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6. AUTHOR(S) N.N. Thadhani, R. Russell, K. Vandersall				
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13. ABSTRACT (Maximum 200 words) In this program, advanced diagnostic instrumentation was developed for time-resolved pressure measurements for experimentation involving materials synthesis by shock-compression of powders and their mixtures. The instrumentation has been developed with the objective of (a) allowing to determine the shocked state of material in real time with nanosecond resolution, to quantitatively correlate the loading histories with post-shock characteristics of materials produced using recovery experiments; and (b) monitoring the shock-initiated transformation or chemical reaction processes in real time by observing the response of in-situ probes to obtain a detailed understanding of the mechanisms and kinetics of processes occurring during the shock-compression state. The instrumentation developed is based on time-resolved stress-wave measurements using the piezoelectric polyvinylidene-fluoride (PVDF) gages, and includes: two Tektronix 784A digital oscilloscopes (1 GHz bandwidth, 1 GS/s rate, and 4 channels) and the associated hardware including high-frequency low-loss coaxial transmission cables, HP pulse and delay generator, HP electronic counters, and an independent circuitry several sets of gages. The instrumentation will be extensively useful for enhancing the quality of on-going research supported by ARO under Grant No. DAAH04-94-G-0192, entitled "An Investigation of the Mechanisms, Kinetics, and Energetics of Shock-induced Chemical Reactions in the Ti-Al/Si/B System," and the AASERT program on "Combustion Synthesis of High-density Powder Mixture Compacts under Conditions of Dynamic Stress and Electric Field"				
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**DURIP/ARO PROGRAM ON
TIME-RESOLVED PRESSURE-MEASUREMENT
INSTRUMENTATION FOR
HIGH-STRAIN-RATE MATERIALS SYNTHESIS**

FINAL REPORT

N.N. Thadhani, R. Russell, K. Vandersall

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1. INTRODUCTION

Synthesis of materials by shock-compression of powders and powder mixtures has become a subject of increasing attention, as a non-equilibrium process having the potential of yielding novel compounds and metastable phases [1-4]. The unusual combination of high pressure and high-loading rates, producing the large amounts of strain in powders, can not only lead to consolidation of metal and ceramic powders [5], but solid-state phase transformations [6] and chemical reactions [7], can also occur during the microsecond duration of the shock-compression state. Thus, materials with non-stoichiometric compositions, non-equilibrium phases, and unique/modified microstructures can be synthesized [1,4].

The lack of a fundamental understanding of the shock-synthesis mechanisms has limited the advances in the development of shock-compression as a viable materials processing approach. It has been difficult to attain a detailed understanding of the mechanistic processes that lead to synthesis of materials during high-strain-rate loading, because of problems associated with correlating the shock response of the material with shock-loading histories. Particularly in the case of porous materials (powders), the correlation of shock response with loading histories has been most ambiguous. This is not only due to difficulties in interpreting the information generated from available test and measurement techniques, but also, because the shock response of powders is uniquely different. Unlike in solid materials, shock waves in powders may be highly dispersed (i.e., have long rise times) [8,9], making shock-compression to be less than a truly hydrodynamic process. Shock wave propagation velocities in powders are also considerably slower than in solids. Furthermore, shock compression of powders can result in a large amount of energy being dissipated in plastic deformation in the process of void annihilation, giving rise to significant increases in temperature. Increases in temperature are also possible if exothermic chemical reactions occur during shock-compression of powder mixtures. The resulting effects, therefore, produce microstructures that are reminiscent of cooling from high temperatures rather than the effects of shock compression.

However, shock compression of powders and powder mixtures also result in various types of unique mechanical, physical, and chemical effects [10,11]. A large number of defects are introduced in the powders due to the kinetic energy of the shock pulse. Extensive plastic deformation, fluid-like turbulent flow, heating, particle comminution, and mixing of constituents with fresh and cleansed surfaces is possible. These effects significantly alter the mechanical, physical, and chemical characteristics of powders, thereby enhancing their solid-state reactivity [12]. The highly reactive states produced can, thus, undergo chemical reactions or phase transitions by mechanisms different from conventional processes involving formation and growth of the nuclei from the liquid or the solid, thereby resulting in synthesis of materials with highly refined microstructures.

Therefore, a need exists to: (i) develop in-situ time-resolved diagnostic techniques to probe the phase transformation and chemical reaction processes occurring during the

shock-compression state to determine the mechanistic and kinetic aspects of these phenomena, and (ii) develop methods that allow better correlation of the loading histories with the shock response of powders and characteristics of recovered shock-compressed materials. In this DURIP program, a unique facility was developed involving advanced diagnostic instrumentation for time-resolved pressure measurements combined with shock-recovery experiments, for applications relevant to materials synthesis by shock-compression of powders and their mixtures. The time-resolved instrumentation is based on stress-wave measurements using the piezoelectric polyvinylidene flouride (PVDF) polymer gages. A two-dimensional hydrodynamic code (AUTODYNE-2D) has also been acquired to design and develop reliable shock recovery fixtures which can be adapted to the Georgia Tech gas gun facility and used with good reproducibility. While the hydrodynamic code will be used for predicting shock conditions in the fixtures, the design of the fixtures will also include placement of embedded gages to monitor the stress-histories at prescribed positions. This will allow direct correlation of numerically simulated shock conditions with the actual measured shock histories in the recovery fixtures.

2. INSTRUMENTED EXPERIMENTS WITH REAL-TIME MEASUREMENTS

Analysis of shock-compression induced microstructural or chemical changes can be performed by post-shock characterization of the recovered products. However, post shock analysis possesses the inherent limitation that the observed phenomena may not necessarily be attributed to shock compression effects alone, since the recovered materials have been exposed to both loading and unloading effects. Direct time-resolved (in-situ) measurements allowing real-time observations of the shock phenomena are, therefore, essential to distinguish the effects occurring under shock-compression, unloading, and at later times. Time-resolved materials response diagnostics have been based on direct measurements of stress and particle wave profiles, enabling studies of the mechanical response with time resolutions approaching 1 ns. Capabilities are also being developed to provide direct measurements of the rate of change of stress. With such measurement of derivative functions, detailed studies of rate-dependent phenomena are possible. Spectroscopic and diffraction based techniques for direct measurements of transformation/reaction phenomena have also been successfully attempted [13] in recent years.

The devices that have been widely used for measurements of pressure or particle wave profiles for materials science studies can be categorized by the physical phenomena utilized in the measurements, e.g., piezoelectric, piezoresistant, electromagnetic, or optical. In each case the phenomena provide a signal corresponding to the shock-compression response, with time resolutions of 1 nanosecond. The piezoresistant stress gages have active sensing elements of manganin, ytterbium, or carbon and respond to shock pressures by changes in electrical resistivity. Manganin gages are typically the high pressure piezo-resistive gages and have been successfully applied for stresses up to 30 GPa, with a resolution of 50-100 ns. Piezo-resistive gages are embedded in the material and are protected from the surrounding by insulating

materials. Piezoelectric stress gauges have active sensing materials of crystalline quartz, lithium niobate, or of the piezoelectric polymer polyvinylidene difluoride (PVDF). Piezoelectric materials generate electric current when stressed, thus, these gauges do not require an external power supply. The piezoelectric gauges can be employed either as thin-element or thick-element configurations. In the thick-element gauges the entire shock pulse occupies only a fraction of the gauge thickness, while in the thin-element configuration, the gauge thickness is much smaller than the thickness of the pulse travelling through it. In particular, the PVDF piezoelectric gauges developed by Bauer are very thin (25 μm), thus, just like in the case of piezo-resistive gauges, they can be used as totally embedded material probes [14]. Electromagnetic gauges provide a measure of the integral of stress, and include electromagnetic particle velocity and axisymmetric magnetic gauges. They are based on Faraday's law of induction and rely on the electromagnetic force (emf) generated as a conductor moves in a magnetic field.

Optical devices are the most widely used systems in fundamental studies of explosives, and have perhaps provided most of the available shock data. The earliest approach utilizing the flash gap and mirror systems provided discrete or continuous measurements of displacement versus time. VISAR systems (velocity interferometer system for any reflector) have become the most powerful modern device for measurement of the shock response. Optical systems such as the VISAR are based on interference fringes produced when different laser beams interact. The fringes are then related to the change in velocity of the reflecting surface. Visar systems have accuracies of ~2% for peak surface velocities of about 100 m/s and even better at higher velocities.

3. CURRENT EXPERIMENTAL CAPABILITIES

3.1 Georgia Tech high-strain-rate facility

The Georgia Tech high-strain-rate impact facility includes an 80 mm (barrel) diameter compressed-gas gun, built and designed by Physics Applications, Inc., in Dayton, Ohio. The gun was purchased at a cost of \$ 105,000 using the principal investigator's research start-up funds. A digital oscilloscope, vacuum system, and a lapping machine for sample preparation have also been acquired since then. The gun is routinely used for several types of shock recovery experiments. The single-stage cold gas gun has an 8000 mm long launch tube connected to a 28.5 liter gas chamber via a double diaphragm. The gun is equipped with a novel design of a catcher tank, allowing the capability to perform well-controlled recovery experiments at ambient and other selected environmental conditions (low temperature or gas pressure). Impact velocities of up to 1000 m/s are possible with a 500 g projectile. Predictions of shock conditions is obtained using the AUTODYNE-2D computation program [15].

A key to performing well-controlled shock-recovery and instrumented experiments is the need to ensure reliable impact planarity and velocity measurements. A newly designed muzzle (by Mark Anderson at Sandia National Laboratories), allows holding the test fixture on to the gun barrel with a method that permits careful alignment of

the fixture and measurement of the impact velocity and planarity using four sets of velocity pins. It includes a sacrificial plate on to which the test fixture is fastened. The contact surface is lapped in between experiments, with the sacrificial plate needing replacement perhaps after every 50 experiments. With this attachment well-controlled and reproducible experiments with impact planarity better than 50 milli-radians and velocity measurements, reproducible within 3% over velocity range of 100 to 1000 m/s, can be performed. Impact velocity measurements are performed using 4 sets of velocity pins, connected to electronic counters and a digitizer (for redundant measurements).

3.2 Instrumentation Developed

The instrumentation developed includes capabilities to perform stress wave measurements using the piezoelectric PVDF gages. The following two sets of instrumentation were developed.

(a) Instrumentation for performing planar-impact experiments with reliable measurements of impact velocity and planarity: This task was accomplished with a new sample-holding muzzle designed and fabricated at Sandia National Laboratories. Three high-performance **electronic counters** (HP Model 53131A, with a two-channel frequency and time measurements to 225 MHz and time-interval resolution of 500 ps) were purchased for impact velocity/planarity measurements using velocity pins.

(b) Instrumentation for monitoring the response of PVDF gages: In order to monitor the current generated due to applied stress for subsequently determining the stress-wave profile via integration of the current-versus-time profile, two sets of very fast digitizers were acquired, in addition to a pulse/delay generator, and a set of ~ 50 foot length, high-frequency, low-loss transmission cable plant. The cable plant is the most important, and often most commonly overlooked, part of successful data acquisition. Since, the recording digitizers are placed in the control room at a distance of ~50 feet from the target holding device on to which the PVDF gages are mounted, it was essential that the cables not be the bandwidth limitation of the recording system. Thus, with a design goal of a 1 nanosecond rise-time (0-95%) at 1 GHz for the cables, eight Andrews LDF5-50 cables (50 foot long) and sixteen connectors (one at both ends of cable) were acquired. The cable diagnostics that need to be used, consists of a Stanford Research Center's Digital Delay/Pulse Generator (Model No. DG535) that sends a 1 nanosecond rise-time, 10 nanosecond wide pulse down a spare transmission cable from the control room to the target chamber, then returns via short disposable cables at the target, and the appropriate transmission cables. This method of pulsing the cables identifies faulty or damaged cables and connectors, as well as verifying the proper cable connections from the target to the digitizers. The delay/pulse generator is also used for controlling the detection window of the digitizers. Two Tektronix digital oscilloscopes, Model TDS784A (1 GHz bandwidth), which sample twice as fast as the DSA digitizers available a couple of years ago, were purchased for the type of nanosecond, time-resolved PVDF measurements, that need to be performed. With the two TDS784A's, we have the capability to record two gauges (four channels) at 0.5 nanoseconds per point.

3.3 PVDF-gage Stress-wave Measurement with Nanosecond Resolution

A capability for direct time-resolved stress-wave measurements using PVDF gages has been successfully set-up with our gas-gun facility, for real-time observations of shock-compression phenomena. The piezoelectric PVDF gages have been successfully used for measurements of stress wave profiles with pressures ranging from 0.3 GPa to 25 GPa and time resolution of the order of a nanosecond. Furthermore, because these are very thin gages ($\sim 25 \mu\text{m}$), they can be used as embedded material gages. Our objective of using the piezoelectric PVDF gages is to both, use them as embedded gages to simply monitor the amplitude of the stress wave, and also to use the gages as probes placed in front of and behind a powder sample, to monitor the input and propagated stress waves, as well as the speed of the stress wave through the powder sample sandwiched between the two sets of gages. The latter situation is particularly useful for monitoring the changes in pressure or wave speed associated with the phase transformation or chemical reaction phenomena occurring during shock-compression.

The first use of the Bauer piezoelectric polymer PVDF stress-wave gages for measurements of stress-wave profiles in 50-60% dense powder-mixture compacts was performed in mid-June. The powder-mixture sample was pressed directly into the copper capsule with PVDF gauge packages placed in intimate contact with powder (at opposite surfaces), to monitor **input-shock** and **propagated-shock** wave characteristics. Typical PVDF gauge package configurations consisted of insulating films of FEP Teflon on both sides of the $25 \mu\text{m}$ PVDF elements, with Al sputtering of 2000 \AA on powder sides of the gauge package to prevent pyroelectric effects from affecting gauge response during possible reaction of powder mixture. The gages were of high quality, biaxially stretched PVDF film, poled using Bauer process to a $9.2 \mu\text{C}/\text{cm}^2$ remnant polarization, and having identical gold over platinum electrodes [14]. The capsule was placed on the impact surface of the gun, in which a copper-faced, smooth-bored projectile was accelerated to a pre-selected impact velocity. The target assembly is shown in the schematic in Figure 1 (a-e). Representative traces of the gauge output in current versus time and the integrated trace of stress versus time for gauge packages located at both **input-shock** and **propagated-shock** locations are shown in figure 1 (b-e).

The shock wave produced upon impact enters the capsule from the left, as shown in the schematic in Figure 1 (a). It propagates through the powder sample with PVDF gauge packages monitoring the **input-shock** and **propagated-shock** wave profiles, and identifying the transit time between the two gauge locations. The input PVDF gauge generates a piezoelectric current as the shock wave transits the gauge, with a rise time less than the shock transit time through the $25 \mu\text{m}$ film thickness. The input shock propagates through the $125 \mu\text{m}$ Kel-F insulation film to the Kel-F/Ti-Si powder mixture boundary, where a reflection is caused due to the impedance mis-match between the Kel-F and the powder. The reflected release wave then arrives back at the input PVDF gauge $\sim 40 \text{ ns}$ after the initial input shock as shown in Figure 1 (b). The piezoelectric current is recorded with two complementary amplifier sensitivities connected to a current viewing resistor at the PVDF gauge. The combined recordings of both amplifiers provides a high resolution current-versus-time profile which is then

numerically integrated and converted to stress-versus-time (Fig. 1(d)) using the PVDFSTRESS computer code [16]. The shock wave after passing through the 4 mm thick powder sample arrives at the **propagated-shock** PVDF gage location as a dispersed loading wave and generates a piezoelectric current with magnitude and duration altered by an order of magnitude from the input gage, as shown in Fig. 1 (c), and the resulting stress-versus-time profile in Fig. 1 (e).

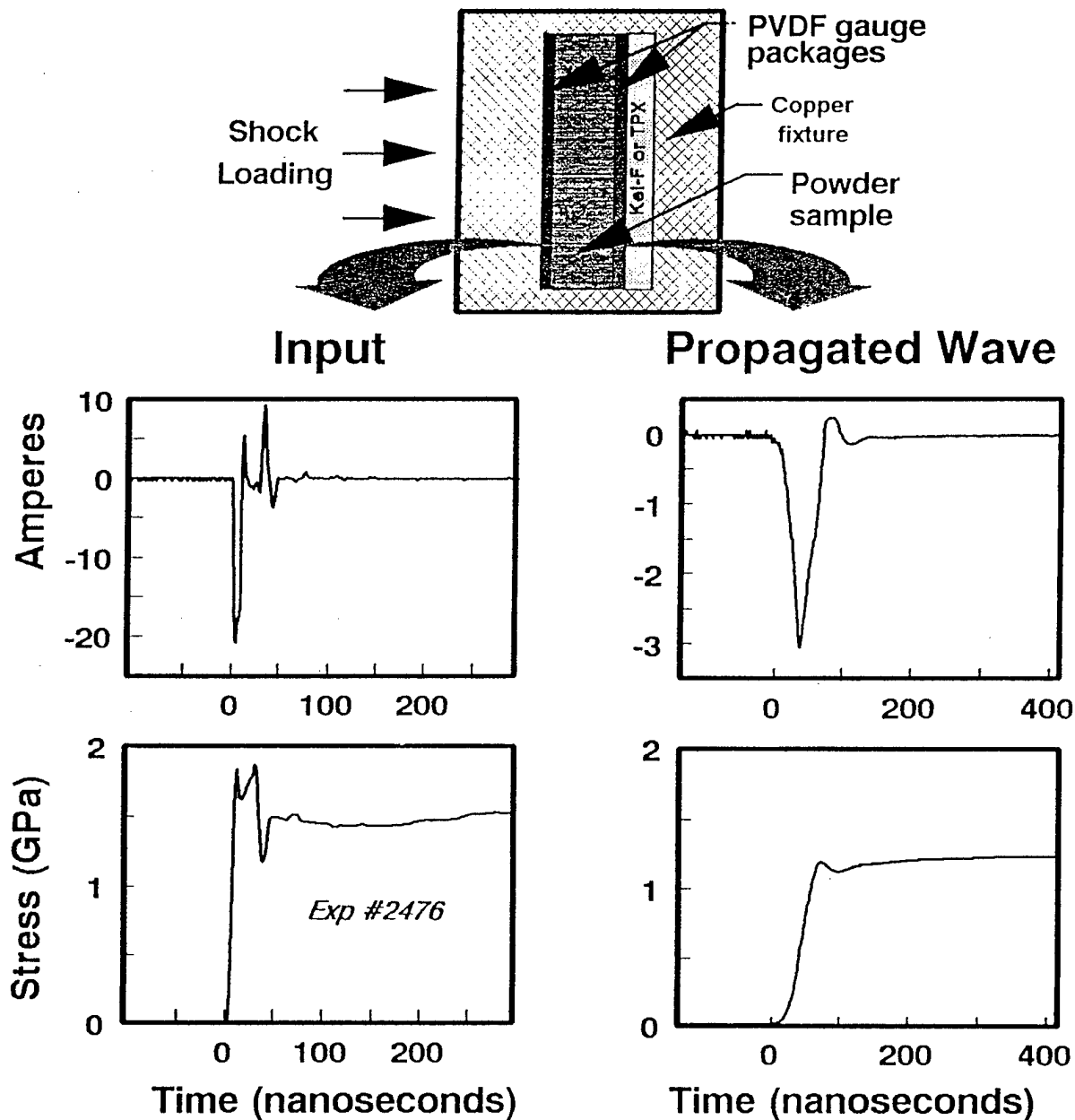


Figure 1. (a) Typical configuration for instrumented experiment showing powder sample encased in copper capsule, with PVDF gauges at input and propagated shock locations in direct contact with powders; (b) and (c) characteristic current pulses generated by PVDF gauges, measuring wave velocity and stresses at input and propagated shock locations; (d) and (e) Corresponding stress profiles obtained by numerical integration of current pulses in (b) and (c).

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