Comparative Closed Vessel Firing-Ballistic Parameters Evaluation for Development of Base Bleed Composite Solid Propellant

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***Abstract*—Closed vessel test (CVT) is widely used for the measurement and comparison of ballistic and energetic properties of propellants by the ignition of a specific sample mass in a closed high-pressure vessel. In our research, comparative CVTs were performed in a vessel, where an internationally accepted composite propellant base bleed grain sample served as a reference (Ref) for recording the standard values of the under investigation propellant composition, in relation to which newly developed samples were characterized. These comparative CVT experiments were performed under chamber volume of 100cm3, sample mass of 10g for all samples and identical ignition system having 1.5g igniter bag of gunpowder. We used closed vessel with working pressure limit of 5000 bars for recording the ballistic parameters of various composite solid propellant samples with reference to a standard sample. It was found that the Ref sample at 50% propellant loading recorded mean maximum pressure (Pm) of 1040 bars in complete combustion time (tPm) of 120ms and vivacity of 0.038 (1/bar·s). The measured mean Pm was taken as relative force (%) and measured mean vivacity was taken as relative vivacity (%). This data has been used to tune and study the ballistic parameters to develop Ammonium perchlorate (AP) and hydroxyl-terminated polybutadiene (HTPB) based composite solid propellant (CSP) base bleed grain for artillery projectile.**

Keywords-composite propellant; closed vessel

I. Introduction

Closed vessel test (CVT) of gun propellants is a technique employed for the experimental evaluation of propellant ballistic parameters at laboratory level [1, 2]. The equipment consists of a vessel made of very high strength steel, equipped with piezo-electrical pressure transducer, outlet valves, special ignition system fitted in breech screw and data acquisition device. Tests are mainly used to measure pressure history and pressure maximum of propellants [3]. Dynamic firing for testing any propellant is an expensive method so the closed vessel (CV) provides a very safe, quick and economical way for research and development of propellants [4-6]. It involves combustion of a known mass of propellant in a closed chamber of a specific volume. The pressure rises with time inside the vessel, it is measured, recorded and processed by the data acquisition system. Recorded P-t data are used to calculate the ballistic parameters including differential pressure (dP/dt), relative force and relative vivacity (quickness) of the propellant [7]. The ballistic assessment of the propellant charge fired in closed vessel is the mean result of its force and value of its vivacity compared to that of the reference or standard propellant. The force is the maximum pressure developed in CV chamber by firing a propellant charge and it is considered as the energy of the propellant. It also indicates that the developed composition is correct [4]. Propellant vivacity is defined as the differential pressure (dP/dt) divided by maximum pressure (P_m). Vivacity is the measure of quickness and efficiency of a propellant to produce energy while undergoing combustion. The development of new propellants with desired properties and the quality control in production of propellants at industrial scale requires the measurement of pressure, vivacity and ballistic properties [8]. Comparative tests are also performed in closed vessel where a sample is considered as reference (Ref) and the obtained data of other propellant samples are evaluated in comparison to the reference sample. Comparative tests are performed in identical conditions including closed chamber capacity, loading conditions, loading density and ignition system. A specific amount of propellant charge is loaded in the CV and fired remotely [9].

Range enhancement of artillery shells is a basic need of all modern armies. One out of the many methods to extend the range is employment of base bleed (BB) unit housing a BB composite propellant grain at the base of artillery projectile [10-12]. The projectile travels at supersonic velocity during its flight which creates an under-pressure region at its base which is about 50% of total drag acting on shell body. BB grain inside the BB-unit acts as a gas generator that fills the under pressure zone behind the shell with hot gases and increases the pressure consequently overcoming the base drag giving the projectile increased range [13, 14]. Ammonium perchlorate (AP) and hydroxyl-terminated polybutadiene (HTPB) based composite solid propellant (CSP) is employed as BB grain in the BB unit for large caliber artillery gun projectile. CSP contains AP (oxidizer) particles embedded in HTPB binder with metal powders like aluminum as fuel [15, 16]. Burning rate is the most important characteristic of the CSPs. The combustion behavior and ballistic performance of the propellants depends upon the oxidizer, burning rate modifiers and fuel type [17-19]. The combustion process is mainly the decomposition of the AP which is a self-sustained process [20]. Being at the bottom of the projectile, the BB-unit starts burning as the projectile is fired under high pressure conditions. The BB-grain travels through the bore under high pressure. CVT can be employed for the evaluation and modification of ballistic parameters of BB grain under high pressure conditions [21, 22]. We used for the first time CV firing to record the ballistic parameters of various CSP samples with reference to a standard sample for BB unit of 155mm artillery projectile. Measured data have been used to tune and study the ballistic parameters in order to develop a composition with required properties. Different compositions were developed based on AP and HTPB cured with IPDI. CVT was performed by firing three rounds of each sample and the mean values were recorded to study the tuning of ballistic parameters during the development process of BB propellant grain according to the Ref sample.

II. EXPERIMENTAL

A. Composite Propellant Preparation

CSP samples were synthesized based on HTPB binder cum fuel over a solid loading range of 68%-78% including AP as oxidizer. Aluminum powder was used to opacify the propellant and for stable combustion. HTPB binder and various additives including Dioctyl Sebacate (DOS), 1-(2-methyl) Aziridinyl Phosphine Oxide (MAPO), Trimethylol Propane (TMP) and Fe_2O_3 as plasticizer, bonding agent, crosslinker and burning rate promoter respectively were charged in a horizontal kneading machine for 30min followed by the addition of Al powder and AP which is a bimodal mixture of two different partial sizes. After kneading the complete mixture for 40min IPDI was added and mixed for 30min under vacuum. Finally, the completely homogenized mixture was poured in Teflon coated stainless steel molds and cured in oven at 60°C for 170 hours. Propellant compositions developed for this study are given in Table I. SEM images show that the solid AP particles have been homogeneously mixed and embedded in polyurethane matrix in Figure 1.

TABLE I. COMPOSITE PROPELLANT FORMULATION

Sample	Propellant Composition wt. %				
	HTPB + Additives	Al (22μm)	AP (256μm)	AP (130μm)	Fe_2O_3
A	28.00	02.00	35.00	35.00	-
B	26.00	02.00	36.00	36.00	-
C	24.00	02.00	37.00	37.00	-
D	22.00	02.00	38.00	38.00	-
E	20.00	02.00	39.00	39.00	-
F-1	26.00	02.00	35.80	36.00	0.20
F-2	26.00	02.00	35.60	36.00	0.40
F-3	26.00	02.00	35.40	36.00	0.60
F-4	26.00	02.00	35.20	36.00	0.80

B. High Pressure Clossed Vessel

CV is a widely used technique for ballistic evaluation of propellants at laboratory level. It is a safer, quicker and less expensive process instead of field firing with gun when dealing with the research and development of propellants and high energy pyrotechnic materials [4]. A sample of known loading density is test fired in CV of known volume for valuable ballistic performance evaluation [7]. The CV used in this research work is a conventional CV which has chamber volume of $100cm^3$ with pressure recording range up to 5000bars. The vessel has a loading density of Δ=$0.20g/cm^3$ which is the ratio of propellant mass being tested to the volume of CV chamber. The CV is equipped with a pressure block and a firing block. The firing block has a nickel chrome wire soldered to firing terminals and passing through an ignition bag containing 1.50g of gun powder. Gun powder bag is initiated by the firing control unit to provide ignition to the propellant sample. Pressure block is equipped with high pressure quartz direct measuring pressure transducer type 6203 with maximum recording range of 5000bar. The CV is connected to a Kistler charge amplifier type 5018 and an electronic signal acquisition module for P-t data recording. A schematic diagram of CV is shown in Figure 2.

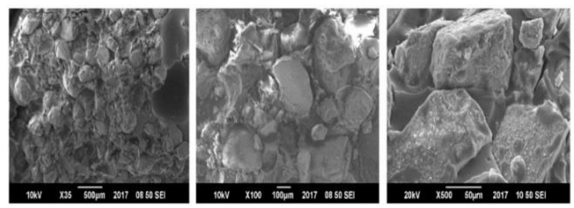

Fig. 1. SEM images of prepared CSP.

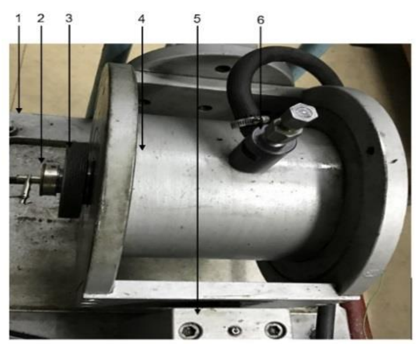

Fig. 2. High strength steel CV: 1) Exhaust valve, 2) Vessel station, 3) Firing block, 4) Ignition electrode, 5) Vessel body, 6)Vessel rotating arm.

C. Clossed Vessel Sample Firing Method

According to the theory of constant volume, loading density is used to calculate the total sample loading mass and required quantity of ignition powder for CVT. The vessel has a loading density of Δ=0.20g/cm3 with full loading charge capacity of 20.00g. Propellant samples were fired in CV at half loading charge mass of 10g with 1.5g of gun powder ignition bag for all tests. CSP is different from gun propellants in geometry and composition, therefore identical square shaped samples were cut and loaded in CV (Figure 3). Firing control unit gives an electrical pulse to the ignition head which generates thermal impulse to ignite the gun powder bag for initiating the propellant samples in CV chamber. The pressure produced is measured by the piezoelectric pressure transducer which is further amplified and recorded by the data acquisition system.

Figure 4 shows a schematic diagram of loaded CV ready for test firing.

Fig. 3. Prepared samples for CVT.

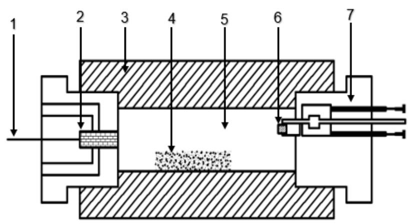

Fig. 4. Schematic diagram of CV: 1) Coaxial data cable, 2) Pressure transducer, 3) Vessel wall, 4) Igniter sample, 5) Vessel chamber, 6) Ignition bag, 7) Firing block.

III. RESULTS AND DISCUSSION

CVT experiments were performed with 10g of sample mass for each sample which is the 50% of loading charge capacity of the vessel. Five rounds were test fired for reference and each batch of AP/HTPB CSP under identical conditions and results were averaged. Single fire data are as represented in Figure 5.

CV Detail Results

p / pm	Time ms	Pressure bar	dp / dt bar / ms	dz / dt 1 / s	Vivacity 1 / bar * s
0.05	21.50	50.45	7.97	3.949	0.0391
0.10	28.00	100.90	7.42	3.678	0.0182
0.15	35.00	151.35	6.68	3.310	0.0109
0.20	42.00	201.80	7.85	3.891	0.0096
0.25	48.00	252.25	9.38	4.646	0.0092
0.30	53.00	302.70	9.69	4.801	0.0079
0.35	58.00	353.16	10.78	5.343	0.0076
0.40	62.50	403.61	11.84	5.865	0.0073
0.45	66.50	454.06	12.70	6.291	0.0069
0.50	70.50	504.51	13.36	6.620	0.0066
0.55	74.00	554.96	14.02	6.949	0.0063
0.60	78.00	605.41	14.77	7.317	0.0060
0.65	81.00	655.86	15.47	7.666	0.0058
0.70	84.50	706.31	15.27	7.569	0.0054
0.75	87.50	756.76	15.43	7.646	0.0051
0.80	91.00	807.21	14.06	6.969	0.0043
0.85	95.00	857.66	12.77	6.330	0.0037
0.90	99.00	908.11	10.63	5.265	0.0029
0.95	105.00	958.56	7.46	3.697	0.0019
1.00	120.00	1,009.00	0.23	0.116	0.0001
Max		1,009.00	15.47	7.666	0.0391

Rise Time

p / pm	ms
10 % - 30 %	41.20
10 % - 60 %	55.95
10 % - 80 %	63.97

Vivacity @ 450.0 bar: 0.0070
Vivacity @ 551.6 bar: 0.0063

Mean Vivacity

p / pm	1 / bar *s
30 % - 30 %	0.0073
30 % - 70 %	0.0066
30 % - 80 %	0.0063

Fig. 5. Recorded data for single CVT.

A. *Effect of Oxidiser/Fuel Ratio*

Most valuable output from the CVT data acquisition system comes in the form of P-t profile. In the initial phase, five propellant compositions with different bimodal AP content (wt. %) were fired comparatively with the standard Ref sample. Recorded and calculated values including P_m, maximum differential pressure $(dP/dt)_m$, t_{Pm} relative force (F) and relative vivacity (V) from first phase of CV firing are reported in Table II. Recorded P-t profiles from firings at identical conditions and loading density are plotted in Figure 6. Recorded data were divided in three parts, the first part is taken from 10%-30% of the maximum recorded pressure and second part at 10%-60% and the final at 10%-80%. The phase of propellant charge combustion used to determine the ballistic parameters of solid composite propellant is taken from 10%-80% and finally the maximum obtained pressure at the end of the complete combustion. The vivacity is taken at 30%-80% of the recorded curve. P-t data are used to calculate dP/dt which is plotted against pressure in Figure 7. Mean values of vivacity obtained from CV firing data is plotted against P/P_m in Figure 8.

TABLE II. CVT RESULTS PHASE-I

Sample	P_m (bar)	t_{Pm} (ms)	$(dP/dt)_m$ (bar/ms)	F %	V %
Ref	1040.80	120.00	15.74	100.00	100.00
A	980.09	281.00	14.69	93.40	44.44
B	1043.10	220.00	16.00	100.48	56.13
C	1098.20	178.00	22.58	105.75	70.32
D	1160.60	158.00	26.64	112.25	72.90
E	1218.20	114.00	35.20	114.00	89.68

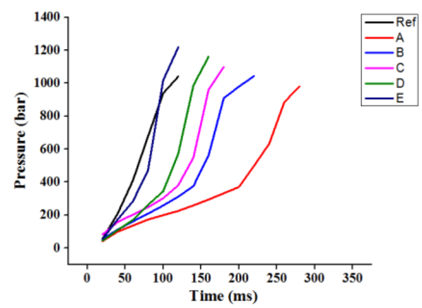

Fig. 6. Comparative P-t profile.

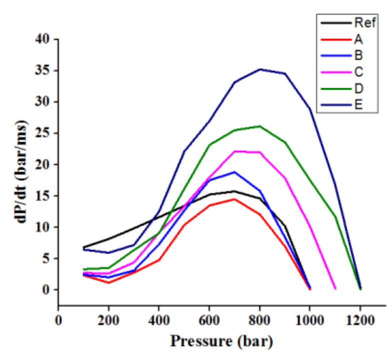

Fig. 7. Rate of change of pressure against pressure.

In compositions A to E, the composite propellant solid loading of bimodular oxidizer content has been gradually increased and no burning rate modifier has been employed. From Figure 6, it is observed that recorded maximum pressure rises along with oxidizer content. This is an obvious result as higher solid loading in composite propellant resulted in the release of higher amount of energy. As the percentage of oxidizer mass increases, the pressure generated increases and the complete combustion time reduces. The quickness or higher rate of regression of burning surface is caused by gradual positive change in oxidizer to fuel (O/F) ratio. 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.

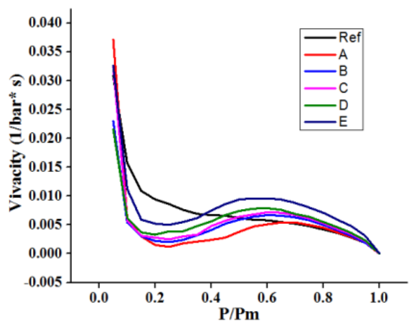

Fig. 8. Vivacity against P/P_m.

From Figure 7 can be observed that the rate of increase of pressure rises with the rise in O/F ratio. It is seen that the slope of dP/dt against P rises with the rise in wt. % of oxidizer content which indicates that the higher the solid loading, the higher is the energy produced. Vivacity is the quickness and rate at which energy is released. The higher the vivacity the higher is the rate of regression of burning propellant surface in the high pressure chamber. Figure 8 shows the vivacity against P/P_m. Figures 6-8 show that the change in oxidizer resulted in rise in pressure and quickness. At a fixed O/F ratio we achieved the requisite pressure at slower rate of propellant combustion taking longer time than the Ref. With gradual increasing of the O/F ratio the rate of pressure rise increased, taking shorter time for the whole propellant mass to burn (closer to Ref). The combustion process grew faster getting closer to Ref but pressure rose beyond the standard limit. Therefore, the O/F ratio was fixed based on the P-t profile and composition B with requisite force was selected for further development.

B. Addition of Burning Rate Promoter

In the second phase we added the burning rate modifier Fe_2O_3 in chosen composition B for studying the effect on P-t profile, with fixed O/F ratio. Fe_2O_3 was employed to increase the quickness or combustion to shorten the burning time to match that of the Ref. The measured values of the experimental results are given in Table III. The recorded and calculated data for P-t, dP/dt against P_m and vivacity against P/P_m respectively are plotted in Figures 9-11.

TABLE III. CVT RESULTS PHASE-II

Sample	P_m (bar)	t_{Pm} (ms)	$(dP/dt)_m$ (bar/ms)	F%	V%
Ref	1040.80	120.00	15.74	100.00	100.00
F-1	1030.50	131.50	24.69	99.01	91.25
F-2	965.34	126.00	22.77	92.74	95.24
F-3	1011.80	120.50	17.23	97.21	99.58
F-4	1039.70	119.00	16.00	99.89	01.00

P-t profile in Figure 9 clearly shows that at a fixed solid loading of AP with bimodular particle distribution, the pressure generated remained within given limits whereas the rate of regression of burning surface changed. The burning rate modifier acts as a burning rate promoter by reducing the temp at which ammonium perchlorate decomposes, consequently the propellant mass combustion grew faster. Gradual increasing percentage of Fe_2O_3 at a fixed O/F ratio affected the relative vivacity. The higher rate of regression of burning surface is indirectly observed in Figure 10. The rate of change of pressure is higher when compared to the composition without burning rate promoter at the same O/F ratio. This means that the same mass of propellant charge is undergoing combustion process at a faster pace.

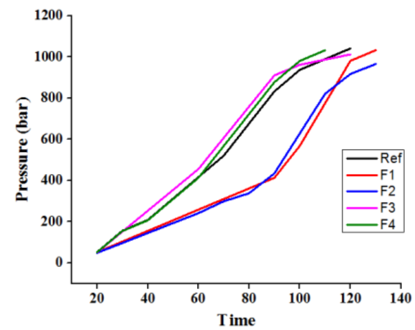

Fig. 9. Comparative P-t pofile.

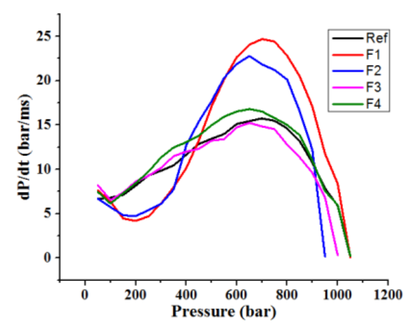

Fig. 10. Rate of change of pressure against pressure.

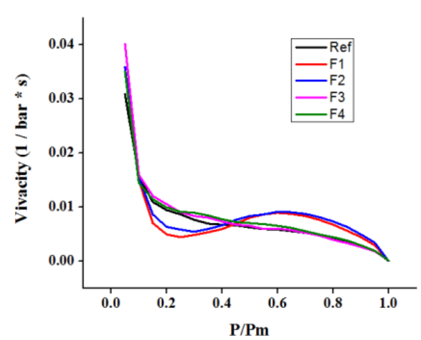

Fig. 11. Vivacity against P/P_m

Figure 11 represents the comparative vivacity and the relative vivacity increased with increase in the catalyst wt. %.

By observing the change in relative vivacity, the final composition F-4 gave acceptable relative force (%) and relative vivacity (%). By comparative firing of newly developed compositions and evaluating the recorded P-t profiles we were able to develop a composition for base bleed composite propellant. The technique is applied to gun propellants and it is very effective in quality control and production of newly developed gun propellants. Our research work proves that this technique is very useful for the development and study of composite propellants.

IV. CONCLUSION

This research work focused on employing CVT technique for the study and development of AP/HTPB based slow burning rate composite solid propellant compositions. Comparative testing of samples with a standard reference sample has been carried out. Based on the experiments it was observed that higher oxidizer content resulted in higher pressures at higher rates of rise of pressure. Higher oxidizer meant higher O/F ratio resulting in more efficient combustion process. By observing the trend in P-t profiles and data calculated the burning rate of propellant sample was enhanced at a fixed O/F ratio by employing Fe_2O_3. This resulted in achieving the desired results and development of base bleed grain was accomplished. Comparative analysis proved to be a very successful method of studying and fine tuning of the ballistic parameters of newly developed propellant composition.

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