Pressure, Velocity, and Geometry Effect on Al_2O_3 Produced During Aluminized Propellant Combustion

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The effect of Al_2O_3 particles on rocket motor stability is critical, but the mechanisms of droplet formation and roles played therein by pressure, velocity, and geometry are not well characterized. An experimental study is reported which was designed to increase this understanding. Size distribution of Al_2O_3 particles as a function of pressure and velocity along with the effects of flow geometry was determined. There was some effect on the particle size as a function of pressure. Moderate-to-low parallel flow velocity resulted in significant changes to smaller particle size distribution as did flow vortices across an otherwise perpendicular flow system. Upon quenching of combustion products as a function of distance from the end-burning propellant surface, it was found that only at quench distances less than about 1 in. was the size distribution affected. The trend was for less smoke ($d < 1$ μm) to be collected after early quench.

Introduction

In recent years, considerable attention has been given to the subject of the state, size, and number density of the particulate matter resulting from the combustion of aluminized solid propellants.[1-14] This is because these particulates provide a major source of damping of the acoustic energy driven by the combustion of these highly energetic propellants, thereby stabilizing otherwise unstable systems. The degree of optimization of this damping is of importance since the stability achieved is often marginal at best.

Two key parameters in the stability of motors and laboratory burners using aluminized propellants are the size distribution of the aluminum oxide reaction products and the spatial dependence of this distribution.[9,10,16] It is believed that these are critically dependent on the way the flow in the combustor affects the accumulation of aluminum on the propellant burning surface, and on the subsequent combustion and formation of Al_2O_3 droplets.[3,7,11,12] Assuming this line of reasoning is correct, the effect of flow would lead to a spacewise variation in size distribution of Al_2O_3 which would have to be known in order to make combustor stability calculations.[16] Such a position dependence would greatly complicate the calculation of damping even if the Al_2O_3 droplet size-space distribution were known, simply because a volume integral would then be required.

To probe the extent of this problem, *T*-burner tests have been made on different laboratory burner configurations[6,8,17,18] and corresponding effects have been observed on damping. Likewise, changes have been made in propellant formulation aimed at changing aluminum agglomeration and Al_2O_3 droplet size distribution[1,9-11,19,20] Again, corresponding changes in damping were observed. Previous observations[1,17,18] have supported the contention that changes in damping were due to changes in Al_2O_3 particle size distribution. However, the supply of such data, showing that the Al_2O_3 particle size distribution is indeed sensitive to flow and propellant variables, was very sketchy in most studies because of the difficulties of getting good particle sampling and size measurements.

A study has been conducted which was designed to assess the effects of pressure and velocity on the particles produced during the combustion of two operational propellants of broad interest and to assess the spatial distribution of the combustion of aluminum particles. The study is undertaken anew in spite of previous work in this area with these and similar propellants because of the disparity of results and variance of opinion that prevails. This disparity has resulted from both collection and counting techniques. For instance, particulate residue has previously been collected for analysis in a variety of ways which have left the findings uncertain. These include sampling from an exhaust plume after passing through a sonic constriction[9,13]; sampling residue remaining in vented burners after burnout,[1] and thus ignoring particles formed during the steady burning period; sampling from horizontally oriented burners with extended areas of exposed metal to quench large particles falling out of the gas stream[1]; sampling from systems of complicated geometry without adequate control tests for assessment of these effects,[1,9,10] and sampling on small plates.[9,12] The present study attempted to avoid the problems associated with these previous studies by firing in a vertical configuration and catching in a pan at the bottom of a surge tank most (up to 70%) of the particles produced. Further, the series of experiments, although including some of complex geometry, had straightforward tests for control and comparison.

Experimental Set Up and Procedure

For the mean pressure and velocity effect tests, the propellant combustion and particle collection was done in a vertically oriented, 12-in. long, 1½-in. i.d. pipe mounted on top of a 1 cubic ft surge tank pressurized with nitrogen. The propellant was mounted in the top end of the tube in either an end-burning (see Fig. 1) or internal-burning cylinder (side-burning) configuration, and the settled particles were collected from a pan in the bottom of the tank. By such an arrangement it was possible to collect about 70% of the

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Al_2O_3 produced. Large particles leaving the surface fell vertically without the tendency to drop against a "cold" metal surface and quench, as they might, in a horizontally oriented *T*-burner; no sonic constriction was present to influence the size of the particles collected; and by collection of "all" the particles, no bias was included toward particles produced at or near burnout.

The propellant samples were FKM (from a 600-lb casing), a composite modified double base (CMDB) propellant containing about 17% aluminum, and ANB 3066, an ammonium perchlorate-based composite propellant containing 15% aluminum. The end-burning samples used were, in general, ⅛-in. thick and 1½-in. in diam, potted into standard Naval Weapons Center (NWC) *T*-burner plastic holders. Ignition was achieved using standard NWC *T*-burner techniques, i.e., a thin coating of pyrotechnic paste was applied to the grain surface and initiation was by ¼-in. long nichrome bridgewire dipped in the same pyrotechnic material. Samples were kept as thin as possible to minimize the pressure rise during a test; about 25-50 psi depending on mean pressure and area ratio.

The system was pre-pressurized with nitrogen to the desired pressure and fired. The closed, pressurized system was allowed to settle for at least 4 hr, during which at least all but the very finest particles (which were insignificant in terms of both total mass and effectiveness of damping at the frequencies of interest) had settled. The 30% of theoretical Al_2O_3 was presumed lost primarily as $d < 1\mu m$ particles on the tank walls and to lesser degree during various separation and transfer processes.

For the investigation of velocity effects the samples were machined to ⅛-in. thick cylinders, 1½-in. o.d. (see Fig. 1b). Lengths were either ⅜-, ¾-, or 1⅛-in. corresponding to area ratios (A/A_o) of one, two, and three, respectively (relative to a single, full diameter, end-burning surface area). These samples were potted into correspondingly deep plastic cups and prepared for ignition as the end-burners were. The dimensions of these grains were chosen to be conservative. That is, the reported area ratio was not achieved until burnout so that any effects observed were also conservative. In addition, no attempt was made to provide these samples with a uniform velocity field, thus the effects were again lessened since the parallel velocity seen at the head end was zero in all cases.

To investigate the spatial extent of the combustion of aluminum particles, a series of tests was run in which the length of the cylindrical burner tube was shortened to such an extent that the initial end-burning surface was from ½ to 4½-in. away from a pan of liquid nitrogen standing in the surge tank. Sample preparation and firing was the same as in the mean pressure effect series except that pre-cooling of the coolant pan was necessary, and a certain amount of speed was required so that the liquid was still present upon ignition. It was found that the inertia, both thermal and mass, of the nitrogen was sufficient to maintain the liquid quench throughout the test. Although most of the solid combustion products were trapped in the coolant pan, some fine smoke and mid-range particles were collected, as in the pressure tests, at the bottom of the tank.

The effect of protrusions in the flow stream away from an end-burning grain was investigated also, because such a device had in other investigations produced surprising results.[8] A "vortex ring" was inserted in a burner tube ¾-in. away from the initial burning surface (see Fig. 1c). It consisted of a ⅛-in. thick annular ring whose i.d. was 1¼-in. leaving a ⅛-in. protrusion from the burner wall. Such a device has significantly changed the damping in previous *T*-burner tests, but not in the way anticipated. It had resulted in lower rather than higher damping, and therefore, greater instability of the system. The explanation at the time was that a steady toroidal vortex had been created (hence, the name of the device) and had caused flow across the end-burning surface resulting in a changed particle size. No firm evidence of such a mechanism was available and the explanation was not accorded universal acceptance at that time although no alternate explanation was presented.

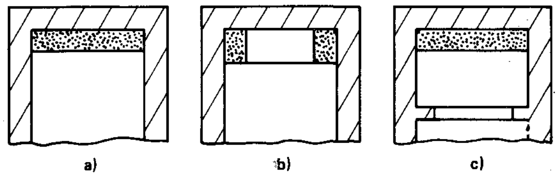

Fig. 1 Particle collection burner configurations: a) flat-end burning; b) internal-burning cylinder (side-burning); c) flat-end burning with vortex ring.

Separation of Particles

Particles collected in the collection pan were washed with ethanol into a 100 ml centrifuge tube, subjected to ultrasonic bath and then washed through a Tyler number 400 sieve. This process was repeated three times, by which time the sample had been divided into particles greater than and less than 37 μm. The fine fraction was again subjected to ultrasonic vibrations in order to break up agglomerated and electrostatically held particles. This material was then placed in a centrifuge and rotated at 2000 rpm for 5 min. The material still dispersed in the ethanol after this process was either in solution or a suspension of extremely fine particulates. This material was very small in quantity and included polymeric material which if left for an extended period of time caused the sample to become gummy to the point of not being separable. This solution was discarded. This process was repeated three times. The remainder of the particulates was separated into greater than and less than 2 μm by repetitive cycles of ultrasonic bath, centrifuge at a fixed rate for a fixed time of a fixed column of ethanol-Al_2O_3 suspension followed by separation of the settled material from the suspended particles.

Although the centrifuge was timed and provided with an electric brake so that cycles were repeatable, there was a transient start up (also repeatable) which came to equilibrium only at the end of the cycle, so that although the final speed was 600 rpm, the speed setting was determined empirically. The determination was based on the requirement that a spherical Al_2O_3 particle 2 μm in diameter at the top of the 4 cm column would just travel to the bottom in 1 min. Thus, all of the material still in suspension was assumed to be <2 μm. The settled material was again suspended in 4 cm of ethanol and the process repeated until the ethanol above the settled material was clear with respect to an arbitrarily set standard. Effectiveness of the separation was determined microscopically. The particular size cuts chosen corresponded to the finest sieve available, and the size below which very little damping occurs at the frequencies of interest in this work, since most particulate damping occurs between 1 and 15 μm at frequencies from a few hundred to tens of thousands of cycles per second.[15]

Experimental Results

Chemical analysis of the residue collected and separated indicated about 95% Al_2O_3 with the remainder assumed to be mainly aluminum and igniter fragments. Figures 2 and 3 show the results of the mean pressure investigation for FKM and ANB 3066 propellants, respectively. Note that the character of the curves is similar, with a less pronounced effect for ANB 3066 propellant. Even over the steepest portion of the FKM propellant curve the variation over the 30-50 psi pressure rise occurring during the course of a test is not significant with regard to the mass fractions measured. The uncertainty is less than 5 percentage points. The trend in both cases is toward a decreased particle size at increased pressures.

Figures 4 and 5 show the effect of gas velocity parallel to the propellant burning surface on the particulates formed

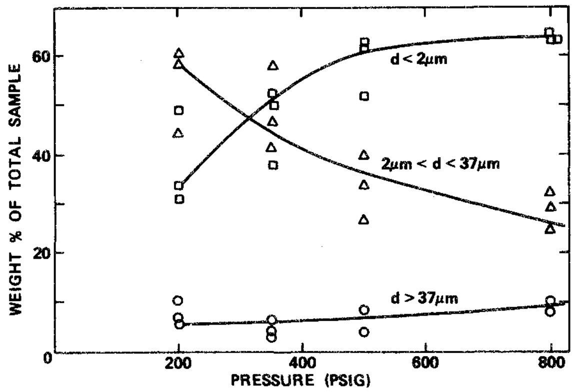

Fig. 2 Effect of mean pressure on particle size distribution; FKM propellant.

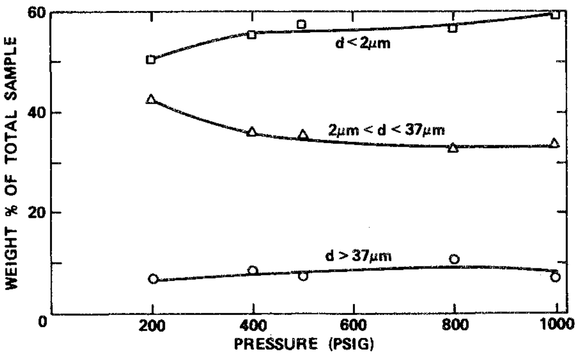

Fig. 3 Effect of mean pressure on particle size distribution; ANB 3066 propellant.

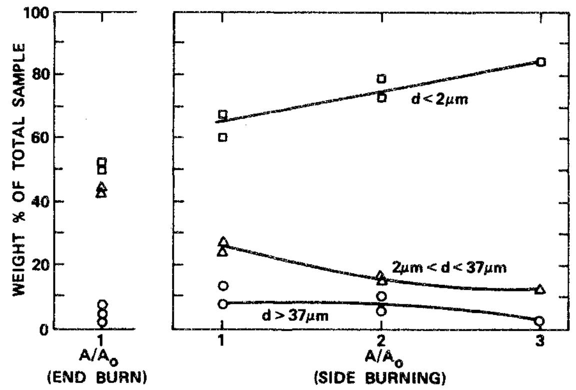

Fig. 4 Effect of parallel gas flow on particle size distribution; FKM propellant. Increased parallel gas velocity produced by increasing lengths of internal-burning cylindrical charges. Pressure was 350 psig.

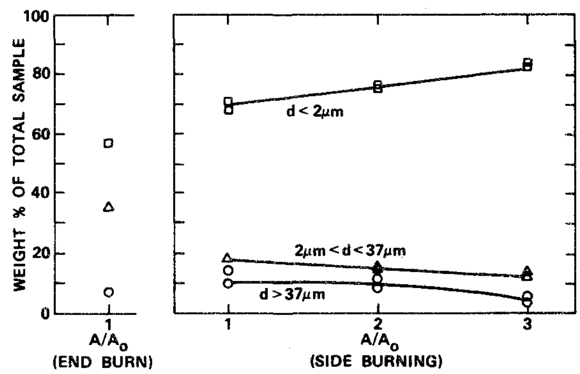

Fig. 5 Effect of parallel gas flow on particle size distribution; ANB 3066 propellant. Increased parallel gas velocity produced by increasing lengths of internal-burning cylindrical charges. Pressure was 500 psig.

during combustion. The parallel velocity was created by burning on the inner surface of cylindrical grains as pictured in Fig. 1b and was varied by varying the length of the cylindrical grain thereby varying the mass flow rate through the standard channel. The results in the figures indicate an increase in the fraction of very fine particles and an associated decrease in the fraction of "good dampers."

For both FKM and ANB 3066 propellants there is a significant increase in the fraction of fine particles produced when one changes from a flat, end-burning grain to one of an internal-burning cylinder (Figs. 1a and 1b, respectively). This change toward finer particulates with increased parallel velocity is shown to continue nearly linearly as far as to $A/A_o = 3$ and to extrapolate to 100% at about $A/A_o = 5$. This latter, of course, is not proposed as fact, but does indicate the minimum where a limiting velocity might exist in such a burner.

One possible interpretation must be proffered. If, at the surface under normal flow conditions, agglomeration of partially oxidized aluminum takes place resulting in large alumina particles, as seen in high speed, high magnification motion pictures, then as the parallel component of flow is increased an increasing tendency toward removal of partially formed agglomerates might well occur resulting in a decreasing size of the agglomerate residual droplets. Such a process might well result eventually in a "bald surface." Such behavior was seen when a stream of either cold N_2 or O_2 was passed parallel to a burning FKM propellant surface.

Note in all cases reported, the coarse fraction, $d > 37$ μm, is nearly constant within the scatter. Tests were run in which the grain thickness was varied by a factor of two. Upon separation of the residue it was found that the mass of $d > 37$ μm particles remained about constant. The conclusion reached was that the coarse material was produced during the transient periods, beginning and ending. There is a logical preference for the burnout transient at which time, due to heat loss and inadequate preparation time, aluminum and partially burned aluminum would be left. This is in agreement with the inference made by Kraeutle.[1]

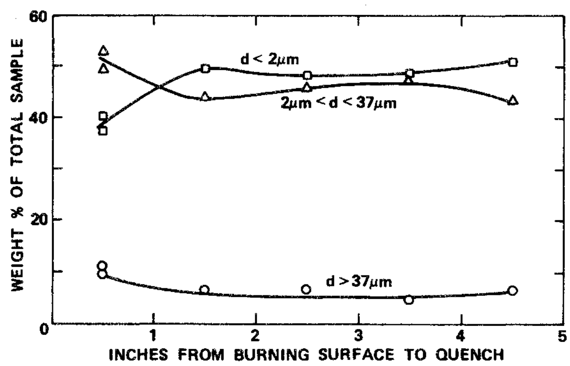

Fig. 6 Distribution of aluminum combustion. FKM propellant. Pressure was 350 psig.

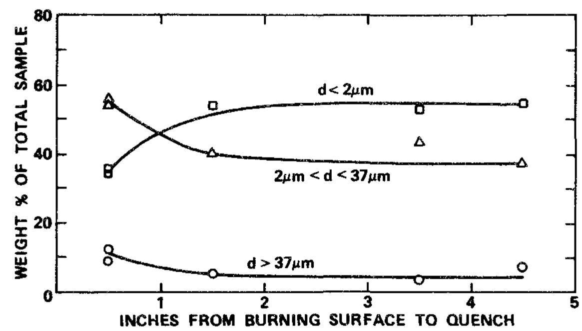

Fig. 7 Distribution of aluminum combustion. ANB 3066 propellant. Pressure was 500 psig.

Table 1 Effect of vortex ring on gross mass distribution of combustion products and combustor stability for FKM propellants at 350 psig

	$d>3\mu$m %	2μm$<d<3\mu$m %	$d<2\mu$m %	α_1 (sec^{-1})[a]	α_c (sec^{-1})[a]
Flat ended	3	52	47	−128	17
Configuration	5	50	41		
	4	38	58		
Vortex ring	8.2	29.5	62.3	−54	43
Configuration	6.8	28.8	64.4		

[a]From Ref. 11.

Figures 6 and 7 show the results of the distribution-of-aluminum-combustion experiment. It was noted that only at distances less than 1-in. from propellant surface to liquid nitrogen quench did this mass distribution appear to change. At distances less than 1-in. from the end-burning surface fewer $d<2$ μm particles, and more 2 μm$<d<$ 37 μm particulates were present than at greater distances.

Table 1 summarizes the results of the vortex ring tests using FKM propellant. The top numbers are taken from the data of Fig. 2 for end-burning tests at 350 psig. The lower numbers represent the comparable particle size distribution for the vortex ring tests. As can be seen there is a significant change in particle weights as a result of the addition of the vortex ring. Reference to Fig. 4 indicates that this flat-end vortex ring configuration results in a mass distribution very nearly identical to the distribution which resulted from $A/A_o=1$ side-burning tests.

Discussion and Conclusions

It is well known that in the control of unstable combustion of aluminum-containing propellants, it is the aluminum oxide particulate matter that is most effective in damping oscillations. Further, for a given frequency of instability the size of the damping particles is a critical consideration. It is, therefore, of extreme importance that the mechanisms of particle formation, or at the very least, the manner and degree in which particle size can be affected is understood. Referring to the Introduction, it seems quite clear that any serious calculation of combustion instability for aluminized propellants would require currently unavailable spatial dependence of particle size distribution for inclusion in the analysis.

For a number of years, several laboratories have been busy assessing stability of new propellants using T-burners. The flat ended T-burner evidently reliably represents conditions typical of stagnation points in a motor, while various other tubular T-burners represent some undefined position of a motor volume.

Many operational propellants are heavily loaded with aluminum and the flat, end-burning T-burner does not provide spontaneous oscillations for such systems. Two divergent approaches have therefore been taken: a pulsed T-burner which fires pressure pulses during burning and after burnout for comparison of damping rates of induced oscillations; and the cupped grain T-burner (variable area T-burner) which places propellant on the cylindrical walls of the burner, as well as on the ends, thereby providing sufficient driving to produce spontaneous oscillations. The results of these two systems have rarely been the same, and therefore, the reputation of the T-burner, in general, has suffered. There are admittedly drawbacks associated with both types of systems, but both have usefulness in propellant evaluation. The present results yield clear understanding of a major reason for the disagreements. To wit, the major source of damping is sensitive to just such geometrical variations.

The results of the present study indicate that the processes leading up to the combustion of the aluminum and size distribution of the Al_2O_3 were decidedly dependent on a) the orientation of the sample surface relative to the mean flowfield, b) the velocity of the mean flow parallel to the burning surface (or at least sample length), and c) the presence of the vortex ring in an end-burning T-burner. In addition, there was some effect of mean pressure (especially with the FKM propellant) but little effect of distance from the end-burning surface at which products were quenched except at distances less than 1 in.

The use of a variable area T-burner requires tests be made at a series of different area ratios and the measured growth rates of the spontaneous oscillations be plotted against area ratio and extrapolated to $A/A_o=0$. The intercept of this extrapolation is then assumed to be the damping present in the burner. The explicit assumption[19-21] is that damping is constant and only the driving varies with varying area ratio. The present results (Figs. 4 and 5) indicate that since damping is proportional to the number of dampers it would be fortuitous indeed for damping to remain constant when the mass fraction in the size range of interest varies drastically and non-simply with area ratio. It has not yet been demonstrated that either the rate of change of mass fraction or the mass fraction of favorable dampers itself becomes constant with increasing area ratio nor have the details of size distribution been determined. With luck, it may emerge that the effects of velocity (area ratio) may level off at some modest threshold velocity.[17] This remains to be determined, but it is clear that the effect of mean velocity cannot be ignored on the low velocity extreme.

It must also be noted that a rather constant fraction of very coarse dampers was present in all samples taken. It was argued that these were produced during the ignition and/or the burnout transients. If they were produced after burnout, it has been argued that the pulse-after-burnout decay of the pulsed system might be questioned. The response is two-fold. First, the particles are so large as to be almost completely ineffectual as dampers. Second, their presence must be limited to a short region near the burning surface due to the short time until the pulse, the lack of mean flow (zero), and the inertia of the particles themselves. The result is that the greatest part of the burner volume is filled with normally produced particles with only a second-order effect possible.

The vortex ring was first used to investigate the effect of the leading edge of the cupped grain. The present result using the vortex ring may not relate directly to the fluid dynamics of the cupped T-burner, but shows dramatically that what can initially only be a fluid dynamic effect has a very large effect on the very parameter most critical in controlling instability of a given propellant. A change from about 51% to about 29% in the fraction containing the most effective dampers occurred for FKM propellant so that regardless of whether the distribution within this size range changed at all, the number of dampers was changed drastically accounting for the earlier result of decreased damping when the ring was used in a flat-ended T-burner (see Table 2 taken from Ref. 11) as postulated in Ref. 8.

As for the distribution of combustion of aluminum particles, the reason for making this investigation was that the assumption that all combustion, and therefore all driving of instability, takes place at or very near the burning surface has been questioned and might be in considerable error if the aluminum actually burns in the gas stream. On the basis of the results, it is clear that from 1-5 in. away from the surface, no effect is seen in the mass fractions in the various size ranges. However, as the distance from the burning surface to the quench is reduced to a ½ in., the quantity of $d<1$ μm particles is decreased indicating interruption of burning with the resultant reduction of smoke produced. Derr, et al.[14] found that upon quenching of particles at ¼ in. distance from a propellant "similar to ANB 3066" only about 10% of the collected material lay in the range below 1 μm. The possibility of gas stream burning of aluminum has not been addressed for high parallel flow systems.

The results of this limited study need to be improved by: a) studying more propellants, b) more detailed particle size

Table 2 Summary of comparison *T*-burner tests in end-burning and cylindrical burners.[b]

Propellant	Configuration	f_1	$\bar{p}$	α_1	α_2	α_c
A-13 (no Al)	Flat disk	620	350	6.4	−11.2	17.6
A-13 (no Al)	Cylinder[a]	620	350	6.6	−11.9	18.5
FKM	Flat disk	620	350	−128	−143	17
FKM	Cylinder[a]	620	350	−93		43
ANB 3066	Flat disk	810	500	−120	−127	8
ANB 3066	Cylinder[a]	825	500	−75	−98	26

[a]Cylinders were flush with burner walls when data were taken.
[b]From Ref. 11.

measurements of the size fractions, c) extension to shorter burning distances, d) extension to lower pressure, e) determination of the nature of Al_2O_3 not collected in the test sample, and f) extension of the study of the effect of area ratio to larger areas and mean flow velocities. In the meantime, the present results seem decisive, and show a critical dependence of particulate damping on flow conditions in the motor or laboratory combustor.

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