Borides and vitreous compounds sintered as high-energy fuels

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Abstract

Boron was chosen as fuel in view of its excellent thermodynamic values for combustion, as compared to traditional fuels. The problem of the boron in combustion is the formation of a surface layer of oxide, which delays the ignition process, reducing the performance of the rocket engine. This paper presents a high-energy fuel for rocket engines. It is composed of sintered boron (borides and carbides and vitreous compounds) with a reducing chemical agent. Borides and boron carbide were prepared since the combustion heat of the latter is similar to that of the amorphous boron (in: K.K. Kuo (Ed.), Boron-Based Solid Propellant and Solid Fuel, Vol. 427, CRC Press, Boca Raton, FL, 1993). Several chemical reducing elements were used, such as aluminum, magnesium, and coke. As the raw material for boron, different compounds were used: amorphous boron, boric acid and boron oxide.

1. Introduction

Boron was chosen as a fuel in solid propellants in view of its excellent thermodynamic values for combustion per unity of volume (Table 1), as compared with traditional fuels. The problem of boron in the combustion process is the formation of a surface layer of oxide, which delays the ignition process by preventing the arrival of oxygen to the boron surface. The final effect is that many burning particles of fuel are left outside the combustion chamber, decreasing the motor output [1,2].

The basis of our study is the incorporation of reducing agents such as aluminum, magnesium, etc. into the boron net, to avoid the formation of an oxide layer in the first combustion stage (ignition). Special crucibles were designed for the controlled reactive sintering with thermic shock conditions. The incorporation of the reducing chemical agents was done by powder technology. Suitable working conditions were designed to obtain a sintered aluminum boride or boron carbide, free of oxide and with a high combustion rate,

higher than that of the propellants used in its formulation: amorphous boron mixed with aluminum. The prepared fuel was mixed with an oxidizer plus a polymer, which together with the action of another metallic fuel, constitute a propellant in composite form.

The Ellingham diagrams [4] show us which reducing agents could be used, aluminum or magnesium. Aluminum was chosen for its high combustion values, (see Table 2).

Crystallization was avoided by working in solid state condition. Working conditions (temperature used) were designed in accordance with the phase diagram of the Al–B system [5,6].

2. Experimental section

The incorporation of reducing elements into the boron in an amorphous state was done by powder technology, and the compaction was by Cold Isostatic Pressing (CIP) which gives a high uniform pressure distribution in the sample [8] and a compact density near to the theoretical value [9]. Low compaction pressures (between 200 and 350 MPa) [10] are normally used with

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Table 1
Combustion values of different fuels [3]

Fuel	Gravimetric combustion heat (J/g)	Volumetric combustion heat (J/cm^3)
JP5	42.427	34.401
Carbon	32.746	74.195
Aluminum	31.015	83.600
$(CH_2)_n$	43.472	40.128
Boron	57.684	134.596
Magnesium	24.703	43.054

Table 2
Heats of formation of inorganic oxides [7]

Reaction	Cal/mol	KJ/mol
$2Al(s) + 3/2O_2(g) = Al_2O_3(s)$	−404.080	−1691.5
$Mg(s) + 1/2O_2(g) = MgO(s)$	−144.090	−603.0
$Ti(s) + O_2(g) = TiO_2(s)$	−288.360	−955.9
$Zr(s) + O_2(g) = ZrO_2(s)$	−262.980	−1100.9
$C(graphite) + O_2(g) = CO_2(g)$	−93.690	−392.0

materials of ceramic behavior such as boron. Since aluminum is the minority element in this case, we raised the pressure to 700 MPa to make sintering practicable.

The aluminum particles used as raw material are of irregular shape [11] on account of the rapid cooling during atomization, and they contained material with a thin layer of oxide. This oxide, another consequence of the atomization process [12], impedes particle contact and retards sintering [13]. Small particles were used, so the volumetric diffusion and the heating rate are high [14]. The oxidation of the aluminum samples is exothermic [7] and the crucible temperature raises. The aluminum oxide generated is reduced in the whole temperature scale [4] by the magnesium content on the boron raw (1–2%). The manufacture of borides is by aluminum excess [15–18].

The heat given off by the formation of aluminum oxide during sintering, raises the temperature in the crucible and the aluminum borides are formed. Of the two major borides, AlB_{10} and AlB_{12}, the more thermo-dynamically useful is the AlB_{10} which is stable between 1933 and 2123 K [5], as was confirmed by the diffraction patterns obtained.

Industrial sintering atmospheres containing nitrogen and dissociated ammonia [19] were not appropriate in our case since they produce boron nitrites and hydrides, so green sintering was made possible by the use of specially designed crucibles.

Metallothermic techniques and nonreactive sintering were found suitable for preparing borides and boron carbides. The main steps in these procedures are: mixture and homogenization of powders by using organic solvent and ultrasounds, among others,
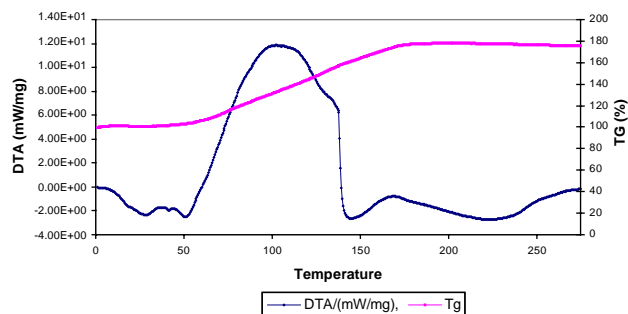

Fig. 1. Thermogravimetric analysis of a sample of B_4C (stoichiometric composition).

multicycle "compaction–sintering–milling" and thermal shock sintering. These procedures supply high activation energy to the aluminum.

To find the average temperature for sintering the carbibes, a series of thermogravimetric measurements were made (Fig. 1).

To minimize the heating time and to prevent the formation of Al_2O_3 and B_2O_3, a thermal shock was applied at 1373 K. At this temperature, the line of liquidus of the phase diagram is not reached. In these working conditions, no crystalline compounds are formed during cooling of the sintered materials.

The composition of the aluminum–boron mixtures covered a wide range, even up to 90% boron content. In the case of B_4C, the composition is stoichiometric.

The fuel sintered was mixed with an oxidizer plus a polymer, which together with the action of another metallic fuel, constitute the propellant in a composite form. Once the fuel was prepared, we proceeded to its combustion. The composite was subjected to trials of controlled combustion in a Crawfor bomb. The results were compared to those of a propellant with an identical composition but incorporating amorphous boron. Fig. 2 shown a list of the operations carried out in this work.

3. Results

3.1. Boride sintering

The mix oxides identified in Figs. 3 and 4 are $Al_{18}B_4O_{33}$ and $Mg_3B_2O_3$. The former was obtained in the preheating [20] of sintering:

$$2Al_2O_3 + 2B_2O_3 \rightarrow 2Al_2O_3B_2O_3,$$

$$9(2Al_2O_3B_2O_3) \rightarrow 2(9Al_2O_32B_2O_3) + 5B_2O_3.$$
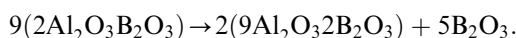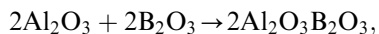
Ordinary crucibles and preheating produce oxides and borides in the sintering process as shown in Figs. 3 and 4.

Fig. 3 shows the aluminum oxides (columnar filament), and small mixed oxides scattered throughout the
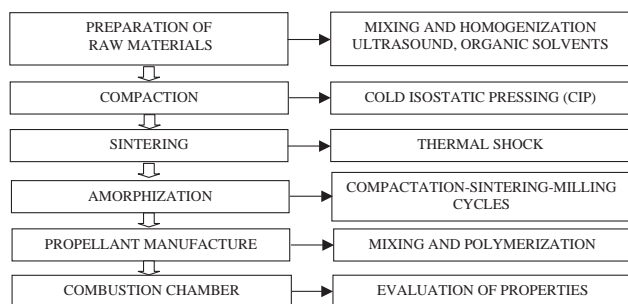
Fig. 2. List of operations carried out.

Fig. 4. Aluminum oxides, sintering at 1373 K for 30 min.

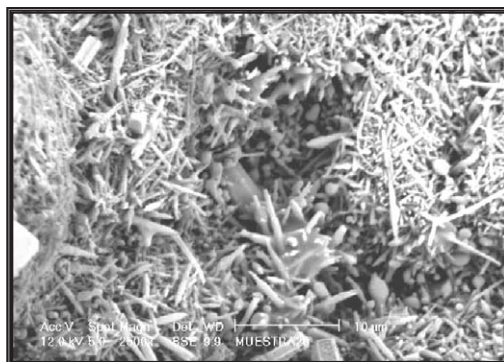Fig. 3. Aluminum, boron and mix oxides, sintering at 1373 K for 30 min.

Fig. 5. Aluminum borides in formation.

matrix (white ellipse). Fig. 4 shows aluminum oxides (black sphere) and mixed oxides (small white agglomerates) of different shapes and sizes. These were formed in oxidizing conditions of sintering, i.e., with inadequate sealing of the crucibles. In both the samples, the initial material was amorphous boron and atomized aluminum.

Crucibles specially designed for this work promote the formation of aluminum borides and boron carbides, free of oxides.

Boric acid and boron oxide [21,22] were used. Isostatic compaction prevented the formation of interconnected porosity.
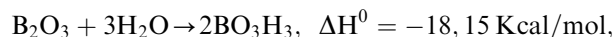
Sintering was done at temperatures favorable to the formation of $Al_4B_2O_9$ between 1073 and 1273 K [23]. This mixed oxide is decomposed at 1373 K, releasing B_2O_3 [23] and hindering the formation of borides since this B_2O_3 reacts with aluminum at any temperature above its melting point and produces $Al_{18}B_4O_{33}$ [24,25]. The only way to avoid the formation of $Al_4B_2O_9$ is to regulate the initial temperature [26,27].

We bypassed this problem by using thermal shocks that raise the temperature to 1673 K in a matter of seconds, with no preheating phase, and this avoids the formation of B_2O_3. This technique required a modification of the previously designed air-tight crucibles to make them resistant to the thermal shocks; it was used for the samples shown in Figs. 5–8.

Figs. 5 and 6 show the initial and final stages of the formation of borides of aluminum, sintered from boric oxide and atomized aluminum. Fig. 5 show us the initial stage of the formation of AlB_{10}. Fig. 6 shows the final stage of the formation of the boride. We can see a lot of aluminum boride completely formed. Few oxides are visible, apart from some small white spheres of aluminum oxide.

Figs. 7 and 8 illustrate the formation of borides of aluminum, again sintered from boric oxide and atomized aluminum. No oxide appears in the matrix which is composed entirely of mixtures of borides of aluminum AlB_{10} and AlB_{12}, mainly of the former.

The reactions of the two processes were

$$B_2O_3 + 3H_2O \rightarrow 2BO_3H_3, \quad \Delta H^0 = -18,15 \, \text{Kcal/mol},$$

$$B_2O_3 + 2Al \rightarrow Al_2O_3 + 2B, \quad \Delta G = -320 \, \text{KJ/mol} (1525 \, \text{K})$$

(see respectively Refs. [28] and [29]) In the conditions of sintering with thermal shock, no B and Al_2O_3 were produced in the second reaction. X-ray diffraction showed the sum of Al_2O_3 and AlB_{10}

The tests were done as follows:

1. Boron oxide was obtained from boric acid by stove heating for 120 min. At 673 K.
2. The boron oxide was ground to a particle size of 50 μm in a high-energy mill.

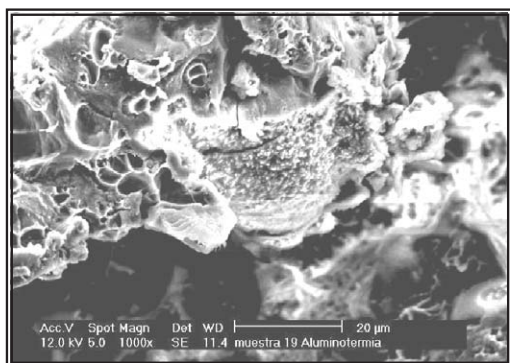Fig. 6. Aluminum borides already formed.
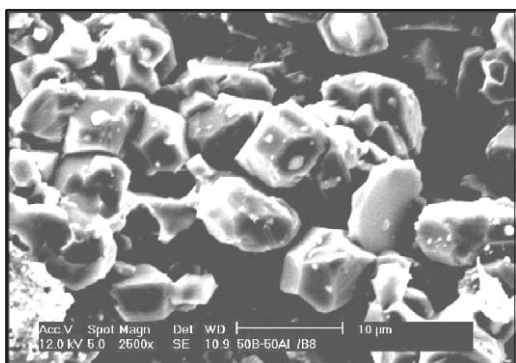

Fig. 7. Aluminum borides.

Fig. 8. Aluminum borides free of oxides.

3. The amounts of boron oxide and boric acid were those of the stoichiometric composition. The added aluminum was over 1–2% by weight.
4. The powders were mixed and homogenized mechanically in a low-speed mill. Steel balls were added to the powder mix in a ratio of 1/10 by weight.
5. The isostatic compaction of the mix consisted of several cycles of compaction–grinding–compaction.
6. After vitrification, the compact was sintered at 1373 K for 30 min.

Fig. 9 shows the diffraction diagrams of the samples of the 1–3 series of B–25Al% by weight. Sample 1 was

prepared by isostatic compaction with alternating cycles of compaction–sintering–milling in oxidizing atmosphere at 1373 K, which produce little oxidation. Samples 2 and 3 were prepared by isostatic compaction with only one cycle of compaction–sintering–milling. Sample 2 was sintered at 1693 K and sample 3 at 1713 K, both of them in nonoxidizing atmosphere which produced borides and mixed oxides.

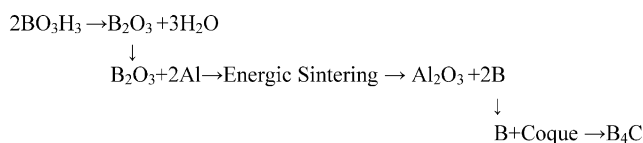The magnesium borides in samples 2 and 3 are from the traces of magnesium in the initial amorphous boron.

The diffraction patterns in Fig. 10 are those of two sintered samples made from B_2O_3 and Al by cold isostatic compaction (sample 4) at 1373 K for 45 min, which promoted the formation of oxide and vitreous B–Al. Sintering with gravity compaction at 1373 K for 30 min (sample 5) promoted the formation of aluminum borides.
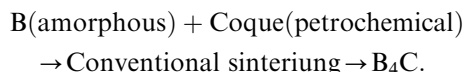
3.2. Sintering carbides

Sintering was done to obtain amorphous boron carbides using B_2O_3 and BO_3H_3 to contribute boron, and coke to contribute carbon. Both these products were milled to increase surface contact and reduce porosity for better sintering. The samples were compacted by energetic isostatic pressing (at pressures up to 375 MPa) with multiple cycles of compaction–milling–compaction.

The various routes towards obtaining B_4C are presented below:

Route 1:

$$2BO_3H_3 \rightarrow B_2O_3 + 3H_2O$$
$$\downarrow$$
$$B_2O_3 + 2Al \rightarrow \text{Energic Sintering} \rightarrow Al_2O_3 + 2B$$
$$\downarrow$$
$$B + \text{Coque} \rightarrow B_4C$$

Route 2:

$$B(\text{amorphous}) + \text{Coque(petrochemical)}$$
$$\rightarrow \text{Conventional sinteriung} \rightarrow B_4C.$$

Route 3:

$$BO_3H_3(\text{commercial}) + \text{Coque(petrochemical)}$$
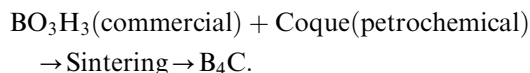$$\rightarrow \text{Sintering} \rightarrow B_4C.$$

Fig. 11 presents the diffractograms of samples variously produced with different carbon sources, compared with commercial boron carbide.

- Pattern no. 1 is that of a sintered sample of amorphous boron carbide. It was produced by Route 2 with an excess of the mesophase in its composition to provide carbon. Compacting was by impact.
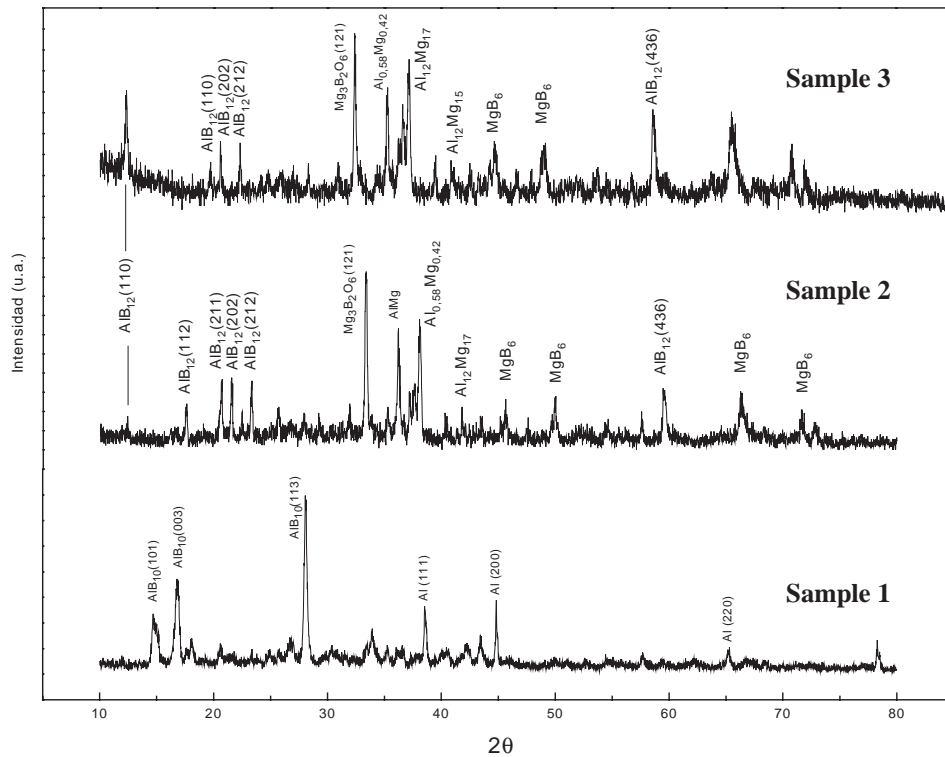
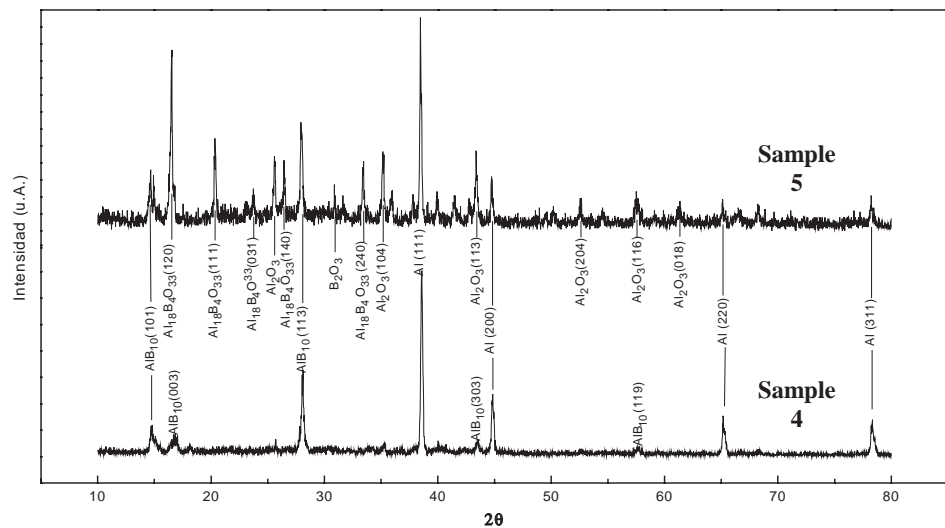Fig. 9. Diffraction patterns of the samples of series 1–3. B–25Al%.

Fig. 10. Diffraction patterns of samples 4 and 5. B_2O_3–Al at 1373 K for 45 min.

- Pattern no. 2 is that of a sample sintered from commercial boron carbide compacted by CIP.
- The B_4C (mesophase) pattern is that of an amorphous boron carbide produced by Route 2. The mesophase was used to contribute the carbon. Compaction by impact.
- The B_4C (coke) type is again an amorphous boron carbide produced by Route 2 but with coke as the source of carbon. Compacted by CIP.

Figs. 12–14 show us these carbides. All the matrices except the commercial one are to have amorphous structures free from oxides.

3.3. Propellant manufacture

Once the sintered fuel was obtained, it was mixed and homogenized. A second step, was to aid to hydroxyl-terminated polybutadiene (HTPB) the polymerization
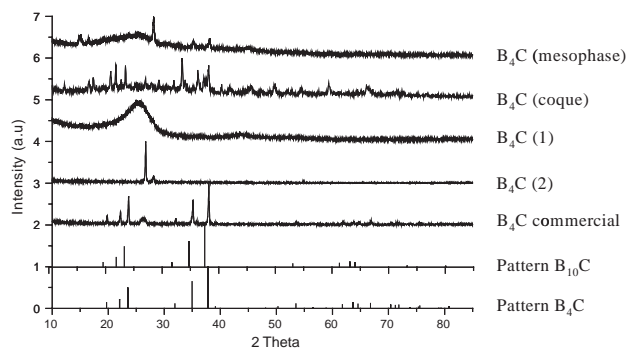
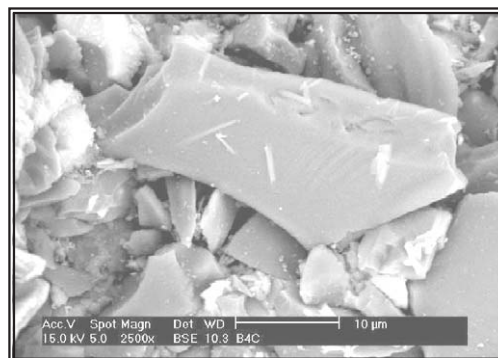Fig. 11. Diffraction patterns of the carbides produced.

Fig. 12. Commercial boron carbide (pattern 2).

Fig. 13. Stoichiometric boron carbide (route3).

catalyst to form the propellant. The raw material were as follows.

The binder composition involved 10% HTPB; 2% of a plasticizer (dioctyl adipate) and a small percentage of a bifunctional azidirine, which was used as a bond promoter. The rest of the propellant is ammonium perchlorate (60%) and the aluminum–boron sintered fuel (30%).

The cure reaction was between the hydroxyl groups of the prepolymer and the isocyanate groups of the curing agent (isophorone diisocyanate). This reaction was prepared by means of a vertical mixer and cured during 10 days at 60°C [30].
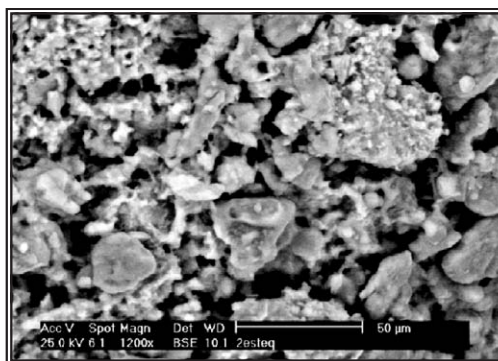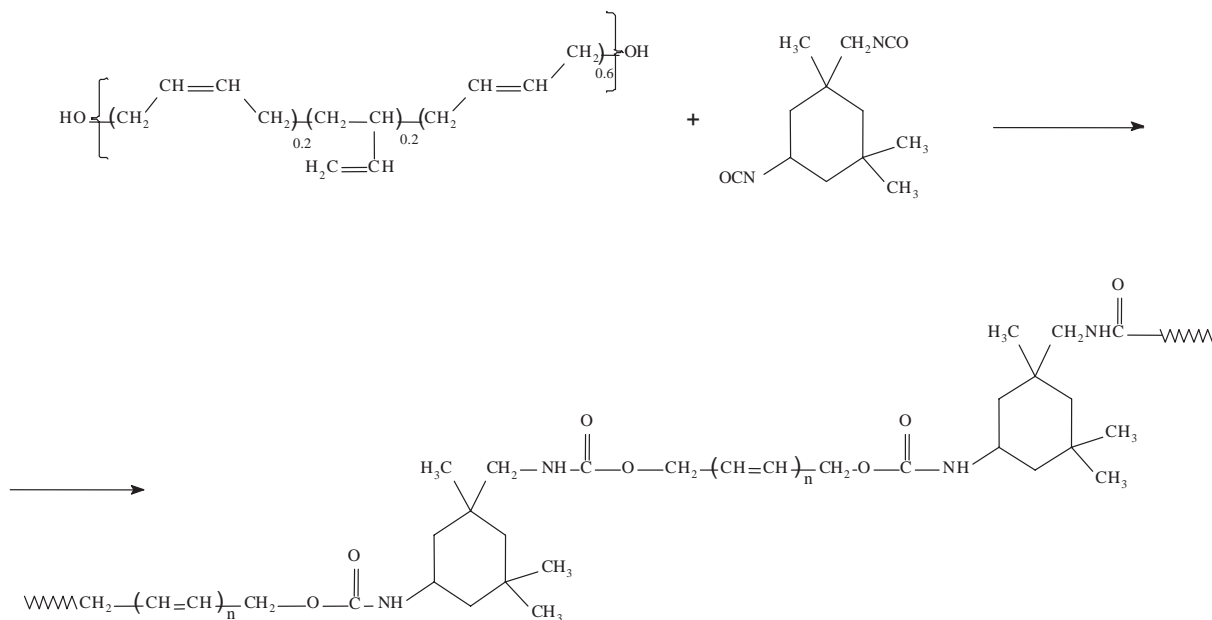

The segregation of hard (diisocyanate) and soft (polybutadiene) segments and the formation of domain structures for this type of polymer have to be taken into account [31].

The urethane groups show strong interactions among them, due to their polar behavior, and also due to the possible formation of hydrogen bonds among the NH hydrogen atoms and the carbonyl groups.

In order to enhance the affinity between the introduced inorganic charges and the elastomeric base; a coupling agent (aziridine) is also introduced. This type of additive, is highly reactive [32,33] especially with
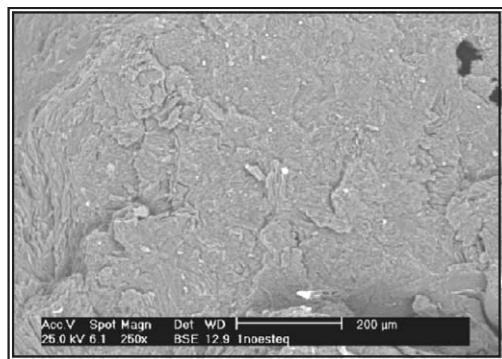
Fig. 14. Nonstoichiometric boron carbide (pattern 1).

hydroxyl groups from HTPB, giving as a result the aperture of the aziridinic ring.

This type of aziridines can also participate in secondary reactions such as transpositions which give as a result oxyazolynes and ammonium ion catalysed homopolymerizations [30]. Taking into account the above-mentioned information, it is deduced as a conclusion that the coupling agents produce an increase of interbreeding inside the propellant polymer bulk in the surroundings of the particles of ammonium perchlorate by reaction with OH prepolymer groups.

3.4. Combustion chamber results

The propellant burning composite generated solid ashes and agglomerates which come from boron–aluminum particles combustion. Agglomerating polymer combustion only produces gaseous mixes, such as CO and CO_2. The ammonium perchlorate produces N_2, H_2O, HCl and O_2:

$$2NH_4ClO_4 \rightarrow N_2 + 3H_2O + 2HCl + 2,5O_2.$$

Ammonium perchlorate undergoes a complex chemical reaction on heating, with decomposition occurring over a wide range, beginning near 200°C [34]. Decomposition occurs prior to melting, so a liquid state is not produced.

A boron particle has an oxide coat that thickens as the particle is heated in an oxidizing atmosphere. This condition is a characteristic of many metals, even those that will burn in the vapor phase. For the efficient combustion of the boron particle, the oxide coat must be removed. The practical means for removing the coat is to undertake the oxidation at temperatures greater than the saturation temperature of the boron oxide B_2O_3. This temperature is about 2300 K at 1 atm.

The control burning time of a boron particle is the heterogeneous oxidation of the clean particle after the oxide has been evaporated [35,36].

The efficient burning of the particle and oxidizing medium temperatures must be close to the saturation temperature of the B_2O_3.

Gaseous boron monoxide BO forms at the surface and this product is oxidized further to gaseous B_2O_3 by vapor-phase reactions. The gaseous B_2O_3 diffuses back to the clean boron surface and reacts to form three molecules of BO.

In a high-temperature atmosphere created by combustion of a polymer fuel with abundance of hydroxyl radicals (HTPB), boron monoxide reacts with hydroxyl radicals to form gaseous metaboric oxide HOBO:
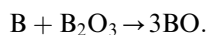
$$M + BO + OH \rightarrow HOBO + M.$$
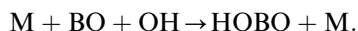
HOBO then reacts with BO to form gaseous boron oxide hydride HBO and boron dioxide BO_2:

$$OBOH + BO \rightarrow OBO + HBO.$$
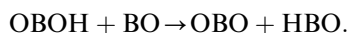
The boron dioxide then reacts with another BO to form boron oxide B_2O_3:

$$OBO + BO \rightarrow B_2O_3.$$

After the gaseous reaction system is established, the B_2O_3 diffuses back to the nascent boron surface to form BO:

$$B + B_2O_3 \rightarrow 3BO.$$

On the other hand, and regarding aluminum; the burning propellant produces an Al/Al_2O_3 agglomerate. The agglomerate size depends on many factors such as the original particle size, chamber pressure, burning rate and aluminum concentration. When chamber pressure increases, the agglomerates become smaller [37]. Only a small fraction of the aluminum in such large agglomerates has time to burn inside the chamber. When aluminum is used as a sintered boron–aluminum
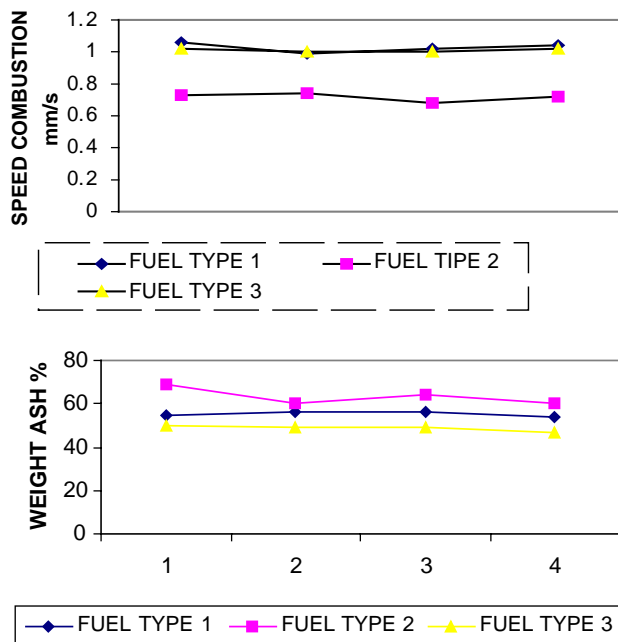

Fig. 15. Results of controlled combustion.

Table 3
Combustion chamber results for two series of propellants at 295 K and 27 kg/cm^2 pressure

Propellant	Fuel	Speed combustion (mm/s)	Ash (% weight)
Type 1	Series 1 (AlB$_{10}$ sintered)	1.06 ± 0.01	55
	Series 2 (AlB$_{10}$ sintered)	0.99 ± 0.01	55
	Series 3 (AlB$_{10}$ sintered)	1.02 ± 0.01	56
	Series 4 (AlB$_{10}$ sintered)	1.04 ± 0.01	54
Type 2	Series 5 (amorphous boron)	0.73 ± 0.01	69
	Series 6 (amorphous boron)	0.74 ± 0.01	60
	Series 7 (amorphous boron)	0.68 ± 0.01	64
	Series 8 (amorphous boron)	0.72 ± 0.01	60
Type 3	Series 9 (boron carbide)	1.02 ± 0.01	50
	Series 10 (boron carbide)	1.00 ± 0.01	49
	Series 11 (boron carbide)	1.00 ± 0.01	49
	Series 12 (boron carbide)	1.01 ± 0.01	47

combustible, the amount of nonburnt aluminum that is recovered from the chamber is certainly reduced, due to the smaller amount of agglomerates that are produced.

Regarding the boron carbides, it is necessary to note, that no agglomerates are produced, so the combustion appears to be complete.

In order to test the combustion process, three series of the test tubes were made. The first one with amorphous boron as fuel, the second with aluminum boride as fuel and the third using boron carbide as fuel. The results of the controlled combustions (see Fig 15 and Table 3) are as follows:

1. The combustion speed of the propellant which incorporate aluminum boride or boron carbide sintered as a fuel is 40% above that with amorphous boron as fuel.
2. The weight of the ash from the combustion is 20% lower in the propellant that contains sintered aluminum boride than in the composite with amorphous boron.

The propellant with boron carbide as a fuel produced less ash than the other types.

4. Conclusions

1. A high-energy propellant for motor rockets was developed, of boron plus a chemical reductant.
2. The reducing elements were incorporated by powder technology. Suitable working conditions were designed to obtain sintered aluminum borides and boron carbides, amorphous, free of oxide.
3. Successive cycles of compaction–sintering–milling and thermal shock were found suitable for boride and carbide sintering and for vitreous boron–aluminum.

4. The preheating in the sintering can be avoided by the use of special crucibles developed for this work. These crucibles were chosen because they supply all nonequilibrium conditions.
5. Boron acid and boron oxide were used as raw material for boron. In isostatic and gravity compaction, the results were as good as those of amorphous boron sintering.
6. The combustion speed of the propellant with sintered aluminum boride and boron carbide was 40% above that with amorphous boron.
7. The weight of the ash obtained in the combustion is 20% lower in the propellant that contains sintered boron or boron carbide than in that prepared with amorphous boron.
8. Sintermetallothermic and reactive sintering were the procedures used for preparing the borides and boron carbides.

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