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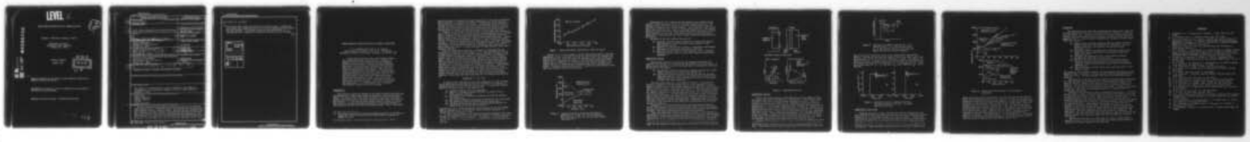

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Technical Report
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Liquid carbon disulfide (CS_2) was shocked by reflection to pressures where reaction occurs. Progress of shocks and reaction was monitored with electromagnetic particle velocity probes. Particle velocity waveforms show that a two-wave structure evolves in the reflected shock wave at pressures above about 75 kbar, with the character depending upon the pressure. Two wave evolution is accompanied by an induction time, followed simultaneously by particle velocity decay and a burst of decaying electrical noise; the

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noise decay takes longer than the particle velocity decay. Equation of state models were developed and used to calculate temperatures both before and after reaction. From these calculations and observations, it is hypothesized that a decomposition chemical reaction occurs in the shock.

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Liquid carbon disulfide (CS_2) was shocked by reflection to pressures where reaction occurs. Progress of shocks and reaction was monitored with electromagnetic particle velocity probes. Particle velocity waveforms show that a two wave structure evolves in the reflected shock wave at pressures above about 75 kbar, with the character depending upon the pressure. Two wave evolution is accompanied by an induction time, followed simultaneously by particle velocity decay and a burst of decaying electrical noise; the noise decay takes longer than the particle velocity decay. Equation of state models were developed and used to calculate temperatures both before and after reaction. From these calculations and observations, it is hypothesized that a decomposition chemical reaction occurs in the shock.

INTRODUCTION

The utility of shock-wave methods in the study of phase transitions was established early through both dynamic measurements[1] and recovery experiments.[2] Yet their value in the study of chemical reactions in condensed matter has not been widely recognized, despite the role they have played in gas reactions.[3,4] Most work on shock-induced chemical changes in liquids and solids has involved post-shock examination of recovered specimens,[5-7] where pressure and time histories are but poorly known. Part of the reason

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that shock-dynamic techniques have been little used in the study of condensed matter reactions is probably the limited information that can be obtained from measurement of macroscopic variables. If parameters being measured are limited to pressure, density, particle velocity and shock velocity, there is no sure way to distinguish between phase transitions and chemical reactions. Consideration of related information can help in making the distinction.

Liquids are more likely than most solids to react under shock because of their greater compressibility, which leads to high temperatures in the shocked state. A number of liquids have been studied and considerable effort has been directed toward the subject of liquid-to-solid phase transitions. Shock-induced changes have been reported in opacity[8-10] and in electrical conductivity;[11-17] anomalous temperatures have been measured,[10] and cusps have been reported in the (p,v) Hugoniot curves of a number of liquids.[18-20] Discontinuous changes in macroscopic properties have frequently been interpreted as phase transitions; but it is likely that many of these anomalies result from chemical reactions.

Explicit reference to reactions, particularly to decomposition and pyrolysis, has been made in some cases.[17,20-22] The most complete study of a shock-induced reaction which has been reported to date is a lengthy study on acrylonitrile by Yakushev, Nabatov, and Yakusheva[22] in which they observed a cusp in the Hugoniot at 43 kbar, based on shock velocity measurements. They also used an electromagnetic particle velocity gage and measured what appears to be a two-wave structure but they said the scatter in the data was so great that it was not possible to "establish unambiguously the nature of the second discontinuity in the mass velocity." Among several other tests, recovery experiments were done where the acrylonitrile was shocked to 240 kbar and then recovered. On examining the sample, they discovered the evolution of large amounts of ammonia, with a black velvet powder remaining, which was identified by x-ray diffraction as hexagonal graphite. Using these data they speculated that the decomposition reaction is

$$CH_2{=}CH\text{-}CN \longrightarrow 3C + NH_3.$$

A review of this and other earlier work suggested to us that an effort to use shock-dynamic techniques to unravel the kinetics of a reaction in condensed matter might be profitable at this time. In screening candidate materials for such a study, it was required that

i) anomalous behavior should be documented
ii) there be a reasonable probability that the anomaly results from a chemical reaction
iii) the material be of interest to chemists and that the molecule be a simple one
iv) the ordinary chemical and physical properties be well established
v) the material be readily available and not highly toxic
vi) the shock pressures required to produce anomalous behavior be within reach of the WSU gas gun facility.

Acrylonitrile was a serious candidate, largely because of the work already done by Yakushev et al.,[22] but CS_2 was finally selected because of its simpler molecular structure: $S=C=S$.

R. Dick, using explosives, has measured the Rankine-Hugoniot (p,v) curve for CS_2 and determined that a cusp exists at 62 kbar.[20] A graph of his data in the U_s-U_p plane is shown in Figure 1. The existence of a cusp implies that the end product of the reaction or transition has a higher density than the CS_2.

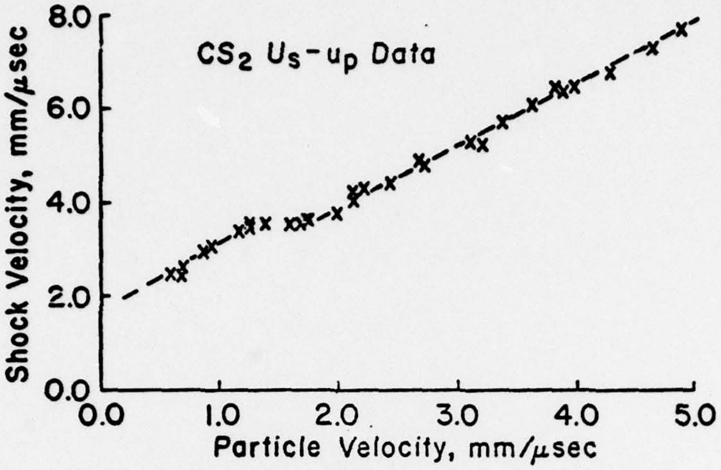


Figure 1. Shock measurements reported by R. Dick for CS_2.[20]

Butcher et al.[23] did a number of static high pressure tests at elevated temperatures on CS_2 to produce and study the "black solid CS_2" Bridgman had discovered earlier. From their data and Bridgman's data, they constructed a diagram that is reproduced in Figure 2. They indicate that a black solid form of CS_2 is formed only in the relatively small region indicated in the figure and that, at temperatures and pressures above this, decomposition occurs. The region marked "decomposition" is where the shock results of Dick (with the reaction occurring) are located.

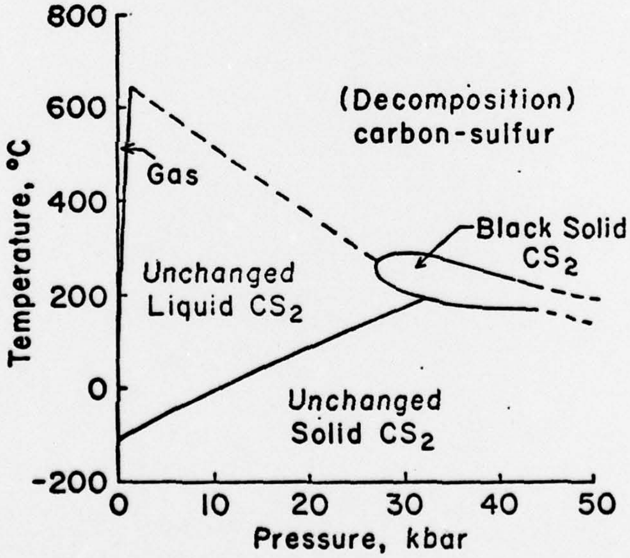


Figure 2. Temperature-pressure diagram constructed by Butcher et al. in Ref. 23. The cusp observed by R. Dick occurs well up into the region marked "Decomposition."

Afanasenkov et al.[21] assumed that the Hugoniot cusp reported by Dick resulted from decomposition of CS_2 to elemental carbon and sulfur. They combined carbon and sulfur Hugoniots, using the stoichiometric ratio, to calculate a Hugoniot curve for the products. This curve went right through Dick's data above the cusp. They also measured temperature by a brightness technique and found it to be 3000°K, about 500°K higher than their calculations indicated it should be. From these results and the Butcher et al. measurements, they concluded that their initial assumption of decomposition was correct.

The goals of the work reported here were to

(1) establish an experimental procedure for shocking the material into the region of reaction which can be adapted to later spectroscopic studies
(2) establish by pressure and velocity, or equivalent, measurements the range of interesting experimental conditions
(3) establish overall features of the reaction, such as induction time and reaction rates, as they affect observable macroscopic parameters.

EXPERIMENTAL DESIGN

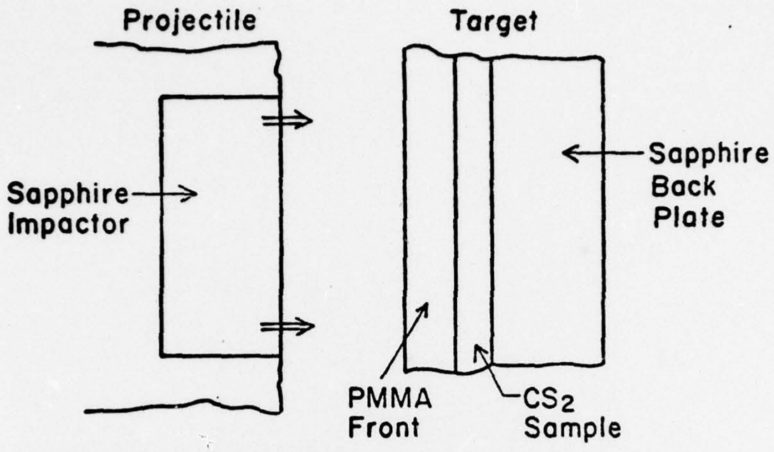
The particle velocities at which the CS_2 undergoes transition are typically 1.35 mm/μsec for a single shock and greater for a double shock. A double shock experiment, in which the second shock is produced by reflection, was chosen for two reasons.

(1) The particle velocity required for reaction produced by a single shock is barely within reach of the WSU gas gun.
(2) A reflection experiment gives finer control over the reacting state and opens the possibility for generating and monitoring a two-wave structure, which, according to R. Dick, is not present in the single shock experiment.

The experimental design is illustrated in Figure 3. The target was placed in a magnetic field during the shots so it was necessary to use a sapphire impactor when a high impedance material was required. A sapphire faced projectile drives a shock into the PMMA cell front, which then traverses the CS_2 as a relatively low pressure wave (state 1). When the shock reaches the sapphire back plate, a high pressure wave (state 3') is driven back into the already shocked CS_2. The experiment can be designed so the high pressure wave is above the transition pressure and a reaction is observed. In this case the reflected wave splits into two waves, producing state 2 behind the first wave and state 3 behind the second wave. References made to a particular state in the remainder of this paper will be consistent with the designations of Figure 3c.

Progress of the shocks and the reaction were measured by imbedding several electromagnetic particle velocity (EMV) probes in the liquid sample so that both particle velocity and shock arrival times could be continuously recorded. From these records velocity can also be determined.

Since the experiments involve reshocking of the CS_2 it is important to understand that the reshock Hugoniot is different from the principal Hugoniot, how different depends upon the compressibility of the material. CS_2 is quite compressible, so it is necessary to take into consideration the difference. Both the reshock and principal Hugoniots lie on the equilibrium equation of state surface for unreacted CS_2 so the approach taken has been to develop a model for the equation of state and use it to calculate the reshock Hugoniot.



(a) Cross section of experiment

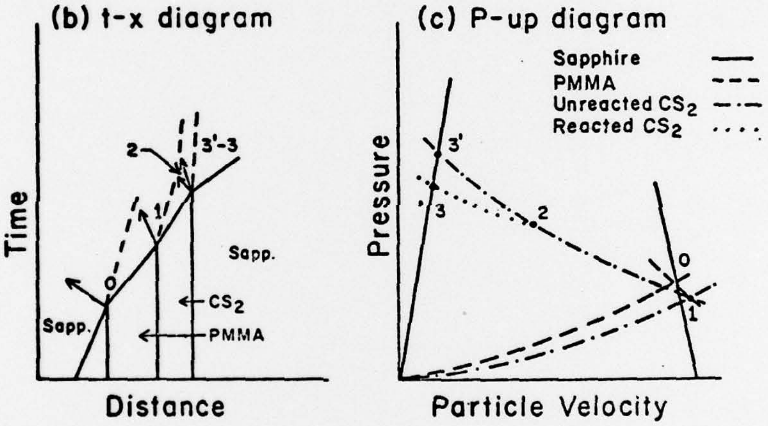


Figure 3. Experimental design.

EXPERIMENTAL RESULTS

Of all the records obtained, those obtained at the CS_2-sapphire interface reveal most clearly the features of the reaction. An idealized record is shown in Figure 4; it displays three interesting features. First is a region of uniform particle velocity, AB, immediately following reflection. Its duration, t_I in the Figure, depends on shock pressure and temperature in unreacted CS_2; it is called an "induction time." Second, BC, is decay of the average particle velocity to a final uniform state, lower than the induction state by an amount Δu_p. There is also a characteristic time, t_{u_p}, associated with this change. Finally, and quite unexpectedly, is a coherent electrical signal which begins suddenly at the time particle velocity starts to decay, B, and decays in amplitude to D with a characteristic time, $t_N > t_{u_p}$, which also depends upon pressure and temperature in the induction state.

Because of the electrical signal it was impossible to determine the exact nature of the particle velocity decay. Data for this decay were estimated by drawing a curve through the signal at what appeared to be the mean position. Signal decay time was also estimated but it was difficult to tell the

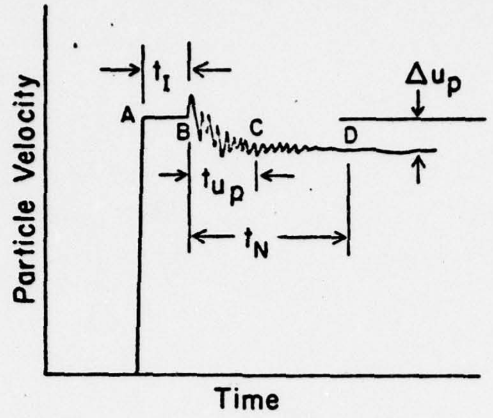


Figure 4. Idealized CS_2-sapphire gage record. t_I is induction time, t_N is the noise decay time, t_{u_p} is the particle velocity decay time, and Δu_p is the particle velocity decrease.

end of the signal from normal noise on the approximately 100 millivolt signals. For many of the shots the induction time was so small that it was not resolvable in the 10 to 20 nsec risetime of the gage. Two records are shown in Figure 5. Frequency of the oscillatory signal is not periodic, but its average frequency varies from about 40 to 150 MHz among records. Induction times range from < 6 to ∿310 nsec; t_{u_p} ∿ 50 to 400 nsec; t_N ∿ 110 to 340 nsec.

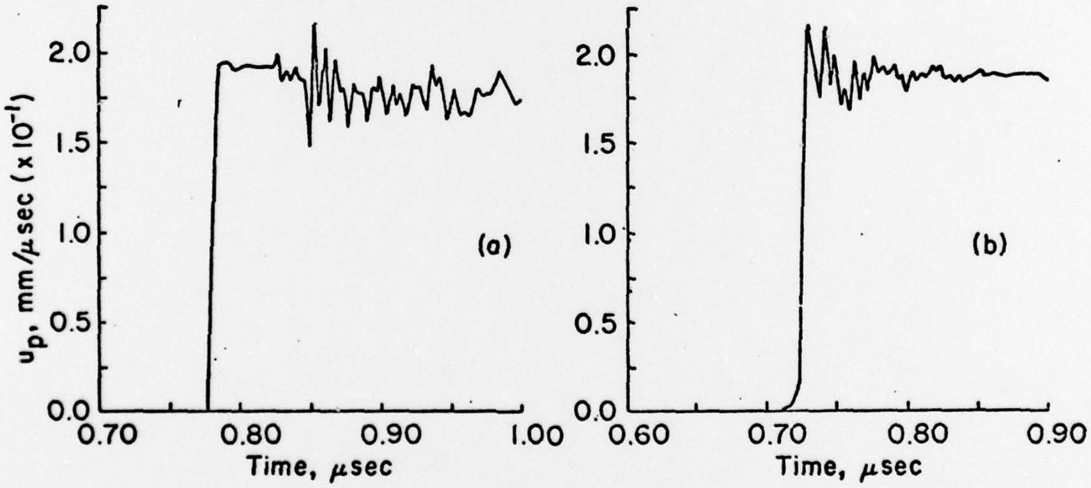


Figure 5. Records from the CS_2-sapphire interface. (a) shock pressure is 87 kbar; (b) shock pressure is 106 kbar.

TEMPERATURE CALCULATIONS

Complete equations of state, suitable for numerical computation, have been constructed from shock and static data for CS_2 and for reaction products, assumed to be a stoichiometric mixture of carbon and sulfur. Temperatures and pressures calculated for the principal Hugoniot centered at room temperature and for reshock Hugoniots centered at various points on the principal Hugoniot, all for unreacted CS_2 are shown in Figure 6a. States lying on the CS_2 curve at point 3' in Figure 3 are constrained to lie on the dashed line in Figure 6a. These represent metastable points in the CS_2 in the reacting

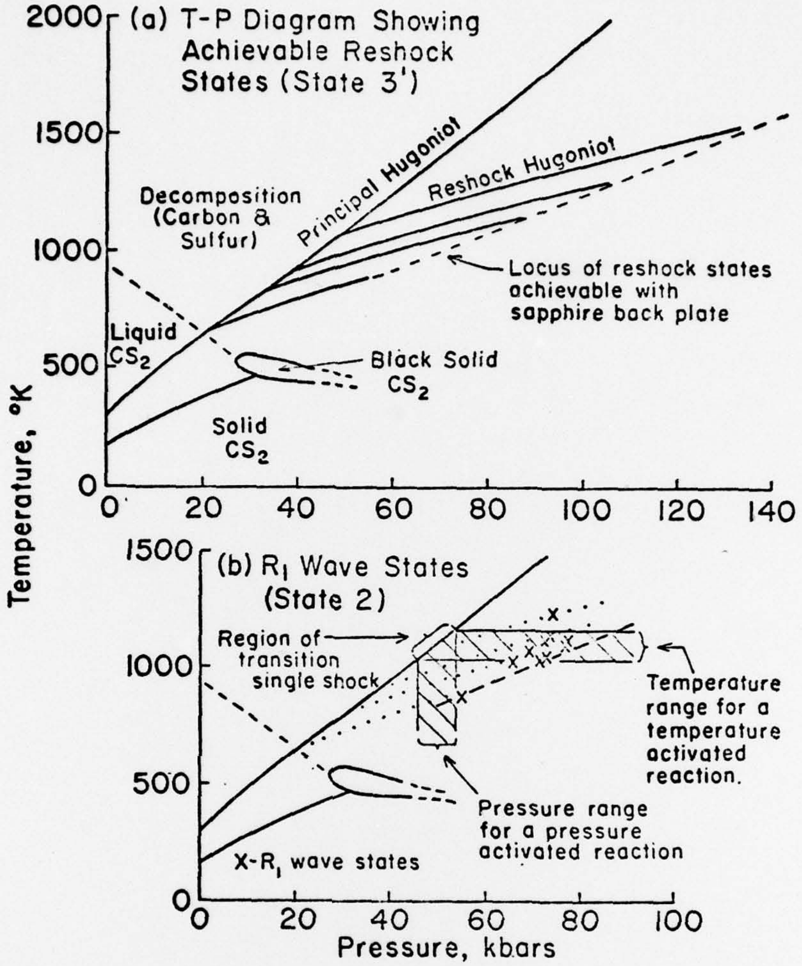


Figure 6. Temperatures and pressures in the reflected shock wave.

region but before reaction has started. In Figure 6b are plotted measured points (X) corresponding to point 2 of Figure 3. The band of pressures and temperatures indicated on the principal Hugoniot in Figure 6b represents what we believe is the region of onset of reaction in a single shock. These are somewhat lower than R. Dick's estimate of 62 kbar. If the reaction were activated solely by pressure, measured points "2" would lie in the vertical cross-hatched band. If the reaction were temperature-activated, they would lie in the horizontal band. Actually they do neither, and this is quite reasonable since activation energy can reasonably be expected to be a function of pressure. The phase diagram given by Butcher et al. is shown in Figure 6 for comparison with measured states. The measured states are well into the region of decomposition indicated by Butcher et al.

DISCUSSION

The measurements that have been made demonstrate clearly that liquid CS_2, on being shocked to sufficiently high temperature and pressure, is decaying to a more dense state. One must be cautious in leaping to conclusions about internal mechanisms responsible for the decay, but there are some grounds for supposing that it is due to chemical decomposition instead of a phase change. These are, briefly,

(1) Velocity-time histories recorded at the CS_2-sapphire interface differ in form from those expected for transition. The most notable differences are induction time and the reaction electrical signal.
(2) Calculated pressures and densities of the final states are in accord with those expected from a stoichiometric mixture of carbon and sulfur.
(3) Pressures and temperatures fall in the region of chemical decomposition described by Butcher et al.
(4) The array of final pressure and temperature states shown in Figure 6b appears to be more nearly in accord with characteristics of a temperature-dependent process than with a pressure dependent one.
(5) CS_2 is chemically unstable relative to carbon and sulfur in the reaction region.

The weight of this evidence is grounds for the tentative conclusion that the observed effect is chemical in origin, probably representing decomposition to carbon and sulfur. Absolute assurance that this is so awaits more direct evidence.

The character of particle velocity records at the CS_2-sapphire interface, viz. the existence of an induction time followed by decay of pressure and particle velocity, suggests that there may be two principal stages in the reaction. The initial reaction proceeds without significantly affecting pressure, and the second is associated with a decrease in pressure and increase in density. Here, too, a more detailed analysis is required before firm conclusions are possible.

Presence of the electrical signal is unexpected and puzzling. Its almost-periodic nature implies a coherent motion of electrified particles, which may be induced by the magnetic field of 2000 gauss used for the EMV gauge. If the average frequency of the signal is taken to be 100 MHz and the signal is assumed to represent the cyclotron frequency of a particle of mass M and charge ze in the 2000 gauss field, then $M/z \simeq 56$ electron masses, which corresponds to nothing reasonable. Some experiments were done to affirm that the reaction is the source of the signal. The signal is picked up by a probe outside the CS_2 at the instant the signal appears at the CS_2-sapphire gauge. Shorting pins and other sources of electrical noise were removed from the experiment. PMMA was substituted for CS_2 and no noise was observed. The possibility that this electrical signal may provide a means of monitoring the reaction appears real, but much further work will be required to identify its source.

The work reported here is a first step in a study of shock-induced reactions in CS_2. It will provide the basis for further and more detailed studies of microscopic aspects of the transition.

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