

## Supplementary Materials for

### **Vertical graphene and nanodiamond composited films prepared by monodispersed molybdenum atoms loading and annealing**

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**Fig S1**

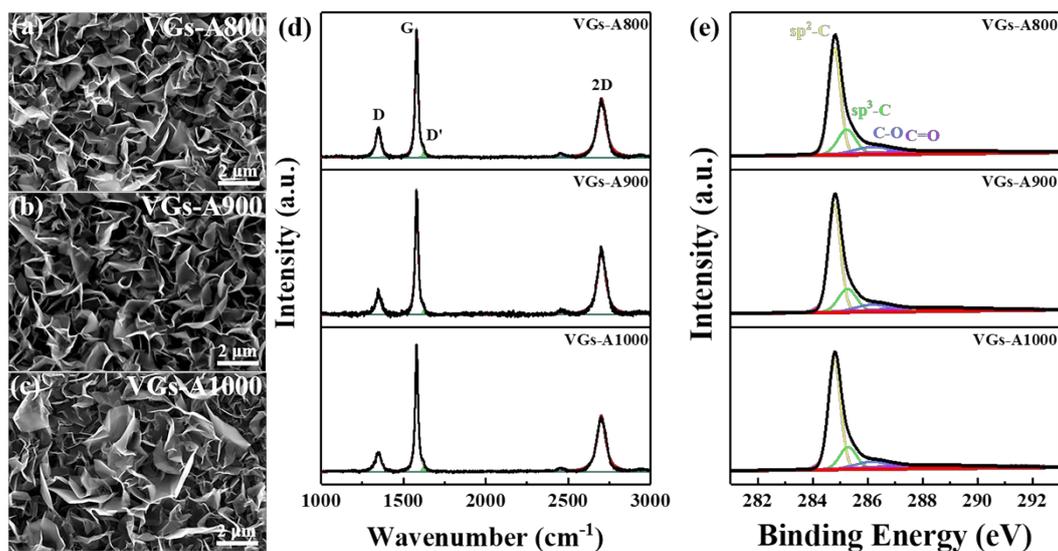


Fig. S1. SEM images of (a) sample annealed at 800 °C, (b) sample annealed at 900 °C and (c) sample annealed at 1000 °C, and their (d) Visible Raman spectra as well as (e) the XPS results of C 1s core-energy level spectra.

Fig S1(a-c) shows that after annealing at 800-1000 °C, the surface morphology of the samples did not undergo significant changes compared to VGs-UA. It still consists of uniformly vertical sheets, and the vertical sheets are supported by each other to form a self-supporting network structure.

The Raman spectra in Fig. S1d show that there are four characteristic Raman peaks, at 1350 cm<sup>-1</sup> (D-peak), 1580 cm<sup>-1</sup> (G-peak), 1620 cm<sup>-1</sup> (D'-peak) and 2700 cm<sup>-1</sup> (2D-peak), after annealing treatment, indicating that the main component of the samples is graphene. That is, VGs films have been successfully prepared. Fig S1e shows the XPS results of the samples. The C 1s core-energy level spectra of all the samples can be deconvoluted into four peaks attributed to sp<sup>2</sup>-C, sp<sup>3</sup>-C, C-O and C=O bonds, respectively. sp<sup>3</sup>-C peak area did not change significantly after the annealing treatment at 800-1000 °C, indicating that the sp<sup>3</sup>-C bonds did not increase significantly after the annealing at 800 -1000 °C.

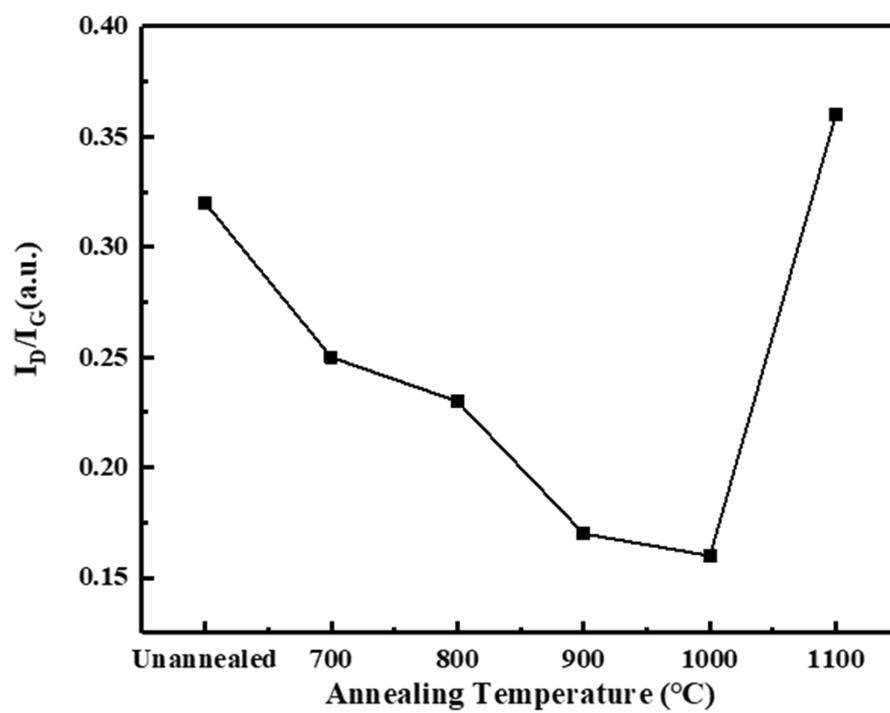


Fig. S2 Dependence of the value of  $I_D/I_G$  on annealing temperature

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**Fig S2**

**Table S1** Corresponding parameters for a sweep of 100 mV/s in Fig. 5

| Sample Name | Hydrogen precipitation potential(V) | Oxygen removal potential(V) | Potential window (V) | Background current (mA/cm <sup>2</sup> ) | Electrochemically active area (μC/cm <sup>2</sup> ) |
|-------------|-------------------------------------|-----------------------------|----------------------|--|---|
| VGs-UA      | -1.41                               | 1.57                        | 2.98                 | 23.96                                    | 721.93  |
| VGs-A 700   | -1.67                               | 1.77                        | 3.44                 | 6.13                                     | 282.85  |
| VGs-A 1100  | -1.56                               | 1.48                        | 3.04                 | 28.97                                    | 1617.44   |