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### ABSTRACT

Explosibility of micron- and nano-titanium was determined and compared according to explosion severity and likelihood using standard dust explosion equipment. ASTM methods were followed using a Siwek 20-L explosion chamber, MIKE 3 apparatus and BAM oven. The explosibility parameters investigated for both size ranges of titanium include explosion severity (maximum explosion pressure ( $P_{max}$ ) and size-normalized maximum rate of pressure rise ( $K_{St}$ )) and explosion likelihood (minimum explosible concentration (MEC), minimum ignition energy (MIE) and minimum ignition temperature (MIT)). Titanium particle sizes were –100 mesh (<150 µm), –325 mesh (<45 µm),  $\leq 20$  µm, 150 nm, 60–80 nm, and 40–60 nm. The results show a significant increase in explosion severity as the particle size decreases from –100 mesh with an apparent plateau being reached at –325 mesh and  $\leq 20$  µm. Micron-size explosion severity could not be compared with that for nano-titanium due to pre-ignition of the nano-powder in the 20-L chamber. The likelihood of an explosion increases significantly as the particle size decreases into the nano range. Nano-titanium is very sensitive and can self-ignite under the appropriate conditions. The explosive properties of the nano-titanium can be suppressed by adding nano-titanium dioxide to the dust mixture. Safety precautions and procedures for the nano-titanium are also discussed. © 2013 Elsevier Ltd. All rights reserved.

#### 1. Introduction

The current research is aimed at investigating the explosion behaviour of hazardous materials in relation to particle size. The materials of study are titanium powders having size distributions in both the micron- and nano-size ranges. Dust explosions have been well documented for particles falling in the micron-range for different types of metals (Cashdollar & Zlochower 2007). As the size is further decreased into the nano-range, new physical and chemical properties can occur changing the severity and likelihood of a dust explosion.

At the nano-scale, typically between 1 and 100 nm, properties of well-known substances change and they may exhibit physical (added strength) and chemical (increased reactivity, fluorescence and conduction) changes. The bulk properties no longer hold and the surface properties of the material take precedence. For example, bulk titanium and zinc are solid, opaque metals. Once in the nano-scale, the surface properties allow the metal to become transparent (Pritchard, 2004). The small size of the nano-particles

increases the reactive surface area within a bulk sample. This allows for the different physical and chemical properties of nanoparticles. These properties are highly desirable for technological advancements but also come at a cost, with an increased explosion potential. In the current paper, six titanium samples (-100 mesh, -325 mesh,  $\leq 20 \,\mu$ m, 150 nm, 60–80 nm, and 40–60 nm) are investigated with standard dust explosion equipment to determine explosion severity and likelihood. The results, limitations of the research, and safety precautions are all discussed.

### 2. Literature review

Dust explosion research involving nano-materials has been limited, but some research groups have performed tests with various materials to better understand the properties of nano-size powders. Holbrow et al. (2010), with the UK Health and Safety Executive, performed dust explosion tests with different types of nano-materials including metals (aluminum, zinc, copper and iron) and carbon nanotubes. Experiments were performed in a specially designed 2-L explosion chamber (Holbrow et al. 2010). Explosions using a 20-L sphere with various nano-particles were also investigated by Vignes et al. (2009). Materials included carbon black, multi-walled carbon nanotubes and aluminum.







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Using the nano-aluminum (100 nm and 200 nm) results from Vignes et al. (2009), Dufaud, Vignes, Henry, Perrin and Bouillard (2011) compared the nano-aluminum to micron-size aluminum. The results indicated a maximum explosion pressure,  $P_{\text{max}}$ , of 8.2 bar(g) and 9.5 bar(g) while the maximum rate of pressure rise, (dP/dt)<sub>max</sub>, was 1340 bar/s and 2480 bar/s for the 100 nm and 200 nm samples, respectively. At the micron-scale, 3 µm and 7 µm aluminium gave a  $P_{\text{max}}$  of 9.8 bar(g) and 9.1 bar(g) and (dP/dt)<sub>max</sub> was 2090 bar/s and 1460 bar/s, respectively. Explosion severity in this case was limited by the size of the particles with the peak occurring around 1 µm (Dufaud et al., 2011).

Likewise, Wu, Ou, et al. (2010), Wu, Kuo, Wang, Wu & Hsiao (2010) performed nano-dust explosions with aluminum having average particle sizes of 35 nm and 100 nm, resulting in a  $P_{\text{max}}$  of 7.3 bar(g) and 12.5 bar(g), and (dP/dt)<sub>max</sub> of 1286 bar/s and 1090 bar/s, respectively. These results can be compared to larger micron-aluminum powder data obtained from Eckhoff (2003). For a mean particle size of 22 µm,  $P_{\text{max}}$  was 12.5 bar(g) and (dP/dt)<sub>max</sub> was 1474 bar/s (Eckhoff, 2003).

Wu, Chang, and Hsiao (2009) tested the minimum ignition energy (MIE) of micron- and nano-titanium. As the particle size was decreased from 45  $\mu$ m to 3  $\mu$ m, the MIE dropped from 21.9 mJ to <1 mJ. For three nano-titanium sizes (35 nm, 75 nm and 100 nm), the MIE was lower than 1 mJ (Wu et al., 2009). Nano-aluminum and carbon nanotubes also have similarly low MIE values. Although agglomeration of nano-particles may occur and thus become a limiting factor in determining values of  $P_{max}$  and  $(dP/dt)_{max}$ , nano-particle agglomeration does not seem to affect the minimum ignition energy of these materials.

#### 3. Equipment and methodology

The explosibility parameters investigated for the nano- and micron-size ranges of titanium include maximum explosion pressure ( $P_{max}$ ), size-normalized maximum rate of pressure rise ( $K_{St}$ ), minimum explosible concentration (MEC), minimum ignition energy (MIE), and minimum ignition temperature (MIT). American Society for Testing and Materials (ASTM) methods were followed using standard dust explosibility test equipment: Siwek 20-L explosion chamber, MIKE 3 apparatus and BAM oven. The applicable ASTM methods are ASTM (2003, 2006, 2007 and 2010).

Fig. 1 shows a schematic of the Siwek 20-L chamber with its corresponding components. Nitrogen was used in place of air as the dust dispersion medium for all nano-titanium testing. This was done to prevent pre-ignition of the powder in the dust storage chamber (item 3 in Fig. 1) as observed by Wu, Ou, et al. (2010), Wu,

Kuo, Wang, Wu & Hsiao (2010). A lower-than-usual vacuum was therefore created in the 20-L chamber and oxygen was backfilled to create an elevated oxygen atmosphere (prior to dust dispersion). Once the dispersing nitrogen and the elevated oxygen level mixed, atmospheric conditions would be achieved with 79% nitrogen and 21% oxygen. CaRo11 (Kuhner) dust was tested using this method to establish the procedural validity. The results from nitrogen dispersion and air dispersion were well-correlated. Therefore, all nano-titanium tests were performed with nitrogen dispersal.

## 4. Materials

Titanium was chosen for the current research due to its reactivity and the availability of existing data for dust explosibility at the micron-scale. Six sample sizes were selected: -100 mesh ( $<150 \mu$ m), -325 mesh ( $<45 \mu$ m),  $\leq 20 \mu$ m, 150 nm, 60–80 nm, and 40–60 nm.

Particle size distributions were determined to characterize the micron-size titanium. The powder manufacturer's literature states that the two smaller micron-titanium samples are nominally  $\leq$ 20 µm and <45 µm (-325 mesh) – but are sized differently; a single-point BET surface area analysis is used to determine the size of the  $\leq$ 20 µm titanium while a sieve analysis is used to analyze the -325 mesh titanium. According to the manufacturer, the BET surface area analysis measures the average unagglomerated particle size. However, traditional sieve analysis measures what is essentially the particle size distribution of agglomerated particles. This can be seen in the sieve analyses performed in the current work and shown in Table 1. Particle sizes of the nano-titanium were taken to be as reported by the manufacturer and no further size analysis was performed.

A scanning electron micrograph (SEM) of the  $\leq 20 \ \mu m$  titanium sample is shown in Fig. 2. Individual particles of titanium are not spherical but have a granular shape. Agglomerates occur for this sample, as illustrated in Fig. 2b. Two types of agglomerates are present; larger titanium particles are covered by smaller titanium "bits", and medium-sized particles are joined to form a larger particle. Fig. 3 shows the 150 nm sample. While the individual particles are of course much smaller, agglomerates are still clearly present. The nano-agglomerates varied in composition between approximately 50 particles and thousands of particles.

## 5. Results

Testing of the micron-size samples has been completed; nanotitanium testing is currently underway and will be completed by



Fig. 1. Siwek 20-L chamber.

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