Explosibility of Micron- and Nano-Size Titanium Powders

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 ($P_{\text{max}}$) and size-normalized maximum rate of pressure rise ($K_{\text{St}}$)) 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), $\leq20$ 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 $\leq20$ 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.

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 nano-particles. 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, ≤20 µm, 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.

Using the nano-aluminum (100 nm and 200 nm) results from Vignes et al. (2009), Dufaud et al. (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, \((dP/dt)_{\text{max}}\), 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 \((dP/dt)_{\text{max}}\) 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 et al. (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 \((dP/dt)_{\text{max}}\) 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 \((dP/dt)_{\text{max}}\) was 1474 bar/s (Eckhoff 2003).

Wu et al. (2009) tested the minimum ignition energy (MIE) of micron- and nano-titanium. As the particle size was decreased from 45 µm to 3 µm, 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 \( P_{\text{max}} \) and \((dP/dt)_{\text{max}}\), 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 ($P_{\text{max}}$), 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).

Figure 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 Figure 1) as observed by Wu et al. (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 μm), -325 mesh (<45 μm), ≤20 μm, 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 ≤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 ≤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.

**Table 1** Sieve analysis of micron-size titanium powders.

<table>
<thead>
<tr>
<th>Sieve Size</th>
<th>Micron Size</th>
<th>Wt. % Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>≤20 μm</td>
</tr>
<tr>
<td>+325</td>
<td>&gt;45 μm</td>
<td>0.98</td>
</tr>
<tr>
<td>-325+400</td>
<td>45-38 μm</td>
<td>10.55</td>
</tr>
<tr>
<td>-400+450</td>
<td>38-32 μm</td>
<td>5.96</td>
</tr>
<tr>
<td>-450+500</td>
<td>32-25 μm</td>
<td>23.54</td>
</tr>
<tr>
<td>-500</td>
<td>&lt;25 μm</td>
<td>58.98</td>
</tr>
</tbody>
</table>

A scanning electron micrograph (SEM) of the ≤20 μm titanium sample is shown in Figure 2. Individual particles of titanium are not spherical but have a granular shape. Agglomerates occur for this sample, as illustrated in Figure 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. Figure 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.

**Figure 2** Scanning electron micrograph of ≤20 μm titanium powder.
5. RESULTS

Testing of the micron-size samples has been completed; nano-titanium testing is currently underway and will be completed by the time of the symposium. A full description of all experimental results and analysis will be given during the paper presentation.

5.1. Explosion Severity

The explosion severity of the micron-size titanium samples can be seen in Table 2. $P_{\text{max}}$ and $K_{\text{St}}$ both increase significantly (40% and 400% increases, respectively) with decreasing size from -100 mesh to -325 mesh. As the particle size is further decreased to $\leq 20 \mu\text{m}$, $P_{\text{max}}$ decreases by approximately 10% and $K_{\text{St}}$ remains essentially constant. The micron-size data therefore show the expected increase in $P_{\text{max}}$ and $K_{\text{St}}$ with decreasing particle size, but to a limit that may be imposed by particle agglomeration effects (recall the earlier discussion on sample particle size distributions).

<table>
<thead>
<tr>
<th>Sample</th>
<th>$P_{\text{max}}$ [bar(g)]</th>
<th>$(dP/dt)_{\text{max}}$ [bar/s]</th>
<th>$K_{\text{St}}$ [bar·m/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>-100 Mesh</td>
<td>5.5</td>
<td>84</td>
<td>23</td>
</tr>
<tr>
<td>-325 Mesh</td>
<td>7.7</td>
<td>436</td>
<td>118</td>
</tr>
<tr>
<td>$\leq 20 \mu\text{m}$</td>
<td>6.9</td>
<td>420</td>
<td>114</td>
</tr>
</tbody>
</table>

It is difficult to make a direct comparison of explosion severity between the micron- and nano-titanium due to pre-ignition of the nano-dust. Frictional or static sparking during the dispersal sequence ignited the dust before the chemical ignitors were fired. (Dust ignition in the Siwek 20-L chamber is normally accomplished via one or more
chemical ignitors activated at a preset ignition delay time.) As previously described, nitrogen was used as the dispersing gas to prevent an explosion in the external dispersion reservoir (item 3 in Figure 1). However, when the nano-titanium/nitrogen mixture encountered the elevated oxygen concentration in the 20-L chamber (see earlier discussion on this point), immediate ignition occurred.

Consistent with the above discussion, Figure 4 shows a 40-60 nm titanium explosion at a dust concentration of 100 g/m³. Here one sees an overlap of the dust dispersion and explosion steps, with the ignitors firing after the dust has exploded. The pressure values in Figure 4 must be interpreted in light of the fact that the pressure transducers used are piezoelectric and hence measure only dynamic pressure changes, not static values.

![Figure 4 Sample pre-ignition pressure/time trace for nano-titanium.](image)

The pre-ignition of the nano-titanium causes all the dust to be consumed and effectively prevents a typical evaluation of the maximum explosion pressure. The maximum rate of pressure rise can be estimated by a manual evaluation of the pressure/time trace. The explosion is very violent and causes complete combustion – again, even before the chemical ignitors are activated.

All subsequent trials of this type involving nano-titanium were performed without ignitors; results similar to those shown in Figure 4 were obtained. Because of the pre-ignition phenomenon, typical $P_{\text{max}}/K_{\text{st}}$ testing was simply not possible with these materials. Therefore, estimates of the maximum explosion pressure and maximum rate of pressure rise were obtained from the pre-ignition phase of the pressure/time trace (the region to the left of the vertical line at 96 ms in Figure 4). It is particularly noteworthy that these explosibility parameters arise from an explosion scenario with
no external ignition source (i.e., no chemical igniters as per typical Siwek 20-L chamber testing).

Figure 5 gives the maximum rate of pressure rise data acquired to date. For all nano-sizes, a quasi-plateau in the rate of pressure rise occurred at low concentrations. At approximately 125 g/m$^3$, the maximum rate of pressure rise for all three nano sizes was between 550 – 650 bar/s. In the case of the 150 nm titanium, the rate of pressure rise displayed a decrease at concentrations higher than 500 g/m$^3$. Micron-size titanium did not reach its maximum rate of pressure rise until concentrations in the vicinity of 1500 g/m$^3$.

![Figure 5 Maximum rate of pressure rise for nano-titanium dusts.](image)

Surface area clearly plays a major role in the combustion of the different sizes of titanium studied in the current work. At the micron-scale, larger amounts of dust are needed to achieve the same effective surface area as with a smaller mass of nano-titanium. For complete combustion of all the oxygen in the 20-L chamber, high concentrations of micron-size titanium are needed. To react with the same amount of oxygen, much lower amounts of nano-titanium are required. There is thus a high reactivity at very low concentrations for nano-size titanium.

Maximum explosion pressure can be estimated for the nano-titanium samples via the $P_d$ value given by the Kuhner (equipment manufacturer) software. In the usual 20-L procedure, the chamber is partially evacuated to 0.4 bar(a) so the resulting chamber pressure upon dust dispersion and at the time of ignition (chemical ignitor firing) is approximately 1 bar(a). The parameter $P_d$ in the Kuhner software reports the rise in pressure in the 20-L chamber due to dust dispersion. Acceptable values of $P_d$ are in the range of 0.55 – 0.7 bar, meaning that when the value of $P_d$ is added to the initial chamber pressure of 0.4 bar, the sum is approximately 1 bar.
Recalling that the piezoelectric pressure transducers measure only dynamic pressure changes (i.e., not static pressures), $P_d$ values as shown in Figure 4 are a combination of the pressure rise due to dust dispersion and the actual explosion itself. The nano-size $P_d$ results are therefore not directly comparable to the micron-size $P_{\text{max}}$ results. They are, however, self-consistent as a data set as shown in Figure 6. Similar to the case for rate of pressure rise shown in Figure 5, nano-titanium reaches its highest values of “maximum explosion pressure” at lower concentrations than the micron-titanium, which again required dust concentrations around 1500 g/m$^3$.

![Graph showing $P_d$ values vs. concentration for different dust sizes](image)

**Figure 6** $P_d$ values (“maximum explosion pressure”) for nano-titanium dusts.

### 5.2. Explosion Likelihood

The explosion likelihood of a dust can be assessed by measurement of three parameters: minimum ignition energy (MIE), minimum ignition temperature (MIT) and minimum explosible concentration (MEC). Results from the work reported here are given in Table 3.

<table>
<thead>
<tr>
<th>Sample</th>
<th>MIE [mJ] (no inductance)</th>
<th>MIT [°C]</th>
<th>MEC [g/m$^3$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>-100 Mesh</td>
<td>1 – 3</td>
<td>&gt;590</td>
<td>60</td>
</tr>
<tr>
<td>-325 Mesh</td>
<td>1 – 3</td>
<td>460</td>
<td>60</td>
</tr>
<tr>
<td>≤20 µm</td>
<td>&lt;1</td>
<td>460</td>
<td>50</td>
</tr>
<tr>
<td>150 nm</td>
<td>&lt;1</td>
<td>250</td>
<td>40 – 50</td>
</tr>
<tr>
<td>60-80 nm</td>
<td>&lt;1</td>
<td>240</td>
<td>Not determined yet</td>
</tr>
<tr>
<td>40-60 nm</td>
<td>&lt;1</td>
<td>250</td>
<td>Not determined yet</td>
</tr>
</tbody>
</table>
Micron-size titanium has been previously shown to be ignitable at very low spark energies. Randeberg and Eckhoff (2006) demonstrated that 3 µm and 9 µm titanium can ignite at energies as low as 0.012 mJ and 0.36 mJ, respectively. Low MIE values for titanium were also measured in the current work as shown in Table 3 (1 mJ being the lowest spark energy attainable with the MIKE 3 apparatus).

There is, however, a fundamental difference between the MIEs of the micron- and nano-size samples. As shown in Figure 7 for the ≤20 µm sample, ignition at 1 mJ occurred only at a high dust loading of 3000 mg. Figure 8, a MIKE 3 ignition graph for the 150 nm titanium, illustrates the significantly lower dust loadings (as low as 100 mg) required for ignition at 1 mJ. (In Figures 7 and 8, dust amount appears on the x-axis and spark energy on the y-axis; the open boxes indicate no ignition at the particular delay time and the solid boxes indicate ignition.)

MIE tests with dust loadings >900 mg were not attempted for the nano-titanium samples. At even low dust loadings with these materials, the explosion in the MIKE 3 glass tube (modified Hartmann tube) was violent and produced an audible ‘boom’.

**Figure 7** MIE data (no inductance) for ≤20 µm titanium.

**Figure 8** MIE data (no inductance) for 150 nm titanium.
A significant increase in explosion likelihood occurs as MIT decreases with decreasing particle size (Table 3). Large -100 mesh (<150 µm) particles could not be ignited in the BAM oven at 590 ºC (which is the highest temperature attainable with our apparatus). Smaller micron-size particles at -325 mesh (<45 µm) and ≤20 µm ignited at a lower oven temperature of 460 ºC. The minimum ignition temperature of the nano-titanium (all sizes) was in the range of 240 – 250 ºC. These temperatures are obviously much lower than those required to ignite the micron-titanium, thus clearly demonstrating the enhanced potential for nano-titanium ignition by hot surfaces.

It was in fact difficult to determine the minimum ignition temperature of the nano-sizes due to the nature of the powder. Titanium ignition produces flames that are very bright and fast, and temperatures exceeding 350 ºC would cause the nano-titanium to flash-off rapidly. In these tests, a ‘hissing’ sound could be heard as the powder entered the BAM oven upon dispersal. Visual observation of flames exiting the oven was more readily accomplished at temperatures below 300 ºC.

While MEC determination for the micron-size samples in the Siwek 20-L chamber was relatively straightforward, the nano-size samples again posed an experimental challenge – this time due to the previously described pre-ignition of the powder during the dispersion sequence. With the 150 nm titanium, pre-ignition did not occur at low concentrations of 20 and 30 g/m³; these concentrations could therefore be tested with nitrogen dispersion and the chemical ignitor (5 kJ) in place. No explosions in the 20-L chamber were recorded at these conditions, meaning that the MEC of the 150 nm sample is >30 g/m³.

With a dust concentration of 40 g/m³, and again nitrogen dispersion and a 5-kJ ignitor in place, pre-ignition occurred as evidenced by a P_d value of 1.64 bar. (Recall the previous discussion with respect to Figure 4). Given that approximately 0.6 bar of the P_d value would normally be associated with dust dispersal, and a 1 bar overpressure is the accepted explosion criterion for MEC testing, it can be argued that a P_d value >1.6 bar indicates that an explosion occurred. The main difference from this procedure and usual MEC testing is that the nano-dust ‘MEC explosions’ occur with no external ignition source.

5.3 Explosion Inerting

Explosion inverting tests were performed for the 150 nm titanium using nano-size titanium dioxide (10-30 nm). The TiO₂ was first tested by itself with two 5-kJ igniters in the 20-L chamber and was found, as expected, to be chemically inert. Different amounts of TiO₂ were then admixed while the 150 nm titanium concentration was held constant at 125 g/m³. Like all other nano-material tests, nitrogen gas was used during dispersal.

For admixed amounts of TiO₂ up to 65% of the total dust mixture, pre-ignition still occurred. At 70% TiO₂, some pre-ignition activity occurred with a P_d of 0.81 bar (just above the normally acceptable upper-end P_d value of 0.7 bar). At 75% and 80% TiO₂,
the mixture was successfully dispersed with no pre-ignition, but the resulting dust cloud was ignitable with a 5-kJ ignitor. At 85% TiO₂ the mixture was rendered inert from pre-ignition and from the external 5-kJ ignition source. These results can be seen in Figures 9 and 10, drawn for the maximum rate of pressure rise and maximum explosion pressure, respectively.

**Figure 7** Maximum rate of pressure rise of 150 nm titanium at a concentration of 125 g/m³ for varying amounts of TiO₂.

**Figure 8** Maximum explosion pressure of 150 nm titanium at a concentration of 125 g/m³ for varying amounts of TiO₂.
This type of explosion suppression behaviour is similar to that observed for coal dust inerting with limestone or dolomite (rock dust). Investigations by Dastidar et al. (1997) determined that Pittsburgh pulverized coal dust was inerted in a Siwek chamber with 75 – 80% limestone. Limestone inerts a coal dust explosion by acting as a heat sink and absorbing energy from an ignition source or any combusting coal dust. The data shown in Figures 9 and 10 suggest that TiO$_2$ acts as a thermal explosion inhibitor in a manner similar to limestone.

6. EXPERIMENTAL CHALLENGES

There are many experimental challenges that arise when performing dust explosion research with micron- and nano-titanium. These include challenges related to the experimental methods, material handling, waste disposal and laboratory safety. These challenges were especially apparent with the nano-materials.

6.1 Experimental Methods

Issues with titanium powder were first noticed during the 20-L $P_{\text{max}}/K_\text{St}$ testing of the -325 mesh and $\leq$20 µm samples. Ignition at high concentrations would cause explosion residue to be lodged beneath the solenoid valve (item 2 in Figure 1) and disrupt subsequent trials. The dense titanium could not be completely dispersed, leaving a few grams beneath the solenoid valve. It seems that this remaining powder would then be ignited by the intense heat of the explosion occurring in the 20-L chamber and transferred through the solenoid valve.

Figure 11 shows a ‘normal’ pressure/time trace for a test in the Siwek 20-L chamber.

![Sample pressure/time trace for micron-titanium.](image-url)
In this routine trace for one of the micron-size samples, one can see the pressure rise due to dust dispersion into the 20-L chamber followed by the ignitors firing and then the dust itself exploding. This may be compared with the pre-ignition behaviour displayed in Figure 4.

Table 4 illustrates that no suitable combination of experimental conditions could be identified so as to disperse the nano-titanium into the 20-L chamber and achieve a pressure/time trace of the type shown in Figure 11. Numerous attempts were made by varying: (i) dust concentration, (ii) dispersion gas (nitrogen or air), (iii) dispersion gas pressure and ignition delay time (both of which affect dust cloud turbulence intensity), (iv) location of the dust prior to dispersion (placed in either the external dust storage container or the 20-L chamber itself), and (v) expected oxygen concentration in the 20-L chamber once dispersion was complete. Although no external ignition source (i.e., chemical ignitors) was used in these tests, the result was an explosion similar to that shown in Figure 4 in every case.

Table 4  Trials with various experimental conditions for 150 nm titanium (no ignitors).

<table>
<thead>
<tr>
<th>Dust Concentration [g/m³]</th>
<th>Dispersion Gas</th>
<th>Dispersion Pressure [bar(g)]</th>
<th>Ignition Delay Time [ms]</th>
<th>Dust Placement (Figure 1)</th>
<th>Final O₂ % in 20-L Chamber</th>
<th>Explosion During Dispersion?</th>
</tr>
</thead>
<tbody>
<tr>
<td>125 N₂</td>
<td>20</td>
<td>60</td>
<td>In Item 3</td>
<td>21.0</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>125 N₂</td>
<td>20</td>
<td>60</td>
<td>In Item 3</td>
<td>19.5</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>125 N₂</td>
<td>20</td>
<td>60</td>
<td>In Item 3</td>
<td>17.6</td>
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<td>125 N₂</td>
<td>20</td>
<td>60</td>
<td>In Item 3</td>
<td>15.0</td>
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<td></td>
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<tr>
<td>125 N₂</td>
<td>20</td>
<td>60</td>
<td>In Item 3</td>
<td>9.0</td>
<td>YES</td>
<td></td>
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<tr>
<td>500 Air</td>
<td>20</td>
<td>60</td>
<td>Under Item 8</td>
<td>21.0</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>500 Air</td>
<td>20</td>
<td>60</td>
<td>On Item 8</td>
<td>21.0</td>
<td>YES</td>
<td></td>
</tr>
<tr>
<td>500 Air</td>
<td>10</td>
<td>60</td>
<td>On Item 8</td>
<td>21.0</td>
<td>YES</td>
<td></td>
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<tr>
<td>500 Air</td>
<td>10</td>
<td>120</td>
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<td>21.0</td>
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<tr>
<td>500 Air</td>
<td>5</td>
<td>120</td>
<td>On Item 8</td>
<td>21.0</td>
<td>YES</td>
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<tr>
<td>500 N₂</td>
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<td>60</td>
<td>On Item 8</td>
<td>9.0</td>
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<tr>
<td>250 N₂</td>
<td>10</td>
<td>60</td>
<td>On Item 8</td>
<td>9.0</td>
<td>YES</td>
<td></td>
</tr>
</tbody>
</table>

6.2 Material Handling

Titanium has been shown to react with water at elevated temperatures around 700 °C. The metal has a high affinity for oxygen, stripping the oxygen from water to form titanium dioxide and hydrogen gas (Patnaik 2003). Hydrogen explosions have occurred in the past when cooling water unintentionally comes in contact with molten
titanium (Poulsen 2000). At ambient temperatures, micron-size titanium does not pose a hazard in this regard. Nano-size titanium, however, can have the same effect as molten titanium; it reacts with moisture to create hydrogen gas but this occurs at ambient temperatures. Hydrogen co-existing with easily ignitable nano-dust causes many concerns for handling the nano-titanium. Therefore, nano-titanium should have limited exposure to air or moisture and should be kept under dry nitrogen or argon. A nitrogen-filled glove bag was an essential feature of the safety precautions taken for the handling of nano-titanium in the current work. This reduced the potential nano-titanium contact with moisture and air.

6.3 Waste Disposal

Disposal of unused nano-titanium powder was important to minimize storage of the material for extended periods of inactivity. The nano-titanium should be deactivated and stored in a fume hood. Nano-titanium was carefully mixed in small quantities with water or dilute nitric acid. Mixing the titanium with water creates stable titanium dioxide while forming hydrogen gas within a vented and controlled environment. Adding nitric acid to the mixture promotes deactivation of the nano-titanium.

6.4 Laboratory Safety

Before any experimentation began, the laboratory was assessed for proper ventilation equipment and personal protective equipment (PPE). An improved ventilation extraction arm was installed and the vacuum exhaust sent through the ventilation system. Appropriate PPE included: a filtered mask, safety glasses, face shield (when working with chemical ignitors), lab coat, and double nitrile gloves.

Air quality measurements for airborne nano-particles were performed during normal operation of the 20-L chamber. Two types of particle counters were used: a FLUKE 983 particle counter for particles in the range of 300 nm to 10 µm, and a KANOMAX handheld CPC model 3800 for particles in the range of 15 nm to 1 µm. It was determined that no excess particles become airborne as a result of the use of nano-materials.

It is also important to apply the principles of inherently safer design when handling and working with nano-titanium and nano-materials in general. Principles of minimization, substitution, moderation and simplification should all be applied to reduce the risk involved with nano-titanium (Amyotte 2011). Examples include:

MINIMIZATION – Nano-titanium was received from the manufacturer in packages of 100 g each, thus reducing the amount of material handled at any given time.

SUBSTITUTION – Nitrogen was used in place of air for nano-dust dispersion in an attempt to prevent pre-ignition in the 20-L chamber tests.

MODERATION – Unused nano-titanium was deactivated in 0.1M nitric acid, eliminating the potential risk of storing an already opened package of material.

SIMPLIFICATION – Clear and straightforward procedures were developed and implemented.
7. CONCLUSION

The results obtained in the current work have shown that nano-titanium is much more sensitive than micron-titanium. Relatively low quantities and very low spark energies can cause nano-titanium to ignite yielding severe explosions. Nano-titanium is so reactive that traditional explosion test procedures using the Siwek 20-L chamber were not possible. Modifications to these procedures were attempted with limited success at preventing pre-ignition of the powder. Although nano-titanium is easily ignitable, explosions can be inerted and thus prevented. Adding nano-titanium dioxide to the dust mixture can reduce or eliminate the threat of an explosion.

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