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Effect of Coated Nano-Aluminum Powders on the Agglomeration in Solid Propellant

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Abstract

Coated nano-aluminum particles are added to composite aluminized solid propellant in order to investigate their effect on the agglomeration phenomenon. Two of the powders which are considered are V-ALEX, a fluoropolymer (Viton) coated nano-aluminum, and L-ALEX, a stearic acid coated nano-aluminum. It is believed that a thin layer of coating should protect the aluminum core from premature oxidation, and that when this layer is removed during combustion, aluminum ignition is faster and more prominent.

This paper investigates the effect of changing 5% propellant mass from micron aluminum to nano-powder. ALEX, V-ALEX and L ALEX powders are considered, in propellant samples containing 20% HTPB, 65% AP and 15% aluminum. Propellant strands were burned inside a windowed pressure chamber, allowing measurements of the agglomerates size and number using high-speed photography.

The collected data indicates that propellants containing 5% nano-Al exhibit reduced agglomeration, in terms of both number and volume. The reason is presumed to be the lower ignition time and temperature of the nano-powders, which result in aluminum ignition prior to aggregating into agglomerates. Propellants containing V-ALEX and L-ALEX powders experienced the most profound reduction, with the reasons mainly attributed to the protective coating layers, but also to the increased burning rate of these propellants. It can be concluded that by replacing some of the micron-aluminum with coated nano-aluminum in solid propellants, it is possible to significantly reduce the propulsive losses due to agglomeration

1. Introduction

Aluminum powder is generally added to composite solid propellants in order to increase their performance, due to elevated combustion temperature and higher propellant density. Additionally, aluminum powder inhibits combustion instabilities developed during propellant combustion, due to acoustic damping of the condensed-phase particles. Nevertheless, during combustion of aluminized solid propellants, aluminum particles accumulate and form much larger particles, i.e. agglomerates. Agglomeration may cause loss of potential chemical energy due to incomplete combustion; two-phase flow losses in the nozzle resulting from large particles in the exhaust gases; and accumulation of slag in the motor [1].

The agglomeration phenomena is considered to be a very complex one, incorporating issues such as multi-stage kinetics, phase transformations, significant temperature gradients and heat transfer, two- and even three-phase flow, surface and micro-surface forces, as well as other subjects related to “classic” propellant combustion such as pressure, burning rate, mass/volume loading and others. For these reasons, many attempts have been made in order to model agglomeration, with most works using different assumptions allowing some simplification of the problem.

One of the earlier and well known models is the “Pocket model” by Crump and Price [2], which takes the coarse AP particles as a boundary to form pockets of free volume between them, with the aluminum particles aggregating and agglomerating inside. The model presumes that the agglomerate size is affected by the pocket size, and was later elaborated and further detailed by Beckstead [3] and by Cohen [4-5]. Gany and Caveny [6] developed a model assuming that aluminum particles accumulate in a mobile thin layer on the propellant surface. The un-ignited aluminum particles accumulate and gather as the layer regresses, to form a large aggregate which then ignites, as illustrated in Fig. 1 [1]. Further works by Yavor et al. [7-8] extended this model to include elements from a modified pocket model, and were able to predict the agglomerate size distribution for a given composition and operating pressure, as seen in Fig. 2 [1].

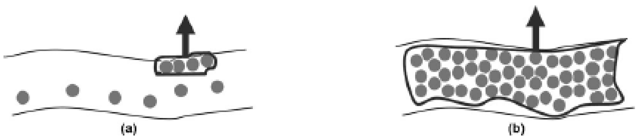

Figure 1: Accumulation of aluminum particles within the mobile layer at ignition. In (a), the layer is partly filled, resulting in a small agglomerate. In (b), the mobile layer is fully saturated, resulting in a large agglomerate [1]

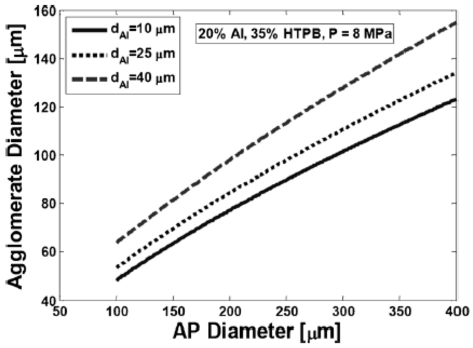

Figure 2: Predicted average agglomerate diameter vs. AP particle size for a propellant containing 20% Al and 35% HTPB at P=8 MPa, for different Al particle sizes [1]

Reducing agglomeration has been, and still is, one of the key motivations to studies in this field. Coated aluminum particles had been suggested by several scientists [9-10], with the coating providing (in some cases) extra exothermic source which accelerates particle ignition. It is believed that for some cases, the coating also improves the mechanical properties of the surrounding polymer matrix and the aging characteristics of the powders [11]. An additional technique to reduce agglomeration is using aluminum nano-powders (diameter of 50 nm – 200 nm) in solid propellants [12-14]. The oxidation onset temperature of aluminum nano-powders is about 100-200°C lower than the aluminum melting point (660°C) [15], resulting in faster ignition and shorter burning time, which leads to higher burning rates and reduced two-phase-flow losses compared to propellants containing micron-aluminum. De Luca et al. [16] analyzed the aggregation and agglomeration of aluminum particles in composite propellants, showing that burning rates were significantly faster as nano-Al fraction was increased within the compound. The high-speed images showed that compared to micron-Al, the nano-Al combustion occurs within a thin and luminous layer adjacent to the burning surface, and very little agglomeration is observed.

Despite the significant benefits of aluminum nano-powders, it still poses several disadvantages, such as higher cost, lower active aluminum content, and increased mixture viscosity. Shalom et al. [17] measured the viscosity of the uncured propellant and found that it increases with the nano-Al content, where a compound containing 12% nano-Al could not mix homogenously.

Studies conducted on the characteristics of coated nano-Al powders [15, 18] showed that the Al particles coated with an organic layer had higher combustion enthalpy compared to standard uncoated nano-Al powder (ALEX, aluminum nano-metric particles produced by electrical explosion method. Hence – ALuminum EXplosion). Dossi et al. [19] investigated three types of nano-Al powders (50 nm ALEX, 100 nm ALEX and 100 nm ALEX coated by stearic acid), and showed a reduction in ignition temperature when stearic acid coated nano-Al was used, as well as 60-85% increase in burning rate of AP/HTPB/AL propellant containing coated nano powder. In another study [20], it was found that for a propellant that contained only stearic acid coated nano-Al powder, the regression rate was doubled and the pressure sensitivity was increased, compared to the micron-Al. The agglomeration of the different propellants was measured at pressures of 1-2 MPa, and a decrease in agglomerate size was noted for propellants containing even small fraction of stearic acid coated nano-Al.

The current work investigates the agglomeration phenomena of different nano-aluminum powders in solid HTPB/AP/Al propellants at various operating pressures. The powders are ALEX, uncoated nano-aluminum; V-ALEX, a fluoropolymer (Viton) coated nano-aluminum; and L-ALEX, a stearic acid coated nano-aluminum. The purpose is to examine whether the Viton and the stearic acid coatings of the nano powders improve the agglomeration phenomena, compared to the micron-Al and the uncoated nano-Al. The work further extends a previous study [21], which was conducted with similar powders but at relatively low pressures.

2. Experimental method

2.1 Materials

Since this study is the extension of a previous research [21] at elevated pressures, compositions of the different solid propellant samples are similar. The compound includes 18% HTPB binder + 2% cross linker (IPDI), 65% AP oxidizer (with ratio of 4:1 coarse to fine AP) and 15% aluminum powder. The baseline composition contains 15% of 6 µm Al powder and the other compositions contained 10% of 6 µm Al powder and 5% nano-Al powder, as listed in Table 1.

Table 1: Propellant composition

Material	Function	Size	Mass Fraction [%]
HTPB	Fuel binder	-	18
IPDI	Cross-linker	-	2
AP	Oxidizer	400 µm	52
		90 µm	13
Aluminum powder	Metalic fuel	6 µm	10 / 15
		50-70 nm	5 / -

Nano powders used in this research were delivered from APT (Advanced Powder Technologies), and include the three powders listed in Table 2. TGA/DSC analysis of all powders in air atmosphere (taken with SDT-Q600 device) are presented in Fig. 3 [22]. Rapid ignition is observed for all powders at ~600°C, a trend which is consistent with results obtained by Lerner et al. [23] for similar powders. Active aluminum content was derived from the TGA [24] and is presented in Table 2, together with TEM analysis which determines the actual coating thickness of each powder.

Table 2: Propellant composition ingredients

Powder	d_{50} [nm]	Active Al content [% mass]		Coating	Coating thickness [nm]	
		supplier	TGA		Supplier	TEM
ALEX	50-70	88	83.5	None (natural Al_2O_3)	3-5	2-6
L- ALEX	50-70	94	93.2	Stearic acid ($C_{18}H_{36}O_2$)	3-5	2-5
V- ALEX	50-70	75	72.3	Viton (~$C_{14}H_{11}F_{17}$)	5-7	3-6

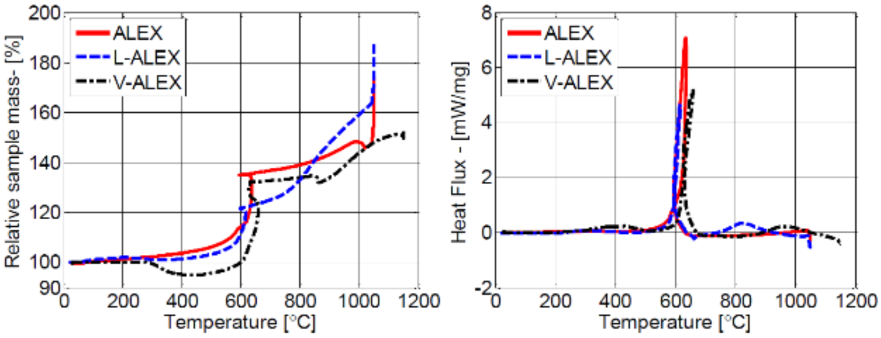

Figure 3: TGA (left) and DSC (right) data in air atmosphere for three nano-aluminum powders (ALEX, L-ALEX and V-ALEX [22])

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2.2 Strand preparation

Strand propellants were manually prepared, with particles gradually added to the HTPB thick binder in a descending particle size order – first the coarse AP (400 µm), then the thinner AP (90 µm), followed by the micro-Al (6 µm) and the nano-Al. After all solids were added, the cross-linker (IPDI) was poured. In every step, the mixture was stirred well for several minutes until uniform texture was achieved, as can be seen in Fig. 4. The propellant was then casted into 5x5x80 mm^3 molds and placed to cure for two weeks at room temperature, as shown in Fig. 5. After the strands were cured, they were cut to 15-mm-long strands, which were later used in the combustion chamber. The strands density was measured and found to be uniform and repeatable among the different samples, with the measured values matching well (within 5%) the theoretical density (1.6354 g/cm^3 – taken from ProPEP [25]). X-ray micro-CT (“Easy-Tom 150” of RX Solutions) was used to acquire images of the different propellants, with a typical image presented in Fig. 6. The measured porosity ranged between 5-7%, corresponding well to the above- mentioned density values.

Figure 4: Mixing process of the AP/HTPB/Al solid propellant composition

Figure 5: Propellant mixture casted into the mold

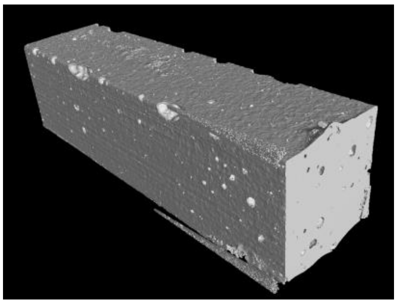

Figure 6: X-ray micro-CT scan of a typical aluminized solid propellants containing L-ALEX powder

2.3 Experimental setup

The experimental setup illustrated in Fig. 7 includes of a ~ 1 liter nitrogen-pressurized chamber with a 25-mm-thick glass window, enabling the visualization and photography of the strand combustion. The strand was installed on a custom-made Teflon strand holder within the pressure chamber, and nitrogen was injected in order to reach the required chamber pressure. The propellant surface was ignited using a filament and a 10 W power supply that was used to heat the filament to the propellant ignition point. A Chronos 2.1 high-speed camera was set up facing the window of the pressure chamber. The focus was on the burning surface area and the area above it, in order to examine the quantity and the size of the emitted agglomerates. The high-speed camera provided images at a frame rate of 5000-5400 frames per second, with exposure time of 15 µs.

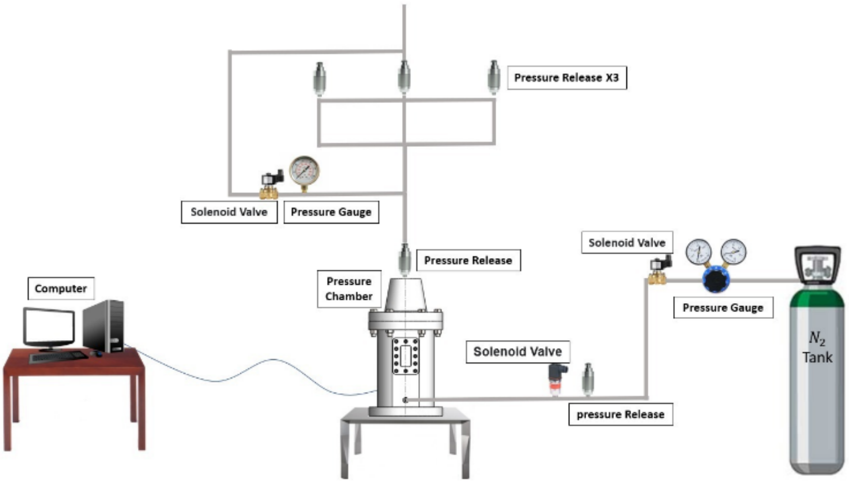

Figure 7: Schematic illustration of the experimental setup used for high-speed imaging experiments

The images taken during combustion experiments were carefully analysed, with measurements of particles size, amount and flux. Single particles were detected, identified and measured, as can be seen in Fig. 8.

Figure 8: A typical image of the propellant combustion process taken with Chronos 2.1 camera at 5400 frames per second. The photographed strand includes only micro-Al powder and burns at a pressure of 4 MPa

The calibration of the measurements to real size was performed using an image of the propellant before ignition, for which its dimensions were known (the original strand dimensions). Pixel size was 13-15 µm, depending on the calibration.

The regression rate was obtained from two different frames with known time tags, and by measuring the distance between the locations of the flame front in these two frames. The calculated burning rate values may include large uncertainties, due to the fact that the photographed images encompass only two dimensions, while the actual combustion process is three dimensional.

3. Results and discussion

In total, twelve tests were conducted for each propellant composition, at three different operating pressures: 2, 4.2 and 5 MPa, in an attempt to find the effect of the powder on the agglomeration phenomena. Several frames were analysed for each test, in order to reduce statistical uncertainties. The agglomerate measurement resolution is limited by the size of a single pixel of the recorded images, and agglomerates smaller than 25 µm were difficult to detect, thus for smaller sizes no agglomeration is assumed.

Figure 9 presents the measured burn rate values for all the tests, together with data collected from [21] for the lower pressures. The micron powder exhibits the lowest burn rate at all pressures. Among the different nano-powders, there is no clear trend. The goal of the burn rate analysis is to examine its effect on the agglomeration, and determine whether this effect is only pressure-dependent or other factors should also be taken into account.

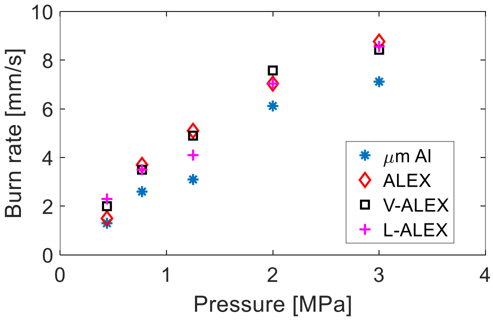

Figure 9: Burn rate vs. Pressure of the different propellants used in this study

The agglomerates quantity for each test is presented in Fig. 10, together with data collected from [21] for the lower pressures. It can be seen that the number of agglomerates decreases with the increase in pressure. As expected, the micron powder presents the largest amount of agglomerates, while the ALEX and L-ALEX produce slightly more agglomerate quantity than the V-ALEX. It is important to note that the main relevance and significance of these results is comparison between the various powders, and not the exact agglomerate quantity which depends on the sample size and number of analysed images.

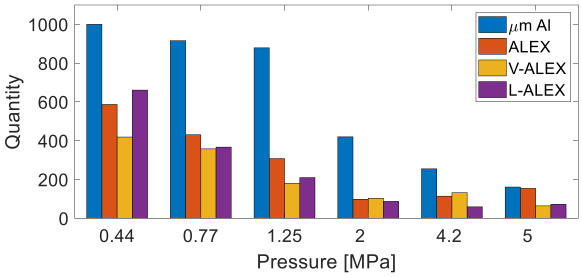

Figure 10: Agglomerates quantity vs. operating pressure

The effective diameter of each agglomerate was derived by the measured area and circumference. Using this equivalent diameter, the volume was approximated under the assumption of spherically shaped particles. Figure 11 shows the comparison of the calculated total agglomerates volume, including data collected from [21] for the lower pressures. The propellant with micro-Al only exhibited the highest volume, as expected, while addition of 5% V-ALEX produced the lowest volume. Similarly to the agglomerate quantity, the volume decreases with pressure increase.

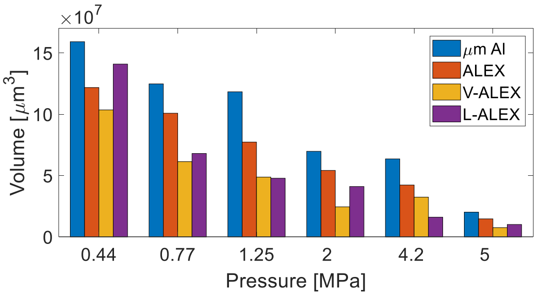

Figure 11: Agglomerates total volume vs. operating pressure

Figure 12 presents the volume fractions relative to the total volume obtained for the reference propellant containing only micron-Al, to emphasize the effect of nano-powders addition. Again, data collected from [21] for the lower pressures is also presented.

The results clearly show that the use of nano-powders reduces the agglomeration phenomenon in both quantity and volume, and therefore, is expected to produce better performance characteristics. The coated nano-powders showed an additional improvement compared to the uncoated ALEX, with the V-ALEX showing slightly better characteristics than L-ALEX in some cases, and vice versa in others.

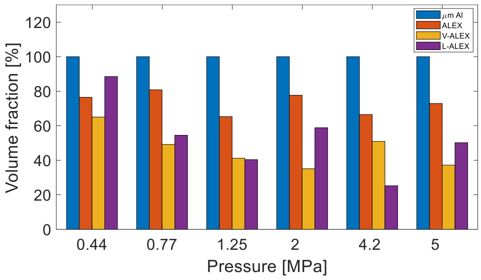

Figure 12: Agglomerates total volume (% of micron) vs. operating pressure

Figure 13 describes the size distributions at 4.2 MPa for each aluminum powder. Agglomerate diameter values shown along the horizontal axis represent the mean diameter for each bin.

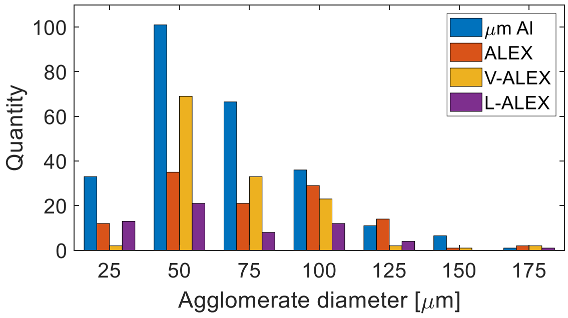

Figure 13: Agglomerate diameter distribution (quantity) for different aluminized propellants, at P = 4.2 MPa

As can be seen, the majority of the agglomerates are concentrated at diameters up to 75 µm. The presence of nano-powders reduces the quantity of agglomerates with a mean diameter lower than 100 µm. There is a visible trend in the agglomerate size distribution between the nano-powders, with the addition of L-ALEX resulting in the lowest amount of agglomerates for almost all particle sizes. The addition of V-ALEX results in slightly higher amount of agglomerates at smaller diameters. This trend is different than the one seen in lower pressures [21], with V-ALEX producing lower amount of agglomerates throughout all diameters. Volume size distribution is presented in Fig. 14, showing similar trends. It is important to note that for the larger diameter bins, even small amounts of agglomerates result in relatively high volume, as observed for the propellants containing ALEX and L-ALEX.

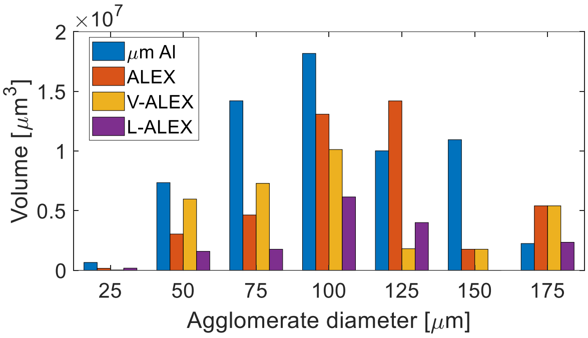

Figure 14: Agglomerate diameter distribution (volume) for different aluminized propellants, at P = 4.2 MPa

Assuming that agglomerates are generated solely from the micron Al particles, propellants containing 15% micron aluminum should generate 50% more agglomerates than a propellant containing 10% micron aluminum. Multiplying the results of the nano powder propellants (which contain 10% micro-Al) by 1.5, and compare the results to the micron-Al propellant (which contains 15% micro-Al), should reveal whether there is another mechanism reducing agglomeration, apart from the total amount of micro-Al powder. For the data presented in Fig. 14, it can be noted that the total volume of agglomerates in the nano-powder propellants (compared to the micro-Al propellant) reduces in 34%, 49% and 75% for the ALEX, V-ALEX and L-ALEX propellants, respectively. It is believed that the lower ignition temperature of nano-powders results in shorter aluminum residence time in the “pocket”, with the micro-particles igniting before aggregating to agglomerates.

As for the different nano-powders used in this work – propellants with coated particles had clearly experienced reduced agglomeration compared to the propellant containing ALEX powder. This observation indicates that there is an influence of the powder coating on the agglomeration processes.

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Previous works studying compounds containing aluminum and fluoro-polymers [26-28] state that inter-particle reactions between the aluminum core and fluorine-containing decomposition species promote the degradation of protective alumina shell and shorten aluminum ignition delay time. Similarly, it is assumed that in the current study, Viton coating decomposes during heating and its thickness decreases, with decomposition species that can serve as reaction precursors to aluminum oxidation [21]. A similar mechanism is presumed to exist for the stearic acid coating, though in this case the decomposition products hardly contain oxidizing species [21]. Nevertheless, aluminum ignition delay is still expected to be shorter than for uncoated ALEX, and in some cases even shorter than V-ALEX, as observed in a research studying nano-aluminum-water reactions [29]. An additional reason for the increased agglomeration reduction in propellants containing L-ALEX, is the thinner coating thickness compared to that of V-ALEX, together with its higher active aluminum content. The absence of a thick alumina layer increases the heating rate to the aluminum core, as well as enables the oxidizing species to diffuse and directly reach the unreacted aluminum – which results in early ignition, relative to the uncoated ALEX.

4. Conclusion

The effect of coated nano-aluminum powder on the agglomeration phenomena in HTPB/AP aluminized solid propellants is investigated. Four different types of powders were used in the tests: micron-aluminum, nano-aluminum, stearic-acid coated nano-aluminum and Viton coated nano-aluminum. A windowed pressure chamber was used for combustion experiments, with various operating pressures. The combustion process was photographed using a high-speed camera, to capture the burning surface of the propellant and the ejected ignited aluminum agglomerates.

The images were analysed to measure the number of particles and their diameters, thus determine the size distribution, as well as the burning rate for each combustion experiment.

The total volume and quantity distribution of ejected agglomerates were measured and analysed for the different propellant compositions at various pressures. Agglomerates quantity and volume results showed that the number of agglomerates decreases with the increase in pressure, and that propellant containing only micron-Al presented the largest amount of agglomerates, while inclusion of L-ALEX to the propellant presented the smallest quantity, followed by the inclusion of V-ALEX.

The addition of nano-powders to propellants showed significant improvement in the agglomeration phenomena compared to the use of micron powder only. The ignition time and temperature are lower for nano-powders, resulting in shorter residence time in the “pocket”, which leads to reduced aggregation and agglomeration. The coated nano-powders exhibited higher reduction in agglomeration compared to the uncoated ALEX, with the L-ALEX showing slightly better performance than the V-ALEX, possibly due to its thinner coating thickness, and its higher active aluminum content. For V-ALEX, the reduction is much attributed to the fluorine containing pyrolysis products of the Viton, and their exothermic reaction with pure aluminum, which promote particle ignition and reduce aluminum residence time. Additionally, the absence of a thick alumina layer enables early ignition and a higher heating rate to the aluminum particle, relative to the uncoated ALEX.

Despite the lower agglomerate volume and quantity in the propellants with nano-Al powder, in some cases they exhibited higher volume fraction of large agglomerates, relative to the micron powder only. This should be further studied for a wider database of analysed images, but may also be attributed to possible non-uniform distribution due to the manual propellant mixing process.

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