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Efficient Sensitivity Reducing and Hygroscopicity Preventing of Ultra-Fine Ammonium Perchlorate for High Burning-Rate Propellants

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Abstract: In this research, several inert materials, including some functional carbon materials, paraffin wax and the well-known insensitive energetic material 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) were selected to reduce the undesirable high sensitivity and hygroscopicity of ultra-fine ammonium perchlorate (UF-AP) via polymer modified coating. Structure, sensitivity, thermal and hygroscopicity performances of the UF-AP based composites were systematically studied by scanning electron microscopy, sensitivity tests, thermal experiments, contact angle, and hygroscopicity analysis. The results showed that both the impact and friction sensitivity of UF-AP can be remarkably reduced, respectively, with only a small amount of 2% (in mass) desensitization agents. Meanwhile, improved thermal decomposition was gained, and the hygroscopicity can also be reduced to a large extent. Propellants containing 10% coated UF-AP in mass were processed and tested, the burning rate reached 45.7 mm s^{-1}, 50% higher compared with that of normal AP, with remarkably reduced impact sensitivity from 11.5 J to 29.6 J and friction sensitivity from 76% to 28%.

Keywords: Ammonium perchlorate (AP) · Propellants · Sensitivity · Hygroscopicity

1 Introduction

As an oxidizer with high energy, ammonium perchlorate (AP) has been widely used in solid propellants, blasting explosives and pyrotechnics, especially plays an important role in high burning-rate composite solid propellants [1,2]. The burning of AP in the monopropellant is a complex process including a series of gaseous and solid-state chemical reaction occur simultaneously, also combining with some physical factors such as heat transfer, diffusion, etc [3]. For decades, efforts have been devoted to investigate the burning mechanism of AP. It was found that the burning rate of propellant grain can be greatly increased with the decreased particle size of AP, owing to the high reactivity caused by enhanced specific surface area and the reduced diffusion distance [4,5]. However, the contradiction between high energy and low sensitivity is the eternal theme for energetic materials [6]. Although the burning performance can be visibly improved as the ultra-fine ammonium perchlorate (UF-AP, commonly < 5 μm) is largely adopted to replace the AP in normal size (> 100 μm) [5], the mechanical sensitivity will also be increased, especially for the friction sensitivity of propellant charge containing catocene as the catalyst [7].

For modern weapon systems, safety performance and environmental adaptability are of great significance, even more important than high energy output [8]. For a dozen of years, considerable works have been done to improve the safety of propellants on the premise of high burning rate, including the exploration of novel materials, structures, and manufacturing technologies [9]. For instance, hydrazinium nitroformate (HNF) was synthesized to replace AP [10], in spite of high energy and specific impulse, the friction sensitivity remained a thorny problem [11]. In addition, ammonium dinitramide (ADN) was explored as another powerful oxidizing agent and a potential halogen-free replacement for AP in solid rocket propellant [12], but the hygroscopicity of ADN crystal was found more severe [13]. In view of the high sensitivity caused by UF-AP, desensitization techniques have been investigated largely. Kohga synthesized fine porous or hollow AP by spray-drying method [14], in this case, the void structure inside the porous AP particles exhibited positive effects on the burning rate, nevertheless, the loading density would be in a low level. Nandagopal developed a precipitation technique to coat AP using a copolymer of hexafluoropropylene and vinylidene fluoride [15], resulting the increased burning rate, tensile strength and thermal stability after coating. Unfortunately, little change was observed in friction sensitivity value. In addition, as a kind of

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salt, the hygroscopicity and clustering of UF-AP can undoubtedly cause limitations during its transportation, storage and use, which should be inhibited via effective surface modifications. Therefore, exploring novel desensitizing materials as well as coating techniques becomes critical and urgent for the application of UF-AP in high burning-rate propellants.

Recently, functional carbon materials, such as activated charcoal (AC), graphite (G), graphene (GR), carbon nanotubes (CNTs), graphene oxide (GO), graphene nanosheets (GNs), have been extensively studied as additives to improve the burning, sensitivity and thermal performances of propellants and explosives [16–19]. Thanks to the high thermal conductivity and lubricating effect resulted from the layered structure [20], some functional carbon materials can provide great potential to reduce the friction sensitivity of UF-AP. Besides, 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) is known as the most insensitive energetic material, which has been proved efficient to reduce the sensitivity of high explosives with slight energy loss [21]. Meanwhile, paraffin wax has also been generally used as a desensitizing agent because of its endothermic and buffering capacity. Herein, these functional materials and their composites were employed to reduce the mechanical sensitivity and hygroscopicity of UF-AP by surface modification followed by a solvent suspension coating process. Coating structure, sensitivity, hygroscopicity, thermal decomposition properties and performances of propellants containing UF-AP were systematically studied.

2 Experimental Section

2.1 Materials

UF-AP (analytical grade) with an average particle size of 1 μm was purchased from Dalian North Potassium Chlorate CO., Ltd. China. Graphite, graphene, GO and GNs were purchased from Beijing DK Nano Technology Co., Ltd. China, nano TATB (nTATB) was prepared in our institute according to the literature [22]. Paraffin wax with melting point at 70 °C, Estane 5703 (poly [ester urethane] block copolymer), hydroxyl-terminated polybutadiene (HTPB) having number average molecular weight of 2500, aluminum powder (Al) with average particle size of 15 μm and the other reagents of analytical grade were commercially obtained and used without further purification.

2.2 Surface Modification and Coating of UF-AP

The coating of UF-AP was carried out via a solvent suspension process. UF-AP (25.0 g) was firstly dispersed in a Estane/1,2-dichloroethane solution (200 g, 0.5 % in mass) at room temperature to form a suspension, followed by vigorous stirring (600 rpm) for 0.5 h in a 1000 mL three-necked round-bottomed flask connected to a reflux condenser and equipped with magnetic stir bar. The UF-AP after surface modification was then obtained by filtering, washing with 20 mL 1,2-dichloroethane and drying in vacuo at 45 °C for 3 h. In this case, only negligible amount of polymer Estane (< 0.02 %) was kept, leaving an Estane coating film attached to the surface of UF-AP. Secondly, the modified UF-AP (24.5 g was crushed to powder and added into 150 g toluene to get a suspension under mechanical agitation (450 rpm), which was heated to 60 °C. At this time, 0.45 g or 0.5 g (1.8 % or 2 %) desensitizing agent (consists of carbon materials, nTATB or their composites) experienced an ultrasonic treatment (40 kHz, 20 °C, 10 min) to acquire uniform dispersion. Afterwards, the slurry containing desensitizing agent was added to the UF-AP suspension whilst stirring at 60 °C, and the vacuum (200 mba) was introduced for several min. Therefore, the 1,2-dichloroethane could be removed, resulting the coated desensitizing agent on the modified UF-AP. After that, for some samples containing only 1.8 % desensitizing agent, an additional amount of Estane/1,2-dichloroethane solution containing Estane (0.05 g, 0.2 % in the AP based composites) was added with the kept vacuum for a short time to remove the residual 1,2-dichloroethane, thus the composites with components of AP/desensitizing agent/Estane = 98/1.8/0.2 could be achieved. Finally, the products were filtered, washed by toluene and dried under vacuum for 48 h. The paraffin wax coated sample was prepared similarly via solvent suspension process by using petroleum ether as the solvent of wax. For comparison, the physical mixed samples with the same components were also prepared by mechanical grinding.

2.3 Characterization

The morphology of samples was performed by scanning electron microscopy (SEM) measurements with a LE0438VP instrument at an operating voltage of 25 kV. Thermogravimetry (TG) and the differential scanning calorimeter (DSC) test were recorded with a Mettler TG/DSC instrument from 50 to 500 °C in a nitrogen (40 mL min^{-1}) atmosphere with ramp of 10 °C min^{-1}. Impact sensitivity test was conducted with a WL-1 type instrument according to GJB-772A-97 standard method 601.2 (National Military Standard of China). The widely used up-and-down method was adopted generally, a series of 25–30 trials were performed in this method. For explosive powders, the test conditions are: drop weight, 5 kg; sample mass, 35 mg. The impact sensitivity of each test sample was expressed by the drop height of 50 % explosion probability (H_{50}) and impact energy (E_{50}). Friction sensitivity test was determined with a WM-1 type f instrument according to GJB-772A-97 standard method 602.1 (National Military Standard of China). The test conditions are: relative pressure, 3.92 MPa; sample mass: 30 mg, pendulum weight: 1.5 kg; pendulum angle: 90°. The friction sensitivity of each test sample was expressed by explosion probability (*P*). In

Table 1. Impact and friction sensitivity of UF-AP composites.

Group	ID	Composition[a)]	Processing[b)]	Impact E_{50}/J	Friction P/%
Raw	UF-AP	AP = 100	MC	7.9	94
I	AP-1	AP/G = 98/2	MC	8.7	10
	AP-2	AP/GR = 98/2	MC	27.3	54
	AP-3	AP/GO = 98/2	MC	51.2	82
	AP-4	AP/GNs = 98/2	MC	31.8	80
	AP-5	AP/nTATB = 98/2	MC	10.8	0
	AP-6	AP/W = 98/2	MC	21.2	56
II	AP-7	AP/G/W = 98/1/1	MC	59.6	16
	AP-8	AP/GNs/G = 98/1/1	MC	20.7	8
	AP-9	AP/GO/nTATB = 98/1/1	MC	51.4	0
III	AP-10	AP/G/Es = 98/1.8/0.2	MC	24.6	26
	AP-11	AP/GR/Es = 98/1.8/0.2	MC	53.3	58
	AP-12	AP/GO/Es = 98/1.8/0.2	MC	49.2	56
	AP-13	AP/GNs/Es = 98/1.8/0.2	MC	30.1	26
	AP-14	AP/GO/nTATB/Es = 98/0.8/1/0.2	MC	57.7	0
IV	AP-15	AP/GO = 98/2	PM	12.4	88
	AP-16	AP/nTATB = 98/2	PM	9.1	72
	AP-17	AP/GO/nTATB = 98/1/1	PM	13.4	80

a) Composition given in mass ratio; Es: Estane 5703. b) MC: modified coating; PM: physical mixing.

this way, higher E_{50} and lower P values represent reduced impact and friction sensitivity, respectively.

2.4 Hygroscopicity Analysis

Typically, raw or coated AP (5.0 g) was placed in a weighing bottle and exposed to the atmosphere of saturated steam at 20 °C, which was achieved in an airtight desiccator with suitable water in the lower layer. The weight of AP powders was recorded at different times, and then the hygroscopicity was calculated. The contact angle test was conducted with a Kono SL200B instrument, a molding tablet (Ø 20 mm × 2 mm) was prepared under specific pressure of 320 MPa, tested with water droplets and then the contact angle was calculated.

2.5 Propellants Processing and Performance Tests

The tested propellant formulation is AP/Al/HTPB/catocene/processing aids/bonding agents = 70/16/7/3/3/1 (in mass). The propellants containing different AP were prepared via homogeneous mixing and curing. For normal formulation, the 70% AP was used in grain gradation of trimodal distribution with particle size of 240 μm, 100 μm and 10 μm, the average particle size was about 100 μm. For high burning-rate propellants, 10% untreated or coated UF-AP was employed to substitute AP in normal size (about 100 μm), the other 60% AP was kept the same in the formulation. Impact and friction sensitivity tests were conducted also according to the GJB-772A-97 standard method 601.2 and 602.1, respectively. However, it should be noticed that the sensitivity test conditions should be different as the test objects have been changed from explosive powders to propellants. Herein, test conditions for impact (2 kg drop, 30 mg sample) and friction sensitivity (2.45 MPa, 1.5 kg pendulum, 66° angle, 20 mg sample) were adopted as the test objects are small table of propellants. Afterwards, impact energy (E_{50}) and explosion probability of friction (P) were recorded. For burning rate test, strips with size of 5 mm × 5 mm × 80 mm were processed and tested under 6.18 MPa. For the determination of pressure exponent value, the burning rate for each formulation was tested under pressure of about 3 MPa, 4.5 MPa, 6 MPa, 7.5 MPa, and 9 MPa. Finally, the pressure exponent could be calculated in this burning rate-pressure curve fitted by five points.

3 Results and Discussion

3.1 Appearance and Sensitivity Studies of UF-AP Composites

To investigate the desensitization effect of UF-AP, nTATB, paraffin wax (W), functional carbon materials including graphite (G), graphene (GR), graphene oxide (GO) and graphene nanosheets (GNs) were used. As large amount of desensitizing materials might reduce the energy and cause obstacles between UF-AP particles and catalysts in the propellants, by combination of the effectiveness of the passivation and energy maintenance, 2% total amount of the inert additives was adopted. To display the results clearly, samples of the UF-AP composites can be classified in four groups: (I) with single desensitizing agent (AP-1–AP-6), (II) with desensitizing complex (AP-7–AP-9), (III) with extra 0.2% polymer Estane (AP-10–AP-14), and (IV) physical mixed ones (AP-15–AP-17), as summarized in Table 1. From the results, it

is clear the raw UF-AP exhibits high impact and friction sensitivity, after coating with surface modification, the sensitivity can be visibly reduced.

Among the Group I with single desensitizing agent, it can be concluded that GR, GO, and GNs display efficient desensitization of impact, while G and nTATB definitely decrease the friction sensitivity. The well-known passivation agents, wax, shows moderate desensitization for both impact and friction. It should be noticed that high-extent sensitivity reduction can be obtained via such coating, especially compared with the samples by physical mixing. For instance, the impact energy E_{50} of AP-3 is dramatically increased from 7.9 J to 51.2 J, and the friction sensitivity of AP-5 is considerably decreased from 94% to 0%, while the physical mixed ones with same components show only slight desensitization effect. In view of the seemingly existing selectivity of sensitivity reduction for single desensitizing agent, the desensitizing composites were explored to pursuit a comprehensive desensitization. As can be seen in the results of Group II (Table 1), both the impact and friction sensitivity can be further reduced as two functional agents were introduced. As GO and nTATB are proved profitable to cut down the impact and friction sensitivity, respectively, their composites (AP-9) achieved both fairly low sensitivity (E_{50} = 51.4 J, P = 0%) as expected. Such remarkable desensitization capability might be caused by the combination of high buffer capacity and lubricating effect, which are resulted from the well dispersion of GO nanoparticles and the layered structure of TATB, respectively, thus facilitating the reduction of impact and friction sensitivity. For physical mixed samples (Group IV), inert agents exist in aggregates, and the effectiveness can be greatly weakened. By contrast, the inert agents can be greatly well dispersed after such modified coating as the ultrasonic treatment and solvent suspension technique were introduced. Furthermore, the surface modification by a trace of polymer can largely improve the interfacial interaction between UF-AP particles and desensitizing materials [23], leading to markedly decreased sensitivity.

For energetic composites, the mechanical strength of coating materials is important to maintain the nice performances. Besides, voids on the coating interface can cause larger probability for the formation of hot spot when external mechanical stimulation occurs, which is detrimental for sensitivity. Hence, in this work, 0.2% polymer Estane was introduced in the desensitizing system during the coating process. Despite a small amount was used, the Estane was expected to realize three functions: fixating of coating shell, filling the voids on the interface, and adjusting the hygroscopicity of UF-AP. The results of Group III (AP-10–AP-14) reveal that with the addition of Estane, both the impact and friction sensitivity can be further decreased, probably due to the favorable effects as mentioned above.

Figure 1 displays typical SEM images for raw UF-AP and the composites. The UF-AP particles are spherical in morphology, with the particle size of about 1 μm. After coating

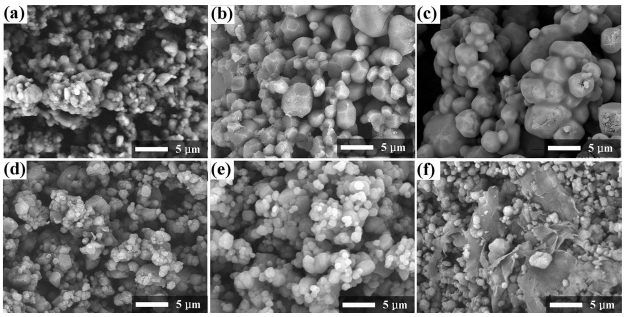

Figure 1. SEM images of UF-AP and composites, (a) UF-AP, (b) AP-5, (c) AP-6, (d) AP-9, (e) AP-10, and (f) AP-15.

by different desensitizing materials, we can see the uniform distribution of nTATB, carbon materials and wax on the surface of UF-AP; namely, the desensitizing agents are in nanoscale and well dispersed, instead of in large bulk, attributing to the assistance of ultrasonic treatment before the addition. Consequently, the sensitivity can be greatly reduced, corresponding to the results in Table 1, although the 0.2% amount of Estane is too low to be observed (Figure 1e). Moreover, it is noticeable that only a moderate size increase of UF-AP (from 1 μm to 4 μm) are observed for the composites after such surface modified coating, thus the unfavorable influence caused by particle clustering or size increment on burning rate in propellants can be eliminated. For the physical mixed AP-15 (Figure 1f), GO exists largely in bulk, thus showing negligible coating and desensitization effect.

3.2 Hygroscopicity Properties of the UF-AP Composites

As an inorganic salt, AP can easily dissolve in water, and the deliquescence of UF-AP can be strikingly deteriorated with decreasing particle size. When exposed to atmosphere, it absorbs moisture and starts to cluster [24], resulting restrictions for its application. Figure 2 shows the hygroscopicity performance of UF-AP and the representative composites (involve four groups in Table 1) after modified coating. Clearly, UF-AP exhibits apparent water absorption under the atmosphere of saturated steam, the hygroscopicity increases sharply to about 12% after 400 h. For the UF-AP based composites after modified coating, the deliquescence can be suppressed noticeably, ascribing to the hydrophobic polymer of Estane as a modified layer and the insensitive coating shell on the surface of AP. Different composition of coating shell can result different resistance to moisture absorption, AP-2 and AP-9 exhibit relative low resistance (9% after 400 h), probably due to the large specific surface area of GR and hydrophilicity of GO. The difference for the formulation of AP-9 and AP-14 is that 0.2% GO has been changed to Estane, forming a protecting polymer film, therefore, the restriction of hygroscopicity can be

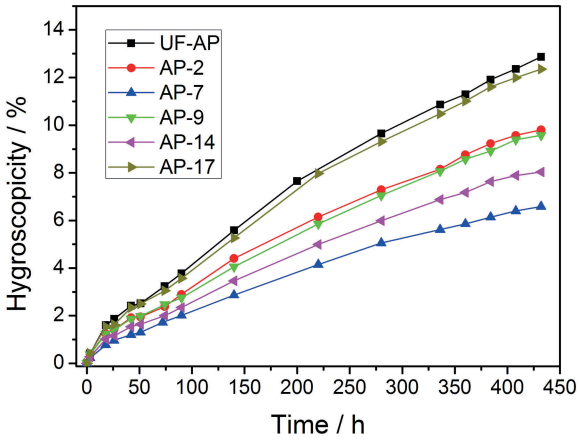

Figure 2. Hygroscopicity of UF-AP and composites after modified coating.

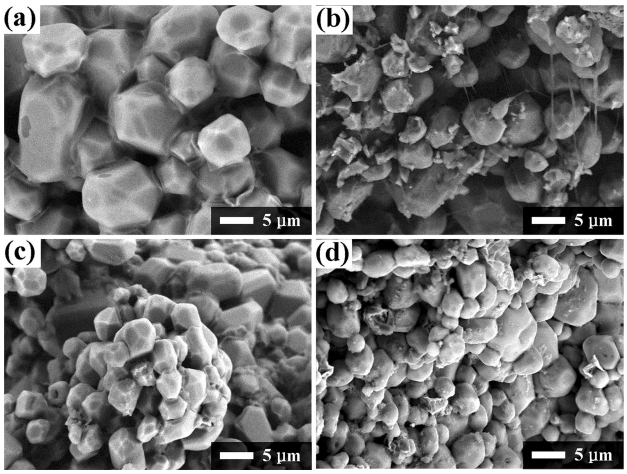

Figure 4. SEM images of UF-AP and composites after 120 h in saturated steam, (a) UF-AP, (b) AP-7, (c) AP-9, and (d) AP-14.

strengthened (7.7% after 400 h). AP-7 displays the lowest hygroscopicity of 6% after 400 h, owing to the protection of the hydrophobic 1% wax coated on the surface. It is worth to mention that the sample prepared by physical mixing (AP-17) brings little suppression for the hygroscopicity, as compared with raw UF-AP. In brief, both the unfavorable hygroscopicity and high sensitivity of UF-AP can be decreased by modified coating in this work.

Contact angle analysis was conducted to further study the hygroscopicity of UF-AP and the composites, the results are shown in Figure 3. The water droplet can diffuse easily toward the surface of UF-AP tablet, causing a small contact angle of 8.6°, whereas the angle values are 45.1°, 29.1°, and 35.4° for AP-7, AP-9, and AP-14, respectively. As large contact angle reveals a higher hydrophobicity, the sequence of water absorption suppression can be obtained as AP-7 > AP-14 > AP-9 > UF-AP, which is in good accordance with the hygroscopicity results in Figure 2. SEM results of these four samples after 120 h in saturated steam atmosphere are shown in Figure 4. It is obvious that UF-AP particles witnessed an apparent clustering during the water absorbing process. The crystal surface and interface of UF-AP were rebuilt, with the particle size increased to approximately 10 μm. For the composites after modified coating, such particle clustering was less serious. Filamentous additives can be seen in Figure 4b because of the breakage of wax shell in AP-7 during the water absorption. Although slight clustering and crystal growth for AP-9 and AP-14 also occurred, the protection of such insensitive coating shell can be beneficial to maintain the crystal structure compared with raw UF-AP, corresponding to the hygroscopicity and contact angle results.

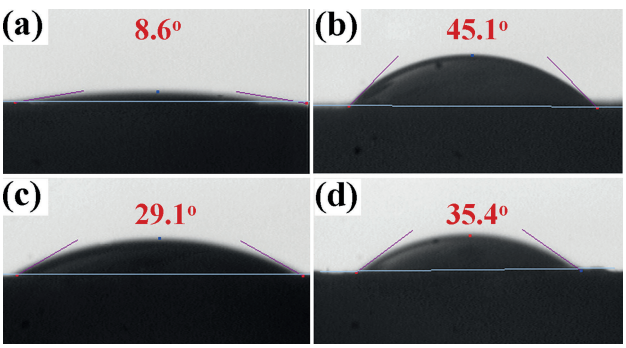

Figure 3. Contact angle of UF-AP and composites after modified coating, (a) UF-AP, (b) AP-7, (c) AP-9, and (d) AP-14.

3.3 Thermal Properties of the UF-AP Composites

Thermal decomposition behavior of AP plays a key role for the combustion of propellants. Figure 5 shows TG and DSC curves for UF-AP and composites, an endothermic peak at about 243 °C is observed in every sample, attributing to the polymorphic transformation of AP from orthorhombic to cubic crystalline [25], such transformation is almost unaffected by coating. A broad exothermic peak at about 320 °C is found in some DSC curves (AP-2, AP-3, AP-5, AP-9), due to the typical low-temperature decomposition of AP. The corresponding decomposition in two stages can also be observed in TG curves, and it is common such peak disappear for ultrafine AP particles (e.g. raw UF-AP) [26]. The exothermic peak at 441.8 °C for UF-AP belongs to its high-temperature decomposition, which encounters a decreasing shift for those composites after coating. Specifically, the decomposition temperature decrement of samples coated by 2% G (AP-1), GR (AP-2), GO (AP-3) and nTATB (AP-5) is 4.6 °C, 22.0 °C, 12.3 °C and 6.0 °C, respectively, compared with raw UF-AP. Such apparent decomposition in advance is caused by the catalysis of functional additives, which provides favorable effects on the energy release during the combus-

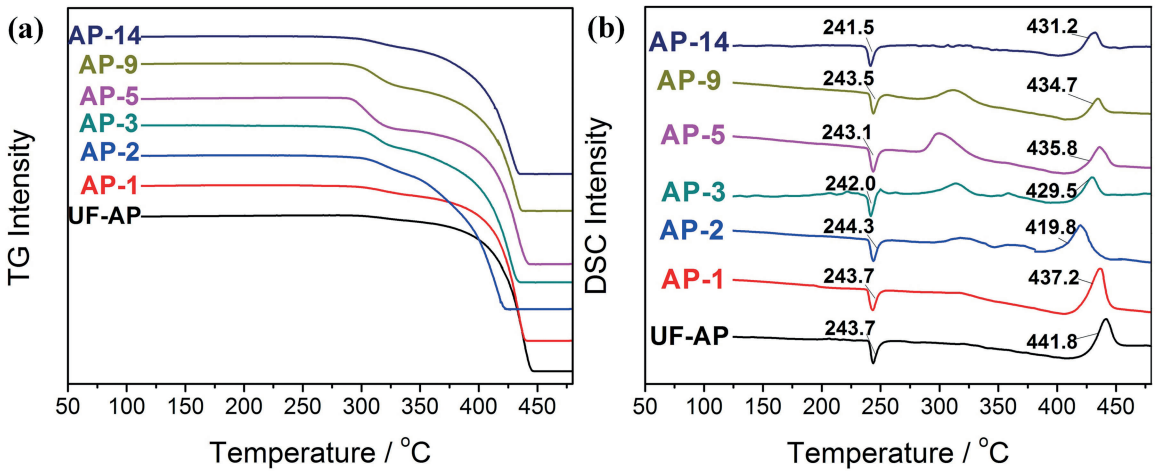

Figure 5. Thermal analysis of UF-AP and composites, (a) TG, (b) DSC.

tion of propellants. As GR and GO possess high specific surface area, their catalytic capability can be strengthened evidently. The decomposition temperature decrement of AP-9 and AP-14 can be calculated as 7.1 °C and 10.2 °C, respectively, indicating a further decrease of decomposition when a small amount of Estane is added in the composites, due to the inducing of free radicals formed during the depolymerization and thermolysis of Estane [21,27]. In summary, the energy release of coated AP can be improved, facilitating the combustion of solid propellants.

3.4 Performance of Propellants Containing Coated UF-AP

UF-AP, representative coated UF-AP and AP in normal size (about 100 μm) were selected to study the combustion and sensitivity performance, 10% UF-AP was employed in the processing of solid propellants and the results are shown in Table 2. For UF-AP, with the particle size decreases, burning rate can be definitely improved from 30.1 mm s^{-1} to 41.6 mm s^{-1}, compared with normal AP. Besides, the burning rate of AP-7 and AP-14 exhibits further increase to 44.8 mm s^{-1} and 45.7 mm s^{-1}, respectively, owing to the catalysis effect of functional additives. As has been noted, the impact and friction sensitivity of solid propellants increases dramatically as the particle size of AP decreases, the impact (E_{50}) and friction *P* sensitivity values are 11.5 J and 76% for propellant containing UF-AP in this work.

As for the desensitized UF-AP after coating, the impact and friction sensitivity can be noticeably reduced. For example, the E_{50} and P values of AP-14 are 29.6 J and 28%, respectively, even superior than those of normal AP, with 50% improved burning rate. In a word, the safety performance of current high burning-rate propellants containing UF-AP can be greatly improved by such surface coating. Moreover, the pressure exponent value generally increases with largely increased burning rate, but the increment seems moderate in this work. For UF-AP, the pressure exponent is increased by about 0.04 as compared with normal AP, and the increment for the representative coated AP-7 and AP-14 is about 0.08. As it is known that lower pressure exponent can be beneficial for the design of engine [28], such high burning-rate propellants are further proved favorable for the application.

Table 2. Burning and sensitivity performances of propellants containing 10% different AP.

AP used	Burning rate/ mm s^{-1}	Impact E_{50}/J	Friction *P*/ %	Pressure exponent
Normal AP	30.1	24.0	36	0.453
UF-AP	41.6	11.5	76	0.492
AP-7	44.8	21.9	54	0.537
AP-14	45.7	29.6	28	0.533

4 Conclusions

A series of UF-AP based composites were prepared by coating 2% different desensitizing materials after surface modification by a little polymer Estane, additives were dispersed well in the composites. Both the mechanical sensitivity and hygroscopicity can be greatly improved for the coated UF-AP based composites, compared with the raw material, as confirmed by sensitivity tests, SEM, contact angle and hygroscopicity analysis, indicating preferable safety and storage stability performances. Thermal analysis results by TG/DSC show that the resultant UF-AP composites also bring favorable effects for their thermal decomposition, which is profitable for the energy release during combustion. Sensitivity of propellants containing UF-AP can be visibly reduced, with the more than 50% higher burning rate maintained and only moderate increment of pressure exponent.

In conclusion, through this facile coating technique, current high-performance propellants can be obtained with high burning-rate, low sensitivity and low hygroscopicity, indicating evident application prospects in modern weapon systems.

Acknowledgments

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