
GRAM-SCALE PRODUCTION OF GRAPHENE BASED ON SOLVOTHERMAL SYNTHESIS

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Graphene is a monolayer of carbon atoms arranged in a 2-dimensional (2D) hexagonal, honeycomb, lattice. In this respect, the bi-dimensionality does not imply that the lattice has zero inherent depth (the thickness being orthogonal to the graphite plane), rather that the thickness of a single layer corresponds to the thickness of a single carbon atom. The act of exfoliating carbon flakes from bulk graphite by simply tracing the material across a substrate has led to the emergence of free graphene sheets as a physical substance [1,2]. The top-down approaches [1-4] to obtaining graphene have not yet addressed the important tenet that potential graphene based applications will require: large scale production methods. The need for a new process of making graphene is evident in order to allow the technology to progress.

In this paper we report the synthesis of single-layer graphene by chemical means in an atomistic bottom-up approach. The process can be summarised as the low-temperature pyrolysis of a solvothermal product of sodium and ethanol. The product of the pyrolysis reaction is then washed in water and dried to obtain pure graphene. Rapid pyrolysis of the precursor to obtain graphene appears not to be simply combustion of the sodium ethoxide, but rather the ignition of the nano-dispersed ethanol, thermolysing the ethoxide and thus resulting in its decomposition; this then gives rise to fused monatomic sheets of graphene. Due to the small isolated nature of the ethanol ignition points, nucleation of the sheets occurs around those regions rich in ethanol in what we have labelled a 'popcorn effect'.

Multiple cross-sections were recorded of the sample laid onto a mica substrate and a number of step heights were measured across the relief of the sample. The step heights measured between the surface of the sheets and the substrate were consistently found to be 4 ± 1 Å, indicating them to be only a single atom thick. The image shown in Figure 1 highlights a region of the sample in which a narrow ridge of around 10^{-7} m width extends beyond the bulk of the sheet. The profile across this point of the image is fully consistent with those observed elsewhere and clearly shows the narrow valley between the ridge and the neighbouring sheet.

The presence of irregularly shaped sheets is a result of the sonication of the coral-like fused sheets obtained from the pyrolysis stage. Sonication leads to the fragmentation and eventual isolation of free graphene sheets.

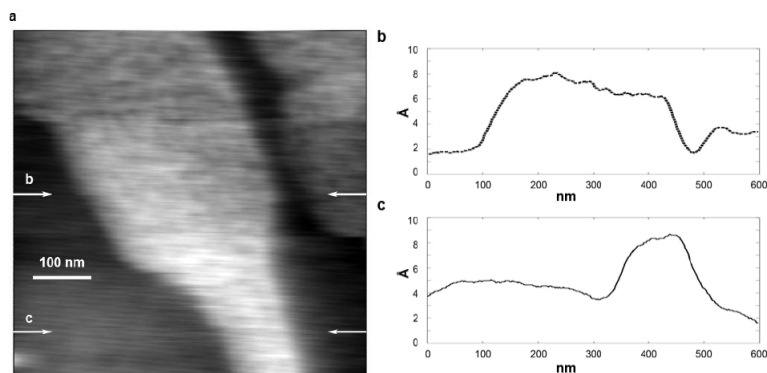


Figure 1: Atomic force microscope (AFM) image of graphene in tapping mode. a, Topography image (600 x 600 nm, height scale: 0-10 Å). b-c, height profiles (600 nm along x-axis) obtained from positions b and c in Figure 1a as indicated by white arrows.

Scanning electron microscopy (SEM) images of the sample before sonication are shown in Figure 2, which illustrates the extensive nature of the graphene produced in the pyrolysis reaction. The whole of the image consists of individual graphene sheets held into a porous structure that typically extends over more than 10-4 m, with the presence of numerous cavities, or holes. As such, the graphene is initially obtained as fused sheets, weakly held into a coral-like structure that are easily separated into individual sheets by sonication in distilled water for several minutes.

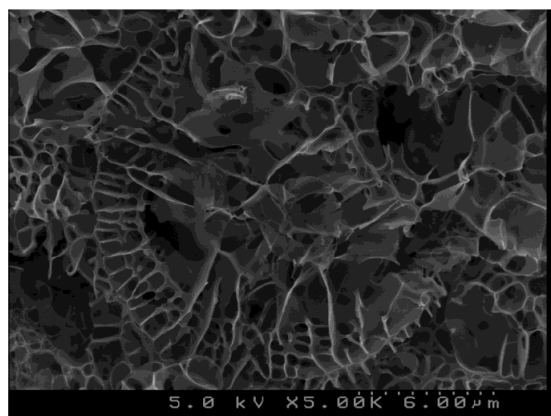


Figure 2: SEM image of the as-synthesised graphene structures. The bulk graphene product obtained from the pyrolysis of the solvothermal product is highly porous, but consists of individual sheets.

By chemically synthesising the material, large quantities of graphene can now be produced in very little time, in a readily up-scalable process that can be environmentally carbon-neutral. We anticipate our work to be a starting point for further advances in the development of new routes to graphene and other carbon nanostructures [5].

References

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