



Combustion of mechanically activated Ni/Al reactive composites with microstructural refinement tailored using two-step milling



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ARTICLE INFO

Article history:

Received 9 March 2015

Received in revised form

10 June 2015

Accepted 11 June 2015

Available online xxx

Keywords:

Aluminides

Reaction synthesis

Mechanical alloying and milling

Microstructure

ABSTRACT

Metal-based reactive composites are high energy-density materials that have potential uses as multi-functional energetics. However, when composed of micron size particles they can be difficult to ignite and have slow reaction rates. Recent work has shown that mechanically activated (MA) materials can have increased ignition sensitivity and reaction rate, yet the role of microstructure refinement (i.e., mechanical activation duration) in controlling combustion behavior is not well understood. In this work, the combustion velocities and flame temperatures were measured for equiatomic MA Ni/Al reactive powders produced using different milling durations in a two-step dry/wet milling process. For MA Ni/Al pellets pressed to 70% of the theoretical maximum density, it was shown that the combustion velocities increase as the milling time increases from ~9.4 cm/s at 25% of the critical reaction milling time (t_{cr}) to ~20 cm/s at a milling time of 97% t_{cr} . For the cases considered, the average maximum flame temperatures were measured to be -1873 ± 30 K for samples milled for 25% t_{cr} to 1786 ± 30 K at 97% t_{cr} . It was also found that hydrocarbon contaminants are milled into the MA Ni/Al composite particles during the wet milling step and result in expansion of the pellets during combustion. Differential scanning calorimetry coupled with Fourier transform infrared spectroscopy showed that the release of hydrocarbon contaminants occurs at a temperature of ~630 K. It was also shown that the concentration of hydrocarbon contamination decreased as the dry milling times increased, which suggests particle structure and mechanical property evolution during initial dry milling also affects contamination during subsequent wet milling.

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1. Introduction

Metal-based reactive powder composites, such as those consisting of nickel (Ni) and aluminum (Al), are a class of energetic materials that have high-energy densities and can produce significant heat release during reaction by thermal or mechanical initiation. Their potential use as structural energetic materials, as enhanced blast materials and for the synthesis of novel metastable non-equilibrium materials has driven research focused on their mechanical impact behavior [1–7]. However, these materials typically have slow reaction rates and are difficult to ignite when micron particle size ranges are used, limiting their application.

Therefore, significant effort has been focused on creating materials with high interfacial surface areas and smaller characteristic dimensions in order to increase combustion velocity and ignition sensitivity. Reactive powder composites with these features have been fabricated by using nanopowders [7–10], magnetron sputtering [11–16] and mechanical activation techniques [1–3,17,18].

Mechanical activation techniques are of particular interest, as they can increase combustion velocities and ignition sensitivities by modifying the system's morphology and mechanical properties through a process that is scalable and inexpensive. This is achieved by high-pressure collisions between the particles and the milling media in which the particles are simultaneously cold worked and welded together, resulting in composite particles. If milled for a long enough time, the individual components form stable intermetallic compounds. The milling time needed to completely react the material is termed the “critical reaction time” (t_{cr}). When milling is arrested prior to t_{cr} , composite particles are produced that

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consist of intermixed material with nano/microscale structures/laminates. Manukyan et al. [2] have shown the ratio of nano-to microscale laminate structures within particles can be controlled by varying the dry milling duration of a two-step dry/wet milling procedure. Further, they have shown that composite particles milled for $\sim 25\%$ t_{cr} or less consist primarily of structures/laminates intermixed on the micron-scale. As the milling duration increases, nanoscale laminates form and the ratio of nano- to micron-scale structures/laminates increases until t_{cr} is reached and the material reacts. It has been shown that the intermixing of Ni and Al at both the micron- and nano-scale increases thermal and impact sensitivity due to smaller diffusion distances and increased contact areas [1–3,17,19–23]. However, the role and extent to which the modified microstructural properties affect the reaction velocity and combustion temperature has yet to be fully investigated.

Prior work involving mechanically activated (MA) Ni/Al has focused on the 3Ni/Al system. Korchagin et al. [24,25] reported combustion velocities that ranged from ~ 0.1 to 1.4 cm/s depending upon the milling time and milling energy. The combustion velocities initially increased with extended milling until a threshold was reached. With further milling beyond the threshold, the combustion velocities would decrease due to the formation of Ni/Al intermediates and solid solutions during milling or “poisoning.” Combustion temperatures ranged from ~ 850 to 1070 K and followed the same trend as the combustion velocities. Also using the 3Ni/Al system, Hadjiafxenti et al. [20,26] reported that increasing compact density increased combustion velocity from ~ 0.9 to 5 cm/s due to increased thermal diffusivity [20]. When compared to non-milled samples these studies show a slight increase in combustion velocities under analogous conditions [27].

As compared to non-milled samples, MA significantly increases the combustion velocities of the more exothermic equiatomic Ni/Al system. For equiatomic Ni/Al milled to $\sim 90\%$ t_{cr} , Bacciochini et al. [18] reported combustion velocities that increased with compaction density from ~ 3 cm/s to 6 cm/s. These samples were cold-pressed to densities of ~ 42 – 75% of the theoretical maximum density (TMD) respectively. Near-true ($\sim 99\%$) density compacts formed by cold spray compaction were found to have ~ 24 cm/s velocities [18]. Also using equiatomic Ni/Al, Hadjiafxenti et al. [22] reported similarly high combustion velocities of 20 – 24 cm/s and ~ 14 cm/s in lower density (68%) compacts using low energy ball milling ($\sim 87\%$ t_{cr} and $\sim 95\%$ t_{cr} , respectively). Depending on compact density, particle size, and pre-heat temperature, combustion velocities of typical non-milled Ni/Al pellets are on the order of 1 – 12 cm/s [28–30] for micron-sized powders. These studies show MA significantly increases combustion velocities as compared to similar non-milled materials. However, for MA Ni/Al, studies demonstrating the effects of microstructure on combustion are limited [18,22]. A full study investigating how varying microstructure refinement via milling duration affects the combustion behavior of equiatomic MA Ni/Al has not been done.

The objective of this work is to characterize how modifying dry milling duration, influences the resulting combustion velocity and temperature of pressed, equiatomic MA Ni/Al reactive composite powders manufactured using a two-step dry/wet milling process. With this information, a more comprehensive understanding of these materials can be gained along with data that can be used in continuum and molecular dynamics models that are currently being developed in conjunction with this work [6,31].

2. Experimental

2.1. Mechanical activation procedure

For this study, 3 – 7 μm Ni (Alfa Aesar) and -325 mesh Al (Alfa

Aesar) were used to prepare an equiatomic, MA Ni/Al reactive composite powder using a two-step dry and wet milling procedure as detailed in previous work [2]. Briefly, the Ni/Al was milled by high-energy ball milling in an argon (99.998 vol. %) atmosphere using a PM100 (Retsch) planetary ball mill. The reactant powders were subjected to dry milling durations of 4.25 , 8.5 , 12.75 and 17 min corresponding with roughly 25% , 50% , 75% and 97% of the critical milling time. Because a process control agent (PCA) was not present in this stage, $\sim 50\%$ of the Ni/Al material is cold-welded to the milling jar and media and the remaining material consists of particles that are 100 – 3000 μm in size. In order to increase the material yield and to reduce the particle size range to <53 μm the material was then wet milled with hexane (98.5% hexane isomers, Mallinckrodt Chemicals) for a total of 10 min. After milling with hexane, the material was dried under vacuum (~ 0.01 atm) for 24 h (hr) at room temperature. Morphology and phase content has been previously reported [2].

2.2. Combustion experiments

Combustion experiments were performed on cylindrical pellets of sieved (25 – 53 μm) Ni/Al particles that were pressed to $70\% \pm 2\%$ TMD with a height 13.5 ± 0.2 mm and a diameter of 12.7 mm. Shown in Fig. 1a, pellets were thermally ignited to measure burning rates and combustion temperatures. To ensure consistent ignition and to promote a planar combustion front, the samples were ignited using a hot wire and ignition increment that ignited a 2 mm thick compact of 97% t_{cr} MA Ni/Al pressed at $65\% \pm 2\%$ TMD. The 2 mm compact would subsequently ignite the experimental sample. The ignition increment was A1A, a pyrotechnic igniter with a composition of 65 wt.-% Zr, 25 wt.-% Fe_2O_3 and 10 wt.-% diatomaceous earth. Combustion propagation was visually recorded using a high-speed color Phantom v7.3 camera at a frame rate of 1000 frames/s (fps). Temperature measurements were taken with a FLIR series SC2500 infrared (IR) camera at 1000 fps and an Omega Type B (platinum/rhodium with 0.20 mm dia.) thermocouple. The thermocouple was inserted into a ~ 0.5 mm \times 0.6 mm diameter hole, drilled 4 mm from the top of the pellet. For repeatability, three separate experiments were conducted for each experimental condition.

A second set of combustion experiments were performed in order to observe the reaction front propagation on a microscopic scale. This was done using 12.7 mm diameter cylindrical pellets

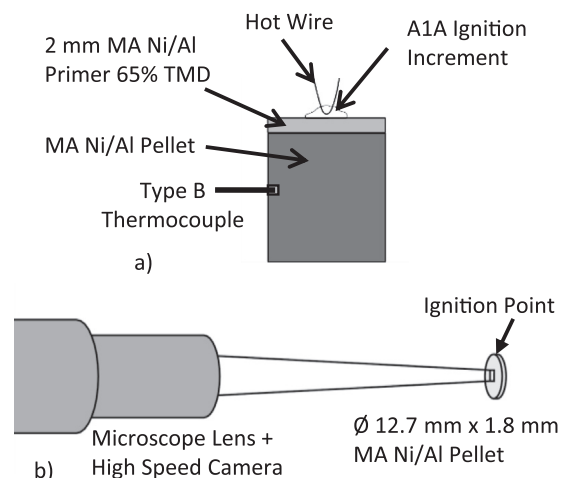


Fig. 1. Schematics for (a) pellet combustion experiment and (b) reaction front combustion experiment.

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