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SHOCK CHARACTERIZATION OF EXPLOSIVE MATERIALS

VOL. II: 5-CYANOTETRAZOLATOPENTAAMINE-COBALT (III) PERCHLORATE (CP)

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ABSTRACT

The results of an experimental program to generate dynamic mechanical properties data for an unreacted porous explosive material are discussed and the data presented. Stress-particle velocity Hugoniot data were experimentally determined for 5-Cyanotetrazolatopentaamine-Cobalt(III) Perchlorate (CP) using flat plate impact techniques. The CP samples were fabricated to the desired bulk sample density and size using a pressing technique. Samples with nominal densities of 1.47 and 1.66 g/cm³ were investigated. The Hugoniot data for both sample densities showed essentially the same compaction behavior at stress levels above 5 kbar. The stress-time data showed major stress increases following impact, indicating initiation of a lower order chemical reaction. A constant high amplitude stress plateau was achieved after impact in a number of the experiments. These data provide both shock initiation and reaction rate information for CP.

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PREFACE

The experimental work described in this report was conducted under Contract F29601-79-C-0017 for the Air Force Weapons Laboratory (AFWL). The technical program was performed for Sandia Laboratories, Albuquerque, NM. The AFWL Project Officer was Mr. Kenneth D. Smith and the Sandia Laboratories Technical Consultant was Dr. Philip L. Stanton. The authors are indebted to Dr. Stanton for his helpful suggestions regarding explosive material physical characteristics and safe handling procedures. The experimental work was performed at the AFWL Impact Facility by Ktech Corporation personnel. Mr. L. M. Lee was the Principal Investigator and was assisted by Messrs. B. D. Jenrette and C. G. Newcomb.

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SECTION I

INTRODUCTION

Explosive materials are utilized in a number of system and component applications. Effective design and performance analyses of systems employing explosive materials are dependent upon proper modeling of the materials' thermodynamic behavior. A complete thermodynamic equation of state is based on numerous types of material properties data, including the shock response or Hugoniot of the unreacted explosive. The primary purpose of the study reported here was to experimentally determine the shock loading behavior of unreacted explosive materials and provide information to support reaction rate modeling.

The porous explosive studied was 5-Cyanotetrazolatopentaamine-Cobalt(III) Perchlorate (CP). Pressed CP samples were supplied to the AFWL Material Response Impact Facility in cylindrical shape. Two sets of samples were made, with one set having a nominal bulk density of 1.47 g/cm^3 and the other set 1.66 g/cm^3 . Theoretical solid density of the CP mixture was determined to be 1.97 g/cm^3 . Binder was used in fabricating the samples; however, the low density samples had minimal structural integrity.

The impact experiments, conducted using the gas gun facilities at the AFWL Material Response Impact Facility, provided Hugoniot data for both densities of CP. The low density explosive samples had limited structural integrity and were not flat, which hampered the experimental setup. The Hugoniot data, generated using a projectile-mounted piezoelectric transducer technique, were used to define the shock compaction characteristics of the porous explosive up to 60 kbar. The Hugoniot data for both sample densities indicated essentially full compaction was achieved at about 5 kbar. The stress-particle velocity data were transformed

to the stress-specific volume and shock velocity-particle velocity planes using the Hugoniot equations to facilitate utilization of the experimental results.

The experimental technique used to generate the Hugoniot data was designed to measure essentially an instantaneous change in stress, which remained at a constant amplitude during the recording time of the transducer (1.0 μ s). Behavior of this type is typical for unreactive porous and solid materials. Experimental data generated in this program showed substantial stress increases as a function of time following the initial stress jump produced by the impact. Both densities of explosive displayed the same type of behavior. The stress increase was attributed to initiation of a lower order (deflagration) chemical reaction. Initiation was observed in the lower density material when impact stress exceeded 8 kbar, with 12 kbar being required to produce the same type of behavior in the higher density samples. The increasing stress reached a plateau during the recording time of the experiment when impact stresses were greater than 15 and 20 kbar in the 1.47 and 1.66 g/cm³ samples, respectively. The high amplitude stress (70 to 100 kbar) plateau was achieved approximately 0.3 μ s after impact and remained essentially constant during the one-dimensional read time of the experiment. The experimental results provide a unique set of data to support reaction rate modeling of the CP, in addition to providing unreacted Hugoniot data.

SECTION II

MATERIAL DESCRIPTION

The explosive samples used in the test program were pressed for Sandia Laboratories by Unidynamics* using material from production lot number EL-47344. The CP (5-Cyanotetrazolatopentaamine-Cobalt(III) Perchlorate) was formulated to the specifications in Reference 1. A solid particle density of 1.965 g/cm^3 has been determined using a picknometer technique[†], while 1.99 g/cm^3 has been reported for single crystal CP (Ref. 2).

The low density CP samples were fabricated using a one percent Viton A binder and a consolidation pressure of approximately 0.7 kbar. The average density of the ten samples fabricated for the impact test program was $1.47 \pm 0.01 \text{ g/cm}^3$. The impact surface of the low density samples was not flat, which is a requirement for conducting Hugoniot tests. The samples typically were high in the central region of the disk by as much as 0.1 mm. Curvature of the sample impact surface negated accurate target and projectile alignment and produced long rise times of the initial stress pulse ($0.05 \mu\text{s}$). The higher density CP pellets were fabricated from the same material lot and were also provided as free-standing disks. The higher density samples were nominally $1.66 \pm 0.01 \text{ g/cm}^3$. A one percent Viton A binder was used in conjunction with a consolidation pressure of 2.58 kbar. The higher density CP samples were not flat either; however, they were substantially better than the 1.47 g/cm^3 samples.

* Unidynamics/Phoenix, Inc., Box 2990, Phoenix, Arizona 85062.

† Marchi, D. L., Sandia Laboratories, Private Communication.

1. Fleming, W. and Fronabarger, J. W. (Unidynamics) and Searcy, J. Q. (Sandia Laboratories), "Preparation of 5-Cyanotetrazolatopentaamine-Cobalt(III) Perchlorate, (CP): A New Material for Detonator Applications," Proceeding of ADPA Meeting, Albuquerque, NM, May 15-17, 1979.
2. Graber, E. J. and Morrison B., "The Secondary Explosive 5-Cyanotetrazolatopentaamine-Cobalt(III) Perchlorate," Proceedings, American Crystallography Association, University of Oklahoma, Norman, OK, March 19-24, 1978.

A backer plate of plexiglass was attached to the free-standing samples to facilitate target fabrication and minimize sample breakage. The backer plate and explosive sample were bonded together using 5-minute cure epoxy* applied to the circumference of the recess in the backer plate (Figure 1). A minimum amount of epoxy was used on the circumference and a thin film was applied to the flat surface in contact with the sample. The small amount and location of epoxy used in mounting the sample did not influence the Hugoniot measurements.

Figure 1. Schematic of explosive sample and plexiglass backer used to hold the sample.

* Devcon "5 Minute" epoxy, Stock No. 14250, Devcon Corp., Danvers, MA 01923.

SECTION III

EXPERIMENTAL PROCEDURES AND ANALYSIS

The dynamic response of the porous, unreacted CP was determined under conditions of uniaxial-strain shock loading. The shock loading characteristics of the explosive material were obtained from plate-impact experiments performed with a 102-mm bore compressed gas gun (Ref. 3). Analysis of the shock loading data employed the Rankine-Hugoniot jump equations, which assume steady wave behavior and thermodynamic equilibrium behind the shock front (Ref. 4).

1. HUGONIOT EXPERIMENTS

The basic experimental configuration used to generate the Hugoniot data is shown schematically in Figure 2. In this configuration the piezoelectric transducer used to measure stress was mounted in the projectile rather than the target. The projectile-mounted transducer design was adopted so that the explosive sample could be placed in the target and not subjected to any acceleration loads. Previous experience indicated that pressed pyrotechnic and explosive samples cannot be mounted on projectiles and directly impacted into targets successfully because of their limited structural integrity (Ref. 5).

The explosive sample was impacted with either a 4340 R_c52 steel or a tungsten carbide (WC)* plate backed with a shunted quartz gauge (Ref. 6) to make

* Type K-68, Kennametal Inc., Latrobe, PA.

3. Lee, L. M., Operation of the Air Force Weapons Laboratory Material Response Impact Facility, AFWL-TR-75-287, Air Force Weapons Laboratory, Kirtland AFB, NM, March 1976.
4. Rice, M. H., McQueen, R. G. and Walsh, J. M., in Solid State Physics, "Compression of Solids by Strong Shock Waves," F. Seitz and D. Turnbull, Eds., Academic Press, New York, 1958, Vol. 6, pp. 1-63.
5. Lee, L. M., Shock Compaction Behavior of Porous Titanium Hydride-Potassium Perchlorate, Ktech TR-76-06, Albuquerque, NM, July 1976.
6. Graham, R. A., Neilson, F. W. and Benedick, W. B., "Piezoelectric Current from Shock-Loaded Quartz--A Submicrosecond Stress Gauge," J. Appl. Phys., 36, No. 4, 1965, pp. 1775-1783.

Figure 2. Schematic of the projectile and target configuration used to generate the explosive Hugoniot data.

the stress measurement. Impact stress data below 20 kbar were obtained using a steel buffer plate, and above 20 kbar using WC. The buffer plate separated the quartz gauge from the porous explosive sample, which minimized gauge failure caused by local discontinuities at the gauge front electrode. The thicknesses of the sample, buffer plate, and quartz gauge were designed such that the initial stress wave generated at impact could propagate into the quartz gauge without being influenced by side or edge rarefaction waves. The measured experimental quantities included impact velocity and the time resolved current output of the quartz gauge as measured by the voltage across a 50-ohm termination resistor. Impact velocity was determined from time interval readings with ± 0.1 ns resolution, obtained from three counters triggered by charged pins with known distance intervals. Impact velocity was calculated from these data with an estimated maximum error of ± 0.4 percent. The maximum experimental error associated with the quartz gauge measurements was estimated to be ± 3 percent.

2. DATA ANALYSIS

The buffer plate experiments yielded stress-particle velocity data directly, as shown schematically in Figure 3. Continuity of stress and particle velocity across the buffer plate-explosive sample interface requires:

$$\Delta\sigma_s = \Delta\sigma_b$$

and

$$\Delta u_s = IV - \Delta u_b$$

where $\Delta\sigma_s$ and $\Delta\sigma_b$ are the stress changes in sample and buffer plate, respectively, and Δu_s and Δu_b are the particle velocity changes in the sample and buffer plate, respectively. Impact velocity is denoted IV. The stress and particle velocity state reached in the buffer after impact was calculated from the measured quartz

Shot 1253
Impact Velocity = 0.272 mm/ μ s

0.1 μ s/Time Mark
1.5008 Volts Cal.

Shot 1251
Impact Velocity = 0.515 mm/ μ s

0.1 μ s/Time Mark
5.004 Volts Cal.

Figure 3. Representative quartz gauge records from explosive Hugoniot shots and schematic diagram data analysis technique.

gauge stress amplitude, σ_q , and the known Hugoniot for the steel (Ref. 7) and WC* buffer material and quartz crystal. These experiments do not provide explicit information on stress wave shape or the loading path followed by the porous explosive. Measurements of the wave profile after it has propagated through the unreacted material are necessary to suitably define this portion of material response. The stress-particle velocity data from the buffer plate experiments were transformed to the shock velocity-particle velocity and stress-specific volume plane using the Hugoniot equations and assuming a zero Hugoniot elastic limit.

The initial stress jump recorded by the quartz crystal was used to determine the Hugoniot point. The initial stress jump due to impact was clearly defined for Shot 1253 in Figure 3; however, the point was not so easily defined for Shot 1251 because of the increase in stress due to chemical reaction. The approach taken in the data analyses was to use the point defined by the intersection of a line drawn through the initial rise with a line drawn through the second major slope. The analysis technique allowed all of the experimental data to be evaluated; however, when the stress increased at a rapid rate immediately following impact, definition of a discrete Hugoniot point was somewhat subjective.

* Karnes, C. H., Sandia Laboratories, Private Communication.

7. Butcher, B. M. and Canon, J. R., "Influence of Work-Hardening on the Dynamic Stress-Strain Curves of 4340 Steel," AIAA Journal, 2, No. 12, p. 2174.

SECTION IV

EXPERIMENTAL RESULTS AND DISCUSSION

The experimental program provided information defining the shock compaction behavior of porous CP up to 60 kbar, as well as reaction rate information. The impact testing portion of the program was broken into three phases: (1) system check shots; (2) low density explosive Hugoniot shots; and (3) high density explosive shots. The system check shots were fired to verify the experimental design and to check all recording systems. Two check shots were conducted using a plexiglass sample instead of CP. The well documented plexiglass Hugoniot (Refs. 8 and 9) was used in conjunction with the steel Hugoniot to calculate the impact stress amplitude using the measured impact velocity. The calculations were then compared with the quartz gauge results. Differences of 1.1 and 2.0 percent existed between the calculations and data. The degree of nonplanarity at impact was also obtained from these two check shots, with tilt angles of between 1.0 and 1.7×10^{-3} radians being measured. A data trace from one of the check shots is shown in Figure 4.

Figure 4. Data trace from check shot 1230 with steel impacting a plexiglass sample.

8. Barker, L. M. and Hollenbach, R. E., "Shock-Wave Studies of PMMA, Fused Silica, and Sapphire," J. Appl. Phys., 41, No. 10, Sept. 1970, p. 4208.
9. Schuler, K. W. and Nunziato, J. W., "The Dynamic Mechanical Behavior of Polymethyl Methacrylate," Rheologica Acta, Vol. 13, 1974, pp. 265-273.

The stress plateau resulting from chemical reaction was substantially higher in amplitude than any stress level considered during the experiment design. The impact experiments were designed to exercise the buffer plates and quartz gauges in the elastic range. However, the 100-kbar plateau was beyond the elastic range of the tungsten carbide buffer plates used in the experiment. The particular carbide (K-68) responds in a linear elastic fashion to 35 kbar before nonlinear effects are measurable. The propagation speed of a stress increment at 100 kbar in WC is 12 percent less than the elastic propagation speed associated with stresses from 0 to 35 kbar. The net effect of operating the carbide at stresses beyond the elastic limit was an additional spreading of the input wave above 35 kbar as it propagates through the carbide. The buffer plate thickness used in the test program was 3.18 mm. If a 100-kbar stress wave was instantaneously applied to the front surface of the WC buffer, the nonlinear material response would cause a 0.06- μ s spreading of the wave when it arrived at the rear surface of the buffer.

An experiment using a WC target and impactor was conducted to evaluate the transmitted wave shape for a step input of 100 kbar. The results of the test are shown in Figure 5. A slight deviation in the rise of the stress pulse is evident and correlates to the nonlinear behavior of the carbide. The time correction produced by the nonlinear material behavior was not applied to the experimental results obtained on CP because it was small (0.03 to 0.06 μ s) and could not be clearly separated from the effects of nonplanar impact. The results of Shot 1259 clearly show that the nonlinear behavior of WC at 100 kbar did not compromise the validity of the recorded CP stress-time data.

Figure 5. Stress-time data for Shot 1259, using a WC target and impactor to evaluate the transmitted wave shape for a step input of approximately 100 kbars.

The results of the impact experiments on the 1.47 and 1.66 g/cm³ CP samples are given in Tables 1 and 2, respectively. The sample number and type of experiment used to generate the data are also listed. Some of the results listed as Hugoniot data are questionable, as indicated in the tables. Definition of the unreacted CP Hugoniot state was questionable when chemical reaction produced a rapid stress increase immediately after impact. The stress amplitude determined in these experiments, using the data analysis technique described in Section III, was larger than the Hugoniot state for the unreacted material because it was a combination of impact induced stress and pressure due to chemical energy release. Utilization of an artificially high stress amplitude in the Hugoniot relations produced unrealistically large values of shock velocity and specific volume. All of the experimental results for both densities of CP are included in Tables 1 and 2 and plotted in Figure 6. Also shown in Figure 6 are polynomial fits to the Hugoniot data determined using a least squares technique and constraining the fit through the origin. The data influenced by the chemical reaction were not included in the fit, but are shown in Figure 6. The stress-particle velocity relation for the unreacted 1.47 g/cm³ CP is:

$$\sigma = 6.90u + 34.34u^2$$

and for the 1.66 g/cm³ density material:

$$\sigma = 10.55u + 56.28u^2$$

where stress σ is kbar, and particle velocity u is mm/ μ s.

The stress-particle velocity data for the unreacted material were transformed to the shock velocity-particle velocity plane by using the Hugoniot jump equation. Shock velocity was calculated from:

$$U_1 = \frac{\Delta\sigma_1}{\Delta u_1 \rho_0}$$

TABLE 1

SUMMARY OF HUGONIOT DATA FOR LOW DENSITY EXPLOSIVE (CP)

Shot No.	Type of Experiment	Sample Number	Initial Conditions			Hugoniot Data			
			Impact Velocity (mm/ μ s)	Sample Thick. (mm)	Sample Density (g/cm ³)	Stress (kbar)	Particle Velocity (mm/ μ s)	Shock Velocity (mm/ μ s)	Specific Volume (cm ³ /g)
1243	XQ/4340 $\rightarrow$ CP	1	0.511	0.302	1.47	11.6	0.485	1.63	0.477
1244	XQ/4340 $\rightarrow$ CP	2	0.427	0.300	1.48	8.2	0.409	1.35	0.471
1245	XQ/4340 $\rightarrow$ CP	3	0.268	0.305	1.45	---- no data due to gauge failure ----			
1246	XQ/4340 $\rightarrow$ CP	4	0.269	0.302	1.47	4.2	0.260	1.10	0.519
1247*	XQ/WC $\rightarrow$ CP	5	0.693	0.302	1.46	25.9	0.668	2.66	0.513
1248*	XQ/WC $\rightarrow$ CP	6	0.616	0.297	1.49	21.0	0.595	2.37	0.502
1249*	XQ/WC $\rightarrow$ CP	7	0.617	0.302	1.46	18.3	0.599	2.09	0.489
1250*	XQ/WC $\rightarrow$ CP	8	0.695	0.302	<u>1.46</u>	19.0	0.676	1.93	0.444
					1.47 avg.				

* Data point influenced by explosive reaction.

TABLE 2

SUMMARY OF HUGONIOT DATA FOR HIGH DENSITY EXPLOSIVE (CP)

Shot No.	Type of Experiment	Sample Number	Initial Conditions			Hugoniot Data			
			Impact Velocity (mm/ μ s)	Sample Thick. (mm)	Sample Density (g/cm ³)	Stress (kbar)	Particle Velocity (mm/ μ s)	Shock Velocity (mm/ μ s)	Specific Volume (cm ³ /g)
1251	XQ/4340 $\rightarrow$ CP	11	0.515	0.297	1.66	17.3	0.477	2.19	0.471
1252	XQ/4340 $\rightarrow$ CP	12	0.417	0.297	1.65	12.4	0.389	1.93	0.483
1253	XQ/4340 $\rightarrow$ CP	13	0.272	0.297	1.65	6.2	0.258	1.44	0.498
1254	XQ/WC $\rightarrow$ CP	14	0.619	0.297	1.66	27.9	0.591	2.84	0.477
1255	XQ/WC $\rightarrow$ CP	15	0.698	0.297	1.66	30.9	0.668	2.79	0.458
1256	XQ/WC $\rightarrow$ CP	16	--	0.297	1.65	Sample cracked during target alignment--no data.			
1257*	XQ/WC $\rightarrow$ CP	17	0.800	0.297	1.66	51.0	0.740	4.15	0.495
1258*	XQ/WC $\rightarrow$ CP	18	0.796	0.297	<u>1.66</u>	62.1	0.734	5.10	0.516
					1.66 avg.				

* Data point influenced by explosive reaction.

Figure 6. Stress-particle velocity Hugoniot data for porous CP explosive.

where U_1 is shock velocity, $\Delta\sigma_1$ is the change in stress, Δu_1 is the change in particle velocity, and ρ_0 is the initial sample density. Linear fits to the two sets of data were obtained, as shown in Figure 7, with the relations being:

$$U = 0.49 + 2.26u$$

for the 1.47 g/cm³ density material, and

$$U = 0.54 + 3.57u$$

for the 1.66 g/cm³ density material, where U and u are mm/ μ s. The data were also transformed to the stress-specific volume plane using the Hugoniot equation:

$$\rho_1 = \frac{\rho_0}{1 - \frac{\Delta u_1}{U_1}}$$

where ρ_1 is the material density at shock state 1. Both densities of explosive material displayed essentially the same compaction response above 5 kbar as shown in Figure 8.

The data traces recorded on both densities of CP are summarized in Figures 9, 10 and 11 to show the rapid rise in stress following the initial impact. The slope of the stress-time data after the initial step is clearly increasing with increasing impact stress. The dynamic behavior of the high density CP was similar to the low density material. The slope of the stress-time data following the initial stress jump was lower for the 1.66 g/cm³ than for the 1.47 g/cm³ for a given impact stress. The data indicated the more porous, lower density samples were more sensitive to impact initiation than the higher density material.

A high amplitude stress plateau was recorded approximately 0.2 μ s after impact, which was attributed to the chemical reaction taking place in the explosive sample. A nominally 70-kbar plateau was observed in two experiments conducted on the low density CP when the impact stress was above 18 kbar. A

Figure 7. Shock velocity-particle velocity Hugoniot data for porous CP explosive.

Figure 8. Stress-specific volume Hugoniot data for porous CP explosive.

Figure 9. Summary of low density CP impact test data.

Figure 10. Summary of high density CP impact test data.

Shot 1254
Impact Stress
27.9 kbar

0.1 μ s/T.M.

Shot 1255
Impact Stress
30.9 kbar

0.1 μ s/T.M.

Shot 1258
Impact Stress
62.1 kbar

0.1 μ s/T.M.

Shot 1254
Peak Stress
95 kbar

0.2 μ s/T.M.

Shot 1255
Peak Stress
100 kbar

0.2 μ s/T.M.

Shot 1258
Peak Stress
103 kbar

0.2 μ s/T.M.

Figure 11. Summary of high density CP impact test data.

plateau of approximately 100 kbar was recorded in four experiments conducted on the high density CP with impact stresses ranging from 28 to 62 kbar. The experimental data were converted to stress-time at the quartz gauge/buffer intersection, as shown in Figures 12 and 13, with the peak stress values determined from the plateaus listed in Table 3. The experimental data defined the stress-time history at the gauge/buffer plate interface resulting from impact and chemical energy release of the CP sample. The stress-time data shown in Figures 12 and 13 can be used to support reaction rate modeling for CP.

Figure 12. Summary of low density CP stress-time data from impact experiments.

Figure 13. Summary of high density CP stress-time data from impact experiments.

TABLE 3

SUMMARY OF PEAK STRESSES RECORDED IN CP IMPACT TESTS

Shot Number	Sample Number	Sample Density (g/cm ³)	Impact Velocity (mm/ μ s)	Peak Stress * (kbar)
1249	CP #7	1.47	0.617	71.5
1250	CP #8	1.47	0.695	69.9
1254	CP #14	1.66	0.619	94.5
1255	CP #15	1.66	0.698	99.6
1257	CP #17	1.66	0.800	99.8
1258	CP #18	1.66	0.796	103.1

* Stress amplitude at quartz gauge/WC buffer interface.

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

Hugoniot data defining the shock loading behavior of the unreacted explosive were obtained up to 30 kbar. Experiments at higher impact stress levels did not produce unreacted material data because reaction was initiated essentially at impact. Chemical reaction started at stress levels of 12 and 18 kbar for low and high density CP, respectively. Shock induced chemical reaction was expected at relatively low impact stress levels. However, existence of the high amplitude stress plateau following the rapid stress increase was not anticipated. The stress-time data indicated essentially a constant high amplitude stress state was produced by the explosive at the quartz gauge/buffer plate interface. The experimental results provide a unique set of data that can be used to study deflagration to detonation kinetics. The time resolved data showed the material state produced by impact and the increase of interface pressure due to chemical reactions followed by a high amplitude, essentially constant amplitude pressure state. A number of physical processes included in explosive material modeling can be evaluated using the experimental results.

All of the CP samples were consumed during the impact experiment, either by burning or detonation, based on posttest inspection of the test chamber. This result indicated that the samples tested at low impact stress levels reacted at times later than the recording time of the stress transducer. Major stress increases were observed at times greater (1.5 to 3.0) than the one-dimensional recording time of the gauge on the low stress level shots. These results indicate that the sample reaction might have progressed to detonation even for the 3 to 4 kbar impacts. Longer recording time experiments should be conducted at low stress levels to investigate this possibility.

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