Extending the Compositional Limit of Combustion-Synthesized B₄C-TiB₂ Composites by Field Activation

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Composites of B₄C-TiB₂ with molar ratios B₄C/TiB₂ of up to 8 were synthesized by field activated combustion. The combustion wave velocity depended on this ratio and increased approximately linearly with the applied field. The effect of the field was analyzed and its contribution to the combustion temperature was calculated. X-ray and microscopic analyses showed the synthesis to be a two-step process. Titanium boride forms at or near the leading edge of the combustion zone and B₄C forms at or near the trailing edge.

I. INTRODUCTION

The feasibility of synthesizing composite materials directly from elemental reactants is one of the primary reasons for the current interest in the self-propagating combustion method. The use of this method to synthesize composite materials has been amply demonstrated in numerous investigations. These include the synthesis of metal-matrix and ceramic-ceramic composites. An example of the latter is the system B₄C-TiB₂, which has been previously investigated as a preform for liquid metal infiltration in armor applications. In this regard, the specific gravity of the composite is of concern, and hence the molar ratio, y, of B₄C/TiB₂ is an important factor. Using elemental reactants, such a composite can be synthesized by a self-sustaining combustion method for y ≤ 0.5 when the reactants are at an initial temperature, T₀, of 298 K. For y > 0.5, no self-sustaining combustion wave can be established without raising T₀. For example, for y values of 1.0 and 2.0, T₀ must be 800 and 1200 K, respectively. However, the preheating of the reactants to such temperatures has been shown to result in the formation of additional phases, in this case, TiC and TiB₁₉. The formation of these phases is attributed to diffusion processes occurring prior to combustion, as has also been observed in the synthesis of MoSi₂.

The fact that composites of B₄C-TiB₂ with y > 0.5 cannot sustain self-propagating combustion when T₀ = 298 K is the consequence of a low adiabatic combustion temperature, Tₚₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑₑelectronically activated synthesis was first demonstrated for the synthesis of β-SiC and has since been used to synthesize MoSi₂-SiC and other composites. The result of these studies showed that the effect of the field is largely confined to the combustion zone. The wave velocity increased linearly with an increase in field strength. On the basis of a combustion-zone confinement, a model was developed to simulate field activation. Modeling and experimental results were in qualitative agreement.

In this work, we investigate the field activation of the combustion synthesis of B₄C-TiB₂ composites with B₄C/TiB₂ ratios as high as 8.

II. EXPERIMENTAL MATERIALS AND METHOD

Powders of boron, graphite, and titanium were used in the appropriate stoichiometric ratios to form the reactant

Fig. 1—The dependence of the adiabatic combustion temperature on the B₄C/TiB₂ ratio in the product.
mixtures. The boron powders (obtained from Alfa Products, Danvers, MA) were amorphous with a mean particle size of 1 μm. These powders contained about 3 pct Mg and 5 pct B₂O₃ (amorphous) as impurities. The carbon (graphite) powders (Superior Graphite, Chicago, IL) were 99.8 pct pure and had particles of <20 μm, and the titanium powders (Alfa Products) were -325 mesh with a reported purity of 99 pct. Stoichiometric amounts of these powders were weighed to give composites B₄C-TiB₂ with y values of 0.5, 1.0, 2.0, 3.0, 5.0, and 8.0. The powders were mixed in a vibratory mixer for 15 minutes and then ball-milled for 10 hours. Powder mixtures were then uniaxially pressed into orthorhombic pellets of 21 × 12 × 8 mm dimensions. The relative density of these pellets was maintained at about 50 pct.

The pellets were placed between graphite electrodes inside a combustion chamber equipped with a tungsten coil ignition source. Figure 2(a) shows a schematic representation of the sample, electrodes, and ignition source. The other geometries, shown in Figures 2(b) and (c), will be discussed later. When a voltage is applied across the electrodes and the ignition coil is activated, a self-propagating wave is initiated at the end nearest the heat source and the wave propagates in a direction perpendicular to the field. Combustion wave velocities were measured from video recordings using a time-code generator. Temperatures were measured by a two-color pyrometer with a response time of 0.01 seconds. These measurements, along with those of current and voltage were synchronized through a computerized data acquisition system. Product phase analysis was made by X-ray diffraction and microstructural information was obtained through scanning electron microscopy.

III. RESULTS AND DISCUSSION

In the absence of a field, only pellets with y = 0.5 can be ignited to produce an SHS wave when the reactants are at room temperature. On the other hand, the application of a field without the ignition source produces no combustion wave or any detectable reaction. This can be seen from Figure 3(a), which shows the time dependence of the applied voltage (38 V) and current for a sample with y = 2.0. The exceedingly low value of the current excludes the possibility of significant Joule heating of the reactants. The initial resistance of the sample was about 5000 ohm. When