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SHOCK-INDUCED REACTION IN CARBON DISULFIDE

George E. Duvall

Department of Physics
Washington State University
Pullman, WA 99164

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Annual Summary Report
for the period
4/1/79 - 3/31/80

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The purpose of this work is to study the shock-induced chemical decomposition of liquid CS₂. Studies of mechanical response have shown that the liquid under compression transforms to a higher density form. Past work has shown that this transformation occurs under static pressure at 45 kbar and 175°C. Previous shock experiments have shown that a similar transformation occurs in a single shock at approximately 60 kbar and 1000°C. Sheffield at WSU has shown that the transformation occurs in a reflected		

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shock at pressures greater than 75 kbar and at temperatures greater than 750°C. The transformation or reaction time was determined by Sheffield in various ways and found to decrease from about 400 nsec at 79 kbar to less than 10 nsec at 130 kbar. At the lower pressures there is an induction time decreasing from 300 nsec at 79 kbar to less than 6 nsec at 106 kbar.

In the year just completed we have finished and reported the work on shock-induced light emission in CS₂ experiments and gone on to redesign the absorption experiment to overcome two principal problems. One was the inadequacy of the light source being used. The present source has an effective temperature of about 15000°K. With this the spectrograph-streak camera produces a satisfactory image with time and spectral resolutions of about 30 nsec and 30 Å, respectively. The other difficulty lay with the effects of shock on the light reflection. This has been resolved by placing the reflector in the projectile, where it is shock-free during the significant part of the experiment. All parts of the new design have been tested, and we are now doing complete experiments in the full-density liquid.

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I. INTRODUCTION

The use of shock waves for generating and analyzing new chemical species, other than explosive products, is still a relatively undeveloped subject. It has considerable potential for both practical applications and understanding, but its limitations are not yet completely defined. Time resolution obtained conventionally in shock experiments is one to ten nanoseconds, but recent studies with laser generated shocks suggest that it may be possible to improve this by nearly two orders of magnitude. The duration of a conventional shock experiment is one or two microseconds, though this can be stretched by at least an order of magnitude in large-scale explosive experiments. Time resolution and experiment duration define the kinetic regime in which shock experiments will be useful. Limitations arise principally because the technical arts associated with shock wave spectroscopy have not yet been fully explored: Suitable light sources remain to be developed; optical transmittance of shocked windows must be established for a variety of materials; luminescence of structural materials under shock is a recurring problem; the conditions under which mirrors retain their reflectivity under shock are not well understood. These and a host of other new problems arise; the procedure followed in our program at WSU has been to thread our way through this maze of difficulties without trying to explore alternative solutions or materials, once a path that serves has been found.

Aside from these laboratory difficulties, the scientific problems to be solved in a shock-spectroscopy program are

- Target and projectile design to provide suitable control of temperature and pressure histories in a geometry which can be adapted to absorption measurements.
- Development of equations of state for reactants and products so that temperatures can be calculated.

- Measurement of mechanical parameters--particle velocity, pressure, etc. in the selected geometry.
- Choice of suitable combinations of light source, optics and detector for the continuous time measurement of broad band spectral absorption.

Initial measurements must be continuous in both time and wavelength because, in general, the spectral region of interest is not known and continuous time measurement provides the simplest information for rate determinations.

Work in progress at WSU is presently limited to a study of the shock-induced decomposition of liquid CS_2 . The purpose of these experiments is twofold: to determine the rate at which CS_2 disappears, and to identify new species which are formed. The degree of success which will be achieved is unpredictable at present, principally because of uncertainties in the decomposition and absorption processes. The liquid form of CS_2 is strongly absorbing in the ultraviolet for $\lambda > 2500 \text{ \AA}$, except for a window centered at about $2700 \text{ \AA}$. The vapor spectrum of CS shows a strong absorption line near $2700 \text{ \AA}$. The intent of the experiment is to monitor the absorption spectrum between 2500 and $3000 \text{ \AA}$ following the shock and look for the development of CS lines and disappearance of CS_2 .

The research now in progress at WSU is an outgrowth of work started here in 1976 by Stephen A. Sheffield, now at Sandia Laboratories in Albuquerque. His work was principally concerned with thermodynamics and mechanical properties of shocked liquid CS_2 . He saw this as a preamble to experiments which would ultimately unravel the reaction processes, and it is toward this goal that we continue to work. Sheffield postulated that a cusp in the Hugoniot at 60 kbars reported by R. Dick (1970) is an indication of chemical decomposition. His work defined the region of interest and provided important data on thermodynamic and kinetic parameters. Work since that time has been

directed to development of equipment and techniques for absorption spectroscopy under shock conditions.

Details of earlier work are set forth in Sheffield's thesis (1978), in the Annual Summary Reports on this contract for the periods 4/1/77 - 3/31/78 and 4/1/78 - 3/31/79, and in a paper presented at a conference in Paris in 1978 (Sheffield and Duvall). These sources should be consulted for more detailed statements of motivation and progress.

II. SHOCK-STIMULATED RADIATION (D. A. Cremers and P. L. Marston)

Shock tube studies of light emission from CS_2 vapor have shown that useful reaction kinetic information can be obtained from its temporal and spectral characteristics (Arnold and Kimbell, 1969; Arnold et al., 1970). Motivated by these results, we have observed light emitted from liquid CS_2 samples shocked to more than 46 kilobars, i.e. into the regime of pressure and temperature at which decomposition occurs. Correlation of this emission with a reaction event involving CS_2 has been complicated by the presence of simultaneous emission from other sources. For example, some materials used in the construction of cells to contain the CS_2 apparently produce light under shock conditions. The purpose of this communication is to report on those sources of extraneous light that we have been able to identify.

The gas gun at WSU can generate projectile velocities up to 1.3 mm/ μsec , which is not sufficient to induce the CS_2 reaction on a single shock. A reshock technique is necessary to generate pressures above 46 kbar. Sapphire single crystals were used at the rear of the CS_2 cell to provide the reshock as well as an optical path from the cell interior. Two cell designs are shown in Fig. 1. Light emission from the CS_2 cell was detected by two RCA 7326

photomultiplier tubes (PMT). The signals from these tubes were recorded on several fast oscilloscopes (Tektronix 7900-7800 series). Photomultiplier tubes were chosen for this initial work because they provide great sensitivity and high frequency response (>100 MHz) (RCA 1970). The spectral bandwidth of the detection system, determined by the photomultiplier S-20 photocathode and the glass optical elements, was limited to the region of 370-700 nm. Filters were placed in front of each PMT to permit independent monitoring of the red and blue parts of the visible spectrum. The photomultiplier tubes were calibrated at 595 nm to permit comparisons of light intensities.

The first experiments of this study revealed that immediately before projectile impact an intense flash of light was produced at the impact plane. This light entered the detection system through the transparent cell front (Figure 1a). The flash was probably the result of blackbody radiation from residual gases trapped at the impact plane and heated to high temperatures. The pressure in the target chamber was about 20μ . Light emission from residual gases has been noted previously (Erkman, 1963). Our photographic recording of this light flash by a Beckman and Whitley streak camera (model 339B with model 1500 dispersion unit) indicated it contains spectral components as far as 270 nm into the ultraviolet. The width of the streak on the film revealed a flash duration of not more than 30 nsec, which was confirmed by a simultaneous photomultiplier measurement.

Calculations based upon film sensitivity and losses in the optical system revealed that the flash of light corresponded to a blackbody of at least 7000°K . Since the flash occurred at impact and was of short duration it could not directly interfere with measurements of CS_2 emission, which should have begun no sooner than two microseconds after impact. The short

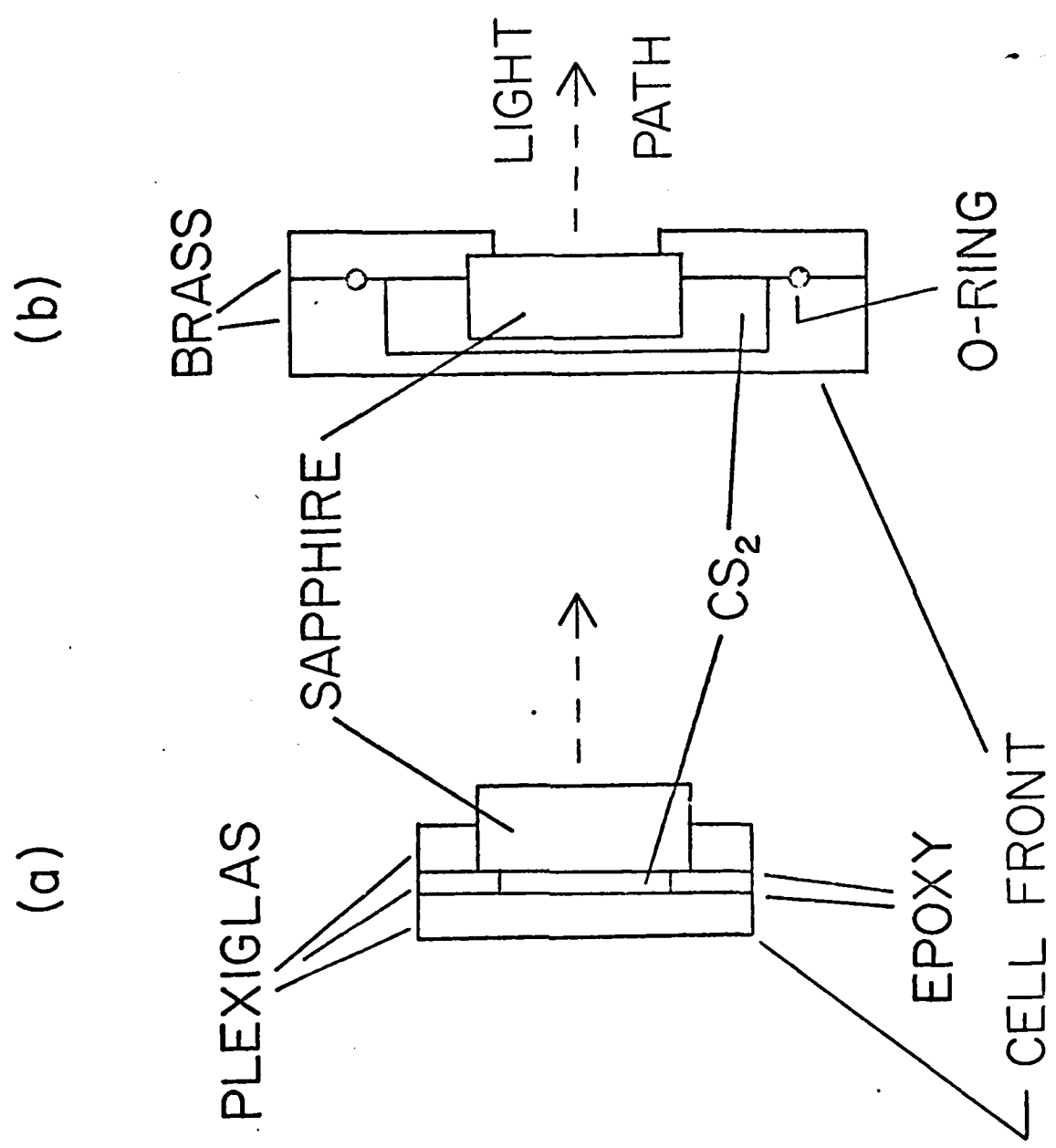


Figure 1

Schematic of two CS₂ cells: the (a) plexiglas-sapphire cell which was redesigned into a (b) brass-sapphire cell.

duration also limited the possibility of permanent photomultiplier damage from such an intense source. However, there was the possibility of a temporary loss of PMT performance due to the flash, which could have affected subsequent measurements of CS_2 luminescence. For this reason it is advisable to design any experiment, which monitors light from shocked samples using sensitive photodetectors, to exclude the impact light from the detection system. For the CS_2 work this involved the use of cells with opaque fronts.

When shocked to a pressure of 2 Mbar, plexiglas emits thermal radiation corresponding to a blackbody at 8300°K (Zel'dovich et al., 1958). There is evidence in the present work that shocked plexiglas generates light at pressures as low as 50 kbar. We have observed light emission during the propagation of shock waves through plexiglas sections of blank cells (cells having no cavity for the CS_2) made of clear and black plexiglas (pigment: carbon black). During the initial shock ($P \approx 50$ kbar), the total emitted power from plexiglas was low (≈ 1.5 ergs/sec). As the plexiglas was reshocked to greater pressures ($P \approx 100$ kbar) the total power increased significantly (24 ergs/sec). The increased emission cannot be directly attributed to plexiglas however, for during the time of reshock the sapphire rear cell piece and epoxies used in cell construction were also shocked. They may have contributed to increased emission. It is apparent then that emission from shocked cell materials, although of weak intensity, may prevent unambiguous interpretation of CS_2 luminescence. To remedy this situation the CS_2 cell was redesigned as shown in Fig. 1b. This cell was made of brass and sapphire and was constructed so that up to and during the time of CS_2 reshock, only brass, CS_2 , and sapphire have been shocked. This limits the possibility of extraneous emission from other cell materials, such as epoxy.

The use of brass rather than plexiglas in CS_2 cell construction significantly reduced the levels of extraneous light. In one experiment, using the cell design of Fig. 1b, it was possible to correlate light emission with shock arrival at the brass- CS_2 and CS_2 -sapphire interfaces. A t-x diagram for this experiment is shown in Fig. 2, along with tracings of the photomultiplier signals to illustrate this effect. Since the emission began as the shock reached the interfaces and then dropped off in intensity as the shock moved away, it would appear to be an interface related phenomenon rather than evidence for the CS_2 reaction. This behavior is clearly illustrated on the red PMT signal between 0.7 and 1.5 μsec and on the blue PMT signal between 1.55 and 1.70 μsec . In the case of a shock induced reaction the light intensity would be expected to increase or remain constant as the shock propagates through the CS_2 . The light may have been caused by heating of minute gas bubbles trapped at the interfaces. The bubbles would cool by conduction to the surrounding media. It is conceivable that small bubbles were present at the microscopically rough, lapped brass- CS_2 interface but the sapphire crystal at the rear interface was highly polished. The emission observed at the CS_2 -sapphire interface could be interpreted in terms of a reaction if it is noted that above 65 kbar CS_2 becomes opaque (Yakusheva et al., 1977). The reduction in light intensity during reshock may have only been apparent, caused by a loss of transparency of shocked CS_2 to radiation at the emitted frequencies.

Light emission has been reported from shocked fused and crystalline quartz (Brooks, 1965; Hamann, 1966) and simultaneously shocked and illuminated aluminum oxide surfaces (Coleburn et al., 1965). We have examined the possibility that the sapphire single crystals (random crystallographic orientation; Adolph Meller Co.) used at the rear of the CS_2 cell may be the source of some extraneous light. A sapphire crystal (38.8 mm diameter by

6.4 mm thick) was shocked to pressures around 110 kbar, which corresponds to pressures present in the sapphire during the actual CS_2 experiments. The sides and impact surface of the sapphire were silver plated to exclude impact light from the detection system. A small brass impactor of 15.9 mm diameter was used to prevent shocking of the epoxy at the edge of the sapphire prior to arrival of the shock at the rear surface. Since shocked epoxy may emit light, the experiment would have been ambiguous. However, the use of the small impactor may have introduced a light producing fracture of the sapphire at the periphery of the impact area. The light emission observed from shocked sapphire is shown in Fig. 3. The burst of light at impact, designated by the arrow, may have resulted from rupture of the silver plating or fracture of the sapphire. Traveling at a velocity of 12 mm/ μsec , the shock wave should have reached the rear of the sapphire 0.5 μsec after impact. The steady increase in light intensity following impact would indicate that it was uniformly generated as the shock propagates through the sapphire. The maximum total emitted power of red or blue light recorded during this time was 3.0 ergs/sec.

In conclusion, we have identified several materials which emit light when shocked to pressures above 50 kbar. Two light producing phenomena have also been identified. With the exception of the impact light, the intensities were relatively weak. Their existence should cause no problems if strong light signals are anticipated from the shocked sample. However, if the strength and character of the light emission of interest are unknown, the extraneous light may confuse interpretation.

This work is to be published in Vol. 11, No. 5 of High Temperatures-High Pressures. It was concluded that further work on emitted light is inappropriate at this time because of the difficulty of detailed, time-resolved spectral analysis at such low light levels.

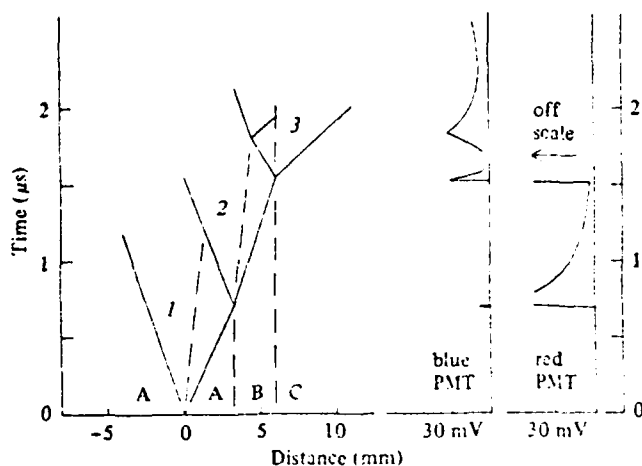


Fig. 2 Analysis of light observed from a shocked CS_2 cell of Fig. 1b. For the x-t diagram on the left: ²A, brass; B, CS_2 ; C, sapphire. The pressures are: state 1, 235 kbar; state 2, 44 kbar; state 3, 111 kbar. Signals (voltage versus time) from red and blue filtered photomultiplier tubes (PMTs) are on the right.

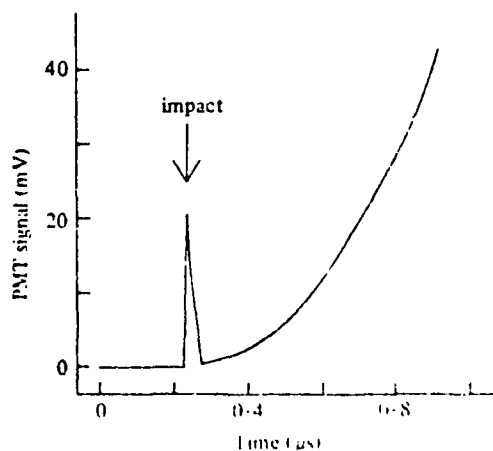


Fig. 3 Tracing of oscillogram showing red light emission from shocked sapphire ($P \approx 110$ kbar). Oscillogram for blue emission was similar. The shock wave traverses the 6.4 mm thick sapphire crystal in about 0.5 μs .

III. ABSORPTION EXPERIMENTS

3.1 Light Source

It was reported in the last Annual Summary Report that there was some doubt about the adequacy of our 6000°K Xe flash lamp for absorption experiments in the ultraviolet. This concern turned out to be well-justified and some effort was devoted to developing a more intense source.

The rate at which radiation is emitted from unit area of a black body into unit solid angle at temperature T and wavelength λ is

$$I(\lambda)d\lambda = .5884 \times 10^{-12} \lambda^{-5} (e^{-1.4385/\lambda T} - 1)^{-1} d\lambda, \frac{\text{watts}}{\text{cm}^2 \text{steradian}}$$

This emission function has a maximum at

$$(T\lambda)_{\max} = 28.97 \times 10^6 \text{ \AA deg.}$$

For $\lambda T \gg (T\lambda)_{\max}$

$$I_{\lambda} d\lambda \approx (.4090 \times 10^{-12} / \lambda^4) T d\lambda.$$

For $\lambda T \ll (T\lambda)_{\max}$

$$I(\lambda) \approx (.5884 \times 10^{-12} / \lambda^5) e^{-1.4385/\lambda T}.$$

That is, when the temperature of the black body is such that the wavelength of interest is less than $\lambda_{\max}$, $I(\lambda)$ increases very rapidly with T . When T has been increased enough that $\lambda_{\max}$ is less than the wavelength of interest, $I(\lambda)$ increases only linearly with T .

In our case the minimum wavelength of interest is 2500 Å at $T = 11,600^\circ\text{K}$, so increasing the temperature much beyond this value is not very effective at increasing the light output in the region of interest to us. Some relative values of I_{λ} at 2700 Å and at several temperatures are shown in the following

table:

T, °K	6,000	10,000	15,000	20,000	25,000
I(λ)	1	35	210	530	950

By choosing the length and diameter of the Xe lamp so that its image just matched the entrance slit to the spectrograph, it was possible, with minor electrical modifications of the power source, to obtain an estimated source temperature of about 15,000°K. This gave a two hundred fold increase in radiation intensity, which we estimated to be enough for our immediate purpose. With this source, time resolution of 30 nsec and spectral resolution of 30 Å, the photo reproduced in Fig. 4 is obtained. This was made under conditions in which the light passes twice through the CS₂ sample and is reflected from a mirror in the projectile. A substantial fraction of light was reflected from sapphire-vacuum interfaces. There is some uncertainty in interpretation of the record in Fig. 4. The assembly at impact consisted of a 1/4" sapphire impactor, a .080" sapphire plate, a .004" cavity filled with CS₂, and a 1/4" rear plate of sapphire. The time between A and B is 1.2 μsec, which is very nearly equal to the time between impact and the meeting of rarefactions from the back face of the impactor and the rear face of the cell. At this meeting the cell should start to split at the CS₂ cavity. The time from B to C is the ring time for the .080" front face of the CS₂ cell. So one interpretation is that the lamp was late in turning on, that impact occurred at A, and the cell started to come apart at B. This leaves unexplained the questions: Why is there no indication of absorption by the CS₂ between A and B? Why is there no change of intensity when the CS₂ is compressed? Why is there no indication of the mirror popping off the back of the impacting sapphire when the shock hits it? In spite of

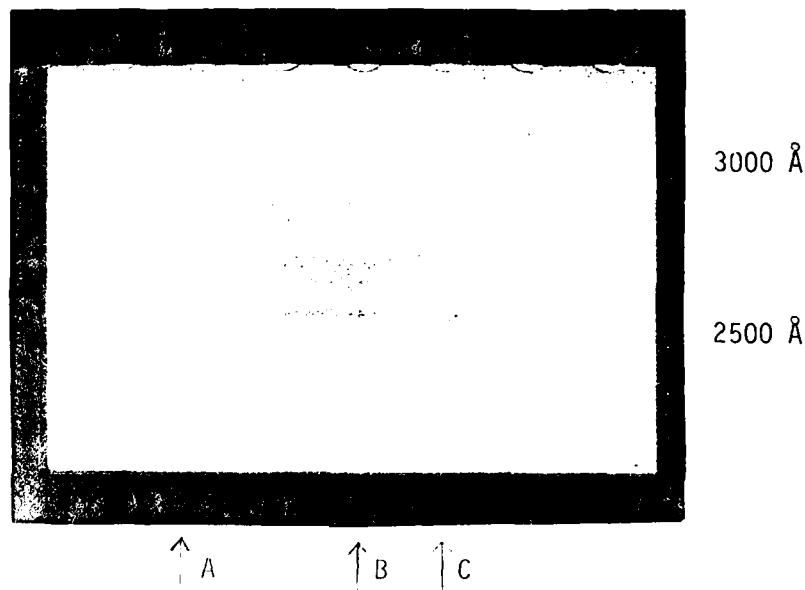


Fig. 4 Record of light source intensity in model
A. Impact
B. Rarefactions from front and rear
surfaces meet.

these unanswered questions the experiment was considered successful because, for the first time, everything seemed to work. The record is suitable for densitometer analysis, but a reduction of intensity by an order of magnitude would render it unusable. To produce much higher temperatures would require a new power source and would give only modest increases in intensity. It appears that laser sources should be considered in applications where much higher intensities are required.

3.2 Reflector Problems

The nature of the shock experiment is such that the influence of the shock on light transmission through a sample must be measured in a direction normal to the plane of the advancing shock front. Any measurement made transversely through the shock is perturbed by an unknown extent by lateral expansion behind rarefaction encroaching on the region of uniaxial strain. These produce regions of non-uniform sample properties. They also produce severe refraction effects which render the interpretation of measured intensities questionable.

When shocks are generated by projectile impact, as at WSU, there are two choices for transmission experiments. For one-way transmission measurements through the sample, the light source and detector (which may be a deflecting mirror) must be mounted on opposite sides of the sample, Fig. 5.

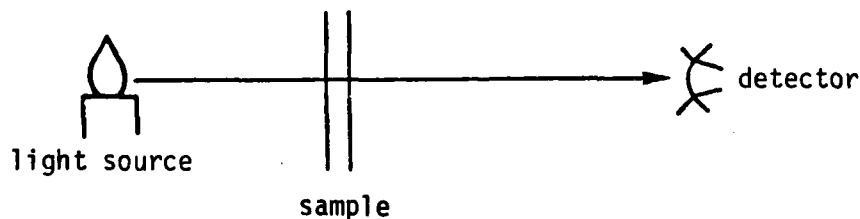


Fig. 5 Experiment with one-way transmission

It is not out of the question to do such an experiment. In fact one such experiment was tried in which the light source was an argon gap squeezed between impactor and a following mass, Fig. 6. After impact, the sapphire

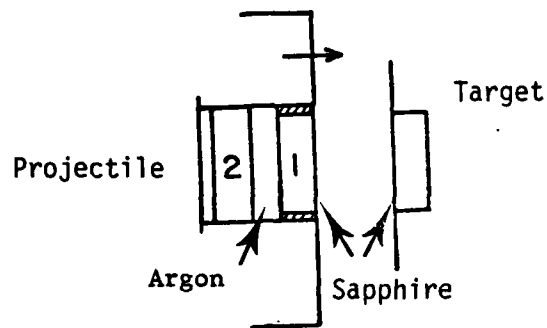


Fig. 6 Argon flash gap in projectile

in the projectile, 1, is stopped and the freely moving mass, 2, continues its forward motion, squeezing the argon between 1 and 2 to incandescence. After one experiment this was abandoned because of non-uniformity and short duration of the flash produced. But variants are possible and may be worth considering in future experiments. One is to mount a flash tube in the projectile in such a way as to be free from shock until the measurement is complete.

The second choice is to direct the light through the rear face of the sample and bring it out the same face after reflection from a mirror in front of the sample, Fig. 7. It has been common practice in the past to incorporate the mirror in the sample assembly, as in Fig. 7a. The trouble with this is that there is no assurance that the mirror will maintain its integrity when shocked. Examination of records taken by others (Yakusheva et al., 1971; Gato et al., 1979) suggests that in at least some

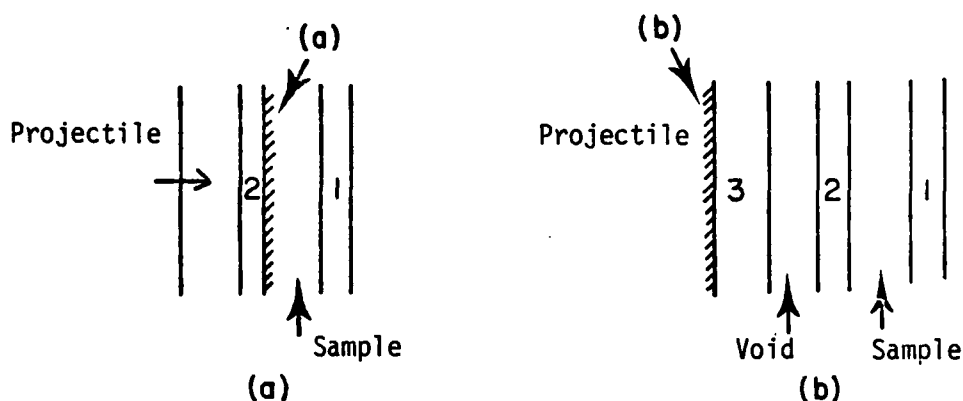


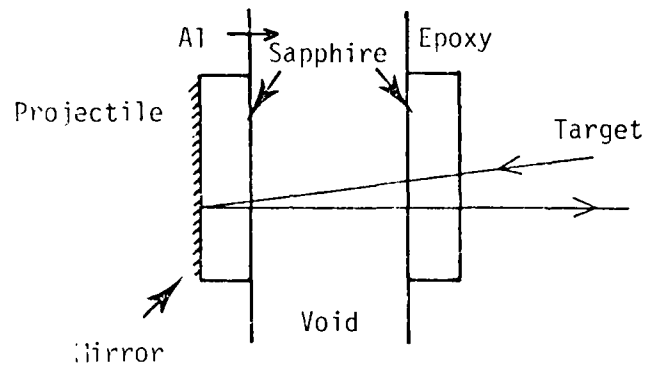
Fig. 7 Two way transmission through the sample. 1 and 2 are transparent faces of sample cell; 3 is a transparent impactor.

(a) Mirror incorporated in sample.

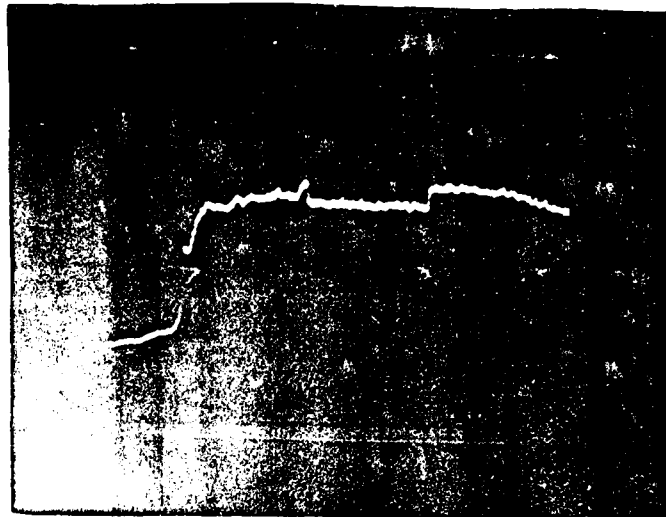
(b) Mirror in projectile.

instances the loss of reflectivity by the mirror may be the major observed effect.

The alternative is to place the mirror in the projectile in such a way that it is protected from shock until the measurement is complete, as in Fig. 7b. Here the mirror is plated on the back surface of the transparent impactor where it is pressure-free until the shock reaches the rear face of the impactor. With a thick impactor and a thin face on the front of the sample cell, the transmission measurement can be completed before the mirror is destroyed. One question of concern about such a system is whether or not the system continues transmitting during and after impact. Fig. 8 is a record of total light intensity through two sapphires with reflection from a mirror as in Fig. 8a. The record of Fig. 8b shows a small reduction of transmitted intensity at impact, but not enough to interfere with experiments.



(a)



↑ A ↑ B ↑ C

(b)

Fig. 8 Transmission of UV light through two pieces of sapphire during impact.

(a) Experimental arrangement

(b) Total intensity in 2500-3000 Å interval vs. time.
Sweep rate 200 nsec/div.

A. Time of impact

B. Shock arrival at rear surface

C. Approximate time of meeting of rarefactions.

3.3 Current Experimental Design

The problems described in Sections 3.1 and 3.2 dictated changes in design of the CS_2 target cell and the impactor. Details of cell and impactor construction are shown in Fig. 9. The cavity containing CS_2 is made very thin to decrease ring-up time and to decrease total absorption in the UV. The rear surface of the back piece of sapphire is beveled to reduced surface reflections back into the spectrograph. The front sapphire plate on the CS_2 cell is thin and the impactor is thick so that transmission measurements are completed before the mirror is shocked.

The total experimental setup is shown schematically in Fig. 10. The PM tube affixed to the streak camera monitors the total light received by the streak camera in the wavelength interval 2500-3000 Å. Pressure-time histories in the CS_2 layer are illustrated in Figs. 11-13.

In Fig. 11, A is the impacting sapphire which strikes the front-face sapphire, B, at $t=0$, $h=0$. C is an inert, transparent substance with shock impedance which closely matches that of the CS_2 in D. The rear face of the cell, E, is also sapphire. Pressure in D rings up to its final value through states 2, 3, 4, 5, etc., shown in Fig. 12 and 13. In Fig. 13, successive pressure states at the C-D boundary are shown by the solid line; mean pressure, as seen by a light ray normal to the cell, is indicated by the dotted line. Duration of states 3, 5, etc. can be adjusted by varying thicknesses of C and D. This permits considerable flexibility in varying pressure and temperature. Its principal disadvantage is that temperature at a given pressure is somewhat less than in the Sheffield experiment.

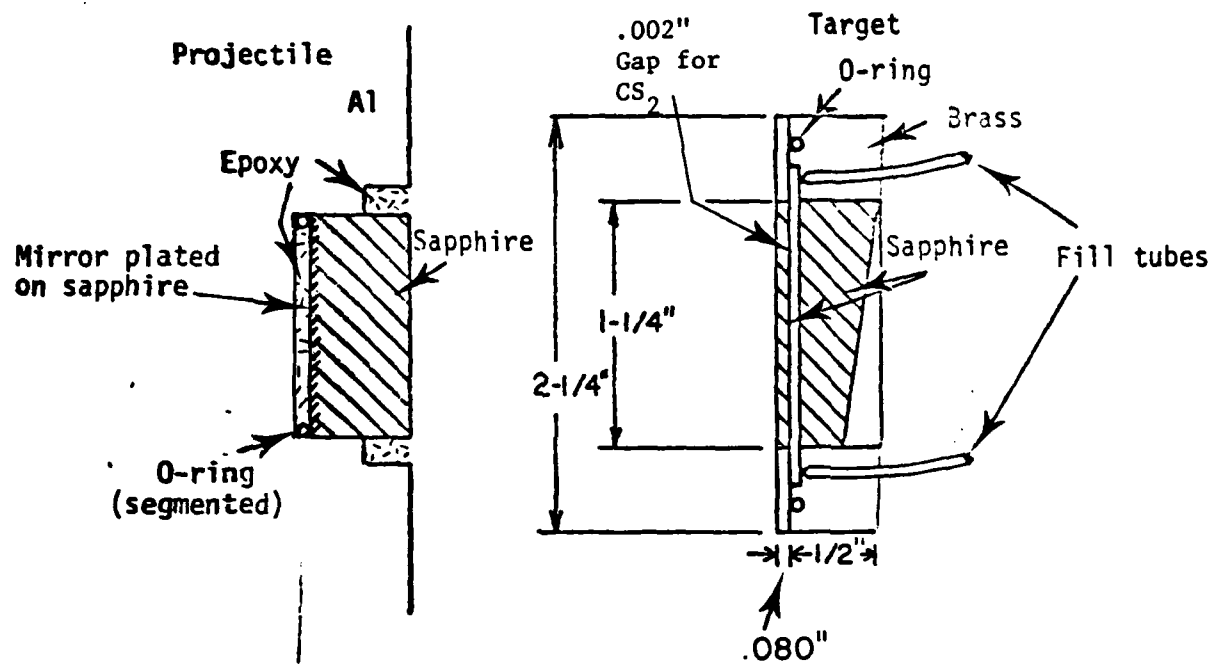


Fig. 9 Target Cell and Projectile for CS_2 Experiment

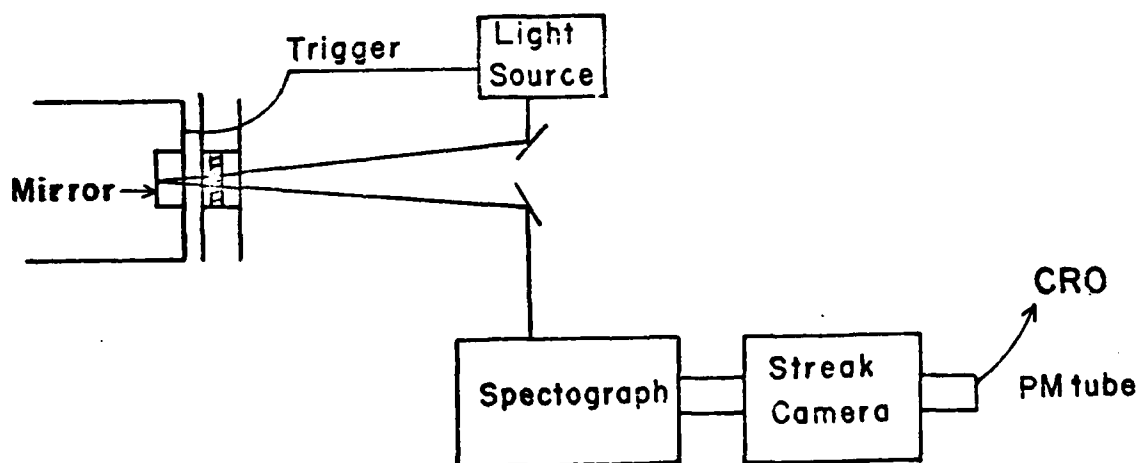


Fig. 10 Design for CS₂ Experiment--Schematic.
Records consist of (i) a two dimensional display on 35 mm film with time running along the film and wavelength across, (ii) total light intensity vs. time received by PM tube.

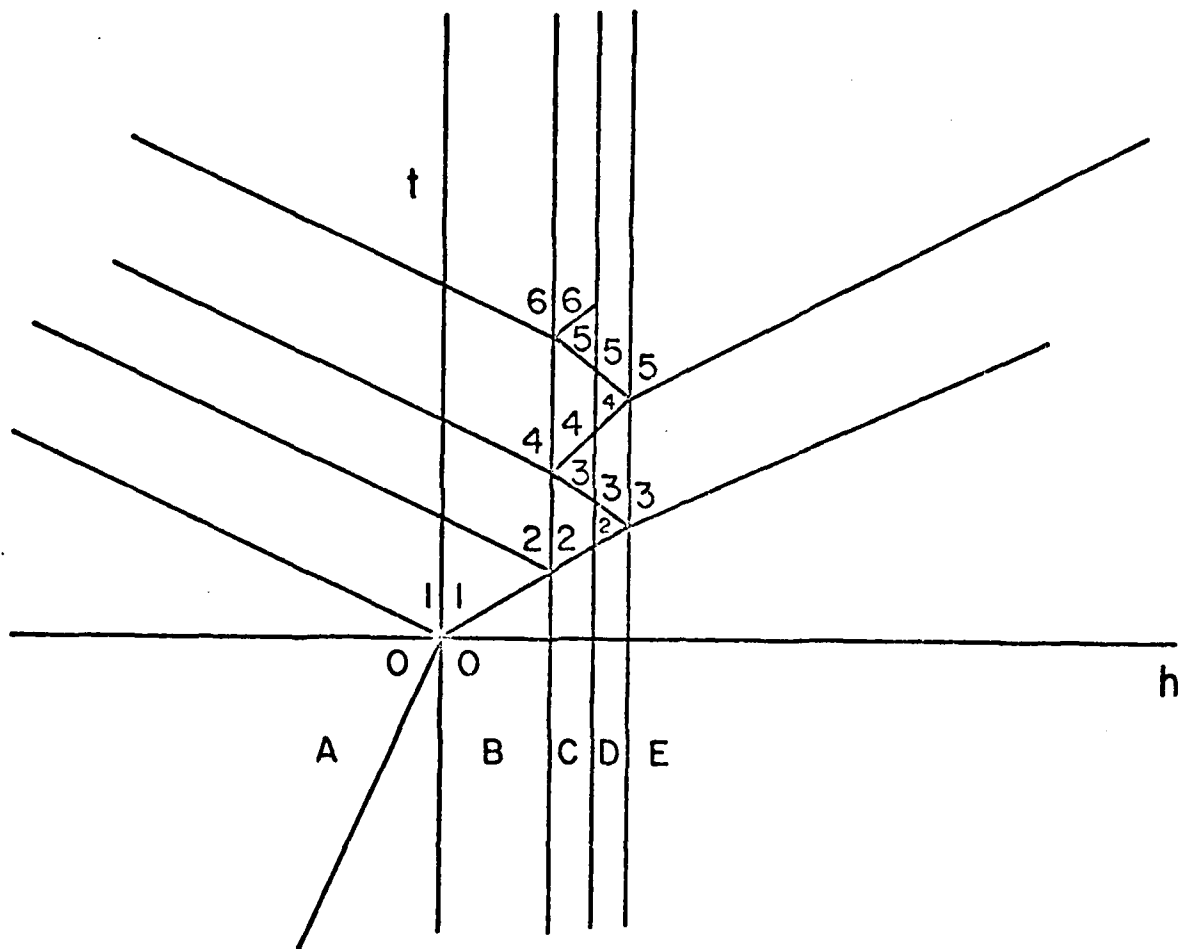


Fig. 11 Cell Design

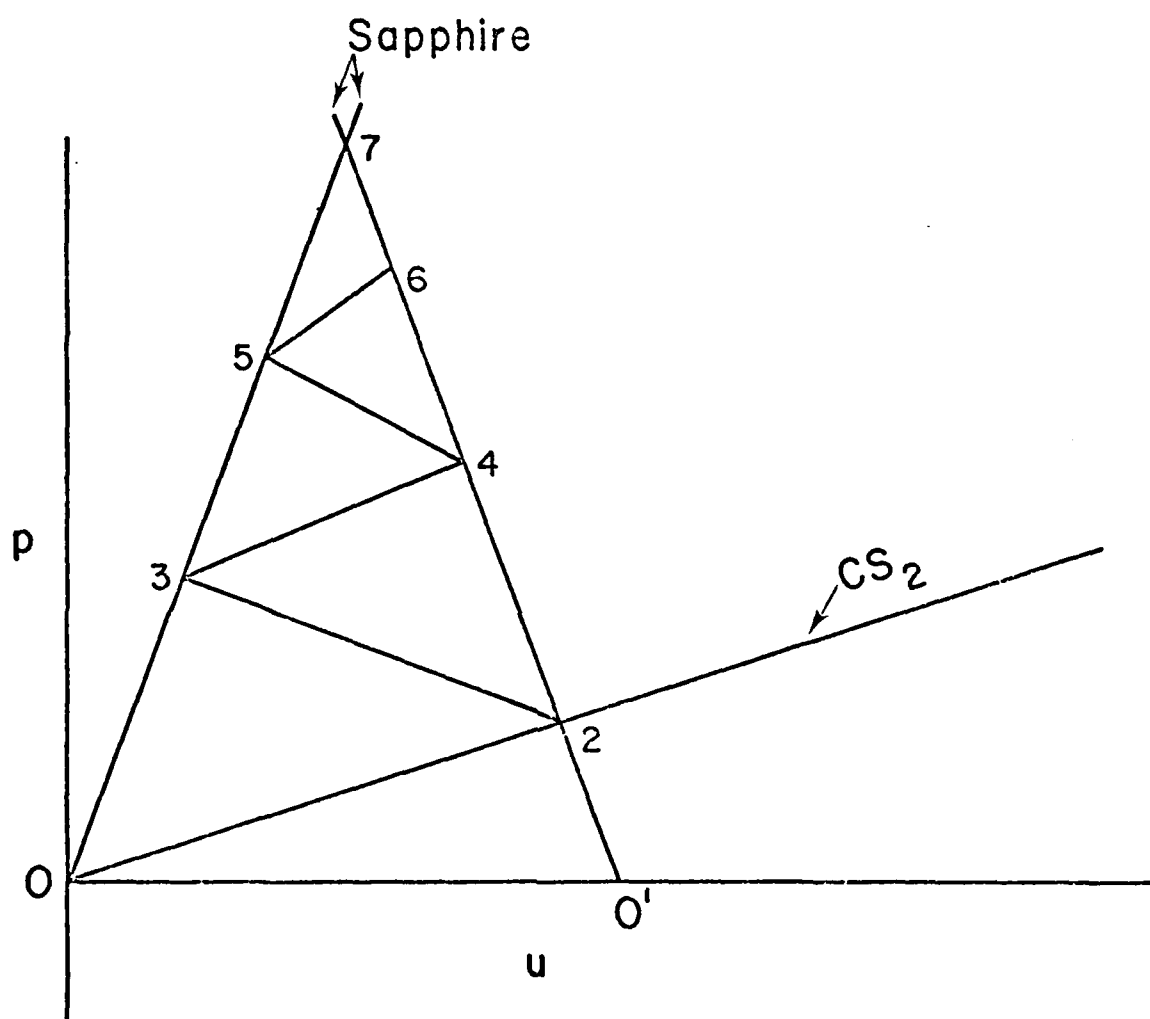


Fig. 12 Pressure states in CS_2

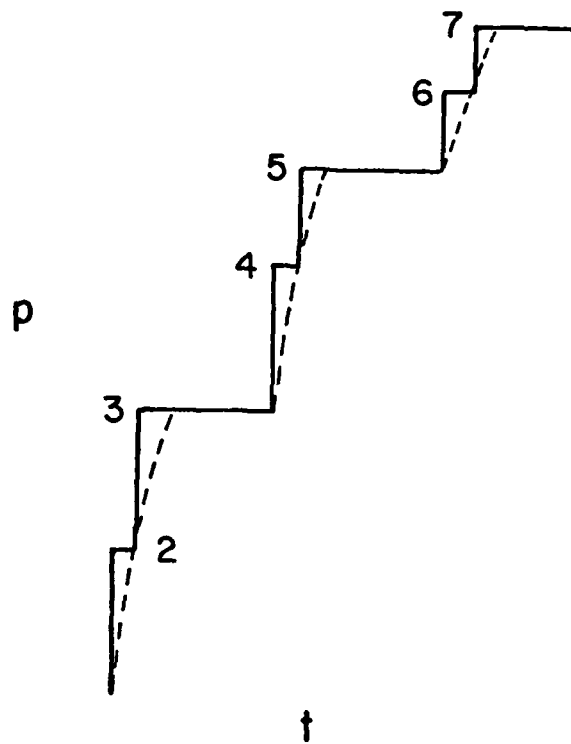


Fig. 13 $\bar{p}(t)$ in sample is represented by dotted curve

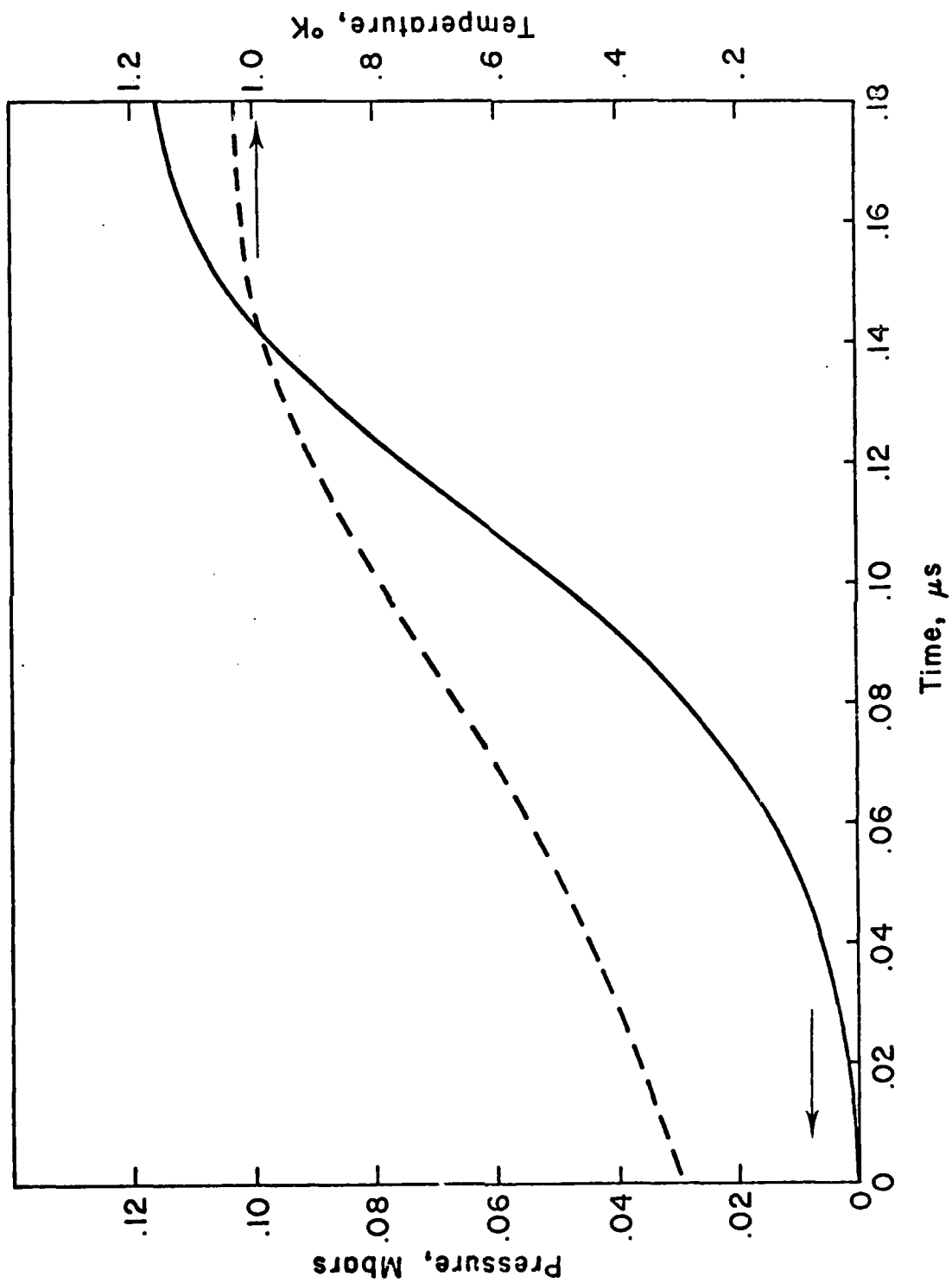


Fig. 14 Temporal record of pressure and temperature in CS_2 compressed by 120 kbar shock in sapphire.

IV. STATUS AND PLANS

The new cell design, shown in Fig. 9, appears to be quite satisfactory for study of absorption in highly absorbing materials when used in conjunction with the new light source, described in Section 3.1. Placement of a mirror in the projectile appears to be a satisfactory method for avoiding loss of reflectivity during the experiment. All parts of the experiment have been tested and performed satisfactorily. A complete experiment was fired in March without success. The "crowbar" in the power supply was apparently triggered by a spurious pulse before the shock reached the CS_2 and the light intensity was substantially diminished so that no film record was obtained. A new cell is being built and will be fired soon.

We think that all the essential experimental difficulties have been overcome, within the limit of our resolution, and the next problem will be one of measuring and understanding the absorption spectrum as it unfolds. We have made preliminary plans to study the CS_2 decomposition in dilute solutions in hopes of distinguishing between first and second order reactions. We have also looked at other promising candidates for shock experiments. But it is not certain that either of these will be done during the next contract period.

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