



Microexplosions and ignition dynamics in engineered aluminum/polymer fuel particles



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ABSTRACT

Aluminum particles are widely used as a metal fuel in solid propellants. However, poor combustion efficiencies and two-phase flow losses result due in part to particle agglomeration. Recently, engineered composite particles of aluminum (Al) with inclusions of polytetrafluoroethylene (PTFE) or low-density polyethylene (LDPE) have been shown to improve ignition and yield smaller agglomerates in solid propellants. Reductions in agglomeration were attributed to internal pressurization and fragmentation (microexplosions) of the composite particles at the propellant surface. Here, we explore the mechanisms responsible for microexplosions in order to better understand the combustion characteristics of composite fuel particles. Single composite particles of Al/PTFE and Al/LDPE with diameters between 100 and 1200 μm are ignited on a substrate to mimic a burning propellant surface in a controlled environment using a CO₂ laser in the irradiance range of 78–7700 W/cm². The effects of particle size, milling time, and inclusion content on the resulting ignition delay, product particle size distributions, and microexplosion tendencies are reported. For example particles with higher PTFE content (30 wt%) had laser flux ignition thresholds as low as 77 W/cm², exhibiting more burning particle dispersion due to microexplosions compared to the other materials considered. Composite Al/LDPE particles exhibit relatively high ignition thresholds compared to Al/PTFE particles, and microexplosions were observed only with laser fluxes above 5500 W/cm² due to low LDPE reactivity with Al resulting in negligible particle self-heating. However, results show that microexplosions can occur for Al containing both low and high reactivity inclusions (LDPE and PTFE, respectively) and that polymer inclusions can be used to tailor the ignition threshold. This class of modified metal particles shows significant promise for application in many different energetic materials that use metal fuels.

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1. Introduction

Development of engineered composite metallic fuels that have enhanced ignition and combustion characteristics is of critical need to improve the performance of current systems that use aluminum (Al). Al is a widely used metallic fuel in propellants because of its high gravimetric and volumetric oxidation enthalpy [1–3]. However, microscale Al particles exhibit relatively slow combustion rates [2,3] and long ignition delays, resulting in incomplete combustion and ignition failure in some cases [4,5]. Additionally,

molten Al particles coalesce on the propellant surface and form agglomerates orders of magnitude larger than the original particles, resulting in increased two-phase flow losses in rocket motors [6,7]. The combustion rates of microscale Al can be increased by a reduction in average particle size. For example, replacing Al with nAl in composite propellants has been shown to increase propellant burning rate [8], decrease Al particle ignition delay [1], and reduce agglomerate sizes in combustion products [9]. However, resulting agglomerates are still much larger than initial nAl particles and the relative increase in native oxide content with decreasing particle size results in a large reduction of available combustion enthalpy and much lower theoretical specific impulse (ISP). During processing, nAl particles form difficult to disperse aggregates resulting in high uncured propellant viscosity and poor

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mechanical properties [10–13]. Processing issues resulting from the much higher specific surface area of nAl and safety concerns related to ignition sensitivity can nullify the benefits of improved combustion efficiency. Composite propellants manufactured with nAl have also been shown to suffer from long-term degradation.

An alternative approach to enhancing reactivity and reducing two-phase flow losses is to engineer composite fuel particles through mechanical activation (MA) with tailored combustion characteristics. High energy density reactive powders with ignition temperatures and ignition delays comparable to those of nAl can be produced by selecting the proper choice of inclusion materials and milling parameters [2]. For example, replacing neat Al with mechanically activated Al particles containing reactive (polytetrafluoroethylene, PTFE) or non-reactive (low-density polyethylene, LDPE) inclusions has been shown to reduce ignition delay and metal particle ignition temperatures in propellant formulations [14,15]. Additionally, observed decreases in particle agglomeration at the burning surface of propellants using MA Al with PTFE or LDPE inclusions can potentially reduce two-phase flow losses in rocket motors [16,17]. Reduced agglomerate sizes result from fragmentation of the reacting composite particles due to microexplosions at the propellant surface. It is possible that intraparticle evolution of decomposition gases and/or reaction products results in internal particle pressurization, shattering the particles into smaller fragments. Typically, ignition studies on MA composite fuel particles containing inclusion materials focus on bulk ignition properties of particles, but the role of inclusion reactivity on dynamics (e.g. microexplosions) is unclear [18]. The effects of milling energy and other particle parameters, such as initial particle size, stoichiometry, milling duration, and thermal environment on microexplosion tendency also remain unknown. In order to engineer composite fuel particles and tailor combustion characteristics to reduce ignition delay, increase Al combustion rate, and produce smaller agglomerates than conventional metal fuels, the ignition dynamics of engineered composite particles must be characterized.

The objective of this work is to understand the role of polymer inclusion types, particle parameters, and incident heat flux on microexplosion behavior during combustion experiments. Reaction dynamics will be qualitatively described and measurements of ignition delay and product size distributions at different laser heating fluxes as a function of milling duration, initial particle size, and varying inclusion materials will be reported.

2. Experimental methods

Mechanical activation of Al (Valimet H-30, 35 μm)/PTFE (Sigma Aldrich, 468096, 35 μm) was performed using a SPEX 8000M high-energy mill. One-gram batches of Al/PTFE particle blends at mixture ratios of 70:30 and 90:10 wt% were milled in argon-filled (99.997 vol%), high-density polyethylene (HDPE) plastic milling containers (Cole Parmer EW-62201-01). A crash ratio of 24:1 was used with five 9.5 mm and fifteen 4.8 mm diameter 440C stainless steel media (McMaster Carr). Batches were milled with a duty cycle of 1 min ON and 4 min OFF while being cooled continuously by a fan for milling durations of either 20 min or 60 min. Critical milling time for the 70:30 Al/PTFE mixture was 60 min and longer milling caused the powders to react during milling. Particle fabrication procedures are similar to previous work [19], where XRD and EDS analysis of as-milled composite particles showed no detectable levels of media/container contamination.

Scaled production of MA composite particles is most advantageously done via roller milling. Low-energy milling techniques can be used to reduce ignition sensitivity in reactive mixtures, providing additional safety margins during manufacture and propellant processing. As such, Al (Valimet H-30, 35 μm)/LDPE (IASCO,

LDP5, 500 μm) MA was carried out using an intermediate-scale US Stoneware CV-90116 low-energy roller milling technique developed previously. In this technique, milling parameters (charge ratio, milling duration, rotational rate, milling duration) are chosen such that DSC oxidation heat release characteristics from low-energy milled particles is matched to that of similar Al/LDPE particles milled for 60 min using the high-energy manufacturing technique described previously. Briefly, 200 g batches were milled using the same media type in a one-gallon argon-filled HDPE container (6 inch diameter) at a crash ratio of 70:1 and a rotational speed of 90 rpm for 52 h. The milled materials were handled in an argon-filled glove box after milling and were air passivated in hexane, which was allowed to slowly evaporate in air.

Microstructures of the synthesized Al/PTFE and Al/LDPE particles were characterized using a 300 kV FEI Titan S/TEM 80-300 equipped with a Gatan imaging filter (GIF) and a 300 kV Hitachi HF-3300 S/TEM equipped with a secondary signal detector. To visualize the local distributions of Al and polymers within a single particle, a three-window energy-filtered transmission electron microscopy (EFTEM) mapping technique was performed. In this technique, the inelastic-scattered electrons containing element specific ionization edges are selectively collected using an energy slit to construct a chemical map of a selected element. Specifically, three EFTEM images (two images from the pre-ionization edge region and one from a post ionization edge region) were sequentially collected. Then two pre-edge images were used to subtract the background signal from a post-edge image to get a quantitative elemental map. Al maps (L edge) and C maps representing the distribution of polymers (K edge) were collected with a 20 eV width energy slit for 5 and 10 s, respectively. In addition, secondary signal images (SSI) were also collected using a Hitachi HF-3300 S/TEM equipped with a secondary electron (SE) detector to gain insight in the three-dimensional morphology of the particles.

In contrast to previous studies where bulk ignition characteristics were identified for aerosolized powder flows [6,20–23, loose powders [24], and densified compositions [25], here we study CO₂ laser ignition of single particles on a ceramic substrate (OZM Research, FSK 50-20 K) in order to observe intraparticle combustion dynamics. The thermal conditions are more similar to those burning on a propellant surface with associated conductive and convective feedbacks. These are primarily the radiant heating of the particle and the substrate around the particle, the indirect heating of the particle from the substrate, heat losses from the reacting particle to the substrate, as well as convective losses. A Coherent GEM 100A, 10.6 μm wavelength, 100 W CO₂ laser with a Gaussian beam was used to heat particles of composite materials with $D_{\text{Eq,CSA}}$ ranging from 100 to 1200 μm . A ThorLabs ZnSe plano convex lens (500 mm focal length) was used to focus the laser beam to a $1/e^2$ radius of 1.3 mm for experiments with laser flux between 78 and 600 W/cm² and 0.6 mm for experiments between 2400 and 7700 W/cm². LabVIEW was used to control the laser power output and laser pulse time. The power output of the laser was measured with a Coherent LabMax TOP laser power and energy meter. An initial high flux (>9000 W/cm²) pulse was used to mark the incident beam location on the ceramic tile prior to experiments. The composite fuel particles were placed in the center of the incident beam location. The plate-like particles mostly consist of aluminum and we expect their absorptivities to be similar.

Prior to ignition experiments, a Hirox KH-8700 digital optical microscope was used to image the particles and determine the top surface area of each particle used in experiments. As the particles used in experiments were plate-like, only the top surface area of a particle is exposed to laser radiation. The profiles of particles were irregular, and the equivalent cross-sectional diameter of single particles was used as a measure of particle size and was

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