Burning Rates of Viton-Coated Aluminum and Water Mixtures

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Abstract

Burning rates of solid propellant containing Viton-coated aluminum powder and distilled water were examined at pressures up to 75 bar. Two powder sizes were tested, $d_{50} = 60$ nm and $d_{50} = 100$ nm. Both propellants demonstrated two different burning rate regimes when varying the pressure. For pressures lower than 20 bar, burning rates were found to be independent of pressure, reaching ~4 mm/s for both powders. At higher pressures, the burning rate of both propellants followed well the power law behavior, exhibiting relatively high pressure exponents of n=0.7 for the 60 nm propellant, and n=0.81 for the 100 nm propellant.

Nomenclature

- \dot{r} Burning rate [$\frac{mm}{s}$]
- P_{C} Chamber pressure [bar]
- n Pressure exponent
- *a* Burning rate coefficient $\begin{bmatrix} mm/\\ s \cdot bar^n \end{bmatrix}$
- η_{C} Combustion efficiency

1. Introduction

Solid propellants comprised of aluminum powder and water show high energetic theoretical performance and are characterized as environmentally friendly due to their non-toxic combustion products [1-2]. These propellants have therefore been considered for use in space and marine propulsion systems [3], as well as for power supply applications and hydrogen generators [4-5].

Thus far, most studies on the exothermic aluminum-water reaction focused mainly on hydrogen production for different energetic systems [4-8], and did not consider the combustion regime of the reaction, which may be attractive for propulsion applications. In these works, excess water was almost always used in order to enable complete oxidation of the aluminum, as well as to control the reaction temperature. Nevertheless, in recent years, there has been a substantial increase in research on the behavior of Al-water (aluminum-water) solid propellants, which comprises of mixtures with a mass ratio close, or equal to the stoichiometric one $(m_{water}/m_{Al}=1)$ [9-10]. Starting from the late 90's, Al-water propellants were the subject of underwater propulsion system research [11-12], where initial development of underwater engine using aluminum and seawater has been conducted. At the beginning of the 21st century, alongside with the growing awareness to global warming, an increase was noted in works conducted for alternative fuels and propulsion systems, among them motors involving aluminum-water propellants for space vehicles [3, 9-10]. In 2009, solid rocket motor using aluminum and ice (ALICE) propellant was successfully designed and fired by groups from Perdue University and Pennsylvania State University, USA [13-15]. Most studies on Al-water solid propellants investigated the burning rates of compositions of different O/F ratios and various powder diameters [15-20]. In works led by Risha [15, 18-19], pressurized experiments were conducted using compositions containing water and uncoated nano aluminum powders (ALEX), with powders size of d₅₀=38 nm and d_{50} =80 nm. For the 38 nm aluminum powder, their data indicated a pressure exponent of n=0.47 and very high burning rates (8.6 \pm 0.4 cm/s at 43 bar). When a larger powder was used (80 nm), the observed pressure exponent was n=0.27, and burning rates were lower. The work of Georges et al. [20] focused on the coating effects of aluminum powder on the burning rates of Al-water solid propellants under high pressure conditions (up to 345 bar).

Their study examined three different powders: uncoated (ALEX), Viton coated (V-ALEX) and palmitic acid coated (L-ALEX). For the ALEX powder, two burning rate regimes were observed: Below 140 bar, burning rate increases with pressure, with a very high pressure exponent (n=1.04); whereas above that pressure (140 bar) the burning rate surprisingly decays with pressure. Propellants containing V-ALEX powder followed well the power law behavior, noting a pressure exponent of n=0.57. L-ALEX propellants showed no coherent trend with pressure, and the need for further studies and experiments was suggested by the authors.

The effect of powder coating was also investigated by Trowell et al. [21]. They showed that when using a coated powder, an increase is seen in both ignition and on-set temperatures of mixtures containing water and nano powders. The mixture containing L-ALEX powder exhibited relatively high ignition temperature (~150°C), whereas the ALEX mixture presented lower ignition temperature (~80°C). The V-ALEX mixture exhibited an ignition temperature of ~120°C.

Many of the works that measured propellant burning rates used quartz or glass tubes [15-20], which do not burn during the combustion of the strand. This may be undesirable since the thick glass tube does not simulate real motor conditions, and may affect the measured burning rates due to its different thermal properties. Recent works by Wollmark et al. [22-24], introduced the use of consumable strands made from plastic straws which burn as the propellant propagates, leading to more realistic motor conditions. In these experiments, two different powder coatings were investigated: uncoated aluminum powder (ALEX) with d_{50} =80 nm, and stearic acid coated (L-ALEX) with d_{50} =100 nm. Both propellants were tested in pressure conditions varying from 5 bar to 60 bar. ALEX propellants resulted in pressure exponent of n=0.38 with burning rates of ~10 mm/s at 55 bar, while the propellants containing L-ALEX powder exhibited a slightly higher pressure exponent (n=0.47), but slower burning rates, reaching ~6 mm/s at 60 bar.

In order to compare the theoretical performance of Al-water propellants to other compositions, thermochemical analysis was conducted using codes of NASA CEA [25] and PEP [26]. Calculations showed that the specific impulse (I_{sp}) of Al-water propellants can reach 234 s at 70 bar, with a volumetric specific impulse (ρI_{sp}) of 341 g · s/cm³. Both parameters are comparable to those of conventional propellants, as seen in Fig. 1, and exceed those of the toxic hydrazine currently used in many space propulsion systems. Moreover, they are higher than the values obtained by other conventional metal water combustion reactions, under similar conditions (see Fig. 2).



Figure 1: Specific impulse (I_{sp}) and volumetric specific impulse (ρI_{sp}) for different propellant compositions. Chamber pressure is 70 bar, and matched nozzle at sea level is assumed



Figure 2: Specific impulse (I_{sp}) and volumetric specific impulse (ρI_{sp}) for different metal-water compositions. Chamber pressure is 70 bar, and matched nozzle at sea level is assumed

In the presented paper, propellant burning rates of mixtures containing distilled water and Viton coated nano-aluminum is studied and analysed, via the combustion of consumable propellant strands at various operating pressures.

2. Experimental Setup

a. Powder analysis:

Propellant strands were prepared using nano-aluminum powder coated by fluoro-carbonic polymer (Viton). The two powders used in this work (manufactured by APT, Russia) are of median diameters of 60 nm and 100 nm, and are reported to contain 75% and 72% of active aluminum, respectively. Nano-sized powder was chosen due to its high reactivity [27], as well as for the relatively high viscosity of the nano-Al-water mixture, which unlike micon-size powder, does not precipitate to the bottom of the vessel when mixed with water.

Prior to combustion experiments, TGA tests were conducted in order to measure the active aluminum content within the powders, and compare it to the data provided by the manufacturer. The Thermal Gravimetric Analysis (TGA) was carried out in the SDT Q600 device, and consisted of two regimes: A constant heating at 20°C/min up to 1150°C, and then an isotherm for three to four hours. During the first section, at 450-550°C, a drop in mass is noted (Δm_1 in Fig. 3), alongside an indication for a slightly exothermic reaction (Δh_1). These indications were also reported in previous works involving TG/DSC analysis on coated aluminum powders [28-29]. It is believed that the Viton coating reacts exothermically with the oxygen in the air, and releases gaseous products (mainly CO₂ and HF), resulting in a reduction of sample mass (~4-6%), and an increase in heat flow. From that decrease in mass, the Viton mass fraction can be derived using basic calculation.

During the second section of the TG/DSC analysis, mass gains are noted (Δm_2 , Δm_3 in Fig. 3), accompanied by heat releases (Δh_2 , Δh_3). These processes can be attributed to the exothermic reaction of aluminum oxidation into Al₂O₃. Figure 3 illustrates the mass gain and heat release during a typical TGA/DSC test of V-ALEX powder. The active aluminum content was determined by comparing the relative sample mass to the maximal theoretical value calculated for a sample with 100% active aluminum, which is roughly 189% of the original mass.



Figure 3: Typical data from a TGA/DSC test of V-ALEX powder (d₅₀=100 nm)

In addition to the TGA tests, TEM (Transmission Electron Microscopy) imaging was also conducted. The images presented in Fig. 4 show that the particle sizes and coating thicknesses are within the range of the dimensions specified by the manufacturer.



Figure 4: TEM images of V-ALEX powder (a) $d_{50}=60$ nm: full image (b) $d_{50}=60$ nm: zoom in (c) $d_{50}=100$ nm: full image (d) $d_{50}=100$ nm: zoom in

The characteristics of the different powders used in this work are presented in Table 1, including the specifications provided by the manufacturer (APT) and the TGA/DSC and TEM results.

d ₅₀ [nm]		Coating thickness [nm]		Active Al [% mass]	
APT	TEM	APT	TEM	APT	TGA
60	68	5-7	3-6	75	72.3
100	94	5-7	3-6	72	71.6

Table 1: Properties of the Viton coated nano-aluminum powders used in this work

According to the TGA results shown in Table 1, the active aluminum in each powder is in good agreement with the data provided by the manufacturer, with minor differences error for each powder. It can also be seen that powder size and coating thickness measured using TEM analysis are very similar to the values provided by the supplier.

b. Propellant preparation:

During mixing of the aluminum powder with water, a surfactant was added to the mixture (Neodol 91-8, 1.5% with respect to the water mass), in order to form homogeneous slurry and achieve the desired mixture consistency. A stoichiometric mass ratio (aluminum/water = 1:1) was maintained in order to reach complete combustion and maximize the flame temperature. After mixing, the mixture was poured into plastic straws, 7 mm in diameter (as seen in Fig. 5), and the bottom end of each strand was sealed using epoxy in order to prevent mixture spillage. Average length and mass of each strand were 9-10 cm and 5-6 grams, respectively.



Figure 5: Propellant strand before mounted to the test facility

c. Experimental apparatus:

The experimental setup, seen in Fig. 6, is widely described in [22-24] and includes a custom pressure chamber, an inert nitrogen tank and data acquisition system connected to a computer. Before each experiment, the strand is mounted on the stand inside the pressure chamber and its wires are connected to the electric circuits of the data acquisition system. Prior to ignition, nitrogen is flowed into the chamber in order to reach the desired pressure. The strands were ignited by a hot wired igniter made of standard aluminized AP/HTPB propellant charge. The burning rate measurements are obtained by three thin fuse wires passing through the propellant horizontally, in even gaps. As the flame passes each wire, the wire melts and triggers the electrical circuit to open and a voltage drop is noticed in the system. The data from each burn rate experiment is transferred using an acquisition card (with data acquisition frequency of 0.1 s) a to computer software (LabView) for further analysis.



Figure 6: Schematic diagram of the experimental setup

3. Results

Before ignition, the length and mass of each strand was measured, to obtain the propellant density. Figure 7 presents the measured densities of the propellant strands for the two different powder sizes, compared to the theoretical density of aluminum water propellant at stoichiometric ratio (assuming pure aluminum).



It can be seen that all densities were in similar range, and the average values were 1.45 g/cm³ and 1.48 g/cm³ for the $d_{50}=60$ nm and $d_{50}=100$ nm propellants, respectively.

In order to closely examine the combustion process of the Al-water propellant, strand samples were ignited at atmospheric pressure, and the combustion was photographed using a high speed video camera (Phantom V-310) in a frame rate of 400 frames per second. The different stages of the combustion are presented in Fig. 8, for the 60 nm powder propellant.



Figure 8: Combustion stages of a burning V-ALEX propellant ignited at atmospheric pressure ($d_{50} = 60$ nm)

As the flame propagates through the strand, the plastic walls are consumed and the combustion products are falling to its sides (t=6 and 14.9 s in Fig. 8), unlike the ALEX propellant products which kept a solid skeletal form during their strand combustion [22,24]. This may indicate that the combustion products of V-ALEX propellants are more prone to follow streamlines inside the combustion chamber and eject through the nozzle, rather than accumulate as undesirable slag within the motor.

Combustion experiments were also conducted for strands in pressurized environment, and data regarding flame propagation time were recorded using data acquisition system. Data from a typical experiment are presented in Fig. 9, and can indicate the strand burning rate throughout its combustion.



Figure 9: Data from a typical V-ALEX burning rate experiment conducted at 50 bar

As the flame propagates through the strand and reaches the fuse wires, they are melted and a voltage drop is seen. For most experiments, the bottom half of the strand (between the 2^{nd} and 3^{rd} wires) was characterized in a higher burning rate than the top half, probably due to the higher pressure within the chamber, caused by the hydrogen release during propellant combustion propagation. Figure 10 demonstrates the change in pressure during typical strand combustion, and may explain the increased burn rate values seen for the bottom part of the strand in most experiments. When the collected data was analyzed, the experiment operating pressure point was taken as the average pressure throughout strand combustion, as illustrated in Fig. 10.



Figure 10: Change in pressure during a typical V-ALEX burning rate experiment conducted at 50 bar

The results of the different experiments conducted for the propellant strands containing $d_{50}=60$ nm V-ALEX are presented in Fig. 11. The combustion of the propellant strands was characterized by two different regimes: At pressures lower than 20 bar, burning rates were found to be nearly constant at roughly 4.4 mm/s, and independent of chamber pressure. At higher pressures, the burning rate followed well the power-law behavior, with a high pressure exponent of n=0.7 and a moderate burning rate of 8.8 ± 0.2 mm/s at 66 bar. The burn rate equation for this propellant is shown in Eq. 1, where \dot{r} and P_C are measured in [mm/s] and [bar], respectively.

$$P_c > 20 \ bar: \dot{r} = 0.447 \cdot P_c^{0.7} \tag{1}$$

While the power law behavior at pressures starting from 20 bar is not surprising, the somewhat constant (and slightly higher) burning rate at lower pressures is unexpected, and hasn't been previously reported in the literature. This unintuitive finding is unique to the V-ALEX powder propellants, and was not seen in previous studies which used the same apparatus and working procedures with different nano-aluminum powders (ALEX, L-ALEX) [22-24]. The 100 nm V-ALEX propellant, as can be seen in Fig. 12, experienced similar behavior at a similar pressure point. A possible explanation to the constant burning rate at low pressures is partially supported by the experiments conducted at atmospheric conditions. In these tests, it was seen that the hydrogen gas produced during combustion was able to "push" upward and aside layers of ignited propellant, thus removing burning parts of the strand and, hence, reducing the heat transfer from the flame to the unburned propellant surface. At the low pressure regime, the momentum of the produced hydrogen is sufficient to elevate and remove propellant burning layers, thus keeping a constant heat transfer mechanism from the flame to the propellant surface, regardless of the exact operating pressure value resulting in little change in propellant burning rate. At higher pressures, the flame is closer to the propellant surface, and the momentum of the produced hydrogen is not high enough to overcome and remove burning layers. From this pressure point and higher, the Al-water propellant combusts in a similar manner to conventional solid propellants, as it increases its burning rate with increasing pressure, due to the increased heat feedback from the flame to the propellant strand. Although reasonable and supported by observations, the unexpected trend seen at low pressures has to be further studied and analyzed, in order to substantiate and better understand the observed phenomena.



Figure 11: Burning rate measurements of V-ALEX propellants (d₅₀=60 nm) at various pressures

Propellants containing $d_{50}=100$ nm V-ALEX powder demonstrated the same independence of pressure up to 18 bar, noting rates of ~4.1 mm/s at this range. At $P_c=18$ bar, a significant drop in burning rate was seen, to about 2.5 mm/s (more than 30% drop). For higher combustion pressures, the power-law trend was observed throughout the pressure range, with a very high pressure exponent of n=0.81, and a slightly low burning rate of 7.0 ± 0.1 mm/s at 75 bar. The results of the different experiments conducted for the 100 nm powder propellant are presented in Fig. 12, and the observed trend looks similar to that of the propellant with finer V-ALEX powder. The burn rate equation for this propellant is shown in Eq. 2, where \dot{r} and P_c are measured in [mm/s] and [bar], respectively.

$$P_{c} > 18 \ bar: \dot{r} = 0.196 \cdot P_{c}^{0.81} \tag{2}$$



Figure 12: Burning rate measurements of V-ALEX propellants (d₅₀=100 nm) at various pressures

In addition to burning rate tests, combustion efficiency was also measured and calculated, using a TG analysis conducted on the propellant combustion products. Ideally, 100% of the aluminum would oxidize during propellant combustion, and no mass gain will be seen when a small sample of the condensed products will be placed inside the TGA in an oxidizing environment. Any mass gain noted by the TGA, will indicate that the sample includes unreacted aluminum powder within the combustion products. The samples were grinded using a mortar and pestle in order to reach a fine powder of homogeneous propellant combustion products. A sample of 40-50 mg was inserted

into the device and heated up using the same procedure used with the aluminum powders. It is assumed that, if present, the un-oxidized aluminum powder will be oxidized during heating, thus increasing the sample mass. From the relative mass increase, the combustion efficiency can be calculated. It can be seen in Fig. 13, that the efficiency is roughly independent of combustion pressure, and ranges around 85%-95%, indicating that the aluminum had combusted almost fully. It is possible that some of the aluminum had not reacted due to water evaporation during the combustion and, therefore, there is not enough oxidizing species to react with the aluminum. This assumption is supported by the higher combustion efficiencies of the 60 nm propellant compared to those of the 100 nm propellant, since a faster reaction time (which is typical for smaller size particles) will result in decreased water evaporation, thus more powder could react with the surrounding water prior to its vaporization. Future experiments may include mixtures with some excess water, in order to substantiate this hypothesis.



Figure 13: Combustion efficiency of V-ALEX propellants at different pressures

In order to compare the burning rates obtained in this work to those of previous studies, results of the relevant data are plotted in Fig. 14.



Figure 14: Burning rate results (power law trends) from [15, 19-20, 24] and from this work

It can be seen that in this work, as in others ([15, 19]), the burning rate increases as particle diameter decreases. In fact, for operating pressures higher than 18 bar, the burning rate of the 60 nm propellant is 40-60% higher than that

of the 100 nm propellant, for a given pressure point. In general, burning rates obtained in this work are in the same order of magnitude with burning rates obtained in previous works with consumable strands [24], but much lower than those obtained in [15, 19], which used uncoated ALEX powders. The differences in burning rates may also be attributed to the type of strands used: at works [15, 19], the non-consumable strands (quartz or glass tubes) could have increased the burning rate due to the protective nature of the tubes, which decreases heat losses from the flame to the surroundings.

The study by Georges et al. [20] on V-ALEX propellants introduces high pressure combustion experiments, resulting in relatively high burn rate values. The power law trend seen in their work seems to elaborate on data from this work for the V-ALEX 60 nm propellant, suggesting that similar mechanisms control the combustion processes of the similar (but different size) powder propellants.

4. Conclusions

The effect of Viton coated nano-aluminum powder on the burning rate of solid Al-water propellants was investigated in this work. Burning rate experiments using consumable strands were conducted at pressures up to 75 bar. Two different particle sizes were tested, with $d_{50}=60$ nm and $d_{50}=100$ nm. Both propellants demonstrated two burning rate regimes throughout the pressure range: a constant rate of ~4 mm/s at pressures lower than 20 bar; and a power law behavior with large pressure exponents at higher pressures. It is believed that at low pressures, a mechanism which involves the produced hydrogen gas removes layers of burning propellant during combustion. At high pressures, this mechanism is not dominant enough to overcome the conventional heat transfer mechanisms of burning solid propellants, and the burning rate starts to follow the well-known power law relation with operating pressure.

Both propellants exhibit relatively high pressure exponents. This is a less desirable property in terms of combustion instability, but it allows, on the other hand, reaching a broad range of burning rates at reasonable pressures, which could be suitable and attractive for various applications.

The main innovation of this research compared to previous studies is the use of consumable strands, which burn with the propellant while it combusts. This type of combustion may simulate more realistically the conditions inside a rocket motor, thus making this work an important tool for designing and analyzing Al-water motors.

Previous studies working with consumable strands [22-24] reported large amounts of slag (combustion products) remaining on the strand after combustion. This can greatly affect the performance, and might damage motor insulation or worse, result in motor failure due to nozzle blockage. The V-ALEX propellants seen in this work, however, ejected finer and less bulky combustion products, which will be less inclined to form undesirable slag inside a rocket motor. This will, of course, increase the expected performance, but perhaps more importantly – will allow safer and more reliable motor designs.

The present study focuses, naturally, on Al-water propellants for solid rocket motors. However, the basic Al-water combustion properties may be interesting and attractive for other applications. The hydrogen released during combustion could be directed to a wide variety of power and energetic systems, which require clean, non-polluting and particle-free supply of the highly energetic fuel.

Further research still has to be performed in this growing field of aluminum-water combustion. Properties like sensitivity to initial temperature, aging phenomena, and safety aspects, should all be addressed as well, in order to better characterize this novel propellant before commercial use is considered.

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