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**Measurements of Pressure
for the $\text{TiH}_x/\text{KClO}_4$ System**

Clyde H. H. Chong and Jerome E. Glaub

April 10, 1978

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Summary

An investigation was conducted to measure the pressure obtained when the pyrotechnic mixture $\text{TiH}_x/\text{KClO}_4$ was ignited in a confined variable-volume system. It was possible to derive an expression of the form $PV^\gamma = k$ for the pressure-volume relationship obtained. This expression is a polytropic expansion of the ideal gas equation that best fits the data generated. For this particular work, values of $\gamma = 0.53$ and $k = 69.3$ were obtained where P is in megapascals and V is in cubic centimeters. In addition, estimates of the reaction rates were calculated based on the times to achieve maximum pressure for a given volume system. An explicit expression relating rate to pressure was derived by a simplified least-squares fit of the data obtained.

Introduction

Generally, investigations of pyrotechnic materials are concerned with such critical attributes as determining the ignitability of various compositions from bridgewire systems utilizing different ignition modes, or measuring the degree of safety in terms of their sensitivity to such standard tests as impact, friction, or electrostatic spark conditions. Other pertinent data relative to any particular pyrotechnic system under consideration would naturally include its thermal stability, fuel-to-oxidant ratios versus calorific output, and other related pieces of information.

Recently, investigations have been performed in the pyrotechnic area regarding the characteristics and analysis of pyrotechnic reaction products, and their possible utilization and/or applicability in selected ordnance devices [1]. This report is intended to supplement previous experimental work. Attention and efforts are directed primarily to the pressure effects obtained in confined volume systems. The ability to measure the time interval from ignition to attainment of maximum pressure permits calculation of a reaction rate for the pyrotechnic. Accordingly, this work focuses on two major activities: one dealing with pressure aspects, and the other related to rate of reaction as it pertains to the given system.

The experimental work involved taking a given quantity of the pyrotechnic charge and initiating it directly with a hot bridgewire. The pyrotechnic was confined in a closed system to which was affixed a pressure sensing device. Pressure was

measured as a function of time in these experiments.

Again, to emphasize, it is not the purpose of this report to present data related to the physical characteristics of the pyrotechnic itself, since this has been done by others, but rather to discuss the findings encountered when the pyrotechnic was subjected to a set of conditions in a limited (confined) geometry. This investigation deals primarily with a possible application of the pyrotechnic and not with intrinsic characteristics of the material.

Experimental procedure

Equipment

Several stainless steel reactor-receiver systems of varying volumes were employed in the pyrotechnic firing studies (Figure 1). The reactor portion of the system consisted of a threaded hole for inserting a loaded bridgewire unit 0.62 in. (1.57 cm) in length with 9/16-18 threads. The pressure measurements and propagation rate studies were performed on cylindrical compacts 0.15 in. (0.381 cm) in diameter by 0.145 in. (0.368 cm) in length. Ignition was achieved by electrically heating the bridgewire. All reaction mixtures could be ignited without difficulty. The typical loaded bridgewire header assembly is shown in Figure 2.

The bridgewire header assembly consisted basically of a cavity 0.236 in. (0.600 cm) in diameter and 0.420 in. (1.067 cm) deep. A ceramic (Al_2O_3) bridgewire unit and charge holder sleeve were inserted into this cavity and were glued together with epoxy cement. The pyrotechnic charge was pressed into the sleeve of 0.042 cm^3 volume [0.150 in. (0.381 cm) i.d. x 0.145 in.

FIGURE 1 - Reactor-receiver system used in pyrotechnic firing experiments.

FIGURE 2 - Sketch of bridgewire (loaded) header assembly (not to scale).

(0.368 cm) long] using approximately 10 kpsi (69 MPa) pressure. The bridgewire was 1.7-mil (0.0043-cm) nickel-chromel (Tophet-C) wire, the resistance of which varied from 0.952 to 1.094 ohms.

The reactor-receiver system was sealed together with a Neoprene O-ring using a 2-in. (5.1-cm)-diameter flange to ensure positive seating of the gasket and flange and was compressed by tightening six 1/4-20 screws. Attached to this flange was a Sundstrand Data Control Inc. quartz piezoelectric pressure transducer, Model 207C2. The maximum pressure rating for this type of transducer is 70 kpsi (480 MPa). An ablative coating [about 0.10 in. (0.25 cm) thick] on the diaphragm of the transducer was used to protect the diaphragm from melting or permanent deformation at flash temperatures up to 5000°C. The ablative coating consisted of GE SS-4004 primer and GE RTV-88 silicone rubber.

Volume variations ranging from 0.25 to 5.0 cm³ were provided by varying the distances between the assembly header and the transducer while maintaining a constant diameter of 0.626 in. (1.59 cm). Distances of 0.05 to 1.0 in. (0.13 to 2.54 cm) resulted in volumes of 0.25 to 5.0 cm³ respectively.

The pressure generated in the closed system and the time from ignition to maximum pressure for the pyrotechnic were measured by an elaborate array of electronic and photographic components. A schematic of the firing and measuring systems used in these studies is shown in Figure 3. The function and purpose of each major component in the sketch is given in outline form below to provide a better understanding of the need for incorporation of that particular item in the overall system.

FIGURE 3 - Schematic of firing and measuring system for pyrotechnic studies.

Systron Donner Pulse Generator - Supplies the necessary initiating pulse for the firing system.

Hewlett-Packard Pulse Generator - produces gate pulse for the emitter follower. The pulse width determines the firing pulse width.

Emitter Follower Fire Set - supplies power to the bridgewire header assembly under test. The current output into a 1-ohm load can be adjusted between 1 and 20 A. Throughout this work a constant-current pulse of 3.5 A was utilized to initiate the bridgewire.

Time Interval Counter - displays time for the device bridgewire to burnout. The usual time interval is in the 0 to 50-msec range.

TEK 556 Oscilloscope - a dual-beam unit which monitors the bridgewire voltage and current pulses as a function of time in milliseconds. An attached camera records those traces for subsequent data analysis.

Charge Amplifier - converts pressure transducer output readings into a calibrated pulse which can be monitored by an oscilloscope.

TEK 544M Oscilloscope - a single-beam unit that monitors the pressure pulse as a function of time in microseconds. An attached camera records this pulse for subsequent data analysis.

Materials

The pyrotechnic material used throughout this study was $\text{TiH}_{0.68}/\text{KClO}_4$. The procurement, synthesis techniques, fabrication,

analysis, etc. have been developed [2]. The fuel ($\text{TiH}_{0.68}$) and oxidizer (KClO_4) were combined in the weight percent ratio of 33 to 67, respectively. The fuel and oxidizer were blended and pressed. The pressed compacts appeared to be uniform and without visible voids. Reaction mixtures will react readily without compression but compressed shapes were used in this study in order to provide compacts of well-defined geometry and density. The densities of the charge compacts were calculated to be approximately 2.6 g/cm^3 . This value was obtained from the weight of charge mix used and from the volume calculations (i.e., approximately 110 mg of pyrotechnic charge and a calculated volume of 0.042 cm^3).

Results and discussion

The ability to fire the pyrotechnic system $\text{TiH}_{0.68}/\text{KClO}_4$ was demonstrated in the unique reactor-receiver chamber. All of the experiments proceeded satisfactorily. A typical example of the kind of data trace generated from the oscilloscope (camera) is shown in Figure 4. A summary of the data obtained in these experiments is shown in Tables 1-3. Essentially, three independent sets of data were generated from the studies performed and have been labeled accordingly. The firings were conducted in a reproducible manner using three different batches of pyrotechnic material. The three batches were of the same composition and were prepared identically in terms of material preparation, bridgewire header assembly fabrication, and loading and compacting of the pyrotechnic. The firings for each particular set were performed randomly using various volume sizes.

Pyrotechnic firings were conducted in volumes of 0.25, 0.50, 1.25, 2.50, and 5.00 cm^3 . Within each set, two or more firings

FIGURE 4 - Oscilloscope trace of typical pressure/time curve.

were conducted for each volume size and the average pressure values were determined.

Varying the volume size of the reactor-receiver system while employing a nearly constant quantity of the pyrotechnic charge resulted in pressure values that increased with decreasing volume. For a given set of pyrotechnic charges a plot of the average pressure observed as a function of the volume was made. Pressure increased asymptotically as volume size decreased (Figure 5). There appear to be no significant differences in the shapes of the three curves generated despite the fact that these sets of experiments were performed at different time periods. Average

pressures ranged from a low of 30 MPa for the 5.00-cm³ system to nearly 160 MPa in the 0.25-cm³ system.

It follows, upon close examination, that the family of curves generated in Figure 5 can be expressed in some form of the ideal gas law equations. As a first approximation it would appear that a reversible, isentropic (constant-entropy) expansion of the thermodynamic expression for an ideal gas can be assumed. However, since the expansion is not ideal (the gas is expanded through a nozzle), the process is not isentropic, and a polytropic expansion of the gas law equations appears to be more likely to best fit the given data.

Table 1 - DATA SUMMARY OF PYROTECHNIC FIRINGS IN A CLOSED SYSTEM (SET I)

Experiment No.	System Volume (cm ³)	Calculated Path Length (cm)	Bridgewire Burnout Time ^a (msec)	Reaction Time ^b (μsec)	Peak Pressure Observed (MPa)
1	1.25	0.96	1.855	--	54.0
2	1.25	0.96	2.428	19.84	60.6
3	1.25	0.96	2.166	17.96	57.8
4	2.50	1.64	2.001	31.51	35.8
5	2.50	1.64	3.595	28.58	39.8
6	0.25	0.49	2.208	21.49	146.3
7	0.25	0.49	2.522	25.30	157.0

^aTime from ignition to "bridgewire burnout".

^bTime from ignition of charge to complete reaction.

Table 2 - DATA SUMMARY OF PYROTECHNIC FIRINGS IN A CLOSED SYSTEM (SET II)

Experiment No.	System Volume (cm ³)	Calculated Path Length (cm)	Bridgewire Burnout Time ^a (msec)	Reaction Time ^b (μsec)	Peak Pressure Observed (MPa)
7 ^c	0.25	0.49	3.411	21.40	93.4
8	0.25	0.49	3.040	21.23	129.6
9	0.25	0.49	2.978	21.51	137.7
1	0.50	0.62	--	20.38	91.8
2	0.50	0.62	--	18.56	94.5
10 ^c	0.50	0.62	1.798	20.40	122.3
3	1.25	0.96	2.768	18.76	63.8
4	1.25	0.96	3.089	19.14	71.9
14	1.25	0.96	3.301	19.98	70.9
11	2.50	1.64	3.166	24.33	44.9
12	2.50	1.64	3.076	25.91	39.1
13	2.50	1.64	4.204	28.58	39.4
5	5.00	2.91	2.655	25.46	32.3
6	5.00	2.91	2.824	31.49	28.4
15	5.00	2.91	3.335	31.98	23.8

^aTime from ignition to "bridgewire burnout".

^bTime from ignition of charge to complete reaction.

^cExperiments No. 7 and 10 reflect the effect when less than or greater than the normal quantity of pyrotechnic charge mix weight is used.

Table 3 - DATA SUMMARY OF PYROTECHNIC FIRINGS IN A CLOSED SYSTEM (SET III)

Experiment No.	System Volume (cm ³)	Calculated Path Length (cm)	Bridgewire Burnout Time ^a (msec)	Reaction Time ^b (μsec)	Peak Pressure Observed (MPa)
4	0.25	0.49	1.688	18.67	146.2
5	0.25	0.49	1.964	20.09	174.0
6	0.50	0.62	2.420	19.04	119.1
7	0.50	0.62	2.012	19.36	107.9
12	1.25	0.96	3.061	21.61	56.8
13	1.25	0.96	2.690	21.16	62.5
8	2.50	1.64	1.820	16.26	42.2
10	2.50	1.64	1.889	29.12	46.4
9	5.00	2.91	3.277	31.06	35.1
11	5.00	2.91	2.964	27.64	28.9

^aTime from ignition to "bridgewire burnout".

^bTime from ignition of charge to complete reaction.

In order to derive a relation between P and V , one can utilize the equation [3]:

$$dQ = C_V dT + PdV \quad (\text{ideal gas}) \quad (1)$$

and

$$dQ = C_P dT - VdP \quad (\text{ideal gas}) \quad (2)$$

Since, in an adiabatic process, $dQ = 0$,

$$PdV = -C_V dT$$

and

$$VdP = C_P dT$$

Dividing the second equation by the first and rearranging results in:

$$\frac{dP}{P} = -\frac{C_P dV}{C_V V} = -\gamma \frac{dV}{V} \quad (3)$$

This equation cannot be integrated until something is known about the behavior of γ . For monatomic gases γ is constant, whereas for diatomic and polyatomic gases it may vary with temperature. It requires, however, a large change in temperature to produce an appreciable change in γ . Most

adiabatic processes normally do not involve large temperature changes. Accordingly, one is entitled, in an adiabatic process that involves only moderate temperature change, to neglect the small accompanying change in γ . Regarding γ , therefore, as a constant and integrating yields:

$$\ln P = -\gamma \ln V + \ln \text{constant}$$

or

$$PV^\gamma = k \quad (4)$$

where k is a constant, different in general for each particular process. It must be emphasized that a free expansion is an adiabatic process and is not quasi-static. It is therefore incorrect to apply Equation 4 to the states traversed by an ideal gas during free expansion.

A family of curves representing quasi-state adiabatic processes may be plotted on a PV diagram, each curve corresponding to a different value of the constant k . The slope of such a curve is given by

FIGURE 5 - Pyrotechnic firing experiments - pressure generated as a function of volume size.

$$\left(\frac{\partial P}{\partial V}\right)_S = -\gamma k V^{-\gamma-1} = -\gamma \frac{P}{V}$$

where the subscript S denotes a quasi-static adiabatic process. Since

$$\left(\frac{\partial P}{\partial V}\right)_T = -\frac{P}{V}$$

it follows that an adiabatic curve has a steeper negative slope than does an isothermal curve at the same point.

An additional constraint was imposed in Equation 4. The compressibility factor z should be included in the equation. Thus,

$$(z)PV^\gamma = k \quad (4a)$$

However, over the investigative range of interest, z is taken to be approximately 1 ($\pm 10\%$) because it is assumed that the principal contributing gaseous species in the system during the reaction is H_2O . If such is the case, then from tables [4], the reduced temperature T_r takes on values of $3 < T_r < 15$ and the reduced pressure P_r takes on values of $3 < P_r < 10$ since the critical temperature and pressure (T_c and P_c) of water are $374^\circ C$ and 218 atm (22 MPa), respectively.

From the PV data generated in the experiments performed, values of γ and k can be determined. The values for γ and k were 0.53 and 69.3, respectively. The former value can be compared with the value obtained by Reed et al. [5] of approximately 0.9 from equilibrium thermodynamic calculations for the heat capacity ratio C_p/C_v . Reed's theoretical calculation assumes an isentropic process whereas, as stated earlier, the process is not really isentropic.

A plot of Equation 4 using the optimum values for γ and k is shown as a dot-dash line in Figure 5.

The effect of varying the quantity of the pyrotechnic mix used is apparent. An increase or decrease in the amount of mix will cause an appreciable change in the observed pressure. Such were the cases for experiments 7 and 10 in Set II. The pressure variations in these particular firings are obvious. The time for complete reaction to occur, however, does not appear to be affected by changes in pyrotechnic mix weights.

To more completely characterize the system, a reaction rate was calculated for the pyrotechnic in the geometry utilized. Provisions were made to measure the time interval from: (1) bridgewire firing pulse to bridgewire burnout, and (2) ignition of the pyrotechnic charge to time of maximum pressure. The former event occurred in milliseconds; the latter, in microseconds. Consequently, by synchronizing the cameras on the oscilloscopes to record the two events (1) input signal to the bridgewire to its burnout time followed by (2) triggering event of the bridgewire burnout to maximum (peak) pressure observed, it was possible to obtain reasonably accurate values for the latter event. These values enabled computation of the reaction rate in centimeters per second. The length, which included the charge depth, varied for each particular system, ranging from 0.195 to 1.145 in. (0.495 to 2.908 cm) overall. These lengths were determined from machine fabrication of the system components used.

Compilations of the reaction rate as a function of volume size are shown in Table 4. The values represent the average

Table 4 - CALCULATED REACTION RATES FOR PYROTECHNIC FIRINGS IN VARIOUS VOLUME SYSTEMS

System Volume (cm ³)	Reaction Rate (cm/sec)		
	Set I	Set II	Set III
0.25	21,100	23,100	25,300
0.50	--	31,500	43,300
1.25	51,000	50,000	45,000
2.50	54,600	62,500	76,700
5.0	--	94,700	99,100

of two or more experimental firings. The marked differences in results upon going from the 0.25-cm³ to the 5.0-cm³ system in a given set are obvious. These changes appear to increase as the volume is increased. For a given volume, however, there is no major difference in the rates observed for the sets. The drastic changes from the 0.25-cm³ to the 5.0-cm³ system can be ascribed to the higher pressure built up in the smaller system as compared to the larger volume systems. It must be emphasized that the rates obtained are for a particular type of system and are not true "burn rates" in the strict sense.

The possibility of determining a linear burning rate was not overlooked in these studies. Burning rate is defined as the velocity at which the burning surface recedes in a direction normal to the surface as the solid is transformed to gaseous products. This linear burning rate (r) has been found, empirically, to increase with increasing gas pressure (P) on the pyrotechnic, according to the equation [6]:

$$r = aP^b \quad (5)$$

where a and b are constants.

However, the time to burnout for a constant charge length is essentially unaffected by

pressure in the high-pressure region (30-160 MPa) for the experiments performed in this study. A plot of the burning rate relationship incurred for this particular system as a function of pressure is shown in Figure 6 where a least-squares straight-line fit has been drawn. The burning rate in this figure is expressed for the constant charge length (0.368 cm) divided by the actual time incurred from initial pressure inflection to maximum inflection as determined from photographic data analysis (see also Figure 4 for a typical pressure/time trace). Essentially a straight line is depicted, indicating the value of b must be very nearly zero. Therefore, the value of b in the expression $r = aP^b$ cannot be determined at these high pressures. The dashed line in Figure 6 shows the expected behavior at lower pressures.

Conclusions

Ignition of the pyrotechnic $TiH_x/KClO_4$ can be achieved with the system hardware and techniques employed. In the series of experiments performed, pressure as a function of volume size was measured. An expression of the form $PV^\gamma = k$ was derived for the P-V relationship observed with this kind of a system. The value for γ , 0.53, compared reasonably well with a value reported by Reed [5].

FIGURE 6 - Linear burning rate as a function of pressure for the system $\text{TiH}_{0.68}/\text{KClO}_4$ (average times and pressures used for each volume system).

The pressure/time data from these experiments appear to be in agreement with the work done by Ng [7,8] employing different measuring techniques for pressure output generated. Although different kinds of information were obtained by the methods used, they both describe behavior of the pyrotechnic systems in a given configuration.

It was also possible to determine a reaction rate from the studies conducted. This piece of information was readily attained by measuring the time required to ignite and react the pyrotechnic charge completely. From the length of the reaction system and the time required to achieve maximum pressure, the reaction rate can be calculated. It was not possible to determine a true linear burn rate for the system. Since these experiments were done at high pressures, the typical

direct relationship between linear burn rate and pressure was not observed; instead, burn rate was found to be essentially unaffected by pressure.

Of even greater concern is the need to measure the actual temperature obtained in these systems. Work on this parameter would be of major benefit in understanding more fully the pressure data obtained and would provide a more complete and unified explanation of the reaction that is occurring in the system.

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