Pressure-Time Study of Slow Burning Rate AP/HTPB Based Composite Propellant by Using Closed Vessel Test (CVT)

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Abstract. The Closed vessel (CV) is an equipment used to study the ballistic parameters by recording burning time history, pressure buildup during the process and vivacity of the propellants. It consists of strong pressure vessel, piezo-electric pressure transducers, sensors and dedicated software. To save time and resources this method is employed instead of dynamic firing while doing research and development of propellants. A measured amount of propellant charge is loaded in the vessel and fired remotely. Ignition is provided by the filament which ignites the black powder charge. In this study, we have used Closed Vessel Tests (CVT) for the first time for recording the ballistic parameters of slow burning composite rocket propellant. We developed a set of composite solid propellant samples containing a mixture of bimodal Ammonium Perchlorate (AP) as an oxidizer, Hydroxy-terminated Polybutadiene (HTPB) as a binder as well as fuel, Dioctyl Sebacate (DOS) as plasticizer, 1-(2-methyl) Aziridinyl Phosphine Oxide (MAPO) as bonding agent and Toluene Diisocyanate (TDI) as curator. Samples were developed by changing the solid loading percentage of bimodal AP particles. By increasing the percentage of AP, the oxidizer-fuel ratio (O/F) increases which affects the ballistic parameters. It is observed that maximum pressure and vivacity increases with increase in solid filler in the propellants. As quantity of AP increases, rate of rise of pressure also increases. CVT firing of each sample was done three times to obtain average burning time and pressure buildup history to evaluate the effect of oxidizer loadings on ballistic parameters of the composite propellant.

Introduction

Composite solid propellants with very high rate of burning produce a higher impulse by releasing combustion products in very short time enabling missiles to fly at high velocity. The propellants with slower rate of burning are less powerful and are produced for generating gases to enable flight control and in base bleed projectiles for artillery guns. Therefore, many studies have been directed toward the development of propellants with wide range of burning rates [1]. This is a class of energetic materials which produce high temperature combustion gaseous products and it finds application mainly in guns and rocket propulsion. The production of new kinds of propellants with improved properties as well as to maintain the quality of propellants being produced at large level demands the knowledge of ballistic parameters, pressure produced and vivacity [2]. There are many sophisticated and reliable experimental techniques for correct estimation of burning rate [3].

Composite propellant is mainly composed of HTPB binder, AP as oxidizer and Al powder as metal fuel [4]. Binder is cured commonly with Isophorone diisocyanate (IPDI) or Toluene diisocyanate (TDI). Plasticizer, bonding agent and burn rate modifiers are also important additives. Propellant burning rate is the most vital characteristic which determines the ballistic performance. It is affected by the oxidizer, burn rate modifier, and any metal fuel if used [5-7]. AP is very good oxidizer with long history of usage in solid composite propellants for space exploration, military

and high powered rocket systems for research. Researchers have been working on combustion mechanism of AP/HTPB propellants for years and the burning behavior is well known [8, 9]. The combustion process starts with the thermal decomposition of AP which generates gases at the propellant surface and the process is self-sustained [10, 11]. Burning characteristics of an AP/HTPB composite propellant is considerably affected by size of AP particles and its total percentage in propellant composition. It increases by reducing the AP particle diameter and it rises with increase in AP content. The burning rate of low burning rate composite propellants is very vital factor and becomes extremely important parameter to be considered for base bleed applications. Base bleed composite propellant fitted with artillery shells, starts burning as soon as the projectile is fired inside gun bore. There is very high pressure inside the gun bore that impact the performance of the base bleed propellant grain. Therefore, in order to achieve desired performance it is imperative to study combustion behavior of low burning rate propellant in high pressure conditions [12].

While doing research work on development of composite propellants for base bleed applications, dynamic firing is expensive, time consuming and unsafe. Therefore, the closed vessel examination of propellants is adopted to save time and resources, and for efficient analysis of propellant under high pressure conditions [13, 14] It is conducted by burning a specific mass of propellant charge as per loading charge density of the CV of known volume. The high-pressure buildup inside the CV is recorded by a piezo-electric pressure transducer against time from the time of ignition till the maximum pressure is reached. A dedicated system of charge amplifier and data acquisition system records the pressure- time ($p-t$) data for further evaluation.

Experimental

Propellant Formulation. All composite propellants investigated in this paper are composed of HTPB (15-19%) binder cum fuel, AP (75-84%) as oxidizer and Al powder (1%) as burning stabilizer and opacifier. TDI, DOS and MAPO were used as curing agent, plasticizer and bonding agent. Four propellant compositions were prepared by gradually increasing the percentage of AP filling. Each propellant composition is based on a bimodal AP size 250 μm (40%) and 340 μm (60%). Set of four samples with gradually increasing weight percentage of AP were synthesized, A-1 with 75%, A-2 with 78%, A-3 with 81% and A-4 with 84%. The burning rate is the most important property affecting the ballistic performance of composite propellant motors and most contributing ingredient is the oxidizer. It increases with increase in the AP content in the composition which ranges from 70 to 90 % by mass [8, 15].

Lab scale composite propellants were prepared. The binder and rest of the liquid additives excluding curing agent (TDI) were properly mixed for 30 minutes followed by addition of Al powder. This mixture was mixed for another 20 minutes then AP was added in two steps to ensure proper mixing. After adding complete solid ingredients, the composition was mixed for 30 minutes and placed in vacuum chamber to remove air bubbles. Finally curing agent was added and mixed followed by vacuum treatment for 15 minutes. All the samples were of 100 grams mass and prepared in Porcelain dishes by hand mixing with glass stirring rods. This final propellant material was cast into specific molds and was cured at 60 °C for 78 hours.

Closed Vessel System for Ballistic Parameters. A CV of 100 cm^3 volume with very strong stainless-steel body equipped with water cooling jacket on its outer surface was constructed shown as in Fig. 1. Vessel is equipped with piezo-electric pressure transducer, outlet valve, and breach screw fitted with ignition system. The closed vessel is positioned on a stand which allows it to move in 180-degree range for ease of handling, loading and unloading operations. This closed vessel is connected to an amplifier unit and complete data acquisition system for recording the change in $p-t$ profile. An ignition system is also connected to the CV to initiate the burning of black powder igniter bag inside the vessel for initiating the deflagration of propellant charge under safe and controlled condition. Complete CV system is as shown in Fig. 2.

Experimental Test Method. The AP/HTPB propellant were fired inside the CV at one standard solid loading which is the ratio of mass of the propellant charge to chamber volume. The firing block of the vessel has a nickel chrome wire which is soldered on the firing pin terminals. Black

powder bag is attached to the wire which gives ignition to the propellant charge. Constant vessel volume being 100 cm^3 was used to calculate the full loading mass. For each sample of composite propellant charge loadings of 50% of the full charge mass were fired in triplicate. The composite propellant samples were cut in equal small square sheets which were loaded in the vessel. According to the theory of constant volume, the propellant charge as per the loading density and required quantity of ignition powder of all the four is as shown in the Table 1.

Fig. 1. High pressure vessel.

Fig. 2. Schematic diagram of closed vessel with data recording system.

Table 1. Loading charge of closed vessel with ignition charge and propellant.

Loading Density $\Delta \text{ [g/cm}^3\text{]}$	Vessel Volume $[\text{cm}^3]$	Ignition Powder $[\text{g}]$	Full Propellant Loading Mass $[\text{g}]$	Propellant Mass to be Fired $[\%]$
0.2	100	1.5	20	50

Result and Discussion

For each sample with varying filler percentage, closed vessel firing average results are tabulated in Table 2. The CV firing gave results in the form of p - t profile. The recorded p - t profiles of four composite propellants having gradual increase in AP percentage are recorded and the comparative p - t curve based on mean values taken from three separate firing of each sample is shown in Fig. 3. From Fig. 3, it was observed that at standard loading density and fixed propellant charge mass, the pressure generated has an increasing trend with increasing contents of AP. Similarly, time to achieve maximum pressure (P_m) also reduces with increase in AP content.

Table 2. Closed vessel (100 cc) results.

Sample	AP [%]	Max Pressure [bar]	Rise Time [ms]	Maximum Differential Pressure, dP/dt
A-1	75%	374.70	718.00	4.69
A-2	78%	448.61	436.50	5.20
A-3	81%	611.97	124.00	10.98
A-4	84%	632.32	63.00	22.85

Fig. 3. Comparative p - t profile.

The combustion of higher content of oxidizer at a fixed charge weight in CV resulted in release of more energy. From the data obtained from p - t curve, differential pressure vs pressure is obtained as shown in Fig. 4. It can be observed that the rate of pressure rise is increasing with increase in the oxidizer percentage. This implies that the slope showing rate of change of pressure vs pressure rises with increasing AP loading of composite propellant.

Gradual increasing percentage of AP is the major factor here that affects the (O/F) ratio, as with increasing (O/F) ratio the rate of decomposition and pressure also increases. Consequently, this increase raises the rate of regression of burning surface area. This is also apparent in shape of rate of change of pressure (dP/dt) as shown in Fig. 4. The rate of increase of pressure vs pressure shown in Fig. 4. It is obvious that the propellant with higher oxidizer loading releases more energy and peak pressure is higher.

Propellant Vivacity. Vivacity is the rate of change of pressure with time (dP/dt) divided by P_m . The vivacity of a propellant is its efficiency in its deflagration and the capability to release energy. It is the rate of energy released on combustion of a propellant charge, it shows the complete and effective burning behavior of a propellant charge. The vivacity obtained from CVT has been plotted against P/P_m to explain the complete propellant charge burning behavior as shown in Fig. 5.

Burning behavior of propellant is an important parameter for propellant development and its diagnostics to compare it with reference or standard propellant. The pressure-time history gives the information about ballistic characteristics which can be used for experimental evaluation of propellant compositions. These ballistic characteristics help to differentiate the propellants on the basis of force and vivacity which ultimately give the quickness of a propellant charge. This high pressure leads to the higher vivacity which means propellant surface regression rate is higher and the propellant burns efficiently.

Fig. 4. The rate of change of pressure against pressure.

Conclusions

The experimental results show that the closed vessel technique is an efficient method to study the ballistic parameters of gun as well as solid composite propellants. A small amount of sample is loaded in vessel and is fired remotely with complete safety and process can be repeated in very short time for average and comparative data. The burning characteristics can be determined by firing propellants in closed vessel at predetermined loading mass. The closed vessel results showed pressure rise with time, rate of change of pressure and vivacity of the propellant samples under investigation. This method is very convenient for development and study of new propellant compositions and their behavior at high pressure and temperature conditions under controlled and safe firing. The study of prepared samples in comparison with a standard available reference propellant sample can also be done. This method is suitable for comparative evaluation of combustion behavior, $p-t$ profile and vivacity for development of improved propellant composition for specific applications.

Fig. 5. Vivacity vs P/P_m of complete samples.

References

- [1] S. Shioya, M. Kohga, T. Naya, Burning characteristics of ammonium perchlorate-based composite propellant supplemented with diatomaceous earth, *Comb. Flam.*, 161 (2014) 620-630.
- [2] A. Bougamra, H. Lu, Determination of Pressure Profile During Closed-Vessel Test Through Computational Fluid Dynamics Simulation, *J. Therm. Sci. Engineer. Appl.*, 8 (2016) 021005.
- [3] G. Gupta, L. Jawale, D. Mehilal, B. Bhattacharya, Various Methods for the Determination of the Burning Rates of Solid Propellants: an Overview, *Cent. Europ. J. Energ. Mater.*, 12 (2015).
- [4] S. Jain, M. Mehilal, S. Nandagopal, P. Singh, K. Radhakrishnan, B. Bhattacharya, Size and shape of ammonium perchlorate and their influence on properties of composite propellant, *Defen. Sci. J.*, 59 (2009) 294.
- [5] N. Kubota, T. Kuwahara, S. Miyazaki, K. Uchiyama, N. Hirata, Combustion wave structures of ammonium perchlorate composite propellants, *J. Propul. Pow.*, 2 (1986) 296-300.
- [6] E.W. Price, J. Sambamurthi, R. Sigman, R. Panyam, Combustion of ammonium perchlorate-polymer sandwiches, *Combust. Flam.*, 63 (1986) 381-413.
- [7] H. Hasegawa, M. Fukunaga, K. Kitagawa, T. Shimada, Burning rate anomaly of composite propellant grains, *Comb. Explo. Shock Wav.*, 49 (2013) 583-592.
- [8] M. Kohga, Burning characteristics and thermochemical behavior of AP/HTPB composite propellant using coarse and fine AP particles, *Prop. Explo. Pyrotech.*, 36 (2011) 57-64.
- [9] K.K. Kuo, M. Summerfield, *Fundamentals of solid-propellant combustion*, American Institute of Aeronautics and Astronautics New York, 1984.

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- [10] H. Bazaki, N. Kubota, Effect of binders on the burning rate of AP composite propellants, *Prop. Explo. Pyrotech.*, 25 (2000) 312-316.
- [11] H. Bazaki, N. Kubota, Effect of binders on the burning rate of AP composite propellants, *Prop. Explo. Pyrotech.*, 25 (2000) 312-316.
- [12] H. Shen, Y.G. Yu, Study on Measurement and Simplified Model of Burning Rate of AP/HTPB Base Bleed Propellant under High Pressure, *Advan. Mater. Resear.*, vol. 724, Trans Tech Publ, 2013, pp. 1176-1180.
- [13] P. Mehta, C.P. Shetty, R.N. Pundkar, H. Shekhar, Effect of Loading Densities in Closed Vessel Tests on the Burning Rate of a Propelling Charge, *Defen. Sci. J.*, 65 (2015) 126-130.
- [14] Defence Standard. The closed vessel ballistic assessment of gun propellants. Ministry of Defence, UK; Def-Stan 13-191/1,, (1996).
- [15] A. Mezroua, K. Khimeche, M.H. Lefebvre, M. Benziane, D. Trache, The influence of porosity of ammonium perchlorate (AP) on the thermomechanical and thermal properties of the AP/polyvinylchloride (PVC) composite propellants, *J. Therm. Anal. Calorim.*, 116 (2014) 279-286.