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UAH Solid Propellant Characterization

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One of the main properties of solid propellants that must be known before a solid propellant design can be implemented into any use is the burning rate law. The objective of this works is to investigate the temperature sensitivity as a function of pressure for a composite propellant. An ultrasonic pulse-echo device measured the burning rate of 20-gram samples in a closed bomb at pressures from 250 to 3,000 pounds per square inch. Repeated tests show sample-to-sample variations in the burning rate measurements. The standard deviation of burning rates at a reference condition ranged from 0.4% to 0.6% at initial propellant temperatures of 145°F and 75°F respectively.

Nomenclature

n	=	burning rate exponent
P	=	pressure
r	=	burning rate
T_P	=	initial propellant temperature
σ_p	=	temperature sensitivity at constant pressure
τ	=	propagation time

I. Introduction

Recent interest in new solid boosters from NASA has resulted in a renewed interest in ballistic evaluation methods for solid propellants. As part of a program to get capabilities back on line, NASA Marshall Space Flight Center (NASA MSFC), the U.S. Army's Aviation and Missile Research, Development and Engineering Center (AMRDEC), and The University of Alabama in Huntsville (UAH) developed a small program to demonstrate and compare their respective capabilities. Part of this program involved preparing small samples of propellant for ballistic evaluation at UAH. The UAH work involved an ultrasonic burning rate technique developed by ONERA[1] and utilized at UAH[2,3,4] The objective of the UAH work was to characterize the burning rate of one propellant formulation as a function of pressure and initial bulk temperature of the propellant. Initial propellant temperatures of 75°F (23.9°C) and 145°F (62.8°C) were specified for this study.

II. Approach

A. Propellant Samples

The investigation used an ammonium perchlorate (AP)/Aluminized/hydroxyl-terminated polybutadiene (HTPB) composite propellant. The bimodal-oxidizer propellant was prepared in a one gallon mixer and cast into three cardboard tubes 12 in. long by 1.25 in. in diameter. After curing, the tubes were cut into approximately 0.5 in. thick samples with a lathe; each tube yielded eighteen samples. The average mass of a sample was 20 grams. Each sample was labeled with the tube it came from, its position in the tube, and an arrow to indicate its orientation in the tube.

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B. Testing Hardware and Instrumentation

The propellant samples were burned in a closed combustion bomb (Figs. 1 and 2) that is rated up to 5000 psi. The combustion bomb is inside an environmental test chamber which allows the temperature of the propellant sample and the test hardware to be controlled at a constant level. The environmental test chamber sits in a 5'x 6.5' test cell with one foot thick steel-reinforced walls with a 2.5" thick sliding steel door. The combustion bomb is made of stainless steel since it has a high durability against the volatile gasses produced during the burning. In the event of over-pressurization, the combustion bomb has a check valve with a cracking pressure of 5000 psi which will allow the excess gas to be vented into the test cell.

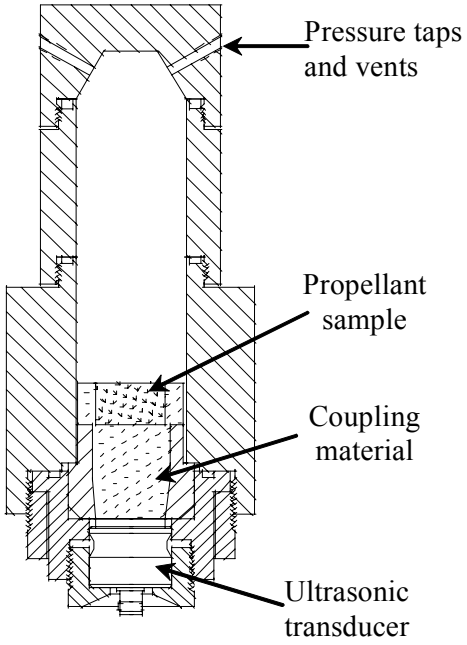

Figure 1. Schematic of combustion bomb showing the location of the propellant sample and ultrasonic transducer.

The combustion bomb has three ports for connecting two 5000 psi Setra pressure transducers and a ventilation system (Fig. 2). One pressure transducer is connected directly to the combustion bomb inside the test chamber, while the other one is situated outside the test chamber and connected to the bomb through stainless steel tubing. The reason for inside pressure transducer is minimize the pressure lag time of the outside pressure transducer due to the tubing connection. Before performing a test, the outside pressure transducer is calibrated. Using a transfer calibration from the outside to the inside transducer under lower pressurization rates, the outside transducer calibration is transferred to the inside transducer.

A panel outside the test cell controls the ventilation system which is used to purge the combustion bomb of the volatile gasses produced during a test. During a purge, nitrogen is supplied to the combustion bomb at high pressure through the ventilation port, held for 2-3 seconds, and then exhausted back out the same port. A check valve on the supply line ensures the gasses stay in the exhaust line when exhausting. The gasses are then sent to a surge tank for depressurization. Finally, the gasses pass through a check valve into a tank filled with water where they are scrubbed and vented to the outside of the building. This process is repeated four times to get most of the volatile gasses out of the combustion bomb, so that it can be safely cleaned.

Figure 2. Picture of combustion bomb inside the environmental test chamber.

The heart of the pulse-eco ultrasonic method is the Electronic Device for Ultrasonic Measurement (EDUM) developed by the Office National d'Etudes et de Recherches Aerospatiales (ONERA) in France.[1] The EDUM sends a signal to the ultrasonic transducer (Panametrics V102 1.0/1.0) to send out an ultrasonic pulse at 1000 Hz. The pulse travels through a coupling material in the propellant sample holder and then through the propellant (Fig. 1). When the pulse encounters a difference in impedance, part of the signal is reflected back and detected by the ultrasonic transducer. The EDUM also receives the signal and measures the propagation time, τ, and outputs a voltage proportional to τ.[1]

The reflections occur at the coupling material/propellant interface and at the burning surface. The coupling material causes a delay in the burning surface echo return, so that the signal does not get lost in the internal echoes of the ultrasonic transducer. The coupling material also protects the ultrasonic transducer from the high temperature of the burning propellant. Figure 3 shows a schematic of the ultrasonic transducer setup and the waveform it produces as seen on an oscilloscope. Point 1 in Fig. 3 is the signal produced by ultrasonic transducer and it travels in both directions. Point 2 is the reflection off of the back of the ultrasonic transducer. This surface is generally attenuated to reduce the echo. Point 3 is the echo from interface of the coupling material and the propellant sample. In order to reduce this echo the coupling material and propellant should have a similar density. Point 4 is the return echo from the propellant burning surface and is tracked during the test.[1] Using the propagation time of the signal and the speed of sound in the propellant and coupling material, the instantaneous thickness of the propellant can be found. The burning rate of the propellant can then be calculated by taking a time derivative of the instantaneous thickness.

A Hewlett Packard 54602B oscilloscope is used to visualize the ultrasonic waveform. The data from the two pressure transducers and the EDUM are sent to a National Instruments BNC-2090 block and then routed to a National Instrument AT-MIO 16 F 5 A/D board. LabVIEW virtual instruments are used to acquire and record all the data from a test. A Mathcad 13 program is used to analyze the resulting data.

The waveform seen in Fig. 3 is also recorded and stored using the data acquisition system. When the data acquisition program is triggered, it takes a data point from the different channels at 1000Hz. Every 25th time it records data points it also records and stores the waveform that it sees at that time. The waveform data is then put into a Mathcad file which produces a simulated movie of what the waveform does as the propellant sample burns during the test.

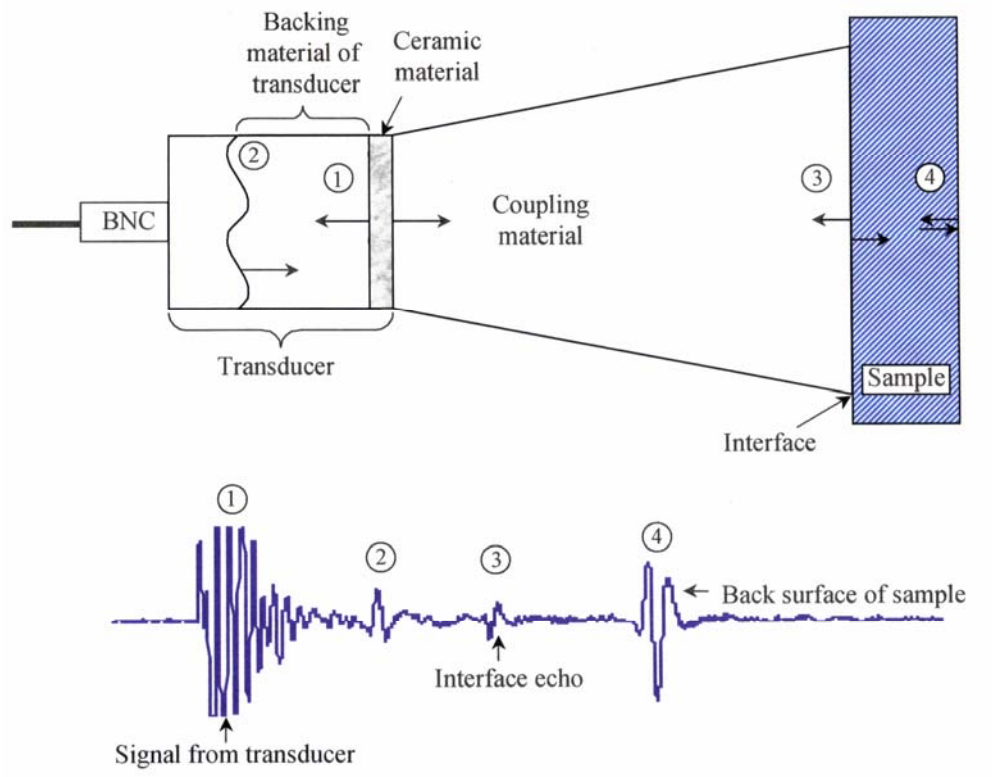

Figure 3. Schematic of the setup of the ultrasonic transducer and the waveform produced.

C. Testing Procedures

The complete execution of a test involves three stages: sample preparation, setting up the testing equipment, and running the test. A sample is prepared by first recording its thickness along with its other pertinent data. To attach the sample to the sample holder, an Araldite D epoxy is used. The epoxy is mixed and degassed in a vacuum chamber to remove any voids that could increase its impedance. After degassing, a thin layer of the epoxy is poured onto the sample holder and degassed again. At this point the sample holder has a Teflon/rubber sleeve attached aoround it in order to contain the epoxy. After degassing, the sample is placed on the thin layer of epoxy with a small weight on top. The entire assembly is then degassed again. This helps the propellant bond more firmly to sample holder and reduces the chances of a debond during testing. Finally, epoxy is poured around the side of the sample until it is even with its surface. This holds the sample in place and inhibits lateral combustion. After soaking at room temperature for 24 hours, the sample is ready to test (Fig. 4).

Two different epoxies are used for the sample holder coupling material for this testing. The first epoxy type is the aforementioned Araldite D formulation. Since this epoxy tends to soften as the temperature rises, it can only be used for ambient testing. If it were soaked at 145°F it would soften to the point where it could come out of the sample holder during a test (due to the high pressures) and could possibly crush the ultrasonic transducer. Taking this into account, a high-temperature epoxy (Ciba-Geigy 4500/1510) is used for the hot tests. This epoxy can maintain the desired stiffness during soaking at the high temperatures.

Figure 4. Propellant in sample holder.

To setup a test, the combustion bomb is assembled with the propellant sample holder and a pyrogen igniter installed. The combustion bomb is then placed in the test chamber and the inside pressure transducer, a line to the outside pressure transducer, the nitrogen ventilation system, and the ultrasonic transducer are attached. The test chamber is then shut and the equipment is allowed to thermally soak for the requisite amount of time.

Before running a test, the outside pressure transducer is calibrated with a dead weight pressure tester and attached to the line coming out of the test chamber. The EDUM is then calibrated so that propagation times to the coupling material/propellant interface and the burning surface can be correlated to the desired voltages.

Since the speed of sound in the propellant and the coupling material varies with pressure, a pre-test is conducted to determine its acoustic properties.[1] The combustion bomb is pressurized with nitrogen to the approximate test pressure, and the resulting data are used to determine a linear calibration curve. After satisfactory pre-tests, the burn test takes place. The igniter is hooked up to a battery and a safety check is performed. The data acquisition system is initiated and the sample is burned. After the test, the combustion bomb is purged to expel the volatile gasses produced during the combustion. During a test large amounts of heat are produced, therefore the setup is allowed to cool until it returns to the initial testing temperature. A post-test is then performed in the same manner as the pre-test, in order to determine the acoustic properties of the coupling material.

III. Results and Discussion

Two sets of three tests were conducted, with the initial propellant temperature controlled for each set. The first set of tests was performed on samples with an initial temperate of 145°F. The second set of tests was performed on samples with an initial temperature of 75°F. The results of the six tests at 1000 psi are summarized in Table 1. Figure 5 shows the burning rate curve of all six tests.

Table 1. Summary of Test Data at 1000 psi

Test	T_P, °F	r, in/s	n	Test	T_P, °F	r, in/s	n
A	145	0.4713	0.3701	D	75	0.4459	0.3174
B	145	0.4717	0.3863	E	75	0.4466	0.3503
C	145	0.4770	0.3421	F	75	0.4433	0.4016
Average		0.4733	0.3662			0.4453	0.3564
Standard deviation		0.002598	0.02237			0.001660	0.04242
σ_P, °F^{-1}	0.0008732						

For the hot tests, the propellant samples and testing hardware were thermally soaked at 145°F for seven hours. The hot tests produced very similar burning rates at 1000 psi with Test C being slightly higher. The average burning

rate of the three samples was 0.4733 in/s. The burning rate exponents were also similar with Test B being higher than the others. The average burning rate exponent was 0.3662. Figure 6 shows the burning rate curves of the hot tests ranging from 240 psi to 2738 psi. The curves and associated data correspond well with each other, establishing the repeatability of the ultrasonic technique.

The second set of tests was performed at 75°F, the reference temperature of the data provided by AMRDEC. For these tests the samples and hardware were again thermally soaked for seven hours. The average burning rate for these tests was 0.4453 in/s. This burning rate compares well with the burning rates supplied by AMRDEC of 0.4450 in/s for a strand burner and 0.04300 in/s for a 2 x 4 motor. The burning rate exponents for the 75°F tests had a larger range than those of the hot tests. The average exponent for the ambient tests was 0.3564. However, this value again compares favorably with data provided by AMRDEC of 0.3400 and 0.3200 for exponents from 1000 psi to 2000 psi for a strand burner and a 2 x 4 motor respectively. Figure 7 shows the burning rate curves of the 75°F tests ranging from 500 psi to 2400 psi. Again the curves match well with each other showing the repeatability of the ultrasonic technique.

Using the average burning rates from the two sets of tests, the temperature sensitivity, σ_p, was calculated from 800 psi to 1900 psi at increments of 100 psi using

$$\sigma_p = \left(\frac{\partial \ln r}{\partial T_P} \right)_P . \tag{1}$$

The average of the σ_p values was 0.0008805°F^{-1}. This value is lower than the typical values of 0.002°F^{-1} to 0.04°F^{-1} .[3,5] The σ_p values are shown in Table 2 and displayed in Fig. 5.

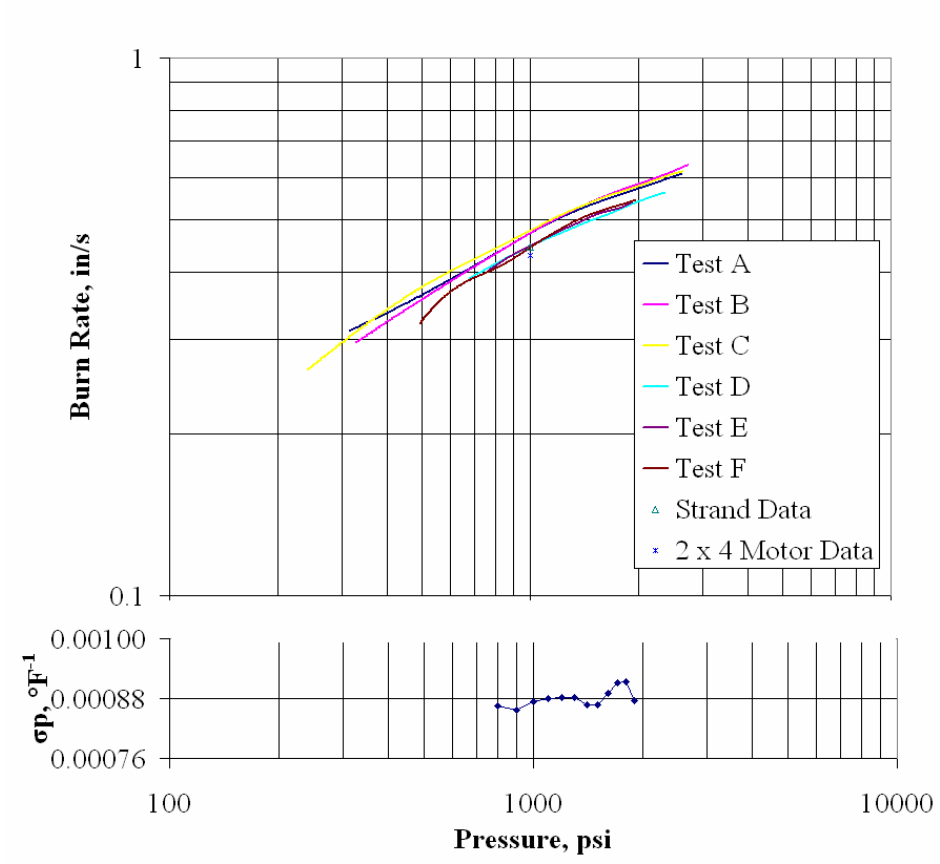

Fig. 5 Burning rate curves for all tests and temperature sensitivity, σ_p.

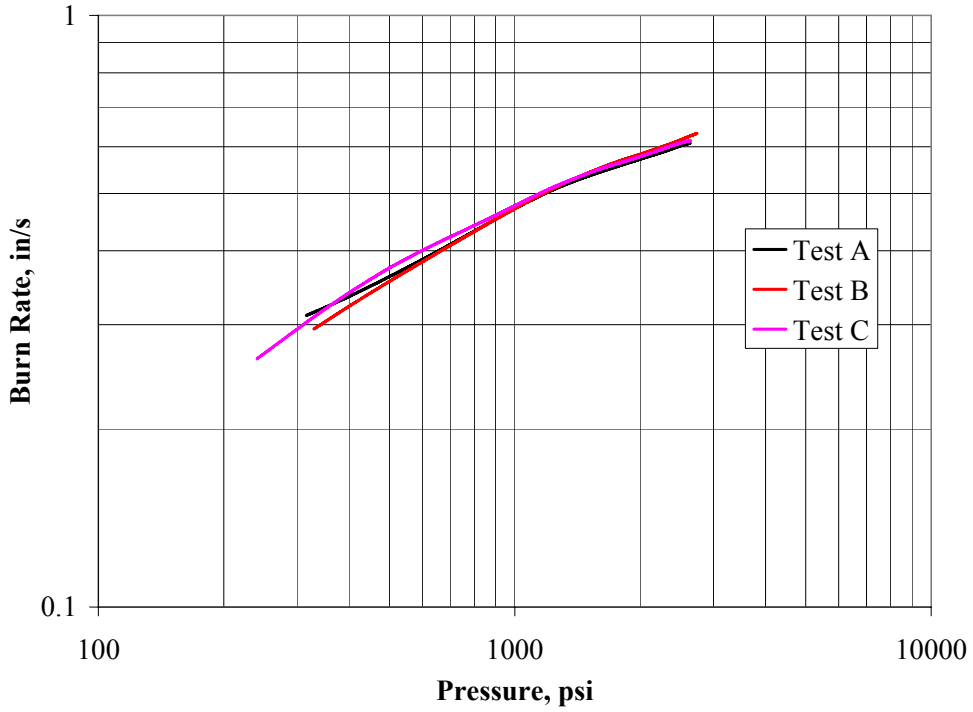

Figure 6. Burn rate curves for tests performed at 145°F.

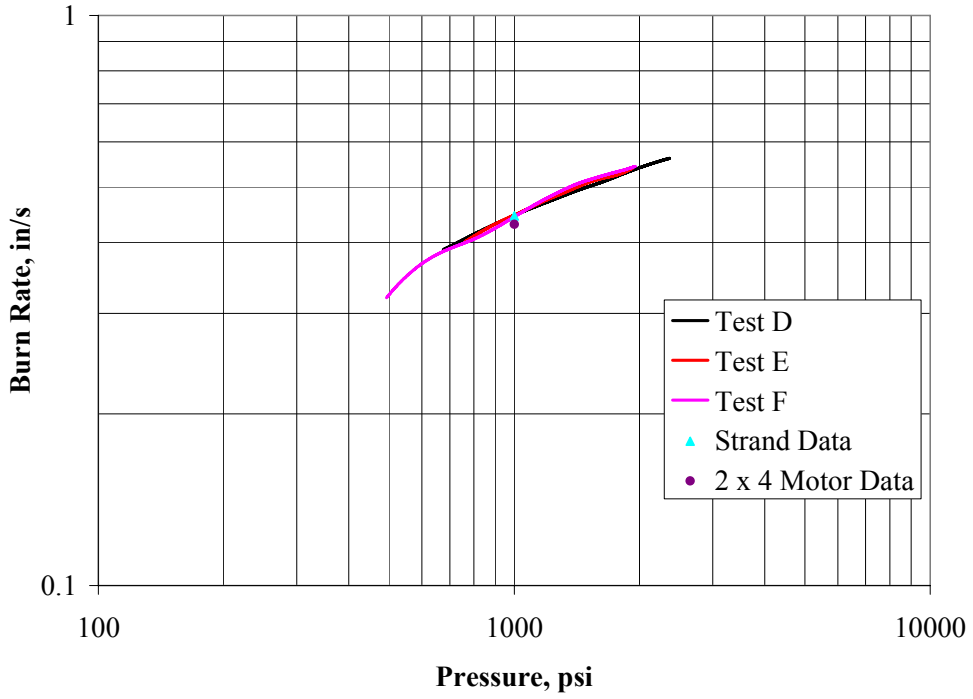

Figure 7. Burn rates curves for tests performed at 75°F.

Table 2 Temperature Sensitivity Data

Pressure, psi	145°F Tests		75°F Tests		σ_p, °F^{-1}
	r, in/s (average)	Standard Deviation, in/s	r, in/s (average)	Standard Deviation, in/s	
800	0.4351	0.004611	0.4095	0.002557	0.0008651
900	0.4548	0.003371	0.4284	0.003674	0.0008565
1000	0.4733	0.002598	0.4453	0.001660	0.0008732
1100	0.4901	0.002099	0.4608	0.000600	0.0008806
1200	0.5049	0.002125	0.4747	0.002146	0.0008820
1300	0.5179	0.002177	0.4869	0.003378	0.00088.18
1400	0.5293	0.002546	0.4981	0.003868	0.0008679
1500	0.5393	0.003044	0.5075	0.003398	0.0008683
1600	0.5483	0.003537	0.5152	0.003227	0.0008896
1700	0.5564	0.004027	0.5220	0.003536	0.0009118
1800	0.5638	0.004216	0.5288	0.003282	0.0009138
1900	0.5706	0.004410	0.5367	0.001861	0.0008750

IV. Conclusions

The pulse-echo ultrasonic technique is an effective method of characterizing solid propellant properties. The method showed repeatability at each temperature tested, and accuracy since it compared well with the reference data supplied. Future work will entail testing in the 30°F to 50°F temperature regime.

Acknowledgments

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