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Particulate Matter in the Exhaust of a Boron-Loaded Solid Propellant

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Particles issuing from the primary solid-propellant combustor of a typical boron-loaded air-augmented rocket motor have been collected and analyzed for chemical composition and size distribution. It was found that substantial changes can occur in the chemical composition of the particulate effluent (notably, ammonium pentaborate and carbon were found), but the size analyses suggest that no important change in the size distribution of the remaining boron particles occurs. However, this last conclusion is subject to the uncertainties involved in any particle collection technique's influence on the size distribution results.

I. Introduction

BASIC engineering design techniques for air-augmented rockets have come under study in the last few years as a result of certain propulsion applications; in such devices a fuel-rich primary solid-propellant rocket motor is exhausted into an after-burner, or secondary combustor, where the exhaust is burned with air which is obtained from the ambient atmosphere. One of the critical design parameters is the volume or length of this secondary combustor necessary for complete combustion of the primary exhaust, and both analytical and experimental studies have been directed toward obtaining estimates of this parameter as a function of the overall engine operating parameters.^{1,2}

Since the addition of boron (B) particles to the primary solid propellant has been used extensively to maintain its exhaust fuel-rich, and since these solid or liquid particles are the major combustible constituent of the primary exhaust, any analytical treatments of the secondary combustor are extremely complicated and must consider simultaneous turbulent mixing and combustion of the gas-particle flow. The conditions at the primary rocket exit serve as boundary conditions for analysis, and in this context it is of interest to characterize the state of the particles in the exit plane. In particular it is desired to know of any velocity or temperature lag of the particles, and their size and spatial distributions in the plume.

In general, because such information is not available, it has been assumed that initially the temperature and velocity of the particles equal the local values in the gas and that a unimodal size of particles is distributed uniformly over the plume.² Also, the chemical composition of the particles is postulated not to have changed during their residence time in the primary motor, that is, the B is assumed to remain chemically inert until it is exposed to air in the secondary combustor.

In this paper results dealing with the size distribution and chemical composition of these particles are presented. A typical B-loaded solid-propellant primary rocket was fired into a particle collection chamber, and the resulting samples were analyzed chemically by x-ray diffraction; size distributions were obtained both by electron micrography and photo-sedimentometry. In addition, optical methods which could lead to in situ determinations of particle size and spatial distribution in the primary exhaust have been discussed elsewhere.³ Such methods are of interest because size measurements similar to those described here, due to unknowns such as effects of the sample collection and measurement techniques, cannot yield noncontroversial conclusions. Primarily because of the high optical depths involved in the primary motor plume, however, it was concluded that such experimental techniques would be unsuccessful.³

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II. Experimental Apparatus

The basic design of the primary combustion chamber and the specifications of the boron-loaded propellant have been detailed elsewhere.⁴ The combustor was of approximately 19 in.³ volume when unloaded, and the propellant was mounted so as to burn in cigarette fashion. The only significant change

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Index categories: Properties of Fuels and Propellants; Subsonic and Supersonic Airbreathing Propulsion.

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Table 1 Sample nomenclature for size distribution and chemical analysis

Sample number	Firing	Source
1	...	As-received boron
2	1	Cyclone separator
3	1	Filters
4	...	As-received boron
5	2	Cyclone separator
6	2	Filters

in the combustor for this particular portion of the study was that a single converging nozzle replaced the dual impinging nozzles used in other studies⁴; steady-state primary chamber pressures during the firings were measured to be about 250 psia.

The nozzle exhausted into a mixing insert in which cold N₂ was added to the exhaust stream to quench any chemical reactions and to cool the particles before collection. The resulting stream was directed into a large cyclone separator and subsequently filtered (through two Pall Corporation MDF 1000B6 sintered stainless steel bayonet filters mounted in parallel) for removal of the majority of the particulate matter. The filters were used to collect the fine particles which could not be trapped in the cyclone separator. A schematic diagram of the test apparatus is shown in Fig. 1; the cyclone separator consisted of an 11 ft pipe of 4 in. inside diameter with a 0.75-in. exhaust tube leading to the filters. The volume of the separator was approximately 90 times that of the unloaded primary combustion chamber.

After each firing, the cyclone separator was carefully opened and emptied into a large plastic bag; the residue collected in the filters was placed in a separate bag. The samples thus collected in each bag were coned and quartered⁵ in order to obtain smaller amounts more convenient for size distribution and chemical analyses. These final samples will be referred to by number as defined in Table 1.

Two primary rocket firings were conducted; in the next section the results of the analysis are presented and discussed.

III. Results and Discussion

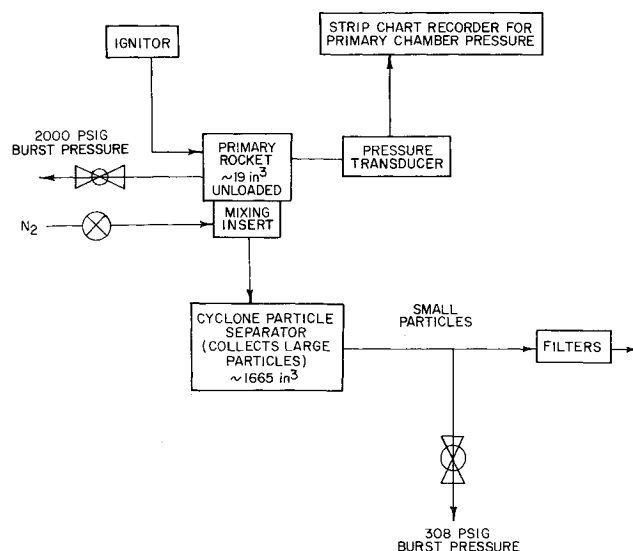
A. Chemical Analyses

X-ray diffraction analyses of Samples 1-6 were conducted by Sloan Research Industries of Santa Barbara, Calif. Calibration standards were prepared in 1% increments of B₂O₃ ranging from 2% to 10% with the remainder leached Kawecki boron for samples 1 and 4. For the other samples standards containing 20-60% B in 10% increments, with the balance amorphous C and NH₄Cl, were used. To insure consistency, an internal standard of 3.13% Mo was added to each calibration standard. Leaching was used to estimate the water soluble component concentrations; note that this last percentage will include B₂O₃.

Results of the chemical analyses for Samples 1-6 are presented in Table 2. In addition to the data listed in Table 2, it should be noted that no boron carbide or boron nitride was detectable within experimental accuracy (2%).

In general, the following conclusions can be drawn from Table 2: firstly, the as-received B results for Samples 1 and 4, which were both analyzed to ascertain the reproducibility of the method, are equivalent within experimental error.

Secondly, by comparison of the separator and filter analyses, it is seen that the B particle size is larger than that of the C, since more B was found in the cyclone separator. This conclusion is also suggested by examination of the electron micrography results which are presented in the next section. Note also that substantial reaction of the B to ammonium pentaborate (NH₄B₅O₈·4H₂O) occurred during both primary firings. This compound is most probably formed on the surface of the B particles by reaction with ammonium perchlo-

**Fig. 1 Schematic diagram of particle collection apparatus.**

rate or its decomposition products during combustion of the solid propellant.⁶ If this compound were a constituent of the B particles, then proportionally more of it should be found in the separator, as is the case for Samples 2 and 5.

Finally, the higher percentage of C relative to B in Sample 5 than in Sample 2 is attributed to partial blockage of the filters as a result of the first firing; thus finer particles were collected in the cyclone separator.

It is concluded that thermochemical studies of the secondary combustor should not assume that the "boron" particles issuing from the exhaust of the primary rocket are composed of pure B or 95% B with the remainder B₂O₃ (which approximates the composition of the as-received particles); rather some allowance for the presence of NH₄B₅O₈·4H₂O on the particle surface must be made. However, because samples of unknown size distribution were collected in the cyclone separator and in the filter, it is not possible to estimate an absolute B concentration extant in the primary exhaust on the basis of the present results.

Table 2 Results of the x-ray diffraction analyses

Source/Constituents	Sample number	
As-received boron	1	4
B	95.5-97.5% ^a	96-98%
B ₂ O ₃	4.0-6.0%	3-5%
Organic	...	1-2%
H ₂ O soluble	3-7%	3-7%
C (by difference)
Cyclone separator (larger particles)	2	5
B	50-55%	45-50%
B ₂ O ₃	2.5-4.5%	5.5-7.5%
Organic	4-6%	5-7%
H ₂ O soluble	20-25% ^b	20-30% ^c
C (by difference)	10-20%	20-30%
Filter (small particles)	3	6
B	20-35%	25-35%
B ₂ O ₃	5-7%	6.5-8.5%
Organic	10-20%	10-20%
H ₂ O soluble	20-30% ^d	20-30% ^e
C (by difference)	20-30%	25-35% ^f

^a Weight percents are listed; accuracy is $\pm 1-2\%$.

^b 15-20% NH₄B₅O₈·4H₂O; <5% NH₄Cl; balance B₂O₃.

^c <5% NH₄Cl; balance NH₄B₅O₈·4H₂O and B₂O₃.

^d <10% NH₄B₅O₈·4H₂O; 12-18% NH₄Cl; balance B₂O₃.

^e <10% NH₄Cl; balance NH₄B₅O₈·4H₂O and B₂O₃.

^f There is possibly another amorphous species present (which would lower the actual C).

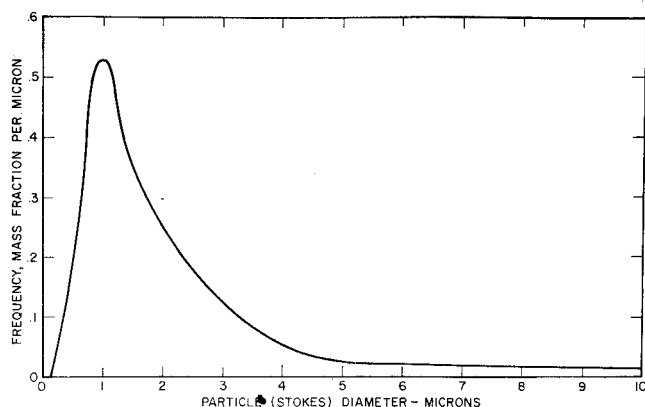


Fig. 2 Mass frequency vs diameter, sample 1.

B. Size Distribution Analyses

Size distributions for some of the samples were obtained using electron micrography or photosedimentometry; the former analyses were performed by Sloan Research Industries and the latter by J. William Shelnutt of the Aerospace Research Laboratories at Wright-Patterson Air Force Base.

For the electron micrograph studies, Sample 4 was examined as received, but, because Samples 5 and 6 contained particles of species other than B (the size distribution of which is of primary interest here), they were leached with both water and organic solvents to remove all constituents except C, which could not be removed, and B. Results for Sample 4 are presented in Table 3. The mode of the distribution presented in Table 3 is on the order of 0.25μ .

Sloan Research Industries noted difficulties in obtaining a similar analysis for either the separator or filter samples because of the presence of the C particles, but did indicate that "we have looked at enough preparations of these samples (5 and 6) to feel firmly convinced that the general statistical mode of the boron particles does not vary significantly from that put forth (in Table 3)."⁷ In addition, in initial studies which were conducted with unleached samples of 5 and 6, large amounts of particles in the $\frac{1}{3} \mu$ range were noted and were attributed to either the C or one of the soluble species.

Stokes' diameters were measured for samples 1 and 2 using photosedimentometry⁸ (see Ref. 5 for a discussion of this technique). Typical frequency curves for each sample are presented in Figs. 2 and 3. It is seen that the mode for sample 1 is approximately 1μ and that for sample 2 is about 2.3μ . For these data the sample preparation consisted of simply mixing a known weight of the original sample with a known volume of the sedimenting solution (1% Liqui Nox soap in H_2O) followed by vigorous shaking by hand. If for two minutes the sample was ultrasonically dispersed at 22,000 Hz before testing, however, the modes for both samples appeared

Table 3 Results of the electron micrography analyses of Sample 4

Particle size, μ	Incremental %
0-0.1	0.5
0.1-0.2	9.5
0.2-0.3	26.5
0.3-0.4	21.0
0.4-0.5	11.0
0.5-0.6	9.0
0.6-0.7	7.5
0.7-0.8	5.5
0.8-0.9	2.0
0.9-1.0	2.0
1.0-1.1	1.0
1.1-1.2	1.0
1.2-1.3	0.5
1.3-1.4	1.0
1.4-1.5	0.5
	98.5%

to shift towards smaller diameters, but because the frequency curves were not smooth, no numerical value was given.

Although it was argued in the previous section that because relatively more C was collected in the cyclone separator in the second firing, due to partial blockage of the filters, it is not felt that the discrepancy between the size analysis for the second firing (Sample 4, electron micrography, mode about 0.25μ) and that for the first firing (samples 1 and 2, photosedimentometry, mode equal to or greater than 1μ with manual dispersion, but somewhat less with ultrasonic dispersion) results completely from this effect. Indeed, since Samples 1 and 4 were taken from the same source, there is clearly an influence of the type of analysis used (it is well known that different techniques can give differing results even for the same sample).⁵

Rather, the larger distribution modes obtained with the photosedimentometer are attributed to the following points: firstly, in the photosedimentometer the settling rate of the particles will be influenced by entrainment of the sedimenting solution; thus the effective particle diameter will be larger as determined by this technique than by micrography. Secondly, for photosedimentometry the density of the particulate sample is required as input, and difficulties were noted in determining this value accurately: variations in the measured densities for sample 2 were as large as $\pm 6.3\%$ of the average, which could lead to errors of $\pm 2.5\%$ in the specified diameters.⁸ Thirdly, the method is limited by the assumptions that the extinction coefficient for the particles is independent of particle size in the range of interest and that the particles are not significantly soluble in the sedimenting fluid. This last assumption is most likely appropriate for the B particles, but not for the water soluble components.

Consequently, because of all of these effects it is generally agreed that photosedimentometry is a comparative technique, but cannot be applied for absolute size measurements,⁵ and in the present context no significance is placed in its yielding a larger distribution mode than electron micrography. It is thus concluded that the latter results are more valid, that is, the B particles in Samples 4 (as-received), 5 (removed from the cyclone separator), and 6 (removed from the filters) all possess a distribution mode on the order of 0.25μ . The result of the photosedimentometric analyses that the separator sample (sample 2) has a larger mode than the as-received B may be valid insofar as this technique cannot distinguish between particles of differing composition; however, the distributions for the B particles are thought to be the same on the basis of the electron micrographs.

It must be emphasized that these conclusions do not infer that the B particles in the exhaust of the primary rocket possess the same size distribution as those used for formulation of the propellant, since the effects of the particle collection method upon the actual distribution in the exhaust are

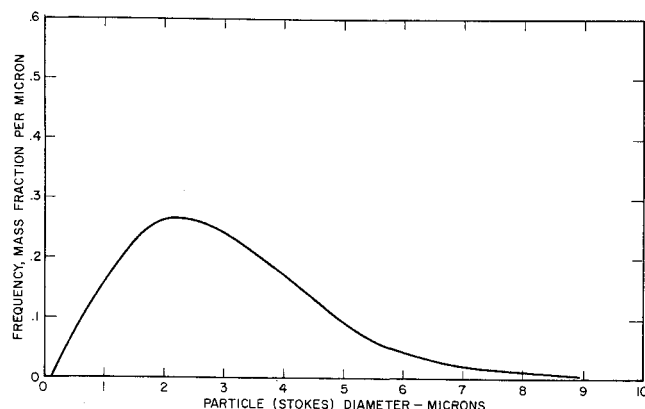


Fig. 3 Mass frequency vs diameter, sample 2.

unknown; only in situ measurements which have minimal effects on the local flow can yield this information.

IV. Summary

In any characterization of the performance of an engine such as the secondary combustor of an air-augmented rocket, a combustion efficiency is defined as the experimentally determined change in some parameter (e.g., temperature) divided by its theoretically calculated value. One contributing factor to discrepancies in efficiency can arise in calculation of this latter value, because the composition of the fuel, specifically its particulate components, is frequently unknown. In the present study it has been demonstrated that these particles can contain $\text{NH}_4\text{B}_5\text{O}_8 \cdot 4\text{H}_2\text{O}$, NH_4Cl , C, and other organics in addition to B and B_2O_3 . Clearly, before the performance of a motor is compared with theory, tests similar to those reported here must be conducted to determine the input for the analytical calculation.

Size analyses of the particles collected in the present investigation have also been discussed. The as-received B has a mode on the order of 0.25μ , and electron micrograph studies of the particles collected from the primary motor exhaust suggest that no significant change in this mode has occurred. However, since the influence of the experimental

collection method on the measured particle size distribution is unknown, this conclusion cannot be stated unequivocally.

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OCTOBER 1971

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Magnetohydrodynamic Instabilities in a Weakly Ionized, Radiating Plasma

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A unified, linear theory is formulated for MHD waves at low magnetic Reynolds number, propagating transverse to the magnetic field in a weakly ionized, radiating plasma. The set of equations obtained from a two-fluid model yields a fifth-order dispersion relation whose roots correspond to four waves: magnetoacoustic (a paired wave), thermal, electrothermal, and "ionization-rate." To bring out the physics of each of these waves, simplifying assumptions are made, and "simple" analytical wave solutions are found. By taking advantage of the wide separation of the roots in the complex plane, "distinct" wave solutions are obtained in which many limitations of the "simple" wave solutions are absent and which are in excellent agreement with the roots of the full dispersion relation, obtained numerically. Both thermal and magnetoacoustic waves are shown to require a two-fluid model for proper description. In particular, the latter mode requires the inclusion of the rate of change of electron enthalpy for wavelengths $\lesssim 0.1$ m, at typical MHD generator conditions. The electrothermal wave instability region is much larger than previously thought, whereas the "ionization rate wave" is always stable.

Nomenclature

a_0 = steady-state speed of sound
 \mathbf{B} = magnetic field
 B_{00} = Planck function at line center
 C_{en0}, C_{ei0} = steady-state collisional loss
 C_r = C_{ei0}/C_{en0}
 d = width of MHD channel
 D/Dt = $(\partial/\partial t) + \mathbf{u}_g \cdot \nabla$

e = elementary charge ($e = 1.60210 \times 10^{-19}$ coul)
 e_i = internal molecular energy per unit mass
 \mathbf{E} = electric field measured in laboratory frame
 \mathbf{E}' = electric field measured moving at the heavy-gas velocity
 h = Planck's constant ($h = 6.624 \times 10^{-34}$ joule-sec)
 H = $\lambda_0 T_e k^2 / (1 + \beta_0^2)$, electronic heat conduction loss as function of wave number
 I = $J_0 B_0 / k p_{g0}$, interaction number

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