

Research on Dust Explosion Behavior of Nano-Sized Titanium Powders

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ABSTRACT

The purpose of this study is mainly to explore the dust explosion behavior of nano-sized titanium (Ti) powder. First, three kinds of Ti powders with average particle sizes of 35, 75 and 100 nm were examined by scanning electron microscopy (SEM) and X-ray diffraction (XRD) to confirm the particle size and purity. An environment operation box was used to avoid the oxidation of nano-sized Ti powder samples during the weighing process. Subsequently, the dust explosion characteristics were measured by 20-L explosion sphere apparatus, including the maximum explosion pressure ($P_{\max}$), maximum rate of pressure rise $[(dP/dt)_{\max}]$, minimum explosion concentration (MEC) and deflagration index (K_{St}), and the minimum ignition energy (MIE) and flame propagation velocity (FPV) were evaluated by modified Hartmann tube. The experimental results showed that the maximum $P_{\max}$, $(dP/dt)_{\max}$ and K_{St} values increased with decrease of average particle size. The MEC value increased with increase of average particle size. The MIE values were all less than 1 mJ. Therefore, it is very dangerous to handle nano-sized Ti powder in the process industries. In addition, the maximum FPV decreased with increase of particle size, and increased with increase of ignition energy used. According to the dust explosion class, these three nano-sized Ti powders were classified as Class 2, which means that they have a strong explosive power.

Keywords: nano-sized Ti powder, dust explosion behavior, flame propagation velocity

奈米級鈦粉粉塵爆炸現象之研究

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摘 要

本研究主要探討奈米級鈦粉的粉塵爆炸現象，平均粒徑分別為 35、75 和 100 nm 的三種奈米級鈦粉，先藉由掃描式電子顯微鏡(SEM)和 X 光繞射分析儀(XRD)確認其尺寸和純度，並使用環境操作箱在稱量過程避免奈米級鈦粉樣品的氧化。後續，粉塵爆炸特性使用 20 公升爆炸鋼球來量測，包括最大爆炸壓力($P_{\max}$)、最大升壓速率 $[(dP/dt)_{\max}]$ 、最小爆炸濃度(MEC)及爆燃指數(K_{St})，最小點火能量(MIE)和火焰傳播速度(FPV)則使用改良型 Hartmann 管來評估。實驗結果顯示，最大的 $P_{\max}$ 、 $(dP/dt)_{\max}$ 和 K_{St} 值隨平均粒徑的減小而增加，MEC 值則隨著平均粒徑的增大而增加，MIE 值均小於 1 mJ，因此，在工業程序中處理奈米級鈦粉是非常危險的；此外，最大的火焰傳播速度隨著粒徑的增加而減小，並且隨著所使用點火能量的增加而增加；根據粉塵爆炸等級的分類，這三種奈米級鈦粉被歸類為第 2 級，亦即具有強的爆炸威力。

關鍵詞：奈米級鈦粉、粉塵爆炸現象、火焰傳播速度

I. INTRODUCTION

The dust explosion is the very rapid combustion of fine combustible dust particles suspended in air, often but not always in an enclosed location. Some basic conditions must exist at the same time for the occurrence of dust explosion: combustible dust, dispersed in air, a concentration above the flammable limit, the presence of sufficiently energetic ignition source, and confinement of dust cloud [1]. If one of these conditions is missing, no dust explosion will occur. Combustible dust is any fine substance that can catch fire and cause an explosion when it is mixed with air, which usually comes from solid organic materials (such as sugar, flour, grain, wood dust, etc.), metal and non-metal inorganic materials. In the processing industry that handles combustible powders, dust explosions are a serious hazard that can cause death, injury, and property damage. The catastrophic accidents related to dust explosions are reported every year. According to the data collected in the accident database and reported in the 2020 Combustible Dust Accident Report, between the years of 2016 and 2020, there were 159 accidents involving dust explosions in the U.S., resulting in 13 worker deaths and 145 injuries. Looking at the global loss overview in 2020, 70% of the fatalities recorded occurred due to dust explosions. Of the injuries, 73% occurred due to explosions and 27% occurred due to fires [2]. Therefore, it is necessary to check any dust-generating activities to confirm whether there is a risk of dust explosion.

Compared with micron-sized metal particles, nano-sized metal particles have recently attracted considerable attention due to their unique and excellent physical and chemical properties. [3-4]. Nano-sized titanium (Ti) powder has high purity, uniform small particle size and high surface activity [5-8]. Therefore, it is widely used in high-tech industries such as machinery, electronics, aviation, metallurgy, chemistry and environment [9-13]. In addition, nano-sized Ti powder is also used in solid-fuel rocket propellants and pyrotechnics. Recently, polymer coatings with nano-sized Ti powder

additives have been gradually used in water injection systems, oil well pipes and oil pipelines to prevent corrosion [14]. Like many metal powders, Ti powder has flammable and explosive properties, and due to its increasing use and insufficient awareness of specific hazards, many dust explosion accidents have been caused in recent years [2, 15-17]. In general, the dust explosion process of nanometal particles involves complex chemical reactions and belongs to a kinetically controlled combustion regime, which is different from the reaction of micro metal particles by diffusion control [18-21]. Therefore, the surrounding pressure plays an important role. This is because increasing the surrounding pressure can inhibit the gas phase reaction to enhance the heterogeneous surface reaction, thereby increasing the flame temperature and reducing the burning time [22, 23]. This also means that the condensed phase remains above surrounding temperature.

At present, studies on the dust explosion of nano-sized Ti particles mainly focus on the effects of particle characteristics and external conditions on the reaction, such as particle size, dust concentration, temperature, pressure, and O₂ concentration [24-28]. Boilard et al. have reported that nano-sized Ti powder is very sensitive and can self-ignite under the appropriate conditions [24]. The explosive properties of nano-sized Ti powder can be suppressed by adding nano-sized titanium dioxide (TiO₂) powder to the dust mixture. The experimental results reported by Wu et al. showed that the minimum ignition energy (MIE) of all nano-sized Ti powders was less than 1 mJ [25]. Boilard has also used experimental methods to obtain the explosion parameters of explosion severity [maximum explosion pressure ($P_{\max}$) and maximum rate of pressure rise ($(dP/dt)_{\max}$)] and explosion probability [minimum ignition energy (MIE), minimum explosion concentration (MEC) and minimum ignition temperature (MIT)] [28]. It is clear from this study that greater dust explosion hazards are present when nano-sized Ti dust is involved. Furthermore, there are many reports of dust explosion accidents in related literatures involving flame propagation and secondary

explosions [29-31]. However, the details of the flame propagation of nano-sized Ti powder are still unclear. In order to reduce the hazards of dust explosions, this is a topic worthy of discussion.

In this study, the dust explosion behavior of three nano-sized Ti powders with different particle sizes was investigated by experimental methods. An environment operation box was used to prevent the oxidation of nano-sized Ti powder samples during the weighing process. Therefore, the experimental results were more accurate than previous literatures. First, the morphology and size were observed by scanning electron microscopy (SEM) and the purity was identified by X-ray diffraction (XRD). Subsequently, the dust explosion characteristics were measured by 20-L explosion sphere apparatus, including $P_{\max}$, $(dP/dt)_{\max}$, MEC and deflagration index (K_{St}), and MIE and flame propagation velocity (FPV) were determined by modified Hartmann tube. Finally, these experimental results were used to analyze the dust explosion hazard of nano-sized Ti powder.

II. EXPERIMENTAL

2.1 Materials

Three kinds of nano-sized titanium (Ti) powders with average particle sizes of 35 nm (particle size range: 15-45 nm, specific surface area: 50-70 m^2/g , apparent density: 0.15-0.18 g/cm^3), 75 nm (particle size range: 60-85 nm, specific surface area: 40-60 m^2/g , apparent density: 0.16-0.19 g/cm^3) and 100 nm (particle size range: 90-110 nm, specific surface area: 35-55 m^2/g , apparent density: 0.18-0.21 g/cm^3) and purity of 99.9% were used to investigate the dust explosion behavior, which were obtained from Yong-Zhen Technomaterial Co., Ltd. (Taiwan manufacturer).

2.2 Equipments and Experimental Procedures

In the weighing process, an environment operation box made by Yong-Hsin Co., Ltd. (Taiwan manufacturer) was used to avoid the

oxidation of nano-sized Ti powder samples, as shown in Figure 1. It has a size of 80×55×50 cm^3 and is assembled from acrylic plates with a thickness of 1 cm, and can be operated in an inert gas or vacuum environment. Furthermore, the morphology and size were observed by scanning electron microscopy (SEM, Hitachi S-3000H) and the purity was identified by X-ray diffraction (XRD, Bruker D2 Phaser).



Fig. 1. Photo of environment operation box

The dust explosion characteristics of three kinds of nano-sized Ti powders were measured by 20-L explosion sphere apparatus (20-L-Apparatus, Kühner AG), including $P_{\max}$, $(dP/dt)_{\max}$, MEC and K_{St} , as shown in Figure 2. It consists of a hollow sphere made of stainless steel with an internal volume of 20 liters, a dust storage container connected with the chamber through a dust outlet valve, a pair of electrodes holding two pyrotechnic igniters of 5 kJ, and two piezoelectric pressure transducers [32].

The experimental procedure of the American Society for Testing and Materials (ASTM) E1226 test method is as follows [33]. First, a certain amount of nano-sized Ti powder sample was put into the dust container. The explosion chamber was evacuated to 0.4 bar absolute pressure, and then an automatic test sequence was started to pressurize to 20 bar absolute pressure. Afterward, the nano-sized Ti powder sample was dispersed from the dust container to the explosion chamber through the quick acting valve and the rebound nozzle. The rebound nozzle ensured an even distribution of dust in the explosion chamber. The control system activated the igniters located in the center of the explosive sphere, and the ignition delay time was 60 ms after the dust was dispersed. Two pressure transducers were used

to measure $P_{\max}$ and $(dP/dt)_{\max}$. The severity of dust explosions is usually expressed by the deflagration index (K_{St}), which is defined as

$$K_{St} = (dP/dt)_{\max} V^{1/3} \quad (1)$$

where V represents the chamber volume (m^3) of the experimental apparatus.



Fig. 2. Photo of 20-L explosion sphere apparatus (Manual for 20-l-Apparatus)

The MIE values of three kinds of nano-sized Ti powders was measured by modified Hartmann tube (MIKE 3, Kühner AG), as shown in Figure 3. It consists of a vertical cylindrical glass tube with an internal volume of 1.2 liters and a diameter of 7 cm mounted onto a dust dispersion system, and is fitted with two electrodes. The electrodes are connected to a circuit that produces an electrical spark of known energy. On top of the glass tube, a hinged cover allows the pressure to be released in the case of a dust fire or explosion inside the tube.

The experimental procedure of the BS EN 13821 experiment standard (British Standards Institution, 2006) is as follows. First, a certain amount of nano-sized Ti powder sample was put into the dust dispersion cup, which was blown into the tube with compressed air, and passed through the ignition source. If a flame was observed, the energy of electrical spark was reduced until no flame was seen for 10

consecutive tests. The MIE value was between the lowest energy (IE) value at which ignition occurred and the energy (NIE) value at which ignition was not observed in at least 10 consecutive experiments.

Cesana and Siwek developed a statistical model to calculate MIE based on experimental results, as shown below [34].

$$\log MIE = \log IE - N_I \times \frac{\log IE - \log NIE}{N+1} \quad (2)$$

Where NI is the number of successful ignitions under IE , and N represents the total number of tests performed at the NIE energy level. At least five concentrations need to be measured to calculate the probability of ignition.



Fig. 3. Photo of modified Hartmann tube (Manual for modified Hartmann tube)

The modified Hartmann tube was also used to measure the FPV of three kinds of nano-sized Ti powders. A high-speed video camera (FASTCAM SA1.1, Photron) was employed to record the flame propagation process, which was composed of a processor, an imager and a keypad, and could shoot 3000 frames per second. Furthermore, the digital-image processing device was connected to the video camera

through an image acquisition card, and the shot video was edited by image processing software to determine the FPV.

III. RESULTS AND DISCUSSION

3.1 Identification of Nano-sized Ti Powder Samples

Figure 4 shows the SEM images of Ti powders with average particle sizes of 35, 75 and 100 nm. It can be seen that the three nano-sized Ti powders are all spherical and easy to agglomerate. The XRD patterns are shown in Figure 5. The diffraction peaks appear at 35.17° , 38.46° , 40.25° , 53.09° , 63.11° , 70.77° , 76.10° and 77.55° corresponding to the (100), (002), (101), (102), (110), (103), (112) and (201) planes, which matches well with the standard pattern of Ti (JCPDS No. 89-5009). In addition, the standard XRD pattern of TiO_2 powder (JCPDS No. 21-1272) is also attached to Figure 5 for comparison. The diffraction peaks appear at 25.27° , 36.91° , 37.77° , 38.52° , 48.01° and 62.07° corresponding to the (101), (103), (004), (112), (200) and (204) planes. The XRD patterns of three nano-sized Ti powders do not show obvious diffraction peaks of TiO_2 powder. This result indicates that the purity of nano-sized Ti powders is very high, and the content of TiO_2 is very low.



Fig. 4. SEM images of nano-sized Ti powders

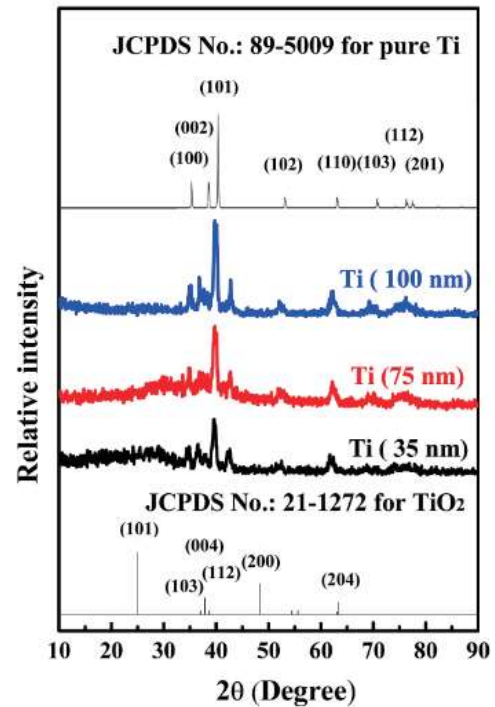


Fig. 5. XRD patterns of nano-sized Ti powders

3.2 Measured Results of $P_{\max}$, $(dP/dt)_{\max}$, and MEC

20-L-Apparatus was used to measure the dust explosion characteristics of three kinds of nano-sized Ti powders, including $P_{\max}$, $(dP/dt)_{\max}$, and MEC. The tested concentration range was selected as 10 to 1000 g/m^3 , including 10, 20, 30, 60, 125, 250, 500, 750 and 1000 g/m^3 . Figure 6 and Figure 7 display the relationships between $P_{\max}$ and $(dP/dt)_{\max}$ versus dust concentration, respectively. The measured results show that the Ti powder with an average particle size of 35 nm has a maximum $P_{\max}$ of 7.0 bar at a dust concentration of 500 g/m^3 , and a maximum $(dP/dt)_{\max}$ of 984 bar/s at a dust concentration of 750 g/m^3 . The maximum K_{St} calculated by equation (1) is 267 mbar/s . According to the OSHA hazard communication guidance for combustion dusts, the dust explosion of Ti powder with an average particle size of 35 nm can be classified as Class 2, which means that it has a strong explosive power. When the average particle size of Ti powder is 75 nm, the maximum $P_{\max}$ and $(dP/dt)_{\max}$ are 6.6 bar and 786 bar/s at a dust concentration of 750 g/m^3 , respectively. Its maximum K_{St} is 213 mbar/s .

bar/s, which can also be classified as Class 2. When the average particle size of Ti powder is 100 nm, the maximum $P_{\max}$ is 6.2 bar at a dust concentration of 750 g/m³, and the maximum $(dP/dt)_{\max}$ is 739 bar/s at a dust concentration of 1000 g/m³. Its maximum K_{St} value is 201 m bar/s, which can also be classified as Class 2. In addition, it is found that the maximum $P_{\max}$, maximum $(dP/dt)_{\max}$ and K_{St} values increase with the decrease of the average particle size of nano-sized Ti powder. Boilard et al. have reported that the maximum $P_{\max}$ and maximum $(dP/dt)_{\max}$ of Ti powders with particle sizes of 40-60, 60-80 and 150 nm are 6.1, 5.7, 5.4 bar and 884, 752, 713 bar/s, respectively [24]. These experimental results are similar but smaller than those of this study. The MEC of Ti powders with average particle sizes of 35, 75 and 100 nm are 30, 30, and 60 g/m³, respectively. All the experimental results of dust explosion characteristics are listed in Table 1.

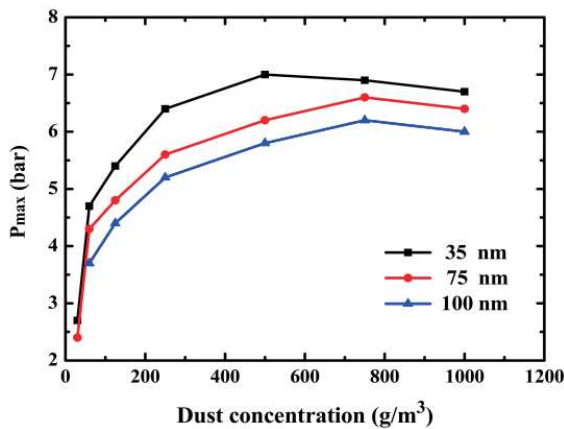


Fig. 6. Relationships between $P_{\max}$ versus dust concentration for dust explosions of Ti powders with average particle sizes of 35, 75 and 100 nm

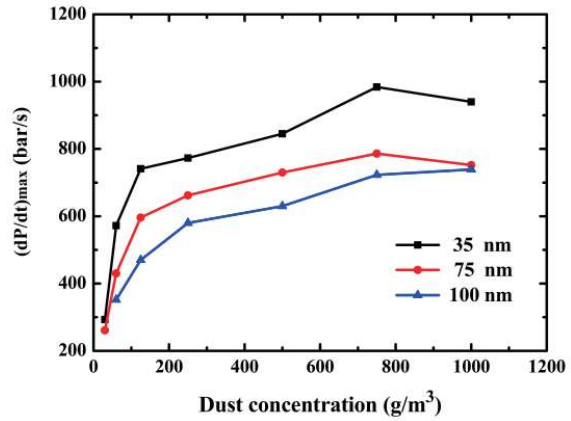


Fig. 7. Relationships between $(dP/dt)_{\max}$ versus dust concentration for dust explosions of Ti powders with average particle sizes of 35, 75 and 100 nm

3.3 Measured Results of MIE and FPV

Modified Hartmann tube was used to measure the MIEs and FPVs of three kinds of nano-sized Ti powders. The test conditions selected seven kinds of electric spark ignition energies of 1, 3, 10, 30, 100, 300 and 1000 mJ, and six kinds of dust weights of 300, 600, 900, 1200, 1500 and 1800 mg. The ignition delay time was set to 120 ms. The MIE measurement results of Ti powders with average particle sizes of 35, 75 and 100 nm are shown in Figure 8. The solid square indicates successful ignition at this ignition energy. The MIE values calculated by equation (2) are all less than 1 mJ, which are also listed in Table 1. Therefore, it is very dangerous to handle nano-sized Ti powder in the process industries. In addition, the MIE of nano-sized Ti powder is smaller compared with that of micro-sized Ti powder, and is consistent with the report of Eckhoff that the smaller the particle size, the smaller the MIE [17].

Table 1. Explosion characteristics of nano-sized Ti powders

Average particle size (nm)	Maximum $P_{\max}$ (bar)	Maximum $(dP/dt)_{\max}$ (bar/s)	Maximum K_{St} (m bar/s)	Explosion class	MEC (g/m ³)	MIE (mJ)
35	7.0	984	267	2	30	<1
75	6.6	786	213	2	30	<1
100	6.2	739	201	2	60	<1

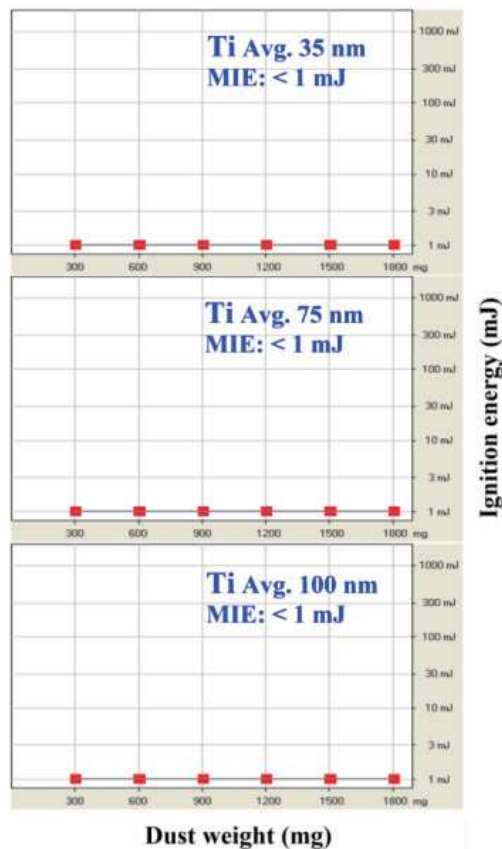


Fig. 8. MIE measurement results of nano-sized Ti powders

The FPV test conditions selected three kinds of electric spark ignition energies of 1, 30 and 100 mJ and six kinds of dust weights of 300, 600, 900, 1200, 1500 and 1800 mg. The ignition delay time was also set to 120 ms. Figure 9 shows the snapshots of flame propagation of dust explosion taken by a high-speed video camera for Ti powder with an average particle size of 35 nm under the conditions of different dust weights and an ignition energy of 1 mJ. The relationships between flame propagation velocity versus time were measured and calculated as shown in Figure 10. It is found that the Ti powder with an average particle size of 35 nm has a maximum FPV of 2.77 cm/ms at a dust weight of 1500 mg using an ignition energy of 1 mJ. In addition, under the conditions of ignition energies of 30 and 100 mJ, the maximum FPVs are also found to be 2.83 and 2.94 cm/ms at a dust weight of 1500 mg, respectively. Figure 11 shows the relationships between maximum FPV versus dust weight for dust explosion of Ti

powder with an average particle size of 35 nm at ignition energies of 1, 30 and 100 mJ. There is a noteworthy phenomenon in Figure 11 that the maximum FPVs of these three ignition energies exhibits a trend of increasing first and then decreasing with increase of dust weight. This means that the maximum FPV increases with increasing amount of dust, but too much dust delays the flame propagation of dust explosion. Furthermore, the maximum FPV increases with increasing ignition energy.

For the Ti powder with an average particle size of 75 nm, Figure 12 shows the snapshots of flame propagation of dust explosion under the conditions of different dust weights and an ignition energy of 1 mJ. Figure 13 display the corresponding relationships between flame propagation velocity versus time. The maximum FPV is 2.14 cm/ms at a dust weight of 1200 mg. In addition, under the conditions of ignition energies of 30 and 100 mJ, the maximum FPVs are also found to be 2.25 and 2.43 cm/ms at a dust weight of 1200 mg, respectively. Figure 14 shows the relationships between maximum FPV versus dust weight for dust explosion of Ti powder with an average particle size of 75 nm at ignition energies of 1, 30 and 100 mJ. It also presents a similar trend to Ti powder of 35 nm, the maximum FPVs of these three ignition energies is increasing first and then decreasing with increasing dust weight. In addition, the maximum FPV also increases with increasing ignition energy.

For the Ti powder with an average particle size of 100 nm, Figure 15 shows the snapshots of flame propagation of dust explosion under the conditions of different dust weights and an ignition energy of 1 mJ. Figure 16 display the corresponding relationships between flame propagation velocity versus time. It can be seen that the maximum FPV is 1.59 cm/ms at a dust weight of 900 mg. In addition, under the conditions of ignition energies of 30 and 100 mJ, the maximum FPVs are also found to be 1.85 and 2.01 cm/ms at a dust weight of 900 mg, respectively. The variation trends of maximum FPV with dust weight and ignition energy are the same as those of 35 and 75 nm Ti powders, as shown in Figure 17.



Fig. 9. Snapshots of flame propagation of dust explosion for Ti powder with an average particle size of 35 nm [Dust weight: (A) 300, (B) 600, (C) 900, (D) 1200, (E) 1500, and (F) 1800 mg; Ignition energy: 1 mJ]

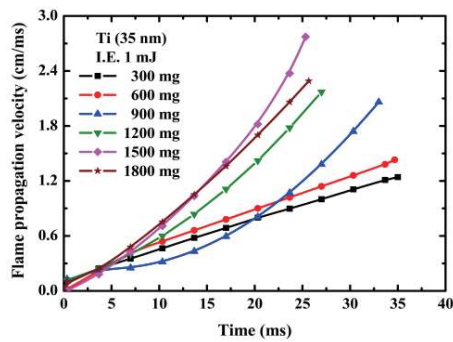


Fig. 10. Relationships between FPV versus time for dust explosion of Ti powder with an average particle size of 35 nm under the conditions of different dust weight and ignition energy of 1 mJ

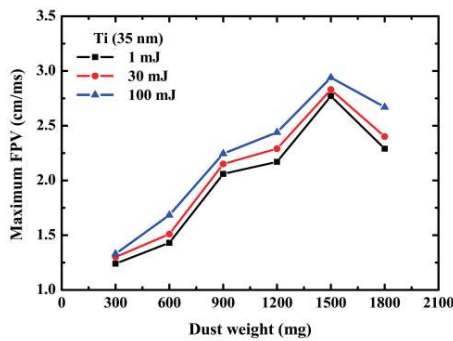


Fig. 11. Relationships between maximum FPV versus dust weight for dust explosion of Ti powder with an average particle size of 35 nm at ignition energies of 1, 30 and 100 mJ

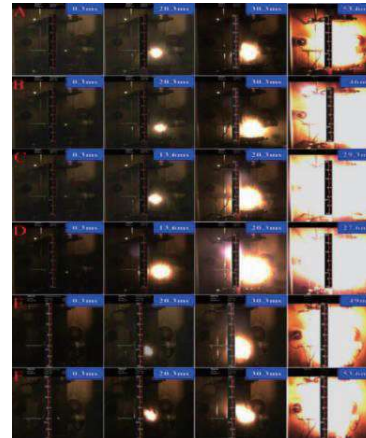


Fig. 12. Snapshots of flame propagation of dust explosion for Ti powder with an average particle size of 75 nm [Dust weight: (A) 300, (B) 600, (C) 900, (D) 1200, (E) 1500, and (F) 1800 mg; Ignition energy: 1 mJ]

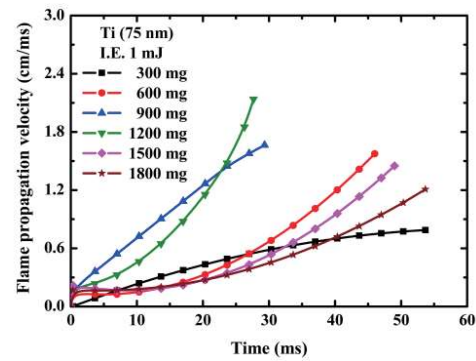


Fig. 13. Relationships between FPV versus time for dust explosion of Ti powder with an average particle size of 75 nm under the conditions of different dust weight and ignition energy of 1 mJ

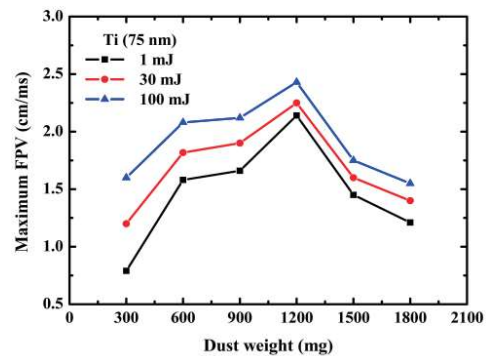


Fig. 14. Relationships between maximum FPV versus dust weight for dust explosion of Ti powder with an average particle size of 75 nm at ignition energies of 1, 30 and 100 mJ

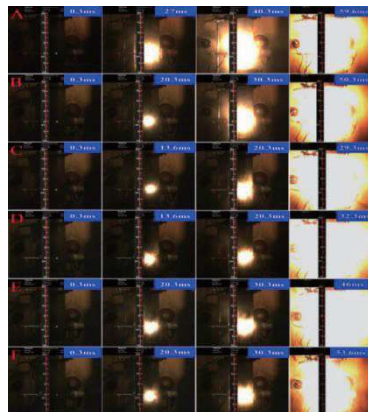


Fig. 15. Snapshots of flame propagation of dust explosion for Ti powder with an average particle size of 100 nm [Dust weight: (A) 300, (B) 600, (C) 900, (D) 1200, (E) 1500, and (F) 1800 mg; Ignition energy: 1 mJ]

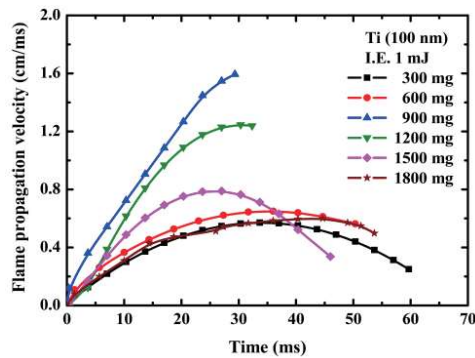


Fig. 16. Relationships between FPV versus time for dust explosion of Ti powder with an average particle size of 100 nm under the conditions of different dust weight and ignition energy of 1 mJ

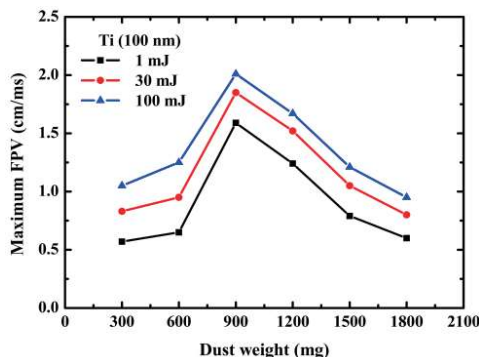


Fig. 17. Relationships between maximum FPV versus dust weight for dust explosion of Ti powder with an average particle size of 100 nm at ignition energies of 1, 30 and 100 mJ

The maximum FPVs of these three nano-sized Ti powders at the ignition energies of 1, 30 and 100 mJ are listed in Table 2. It is also worth noting that the maximum FPV decreases with increasing average particle size of nano-sized Ti powder.

Table 2. Maximum FPV of dust explosion of Ti powders

Average particle size (nm)	Ignition energy (mJ)	Maximum FPV (cm/ms)
35	1	2.77
	30	2.83
	100	2.94
75	1	2.14
	30	2.25
	100	2.43
100	1	1.59
	30	1.85
	100	2.01

VI. CONCLUSIONS

This study used the experimental methods to explore the dust explosion behavior of nano-sized Ti powder. Based on experimental and analytical results, the following conclusions are obtained:

- (1) The Ti powder with an average particle size of 35 nm has a maximum $P_{\max}$ of 7.0 bar, a maximum $(dP/dt)_{\max}$ of 984 bar/s and a maximum K_{St} of 267 m bar/s. When the average particle size of Ti powder is 75 nm, these values are 6.6 bar, 786 bar/s and 213 m bar/s, respectively. When the average particle size of Ti powder is 100 nm, these values are 6.2 bar, 739 bar/s and 201 m bar/s, respectively. The maximum $P_{\max}$, maximum $(dP/dt)_{\max}$ and K_{St} values increase with the decrease of average particle size. The dust explosion of Ti powders with average particle sizes of 35, 75 and 100 nm can be classified as Class 2, which means that they have a strong explosive power. In addition,

the MEC values of Ti powders with average particle sizes of 35, 75 and 100 nm are 30, 30, and 60 g/m³, respectively.

- (2) The MIE values of Ti powders with average particle sizes of 35, 75 and 100 nm are all less than 1 mJ. Therefore, it is very dangerous to handle nano-sized Ti powder in the process industries.
- (3) Under the conditions of ignition energies of 1, 30 and 100 mJ, the maximum FPs of Ti powder with an average particle size of 35 nm are 2.77, 2.83 and 2.94 cm/ms, respectively. When the average particle size of Ti powder is 75 nm, the maximum FPs are 2.14, 2.25 and 2.43 cm/ms, respectively. When the average particle size of Ti powder is 100 nm, the maximum FPs are 1.59, 1.85 and 2.01 cm/ms, respectively. A noteworthy phenomenon is that the maximum FPV of nano-sized Ti powder under these three ignition energy conditions exhibits a trend of increasing first and then decreasing with increasing dust weight. This means that the maximum FPV increases with increasing amount of dust, but too much dust delays the flame propagation of dust explosion. In addition, the maximum FPV of these three nano-sized Ti powders decreases with increase of particle size, and increases with increase of ignition energy used.

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