A novel energetic composite with a special sandwich microstructure: RDX/expanded graphite intercalation composite

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Abstract. In this paper, the RDX/expanded graphite (EG) intercalation composites were prepared by solvent/anti-solvent process to recrystallize RDX crystals into the holes, gaps and grooves of EG with controllable embedding ratio (maximum 87.0 wt%) and crystal size. RDX in the composites was in the most stableα-phase, which was benefit for its further application in the military industry. The stability and sensitivity of RDX were improved with higher melting and thermolysis temperature, activation enthalpy (ΔHf), critical temperature of thermal explosion (Tc) and lower mechanical sensitivity. The advanced half-coated parallel multi-sandwiches microstructure of RDX/EG intercalation composites possessed two effects: (i) high heat conduction and (ii) hot spots isolation of the carbon microstructure, which were the key to higher performance.

1. Introduction

As the energy source of weaponry to fire, launch, actuate, motorize, explode and destroy, energetic materials (EMs) have received great attention from both militaries and scientists. Nowadays, some novel EMs with high density, decent thermal stability, positive heat of formation and excellent detonation properties have been synthesized successfully and show a great promise for potential applications in the military industry [1-6]. Besides higher performance, the safer properties of EMs have of equal importance in the field of EMs, especially the stabilization and desensitization of EMs are essential for their practical applications [7-8]. Recently, a variety of carbon materials have been developed as carriers of EMs to form advanced structures with higher performances. Some of them, especially graphene [9,10], graphene oxide (GO) [11-15], reduced graphene oxide (rGO) [16], carbon nanotubes (CNTs) [17] and their composites, as coating or encapsulating materials, are reported as effective choice in improving thermal stability and decreasing sensitivity of energetic systems. Theoretical studies have shown that the encapsulation of EMs molecules or crystals in carbon...
nanostructure can improve their stability because of the interaction between molecules and carbon wall [18-19].

Among carbon materials, expanded graphite (EG) has porous worm or vermicular appearance in nature and possesses outstanding properties including lower percolation threshold, good electrical conductivity [20-25] and thermal conductivity [26-33], remarkable thermal stability, [34-36] mechanical strength, large specific area, low density, special 3D structure (full of holes, gaps, grooves on its matrix) [37] and the ability to absorb organics [38,39]. In addition, compared to graphene, GO, rGO and CNTs, the relatively much lower price of EG facilitates its further scale-up preparation and applications. Considering such many outstanding properties of EG, we employed the solvent/anti-solvent process to recrystallize RDX crystal into the holes, gaps and grooves of EG to form a half-coated parallel multi-sandwiches microstructure with improved stability and sensitivity.

The prepared RDX/EG intercalation composites were characterized by scanning electron microscope (SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), infrared spectroscopy (IR), Raman, thermo-gravimetry analysis (TG), differential scanning calorimeter (DSC) and sensitivities tests to get the information of microstructure, crystal phases, chemical compositions, thermal behavior and sensitivities. SEM images confirmed that RDX crystal was intercalated into EG successfully and showed the effect of preparation conditions on the intercalation process. The embedding ratio of RDX in composites could be adjusted by varying preparation conditions and the maximum was 87.0 wt%. IR and XPS analysis revealed that oxygen-containing groups and H₃PO₄ anion were remained on the surface of EG, which promoted the interaction and adsorption between the EG surface and RDX molecules. Raman and XRD analysis showed that RDX in the composites was in the most stabler-phase. Compared to raw RDX, the RDX/EG intercalation composites showed improved stability and sensitivity due to their special structure with higher activation energy \( E_K \), thermolysis rate constant \( k \), activation enthalpy \( \Delta H^* \), activation entropy \( \Delta S^* \) and critical temperature of thermal explosion \( T_k \). The advanced half-coated parallel multi-sandwiches microstructure possessed two effects: (i) high heat conduction and (ii) hot spots isolation of the carbon microstructure, which were the key to higher performance. To the best of our knowledge, this is the first report of energetic composites based on EG material.

2. Materials and methods

2.1. Materials

The EG was obtained by a modified sulfur-free method to reduce the cauterization to the ammunition considering its further appalement in the military industry. Natural flake graphite, potassium permanganate, nitric acid, phosphoric acid and acetic anhydride were obtained from J & K. Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) was obtained from Gansu Yinguang Chemical Group Co., Ltd. All the raw materials were of analytical grade and used as received without further purification. SEM images of raw materials EG and RDX were shown in figure A1. As shown in figure A1, EG exhibited a worm-like cylinder and was full of holes, gaps and grooves on its matrix. RDX used as raw material was the type 5 and 325 mesh with ellipsoidal morphologies from figure A1c.
2.2. Preparation of sulfur-free EG

Typically, KMnO₄ (6.0 g) was dissolved into the mixed liquid consisting of HNO₃ (75.0 mL), H₂PO₄ (225.0 mL) and acetic anhydride (18.0 mL). Then natural flake graphite (30.0 g) was added to the solution and the reaction was carried out at 45°C for 80 min under continuous stirring to form expandable graphite. Afterwards, the resulting mixture was carefully washed and filtrated with deionized water to neutral. After being dried at 80°C in a vacuum oven for 24 h, the expandable graphite was put into a quartz crucible and heated in a muffle furnace under 900°C for 1 min to produce EG.

2.3. Preparation of RDX/EG Intercalation Composites

The solvent/anti-solvent process was employed to prepared RDX/EG intercalation composites as following steps. Firstly, EG was dispersed in RDX solution and soaked for about 1 h to absorbing the solution fully, followed by the removal of excess solution outside of EG. Then, the filter residue was poured into deionized water rapidly with the agitation for about 5 min. After the solvent/anti-solvent process, the production was filtered out and washed with deionized water to remove the remaining RDX crystal on the surface of EG. Finally, the sample was dried at 60°C to the constant weight.

2.4. Characterizations

2.4.1. The embedding ratio of RDX in the intercalation composites

In order to investigate the embedding ratio of RDX in the intercalation composites, the thermo-gravimetry (TG) curves of composites was measured assuming that RDX decomposed into gas phase completely and EG didn’t lose weight during heating in TG test.

2.4.2. Other Characterizations

The morphologies of raw EG, RDX and RDX/EG intercalation composites were characterized by scanning electron microscopy (SEM, TESCAN, S8000). Raman spectroscopy of the samples were recorded on a Raman spectrometer (RAMAN, Renishaw, inVIA). The crystal structures and chemical compositions of the samples were determined by X-ray diffractometer (XRD, Panalytical, Xpert Powder) and Fourier Transform infrared spectroscopy (FTIR, Bruker, ALPHA II), respectively.

The thermal behaviors of raw RDX and RDX/EG intercalation composites were carried out by thermo-gravimetry analyzer (TG, SHIMADZU, DTG-60A) and differential scanning calorimeter (DSC, NETZSCH, DSC 200 F3). Each experiment was accomplished by heating about 1.0 mg sample in an aluminium sample cell under nitrogen flow (50 mL·min⁻¹).

The impact sensitivity was tested by using the WL-1 drop hammer apparatus. According to GJB-772A-97 601.2 drop height (H₅₀, cm) method, a mass of 35.0 mg sample was impacted by 5.0 kg hammer from various heights using an up and down method. The impact sensitivity was expressed by H₅₀ which is the height of 50% probability of the sample initiation. Therefore, the higher H₅₀ means lower impact sensitivity. The friction sensitivity was recorded by using MGY-1 pendulum friction apparatus. Refer to GJB-772A-97 602.1 explosion probability method. A mass of 20.0 mg sample was fixed between steel anvils and hit by a 1.5 kg pendulum hammer. The experiment tested twenty-five times below 90° tilt angle and 3.92 MPa pressure. The Friction sensitivity was expressed by explosion
The higher $P_F$ is, the more sensitive appears. To get the reliable data, friction sensitivity tests were carried out twice and the average values were recorded as results.

3. Results and Discussion

To investigate the effect of preparation conditions such as volume ratio of solvent to anti-solvent and concentration of solution on intercalation, the preparation conditions were systematically varied.

3.1. Effect of volume ratio of solvent to anti-solvent on intercalation

![Figure 1](image.png)

Figure 1. SEM images of RDX/EG intercalation composites prepared under different volume ratio of solvent to anti-solvent conditions: (a) 1:2; (b) 1:6; (c) 1:10; (d) 1:20.

The effect of volume ratio of solvent to anti-solvent was studied by varying volume ratio from 1:2 to 1:20 and keeping the RDX solution concentration constant (saturation). SEM images of RDX/EG intercalation composites prepared under different volume ratio of solvent to anti-solvent conditions were shown in figure 1.

The particle size was larger and lots of huge crystal that even could be seen with naked eyes was found to attach on the surface of EG when the volume ratio was 1:2 (figure 1a). When the volume ratio was enhanced to 1:6, 1:10 and 1:20 (figure 1b, c, d), the particle size became smaller and no more the huge crystal appeared due to the shorter induction time and faster nucleation rate. Considering saving anti-solvent and dealing with less recycling liquid, the volume ratio of solvent to anti-solvent was determined to be 1:6. All further experiments were performed at this ratio.

3.2. Effect of concentration of RDX solution on intercalation

The concentration of RDX solution was varied systematically from 0.1 mol·L$^{-1}$ to saturation. SEM
images of RDX/EG intercalation composites prepared under different RDX solution concentration conditions were shown in figure 2.

![Figure 2](image_url)

**Figure 2.** SEM images of RDX/EG intercalation composites prepared under different RDX solution concentration conditions: (a, b) 0.10 mol·L⁻¹; (c, d) 0.90 mol·L⁻¹; (e, f) saturation.

It could be found that the quantity of RDX crystal intercalating into EG increased with the increase of solution concentration obviously. When the concentration was 0.10 mol·L⁻¹, only tiny amounts of crystal attached at the graphite sheets of EG, the holes, gaps and grooves of EG were empty (figure 2a, 2b). When the concentration was increased until to the saturation, RDX occupied the space intimately (figure 2d-2e). In addition, graphite sheets stick to each other at the central axis constructing the EG worm. The RDX crystal intercalating into EG was oval shape with smooth surface and about 10 µm in major axis under the rapid crystallization process. To obtain the embedding ratio, thermo-gravimetry curves of EG and RDX/EG intercalation composites prepared under the different solution concentration conditions were measured (shown in figure A2).

From TG tests in figure A2, a maximum intercalation with the embedding ratio of 80.0wt% ~ 87.0 wt% when the concentration was over 1.1 mol·L⁻¹ was observed.

3.3. **Infrared Spectroscopy and XPS Analysis**

Figure 3 showed the IR spectroscopies of EG, raw RDX and one representative RDX/EG intercalation composite (maximum intercalated one prepared under the saturated RDX solution and 1:6 of volume ratio of solvent to anti-solvent) as KBr pellets, measured in the wave number range 4000 ~ 400 cm⁻¹ with a resolution of 4 cm⁻¹.
Figure 3. IR spectroscopies of EG, raw RDX and the maximum intercalated RDX/EG intercalation composite.

From figure 3, it could be found that the IR spectrum of composite perfectly consist of raw RDX and raw EG, the significant C-H equatorial asymmetrical stretching peaks 3080 and 3000 cm$^{-1}$, NO$_2$ asymmetrical stretching peaks 1592, 1571 and 1528 cm$^{-1}$, the methylene deformation vibrations in the ring of RDX (1456 and 1423 cm$^{-1}$)[40-42] suggested that antisolvent precipitation of RDX did not lead to any chemical evolution of RDX. Furthermore, the XPS spectroscopies of EG (figure 4) involved three characteristic peaks with the binding energies of 284.78, 532.78, 134.78 eV, which corresponded to C1s, O1s and P2p, respectively. The presence of these oxygen-containing groups promoted the interaction (i.e. hydrogen bond) and adsorption between the EG surface and RDX molecules, which could be the formation mechanism of RDX/EG intercalation composites.

Figure 4. The XPS general spectroscopy and the C1s, O1s, P2p spectroscopies (in the inserts) of EG.

3.4. Raman Spectroscopy and XRD Analysis

The normalized Raman spectroscopies of EG, raw RDX and the composite were collected using a
514.5 nm laser excitation source and shown in figure 5, revealed both raw RDX and the RDX in the composite sample to be α-phase [43]. To get a further evidence of the crystal phase, the crystal structures of EG, raw RDX and RDX/EG intercalation composites prepared under the different solution concentration conditions were characterized by using powder XRD patterns (shown in figure A3).

![Figure 5](image_url)

**Figure 5.** Normalized Raman spectroscopies of EG, raw RDX and the maximum intercalated RDX/EG intercalation composite.

3.5. **Thermal Analysis**

The DSC curves of the maximum intercalated RDX/EG intercalation composite and raw RDX tested at a heating rate of 10 K·min⁻¹ were shown in figure 6.

![Figure 6](image_url)

**Figure 6.** DSC curves of the maximum intercalated RDX/EG intercalation composite and raw RDX at 10 K·min⁻¹.

From figure 6, both the composite and raw RDX exhibited an endothermic peak and an exothermic
peak which related to the melting and thermolysis course of RDX, respectively. The melting and thermolysis temperatures of raw RDX were 205.53 and 243.03 °C, while these temperatures of the composite increased to 206.72 and 246.22 °C, respectively. Considering the structure of the composites, all RDX crystal was packed into the various cabins constructed by graphite sheets to form the half-coated parallel multi-sandwiches microstructure of RDX/EG intercalation composites. Moreover, graphite sheets connect to each other at the central axis constructing the EG worm as shown in following SEM image and sectional view schematic diagram, figure 7.

Figure 7. SEM image and sectional view schematic diagram of the RDX/EG intercalation composite.

EG is a three-dimensional material but constructed by two-dimensional graphite sheets. The sheets can realize heat transfer along x and y axis, accelerating the in-plane thermal conductivity [33]. The parallel thermal conductivity is much higher than that in the perpendicular direction, revealing that the thermal conductivity depends strongly on the orientation of the graphite sheets. From figure 7, RDX crystal attached both sides of sheets tightly and spread out uniformly without much stacking. When heated in DSC tests, the energy would be transferred and absorbed quickly by graphite sheets and central axis due to the excellent paralleled thermal conductivity and heat capacity. So, RDX could survived in higher temperature, i.e., the melting and thermolysis temperatures increased.

To obtain a specific evaluation of thermolysis of RDX/EG intercalation composites, the kinetics, thermodynamics and thermal stabilities parameters (table1) of raw RDX and the maximum intercalated RDX/EG intercalation composite were derived from their DSC curves (figure 11) under heating rates of 1, 2, 5, 10 K·min⁻¹. The kinetics parameters including activation energy (Eₖ), preexponential factor (Aₖ) and thermolysis rate constant (k) were calculated by Kissinger equation (1) and Arrhenius equation (2). The thermodynamics parameters like thermolysis activation enthalpy (ΔHᵣ), thermolysis activation free energy (ΔGᵣ) and thermolysis activation entropy (ΔSᵣ) came from the equation (3), (4) and (5). The thermal stability parameter critical temperature of thermal explosion (Tₑ) was obtained through the equation (6) and (7).

$$\ln \frac{\beta_i}{T_{pi}^2} = \ln \frac{R \cdot A_K}{E_K} - \frac{E_K}{R} \cdot \frac{1}{T_{pi}} \cdot i = 1, 2, 3, 4$$  (1)
\[ k = A_K \cdot \exp \left( -\frac{E_K}{T_P \cdot R} \right) \]

\[ A_K \cdot \exp \left( -\frac{E_K}{T_P \cdot R} \right) = \frac{K_B \cdot T_P}{h} \exp \left( -\frac{\Delta G^*}{R \cdot T_P} \right) \]

\[ \Delta H^* = E_K - T_P \cdot R \]

\[ \Delta G^* = \Delta H^* - T_P \cdot \Delta S^* \]

\[ T_b = \frac{E_K - (E_K - 4E_K \cdot R \cdot T_{P0})^{1/2}}{2R} \]

\[ T_{P0} = T_P + b\beta_i + c\beta_i^2 + d\beta_i^3, \quad i = 1, 2, 3, 4 \]

where \( \beta \) is the heating rate, K \cdot \text{min}^{-1}; \( T_P \) is the peak thermolysis temperature in the DSC curves, K; \( R \) is the ideal gas constant (\( R = 8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1} \)); \( E_K \) and \( A_K \) are the activation energy and preexponential factor calculated by the Kissinger equation, kJ \cdot \text{mol}^{-1} \) and s^{-1}, respectively; \( k \) is the thermolysis rate constant; \( K_B \) and \( h \) are Boltzmann’s \( (K_B = 1.381 \times 10^{-23} \text{ J} \cdot \text{K}^{-1}) \) and Planck’s \( (h = 6.626 \times 10^{-34} \text{ J} \cdot \text{s}^{-1}) \) constants, respectively; \( \Delta H^* \) is the thermolysis activation enthalpy, kJ \cdot \text{mol}^{-1}; \( \Delta G^* \) is the thermolysis activation free energy, kJ \cdot \text{mol}^{-1}; and \( \Delta S^* \) is the thermolysis activation entropy, J \cdot \text{mol}^{-1} \cdot \text{K}^{-1}; \( T_b \) is the critical temperature of thermal explosion, K; \( T_{P0} \) is the extrapolated peak temperature when \( \beta \to 0 \), K; \( b \), \( c \) and \( d \) are coefficients.

Figure 8. DSC curves of (a) the maximum intercalated RDX/EG intercalation composite and (b) raw RDX at different heating rates. Kissinger plots of \( \ln(\beta/T_P^2) \) versus \( 1/T_P \) were shown in the inserts. As shown in figure 8, two peaks appeared in all the DSC curves for the as prepared composite and raw RDX as the same with figure 6 and the melting and thermolysis peaks shifted to the higher temperature as heating rates increased. Compared to raw RDX, both the melting and thermolysis temperature of the maximum intercalated RDX/EG intercalation composite increased even at the varying heating rates.

From the table, all the kinetics parameters values including \( E_K \), \( A_K \) and \( k \) of the prepared composite were larger than those of raw RDX. The higher activation energy (improved by 33.37 kJ \cdot \text{mol}^{-1} and
20%) indicated that it needed more energy to activate the composites to the activated state. This result consisted with the calculation for thermal stability. The larger value of thermolysis rate constant \((k)\) at the same heating rate suggested that RDX/EG intercalation composites decomposed faster than raw RDX. Additionally, the value of \(k\) became larger at higher \(T_{b}\), i.e., higher heating rate.

Table 1. Kinetics, thermodynamics and thermal stabilities parameters of raw RDX and the maximum intercalated RDX/EG intercalation composite derived from their DSC curves.

| Samples     | \(\beta\) (K·min\(^{-1}\)) | \(T_{b}\) (°C) | Kinetics | Thermodynamics | Thermal stabilities |
|-------------|-----------------------------|----------------|----------|----------------|---------------------|
|             | \(E_k\) (kJ·mol\(^{-1}\)) | \(\lg A_k\) (s\(^{-1}\)) | \(k\) (s\(^{-1}\)) | \(\Delta H^f\) (kJ·mol\(^{-1}\)) | \(\Delta G^\theta\) (kJ·mol\(^{-1}\)) | \(\Delta S^\theta\) (J·mol\(^{-1}\)·K\(^{-1}\)) | \(T_b\) (°C) |
| Composite   | 1                           | 222.86         |          | 0.10           |                     |                     |             |
|             | 2                           | 229.46         | 201.43   | 20.2           | 0.19               |                     |             |
|             | 5                           | 239.14         | 1        | 0.47           | 197.21             | 131.61             | 129.24       | 224.09       |
|             | 10                          | 246.22         |          | 0.89           |                     |                     |             |
|             | 1                           | 215.76         |          | 0.08           |                     |                     |             |
| Raw RDX     | 2                           | 223.84         | 168.06   | 16.8           | 0.16               | 163.88             | 130.94       | 65.56        | 216.44       |
|             | 5                           | 235.01         | 8        | 0.40           |                     |                     |             |
|             | 10                          | 243.03         |          | 0.74           |                     |                     |             |

Meanwhile, the activation enthalpy \((\Delta H^f)\) of the composite was higher than that of raw RDX, which possesses of a similar physical meaning with the activation energy. The activation free energy \((\Delta G^\theta)\) for both the composite and raw RDX were positive and close to each other. This means that the thermolysis of composite is thermodynamically unfavorable, further demonstrated the composite is more stable than raw RDX.

The critical temperature of thermal explosion \((T_b)\) is a crucial parameter defined as the lowest temperature for a specific charge that may be heated without undergoing thermal runaway which is required to ensure safe storage, process operations and thermal stability of the explosives, propellants and pyrotechnics. The value of \(T_b\) of the RDX/EG intercalation composite was somewhat higher than that of raw RDX. All the calculated kinetics, thermodynamics and thermal stabilities parameters showed that the thermal stability of raw RDX was improved by the advanced half-coated parallel multi-sandwiches microstructure, i.e., the RDX/EG intercalation composites.

3.6. Impact and Friction Sensitivity

Sensitivity is one of the most important proprieties for energetic materials which influences their safety and application. Impact and friction sensitivities of raw RDX and the RDX/EG intercalation composite were shown in table2.

From the table, both the higher \(H_{50}\) value (maximum 28.40 cm) and lower explosion probability \(P_F\) value (minimum 20%) of composites than those of raw RDX, indicated that the special microstructure of samples could reduce the sensitivity obviously by (i) high heat conduction and (ii) hot spots isolation of the carbon microstructure.[44] When RDX crystal was stimulated accidentally, the energy of hot spots would be transferred and absorbed quickly by graphite sheets due to the excellent
paralleled thermal conductivity and heat capacity. In addition, RDX crystal was limited in the cabins constructed by graphite sheets, which prevented hot spots spreading everywhere to heat the vicinal RDX particles.

Table 2. Impact and friction sensitivities of raw RDX and prepared RDX/EG intercalation composites.

| No.   | Concentration of RDX solution (mol·L⁻¹) | Volume ratio of solvent to anti-solvent | Embedding ratio (wt%, TG Method) | Impact sensitivities | Friction sensitivities |
|-------|----------------------------------------|----------------------------------------|---------------------------------|----------------------|-----------------------|
| Raw RDX | -                                      | -                                      | -                              | 10.80                | 72                    |
| Composite | 0.9                                   | 1:6                                    | 73.5                            | 28.40                | 20                    |

4. Conclusions

In conclusion, sulfur-free expanded graphite was chosen to be the carrier of RDX crystal forming an advanced half-coated parallel multi-sandwiches microstructure, and the physicochemical properties of the composites were characterized. The embedding ratio (maximum 87.0 wt%) and particle size of RDX intercalated into EG could be controlled by preparation conditions of solvent/anti-solvent process. IR and XPS analysis revealed that there remained oxygen-containing groups and H₂PO₄⁻ anion on EG, which promoted the interaction and adsorption between the EG surface and RDX molecules. Raman and XRD analysis showed that RDX in the composites was in the most stable α-phase. After being embedded into EG, the thermal stability, impact and friction sensitivities of RDX were dramatically improved, suggesting that the prepared RDX intercalated EG composites could be applied as a novel electromagnetically energetic material in the field of multi-functional damage.

Appendix A

The Appendix A contains SEM images of raw materials (figure A1) and thermo-gravimetry curves of EG and RDX/EG intercalation composites prepared under the different solution concentration conditions (figure A2). Figure A3 shows the XRD patterns of EG, RDX/EG intercalation composites prepared under the different solution concentration conditions (the volume ratio of solvent to anti-solvent was 1:6) and raw RDX.

Figure A1. SEM images of raw materials: (a, b) EG at low- and high-magnification; (c) raw RDX.
Figure A2. Thermo-gravimetry curves of EG and RDX/EG intercalation composites prepared under the different solution concentration conditions.

Figure A3. XRD patterns of EG, RDX/EG intercalation composites prepared under the different solution concentration conditions (the volume ratio of solvent to anti-solvent was 1:6) and raw RDX.

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