NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Technical Memorandum 33-352

Recent Measurements at JPL of Particle Size of Al₂O₃ From Small Rocket Motors

R. A. Dobbins L. D. Strand

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CALIFORNIA IN	STITUTE OF TECHNOLOGY	
PASAD	ENA, CALIFORNIA	

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Contents

I.	Intr	$roduction \dots \dots$	•	•	•	•	•	•	•	•	•	•	1
11.	Mo	difications to Emission-Scattering Photometer .		•			•	•	•	•	•	•	1
111.	Inte	erpretation of Optical Measurement Results .		•	•	•		•	•	•		•	2
	Α.	Tests Without Aluminum Present in Fuel	•	•	•		•	•				•	2
	B.	Tests Approximately Duplicating Previous Firings	•	•	•			•			•	•	2
	C.	Tests at Various Radial Positions	•	•	•	•	•	•		•	•	•	3
	D.	Tests With Varying Aluminum Content	•	•	•		•	•		•	•	•	3
	E.	Test Results Using New Infrared Channel	•	•	•	•	•	•	•	•	•	•	4
IV.	Soli	id Exhaust Products Recovery Tests	•	•	•	•	•	•	•	•	•	•	5
No	men	clature	•	•	•	•	•	•		•	•	•	10
Ref	eren	ices									•		10

Tables

1.	Characteristics of the three optical channels	1	2
2.	Information on refractive index	1	2
3.	Firings without aluminum present	:	3
_4.	Tests with 12% aluminum (arranged in order of increasing pressure)	_ :	3
5.	Tests with 2% and 20% metallic aluminum concentration in propellant formulation	4	4
6.	Mean particle size indicated by channels 2 and 3 (λ_2 and λ_3) compared with size indicated by channels 1 and 2 (λ_1 and λ_2)	4	4
7.	Tank test conditions	5	5

Figures

1.	Tank configuration 1	•	•	•	•	•	٠	•	•	•	•	•	•	•	•	•	•	•	•	•	•	6
2.	Tank configuration 2		•	•		•	•	•	•	•	•	•	•		•	•		•	•	•	•	7
3.	Tank configuration 3		•				•	•		•	•	•	•	•		•	•	•	•	•	•	8
4.	Tank configuration 4	•	•	•		•		•	•		•	•	•	•				•		•	•	9
5.	Typical test record .	•				•				•					•			•		•	•	9

;

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Abstract

This second series of 28 test firings was a continuation for determining the particle size of Al_2O_3 from small rocket motors. After modifications were made to the emission-scattering photometer, the tests were conducted with varying amounts of aluminum, with optical beam geometry variations, and with the new infrared channel. Results of the tests with 12% aluminum agreed with the 1964 results; as did those results of tests with varying amounts of aluminum. Results of the tests with various radial positions indicated that the particle size did not vary in relation to radial positions. Results of the new infrared channel tests are inconclusive until further computer analysis can be conducted. The particle recovery tests are presently being conducted to determine whether the particle size distributions are dependent upon the receiver tank size.

Recent Measurements at JPL of Particle Size of Al₂O₃ From Small Rocket Motors

I. Introduction

This report summarizes the results of the second test series, performed at the Jet Propulsion Laboratory during the summer of 1966, using the emission-scattering photometer to measure the particle size of Al_2O_3 from small rocket motors. Also, it reports the progress of a series of solid-exhaust-products recovery tests that are currently being carried on at the Jet Propulsion Laboratory (Edwards Test Station). The report is brief in nature and merely presents the photometer modifications and the current results, without discussing the history of the tests, or describing the apparatus or procedure in detail. These topics are treated in Refs. 1–5, which are used as the basis for the present report.

A total of 28 test firings were made on five different days. These 28 tests have increased the number of tests with the emission-scattering photometer to a total of 51. The recorded data proved good in all but two firings during the current series; in one case, the photometer was adjusted incorrectly, and in the other, the rocket motor developed low-frequency nonacoustic instability.

The propellant used in these tests was similar to that used by Sehgal in the tank tests (Ref. 5), but with the

JPL TECHNICAL MEMORANDUM 33-352

amount of aluminum in the fuel varied to be either 0%, 2%, 12%, or 20% (see Tables 3, 4, and 5). The mass median diameter of the metallic aluminum in the fuel was 6.8 μ m.

II. Modifications to Emission-Scattering — Photometer — — — — — —

As a result of a conference at JPL in May 1966, a decision was made to modify the emission-scattering photometer to permit observations of optical transmission at 2.35 μ m. These modifications included: (1) the installation of a monochromator as previously recommended for the infrared channel 3 (Ref. 4); and (2) the use of a phasesensitive amplifier and an improved chopper motor unit on channel 3 to process the signal from that channel.

A Farrand monochromator, Catalog No. 117187, which operates in the 1 to 3 μ m wavelength interval, was installed. This monochromator was used with the interference filter that had proved inadequate by itself to eliminate the abundant radiation due to CO₂ and/or H₂O. The combination of the monochromator and the interference filter assured a high degree of monochromaticity in the narrow band passed by the filter. Additional improvements included the installation of a higher quality chopper motor and chopper wheel on the channel 3 and the attempted use of a phase-sensitive, lock-on type amplifier. This amplifier failed to perform as expected because it did not pass the 5-Hz modulation as a well-defined square wave. A dc amplifier, Model 3420 (Dana Laboratory Inc.) was used instead of the phasesensitive amplifier. The output signal of the dc amplifier was connected through a $1.0-\mu$ F capacitor to the recorder. These changes greatly improved the performance of channel 3 and allowed the desired optical observations to be performed.

A summary of the characteristics of the three channels is given in Table 1.

Channel No.	Wavelength, µm	Bandpass, µm	Transducer
ı	0.365	0.005	Photomultiplier, Type 1821
2	1.01	0.020	Photomultiplier, Type 7102
3	2.35	0.040	Lead Sulphide Photo- conductor, Type N-2

Table 1. Characteristics of the three optical channels

The overall linearity of the individual channels was tested before each series of runs by inserting a calibrated neutral-density filter in the beam adjacent to the beam intersection with the rocket motor axis. The signals from the transducers were processed and recorded as used for a test. In this manner, the system linearity of each channel was tested and found to be good in all cases.

III. Interpretation of Optical Measurement Results

The measured optical transmission was interpreted using the plotted curves for r_{τ} (2, 1) and r_{τ} (3, 2) vs D_{32} (see Figs. 3 and 4 of Ref. 1). These curves were prepared with the values of refractive index *m* then available to the author. Since that time, the values of *m* at high temperatures were investigated by Gryvnak and Burch (Ref. 6). The values used by the author and those recommended in Ref. 6 are summarized in Table 2.

Although the following data reduction was performed with values of m that do not exactly agree with the best data now available, investigation of the magnitude of the incurred error found the error to be 5%-10%. This error

Wavelength, µm	m used by author	<i>m</i> in Ref. 6
0.365	1.77	1.85
1.01	1.75	1.81
2.40	1.73	1.78

was considered to be an insignificant magnitude that would not warrant correction at this point; however, this correction should be performed in the future.

All 28 tests fall into one, or possibly two, of the following categories. The optical beam geometry was varied during the test program and included position 1 used earlier (0.35×1.22 -in. beam at exit plane, see Fig. 5 of Ref. 4) and the following:

- (1) Position 6: 2.0×1.8 -in. oval beam, 38 in. from rocket nozzle exit plane.
- (2) Position 7: 0.33-in. high × 1.25-in. wide beam, 38 in. from rocket nozzle exit plane.
 - (a) 7a: on rocket axis of symmetry.
 - (b) 7b: 1.25 in. below axis.
 - (c) 7c: 2.50 in. below axis.
 - (d) 7d: 4.00 in. below axis.

A. Tests Without Aluminum Present in Fuel

The purpose of the first several tests was to determine the most advantageous wavelength in the vicinity of 2.40 μ m. After several runs, a monochromator setting of 2.35 μ m was chosen, because the emission at this wavelength was substantially less than 2.40 μ m. Transmissions (T_1 , T_2 , T_3) at all three wavelengths (λ_1 , λ_2 , λ_3) were nearly 100%, as shown in Table 3. Accuracy of measurements was based on a $\pm 10\%$ transmission error, as shown in Table 3.

B. Tests Approximately Duplicating Previous Firings

During the summer of 1964, 15 tests were made in which the particle size of Al_2O_3 was measured with 12% metallic aluminum in the propellant (22.7% Al_2O_3 by mass in the product of combustion). Those tests indicated that the mean particle size D_{32} varied from 0.40 to 0.55 μ m as chamber pressure was increased from 90 to 750 psia.

Test	Wavelength		Beam		Transmission			Emission	
No.	setting for λ₃, μm	P _c , psig	geom	T ₁ T ₂		T ₃	C ₁ / A ₁	C ₂ /A ₂	C ₃ /A ₃
24	2.40*	33	1	0.99	0.965	0.978	0.06	0	1.19
25	2.35*	310	1	0.98	0.965	0.970	0	0	0.12
26	2.35*	29	1	1.07	1.00	0.97	0.02	0	0.25
27	2.35*	102	1	0.985	0.938	0.975	0.06	0	0.34
50	2.35	77	6	0.89	0.94	1.00	0	0	0
51	2.35	518	6	0.93	0.93	1.00	0	0.	0

Table 3. Firings without aluminum present

The current series of tests, using the same channels 1 and 2 of previous tests, produced results that are in close agreement with the 1964 tests (see Table 4). All runs listed in Table 4 were performed in the far plume at a distance of 38 in. from the rocket motor to allow for greater cooling of the particles. The scatter in the data was somewhat higher than before, but the weak-pressure dependency was, again, conspicuously indicated. A slight tendency for the particle size to increase at the end of burning was indicated in all cases, except Tests 32 and 33. This tendency was, as noted previously, so weak that it is of questionable significance.

_ Test _	Beam		Pc, Beam and 2, µ					
No.	psig	geom	t _x , after ignition	ty, before burnout				
30	66	6	0.44	0.46				
33	238	6	0.49	0.49				
32	242	6	0.48	0.48				
31	590	6	0.52	0.55				
29	594	6	0.55	0.57				
37	706	7a	0.55	0.58				
38	710	7b	0.55	0.58				
39	720	7c	0.55	0.56				
40	725	7d	0.55	0.57				

Table 4. Tests with 12% aluminum (arranged in order of increasing pressure)

C. Tests at Various Radial Positions

Tests 37, 38, 39, and 40 (Table 4) were performed with the optic axis at distances of 0, 1.25, 2.50, and 4.00 in.

below the symmetry axis of the rocket motor, respectively. The small-beam $(0.35 \times 1.22 \text{ in.})$ shape was used and the horizontal position of the beam was placed 38 in. aft of the nozzle exit plane. The data showed no variation of particle size with radial position, thus indicating that the particles are in dynamic equilibrium with the gas. These test results paralleled the observations made in 1964, when the radial variation of particle size was examined at the near plume. In the former tests, the chamber pressure was low; in this case, the chamber pressure was over 700 psia.

Kliegel gave a criterion (Eq. 43 of Ref. 7) to estimate the magnitude of particle lag effects. From this relation, the particle velocity in these tests should be about 97% of the gas velocity at the nozzle throat. The test results are in agreement with this calculation in that both experiment and theory indicate no lag effects.

D. Tests With Varying Aluminum Content

The mass fraction of aluminum in the propellant was varied from the previous 12% value (Table 4) up to 20% and down to 2% values.

At the high aluminum loading, the optical density of the plume required an increased sensitivity on the photometer to measure accurately the transmitted light signal. This capability, which had been built into the photometer, was used successfully for the first time during the firings with 20% aluminum. Because the light to the photometer was cut off before the end of burnout, a post-run calibration of the photometer was not obtainable. Thus, only a measurement of particle size after ignition was obtained. The tests with the 2% aluminum loading of the propellant frequently resulted in only a small amount of attenuation due to scattering. Under these circumstances, the accuracy of the experiment was less favorable and the scatter in the data was greater. This increased scatter is evident in the data presented in Table 5.

Task		Paarm		D ₃₂ from	λ_1 and λ_2
No.	AI, %	geom	Pc, psig	After ignition, μm	Before burnout, μm
34	2	6	40	0.37	0.39
45	2	1	61	0.41	0.46
49	2	6	92	0.41	0.46
35	2	6	162	0.45	0.52
36	2	6	332	0.41	0.41
48	2	6	524	0.44	0.44
44	20	6	o	0.275	_
47	20	6	77	0.49	_
42	20	6	235	0.50	-
41	20	6	566	0.55	_
43	20	6	775	0.58	—

 Table 5. Tests with 2% and 20% metallic aluminum concentration in propellant formulation

In general, however, the results of the tests with both the 2% and 20% aluminum content are in fairly close agreement with the results of the tests with 12% aluminum content.

Test 44 with 20% aluminum (Table 5) was fired into the open atmosphere without any nozzle. This test indicated a particle size of 0.275 μ m, which is in close agreement with the United Technology Center tank test measurements of a nozzleless firing at a pressure of 1 atm.

E. Test Results Using New Infrared Channel

Most tests yielded information with the new infrared channel λ_3 , 2.35 μ m. The results of the data reduction, using the transmission measurements from channels 2 and 3 (λ_2 and λ_3) and the curve of r_{τ} (3, 2) vs D_{32} (Fig. 4 of Ref. 1), is summarized in Table 6. The mean size was calculated at a specific time after ignition t_x and before burnout t_y , except for the runs with 20% aluminum because of the previously discussed reasons. The data obtained from channels 1 and 2 are tabulated in Table 6 for comparison.

					Indicated	size D ₂₂ , μ	m
Test No.	Beam geom	AI, %	P _c , psig	With A	$_2$ and λ_3	With λ	$_1$ and λ_2
				t _x	t _y	t _x	ty
34	6	2	40	0.84	0.86	0.37	0.39
49	6	2	92	0.86	0.89	0.41	0.46
48	6	2	524	1.0	0.92	0.44	0.44
30	6	12	66	0.90	0.84	0.44	0.46
33	6	12	238	1.01	1.01	0.49	0.49
31	6	12	590	1.18	1.10	0.52	0.55
37	7a	12	706	1.25	1.25	0.55	0.58
38	7Ь	12	710	1.15	1.28	0.55	0.58
39	7c	12	720	1.15	1.20	0.55	0.56
40	7d	12	725	1.25	1.25	0.55	0.57
47	6	20	77	0.92	-	0.49	_
42	6	20	235	1.05	_	0.50	_

41

6

20

566

1.10

0.55

Table 6. Mee	an particle size indicated by channels 2 and 3
(λ_2 and	id λ_3) compared with size indicated by
	channels 1 and 2 (λ_1 and λ_2)

The systematic discrepancy between the two sets of data is shown in Table 6. In all cases, the particle size indicated by channels 2 and 3 is about twice the size indicated by channels 1 and 2. The estimate of the difference in size, using the latest available information on refractive index of Al_2O_3 (see Table 2), is a reduction of about 10% for channels 2 and 3, and about 5% for channels 1 and 2. The discrepancy between the results from the two combinations of wavelengths is not attributable to the differences in the real part of the refractive index. The particles are assumed to be dielectric, i.e., the imaginary portion of the refractive index is assumed to be zero. Latest estimates indicate that the use of an imaginary portion equal to zero is justified. A computer program is nearing completion at Brown University that will calculate the magnitude of the mean extinction coefficients for particles whose refractive indices are complex. When this program becomes available, a check of the previous calculations of mean scattering coefficients and a more careful appraisal on the influence of a finite, but small imaginary, portion of the refractive index will be conducted. The future JPL tank tests may produce more information on this matter. In the meantime, the sizes measured by the transmission tests from channels 1 and 2 are considered to be more credible.

Also indicated in Table 6 is a small increase in particle size at higher pressures. Again, the important point of disagreement between optical and tank tests is found to be present. Finally, the channels 2 and 3 data (as well as the channels 1 and 2 data) indicate a very slight increase of particle size at higher aluminum concentrations.

An error analysis was made to investigate the sensi-*tivity of the indicated size to an error in the measurement of optical transmission with the assumption of only this type of uncertainty being present. The most unfavorable result developed when the transmission at one wavelength was 10% high (or low) while the transmission at the other wavelength was 10% low (or high). Using Test 30, the indicated size was 0.44 μ m; but, if an error of +10% occurred in T_1 and -10% in T_2 , the indicated size would be 0.42 μ m, or 5% lower. If T_1 and T_2 were both high (or low) by 10%, then the indicated size would be reduced from 0.44 to 0.43 μ m. Thus, the size measurement was not strongly sensitive to errors in transmission measurements; this lack of sensitivity is due to the steepness of the slope in the curves of r_{τ} vs D_{32} .

IV. Solid Exhaust Products Recovery Tests

A series of small motor firings are being conducted, using the tank collection technique, to determine whether the particle size distributions of the aluminum oxide exhaust are dependent upon the size of the receiver tank. The test firings are being conducted using five different size configurations for the collection tank:

-(1)-Configuration-1 (Fig. 1) Original tank for work done at JPL (Ref. 5)	-OD = -3.5 ft length = 8 ft vol = 75 ft ³
(2) Configuration 2 (Fig. 2) One-half of original tank with closure section	$\begin{array}{rl} \text{length} = & 5.5 \ \text{ft} \\ \text{vol} = & 40 \text{ft}^3 \end{array}$
(3) Configuration 3 (Fig. 3) Original tank plus 4-ft long cylindrical section	$length = 12 ft$ $vol = 112 ft^{3}$
(4) Configuration 4 (Fig. 4) Original tank plus 6-ft long cylindrical section	$length = 14 ft$ $vol = 130 ft^{3}$
(5) Configuration 5 Original tank plus 4-ft and 6-ft cylindrical sections combined	$length = 18 ft$ $vol = 168 ft^{3}$

The test program consists of two test firings carried out at each of three motor-chamber pressures (100, 300, and 800 psi) for each of the five tank configurations. The test motor and solid propellant are the same as used in Ref. 5, except for the use of 30° converging-15° diverging conical copper nozzles with molybdenum inserts and 4/1expansion ratios.

Instrumentation consists of two motor pressure gages (Taber), one tank pressure gage (Taber), and an unshielded chromel-alumel thermocouple extending into the tank approximately 6 in. from the tank wall. All data are recorded on a CEC oscillograph recorder. Figure 5 shows a typical test record.

The test procedure is basically the same as that reported in Ref. 5. The tank is pressurized with 1-atm nitrogen before firing the motor. After motor firing, the tank is vented and then is pumped down until the inside of the tank is completely dry. All deposited material is loosened by brushing. The tank walls are lightly watered and all remaining deposits are removed, using a squeegee. Also, the tank bleed valve is run through a scrubber to catch all suspended material that might otherwise be lost during tank ventdown.

These particle size distributions from the representative samples of both the dry and the wet collections are being obtained by Applied Space Products, Incorporated, Palo Alto, California, using the same electron microscope procedure as reported in Ref. 5.

Representative tank test conditions, as measured to date, are shown in Table 7.

Tank configuration No.	Maximum tank pressure, psig	Maximum thermocouple temperature, °F
1	70	1250
2	80	1300
3	55	1030
4	50	1000

Table 7. Tank test conditions

The complete results of the determination and analysis of the test particle size distributions will be reported at a later date.



Fig. 1. Tank configuration 1



Fig. 2. Tank configuration 2



Fig. 3. Tank configuration 3



Fig. 4. Tank configuration 4



Fig. 5. Typical test record

Nomenclature

Α	full scale prerun or postrun calibration (see	T_1	transmission $(B_1 \div A_1)$ for channel 1, $\lambda = \lambda_1$
	Fig. 2 of Ref. 1)	T_2	transmission $(B_2 \div A_2)$ for channel 2, $\lambda = \lambda_2$
В	signal from photometer with lamp on (see $F(x) = 0$, $f(x) = 0$)	T_{3}	transmission ($B_3 \div A_3$) for channel 3, $\lambda = \lambda_3$
ľ	rig. 2 of net. 1)	t_b	time between pressure buildup and decay
С	signal from photometer with lamp off (see Fig. 2 of Ref. 1)	t_x	time measured 10% of t_b ahead of time at which pressure buildup occurs
$D_{_{32}}$	mean particle size (see Ref. 1 or 2)	t_y	time measured 10% of t_b back from time at
m	refractive index		which pressure decays
P_c	chamber pressure, psig	λ	wavelength of light
P_{T}	tank pressure, psig	$\mu \mathbf{m}$	microns
(0.1)		Subscripts	
r_{τ} (2, 1) or	lengths 2 to 1, or 3 to 2 (see Ref. 1)	1	channel 1
r_{τ} (3, 2)		2	channel 2
T_c	tank thermocouple temperature	3	channel 3

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