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# ELEVATED TEMPERATURE BEHAVIOR OF GLASS AND CERAMIC MATRIX COMPOSITES

(Contract No. 87-0383A)

**Principal Investigators:** 

Tsu-Wei Chou Azar Parvizi-Majidi

Center for Composite Materials and Department of Mechanical Engineering University of Delaware Newark, Delaware 19716

September 1991

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### ABSTRACT

This report summarizes research conducted under AFOSR contract No. 87-0383A during the period July 1, 1987 to May 31, 1971. The objective of research was to better understand the performance of ceramic and glass matrix composites through experimental characterization and analytical modeling of the composite mechanical properties. Although not a research objective originally, processing was also added to the research effort at a later stage primarily in the area of textile ceramic matrix composites.

The research effort can be categorized into three major areas. Section 1 of the report summarizes research on continuous fiber reinforced ceramic matrix composites. A systematic investigation of the damage development and failure behavior of Nicalon SiC fiber reinforced calcium aluminosilicate composites was first conducted through mechanical testing and microscopy. Then, an analytical effort was made to examine the fracture behavior and thermal shock resistance of continuous fiber reinforced ceramic matrix composites. Section 2 is on whisker and/or short fiber reinforced ceramic matrix composites. The effort included analytical modeling of crack deflection and creep behavior of whisker reinforced composites, characterization of fracture mechanisms in whisker/short fiber ceramic composites at elevated temperatures and experimental investigation of  $h_{1,-1}$  temperature creep behavior of the SiC<sub>w</sub>/A/<sub>2</sub>0<sub>3</sub> composite system. Section 3 is on processing. Two research efforts have been reported. In one, the kinetics of the chemical vapor infiltration (CVI) process for fabricating continuous fiber reinforced ceramic composites continuous fiber reinforced ceramic generative continuous fiber reinforced ceramic matrix composites. In the other effort, solgel processing route was used to develop three-dimensionally reinforced textile (woven and braided) ceramic composites.

The results of this research program have been shared with the ceramic composites community through 26 research publications and 22 conference presentations. A total of six doctoral students, one masters student, and one research associate have devoted their effort to this research program; three of them have received partial or full financial support from the program. All of their work is cited in this report.

#### **1. CONTINUOUS FIBER REINFORCED CERAMIC MATRIX COMPOSITES**

# 1.1 TENSILE BEHAVIOR OF NICALON SIC FIBER REINFORCED CALCIUM ALUMINOSILICATE COMPOSITES

#### Shaio-Wen Wang

Azar Parvizi-Majidi

An investigation of processing and mechanical behavior, particularly damage evolution and failure mechanisms, of continuous fiber reinforced glass and glass-ceramic composites was conducted.

The processing effort utilized a slurry infiltration/hot pressing technique to fabricate carbon fiber reinforced borosilicate glass and Nicalon SiC fiber reinforced borosilicate glass composites. A slurry infiltration prepregging setup was designed and successfully operated. Processing parameters including slurry composition, binder burnout cycle and hot-pressing schedule were systematically studied and optimized.

Mechanical behavior studies were conducted on Nicalon SiC/Calcium Aluminosilicate (CAS) composites. The nature and the strength of the fiber/matrix interface were first characterized using a variety of fiber indentation test methods and transmission electron microscopy. Both four-point flexural test and uniaxial tensile test were then carried out to study the tensile stress-strain behavior, as well as to identify the failure mechanism, of unidirectional SiC/CAS composites. A tensile test specimen was designed in this study to avoid end tab shear failure and expensive machining as well as to reduce the effect of bending due to misalignment. Theoretical predictions of the composite stiffness

reduction during damage evolution and experimental measurements were compared and used to provide more information concerning the nature of the interface in the SiC/CAS composite.

The study of the mechanical behavior was then extended to (0,90,0) cross-ply laminates of Nicalon SiC/CAS composite under tensile loading. The major failure events were identified as transverse cracking and edge delamination in the 90° ply and matrix cracking in the 0° ply. The evolution of these damages as well as the synergistic effects among thern were investigated. The effect of the 90° ply thickness on these damage modes was also determined. The composite stiffness reduction was again evaluated both experimentally and theoretically as a function of the extent of damage.

The results of this work provide insight into the stress-strain behavior and damage mechanisms of continuous fiber reinforced ceramic composites which can be very valuable in design with these materials. They also lead to recommendations for material improvement.

### **1.2 MATRIX CRACKING IN FIBER REINFORCED CERAMICS**

Yih-Cherng Chiang Tsu-Wei Chou

This work is concerned with the critical stress at the propagation of a fiber-bridged matrix crack of arbitrary length in fiber-reinforced b ittle matrix composites. The formulation of the problem follows the approach adopted earlier by Marshall, Cox and Evans (MCE), but a new shear-lag model that accounts for the matrix shear deformation above the slipping region is used here to derive the relationship between the crack opening

displacement and the crack surface closure traction. The inclusion of the matrix shear deformation above the slipping region significantly affects the calculated crack-tip stress intensity factor and the prediction of the critical stress at the propagation of the crack.

In the MCE model, a simple shear-lag model was used where the matrix shear deformation above the slipping region was neglected. The model provided a simple crack opening displacement (u) verse crack surface closure traction (T) relationship in the form of  $u \approx T^2$ . Thus, at the crack-tip u=0 and T=0. In reality, however, the bridging fibers need to carry the additional load originally born by the matrix. Hence, the crack surface traction T cannot vanish at the crack-tip. The present analysis has shown that the new surface traction T remains finite at the crack-tip. And, this significantly affects the calculated stress intensity factor and the prediction of the critical stress at the propagation of the crack.

A new crack growth criterion is proposed based upon the fracture of the matrix. The present model is shown to reduce to the Aveston, Cooper and Kelly model for long cracks. But for short cracks, there are differences between the present model and the MCE and the McCartney models. These differences are discussed by means of illustrations using three available composite systems of SiC/borosilicate, C/borosilicate and Nicalon/lithium-aluminosilicate (LAS) for which experimental results have been compiled from the open literature.

# 1.3 ANALYTICAL MODELING OF THERMAL SHOCK REJISTANCE OF LAMINATED CERAMIC MATRIX COMPOSITES

#### Yuan Ruo Wang

Tsu-Wei Chou

Ceramics and ceramic is atrix composites have demonstrated the desirable characteristics of high-temperature strength, and resistance to creep and corrosion. But they also display an unfavorable property, namely, brittleness or notch-sensitivity, which can render such materials highly susceptible to catastrophic failure under thermal shock.

The thermal shock resistance capability of monolithic ceramics has been studied since the 1950s. Cheng in 1951, first demonstrated that the thermal shock resistance of ceramics can be quantified by analyzing the nonsteady state thermal stresses in the material. The thermal shock resistance parameter,  $R \sim (1 - v) \sigma K/\alpha E$ , for the ceramics was proposed; where  $\sigma$  is the tensile strength, v Poisson's ratio, K the thermal conductivity,  $\alpha$  the thermal expansion coefficient, and E Young's modulus.

From a literature review, we found that the area of thermal shock resistance of ceramics and ceramic matrix composites is often one of conflicting results in terms of agreement between the experimental observations and theoretical predictions based upon material parameters. Agreement or disagreement between analytical calculations and experiments may be fortuirous depending upon the particular values estimated for the various properties.

The thermal response of composites is an important consideration not only in their fabrication and processing but also for their durability and long-term performance.

However, there is a lack of basic understation of thermal shock resistance characteristics of anisotropic materials or fibre-reinforced composites in general. This investigation is intended to provide some basic understandings of thermal shock resistance capability of fibre-reinforced composite laminates based upon both thermal stress and failure analyses. The thermal shock resistance capability of laminated ceramic matrix composites has been investigated through the study of three-dimensional transient thermal stresses and laminate failure mechanisms. A ( $-45^{\circ}/45^{\circ}$ )<sub>s</sub> SiC/borosilicate glass laminate is utilized as a reference composite system to demonstrate the analytical results. The maximum allowable temperature change,  $\Delta T_{max}$ , has been taken as a measure of the thermal shock resistance capability of composites. The effects of fibre orientation, volume fraction, thermal shock resistance capability, expressed in terms of the maximum allowable temperature change,  $\Delta T_{max}$ , have been assessed. Numerical computations are also performed for six composite systems.

#### 2. WHISKER/SHORT FIBER REINFORCED CERAMIC MATRIX COMPOSITES

## 2.1 MODELING OF CRACK DEFLECTION IN WHISKER REINFORCED CERAMIC MATRIX COMPOSITES

### Yih-Cherng Chiang Tsu-Wei Chou

In some whisker-reinforced ceramics, bonding between the whisker and the matrix is weak; cracks in the matrix can be deflected along the whisker/matrix interface. This is an effective mechanism for enhancing the apparent fracture toughness of composites with brittle ceramic matrices. For a class of whisker-reinforced ceramics fabricated by the hot-press procedure, the distribution and orientation of whiskers are directionally dependent. As a result, the apparent fracture toughness of the composite is highly anisotropic. This research project is concerned with the analysis of crack deflection effects in whisker-reinforced ceramic composites with a biased whisker orientation distribution. Using a crack-deflection model, the fracture toughness enhancement is examined in terms of the volume fraction, orientation distribution and aspect ratio of whiskers.

For the present problem, the size of the main crack is assumed to be much larger than the length of the whiskers. Further, the main crack is assumed to be deflected locally in the plane defined by two neighboring whiskers, which are assumed to have the same diameter and legth. To describe the geometrical relations between the main crack plane and the deflected crack plane, three independent parameters are introduced, each of which can have some degree of randomness depending on the local whisker distribution pattern. In order to proceed with a stress analysis of the crack configuration, the overall composite is assumed to be a homogeneous medium, and the three-dimensional stress field together with the associated stress intensity factors at the deflected crack front are determined, given the main crack-deflected crack configuration.

A numerical procedure involving step-increments is devised to determine the incremental advancements of the deflected crack front. Maximum toughening effect is reached when the deflected crack front is at the position where the associated strain energy release rate becomes a minimum. Since the main crack-deflected crack configuration is defined by three random parameters, a process is employed to determine the probable deflection induced toughening by considering all the possible main crack-deflected crack configurations.

The anisotropic fracture toughness behavior of hot-pressed composites has been reported for experimental measurements and the observations of fracture surface morphology for both  $SiC_w/Al_2O_3$  and  $SiC_w/t$ -ZrO<sub>2</sub> composites. A numerical procedure is established to simulate the fracture toughness for three distinct main crack propagation directions with respect to the hotpressing direction. Results of analysis have shown that the fracture toughness values are sensitive to not only the aspect ratio and volume fraction of whiskers, but also the relative orientations between the crack plane and whiskers. The simulated fracture toughness results are then compared with the experimental data of SiC whisker reinforced  $Al_2O_3$ .

# 2.2 HIGH TEMPERATURE FRACTURE MECHANISMS IN WHISKER REINFORCED CERAMIC MATRIX COMPOSITES

#### Andrew A. Wereszczak

#### Azar Parvizi-Majidi

The overall objective of this research was to examine the behavior of fracture mechanisms in whisker or short-fiber reinforced ceramic composites subject to fracture at high temperatures. Fracture or toughening mechanisms are grouped into two categories; (1) crack-tip mechanisms are those that perturb the path of a naturally propagating crack front and may be induced by fibers perpendicular to, or in, the crack plane, and (2) crack-wake mechanisms which are those that reduce the stress intensity at the crack front by reducing the crack opening displacement and may be induced by only perpendicularly oriented fibers.

In this work, two different composite systems were chosen to examine either the crack-tip or crack-wake toughening mechanisms. A commercially available hot-pressed SiC-whisker / Al<sub>2</sub>O<sub>3</sub> composite was fractured at high temperatures to investigate the crack-tip fracture mechanisms and an Al<sub>2</sub>O<sub>3</sub>-short fiber / cordierite composite was selected and fabricated to examine crack-wake

The Al<sub>2</sub>O<sub>3</sub>-short fiber / cordierite composite was fabricated by extrusion in order to achieve a virtually one-dimensional alignment of the fibers since transverse isotropy such as observed in the hot-pressed SiC-whisker / Al<sub>2</sub>O<sub>3</sub> composite introduces coupling or interaction of the crack-tip and crack-wake mechanisms, hence complicating the study of each toughening mechanism.

All composite samples were chevron-notched and loaded in four-point bending to induce stable crack propagation for toughness determination. Fractography was conducted on the broken specimens by means of scanning electron microscopy.

In the hot-pressed SiC-whisker / Al<sub>2</sub>O<sub>3</sub> composite, ~ack deflection (a crack-tip toughening mechanism) was observed depend on the specimen orien ation. The toughness was approximately 30% less when whiskers were oriented parallel with the crack propagation direction than when they were predominantly perpendicular to the crack propagation direction.

Also in SiC-whisker / Al<sub>2</sub>O<sub>3</sub> composites, the effect of a relatively slow crack propagation rate at fracture temperatures in excess of 1200°C appeared to have permitted more extensive length of pullout and therefore, crack bridging (both crack-wake toughening mechanisms). This may be attributed to lessening of radial compressive stresses on the whisker by either a change in the thermal expansion mismatch or softening of an amorphous interphase between whisker and matrix. This effect is being modelled as drag flow between concentric cylinders, separated by a viscous media. To complement this model, an extruded composite was fabricated containing short alumina fibers with an amorphous silica coating in a cordierite matrix. Bend bars of this composite are chevron-notched and fractured at predetermined temperatures to allow for crack propagation perpendicular to the one-dimensionally aligned short-fibers in the presence of a softened interphase. Thus, crack-wilke mechanisms (bridging and pullout) may be investigated without the complicated crack-tip interactions associated with the transversely isotropic SiC-whisker / Al<sub>2</sub>O<sub>3</sub> composite system.

# 2.3 CREEP BEHAVIOR OF SIC WHISKER REINFORCED AI 203 COMPOSITES

David S. Liu

Azar Parvizi-Majidi

High temperature creep tests were conducted on a silicon carbide whisker reinforced aluminum oxide composite in an air environment. Three modes of testing were employed for the studies: flexure, compression, and tension. Test temperatures ranged from 1150 to 1350°C, and stresses ranged from 80 to 250 MPa.

Flexural creep behavior was dictated by the nucleation, growth, and coalescence of creep cracks on the tensile surfaces of the specimens. Coarse-grained processing inhomogeneities served as the origins of these cracks. Crack deflection and whisker bridging were operant as toughening mechanisms under creep loading conditions.

Compressive creep tests were used to elucidate "intrinsic" creep mechanisms, or mechanisms of deformation other than creep cracking. Models were developed which showed that the creep strain rate was proportional to the impurity content and decreased with increasing matrix grain size and whisker volume fraction. Low rates of diffusion and the suppression of grain boundary slidir g in the composites were used to explain discrepancies between predicted and measured unreinforced matrix strain rates.

Tensile creep tests revealed a critical stress below which creep was accommodated and deformation occurred without cracking; above this stress of approximately 100 MPa, slow crack growth of a single crack caused failure of the tensile specimens.

# 2.4 MODELING OF CREEP OF SHORT FIBER REINFORCED CERAMIC COMPOSITES

John R. Pachalis

Yuan Ruo Wang

Tsu-Wei Chou

Several analyses have been reported in the literature for modeling the steady-state creep deformation of fiber reinforced composites. Most of these works are concerned with polymer and metal matrix composites. Many analyses assume that there is a perfect bond at the fiber/matrix interface. However, in ceramic matrix composites, it is often desirable to minimize the chemical bonds at the fiber/matrix interface in order to improve the composite toughness. Thus, sliding will occur along the interface. Next, the fibers are often assumed to be rigid and do not creep. Other researchers assume that there is a fiber creeping length where the fiber and matrix are creeping at the same constant rate. Outside of this length, the fiber is assumed to be rigid. Also, the existing analyses in the literature often assume that the matrix does not carry the applied load; the function of the matrix is merely for transferring the load to the fibers through its stress relaxation due to creep.

The purpose of this research effort is therefore, for the better understanding of the creep behavior of ceramic matrix composites through analysis and modeling. We first established a model to predict the steady-state creep behavior of aligned short-fiber reinforced ceramic matrix composites. The approach is based on an advanced shear-lag model. The analysis incorporates some unique characteristics of ceramic matrix composites, such as the fiber/matrix interface sliding effect, shear and axial loads carried by the matrix, and the fact that both the fibers and matrix creep

at elevated temperatures. Several parameters, such as fiber volume fraction, fiber aspect ratio and the sliding factor at the interface, are varied to determine their effect on creep behavior.

Next, the steady-state creep behavior of misaligned short fiber reinforced ceramic composites has been investigated. The approach is based upon an advanced shear-lag model and uses the multiaxial creep law for the fiber and matrix. It has been concluded that the creep rate is most sensitive to low values of the coefficient of sliding friction, fiber volume fraction, and fiber aspect ratio, and for misorientation angles from 10° to 60°.

Finally, the modeling analysis has been extend to consider creep of randomly oriented short fiber reinforced ceramic matrix composites.

### 3. PROCESSING OF CERAMIC MATRIX COMPOSITES

# 3.1 MODELING OF CHEMICAL VAPOR INFILTRATION (CVI) IN CERAMIC COMPOSITES FABRICATION

#### Nyan-Hwa Tai

#### Tsu-Wei Chou

The objective of this work is to analyze and model the chemical vapor infiltration (CVI) process in ceramic composites fabrication. In order to gain some fundamental understanding of the CVI process, two model have been proposed for both the traditional (isothermal) process and the improved (pressure and temperature gradients) process.

The first model for CVI process is applied to investigate the matrix growth within a ceramic fiber bundle which is situated in an isothermal reactor. The second model is applied to investigate

the fabrication process of a woven fibrous preform which is situated in a reactor with pressure and temperature gradients.

In the first model, the pore space between the fibers is simulated by cylindrical capillary tubes. The model considers binary diffusion of CO2 and H2, chemical reaction on the inner surface of the tube, and deposition film growth. Furthermore, diffusion-controlled and chemical-reaction-controlled processes are taken into account to determine the dominating process in chemical vapor infiltration. Both molecular diffusion and Knudsen diffusion are considered sequentially in this model during the infiltration process.

The parameters considered in the second model include concentration of the vapor species, pressure gradient between the inlet and the outlet, temperature boundary conditions, thermal properties of the fibrous preform, fiber diameter, fiber content, and the spatial arrangement of fibers in the preform. Furthermore, a 3-D unit cell has been adopted for simulating the fiber arrangement in the preform; and the Darcy's law is applied to simulated the flow field in the fibrous preform.

According to the results of the first model, the matrix growth history, density and porosity of the final product, and the influence of reactor temperature to final deposition profile can be predicted. Furthermore, based upon the definition of the Sherwood number, the relationship between the reactor condition, porosity of final product and the total processing time can also be predicted.

The analytical results of the second model indicate that the final density distribution of the composite is highly influenced by the position of the heating elements in the reactor. Based upon this approach, the final density and total process time of ceramic composites fabricated by the improved CVI process can be predicted.

## 3.2 SOL-GEL PROCESSING OF TEXTILE CERAMIC MATRIX COMPOSITES

Sujata Jagota

Azar Parvizi-Majidi

Tsu-Wei Chou

Sol-gel derived matrices of silica, aluminosilicate or borosilicate based compositions were incorporated into 3-dimensional woven preforms of Nextel and Tyranno fiber by an infiltration process under suction and pressure. The composites were hot pressed at 1100-1500°C to achieve final consolidation. The green structure of the composite was optimized for high green densities and homogeneity of filling by varying the solids content of the sol, adding colloidal particles to the infiltrate and tailoring the pore size distribution within the preform. The chemical composition and microstructure of the matrix could also be tailored to achieve the right consolidation conditions with a glassy state present during hot pressing with subsequent crystallization. for better refractory properties. Two such systems were studied: an aluminosilicate gel and a borosilicate-mullite gel. The composite was characterized for final density, mechanical strength and microstructure of matrix.

#### 4. PUBLICATIONS

Wereszczak, A.A. and Parvizi-Majidi, A. "Alumina Short Fiber Reinforced Cordierite Model Composites for the Study of Interphase Effect on Toughness," In *Processing and Manufacturing* of Advanced Materials for High Temperature Applications, Proceedings of the 1991 TMS Fall Meeting, Cincinnati, OH, to be held October 8-11, 1991, submitted for publication

Wereszczak, A.A. and Parvizi-Majidi, A. "Thermophoretic Deposition of Silica on Chopped Alumina Fibers," *Communications of the American Ceramic Society*, submitted for publication.

Wereszczak, A.A. and Parvizi-Majidi, A. "Effect of Fracture Temperature and Relative Crack Propagation Rate on the Fracture Behavior of Whisker Reinforced Ceramic Matrix Composites," In Ceramic Engineering and Science Proceedings, Pages 721-733, Vol. 11, No. 7-10, 1990.

Liu, D. and Majidi, A. "Creep Behavior of SiC Whisker Reinforced Alumina," In Proceedings of the 12th Conference on Composites, Materials and Structures, American Ceramic Society, Cocoa Beach, FL, January 20-22, 1988.

Liu, D. and Parvizi-Majidi, A. "Creep Behavior of SiCw/Al2O3 Composites," In Proceedings of the Third International Symposium, Ceramic Materials and Components for Engines, American Ceramic Society, Las Vegas, Nevada, November 27-30, 1988.

Liu, D. and Parvizi-Majidi, A. "Creep of Whisker Reinforced Alumina Under Compressive Loading," In Proceedings of the 14th Annual Conference on Ceramics and Advanced Ceramics, American Ceramic Society, Cocoa Beach, FL, January 14-17, 1990.

Wang, S-W. and Parvizi-Majidi, A. "Mechanical Behavior of Nicalon Fiber Reinforced Calcium Aluminosilicate Composites," In Proceedings of the 14th Annual Conference on Ceramics and Advanced Ceramics, American Ceramic Society, Cocoa Beach, FL, January 14-17, 1990.

Wang, S-W. and Parvizi-Majidi, A. "Experimental Characterization of the Tensile Behavior of Nicalon Fiber Reinforced Calcium Aluminosilicate Composites," Journal of Materials Science, Submitted for Publication.

Majidi, A.P. and Chou, T. W., "Elevated Temperature Studies of Continuous and Discontinuous Fiber Reinforced Ceramic Matrix Composites," Paper No. 89-GT-124, The American of

Mechanical Engineers, Gas Turbine and Aeroengine Congress and Exposition, Toronto, Canada, June 4-8, 1989.

Jagota S. and Parvizi-Majidi, A., "In-Situ Ceramic Composites from Ulticomponent Gels," In Proceedings of the 1990 Spring Annual Meeting of the Materials Research Society, San Francisco, CA, April 17-20, 1990.

Jagota S., Parvizi-Majidi, A., and Chou, T.W., "Infiltration of Sol-Gel Derived Glass-Ceramic Matrices for Multi-Directionally Reinforced Ceramic Matrix Composites," Journal of the American Ceramic Society, submitted for publication.

Yih-Cherng Chiang and Tsu-Wei Chou, "Toughness Models of Whisker Reinforced Ceramic Matrix Composites," ASTM, STP1080, 1011-15, 1990.

Yih-Cherng Chiang, "On Crack Deflection by Rod-Shapted Inclusions," Journal of Materials Science, in press.

Yuan Ruo Wang and Tsu-Wei Chou, "Creep Behavior of Short Fiber Reinforced Ceramic Matrix Composites", Journal of Composite Materials, in press.

Yuan Ruo Wang and Tsu-Wei Chou, "Thermal Shock Resistance of Laminated Ceramic Matrix Composites," *Journal of Materials Science*, Vol. 26, 2961-2966 (1991).

Yuan Ruo Wang and Tsu-Wei Chou, "Three-Dimensional Transient Interlaminar Thermal Stresses In Angle-Ply Composites", *Journal of Applied Mechanics*, Vol. 10, 1154-1163 (1989).

Nyan-Hwa Tai and Tsu-Wei Chou, "Theoretical Analysic of Chemical Vapor Infiltration in Ceramic/Ceramic Composites", in High Temperature/High Performance Composites, Materials Research Society, Vol. 120, p. 185 (1988).

Nyan-Hwa Tai and Tsu-Wei Chou, "Analytical Modeling of Chemical Vapor Infiltration (CVI) in Fabrication of Ceramic Composites", J. American Ceramic Society, 72, 414 (1989).

Nyan-Hwa Tai and Tsu-Wei Chou, "Modeling of Chemical Vapor Infiltration (CVI) in A1<sub>2</sub>0<sub>3</sub>/SiC Composites Processing", Proceedings of the 12th Conference on Composites Materials and Structures, Cocoa Beach, Florida, NASA Conference Publication 3018, p. 237 (1989).

Nyan-Hwa Tai and Tsu-Wei Chou, "Effects of Manufacturing Parameters on the Chemical Vapor Infiltration of Ceramic/Ceramic Composites", Proceedings of the American Society for Composites Fourth Technical Conference, p. 317 (1989), Technomic.

Nyan-Hwa Tai and Tsu Wei Chou, "Analytical Simulation of an Improved CVI Process for Forming Highly Densified Ceramic Composites", Proceedings of the Materials Research Society Conference, (1989).

Nyan-Hwa Tai and Tsu-Wei Chou, "Modeling of an Improved CVI Process for Ceramic Composites Fabrication", J. Am. Ceramic Society, 73, 1489 (1990).

Nyan-Hwa Tai and Tsu-Wei Chou, "On the Deposition Mechanism of  $A1_20_3$  in the CVI Process for Forming Ceramic Composites", J. Material, Res. 5, 2255 (1990).

Nyan-Hwa Tai and Tsu-Wei Chou, "Theoretical Modeling of the Chemical Vapor Infiltration Process for Forming Highly Densified Ceramic/Ceramic Composites", Proceedings of the International Conference on Advanced Metals and Ceramic Matrix Composites: P/M Processing, Processing Modeling & Mechanical Behavior, TMS, Anaheim, CA, 1990, in press.

John R. Pachalis, Jin Kim and Tsu-Wei Chou, "Modeling of Creep of Aligned Short-Fiber Reinforced Ceramic Composites," Composites Science and Technology, Vol. 37, 329-346 (1990).

John R. Pachalis and Tsu-Wei Chou, "Modeling of Creep of Misaligned Short Fiber Reinforced Ceramic Composites," J. Appl. Mech., in press.

### 5. PRESENTATIONS

"Alumina Short Fiber Reinforced Cordierite Model Composites for the Study of Interphase Effect on Toughness," to be presented at 1991 TMS Fall Meeting, Cincinnati, OH, October 8-11, 1991

"Design, Processing and Mechanical Testing of a Model Ceramic Composite for the Investigation of High Temperature Fiber Pullout," to be presented at 13th Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 24-26, 1991

"High Temperature Whisker Pullout in Ceramic Composites," 93rd Annual Meeting of The American Ceramic Society, Cincinnati, OH, April 28-May 2, 1991

"High Temperature Fracture Mechanisms in Short-Fiber Reinforced Ceramic Matrix Composites," 12th Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 11-13, 1990

"Thermophoretic Deposition of Silica on Chopped Alumina Fibers," 92nd Annual Meeting of the American Ceramic Society, Dallas, TX, April 22-26, 1990

"Effect of Fracture Temperature and Relative Crack Propagation Rate on The Fracture Behavior of Whisker Reinforced Ceramic Matrix Composites," 14th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, FL, January 14-17, 1990

"Elevated Temperature Fracture Behavior of Whisker-Reinforced Ceramic Composites," 11th Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 19-21, 1989

"Fracture Behavior of Whisker-Reinforced Ceramic Composites," 10th Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 19-21, 1988

"Creep Behavior of SiC Whisker Reinforced Alumina," 12th Conference on Composites, Materials and Structures, American Ceramic Society, Cocoa Beach, FL, January 20-22, 1988. "Creep Behavior of SiCw/Al2O3 Composites," Third International Symposium, Ceramic Materials and Components for Engines, American Ceramic Society, Las Vegas, Nevada, November 27-30, 1988.

"Creep of Whisker Reinforced Alumina Under Compressive Loading," 14th Annual Conference on Ceramics and Advanced Ceramics, American Ceramic Society, Cocoa Beach, FL, January 14-17, 1990.

"Creep Behavior of Whisker Reinforced Ceramic Composites," 12th Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 11-13, 1990.

"High Temperature Creep of Ceramic-Matrix Composites," Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 19-21, 1988.

"Creep Characteristics of SiC Whisker-Reinforced Al<sub>2</sub>O<sub>3</sub> Composites," Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 19-21, 1989.

"Processing and Properties of Fiber-Reinforced Glass and Glass-Ceramic Composites," Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 19-21, 1989.

"Material Science Issues in Ceramic Matrix Composites" Annual University-Industry Research Symposium, Center for Composite Materials, University of Delaware, Newark, DE, September 11-13, 1990.

"Sol-Gel Processing of Ceramic Matrix Composites," Annual University-Industry Symposium, University of Delaware, Newark, DE, September 19-21, 1989.

"Sol-Gel Processing of Ceramic Matrix Composites," Annual University-Industry Symposium, University of Delaware, Newark, DE, September 11-13, 1990.

"In-Situ Ceramic Composites from Multicomponent Gels," 1990 Spring Annual Meeting of the Materials Research Society, San Francisco, CA, April 17-20, 1990.

"Ceramic Matrix Composites Made by Sol-Gel Infiltration of Braided Fiber Preforms," 92nd Annual Meeting of the American Ceramic Society, Dallas, TX, April 23-26, 1990.

"Processing and Characterization of 3-D Woven Composites with Sol-Gel Derived Matrices," 93rd Annual Meeting of the American Ceramic Society, Cincinnati, OH, April 28 - May 2, 1991.

"Mechanical Behavior of Nicalon Fiber Reinforced Calcium Aluminosilicate Composites," 14th Annual Conference on Ceramics and Advanced Ceramics, American Ceramic Society, Cocoa Beach, FL, January 14-17, 1990.

## 6. GRADUATE STUDENTS AND RESEARCH ASSOCIATE PARTICIPATED IN THE PROGRAM AND DEGREES GRANTED

#### (A) Doctorate Degree :

Nyan-Hua Tai, August 1990 Dissertation Title: "Modeling of Chemical Vapor Infiltration (CVI) in Ceramic Matrix Composites Processing"

Shaio-Wen Wang, January 1991 Dissertation Title: "Processing and Characterization of Continuous Fiber Reinforced Ceramic Matrix Composites"

David S. Liu, January 1991 Dissertation Title: "High Temperature Creep of Silicon Carbide Reinforced Aluminum Oxide"

Yuan-Ruo Wang, June 1991 Dissertation Title: "Analysis and Modeling of High Temperature Performance of Advanced Fiber Composites"

Yih-Cherng Chiang<sup>\*</sup>, January 1992 (expected) Dissertation Title: "Analysis and Modeling of Strength and Toughness of Ceramic Matrix Composites"

Andrew A. Wereszczak\*, May 1992 (expected) Dissertation Title: "High Temperature Fracture Mechanism of Whisker/Short Fiber Reinforced Ceramic Matrix Composites"

#### (B) Masters Degrees

John Pachalis, May 1990

Thesis Title: "Analytical Modeling of Creep Behavior of Short Fiber Reinforced Ceramic Matrix Composites"

Partially or fully supported by the research program.

### (C) Research Associate

Dr. Sujata Jagota\*

## 7. AWARD

Shaio-Wen Wang, Best Student Presentation Award American Ceramic Society, 14th Annual Conference on Ceramics and Advanced Ceramics, Cocoa Beach, FL, January 14-17, 1990.

Partially or fully supported by the research program.

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