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A CRITICAL REVIEW

OF RECENT

RESEARCH ON THE MECHANISM

OF IGNITION

OF SOLID ROCKET PROPELLANTS

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A CRITICAL REVIEW OF RECENT RESEARCH ON THE MECHANISM OF IGNITION OF SOLID ROCKET PROPELLANTS¹

by

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I. INTRODUCTION

An understanding of the details of the physical and chemical processes by which a solid propellant rocket motor is brought to a state of steady combustion is obviously of considerable interest to the designer of propellant ignition systems. By such understanding he may, for example, avoid excessive ignition pressures, minimize the weight of an igniter for a given rocket, eliminate excessive ignition delays, program the shape of the ignition transient, or accomplish other desirable objectives.

In general, the overall solid rocket ignition transient consists of three phases after the igniter itself has begun its action. At first, the products of igniter combustion heat the exposed surfaces of the propellant grain and bring some areas to ignition. As this process continues, there is an interval of so-called flame spreading during which the burning area increases as a consequence of propellant combustion itself. Finally, after the entire surface is

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⁵Member, Research Staff, Department of Aeronautical Engineering, Princeton University, Princeton, New Jersey ignited, there is a chamber filling period during which the equilibrium pressure is established. Figures 1 and 2 illustrate the events occurring during these intervals in a schematic manner. In this review attention will be confined to the mechanism of the process by which the first flame in a solid propellant sample is ignited.

In a practical situation the energy impulse which leads to the first flame is always complex and dependent on the type of igniter employed. The processes which contribute to the heat transfer include convective heating by igniter gases, radiation and conduction from incandescent particles, heat liberated by the condensation of metallic vapors, the action of hypergolic materials, and combinations of these and others. In experimental studies of solid propellant ignition, one or more of these processes are used.

Such studies have occupied a number of investigators in recent years. A number of experimental techniques varying in form and concept have been used to study the ignition of composite and nitrate ester propellants. Both slow and fast ignitions have been studied, and ignition delays have ranged from 20 seconds down to less than a millisecond.

In the experiments which have been conducted, the propellants have been heated by hot wires, test furnaces of various types, flowing gas streams at elevated temperatures, shock tubes and shock tunnels, highly reactive oxidizing gases, arc-image furnaces, etc. This review will contain a description of the various theories which have been used to account for the phenomenon of propellant ignition, a discussion of the experimental methods which have been applied to this study, and an evaluation of the reported results.

II. THEORIES OF PROPELLANT IGNITION

A. Discussion of Ignition Criteria

A solid rocket propellant is a solid substance which is capable of burning at a constant rate varying from approximately 0.1 to 5 cm/sec at a fixed pressure. In the steady-state burning of such materials, it is commonly postulated that for components of the solid gasify at the surface and then thin, intense gaseous flame in the vicinity of the surf This extremely simplified picture of the combustion of a field propellant, of course, ignores complications due to field and flame zone structure and it ignores the participation of surface and subsurface reactions in the overall combustion process.

The ignition of such a solid propellant is the process whereby the quiescent solid achieves a state of steady combustion. Experimentally, of course, it is difficult or impossible to define precisely when a propellant may be said to be ignited. However, in experimental studies of the ignition process, two criteria to define the onset of ignition are in general use. The first of these criteria is the detection of the first luminous flame or the first light output from the igniting propellant. The second criterion of ignition is based on the initiation of the pressure rise in a combustion chamber. The effectiveness of the light detection experiment depends, of course, on the sensitivity of the detecting system, whereas the pressure record depends on the response time of the chamber as well. It should be pointed out that the two measurements may detect different states of the ignition event.

When the ignition process being studied is a rapid one, the difference between the results obtained from the two ignition criteria may be quite important. However, when the ignition being studied is a slow one, this distinction is probably unimportant. At the same time, if the ignition of a solid propellant is marginal or incomplete, the difference between the times obtained by the two ignition criteria will be quite significant. (See for example the remarks under Section III-E below.)

Three basic models for the physico-chemical processes which control solid propellant ignition have been proposed. These models consider chemical reactions: a) in the solid phase, b) in the gas phase adjacent to the solid surface, and c) at the solid-gas interface. These three models will be discussed in the following sections.

B. Solid Phase Ignition

In steady state burning of a solid propellant a significant part of the reaction always occurs in the gas phase. The assumption that the ignition process is controlled in the solid phase only implies that the ignition is triggered by an exothermic solid phase reaction and that the time needed to establish a day phase reaction as were smalling compared to pro-

It is generally assumed that such reactions contribute to the

However, in ammonium perchlorate propellants, the situation is much less clear and most investigators claim that endothermic sublimation precedes any exothermic decomposition reaction. The most complete mathematical treatment of ignitions caused by exothermic solid phase reactions, specifically applied to solid propellants, has been reported by Hicks (19). His treatment is similar to that used by others (1,2,3,4) in studies of the thermal initiation of explosion. Hicks considered the case of a solid decomposition of zero order (with respect to both external pressure and reactant concentration) and a convective heat flux at the surface. It was assumed that the temperature dependence of the reaction rate could be described by an Arrhenius function. The partial differential equation describing the flow of heat for this case is

$$\frac{\partial T}{\partial t} = \alpha_p \frac{\partial^2 T}{\partial x^2} + \frac{9 Z}{\rho C_p} e^{-\frac{E}{R}T}$$
[1]

The boundary conditions stated by Hicks were:

 $T = T_0$; $X \rightarrow \infty$ (in the solid)

and

$$-\lambda_{p}\frac{\partial T}{\partial x} = h\left(T_{c} - T_{p}\right); x=0 \text{ (at the surface)}.$$

Hicks solved this equation numerically. He also found an easily calculated, empirical method of estimating the propellant ignition delay. In this method, the solid was considered nonreactive until purely conductive heat transfer brought the propellant surface to a certain temperature at which temperature ignition occurred. In other words, the heat evolution due to exothermic decomposition is neglected up to the time at which its effect on the energy transport is comparable to that of heat transfer. A parameter \prec was defined by equation [2] and it was found by comparison with the numerical solution that ignition occurred when a $7\frac{4}{34}$ was reached such that α' had a value of 0.833.

$$\alpha^{L} = \frac{\frac{2e^{-E/RT_{sl}}}{\sqrt{2}e^{E/RT_{sl}} + \left(eC\frac{\partial T_{s}}{\partial t}\right)^{L}}}$$
[2]

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The surface temperature was calculated from equation [1]

at the same temperature For any $T_{\rm sc}$, equation [2] may be rewritten, (10), to obtain a relation between the applied heat flux and the ignition delay.

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$$\frac{d \, \ell_{m} \, t_{sen}}{d \, \ell_{m} \, \dot{q}} = \frac{\frac{E}{R} \left(T_{si}^{L} - T_{o} \right) - \left(T_{si}^{L} \right)^{2}}{\frac{E}{R} \left(T_{si} - T_{o} \right) + \left(T_{si}^{L} \right)^{2}}$$
[3]

Equation [3] implies that a plot of log $t^{\frac{1}{2}}$ against log (\dot{g}) should be linear with a negative slope ign having an absolute value less than one.

Several properties of this empirical method must be emphasized. Hicks showed that the influence of various physical parameters on t depends on the mathematical choice of ignition criterion. ign Also, it should be pointed out that the ignition criterion specified by equation [2] does not correspond directly with any ignition event commonly measured. In addition, the method of plotting expressed by equation [3] must be limited to cases in which the time delay between the start of appreciable reaction and fast burning is very small compared to the time, t_{ign} , needed to raise the surface temperature to (7 t).

A common feature of many high activation energy ignition processes is that the exact definition of the ignition event is of minor importance when the heating rate is low. The same argument applies in the adiabatic case when the initial temperature is very low. Exact definition and measurement of the ignition event does become important when high heating rates or high initial temperatures prevail and ignition times are relatively short.

The work of Hicks is a valuable contribution to our understanding of the ignition process, for it provides the first accurate numerical solution of the pertinent, nonlinear differential equation. His results have been widely misinterpreted, however.

The relation between ignition time and heat flux given by equations [2] and [3] were derived for an ignition controlled by solid phase reactions, but the experimental demonstration of the linear relation predicted by equation [3] neither proves nor disproves the solid phase mechanism. Indeed, it can be shown that this relation will hold for many high activation energy ignition processes, particularly if they are slow. Thus, the same behavior might be expected for all the previously postulated ignition models. A proper experimental discrimination among the models can be approached only through the variation of parameters other than the heat flux.

C. Gas Phase Ignition

Ambient pressure and oxygen concentration have been found to be important parameters influencing solid propellant ignition

of gas phase ignition will include only ignitions in which the major exothermic processes leading to temperature rise occure in the gas phase and are governed by gas phase kinetics. In the description of this type of model it is postulated that the heating of a solid propellant by an external heat flux causes evaporation or chemical decomposition at the surface. Such gas evolutions have indeed been observed at the temperatures estimated to prevail during ignition processes. These gases then enter into a rapid exothermic reaction in the small region adjacent to the surface. Heat evolved by this rapid reaction then increases the temperature of the surface by heat feedback until a steady state of combustion is reached. This type of reaction would be very rapid indeed at the high gas temperatures that exist during the ignition of current solid rocket motors. It is evident that the presence of oxidizing gases and increases in ambient pressure would tend to accelerate these reactions.

An interesting and practical case of the gas phase ignition of a solid propellant would be an ignition caused by propellant decomposition and subsequent gas phase reaction of oxidizer and fuel product gases. Such an ignition is quite possible conceptually, but has never been treated. A quantitative treatment of such a model would be quite interesting.

Attempts have been made to treat another gas phase ignition model. This model involves the ignition of the fuel binder of a composite propellant through the gas phase oxidation of the binder decomposition products by externally supplied oxygen. The ignition of fuel vapors could be the controlling process in the ignition of a composite propellant having an easily vaporizable binder exposed to a hot oxidizing gas.

Such an ignition process could, of course, take place under the influence of conductive, radiative or convective heat sources in either stagnant or moving gases. In a simple formulation of such a model, the propellant is brought into sudden contact with a hot stagnant gas. Under assumed conditions of conduction heat transfer and constant surface temperature, the following set of diffusion equations may be written for events occurring in the gas phase and at its boundary.

Mana Diffusion. 1.5

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2 Con = D 2 Cor - A Cr Con E € FIRT

(5)

Energy Diffusion:

$$\frac{\partial T}{\partial t} = \propto \frac{\partial^2 T}{\partial x^2} + \frac{9}{PC_0} G_0 C_0 Z_0^{-E/RT} \qquad (c)$$

with boundary conditions:



The following assumptions are, of course, inherent in this model:

- 1) all transport properties are constant,
- convective mass transport can be neglected everywhere,
- the gas density is independent of temperature,
- the reaction is second order and the rate is described by an Arrhenius equation.

McAlevy, Summerfield, and Cowan (12) have treated this case of a cold fuel coming into contact with a stagnant, hot oxidizing gas. They assumed that the surface mass flux of fuel was constant and that oxidizer diffusion was unimportant. However, because of simplifying assumptions made in the analysis, the applicability of their result is rather limited.

A more general treatment of equations [4] [5] and

(2,5,6) with two different boundary conditions on the fael supply.

Previous investigators have inferred the mechanism of the ignition process from the magnitude of the dependence of ignition delay on oxygen concentration. Among the findings of Hermance, Summerfield, and Shinnar was the observation that this magnitude depends on the exact specification of the ignition event. Another finding was that the slope of a plot of the logarithm of ignition delay versus the logarithm of oxygen concentration is not necessarily constant and can vary over wide ranges. These effects occur as a consequence of the influence of oxygen diffusion and consumption. They are important because they indicate that the slopes of the ignition delay-oxygen concentration curves cannot in themselves prove or disprove the validity of any ignition model. Although the available theoretical treatments of gas phase ignition are limited to cases of externally supplied oxygen, the concept of the gas phase ignition may realistically be applied to other cases.

D. Ignition Due to Heterogeneous Reactions At The Gas-Solid Interface

It has been suggested that the effect of gaseous oxygen on ignition delay, which will be described more fully below, could be due to an exothermic oxygen attack on the solid material at the gas-solid interface. The generation of heat there would lead to a subsequent increase in the heat flux to the solid and thus, to ignition. Similar reactions could occur in composite propellants, even in neutral gas environments, where oxidizing gases from the decomposing ammonium perchlorate could react with the organic binder at the interfaces between the binder and oxidizer particles. Some support for this assumption can be found in the fact that ammonium perchlorate starts to decompose at much lower temperatures than most of the polymeric binders in use, (20).

A mathematical model for such a surface reaction can be formulated under the assumption of a one-dimensional, semi-infinite domain of stagnant, oxidizing gas, bounded at one side by a semi-infinite solid with which the gaseous oxidizer reacts at the surface. With this picture in mind, the following transient diffusion equations may be written.

Energy Diffusion.

Mass Diffusion:

Solid:

 $\frac{\partial T_F}{\partial t} = \propto_F \frac{\partial^2 T_F}{\partial x^2}$

 $\frac{\partial C_{\text{OX}}}{\partial t} = D \frac{\partial^2 C_{\text{OX}}}{\partial x^2}$

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These equations are assumed to have the following constraints:

$$\frac{C_{ox}}{C_{ox}} = C_{ox}^{\infty}$$

$$T_{c} = T_{c} = T_{c}$$

+ 40:



In writing these equations, the following assumptions are made:

- 1) Convective mass transport can be neglected
- 2) Density is independent of temperature
- 3) Molecular weights and diffusion coefficient of all gases are equal
- 4) The surface reaction is of order "n" and
- can be represented by an Arrhenius function5) The temperature of the system is initially uniform.

Anderson and Brown (21) solved a similar set of equations numerically, and found that the ignition delay was proportional to the oxygen concentration raised to a power approximately twice the order of the surface reaction. This solution was fitted to some of the data reported in reference (12) with the assumption of a first order surface reaction. This fit was offered in support of the validity of the model.

Because oxygen also affects reactions occurring

ignition delay may be greater than the assumed order of chemical reaction. Therefore, the possibility of experimentally distinguishing between these two processes, each involving an oxidizing gas, is difficult. Such differentiation could be made by the separate variation of surface temperature and gas temperature adjacent to the surface in the same experiment. No such experiment has been performed.

III. EXPERIMENTAL STUDIES OF SOLID PROPELLANT IGNITION

A. General Considerations

In this section an attempt is made to sum up some recent experimental studies which are pertinent to the understanding of solid propellant ignition. This paper is not intended as a complete survey of the literature of this field. Before discussing the individual experiments, consideration will first be given to what experimental evidence is actually needed in order to distinguish between the different mechanisms outlined in the previous chapter.

Almost all experiments measure a total ignition time as characterized by either pressure or luminosity. If the heating is not immediate (as it is, for example, in some shock tube experiments), there will always be a time span during which all chemical kinetic processes can be neglected. This is true for all the processes mentioned, except ignition due to low temperature hypergolic reactions. The total ignition time can, therefore, be divided into several parts:

 $t_{ignition} = t_1 (heat-up) +$

t₂(triggering reaction solid, solid-gas or gas phase) +

t₃(final flame development)

For high activation energy and low heat flux processes, the heat-up time will control the ignition event, as both t and t, will be small as compared to t, in most cases. Slow² ignition experiments provide some ¹ valuable information and sometimes allow an estimate of a so-called ignition temperature. Actually this is not a fixed temperature, but the narrow temperature range in which chemical reactions first become rapid enough to contribute to ignition. This could be the temperature at which an exothermic reaction starts in the solid or it could be the temperature at which significant evaporation starts to occur. The disadvantage of relatively slow ignition experiments is that they provide little insight as to the nature of the ignition process and may obscure the effect of some physical parameters which are important in fast ignition processes.

An important clue which would permit a distinction to be made between a different mechanisms mentioned previously is the affect of pressure on ignition time. In order to establish, if such an affect exists two difficulties have to be overcome.

- t₁ (heat-up time) must not be large as compared to t₂ and t₃ mentioned before.
- The affect of pressure on the heat transfer process itself must be known very accurately.

Other important evidence is the influence of oxidizing gases on the ignition. Again a distinction must be made between slow and fast ignition, only the latter being of real significance. An unfortunate limitation of most experiments is that they do not provide means for independently varying over wide limits the solid surface temperature and the nearby gas temperature. The dependence of ignition delay on these two temperatures as independent variables would be an important clue to the process.

In all these researches, it must be remembered that the objective is to determine the processes that control ignition in a rocket motor. The conditions should be chosen to bring out the effects of the pertinent processes. In order for an ignition experiment to simulate conditions in a rocket motor as closely as possible, the following requirements should be met:

- a) the total ignition time should be very small (several milliseconds and lcss)
- b) the heat flux should be high
- c) the gas temperature should be very high (otherwise a gas phase delay might be introduced which does not necessarily exist in practical situations)
- d) pressure should be varied over a wide range, including near atmospheric pressures.

It is very hard to design such an experiment. In experiments which do not fulfill the above conditions it must always be remembered that in complicated reactions the controlling mechanism often changes with conditions. However, by summing up all the experimental evidence available, some significant insight as to the nature of the ignition process is possible. B. Hot Wire Ignition of Composite Solid Propellants

Altman & Grant, 1952, (5) (JPL, CalTech)

Altman and Grant (5) reported a study of the ignition of composite ammonium perchlorate propellants by heat transfer from a straight, electrically heated wire embedded in the propellant. The temperature of the propellant adjacent to the wire at ignition was computed from the measured ignition delay. They attempted to show that ignition was governed by a specific ignition temperature regardless of the magnitude of the heat flux.

In the experiments, the ignition delay was the interval between the onset of heating and the burn-out of a fuse wire. The operation of this system was checked, in early experiments, by high speed photographs.

Data obtained from this experiment are presented in a graph of power input versus ignition delay, see Figure 3 abstracted from reference (5). This curve is the authors' correlation of their results, using a one-dimensional heat conduction solution and an ignition temperature of 368°C. The results are in fairly good agreement with their hypothesis.

It should be pointed out that the results do not prove anything with regard to the propellant ignition mechanism. At these ignition times, longer than one second, almost any process having a high activation energy should be correlated by an ignition temperature, provided the pressure and gas composition are not varied.

In view of the preceding discussion of ignition theories, it must be noted that the experiment was incomplete with respect to possible effects of total pressure and test atmosphere composition. It would be interesting to have results at higher pressures and also at higher heating rates. An incidental observation reported by the authors which could be significant for the interpretation of the ignition process is that in these slow ignition tests a considerable gas evolution was noticed prior to the appearance of a flame. This could indicate that the decomposition vapors play a role in ignition, according to one of the models discussed above.

C. Explosion Tube Propellant Ignition Experiments

Cook & Olson, 1955, (6)

Cook and Olson (6) reported a series of experiments in which ignition of propellants was caused by impingement and reflection of a complex explosion or detonation front from a nitrate ester propellant sample mounted in an explosion tube. The tube was filled with various detonable mixtures of hydrogen and oxygen. The experiment was conceived as a test procedure for determination of propellant ignitability, with no consideration given to separation of pressure, temperature, and chemical effects from effects caused by the hydrodynamic situation or model mounting methods. However, the authors were cognizant of the importance of the ignition criterion in ignition studies, and observed that any criterion must be such that full scale burning always develops after its attainment.

The significant experimental results were reported in terms of the minimum hydrogen-oxygen charging pressure necessary to cause ignition of the propellant in the explosion tube, see Figure 4 abstracted from (7).

The most interesting feature of the results given in Figure 4 is the influence of oxygen concentration on the minimum charging pressure necessary for ignition. It is impossible to get any quantitative estimate of this effect as the final temperature, pressure, and wave front speed depend upon oxygen concentration also. The fact that the curves have a minimum at oxygen concentrations much above stoichiometric clearly indicates an influence of oxygen concentration on propellant ignition. If oxygen concentration were to have no influence, an approximate symmetry would be expected around the stoichiometric region.

D. Ignition of Nitrate Ester Propellants by Forced Convection

Churchill, Kruggel, & Brier, 1956, (8) (University of Michigan)

Churchill, Kruggel, and Brier (8) reported experiments in which cylinders of nitrate ester propellant were ignited in a crossflow of a heated test gas at atmospheric pressure. The gas composition and temperature were varied to include several oxygen-nitrogen mixtures, carbon dioxide, and pure nitrogen. The experimental ignition criterion used in the ignition delay determinations was the establishment of a luminous flame, close to the propellant surface, which led to combustion of the propellant sample. Their results are reported in Figure 5 in terms of an "ignitability function" versus the percent oxygen in the test gas.

Experimentally, it was observed that the ignition delay depended upon test gas temperature, flow rate, and composition; separate increases in test gas temperature, flow rate, and oxygen mole fraction were all observed to decrease the ignition delay. It was concluded that oxidation reactions were the controlling mechanism in double base propellant ignition under these experimental conditions.

A very interesting observation not stressed in the paper was the fact that consistent ignitions could not be obtained in inert atmospheres under the conditions employed. Also, ignition as defined by the appearance of a luminous flame, was not obtained at high flow rates and low temperatures. In some cases, the grain decomposed completely without the appearance of a luminous flame. This illustrates how strongly the ignition time measured in an experiment can depend upon the ignition criterion and the method used for measurement. The results also show that the time needed to establish a luminous flame is not necessarily negligible, compared to the time needed for the start of solid decomposition.

The influence of such parameters, such as test gas composition and temperature, could be completely different under the high pressure and temperature conditions which prevail in actual rocket motors. Thus, for example, in other experiments, propellants ignite consistently in inert atmospheres at high pressures. It would have been valuable if additional experiments of this type had been performed at higher pressures.

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E. Ignition of Nitrate Ester Propellant In A Pressurized Oven

Roth & Wachtell, 1962, (9)

Roth and Wachtell (9) studied the ignition of nitrate ester propellant samples subjected to radiant and convective heat transfer inside an oven in which pressure, temperature, and test gas composition were varied separately. Oven pressure and luminosity were monitored simultaneously to determine the total ignition delay and the delay between the start of fizz decomposition and a luminous flame reaction. Ignition delays between one and thirty seconds were obtained.

Little effect of oxygen concentration on the overall ignition delay was noted in experiments with M2 propellant tested in nitrogen, air, and helium over a 300 psi pressure range. Two stage ignitions, and a marked effect of oxygen concentration on the fizz-to-flame delay period, was observed in ignition tests of M9 propellant. This effect was observed in tests of M10 propellant at an initial propellant temperature of 0°C, but the effect disappeared with an increase in the initial temperature to 30°C. The experimental data are shown in Table I and Figure 6 which were abstracted from reference (9).

Because of the good fit which was obtained between the ignition delay and the heat flux, Roth and Wachtell concluded that pressure and oxygen concentration had no detectable effect and, therefore, that the overall ignition reaction was controlled by a solid phase decomposition. However, further examination of the data of reference (9) show that the fizz-to-flame delay could be quite significant, depending upon the test gas composition and pressure. It should be mentioned that the observed fizz-to-flame delay was, in itself, large compared to the total ignition delay in a rocket motor. The dependence of the fizz-to-flame delay on oxygen concentration has been replotted from the reported data in Figure 7. It can be seen that pressure and oxygen concentration have a marked effect on this delay.

The conclusions in reference (9) about the dependence of the fizz reaction delay on pressure and gas composition are at least open to question for, as mentioned previously, it is hard to measure such effects at low heat fluxes. The apparent effect of pressure which can be noted in Table I, was explained in terms of an increased heat transfer rate; however, the confidence limits of the heat transfer calculations are not sufficiently narrow to allow a definite conclusion with respect to a pressure effect.

F. Propellant Ignition By High Convective Heat Fluxes

N. W. Ryan, A. D. Baer, & D. L. Salt, 1960, (10) A. D. Baer, 1959, (11)

Ryan, Baer, and Salt (10), (11) have reported studies of propellant ignition. In one series of tests (10), composite propellant samples mounted in a constant area duct were ignited by a hot gas flow which was generated by the exhaust of shock tube driven gas. This gas was generated by a shock tube operating in a tailored interface mode. Test gas pressure and flow velocity were varied to produce a range of heat flux conditions and the gas composition was either nitrogen, air, or oxygen.

In another series of experiments (11), use was made of a low pressure radiation furnace in which temperature and gas composition were varied. No gas composition effect on ignition delay was observed in the furnace tests. At reportedly equal heat transfer rates, ignition delays were larger, by a factor of two to three, in the furnace than in the shock tube experiments.

Data from both of these experiments, abstracted from references (10) and (11), are shown in Figures 8, 9, and 10. The data treatment reported was suggested by an interpretation and development of Hicks' (19) linear surface temperature criterion in which a solid phase decomposition ignition mechanism was assumed. This method is illustrated by equation [3] above.

Ryan, Bacr, and Salt concluded that composite propellant ignition data were explained by Micks' empirical ignition temperature criterion, and that the overall activation energy was fairly well determined by their tests. They also concluded that the more rapid ignition of propellants exposed to pure oxygen test gas was due to an enhanced heat transfer to the propellant caused by an oxygen-fuel binder reaction.

The total pressure range (250 and 350 psig) in these experiments was much too small to permit any definite conclusion regarding the pressure dependence of the ignition reaction. The fact that the ignition delay in the radiation furnace at atmospheric pressure is three times longer than at elevated pressure in the shock tube is interesting, but not conclusive. The difference only illustrates the danger of using heat flux as the sole variable for comparison of ignition times measured under different experimental conditions. It has been pointed out already that, by itself, the form of the dependence of ignition times on heat flux gives no indication as to the ignition mechanism. G. The Ignition of Composite Solid Propellants In A Shock Tube

R. F. McAlevy, III, P. L. Cowan, & M. Summerfield, 1960, (12)

McAlevy, Cowan, and Summerfield (12) investigated the ignition of a composite propellant sample mounted flush with the end wall of a simple shock tube which was operated such that the pressure after the reflected shock remained constant (tailored interface operation). The action of the shock tube exposed the propellant sample instantaneously to a stagnant, doubly shocked, high temperature, high pressure gas. Ignition was detected by a filter-photocell system sensitive to radiation in the violet region of the spectrum.

The effect of variations in the oxygen content of the test gas on ignition delay was obtained under constant shock tube operating conditions. The results are shown in Figure 11as abstracted from reference (12). Tests in pure oxygen in which the pressure level behind the reflected shock was varied, gave the results shown in Figure 12. It was observed that no ignition was possible with an oxygen concentration less than 3 x 10^{-3} gm/cc in the test gas behind reflected shock. This might be explained by the limited test time of only five milliseconds. Samples of the propellant fuel binder tested under similar conditions also ignited, but with slightly longer ignition delays. Parallel experiments, as reported in reference (13), were carried out with nitrate ester propellants under the same test conditions. The data which was obtained are also shown in Figures 9, 10, 11. The nitrate ester propellants ignited more slowly than the composite propellants. It is interesting to note that some of the results reported in reference (22) seem to indicate that the dependence of ignition delay on oxygen concentration decreases at higher concentrations.

On the basis of these results and a simplified analysis of heterogeneous ignition, it was concluded that the dominant ignition delay for composite propellants was caused by a gas phase reaction between pyrolized fuel binder and the oxygen present in the test gas. The experiments do not allow such a definite conclusion as to the mechanism of the ignition and the nature of the oxygen effect. They do, however, clearly indicate that solid phase reactions do not dominate the composite propellant ignition process.

An important feature of these experiments compared to all other solid propellant ignition experiments, was the instantaneous establishment of high propellant surface temperatures by the shock reflection process. After the initial temperature jump, the surface temperature remains quite constant in the absence of any chemical reactions. This makes it difficult to compare these results with those of constant heat flux experiments.

H. Ignition of Composite Propellants By A Radiant Energy Flux

Fishman & Beyer, 1960, (14) Wise & Evans, 1963, (15)

The ignition of composite propellants by intense radiant energy in an arc-image furnace has been studied in considerable detail at the Stanford Research Institute (14), (15). In these experiments the intensity of radiant flux incident on the propellant sample was varied over a wide range, as was the pressure at a single flux level.

Fishman and Beyer (14) measured minimum ignition energy defined as the minimum quantity of radiant energy, applied in a pulse, needed to produce ignition some time after the application of radiant flux was terminated. Specific attention was paid to the important problem of the minimum time for which the radiant flux had to be applied for propellant ignition to occur. In reference (15), ignition delays were measured; the radiant flux was maintained at a constant level until ignition occurred. Ignition was detected photoelectrically in both cases. High speed photographic observations (14) seemed to indicate that at least the final stages of composite propellant ignition occurred in the gas phase. Voluminous evolution of gases was noted considerably before the appearance of a flame. The visible reaction started at a considerable distance from the surface and flashed back to it.

In Figure 13 the minimum exposure time causing ignition is plotted against pressure at a constant flux level. It should be pointed out that the exposure time reported was not an ignition time, as ignition occurred after the radiant flux was removed. The ignition times themselves were not reported. It is evident from this figure that the ignition processes are strongly procesure dependent, especially at low pressures. At high pressures, minimum ignition exposure times are less sensitive to pressure. This can be explained because as previously mentioned, the magnitude of the pressure effect depends on the ratio of flame development time to heat-up time. If this explanation is correct, the pressure dependence could be expected to become steeper at higher flux levels. That this is actually the case is seen in Figure 14 in which minimum ignition exposure time for different pressures are plotted as a function of the flux. In addition, it is apparent from this figure that at high flux levels the minimum ignition exposure time does not continuously decrease, but tends to a constant value. As yet there is no quantitative explanation for the apparent plateau in the dependence of minimum ignition exposure time on flux at low pressures as shown in Figure 14.

Figure 15, obtained from reference (15), shows that even with a constant flux applied up to and including ignition, the ignition delay is pressure sensitive. A comparison of Figures 13 and 15 indicate that in Fishman and Beyer's short ignition time experiments, ignition occurred at the end of the minimum energy exposure interval. Measurements of minimum ignition energy with a constant flux level at atmospheric pressure in a test gas containing oxygen, show a very pronounced oxygen effect. This is illustrated in Figure 16.

The above results indicate very convincingly the importance of gas phase reactions in the ignition of composite propellants. It is interesting to note that a simplified model based on the ignition temperature concept would lead to erroneous conclusions for the last case. The assumption of an ignition temperature is justified only if heat-up is the controlling element in the total ignition delay.

Ignition by radiant flux is advantageous because it allows variation of ignition time over a wide range at reproducible and controlled conditions. Its main disadvantage is that it involves the presence of a cold gas near the surface. This introduces an additional gas phase resistance which is not present in rocket motors where the temperature close to the surface is very high. For gas phase ignition processes, there is a basic difference between a case where the dimensionless temperature (RT/E) is high (0.1 or larger) at the beginning, and one in which (RT/E) is very small. In the first case, any gases coming off from the surface will tend to react immediately while diffusing into regions of high gas temperature. The heat fed back from the reaction will then increase the temperature at the surface causing a still higher gas evolution. In the second case, there must be a build-up of concentration of reactive gases near the surface, before a runaway reaction may occur.

I. Composite Propellant Ignition In A Small Rocket Motor

E. H. Grant, 1963, (16) R. W. Lancaster, 1961, (17)

Grant and Lancaster (16) and (17) have reported a study of the ignition of ammonium perchlorate composite propellants in a small rocket motor. Hot gaseous products from a gas fed, pyrogen-type igniter were used to apply a convective ignition stimulus to the internal surface of a thin-webbed, cylindrical solid propellant grain. A schematic diagram of the experimental system is shown in Figure 17.

Ignition was detected from pressure records and was identified as the first rise over the dummy chamber pressure established by the igniter. An idealized pressure record is shown in Figure 18. The gas mixtures introduced into the igniter contained various proportions of methane, oxygen and nitrogen. The mixtures were selected to have the same adiabatic flame temperature, but a wide range of equilibrium oxygen concentration in the burnt gases. The mass flux through the motor was also held constant in an attempt to maintain a constant heat transfer rate in all experiments.

Ignition delays for a series of experiments conducted with a PBAA-80% ammonium perchlorate composite propellants are shown in Figure 19, plotted as a function of the oxygen weight fraction in the burnt igniter gas. It can be noted that strong pressure and oxygen effects on ignition delay are evident even with an ostensibly constant heat flux. The pressure effect even persists when there is no oxygen in the igniting gases. These factors strongly support the contention that the controlling step in the ignition involves a gas phase process.

It is also evident that the effect of oxygen weight fraction changes with pressure. At a pressure of 35 psig the oxygen effect is hardly significant, whereas at 110 psig there is a strong oxygen effect with the ignition delay decreased almost by a factor of five. At each pressure, a threshold effect was noted such that below a certain value, the effect of oxygen concentration was negligible.

Certain aspects of this experimental technique are open to some criticism. The gas temperatures and oxygen concentrations were calculated from equilibrium thermodynamic data and were not measured. These properties could vary due to changes in flow rates, poor mixing or failure to establish equilibrium. Based upon empirical correlations, the heat flux was kept constant by maintaining constant mass flow. Heat flux and oxygen concentrations in these experiments might have differed from their assumed behavior. There might have been variations in the heat flux in different experiments, and the actual oxygen concentrations might have been larger than were calculated to exist. However, the pressure and oxygen effects are so large that they cannot be explained away by experimental error. In order to explain the difference between maximum and minimum ignition delays, one would have to assure that the actual heat flux in one case was 400 times larger than in the other case.

J. Convective Ignition Experiments On A Composite Propellant

C. E. Hermance, 1963, (18)

Solid propellant models were exposed to the high temperature and pressure gas flow through a shock tunnel exhaust nozzle. Both supersonic and subsonic gas flows were used. The shock tunnel was operated in a manner which produced constant tunnel test pressure with an available testtime of 25 milliseconds. Various test gas mixtures of oxygen and nitrogen were used, and data were gathered by high speed photography.

It was found that no ignition of a fuel or propellant could be obtained in supersonic flow, even in pure oxygen. The ammonium perchlorate in the stagnation point region of hemisphere-cylinder propellant models was found to have been decomposed completely, regardless of the test gas composition. It was not clear whether the changes in the appearance of the fuel surface, both in propellant and fuel model tests under the same conditions, was due to a chemical reaction or a melting and erosion process.

Ignition was obtained in all subsonic flow ignition tests of propellant models shaped in the form of a flat plate with a sharp leading edge. The point of ignition was just behind the leading edge regardless of test gas composition or velocity. Though the reproducibility of these tests was low. a general trend of decreased ignition delay with increases oxygen mole fraction was found, see the circled data points in Figure 20.

The most interesting result of these experiments is the fact that the propellant did not ignite when exposed to the supersonic flow. This can be explained by the extremely small boundary layer thickness near the solid surface. Even if the gas were hot, it tended to quench any reaction due to dilution of the reacting gases. The results strongly indicate that hetergeneous solid phase reactions are not an important factor in composite propellant ignition. Both the propellant and fuel tests seem to cast doubt upon the existence of solid fuelgaseous oxygen reactions at the surface.

IV. DISCUSSIONS AND CONCLUSIONS

In the preceding sections the reader has been presented with an intricate and bewildering array of facts and speculations. The experiments have been quite varied as have the methods of data acquisition and treatment. Despite the apparent complexity which beclouds the experimental situation, several definite trends are clearly evident in the collected results.

The enhancement of composite solid propellant ignition by increased pressure has been observed by several investigators. This phenomenon is important in a practical sense; it has been clearly demonstrated and should be considered seriously in all future theoretical evaluations of the solid propellant ignition process. The enhancement of propellant ignition by the addition of oxidizer to the igniting environment is also clearly demonstrated although the practical consequences of this effect are less evident.

Several other conclusions become evident as a result of these two, well substantiated, observations. The assumption that a specific ignition temperature, independent of heating rate and pressure, is the sole parameter controlling ignition experiments is demonstrably invalid. The ignition temperature concept is still valid if it is regarded as an approximate temperature at which decomposition reaction rates become significant. A careful distinction should be made, however, between the heat-up process and the actual ignition event.

The assumption that composite solid propellant ignition is completely controlled by solid phase decomposition is refuted by the mass of experimental evidence cited above. The evidence clearly indicates that gas phase chemical kinetic processes dominate the ignition event, even in a neutral environment. There is not enough evidence available at present to make a proper evaluation of the role of solid phase processes in nitrate ester propellant ignition. However, it has been shown, even for nitrate ester propellants, that the time needed to establish a steady state flame in the gas phase cannot be disregarded and may be quite important. The time needed to establish a luminous flame in nitrate ester propellants is comparable to the ignition time of rocket motors.

No pressure effects were detected in some of the low heat flux experiments with nitrate ester propellants . However, in these cases pressure was not varied over wide ranges independent of heat flux. This result is inconclusive with regard to the ignition mechanism.

The effect of oxygen concentration on ignition delay which has been noted for both composite and nitrate ester propellants, is not constant and varies with experimental conditions. At low oxygen concentration the effect of additional oxygen on ignition time is generally very small, though it may affect flame stability at low pressure. The exact nature of the mechanism by which oxygen effects propellant ignition is not clear at present.

It is sometimes assumed that the hot gases produced by practical igniters are chemically inert with respect to the solid propellant surface. It would be interesting to investigate the validity of this assumption for chemically reactive fragments could influence the ignition process.

Though the experiments stress the importance of gas phase processes in solid propellant ignition, there is no conclusive evidence as to the nature of the controlling gas phase process. None of the existing experiments allow a clear experimental distinction between the alternative models outlined in sections II-C and II-D above. All the experimental evidence, both in neutral and oxidizing atmospheres, could be explained by gas-solid interface reaction as well as by a pure gas phase reaction. The experimental evidence is not accurate enough, and theoretical knowledge is not complete enough, to distinguish between the two mechanisms on the basis of the form of the pressure and concentration dependence of the ignition time.

In view of the essential lack of resolution of ignition delay experiments for the purpose of distinguishing between surface and gas phase processes, some different experimental approaches should be explored. What is needed now are experiments to measure some of the events which occur during the ignition process rather than the overall event. Either subtle, indirect experiments, or more refined direct experiments, are necessary to clarify this problem.

SYMBOLS

С	specific heat of solid				
c _p	specific heat of gas				
° _F	fuel concentration in gas phase				
cox	oxidizer concentration in gas phase				
Cox	initial value of C _{ox}				
D	mass diffusion coefficient				
Е	activation energy				
$f(C_F)$	any specified function of $C_{\overline{F}}$ or its spacial gradient				
h	convective heat transfer coefficient				
n	stoichiometric ratio or surface reaction order				
ģ	heat transfer rate or heat flux				
9 9 _R	heat of reaction				
R	universal gas constant				
т	temperature				
t	time				
tign	ignition delay				
Z	reaction rate pre-exponential				
× × ×	value of empirical ratio, from B. L. Hicks (19)				
\sim	thermal diffusivity				
à	heat conductivity				
6	density				
μ	viscosity				
Subscripts:					

F	fuel,	solid	or	gas
G	gas			

P propellant

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IGNITION OF M2 PROPELLANT

Roth & Wactell, 1962, Ref. (9)

		Ignition Time, Seconds				
	Results in Nitrogen					
ľξr _{en} p.,	0	50	100	200	300	
κ.	psig	psig	psig	psig	psig	
7, 72 3	30.50	17.50	12.30	8.80	5.40	
7, 79 8	15.40	9.30	6.80	5.00	3.10	
6 87 3	8.90	5.60	4.10	3.10	2.00	
94-8	5.50	3.70	2.70	2.10	1.35	
10102:3	3.70	2.45	1.85	1.50	1.10	
10102:3	2.60	1.80	1.40	1.10	0.75	

	Re	Results in Air			Results in Helium		
	0	100	300	0	50		
	psig	psig	psig	psig	psig		
123	25.80	12.02	4.40	9.12	8.54		
198	11.84	5.84	2.94	5.82	4.41		
6673	7.69	3.62	1.80	3.52	3.22		
948	5.22	2.41	1.36	2.41	2.02		
101023	3.36	1.57	1.00	1.71	1.28		
101028	2.17	1.25	0.74	1.08	0.90		

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SCHEMATIC REPRESENTATION, FLAME SPREADING PHASE OF IGNITION INTERVAL





PRESSURE TRANSIENT DURING

JPR 1552

FIGURE 2

JPR 181





FIGURE 3

20

NITRATE ESTER PROPELLANT IGNITION IN AN EXPLOSION TUBE

COOK AND OLSON, 1955, REF.(6) UNIVERSITY OF UTAH

PLOT OF MINIMUM CHARGE PRESSURE, PI, REQUIRED FOR IGNITION VERSUS MOLAR H2/02 RATIO OF CHARGING MIXTURE



JPR 1812

FIGURE 4

CONVECTIVE IGNITION OF NITRATE ESTER PROPELLANTS

CHURCHILL, KRUGGEL, & BRIER, 1956, REF. (18) UNIVERSITY OF MICHIGAN

PLOT OF AN INVERSE FUNCTION OF IGNITION DELAY VERSUS TEST GAS OXYGEN PERCENTAGE



JPR 1813
M2 NITRATE ESTER PROPELLANT IGNITION AT ATMOSPHERIC PRESSURE

ROTH & WACHTELL, 1962, (9) FRANKLIN INSTITUTE

PLOT OF SQUARE ROOT OF IGNITION DELAY VERSUS CALCULATED HEAT FLUX VALUES



M9 NITRATE ESTER PROPELLANT IGNITION AT ATMOSPHERIC PRESSURE

ROTH & WACHTELL, 1962, (9) FRANKLIN INSTITUTE

PLOT OF FIZZ-TO-FLAME DELAY VERSUS OXYGEN PARTIAL PRESSURE IN TEST GAS (M9 PROPELLANT DISCS)



JPR 1815

COMPOSITE PROPELLANT IGNITION BY CONVECTION IN A SHOCK TUNNEL

RYAN AND BAER, 1960, REF. (10) UNIVERSITY OF UTAH

SQUARE ROOT OF IGNITION OELAY VERSUS MEAN CONVECTIVE HEAT FLUX



JP R 1816

COMPOSITE PROPELLANT IGNITION IN A RADIATION FURNACE

BAER, 1960, REF. (11) UNIVERSITY OF UTAH

SQUARE ROOT OF IGNITION DELAY VERSUS APPLIED HEAT FLUX



JPR 1817

COMPOSITE PROPELLANT IGNITION BY CONVECTION IN A SHOCK TUNNEL

RYAN AND BAER, 1960, REF'S (10) AND (11) UNIVERSITY OF UTAH

IGNITION DELAY VERSUS OXYGEN CONCENTRATION IN TEST GAS



COMPOSITE AND NITRATE ESTER PROPELLANT IGNITION DATA FROM END WALL SHOCK TUBE TESTS

MCALEVY, 1960, REF'S (12) AND (13) PRINCETON UNIVERSITY

IGNITION DELAY VERSUS OXYGEN

MOLE FRACTION IN TEST GAS 5.0 4.0 2.0 t,msec 1.0 0.8 0.6 PROPELLANT KEY 0.4 **D** - P-13 COMPOSITE ▼ - NITRATE ESTER # 1 O - NITRATE ESTER # 2 △ - NITRATE ESTER # 3 0.2 0.2 0.1 0.4 0.6 O.B 1.0 OXYGEN MOLE FRACTION

JP R 1819

FIGURE II

COMPOSITE AND NITRATE ESTER PROPELLANT IGNITION DATA, END WALL SHOCK TUBE TESTS

MCALEVY, 1960, REF'S (12) AND (13) PRINCETON UNIVERSITY

IGNITION DELAY VERSUS OXYGEN CONCENTRATION IN TEST GAS



FIGURE 12

JPR 1820

IGNITION OF COMPOSITE PROPELLANTS BY MEANS OF RADIANT HEAT FLUX

FISHMAN AND BEYER, 1960, REF (14) STANFORD RESEARCH INSTITUTE

PLOT OF MINIMUM EXPOSURE TIME OF PROPELLANT SAMPLES TO A CONSTANT FLUX OF 75 CAL/CM² SEC FOR IGNITION AT VARIOUS PRESSURES VS THE PRESSURE LEVEL



JPR 1821

IGNITION OF COMPOSITE PROPELLANTS BY MEANS OF RADIANT HEAT FLUX

FISHMAN AND BEYER, 1960, REF (14) STANFORD RESEARCH INSTITUTE

PLOT OF MINIMUM TIME OF PROPELLANT EXPOSURE TO RADIANT FLUX NECESSARY FOR IGNITION VS. THE RADIANT FLUX LEVEL USED, PARAMETRIC WITH PRESSURE LEVEL



JPR 1822

IGNITION OF COMPOSITE PROPELLANTS BY MEANS OF RADIANT ENERGY

WISE AND EVANS, 1963, REF. (15) STANFORD RESEARCH INSTITUTE

PLOT OF IGNITION DELAY VERSUS ARC-IMAGE FURNACE PRESSURE LEVEL; CONSTANT RADIANT FLUX OF 70 CAL/CM²SEC



JP R 1824

IGNITION OF COMPOSITE PROPELLANTS BY MEANS OF RADIANT HEAT FLUX

FISHMAN AND BEYER, 1960, REF (14) STANFORD RESEARCH INSTITUTE

PLOT OF MINIMUM EXPOSURE TIME FOR IGNITION OF PROPELLANT SAMPLES EXPOSED TO A CONSTANT FLUX OF 75 CAL/CM² SEC VERSUS GAS COMPOSITION AT ATMOSPHERIC PRESSURE



JPR 1823



IGNITION ROCKET MOTOR EXPERIMENTS ON COMPOSITE PROPELLANTS GRANT, 1963, REF (16) & LANCASTER, 1961, REF, (17) PRINCETON UNIVERSITY

SCHEMATIC DIAGRAM OF IGNITION ROCKET MOTOR

MODIFIED SPARK PLUG

PRESSURE GAUGE





GRANT, 1963, REF (16) & LANCASTER, 1961, REF,(17) PRINCETON UNIVERSITY

IDEALIZED AND INTERPRETED IGNITION ROCKET MOTOR PRESSURE TRACE



ELAPSED TIME

JPR 1826

IGNITION ROCKET MOTOR EXPERIMENTS ON COMPOSITE PROPELLANTS

GRANT, 1963, REF. (16) & LANCASTER, 1961, REF. (17) PRINCETON UNIVERSITY

IGNITION DELAY, T, VERSUS WEIGHT FRACTION OF OXYGEN PRESENT IN COMBUSTION PRODUCTS OBTAINED FROM IGNITER TORCH



JP R 1827

CONVECTIVE IGNITION OF A COMPOSITE PROPELLANT IN A SHOCK TUNNEL

HERMANCE, 1963, REF. (18) PRINCETON UNIVERSITY

IGNITION DELAY VERSUS OXYGEN PERCENTAGE OF AN OXYGEN-NITROGEN TEST GAS MIXTURE; CONSTANT PRESSURE AND TEMPERATURE, SUBSONIC FLOW



JPR 1828

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