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Factors Affecting the Measurement of the Percentage of Gaseous Products from Boron-based Fuel-rich Propellants

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Abstract: Acid-washed asbestos, carbon fibre, and MgO with carbon fibre were used as the filter media in order to compare their filtering qualities in the estimation of the percentage of gaseous products (PGP) arising from the combustion of single-base propellants, double-base propellants, and boron-based fuel-rich propellants. The comparison was based on an analysis of the experimentally registered influence of the propellant formulation, the propellant load, the maximum chamber pressure and the thickness of the MgO filter layer on the PGP from the fuel-rich propellant, and in particular on the PGP produced by combustion of boron-based fuel-rich propellant. The results showed that the experimental values of the PGP were closer to the theoretically predicted values when carbon fibre mixed with MgO powder was used as the filter medium. The PGP of boron-based fuel-rich propellant increased when the AP was in part replaced by HMX, when the AP content was increased and when boron was in part replaced by magnesium-aluminum alloy. In terms of the apparatus used in these experiments, the propellant loading density was found to have little correspondence with the PGP for boron-based fuel-rich propellant. The optimal propellant loading density for the chamber volume of 85 cm^3 was found to be 2-2.5 g, in view of the reliability and safety of the experiment. It is emphasised that the thickness of the MgO filter layer is very important for the accuracy and reliability of the experiment, and that the optimum should be determined by experiment.

Keywords: fuel-rich propellant, percentage of gaseous products, combustion, filter media

1 Introduction

In the development of solid rocket ramjets, many researchers have studied the combustion behavior of solid fuel-rich propellants, including both aluminum magnesium and boron-based fuel-rich propellants [1, 2]. Boron-based fuel-rich propellants are regarded as the best energy source for solid rocket ramjets because they offer a high energy density [3-5]. The combustion of fuel-rich propellants can be divided into two processes, the primary combustion process carried out in the gas generator with the oxygen needed coming from the oxidizer in the propellant. Primary products with high energy are generated during the primary combustion process, and the secondary combustion process takes place when these products, ejected to the secondary combustion chamber, react with the inhaled oxygen from the environment. Therefore, the study of the primary combustion process could have significance not only for research into the combustion mechanism of a fuel-rich propellant during the primary combustion process, but also provide an underlying basis for the research of the secondary combustion process [6, 7].

Considering the lower oxidizer content of fuel-rich propellants, most of the primary combustion products exist in the form of a condensed phase. The percentage of the gaseous products (PGP) refers to the percentage of gaseous products formed during the primary combustion process of a fuel-rich propellant. Consequently PGP could provide not only a parameter for estimating the combustion performance of fuel-rich propellants, but also yield experimental data for comparison with the thermodynamically calculated results for a given propellant [8]. The PGP of fuel-rich propellants has usually been measured in a closed bomb, but such experiments cannot provide accurate results. Firstly because the condensed phase products and the gaseous products cannot be separated during the combustion process and they will therefore react with each other after the combustion because of the high temperature and low volume. Secondly because a closed bomb is a constant volume system and the pressure can change greatly during the combustion process. Usually, the PGP of fuel-rich propellants changes with pressure and only the PGP under an identified pressure is helpful.

Wang *et al.* [9] established a new PGP test apparatus for fuel-rich propellants, which can separate gaseous products from condensed products under the hot conditions and the chamber pressure can be controlled to some extent. In the present paper, different filter media were chosen to validate their filtering ability by measurements on single-base and double-base propellants. The measurement of the PGP for boron-based fuel-rich propellant was carried out after the optimization of the filter medium, and the factors that affect the PGP were also studied in this investigation.

2 Measurement of the PGP for Fuel-rich Propellants

2.1 Principle and test apparatus

Fuel-rich propellants generate plenty of condensed phase products during the primary combustion process because of the low oxidizer content which results in a low PGP (about 30%). Therefore, an effective separation of condensed phase products from the gaseous ones is very important for the measurement of PGP. Moreover, some gaseous products with low boiling points (H_2O, $MgCl_2$, *etc.*) are generated, and the temperature of the test apparatus may affect the state of these products. These products exist in the gaseous state in the gas generator because of the high combustion temperature (about 2000 K), so the condensed phase products and gaseous products must be separated in the hot state in order to avoid any phase change during the combustion and separation process.

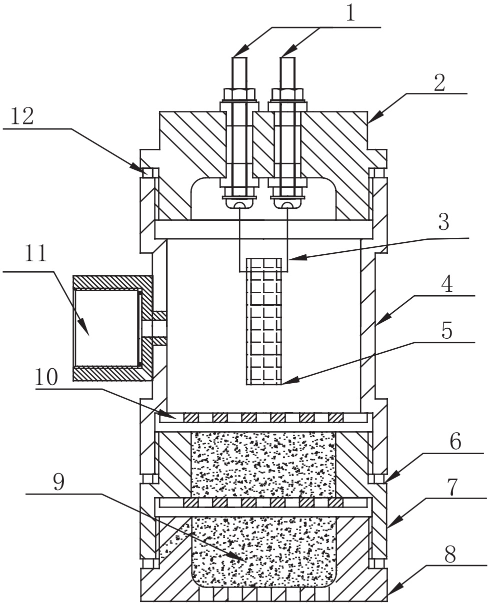

Figure 1. PGP test apparatus for fuel-rich propellants: 1 – ignition electrodes, 2 – top cap, 3 – ignition fuse, 4 – combustion chamber, 5 – propellant, 6 and 12 – copper shim, 7 – the first filter, 8 – the second filter, 9 – filter medium, 10 – plate with round holes, 11 – pressure tap.

Figure 1 is the PGP test apparatus for fuel-rich propellants. It is mainly composed of an ignition module (1 and 3), a combustion chamber (4) and a filter module (7-9). The filter medium is loaded into each filter and prevents the condensed phase products from passing through the filters, while the gaseous products can pass easily. The number of filters and the type of filter medium can be varied to capture all of the condensed phase materials; the thickness of the filter medium in each filter is about 30 mm.

According to the test runs, three filters are usually required to guarantee the filtering quality and the accuracy of the measurement. Plates with 5 mm round holes (10) between the filters prevent the filter medium from moving from one filter to another, thus preserving different filter layers. High-temperature silica glass cloth is used to prevent the filter medium from being ejected from the apparatus. N_i-C_d ignition fuse is employed to ignite the fuel-rich propellant. The propellant combusts after ignition and the pressure difference causes the gaseous products to pass rapidly through the filters. Because of the high temperature of the combustion products and the thinness of the filter layer, the high temperature of the gaseous products ejected from the test setup is maintained, ensuring that the products with low boiling points do not condensed.

2.2 Experimental procedure

The experimental procedure of the PGP measurement is as follows:

(1) Weigh the propellant sample; the mass is expressed as m_p. Cut one end of the propellant sample with a sharp blade and install the ignition fuse in the cut. Bind the ignition fuse and the ignition electrode strongly to ensure successful ignition.

(2) Load the dried filter medium into the filters; pack the filter medium tightly in each filter to guarantee the filtering quality.

(3) Assemble the PGP test apparatus tightly according to the assembly drawing; weigh the apparatus, the result being specified as m_0.

(4) Ignite the propellant using a 24 V power source.

(5) Weigh the mass of apparatus after combustion, the result being expressed as m_1.

(6) Calculate PGP from: $\psi = \dfrac{m_0 - m_1}{m_p}$.

(7) Repeat the experiment no less than five times; the averaged value of PGP is regarded as the final result.

3 Optimization of the Filter Media

3.1 Selection criterion of filter media

Acid-washed asbestos is usually used as the filter medium in industry and the laboratory, and it is employed as the filter medium in the measurement of PGP due to it having the following advantages [10]: (1) acid-washed asbestos is resistant to high temperatures, acidic and basic materials, and does not react with the hot combustion products; (2) the loose texture and the fibrous form could facilitate the filtering quality; and (3) the low density decreases the mass of the test facility, which could improve the accuracy of the experiment.

Although acid-washed asbestos has many advantages, the measured PGP value is usually smaller than the theoretically calculated one and the deviation of each experimental value is significant. Acid-washed asbestos has high hygroscopicity and some moisture cannot be eliminated from the test apparatus. Furthermore, the thermal conductivity of acid-washed asbestos is high; therefore the temperature of the gaseous products decreases significantly when they are expelled through the apparatus and a portion of the water vapour can be absorbed by the acid-washed asbestos. In addition, as a potential health hazard, acid-washed asbestos has been banned in many countries [11]. So using acid-washed asbestos as a filter medium cannot meet all requirements.

Carbon fibre has the excellent advantages of thermostability, corrosion resistance, and heat resistance [12]. Moreover, the hygroscopicity of carbon fibre is lower than that of acid-washed asbestos. Thus, carbon fibre may be a better filter medium. In this research, carbon fibre (7 µm in diameter and 2-20 mm in length, fabricated by NanJing WeiDa Composite Material Co., Ltd) was chosen as the candidate filter medium. It had a surface area of 352 m^3/g.

Considering that the surface of carbon fibre is smooth and that the interspace between the fibres is to some extent large, the filtering quality of carbon fibre alone may not be reliable. In order to enhance the filtering quality, one experimental strategy was to use fine granular MgO particles (AR, Aladdin), with a median grain diameter of 0.85 µm and surface area of 78 m^3/g, as part of the filter system. MgO particles were placed in the first filter with some carbon fibre being placed at the front of the MgO layer to prevent the flame from touching the MgO particles. Moreover, carbon fibre was placed at the back of the final filter to ensure that the MgO was not expelled from the apparatus.

3.2 Filtering quality of different filter media for single-base propellant and double-base propellant

A large amount of gaseous products are generated during the combustion of single-base propellant and double-base propellant, and the filter medium will be appropriate for the measurement of fuel-rich propellants if the filter medium performs well for single-base propellant and double-base propellant. In addition, the PGPs of single-base propellant and double-base propellant can be obtained accurately by calculation, and thus the reliability of the measurements can be easily validated. The single-base propellant (provided by Xi'an Modern Chemistry Research Institute) consisted of nitrocellulose (94%), volatile matter (1.8%), diphenylamine (2%), camphor (1.7%) and graphite (0.3%); the volatile matter refers to residues of the alcohol-ether solution. The formulation of double-base propellant (also provided by Xi'an Modern Chemistry Research Institute) was nitrocellulose (54.5%), nitroglycerine (26.5%), dinitrotoluene (12%), centralite (3.0%), petroleum (1.0%), calcium carbonate (1.8%) and lead oxide (1.2%).

The PGPs of the two propellants were calculated by CEA software [13, 14] at a pressure of 2 MPa (99.5% and 90.03%, respectively). A 2 g propellant sample was used for each measurement and the results are shown in Table 1.

Table 1. PGPs of single-base propellant and double-base propellant

Experiment No.	Propellant species	Filter media	PGP/%	Relative error /%
1	single-base propellant	acid-washed asbestos	90.71	8.83
2	single-base propellant	carbon fibre	89.83	9.72
3	single-base propellant	MgO + carbon fibre	98.47	1.04
4	double-base propellant	acid-washed asbestos	86.53	3.89
5	double-base propellant	carbon fibre	87.67	2.62
6	double-base propellant	MgO + carbon fibre	89.36	0.744

Table 1 indicates that the experimental value of PGP using MgO + carbon fibre as the filter medium for single-base propellant is in good agreement with the theoretically calculated one. The gaseous products expelled from the test apparatus were colourless with MgO + carbon fibre as the filter medium, whilst they were orange coloured with the other filter media. The double-base propellant generates Pb^+ which leads to the orange colour of the gaseous products released, so the filtering ability of MgO + carbon fibre is the best of the three filter media.

The PGP value is lower when acid-washed asbestos or carbon fibre is used as the filter medium alone because they absorb the water vapour in the gaseous products. As the hygroscopicity of acid-washed asbestos is higher than that of carbon fibre, the experimental value is consequently lower. MgO has excellent filtering qualities because of its fine granularity and strong adsorption capacity. The experimental value of PGP with MgO + carbon fibre as the filter medium is higher than the one with only carbon fibre as the filter medium. The MgO layer is compact when MgO is put in the filter, which could increase the pressure difference between the filters, and higher pressure differences result in a higher transit speed of the gaseous products. In addition, the fine granularity of the MgO particles could decrease the flow distance of the gaseous products in a carbon fibre filter medium, so the amount of water vapour absorbed by the filter medium would be obviously decreased.

In order to determine the moisture adsorbed by different filter media during the experiments, the moisture content of the different filter media before and after three experiments was tested using Sartorius scale MA-100 moisture analyzers; the results are shown in Table 2.

Table 2. Moisture content of filter media

	Acid-washed asbestos	Carbon fibre	MgO + carbon fibre	
			MgO	carbon fibre
Moisture content before experiment / %	0.82	0.33	0.50	0.33
Moisture content after three experiment / %	6.50	1.89	0.58	0.45

From Table 2, it can be seen that the hygroscopicity of the acid-washed asbestos is greater than that of the carbon fibre, which makes the PGP values much lower than the ideal condition without moisture adsorption. Moreover, the moisture content of the carbon fibre decreases when fine MgO particles are added as the filter medium. The moisture content of MgO particles increases slightly after three experiments because of its low hygroscopicity and the higher temperatures of the MgO particles, since they are close to the propellant flame. Thus the MgO + carbon fibre can be regarded as the best filter medium from the viewpoint of hygroscopicity .

As the boiling point of water at atmospheric pressure is only 100 °C, attempts were made to decrease the water absorption of the filter medium by heating the apparatus. The apparatus with 2 g double-base propellant was placed in a muffle furnace at 110 °C (thermal decomposition may happen if the temperature is higher) with the furnace door unlocked, and MgO + carbon fibre was chosen as the filter medium. The propellant was ignited after half an hour and the mass of the test apparatus was measured immediately. The experimental results showed that the PGP of double-base propellant was 90.30%, and that the moisture content of the MgO and carbon fibre was 0.61 and 0.41, respectively. So water absorption cannot be avoided by increasing the temperature (110 °C).

High pressure DSC (METTLER TOLEDO HP 827) was used to obtain the vapour pressure diagram of water, at a heating rate of 5 °C /min; the results are shown in Figure 2.

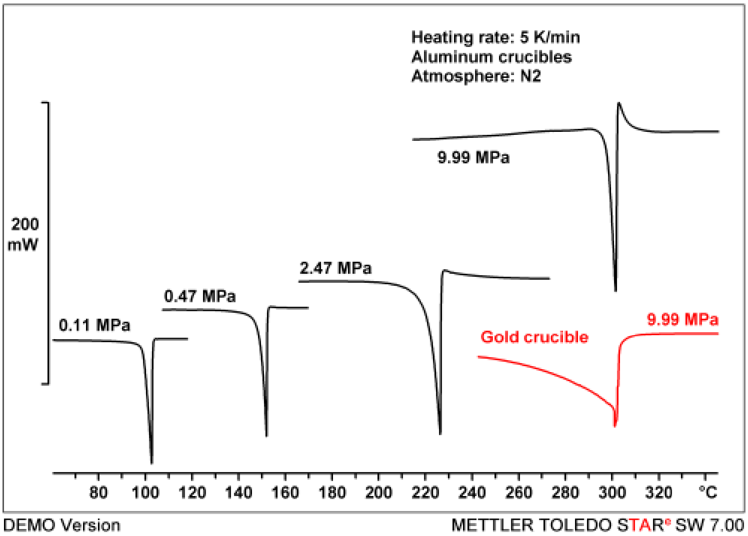

Figure 2. Vapor pressure diagram of water.

Figure 2 shows that the boiling point of water at 2.47 MPa is about 225 °C. The combustion temperature of double-base propellant is 2351 °C (measured by a fine tungsten-rhenium thermocouple), and the temperature of the gaseous products should not change greatly during the gas discharge because the filter medium has low thermal conductivity and the time that the gaseous products remain in the filters is very short. When the gaseous products flow through the first filter with an aggregate of MgO particles, the temperature of the gaseous products is much higher than 225 °C. However the temperature of the gaseous mixture obviously decreases and the boiling temperature is decreased slightly when the gaseous products pass through the carbon fibres. Therefore, the moisture content of the filter medium does not change significantly when the test apparatus is heated to 110 °C. Moreover, the chamber pressure during the combustion process was not measured because the operating temperature of the pressure transducers cannot exceed 80 °C, but the higher PGP values may be the result of higher chamber pressure caused by the increased operation temperature.

3.3 Filtering quality vs PGP value of boron-based fuel-rich propellants with different filter media

The apparatus with different filter media was developed to measure the PGP value of boron-based fuel-rich propellants, in order to check the filtering qualities when large amounts of condensed phase products are present. Two different boron-based fuel-rich propellants were chosen, and the formulations of these are shown in Table 4. A 2 g propellant sample was used in each experiment, and the results are shown in Table 3.

Table 3. PGP values of boron-based fuel-rich propellants

Propellant sample	Filter media	PGP / %	Relative error / %
#1	acid-washed asbestos	25.57	1.07
#1	carbon fibre	26.59	1.85
#1	MgO + carbon fibre	30.99	1.19
#2	acid-washed asbestos	26.40	1.15
#2	carbon fibre	27.67	1.39
#2	MgO + carbon fibre	33.37	0.30

Table 3 indicates that the PGPs of #1 propellant and #2 propellant are highest when MgO + carbon fibre is used as the filter medium. A transparent plastic bag was used to collect the gaseous products expelled from the test apparatus; neither smoke nor carbon fibre was expelled from the test apparatus. Moreover, the experimental values were closer to the theoretically calculated ones based on thermodynamic laws. So MgO + carbon fibre can be supposed to be the best filter medium for the measurement of PGPs for fuel-rich propellants because of its low hygroscopicity and excellent filtering qualities.

4 Factors Affecting the PGP of Boron-based Fuel-rich Propellant

4.1 Influence of the propellant ingredients

The propellant ingredients could have a significant influence on the PGP. This influence is complex because there are many ingredients in a boron-based fuel-rich propellant. In this paper, six different boron-based fuel-rich propellants were processed in order to study the effect of ingredients on the PGPs of these propellants and the formulations of these propellants is shown in Table 4. The purity of boron was 95%, and the purity of all of the other added metal components was 97%.

Table 4. The formulations of the boron-based fuel-rich propellants

Sample No.	HTPB / %	AP / %	B / %	Al / %	Mg / %	Mg-Al alloy / %	HMX / %
#1	29	33	33	0	0	5	0
#2	29	28	33	0	0	5	5
#3	29	33	33	5	0	0	0
#4	29	33	33	0	5	0	0
#5	29	33	30	0	0	8	0
#6	29	28	38	0	0	5	0

Mg-Al alloy: Mg:Al=1:1 (by wt.)

The PGPs of these propellants were measured with MgO + carbon fibre as the filter medium, and a 2 g propellant sample was used in each experiment. The results are shown in Table 5.

Table 5. PGPs of different boron-based fuel-rich propellants

Propellant No.	PGP / %	Relative error / %
#1	30.99	1.19
#2	33.37	0.30
#3	28.53	0.87
#4	29.22	0.96
#5	32.74	0.72
#6	26.83	0.46

AP and HTPB are usually used as oxidizer and binder respectively in composite propellants, which is also the case for boron-based fuel-rich propellants, and metal additives are used to improve the performance of the ignition and combustion of the propellant.

HMX is usually added to composite propellants to enhance their energy, because HMX produces more gaseous products (908 L/kg) than AP (790 L/kg) and the standard enthalpy of formation of HMX (252.8 kJ/kg) is higher than that of AP (-2527.9 kJ/kg) [15]. Table 6 indicates that the PGP increases when 5% HMX is used to replace part of the AP, but HMX could not be added in large amounts to boron-based fuel-rich propellant as HMX cannot provide enough oxygen for the combustion of the propellant.

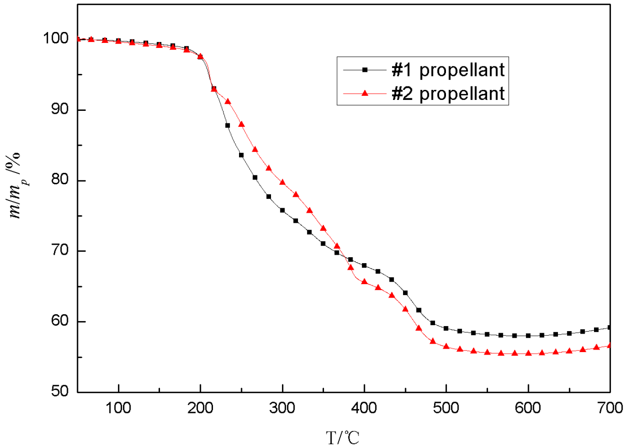

Figure 3. The TGA curves of #1 propellant and #2 propellant.

Figure 3 shows the TGA curves of #1 propellant and #2 propellant under an argon atmosphere (heating rate 20 °C/min, sample mass 3 mg). Figure 3 suggests that #2 propellant has a higher mass-loss rate than #1 propellant, which supports the conclusion that replacing a small part of AP with HMX could increase the PGP value of boron-based fuel rich propellants to some extent.

As metal additives, Mg could increase the PGP compared with aluminum, and magnesium-aluminum alloy also increases the PGP in relation to the magnesium-aluminum alloy content (the boron content is decreased correspondingly). Although the heat of combustion of magnesium is lower than that of aluminum, magnesium could produce more heat in a fuel-rich environment because of its low oxygen consumption and high combustion efficiency. The thermal decomposition of AP and HTPB is more extensive with an increased heat of reaction, and leads to an eventual increase in PGP. Similarly, an increase in magnesium-aluminum alloy content also increases the energy released, which also results in an increase in PGP to some extent.

Table 5 also shows that the PGP decreases with an increase in boron content (AP is decreased correspondingly). Boron is somewhat inactive during the primary combustion process because the ignition temperature of boron is about 1900 K and the combustion temperature of the propellant used here is about 1800 K. The combustion process is less complete when there is a decrease in the effective oxygen content of the propellant.

4.2 Effect of propellant load and maximum pressure on the PGP of boron-based fuel-rich propellant

The PGP of boron-based fuel-rich #1 propellant was measured with different loads of propellant, with MgO + carbon fibre as the filter medium. The results are shown in Table 6.

Table 6. PGP at different loads of boron-based fuel-rich #1 propellant

Propellant load / g	PGP / %	Relative error / %	Maximum pressure / MPa
1.5	30.97	3.20	1.8
2.0	30.99	1.19	2.5
2.5	31.25	1.67	2.9

Table 6 indicates that the propellant load has a mild influence on the PGP of boron-based fuel-rich propellant. However, the relative error is higher when the propellant load is 1.5 g because the lower chamber pressure could lead to less stable combustion to some degree.

The experimental hazard increases when the propellant load is more than 3 g because of the higher chamber pressure. In addition, boron-based fuel-rich propellant usually combusts at low pressure (less than 3 MPa), and the experiment cannot reflect the realistic combustion process if the propellant load is above 3 g. So the PGP should be determined at propellant loads between 2 and 2.5 g.

In order to explore the effect of the maximum pressure on the PGP of the boron-based fuel-rich propellant, strips of #1 propellant with different length-to-diameter ratios (from 1.5:1 to 6:1) were used, ensuring that the maximum chamber pressure and the mass of the propellant remained nearly constant. The results of experiments when the maximum chamber pressure was about 4.5 MPa and the weight of the propellant was 2 g are shown in Table 7.

Table 7. PGP of boron-based fuel-rich #1 propellant with different length-to-diameter ratios

No.	Weight of propellant / g	length-to-diameter ratio	Max pressure / MPa	PGP / %
1	2.01	1.5:1	4.5	30.99
2	2.03	3:1	3.2	30.62
3	2.00	4.5:1	2.3	30.01
4	2.03	6:1	1.6	29.73

Table 7 shows that the maximum pressure (or average pressure) has a negligible effect on the PGP of boron-based fuel-rich propellant; the PGP increases only very slightly with an increase in maximum pressure (or average pressure). This indicates that the higher pressure causes the chemical reactions to become more complete and to produce more gaseous products.

4.3 Influence of the thickness of the MgO layer on the PGP for boron-based fuel-rich propellant

At present, three layer filters are used in measuring the PGP in order to not only ensure adequate filtering quality but also to decrease the systematic error. The thickness of the MgO layer is important when MgO + carbon fibre is used as the filter medium. The PGP of boron-based fuel-rich #1 propellant (2 g samples) with different thickness of the MgO filter layer was measured. The results are shown in Table 8.

Table 8. PGP of #1 propellant with MgO filter medium of different thicknesses

Thickness of MgO filter layer	PGP / %	Relative error / %
Half a layer	33.54	2.68
One layer	30.99	1.19
Two layers	40.83	6.34

Table 8 shows that a thin layer of MgO has a different influence on the measured PGP. For one thing, the filtering quality is decreased when the filter layer is very thin, and some of the solid-phase products could have been expelled from the test apparatus. For another, the decreased pressure difference between the filters could lead to an increase in the moisture content in the filter medium. The thicker layer of MgO also has an influence on the PGP. On the one hand, the higher chamber pressure could increase the experimental hazard. On the other hand, the fine MgO could be expelled from the apparatus causing the PGP to have a higher value. Then the thickness of MgO layer is important for the measurement of PGP and the optimal thickness should be determined by the experiment.

5 Conclusions

(1) The experimental value of the PGP is closer to the realistic conditions with MgO + carbon fibre as the filter medium because of its excellent filtering qualities.

(2) Replacing a part of the AP with HMX in the boron based fuel-rich propellant could increase the PGP levels, and the PGP level could be increased with magnesium or magnesium-aluminum alloy.

(3) The propellant load has a mild influence on the PGP of boron-based fuel-rich propellant. However, the best propellant load is 2.0-2.5 g in view of the accuracy and safety of the experiment. The PGP level increases with an increase in the maximum chamber pressure.

(4) The thickness of the MgO layer is important when MgO + carbon fibre is chosen as the filter medium. The optimal thickness should be determined by the experiment.

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