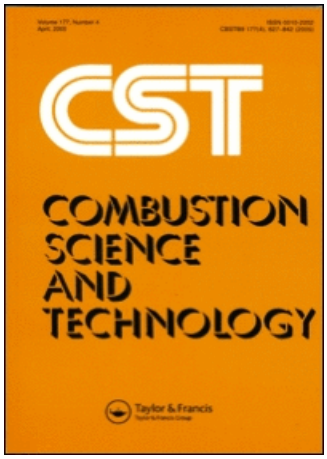

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Regression Rate Studies in Hypergolic System

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Regression Rate Studies in Hypergolic System

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Abstract—Regression rates of a hypergolic combination of fuel and oxidiser have been experimentally measured as a function of chamber pressure, mass flux and the percentage component of the hypergolic compound in natural rubber. The hypergolic compound used is difurfurylidene cyclohexanone (DFCH) which is hypergolic with the oxidiser red fuming nitric acid (RFNA) with ignition delay of 60–70 ms. The data of weight loss versus time is obtained for burn times varying between 5 and 20 seconds. Two methods of correlating the data using mass flux of oxidiser and the total flux of hot gases have shown that index n of the regression law $\dot{r}=aG_{ox}^n$ or $\dot{r}=aG^n x^{n-1}$ (x the axial distance) is about 0.5 or a little lower and not 0.8 even though the flow through the port is turbulent. It is argued that the reduction of index n is due to heterogeneous reaction between the liquid oxidiser and the hypergolic fuel component on the surface.

INTRODUCTION

Regression rate measurements in hybrid rocket engines are pre-requisite to the design of such systems. It is well known that the regression rate at low chamber pressures depends strongly on the mass flux of oxidiser through the port of the fuel grain and on chamber pressure. Above a certain pressure level the regression rate is a function of only the mass flux. Investigations on regression rate have been fairly extensive and many studies have been conducted with classical oxygen-polymer systems particularly at ambient pressure (Marxman, 1964; Houser and Peck, 1964; Rastogi *et al.*, 1974; Gupta, 1974; Srivastava, 1974; Joulain *et al.*, 1977). In some studies results have been obtained at higher pressures of the order of 10–16 kg/cm^2 (Wooldridge *et al.*, 1969; Smoot and Price, 1966, 1967). Studies on hypergolic systems have not been very extensive (Smooth and Price, 1966; Ankarsward, 1964; Schmucker, 1971).

Table I shows a brief summary of the results of several investigators including those of the present authors. Ankarsward (1964) describes studies on hypergolic fuels using red fuming nitric acid (RFNA) as oxidiser and Tagaform in the form of pressed blocks as fuel. Though ultrasonic technique was used to find regression rate, it was found to be less accurate compared to weight loss technique. The results as deduced from the pressure versus time traces of rocket motor test showed the index n in

$$\dot{r} = a\,G^n \qquad (1)$$

as 0.8 at high pressures and 0.5 at low pressures. In the former case, the mass flow rate of fuel decreases with time and the chamber pressure decreases as a consequence, and in the latter the mass flow rate remains constant with time and hence the pressure also remains constant.

Nadaud and Baisini (1966) conducted experiments on a two-dimensional motor in a manner different from the ones conducted by others. A gas generator which functioned on nitric acid and a solid hypergolic fuel was used to obtain hot gases of desired oxidiser to fuel ratio (O/F). These gases were passed through the test cell containing blocks of PVC or the hypergolic fuel as the candidate fuel for regression. The results were obtained over a wide range of pressures and mass fluxes. A curve fit technique was used to obtain the exponent n of Eq. (1). The principal results are that the regression rate is independent of pressure beyond about 20 atm in all cases and the dependence on pressure comes in below 20 atm only at relatively high fluxes ($\sim$100 g/cm^2 s). This fits into the classical theory proposed by Smoot and Price (1965) and is explained by chemical kinetics (heterogeneous or homogeneous) becoming the rate controlling mechanism of regression. They also observe another peculiar feature, namely that at relatively high fluxes of hot gas flow, its composition matters; near about slightly oxidiser richness the regression rate attains a peak of 20–30 percent more than the asymptotic rates for the hypergolic fuel. This feature in all likelihood is due to the heterogeneous attack in the oxidiser-rich zone combined with reducing flame

TABLE I

Summary of the results of regression rate measurements in hypergolic systems by different authors

Sl. no.	Authors	Experimental rig	Fuel and oxidiser	Regression rate law	Data analysis scheme	Flux, burn time pressures, etc.
1	Ankarsward (1964)	Tabular burner OD of the motor 100 mm	RFNA hypergolic fuel (TAGA form)	$\dot{r}=aG^{0.8}$ at low pressure	Weight loss measurements	G_{ox}: 40 g/cm² s P_c, 30 kg/cm² and 7 atm t_b: up to 20 s
2	Smoot and Price (1965, 1966, 1967)	Two-dimensional window motor, 1″wide × ½″thick × 15″long (up to)	Butyl rubber (BR) PBAA PU–oxygen + fluorine + nitrogen	$\dot{r}_{BR}=0.017G^{0.8}$ (fluorine) × $0.0088G^{0.8}$(oxygen): diffusion limited	y/t: micrometer measurements on regression over slab length	G_{ox}: 1–12 g/cm² s P_c: 2–12 kg/cm² t_b: 3–10 s
3	Nadaud and Baisini (1967)	Gas generator to produce fuel-rich or oxidiser-rich gases over the fuel block (two-dimensional window motor) 9.5 × 70 mm length	PVC and amine-filled plastic		D^n vs t curve fit and subsequent reduction of data	G_{ox}: 20–150g/cm² s P_c: 11–40 kg/cm² t_b: 4 s
4	Schmucker (1971)	Two-dimensional window motor	HNO_3 with toluidine	$\dot{r}=0.02G_{ox}^{0.5}\times P_c^{0.2}$	(1) measuring chamber pressure variation and analysing it; (2) optical examination of the burning grain	G_{ox}: 5–50 g/cm² s P_c: up to 25 kg/cm²
5	Present work	20 mm ID × 40 mm OD × up to 320 mm length cylindrical configuration	Natural rubber + DFCH (varying fraction) with RFNA (84%HNO_3 + 13%N_2O_4 + 3%H_2O	$\dot{r}=0.026G_{ox}^{0.5}$ (85% DFCH) $\dot{r}=0.024G_{ox}^{0.5}$ (75% DFCH) $\dot{r}=0.016G_{ox}^{0.5}$ (50%DFCH)	Weight loss for different burn times; Analysis based on curve fit of integrated regression rate expression	G_{ox}: 5–11 g/cm² s P_c: 4–25 kg/cm² t_b: 5–25 s

temperature towards the increasing oxidiser richness zone. The results of their experiments on hypergolic fuel seem to indicate that $n=0.72\pm0.1$ conforms to the dynamics of turbulent flow.

Schmucker (1971) determined regression rates of nitric acid − 30 percent *p*-toluidiene + 70 percent *p*-aminophenol system by optical and electrical methods, and also by the method of shifting chamber pressures. In the optical method, the engine was test-fired and regressing surface was photographed. The photographs were analysed and the regression rate calculated. The electrical method used a special printed circuit probe embedded in the fuel. There were many leads at set distances in the circuit. The destruction of a lead gives a signal recorded as a step function. This was analysed to give the regression rate. The results of optical and chamber pressure shifting agreed well with one another, but the electrical method seemed to give lower regression rate. These experiments seem to give n of 0.5 (see Table I).

As may be noticed from the examination of the literature there seem to be differing results with regard to the exponent of the mass flux. Some studies seem to conclude that $n=0.5$ while others conclude that $n=0.8$. Breslau (1968) while commenting on the paper by Marxman and Wooldridge (1968) raised a point indicating that flow dynamics may be laminar in cases where $n=0.5$. Marxman and Wooldridge responded by stating that the correlations based on the total mass flux, G, lead to $n\simeq0.8$ whereas those based on oxidiser mass flux, G_{ox}, lead to $n\simeq0.5$. This does not seem to be the accepted position since Barrere and Moutet (1967) as well as Kosdon and Williams (1967) have pointed out that $n=0.5$ could imply either that combustion processes inside the port can be approaching that of

a stirred reactor or that surface reactions modify the regression rate law. Since the question of $n=0.5$ or 0.8 is still uncertain, it was thought that it should be pursued more carefully and this forms the primary motivation for the present work.

THE EXPERIMENTS

Fuel and Oxidiser

Since this study was a part of a development project on hypergolic hybrid rockets, the first task was concerned with the development of hypergolic fuels. The liquid oxidiser was to be a storable one and so RFNA (83–85 percent HNO_3 + 13–11 percent N_2O_4 + rest H_2O) was chosen. Two fuels, namely difurfurylidene acetone (DFA) and difurfurylidene cyclohexanone (DFCH), were developed early in 1973 and they were characterised with respect to ignition delay with RFNA under varying ambient conditions. Some of these properties are summarised in Table II.

TABLE II
Properties of fuels

Property	DFA	DFCH
Colour	Yellow	Yellow
Density	1.18	1.32
Melting point	55 °C	145 °C
Ignition delay (milli secs)	40–45[a]	50–60[b] 60–70[c]

[a] The melted fuel was poured as a sheet and allowed to freeze and this was used for the test.
[b] Powder DFCH form.
[c] Fuel consisting of 75% Powder DFCH and DFA melted (60 °C), mixed and cast into a block.

Since the mechanical properties of these fuels were not expected to be satisfactory, varying proportions of DFCH were incorporated into natural rubber (polyisoprene) in a rubber mill and the cured blocks were used as the fuel in many tests. The amount of DFCH in rubber was varied as 25, 50, 75, and 85 percent. Experiments with these blocks would also bring out the effect of amount of hypergolic component on the regression rate (it should be noted that natural rubber is not hypergolic with RFNA). Experiments of the kind are not available in the literature as yet.

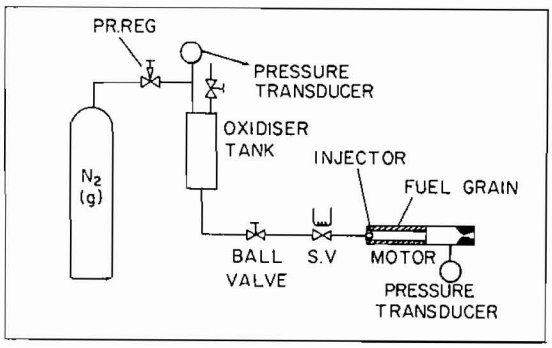

FIGURE 1 Schematic of experimental setup.

Test Details

The schematic of the test setup is shown in Figure 1. Six different motors of 40 mm ID were used. These motors used fuel blocks of 20 mm ID × 40 mm OD and 80 mm length. Any desired length could be obtained by fixing them together. A swirl injector was used to inject RFNA on to the fuel blocks. Mild steel nozzles made with graphite inserts with a wide range of throat diameters varying from 3 mm to 8 mm were kept available for immediate use. During a test, the liquid oxidiser tank pressure and the chamber pressure were recorded and the calibration of the injector was used to deduce the flow rate of the oxidiser. The initial and final weight measurements of fuel block gave the weight loss of fuel. The different motors using identical fuel blocks and oxidiser flow rate were burnt for different times in order to obtain the weight loss versus burn time data. The idea of burning different blocks was essentially to simulate similar conditions of tests for all of them. All the blocks were coated with DFA in order to facilitate smooth ignition. The start and stop of the test were controlled by operating a solenoid valve.

The present tests were conducted over a flow rate range from 15 to 35 g/s implying an initial oxidiser flux of 5–11 g/cm^{2}s, and burn time range of 5–20 s.

The Results

The effect of varying the oxidiser mass flow rate on regression rate is shown in Figure 2. In this figure the weight loss of fuel grain is plotted against burn time for two different oxidiser mass flow rates. Comparison of the slopes of the two fitted straight lines shows that doubling the oxidiser flow rate causes a weight loss increase of about 40 percent. This means

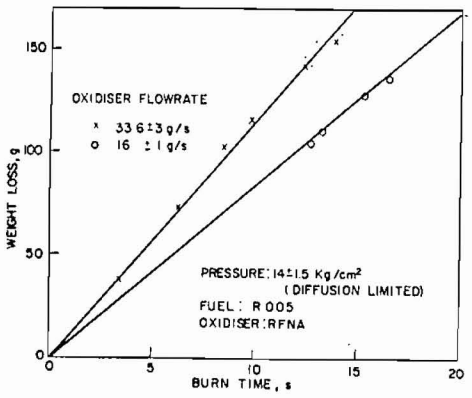

FIGURE 2 Effect of flow rate on regression rate.

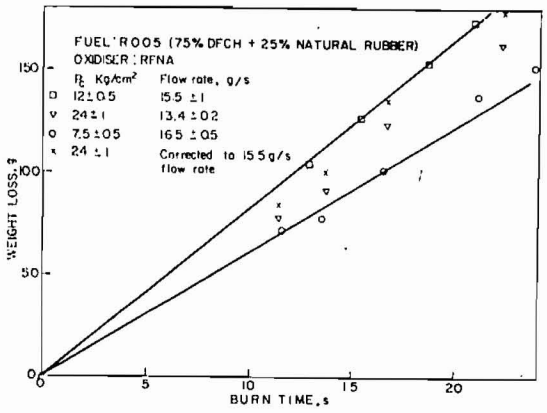

FIGURE 3 Effect of pressure on regression rate.

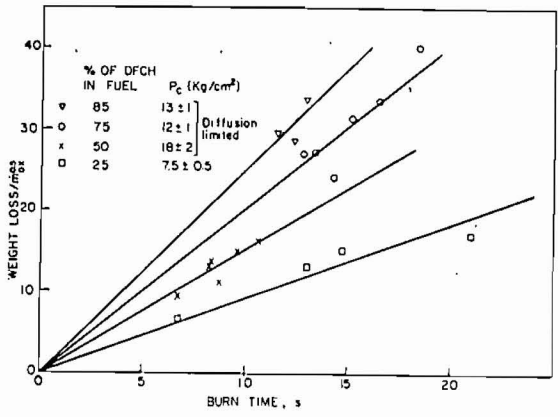

FIGURE 4 Effect of fuel on regression rate.

that the regression rate has a square root dependence on oxidiser mass flux. A more detailed discussion of this aspect is presented later.

Figure 3 shows the effect of pressure on regression rate. Data have been presented for three different pressures. Data points at 24 kg/cm^2 correspond to a lower flow rate (13 g/s) and they have been corrected to an oxidiser flow rate of 16 g/s assuming that weight loss has square root dependence on oxidiser flow rate. It can be seen that the corrected points fall in about the same line corresponding to 12 kg/cm^2 whereas data at 7 kg/cm^2 fall below. Attempts were also made to conduct firings at 3 kg/cm^2, but combustion could not be sustained at this pressure, particularly for the R005 whose composition is 75 percent DFCH +25 percent NR.

The content of hypergolic substance (DFCH) in the fuel also seems to have a strong influence on regression as can be seen from Figure 4. Since data were not available for different DFCH loadings at the same oxidiser flow rate, the points plotted are corrected for mass flow rates by dividing the weight loss with $\dot{m}_{ox}^{0.5}$ since the weight loss has a square root dependence on mass flow rate as mentioned earlier. The points are plotted for DFCH loadings of 85, 75, 50, and 25 percent. All the data except those of 25 percent loading correspond to the diffusion limited regime. The fuel grain with 25 percent DFCH loading is likely to have a higher regression rate at higher pressure. The reduction of mass loss from 75 percent loading to 50 percent loading is about 35 percent. The change in regression rate may be due to the change in mass transfer number B or due to heterogeneous reaction, especially the solid liquid reaction. The heterogeneous reaction is likely to be the more dominant factor since the effect of B is expected to be small (Marxman, 1964) and RFNA and DFCH are known to react in condensed phase significantly.

Calculation of regression rate from the data of weight loss versus time is complicated by the fact that the regression rate is a function of time as well as axial distance. These dependences are the result of changing oxidiser mass flux due to the increasing port area and mass addition. Marxman (1964) has shown that regression rate can be expressed as

$$\rho \dot{r} = a G^{0.8} x^{-0.2} \tag{2}$$

where x is the axial distance from the injector end.

This expression contains two terms: one increasing with axial distance and the other decreasing. From the experiments no discernible trend could be

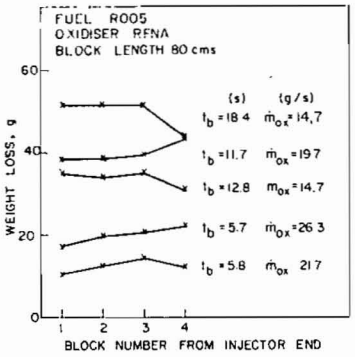

FIGURE 5 Weight loss in individual blocks for different firings.

found in the regression rate with axial distance. As stated earlier the fuel grain is made up of several blocks of 8 cm length joined together. The weight loss of individual blocks in some of the test firings is shown graphically in Figure 5. In most cases any variation of regression rate with axial distance is either too small or not regular enough to be found within the limits of experimental accuracy. Therefore, it was decided that analysis based on overall weight loss data would be satisfactory.

Data Analysis

One of the important points to be noted while conducting data analysis is that the regression rate is not directly measured, but is obtained from the data of weight loss versus time. The dependence of $\dot{r}$ on G will depend to a large extent on the form of the relation used for fitting the data. For example, if we calculate $\dot{r}$ assuming weight loss rate as constant with time, we obtain a result equivalent to $n=0.5$. On the other hand it is equally expedient to treat $n=0.8$, and perform the curve fit to obtain the constant of regression rate law. In many earlier studies (Schmucker, 1971; Wooldridge *et al.*, 1969) a specific value of n was chosen and constants were evaluated. As to whether the data obeys a law with $n=0.5$ better than with $n=0.8$ was not established. In order to do this, one has to integrate the regression rate law keeping the value of n as a parameter and establish the validity of it being 0.5 or 0.8 from a curve fit of the data.

Such an analysis has to be made based on the relation (2) noted above or on a simpler one in which the regression rate dependence on oxidiser mass flux G_{ox} alone is considered. Both analyses will be presented though the simpler analysis will be discussed first. The regression rate relation is

$$\rho \dot{r} = a\, G_{ox}{}^{n} \tag{3}$$

Noting $\dot{r} = d(D/2)/dt$ and $G_{ox} = \dot{m}_{ox}/(\pi D^2/4)$, Eq. (3) can be integrated to give

$$\left(\frac{D}{D_0}\right)^{2n+1} = 1 + \frac{(2n+1)a\dot{m}_{ox}{}^{n} t_b}{(D_0/2)^{2n+1}\pi^n} \tag{4}$$

In terms of W, the weight loss of the fuel grain this can be written as,

$$(1+4W/\pi D_0{}^2 L)^{(n+1/2)} - 1 = (2n+1) \times a\dot{m}_{ox}{}^{n} t_b/\rho\pi^n (D_0/2)^{2n+1} \tag{5}$$

Assigning value for n, a can be calculated for the data of each firing.

The values of a calculated using the above expression for $n=0.5$ and $n=0.8$ in the regime independent of pressure for fuels with 75 percent DFCH loading are given in Table III. The standard deviation of a calculated using $n=0.5$ is 0.00107 (4.6 percent) whereas that with $n=0.8$ is 0.00247 (15.2 percent). The standard deviation with $n=0.5$ is much smaller, and hence an index of 0.5 is taken for correlating all the data. The values of a so obtained for fuels with different DFCH loadings and pressures are given in Table IV.

Now we will examine the variation of O/F with diameter or time. From Eq. (3), we have

$$\dot{m}_f = aA_b\left(\frac{\dot{m}_{ox}}{A_p}\right)^{n}$$

where A_b is the burning area, we obtain the expression for O/F as

$$O/F = \frac{\dot{m}_{ox}}{\dot{m}_f} \sim \frac{\dot{m}_{ox}}{\dot{m}_{ox}{}^{n}(A_b/A_p{}^{n})}.$$

Thus,

$$\frac{(O/F)}{\dot{m}_{ox}{}^{1-n}} \sim \frac{A_p{}^{n}}{A_b} \sim D^{2n-1} \tag{5}$$

This relation implies that if the right value of n is chosen the data points corresponding to different conditions of oxidiser flow rates will fall in a single continuous line on the plot of $(O/F)/\dot{m}_{ox}{}^{1-n}$ versus t_b. One can also expect the curve to have a positive slope if $n>0.5$, and a negative slope if $n<0.5$. These plots for $n=0.5$ and 0.8 are shown in Figures

TABLE III

Values of a calculated in different firings assuming the relation $\dot{r}=aG_{ox}^{n}$

No.	Length of grain (cm)	Weight loss (g)	Burn time (s)	$\dot{m}_{ox}$ (g/s)	a ($n=0.5$)	a ($n=0.8$)
1	32	159.2	11.7	19.5	0.026	0.0181
3	31.1	163.7	15.3	16.47	0.0226	0.0170
4	30.9	135.0	12.8	14.77	0.0236	0.0180
5	30.9	230.5	20.7	15.22	0.0245	0.0199
6	30.9	196.1	18.4	14.67	0.022	0:0195
8	24.6	102.3	10.0	22.50	0.0247	0.0156
11	25.0	110.2	13.3	16.92	0.0238	0.0157
12	24.6	135.6	16.5	16.36	0.0233	0.0167
15	24.2	102.6	8.5	35.29	0.0231	0.0131
16	25.7	141.6	12.4	32.66	0.0231	0.0128
17	24.8	225.4	16.2	30.00	0.0240	0.0186
19	24.5	116.5	9.9	33.33	0.023	0.0133
20	24.7	153.7	14.0	33.00	0.022	0.0129
Results for a:		$n=0.5$	$n=0.8$			
Mean(μ)		0.0233	0.0163			
Standard deviation (σ)		0.00107	0.00247			
σ/μ		0.046	0.152			

TABLE IV

Values of a for different loadings of DFCH and pressures ($n=0.5$)

No.	DFCH	P_2 kg/cm²	a
1	85%	13	0.0259
2	75%	25	0.023
3	75%	7.5	0.0168
4	25%	7.0	0.01095
5	50%	19.0	0.0156

6 and 7 respectively. Only those points which correspond to the diffusion limited regime are shown in the figures.

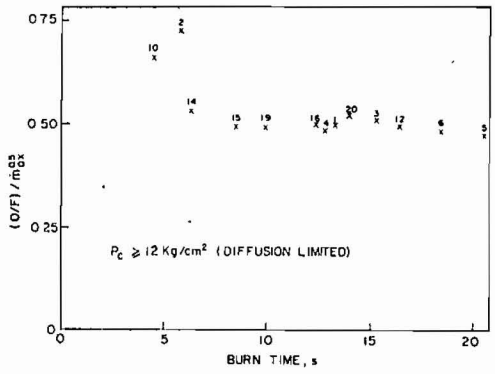

FIGURE 6 Variation of $(O/F)/\dot{m}_{ox}^{0.5}$ with burn time.

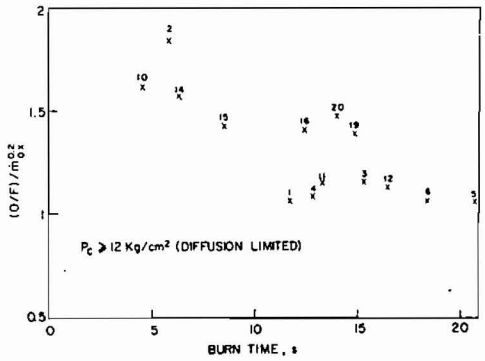

FIGURE 7 Variation of $(O/F)/\dot{m}_{ox}^{0.2}$ with burn time.

Several interesting features can be observed by comparing these two figures. Even a casual look will show that scatter is much larger when $n=0.8$ is used. In Figure 6 where $(O/F)/\sqrt{\dot{m}_{ox}}$ is plotted, most of the points fall on a line parallel to the abscissa whereas in Figure 7, on the other hand, the points seem to indicate two such lines. They in fact correspond to two different flow rates. All the data points numbered less than 12 correspond to oxidiser flow rates ranging from 14 to 12 g/s, whereas points 14–20 correspond to oxidiser flow rates 30–38 g/s. The fact that these two are merged in Figure 6 shows that the right value of n is indeed 0.5. This is also confirmed by the near zero slope of the curve in Figure 6.

In the foregoing analysis, an assumption was made that the regression rate depends on oxidiser mass flux. Boundary layer theory predicts dependence on total mass flux. Such an analysis has also been made using the relation $\dot{r}=aG^n x^{n-1}$. The relation is integrated with respect to x first to get average regression rate over the length of the grain and then it is integrated with respect to time to get the relation between final average diameter and the burn time. The final relation is

$$\int_{D_0}^{Df} \frac{D\,dD}{(1+KD^{2-2n})^{1/(1-n)}-1} = \frac{2\dot{m}_{ox}t_b}{\pi L\rho} \qquad (6)$$

where

$$K=\frac{(1+n)4^n aL^n \dot{m}_{ox}{}^{n-1}}{n\pi^{n-1}},$$

D_f is the final diameter, L = length of the grain.

The final diameter is obtained from weight loss of the fuel grain

$$D_f{}^2 = D_0{}^2 + \frac{4W^{1/2}}{\pi\rho L} \qquad (7)$$

a is evaluated from the expression (6) using an iterative technique. A set of values so obtained is given in Table V for $n=0.5$ and 0.8.

TABLE V

Values of a calculated for different firings using the relation $\dot{r}=aG^n x^{n-1}$

Firing no.	$\dot{m}_{ox}$ (g/s)	a ($n=0.5$)	a ($n=0.8$)
1	19.50	0.132	0.0296
3	16.47	0.117	0.0279
4	14.77	0.121	0.0292
5	15.22	0.125	0.0318
6	14.67	0.122	0.0306
8	22.50	0.110	0.0253
11	16.92	0.102	0.0257
12	16.36	0.103	0.0268
15	35.29	0.106	0.0216
16	32.66	0.104	0.0225
17	30.00	0.130	0.0304
19	33.33	0.106	0.0223
20	33.00	0.100	0.0218
Results for a:	$n=0.5$	$n=0.8$	
Mean(M)	0.114	0.0267	
Standard deviation (σ)	0.0113	0.0038	
σ/μ	0.099	0.142	

Conclusions similar to those of the previous analysis can be drawn from the present one also. The appropriate value of n is obtained when a evaluated becomes independent of $\dot{m}_{ox}$ or burn time. The standard deviation of a in Table V is 0.0113 for $n=0.5$ and 0.0038 for $n=0.8$ respectively and corresponding (σ/μ)'s are 0.099 and 0.142.

Even though σ for $n=0.8$ is lower than for $n=0.5$, the conclusions about the validity of data based on σ cannot be drawn because the basic magnitudes of a are different. The appropriate measure to be considered is the magnitude of σ/μ, the coefficient of variation, a comparison of which leads to the conclusion that the case $n=0.5$ has a lower value of σ/μ and so the value of $n=0.5$ seems to fit the data better. a still seems to be slightly negatively correlated to $\dot{m}_{ox}$, which means that the value of n must be less than 0.5.

The fact that the value of n is near or less than 0.5 does not mean that flow is laminar in the present case. Since the system is hypergolic, heterogeneous reaction, especially the liquid–solid reaction, is likely to control the behaviour of regression. A gas–solid reaction, even if present, will not change the form of relation between the mass flux and regression rate because the transport of oxidiser by diffusion to the surface is similar to convective heat transfer. Therefore, the predominance of liquid–solid heterogeneous reaction is strongly indicated in the results of these experiments.

CONCLUSIONS

Regression rate behaviour in hybrid rocket engines has been experimentally studied using a hypergolic combination of RFNA and rubberised DFCH. Regression rate is found to be pressure dependent only below about 10 atm. The regression rate is also strongly dependent on the content of hypergolic substance (DFCH) in the fuel. This and the fact that the dependence of regression rate on mass flux through the port is less than that predicted by the turbulent boundary layer analysis point to importance of liquid–solid heterogeneous reaction in hypergolic hybrid systems.

NOMENCLATURE

a constant in Eqs. (1), (2) and (3)
A_b burning area
A_p port area

D diameter of grain (cm)
D_0 initial diameter (cm)
D_f final diameter (cm)
G total mass flux through the port (g/cm^2 s)
G_{ox} oxidiser mass flux (g/cm^2 s)
L length of the fuel grain (cm)
$\dot{m}_{ox}$ oxidiser mass flow rate (g/s)
$\dot{m}_f$ mass flow rate of the fuel (g/s)
n index of the mass flux in the expression for regression rate
P pressure (kg/cm^2)
r radius of grain (cm)
$\dot{r}$ regression rate (cm/s)
t_b burning time (s)
W weight loss by the fuel grain during the diring
x axial distance measured from the injector end
ρ density of the fuel grain (g/cm^3)

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