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Experimental Studies for Characterization and Development of Ultrahigh Dynamic Performance Ceramic Composites

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6. AUTHOR(S)

S. Nemat-Nasser

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LaJolla, Ca 92093

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The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

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13. ABSTRACT (Maximum 200 words)

The proposed program sought to study and characterize the mechanical response, fracture, and failure modes of certain ceramics, cermets, and related composites (B_4C-Al, WC-Co, and ceramics obtained by SPS) under ultrahigh loading rates, and to relate the overall material properties to the microstructure, and, hence, to the associated processing techniques. Concomitant with this experimental effort, it was proposed to seek to determine the feasibility of developing a metal-ceramic structure which could be self-healing when subjected to certain high-velocity, impinging penetrators.

Unfortunately, the funding for this project was prematurely terminated on 1/31/90. Nevertheless, some of the projects which closely related to the research objectives of the ongoing URI Center of Excellence for Advanced Materials at UCSD were continued when possible. A number of publications, therefore, continue to appear, which acknowledge partial support under this project.

14. SUBJECT TERMS

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EXPERIMENTAL STUDIES FOR CHARACTERIZATION AND DEVELOPMENT OF ULTRAHIGH DYNAMIC PERFORMANCE CERAMIC COMPOSITES

FINAL TECHNICAL REPORT

SEPTEMBER 1, 1988 - MARCH 31, 1990

GRANT NO. DAAL03-88-K-0118

to

University of California, San Diego

by

ARMY RESEARCH OFFICE

Principal Investigator: Dr. S. Nemat-Nasser

FINAL TECHNICAL REPORT

for the period

September 1, 1988 to March 31, 1990

on

EXPERIMENTAL STUDIES FOR CHARACTERIZATION AND DEVELOPMENT OF ULTRAHIGH DYNAMIC PERFORMANCE CERAMIC COMPOSITES

Grant No. DAAL03-88-K-0118

Dr. S. Nemat-Nasser, Principal Investigator

University of California, San Diego, La Jolla, California 92093

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ABSTRACT

The proposed program sought to study and characterize the mechanical response, fracture, and failure modes of certain ceramics, cermets, and related composites (B_4C-Al, WC-Co, and ceramics obtained by SPS) under ultrahigh loading rates, and to relate the overall material properties to the microstructure, and, hence, to the associated processing techniques. Concomitant with this experimental effort, it was proposed to seek to determine the feasibility of developing a metal-ceramic structure which could be self-healing when subjected to certain high-velocity, impinging penetrators.

Unfortunately, the funding for this project was prematurely terminated on 1/31/90. Nevertheless, some of the projects which closely related to the research objectives of the ongoing URI Center of Excellence for Advanced Materials at UCSD were continued when possible. A number of publications, therefore, continue to appear, which acknowledge partial support under this project.

1.1 RESEARCH OBJECTIVES

The main objectives which were proposed for this research effort may be summarized as follows:

A. To characterize the mechanical behavior of advanced ceramic composites under high-strain-rate loading conditions and to relate this to the microstructure and the associated material processing.

B. To study the feasibility of a structural design for self-healing when subjected to high-velocity impact.

It is expected that the experimental and material characterization research would include the following:

A. Material

1. Obtain material B_4C - Al and WC - Co cermets and prepare samples.
2. Material characterization of samples (Metallography, NDC, XRD,SAM, and TEM).

B. Quasi-Static Experiments

1. Develop a testing frame for ceramics and cermets.
2. Design specimen geometries; specimen preparation.
3. Standardization of mechanical testing procedures for cermets.
4. Compression, tension and torsion testing of cermet specimens; instrumented with acoustic emission gauges for studying microcracking.
5. Micromechanical study of the evolution of damage.

C. Dynamic Experiments

Hopkinson Bar

1. Design specimen geometries for compression, tension, and torsion specimens.
2. Design and testing of special fixtures for the Hopkinson bar; evaluate the validity of Hopkinson bar data for ceramics.
3. Compression, tension, and torsion Hopkinson bar experiments on cermets; micromechanical study of damage in cermets at high strain rates of up to 10^4 s^{-1}.

Plate Impact

1. Design specimen holder for recovery experiments; optimize flyer and specimen geometries.
2. Develop pressure-shear recovery experiments for cermets; instrument experiments on cermets under normal and oblique impact conditions; micromechanical study of damage in, and dynamic behavior of, cermets at ultrahigh strain rates of up to $10^6 s^{-1}$.
3. Develop micromechanical models for dynamic behavior of cermets and construct a model for the evolution of damage (to be used in computer codes).
4. Preliminary experiments related to the development of SHIR materials; tests of SiC-LAS model material.

Laser Stress Pulse

1. Design specimen and stress cell for high energy laser experiments.
2. Preliminary experiments on cermets for studying microcrack initiation.

D. Material Characterization

1. Develop techniques for sectioning and thinning of recovered specimens for electron microscopy observations.
2. Use optical and scanning electron microscopy to quantify damage in terms of the microscopic flaws.
3. Scanning and transmission electron microscopy to identify the sources of failure and modes of failure.
4. X-ray diffraction studies to identify the phases in materials after the experiment.

1.2 DISCUSSION OF PROJECTS INITIATED AND PROGRESS MADE

A number of important projects were initiated to address the research objectives outlined in Subsection 1.1. Important elements are reviewed below.

1.2.1 Dynamic Fracturing of Boron Carbide Aluminum Cermets: Compression Hopkinson Bar Tests

We modified the conventional compression Hopkinson bar for application to dynamic testing of ceramics and ceramic composites which, in general: (1) are extremely hard; and (2) undergo very little strain prior to failing. This new technique involved two essential changes:

1. a thin copper plate is glued to the end of the incident bar;
2. the strain is measured directly by attaching strain gauges to the sample.

The presence of the copper plate introduces a monotonically increasing ramp-like stress pulse in the incident bar. While this limits the strain rates at which the test can be performed, it does preclude sudden straining of the sample. Figure 1a illustrates the stress pulse profile produced by the conventional compression split Hopkinson bar, using a uniform striker bar.

The pulse has a sharp rise and a sharp fall. This results in a step loading of the specimen which is suddenly held at a strain corresponding to the step pulse. Even if the sample fails, it is very difficult to estimate the strain rate and the strain of the sample, and to characterize the test and the material response.

To produce a ramp rise and a ramp fall in the pulse, an appropriately tapered striker bar and proper cushioning of the end of the incident bar are incorporated. This leads to the stress pulse illustrated in Fig. 1b.

Experimental Results

Figure 2 is a typical result for the heat-treated boron carbide-aluminum sample which failed during compressive loading. Failure is marked by the interruption in the strain gauge reading. In this test the incident pulse has essentially a ramp profile. Figure 3 shows the stress-strain relation for the sample which has disintegrated at failure.

Figure 4 shows the stress-strain curve for a boron carbide-aluminum cermet which has not been heat treated, i.e., *as infiltrated*.

In this case, the specimen splits axially, but continues to transmit the axial stress, while the strain gauge remains attached to the specimen. Hence, it is possible to obtain complete loading and unloading, as shown in the figure.

The heat-treated specimen fails by cleavage cracking of the boron carbide and the intermetallics, with the final failure involving decohesion of the boron carbide particles and the disintegration of the boron carbide aggregates. The *as infiltrated* sample shows ductility and fails by cleavage cracking of the boron carbide aggregates and void growth in the aluminum phase. The aluminum ligaments tend to bridge the cracks in the boron carbide.

1.2.2 Dynamic Fracturing of Boron Carbide Aluminum Cermets: Normal Flyer Plate Impact Tests

The experiments were performed using a gas gun with a 2 1/2 inch bore, capable of attaining projectile velocities of up to 200 m/s. The projectile carries a thin flyer plate which impacts the specimen at a predetermined velocity. The flyer plate and the specimen faces are made parallel prior to impact, using an optical alignment technique. The experiments are fully instrumented, allowing for careful measurement of the impact velocity by means of a suitably arranged set of pins, and for the displacement and velocity of the back face of the momentum trap, using interferometry; see the sketch in Fig. 5.

Experimental Results

Data corresponding to four tests are summarized in Table 1, where U_0 is the impact-velocity, σ_0 is the stress amplitude, and t_L is the pulse duration. Figure 6 shows the resulting particle velocity measured at the rear surface of the momentum trap for the experiment designated by BA-14 in Table 1.

Table 1. Summary of experimental conditions

Experiment	Uo m/s	σ_0 MPa	t_L μs
KT-11	45	600	0.47
KT-12	60	800	0.50
BA-14	58	770	0.44
BA-15	71	950	0.43

The specimen in this test was recovered with no visible damage. In this test the measured width of the compressive pulse was about 0.4 μs which is less than 0.44 which is the value expected in the absence of any gaps. The compression pulse was followed by a short pulse of width of about 0.05 μ and of considerably smaller amplitude. This pulse corresponds to the reflected tensile pulse due to the presence of a gap between the specimen and the momentum trap. This tensile pulse propagates back along the sample and causes unloading and then reloading, as it moves toward the impact face. Once it crosses the tail of the incoming primary pressure pulse, it subjects the sample to tension which can produce additional damage and cracking parallel to the impact face.

Figures 7a,b are micrographs showing the damage in the main part of the specimen (Fig. 7a), and close to the impact face (Fig. 7b). The axial crack seen in Fig. 7a typifies cracks observed in the axial compression of heterogeneous brittle solids.

1.2.3 Dynamic Response of Silicon Nitride

A series of systematic tests were conducted (using the new Hopkinson technique) on silicon nitride, in an attempt to obtain structure-property relations and to relate the failure modes to the microstructure. The important parameters associated with the microstructure are:

1. grain size (single crystal to fractions of a mm),
2. grain boundary interface (glassy or crystalline),
3. porosity, and
4. second-phase particles.

Material has been obtained from CERCOM in Vista, California. Microstructural characterization together with measured properties form a good basis for developing suitable micromechanical models for predicting material response under dynamic loading conditions. Some of the results from this work are shown in Figures 8 and 9. This work has been continued under the existing URI funding. A number of scientific papers are now in preparation.

1.2.4 Oblique Plate Impact Tests

The development of a new technique was initiated in order to obtain the shear properties of ceramics directly, using an oblique plate impact configuration as shown in Figure 10. A ceramic sample is impacted by a flyer plate at an angle to the normal at a known velocity. This will generate both pressure and shear waves in the target and the flyer. By measuring both the normal and transverse velocities at the rear surface of the target using laser interferometry, we can deduce the shear response of ceramics under combined pressure-shear loading. The preliminary tests were conducted on alumina. This work has been continued under the existing URI support, with the objective of developing a recovery technique for pressure-shear tests analogous to the normal plate impact recovery test. This enables us to investigate the evolution of microdamage in ceramic materials subjected to combined pressure/shear loading.

1.3 PUBLICATIONS PARTIALLY SUPPORTED UNDER DAAL03-88-K-0118

PAPERS PUBLISHED

"Plate Impact Experiments On Mg-PSZ and Improved Target Configuration" by S.N. Chang, D.T. Chung, G. Ravichandran, and S. Nemat-Nasser, Proceedings for APS Topical Conference Journal, *Shock Compression of Condensed Matter*, (1989), 389 - 392

PAPERS SUBMITTED

"Microcrack Induced Damage In Zirconia Ceramics Under Uniaxial Compression: Experiments and Modelling" by G. Subhash and S. Nemat-Nasser, Proceedings of the ASME Summer Mechanics and Materials Conference: Fracture/Damage Models, June 1992.

"Target Configurations For Plate-Impact Recovery Experiments" by S.N. Chang, D.-T.Chung, Y.F. Li, and S. Nemat-Nasser, *J. Appl. Mech.*, in press 2/92. To be presented at the ASME Summer Mechanics and Materials Conference, June 1992.

"Dynamic Stress-Induced Transformation and Texture Formation in Uniaxial Compression of Zirconia Ceramics" by G. Subhash and S. Nemat-Nasser, *J. Amer. Ceramic Soc.* submitted 8/91.

"Adiabatic Shear-Banding In High-Strength Alloys" by J.H. Beatty, Y.-F. Li, M.A. Meyers and S. Nemat-Nasser, Proc. of Army Symposium in Solid Mechanics, November 1991, in-press.

1.3 ABSTRACTS OF PUBLICATIONS

1.3.1 "Plate Impact Experiments On Mg-PSZ and Improved Target Configuration" by S.N. Chang, D.T. Chung, G. Ravichandran, and S. Nemat-Nasser, Proceedings for APS Topical Conference Journal, *Shock Compression of Condensed Matter*, (1989), 389 - 392

Normal plate impact recovery experiments have been performed on thin plates of partially stabilized zirconia, with and without a momentum trap, using a one-stage gas gun. In the range of 0.5-2.4 GPa compressive stresses, phase transformation was observed, with and without a momentum trap. The free surface velocity of the momentum trap was measured, using a normal velocity interferometer. In all recovered samples, cross-shaped cracks were seen to have been formed during the impact, even though star shaped flyer plates were used. These cracks appear to be due to in-plane tensile stresses which develop in the sample as a result of the size mismatch between the flyer plate and the specimen and the free-edge effects. Finite-element computations based on linear elasticity seem to confirm this observation. Based on numerical computations, a simple configuration for plate impact experiments is proposed, which should minimize the in-plate tensile stresses.

1.3.2 "Microcrack Induced Damage In Zirconia Ceramics Under Uniaxial Compression: Experiments and Modelling" by G. Subhash and S. Nemat-Nasser, Proceedings of the ASME Summer Mechanics and Materials Conference: Fracture/Damage Models, June 1992.

A novel split Hopkinson bar technique called the stress reversal Hopkinson technique developed at UCSD is used to subject zirconia ceramics to predetermined stress levels. During the application of loads, these ceramics undergo stress-induced martensitic transformation from tetragonal to monoclinic crystal structure. The transformation is accompanied by extensive microcracking. By carefully shaping the incident pulse in the Hopkinson bar and controlling the amount of input energy into the sample, one can attain saturation of transformation as well as microcracking in the sample. The cracks are observed to grow parallel to the loading direction under uniaxial compression. In a plane transverse to the loading axis, the cracks are observed to be randomly distributed. Hence, as far as the crack distribution is concerned, the tested samples are transversely isotropic. The same specimen is then subjected to uniaxial loads in a direction perpendicular to the first loading. A new set of cracks is seen to grow parallel to the second loading direction. These experimental results unambiguously resolve the nature of crack growth in brittle materials under uniaxial compression. The resulting degradation of moduli due to microcracking is measured by ultrasonics in all orientations. A micromechanical model assuming a dilute distribution of cracks is then used to estimate the degradation in moduli of the resulting transversely isotropic body. The moduli obtained by measuring the longitudinal and shear wave velocities in the cracked body are compared with the model results and good correlation is obtained.

1.3.3 "Target Configurations For Plate-Impact Recovery Experiments" by S.N. Chang, D.-T.Chung, Y.F. Li, and S. Nemat-Nasser, *J. Appl. Mech.*, in press 2/92. To be presented at the ASME Summer Mechanics and Materials Conference, June 1992.

Normal plate impact recovery experiments have been performed on thin plates of ceramics, with and without a back momentum trap, in a one-stage gas gun. The free-surface velocity of the momentum trap was measured, using a normal velocity (or displacement) interferometer. In all recovered samples, cross-shaped cracks were seen to have been formed during the impact, at impact velocities as low as 27 m/s, even though star-shaped flyer plates were used. These cracks appear to be due to in-plane tensile stresses which develop in the sample as a result of the size mismatch between the flyer plate and the specimen (the impacting area of the flyer being smaller than the impacted area of the target) and because of the free-edge effects. Finite-element computations, using PRONTO-2D and DYNA-3D, based on linear elasticity, confirm this observation. Based on numerical computations, a simple configuration for plate impact experiments is proposed, which minimizes the in-plane tensile stresses, allowing recovery experiments at much higher velocities than possible by the star-shaped flyer plate configuration. This is confirmed by normal plate impact recovery experiments which produced no tensile cracks at velocities in a range where the star-shaped flyer invariably introduces cross-shaped cracks in the sample. The new configuration includes lateral as well as longitudinal momentum traps.

1.3.4 "Dynamic Stress-Induced Transformation and Texture Formation in Uniaxial Compression of Zirconia Ceramics" by G. Subhash and S. Nemat-Nasser, *J. Amer. Ceramic Soc.* submitted 8/91

Transformation plasticity in Mg-PSZ and Y-TZP ceramics is investigated using a novel stress reversal Hopkinson bar technique recently developed at the University of California, San Diego (UCSD) for dynamic recovery experiments. The longitudinal and transverse strains are measured by strain gauges mounted on the sample. Samples are loaded until transformation reaches saturation. Reloading of the same sample to higher stress levels does not reveal additional inelasticity. Cuboid samples which have been loaded initially to attain transformation saturation, are then reloaded in a direction perpendicular to the first loading. The second loading produces additional inelasticity and microcracking, indicating the formation of transformation texture in each loading under uniaxial compression. SEM and TEM observations, and the results of ultrasonic and X-ray diffraction measurements, as well as micromechanical modelling of the damage evolution are discussed.

1.3.5 "Adiabatic Shear-Banding In High-Strength Alloys" by J.H. Beatty, Y.-F. Li, M.A. Meyers and S. Nemat-Nasser, Proc. of Army Symposium in Solid Mechanics, November 1991. in press

A special experimental technique has been developed by which adiabatic shear bands can be generated under controlled conditions to allow for an experimental and theoretical study of adiabatic shear-banding in high-strength alloys. This allows the systematic examination of the shear-band microstructure at various stages of its evolution. The technique has been used to develop shear bands in AISI 4340 steel for a variety of quenched and tempered microstructures. Both the strain and strain rate were controlled. The shear-band microstructure has been analyzed using optical, scanning electron, and transmission electron microscopy. Parallel with this work, Hopkinson bar techniques were used to develop high-strain, high-strain-rate constitutive properties of the material. These properties were then embedded in a viscoplastic constitutive model and used in the explicit finite element computer code PRONTO 2D to study the deformation of the specimen, leading to initiation and growth of shear bands.

This paper presents observations of the microstructure of shear bands, results of the high-strain, high-strain-rate constitutive relations of the material used, and a set of finite-element simulations of the experiment.

1.5 PROFESSIONAL PERSONNEL ASSOCIATED WITH THE RESEARCH EFFORT; DEGREES AWARDED (ARO SUPPORT)

Principal Investigator: S. Nemat-Nasser

Academic, Postdoctoral Research Associates (PGR) and Research Engineers (Partial Support)

Dr. G. Ravichandran , Assistant Professor, (9/88, 6/89 - 9/89), Graduate Aeronautical Laboratories, California Institute of Technology, Pasadena, Ca

S.-N. Chang, PGR, (9/88 - 12/89), Agency for Defense Development, Taejon, S. Korea

D.-T. Chung, PGR, (9/88 - 8/89), Materials Research and Development Center, Agency for Defense Development, South Korea

Y. Mikata, PGR, (9/88 - 8/89), Assistant Professor, Department of Mechanical Engineering and Mechanics, Old Dominion University, Norwalk, VA

Visiting Scholars:

Mr. Keith Wilfinger, 10/89, Lawrence Livermore National Laboratories, Livermore, Ca

Graduate Students (Partial Support):	Degree Awarded
B. Balendran, Research Assistant, 9/88 - 10/89	
H. Cherukuri, Research Assistant, 9/89 - 12/31/89	
A. Machcha - Research Assistant, 8/88 - 3/90	M.S., UCSD

N Yu - Research Assistant, 9/88 - 3/90

M. Yang - Research Assistant, 9/88 - 3/90

1.6 INTERACTIONS (Coupling Activities)

A. PARTICIPATION OF PRINCIPAL INVESTIGATOR AT MEETINGS, PAPERS PRESENTED; LECTURES AT SEMINARS.

"Failure Mode and Mechanism in Cermets under Stress Wave Loading", in collaboration with K.T. Ramesh, B. Altman and G. Ravichandran, ICF7 Conference on Fracture, 1989, Houston, Texas, March 23 - 29, 1989

"Compression-Induced Ductile Flow of Brittle Materials and Brittle Fracturing of Ductile Materials", *Plenary Lecture*, ICF7 Conference on Fracture, 1989, Houston, Texas, March 23 - 29, 1989

"Micromechanics of Dynamic Fracturing of Ceramic Composites: Experiments and Observations," in collaboration with G. Ravichandran, ICF7 Conference on Fracture, 1989, Houston, Texas, March 23 - 29, 1989

"Steady Crack Growth in Elastic-Plastic-Work-Harding Solids", in collaboration with M. Hori, ICF7 Conference on Fracture, 1989, Houston, Texas, March 23 - 29, 1989

"Experimental Studies for Characterization and Development of Ultrahigh Dynamic Performance Ceramic Composites" BTI Advanced Computational Method Program Progress Review, Wash. D.C., November 5 - 7, 1989

B. CONSULTATIVE AND ADVISORY FUNCTIONS WITH OTHER AGENCIES, LABORATORIES AND UNIVERSITIES.

ARO/DARPA Research Coordination Meeting 8/89 (Hosted at UCSD)

Member of DARPA Panel on Material Modeling and Large Scale Computations

Collaboration with Dr. M. Hori, Department of Civil Engineering, Tohoku University, Sendai, JAPAN

Collaboration with K.T. Ramesh, Department of Mechanical Engineering, Johns Hopkins University, Baltimore, MD

Collaboration with Dr. Keith Wilfinger, Lawrence Livermore Laboratory, Livermore, CA

Collaboration with Drs. Alec Pyzik and Arie Cohen of Dow Chemical's Central Research, Midland, Michigan. Dr. Pyzik presented a technical seminar on the processing of boron carbide-aluminum composites.

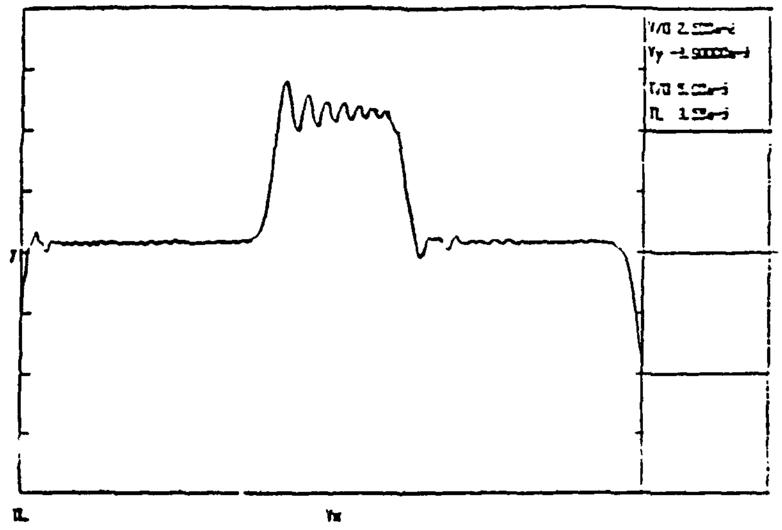

Fig. 1a. Stress pulse produced in the conventional split Hopkinson bar by a uniform striker bar.

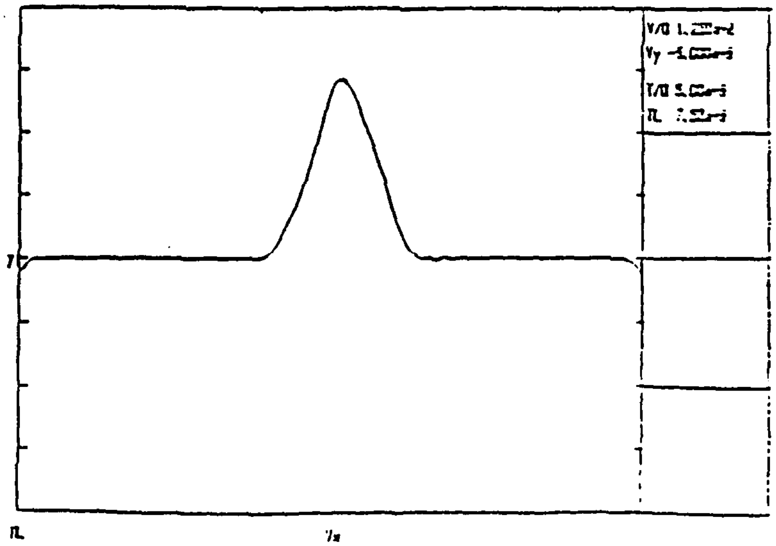

Fig. 1b. Ramped pulse produced by tapered striker bar and ¼" copper piece.

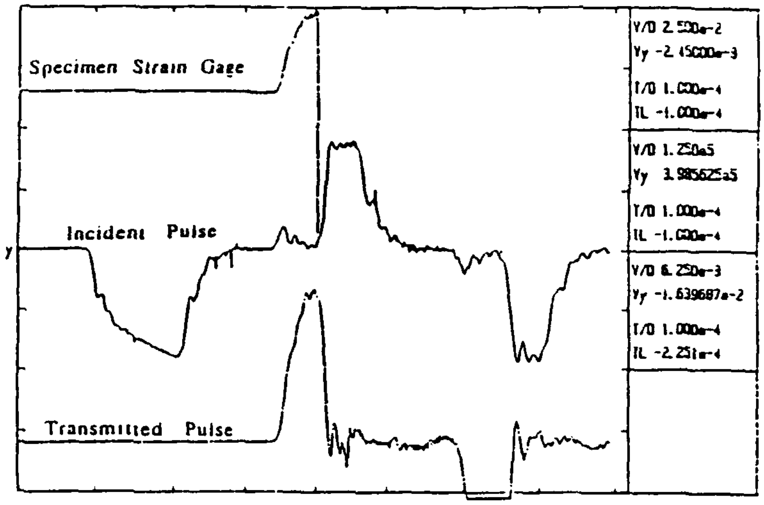

Fig. 2. Typical results for the heat treated B_4C-Al specimen which failed during compression loading.

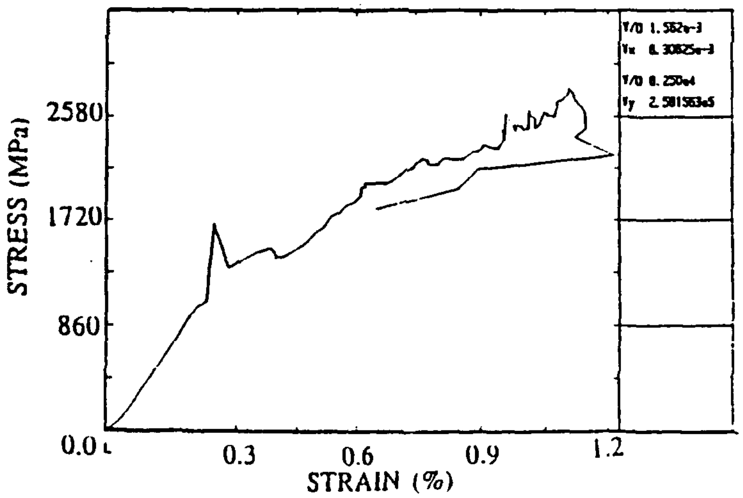

Fig. 3. Stress-strain curve for heat treated B_4C-Al cermet.

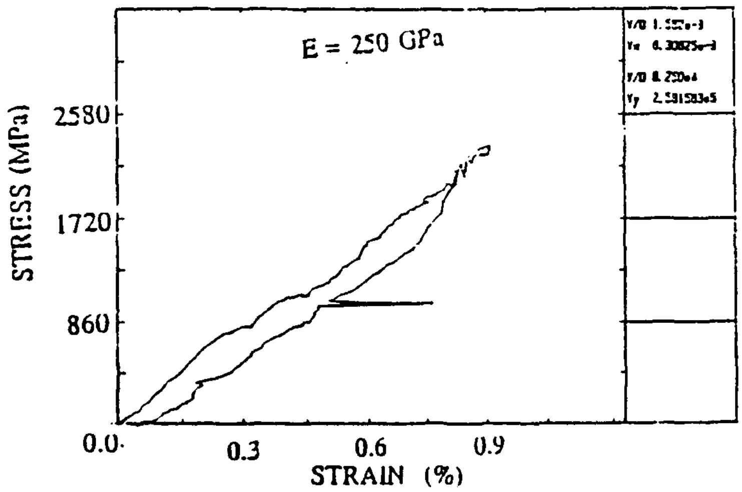

Fig. 4. Stress-strain curve for *as infiltrated* B_4C-Al cermet.

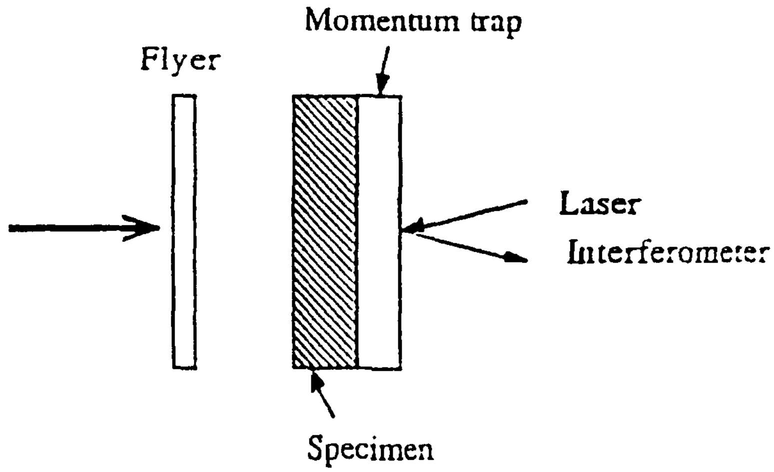

Fig. 5. Plate impact recovery experiment.

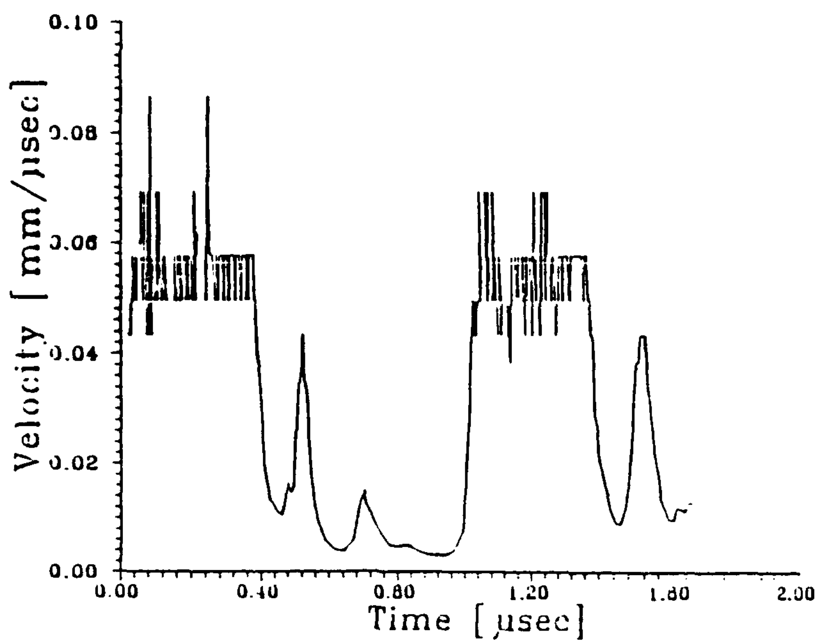

Fig. 6. Measured velocity profile at the back face of the momentum trap.

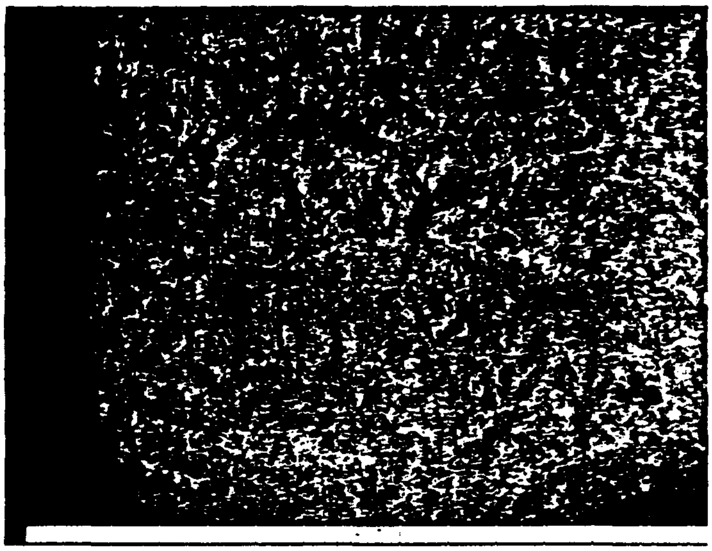

Fig. 7a. Micrograph of recovered specimen; disintegrated boron carbide particles are connected by axial microcracks.

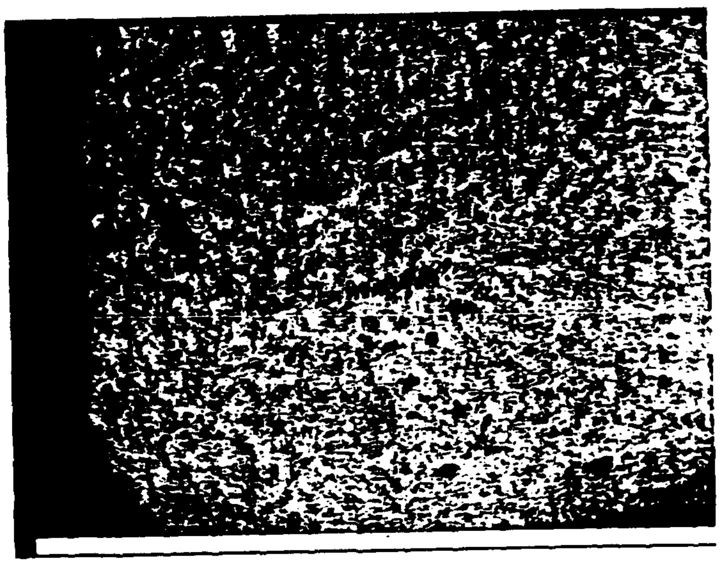

Fig. 7b. Micrograph of recovered specimen; tensile cracks parallel to the impact face, produced by the short tensile pulse which is reflected from the gap between the specimen and the momentum trap.

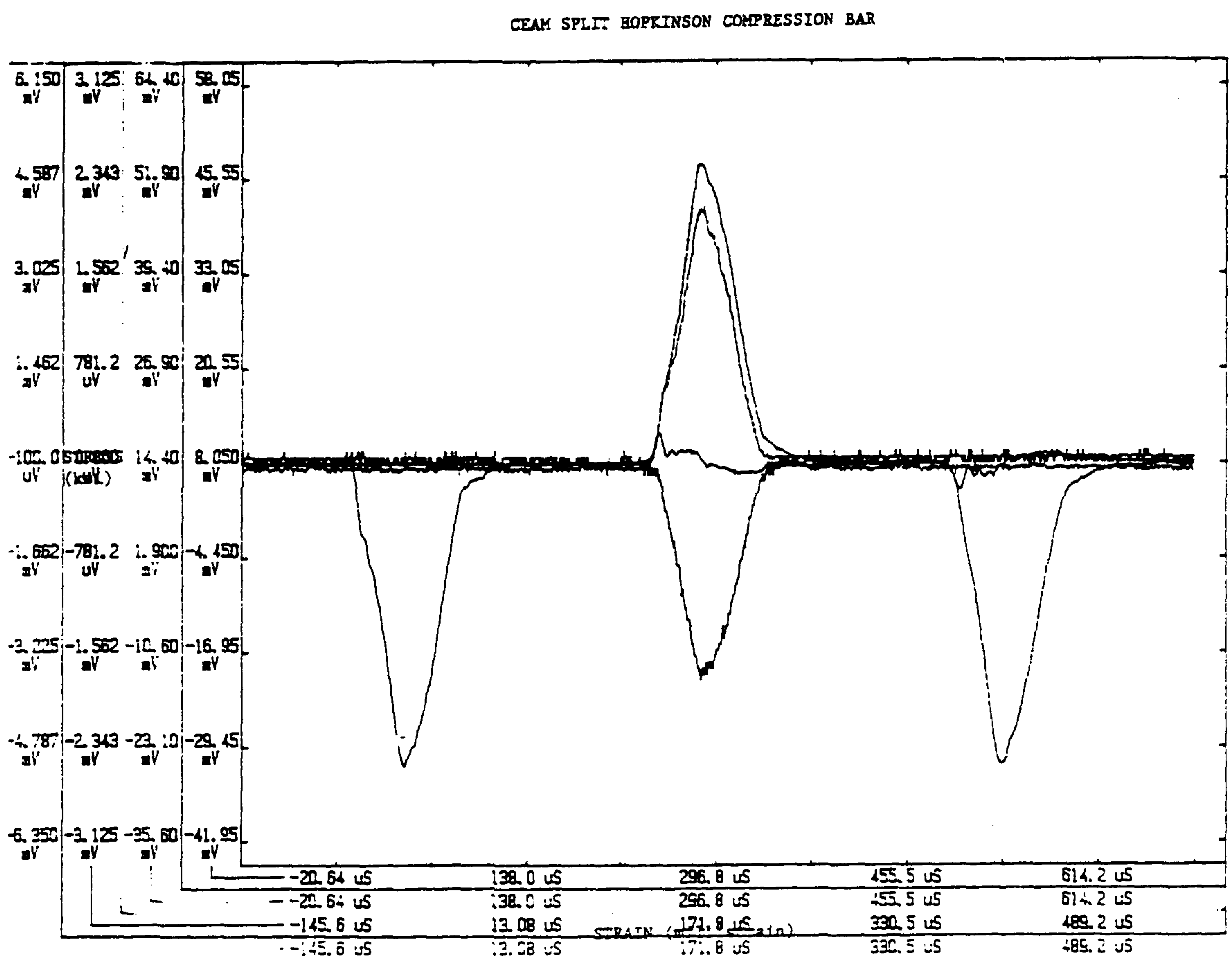

Fig. 8 Typical Stress and Strain Signals in a Compression Hopkinson Bar Test

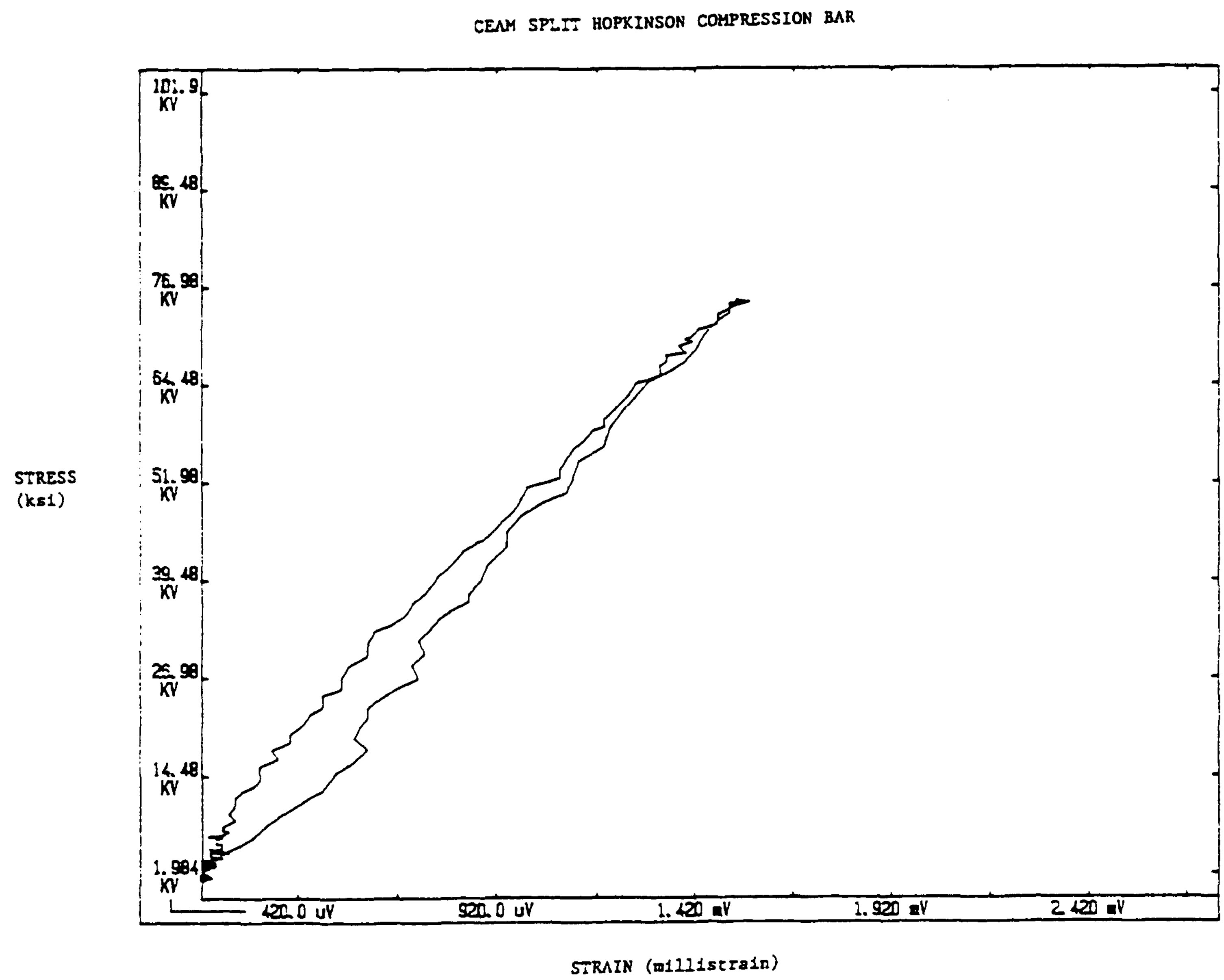

Fig. 9 Stress-Strain curve for Cercom Si_3N_4 Tested in a Hopkinson Bar