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Research Article

The Mechanical and Reaction Behavior of PTFE/Al/Fe_2O_3 under Impact and Quasi-Static Compression

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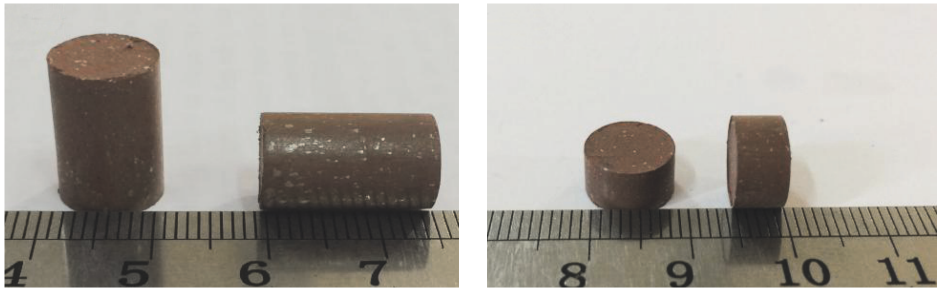
Quasi-static compression and drop-weight test were used to characterize the mechanical and reaction behavior of PTFE/Al/Fe_2O_3 composites. Two kinds of PTFE/Al/Fe_2O_3 composites were prepared with different mass of PTFE, and the reaction phenomenon and stress-strain curves were recorded; the residuals after reaction were analyzed by X-ray diffraction (XRD). The results showed that, under quasi-static compression condition, the strength of the materials is increased (from 37.1 Mpa to 77.2 Mpa) with the increase of PTFE, and the reaction phenomenon occurred only in materials with high PTFE content. XRD analysis showed that the reaction between Al and Fe_2O_3 was not triggered with identical experimental conditions. In drop-weight tests, PTFE/Al/Fe_2O_3 specimens with low PTFE content were found to be more insensitive by high-speed photography, and a High Temperature Metal Slag Spray (HTMSS) phenomenon was observed in both kinds of PTFE/Al/Fe_2O_3 composites, indicating the existence of thermite reaction, which was confirmed by XRD. In PTFE/Al/Fe_2O_3 system, the reaction between PTFE and Al precedes the reaction between Al and Fe_2O_3.

1. Introduction

Reactive materials (RMs) or impact-initiated materials and their applications have attached great interest in recent years due to their uniquely mechanical and chemical properties. The mixture of polytetrafluoroethylene (PTFE) and aluminum (Al) is one of the typical RMs and has been extensively studied, because of its high energy density and a favorable combination of properties of PTFE such as low friction coefficient, high thermal stability, high electrical resistance, high chemical inertness, high melting point (327°C), high thermal energy release when decomposed, and the easiness to deform [1]. A lot of experiments have been conducted to probe the mechanical and reaction behavior of PTFE/Al composites by different means. In order to improve the density and strength of the materials, many researchers have tried to add tungsten (W), the most common "heavy" metal, into the PTFE/Al composites. Dynamic compression experiments were performed by Cai et al. on a pressed PTFE/Al/W mixture to understand the mechanical response and failure at high-strain and high-strain rate [1]. The results showed that failure was proceeded by extensive plastic deformation concentrated mainly in the PTFE matrix, and the initiation as well as the propagation of cracks was caused by the separation of the W particle-PTFE interface. Herbold et al. investigated the particle size effect on the strength, failure, shock behavior, and dynamic properties of PTFE/Al/W composites combining simulation and experiments [2, 3]. They found that the increased strength of the sample with fine metallic particles is due to the formation of force chains under dynamic loading. Numerical modeling of shock loading of this granular composite material demonstrated that the internal energy, specifically thermal energy, of the soft PTFE matrix can be tailored by the W particle size distribution. Xu et al. tested four kinds of PTFE/Al/W with different contents and sizes of W particles [4]. The tensile and impact properties of PTFE/Al/W reactive energetic composites were probed using a universal materials testing machine and an improved pendulum impact tester at room temperature. The results revealed that the failure behavior of PTFE/Al/W included deformation, fracture, disorganization, and reaction. They also drew the conclusion that the addition of 30 wt.% of coarse W particles improved the impact strength of the material but led to the increase of reactive activity and



(a) (b)

Figure 1: Samples of PTFE/Al/Fe_2O_3 composites: (a) for quasi-static compression and (b) for drop-weight tests.

the decrease of perfectibility of the reaction. Other scholars also carried out in-depth study of PTFE/Al/W from other perspectives using different techniques including quasi-static and dynamic compression (both drop-weight test and Split-Hopkinson Pressure Bar), scanning electron microscopy (SEM), X-ray diffraction (XRD), and differential scanning calorimetry (DSC) [5–9].

As can be seen above, much attention has been put to the addition of W particles to increase the strength and density of PTFE/Al, while literatures about the addition of metal oxides, such as CuO and Fe_2O_3, are scarce. In the above experiments about PTFE/Al/W, no reaction between Al and W was observed; only a small amount of WC and W_2C formed, which were activated by the high heat as a result of fluorination of Al [7]. But the heat generated by the reaction between Al and metal oxides (thermite) cannot be neglected. Zhou et al. studied the reactions of Al/CuO, Al/Fe_2O_3, and Al/ZnO systems using T-Jump/time-of-flight mass spectrometer [10]. They found the temperatures of the reactions can reach 2837 K, 3135 K, and 1822 K, respectively. And the enthalpy of combustion is −604.05 kJ/mol, −425.1 KJ/mol, and −312.1 KJ/mol (per mol of Al) correspondingly. Also, Wang et al. revealed that the heat generated from the reaction between Al and Fe_2O_3 can raise the material to a temperature of ca. 3000°C, higher than the melting point of Fe (1535°C) and Al_2O_3 (2059°C) [11]. However, because of the low energy release rate and the difficulty to shape, the application of the thermites is limited. But experiments confirmed that the addition of 10 wt.% PTFE into Fe_2O_3/Al mixture can reduce the time to reach the exothermic peak by two orders of magnitude [12].

The PTFE/Al/Fe_2O_3 composites are promising reactive materials, which have the dual properties of PTFE/Al and Al/Fe_2O_3 and can greatly expand the application fields of the PTFE/Al composites if the Al/Fe_2O_3 reaction can be triggered. But to the best of our knowledge, few researchers investigated the mechanical and reaction behavior of PTFE/Al/Fe_2O_3 systematically. In this paper, the mechanical and reaction behavior of PTFE/Al/Fe_2O_3 under impact and quasi-static compression were investigated, and the failure and ignition mechanism were analyzed. The insensitivity of PTFE/Al/Fe_2O_3 to impact was measured quantitatively, which is of great importance for applications of reactive materials.

2. Experimental Setup

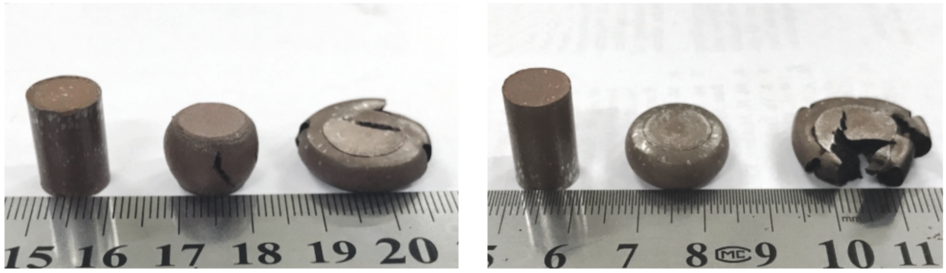
2.1. Specimens Preparation. In this study, two kinds of composites, 50PTFE-28Al-22Fe_2O_3 (wt.%) (type A specimen) and 75PTFE-14Al-11Fe_2O_3 (wt.%) (type B specimen), were fabricated by powder mixture, cold isostatic pressing (CIPing), and vacuum sintering based on the patent of Nielson's [13]. The initial powers have the following average sizes: PTFE: 25 um (3 M, Shanghai); Al: 1-2 um (JT, Hunan); Fe_2O_3: 3–5 um (NAO, Shanghai). Firstly, the PTFE/Al/Fe_2O_3 powders were suspended in an ethanol solution and mixed by a motor-driven blender for 20 minutes and then dried in a vacuum oven for 48 hours at 60°C, followed by a screening process to break up agglomerates. Next, the dried mixture was cold pressed into cylinders (for quasi-static compression and for drop-weight test, Figure 1) under the pressure of 60 Mpa. Finally, specimens were sintered in a vacuum oven at the temperature of 370°C for 4 h with a cooling rate of 50°C/h.

2.2. Methods. Quasi-static compression tests were performed using a SFLS-30T electrohydraulic press with a maximum loading of 30 kN. The sample was placed between two hardened steel anvils and was compressed at the speed of 9 mm/min, corresponding to a normal strain rate of 0.01/s. The load was terminated when the sample was fractured or the maximum load was reached. All tests were conducted at room temperature and the stress-strain curves of all the samples were recorded by the hydraulic press automatically. To ensure repeatability, at least five specimens of each kind were tested.

Drop-weight tests were carried out with a fall hammer impact sensitivity tester with a drop mass of 10 kg which can be dropped from a maximum height of 1.5 m. So the maximum speed of 5.5 m/s of the drop hammer and the strain rate of almost 1000/s can be achieved. In order to observe the reaction process, high-speed photography with the frame rate up to 20000 frames/s was adopted.

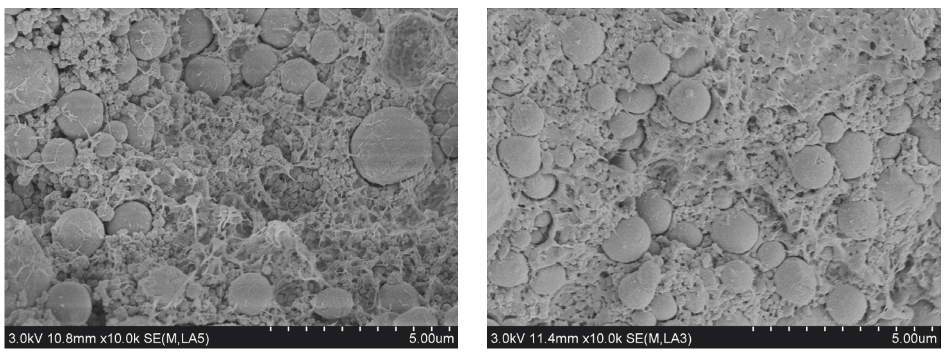
Hitachi S-4800 scanning electron microscope (SEM) was used to observe the microstructure of the materials and to analyze the reaction and fracture mechanisms.

The residues of the specimen after quasi-static compression and drop-weight test were collected and characterized by X-ray diffraction (Bruker D8 ADVANCE, operating at 40 kV and 40 mA with unfiltered Cu Kα radiation).



(a) (b)

Figure 2: Samples before and after quasi-static compression. (a) Type A specimen and (b) type B specimen.



(a) (b)

Figure 3: The microstructures of PTFE/Al/Fe_2O_3 composites. (a) Type A specimen and (b) type B specimen.

3. Results and Discussion

3.1. Mechanical Behavior under Quasi-Static Compression. Typical photographs of uncompressed and compressed PTFE/Al/Fe_2O_3 reactive materials of the two kinds are shown in Figure 2. It can be found that there are quite differences between the two materials. For type A specimens, cracks appeared soon after the compression load was added and the specimens were fractured at a relatively low strain level. But type B specimens stayed intact even when they are pressed to about 0.2 of their original length, indicating a greater plastic deformation. The difference of the failure behavior of the two materials is mainly attributed to the different content of PTFE. More PTFE ensures better matrix continuity and thus leads to a great plasticity, which can be confirmed from the SEM photographs in Figure 3. As can be seen from Figure 3, the combination between Al, Fe_2O_3, and PTFE matrix in type B specimens is better than that in type A specimens, indicating more perfect mechanical properties for type B specimens. The stress-strain curves of the two materials are given in Figure 4, and their corresponding parameters are listed in Table 1. When compressed, both types of specimen underwent the process of elastic and plastic deformation, crack propagation, and fracture. As can be seen, the two specimens have almost identical elastic deformation and possess similar yield stresses. After the pressure exceeded the yield stress, the strain hardening phenomenon occurred for both types of PTFE/Al/Fe_2O_3. However, the strain hardening modulus of type B specimen (34.8 Mpa, averaged from strain 0.1 to 1.72) is 2.88 times higher than that of type A specimen (12.1 Mpa, averaged from strain 0.1 to 1.45), which causes the toughness (the area under the stress-strain curve) of type B specimen to be higher than that of type A specimen. That is, in the case of quasi-static compression, type B specimens can absorb more energy than type A specimens before fracture. So type B specimens are more likely to react in the same condition and the experiments had confirmed this hypothesis, which will be discussed later. As for the failure process, type B specimens failed more suddenly than type A specimens. With the increase of compression load, the axial cracks and shear cracks in type A specimens developed gradually; when the specimens cannot resist the pressure, they fractured. But for type B specimens, no obvious visible macrocracks generated until the maximum strength was reached. However, once the fracture condition was met, type B specimens fractured catastrophically, accompanied with a sharp drop in stress and intensive reaction phenomenon. The ignition point was marked in Figure 4.

3.2. Reaction Behavior under Quasi-Static Compression. Figure 5 shows the initiation phenomenon of a type B specimen under quasi-static compression. When the true stress reached about 75 Mpa, the initiation started from the vicinity of the outer surface of the cylinder where the shear force is the strongest, accompanied by explosion sound and bright lights. Then the reaction propagated from the edge to the interior of the specimen and quenched quickly; the whole ignition process lasted about 1 s, with about 90% of the

Table 1: Parameters of PTFE/Al/Fe_2O_3 reactive material.

Type	PTFE : Al : Fe_2O_3 (wt.%)	Density (g/cm^3)	Yield stress (Mpa)	Average strain hardening modulus (Mpa)	Ultimate strength (Mpa)	Ignition or not
A	50 : 28 : 22	2.23	20.1	12.1	37.1	No
B	75 : 14 : 11	2.33	19.9	38.4	77.2	Yes

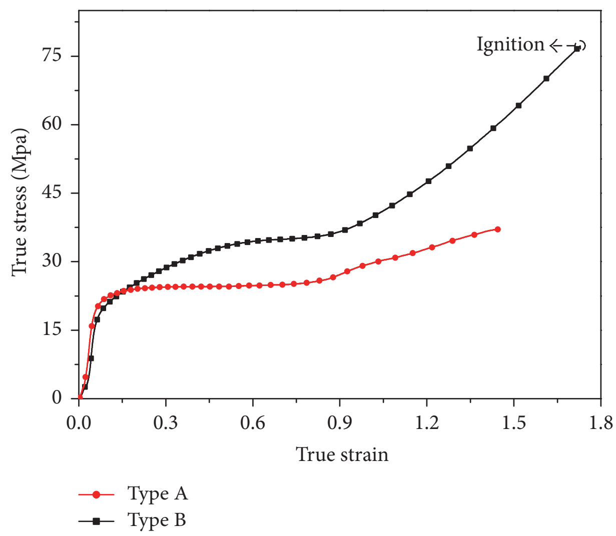


Figure 4: Stress-strain curves of two kinds of PTFE/Al/Fe_2O_3 composites.

specimen unreacted. Feng et al. also observed the initiation phenomenon with PTFE/Al composites under similar experimental condition [14], in which PTFE and Al had reacted with each other to form AlF_3 and C (carbon black). However, no initiation phenomenon was observed for any type A specimen, which could be attributed to the low toughness as mentioned above.

The specimen of type B after reaction and the microstructures of the reacted and unreacted regions are shown in Figure 6. As can be seen from Figure 6(a), the black parts correspond to the reacted regions, which is marked with "reacted region." And there is a clear boundary between the unreacted and reacted region, which can be seen more clearly in Figure 6(b). The microstructures of the unreacted and reacted regions are given in Figures 6(c) and 6(d). In unreacted region (Figure 6(c)), PTFE has a distinct orientation and forms a dense nanofiber network, with Al and Fe_2O_3 particles embedded in the PTFE matrix without significant deformation. In reacted region (Figure 6(d)), the crack section is covered by smooth melted PTFE and carbon black, and PTFE nanofiber network completely disappeared. The specimen after reaction is displayed in Figure 6(a), the black part corresponds to the reacted region, which is marked with "initiation." Moreover, the opening crack and shear plane caused by compression are clearly visible, which are closely related to the ignition phenomenon [15]. Although there has been much controversy about the mechanical initiation of explosions over these years, many scholars believe that the initiation is due to the existence of "hot spots." Swallowe and Field found that the hot spots formed at the polymer crack tip and shear band can activate the explosive [16]. In the PTFE/Al/Fe_2O_3 system, PTFE is the predominant pressure-bearing component; when subjected to compression, PTFE matrix was deformed heavily, causing the crack generation and spread. The crack propagation speed can reach 200 m/s to 800 m/s and the temperature of the crack tip can reach 300°C to 1000°C [17] and the high temperature can greatly promote the reaction. Feng et al. proposed a crack-induced initiation mechanism of Al-PTFE under quasi-static compression, which can also explain the reaction mechanism of PTFE/Al/Fe_2O_3 system [15]. As can be seen from the XRD analysis in Figure 7, the reaction products showed that Fe_2O_3 was not involved in the reaction; the initiation phenomenon was actually caused by PTFE and Al.

The XRD results of the residuals of both type A and type B specimens after compression are shown in Figure 7. No other species were found besides the original three components (PTFE, Al, and Fe_2O_3) in the compressed specimen of type A specimens, which means that there was no reaction between PTFE, Al, and Fe_2O_3. This is due to the brittle nature of this material and the lower strength and toughness. When compressed, type A specimens fractured quickly before the formation of high temperature of the crack tip, and thus there was no enough energy to induce the reaction. However, AlF_3 was found in the compressed specimen of type B materials, and the black powder remaining on the anvil proved to be carbon black, indicating the following reaction had taken place:

$$Al + (\text{-}C_2F_4\text{-}) \longrightarrow AlF_3 + C \tag{1}$$

AlF3 was formed from the Al oxidized by fluorine from the PTFE. But besides iron oxide (Fe_2O_3), no other iron compounds or iron (Fe) was found in the reaction products, illustrating that Fe_2O_3 did not involve in the reaction. That is, the thermite reaction between Al and Fe_2O_3 was not triggered under quasi-static compression. Firstly, the energy or temperature required to initiate the thermite reaction is relatively high. Williams et al. found the ignition temperature of Al and Fe_2O_3 was 800 K–950 K by experiment [18], but Zhou et al. concluded that the ignition temperature of Al and Fe_2O_3 was 1100 K using T-Jump/time-of-flight mass spectrometer [10], while Fan et al. discovered that the exothermic peak of DSC curves of Al and Fe_2O_3 powder mixture appeared at 853.5°C–949.7°C, and the exothermic peak represented the thermite reaction [19]. The differences of ignition temperature in the three studies are attributed to the diversity of materials and the difference of heating rate, but all three studies showed that thermite reaction

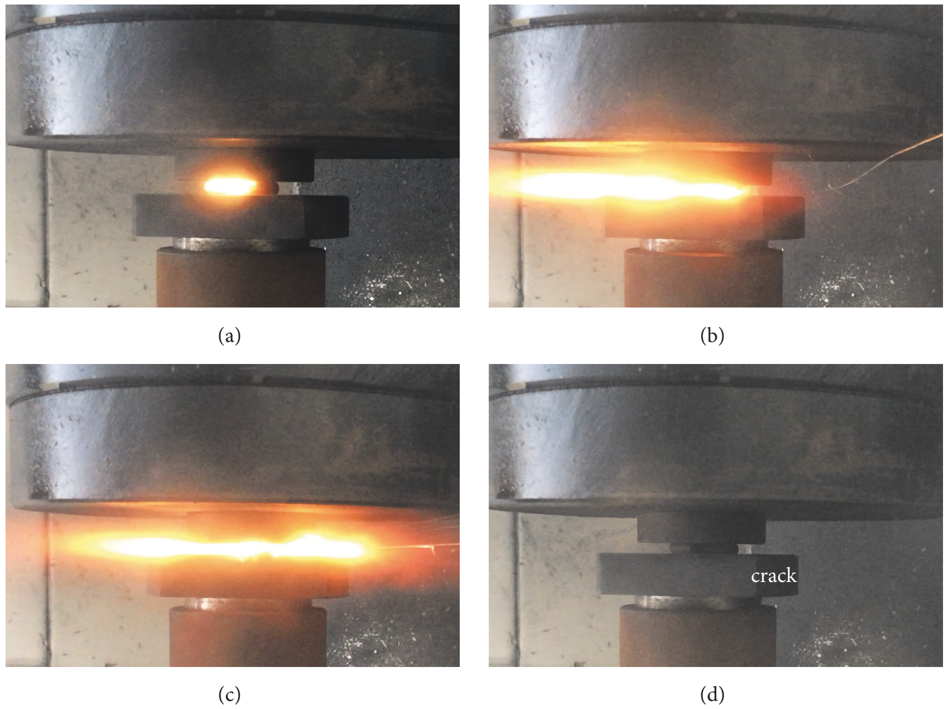


Figure 5: The initiation process of a type B specimen. (a) First sign of initiation, (b) reaction propagated from the edge to the interior of the specimen, (c) violent reaction, and (d) after initiation and the formed crack.

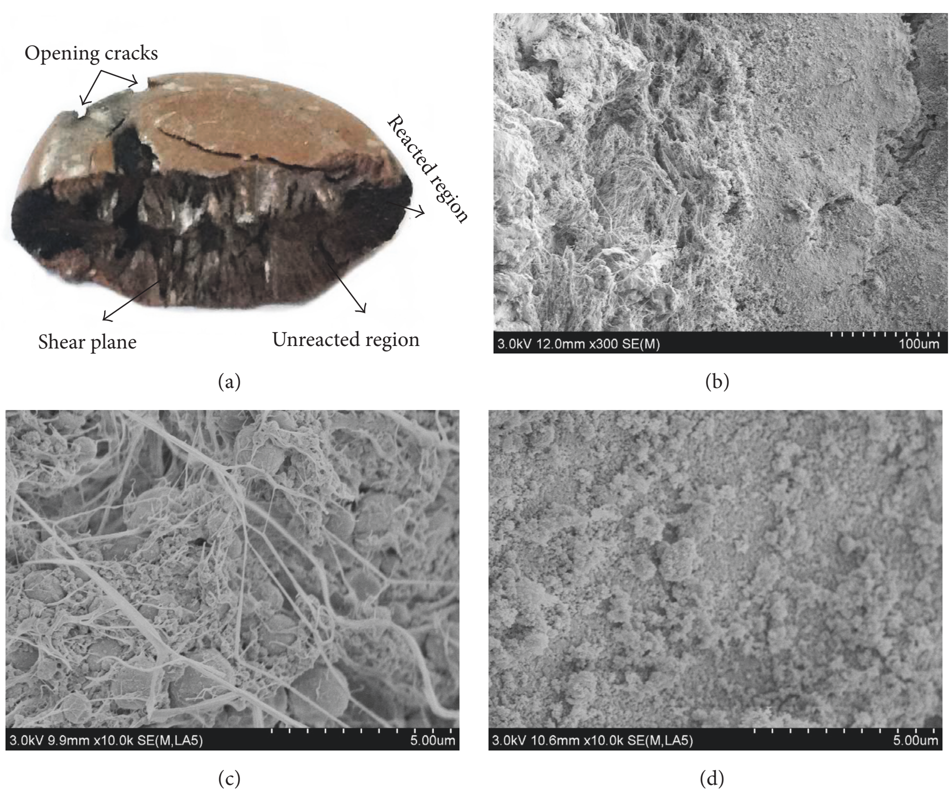


Figure 6: SEM photographs of the initiation region and specimen after reaction. (a) Specimen after reaction, (b) boundary of reacted region (right) and unreacted region (left), (c) unreacted region, and (d) reacted region.

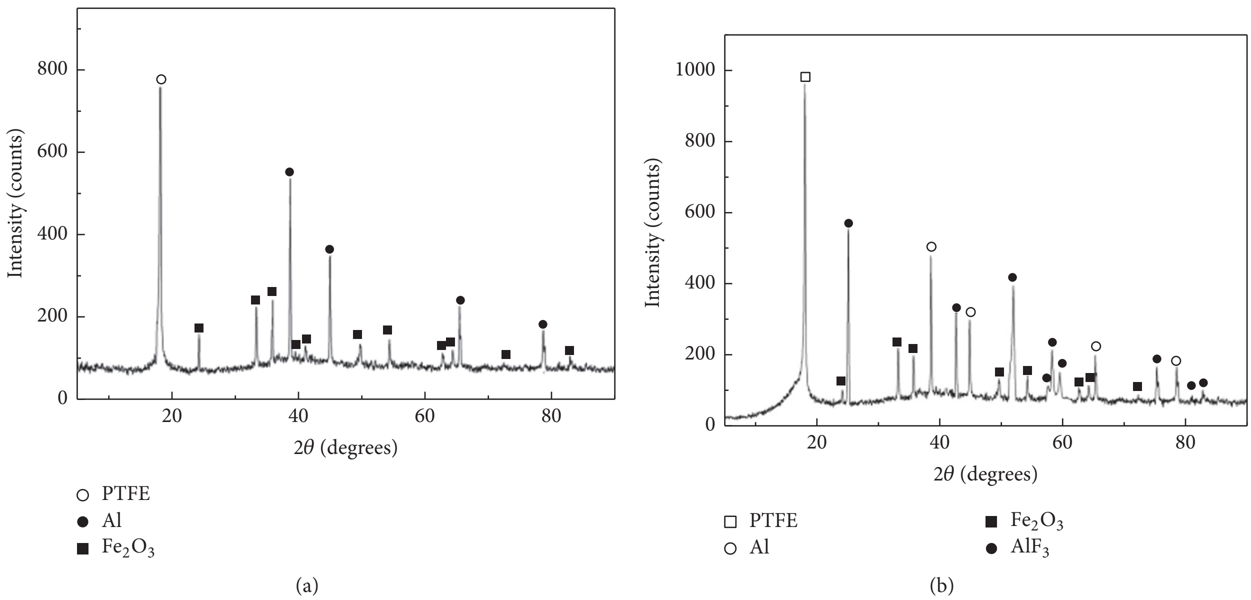


FIGURE 7: The XRD results of specimens after quasi-static compression. (a) Type A specimen and (b) type B specimen.

needs high temperature (hundreds of degrees centigrade) to trigger. However, the temperature rise caused by quasi-static compression is low relatively (tens of degrees centigrade) according to the simulation result of Feng et al. [14]; therefore it is not enough to trigger the reaction of Al and Fe_2O_3. Furthermore, despite the intense reaction of PTFE with Al, the duration may be too short to induce thermite reaction between Al and Fe_2O_3. Besides, the content of Al and Fe_2O_3 was small and they were dispersed in the PTFE matrix; the less contact of the two substances resulted in the difficulty to react. Lastly, compared with other active oxides, such as CuO and Cu_2O, the activity of Fe_2O_3 is relatively lower because of its much less oxygen release, since the analysis by Wang et al. suggested that the initial steps of the thermite reaction are the decomposition of the oxidizer particles to form oxygen as well as Al diffusion through the Al_2O_3 shell [11].

3.3. Reaction Behavior under Drop-Weight Test. The impact sensitivity of both type A and type B materials was investigated using an impact sensitivity tester and a high-speed photography, and the sensitivity of the materials is measured by the lowest drop height from where the drop mass is released to produce greater than 50% reaction, which is on the basis of an abbreviated Bruceton method [20], and the characteristic drop height is represented by H_{50} in Table 2. Characteristic drop height (H_{50}) can be used to calculate ignition energy based on potential energy as in

$$E_p = mgh, \tag{2}$$

where E_p is the potential energy of the drop mass, m is the mass of the drop mass, and h is the optimal height for reaction. To compare the absorbed energy of the materials under quasi-static compression and drop-weight test, the estimated ignition energy under quasi-static compression is also calculated by the work done by the machine along the compress direction, which can be expressed as follows:

TABLE 2: Results of ignition energy under static and dynamic load.

Type	Drop height (H_{50})/cm	Ignition energy (static)/J	Ignition energy (impact)/J
A	80	—	78
B	51	71	50

$$W = \sum F_t \Delta s, \tag{3}$$

where W is the work done by the machine, F_t is the transient force, and Δs is infinitesimal increases of displacement; both F_t and Δs can be retrieved from the testing machine directly. The information of the two materials under the two test conditions is listed in Table 2.

As can be seen from Table 2, the ignition energy of type B materials under quasi-static compression (71 J) is higher than that under drop-weight impact (50 J). But if taking consideration of the energy dissipated into the surrounding environment during the quasi-static compression process, they may be similar, because more heat will be lost during quasi-static compression, while dynamic impact can be considered as an adiabatic process. Comparing the behavior of the two materials under dynamic impact, we can conclude that type B specimens are easier to ignite than type A specimens. More energy or higher drop height is needed for type A specimens to react, which is consistent with the results of quasi-static compression.

Figures 8 and 9 show a sequence of images taken from high-speed camera of the two kinds of PTFE/Al/Fe_2O_3 composites under impact loading, and the drop mass was dropped from the same height of 1.5 m. Obviously, type B specimens reacted more violently than type A specimens.

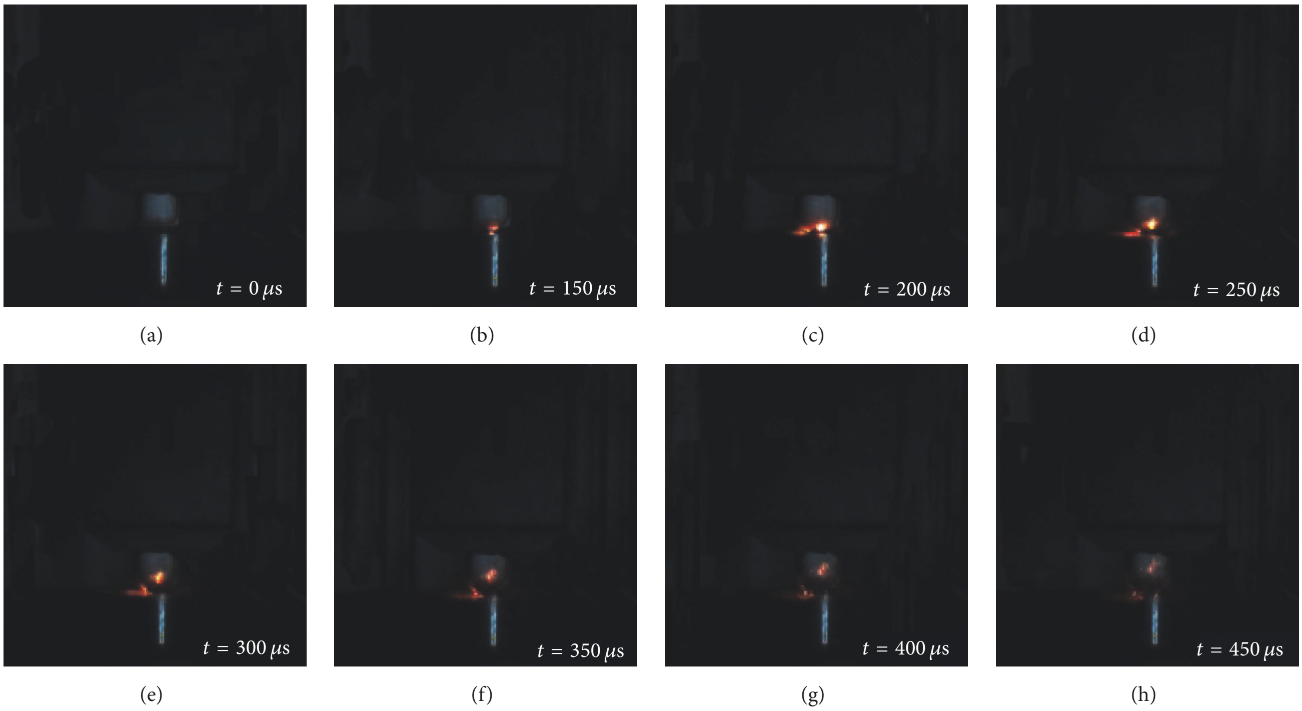


Figure 8: Selected images for a type A specimen reaction recorded by a high-speed camera (from the height of 1.5 m).

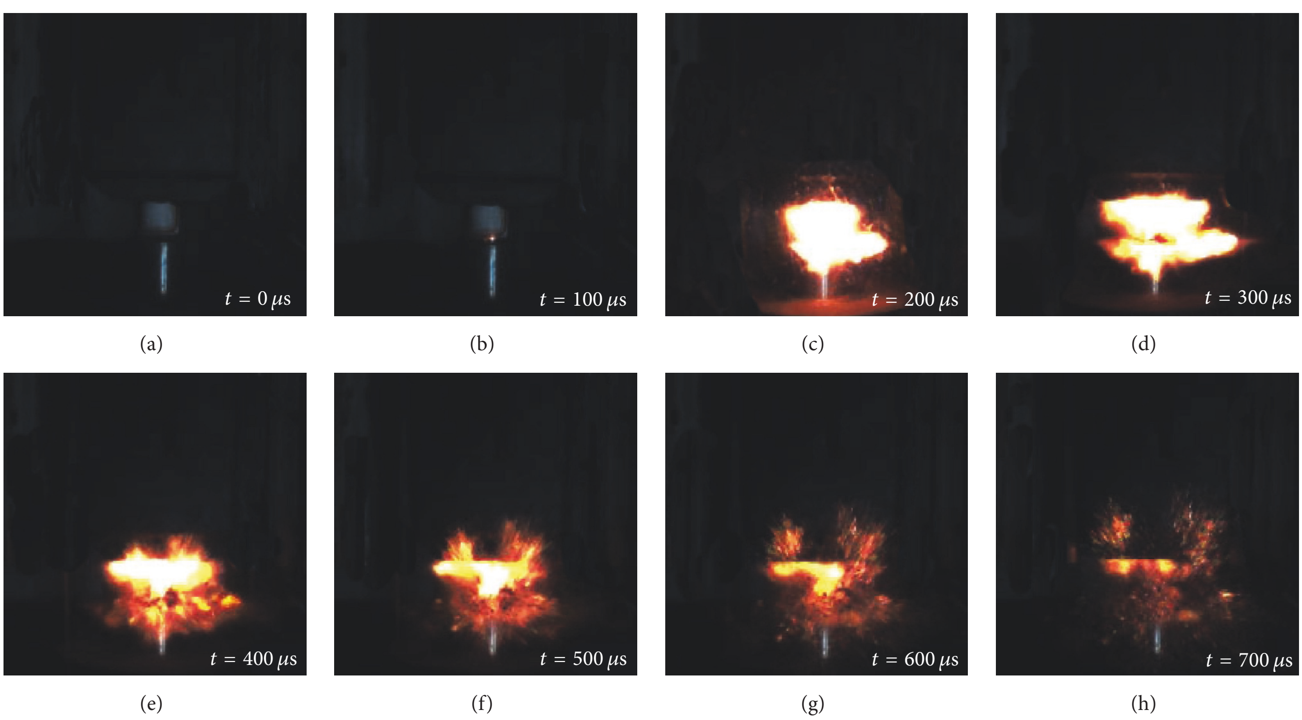


Figure 9: Selected images for a type B specimen reaction recorded by a high-speed camera (from the height of 1.5 m).

For type A specimens, when impacted, only a weak fire was produced and the duration of reaction was only 300 μs. But for type B specimens, intensive light was captured by high-speed photography, accompanied with explosion noise. The reaction time is more than 600 μs, which is much longer than that of type A specimens. The difference in reaction time is mainly due to the difference in completeness of the reaction. For type A, only a small part of the specimen was involved in the reaction, while most of type B specimen reacted. Furthermore, the ignition time of type A specimens

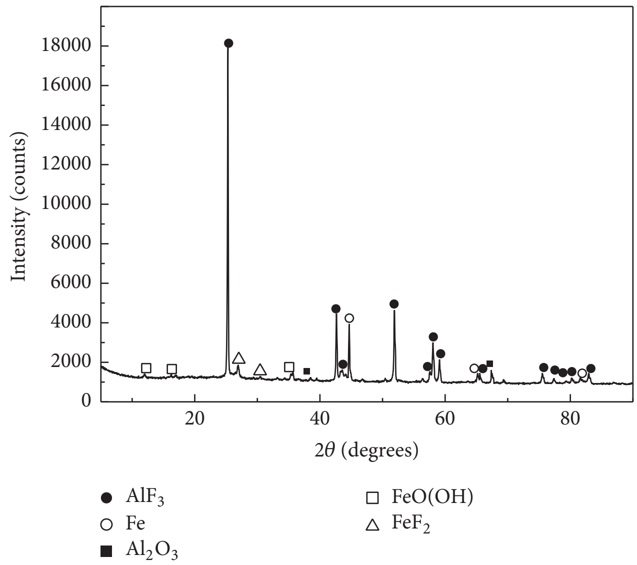


Figure 10: XRD results of type B materials after drop-weight test.

is 150 μs, while the ignition time of type B specimens is 100 μs, indicating that type A specimens are more insensitive.

Interestingly, we observed a High Temperature Metal Slag Spray (HTMSS) phenomenon in both kinds of materials upon dynamic compression. However, when PTFE/Al or PTFE/Al/W was impacted, only bright white light appeared with no HTMSS phenomenon [4, 7, 21]. Therefore, Fe_2O_3 is definitely involved in the reaction and local thermite reaction may be triggered, and the XRD of the reaction products of type B materials (Figure 10) confirmed this. The reaction of type A specimens is too weak to allow the products to be analyzed effectively, so the XRD results are not given here. As shown in Figure 10, Fe and Al_2O_3 were found in the reaction products, demonstrating the occurrence of reaction between Al and Fe_2O_3; that is,

$$Al + Fe_2O_3 = Fe + Al_2O_3 \tag{4}$$

According to the results of quasi-static compression, we believe that the reaction between PTFE and Al occurs preferentially, and the reaction between Al and Fe_2O_3 followed, which is triggered by the huge energy generated from the reaction between PTFE and Al and from the impact of drop mass. However, a small amount of FeF_2 and FeO(OH) was also found in the products of type B specimens. FeF_2 is probably from the complex reaction between Fe_2O_3 and PTFE. FeO(OH) may be from the following reaction:

$$4Fe^{2+} + O_2 + 6H_2O \longrightarrow 4FeO\,(OH) + 8H^+ \tag{5}$$

Fe^{2+} is probably derived from FeF_2, when contacting with O_2 and H_2O in the air; Fe^{2+} will be converted to FeO(OH) under some conditions. However, FeF_2 and FeO(OH) are all traceable, which have little effect on the energy release of the material, so they both can be regarded as impurity and can be ignored temporarily; the specific details will be studied later.

4. Conclusions

In this study, two kinds of PTFE/Al/Fe_2O_3 composites were prepared and their mechanical and reaction behavior under quasi-static compression and dynamic impact were investigated. The main conclusions are shown below.

(1) Under quasi-static compression, with the increase of PTFE content, the strength of the materials is increased (from 37.1 Mpa to 77.2 Mpa), and the fracture mode changes from brittle to ductile. Ignition phenomenon was found in type B materials (75PTFE-14Al-11Fe_2O_3) and not in type A materials (50PTFE-28Al-22Fe_2O_3).

(2) The residuals of the reacted specimen after quasi-static compression were analyzed and no reaction of Al and Fe_2O_3 was found. This is due to the high temperature threshold of thermite reaction, the insufficient contact of Al and Fe_2O_3, and the low reactivity of Fe_2O_3.

(3) In the case of drop-weight test, more energy is needed for type A specimens to ignition than type B specimens. And it takes less time to ignite for the former (100 μs) than the latter (150 μs), indicating that type A specimens are more insensitive.

(4) A High Temperature Metal Slag Spray (HTMSS) phenomenon had been observed in PTFE/Al/Fe_2O_3 composites in the drop-weight test, which indicates that the reaction between Al and Fe_2O_3 was triggered.

(5) In PTFE/Al/Fe_2O_3 system, the reaction between PTFE and Al precedes the reaction between Al and Fe_2O_3.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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