Ignition and Combustion of Boron-based $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites

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Abstract

Stable ternary powders of $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites of interest for agent defeat applications were prepared by mechanical milling. All powders contained 20 wt % of iodine. Powder ignition was characterized using a heated filament experiment. Ignition kinetics was compared to the kinetics of events occurring upon slow heating of these materials in thermo-analytical experiments. Individual particle combustion was studied by seeding the powder into a premixed hydrocarbon-air flame. Both particle burn times and temperatures were measured optically. Aerosol combustion of the powders was tested in a constant volume explosion chamber. Ignition temperatures for the $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites were lower than those for the $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites. Iodine release occurring due to the formation of AlB_2 and MgB_2 was a likely ignition trigger for $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites, respectively. The burn times of these composites were longer than those for pure Al and Mg powders. Burn times for $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ particles were shorter than for the same size particles of $\text{Al}\cdot\text{B}\cdot\text{I}_2$. Combustion temperatures of the composite powders were lower than those of pure Al and Mg. In aerosol combustion, the rate of pressurization and maximum pressure were inversely proportional to the concentration of boron. The combustion efficiency was expressed through a ratio of the experimental maximum pressure to that predicted by a thermodynamic equilibrium calculation. This efficiency was the same for Al and $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites. The efficiency for $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites exceeded that of pure Mg.

Keywords: nanocomposites; reactive materials; biocidal materials; halogens.

1. Introduction

Reactive materials capable of releasing halogenated products upon ignition and combustion are of interest for advanced munitions aimed to defeat stockpiles of biological weapons [1, 2]. Recently, research efforts have focused on preparing iodine-bearing reactive materials [3-14], generating biocidal combustion products. Often, it is difficult to stabilize the biocidal component (e.g., a volatile halogen, such as iodine) in the reactive material, so that the material can be stored and processed along with other components of energetic formulations [15, 16]. Several thermally stable iodine-bearing aluminum-based fuels, including powders of $\text{Al}\cdot\text{I}_2$ [8-10], $\text{Al}\cdot\text{CHI}_3$ [11], and $\text{Al}\cdot\text{B}\cdot\text{I}_2$ [12, 17] were prepared using mechanical milling. The biocidal effectiveness of such reactive materials against challenge bio-aerosols was quantified [17, 18]. Performance of a ternary $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composite [19] (Al:B mass ratio of 6 with 17 wt. % of iodine) was more promising compared to other materials with the same or even greater bulk iodine content. The focus then was placed on improving the thermal stability of ternary $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites [12]. Among composites with a varied Al:B ratio and fixed 20 wt. % of iodine, compositions with higher boron concentrations were identified as more stable. An analogous material development study was later conducted by replacing aluminum with magnesium to prepare ternary $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites [13, 14], which had the thermal stability similar to or even better than that of $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites.

In this study, the most stable ternary compositions of $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites were selected and compared to each other in terms of their ignition kinetics and combustion dynamics. Additionally, the effect of boron concentration on ignition and combustion of these composites

was quantified. The results obtained here are useful to predict combustion behavior of the new reactive materials in various practical configurations.

2. Materials

Starting materials were elemental magnesium powder, -325 Mesh, 99.8% pure, by Alfa Aesar, elemental aluminum powder, -325 mesh, 99.5% pure, by Atlantic Equipment Engineers, amorphous boron powder, 95-97% pure, by SB Boron (nominal size 1 μm), and iodine chips (approximately 1–5 mm in size), 99% pure, by Sigma Aldrich.

Material preparation by mechanical milling was described in detail elsewhere [12-14], and only a brief summary is presented here for completeness. Ball milling at room temperature was performed using a shaker mill (SPEX Certiprep, 8000 series) with two 50-ml flat-ended steel vials cooled by an air jet. The milling vials were loaded and sealed in an argon-filled glovebox. A total milling time of four hours was used to prepare both aluminum and magnesium-based composites. Hardened steel balls with 9.525 mm diameter were used with ball to powder mass ratio of 10. No additional process control agent was used.

Particle shapes, particle size distributions, and stability of the prepared composites were characterized in detail elsewhere [12-14]. In thermo-gravimetric (TG) measurements (using heating rates of 2 - 20 K/min in Ar), all ball-milled powders released iodine in stages. For ignition and combustion studies, two of the most stable ternary compositions for each of the $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites were selected. The compositions of the selected materials are shown in Table 1.

TG traces and their derivatives (DTG) for the selected samples at a heating rate of 10 K/min are shown in Fig. 1. Traces for different samples are shifted vertically relative to one another for clarity. For both types of materials, two stages of iodine loss can be identified in the temperature range covered by the TG measurements. The first stage, associated with release of un-bound iodine [8, 12] occurs at about the same temperature for all materials. The second stage is stronger, and occurs at a higher temperature for Al·B·I₂ composites.

Following the methodology used in Ref. [12], a mass loss observed upon heating to a specific temperature served as an indicator of the material stability. Because the present TG measurements in an inert environment were limited to 450 °C for Mg·B·I₂ composites to minimize evaporation of Mg [13, 14], the mass loss occurring between 50 and 450 °C was treated as the stability indicator. Note that in Ref. [12] the threshold temperature was 400 °C, so that the absolute mass loss values reported there are not directly comparable to those shown in Table 1. Mg·B·I₂ composites exhibit lower absolute mass losses, as shown in Table 1 and Fig. 1, and thus are slightly more stable than Al·B·I₂ composites [12].

Table 1. Most stable ternary compositions of the boron-based Al·B·I₂ and Mg·B·I₂ composite powders prepared by mechanical milling

Sample ID	Mass percentage, %				Mass loss (%) between 50 and 450 °C
	Mg	Al	B	I ₂	
A	0	30	50	20	2.7
B	0	40	40	20	2.2
C	33	0	47	20	1.1
D	50	0	30	20	1.5

Figure 1. TG traces and their derivatives (DTG) of the most stable $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composite powders prepared by mechanical milling (refer to sample IDs in Table 1). Iodine release stages I and II are specified in the DTG traces. Heating rate is 10 K/min.

3. Experiments

Ignition of the prepared materials was studied in air at heating rates ranging from 10^4 to 10^5 K/s using an electrically heated filament [20]. A 0.5-mm diameter 4.5-cm long nickel-chromium alloy wire was heated by a DC current. The heating rate of the filament was adjusted by changing the applied DC voltage and resistor connected in series. A small amount of powder was mixed with hexane, and this slurry was coated onto the filament to form a thin powder layer. The coated portion of the filament was about 1 cm in length. The coating dried prior to experiments. Ignition instant was identified from videos recorded at 500 fps using a high speed camera (MotionPro 500 by Redlake). The temperature history of the heated filament was measured using a high-speed infrared pyrometer (DP1581 by Omega Engineering, Inc.) so that the temperature at the ignition instant was determined as a function of the heating rate.

Combustion of clouds of $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ and $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composite powders aerosolized in air was studied using a constant volume explosion (CVE) experiment [21, 22]. A nearly spherical explosion vessel was initially evacuated. Composites were injected into the vessel with an air blast delivered from a high pressure reservoir; the pressure in the vessel was brought to ca. 1 atm. After a short delay necessary to minimize the turbulence, the aerosolized powder was ignited by a custom-made pyrotechnic initiator placed at the center of the vessel. The initiator used 0.1g of mechanically milled $\text{Al}/\text{Fe}_2\text{O}_3$ thermite with 4:1 $\text{Al}/\text{Fe}_2\text{O}_3$ mole ratio. The thermite was loaded in a 0.5-cm diameter, 3.5 cm height steel tube and mounted on an electrically heated tungsten wire. The pressure inside the vessel was recorded as a function of time using a PX2AN1XX500PSAAX pressure transducer by Honeywell. No detectable pressure pulse was registered for the pyrotechnic initiator alone. The ratio of the maximum pressure over the initial pressure, $P_{\max}/P_0$, and the maximum rate of the pressure rise, $(dP/dt)_{\max}$, were used to characterize the energy released and the rate of combustion, respectively. Three experiments for each powder were conducted at a constant powder load of 4.65 g, corresponding to fuel-rich conditions for all materials. Equivalence ratios for samples A, B, C, and D were 2.5, 2.3, 2.3, and 1.8, respectively.

Burning characteristics of individual composite particles were investigated using a powder seeded air-acetylene flame, following earlier experiments [23-26]. Powders were fed by a nitrogen flow through a central axis of a premixed flame burner. Particles ignited and burned in the acetylene combustion products comprising mostly carbon dioxide and water, while surrounding air was mixed in the combustion environment providing additional oxidizer. Initial flows for both premixed air-acetylene and nitrogen carrying particles were directed vertically. Three filtered photomultiplier tubers (PMTs) including two Hamamatsu R3896-03 and one Hamamatsu R636-

10 were used to monitor radiation of the burning particles. The interference filters with central wavelengths of 700, 800 and 900 nm were used. Recorded emission traces were processed to recover burn times and temperatures for the ignited reactive material particles. It was assumed that the particle emissivity did not depend on the wavelength (gray body assumption).

4. Results and Discussion

4.1 Ignition temperatures

Figure 2 shows the ignition temperatures for all four composite powder samples as a function of the heating rate. Each point represents an individual filament ignition experiment. The ignition instant was identified using high-speed video when a first bright spot was observed on the heated powder coating. The ignition temperatures of all composites increase slightly with increasing heating rate as is expected for a thermally activated ignition mechanism. Clearly, sample D with the greatest concentration of Mg, has the lowest ignition temperature for all heating rates. Higher concentrations of boron in the compositions of all materials consistently correlate with the higher ignition temperatures. For $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites, lower ignition temperatures were observed compared to $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites. The difference in the ignition temperatures for $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ and $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites correlates with difference in the temperatures of their respective Stages II of iodine release (cf. Fig. 1). For both materials, the ignition temperature is substantially lower than that for aluminum and slightly lower than that measured for pure magnesium powder in the same experiment [20].

Figure 2. Ignition temperatures of the composites as a function of heating rate. See Table 1 for compositions of samples with different IDs.

4.2 Reactions leading to ignition

Temperatures marking positions of individual stages of iodine release (minima in the DTG traces shown in Ref. [12-14] and Fig. 1), oxidation steps (maxima in the DTG derivative traces shown in Ref. [13, 14]) and ignition temperatures from Fig. 2 are plotted using the Kissinger method coordinates [27] in Figs. 3 and 4 for all composites. The vertical axis shows $\ln(T^2/\beta)$, where β is the heating rate and T is the specific event temperature; the horizontal axis is the reciprocal temperature, $1/T$. Plots for each sample are shown separately for clarity.

Using Kissinger analysis, kinetic trends were identified for the iodine release stages and for the material oxidation steps observed in the low-heating rate TG experiments. These trends can be extrapolated to higher heating rates to observe whether they correlate with the experimental ignition temperatures. For samples A and B, both iodine release stages I and II could correlate with ignition. For these samples, oxidation steps are omitted in Fig. 2, because the first oxidation

step occurred at a higher temperature, compared to that of the iodine release stage II. Therefore, it was concluded that the oxidation steps observed in TG experiments in oxygen are irrelevant for interpretation of the ignition events, occurring at the same or even lower temperatures, but at much higher heating rates.

For the Mg·B·I₂ composites (cf. Fig. 4), it is apparent that iodine release stage II correlates best with ignition. For sample C, however, neither first iodine release stage nor the first oxidation step could be ignored, if respective trends were extrapolated to higher heating rates.

In an earlier study for the Al·B·I₂ composites [12], the iodine release mechanisms for stages I and II were identified. As noted above, stage I was explained as release of the unattached iodine. Stage II was caused by the iodine release accelerated due to formation of AlB₂. Much more iodine was released in stage II, compared to stage I, as shown in Fig. 1. Therefore, it is considered more likely that ignition is triggered in the Al·B·I₂ composites when AlB₂ is formed causing a substantial iodine release, and thus disruption of the sample surface. Additionally, for both samples A and B stage I occurs at the same temperature while stage II occurs at a higher temperature for sample A than for B, as shown in Fig. 1. Therefore, the observation that the ignition temperature for sample A is higher than for B as shown in Fig. 2, further supports that stage II iodine release is most likely the cause of ignition.

Although a weaker effect, a similar shift in the temperature for stage II is seen for Mg·B·I₂ composites C and D in Figure 1. The iodine release stage II in these composites occurs due to MgB₂ formation [13, 14], correlating best with the ignition temperatures. Thus, it is suggested that formation of MgB₂ in nanocomposite Mg·B·I₂ materials and the associated iodine release

provide their most likely ignition trigger, qualitatively similar to the formation of AlB_2 in the $\text{Al}\cdot\text{B}\cdot\text{I}_2$ composites.

Figure 3. Kissinger plots showing ignition temperatures measured at different heating rates together with peak positions of iodine release stages observed in the DTG traces for samples A and B.

Figure 4. Kissinger plots showing ignition temperatures measured at different heating rates together with peak positions of iodine release stages and oxidation steps observed in the DTG traces for samples C and D.

4.3 Aerosol combustion

All samples ignited inside the explosion vessel and examples of characteristic pressure traces are shown in Fig. 5 for each sample. The initiation of the igniter corresponds to the time of zero. A summary of CVE results is given in Table 2 for the samples A – D and for pure Al, and Mg. Samples B and D, containing less boron, have higher normalized maximum explosion pressure, (P_{max}/P_0) , and exhibit higher maximum rate of pressure rise, $(dP/dt)_{max}$, compared to samples A and C. From earlier work [21, 28, 29], (P_{max}/P_0) and $(dP/dt)_{max}$ are known to be proportional to the flame temperature and combustion rate, respectively. Additionally, shorter ignition delays are observed in Fig. 5 for samples B and D compared to samples A and C with higher concentration of boron. Clearly, the effect of boron concentration on the reactivity of the

samples is observed, where a lower concentration of boron in either composite yields a more reactive material.

A useful assessment of the combustion efficiency for different powders may be obtained comparing the experimental pressure ratios, P_{max}/P_0 with those predicted by thermodynamic equilibrium calculations performed using NASA CEA code [30]. A constant volume configuration was selected in CEA with the sets of reactants available in the CVE experimental conditions. Previous work suggested that for CVE experiments the correction for the powder mass may be as large as 20% [31] due to imperfect aerosolization of the powder. Additionally, some powder could be deposited on the chamber walls and thus be removed from the aerosol. Still, the entire experimental mass load of 4.65 g was considered in the present calculations. Maximum pressures obtained in each calculation are shown in Table 2, where they can be directly compared to the CVE experimental results. Additionally, the efficiency is calculated and presented as a ratio of the experimental and theoretical values of $(P/P_0)_{max}$ for each material.

The CEA-predicted pressures are consistently higher for all composites compared to the experimental pressures. This is an expected result due to the imperfect aerosolization discussed earlier. Furthermore, when comparing the efficiency of the Al·B·I₂ composites to pure Al with comparable particle sizes, similar combustion performances are observed. Considering the efficiency of Mg·B·I₂ composites, a surprising and positive result is seen where combustion performance of the composites surpasses that of the pure Mg powder with comparable particle sizes. Thus, adding a small amount of boron improves combustion performance of Mg-based composites.

For both Al- and Mg- containing composites, higher rates of pressure rise and higher maximum pressures are observed for samples with greater concentrations of Al and Mg, respectively. The effect is particularly strong for Mg-containing composites.

Figure 5. Characteristic pressure traces recorded in CVE experiments for all samples.

Table 2. CVE experiment results compared to other fuels (data represent average values $\pm$ standard deviations).

Material	Volume-based avg. particle size (μm)	$(d(P/P_0)/dt)_{\text{max}}$ (s^{-1})	$(P/P_0)_{\text{max}}$ experim.	$(P/P_0)_{\text{max}}$	
				calculated by CEA	Efficiency
Al (H5) [32]	9.0	439 ± 105	8.5 ± 0.3	12.4	0.69
Mg (-325 mesh)	31.7	76 ± 42	5.7 ± 0.6	12.1	0.47
Sample A	8.6	154 ± 30	6.1 ± 0.1	8.7	0.70
Sample B	13.4	221 ± 74	6.5 ± 0.3	9.1	0.71

Sample C	29.8	80±23	4.8±0.5	8.9	0.54
Sample D	32.0	285±51	6.6±0.8	10.0	0.66

4.4 Particle combustion

Particles were ignited in an air-acetylene laminar flame and their combustion was examined based on their optical emission. The emission pulses collected using the three filtered PMTs were processed using a technique described in detail elsewhere [25, 33]. The distribution of the emission pulse durations representing burn times was correlated with the particle size distribution (PSD) shown in Ref. [12-14], to obtain a trend for the burn time as a function of the particle size. Particle size distribution (PSD) of each sample was measured with low-angle laser light scattering using a Beckman-Coulter LS230 Particle Counter. The results are shown in Fig. 6. For reference, data for the pure Al [23] and Mg [25] are also shown. For the Al·B·I₂ composites, slightly longer burn times were obtained for the same particle sizes for sample A with higher concentration of boron compared to sample B. In contrast, for the Mg·B·I₂ composites, a pronounced effect of boron concentration on burn time is not observed for samples C and D. It is clear that the Al·B·I₂ composites burn longer than the Mg·B·I₂ composites. Also it is apparent that the composite particles burn longer than the reference pure metal powders, possibly extending the time of iodine release from these composites and allowing for longer mixing time of iodine with challenge bioaerosols.

For each material, the experimental trends shown in Fig. 6 were fit with a power law function, $\tau = a \cdot d^n$, where τ is the burn time, d is the particle diameter, parameters a and n are rate coefficient and adjustable exponent, respectively, shown in Table 3. For all the materials, the exponent, n , varies in the range of approximately 0.5-1. The values of exponent, n , for samples A

and B containing aluminum are close to that of pure aluminum. However, the exponent, n , for pure magnesium is clearly lower than those for samples C and D, which are boron-based composites containing magnesium.

Figure 6. Burn times of the composites and pure metal powders as a function of particle size.

Table 3. Parameters of an empirical description of the burn time, τ [ms], as $\tau = a \cdot d^n$ law for different materials (particle diameter d is taken in μm).

Sample ID	Rate coefficient	
	(a, [ms/ μm^n])	
Pure Al	4.55	0.5
Pure Mg	1.88	0.52
A	9.59	0.48
B	7.8	0.55
C	4.59	0.77

An example of the recorded filtered emission traces and respective temperature traces for an individual pulse produced by a burning particle is shown in Fig. 7. Unfortunately, emission filtered at 900 nm was much weaker than 700- and 800-nm signals shown, so that it was poorly suited for temperature measurements. As shown in Fig. 7, for 700 and 800 nm, the emission of the particle is substantially above the background level. Also, temperatures implied by the ratios using the higher amplitudes of the filtered emission intensity signals are rather stable. In this work, multiple emission pulses were processed and the average temperature was determined for each pulse for the period of time while the emission signal exceeded half of its peak value. The correlation between particle sizes and temperatures are shown in Fig. 8. The error bars show standard deviations for the obtained average temperatures. Due to a relatively large scatter, varying the boron concentration does not alter the combustion temperatures significantly for all composites. However, a trend is seen for the composites containing magnesium (samples C and D). As the particle size increases, the combustion temperatures decreases. A similar correlation is much weaker for composites containing aluminum (samples A and B). The present temperature measurements are affected by emission produced by both burning particle surface and surrounding gas phase flame, containing multiple condensing smoke particles. The temperature of the smoke is typically higher than that of the burning particle surface. Respectively, the observed effect of reduced temperatures for coarser particles is most likely representative of a weakening effect of the vapor phase flame emission on the optically measured temperatures for larger size particles. Consistently with this idea, a lower flame temperature was measured for coarser Mg particles in Ref. [25].

Generally, for both Al- and Mg- based composite materials the measured flame temperatures are lower than those for pure Al [34, 35] and Mg [25] powders. This is likely indicative of a greater role of surface reactions for respective composite materials containing boron and iodine.

Figure 7. An example of a single emission pulse of sample D recorded by different filtered PMTs and the respective temperatures inferred by the intensity ratios.

Figure 8. Temperature versus particle size for pulses recorded for all samples in air-acetylene flame

5. Conclusions

Ignition temperature were measured optically and studied as a function of heating rate for the most stable ternary compositions of $\text{Al}\cdot\text{B}\cdot\text{I}_2$ and $\text{Mg}\cdot\text{B}\cdot\text{I}_2$ composites. Increase in the boron concentration affected the ignition temperature negatively, where the ignition temperatures were greater for samples containing higher concentration of boron. In all composites, the ignition was triggered by the iodine release stage II, where formation of AlB_2 or MgB_2 expels iodine from the composite and most likely causes disruption of the particle surface. During aerosol combustion, the composites have similar or better combustion efficiency than respective pure Al or Mg powders with the same particle sizes. Among different composites, the maximum combustion pressures were inversely proportional to the boron concentration. Similarly, as the boron

concentration increased, the aerosol combustion rates decreased consistently for all composite materials.

Both pure metal and composite materials were injected into an air-acetylene flame. From optical measurements, both burn times and temperatures were obtained. The measured trends for burn times, τ , as a function of particle size, d , for all materials were described by a d^n law, where the exponent n varied from 0.5 to 1. All of the composites burned consistently longer than the pure Al and Mg powders with the same particle sizes. Longer burn times may be extending the time available for the iodine release and mixing with challenge bioaerosols. The burn temperatures of the composite powders were lower than those for pure Al and Mg powders, highlighting the diminished role of the vapor-phase reactions for the composite, boron and iodine containing materials.

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