

LA-UR- 05-0241

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Title: High Strain Rate Compression Testing of Ceramics and Ceramic Composites

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Submitted to: 29th International Conference on Advanced Ceramics and Composites
Cocoa Beach, FL
January 24 – 28, 2005
Paper # CB-FS1-31-2005

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HIGH STRAIN RATE COMPRESSION TESTING OF CERAMICS AND CERAMIC COMPOSITES

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ABSTRACT

The compressive deformation and failure behavior of ceramics and ceramic-metal composites for armor applications has been studied as a function of strain rate at Los Alamos National Laboratory since the late 1980s. High strain rate ($\sim 10^3 \text{ s}^{-1}$) uniaxial compression loading can be achieved using the Kolsky-split-Hopkinson pressure bar (SHPB) technique, but special methods must be used to obtain valid strength results. This paper reviews these methods and the limitations of the Kolsky-SHPB technique for this class of materials.

INTRODUCTION

The Kolsky-split-Hopkinson pressure bar (Kolsky-SHPB) technique was originally developed to characterize the mechanical behavior of ductile materials such as metals and polymers where the results can be used to develop strain-rate and temperature-dependent constitutive behavior models that empirically describe macroscopic plastic flow.¹ The flow behavior of metals and polymers is generally controlled by thermally-activated and rate-dependent dislocation motion or polymer chain motion in response to shear stresses.

Conversely, the macroscopic mechanical behavior of dense, brittle, ceramic-based materials is dominated by elastic deformation terminated by rapid failure associated with the propagation of defects in the material in response to resolved tensile stresses. This behavior is usually characterized by a distribution of macroscopically measured failure strengths and strains. The basis for any strain-rate dependence observed in the failure strength must originate from rate-dependence in the damage and fracture process, since uniform, uniaxial elastic behavior is rate-independent (e.g. inertial effects on crack growth).² The study of microscopic damage and fracture processes and their rate-dependence under dynamic loading conditions is a difficult experimental challenge that is not addressed in this paper.

The purpose of this paper is to review the methods that have been developed at the Los Alamos National Laboratory to perform valid, uniaxial, dynamic compression experiments on brittle materials using the Kolsky-SHPB technique and to emphasize the limitations of this technique. Kolsky-SHPB results for several ceramic and ceramic-metal (cermet) materials of interest for armor applications have been measured and show little or no strain rate sensitivity compared to quasi-static compression results.

KOLSKY-SHPB TEST LIMITATIONS FOR HIGH-STRENGTH, BRITTLE MATERIALS

The Kolsky-SHPB technique consists of a pair of long “pressure bars” used to rapidly load a specimen sandwiched between them by a loading pulse applied to one of the pressure bars by a smaller “striker bar”. The classic technique is thoroughly described elsewhere and only highlights or modifications applicable to high strength, brittle materials will be addressed below.¹ A general-purpose Kolsky-SHPB system was used for this study and consisted of 12 mm diameter by 122 cm long pressure bars made of high-strength maraging steel with a nominal

yield strength of 2 GPa; however, this paper also applies to systems with different bar dimensions (or even different bar materials) that have been optimized for testing high-strength, low-failure-strain brittle specimens. Load and displacement are conventionally measured using strain gages mounted on the two pressure bars and used to calculate the applied strain and strain rate and the resulting stress response of the specimen. It is essential that the pressure bars remain completely elastic during the test because elastic analysis is used in the data reduction and because the pressure bars can be damaged by plastic deformation. In particular, the stress generated at the pressure bar-specimen interface must remain below the yield strength of the pressure bars.

A universal condition for valid Kolsky-SHPB testing is that stress-state equilibrium must occur by “ringing up” the specimen stress during the test so that uniaxial stress conditions are achieved throughout the specimen. Early in the test (with respect to time or strain), the stress and strain within the specimen are not uniform and can vary significantly from one end to the other. Hence prior to achieving stress-state equilibrium, the measured specimen stress and strain do not represent uniaxial stress conditions and are therefore invalid measurements until equilibrium is achieved. Because brittle materials fail at extremely low strains, great care must be taken to specially design specimens and choose strain rates that ensure stress-state equilibrium is achieved during the test. A minimum “ring-up time” is required that is approximately equal to three times the specimen length divided by the sound speed of the specimen material (a “ring-up strain” is similarly defined as the product of the ring-up time and the applied strain rate).^{1,3}

Stress-State Equilibrium Considerations

To perform a valid uniaxial compression experiment on brittle materials using the Kolsky-SHPB technique, the specimen length and the applied strain rate must be restricted relative to the strain-to-failure and the sound speed of the specimen material through the ring-up time. Fortunately, the sound speeds of ceramics are very high which aids the rapid attainment of ring-up. Assuming a sound speed of 9.5 km/s (the minimum for materials used in this study), a strain-to-failure of 0.8% under compression (conservative for both dense ceramics and cermets^{4,5}), and a strain rate of 2000 s⁻¹ (readily achievable for conventional Kolsky-SHPB systems), then the maximum specimen length that should be used to ensure stress-state equilibrium is 12.7 mm (with a corresponding minimum ring-up time of four microseconds).

The specimen geometry was therefore designed around a length of 12.7 mm for nominal applied strain rates up to 2000 s⁻¹. For ceramics with the highest sound speeds (~14 km/s) this specimen length allows tests to be conducted at strain rates up to 3000 s⁻¹. Note that further reducing the specimen length involves increasingly difficulty machining to maintain precise dimensional tolerances (especially for the complex geometry discussed later) and samples a smaller volume of material that can change the measured strength distribution. Therefore, the maximum practical strain rate for ceramics based on specimen length is no more than about 5000 s⁻¹ (additional constraints involving in situ strain gage size and data acquisition will be discussed later).

Pressure Bar Maximum Stress Considerations

In order to test advanced ceramics with compressive strengths as great as 8 GPa, the stress at the pressure bar-specimen interface must be reduced by at least a factor of four so that the yield strength of the pressure bars (nominally 2 GPa) is never exceeded. Either high-strength loading platens and/or a reduced-gage-diameter or “dumb-bell-shaped” specimen have been used

for this purpose. Therefore, the ratio of the maximum to minimum diameters of the dumb-bell-shaped specimen or the platen-specimen combination must be ≥ 2 .

The geometry and material for loading platens must also be carefully chosen. First, their length must be short to allow rapid stress-state equilibrium to be achieved in the specimen (as discussed above). However if the platens are too thin, then they will not effectively reduce the stress at the bar interface. Second, if a constant diameter cylindrical specimen is used between platens with different elastic properties, then a non-uniform stress-state will be generated within the specimen instead of a uniaxial stress-state invalidating the stress-strain data. Finally, the ceramics of interest for armor applications are extremely strong in compression so that loading platens need to be made from materials with equal or greater strength. Therefore, to avoid these problems, the obvious solution is to make platens from the same material as the specimens and to taper one end of the platens down to the specimen diameter to minimize stress concentrations in the specimen. In effect, this creates a “segmented” dumbbell-shaped specimen, but with multiple, undesirable interfaces. Each interface surface must be machined to very precise flatness and parallelism (typically at least ± 5 micrometers), which can be very costly and is a potential source of test variability. In addition, a set of platens are likely to sustain damage during the test and must be discarded after a single test. Therefore, a one-piece, reduced-gage section (dumb-bell-shaped) specimen is a very favorable geometry for achieving a high, uniform, uniaxial stress state in the specimen gage section, while it can sufficiently reduce the stress developed at the pressure bar-specimen interface by using a minimum-maximum diameter ratio of two. This approach avoids significant interfacial tensile stresses within the specimen, stress gradients within the high stress gage-section, the costly machining of expendable platens, and finally a one-piece specimen is relatively easy to manipulate and align in the vertical loading configuration of a conventional Kolsky-SHPB system.

The Kolsky-SHPB Compression Specimen Design

The dumb-bell-shaped specimen designed and developed by Tracy⁴ for quasi-static compression testing was modified shortly afterwards by the author⁵ by proportionally scaling the dimensions down to the desired overall length of 12.7 mm necessary to achieve dynamic stress-state equilibrium as discussed above. Downscaling results in a specimen gage diameter of 2.2 mm and a nominal gage length of 3.3 mm. The gage length to gage diameter ratio (L/D) of 1.5 is ideal for general compression failure by either resolved shear or tensile stresses while avoiding buckling failure. Note that the same specimen design was used for both dynamic and quasi-static compression testing so that size effects were eliminated.

Specimen Machining Considerations

The small, intricate geometry and high-precision dimensional specifications of the dumb-bell-shaped specimen make it challenging and costly to fabricate compared to a simple bar or right-circular cylinder, especially for very hard ceramics that require abrasive grinding. Ceramics and brittle materials are also susceptible to premature fracture initiation from surface damage caused by improper machining, so that careful specimen fabrication is a critical step for obtaining accurate, high-quality strength data.

Specimen in this study were fabricated by first cutting and grinding flat a sheet of stock to the final specimen thickness that allows multiple specimens to be created. Individual specimen blanks were then roughed-out using electro-discharge machining (if the material was sufficiently conductive) or by slicing the sheet into rectangular bars. The individual blanks were then

mounted onto a lathe by pressure-padding the ground ends and the final contours were abrasively machined using a precision template. Grinding perpendicular to the compressive loading axis minimizes axially-aligned damage and the probability of failure from this extrinsic flaw source.

KOLSKY-SHPB COMPRESSION TEST METHODOLOGY FOR BRITTLE MATERIALS

Specimen Strain Measurements

The major shortcoming of the dumb-bell-shaped specimen under both quasi-static and dynamic loading conditions is that the uniaxial strain in the reduced gage-section cannot be determined using “far field” displacement measurements (i.e. Kolsky-SHPB reflected bar strain gage analysis) because strain from the entire specimen is averaged and is therefore not representative of the gage section. Furthermore, strain calculated using the Kolsky-SHPB reflected bar strain gage signal is highly uncertain at low strains due to the large amplitude oscillations associated with the leading edge of the reflected wave. Therefore, in situ strain gages mounted within the gage section are necessary to obtain the appropriate strain data. The use of specimen strain gages has drawbacks for dynamic testing: they are time-consuming and difficult to apply, faulty mounting can compromise their accuracy, and they require expensive high-speed signal conditioning and data acquisition equipment. Therefore, the use of strain gages is another significant cost factor associated with this test methodology.

The selection, mounting, and application of strain gages is a substantial subject area and only key aspects will be discussed below. The strain gages selected for this application were stock 120-ohm resistance, general-purpose, miniature gages (designated EA-06-050AH-120) and were manufactured by Measurement Group, Inc. (Raleigh, NC). These gages consist of a constantan foil grid with a polyimide backing that are capable, if properly mounted, of measuring maximum strains of 3-5% (far greater than the largest ceramic or cermet failure strains measured to date). The active grid length and width are 0.05” (1.27 mm) and 0.04” (1.02 mm), respectively. This miniature size allows the strain gage to be centered and fully contained within the specimen gage length. The length of the active grid also governs the time resolution of the strain gage signal. Assuming no influence from the backing or adhesive, the time resolution can be defined as the time required for a stress wave to traverse the entire length of the active portion of the strain gage grid (i.e. the grid length divided by the sound speed of the specimen material). Therefore, the maximum time resolution of the strain signals in this study is calculated to be about 0.1 microseconds (for boron carbide) and is equal to the sampling time used for data acquisition of the strain gage signals (discussed below). Furthermore, the maximum strain resolution (defined as the product of the strain rate and the time resolution) is about 0.00025 (or 0.025%), assuming an applied strain rate of 2500 s^{-1} . This level of resolution of strain is tolerable, but to achieve an improvement (or if significantly higher strain rates are employed) requires the use of even shorter strain gages in combination with higher data acquisition sampling rates.

Three axial strain gages per specimen were generally used for several reasons. First, multiple gages allow the strain signals to be averaged for higher measurement confidence compared to a single measurement. Second, three gages provide substantial redundancy in case one or two gages prove to be faulty. Finally, three gages placed uniformly (at 120 degrees) around the gage section circumference allow loading eccentricity to be evaluated such as due to bending. Bending is an undesirable condition because it causes a non-uniform stress state in the specimen and can invalidate the test if it is greater than the measurement uncertainty. For

example, during a failure test one strain gage signal will often rapidly drop to near zero output first and act as a “break” gage signifying the precise time and strain of failure in the specimen. Both axial and transverse strain gages were used in some cases to explore the volumetric damage and failure behavior of cermet materials. However, only one axial gage can be mounted if transverse gages are used, due to space limitations in the gage section.

The relative output of a strain gage is defined by the gage factor (GF) and specified by the manufacturer for each lot of strain gages, including an accuracy range. The gage factor of constantan gages is nominally $2.05 \pm 2\%$, so the measurement accuracy of the gages used in this study without consideration of various aspects of the actual test method and the instrumentation is only considered “moderate” by the manufacturer at a level of $\pm 2\text{--}4\%$. To maximize reproducibility for a given test, all three gages were selected from the same manufactured lot; however the total uncertainty associated with the dynamic strain measurements was estimated to be about $\pm 5\%$.

The small strain gages used in this study were difficult to mount with precision and reproducibility onto the dumb-bell-shaped specimens (Note that the polyimide backing of the stock strain gages is several times larger than the active gage and the excess is trimmed off prior to mounting). Curvature is high in the specimen gage section due to the small diameter, so that thumb pressure was used to achieve a uniform adhesive thickness over the entire gage surface and to accelerate curing of the adhesive during mounting. Precise alignment of the strain gages to the specimen axis is critical for obtaining accurate axial strain measurements. Strain gages were first precisely aligned on a strip of transparent tape and then the tape was carefully aligned with the specimen axis to within several degrees during mounting. The reduction in the measured versus actual axial strain associated with a two degrees gage misalignment angle is less than 0.2%. If a gage was found to be misaligned by more than two degrees after mounting, then it was removed, the specimen surface re-prepared, and a new strain gage was applied (a difficult and tedious process when applying multiple gages close together on the specimen). M-Bond 200 (Measurement Group, Inc. Raleigh, NC) is a special cyanoacrylate (“super glue”) certified by the manufacturer for use in bonding strain gages. It was chosen to mount the specimen strain gages because it is very thin, fast-setting, and strongly bonding. The recommended curing schedule is one minute of thumb pressure, followed by a minimum two-minute delay before tape removal. Bond strength then increases rapidly during first five minutes. All mounted gages were resistance tested and evaluated for bonding defects and alignment using optical microscopy. Unfortunately, proper bonding of the gages to the specimen (i.e. high bond strength and uniform bond thickness) cannot be entirely verified prior to testing. The output of strain gages mounted to a specimen can be partially checked prior to testing by carefully loading specimens quasi-statically to very low stress levels; however, this was not routinely done for several reasons. First, pre-loading specimens may introduce undesirable changes in the specimen, such as damage or crack growth. Second, this procedure requires high-precision loading equipment and is labor-intensive and therefore costly. Finally, satisfactory strain gage performance at low strain levels did not always ensure proper gage function at higher strain levels.

Each of the three strain gage signals on a specimen was recorded and evaluated separately for consistency. Occasionally one or more of these gage records was disregarded when it was obviously faulty. Variations in stress within the gage section of the specimen due to eccentric loading were calculated when three strain gage signals were available. Typically, these indicated the presence of very small bending moments; however, the strain variations were usually within the uncertainty of the strain measurements, so that these could be artifacts of the

uncertainty. Note that the peak strength measurement without any strain data is meaningful, but incomplete.

High Speed Signal Conditioning and Data Acquisition

The measurement of Kolsky-SHPB pressure bar and in situ specimen strain gage signals requires specialized signal conditioning and data acquisition equipment capable of high bandwidth (≥ 3 MHz), high precision (≥ 10 bits), and high sampling rates (≤ 0.1 microseconds per point as discussed above). Ectron Model 778 whetstone bridge and wideband amplifiers (Ectron Corp. San Diego, CA.) were used to condition and amplify the strain signals. The amplified signals were simultaneously recorded with Tektronix (Beaverton, OR) high-speed digital storage oscilloscopes (models used are now obsolete) and then transferred to a personal computer for analysis. (Note that modern high-speed data acquisition boards are now capable of recording Kolsky-SHPB strain gage signals directly to a personal computer without an oscilloscope interface).

Kolsky-SHPB Striker Conditions and Stress-Strain Analysis

Because ceramics fail at very low strain levels ($\sim 1\%$), it is important to select the proper striker length in order to limit the pulse duration and thereby the amount of displacement generated during a Kolsky-SHPB test. Striker bars that are too long forces excess energy into the specimen after failure that undesirably comminutes the specimen. Ideally, the original fracture surfaces can be preserved for post-test observations. For the present study where a nominal strain rate of 2000 s^{-1} is applied, a striker bar of only 13 mm in length is calculated to generate the required 1% strain in the dumb-bell-shaped specimen. However, to allow for lower applied strain rates and slightly over-drive the applied displacement, various striker bar lengths between 19 and 40 mm were evaluated. It was observed that the 19 mm striker bar produced a “triangular-shaped” pulse with a ramp-up time of about 7-8 microseconds. The ramp time of longer strikers was the same, but was followed by a flat peak lasting about 7 microseconds in the case of the 40 mm length. Consequently, for applied strain rates $\geq 2000\text{ s}^{-1}$, constant loading conditions could not be established and lower strain rates generally provided greater fidelity in the strength measurement. “Over-driving” the specimen was minimized by progressively adjusting the striker velocity (and therefore the peak stress) until specimens just fractured. Each specimen was loaded only once so that some specimens were observed to fully load and unload elastically which validated both the dynamic modulus and strength results.

Specimen strain and strain rate were calculated using the in-situ strain gages and the specimen stress was calculated using the transmitted pressure bar strain gage (i.e. the 1-wave stress analysis¹). Vibrations due to radial ringing in the Kolsky-SHPB strain signals are largest during the initial, low-strain regime and unfortunately cannot be eliminated. These oscillations effectively determine the resolution of the stress measurements and can vary greatly from a few percent up to $\pm 15\%$ depending on the test conditions. The amplitude of these leading-edge oscillations can be reduced by modifying the striker impact conditions. One method is to slightly round the impact end of the striker bar to a radius of curvature of about 15 cm and another is to insert a thin sheet of metal or “tip” material (e.g. 0.4 mm stainless steel) between the incident bar and the striker bar that will deform slightly upon impact.⁶ Both of these methods reduce the amplitude of radial ringing in the incident bar (especially the leading edge oscillations), but also tend to increase the ramp-up time of the pulse. A slow ramping pulse can also be generated by

using thick (~ 2 mm), ductile “tip” materials.^{1,7} However, the maximum strain rate is limited to about 300 s^{-1} and varies significantly during the test so that only the value at failure can be cited.

RESULTS

The results of Kolsky-SHPB compression tests using the methods described above are shown along with quasi-static data using the same specimen geometry for selected ceramics and cermets.^{5,8,9} Commercial-grade TiB_2 and B_4C are shown in Fig. 1. Dynamic strengths are about the same as quasi-static results considering the large scatter and uncertainty in the measurements, but the strains-to-failure are consistently higher in the dynamic data. Nevertheless, the stress-strain paths are in good agreement with the ultrasonically-derived Young’s modulus and, other than differences in the failure strains, there is little indication of strain rate effects.

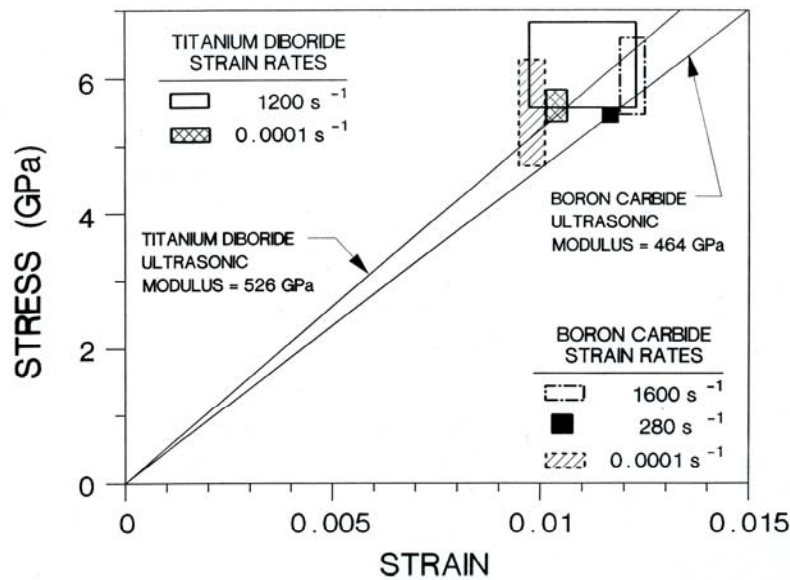


Fig. 1. Compression strengths for TiB_2 and B_4C at quasi-static and dynamic strain rates.

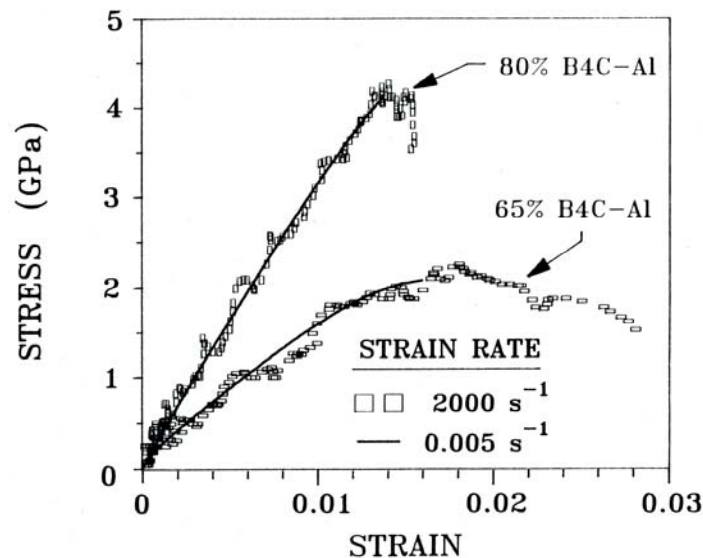


Fig. 2. Compression stress-strain curves for cermets at quasi-static and dynamic strain rates.

Figure 2 shows results for 65% B₄C-Al and 80% B₄C-Al cermets produced by metal infiltration. For these materials, there is very little difference between the dynamic and quasi-static stress-strain behavior, indicating the absence of strain rate sensitivity. The most significant effect is the appearance of a peak followed by a decay in the dynamic stress with further strain indicating damage-tolerance in the 65% B₄C-Al cermet. This effect is likely a result of the superior time resolution of the Kolsky-SHPB diagnostics such that the load decay associated with damage evolution (i.e., microcracking and Al plasticity) can be resolved.

CONCLUSIONS

The use of the Kolsky-SHPB compression technique for testing very high-strength, low-ductility materials, such as ceramics, at high strain rates ($\sim 10^3 \text{ s}^{-1}$) requires special methods and controls to ensure valid test results. The establishment of uniaxial stress-state equilibrium within the specimen and the reduction of stress at the pressure bars-specimen interface below the yield strength of the bars necessitate the use of a specially designed dumb-bell-shaped specimen in addition to limiting the applied strain rate and pulse duration of the tests. In situ strain gages were used to obtain strain-to-failure data and to ensure proper loading of the specimen and specimen stress was calculated via strain measured remotely in the transmitted pressure bar. Unfortunately, the low precision of the stress measurement combined with the inherent distribution of strength of brittle ceramic materials severely limits quantitative comparisons of the ceramic strengths.

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