



High density reactive composite powders

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ABSTRACT

Materials capable of armor penetration and prompt chemical energy release are desired for future weapon systems in order to better couple the kinetic energy of a projectile to its target. High-density metals used today such as tungsten are slow to react, and do not generate as much chemical energy as lower density materials, such as aluminum or boron. To design materials with a high density and reactivity, composites including boron, titanium and tungsten were prepared by mechanical milling. The specific composition density was chosen to match that of steel, 7.8 g/cm³. The proportions of the elemental metals were selected to induce a highly exothermic formation of titanium boride, which would raise the material temperature and assist the initiation and combustion of tungsten. Composite powders were prepared using both single-step and staged milling protocols, and characterized by electron microscopy, x-ray diffraction, thermal analysis, and a custom constant-volume combustion test. Staged milling produced powders with the best degree of refinement while preventing intermetallic reactions during milling. An optimized structure with well-refined components capable of a rapid combustion was prepared by milling elemental B and W for 4 h, followed by the addition of Ti and milling for an additional 2 h in a second stage. The combustion test showed evidence of tungsten combustion upon initiation of all prepared ternary materials in an oxidizing environment. The tungsten combustion occurred most effectively, generating the highest pressure and rate of pressure rise for the material with the optimal microstructure.

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1. Introduction

Reactive materials do not detonate but burn rapidly with a gravimetric and volumetric heat release comparable to or exceeding that of conventional propellants and explosives [1]. They can be used as fuels or customized fuel additives [2–4], reactive liners [5–7], weapons casings [6,8,9], as reactive solders or brazes [10,11], and as components of pyrotechnic devices [12,13]. Most of such materials are based on metals, such as Al, Mg, B, Ti, Zr, etc. Both absolute heat release and rate of reaction are important while utilizing reactive materials in all practical systems. For an important class of applications involving kinetic penetrators [14–16], it is also necessary for the reactive structures to have high density to maximize the momentum of the projectile. The combination of high heat release, high burn rate, and high density is difficult to find while considering individual metals with high heats of combustion;

however, such a combination may be achieved in composite structures.

Although aluminum is by far the most common metal fuel additive [17–19], when selecting components for a composite material, it is natural to consider boron, which possesses the highest volumetric theoretical heat of oxidation of any element at ca. 147 kJ/cm³ [20]. Limitations of boron include its low density (2.5 g/cm³), long ignition delays and relatively low burn rates [17]. It has been shown that combining boron with titanium yields a composite with an accelerated burn rate and increased material density [19,21–26]. The composites were prepared as mixed powders [27], titanium coatings on boron powders [28], and fully-dense composite powders prepared mechanochemically (or via ball milling) [26,28,29]. The latter approach is most versatile and practical; it also enables one to readily control the composition of the prepared composite. Upon initiation, boron begins reacting with titanium forming TiB and/or TiB₂ generating 10.7 and/or 21.6 kJ/cm³, respectively [25]. These are some of the most exothermic intermetallic reactions. Although they are not matching the oxidation heat release of either boron or titanium, they serve to rapidly raise the temperature of the

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composite. When such a fast and nearly volumetric heating occurs in an oxidizing environment, the material goes on to oxidize rapidly and completely [25,26], generating more heat per unit volume than burning aluminum, 103.5 or 111.5 kJ/cm³ for B·Ti and 2B·Ti, respectively (compare to 83.8 kJ/cm³ for aluminum combustion [26]). The densities of B·Ti composites (3.9 and 3.6 g/cm³ for B·Ti and 2B·Ti, respectively), exceed that of aluminum (2.7 g/cm³).

Despite energetic and density advantages, B·Ti composites do not match the density of common structural materials, such as steel, used to construct munition casings. To increase the density of reactive composite material used for kinetic energy penetrators or similar applications, highly reactive B·Ti composites can be combined with tungsten (density of 19.3 g/cm³). Tungsten heavy materials are widely used or explored for the penetrators [25,26,30]; however, they commonly do not rely on tungsten combustion. Combustion of tungsten is used in selected pyrotechnic devices [31,32]; however, tungsten is known to be difficult to ignite and have low burn rates, which mostly restricts the current uses of its combustion to slow-burning delay mixtures [33]. Recently, it was shown that combustion of tungsten can be catalyzed by preparing its composite with zirconium, another readily burning metal [34]. The approach involving catalyzing tungsten combustion is promising for the reactive structural materials and is explored here by combining tungsten with a boron-titanium composite. The composition selected for this study is that having the density of steel, 7.8 g/cm³. It is achieved by combining boron, titanium, and tungsten with the mole percentages of 53, 26, and 21, respectively. The total energy of the intermetallic reaction in this composite is 9.36 kJ/cm³ (formation of TiB₂); its total heat of combustion (or complete oxidation of all metals) is 108 kJ/cm³. The paper describes mechanochemical preparation of the composite powder and its characterization involving electron microscopy, thermal analysis, and combustion experiments.

2. Experimental

2.1. Materials

Starting materials and the overall composition of the prepared composites are described in Table 1. The particular composition used here was chosen in part to maximize the energy release by the TiB₂ formation, using a 2:1 B:Ti atomic ratio. The amount of tungsten was chosen to yield a final theoretical maximum density equaling that of steel, or 7.8 g/cm³. After an initial set of composites prepared with –325 mesh tungsten, a different, finer, tungsten powder was used in order to improve refinement of the components. Binary B·Ti and B·W composites were prepared with the respective component ratios from Table 1.

Composite B·Ti·W powders were prepared by mechanical milling. A SPEX 8000D shaker mill was primarily used. Five grams of powder were milled with 25 g of 3/8" (9.53 mm) balls, giving a ball-to-powder ratio of 5. Milling was performed under argon, and 10 mL of hexane were used as process control agent. Milling vials and media of hardened steel, and of stabilized zirconia were both

used. Milling with steel media introduced noticeable levels of iron contamination in the resulting composite. While not necessarily detrimental to performance, this did make sample analysis more challenging due to the formation of iron-bearing phases. The compositions characterized in detail here were therefore prepared using zirconia vials and milling media.

Two general milling protocols were followed: in one set of experiments, the component powders were loaded into the milling vials together, and then milled for varying periods of time. Materials prepared using this single-step milling protocol will be denoted as B·Ti·W. In a second set of experiments, boron and tungsten powders were milled first in order to minimize the reaction between boron and titanium during milling. Combining mechanically harder boron and tungsten in the first milling step also helped achieving a more homogeneous distribution of tungsten in the final composite material. This first milling step produced binary B·W composites. Titanium powder was added later. Composites prepared using this two-stage milling protocol are designated as (B·W)·Ti. Milling times varied in the 2–7 h range based on experience from prior work [35,36]. For comparison of combustion behavior, a reference material was prepared by blending elemental tungsten with a preliminarily prepared B·Ti binary composite; this reference blend is labeled (B·Ti)+W.

2.2. Characterization

Morphology of, and elemental distribution in the prepared composites were examined on a LEO 1530 VP scanning electron microscope operating at 10 kV, and using a back-scattered electron detector.

Particle size distributions were determined using a Beckman Coulter LS230 particle analyzer. Suspensions were prepared in ethylene glycol. Measurements were repeated three times, and ultrasonic agitation was used for the final repetition. Size distributions were accepted as stable only when all repetitions coincided.

Reactive properties were characterized by differential thermal analysis using a Netzsch STA409-PG thermal analyzer. Samples were heated from 50 to 1100 °C under argon at a rate of 10° per minute, allowed to cool and then reheated. The second heating of the sample was subtracted from the first to compensate for baseline effects. Onset temperatures of exothermic reactions and the net heat release over the observable temperature range were used to compare materials against each other as well as with the literature values. These comparisons also were used to guide the details of the milling protocol.

X-ray diffraction on a PANalytical Empyrean diffractometer with unfiltered Cu-K α radiation was used to assess reaction products formed during thermal analysis.

Bulk energy release upon combustion, and combustion rate in an oxygen atmosphere were assessed in a custom constant volume chamber shown schematically in Fig. 1. In practical applications, reactive materials are expected to be ignited using booster charges or similar high-energy ignition sources. Such igniters are difficult to

Table 1
Elemental Composition.

Composition of W-B-Ti	Description			
	Atomic %	Mass %	Volume %	
Tungsten	21%	68%	27%	Atlantic Equipment Engineers, 99.9%, –325 mesh (<44 μ m) Alfa Aesar, 99.9%, 1–5 μ m
Boron	53%	10%	34%	SB Boron, SB-95, 95%, nominal size –325 mesh (<44 μ m)
Titanium	26%	22%	39%	Atlantic Equipment Engineers, 99.7%, –325 mesh (<44 μ m)

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