Combustion Efficiencies of Aluminum and Boron in Solid Propellants

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A closed-type strand bomb was used to trap all species participating in solid propellant combustion to measure combustion efficiencies of aluminum (Al) and boron (B) using titration techniques. Variations of combustion efficiencies (η) of Al and B with pressure and oxidizers of propellants were investigated. In order to improve B combustion, effects of the addition of Al, granulated propellants, and oxidizers were also investigated. It was found that burning of B particles could be enhanced by using fine powders. Boron particles granulated with potassium nitrate had the highest η_B (40%) in the strand burner. Burning of Al was found to be hindered by B burning when Al and B particles coexisted. These observations could be interpreted by considering the agglomeration and ignition processes of metal particles.

Nomenclature

Al	= aluminum
AN	= ammonium nitrate (NH_4NO_3)
AP	= ammonium perchlorate (NH_4ClO_4)
B	= boron
CTPB	= carboxyl terminated polybutadiene
C^*	= characteristic exhaust velocity, m/s
HTPB	= hydroxyl terminated polybutadiene
KN	= potassium nitrate (KNO_3)
r_b	= regression rate of propellant, mm/s
η	= combustion efficiency

Introduction

METALS are of interest as fuel ingredients because of their high densities and high heats of combustion. Aluminum (Al) is used extensively as a component of solid propellants. Boron (B) has a higher heat of combustion than Al, but it requires a larger amount of oxidizer as compared with Al. Therefore, B is the most promising candidate for air-breathing propulsion in the future. Metals burn in a very different manner than conventional fuels. The unique combustion behavior of metals is due to the physical properties of the metals and their oxides. Comprehensive texts and reviews[1-3] on metal combustion, especially Al combustion[4,5] and B combustion,[6,7] have been published.

High-metal loading is required to achieve useful performance gain in propulsion. This high-particle loading results in particle agglomeration and coalescence, and requires considerable time for completion of burning. A program by Coats et al.[8] for evaluating the performance of solid-propellant rockets embodies an empirical model for Al agglomerates developed by Hermsen.[9] Recently, the performance of aluminized propellants containing a nitramine (HMX) was shown to be related to agglomerate size on the burning surface of the propellants.[10] Studies on the agglomeration process are still being conducted experimentally and theoretically for Al.[11-26] There have been many studies on the burning of single B particles[27-34] (see Ref. 5 for Al). However, there have been few studies on the combustion of B under conditions of high-metal loading. The problem of agglomeration in the combustion of slurry droplets containing B,[35-37,49] as well as in solid propellant combustion, should be investigated.

The combustion efficiency of Al in propellants has been investigated by measuring the amount of unburnt Al in residue collected from plumes. The amount of Al was measured by titration methods using ethylene diamine tetraacetate (EDTA) with xylenol orange[4,19] or Cu(II) α-pyridyl-β-axonaphthol (Cu-Pan)[38] as the indicators. This method has been used to check the completeness of combustion of Al in solid propellants under an acceleration environment.[39] Recently, this chemical titration method was adopted to screen appropriate formulations of propellants containing HMX prior to the development of motors.[40] Effects of two oxidizers, ammonium perchlorate (AP) and ammonium nitrate (AN), on the combustion efficiency of Al were investigated in the present study.

This technique has been extended to study B combustion, and a series of experiments have been conducted to examine the feasibility of enhancing the burning of B particles. The theoretical advantages of B as a fuel can be, of course, only attained with efficient B combustion in a chamber. However, this is extremely difficult since B has an extremely high boiling point (ca. 3930 K). Therefore, ignition of B particles by Al and enhanced burning of B/AP granulated propellants were examined. Effects of potassium nitrate (KNO_3, KN) were also investigated in order to improve the combustion efficiency of ducted rockets. Firing tests using subscale rocket motors were also performed, and the results were compared with those of the strand experiments.

Experimental Method

The closed strand bomb used to trap all condensed and gaseous species participating in solid propellant combustion is shown in Fig. 1. This apparatus consists of a combustion chamber and a surge tank. The combustion chamber and the tank are separated by a free piston. The burning pressure could be maintained at a constant level during burning of the propellant strand. A propellant strand (8 mm in diameter and 20 mm in length) was set upside down in a combustion cell that was submerged in water. The inside of the cell was purged with He and the strand was burnt in He in the combustion cell. The downwardly directed plume impinged on the water, and condensed phase components were retained in the water of this closed bomb. The distance between the propellant surface and the water surface (quench distance) was typically 45 mm (corresponding to a residence time of about 5 ms).

The trapped Al agglomerates were crushed using a high-speed disperser, and unoxidized Al in the agglomerates was then dissolved in boiling hydrogen chloride. The amount of Al

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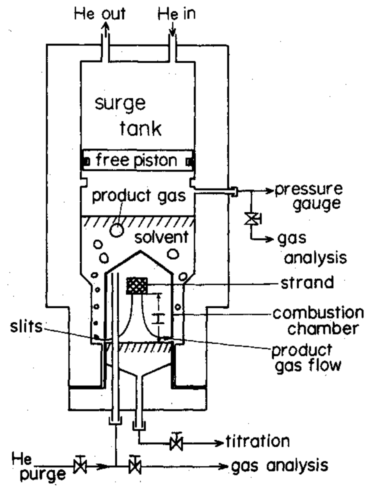

Fig. 1 Experimental apparatus to trap all reactants and products.

was measured by titration using EDTA and Cu-Pan.[38] The detectable limit of Al under these experimental conditions was found to be 0.7 mg, quite small compared with the amount of Al contained in the propellants.

Because the burning rate of B particles is low compared with that of Al, it is more accurate to measure small amounts of combustion products rather than the reactant itself. The major products, boron oxide (B_2O_3) and boric acid (H_3BO_3), were measured using acidimetry with mannitol ($C_6H_{14}O_6$). Effects of CO_2 and other species disturbing the titration were examined. The resolution limit of B compounds was 0.5 mg B in this experiment. The same chemical analysis has been applied for the measurement of combustion efficiency of B in ramjets by Pein and Vinnemeier.[41] The size distribution of B particles in the plumes was also analyzed using a Coulter counter.

Propellants examined in the study are summarized in Table 1. In order to accelerate metal burning, nitro-binder propellants using PEG/BTTN binder were selected (propellants A and B), PEG and BTTN standing for polyethylene glycol and butanetriol trinitrate ($C_4H_7N_3O_9$), respectively. Effects of ammonium nitrate (NH_4NO_3, AN) were investigated using propellants B1, B2, and B3. AP-based propellants C and KN-based propellants D were used to evaluate the combustion efficiency of B in the primary chamber of ducted rockets.

In order to increase the contact surface of B particles with the oxidizer, granulated propellants were formulated. Even-grained granules (approximately 1 mm in diameter) made of mixtures of B/AP or KN (ratio of B/oxidizer = 30/70) powder were prepared as an ingredient for propellants. Then AP (or KN), B, and CTPB binder were mixed with these granules. Propellants C and D have similar compositions (CTPB/oxidizer/B ratio = 30/40/30).

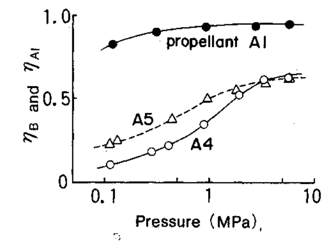

Fig. 2 Combustion efficiencies of Al (propellant A1) and B (propellants A4 and A5).

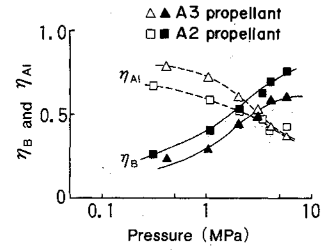

Fig. 3 Variations of combustion efficiencies of Al and B when Al and B particles coexist in propellants.

Experimental Results

Al and B Combustion in Nitro-Binder Propellants

Figure 2 is an illustration of η_{Al} and η_B when only one of these two metals is included in the propellant. The mass fraction of Al is about 20% in usual solid propellants. A preliminary study using this apparatus showed that the η_{Al} varied from 60 to 80% with r_b and burning pressure. Because the content of Al in propellant A1 is low (10%), the value of η_{Al} is much higher than those in usual propellants. The figure shows that 95% of Al burns in the quenching distance of 45 mm at a pressure of 5 MPa. The combustion efficiency of B is lower than η_{Al} and varies from 10 to 60% (A4) as the burning pressure increases. The replacement of crystal B ($\bar{d}$ = 6.8 μm) with an amorphous B ($\bar{d}$ = 1.3 μm) improved the burning characteristics at lower pressures. Because of the high reactivity of amorphous B with the nitro-binder, the mass fraction of amorphous B is limited up to 3% in propellant A5. This small amount of fine B may cause the limitation of improved η_B under high pressure.

A question that arises is whether the ignition of B particles is promoted by Al combustion. The variations of η_{Al} and η_B, when propellants contain both Al and B, are shown for propellants A2 and A3 in Fig. 3. The open symbols indicate η_{Al} and the solid ones η_B. The results show that the replacement of B with Al increases η_B. However, a comparison between A1, A2, and A3 propellants indicates that η_{Al} decreases with increasing η_B. For instance, the value of η_{Al} decreases from 90% (A1) to 70% (A2) and to 55% (A3) under a pressure of 1 MPa.

Table 1 Propellants examined in this study

No.	Binder, %	Oxidizer, %	B, %	Al, %	Equilibrium temperature, K, 5 MPa	Propellant type
A1	Nitro 20	AP[a] 70	0	10[b]	3366	Conventional type
A2	Nitro 20	AP[a] 70	3.5[c]	6.5	3387	Conventional type
A3	Nitro 20	AP[a] 70	6.5	3.5	3372	Conventional type
A4	Nitro 20	AP[a] 70	10.0	0	3311	Conventional type
A5	Nitro 20	AP[a] 70	7[c] + 3[d]	0	3311	Conventional type
B1	Nitro 27	AN[e] 70	0	3[b]	2567	Conventional type
B2	Nitro 26.2	AN[e] 67.8	0	6	2715	Conventional type
B3	Nitro 25.3	AN[e] 64.7	0	10	2905	Conventional type
C1	CTPB 30	AP[a] 40	30[c]	0	2124	Conventional type
C2	CTPB 30	AP[f] 40	30[d]	0	2124	Conventional type
C3	CTPB 30	AP[f] 40	30[c]	0	2124	Granulated propellant
C4	CTPB 30	AP[f] 40	30[d]	0	2124	Granulated propellant
D1	CTPB 30	KN[g] 40	30[d]	0	1886	Conventional type
D2	CTPB 30	KN[g] 40	30[d]	0	1914	Granulated propellant

[a]Trimodal AP (coarse 400 μm, medium 200 μm, and fine 20 μm). [b]Mass-average diameter ($\bar{d}$) = 6.5 μm. [c]$\bar{d}$ = 6.8 μm. [d]$\bar{d}$ = 1.3 μm (amorphous). [e]Monomodal AN($\bar{d}$ = 120 μm). [f]Monomodal AP($\bar{d}$ = 20 μm) for granules. [g]Monomodal KN($\bar{d}$ = 40 μm) for granules.

This tendency becomes evident at pressures greater than 1 MPa (Fig. 3). These results imply that the coexistence with B hinders the Al burning in these propellants.

The variation of particle-size distribution was measured for boron residue. The volume median diameter of boron agglomerates increased from 28 μm to 36 μm as pressure increased from 0.1 to 1 MPa.

C^* Efficiency of B/Al Metalized Propellants

The C^* efficiency of metalized propellants can be calculated for incomplete burning of metal fuels using a chemical equilibrium code, providing that complete burning has been assumed for hydrocarbon fuels (see Discussion). The variation of C^* efficiency with respect to B/Al ratio was obtained using the results shown in Figs. 2 and 3, and is illustrated in Fig. 4. Results in Fig. 4 show a downwardly convex variation of η_{C^*} due to Al burning being retarded by B.

The C^* efficiency evaluated using a standard motor having a diameter of 80 mm and a length of 140 mm is also illustrated in Fig. 4. In firing tests of propellants containing large quantities of B, melted B adhered to the nozzle throats and changed the throat area during firing tests. The large error bars for high B/Al ratio in Fig. 4 were caused by the difficulty encountered in estimating exact nozzle areas for calculation of C^*. The variation given by the upwardly convex curve indicates the improvement of B combustion with the addition of Al in the motor experiments.

Al Combustion in AN-propellants

As shown in Fig. 5, the replacement of AP with AN lowered the burning rate of Al. The peculiar behavior of η_{Al} with regard to pressure and the contents of Al will be discussed later, based on the ignition and agglomeration processes on the combustion surface.

B Combustion in Ducted Rocket Propellants

Effects of B particle size can be seen in C1 and C2 propellants (conventional type propellants) or in C3 and C4 propellants (granulated propellants) in Fig. 6. Augmented burning rates of B in granulated propellants were distinguished for a coarse B powder (C1 vs C3) and a fine powder (C2 vs C4) in AP-based propellants. Such enhanced burning due to granulation was also observed in KN-based propellants using a fine B powder (D1 vs D2). Effects of replacement of AP by KN are revealed by comparing C2 and D1 (conventional-type propellants) or C4 and D2 (granulated propellants), respectively.

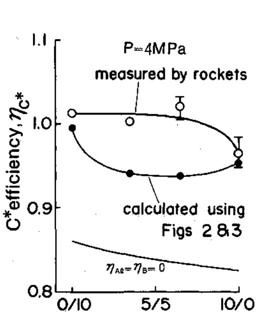

Fig. 4 Comparison of C^* efficiency calculated from Figs. 2 and 3 and C^* efficiency measured using a subscale rocket motor.

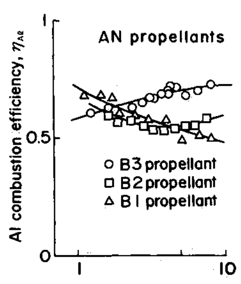

Fig. 5 Variations of combustion efficiency of Al with the contents of Al in AN-based propellants.

Discussion

In this study, the quenching distance was adjusted from 5 to 150 mm to control particle burning time. Results showed a negligible dependence of η_{Al} and a weak dependence of η_B on the quenching distance. These findings imply that the combustion of metals is very rapid during the first few milliseconds and then drops suddenly. Similar results have been reported by Price et al.[4] This protracted burning seems to be due to freezing of reactions on particles due to their mixing with the ambient gas (He or N_2). It can be concluded that this experimental technique is useful in the study of phenomena taking place just above the propellant surface.

Burning of metal particles in solid propellants may be characterized by three processes: agglomeration, ignition, and steady burning of agglomerates. The agglomeration process has been investigated experimentally. As a rule, the degree of agglomeration falls with decreasing metal content or increasing burning rates of propellants. Many experiments have shown that the size of agglomerates decreases as burning pressure increases.[11-17] However, more complicated pressure dependencies have been also reported.[18,21] Dependence of agglomeration on various parameters have been reported.[14-17,20,22]

Several models have been proposed to explain this agglomeration process.[23-25] Grigor'ev et al.[23] proposed a "pocket model" of the accumulation process of metal particles. In that model, agglomerates form from the clusters of particles residing within the pockets surrounded by oxidizer grains. Effects of size distributions of Al and oxidizer and effects of concentration and pressure have been included in a modified model.[24] Sambamurthi et al.[19] indicated the importance of a kinetically limited leading-edge flame (KLLEF) formed between oxidizer and binder for ignition/agglomeration. Kovalev et al.[25] developed a system of differential equations for temperature, diameter, and oxide-film thickness of an agglomerate in an elementary cell (pocket), and postulated ignition to be characterized by thermal runaway of agglomerate. They also found gas temperature and volume content of metal to be important factors. Their model was extended to account for gas temperature distribution near agglomerates.[26]

The experimental results shown in Fig. 2 can be interpreted using these models. The high-combustion efficiency of Al in propellant A1 can be understood as being due to the low Al content and the additional oxidizer in the nitro-binder. The burning rate of the propellant is expressed by $r_b = 6.32P^{0.58}$, where pressure is expressed in units of MPa. The burning rate is high compared with conventional composite propellants using HTPB binder. These factors favor a decrease in agglomerate sizes because of the thin reactive layer trapping Al particles and the promotion of ignition of agglomerates. The

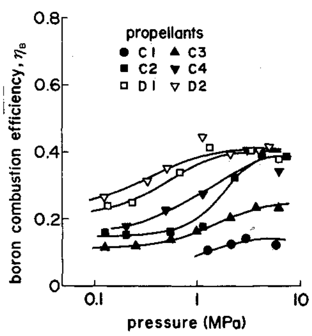

Fig. 6 Effects of particle size of B and granulation of B with oxidizers on the combustion efficiency of B in AP- and KN-based propellants.

exothermic reaction with nitro-binder results in the gaseous flame being closer to the burning surface and in the promotion of ignition. A similar situation can be realized in a less-fuel-rich propellant attained by increasing the content of fine oxidizer in the propellant.[19] Replacement with fine amorphous boron promotes the ignition and the burning of B particles (Fig. 1).

Next, the effects of the addition of Al on the burning of B particles are considered. Although propellant A1 contains sufficient oxidizer to burn the binder and Al, the oxidizer may be consumed by the burning of Al and B in propellants A2, A3, and A4, because boron requires a large amount of oxygen. Therefore, the environment of burning agglomerates was examined using the modified equilibrium calculation discussed below.

In the experiments, hydrogen and hydrocarbons were found from gas analysis to be negligible in the strand experiment. Concentrations of O_2 and CO were found to be low, and a relatively large quantity of CO_2 compared with that expected from chemical equilibrium was detected. These facts indicate that hydrocarbon binder rapidly reacts with oxidizer to produce CO_2 and H_2O just above the burning surface. This enables the evaluation of ***equilibrium*** properties of propellants for the case of *incomplete* combustion of metals.

The temperatures and concentrations of species were calculated for a given η_{metal}, and the distributions of temperature and species above the burning surface of propellants could be read by replacing η_{metal} with the streamwise distance from the surface. The temperature and concentrations of species calculated in this manner differ from the local properties in the KLLEF[19] or the distributions discussed for the microflames.[26] However, these mean properties must be important parameters when considering the ignition of particles above the burning surface. Figure 7 illustrates variations of burning temperature and major species with η_B in propellant C1. The temperature increases from 1179 to 2124 K with η_B as the mole fractions of reactants, i.e., boron, H_2O, and CO_2 decrease. Our calculations showed that oxygen was completely consumed in the propellant, even if boron was not allowed to react at all. Calculations for propellants A2 and A3 show that the mole fraction of oxygen is less than 6% compared with the mole fractions of other oxidizers H_2O (ca. 35%) and CO_2 (ca. 15%), with η_{Al} as a variable and $\eta_B = 0.5$ as a constant. Thus, metal burning has to be initiated in a hostile environment with low oxygen concentrations and temperatures.

Ignition delay results in protracted accumulation and larger agglomerates. The size distributions of agglomerates may be useful when considering the agglomeration and the ignition of B occurring on the burning surface. Mass-averaged diameters of quenched B particles were found to increase from 6.8 μm to approximately 30 μm, and increased slightly with the burning pressure. Microscopic studies indicated that boron residue with original irregular shapes under low pressure was replaced with large spherical boron particles under high pressure. This observation agrees with the measurement of η_B in Figs. 2 and 3. These results show that boron particles may agglomerate in spite of its high melting point (2200 K) and that boron oxide (m.p. 770 K) may play a decisive role as the binder. However, the agglomeration rate of boron particles is low compared with that of Al. The increase of agglomerate size with pressure and the high melting point of boron suggest the possibility of agglomeration of B taking place in the gas phase.

The retarded burning of aluminum caused by boron shown in Fig. 3 can be explained by assuming the competition of ignition between large Al agglomerates and small B agglomerates. The ignition temperature of B particles has been reported to be about 1900 K for single particles[27,28,7] and 1200 K for agglomerates.[35] Because of this low-ignition temperature, in addition to the low agglomeration rate, the ignition of B particles precedes that of Al when Al and B particles coexist. Burning of fine B particles consumes oxidizer, resulting in an increase of the ignition lag of Al agglomerates. This is the reason why the combustion efficiency of Al decreases as B particles burn.

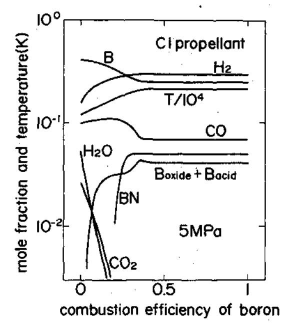

Fig. 7 Variations of temperature and concentrations of major species with the combustion efficiency of boron.

The addition of magnesium or magnesium/aluminum alloy has been reported to enhance the burning of B.[42,43,45] This result, which differs from the effects of Al of the present study, can be rationalized, considering that the ignition temperature of the additives is lower than that of B. This indicates the importance of the relative values of ignition temperatures, as well as of the agglomeration of additive materials, if two kinds of metals participate in propellant combustion.

This retarding effect of B on Al burning is dominant near the burning surface of propellants and decays with increased residence time of particles in motors, because the burning rate of Al is greater than that of B and the handicap in the ignition is overcome as Al particles travel away from the burning surface. Incomplete burning of metal fuels in propellants could be detected by firing tests using subscale motors (Fig. 4). However, definitive analysis with regard to various parameters, such as particle size, was difficult, because precise measurements of the throat area or nozzle efficiency are impossible in subscale motors.

The low combustion efficiency of Al was anticipated from the low regression rates of the AN propellants (typically 3 mm/s at 4 MPa). Since the pressure exponents of the AN-propellants are close to unity, the flame structure is strongly dependent on the burning pressure. High pressure will cause a thinner reaction layer in the condensed phase[24] and result in the KLLEF being closer to the burning surface.[19] This would be a more favorable condition for less accumulation and fast ignition of particles. The experimental results for AN-propellants are, however, difficult to understand, especially those for propellants B1 and B2. Many data points indicate the peculiar turnover of pressure dependence of η_{Al} with respect to the Al content. The equilibrium calculation showed no essential differences between AP-propellants and AN-propellants in the burning environment of Al particles.

A clue to understanding these experimental results is the agglomeration process with a large standoff distance of flames. Kovalev[26] showed that the degree of agglomeration is governed by the temperature distribution above the surface and that the formation of large agglomerates may be possible in propellants with low metal content and large flame standoff distance. The modified equilibrium calculations show that the flame temperature of B1 propellant changes from 2350 K ($\eta_{Al} = 0\%$) to 2567 K (100%), and that the flame temperature of B3 propellant varies from 2200 to 2905 K with increasing η_{Al}. The peculiar minimum in propellant B2 and the increase of η_{Al} with pressure in B3 propellant, as seen in Fig. 5, seem to be due to the competition between the agglomeration and the ignition of Al particles. Environmental temperatures close to the ignition temperature of Al, the high pressure exponents, and the thick melting layer of AN propellants emphasize these two competing processes that govern Al burning.

As seen in Fig. 6, increasing the contact surface of particles, especially with oxidizers, was found to be the most efficient way to improve the combustion of B particles. The granulation of metals and oxidizers is also a useful technique when amorphous powders and binders, such as the nitro-binder, are too reactive to be mixed with each other. For propellants for ducted rockets, the amounts of B that can react in the primary chamber are limited because of the lack of oxidizers. The metal burning is terminated when all the H_2O and CO_2 react with metals to yield H_2 and CO. For instance, the maximum value of η_B in propellant C1 is evaluated to be 0.375 from Fig. 7. The maximum value of η_B is approximately 40% at 4 MPa for propellants D. Therefore, the results in Fig. 6 show that combustion of B in this experiment is completed at pressures higher than 3 MPa.

Schadow[44] doubted the satisfactory B ignition in the primary chamber burning an H_2-O_2-B mixture. The experimental results showing the strong influence of primary chamber temperature on η_B were attributed to a quick ignition of B particles in the secondary chamber. The present strand experiment indicates, however, that burning of B is relatively easy in the primary chambers of ducted rockets if fine B powders or granulated propellants are employed. Pein and Vinnemeier[41] investigated B combustion using a ramjet test facility. HTPB fuels containing amorphous B (typical size of 0.9-1 μm) of varying compositions up to 40% were applied and a fairly high-combustion efficiency of B (30–60%) was reported. It is speculated that a small addition of oxidizer may have improved the combustion of B in their experiment. However, difficulty in burning B particles with air in the secondary chamber has been reported by Mituno et al.[45]

The improvement in η_B due to KN becomes evident at low pressures (Fig. 6), which indicates that KN accelerates the ignition of B. Kuwahara and Kubota[46] revealed that the B particles react with the decomposition gases of AP on and just above the propellant burning surface. On the contrary, an exothermic reaction in the condensed phase is reported between B and KN.[47] Childs et al.[42] suggested that coating the B particles with lithium fluoride might be useful for removing boron oxide by formation of a low-boiling complex. Smoldering combustion of residual B in the atmosphere was only observed for KN-based propellants. These observations imply that introduction of potassium may result in the enhanced chemical removal of the oxide layer coating individual B particles.[43] It was also shown that the removal of the oxide layer can be enhanced by the presence of water vapor in the ambient gases.[27,30,48] A comparison of AP- and KN-based propellants indicates that the enhanced removal rate of KN must be much greater than the case of water vapor.

Conclusions

1) The simple strand burner method in conjunction with chemical titration was found to be useful for investigation of the burning behavior of Al and B. It enables screening of propellants without firing tests of motors and investigation of important parameters controlling burning of metal fuels.

2) Experiments using subscale motors indicated that B burning was enhanced by the introduction of Al. However, the strand experiments showed that burning of Al was hindered by combustion of B. This phenomenon can be explained as being due to the high ignition temperature and the high agglomeration rate of Al particles compared with those of B particles.

3) A maximum value of η_B of about 40% (at 5MPa) was obtained in the strand experiment, where a typical residence time of B particles was 3 to 10 ms. Equilibrium calculations and gas analysis indicate that further burning of B is impossible in primary chambers because of limited oxidizer.

4) Effects of B particle size and embedded B in oxidizers could be measured by AP- and KN-based propellants. Fine B powders were found to improve η_B, and enhanced burning of B in granulated propellants was observed at lower pressures. The results for KN-based propellants suggest the possibility that potassium can accelerate the evaporation of the oxide layer.

5) This experimental study shows that there are many research requirements associated with combustion of aluminum and boron particles in solid propellants. The dependence of critical conditions for ignition and ignition delay times on pressure, temperature, and gas composition should be carried out for the case of Al/B coexistence. Further investigation of AN oxidizer is needed.

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