







# PRODUCTION OF CRACKS IN ZnS UNDER A UNIFORM DYNAMIC STRESS FIELD

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PRODUCTION OF CRACKS IN ZnS UNDER A UNIFORM DYNAMIC STRESS FIELD. = MAR 19 1979 Annual Report P January 1979 10 By:/D. A. Shockey, Y. M. Gupta Prepared for: Office of Naval Research 800 N. Quincy Street Arlington, VA 22203 Attn: Dr. R. C. Pohanka Dr. Arthur M. Diness a decurrent hes been opposite Contrac N00014-76-C-0657 SRI Project PYU-4928 Approved by: G. R. Abrahamson, Director **Poulter Laboratory** P. J. Jorgensen, Vice President Physical and Life Sciences 410 281 LB

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### ABSTRACT

Plate impact and exploding cylinder experiments were performed on CVD ZnS and PMMA, respectively, to establish the applicability of the methods for studying dynamic fracture of brittle materials. Prerequisites for successful plate impact experiments are precise velocity control in the tens-of-meters per second range and specimen encapsulation. Prerequisites for successful exploding cylinder experiments are large specimens (to contain cracks), external compressive hoop stresses, and control over the type and amount of explosive. With these prerequisities, both methods appear to be useful for studying fracture response in fragile materials.



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Conditions and Results of Plate Impact Experiments on CVD ZnS

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#### I SUMMARY

The objective of the work described herein was to investigate the feasibility of applying plate impact and exploding cylinder techniques to study dynamic fracture behavior in fragile ceramics. The research was motivated by problems encountered by the Navy when rain, dust, and ice particles impact laser windows and radomes on flying aircraft. Plate impact and exploding cylinder tests, which are widely applied to study dynamic fracture phenomena in metals, cause fracture under uniform, well-defined dynamic conditions and offer an opportunity to establish material properties governing impact erosion of ceramics.

Using the SRI 100-mm-bore gas gun, we performed seven plate impact experiments on CVD ZnS, a candidate laser window material. We overcame initial difficulties in attaining reproducible velocities in the tensof-meters per second range necessary to cause incipient fracture damage in this fragile material, and demonstrated feasibility of the method in the final two experiments. We designed an encapsulated specimen arrangement (Figure 2) that reduced undesirable radial cracking and permitted intact recovery of severely fractured specimens. Figure 3 shows the fracture damage produced in two impacted specimens. Cracks initiated internally (in the absence of surface influences), and propagated parallel to the loaded surface (in the manner of lateral cracks beneath a particle impact site). Crack size distributions appear to vary through the plate thickness in accordance with the expected tensile stress variation, and the large numbers of cracks may be statistically analyzed. The results suggest that the plate impact method can be applied to ceramics to study lateral crack kinetics and obtain dynamic fracture properties.

Cylindrical specimen geometries allow dynamic fracture to be studied in stress states where large shear strains are concomitant with tensile loads. To investigate whether statistical numbers of cracks could be nucleated, grown, and arrested in thick-walled cylinders of brittle

materials, we performed a number of exploding cylinder experiments, using PMMA specimens and various amounts of explosive, and assessed the resulting fracture damage. The number of radial cracks could be controlled by varying the explosive charge. The 5 to 10 largest cracks (corresponding to the dominant radial cracks around a particle impact site) usually propagated to the cylinder boundaries, and thus crack size was not well controlled (despite an attempt to arrest the cracks in the specimen by applying external compressive hoop stresses). Containment of the cracks might be achieved by using larger specimens, but this would increase material requirements. The exploding cylinder technique thus appears less well suited than plate impact techniques to studies of dynamic fracture of ceramics.

#### **II INTRODUCTION**

Impact erosion is a dynamic fracture phenomenon in which cracks form and grow in material upon passage of a stress pulse generated by the impact of a particle. The phenomenon poses a problem to the Navy when rain, dust and ice particles impinge on aircraft laser windows and radomes. Therefore, the Office of Naval Research is sponsoring research to reduce this problem. As a part of the research effort, SRI International is examining the applicability of plate impact and exploding cylinder techniques for determining the dynamic fracture properties of ceramic materials.

The properties governing a material's fracture response can in principle be deduced by correlating the stress history in the target with the resultant fracture damage. In practice, however, it is difficult to calculate or measure the stress history experienced by material beneath a particle impact site, and even more difficult (because of the high gradients in space and time) to obtain statistically valid local quantitative measures of fracture damage for correlation. Plate impact and exploding cylinder methods may provide a way of deducing dynamic fracture properties by allowing loading of large volumes of material with a uniform stress pulse.

Plate impact experiments produce dynamic stress histories in materials that are similar to those produced by particle impacts, but without the large gradients in time and space that are characteristic of the latter. A further advantage of the plate impact method is that tensions occur first and endure longest in the specimen interiors. This encourages crack nucleation and growth to occur in the absence of surface effects, an attractive feature in view of the sensitivity of semibrittle materials to surface conditions. These desirable aspects of plate impact experiments motivated an effort to establish feasibility of the approach for studying the dynamic fracture response of CVD ZnS, a candidate laser window material.

The exploding cylinder method produces concomitant tensile and shear stresses, and thus allows study of the effect of superimposed shear on dynamic fracture. The applicability of this technique for investigating combined tensile/shear fracture in brittle materials was examined on PMMA specimens.

In this report we first describe briefly the plate impact technique and how the technique has been used to obtain dynamic fracture properties of materials. We then describe the experiments conducted to determine the feasibility of applying this method and the exploding cylinder method to investigate impact erosion phenomena in laser windows.

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#### III BACKGROUND

#### The Plate Impact Technique

If two plates can be made to collide in a planar manner, the response of the plate material occurs under uniaxial strain conditions, a dynamic mechanical state that is simple and mathematically tractable and can easily be related to material response. For this reason, the colliding plate experiment has been widely used by shock wave physicists interested in dynamic material behavior.

Plate impact experiments are usually carried out by mounting a plate on the leading edge of a 50- to 100-mm-diameter cylinder and accelerating the cylinder as a projectile in the barrel of a light gas or an air gun. The second plate is positioned at the gun muzzle and carefully aligned to obtain planar impact. The surfaces of both plates are polished to a surface finish of 0.8  $\mu$ m or better.

Upon impact, a unidirectional compressive wave propagates into both plates, reflects as an unloading wave from each free surface, and produces a short-lived tensile pulse when the two reflected waves intersect. If the impacting plate is made half the thickness of the stationary plate, the tensile pulse occurs symmetrically in the latter, and the impactor plate experiences only compression and release. This allows dynamic material response to be investigated under compression only as well as under compression followed by tension in a single experiment.

If the amplitude and duration of the tensile pulse are sufficiently large, tensile fracture may occur in the stationary plate. Control of the impact velocity and the plate thicknesses allows pulse amplitude and duration, respectively, to be precisely selected and independently varied. In this way microfractures can be initiated and then arrested at various stages, allowing fracture evolution to be studied in a stopaction manner.

#### Fracture Kinetics

Plate impact studies have shown that dynamic fracture in solids (including unalloyed metals and metal alloys, polymers, geological materials, and fiber and particle composites) occurs by nucleation and growth of numercus microfailures, and that these processes could be described by simple rate expressions.<sup>1</sup> The rate of microcrack nucleation  $\dot{N}$  was found to depend exponentially on the locally applied stress  $\sigma$ according to the relation

$$\dot{N} = \dot{N}_{0} \exp \frac{\sigma - \sigma_{n0}}{\sigma_{1}}$$
(1)

where  $N_0$  is the threshold nucleation rate,  $\sigma_{no}$  is the threshold stress for microfracture nucleation, and  $\sigma_1$  is the stress sensitivity.

Similarly, the rate of microfracture growth R was found to be well described by

$$\dot{R} = \frac{\sigma - \sigma_{go}}{4\eta} R$$
 (2)

where R is the characteristic size of the microfracture,  $\sigma_{go}$  is the threshold stress for growth, and  $\eta$  characterizes crack growth resistance. The five dynamic fracture parameters  $\dot{N}_{o}$ ,  $\sigma_{no}$ ,  $\sigma_{1}$ ,  $\eta$ , and  $\sigma_{go}$  suffice to describe the fracture behavior of the material under dynamic loads and are independent of loading geometry. They are dependent on temperature<sup>2</sup> and microstructure<sup>3</sup> and hence act as material properties.

The ability to shock load and recover brittle materials in the gas gun facility was demonstrated<sup>4</sup> during a project concerned with dynamic fracture behavior of Arkansas novaculite, a fine-grained quartzite rock, which is expected to behave much like ceramic materials. A significant finding was that the dynamic fracture process threshold stress  $\sigma_{\rm no}$  was identical with the static tensile strength for this brittle material.

In the novaculite project, crack coalescence and fragmentation, the final two stages of the dynamic fracture process, were also treated,

and material parameters describing these stages were established. An important material property was found to be the incipient flaw structure in the virgin material. This microstructural feature was measured quantitatively and was found to control the fracture strength and fragmentation characteristics of novaculite. The incipient flaw structure was observed in low-magnification composite micrographs. The size distribution of the inherent flaws (which were located at grain boundaries) was determined by counting and measuring the flaw traces on a polished section through a specimen. These data, which represented the size distribution of flaw traces per unit area, were then converted by a statistical transformation<sup>5</sup> to obtain the actual size distribution of inherent flaws per unit volume. An exponential distribution resulted.

In a later research program, the dynamic fracture properties measured under one-dimensional strain conditions were found to predict successfully many features of the dynamic fracture and fragmentation behavior of novaculite under two-dimensional impact conditions<sup>6</sup> similar to the erosion conditions experienced by infrared windows and radomes. Predictions of crater dimensions, fragment size distributions, and subcrater fracture damage agreed well with measured quantities.

The success of this approach in predicting the cratering behavior of polycrystalline quartzite (a material similar in strength and brittleness to window and radome materials) under impact conditions similar to those of interest encouraged us to apply the approach to the rain and dust erosion problem. The following section describes the results of a series of plate impact experiments on CVD ZnS.

#### IV PLATE IMPACT EXPERIMENTS

Seven plate impact experiments were carried out on CVD ZnS<sup>\*</sup> in an attempt to produce a population of microcracks under uniaxial strain conditions and to recover the fractured specimen intact. Conditions and results of these experiments are given in Table 1.

Three experiments were conducted with the SRI 63-mm-bore gas gun. In these experiments, 3.4-mm-thick aluminum flyer plates were mounted on the leading edge of a 150-mm-long by 60-mm-diameter aluminum projectile and accelerated against the specimen plate in a planar manner. Energy-absorbing material was placed behind the specimen to effect a soft recovery; a steel plate prevented the projectile from entering the recovery area and reimpacting the specimen. The nonplanarity of the impact was measured for the first two experiments and found to be 1 mrad and 0.8 mrad, respectively. The initial two experiments resulted in fragmentation.

The cracks produced by these experiments were not of the orientation expected to result from a uniaxial strain tensile pulse. Instead of a profusion of cracks on planes roughly parallel to the plate surfaces, six to eight large radial cracks were produced, as shown in Figure 1. Examination of the fracture surfaces with a microscope revealed that these radial cracks initiated at the impact surface; furthermore, the cracks probably formed quite early in the specimen stress history because an imprint of the specimen fracture pattern was visible on the aluminum flyer plate. Thus, these experiments were of little use for correlating stress history and fracture damage, because of the radial cracks that formed under two-dimensional hoop stresses, propagated

A description of chemical-vapor-deposited ZnS, manufactured by Raytheon Corp., is given in Part II of this report.

Table 1

CONDITIONS AND RESULTS OF PLATE IMPACT EXPERIMENTS ON CVD ZnS

Experiment No.	Specimen Thickness (mm)	Flyer Material/ Thickness (mm)	Impact <u>Velocity (m/s)</u>	Remarks	Fracture Damage
4928-1	5.53	2024 A1/3.47	17.6	ZnS guard ring, no encapsulation	Severe fragmenta- tion
4928-1	5.41	2024 A1/3.39	1.11	ZnS guard ring, no encapsulation	Severe fragmenta- tion
4928-3	5.47	2024 A1/3.43	5.2	ZnS guard ring, no encapsulation	Seven fragments
4928-4	5.69	PMMA/1.48	41.7	Polycarbonate encapsula- tion, ZnS guard ring	Reduced radial cracking
4928–5	12.5	PMMA/1.78	19.0	Polycarbonate encapsula- tion, aluminum guard ring	Reduced radial cracking
4928-6	6.35	Aluminum/0.25	v30	Aluminum encapsulation, epoxy backing (see Fig. 2)	See Fig. 3
4928-7	6.35	Aluminum/0.25	26.5	Aluminum encapsulation epoxy backing (see Fig. 2)	See Fig. 3

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FIGURE 1 RADIAL CRACKS ON THE IMPACT SURFACE OF A CVD ZnS PLATE Impact specimen 4928-3.

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into the specimen interior, and destroyed the simple and calculable uniaxial strain conditions there. It was clear that for the plate impact technique to be useful in identifying dynamic fracture properties for ceramics, radial cracking had to be prevented and impact velocities had to be controlled more precisely.

In the low velocity range (tens of meters/second) required to produce incipient fracture damage in such fragile materials as CVD ZnS, effects of friction between projectile and gun barrel are large and present difficulties in obtaining precise and reproducible velocities. These difficulties were solved by realigning and rehoning an existing 100-mm-diameter gas gun and using a release valve arrangement. We were able to reduce the extent of undesirable radial cracking by encapsulating the ZnS specimen plate in polycarbonate (Experiments 4 and 5). The extra thickness and strength provided to the target assembly apparently reduced the hoop stresses in the ZnS plate; moreover, the tough encasement aided in the soft recovery of the specimen by protecting it against subsequent impacts and holding the fragmented specimen together.

In the final two experiments (6 and 7), a thinner aluminum flyer plate was accelerated to a higher velocity in order to introduce a stress pulse of higher amplitude and shorter duration into the encapsulated specimen. The intent was to activate a larger number of inherent flaws (initiate more cracks) and to arrest their growth at an earlier stage (reduce coalescence and fragmentation). A schematic of projectile and target assembly used in the two final experiments is given in Figure 2. Both the aluminum flyer and the ZnS specimen plates were backed with epoxy plates to reduce flexure. Epoxy, which has a low acoustical impedance, permits a large fraction of the incident shock to be reflected. Great care was taken in aligning the specimen assembly to ensure a planar impact and, hence, uniaxial strain conditions for fracture.

A velocity of about 30 m/s was achieved for the first experiment (Experiment 6). The target assembly was recovered and sectioned to reveal the fracture damage. Figure 3a shows a photomicrograph of the



FIGURE 2 SCHEMATIC OF PROJECTILE AND TARGET ASSEMBLY USED FOR PLATE IMPACT EXPERIMENTS ON ZnS



polished section. The orientation of the crack population indicates that the impact was planar and that fracture occurred predominantly under uniaxial strain conditions. Although some radial cracks were produced, they appear not to have interfered strongly with the uniaxial strain fracture process. Thus, it appears reasonable to use this experiment to correlate crack size distribution and stress history and attempt to identify dynamic fracture properties.

The final experiment (Experiment 7) was performed at a lower velocity in an attempt to activate fewer flaws than were activated in the previous experiment. The resulting fracture damage produced by an impact at 26.5 m/s is shown in Figure 3b. The density of cracks is noticeably reduced, but is still quite high.

These results suggest that the threshold velocity for fracture damage in ZnS is quite sharp, and that damage increases very rapidly with velocity above the threshold. The behavior may be explained by the existence of an inherent flaw size distribution having large numbers of flaws of, or close to, maximum size, and by high crack velocities. Both features are expected in a semi-brittle material such as ZnS. A ramification of this behavior is that precise control of impact velocity is required to produce fracture damage in incipient stages.

#### V EXPLODING CYLINDER EXPERIMENTS

An alternate method of producing incipient fracture damage in large volumes of brittle materials under dynamic loads is to explosively load the axis of a cylinder. Professor Ian Fyfe<sup>8</sup> at the University of Washington has developed this method and applied it to metals with success. Explosive experiments on rocks conducted at SRI showed that 30-cm-dia., thick-walled cylinders having an exploding wire or a thin column of high explosive along a 6-mm-dia. axial borehold sustain a population of internal cracks in a controlled maximum.<sup>9</sup> Fracture damage produced in these experiments, however, occurs under larger shear strains than in the plate impact experiments because of the divergent geometry of the cylinder.

To investigate the applicability of this method to small cylinders of brittle materials, we performed a number of experiments on PMMA. We selected PMMA because it was easier to obtain and machine than ZnS and because its transparency allows internal cracks to be observed without having to section the specimens. The specimens were 22.2 mm in diameter, 10.3 mm thick, and had 2.38-mm-dia. axial holes.

We calculated stress histories with a finite difference wave propagation code to determine the amount of explosive power required. We noted that the tensile stresses produced in the specimens were accompanied by substantial shear stresses, a stress state similar to that produced in certain regions beneath particle impact sites.

We carried out 17 experiments using from 20 to 100 mg of lead styphnate. The explosive was detonated with a bridge wire. The resulting fracture damage consisted of radial cracks, which initiated near the explosive/PMMA cylinder interface and ran to the outer boundary. We found we could vary the number of cracks from about 5 to 10 by varying the quantity of the explosive charge. These numbers are not

conducive to a statistical analysis, but they are representative of the number of large radial cracks produced by an impacting particle. We attempted to arrest the cracks in the cylinders by applying confining pressures of several magnitudes. Compressive hoop stresses, produced by press fitting the cylinders into annular aluminum rings, were not sufficient to prevent most of the cracks from reaching the outer cylinder boundary. Therefore, the experiments were not useful for crack length measurements and, hence, for crack growth property determinations. However, if this problem can be solved by using larger compressive stresses or larger cylinders of material, the exploding cylinder method could be useful for studying dynamic fracture behavior.

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