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Title:

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Development of nano-thermite composites with variable electrostatic discharge ignition thresholds

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Abstract

Presented is a brief description of an apparatus for testing the electrostatic discharge ignition threshold for sensitive materials. This apparatus was used to demonstrate how fluorocarbons could alter the propensity of a nano-thermite to ignite. In addition to these findings, the effect of the fluorocarbon on the pressure output of the material is reported.

Keywords: nanoenergetics, electrostatic discharge

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Abstract

Presented is a brief description of an apparatus for testing the electrostatic discharge ignition threshold for sensitive materials. This apparatus was used to demonstrate how fluorocarbons could alter the propensity of a nano-thermite to ignite. In addition to these findings, the effect of the fluorocarbon on the pressure output of the material is reported.

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1 Introduction

Investigations into energetic nano-materials have been gaining prominence in many areas due to the interesting and sometimes unexpected effect of particle size on material properties. One area of consideration that has largely gone uninvestigated, though, is the change in hazards that may be occurring concomitantly. Researchers developing applications for nano-thermite composites have recently been experiencing issues with electrostatic discharge (ESD) inadvertently igniting composites. Other energetic materials have had to contend with ESD issues, which has brought about spark testing for the approval process of most energetic materials [1]. Typically spark testing apparatuses in use are designed for examining secondary explosives or propellants in production situations. Due to the extremely low ignition energy requirements for many of the nano-material composites, it is nearly impossible to discern if there is a difference in the ignition threshold using these instruments. Determining whether the ESD ignition threshold can be influenced by the addition of minor quantities of materials will be important to furthering the application of these materials.

To address this issue, an instrument has been constructed with a controllable low energy input. This instrument has been tested against an aluminium/copper oxide composite that has been modified using varying amounts of Viton A®. It had been previously postulated that the ESD ignition threshold could be affected by altering a material in three ways: increasing the conductivity of the sample, having the energy from the discharge absorbed by an endothermic reaction, or increasing the electrical resistance of the material. Researchers in the cited reference demonstrated the feasibility of the first two strategies [2] and the positive results on the last approach are reported in this paper. In addition, the effect of the Viton® A on the performance of

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the material, particularly the pressure output and time to peak pressure, will be reported. Ideally, the additive would significantly increase the electrostatic ignition threshold while having no effect on the performance.

2 Experimental

The nano-aluminum purchased from Nanotechnologies Inc. (currently Novacentrix Inc.) had a nominal diameter of 80 nm with 88% active aluminium and the nano-copper oxide purchased from Sigma-Aldrich had a nominal diameter of 33 nm. All values for particle size and active aluminium content are from the manufacturer and were accepted without verification. Materials were used as received except when noted. Nanothermite materials were made to have mass concentrations of 22% Al, and 78% CuO, which was shown to be optimum in previously published results [3]. The two constituents were mixed via sonication (30 seconds) in hexanes using a Branson S-450 Sonifier at a power setting 200W of with intervals of 0.5 s. Viton® A was dissolved in acetone (3.5 mg/ml), added to the nanothermite solution in appropriate amounts, and stirred for 5 minutes to allow for precipitation of the Viton® A on the particle surfaces. The solutions were then dried on a hot plate until the hexanes and acetone appeared to be evaporated. Samples were made to have 0%, 3%, 5%, 10% by mass Viton® A.

A novel ESD tester was developed whereby the voltage can be varied across a constant 10 nF capacitor, thus allowing for control over the energy output by the discharge event. A probe was manually lowered toward the nanothermite, which was resting on a grounded pan, until a discharge event occurred. This led to an ignition event only if sufficient energy is applied. A schematic of the device is shown in Figure 1. A more detailed explanation of this test setup will be provided in a following paper.

Samples were lightly packed into an aluminium pan (ID-6.43 mm, height-1.58 mm) and a razor was used to create a flat top surface. After the pans were placed on the grounded surface, the probe was lowered until the capacitor was discharged. The voltage or energy from the ESD probe was incrementally increased after each discharge event until ignition of the nanothermite was achieved. Once ignition was achieved, the test was repeated 5 times at that energy level to assure repeatability. Moreover, the ignition energy was then decreased incrementally to a level where no ignition occurred and was repeated 5 times to, again, assure repeatability. This provided a threshold at which the energy was sufficient from the ESD to allow for ignition. These tests were repeated for each increment of added Viton® A. After each test, the sample was changed, and after each ignition event the probe needle was changed.

To understand the effects of the Viton A addition on the performance of the material, constant volume pressure cell experiments were performed. Extensive detail about the experimental setup can be found in other publications [3, 4]. Each sample was loaded into the pressure cell whereby the mass of the nanothermite remained constant (17.5 mg) allowing for a constant mass of energetic formulation in the system. Ignition was achieved by pulsing a 1064nm Nd:YAG laser (~9 mJ) onto the material in the cell via an optical fiber and a *PCB Piezotronics* piezoelectric pressure transducer recorded the temporal pressure output. Four tests were run for each concentration of Viton® A to assure repeatability and the peak pressure and time to peak pressure were analyzed.

3 Results and Conclusions

The goal of this work was two-fold: 1) to give a brief description of a novel ESD sensitivity tester, and 2) to use the apparatus to demonstrate how Viton® A will affect the ignition threshold of an Al/CuO nanothermite, while also examining its effect on combustion performance in a constant volume pressure cell. Viton® A was added to an Al/CuO nanothermite at concentrations of 0%, 3%, 5%, and 10% by mass and each mixture was subjected to both the ESD sensitivity tester and the constant volume pressure cell. The preliminary results provided evidence that the novel ESD sensitivity tester was a viable method for characterizing the ESD sensitivity of a nanothermite. Initial performance testing indicates that Viton® A significantly enhances the ignition threshold while modestly decreasing the combustion performance (either decreasing the peak pressure or increasing the time to peak pressure).

Each mixture was subjected to a range of ESD energy values and a go/no go analysis was performed. Five tests were performed for each energy level and the value that did not lead to ignition for any of the five runs was considered its ignition threshold. Table 1 shows the results from the go/no go analysis. These numbers indicate the number of tests, out of the five, that achieved ignition, e.g. a test with 3 ignitions is shown as 3/5.

The ignition threshold increases as the Viton® A concentration increases, as shown in Table 1, indicating that the material is becoming less sensitive. At 0% Viton® A the ignition threshold is well below the lower energy limit of the system and at 10% the energy required to consistently ignite the material is above the upper energy limit of the system. More increments in both energy and Viton® A percentage are planned for future work as well as a larger sampling set for each condition. This preliminary test does give strong evidence, however, that the ESD ignition energy threshold is increased by small additions of Viton® A.

In addition to increasing the ESD ignition energy threshold, the Viton® A could potentially affect the performance of the material. If the performance is significantly hindered relative to the effective increase in ignition threshold, there is no overall benefit to addition of the Viton® A. However, if it can be demonstrated that the increase in ignition threshold is significant relative to the decrease in performance, the addition of Viton® A can be considered beneficial to the current system.

Performance was evaluated by examining both the peak pressure and time to peak pressure in a constant volume pressure cell. Each mixture was repeated four times and the values were averaged. Table 2 shows the combustion performance characteristics as well as the ignition threshold for each mixture.

To assess whether the Viton® A was beneficial to the system these values are normalized to allow for comparison. Normalization was achieved by dividing the change in the specific property by the initial property value, e.g. $\frac{PP_{5\%}-PP_{0\%}}{PP_{0\%}}$, where PP is the peak pressure and the subscript corresponds to the concentration of Viton® A in the sample. The normalization shows the relative increase in ignition threshold and relative decrease in combustion performance characteristics (either decrease in peak pressure or increase in time to peak pressure). Figure 2 shows the relative increase or decrease for each of the normalized properties as the concentration of Viton® A is varied.

As shown in Figure 2 the ignition threshold is significantly increased as the concentration of Viton® A is increased. The combustion performance characteristics do decrease, however, it is small relative to the increase in ignition threshold. To better visualize this comparison, Figure 3 shows the values of the normalized ignition threshold value over absolute value of each normalized combustion performance characteristic to give an ignition threshold enhancement efficiency.

This figure demonstrates that if one were concerned with increasing the ignition threshold while keeping the peak pressure relatively constant, Viton® A would be a good choice for an Al/CuO nanothermite. However, the relative increase in time to peak pressure with added Viton® A is significant when compared to the ignition threshold increase. It is noted, however, that at 10% Viton® A the peak pressure (gauge) of 0.86 atm is too low for any explosive purposes.

This preliminary test gives a brief description of a novel ESD sensitivity tester and demonstrates its capabilities by examining the effect of adding Viton® A to an Al/CuO nanothermite. The ability to incrementally vary the spark energy allows for an ESD ignition threshold to be found for a given material. The performance of the material can then be characterized and an ignition threshold enhancement efficiency can be found.

Future work will be performed to examine various other additives as well as other nanothermite mixtures. Furthermore, the authors realize that the Viton® A may participate in reactions and, thus may affect the overall system. Further experiments are needed to determine whether the ratio of the Al to CuO could potentially be altered from its current value to enhance combustion performance characteristics while keeping the ignition threshold constant for a given mass percentage of the additive.

4 References

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Table Captions

Table 1. Go/No Go Analysis for Various Concentrations of Viton® A

Table 2. Performance and Ignition Threshold Values

Table 1. Go/No Go Analysis for Various Concentrations of Viton® A

	ESD Energy (mJ)									
Percent Viton® A by mass		0.14	0.8	2.11	3.78	5.62	21.22	101.25	125.00	211.25
	0%	**3/5**	5/5	5/5						
	3%		**0/5**	1/5	4/5	5/5				
	5%			**0/5**	3/5	5/5				
	10%					0/5	**0/5**	2/5	2/5	2/5

Table 2. Performance and Ignition Threshold Values

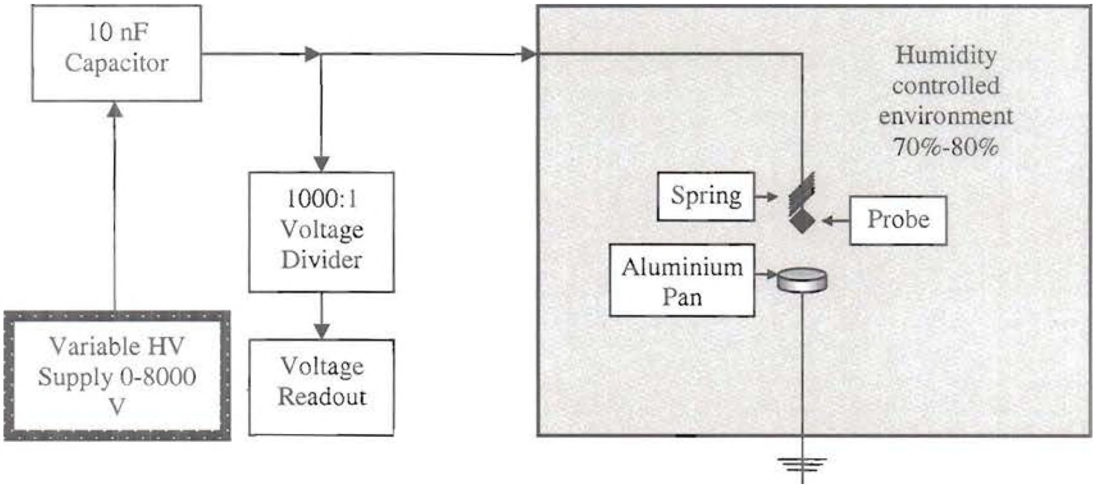
Percent Viton® A by mass	Time to Peak Pressure (μs)	Peak Pressure, gauge (atm)	Ignition Threshold (mJ)
0%	170.82	5.00	<0.14
3%	574.42	2.42	0.80
5%	825.72	2.19	2.11
10%	5077.10	0.86	21.22

Figure Captions

Figure 1. Schematic of ESD sensitivity tester

Figure 2. Normalized time to peak pressure, peak pressure and ignition threshold

Figure 3. Ignition Threshold Enhancement Efficiency for each combustion performance characteristic



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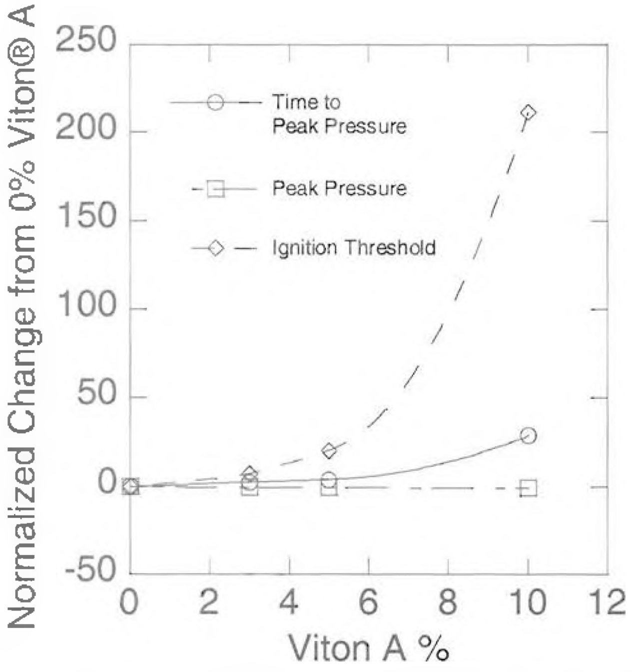
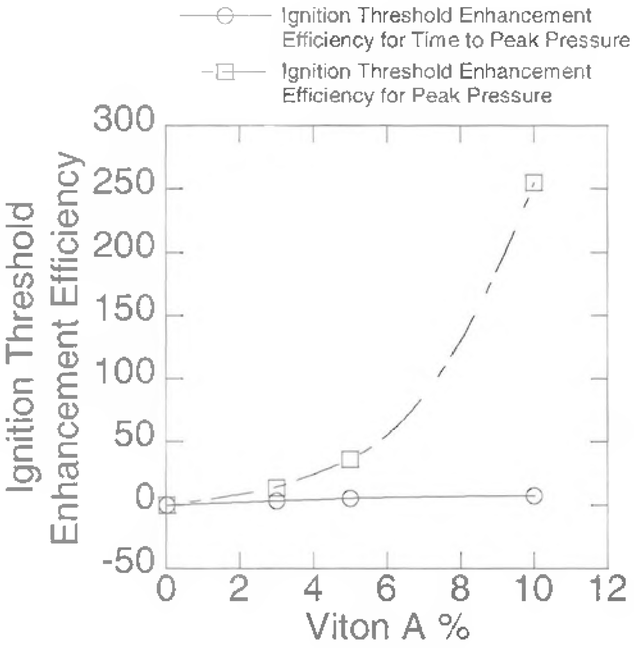


Figure 2. Normalized time to peak pressure, peak pressure and ignition threshold



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