Liquid-phase exfoliation of graphite

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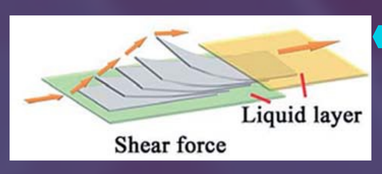
Introduction

Liquid-phase exfoliation (LPE) of graphite is a top-down process to create single layer or few layer graphene. The advantage of this method is the scalability techniques as well as the relatively defect-free graphene compared with most bottom-up synthesis techniques. A vast array of techniques are used, such as ultrasonication, wet ball milling, homogenization, microfluidization and high-shear mixing which is the focus of this poster[1].

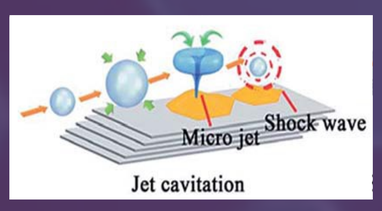
High-shear mixing

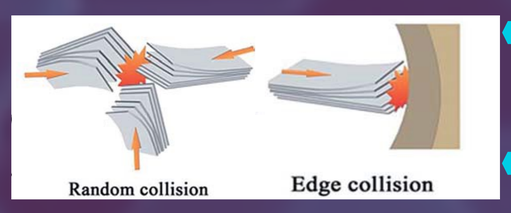
- A high-shear mixer workhead contains two important parts, the rotor and the stator.
- The rotor is a rotating shaft 4 blades on the end, this rotates up to 8000 RPM which results in an end velocity of 12.6 ms^{-1}.
- The stator is a stationary cylinder of metal that surrounds the rotor, the stator can have many different design, such as large circular holes or small square/circular holes which are used for different applications.
- The exfoliation of the graphite occurs between the 100 µm gap between the rotor and stator as this produces a shear rate of ~10^5 s^{-1} ,

Exfoliation mechanism

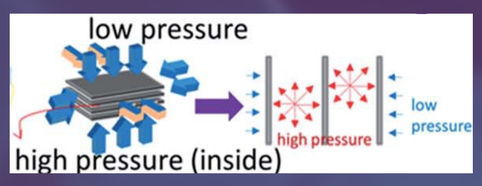


- The primary exfoliation mechanism is due to the viscous shear due to the velocity gradient Exfoliating the graphite with a **lateral** force[2] .
- Jet cavitation causes a **normal force** which sends a shockwave through the layers and exfoliates the graphite[2].
- This can cause defects in the graphene

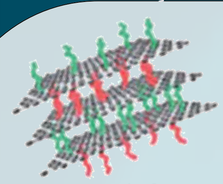


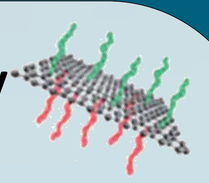


- **Lateral force** graphite-graphite and graphite-edge collision become a considerable problem[2].
- greatly reduces the particle size and damage crystallinity[1].
- Turbulent pressure fluctuations causes a **normal force** pushing the layers apart
- This is more prominent in pressure driven exfoliation methods[3].



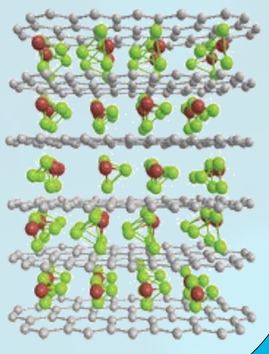
Surface chemistry





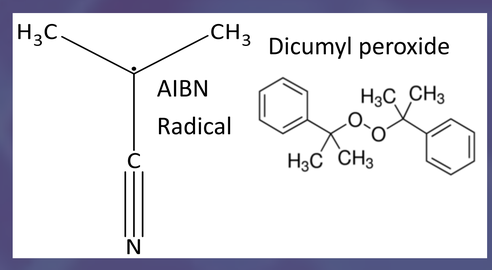
- Van Der Waals forces that hold the layers together need to be overcome for an efficient process.
- NMP has a surface energy of 69 mJ m^{-2} which is close to the 70.5-71 mJ m^{-2} for graphite, this makes a great solvent, but NMP is difficult to remove from the graphene produced[4].

- Water based systems can also be used by the addition of IPA or acetone, or the use of surfactants such as SDS, SBD, PC and polymeric surfactants [2].
- The interlayer spacing is increased with the use of intercalant such as iron chloride or sulfates , however these require extra processing which is costly and time consuming[5].



Current work

- During the process the lateral size of the graphene is also reduced, this makes the edge of the freshly broken graphene more reactive which we have attempted to exploit[6].
- AIBN is broken down into radicals during the shearing process, these radicals can then functionalise the graphene



- However indications show that it is not a radical reaction that is helping stabilise the graphene in the organic solvents
- The interaction of small molecule solvents and the graphene surface could be the interaction that stabilises the graphene

Future work

- Investigate the use of iron chloride intercalated graphite with shear mixing due to it's stability
- Use the material produced from the shear mixer for use in polymer composites with the use of melt extrusion
- Investigate other possible methods for liquid phase exfoliation using higher shear forces

References

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