

# Ignition and Combustion of HTPB-based Solid Fuels Loaded with Micron-sized Metals

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## Abstract

This paper discusses an experimental investigation focused on the effects of innovative micron-sized metal additives on the ignition and burning of solid fuel formulations based on Hydroxyl-Terminated PolyButadiene (HTPB). Gaseous oxygen was selected as oxidizer for ignition delay and regression rate tests. A relative grading of solid formulation performance was performed taking unloaded HTPB as baseline. Three different micron-sized additives were investigated in the experimental activity. Ignition delay is highly depended on pressure, while the linear regression rate was not appreciably affected as the pressure was increased from 1.0 MPa to 1.9 MPa. All of the micron sized additives have a positive effect on enhancing both linear regression rate and mass burning rate: amorphous aluminum (am\_Al) demonstrated a better effect than both magnesium (Mg) and magnesium-boron (MgB) powders.

## 1. Introduction

Hybrid propulsion systems (combining both a fluid and a solid reactant) have many advantages over conventional solid- and liquid- propellant rockets, such as low-cost, safety, throttleability, and a wide range of appealing applications, making them very attractive for both military and commercial applications [1, 2]. The major drawback of the hybrid propulsion systems is the low regression rate of the gasifying solid fuel that yields low thrust levels for limited burning surfaces. However, this disadvantage can be overcome by loading the solid fuel with energetic additives.

HTPB has been considered one of the most promising solid fuels for hybrid propulsion systems due to its large heat of combustion when reacting with oxidizers, safety in manufacturing and handling, and no sensitivity to grain cracks, voids, and other mechanical defects [3]. Risha et al. demonstrated that nano-sized particles added into HTPB fuels can provide considerable regression rate enhancement, but proper manufacturing techniques are required to disperse

the additive down to the nano-scale [4, 5]. On the contrary, the conventional micron-sized additives used for solid fuel loading are usually ineffective in enhancing the regression rate [6].

A sub-micrometric compound of magnesium-boron (MgB) produced by MACH\_I with a proprietary procedure demonstrated that this powder is able to exceed the intrinsic ignition limits of pure boron: even though magnesium combustion efficiency is particularly low, it appears an appropriate additives to enhance the initial combustion temperature in order to allow boron sustained combustion [7, 8].

Amorphous aluminum (am\_Al) is an alloy of aluminum, iron and yttrium. Hence, the melting point is lower than that of aluminum, which may have a positive effect on enhancing regression rate of fuel compared to aluminum.

Ignition is the first step of solid fuel applications to propulsion. However, few works on ignition of hybrid fuel under realistic operating conditions are reported in the open literature. T. J. Ohlemiller and M. Summerfield [9] were pioneers in ignition tests of polymers and conducted radiative ignition of polystyrene and an epoxy in oxygen/nitrogen mixtures as early as 1971. The effect of radiant flux, pressure, oxygen percentage and fuel absorption coefficient on ignition delay were discussed. A radiative ignition model of a solid fuel including the gas phase reaction and the in-depth absorption of the incident radiation by its solid phase was solved by T. Kashiwagi; however, the pressure effect was not considered [10].

The solid-fuel regression represents the key parameter in the study of hybrid rocket engine. The diffusion model developed during 1960s by Marxman and co-workers [11], considers the convective heat transfer as the main mechanism for regression rate determination, and therefore other operating parameters including pressure should exert no (or limited) influence on regression rate. One of the first analysis about pressure effects on regression rate was conducted by Smoot and Price [12-14], during the investigation of rubber and poly-urethane fuels using fluorine and mixtures of fluorine/oxygen as oxidizer, at  $P_c$  below 1.2 MPa. Results show that in the low mass-flux regime, radiant heat transfer may account for the pressure dependence; however, at the high mass-flux regime, reaction kinetics may become the rate-limiting mechanism. C. Paravan [15] reported that pressure exhibits neutral effect on regression rate of HTPB at the back pressure from 0.7 MPa to 1.6 MPa. However, F. M. Favaró et al. clarify that regression rate decreased with the increasing of pressure thanks to numerical simulation [16].

Hybrid propulsion systems have many advantages over conventional liquid- or solid-propellant system. As mentioned, the major disadvantage of the exiting solid fuel is the low regression rate. Regression rates of solid fuels have been shown to be increased by the addition of energetic metal powders. The objective of this investigation was focused on the effects of various innovative micron-sized metal additives on the behaviour of solid fuel formulations based on HTPB. Ignition of fuels under CO<sub>2</sub> laser irradiation was carried out at 0.1 MPa and 1.0 MPa, and ballistic characterization of solid fuel at 1.0 MPa and 1.9 MPa were performed at the same time.

## **2. Experimental**

### **2.1 Energetic Additives**

The types of energetic additives selected in the study, including manufacturer, size and density are given in Table 1. All of the metal additives are in micron size as you can see from the table. MgB is a dual metal composed by Mg and

B. MgB used in this study is composed by 20% magnesium (Mg) and 80% boron (B) with 90% purity. Am\_Al is an alloy of aluminum (Al), iron (Fe) and yttrium (Y), with mass fraction respectively 70.3%, 13.54% and 16.16%.

Table 1: Description of micron sized energetic additives used in this study

<b>Energetic additives</b>	<b>ID</b>	<b>Distributor</b>	<b>Particle Size /<math>\mu\text{m}</math></b>	<b>Density /g/cm<sup>3</sup></b>
Magnesium	Mg	Afa Aesar	<47	1.74
Magnesium-Boron	MgB	MACH_I	5.2	2.189
Amorphous Aluminum	am_Al	CERAM	38~212	3.19

## 2.2 Sample Preparation

Details of solid fuel processing procedure were discussed in [15]. Micron sized energetic powders should be disposed by ultrasonic machine for more than 15 mins before adding to the fluid fuel. Binder, plasticizer and curing agent selected for each fuel is HTPB R-20, dioctyladipate (DOA) and isophorone di-isocyanate (IPDI), respectively. A reference composite fuel consisting of 78.86% of HTPB (mass fraction), 13.04% of DOA, 7.67 % of IPDI and 0.43% of TIN was selected. This composite fuel was regarded as a unit, used in all of the other types of the fuels. Table 2 shows mass fraction of energetic additives and density of the solid fuel. Although mass fraction of additives in each fuel is different, the content of main metal (Mg, B and Al) in each fuel is maintained to 0.37 mol/100 g. The measured density ( $\rho_{MD}$ ) of solid fuel illustrated in Table 2 was obtained thanks to the Gibertini Europe 500 precision balance. The theoretical density ( $\rho_{TMD}$ ) of the fuel was calculated under consideration of the volume occupied by a certain amount of additives.  $\Delta\rho$  defined in equation (1) was used to evaluate tested compound porosity. The measured densities agree well with the theoretical density.

$$\Delta\rho = \frac{\rho_{TMD} - \rho_{MD}}{\rho_{TMD}} \times 100\% \quad (1)$$

Table 2: Composition and density of tested fuels

<b>No.</b>	<b>Additives (by wt%)</b>	<b><math>\rho_{TMD}</math>, Kg/m<sup>3</sup></b>	<b><math>\rho_{MD}</math>, Kg/m<sup>3</sup></b>	<b><math>\Delta\rho</math>, %</b>
1	None	915	$922 \pm 1.9$	$-0.77 \pm 0.21$
2	8.9%Mg	956	$957 \pm 1.8$	$-0.10 \pm 0.19$
3	2.8%MgB	930	$935 \pm 1.1$	$-0.54 \pm 0.12$
4	14.2%am_Al	1018	$1022 \pm 0.5$	$-0.39 \pm 0.05$

Samples for ignition tests were cut into 8-mm-diam, 6-mm-high cylinders. The surface to be irradiated was carefully cleaned by acetone before the ignition. Samples for ballistic tests were manufactured in the shape of cylinder. The cylindrical samples are elements of 30mm of length and 18mm of external diameter casted in a metallic case. The cylindrical grain has a circular central port perforation with an internal diameter of 4mm.

## 2.3 Apparatus

A 2D radial micro burner was designed in SPLab as shown in Fig. 1. Flame visualization and time-resolved regression rate can be achieved by this setup. Mass flow rate of oxidizer and chamber pressure can be controlled independently, thus experiment can easily be carried out under different situations.

All of the regression tests were conducted in a stainless steel cylinder chamber. Progress of combustion was monitored by a 45 degree mirror. Mass flow of oxidizer flow injected into chamber was controlled by a mass flow meter. Nitrogen injected into the chamber was used to provide combustion pressure and prevent soot deposition hindering the burning process visualization. Nitrogen can be used to stop combustion when the oxidizer was cut off as well. Pressure of chamber was sustained at needed value by 4 electro valves.

Ignition was achieved by the combustion of a pyrotechnic primer charge, which was inserted in the central port of the fuel. The primer charge was ignited by CO<sub>2</sub> laser. Progress of combustion was recorded by a high speed camera (500 frames per second), and regression rate could be calculated from this video.

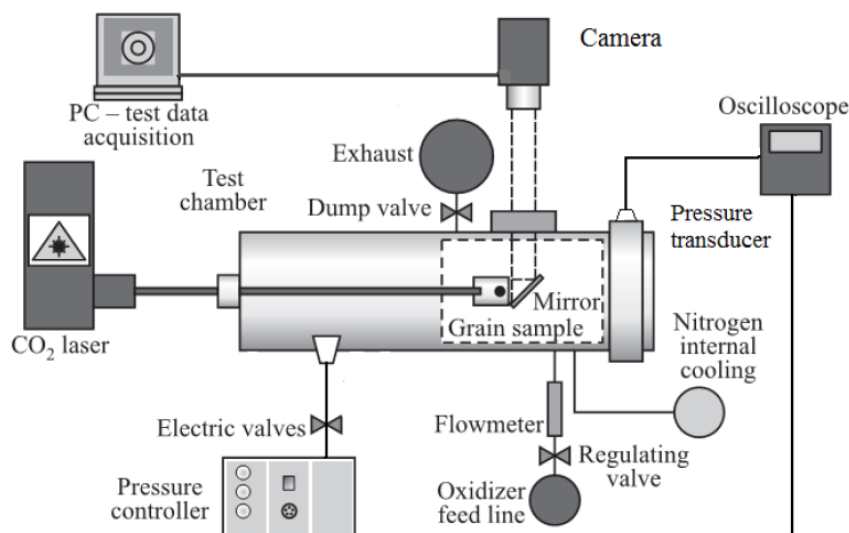


Figure 1: Scheme of experiment instrument

The setup for ignition test is quite similar to that for regression experiment. Mass flow of oxygen injected into the chamber was controlled by a flow meter, and it was adjusted to 5 LPM during all of the ignition tests. A continuous CO<sub>2</sub> laser emits a beam with a wavelength of 10.64  $\mu\text{m}$  and spot diameter of 1.5 cm; however, only 4 mm in the center of the beam which is nearly uniform was used to ignite the fuel. Power of the laser could be varied from 0~180 W by adjusting the current through the laser discharge tube. Two optical sensors were used, one is for the signal of laser and the other is for ignition of fuel.

## 3. RESULTS AND DISCUSSION

### 3.1 Ignition Delay

Typical result of ignition test is shown in Fig. 2. Beam emit from the laser becomes stable in about 5ms, and it could be used for ignition test of hybrid fuels. The time between the start of stable laser and the first flame of the fuel was

regarded as ignition delay. Signal for the flame of the fuel after the ignition is nearly a straight line, suggesting that fuel was burned stably as soon as it was ignited.

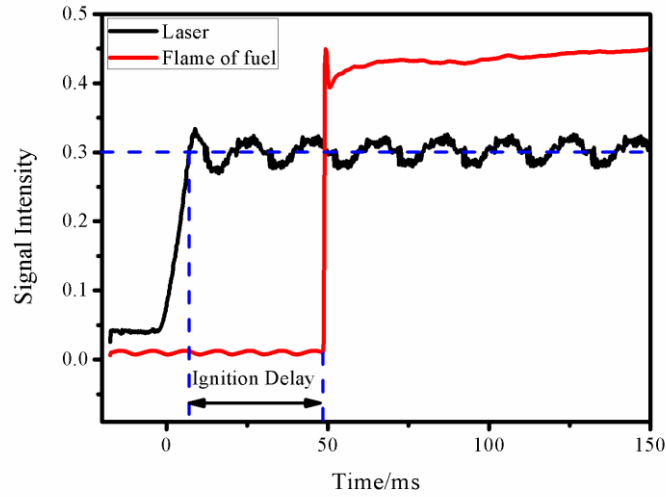


Figure 2: Typical result of ignition test (HTPB with the radiant flux of  $111 \text{ W/cm}^2$  is shown here)

In Fig. 3, ignition delay of solid fuels at 0.1 MPa and 1 MPa is presented. Ignition delay of all of the fuels was decreased with the increasing of radiant flux of laser. Relationship between ignition delay ( $t_i$ ) and radiant flux ( $q$ ) can be described by equation (2), which is quite similar to that of solid propellant.

$$t_i = Aq^{-n} \quad (2)$$

where  $A$  and  $n < 2$  are constants.  $A$  and  $n$  of all the fuels tested are listed in Table 3. As you can see from Table 3, all of the index number  $n$  is very close to 1.0, however, the factor  $A$  for each fuel is quite different.

In general, metal additives have positive effect on decreasing ignition delay of HTPB fuels; nevertheless, ignition delay were not shortened greatly with the help of metal additives (less than 30%) indicating that Mg, MgB and am\_Al are insensitive to  $\text{CO}_2$  laser irradiation, and the absorption effect of these metal particles at  $10.6 \mu\text{m}$  laser irradiation is less than that of carbon black [9, 18-19]. Magnesium, as a conventional metal additive shows better effect on decreasing ignition delay than all of the other additives.

Ignition delay was decreased greatly as the ambient pressure was increased from 0.1 MPa to 1 MPa as you can see from Fig. 3. Taking HTPB for example, with radiant flux of  $80 \text{ W/cm}^2$ , ignition delay was decreased from 48.33 ms at 0.1 MPa to 27.04 ms at 1 MPa. High pressure dependence of hybrid fuel's ignition delay is consistent with the results of epoxy under  $\text{CO}_2$  laser irradiation, conducted by T. J. Ohlemiller and M. Summerfield in 1971 [9]. Since the distance between reaction zone and solid phase was decreased with the increasing of pressure, this could be one reason for the pressure dependence. D. Healy et al. [17] reported that the ignition temperature of  $n\text{-C}_4\text{H}_{10}$  decreased with the increasing of ambient pressure. If this is true to hybrid fuel, lower ignition temperature at higher pressure is main reason for high pressure dependence of ignition delay.

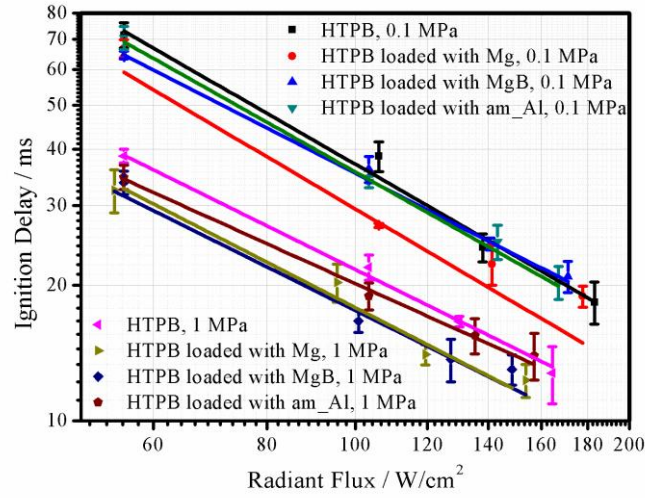


Figure 3: Ignition delay of solid fuels at 0.1 MPa and 1 MPa under CO<sub>2</sub> laser irradiation; O<sub>2</sub>, 5 LPM

Table 3: Solid fuels ignition in oxygen at 0.1 MPa and 1 MPa, relationship between ignition delay vs radiant flux

Fuel	Pressure/MPa	A	n	R <sup>2</sup> , Eq.(1)
HTPB	0.1	7702.7 ± 3387.8	1.159 ± 0.095	0.971
	1.0	2130.48 ± 265.17	0.997 ± 0.027	0.997
HTPB loaded with Mg	0.1	7045.15 ± 7738.19	1.189 ± 0.236	0.847
	1.0	1740.19 ± 1194.69	0.998 ± 0.146	0.903
HTPB loaded with MgB	0.1	4038.45 ± 419.79	1.029 ± 0.024	0.998
	1.0	2100.5 ± 1089.41	1.036 ± 0.114	0.951
HTPB loaded with am_Al	0.1	4123.19 ± 1104.67	1.134 ± 0.047	0.993
	1.0	1334.46 ± 264.16	0.911 ± 0.044	0.991

### 3.2 Ballistic Characterization

More than three tests were carried out for each fuel at both 1 MPa and 1.9 MPa in order to avoid possible errors. Theory and method for measuring regression rate from recorded video were described in full detail elsewhere [1, 15]. Error bar for each ensemble curve was drawn in the range of Gox, where at least two tests were conducted. In order to compare the regression rate of fuel at different pressure, variation of regression rate from 1 MPa to 1.9 MPa ( $\Delta r_f$ ) was defined as:

$$\Delta r_f = \frac{r_{19} - r_{10}}{r_{10}} * 100\% \quad (3)$$

Where  $r_{10}$  is the regression rate of fuel at 1 MPa;  $r_{19}$  is the regression rate of fuel at 1.9 MPa.

### 3.2.1 Pressure Effect

Fig. 4 illustrates that the regression rate of HTPB loaded with am\_Al vs Gox at 1 MPa and 1.9 MPa. Four tests were conducted at 1 MPa, and the same number of tests was carried out at 1.9 MPa as well. As you can see from Fig. 4, regression rate at different pressures are very close over the whole range of oxidizer mass flux considered in this paper. The average  $\Delta r_f$  is less than 10% over the whole range of Gox tested, and  $\Delta r_f$  is decreased to 0 when Gox comes to 262 Kg/(m<sup>2</sup> · s). Thus, we can come to the conclusion that regression rate of HTPB loaded with am\_Al is independent on pressure.

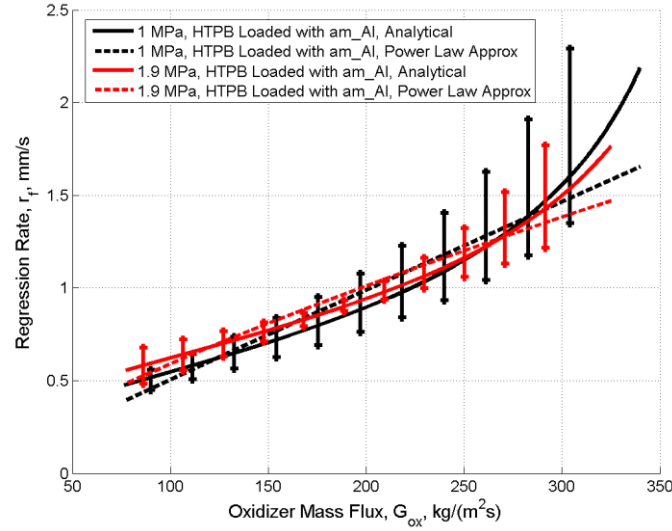


Fig. 4 HTPB loaded with 14.2% am\_Al burning in GOX with pc of 1 MPa and 1.9 MPa,  $r_f$  vs.  $G_{ox}$  (ensemble average;) Variation of regression rate of all of the fuels over the whole range of Gox investigated in this study is presented in Table 4. In general, regression rate of fuel was increased a little as the ambient pressure was increased from 1 MPa to 1.9 MPa (except HTPB loaded with am\_Al with the Gox of 250 Kg/(m<sup>2</sup> · s), however, almost all of the  $\Delta r_f$  are less

or close to 10% as you can see from the table. The biggest  $\Delta r_f$  is  $16.931 \pm 0.640\%$  as HTPB loaded with Mg with the Gox of 250 Kg/(m<sup>2</sup> · s). Thus, one conclusion can be drawn that, regression rates of HTPB and HTPB loaded with micron sized metal particles have a weak pressure dependence.

Table 4: Variation of regression rate of fuels

Gox /Kg/m <sup>2</sup> s	Pressure /MPa	Regression Rate /mm/s			
		HTPB	HTPB loaded with Mg	HTPB loaded with MgB	HTPB loaded with am_Al
100	1.0	$0.435 \pm 0.038$	$0.534 \pm 0.065$	$0.507 \pm 0.074$	$0.535 \pm 0.061$
	1.9	$0.475 \pm 0.034$	$0.586 \pm 0.098$	N/A	$0.619 \pm 0.088$
	$\Delta r_f, \%$	$9.195 \pm 1.578$	$9.738 \pm 4.551$	N/A	$15.071 \pm 2.815$
150	1.0	$0.536 \pm 0.029$	$0.660 \pm 0.052$	$0.623 \pm 0.074$	$0.714 \pm 0.102$
	1.9	$0.590 \pm 0.048$	$0.735 \pm 0.058$	$0.622 \pm 0.042$	$0.770 \pm 0.050$
	$\Delta r_f, \%$	$10.075 \pm 2.725$	$11.364 \pm 0.012$	$-0.159 \pm 5.086$	$7.843 \pm 7.792$

(Table continued on next page)

Table 4 (cont): Variation of regression rate of fuels

G <sub>ox</sub> /Kg/m <sup>2</sup> s	Pressure /MPa	Regression Rate /mm/s			
		HTPB	HTPB loaded with Mg	HTPB loaded with MgB	HTPB loaded with am_Al
200	1.0	0.651 ± 0.044	0.810 ± 0.056	0.755 ± 0.081	0.934 ± 0.163
	1.9	0.722 ± 0.077	0.922 ± 0.033	0.774 ± 0.080	0.946 ± 0.037
	Δ r <sub>f</sub> , %	10.906 ± 3.906	13.827 ± 3.334	2.540 ± 0.383	1.285 ± 13.541
250	1.0	0.801 ± 0.090	1.010 ± 0.103	0.927 ± 0.109	1.243 ± 0.261
	1.9	0.895 ± 0.126	1.181 ± 0.128	0.982 ± 0.171	1.188 ± 0.129
	Δ r <sub>f</sub> , %	11.735 ± 2.842	16.931 ± 0.640	5.915 ± 5.676	-4.425 ± 10.139
300	1.0	1.035 ± 0.181	1.324 ± 0.216	1.191 ± 0.181	1.761 ± 0.449
	1.9	1.165 ± 0.216	N/A	N/A	N/A
	Δ r <sub>f</sub> , %	12.560 ± 1.053	N/A	N/A	N/A

### 3.2.2 Additives Effect

Regression rate and mass burning rate of fuels at 1.9 MPa are similar to that at 1 MPa since regression rate was not increased greatly as the ambient pressure increased from 1 MPa to 1.9 MPa as previously stated. Therefore, additives effect on ballistic characterization will be focused on the pressure of 1 MPa.

In Fig. 5, the linear regression rate of all of the fuels at 1 MPa is presented. In addition, the mass burning rate of fuels at 1 MPa is presented in Fig. 6. All of the energetic material additives have a positive effect on enhancing regression rate. Among all of the additives, Mg is the only conventional material which was designed to compare with the other two innovative micron sized additives. As you see from Fig. 5, HTPB loaded with am\_Al demonstrated higher regression rate over the whole range of G<sub>ox</sub> investigated in this study, however, regression rate of HTPB loaded with MgB is a little lower than that of HTPB loaded with Mg.

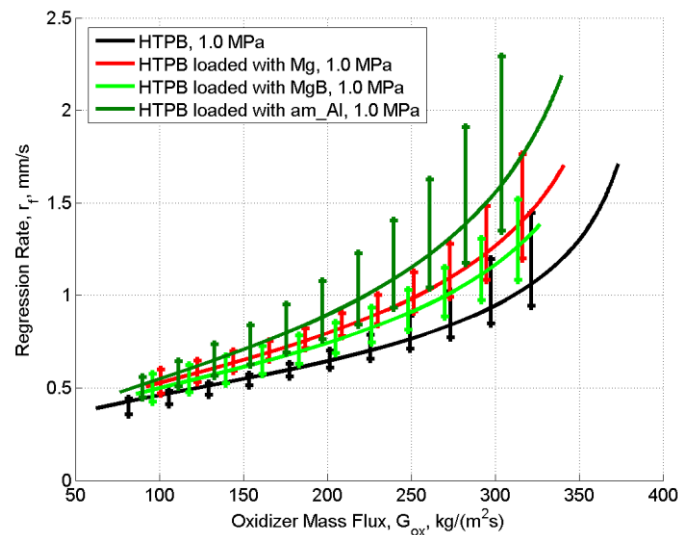


Figure 5: Comparison of linear regression rate for various fuel formulations at 1 MPa (ensemble average)

Similar to linear regression rate, as shown in Fig. 6, HTPB demonstrated the lowest average mass burning rate.

Average mass burning rate of HTPB loaded with Mg is a little higher than that of HTPB loaded with MgB over the whole range of oxidizer mass flux. As expected, HTPB loaded with am\_Al demonstrated higher mass burning rate of



all of the other fuels. Because of small scale of the fuel in our study, mass burning rate of fuels here is much lower than that of G. A. Risha's [4, 5].

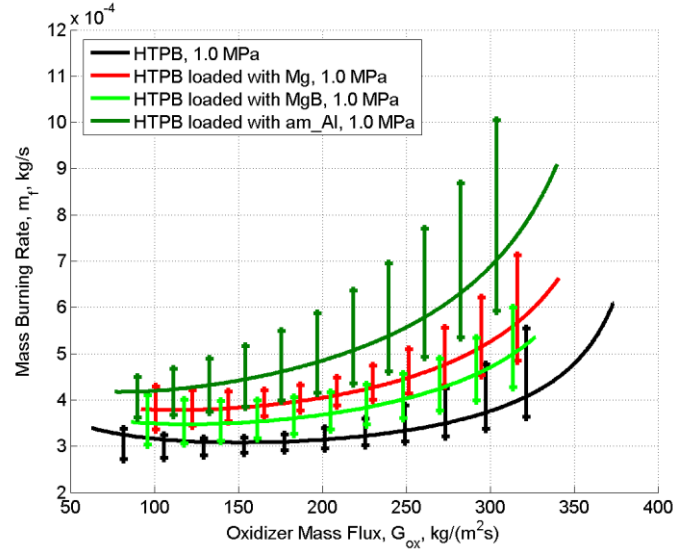


Figure 6: Comparison of mass burning rate for various fuel formulations at 1 MPa (ensemble average)

Percentage increase of linear regression rate of the fuels loaded with energetic additives at different oxidizer mass flux compared to HTPB is presented in Fig. 7. Fig. 8 shows the comparison between the percentage increase of mass burning rate of fuels loaded with micron sized additives and HTPB without additives. Both the percentage increase of regression rate and that of mass burning rate of all of the fuels increased with the increasing of oxidizer mass flux. HTPB loaded with am\_Al demonstrated mass burning rate 24.6% higher than HTPB at the  $G_{ox}$  of 100 Kg/(m<sup>2</sup> · s), which is lower than the value of 61% of HTPB loaded with 13% nano\_Al at the  $G_{ox}$  of 112 Kg/(m<sup>2</sup> · s) conducted by G. A. Risha [5]. However, when  $G_{ox}$  comes to 300 Kg/(m<sup>2</sup> · s), the percentage increase was increase to 45.6%, suggesting that am\_Al is a good promising micron sized material to enhance both linear regression rate and mass burning rate of HTPB based fuels. Percentage increase of mass burning rate of HTPB loaded with Mg is over the range of 16% to 28%. HTPB loaded with MgB demonstrated less than 20% increase of mass burning rate compared to HTPB in the range tested; however, pay attention to the fact that, L. Fanton et al. [8] from SPLab demonstrated that mass burning rate of HTPB loaded with 2.8% MgB (mass fraction) was increased by 51.7% with  $G_{ox}$  of 100 Kg/(m<sup>2</sup> · s) compared to pure HTPB fuel in 2012. Effect of MgB on enhancing regression rate here is quite lower than the result obtained by L. Fanton because of its aging problem as we can see from the series study at different time in SPLab [8, 15, 20].

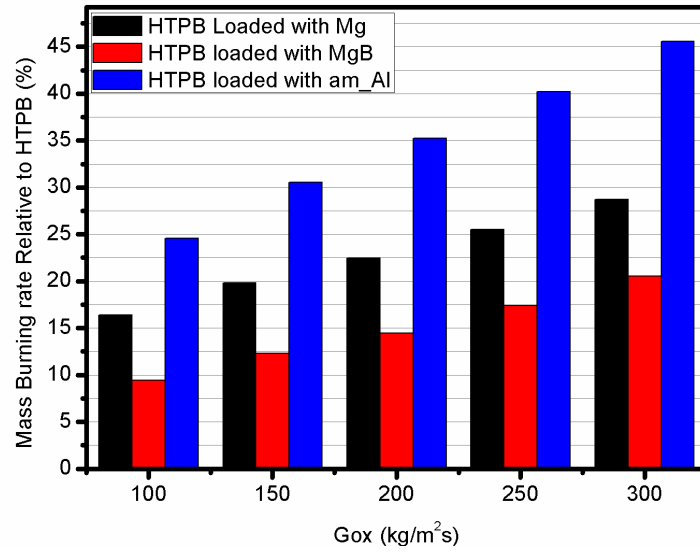


Figure 7: Percentage increase of regression rate of fuels loaded with additives compared to HTPB at 1 MPa

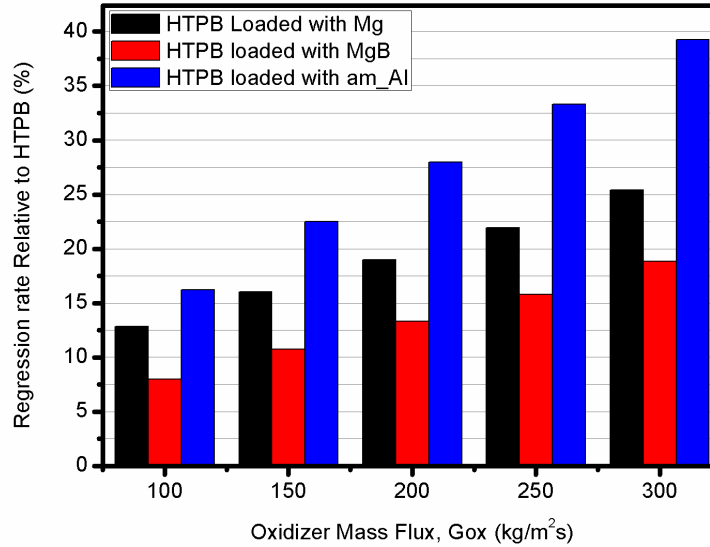


Figure 8: Percentage increase of mass burning rate of fuels loaded with additives compared to HTPB at 1 MPa

#### 4. Conclusions and Future Work

Ignition and ballistic characterization of HTPB loaded with micron sized energetic materials have been carried out. All of the micron sized powders showed a positive effect on shorting the ignition delay, which was decreased with the increasing of radiant flux. Ignition delay of fuels is highly dependent on pressure, and the ignition delay of fuels at 1 MPa is nearly 1/2 of that at 0.1 MPa.

Regression rate of fuels was increased a little when the ambient pressure was increased from 1 MPa to 1.9 MPa, and almost all of the variation of regression rate is less or close to 10%, suggesting that regression rate of HTPB and HTPB loaded with micron sized particles have a weak pressure dependence.

Micron sized additives have a positive effect on enhancing both regression rate and mass burning rate of the fuel.

Am\_Al is the best material to increase the mass burning rate of fuel, and the mass burning rate of it was increased by 45.6% at the Gox of 300 Kg/(m<sup>2</sup> • s) compared to that of HTPB. Due to aging problem, the innovative micron sized

material MgB demonstrated weaker effect on enhancing both regression rate and mass burning rate compared to Mg powder.

An interesting further work would be an extended study on regression of fuels loaded with am<sub>2</sub>Al, especially for the its mechanism on enhancing regression rate.

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