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Laser Ignition and Combustion Study of KNO3-Sorbitol based Solid Propellant

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Abstract

KNO_3-Sorbitol (KNSB) is a simple, cheap and safe solid propellant that is used extensively in the student and amateur rocketry communities. During Ballistic Evaluation Motor (BEM) tests with different propellant compositions it was found that ignition was not achieved with fine KNO_3. This prompted the design of a laser ignition study to investigate the KNSB combustion behaviour. Propellant samples with varying compositions were ignited with a 1064 nm laser. Samples with a fine particle distribution and surfactant showed a different flame structure, and one sample extinguished. Findings were used to improve the fine composition, which led to successful firings.

1. Introduction

The potassium Nitrate (KNO_3)- Sorbitol ($C_6H_{12}O_6$) propellant is used widely in the amateur and student rocketry community as it has significant benefits over other, more commercially viable, propellants. Examples of research on KNSB and KNO_3-sugar propellants in general, are the extensive work by Nakka[10] or the thesis work by Gudnasson.[3] The propellant has also been extensively used by Delft Aerospace Rocket Engineering (DARE), a student society connected to Delft University of Technology (TU Delft). This culminated in the European altitude record in 2009 (12.3 km) for student rockets. To further improve the reliability and performance of KNSB propellant a MSc thesis project was completed,[11] which focused on improving the propellant formulation and determination of the propellant ballistic properties.

In KNO_3-sugar propellants the oxidiser crystals are suspended in a sugar matrix which acts as both binder and fuel. KNSB propellant, similar to Ammonium Perchlorate based propellants, exhibits increased regression rates for smaller oxidiser particle size distributions (PSD). During the study both fine (mean 53 ± 30.7 μm) and coarse (mean 411.4 ± 30.7 μm) KNO_3 was used to create two different KNSB compositions. A progressive burning BEM, developed for this thesis project, was used to determine the propellant ballistic properties such as combustion quality, c^*, nozzle performance, C_f, and regression speed as function of propellant composition, chamber pressure and firing temperature. The tested propellant, its formulation and BEM ignition environment are described in section 2 of this paper.

During these tests it was found that under similar conditions, ignition was not achieved with fine KNO_3, while the coarse composition did ignite. To investigate this, a root cause analysis (RCA) was completed which identified several contributing factors and two likely causes for the observed failures. This investigation is described in section 3.

To study the ignition and combustion behaviour in more detail and establish a final root cause, a laser ignition experiment was performed. Laser ignition sources have been used extensively to study the ignition behaviour of solid propellants as it allows the delivery of fixed radiant fluxes to the propellant sample.[5] The design of this experiment is described in section 4. The results of the laser ignition test can be found in section 5 and will be discussed in section 6. Conclusions as well as recommendations will be presented in section section 7. Lastly, in section section 8 acknowledgements are provided.

2. Potassium Nitrate-Sorbitol Propellant

The propellant used in this study consisted of Potassium Nitrate and Sorbitol with a 65/35 ratio by mass which is equal to an O/F ratio of 1.857. The adiabatic flame temperature is 1600 K at 6.895 MPa (1000 Psi). The theoretical Isp at this chamber pressure is 153 s at sea level conditions. However, I_{sp} performance is significantly reduced by the large

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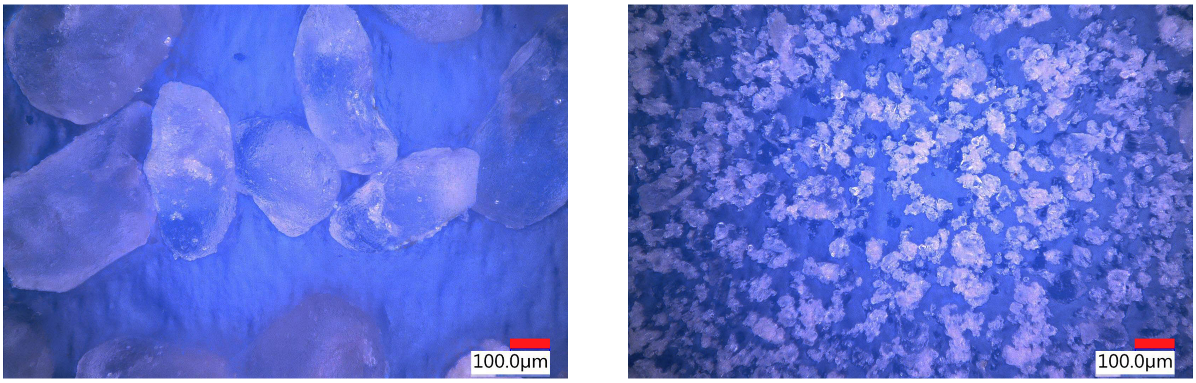
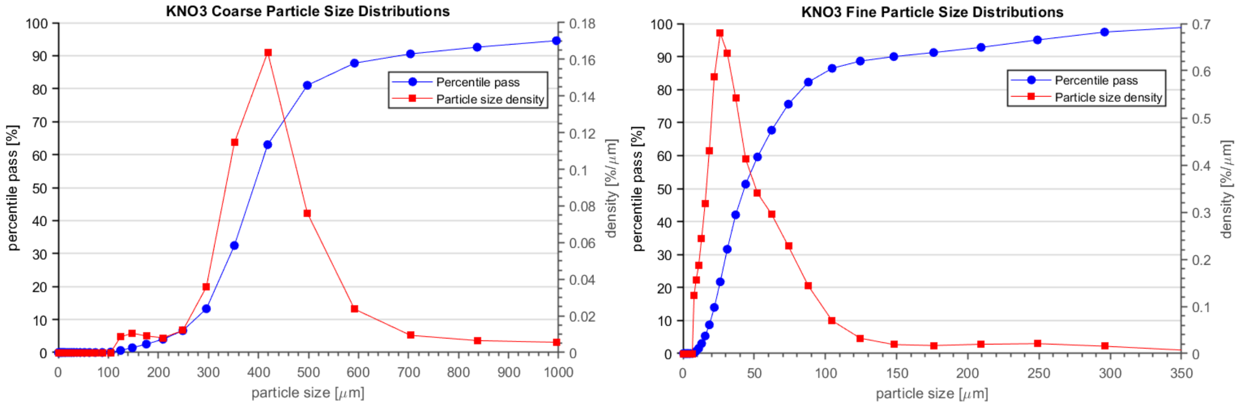


(a) Coarse KNO_3 microscope image.

(b) Fine KNO_3 microscope image.



(c) Coarse KNO_3 PSD.

(d) Fine KNO_3 PSD.

Figure 1: Coarse and fine KNO_3 powders.

amount of condensed phase products in the form of K_2CO_3 which can be in excess of 40% by mass of the exhaust products. With the contribution of condensed phase losses, the measured sea level Isp performance is typically up to 120-130 s.

The propellant is made by heating a KNO_3-Sorbitol powder mixture to above the melting point of sorbitol (110 °C) and casting the mixture into the desirable shape. The propellant remains pliable until 24 hours after casting. Once cured it forms hard and brittle grains which are susceptible to moisture and mechanical impact but can otherwise be stored for several months. Especially Sorbitol is very sensitive to moisture with a critical relative humidity (CRH) around 60%. Thus, the moisture content needs to be specifically controlled during production. Due to its brittle properties and relatively low combustion temperature most KNSB motors are used in non-case bonded BATES configurations.

2.1 Formulation

The KNSB propellant in this study consisted of industrial grade ingredients. Several ingredient and propellant properties are provided in table 1. Microscope images are shown in Figure 1a and Figure 1b. Particle size distributions, as determined with a Microtrac S3500 laser diffraction analyser, are shown in Figure 1c and Figure 1d. To improve the rheological properties of the fine composition, Sodium Laureth sulfate was added as a surfactant (78.9 mg per 100 g of dry propellant mix, σ = 1.6 mg). This improved the viscosity so that also the fine composition was pourable into the desired grain geometry.

KNSB propellant is produced in 9 steps. The ingredients are weighed to batches of 2 [kg]± 1 [g] and thoroughly mixed. Subsequently the premix is heated on an induction hotplate while being stirred until it reaches 125±2 °C. To remove any remaining moisture, which can cause propellant defects and can significantly reduce propellant density, a vacuum is applied (<10 mbar, 8 minutes). Afterwards the propellant is reheated to 125±2 °C and cast into grains. The propellant is allowed to cure for 24 hours to assure the grains have set. The mandrel and tooling is removed. Finally the grains are cleaned of any release agent (bearing grease) and inspected for defects. Grains are stored in sealed plastic bags at dry indoor conditions prior to motor installation and firing.

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Table 1: Properties of the ingredients of the KNO3/Sorbitol propellant

Property	Symbol	unit	KNO3	Sorbitol
Density	ρ_s	kg/m^3	2109[6]	1489[2]- 1542[6]
Decomposition temperature	T_d	°C	400 ($KNO_3 \longrightarrow KNO_2 + O$)[6]	250 (with O_2)[8]
Temperature of boiling	T_b	°C	NA	295[6]
Temperature of fusion	T_f	°C	337[6]	95-111[6]
Specific heat at constant pressure	C_{p_c}	kJ/kgK	0.9535[6]	1.213-1.325[2, 12]
Molar mass	M_{mol}	g/mol	101.103[6]	182.171[6]
Volumetric thermal expansion coefficient	α	1/K	$228\cdot10^{-6}$[7]	-
Critical relative humidity	CRH @20°C	%	93.7[13]	60[1]

2.2 Ignition Characteristics

The BEM design of this thesis project consists of a progressively burning grain with a net explosive mass of around 758 g. The nozzle geometry is variable between tests and adapted for different propellant types and design operating pressures. The grain is inhibited at both ends of the grain with 1.5 mm thick cardboard disks and high temperature silicone glue. The outside of the grain is inhibited by a bonded cardboard tube. The BEM design is shown in Figure 2. The BEM is ignited using a two stage igniter which comprises of an electric squib and a small (0.8-1) g black powder charge (Swiss no.2) mounted in a 25 mm M12 bolt at the head end of the BEM. The grains are coated with an ignition primer which consists of a nitrocellulose (NTC) and black powder (BP) mixture (0.25 g NTC /2 g BP dissolved in acetone) which is liberally applied on the grain port (~2 g per grain).

Thermal events for KNSB propellant are the melting point of sorbitol at 100-110 °C, the boiling point of sorbitol at 295 °C, melting temperature of KNO_3 at 337 °C and decomposition of $KNO_3 \longrightarrow KNO_2 + O$ at 400 °C.[6] Reactions with atmospheric oxygen could introduce additional energy into the system but are only likely to occur in the gaseous phase, beyond the boiling point of sorbitol. The adiabatic flame temperature at ambient pressure is 1167 °C, but due to thermal losses the flame temperature during ignition can range from 400 to 1167°C. From these values it is expected that thermal runaway, as criterion for ignition, will occur between 350 and 400 °C. By comparison the ignition of nitrocellulose and black powder occurs around 150-170 °C.[4]

3. Root Cause Analysis

16 BEM tests were conducted between 9-09-2018 and 20-10-2018. All tests of the fine composition (9) resulted in misfires, while the coarse composition did ignite, even though the conditions were similar. In the moment of igniter firing, the motor builds up pressure as the ignition charge is consumed. During successful BEM firings, the propellant then starts combustion 1-2 seconds after firing. For misfires this first pressure spike is also seen but ignition of the main propellant charge fails. Further observations are:

1. Post-test inspection showed that the primary ignition charge and all ignition primer were fully consumed. The propellant looked slightly caramelised but clean and showed no other signs of degradation.
2. Surface quality of the grains as cast are similar to plastic, very smooth and generally flawless.
3. Changes in the ignition primer composition to a titanium (Ti) augmented mixture (0.25 g NTC/2 g BP /0.3 g KNO_3 /0.7 g Ti) did not significantly improve the ignition delay or solve the misfires.
4. Open air test of the igniter together with the motor geometry (refer to figure 2) shows that the impingement point of the black powder and ignition flame likely occurs towards the nozzle end of the grain.
5. On several grains that were not fired, the primer coating was found to detach from the smooth propellant surface. This was reinforced by the evaporating acetone which caused the coating to shrink resulting in cracks in the primer surface.

Several mechanisms were proposed and discarded based on a failure tree diagram. This included insufficient heat transfer from the ignition train, ejection of uncombusted igniter materials or differences between the fine and coarse KNSB compositions which inhibited the ignition process. Based on the observations of the misfires and failure investigation the following causes were proposed.

1. *A disadvantageous igniter/ grain geometry.* There are no sharp corners or vortex inducing locations in the BEM design which would normally create a hot spot from which full combustion is achieved.

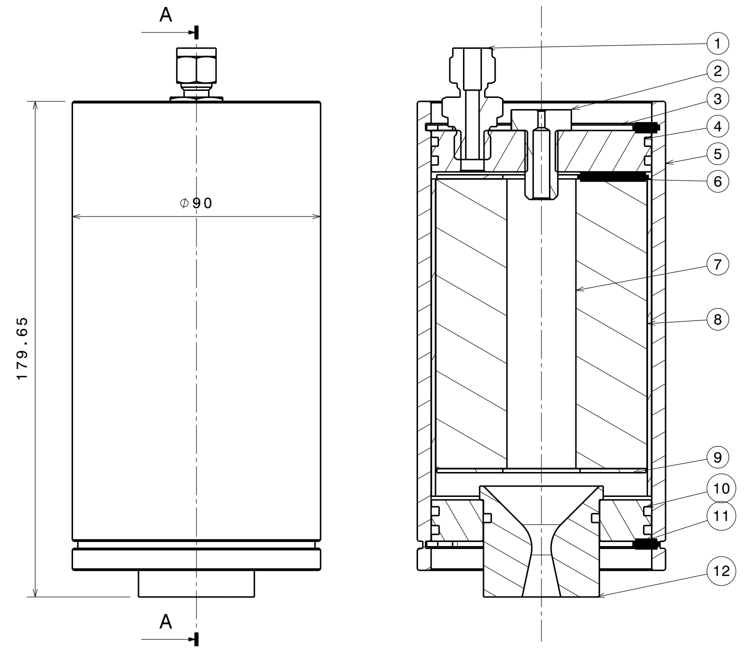

Figure 2: Ballistic Evaluation Motor (BEM) used during thesis study.(1) Swagelock RS-4 Adapter to pressure sensor. (2) M12 Igniter Bolt with Dowdy seal, (3) Aluminium Casing, (4) Circlip, (5) Forward Closure, (6) Cardboard Inhibitor Disk, (7) Propellant Grain, (8) Cardboard Inhibitor Tube, (9) Cardboard Inhibitor Disk, (10) Aft Closure, (11) Circlip, (12) Steel Nozzle.

2. *Insufficient sustained heat flux from the ignition primer/coating.* The baseline nitrocellulose-black powder coating burns very fast compared to other industrial compositions.
3. *Inhibiting effects from the surfactant.* The surfactant was found in several reported experiments to adversely affect the ignition characteristics of KNSB propellant.[9] In addition, the amounts used in this study were larger than those recommended by Nakka.[10]
4. *Other composition differences between fine and coarse KNSB.* As the KNO_3 PSD was dissimilar and the propellant was produced in different batches, other propellant differences could not be ruled out.

The disadvantageous geometry is very likely a strong contributing factor. However, as the coarse KNSB propellant was fired successfully, it is unlikely to be the sole cause. As the root cause analysis is at this stage not fully conclusive it is proposed to investigate the ignition characteristics of propellant directly focusing on propellant variations with fine and coarse KNO_3, variations in the amount of surfactant and the effect of different primer compositions on ignition.

4. Experiment Design

Problems with the ignition of fine KNSB propellant has resulted in several misfires. The RCA suggested that the surfactant, used to improve rheological properties of the propellant for casting, made ignition more difficult, although other composition variations could not be ruled out in the preliminary analysis. A laser ignition experiment was designed to determine if there are any differences between the ignition of coarse and fine KNSB compositions and to what extend this is adversely affected by the surfactant. In addition, the goal is to gain insight into the ignition process of KNSB propellant and how this might be improved with the addition of an ignition primer.

A 1064 nm near infrared laser was made available for this experiment. The use of lasers to investigate propellant ignition has a relatively long history as it allows the delivery of well defined energy fluxes to the propellant surface compared to other methods such as resistance wires, shock tubes or arc-furnaces.[5] Studies of ignition of AP based composite propellant by radiant flux shows ignition to be achieved in 100-1000 ms for radiant fluxes of 10 cal/cm^2s ($\approx$ 42 W/cm^2).[5] As this is around the lower limit of the used welding laser (50 W - 3 kW continuous output) it was decided to make the propellant samples larger than 1 cm^2. At the same time the propellant sample mass was to be

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kept around 3 g excluding ignition primer. The final sample design was a circular pellet of 16 mm ⊘ with a height of 8 mm. The 16 mm sample diameter results in a sample surface area of 2.011 cm^2 providing a range in incidence flux from approximately 25 W/cm^2 up to 1500 W/cm^2 by placing the sample outside the focus point of the beam. As it is unknown what the optical properties of the KNSB propellant or its constituents are, part of the experiment was used to determine the thermal uptake of the propellant. A thermal camera was used to measure propellant surface temperature as a function of time. Based on a short 50 W, 0.1 s pulse the power was interpolated to determine the approximate power requirement to achieve ignition in one second.

4.1 Experiment Setup

The experiment setup is shown in figure 3. It consists of an adjustable table underneath the laser optics (HAAS-Laser 22-24-01AY), with a focal length of 159 mm and a beam divergence <23°, which was tilted away from vertical. The laser beam was fed into the room via a fibre-optic cable. To assure sufficient distance between the optics and samples, the samples were placed beyond the focal point to provide uniform illumination of the sample surface. The optics were protected from contamination by a cross-flow of compressed air, while exhaust gasses were evacuated from the room with a fume arm placed close to the setup.

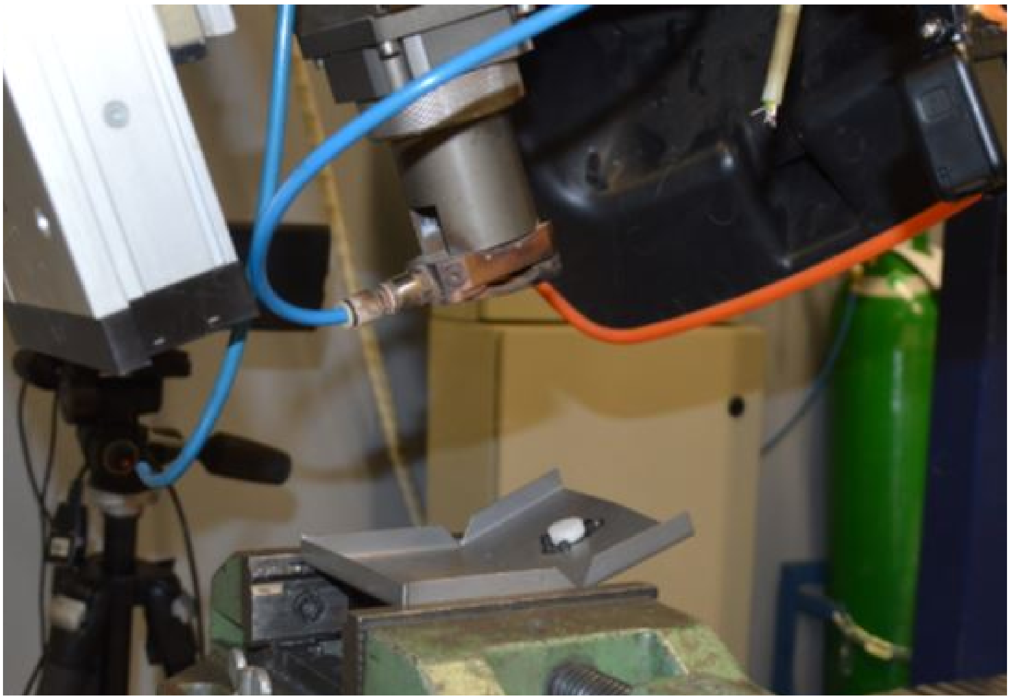

Figure 3: Experimental setup with laser optics visible from the top, and white propellant pellet placed perpendicular to the beam.

Measurements were taken with a TESTO 890 thermal imager, go-pros and a JVC camera. The thermal imager allowed thermal measurements of the samples during ignition with a set measurement range between 0-650 °C.

4.2 Sample Preparation

Samples were prepared a week before the experiment with the manufacturing process described in section 2. Batches were produced from the same ingredient stores used during the BEM tests. Propellant was prepared in batches of 100 g to reduce variations in mixture ratio. The batches consisted of fine and coarse compositions and the fine composition with both 2 and 6 drops (equal to 53.8 mg and 158 mg) of surfactant per 100 g of base ingredients. In addition, coarse and fine samples were made with various primer coatings: P1 the heritage coating, P2 a coating which consisted solely of charcoal and KNO_3 and P3 the titanium augmented coating. Of all mixtures at least three samples were fired. Sample weight showed to be slightly lower than designed at 2.8 ±0.2 g. Curing samples are shown in Figure 4a with some of the finished samples shown in Figure 4b. An overview of the fired samples is provided in Table 2. Samples a-c were half size test samples to verify the system worked as intended.

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(a) Coated propellant samples, sealed in plastic.
(b) Samples curing in aluminium moulds.

Figure 4: Samples prepared for the laser ignition experiment.

Table 2: Samples fired during the laser ignition experiment.

no.	**Composition**	**Surfactant [g/100g]**	**Primer**	Φ [W/cm^2]
a-c	Coarse	-	-	1250
1-3	Coarse	-	-	1250
4-6	Fine	-	-	1250
7-9	Fine	0.053	-	1250
10-12	Fine	0.158	-	1250
13-15	Coarse	-	0.25g NTC/2g BP	100/25/25
16-18	Coarse	-	0.25g NTC/2g BP/0.3g KNO_3/0.7g Ti	25
19-21	Coarse	-	0.6g C,1.4g KNO_3	25

5. Results

The laser ignition experiment was completed on 10-12-2018. The pilot laser was not working, thus alignment of the beam proved difficult. Similarly the distance to the focal point could only be approximately determined with an estimated uncertainty of ±2 mm. Measured distance to the approximate sample surface was determined to be 221 [mm], indicating that the beam was expanded slightly larger than the sample. In the end the uncertainty in beam location and beam width at focal point resulted in a beam width at the sample surface estimated to be ±20 % and approximately 2 mm offset to side of the propellant surface. Calibration of the input power was attempted using the TESTO thermal camera to measure the sample temperature, however this was unsuccessful. In the end a power level was chosen which led to ignition in approximately 1/10th of a second.

A total of 21 samples were ignited using the laser with 19 samples achieving full combustion. One sample (no. 12), with 0.158 g/100g surfactant extinguished after the laser was turned off. Thermal measurements from samples 2 (coarse), 5 (fine, no surfactant) and 12 (extinguished) are shown in Figure 5a. It can be seen that the propellant surface temperature rises nearly instantaneously to the point of decomposition. The estimated flame temperature is much lower than expected at 350-400 °C. The lower temperature plateau in all measurements occurs when the burning surface area is no longer directly measured as the propellant burns away and does not coincide directly with laser illumination, which was 1 second. Surface temperature measurements for the samples with coatings P1,P2 and P3 are shown in Figure 5b and give higher measured surface temperatures compared to the uncoated samples. Samples that were coated with nitrocellulose/ black powder showed ignition at lower power level but thermal graphs show a slight ignition delay. However, this delay was not observed with the coating based on KNO3/charcoal (P2).

During the experiment the following qualitative observations were made:

1. Investigation of fine and coarse propellant samples with and without surfactant,
   (a) Self extinction was observed in a single fine sample (no. 12). The extinguished surface became soft and formed a liquid layer on top of the sample as shown in figure 6b. However, also the other samples with large quantities of surfactant (10-11) initially showed a clear flame as seen in Figure 6a (shown image has

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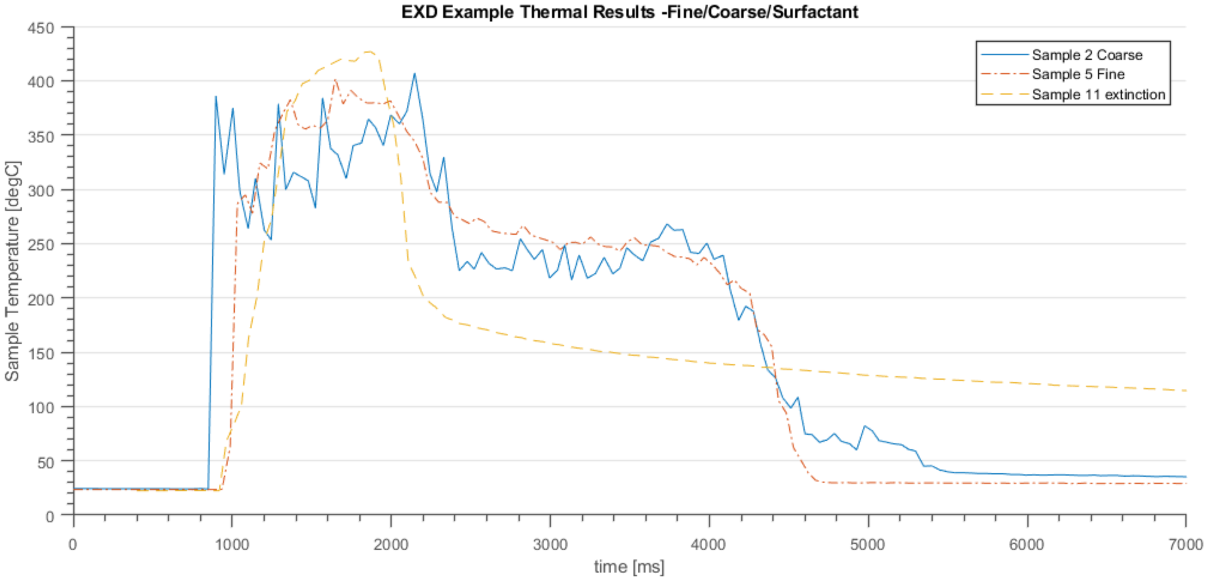
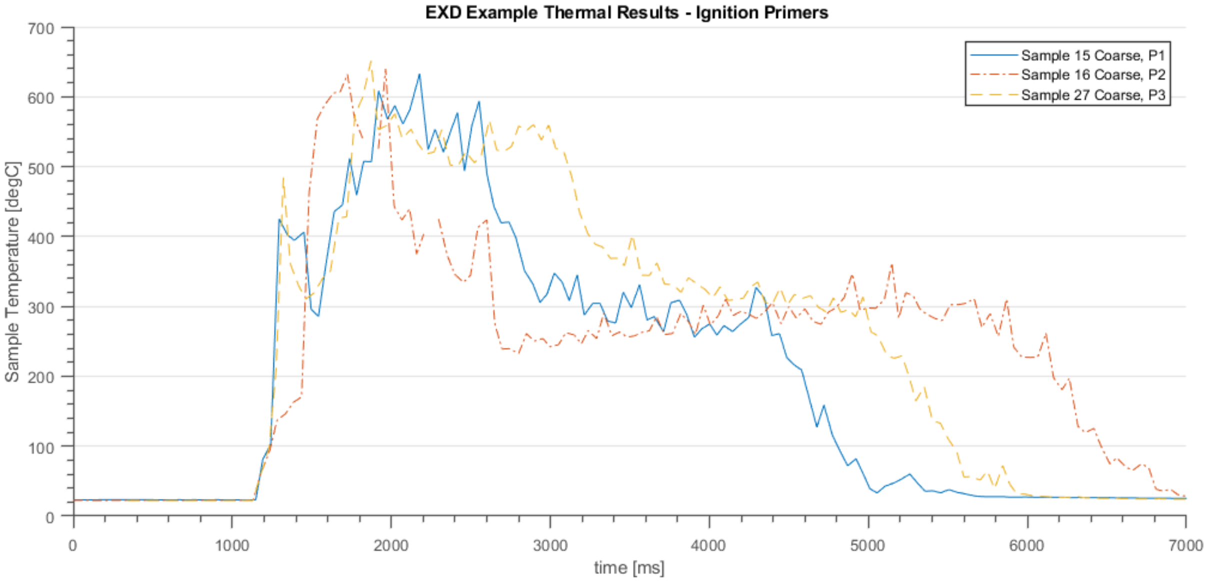


(a) Burning surface temperature measurements for samples 2 (Coarse), 5 (fine) and 12 (fine, 6 drops of surfactant, self extinguished). Radiant flux at 1250 [W/cm^2] for a 1000ms



(b) Burning surface temperature measurements for samples 15 (coarse P1), 16 (coarse P2) and 27 (coarse P3), Radiant flux at 25 [W/cm^2] for 1000 ms

Figure 5: Example surface thermal measurements made during the laser experiment using the TESTO thermal imager system.

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adjusted brightness). The lack of particles in the flame is indicating a low amount of KNO_3 or K_2CO_3 products.

(b) Fine and coarse sample combustion showed considerable difference in the flame structure. During combustion of the coarse composition, particles are visible in the flame (see Figure 6c) and continue combustion as part of the flame indicated by their brightness. The fine propellant showed a smooth uniform flame front 6d. KNO_3 at the burning surface was visibly the brightest and likely hottest location in the flame. The flame front for both fine and coarse propellant is shown in figures 6f and 6e respectively.

2. Investigation into ignition primers

   (a) Even at the significantly reduced power level ignition occurred relatively fast over the entire propellant surface. For the heritage mixture P1:BP/NTC near instantaneous ignition was observed across the entire surface at 200 W (no. 13).

   (b) Ignition for the P2:charcoal/KNO_3 mixture was considerably slower. A visible deterioration occurred where the surface became darker shortly before ignition. Flame-spreading for this coating seemed to occur below the primer surface.

   (c) Similar to P2:Charcoal/KNO_3, P1:BP/NTC in one case seemed to ignite below the propellant surface as could be seen in figure 7a.

   (d) The Titanium coating P3:BP/NTC/Ti, did not appear to improve ignition of the sample although bright sparks were observed to scatter around the sample.

6. Discussion

6.1 Investigation into the different KNSB compositions

Extinction was observed for sample 12. The flame that was observed while the sample was illuminated by laser showed considerable differences to the other samples (fine, coarse, fine with 0.053 g/100g) as it was a less turbulent clear flame compared to the other samples. The other experiment repetitions with same experiment conditions (10-11) showed the same initial flame structure but did not lead to extinction. This could be explained by a lack of KNO_3 present in the flame either as the solid particles remain stuck at the propellant surface or because the KNO_3 particles do not attain the necessary temperature to start decomposition. As the propellant with 0.158 g/100 g was ignited successfully and proceeded to burnout for 2/3 samples and full BEM grains ignited with a blowtorch, it seems that primarily the initial ignition is affected and the effect of the surfactant becomes less once steady combustion is attained.

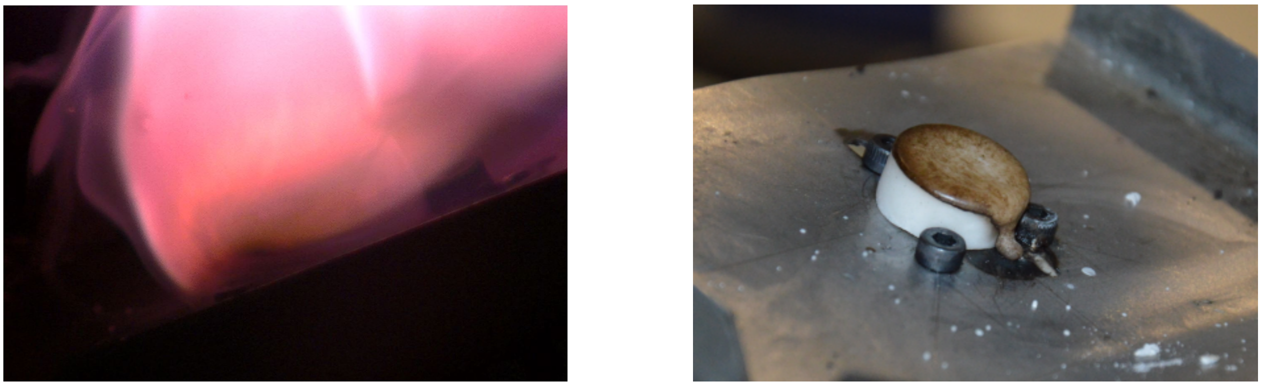
As no other clear differences were found in the ignition behaviour of the other (fine, coarse, fine with two drops) samples this result strongly supports the surfactant as key inhibiting ingredient. Based on observations the ignition of the propellant surface follows the following phases: rapid gasification in selected locations; a gaseous flame (likely with little KNO_3) after which several local initiation points grow from inside outward.

Combustion for coarse propellant showed similarities to the combustion structure of AP composite propellants: the relatively faster decomposition of the binder dislodges oxidiser particles which then continue combustion in the flame, this could be explained by the significantly lower boiling point of sorbitol (295 °C) compared to the decomposition temperature of KNO_3 (400 °C). The small size of the fine KNO_3 particles prevented direct observation of this behaviour although the observed flame was translucent which suggests that at least some condensed particulate was present.

Temperature measurements were reviewed, however, neither the initial temperature rise and absolute magnitude appears to represent the actual decomposition. Maximum measured temperatures of the samples was between 250-450 °C. As this is around or even below the minimum decomposition temperatures of the propellant it is expected that flame temperature was incorrectly measured. This could be explained by partial transparency of the flame, or specific spectral bands distorting the estimation of the peak temperature. This measurement can likely be improved by moving the camera closer to the sample, however, it is recommended to first investigate proper calibration or use equipment better suited to the task.

6.2 Investigation into ignition primers

When comparing the ignition behaviour of coarse samples coated with ignition primers it was found that the compositions P1 and P3 ignited faster than P2 which agreed with expectations. This is likely due to the low ignition temperature



(a) Flame structure during ignition of the fine composition with 6 drops of surfactant (no 11).

(b) Extinguished propellant (no 12) sample with molten propellant surface.

(c) Coarse Propellant flame structure with a more turbulent luminous flame showing uneven regression at the sample edge.

(d) Fine propellant showing more even regression with a smooth edge at which combustion occurs.

(e) Coarse Propellant flame structure showing significant active particles at the burning surface.

(f) Fine flame front with similar active region thinner and more evenly distributed.

Figure 6: Propellant samples prepared for experiment D: laser ignition.

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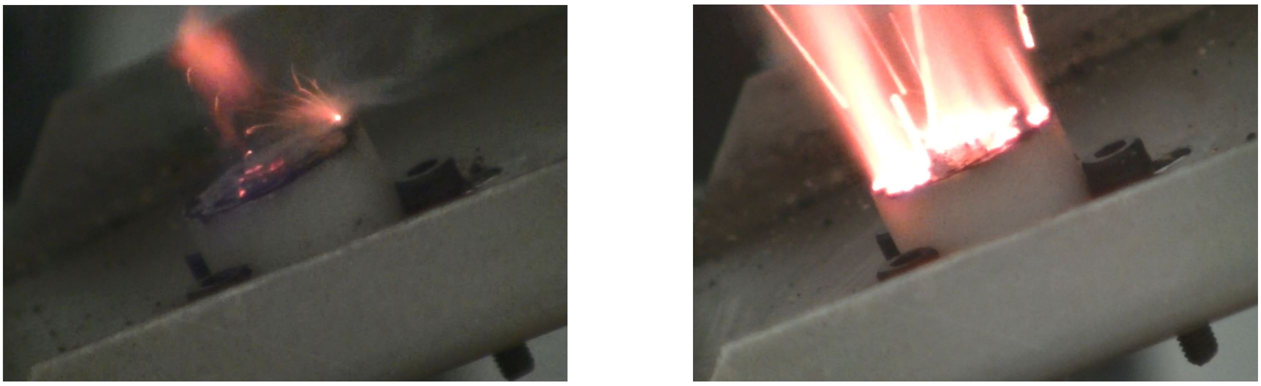

(a) Ignition of the coating (P2) showing ignition to start below the irradiated surface.

(b) Instantaneous ignition (P1) across a large part of the irradiated surface.

Figure 7: Propellant samples prepared for experiment D: laser ignition.

of nitrocellulose (auto ignition at 170 °C) or due to the lack of sulphur (one of the ingredients in black powder) in the composition. In black powder, the sulphur decomposition is the lowest temperature (150 °C), and partially catalytic, decomposition to occur.[4]

Maximum measured temperatures were also much higher for the coated samples even though radiant flux (25 W/cm^2) was a small fraction of that used with uncoated samples. This is likely due to the presence of carbon in all primer mixtures which is specifically noted for its optical activity in infrared. This would increase the absorbance and emissions both resulting in efficient ignition and higher estimated surface temperatures. Although the slower ignition of the samples with P2 might be desirable, the slow flame spreading might also hamper efficient ignition of the full motor. It is recommended to further investigate the various ignition primers with the laser experiment, but also to test these different coatings in the BEM and compare the differences under the representative motor environment to reach a full conclusion.

7. Conclusion & Recommendations

A new method was piloted at the TU Delft during this experiment which allows direct study of the flame structure and combustion characteristics of solid propellant. First and foremost the experiment goal was to identify any differences between propellant samples with and without surfactant, with fine or coarse particle size distributions and between the different ignition primers.

The extinction of fine propellant samples with 0.158 g/100g of surfactant indicate that this is the most likely cause of the observed misfires. Differences were observed in the flame structure of fine (with and without 0.053 g/100g surfactant) and coarse samples, however, this did not appear to affect the likely hood of ignition or combustion. In conclusion to the RCA it is recommended to decrease the surfactant quantity to or below 0.053 g/100g of KNSB propellant.

From the observed flame structure the hottest part of combustion is at the oxidiser particles surface. For the coarse composition these oxidiser particles were pulled into the gas flow while for the fine composition a more even flame was observed without oxidiser.

Although sufficient for the direct goal of the experiment, determining the root cause of the observed BEM failures, the experimental method can be significantly improved to increase the quality of measurements and depth of investigation. An attempt to characterise the beam with thermal paper was unsuccessful during the experiments. Therefore a better method needs to be designed to measure and calibrate the energy delivered to the propellant surface. This would account for the alignment, beam width at the point of incidence, and a verification of beam coherence at the sample surface. The ideal mode, especially considering the relatively large surface area in this experiment is a flat top beam with at most 80-90% incident flux at the pellet edge. Besides more professional calibration equipment insight into the power distribution might be achieved with acrylic pellets with dimensions similar to the propellant samples under test, as both the illuminated surface area and the local radiant flux can be established within the sample holder.

Defining a clear criterion for ignition has been a challenge in studies of solid propellant ignition behaviour. This was made even more difficult by the low relative frame-rate compared to the speed of the physical process. The temperature rise to decomposition occurred within 1/10th of a second for samples 1-12. A second improvement of the experiment can therefore be achieved by increasing either the frame rate at which the ignition behaviour is observed,

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or by reducing the radiant flux used during the first part of the experiment. A decrease in incident flux to 250 W/cm^2 would result in an estimated ignition transient of approximately 0.5 s for uncoated KNSB propellant, as a trade between observing transient behaviour and limiting conduction of heat into the entire sample.

8. Acknowledgements

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