

Electronic Supplementary Information

The Facile Synthesis of Graphene Nanoplatelets and Lead Styphnate Composites for the Depressed Electrostatic Hazards

Zhi-Min Li, Ming-Rui Zhou, Tong-Lai Zhang*, Jian-Guo Zhang, Li Yang and Zun-Ning Zhou

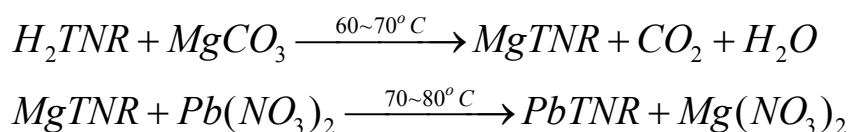
State Key Laboratory of Explosion Science and Technology, Beijing Institute of Technology, Beijing 100081, P. R. China

Fax & Tel.: +86-10-68911202

Email: ztlbit@bit.edu.cn (Tong-Lai Zhang)

Section S1 Preparation of the samples

LS was prepared by reaction of magnesium styphnate (MgTNR) with lead nitrate, the synthetic route of the title compound shows as fellow.



GLS(I):

Graphene nanoplateles was added to the solution of $Pb(NO_3)_2$, ultrasonic dispersion for 10 min, then pour into glass reactor with strong stirring. Solution of MgTNR was dropwise added. Gray or black GLS precipitation was obtained. After filter, washing and dry, GLS(I) products were obtained.

GLS(II):

5 g normal LS was added to 100 mL deionized water in the glass reactor with well stirred at 60 °C. GNP was added to 40 mL ethanol in a small beaker, and ultrasonic dispersion for 15 min. 2.5 mL shellac ethanol solution as the adhesive (3%) was dropped into the reactor slowly in 15 min. After that, GNP dispersed in ethanol was pour into the reactor with strong stirring at 60 °C for 10 min. GLS(II) was obtained after filter, washing and dry.

Section S2 Characterization

Field-emission scanning electron microscope (FE-SEM) images and EDS data of the samples were taken on JSM-7001F SEM unit. X-ray diffraction (XRD) patterns were obtained by using a D8 ADVANCE X-ray diffractometer (Bruker, Germany). Raman spectra were recorded using a RM 2000 Microscopic Confocal Raman Spectrometer (Renishaw PLC, England) with an Ar laser at a wavelength of 514.5 nm.

Section S3 Conductivity

The surface resistance and volume resistance were tested by applying a digital high resistance/micro-current measuring instrument (EST121) with a three electrodes mould. Fig.S1 is the schematic diagram of three electrodes mould.

Surface resistivity and volume resistivity were obtained by the following equations:

$$\rho_s = \frac{2\pi}{\ln \frac{D_2}{D_1}} R_s \quad \rho_v = R_v \frac{S}{b}$$

Where ρ_s is surface resistivity, ohm (Ω); R_s is tested surface resistance, ohm (Ω); D_1 is the diameter of measuring electrode; D_2 is the diameter of shield electrode. ρ_v is volume resistivity, ohm

centimeter (Ω cm); R_v is tested volume resistance, ohm (Ω); S is area of measuring electrode, square centimeter (cm^2); b is the thickness of sample, centimeter (cm).

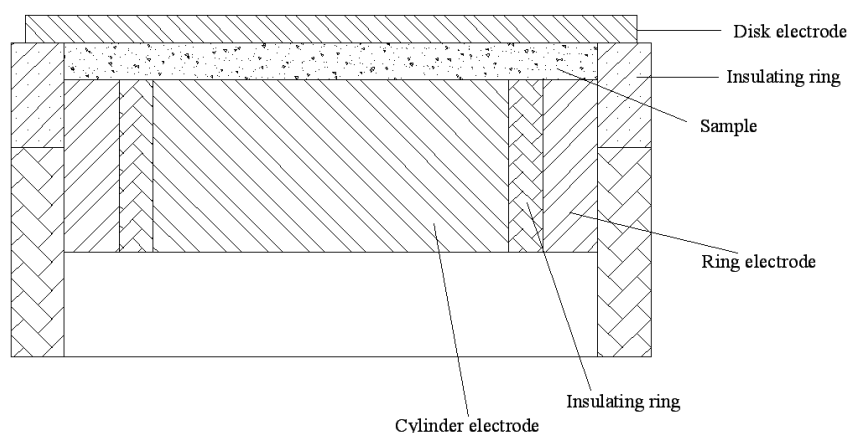


Fig. S1 Schematic of three electrodes sample mould.

Section S4 Electrostatic spark sensitivity

The schematic diagram of electrostatic spark sensitivity tester used in this work is shown in Fig. S2. The charging circuit is mainly constituted by a high voltage power generator and a bank of capacitor. When the capacitor is loaded, the vacuum discharge switch insulates them from the charging circuit and connects them to the discharge circuit, which containing the ignition device through the needle electrode. The sample is placed in a holder which is secured to the ground base electrode. The electrode gap is measured with a dial gauge.

Electrostatic power generator (EST806F), charging condenser (500pF), vacuum discharge switch, the electrode gap length is 0.12 mm, negative charge. The test energy was taken to be the energy stored on the charged capacitor, was given by the formula $E=0.5CV^2$. Where C is the capacitance of the capacitor, farads (F); V is charge voltage, volt (V). A series of 25-30 samples was tested using the up and down method for each condition, and the electrostatic spark sensitivity (E_{50}) for 50 % probability of ignition was calculated according to the usual Bruceton formula.

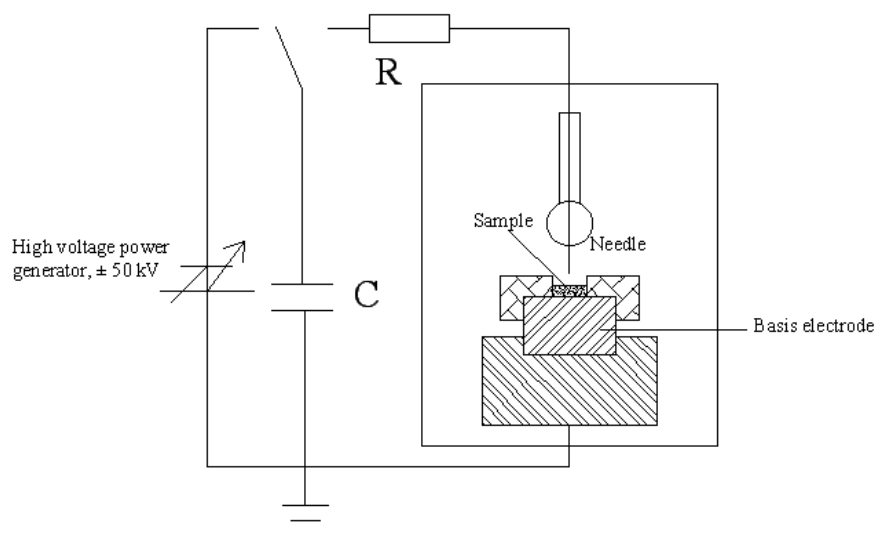


Fig. S2 Schematic of the electrostatic spark sensitivity tester.

Section S5 Static electricity accumulation

The static electricity accumulation testing system is constructed as Fig. S3 in our laboratory. A digital charge meter (EST111) connected to the Faraday canister was used to measure the charge of the sample flow into the Faraday canister. When flowing out from the flume and into the Faraday canister, the static voltage of the sample was measured by a voltage probe connected to the vibration capacitor electrometer (EST102). Below the Faraday canister, there is a digital balance which can continuously test the weight of the samples flowed in the Faraday canister. Signals of charge, voltage and mass were input a computer data gathering and processing system (EST406). And then, the continuous data were collected and processed in the computer. 5 g sample was placed in the sample box, then be poured into the flume and flow through it. Sample charged in the process of friction with the flume and flow into the Faraday canister. Continuous data of static charge (Q), voltage (U), as well as weight (W) of the testing sample were obtained. Various flumes were used in this work. To maintain the accuracy of the obtained static electricity accumulation, eight tests were carried out in each condition, and averaged.

The length of flume is 60 cm, and the angle of inclination is 45 degree. 5g samples were applied to test the static electricity accumulation of the samples.

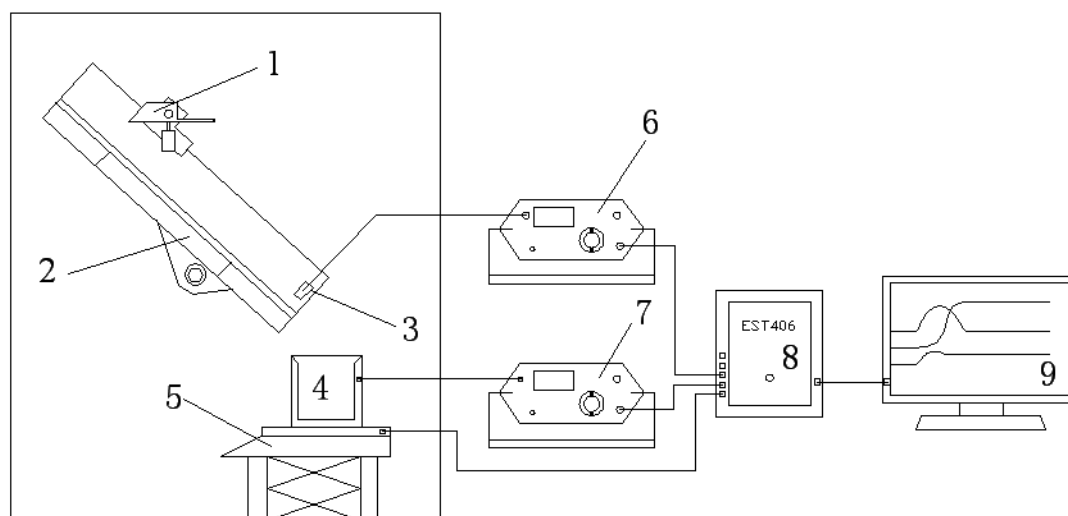


Fig. S3 Schematic of the static electricity accumulation testing system.

1–sample box; 2–flume; 3–voltage probe; 4–Faraday canister; 5–digital balance; 6–vibration capacitor electrometer; 7–digital charge meter; 8–computer data gathering and processing system; 9–computer

Section S6 DSC and 5s delay explosive temperature

Differential scanning calorimeter (DSC) (Pyris-1, Perkin-Elmer, USA) was tested with the heating rate of $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$ from $50\sim 600\text{ }^{\circ}\text{C}$, sample (about 0.2 mg) was placed in platinum sample pans, using dry nitrogen as atmosphere with a flow rate of $20\text{ mL}\cdot\text{min}^{-1}$. The 5s delay explosive temperature (T_5) was measured with a copper detonator shell, wood's alloy bath, 20 mg sample mass.

Section S7 Impact sensitivity, friction sensitivity and flame sensitivity

The impact and friction sensitivities as well as the flame sensitivity were determined on the basis

of the China National Military Standard¹. Impact sensitivity (50 % firing height, $H_{50}(I)$ /cm), measured by Bruceton staircase method, 0.8 kg drop hammer, 20 mg sample mass. Friction sensitivity (probability of explosion, P /%) was determined with a MGY-1 pendular friction sensitivity apparatus by following a standard procedure using 20 mg sample. When sample was compressed between two steel poles with mirror surfaces at the pressure of 1.23 MPa, and additionally was hit horizontally with a 1.5 kg hammer from 70° angle. Flame sensitivity (50 % firing height, $H_{50}(F)$ /cm), 20mg sample was compacted into a copper cap under 58.8 MPa and was ignited with a standard black powder pellet.

References

1. Z. T. Liu, Y. L. Lao, Initiating Explosive Experimental, Beijing Institute of Technology Press, Beijing, 1995, p. 238.