Electronic Supporting Information

Graphene Encapsulated Silicon Carbide Nanocomposites for High and Low Power Energy Storage Applications

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Adiabatic Methodology to Produce Graphene@SiC Nanomaterials

The adiabatic synthetic methodology is a novel single-step method for adiabatic compression that can be used to produce nanoscale materials and compounds of high purity and low particle size polydispersity. The synthetic protocol allows for the mass scale of graphene@SiC nanomaterials.

The adiabatic methodology allows the control of various parameters to produce graphene@SiC nanomaterials with outstanding standard properties of high purity (demonstrated to be 99.99%); the purity depends only on the purity of the precursors and 95% monodispersity, which is to say that less than 5% of particles deviate from the defined diameter. The adiabatic synthetic process is cost-effective, scalable, and a single-step methodology.

The adiabatic method is based on the concept of obtaining graphene@SiC nanopowders by initiating the synthesis of the target products via rapid and uniform temperature rise throughout the volume of a reactor filled with a mixture of gaseous precursors. The method requires neither the use of expensive high-tech equipment (such as laser machines, torches, microwave generators, etc., as used in other methods) nor service by highly qualified personnel. The simplicity of the equipment as well as its easy maintenance, low energy costs, and the use of a single-step process lead to lower production costs compared to alternative technologies, and greatly enhanced the process scalability.

Fabrication and Electrochemical Characterisation of Screen-Printed Electrodes

The values of the heterogeneous electron transfer rate constant, $k^e$, were determined by the Nicholson method through the use of the following equation: $\psi = k^e(\pi DnF/(RT))^{-1/2}$, where $\psi$ is the kinetic parameter, $D$ is the diffusion coefficient, $n$ is the number of electrons involved in the process, $F$ is the Faraday constant, $R$ is the universal gas constant, and $T$ is the temperature. The kinetic parameter, $\psi$, is tabulated as a function of $\Delta E_r$ (peak-to-peak separation) at a set temperature (298 K) for a one-step, one electron process with a transfer coefficient, $\alpha$, equal to 0.5. The function of $\psi$ ($\Delta E_r$), which fits Nicholson’s data, for practical usage (rather than producing a working curve) is given by:
\[ \psi = (-0.6288 + 0.0021X)/(1 - 0.017X), \]
where \( X = \Delta E_r \) is used to determine \( \psi \) as a function of \( \Delta E_r \) from the experimentally recorded voltammetry. From this, a plot of \( \psi \) against \([\pi D_m F/(RT)]^{1/2}\) allows the \( k^0 \) to be readily determined. The heterogeneous electron transfer rate constants were calculated, assuming a diffusion coefficient of \(9.10 \times 10^{-6} \text{cm}^2 \text{s}^{-1}\) for 1 mM hexaammineruthenium (III) chloride/0.1 M KCl.

The screen-printed graphite electrodes (SPEs) utilised throughout this work consisted of a graphite working electrode, a graphite counter electrode, and an Ag/AgCl reference electrode. The SPEs, which have a 3-mm diameter working electrode, were fabricated in-house with appropriate stencil designs using a microDEK 1760RS screen-printing machine (DEK, Weymouth, UK). This SPE design has been previously reported. For experimental continuity, the SPE’s on-board Ag/AgCl reference and carbon counter electrodes were removed and replaced with an external SCE reference and Pt counter electrodes, respectively. The SPEs have been electrochemically characterised previously, and exhibit a heterogeneous electron transfer rate constant, \( k^0 \) of ca. \(1.08 \times 10^{-3} \text{cm s}^{-1}\) using 1 mM hexaammineruthenium (III) chloride/0.1 M KCl. The reproducibility and repeatability of the fabricated batches of electrodes were explored through comparison of cyclic voltammetric responses using a 1 mM hexaammineruthenium (III) chloride/0.1 M KCl supporting electrolyte. Analysis of the voltammetric data revealed the % relative standard deviation (% RSD) to correspond to no greater than 0.82 % \((N = 20)\) and 0.76 % \((N = 3)\) for the reproducibility and repeatability of the fabricated SPEs.

**Figure S1.** Electron diffraction image of n-SiC core in graphene@SiC.
Figure S2. SEM images of the graphene@SiC nanocomposite (A,B) and 5 µg graphene@SiC/SPE (C,D) at magnifications of ×250 and ×20 k, respectively.

Figure S3. Cyclic voltammograms within a 0.1 M H₂SO₄ utilising a graphene@SiC/SPE, Nafion 5% SPE, and unmodified SPE. Scan rate: 50 mV·s⁻¹.
Figure S4. Cyclic voltammograms (vs. Li/Li⁺) of the graphene@SiC nanomaterial within a Li-ion cell. Scan rate: 0.1 mV·s⁻¹.

References

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