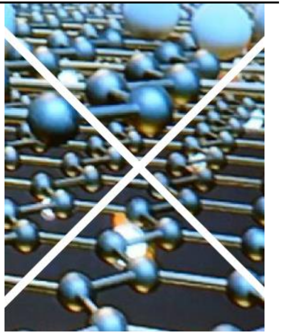
TIJMET, Vol. 003 (2020) 109-119

The International Journal of
**Materials and Engineering Technology**

ISSN: 2667-4033
TIJMET ID: 200302006 ( 11 pages)
Type of the Paper: Article
Received 01.11.2020 Revised: 09.10.2020 Accepted: 09.11.2020 Published (Online): 16.11.2020
Journal homepage: http://dergipark.gov.tr/tijmet



# ENERGETIC MATERIALS AND METAL BORIDES FOR SOLID PROPELLANT ROCKET ENGINES

**MUSTAFA GUVEN GOK** *[1] **and OMER CIHAN** [2]

[1] Hakkari University, Engineering Faculty, Materials Science and Eng. Department, Turkey
*m.guvengok@hakkari.edu.tr
[2] Hakkari University, Engineering Faculty, Mechanical Eng. Department, Turkey

## Abstract

Energetic materials are a kind of materials involving a huge amount of chemical energy and they can release this energy suddenly or slower. In this review study, types, usage areas and compositions of energetic materials are explored. Especially, energetic materials for solid propellant rocket engines are evaluated more extensively. The advantages and synthesis techniques of metal borides as an alternative to fuel in solid propellant rocket engines are criticized in the light of the relevant literature. It is concluded that, metal borides, particularly aluminum and magnesium borides, are promising materials as solid rocket engine fuel. However, new methods should be developed for the synthesis of higher amount and purity of metal boride.

**Keyword:** Energetic Materials, Metal Borides, Solid Propellant, $AlB_2$, $MgB_2$.

## 1. Introduction

Rocket engines simultaneously contain the fuel required for propulsion and oxidizers required for combustion. Therefore, they are different from other engines; they can work both in the Earth's atmosphere and in space where oxygen is absent. Since the fuels used by rockets contain a significant amount of chemical energy and can release this energy quickly, these kind of fuels are classified as "energetic material" [1]. A rocket used to launch a satellite or to carry something into space is called as a launch vehicle, while a rocket carrying a warhead as payload is called as a missile [2].

On the other hand, efforts to produce rocket propellant that will carry launch vehicles and missiles to longer range with more economical and effective methods are gaining importance today. The aims of developed countries in their efforts to produce more efficient rocket engines and propellants according to their sociological, economic and geopolitical situations can be classified as follows;

i. to improve the quality and quantity of deterrent materials in their defense policies,
ii. to earn income through exports in the field of defense industry,
iii. to expand their technology frontiers with satellite and space studies,
iv. to increase their prestige in the international platform.

The way to have information about advanced and efficient rocket engines and propellants and to take them one step further is to follow simple mechanisms, alternative fuel systems and current developments in this regard, as well as to do original researchers [1–4]. Although there are some studies on the use of energetic materials and metal borides as solid rocket fuel, which have gained importance in recent years, there are not many sources prepared as review on this subject. Therefore, this review article will constitute an important scientific resource for those who are curious and/or want to work on "Energetic Materials and Metal Borides for Solid Propellant Rocket Engines".

## 2. Solid Propellant Rocket Engines

In the rocket engine, high temperature and pressure are released with the combustion of the fuel and oxidizer [3]. The energetic materials used as propellants can be divided into three groups in terms of the type of fuel. These are solid fuel, liquid fuel and hybrid fuel systems where solid and liquid fuel are used together. Solid propellant rocket engines contain fewer components and have simple-structure than liquid and hybrid propellant rocket engines. Therefore, they have relatively low production costs, ease of storage and transportation. This situation makes the solid fuel engine to be preferred as an propellant engine in defense and space systems [4, 5]. Another advantage of solid propellant is that a thrust is relatively stable and can therefore be produced and stored for future use. Moreover, they can burn very quickly and have no sensitive to shock, vibration and acceleration. There is no need for a thrust pump, so rocket engines have less complex construction [6–9]. A simple solid propellant rocket engine consists of the engine casing, insulation, igniter, nozzle and solid propellant. Figure 1 shows general view and basic components of the solid propellant rocket engine.

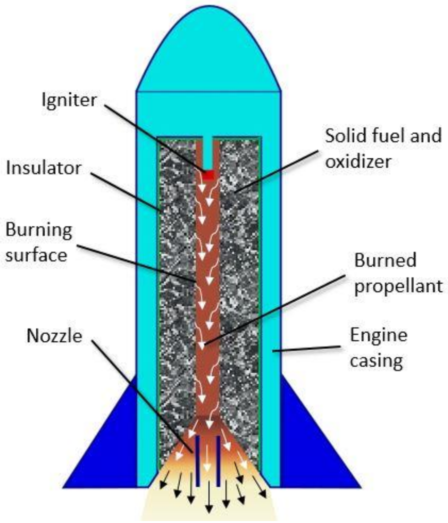


**Figure 1.** Basic components and structure of the solid propellant rocket engine.

The engine casing maintains insulation, igniter and solid propellant in a controlled manner. The casing additionally preserves the structural integrity of the rocket. The engine casings are generally produced from high strength metal alloys (high-resistance steels or high strength aluminum alloys) or composite materials (Glass, Kevlar® and/or Carbon reinforced material). Insulator protects the engine casing against the high temperatures that occur in the combustion chamber. Insulator is a layer made from a composite material having low thermal conductivity and it is located in between the internal surface of the engine case and the outer surface of the propellant. Insulator generally is produced from Ethylene Propylene Diene Monomer (EPDM) reinforced with chopped Kevlar®, peroxide as curing agent (cross-linking agent) and ammonium polyphosphate $(NH_4PO_3)_n$ as a flame retardant agent [10]. Igniter enables the mechanical, electrical or chemical input formation to be transformed into heat energy output. Also, it triggers the first burning with the release of energy. Energetic black powder, nitrocellulose $(C_6H_7(NO_2)_3O_5)_n$ or nitroglycerine $(C_3H_5N_3O_9)$ has been used as igniter material. Nozzle determines the performance of the rocket engine. The main task of the rocket nozzle is to convert the energy obtained as a result of chemical reaction into kinetic energy. High pressures and temperatures are reached with the burning of solid propellant. The thrust occurs when particles with these temperatures and pressures are accelerated by passing through the convergent-divergent nozzle [11]. As nozzle material, generally carbon-based materials such as carbon-carbon composite or amorphous graphite are used. The type of energetic material also affects the pressure-time and thrust-time graphs in parallel with the cross section geometry [12, 13]. Rocket propellant systems contain the mixture of fuel as energetic material and oxidizer required for the combustion of the fuel.

## 3. Energetic Materials

Energetic materials are defined as materials that are in solid, liquid or gaseous state and can rapidly release chemical energy stored in their structures by combustion [1, 14]. Combustion products, heat and pressure emerge with the release of energy in the energetic material. These combustion products are in gaseous state containing some solid matter in the form of smoke or soot. The most important difference between energetic materials and normal flammable materials is very high heat energy released by energetic materials in per unit time.

For example, one gram of coal generates about 32.5 kJ of energy when burned in two minutes (120 seconds) in open air. On the other hand, when one gram of TNT explodes, it releases about 2175 kJ of energy in 1 microsecond (μs). This means about a million times more energy than coal [15]. Since the oldest information about energetic materials was found in a text describing the burning of sulfur and nitrate salt mixtures in powder form written in the IV. century in China, it can be said that the history of energetic materials is very old. In 1867, dynamite, a mixture of nitroglycerin and silica that is more stable and safer to use, was invented and commercialized. Although it is thought that energetic materials are used only in military fields, today they are mostly used in fields such as construction, mining and rocket engines for space exploration. Although they have a very old history and therefore an improvement process, studies to develop more economical and more powerful energetic materials are still being carried out rapidly [16, 17]. It is possible to divide energetic materials into three classes according to their usage areas (Figure 2).

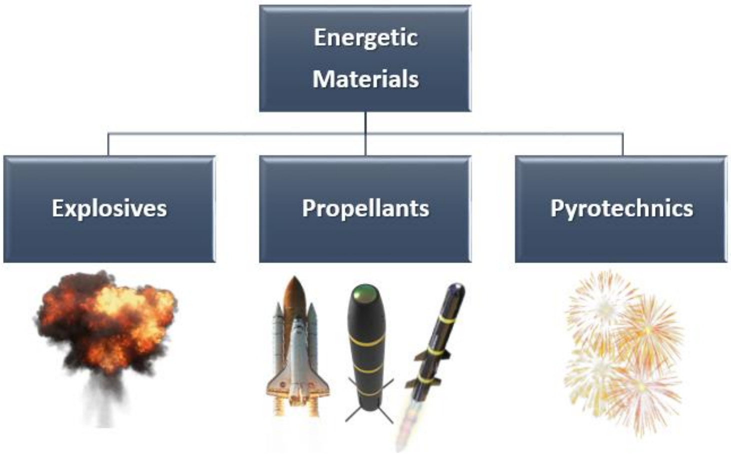


**Figure 2.** Classification of energetic materials according to their usage areas.

Explosives release a great deal of energy in a time period of microseconds and expand greatly in volume. A supersonic shockwave forms and propagates through the material when explosive material starts to detonates. Material is under high pressure at the wave front and this leads an increase temperature and hence exothermic reactions starts. The temperature and pressure further increase due to exothermic reaction and this allows to prolong the propagation of shock wave [17]. Explosive energetic materials are generally in the class of heterocyclic (at least one of the ring atoms is a hetero-atom [O, N, S]) compounds. The terminological abbreviations, chemical names, molecular formulas and energy contents of some energetic materials used as explosives are given in Table 1.

**Table 1.** Some energetic materials used as explosives [18].

| **Abb.** | **Chemical Name** | **Molecular Formula** | **$E_d$, Energy Content (kJ/cm$^3$)** |
|---|---|---|---|
| DATB | 1,3-Diamino-2,4, -6-trinitrobenzene | $C_6H_5N_5O_6$ | 8.6 |
| EDNA | Ethylenedinitramine | $C_2H_6N_4O_4$ | 9.2 |
| HMX | Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetraazacin | $C_4H_8N_8O_8$ | 11.1 |
| HNB | Hexanitrobenzene | $C_6N_6O_{12}$ | 14.3 |
| NG | Nitroglycerine | $C_3H_5N_3O_9$ | 10.0 |
| PETN | Pentaerythritol tetranitrate | $C_5H_8N_4O_{12}$ | 10.5 |
| RDX | Hexahydro-1,3,5-trinitro-1,3,5-triazine | $C_3H_6N_6O_6$ | 10.4 |
| TATB | 1,3,5-triamino-2,4,6-trinitrobenzene | $C_6H_6N_6O_6$ | 8.6 |
| TETRYL | N-methyl-N, 2,4,6-tetranitro Benzenamine | $C_7H_6N_4O_6$ | 8.8 |
| TNA | 2,4,6-trinitroaniline | $C_6H_4N_4O_6$ | 8.1 |
| TNAZ | 1,3,3-trinitroazetidine | $C_3H_4N_4O_6$ | 11.2 |
| TNB | 2,4,6-trinitrobenzene | $C_6H_3N_3O_9$ | 8.6 |
| TNT | 2,4,6-trinitrotoluene | $C_7H_5N_3O_6$ | 7.7 |

Pyrotechnics burn producing special effects such as heat, bright light, sparks, fog, and sound when they are ignited. In this way, they are energetic materials used for purposes such as entertainment, lighting, ground marking with smoke and signaling. The main feature that distinguishes pyrotechnics from explosives is the much slower burning speed of pyrotechnics [19]. A general mixture of pyrotechnic comprise of a fuel, an oxidizer, a carbon source and chlorine donors to improve color and other chemical additives to change the look or sound [20, 21]. Zirconium (Zr), Magnesium (Mg) and Boron (B) are frequently used as fuel in pyrotechnics. Potassium Chlorate ($KClO_3$), Potassium Perchlorate ($KClO_4$) and Potassium Nitrate ($KNO_3$) are frequently used as oxidizers in pyrotechnic systems. Many pyrotechnic compositions contain binders such as viton® and nitrocellulose ($(C_6H_7(NO_2)_3O_5)_n$). Even the slightest change in the formulation of the pyrotechnic composition significantly affects the characteristics of the pyrotechnic material [21, 22].

Unlike explosives, the energetic materials used in rocket fuels are desired a combustion having slow and controlled characteristic. For the reasons mentioned in the previous section, solid propellant rocket engines are considered to be more important in military and space research applications [5]. To give an example of solid rocket fuel compositions, the first of these is black powder that has been used for many years. However, black powder has now been replaced by alternative (modern) solid rocket fuels and it is currently used as a fuel in model rockets and as an igniter in other solid fuel rockets. The black powder is in the composition of potassium nitrate ($KNO_3$), sulfur (S) and charcoal (C). It is reported that the best composition to use black powder as rocket fuel or igniter is 75% $KNO_3$ (oxidizer), 10% S and 15% C by weight. These components must be mixed and pressed mechanically or hardened by adding natural rubber to the composition. Modern solid rocket fuels consist of solid and liquid components during the mixing process. The most preferred energetic material composition in solid rocket fuels consists of ammonium perchlorate ($NH_4ClO_4$) as an oxidizer which is an oxygen-rich salt, and aluminum (Al) powder as fuel. Ammonium perchlorate contains 54.5% oxygen by weight. As the liquid phase used in the mixing process, a long chain industrial polymer is generally preferred. The purpose of this polymer is to make the powder mixture formable by keeping particles together and to ensure their fixation where desired place in the rocket engine. The most widely used polymer in this process is the Hydroxyl Terminated Polybutadiene ($C_xH_yO_z$) industrial polymer which is also known as HTPB. HTPB has a viscosity similar to corn syrup. A hardener (curing agent) such as isophorone diisocyanate ($C_{12}H_{18}N_2O_2$) is used to initiate the cross polymerization. A cast mandrel with a specific geometry is used to pour the mixture having a sticky state into the rocket engine fuel chamber. Exit geometry of the propellant is also given thanks to this cast mandrel. The most preferred geometry is in the form of a star. Then the rocket engine is placed in a curing oven (at a temperature of 60-70 °C) and the curing process is performed. Table 2 shows the name, formula and ratio of the most commonly used energetic materials for modern solid propellant rocket engines. Apart from these, rate and ratio of combustion can be controlled by adding additives such as iron oxide ($Fe_2O_3$), which is called burning rate catalyst, to the structure between 0.5 and 3% [23], [24]. Besides ammonium perchlorate, materials such as potassium perchlorate ($KClO_4$), hexanitrohexaazaisowurtzitane (CL20 - $C_6N_{12}H_6O_{12}$), lithium perchlorate ($LiClO_4$) and potassium nitrate ($KNO_3$) are also used in some applications as solid oxidizers [25].

**Table 2.** A typical solid fuel composition for rocket propellant [24].

| Composition | Function | Formula | wt. % |
|---|---|---|---|
| Ammonium perchlorate | Oxidizer | $NH_4ClO_4$ | 68 |
| Aluminum | Fuel | Al | 20 |
| Hydroxyl Terminated Polybutadiene (HTPB) | Fuel-Binder | $C_xH_yO_z$ | 11.5 |
| Isophorone diisocyanate | Curing agent | $C_{12}H_{18}N_2O_2$ | 0.5 |

On the other hand, elemental metals and their compounds (such as Al, Mg and Mg-Al) as energetic material in solid rocket fuels have low enthalpy of combustion and tendency to accidentally ignite [26]. It is also stated that elemental metals do not completely burn in practical application. The combustion temperatures of energetic metals are given in Table 3. When the table is analyzed, it is understood that Boron and Lithium have the best combustion temperature values (gravimetrically) as energetic materials. However, lithium is not very convenient for use as rocket fuel due to its high toxicity, high cost, low melting point and high chemical activity properties [27]. Studies have shown that it is much more effective to use boron instead of elemental metal additives to increase efficiency of solid rocket propellants [26–28]. The heat of combustion of boron is approximately two times higher than that of aluminum (Table 3). Theoretically, thanks to this feature of boron, it is possible to increase the impulse power while the rocket fuel consumption decreases significantly. However, due to the high oxidation affinity of elemental Boron, a thin solid oxide ($B_2O_3$) film forms on the surface of the Boron particles, and this film prevents the diffusion of oxygen during combustion (the oxidizing phase cannot reach the boron), which significantly reduces the combustion efficiency [20, 28]. To solve these problems, the approach of using metal borides as energetic materials in solid rocket fuels has been developed. Thus, it has been specified that many metal boride compounds increase the combustion efficiency of boron [20, 25–28].

**Table 3.** The heat of combustion values of energetic metals [1, 27].

| Element | Heat of combustion, kJ/g | Heat of combustion, kJ/cc |
|---|---|---|
| Lithium (Li) | 43.5 | 23.2 |
| Boron (B) | 57.2 | 135.5 |
| Magnesium (Mg) | 25.1 | 43.6 |
| Aluminum (Al) | 31.4 | 84.7 |
| Silicon (Si) | 32.2 | 75.1 |
| Titanium (Ti) | 15.7 | 71.1 |
| Zirconium (Zr) | 12.0 | 77.9 |

## 4. Metal Borides as Energetic Materials

It was mentioned in the previous section that the use of elemental boron as an energetic material in rocket fuels is not efficient due to oxide layer on the surface of the boron particle. Since the oxygen in the oxidizer component of the fuel cannot reach the boron by passing through the oxide layer on the surface of the boron and so combustion becomes difficult. As a solution to this problem, the use of metal borides.is suggested. An oxygen-barrier complex oxide layer is not formed on the surface of metal borides.

The oxidation degree and oxidation speed required for combustion in metal borides are high and therefore high combustion temperature values are obtained [20, 29]. The heat of combustion values of energetic metal borides are given in Table 4. Among the metal borides, Al and Mg borides are most suitable for use in rocket propellants due to their relatively cheapness, easy synthesis process and high calorific value. It can be understood that the heat of combustion values of the metal borides in Table 4 are higher than heat of combustion values of the energetic metals given in the Table 3 of previous section.

**Table 4.** The heat of combustion of values of energetic metal borides [1, 20].

| **Boride** | **Heat of combustion, kJ/g** | **Heat of combustion, kJ/cc** |
|---|---|---|
| $AlB_2$ | 39,5 | 125,1 |
| $AlB_{12}$ | 50,9 | 131,4 |
| $MgB_2$ | 37,8 | 99,6 |
| $TiB_2$ | 23,9 | 108,0 |
| $Mg_{0,5}Al_{0,5}B_2$ | 38,6 | 112,2 |
| $ZrB_2$ | 17,7 | 108,3 |
| $AlMgB_{14}$ | 28,3 | 77,8 |

Aluminum diboride ($AlB_2$), which is cheaper and highly energetic than another energetic metal boride compounds, gains importance in this field. $AlB_2$ has the potential to be an effective energetic material due to its high heat of combustion value both gravimetrically (39.5 kJ/g) and volumetric (125.1 kJ/cc). Moreover, Korotkikh et al. [25] determined that if $AlB_2$ is used as energetic material instead of Al, the ignition delay time can be reduced in between 1.7 and 2.2 times. The combustion improvement mechanism of $AlB_2$ in rocket propellant can be explained as follows [1];

(i) $AlB_2$ burns and releases $2Al_2O_3 \cdot B_2O_3$ ($Al_4B_2O_9$) as a combustion product.
(ii) This combustion product forms a solid needle-shaped structure on the surface of the oxidized particle.
(iii) This borate binds the liquid $B_2O_3$ and opens the unoxidized surface.
(iv) As a result, the diffusion rate of the oxidizer towards the surface of the fuel particle increases.

The synthesis of $AlB_2$ from powders its elemental components is relatively low cost and effective. It is reported that the reaction between Al and B for the synthesis of $AlB_2$ starts at 600 °C and reaches a maximum point around 900 °C [30]. Binary phase diagrams between Al-B and Mg-B are given in Figure 3 (a) and (b), respectively. According to these phase diagrams, $AlB_2$ is formed by a peritectic reaction at around 980 °C in the ratio of approximately 44.5% boron by weight. Above 980 ° C, it starts to decompose and transforms into liquid Al and α-$AlB_{12}$ as shown in the following formula (1);

$$6AlB_2 \rightarrow AlB_{12} + 5Al_{[Liquid]} \tag{1}$$

Similarly, magnesium diboride ($MgB_2$) is formed at 1545 °C and in the ratio of approximately 47% boron by weight.

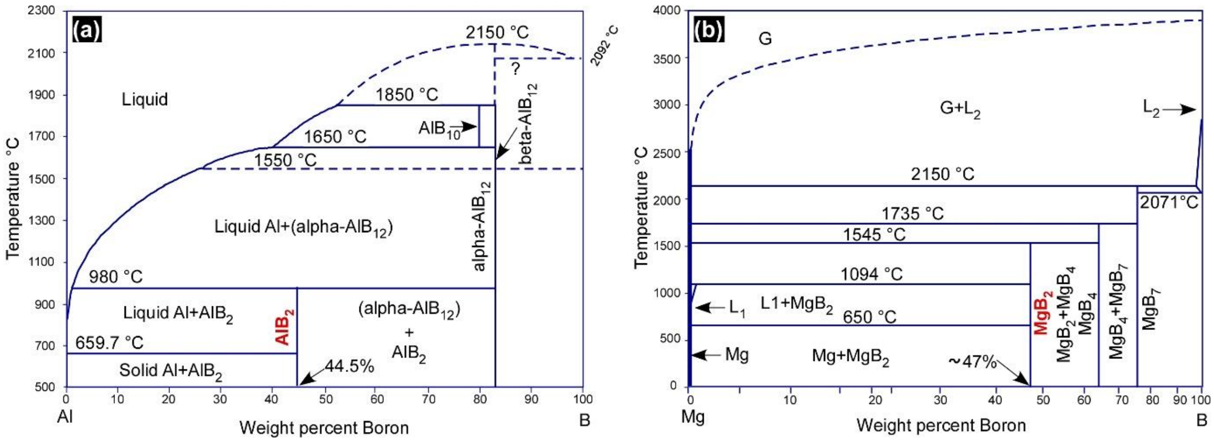


**Figure 3.** Al-B (a) and Mg-B (b) binary phase diagrams (they were redrawn from [31–34]).

Whittaker et al. [30] mechanically mixed elemental boron (1.2 µm in size and 96.8% purity) and aluminum (3 µm in size and 99.7% purity) powders and then pressed isostatically. To synthesize $AlB_2$, the pressed mixtures of powders were heated in different atmospheres (argon, vacuum and reducer) at temperatures ranging from 700 to 975 °C and the time periods ranging from 1 to 12 hours. It is found that, in terms of the purity of the synthesized $AlB_2$, (i) the temperature was the most important factor among the synthesis parameters, (ii) the synthesis at the temperature of 900 °C was the most optimum for the reaction of Al and B, (iii) the time had no effect on the $AlB_2$ formation, (iv) synthesis process fulfilled under vacuum gave best result and (v) the decomposition occurred above 950 °C. Jiang et al. [35] mixed nano-sized elemental Al and B powders to synthesize $AlB_2$ and then heated them in an argon atmosphere at temperatures varying between 750 and 950 °C. As a result, they determined that synthesis temperature of 900 °C, synthesis time of 2 hours and 1:1.18 molar ratio were the optimum parameters for $AlB_2$. In another study [36], the researchers concluded that the synthesis process performed after mechanically alloying of Al and B in a high energy mill was more efficient and the reaction took place at a lower temperature (757 °C for $AlB_{12}$). $MgB_2$ also has been synthesized by similar methods of $AlB_2$. Guo et al. [37] mixed elemental Mg and B powders at a ratio of 1: 2 and heated them at 850 °C for 2 hours to obtain $MgB_2$. In their analysis, it was determined that the vapor of magnesium formed during combustion prevented oxide formation on the $MgB_2$ particle surface and this situation led to high combustion efficiency. In another study [38], solid phase reaction of elemental Mg and B powders was carried out using a high energy mill, and then, synthesis process was completed by heating the powders to different temperature values in an argon atmosphere. Optimum conditions in this process were determined as (i) ball to powder ratio 8:1, (ii) 7 hours of milling time, and (iii) 650 °C of synthesis temperature. In this study, unreacted Mg and impurity phases were removed from the powders by leaching process with a HCl (hydrochloric acid) solution. Moreover, the researchers measured the calorific energy value of the $MgB_2$ as 9.2 kcal/g. Apart from these, there are also studies for the production of $AlB_2$ and $MgB_2$ with Self-propagating high-temperature synthesis (SHS) method. Metal boride production process using the SHS method involves three steps: (i) preparation of the exothermic mixture, (ii) combustion synthesis and (iii) product processing. The first step is to mix the powder compositions in the required stoichiometric proportions. In the second step, the prepared powders are placed in the

SHS reactor and the combustion process is carried out. And in the last step, the synthesized products are subjected to processes such as separation and grinding. Zhukov et al. [20] and Bondarchuk et al. [27] successfully produced energetic $AlB_2$, $TiB_2$, $MgB_2$ and $Al_{0.5}Mg_{0.5}B_2$ materials using the SHS method. In their study, the researchers carried out their production in an inert gas atmosphere and determined that the purity degrees of the $AlB_2$, $TiB_2$, and $Al_{0.5}Mg_{0.5}B_2$ powders were 93.18%, 98.43% and 88.04%, respectively. In the patent literature [39], Al and B powders were mixed by a ball mill process in alcohol at the atomic ratio of B:Al = 2.1:1. Then mixture was dried and pellets were obtained by pressing powders. The reactor was electrically ignited and SHS reaction initiated. After grinding process, the purity degree and average particle size of the synthesized $AlB_2$ was between 93 - 95%, and 1.5 - 6 µm, respectively. On the other hand, as a different method from these, nano-sized boride powders can be produced from elemental Al and B mixture with the plasma recondensation method. However, while the amount of $AlB_2$ in the products obtained by this method is very low (about 2%), high amounts of $AlB_{12}$ (69-76%), free boron (5-6%) and aluminum (15-30%) can be produced [40, 41].

## 5. Conclusions

1- Energetic materials are important in both military and civilian applications.
2- Modern developments in aviation and space industry directly increase the need for new and efficient energetic materials.
3- Elemental boron and metal fuel used in solid and hybrid rocket propellants can be replaced by energetic metal borides due to their higher performance properties.
4- $AlB_2$ and $MgB_2$ are the most effective energetic materials among metal borides.
5- $AlB_2$ and $MgB_2$ are generally produced via mechanical activation of their elemental components, holding them at reaction temperature (700-900 °C) or using SHS method.

Suggestions:

1- New methods should be developed for the production of higher amount and purity of metal boride.
2- The molten-salt route allowing lower reaction temperatures for synthesis of energetic metal borides should be investigated.

## Acknowledgments and Funding

No funding to declare.

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