COMBUSTION OF TETRYL

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In this research we selected for study a typical representative of the secondary explosives-tetryl. Many of its thermophysical characteristics $[1-3]$, its kinetics of thermal decomposition in the liquid state at quite high temperatures [4,5], and the composition of its combustion products [6] are known.

Fig. 1. Oseillograms of temperature profiles obtained for tetryl burning at atmospheric pressure (a), at $p =$ $= 8 \text{ kg/cm}^2$ (b), and at p = 45 kg/cm² (c).

METHOD

The temperature profile was measured with U-shaped ribbon microthermocouples (tungsten + 5% rheniumtungsten + 20% rhenium) 5-7 microns thick by the method described by A. A. Zenin [7]. The thermocouples were welded by discharging a high-capacity electrolytic capacitor (800 μ F). The charges were pressed into plexiglass tubes 40 mm high with an internal diameter of 8 mm and a wall thickness of 2 mm. The thermocouples were inserted in the charge as follows: first, half the weighed portion of explosive was poured into the tube and a trapezoidal impression was formed in it with a shaped punch; the thermocoupIe was placed in this impression, the rest of the explosive was poured in, and finally the charge was compressed in a hydraulic press to a density (1.68-1.70 g/cm³) close to the maximum (1.73 g/cm³). The charges were ignited in a constant-pressure bomb with windows through which it was possible to establish the type of combustion and the burning rate at the same time as the temperature profile was being recorded.

RESULTS OF EXPERIMENTS

Figure 1 presents typical oscillograms of the distribution of temperature in the combustion zones of tetryl of different pressures. In all the oscillograms it is possible to distinguish the following characteristic zones. Zone I from the initial temperature to thebreak corresponding to the melting point, i. e., the solid zone at a temperature less than 130° C; zone II from the melting point to the characteristic point of inflection corresponding to the temperature at the surface of the condensed phase $T_{\rm g}$, i.e., the molten zone and reaction layer; the primary flame zone Ill directly adjacent to the reaction layer of the condensed phase, and, finally, the secondary flame zone IV, which only appears at pressures above 7 kg/cm^2 .

At first, the maximum combustion temperature (Fig. 2) varies very weakly from 900 ~ C at atmospheric pressure to 1000° C at $p = 7$ kg/cm², then it increases sharply to 2100° C (a secondary flame zone appears at 8 kg/cm²), a further increase in pressure to 45 kg/cm^2 leads only to an inconsiderable increase in maximum temperature. Similar values of the maximum combustion temperature for tetryl were obtained in [8] by Polkhil and Mal'tsev. At the point at which the secondary flame appears (8 kg/cm^2) the maximum temperature in it is reached at a distance of 4.5 mm from the surface of the condensed phase, but with increase in pressure this distance quickly diminishes (Fig. 3) and at $p = 45 \text{ kg/cm}^2$ it amounts to approximately 0.25 mm.

The maximum temperature of the primary flame increases somewhat with increase in pressure (see Fig. 2}.

We note that at pressures of $1-20 \text{ kg/cm}^2$ this zone includes a region of temperature oscillation but borders directly on the condensed phase. This region is clearly visible in Fig. 1a, b. At 45 kg/cm² these temperature oscillations are not observed (see Fig. le). As the pressure increases, the amplitude of the temperature oscillations decreases, while their frequency grows, At 1 atm temperature oscillations were also noted in the condensed phase (see Fig. la).

At atmospheric pressure the surface temperature of burning tetryl T_S (Table 1, Fig. 2) coincides with the boiling point of tetryl. An increase in pressure leads to a smooth increase in T_S (see Fig. 2) along a curve similar to the calculated pressure dependence of the boiling point of tetryl. The boiling point (see Fig, 2) was calculated from the Clausius-Clapeyron formula, starting from the fact that, according to Belyaev's data [1], the boiling point T_b of tetryl at

atmospheric pressure is equal to 310" C, while its heat of vaporization is 26 kcal/mole.

The temperature profiles enabled us to determine the temperature gradient close to the surface of the condensed phase $(dT/dx)_{s}$ (this quantity and the surface temperature are determined much less reliably than the combustion temperature and the quantity of heat transferred from the gas phase to the condensed phase by conduction (Table 1).

The temperature distribution in zone $I(T < T_i)$ of the osefllograms deserves attention. It is characteristic of this zone that the temperature distribution in it should apparently strictly obey the Miehelson law, since at such low temperatures (T < 130° C) there should be no appreciabledecomposition oftetryl inthe combustion wave during heating (half-life at 120° C, 8700 hours [4]); therefore, with increase in the burning rate u the width of the heated layer l should decrease so that the product ul remains constant, but an increase in pressure from 6 to 45 kg/cm² unexpectedly leads to a sharp increase in the width of the heated layer from 0.05 to 0.5 mm, which, in its turn, leads to an increase in the product ul by a factor of 20 (Fig. 4).

Fig. 2. Surface temperature {1), temperature of smoke-gas zone (2) maximum temperature (3), and boiling point T_b as functions of pressure.

HEATED LAYER OF SOLID PHASE

It may be assumed that the increase in the product u! is associated with radiative heat transfer from the secondary-flame zone. In fact, the heated layer begins to expand upon the appearance of the secondary flame, which *must* possess considerable radiating power [8], since when tetryI burns, a large amount of carbon is formed [6]. Moreover, tetryl is quite transparent; therefore the intense radiation of the secondary flame, at a temperature of $2000^{\circ}-2300^{\circ}$ C, can heat the inner layers of material and produce an expansion of the heated layer and a substantial deviation of the temperature distribution from the Michelson law. In order to check this assumption concerning the

role of radiation, we conducted experiments with tetryl mixed with 2 and 5% of gas black, which converted it into an almost opaque material, so that the entire radiation flux should be retained in the upper layers of the melt without penetrating to the zone with $T < T_i$.

Fig. 3. Distance from surface of condensed phase to maximum temperature of secondary flame h.

Adding gas black to the tetryl had almost no effect on the burning rate (Table 1), but led to a sharp contraction of the heated layer at $10-45 \text{ kg/cm}^2$, whereas at lower pressures (7 kg/cm²) the width of the heated layer did not change. The product ul for tetryl with gas black does not change with increase in pressure (see Fig. 4), i.e., as was to be expected, heat transfer in the opaque material is realized only by conduction. Thus, it is possible to state with confidence that the expansion of the zone $T < T_i$ is associated with radiation.

To determine the characteristics of the heat flux and the law of temperature distribution in zone I, we shall write the heat balance equation for stationary propagation, assuming that there is no chemical reaction and the radiation flux

$$
\lambda \frac{d^2 T}{dx^2} - u \rho c \frac{dT}{dx} + \frac{r}{S} \exp\left(\frac{x}{S}\right) = 0, \quad (1)
$$

where u is the linear burning rate, cm/sec, ρ is the density, g/cm^3 , C is the specific heat, cal/g.deg, r is the radiant heat flux entering the solid zone, cal/cm²·sec, λ is the heat conductivity, cal/cm-sec-deg, and S is the distance at which the intensity of the radiant flux decreases by e (-2.7) times, cm. It is known that S depends on the wavelength of the light; here, for simplicity, we have selected the average value for the flame spectrum.

The solution of (1) with the boundary conditions $T = T_0$ at $x = -\infty$, $T = T_1$ at $x = 0$ is

$$
T-T_0=(T_1-T_0-6)\exp\left(\frac{x}{t_0}\right)+6\exp\left(\frac{x}{s}\right), (2)
$$

where

$$
\theta = \frac{r l_0}{\lambda \left(1 - \frac{l_0}{S}\right)},\tag{3}
$$

$$
l_0 = \frac{\lambda}{u \rho c} = \frac{\lambda}{u}.
$$
 (4)

The quantity θ shows by how many degrees the solid phase of the burning explosive is heated by radiation.

Taking logs in expression (2) and considering that when S $> l_0$ and $|x| > l_0$ the term $(T_1 - T_0 - \theta)$ exp (x/l_0) becomes negligibly small, we obtain

$$
\ln (T - T_0) = \ln \theta - \frac{|x|}{S} \text{ when } S \geq I_0; |x| \geq I_0, \quad (5)
$$

consequently, in $ln(T - T_0) - x$ coordinates this must represent a straight line which cuts off an intercept In θ on the ordinate axis and has a slope S^{-1} . As may be seen from Fig. 5, at pressures of $20-45 \text{ kg/cm}^2$ in these coordinates the temperature profiles are, in fact, linearized and only at 10 kg/cm² (l_0 approaches S) is there a deviation from the straight line. At $S \gg$ $> l_0$ expression (3) is simplified to

Fig. 4. Variation of the product $u \, l$ for pure tetryl (1) and tetryl with gas black (2).

The values of θ , r, and S obtained are presented in Table 1, where it is shown that S does not depend on pressure. This serves as an indirect confirmation of the expansion of the heating zone, as compared with that predicted by the Michelson law, owing to radiative heat transfer. In fact, the coefficient of absorption of light $1/S$ is a characteristic of the material and the radiation and should not depend on the burning rate (in the region where combustion is complete and the flame has sufficiently large linear dimensions). If the width of the zone were determined by heat conduction and chemical reactions, then it would be inversely proportional to the heating rate.

Since, as a rule, all secondary explosives have a bright hot flame and sufficient transparency $(S \gg l_0)$, we may assume that many explosives and some powders, like tetryl, have anomalously wide heating zones owing to radiative heat transfer. To verify this assumption, we measured by the method described above the temperature distribution in burning PETN at $p =$ = 25 kg/cm². It was found that the width of the heating zone in PETN under these conditions is also anomalously large and is determined by the radiation of the secondary flame and the transparency of the material.

From the experimental data and formulas presented it is clear that owing to radiative heat transfer there is a considerable accumulation of heat inthe condensed phase heating zone (exceeding the accumulation of heat in the Michelson layer) with a maximum at a certain

pressure (for tetryl 20 atm). With change in pressure the accumulation of heat also changes. Consequently, heating of the explosive by radiation from the flame must have a substantial effect on the transient and limiting combustion regimes (combustion with rapid variation of pressure, ignition, quenching, amplification of aceoustic waves in the combustion zone, limiting combustion diameter). In all the experiments described the inequality $A = (L/c) + T_1 - T_0 - \theta > 0$ (T_i is the melting point, T₀ the initial temperature, L the heat of fusion) is satisfied; hence it is clear that the fraction of heat required for heating and melting the solid material is supplied from the molten zone by thermal conduction.

By approximately describing the temperature distribution in the molten zone by means of Michelson's law, we find the distance from the surface of the condensed phase (or from the chemical reaction zone) to the boundary of the solid and liquid phases

$$
x_1 = l_0 \ln \frac{A}{A + T_s - T_1}
$$

As A approaches zero, the distance x_1 tends to infinity, while the heat flux from the molten zone to the solid phase approaches 0. If $A < 0$, then the heat flux, formally oucA, also become negative, i. e., heat will pass from the solid phase to the molten zone, which is impossible. Hence it is clear that in the stationary case the inequality $A > 0$ must be satisfied. Otherwise $(A < 0)$ combustion is nonstationary, the fusion surface being propagated more rapidly than the flame front, while the molten zone expands. This expansion may cease owing to heat losses. Under certain conditions this combustion regime with an anomalously broad molten layer may occur for slowly burning explosives with a hot flame, for example, for PETN. It is not impossible that precisely this regime is connected with the known fact that PETN ceases to burn in a certain pressure interval, a fact attributed by Andreev and Popova [4,9] to combustion instability of the molten phase as described by Andreev [4] and Landau [10].

Fig. 5. Shape of temperature profile in semi-logarithmic coordinates at a pressure of 10 atm (1), 20 atm (2), and 45 atm (3).

HEAT BALANCE OF CONDENSED PHASE

To determine the heat balance of the condensed phase we integrate the heat conduction equation from the initial temperature to the surface temperature of the condensed phase

$$
c(T_s-T_0)+L+Q_e-Q_\lambda-Q_x-Q_\mu=0,
$$

where $c(T_s - T_0)$ is the heat going toward heating the explosive to the temperature T_S , L, Q_e is the heat goint toward fusion and evaporation, Q_x is the heat released owing to chemical reactions in the reaction layer of the condensed phase, and Q_{λ} , Q_{R} is the heat transferred to the condensed phase by thermal conduction and radiation.

The quantities T_s , T_0 , Q_λ are known from the experiments described above; the quantity $L = 20.6$ cal $\cdot g^{-1}$ [3]. An upper bound can be obtained for the quantity \mathbb{Q}_R if we consider that the entire heat flux of the flame, radiating as a black body of the same temperature, reaches the condensed phase and is absorbed in it; a lower bound can be obtain by equating \mathbb{Q}_R to the quantity $r/\rho u$ absorbed in the solid phase (r is measured). Concerning the quantity $Q_{\rm e}$ we know in advance only that it lies between zero and the heat of vaporization of tetryl L_{e} , since it is not clear what fraction of the tetryl is able to evaporate and how much of the material is dispersed or foamed (the decomposition product of tetryl--picric acid--has a heat of vaporization close to that of tetryl: 93 and 91 cal/g, respectively). The heat balance equation was used to compute the quantity Q_x and $Q_1 = c(T_e - T_0) + L$ —the total reserve of heat in the condensed phase; in computing $Q_{\text{X max}}$ it was assumed that $Q_e = L_e$ and $Q_R = r/\rho u$, while in computing Q_{X} min it was assumed that $Q_{\text{e}} = 0$ and $Q_{\text{R}} = R/\rho u$ $(R = 50 \text{ cal/cm}^2\text{·sec})$ (Table 2).

It is clear from Table 2 that at pressures of $10-45$ atm the contribution \mathbb{Q}_R to the heat balance of the condensed phase is not less than the contribution Q_{λ} ; it is still not possible to compute Q_X owing to the indeterminacy of the quantities \mathbb{Q}_R and \mathbb{Q}_e . In order to determine this quantity it would be necessary to make detailed measurements of the temperature distribution in the reaction layer of the condensed phase, which has not yet proved possible.

Table 1

Quantity	p , kg/cm ²				
		7	70	20	45
T_s° C (expt.) T_h° C (calc.)	310 310		350 377	400 400	405 427
$(dT/dx)_{s} \cdot 10^{-4}$, deg/cm 2.7			5	7.5	15
cm/sec u. u_{1} cm/sec °C 0.	0.045 0.18 0	0.19 $_{0}$	0.22 0,25 45	0,35 89	0,55 0.55 78
r, cal/cm2.sec S. cm	0	43	÷ 0.12	Đ 0.08	17 0. I
r/R, %			κ	21	34

Note: u is the burning rate of pure tetryl, u_1 is the burning rate of a mixture of tetryl and 5% gas black, R is the maximum possible luminous flux from the flame zone. $R = \sigma T^4$ (R = 50 cal/cm²-sec.

 $C = 0.23 \text{ cal/g-deg [3]: } \lambda_1 = 1.2 \cdot 10^{-14} \text{ cal/}$ /cm-sec-deg. In computing λ_1 it was assumed that above the surface carbon is present in the form of individual particles surrounded on all sides by gas. The heat was measured in caloties per gram.

Clearly, without improved measurements and more specific knowledge of the combustion mechanism it will not be possible uniquely to determine the heat balance of the condensed phase. We offer certain considerations that make it possible to estimate more accurately the amount of heat going toward evaporation of the explosive. First, we shall consider the limiting case when there are no heat sources or chemical reactions in the condensed phase. The surface of the condensed phase is approached by a heat flux transferred from the gas by the thermal conduction $\mathbf{q}_{\pmb{\lambda}}$ and partially by radiation q_R^{\dagger} (we have in mind the part of the radiant flux absorbed in a quite thin surface layer; the thickness of this layer requires more accurate determination). This heat flux goes toward heating the material to the surface temperature and toward evaporation of material from the surface. In this case we always have $q_{\lambda} + q_{\text{R}} > L_{\text{e}} u \rho$.

We now turn to a consideration of a second limiting case, when there is no heat flux from the gas phase and the net reactions in the condensed phase are exothermic. As a rule, gas bubbles are formed in the chemical reaction zone. A growth of the number and size of these bubbles due to evaporation and decomposition of a relatively small fraction of the condensed material leads to intense dispersion or foaming of the main mass of material; consequently, in this case the consumption of energy for evaporation is inconsiderable (as distinct from the first limiting case} and the surface is formed by dispersion and foaming, not surface evaporation.

We shall cite one more reason in favor of the idea that in the presence of chemical reactions and heat release in the condensed phase only a small fraction of the material is evaporated. In the limiting case considered (no heat flow from the gas phase to the surface of the condensed phase) stationary propagation

of the flame is incompatible with surface evaporation, since in this case the heat going toward evaporation must reach the surface from the interior of the condensed phase, and for this it is necessary that the surface temperature be lower than the temperature in the interior of the condensed phase, which would lead to the formation of a peak and a trough in the temperature profile. In the event of stationary propagation of the flame front the temperature increases smoothly, without change of the sign of the derivative with respect to the coordinate, from the initial value to the maximum value in the gas phase, i.e., in stationary combustion the temperature profile does not have temperature troughs. This principal applies for quite general (but not any) assumptions concerning the kinetics of the chemical reactions. In this paper we shall not consider the limits of applicability of this principle, and therefore it is regarded here as an initial assumption.

In the presence of dispersion and foaming of the surface layer the concept "surface" has a somewhat arbitrary character and relates to the zone where a sharp decrease in density takes place [11-13].

It is also possible to envisage pulsating combustion taking place as follows: A thin surface layer of the condensed phase is overheated and then begins to boil up, the next layer is overheated (or supersaturated with gaseous reaction products), and so on. In the presence of such pulsations (which, instead of being periodic, may also occur from time to time against a background of stationary combustion) it is also unnecessary to expend heat for the evaporation of the entire condensed phase or even a considerable part of it in order to form a new surface.

In the more general case when heat does flow from the gas phase to the surface of the condensed phase and intense chemical reactions take place in the condensed phase with liberation of heat (owing both to chemical reactions and to the absorption of thermal radiation), starting from the assumption of the absence of troughs in the temperature profile, we obtain the conditions

$$
Q_e \sim Q_{\lambda} + Q'_{R^*} \tag{7}
$$

where $Q_{\rm R}$ is the part of $Q_{\rm R}$ absorbed in a surface layer so thin that all the gas formed is able to diffuse to the

surface and the liquid does not boil. These considerations help us to refine the relations between Q_e , Q_x , and Q_B , by imposing on them the above limitation. This limitation is especially important at small heat fluxes from the gas phase $Q_{\lambda} < L_{e}$, since at large fluxes $Q_{\lambda} > L_e$ the inequality obtained above is automatically satisfied.

It may be assumed that in a number of cases the condition

$$
Q_e = Q_\lambda \text{ when } Q_\lambda < L_e,
$$

$$
Q_e = L_e \text{ when } Q_\lambda \ge L_e
$$

is satisfied. In this case the heat flux Q_{λ} begins to have a substantial effect on the burning rate only if $Q_{\lambda} > L_{e}$. As may be seen from the data presented, for tetryl burning under the experimental conditions described we always had $Q_{\lambda} < L_{e}$.

Table 3 presents the heat balance of the condensed phase; in computing Q_{max} we assumed that $Q_{\text{e}} = Q_{\lambda}$ and $Q_R = r/\rho u$, while in computing Q_{min} it was assumed that $Q_e = 0$ and $Q_R = R/\rho u$.

We shall obtain a more accurate value for the quantity Q_R . The heat flux absorbed in the solid phase has been measured. In the molten phase there is almost no absorption of light (this is clear from the results of analysis of the measured temperature profile) owing to the transparency of the material. Nothing is known in advance of the absorption of light in the reaction laver of the condensed phase. The formation of bubbles and a divided uneven surface may lead to considerable absorption of light. However, it is possible to assume that the greater part of the light will be absorbed not in the thin reaction layer of the condensed phase, but in the much wider layer of the smoke-gas phase adjacent to the burning surface, occupied by a two-phase mixture of intermediate reaction products and particles of dispersed and foamed condensed phase.

If we set $Q_R = r/\rho u$, we obtain the following heat balance (Table 4).

By comparing Q_x with the value of Q for tetryl, equal to 341 cal/g $[5]$, we can estimate the fraction of tetryI decomposed in the condensed phase. This quantity is from 13 to 25%. The remainder of the tetry] is dispersed and foamed.

From Table 4, based on the experimental data and the asumptions made above, it follows that the greater part (from 80 to 100%) of the heat of the condensed phase Q_t is associated with the exothermic reactions proceeding in it and also with the absorbed radiation from the flame. Only at 1 kg/cm² is a smaller value (50%) obtained.

BURNING RATE

To obtain a more accurate picture of the heat balance of the condensed phase and the combustion mechanism we shall compare the measured values of the burning rate with the values calculated under certain assumptions concerning the combustion mechanism. Above it was shown that the surface temperature of tetryl coincides with the boiling point (it would be useful to measure the relation between the surface temperature of tetryl and other explosives and the initial temperature).

We shall compute the burning rate of tetryl for the following combustion model:

1) surface temperature equal to boiling point;

2) radiant energy absorbed in the solid phase, but not absorbed in the liquid phase or the reaction layer of the condensed phase;

3) surface of condensed phase formed by dispersion and foaming, i.e., $Q_e = 0$;

4) heat flux from gas phase neglected;

5) a zero-order reaction takes place in the condensed phase.

With these assumptions the burning rate will be described by the following formula obtained by the Zel'dovieh- Frank-Kamenetskii method:

$$
\frac{1}{\left(T_s - T_0 - 0 + \frac{L}{c}\right)} \sqrt{\frac{2 \kappa \, QRT_s^2}{cE} \, z \, \exp\left(-\frac{E}{RT_s}\right)}
$$

If θ and L tend to zero, we arrive at the known formula of Ya. B. Zel'dovich [14],

The value $x = 10^{-3}$ cm²/sec for liquid tetryl was determined by analyzing the temperature profiles in the liquid phase of burning tetryl. In calculating the burning rate we employed the chemical kinetics of thermal decomposition of tetryl in the liquid state obtained by Robertson at temperatures of $210-260$ °C, i.e., $z = 10^{15.4}$ 1/sec; $E = 38,400$ cal/mole [4]. The calculation of the burning rate gives satisfactory agreement with the experimental data (Table 4). Andreev and Plyasunov earlier made a similar calculation of the burning rate of tetryl with several other values of the constants and concluded that the chemical reaction in the condensed phase plays a leading part [6].

COMBUSTION MECHANISM

On the basis of direct measurements it has been established that the solid phase $(T < T_i)$ is heated by radiation from the flame and thermal conduction, the greater part of the accumulation of heat in the heated layer being attributable to radiation (at pressures of $10-50$ atm). The intensity of the radiant flux from the flame to the solid zone has been measured. The temperature profile due to thermal conduction and radiation is in agreement with theoretical profile.

It has been established that the surface temperature is equal to the boiling point of tetryl, which experimentally confirms the known view of A. F. Belyaev [15] concerning the combustion mechanism of volatile explosives. We note that it is much more important to know the surface temperature when a leading role is played by the chemical reactions in the condensed phase than when the burning rate is determined only by reactions in the gas phase.

By constructing the heat balance of the condensed phase and comparing the calculated and measured burning rates it has been shown that during combustion a fresh surface is formed not by evaporation, but chiefly by dispersion and foaming. The burning rate of tetryt

is mainly determined by the chemical reactions in the condensed phase.

Thus, the combustion mechanims of tetryl is very similar to the combustion mechanism of powders described by P. F. Pokhil [II]. The difference evidently consists in the fact that the surface temperature of tetryl is equal to the boiling point, while the surface temperature of a powder is determined by the laws of dispersion and foaming [16].

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