Journal of Electrostatics 72 (2014) 28–32

Contents lists available at ScienceDirect

Journal of Electrostatics

journal homepage: www.elsevier.com/locate/elstat

Synthesizing aluminum particles towards controlling electrostatic discharge ignition sensitivity

Eric S. Collins [a], Jeffery P. Gesner [a], Michelle L. Pantoya [a,*], Michael A. Daniels [b]

[a] Department of Mechanical Engineering, Texas Tech University, Lubbock, TX 79409, USA
[b] Idaho National Laboratory, PO Box 1625, Idaho Falls, ID 83415, USA

ARTICLE INFO

Article history:
Received 9 August 2013
Received in revised form
21 October 2013
Accepted 1 November 2013
Available online 21 November 2013

Keywords:
Aluminum oxidation
Joule heating
Electrostatic discharge
Energetic materials
Ignition delay
Electrical conductivity

ABSTRACT

Aluminum particles were synthesized with shell thicknesses ranging from 2.7 to 8.3 nm and a constant diameter of 95 nm. These fuel particles were combined with molybdenum trioxide particles and the electrostatic discharge (ESD) sensitivity of the mixture was measured. Results show ignition delay increased as the alumina shell thickness increased. These results correlated with electrical resistivity measurements of the mixture which increased with alumina concentration. A model was developed using COMSOL for ignition of a single Al particle. The ignition delay in the model was consistent with the experimental results suggesting that the primary ESD ignition mechanism is joule heating.

1. Introduction

Composite energetic materials (CEM) are defined here as mixtures of aluminum (Al) fuel and metal oxide particles, that ignite to produce exothermic chemical energy. With the advent of nano-technology, nano-Al fuel particles have shown heightened reactivity compared to their micron scale counterparts [1–3]. Safely handling these powder mixtures requires a thorough understanding of their electrostatic ignition sensitivity yet very few studies on electrostatic discharge (ESD) ignition have been reported in the literature [4–6].

Most ESD ignition research is performed for the discharge of electric energy into a sample rather than by pouring induced inter-particle transport (e.g., electrostatic sensitivity that can occur from pouring a powder sample). An interesting finding from studying the literature on electrostatic ignition of powders is that a paradox exists regarding the electrical properties of the powder and the corresponding electrostatic ignition behavior. Glor [7] studied dust particles that had accumulated a charge through inter-particle transport. He found that as the powder's electrical conductivity decreased, so does the minimum ignition energy. In other words, materials with a decreased conductivity are more readily ignited by ESD [7]. In contrast, Foley et al.'s [8] study on Al–CuO showed that increasing electrical conductivity using additives actually decreased the minimum ignition energy of the mixture. A striking difference in these two studies is the way in which the electrical stimuli were introduced to the sample. For Glor [7] electrostatic charge accumulated within the sample, while in Foley et al. [8] the electrostatic charge was discharged into the sample. This paradox poses new research questions that have potential for impactful development in this field.

As a first step, we examined electrostatic discharge (ESD) ignition sensitivity of nine different CEM formulations, limiting the study to only micron-Al inclusion [6]. The results showed that at the highest setting on the ESD apparatus (i.e., which corresponded to 100 mJ), only Al–CuO ignited and its corresponding electrical conductivity was measured to be two orders of magnitude above the next mixture, Al–MoO_3 which did not ignite (i.e., 1246 compared with 40 nS/m, respectively). This was the first study to correlate electrical conductivity to ESD ignition sensitivity in energetic materials [6].

The influence of alumina in ESD ignition sensitivity was further studied in Ref. [9]. Specifically, Weir et al. [9] examined the electrical conductance and ESD ignition sensitivity of aluminum and molybdenum trioxide (Al + MoO_3) with varying Al particle size ranging from nano to micron scales. The results showed that as

* Corresponding author. Tel.: +1 806 742 3563; fax: +1 8067423451.
E-mail address: mpantoya@gmail.com (M.L. Pantoya).

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http://dx.doi.org/10.1016/j.elstat.2013.11.002

particle diameter decreased (and alumina concentration increased) the electrical conductance increased by 7 orders of magnitude and minimum ignition energy required for ESD ignition reduced accordingly. On the other hand, discretely added alumina particles significantly reduce the mixtures electrical conductivity, desensitizing the mixture to ESD ignition. This study revealed that the alumina shell may play a significant role in spurring ignition in Al + MoO_3 by accumulating charge and acting as a capacitive network, in contrast to discretely added alumina particles.

The objective of this work is to understand how the electrical conductivity and ESD ignition sensitivity of Al + MoO_3 varies as a function of the thickness of the alumina passivation shell surrounding the Al particles. To accomplish this objective, nano-scale Al particles were synthesized with varying shell thicknesses and combined with nano-scale MoO_3 particles. The mixture was further studied for electrical conductivity measurements and ESD ignition sensitivity quantified in terms of ignition delay time.

2. Experimental

The Al particles were supplied by Sigma Aldrich and had an average particle diameter of 95 nm. The MoO_3 particles were purchased from Nanostructured and Amorphous Materials Inc. and had an average platelet size of 380 nm. The Al particles were oxidized in an isothermal oven to increase the thickness of the Al_2O_3 shell; thereby synthesizing particles with controlled shell thicknesses. It is noted that the overall particle diameter does not change, such that the shell thickness grows at the interface of aluminum and alumina and as alumina concentration increases, aluminum correspondingly decreases.

2.1. Material synthesis and preparation

A thermogravimetric analyzer (TGA), model STA 409 PC by Netzsch, was used to observe the weight gain of Al particles as the Al oxidized to Al_2O_3. During oxidation in the TGA, 11.48 mg of Al powder was held in a platinum crucible while the temperature increased at a rate of 40 °C per minute in a controlled environment of ultra-high purity oxygen. The oven continued to heat until 480 °C, a temperature that provided reasonable reaction rates where oxidation and shell growth was observed. The Al sample remained in the isothermal environment for 180 min as the mass gain was monitored. The precision of the change in mass of the sample in the TGA had a variance of 0.001 mg. This data was used to control oxide shell growth on larger quantities of aluminum powder.

A Neytech Qex oven was used to oxidize Al particles in an isothermal oxygen environment. Ultra-high purity oxygen was purged in the oven chamber at a flow rate of 180 cm^3/min for 10 min. This procedure ensured that the volume was flushed five times and had a statistical purity of 99% of oxygen in the oven chamber. The Qex oven was set to have a temperature ramp rate of 200 °C/min and a temperature of 480 °C. The settings were programmed in the oven for automated control and repeatability between oxidation procedures. Six samples of 0.9 g of Al powder were prepared for each oxidation cycle. Once the chamber was purged and the temperature of 480 °C was reached, the Al powder remained in the oven for various durations ranging from 8 to 150 min. This variable oxidation time provided different alumina shell thicknesses.

A transmission electron microscope (TEM), model Jeol JEM-2100, was used to examine the Al particles and measure the thickness of the alumina shell with Gatan image analysis software to measure the thickness of the alumina shell. Thickness measurements were taken from at least three different locations on several Al particles. Fig. 1 shows three representative images of the Al particles taken from the TEM.

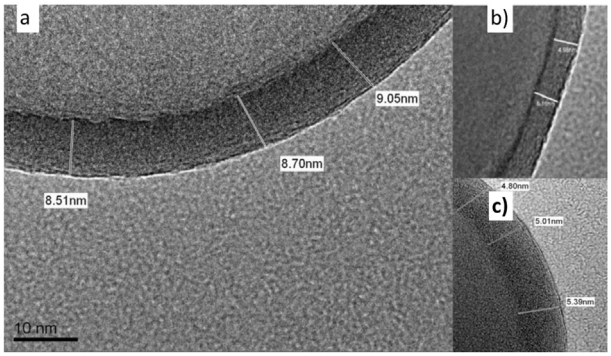

Fig. 1. TEM images of the alumina shell after oxidation times of a) 150 min, b) 8 min, and c) 30 min.

The percent of active Al by weight (Y) was calculated using Eq. (1),

$$\frac{R^3}{(R-\delta)^3} = \frac{\rho_m}{\rho_{mo}}\left(\frac{1}{(Y)} - 1\right) + 1 \quad (1)$$

where R is the average particle radius, δ is the Al_2O_3 shell thickness, and ρ is the density of the metal (m) and metal oxide (mo). The average shell thickness and the percent of active Al content are listed in Table 1 for all treated Al powders.

The Al + MoO_3 mixtures were prepared to an equivalence ratio of 1.0, corresponding to stoichiometric conditions. Details of this mixing procedure are reported in Ref. [10].

2.2. Experimental setup

An acrylic channel was loaded with 58 mg of the powder, which was pressed into a pellet within the channel. The pellet occupied a volume of 31 mm^3 and had a bulk density of 1.89 g/cm^3. The bulk density of the pellet was calculated to be 50% of the theoretical maximum density (TMD). Two copper electrodes were positioned to cover the openings in the channel such that the tips of the electrodes were in contact with the surface of the pellet as shown in Fig. 2. A voltage potential was applied to the electrodes and when greater than the dielectric strength of the pellet material, a spark was generated which ignited the pellet. The voltage source used in these experiments was an electrostatic discharge (ESD) tester that is a human body model developed by Franklin Applied Physics [6]. A human body model transfers charge from a capacitor (human) to another object. The range of voltage output is 1–10 kV which is stored in a 0.002 μF capacitor producing up to 100 mJ of energy.

A current monitor, model 2878 from Pearson Electronics, was used to measure the electric charge released by the ESD tester. A

Table 1
Thickness of Al_2O_3 shell and weight percent active Al content for all oxidized Al particles.

Oxidation time (min)	Al_2O_3 thickness (nm)	Wt % active Al
0	2.7	78.1
8	3.3	73.9
15	4.5	66.3
30	4.6	65.7
60	5.7	59.4
90	6.7	54.2
150	8.3	46.7