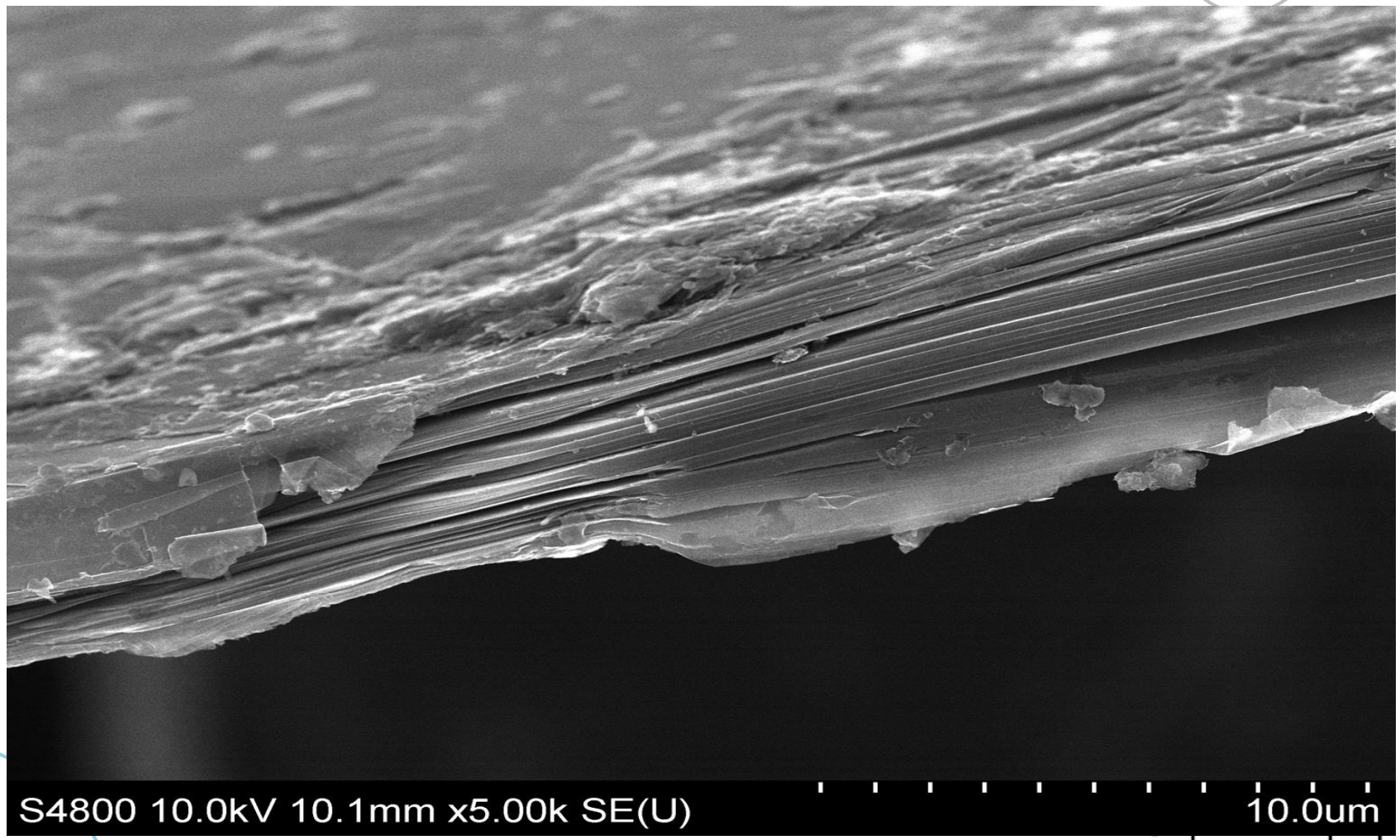
haydale
PART OF THE ICL GROUP

Haydale split plasma processing

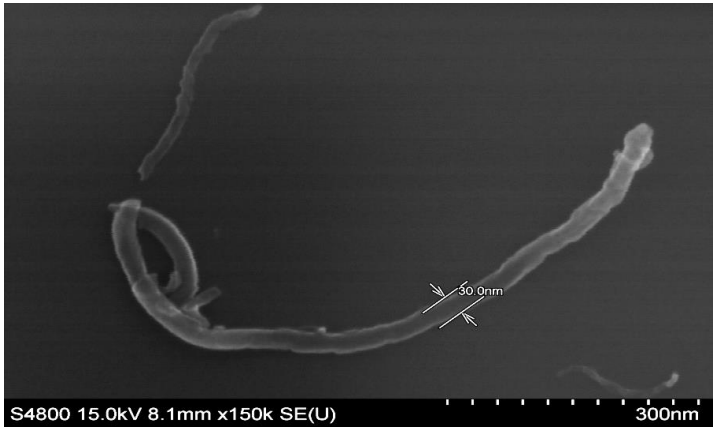
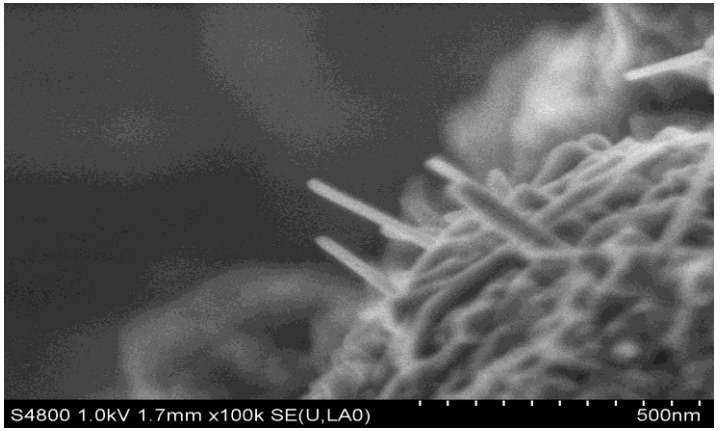
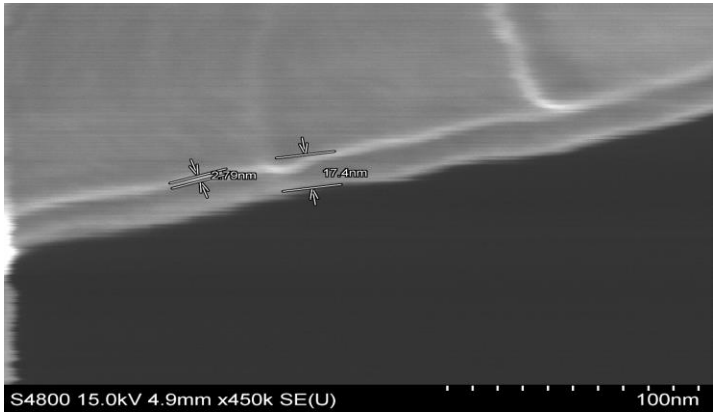
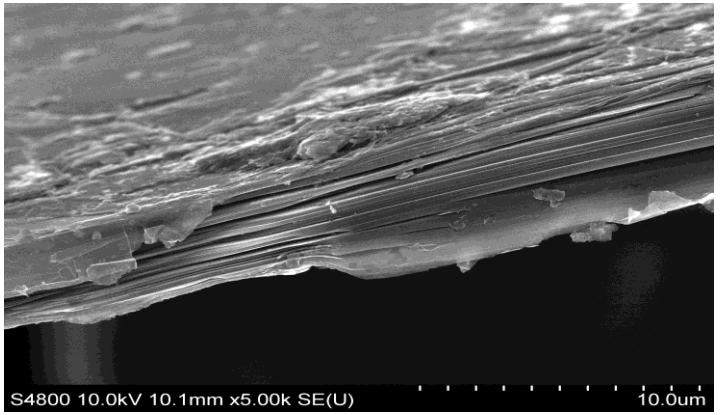
- Low pressure gas plasma generated from controlled gas and vapour mixtures
- Ionised gases (plasma) interact with the CNT / GNP surface , attaching “free radicals”



Mined Natural Graphite



De-agglomeration

	HDPlas™	Industrial
CNTs		
GNPs		

haydale
PART OF THE ICL GROUP

Why Plasma Functionalisation ?

- Correct functionalisation aids dispersion
- Good dispersion optimises properties/performance
- Homogeneous dispersion adds value
- Low temperature processing reduces energy cost
- Plasma processing does not damage material structure
- Haydale processing does not use wet chemistry
- HDPlas materials have no process induced impurities

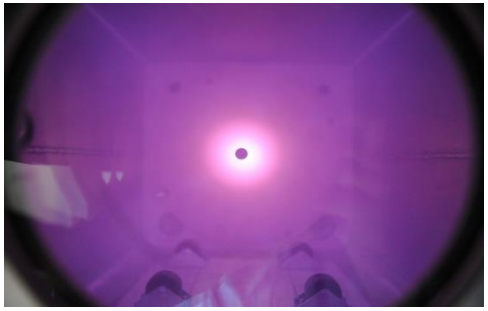
haydale
PART OF THE ICL GROUP

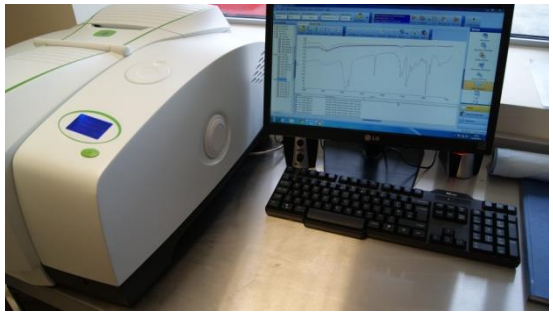
The Haydale Factor : what do we offer ?

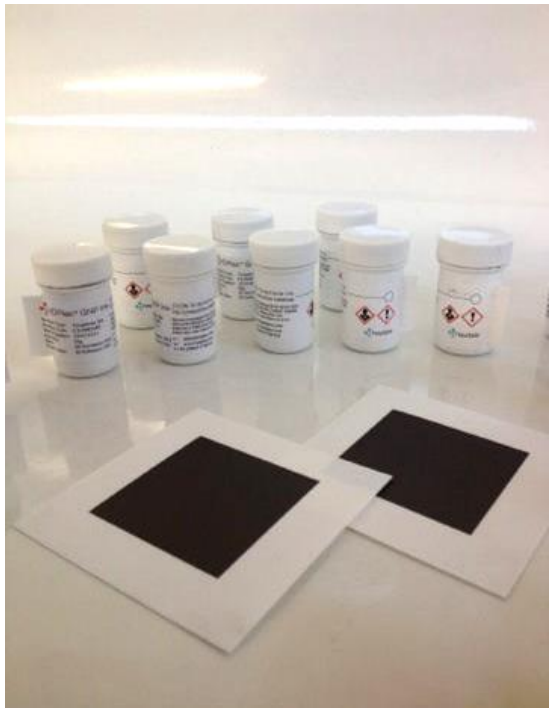
- An energy efficient ‘green’ route to enhance dispersion and thus better utilisation of nano materials.
- A method to functionalise graphite, synthetically produced graphenes and CNTs’ without damaging the structure
- The opportunity to purify and exfoliate the raw CNT and “graphenes”
- A scalable route for the GNP and FLG producer
- Bespoke nano processing in customised facilities (this includes subcontract R&D)

haydale
PART OF THE ICL GROUP

Services







Functionalisation :

- Bespoke plasma treatments
- Application driven development

Analysis :

- Chemical
- Physical
- Mechanical

Prototyping

- Engineering Materials
- Paints and Coatings
- Electrically Conductive Polymers
- Electro-Chemical Components
- Conductive Inks

haydale

PART OF THE ICL GROUP

The Haydale Factor

- No acid processing
- No toxic waste stream
- No post processing drying
- Low temperature processing
- Bespoke materials
- Controlled functionalisation of nano materials

the lean, clean, green, nano processing machine !

haydale
PART OF THE ICL GROUP

Customer comment

"As you know, there is much interest in all things carbon nano, however most of the results I've observed have large scatter with low contributions with regards to mechanical properties. I manufactured 6 loading concentrations for my composites, and I expected to see a degradation or at least a saturation in properties as is usually the case somewhere toward the later half.

I was very surprised to see that your material kept significantly improving both in strength and modulus values. ". US Govt funded researcher

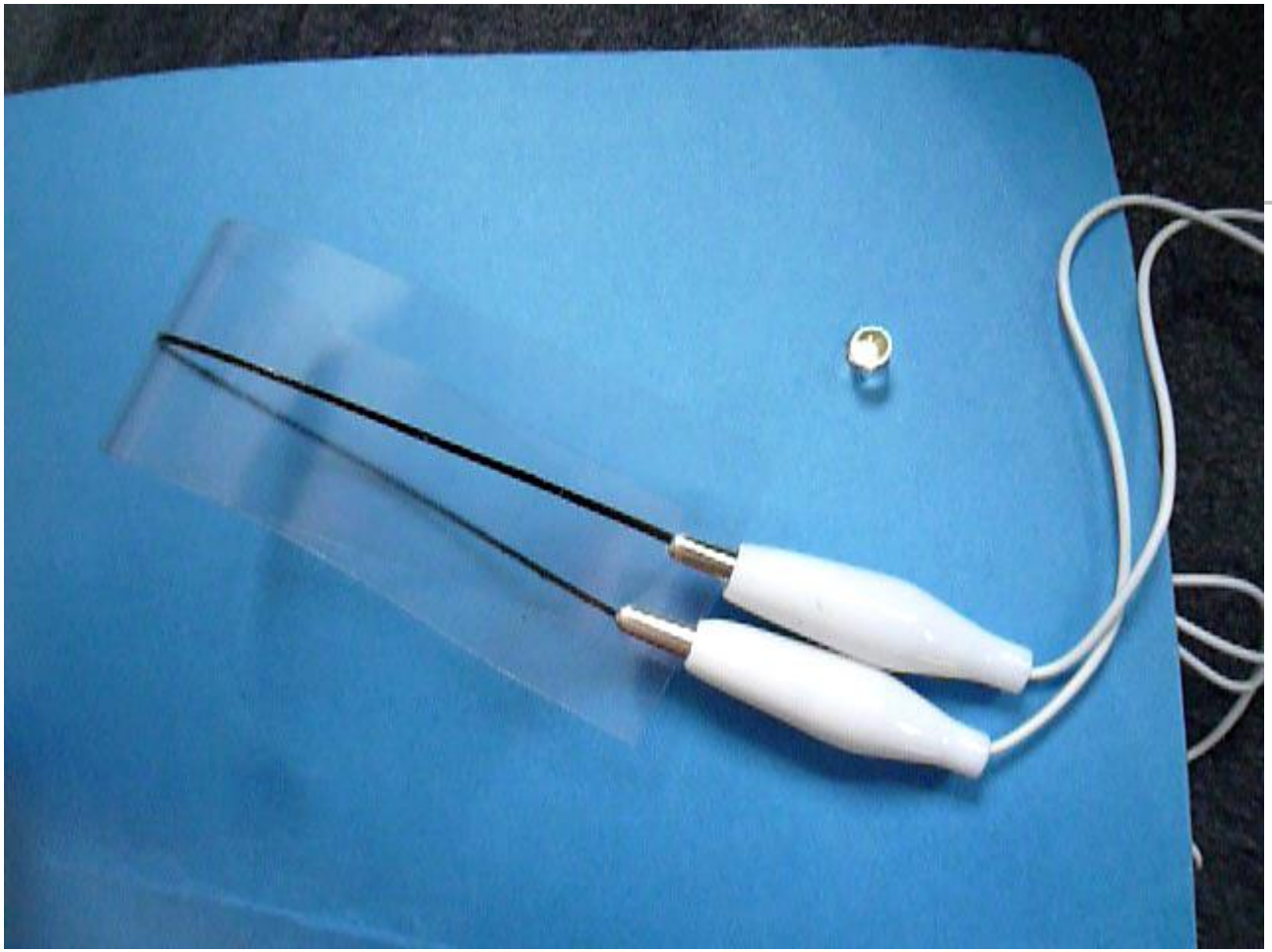
haydale
PART OF THE ICL GROUP

Customer comment

“Our tensile strength and modulus results have been outstanding and increases as a function of loading have shown continuous increases of over 100% at relatively higher loading levels.

Your split plasma method is very efficient with regards to uniformity”.

haydale
PART OF THE ICL GROUP



haydale
PART OF THE ICL GROUP

Thank you for your time !

Diolch Yn Fawr

Ray Gibbs
Haydale Limited
Clos Fferws
Parc Hendre
Capel Hendre
Ammanford
Wales
www.Haydale.com

haydale
PART OF THE ICL GROUP

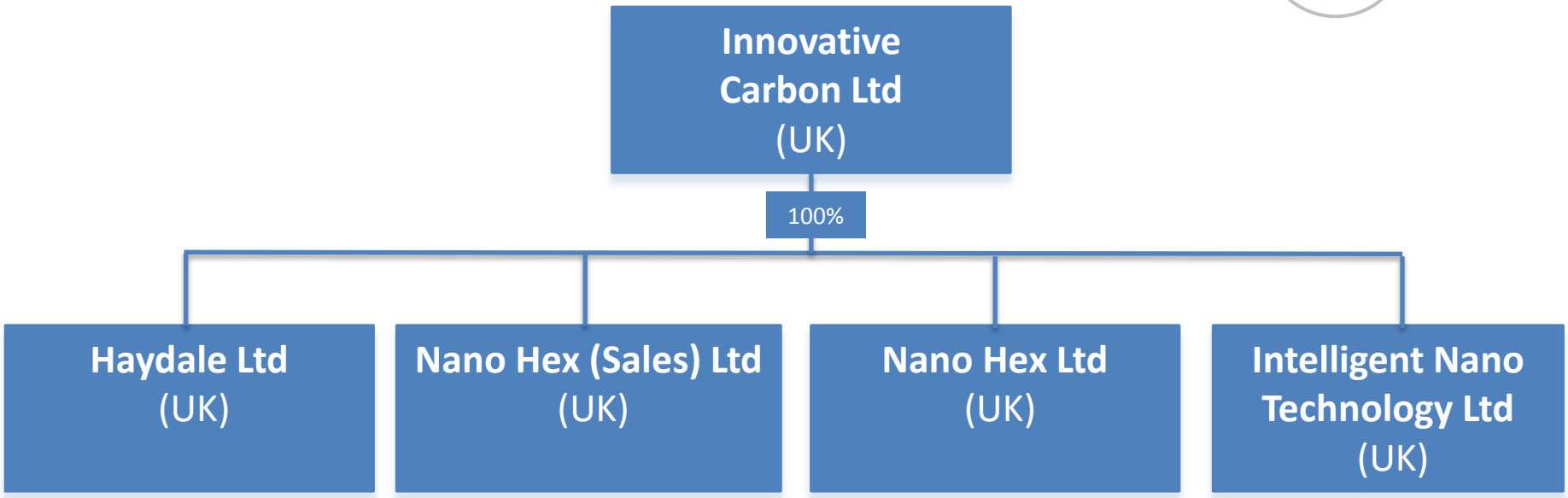
Questions

haydale
PART OF THE ICL GROUP

Appendices

haydale
PART OF THE ICL GROUP

Group structure



Specialist in R&D GNP/Ink sales and operations

HDPlas™ products sold to USA

Dormant

Dormant

haydale
PART OF THE ICL GROUP

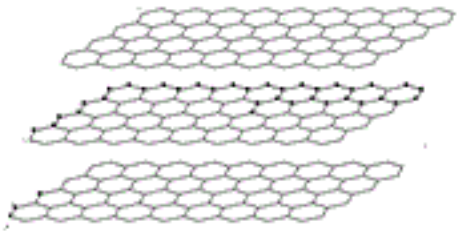
Functionalisation – what and why?

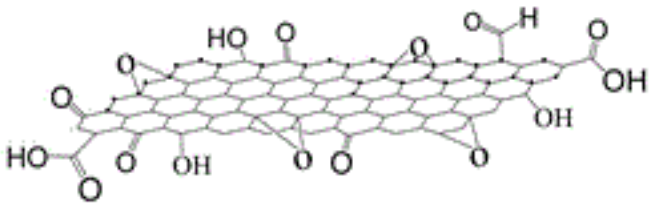
Carbon is its various forms including CNT and GNP is relatively inert.

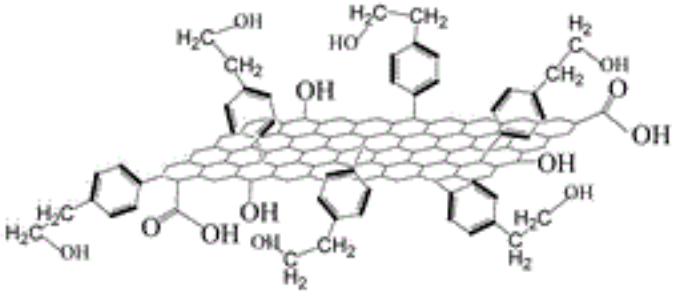
Thus carbon is difficult to bond to and to disperse.

Adding free radicals to the surface of the carbons can :

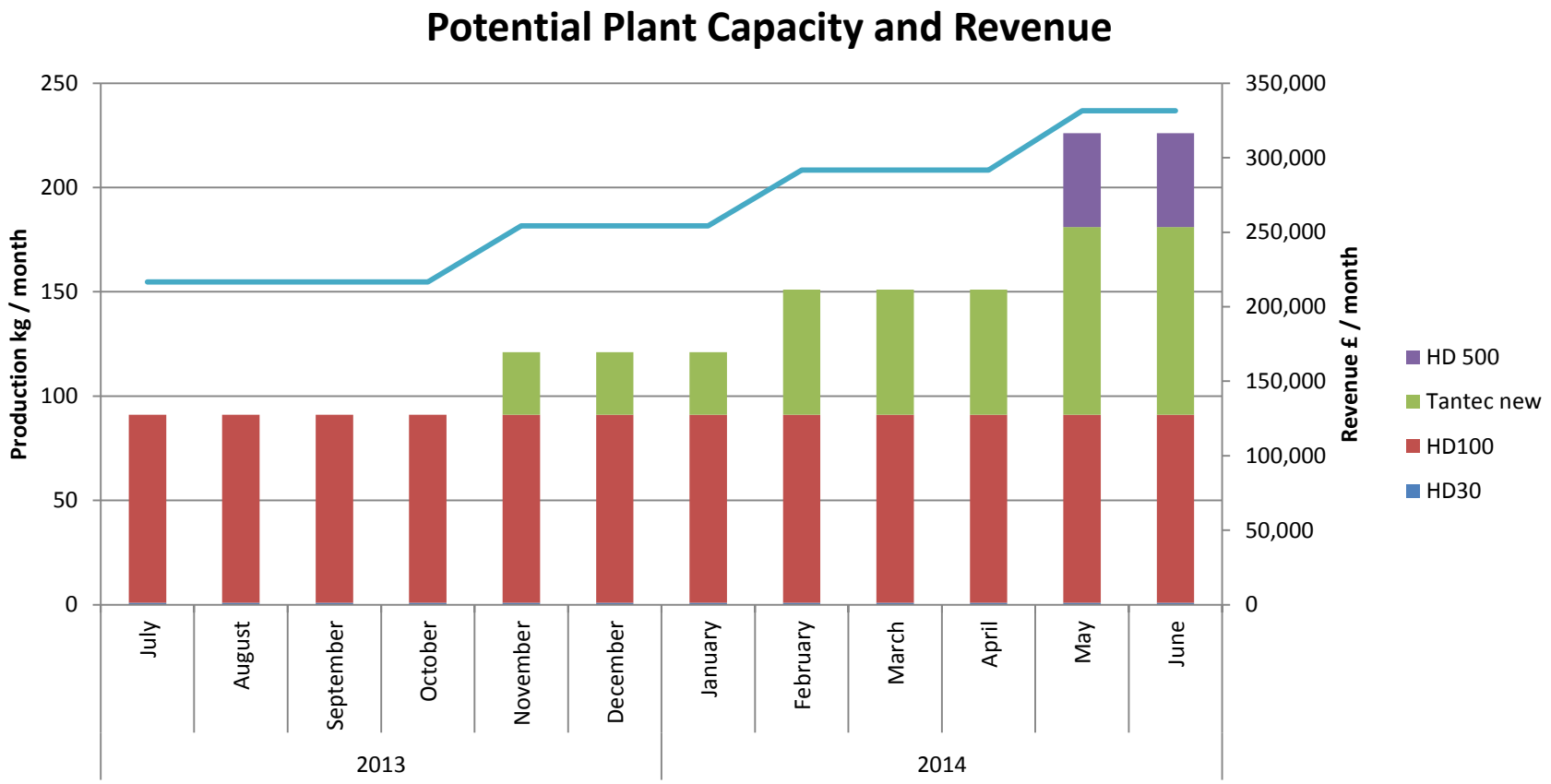
- exfoliate sheets
- enhance particle segregation
- improve dispersion
- enabled tailored interactions



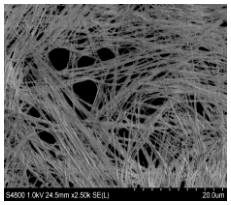




The scale up



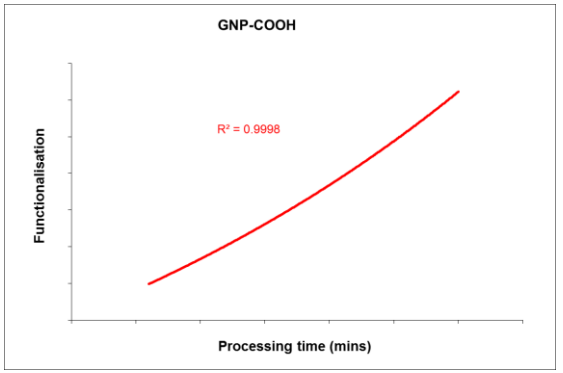
haydale
PART OF THE ICL GROUP

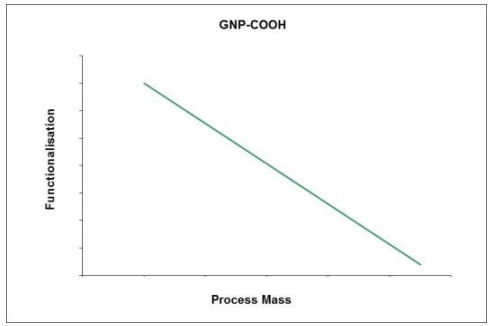


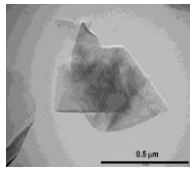
Process Control

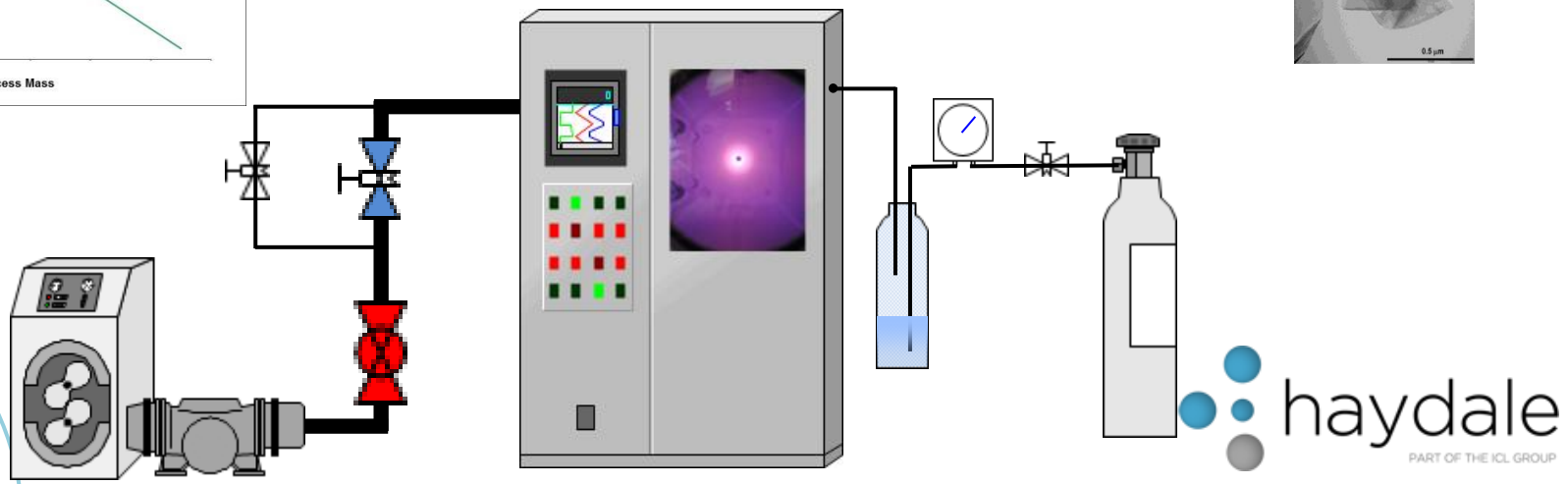
Functionalisation :

- Discrete batches from 25g to 0.5 kg
- Wide range of process gases proven to be effective
- Degree of functionalisation can be controlled
- Material never exceeds 100 °C







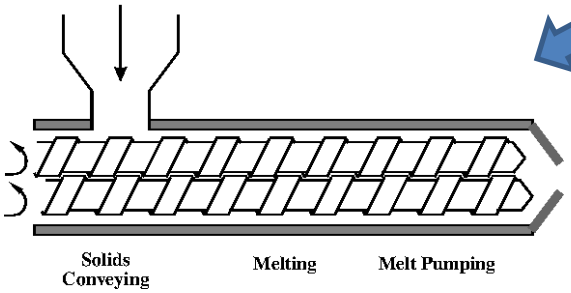


haydale
PART OF THE ICL GROUP

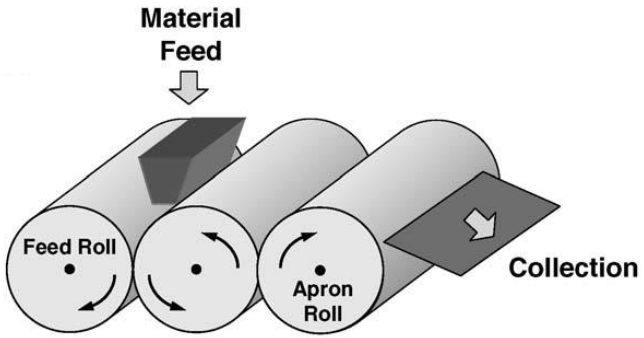
HDPlas™ Materials in Practice

Graphite/CNT Power

Polymer Processing

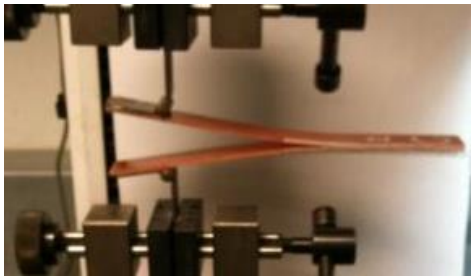


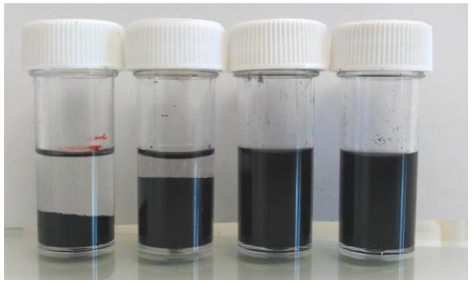
Nano-Enabled Polymer



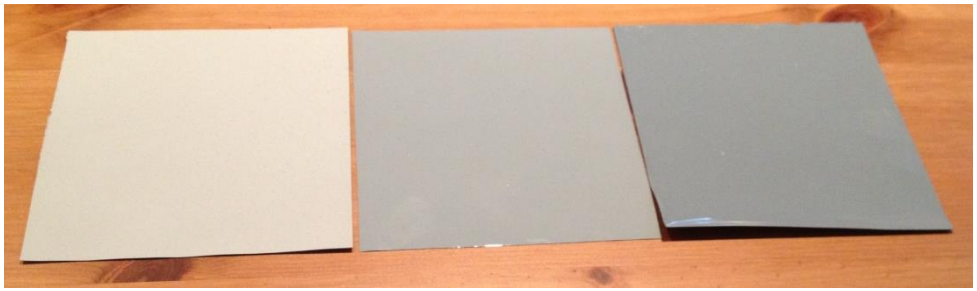
haydale
PART OF THE ICL GROUP

HDPlas™ Materials in Practice



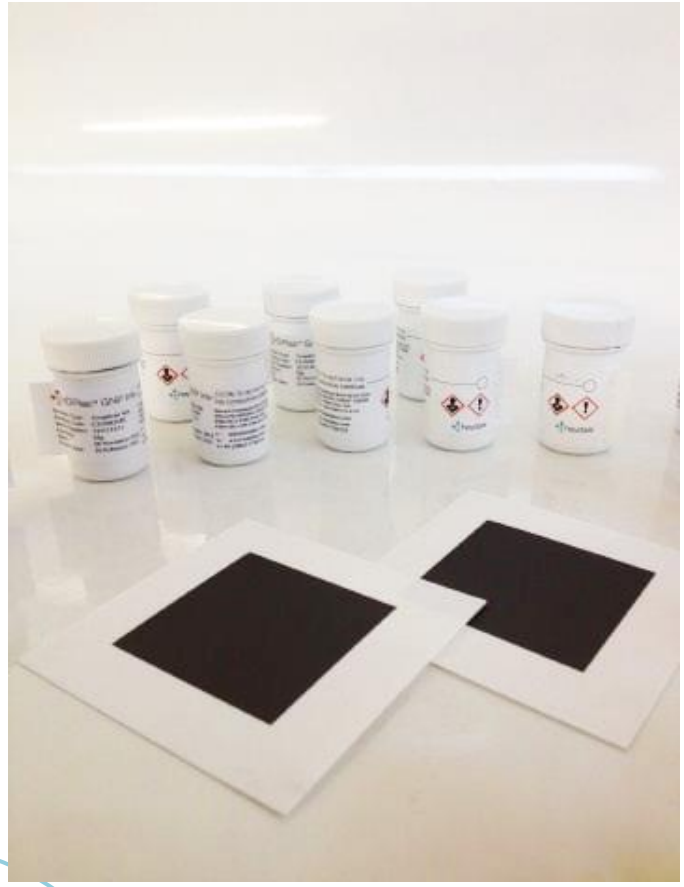


HDPlas™
PART OF THE ICL GROUP



haydale
PART OF THE ICL GROUP

HDPlas™ Graphene Inks



HDPlas™ Graphene Ink Sc213

Screen Printing Conductive Graphene Ink

Solids Content	At 130°C, 90 Minutes	40.0 – 42.0 %
Viscosity	Haake VT550, PK1.1o at 230 sec-1 at 25°C	3.0 – 5.0 Pas
Coverage	Printed through a 230 SS mesh with 13 micron emulsion	1g of ink will cover approximately 350 sq cm
Sheet Resistivity	Printed through a 230 SS mesh with 13 micron emulsion	<10 ohm/sq normalised to 25 micron
Cured Thickness	Printed through a 230 SS mesh with 13 micron emulsion	Typical 12 microns

CUSTOMISATION AND FORMULATION SUPPORT AVAILABLE

haydale
PART OF THE ICL GROUP

Graphene market: overview

- **Market currently limited by supply:**
  - $100m by 2018 and $986.7m by 2022 – estimated, IDTechEX and BCC Research
  - Significant price variance depending on quality and applications
  - Graphene Nano Platelet (GNP) market capacity difficult to establish - estimated at 20 tonnes – growing
- There are two distinct markets for Graphene and GNPs:

Single or few layered Graphene	GNPs
>700m²/gm	Typically 30-100m²/gm
High value, low volume	Lower value, significant volume
Transparent conductive films, sensors, platinum catalyst replacement	Paints, coatings, composites
Eventually Chemical Vapour Deposition competition- synthetic	Mined Graphite- exfoliation

- Competition increasing BUT:
  - **Limitations on dispersions; and**
  - **Use of wet chemistry**

haydale
PART OF THE ICL GROUP

The mechanics of graphene nanocomposites: A review

Robert J. Young[a], Ian A. Kinloch[a], Lei Gong[a], Kostya S. Novoselov

6. Conclusions and prospects

It is clear that both graphene and graphene oxide show promise as reinforcements in high-performance nanocomposites. They have high levels of stiffness and strength and this means that the nanocomposites ought to have outstanding mechanical properties.

There are problems, however, in obtaining good dispersions and there are challenges in obtaining the full exfoliation of graphene into single- or few-layer material with reasonable lateral dimensions o**r producing graphene oxide without imparting significant damage upon the flakes**.

It is also necessary to ensure that there is a strong interface between the reinforcement and the polymer matrix to obtain the optimum mechanical properties.

It should also be remembered that in addition to offering good prospects of mechanical reinforcement there are also possibilities of using graphene to control functional properties such as electrical conductivity, gas barrier behaviour and thermal conductivity, expansion and stability.

haydale
PART OF THE ICL GROUP

HDPlas™ Standard Products

Product	Functionalities	Typical Applications
O_2	Carbonyl [C=O] Hydroxyl [OH] Ether [R-O-R]	Cleaning, activation, etching, reducing undesirable inclusions. Improves compatibility with polar solvents.
Ar	Radicals	Cleaning, activation, pre-cursor for further treatments.
NH_2	Primary Amine [NH2] Amide [RCONH2] Nitrile [C≡N]	Compatibility with a wide range of epoxies, solvent dispersions, electrochemical applications.
N	Nitrous Oxide [NOx] Imine [=C=N] Amines [NHx]	Polymer compatibility, solvent dispersions, surface cleaning, semiconductor doping.
F	Fluorinated Carbon (CF)n	Producing hydrophobic surfaces; aggressive pre-treatment; insulating properties (characteristics similar to Teflon).
COOH	Carboxyl [COOH]	Polymer compatibility, reactive surfaces, pre-treatments.

BESPOKE FUNCTIONALISATION DEVELOPMENT AVAILABLE

haydale
PART OF THE ICL GROUP