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Shock Compression Synthesis of Hard Materials

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**AMARILLO NATIONAL RESOURCE CENTER FOR PLUTONIUM/
A HIGHER EDUCATION CONSORTIUM**

A Report on

Shock Compression Synthesis of Hard Materials

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Shock Compression Synthesis of Hard Materials

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Abstract

The purpose of this research was to adapt the high explosives technology that was developed in conjunction with nuclear weapons programs to subjecting materials to ultra-high pressures and to explore the utility of this technique for the synthesis of hard materials.

The research was conducted in collaboration with researchers at the University of Texas, Texas Tech University and Pantex (Mason & Hanger Corp.). The group designed, modeled, built, and tested a

new device that allows quantitative recovery of grams of material that have been subjected to unprecedented pressures. The modeling work was done at Texas Tech and Pantex. The metal parts and material samples were made at the University of Texas, and Pantex machined the explosives, assembled the devices and conducted the detonations. Sample characterization was carried out at the University of Texas and Texas Tech.

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1. EXPERIMENT

Two conventional explosives have traditionally been used to provide compression in nuclear devices. The early weapons were constructed of formulations based on HMX, tetra(methylenenitramine). This material was replaced by TATB, 1,3,5-triamino-2,4,6-trinitrobenzene in later weapon designs. Our application for these explosives is very similar to the purpose for which they were originally designed, a means of producing intense pressure through shaped charge detonation. The detonation of carefully designed shaped charges can produce enormous compression and temperature pulses. Our intention was to exploit the expertise and the technology that has been developed in the manufacturing and modeling of such devices for use in ignition of nuclear weapons, to adapt the explosives and that technology to generate the high-pressure conditions required for the synthesis of metastable materials. We had to design a new tool (device) for this purpose.

This tool was used to compress C_{60} in an attempt to transform that material into the diamond allotrope. We also used the tool in an attempt to synthesize the cubic phase of carbon nitride. First, principle calculations have predicted that β - C_3N_4 could have a hardness equal to, or greater than that, of diamond. Previous low-pressure synthesis experiments have failed to generate this material on a macroscopic scale. High pressure and high temperature techniques have often been used to generate and study dense and hard solid phases, although their use in the synthesis of carbon nitride has not been extensively explored. Recent theoretical work predicts that the desired β phase of carbon nitride is likely to be more stable at high pressure. Hence, our new shock compression technique provided an exciting potential for generating this valuable, but elusive compound.

Research on the consolidation of powder and synthesis of materials under shock pressure is still in its infancy. The first

report of such research was by the Russian scientists Pashkov, et al. (1979). Reports of pioneering research on shock consolidation of powders in the United States first appeared in the early 1980s in the work performed by Davidson et al. (1982) Schwartz et. al. (1984) and Ahrens et. al. (1986). Regardless of the detailed nature of the research, the major issues that must be considered in performing the shock consolidation and shock synthesis experiments are: (1) the shock delivery system, (2) the size and configuration of the sample, and (3) the sample containment and holding fixture design. The pressing issues are the reproducibility of the experiments, the consistency of shock pressure and temperature achieved, and of course, the recovery of the shocked sample for post-consolidation analysis is a critical issue. The shock delivery systems reported in the literature include planar loading and convergent loading systems. The planar loading techniques use delivery systems such as single stage gas guns, propellant guns, two stage light gas guns, and explosively driven flyer plates as reported by Dodson et. al. (1982), Chabildas and Hills (1986), and Nellis et al. (1986). In these experiments, the shock must produce a controllable, reproducible, high-pressure, and flat shock front.

Sample recovery must be carefully considered in the design of the loading system (Duvall and Graham, 1977). The recovery system is designed to either control the rate of pressure release or to decelerate and capture the sample holding fixture. Recovery systems are generally complex structures that in turn make it difficult to predict or measure the magnitude and direction of the shock delivered to the sample.

Since bulk quantities of C_{60} and other fullerenes have become readily available, many successful experiments have been conducted that have generated diamonds from C_{60} (Hirai, et al., 1993; Sekine, 1992; Bocquillon, et al., 1993; Hodeau, et al., 1994; Regureiro, et al., 1992; Ma, et al., 1994; Yoo,

et al., 1992; He, et al, 1995; Mukhanov et al., 1996; Kozlov, et al., 1996). Regueiro and coworkers (1993) found that the transformation from C_{60} to diamond occurred with "fast kinetics and high efficiency" even at room temperature under "moderate" pressure. Hirai and coworkers' shock loaded C_{60} and recovered diamond crystallites with no evidence of a transition from C_{60} to graphite or reversion to graphite from diamond. Recently, He *et. al.* (1995) reported that C_{60} could be converted to diamond with 100% yield when mixed with nanometer grade nickel and compressed by a one-dimensional plane wave generator. The nanometer grade metal (Kozlov, et al., 1996; Sinfelt, 1977; Du, et al., 1991; Anderson, et al., 1980; Gong, et al., 1991) (metal whose particle size is between 1 nm and 100 nm) was used as a temperature quenching agent, and is designed to suppress any reversion of the high pressure phase during pressure release (Hirai, et al., 1993; Bundy, 1980; Kurdyumov, et al., 1988). The most popular quenching materials have traditionally been metals or alloys from Group VIII due to their high catalytic activity in a number of other processes (Bocquillon, et al., 1993; Kozlov, et al., 1996; Bundy, 1980). This group of metals can not only serve as a quenching material, but can also act as a carbon solvent and thereby promote diamond nucleation (Li, et al., 1993). In particular, Group VIII metals with extremely high surface areas have shown promise as "catalysts" in diamond synthesis by shock compression (He, et al., 1995).

Numerous attempts have been made in the past several years to synthesize β - C_3N_4 in macroscopic amounts. The majority of these synthetic efforts have focused on low pressure, thin film growth. These attempts include sputtering with several variants, (Torng, et al., 1990; Chen, et al., 1992; Nakayama, et al., 1993; Yu, et al., 1994; Yen, et al., 1995; Li, et al., 1995) electron beam evaporation of carbon coupled with nitrogen ion bombardment (Fujimoto and Ogata, 1993; Chubaci, et al., 1993), and delivery of carbon

via pulsed laser ablation to a high flux nitrogen stream (Xiong, et al., 1993; Niu, Lu and Lieber, 1993; Narayan, et al., 1994; Ren, et al., 1995; Lieber and Zhang, 1995; Zhang, Fan and Lieber, 1995; Zhang, et al., 1996). High pressure and high temperature techniques provide an alternative mechanism to probe the formation of the sp^3 bonded carbon nitride. This procedure should be analogous to the synthesis of diamond by shock compression of sp^2 graphite or other carbon containing materials. In fact, recent theoretical studies have suggested that carbon nitride with sp^3 bonding should exhibit greater stability at high pressures (Teter and Hemley, 1996; Badding and Nesting, 1996). However, only a few experimental attempts to synthesize carbon nitride using high pressure have been reported (Sekine, et al., 1990; Badding et al., 1995). No evidence of carbon nitride has been detected in the compression products from these studies. Starting materials have consisted of various organic precursors, with the emphasis placed on the ratio of carbon to nitrogen. We, therefore, chose to investigate the use of the new pressure environment as a possible synthetic route to the elusive, ultra-hard carbon nitride.

Computer modeling was used to assist in the design of an explosive and workpiece configuration that could survive the extreme conditions experienced during the explosion. In this context, the term "workpiece" is used to describe the combination of materials contained within the explosive shell. Dyna East Finite Element Lagrangian (DEFEL) hydro-code was used to determine the best detonation pattern to achieve uniform implosion of the workpiece. DEFEL calculations provided a two-dimensional axisymmetric simulation of the process intended to predict the number and location of the detonators needed to produce the symmetric collapse of a spherical shell. The CTH family of codes was used to accurately model the compression and recovery stages. This three-dimensional code is most commonly used to model the compression required to initiate the

detonation of nuclear weapons. Previous model studies of this process were only concerned with the compression process, since once compression was achieved, nuclear reaction ensued. We had to model both the compression cycle and the important events occurring during decompression.

Modeling the propagation wave provides an estimation of the magnitude and duration of the pressure and temperature pulse delivered to the sample. The magnitude of the pressure pulse is such that it cannot be measured directly by experimental means. It can only be estimated through such modeling. Simulation of the decompression process provided input into the design of a workpiece that could survive the huge tension forces generated during unloading. The simulations allowed us to explore a wide variety of geometries.

Various designs were considered in an attempt to achieve a workpiece that was recoverable after the explosion. Trial 1 was constructed using a pressed 0.25 inch diameter graphite sphere enclosed in a 1.0 inch diameter copper shell. The copper shell was assembled from hemispheres. It was encased in a spherical shell of a 160g explosive that was precisely machined and assembled with epoxy glue. The graphite to diamond transition requires only about 20 GPa. The modeling code showed that the graphite would experience a compression pulse of approximately 150 GPa for 2 microseconds. The calculations showed that the copper shell would experience a tension stress of about 20 GPa on decompression, far exceeding the spall strength (5 GPa) of copper. As predicted, the copper sphere fractured into many pieces (dust)!

The second trial design incorporated a steel shell surrounding the copper sphere and a brass shell surrounding the explosive material. Holes were cut through the brass shell to accommodate the detonators. The steel shell significantly increases the spall strength of the workpiece. (See Figure 1).

The addition of the brass jacket surrounding the explosive was the key to success. It was employed to reduce the magnitude of the tension waves transmitted through the workpiece after compression. Detonation of a spherical shell shaped charge results in the generation of two pressure waves. One is directed inward and results in compression of the carbon sample. The second is an expansion (tension) wave that travels outward from the explosive surface. The brass shell served as a sacrificial structure. It was efficiently destroyed by this expanding pressure wave. Though destroyed, it served to reflect a portion of the expansion wave back toward the sample. This reflected pulse was a compression wave, directed toward the center of the workpiece. The distance from the brass shell to the copper/steel workpiece was precisely controlled such that the reflected compression wave generated from the brass surface reached the surface of the workpiece at the same time as the reflected tension wave that was propagating from the center of the sample. These waves destructively interfered in the workpiece material, thereby reducing the magnitude of the tension waves below the spall strength of the sample holder and preventing failure. The assembled device (Figure 4) was buried in sand. The sand surrounding the workpiece provided a surface that augmented reflection of the expansion waves and it served to contain the sample holder.

Unfortunately, the second compaction experiment resulted in a new failure mode. The copper/steel chamber was recovered in two hemispherical pieces. The workpiece failed at the junction where the metal hemispheres were assembled. This was a dramatic improvement over the first experiment, in which the sample chamber failed catastrophically. The new result showed that the dimension of the gap between the outer brass shell and workpiece was calculated correctly so that the inwardly propagating compression wave was properly timed to destructively interfere with the

outwardly propagating tension wave within the steel shell. The cancellation was sufficient to prevent the steel from fracturing in tension. Although fragmentation did not occur, the workpiece was not recovered intact, resulting in the loss of the carbon sample. The cause of the dissection becomes more evident with a description of the manufacturing process for the workpiece.

The workpiece consisted of five individual pieces: a spherically pressed pellet of C_{60} and two hemispherical shells, one of copper and one of steel. In order to assemble the workpiece, the sample pellet was encapsulated by the copper hemispheres as shown in Figure 1. Figure 2 shows the actual components before assembly. The assembly was placed inside the two steel hemispheres, which were epoxied together at the outer seam to form the final workpiece. It was expected that the high temperature and pressure experienced during detonation would be sufficient to "weld" the two halves together. Analysis of the product showed that unfortunately this junction actually served as a propagation point for failure during detonation.

The third design iteration consisted of a single change from the previous experiment, that being rotation of the steel cover such that its junction was perpendicular to the seam formed by the two copper hemispheres. That workpiece fragmented into four equal, quarter-spherical pieces and the carbon sample was lost. This failure was caused by a phenomenon called "jetting." Machining imperfections preclude the steel hemispheres from fitting together with no resulting gaps. Upon detonation, hot, expanding, high velocity gas accelerated through the gaps and cut through all the material beneath the steel. The high velocity gas has enough energy to cut through all the material beneath the steel. Thus, the copper sphere was cut in half along a plane containing the steel junction. In addition, the copper did not fuse together as predicted, and it failed at its seam. Hence, the four parts.

The fourth workpiece design incorporated a single solid steel sphere designed to minimize jetting. In order to introduce the sample material, a hole was drilled to the center of the steel sphere. The diameter of the drill was the same as the designed diameter of the spherical sample chamber. The drilled section was rounded at the bottom to form a hemisphere. The sample material was inserted into the center of the steel sphere and the hole was then closed with a threaded screw that had the bottom tip hollowed to form a hemisphere. After tightening to the maximum torque possible, the top of the screw was cut, rounded, and smoothed until it was flush with the outer surface of the steel sphere. This resulted in a spherical sample contained in the center of an essentially seamless workpiece as shown in Figure 3. Figure 4 shows what the assembled system looks like just before detonation.

The fourth shot resulted in complete, intact recovery of the workpiece as shown in Figure 5. The markings on the surface of the steel resulted from converging shock fronts generated at each detonator site. Fourteen distinct polygons were formed with the center of each polygon corresponding to the location of a detonator. The spheres recovered from subsequent trials had exactly the same physical characteristics.

The final design subjects the sample to a maximum compression of 230 GPa (as predicted by the CTH code). The duration of this pulse is approximately 4 μ s as shown in Figure 6. A second, less intense compression pulse is evident at between 20 and 30 μ s. This second compression wave is the result of reflection of the expansion waves from the brass shell. The calculations associated with the second peak are complex and somewhat subjective. Therefore, the magnitude and duration of this peak are still being studied. No changes were made to the design for any of the remaining experiments (only the starting materials were varied) so the pressure

Table 1: Summary of Samples Compressed

Diamond Synthesis Experiment	Amount of C ₆₀ ^a (by weight)	Amount and Type of Quenching Material (by weight)
Shot D1	50%	50% -200 mesh copper
Shot D2	50%	50% nanograde nickel ^b
Shot D3	50%	50% -200 mesh nickel
Shot D4	50%	50% nanograde cobalt ^c
Shot D5	20%	80% nanograde nickel ^b
Shot D6	50%	50% nanograde nickel ^b and 0.5 mm diamond seeds
Shot D7	20%	80% -200 mesh nickel [compressed sample]
Shot D8	20%	80% nanograde nickel ^b [compressed sample]
Carbon Nitride Synthesis Experiments		
Shot CN1	2-Amino-1,3,5-triazine - C ₃ N ₄ H ₂	
Shot CN2	Tetracyanoethylene - C ₆ N ₄	

a. C₆₀: 99.5% pure;

b. nanograde nickel: 99.3% pure, particle size of 10-20 nm;

c. nanograde cobalt: 99.5% pure, particle size of 15-25 nm.

delivered to the samples in all shots was the same as that for *Shot D1*.

Eight experiments designed to synthesize diamond and two compactions designed to generate carbon nitride were performed. The starting materials for each experiment are described in Table 1.

In order to extract the sample from the center of the workpiece, the steel sphere was machined into two pieces to expose the sample cavity. This is difficult because the steel becomes extremely hard in areas near the center of the workpiece. The spheres were cut across their entire circumference and sawed radially inward to a point just outside the sample cavity. This process can consume as many as six high speed steel saw blades for a single workpiece! The location of the sample cavity was determined from analysis of an X-ray taken of the recovered workpiece. From the X-ray, the sample chamber appears much lighter than the surrounding steel and the distance from the outer surface of the sphere to the sample chamber can be easily estimated. When the sawing reached the

point when just a thin layer of steel (1/16") was holding the workpiece together, the sphere was "cracked" into two hemispheres using a chisel and hammer. Care was taken to ensure that all particles that were dislodged during this process were recovered for analysis.

The samples were found in one of two states. If the amount of quenching material was low, the sample was recovered in a powder form that could be directly analyzed. If the amount of quenching material was high, the carbon sample was trapped in the molten quenching material during the detonation. Separation of the carbon sample from the metal requires dissolving the metal in an appropriate solvent. The hemispheres can, in principle, be dissolved in boiling HCl and subsequently filtered to provide the carbon product. Stainless steel is not designed to easily corrode, so this process does not easily produce a clean sample. An alternative to dissolving the entire hemisphere was to drill portions of the sample material out of the shell and collect the cuttings. The cuttings

were dissolved in HCl and the resulting mixture centrifuged. The liquid was poured off, leaving only the carbon sample, which was washed to a pH of 7 and then dried for analysis.

X-ray scattering and Raman spectroscopy were used to determine the structural characteristics of the materials recovered from the explosions. X-ray diffraction studies were performed using a Philips PW1729 X-ray generator operating at 40 kV and 40 mA to generate Cu-K α radiation. The diffracted radiation was collected via a Philips APD 3520 scanning detector. A second x-ray analysis for *Shot D7* was done on a Scintag X $_1$ diffraction system (40 kV, 40 mA) with a theta-theta powder diffractometer fitted with a solid state Si(Li) detector. All x-ray samples were prepared by spreading the sample in powder form on glass substrates using amyl acetate (used to help secure the powder to the glass substrate). Spectra were taken using a scan rate of 2°/min, unless otherwise noted.

Raman spectroscopy studies were performed on powder samples using a coherent I-200 Argon laser excitation source irradiating the solid sample at 457 nm. The light was passed through an Applied Photophysics pre-monochromator to filter the plasma lines. The scattered light was collected by a Navitar f/0.95 camera lens and dispersed through a Spex Triplemate three-stage spectrometer containing a 1600

grooves/mm grating. The dispersed light was detected by a Photometrics CH-210 liquid nitrogen cooled CCD. Micro-Raman spectroscopy was carried out on the interior walls of the workpieces as well. Wavelengths of 514.5 nm and 488 nm were generated with an argon-ion laser and were directed on the samples using a backscattering Raman microscope. The scattered light was collected, passed through a holographic notch filter, and dispersed through a single grating monochromator. The light was detected in a multi-channel configuration by a liquid nitrogen cooled CCD detector.

A typical x-ray diffraction pattern of a recovered sample is shown in Figure 7. The (002) diffraction of graphite is evident in the peak at $2\theta = 26.58^\circ$. A typical Raman spectrum of *Shot D1* is shown in Figure 8. The peak at 1570 cm $^{-1}$ is due to zone-center vibrations in graphite and is referred to as the G-band. The peak at 1351 cm $^{-1}$ corresponds to zone-edge vibrations that become Raman active in the disordered and microcrystalline phases of graphite. The Tuinstra and Koenig (1970) model was used to determine the average graphite crystallite size based upon the relative intensities of the D and G bands. The ratio of the areas of these two peaks has been used to estimate a nanocrystallite size of 30nm for *Shot D1*. The results from the ten-compaction experiments are shown in Table 2.

Table 2: Carbon Phases as Determined by X-Ray and Raman Spectral Analysis

Experiment	Product
Shot D1	Nanocrystalline graphite (~ 30 nm)
Shot D2	Nanocrystalline and disordered graphite (~ 25 nm)
Shot D3	No carbon phase detected
Shot D4	Highly amorphous graphite (< 3 nm)
Shot D5	Highly amorphous graphite (< 3 nm)
Shot D6	Crystalline diamond, graphite, and nanocrystalline graphite (~10nm)
Shot D7	Nanocrystalline and disordered graphite (~ 10 nm)
Shot D8	Nanocrystalline and disordered graphite (~ 5 nm)
Shot CN1	Highly amorphous graphite
Shot CN2	No carbon phase detected

Eight compression experiments were conducted with the goal of converting C_{60} to diamond. Diamond was only recovered in the experiment in which diamond seeds were added to the starting material (*Shot D6*). X-ray results showed that some diamond was still present in that sample after detonation. Therefore, the conditions generated are not completely hostile to diamond. The compression in all of the experiments was sufficient to result in the quantitative loss of the fullerene phase. However, only graphitic and amorphous carbon could be recovered. The nature of the compaction produced by this particular workpiece design does not appear to be applicable for the synthesis of the diamond phase.

Two explanations are consistent with the experimental findings: either the carbon samples never entered the pressure/temperature region of diamond stability, or the diamond region of the phase diagram was reached, but the diamond phase was lost due to subsequent phase transitions. We believe that the magnitude of the compression was sufficient to reach the portion of the phase diagram best described as "liquid carbon (Mitchell, et al., 1986; Bundy, 1989). Despite our best efforts to quench the sample into the diamond phase, the design of the workpiece did not allow the temperature to be reduced at the rate necessary to enter the diamond region. It is also possible that even though the sample experienced the pressure/temperature conditions in which diamond is thermodynamically favored, the

time the sample was maintained in this condition was less than that spent within the graphite portion of the phase diagram. This could again be attributed to insufficient quenching, or possibly due to a phase change resulting from the secondary compression.

Two experiments were conducted in an attempt to generate carbon nitride from starting materials containing carbon and nitrogen. The detonation of the shaped explosives did not produce well-crystallized product. Instead, predominantly amorphous material was recovered. These materials are similar to those recovered from the diamond synthesis. The product appears to form while in an undesired region of the phase diagram.

The final shot using this workpiece configuration did not contain a sample. Instead a solid stainless steel sphere was placed at the center of the explosive and detonated. Figure 9 shows that a void was generated in the center of the steel sphere. Hardness measurements show that the material near the void in the center is harder than the starting material.

The pressure pulse delivered to the sample can be altered by changing the amount of explosive and by changing the dimensions of the workpiece. The conditions generated using the current design produce extremely high pressures but is apparently not suited to forming the desired products. Modifying the design could provide the proper compression cycle required to synthesize one or both of the desired ultra-hard materials.

2. CONCLUSIONS

We successfully designed and constructed a workpiece that can withstand the explosion and contain the sample. This device allowed us to recover materials subjected to unprecedented pressures. However, the temperature quenching rates of the current design are insufficient to produce diamond or β -C₃N₄. Graphite and C₆₀ are converted into disordered or nano-crystalline graphite by this process. The extreme compression conditions have produced steel with hardnesses that exceeds that of high-strength steels made by more conventional means.

2.1 Future Work

Extensive hardness testing measurements are still underway. Figure 10 shows Vickers hardness results for the sample (MAH 38) that contained 50% C₆₀ and 50% Ni. The figure shows the hardness dependence as a function of position within the workpiece and sample. We are attempting to correlate hardness with structural characteristics.

A new design has been completed for the work-piece. Our intention was to use this workpiece to produce a super-hard coating on a stainless steel sphere. Figure 11 shows a cross-section of the assembled workpiece. The 2" diameter stainless-steel shell is made in two pieces that are tapped and threaded, respectively. A tungsten carbide powder was pressed into a spherical shell ~ 2 mm thick on the inside of both halves of the workpiece using a spherical die. A stainless steel ball was placed in the center of the cavity, and the two halves were screwed together. Simulations were used to calculate the amount of explosives necessary to produce a uniform compression. Pantex is machining the high explosives necessary for this experiment. Six workpieces are being constructed and may be tested. A design incorporating copper as the workpiece material was also developed. The copper workpiece would facilitate the recovery of carbon phases and the larger geometry would reduce the maximum pressure created at the center of the workpiece. We hope that this configuration will successfully synthesize the diamond phase.

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Figure 1: Second Experimental Design. [Note, only one of the fourteen detonators is depicted.]

Figure 2: Components for the Second Work-Piece Design

Figure 3: Design of the Fourth Compaction Workpiece Consisting of a Solid Steel Sphere which has been Drilled and Tapped to the Center, Filled with Sample, and Closed with a Threaded Screw

Figure 4: Assembled Workpiece and Detonation Wires

Figure 5: Steel Workpiece Recovered from the Fourth Compaction Experiment

The picture on the left shows the location of the steel screw while the picture on the right shows the hemisphere not containing the screw.

Figure 6: Pressure Pulse Duration as Calculated by CTH Code

Figure 7: X-ray Diffraction Pattern of *Shot D7* after Dissolving the Shavings in HCl
 [Note: Pattern was taken on Scintag X-ray diffraction system at a scan rate of 1°/min.]

Figure 8: Raman Spectrum of D1

Figure 10: Vickers Hardness Measurements Using a Micro-Ball Indenter on a Cross-Section of Sample MAH38 [The data shows very high hardness at the inner rim and then decreasing hardness away from the center.]

Figure 11: New Experimental Design