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and Combustion of Aluminum**

J. F. Driscoll, J. A. Nicholls, V. Patel,
B. Khatib-Shahidi and Tzu-Chao Liu,
The University of Michigan,
Ann Arbor, MI

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SHOCK TUBE STUDY OF THE IGNITION AND COMBUSTION OF ALUMINUM

J. F. Driscoll, Associate Professor
J. A. Nicholls, Professor*
V. Patel
B. Khatib-Shahidi
Tzu-Chao Liu**

Gas Dynamics Laboratories
Department of Aerospace Engineering
The University of Michigan
Ann Arbor, MI 48109

Abstract

Addition of aluminum to solid rocket propellants can significantly improve performance but may seriously increase plume visibility and radiative heating of the nozzle.

The goal of this project is to obtain data that will help identify the reaction mechanism and the ignition limits of aluminum at elevated temperatures and pressures. Formidable problems arise when attempting such measurements in rocket motors since conditions are unsteady and not easy to control. Therefore, it was decided to mount a pure aluminum sample to the end wall of a shock tube and to ignite the sample using a reflected shock wave. The pressures and temperatures that can be achieved (40 atm, 5000°K) are typical of rocket motor conditions and are much higher than those obtained in previous studies using incident shock waves in conventional shock tubes. The aluminum sample reacts with a test gas in which the proportions of nitrogen, hydrogen, oxygen and chlorine are the same as found in ammonium perchlorate. A single pulse shock tube was used.

Ignition of the aluminum sample occurred for temperatures above 2400K. A number of intermediate species were detected in the $Al-N_2-H_2$ Cl_2-O_2 reaction at 40 atm., 5000K using emission spectroscopy. Species that were conclusively identified were Al , Al^+ , AlO , H , O , O^+ , and N . Other weak emission bands were believed due to AlH , OH , $AlOH$ or $AlCl$. Time histories of the AlO emission band indicate that AlO reaches a maximum before the continuum radiation reaches a maximum, which is an indicator, but not positive proof, that AlO is a precursor to Al_2O_2 in the reaction scheme.

Solid products of combustion were analyzed using a transmission electron microscope, electron diffraction, and X-Ray diffraction techniques. Spherical Al_2O_3 particles were observed in the 0.1 to 1 μ m size range. The major products were Al_2O_3 , HCl , and H_2O as expected, with the latter two products causing appreciable agglomeration of the Al_2O_3 particles. Levels of NO were 10,000 times larger than the predicted concentrations,

* Fellow, AIAA

** Visiting Scientist, Beijing I. A. A., P. R. C.

indicating that significant deviations from the equilibrium NO chemistry occurred.

I. Introduction

The motivation for the present research project has been the need to obtain fundamental data relating to aluminum combustion in a controlled environment in the pressure range up to 40 atm and the temperature range up to 5000 K, typical of rocket motor conditions. Previous work in controlled laboratory experiments has been confined to pressures less than approximately 10 atm, using such devices as flow reactors, burning ribbons, laser heating, and incident shock waves in shock tubes.¹⁻⁷

For example, a fast flow reactor produces a laminar, premixed aluminum vapor/oxygen flat flame which is several centimeters thick and ideal for kinetics studies. Unfortunately, the pressure must be maintained below 0.1 atm, and the resulting premixed flame may not be representative of the diffusion flames that actually occur around burning aluminum particles in rocket motors. Burning ribbons and incident shock wave studies generally have been limited to pressures below 10 atm. Using incident shock waves, aluminum particles are quickly accelerated in a shock tube, and conditions near the moving particle are difficult to ascertain. Convective effects that dominate such processes were not of interest in the present study.

The primary region of interest is the reaction zone that occurs near the surface of a burning aluminum particle, which is shown schematically in Fig. 1. To simulate this reaction zone, it was decided to mount an aluminum sample on the end wall of a shock tube, and subject to a strong reflected shock wave as shown in Fig. 1. This technique offers the following advantages: (a) The aluminum surface is stationary, the reaction zone is large enough (2cm by 2cm) and the test time long enough (10 msec) to allow spectroscopic techniques to be used; (b) Pressures up to 40 atm are easily controlled and measured, and are uniform in the reaction zone; likewise the gas temperature prior to ignition can be measured and controlled up to 5000 K. These pressures and temperatures are considerably higher than used in previous studies, including previous shock tube work.

(c) Gas composition is easily varied; initially pure oxygen was used; later a mixture of .1 N₂ + .4 H₂ + .1 Cl₂ + .4 O₂ was used because it has the same elemental composition as ammonium perchlorate (NH₄ClO₄).

(d) Solid and gas phase combustion products are easily collected and analyzed.

II. Experimental Facility

A. Design and Instrumentation

The shock tube shown schematically in Fig. 2 is designed to allow for single pulse, "tailored" conditions, similar to those achieved by Brabbs⁸ at NASA and Bauer⁹ at Cornell. It consists of a 14 ft long stainless steel driver section (3.5 in. OD, .30 in. wall) and a 20 ft long driven section (3.5 in. x 2.5 in., .30 in. wall). The driver tube has circular cross section in order to withstand driver pressures up to 3000 psi; the driven tube is rectangular so that the windows that are mounted near the end wall are flush with the sidewalls. In this way the windows will not disturb the shock wave near the end wall.

The effective test time available is labeled on the t-x diagram of Fig. 2. When the primary diaphragm is broken, the incident shock reflects off the end wall; meanwhile, expansion waves reflect off the end wall of the tube. This quenches the reaction at the end wall. In addition, the interface between the driver gas (helium) and the driven gas may reflect off the end wall which also quenches the reaction. The secondary diaphragm is broken using an exploding wire at some time after the expansion wave has reflected off of it, in order to prevent further reheating of the sample.

The aluminum sample (2 cm x 2 cm x .1 cm) was mounted on two copper electrodes which formed the end wall of the shock tube, as seen in Fig. 2. Since the test time available is 10 msec, it is imperative that the thermal inertia of the sample be small enough so that the sample surface will reach the temperature of the gas behind the shock wave in less than 1 msec. This was accomplished by preheating the sample to 700 K by passing a DC current through it.

The temperature of the aluminum sample depends on the thermal properties of material on which it is mounted. At time t = 0 a temperature discontinuity at the end wall occurs. After time zero, a thermal boundary layer propagates, having thickness $\delta_g = \sqrt{\alpha_g t}$ where α_g is the thermal diffusivity of the gas. A thermal boundary layer of thickness $\delta_w = \sqrt{\alpha_w t}$ also propagates to the right through the solid wall, which has thermal diffusivity α_w . Equating heat flux from the gas to heat flux to the wall results in a temperature at the gas-wall interface of:

$$T_{Al} = \frac{\frac{k_g}{\sqrt{\alpha_g}} T_g + \frac{k_w}{\sqrt{\alpha_w}} 300 \text{ K}}{\frac{k_g}{\sqrt{\alpha_g}} + \frac{k_w}{\sqrt{\alpha_w}}}$$

Thus, if the aluminum sample is mounted on a good insulator, $k_w/\sqrt{\alpha_w} \ll k_g/\sqrt{\alpha_g}$ and the aluminum temperature will always remain near T_g (i. e., above 4000 K) whereas if the aluminum sample is mounted directly on a metal wall, $k_w/\sqrt{\alpha_w} \gg k_g/\sqrt{\alpha_g}$ and the aluminum temperature will remain near 300 K. The sample in this study was mounted on a Pyrex plate and preheated to 700 K.

The shock tube is instrumented with piezoelectric pressure transducers, pressure gauges, a photographic system, and a spectrometer. An oscilloscope records the pressures P₂ and P₅ which occur behind the incident and the reflected shock. A HP 5314A time interval counter measures the time between pulses output by two transducers two meters apart, so that incident shock velocities and reflected shock velocities are measured. The photographic system consisted of a Fastax 16 mm movie camera, a Polaroid camera for long time exposure photographs and a series of lenses to magnify the test section region.

B. Range of Run Conditions

To measure the run conditions, the end wall pressures P₂ and P₅ behind the incident shock and the reflected shock were measured. The incident and reflected shock speeds u_i and u_r were measured using pressure transducers mounted two meters apart. Also, the driver gas pressure P₄ was measured.

To determine gas temperatures and composition just prior to ignition, the NASA Equilibrium Gas Composition Program developed by Gordon and McBride was used. This computer program accounts for real gas effects (dissociation and reactions of the test gases---N₂, O₂, H₂, Cl₂) and predicts the gas conditions behind the reflected shock wave. The typical conditions achievable are shown in Fig. 3.

III. Results

A. Ignition of Aluminum in Oxygen

The ignition and combustion of the aluminum sample in a pure oxygen environment was studied first. A typical pressure history of the gas adjacent to the aluminum sample is shown in Fig. 4a. Run conditions for this trace were: Helium driver pressure = 400 psi; oxygen initial pressure = 20 mm Hg; incident shock strength = Mach 7; pressure behind reflected shock = 13 atm; temperature behind reflected wave = 4100 K. The

pressure trace shown in Fig. 4b was obtained using a pressure transducer mounted two meters from the end wall. It clearly shows the gas pressure rising to a value P_2 behind the incident shock and later to a value P_5 behind the reflected shock

Figure 5 shows the ignition of an aluminum sample. The incident shock has traveled from the right to the left and has reflected back from the left to the right. Some hot particles, either Al or Al_2O_3 are seen to travel to the right, away from the reaction zone. These particles may be emitted due to boiling of the aluminum at the sampling surface, caused by gaseous impurities in the aluminum, as proposed by Glassman.⁶ Aluminum and condensed Al_2O_3 drops or particles contribute black body radiation which interferes with spectroscopic measurements.

Low temperature runs in the range $T_5 = 1600 - 3200$ K were made to determine the ignition temperature of aluminum. Data shown in Fig. 6 shows that when the sample is exposed to pure oxygen at temperatures below 2000 K, no ignition was observed. When the oxygen temperature was above 2400 K, ignition occurred on every run. For oxygen temperatures between 2000 and 2400 K, the small particles emitted from the sample emitted bluish-white light, indicating that they ignited. Therefore it is believed that no unique ignition temperature exists, although ignition does occur in the range 2000 - 2400 K. The ignition process is controlled primarily by the melting of the oxide layer, which is generally less than one micron thick and has a melting temperature of 2300 K. As the oxide layer reaches the melting point, any aerodynamic stripping of this oxide layer would aid in the ignition process, and may be the reason why the smaller moving particles are observed to ignite when the large sample does not.

B. Reaction of Aluminum in H_2, O_2, Cl_2, N_2

For the typical run conditions of a Mach 7.8 incident shock wave in a gas mixture of 40% H_2 , 40% O_2 , 10% Cl_2 and 10% N_2 , which corresponds to the elemental composition of ammonium perchlorate, the calculated equilibrium gas conditions are given in Fig. 7. An equilibrium code was run to get some idea of the species that exist during the reaction, and those that later appear after the reaction is quenched to room temperature. The calculations are useful as a guide to indicate which spectral lines to look for in the emission spectrum. Thirty-nine species were determined to have mole fractions above 0.5×10^{-5} . In addition, 41 other species were considered in the calculations but their mole fractions were less than 0.5×10^{-5} . The final products are seen in Fig. 7 to be mostly H_2O liquid, HCl , alumina, N_2 , and ammonia, with traces of H_2O vapor.

The computed chemical equilibrium product of the NH_4ClO_4-Al reaction was compared with the products of the $.1 N_2 + .4 H_2 + .1 Cl_2 + .4 O_2 +$

Al reaction in order to assess how closely the shock tube conditions compare with actual rocket motor conditions. As seen in Fig. 8, both reactions have the same intermediate species in similar rocket motor conditions therefore is justified. The effect of increasing the aluminum content of an ammonium perchlorate-aluminum propellant on the resulting flame temperature is shown in Fig. 9.

C. Spectrographic Results

Three problems arose while measuring the emission spectrum of the aluminum reaction. First, the solid aluminum and alumina particulates gave off strong black body continuum radiation that obscured the discrete bands emitted by gaseous species.¹¹⁻¹⁷ Secondly, the AlO bands were so intense that they too obscured other bands. Thirdly, many of the minor species occur in low concentrations and have low intensity spectral bands.

Initially, the emission spectrum of the aluminum-oxygen reaction was recorded. A typical emission spectrum is shown in Fig. 10. Spectral lines associated with Al and Al^+ are clearly visible, and four bands corresponding to AlO are seen. No spectral bands associated with species Al_2O_2 or AlO_2 could be identified. It is suspected that the emission spectrum emitted by these species lies in the ultra violet region that cannot be recorded using the windows and film of the present experiment.

The emission spectrum of the Al- N_2 - H_2 - O_2 - Cl_2 reaction was recorded. It is difficult to associate the bands with the various species because many of the bands overlap each other. Therefore, runs were made in which various species were removed from the reactant gas mixture in a systematic way. Spectra were recorded for the Al- H_2 , Al- Cl_2 , Al- O_2 , Al- H_2 - O_2 , Al- Cl_2 - H_2 and Al- N_2 - H_2 - Cl_2 reactions. AlO, Al, Al^+ , H, N, O, O^+ , and N^+ have been positively identified. There is some evidence that observed bands belong to OH and AlH also, but the bands were too weak to be positively identified. Other weak bands could not be positively identified, but most probably were associated with AlOH, AlCl, and AlCl₂. A strong continuum also was measured; this continuum has been reported in previous studies¹⁸ and is believed to be associated with Al_2O_2 .

D. Chemical Kinetics

The time history of the AlO emission band at 4846 Å was recorded using a polychromator and photomultiplier tubes. The background continuum at 4500 Å also was recorded. Results are shown in Fig. 11. The intensity of the AlO band peaks at 1.4 msec after ignition; at 2 msec after ignition no more AlO was observed, only background continuum. The background continuum first occurs at approximately 2 msec after ignition and exists for the entire test period of 12 msec in this case.

King¹⁸ has postulated that the background continuum observed by a number of researchers may be due to Al_2O_2 . If this is so, then the results shown in Fig. 11 indicate that AlO is a precursor to Al_2O_2 , which would verify that the mechanism observed by Mann¹ at low pressures also occurs at the 40 atm test conditions of this study. However, additional spectroscopic work is needed to conclusively determine if the background truly represents the presence of Al_2O_2 .

It is felt that much information concerning the $Al-O_2$ reaction and the $Al-N_2-H_2-Cl_2-O_2$ reaction can be obtained by measuring the spatial profiles of the observed emission bands. To do so, an optical multichannel analyzer is needed. To measure intermediate species concentrations, laser fluorescence diagnostics have proven useful in similar studies. For example, fluorescence technique could prove useful in helping to interpret the emission spectroscopy results since the intensity of AlO bands may partly be due to chemiluminescence and may not be an accurate measure of AlO concentration.

E. Solid and Gaseous Combustion Products

The solid and the gaseous products of combustion were collected after shock tube runs and were analyzed using: (a) scanning electron microscope, (b) transmission electron microscope, (c) electron diffraction technique, (d) X-ray diffraction technique and (e) infrared gas analysis.

After the aluminum sample was burned in pure oxygen, the white powder that formed a thin layer on the inside of the shock tube walls was collected and verified to be aluminum oxide (Al_2O_3). To collect the Al_2O_3 , a carbon block was inserted in the electrode flush to the wall. After the run, the carbon block, on which the visible Al_2O_3 powder had settled, was taken to the electron microscope laboratory. Carbon was chosen as the mounting material because it is transparent to the electron microscope.

A transmission electron micrograph of the Al_2O_3 particulates is shown in Fig. 12. The particles are seen to be spherical and in the size range from 0.1 to 0.6 μm .

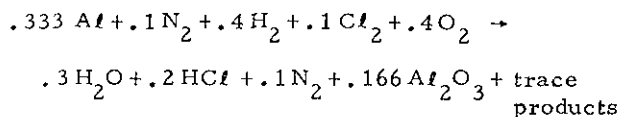
When the aluminum sample was burned in the shock tube with the gas mixture $.1 N_2 + .4 H_2 + .1 Cl_2 + .4 O_2$, the products of combustion were observed to be solid particles suspended in a green colored liquid. The liquid was determined to be H_2O and HCl and the solid particles were Al_2O_3 , with some unburned Al . No traces of other possible solids, such as NH_4Cl or NH_4N_3 were detected, probably because all the available hydrogen combined with the excess O_2 to form H_2O . The solid product $AlCl$ was not detected; it is deduced that the aluminum atoms combined with the excess O_2 to form Al_2O_3 rather than combine

with chlorine. The result of the analysis of solid and liquid combustion products was that the only major products are Al_2O_3 , HCl and H_2O , which is consistent with results of rocket motor sampling, such as done by Strand et al.¹⁹

Various minor species were detected in the solid products of combustion. An electron micrograph of the solid products is shown in Fig. 13. It is seen that the presence of condensed H_2O and HCl causes the Al_2O_3 to agglomerate into long strands, bearing no resemblance to the spherical particle from the $Al-O_2$ reaction which were shown in Fig. 12. The regions that appear grey in Fig. 13 are Al_2O_3 and H_2O ; the regions that appear white contain chlorine, as deduced from quantitative X-Ray analysis. The X-Ray analysis indicates elemental components only and, as seen in Fig. 13, verifies the presence of aluminum and chlorine in large quantities. Trace amounts of copper, iron and silicon are detected, which were contributed by the copper electrodes, the steel diaphragm and the glass windows, respectively.

From the above results, it is concluded that HCl and H_2O have major effects on the agglomeration of Al_2O_3 . Future work in the shock tube facility would be necessary to systematically study the effects of HCl on the nucleation of Al_2O_3 and the resulting particle sizes. It is known that HCl in rocket plumes acts as nucleation sites for H_2O condensation and thereby enhances contrail visibility. The shock tube provides ideal controlled conditions for studying how chemical species affect Al_2O_3 condensation.

The results of the analysis of gaseous products of combustion for the $Al-N_2-H_2-Cl_2-O_2$ reaction are shown in Fig. 14. An evacuated sample bag was filled with the gaseous products after a run. The sample was analyzed using infrared gas absorption analysis. An estimate of the final H_2O and HCl concentrations could be inferred from an overall species balance:



For the shock tube run conditions, .025 moles of the test gas and .008 moles (.21 grams) of aluminum were consumed.

An unexpected result was the large fraction of nitric oxide (NO) and nitrous oxide NO_2 that was measured in the gaseous products. The NO and NO_2 bands are clearly prominent in Fig. 14; the measured level of NO and NO_2 were 0.4% and 0.06% of the products. These levels are at least three orders of magnitude larger than the predicted equilibrium results, some of which are shown in Fig. 7. It is believed that the unexpectedly high

levels of NO and NO₂ are a result of the "prompt NO" mechanism that is well documented in hydrocarbon-air flames.

IV. Conclusions

1. The chemical kinetic shock tube provided controllable combustion of aluminum at conditions that simulate the surface of a burning aluminum particle in a rocket motor. Temperatures and pressures achievable (5000 K, 40 atm) were higher than for any previous controlled laboratory study of aluminum combustion, and were realistic of rocket motor conditions.
2. No distinct ignition temperature for aluminum was observed; small particles of aluminum ejected from the sample ignited at 2000 K while the entire sample always ignited at temperatures above 2400 K. Ignition occurred within a ± 100 K range about the Al₂O₃ layer melting point, which is 2319 K.
3. Intermediate species detected in the Al-O₂ reaction by emission spectroscopy were: Al, Al⁺, and AlO. Intermediate species detected in the Al-N₂-H₂-Cl₂-O₂ reaction at 40 atm, 5000 K were: Al, Al⁺, AlO, H, O, O⁺, N. Weak bands that could not be conclusively identified were believed to be due to AlH, OH, AlOH, and AlCl. A strong continuum was measured which has been associated with Al₂O₂ in other studies.
4. No emission bands were found that could be identified with intermediate species Al₂O or AlO₂. Additional diagnostic techniques such as fluorescence or Raman scattering are needed to detect these species.
5. Time histories of the AlO band at 4846 Å and the continuum at 4500 Å indicate that the AlO emission reaches a maximum before the continuum reaches a maximum. If the continuum can be associated with Al₂O₂, this would verify that AlO is a precursor to Al₂O₂ in the reaction mechanism.
6. Spherical Al₂O₃ particles were formed in the size range .1 - 1.0 microns when aluminum was burned in pure oxygen. When aluminum was burned in the presence of N₂, H₂, Cl₂, and O₂, extensive agglomeration of the Al₂O₃ particles was observed; the major products were Al₂O₃, HCl, and H₂O only. The solid products were analyzed using a transmission electron microscope, and X-Ray and an electron diffraction method.

V. ACKNOWLEDGEMENT

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VI. References

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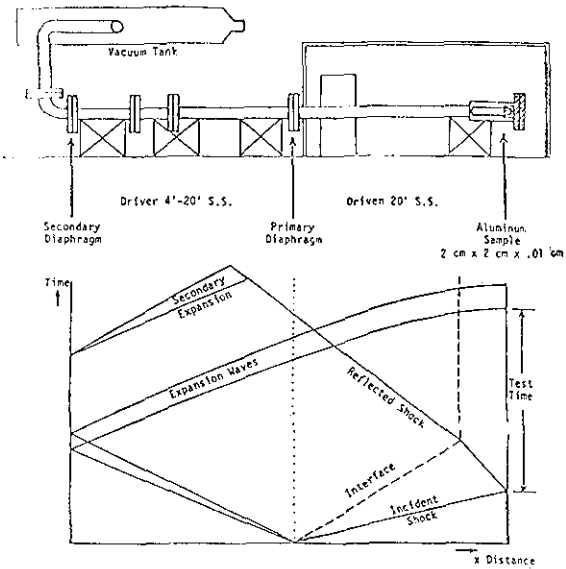


Figure 2. Schematic of Chemical Kinetic Shock Tube

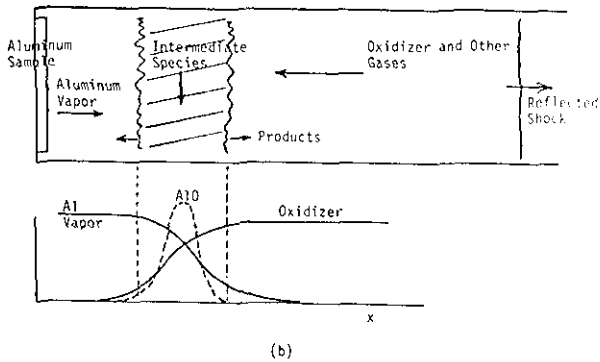
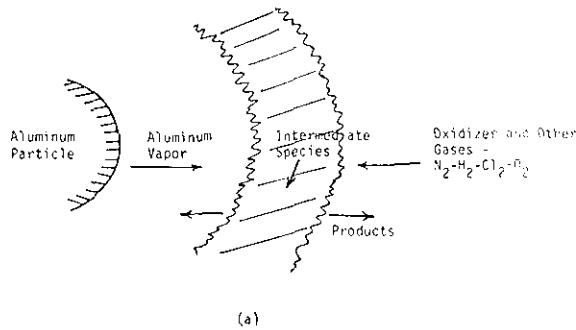


Figure 1. Schematic of the Reaction Zone Near (a) An Aluminum Particle in a Rocket Motor and (b) An Aluminum Sample in a Shock Tube

Typical Run Conditions for Al-O₂ Reaction

Conditions behind reflected shock: 4000 K, 17 atm
 Incident shock: M = 5
 Initial oxygen pressure: 1.16 psi
 Driver pressure: 460 psi helium

Typical Run Conditions for Al + .1 N₂ + .1 Cf₂ + .4 H₂ + .4 O₂ Reaction

Conditions behind reflected shock: 4149 K, 14.3 atm
 Incident shock: M = 7.8
 Initial gas pressure: .386 psi
 Driver gas pressure: 400 psi helium
 Molecular weight of mixture: 25.67 g/mole
 Ratio of specific heats: 1.1179

Gas Composition Prior to Aluminum Ignition (Mole Fraction)

	Percent		Percent
O	17.405	Cl ₂	9.847
OH	14.249	HCl	7.036
H	13.397	N ₂	7.008
O ₂	11.400	H ₂	6.208
H ₂ O	10.392	NO	2.936

Maximum P, T Run Conditions for Al + .1 N₂ + .1 Cf₂ + .4 H₂ + .4 O₂

Conditions behind reflected shock: 5150 K, 40 atm
 Incident shock: M = 10.5
 Initial gas pressure: .386 psi
 Driver gas pressure: 1500 psi

Figure 3 Typical Shock Tube Operating Conditions

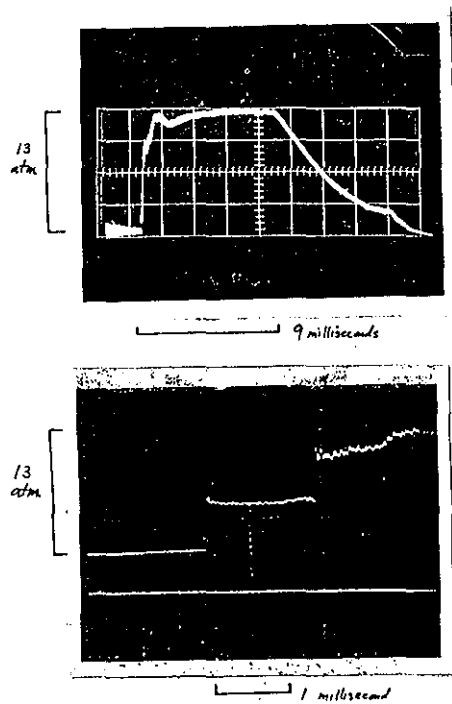


Figure 4 Oscilloscope traces showing (a) pressure history at end wall (b) pressure history two meters from end wall.

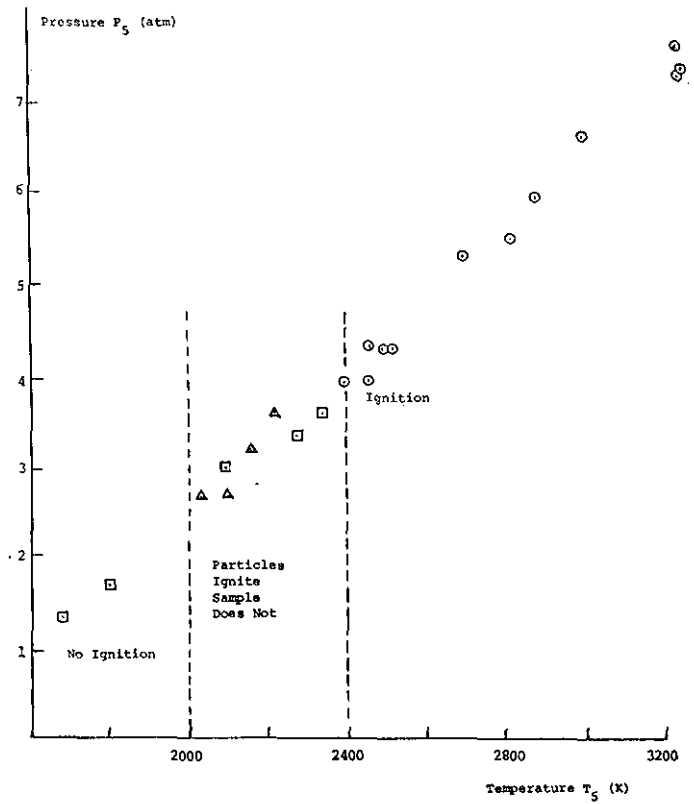


Figure 6 Aluminum ignition data for various pressure and temperatures in oxygen. \circ Ignition observed, \square No ignition observed, Δ Particles only ignite.

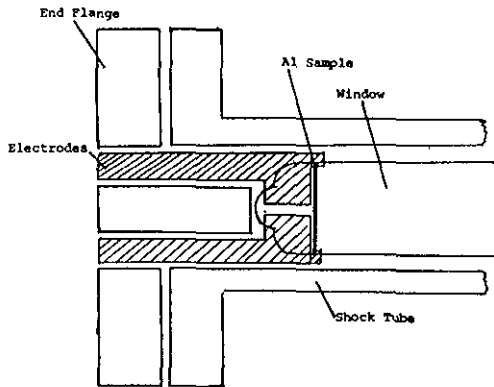
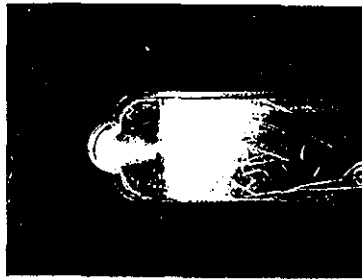


Figure 5 Photograph of the ignition of the aluminum sample mounted on shock tube end wall.

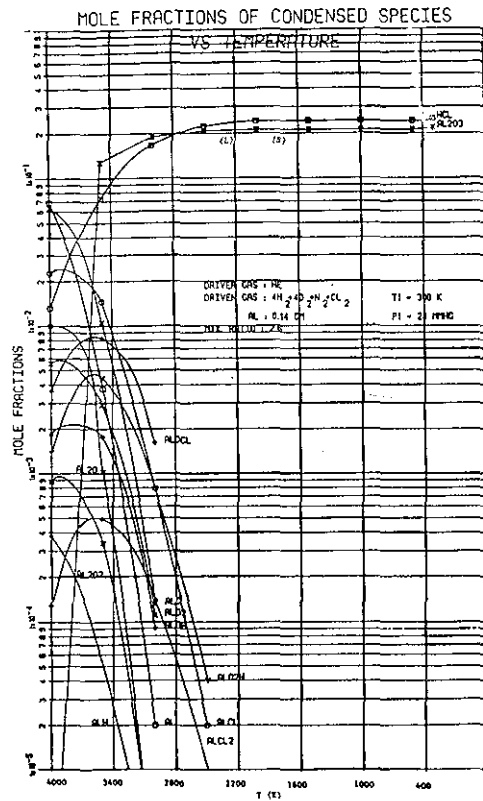


Figure 7 Variation of Reaction Products as Temperature Decreases to 300 K

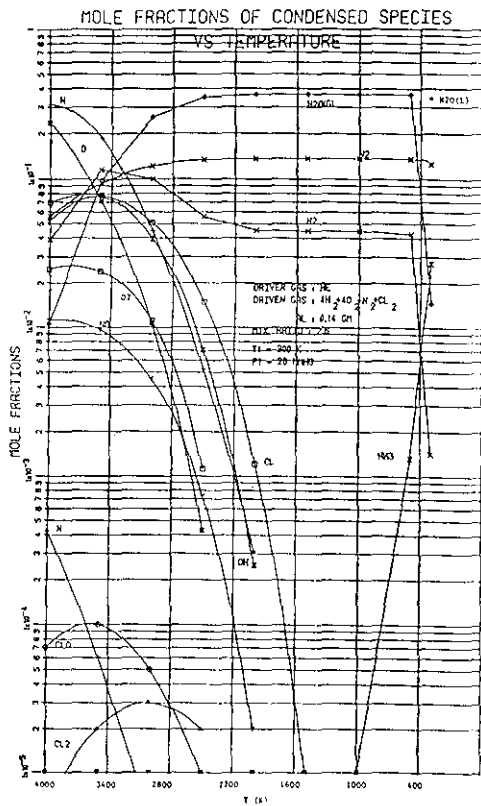


Figure 7 concluded

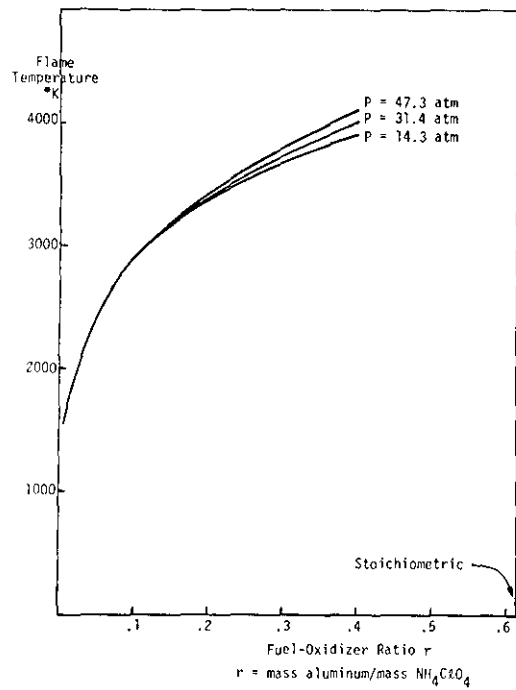


Figure 9 Aluminum- NH_4ClO_4 Flame Temperatures Calculated Using NASA Equilibrium Code

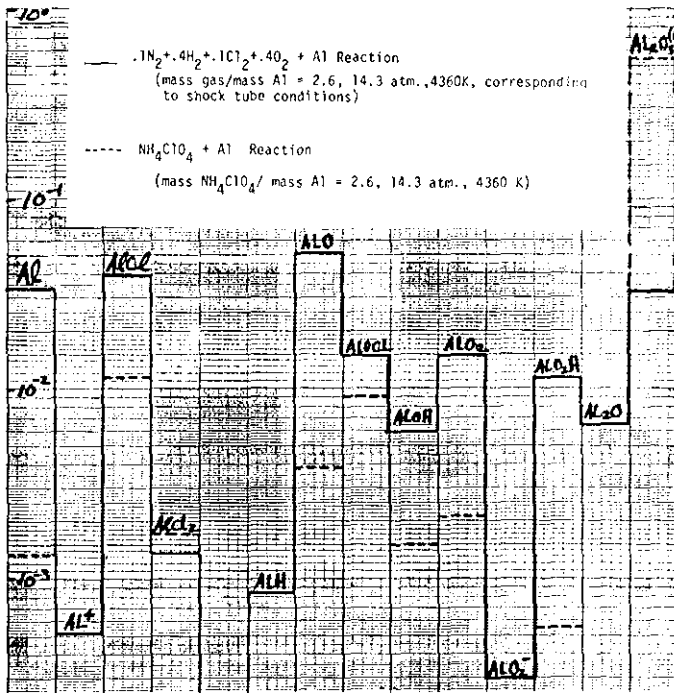
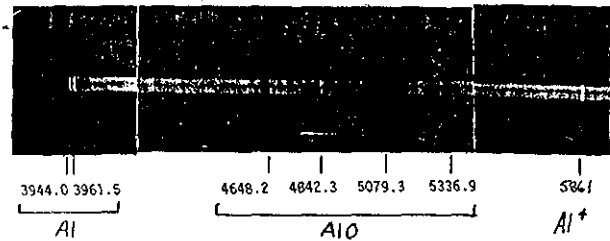
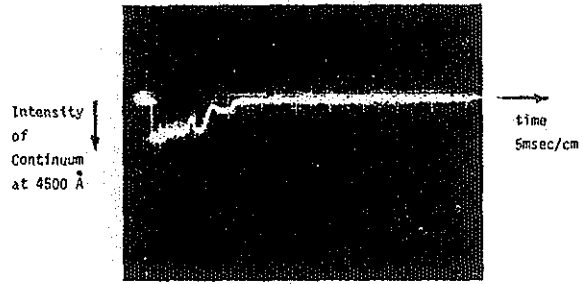
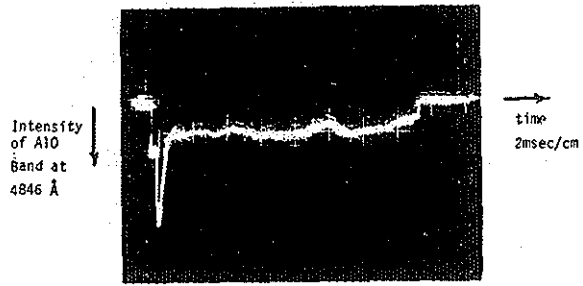


Figure 8 Comparison of Computed Equilibrium Products of the $\text{NH}_4\text{ClO}_4 - \text{Al}$ Reaction with those for the $.1\text{N}_2 + .4\text{H}_2 + .1\text{Cl}_2 + .4\text{O}_2$ Reaction

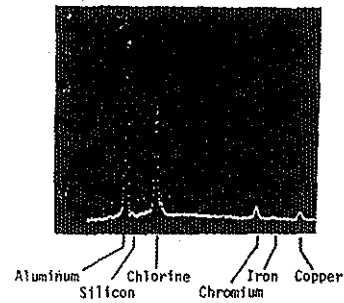


$P_5 = 7.1 \text{ atm}$
 $T_5 = 2700 \text{ K}$
 Exposure time = 8 msec

Figure 10. Emission spectrum of aluminum — oxygen reaction in the shock tube.



(a) Electron Micrograph (600X)
white area = $Al_2O_3 + HCl$
grey area = $Al_2O_3 + H_2O$



(b) X-Ray Analysis

Figure 11 Time History of Emission Spectra

Figure 13 Electron Micrograph and X-Ray Diffraction Analysis of Products of $Al + H_2 + H_2 + Cl_2 + O_2$ Reaction

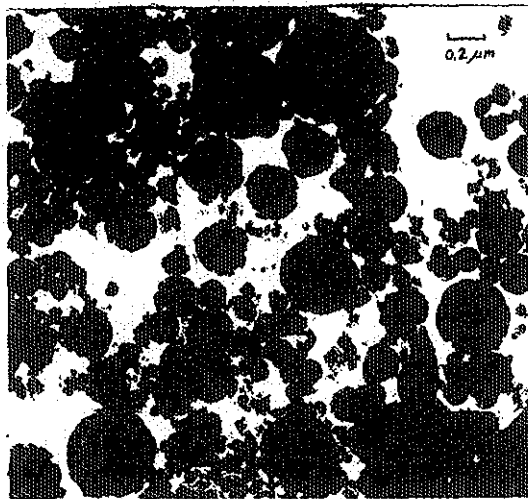


Figure 12 Transmission electron micrograph of Al_2O_3 formed in shock tube.

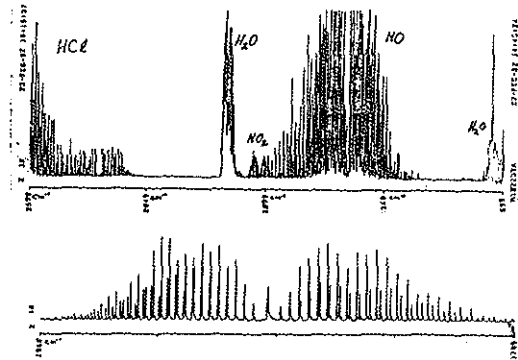


Figure 14 Infrared Spectrometer Analysis of Gaseous Products of Combustion