

**AFRL-ML-WP-TP-2002-407**

**HIGH TEMPERATURE COMPOSITES**  
**Work Unit Directive (WUD) 53**

Ronald J. Kerans



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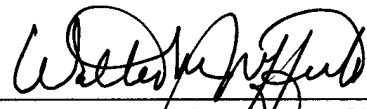
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<b>14. ABSTRACT</b> There were numerous interrelated issues that were addressed as a part of the composite interface control effort. Crack deflection is a complex function of the fracture properties of the constituents. Hence, in addition to designed oxide coatings for both oxide and non-oxide fibers, substantial activity has pursued understanding and controlling fiber degradation, isolating and understanding aspects of the failure process, designing processes, and tests that provide definitive criteria for evaluation. These efforts have resulted in the identification and solution of a persistent and pervasive problem with fiber degradation during coating. This has finally enabled evaluation of the coatings in real composites made both in-house and as part of numerous collaborations with industry, academe, and other government laboratories. This in turn led to the recent definitive proof of the viability of oxide coating approaches in real composites. With this recent definitive demonstration of oxide coating viability, the specific focus is shifting towards understanding how best to design the overall composite system to best employ the new coatings. The word "design" is used in the sense of the choice of the basic constituents of the composites and the choice and execution of the nano and microstructures of the constituents, and the micro and macrostructures of the composites.					
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**Ceramics Research Group WUD 53 (AFOSR Task 2306BL)**  
**Final Report**  
**August 2002**

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**High Temperature Composites Task Leader**

## **1. INTRODUCTION AND BACKGROUND**

The ability to exploit the unique balance of properties offered by the wide variety of compounds and mixtures of compounds that fall under the heading of ceramics has enabled many technological changes, and will enable many more in coming years. The microelectronic revolution, or "Silicon Age", would not be possible without the concurrent advances in ceramic substrate materials and processing. The future of "smart materials" will depend heavily on advanced ceramic systems, as will micromachines for high temperature and chemically aggressive environments. One of the most pervasive and attractive, yet most elusive, objectives has been use of ceramics in high temperature structural applications. The higher operating temperatures thereby enabled will provide substantial increases in efficiency and performance with reduced environmental impact.

The primary problem has been the low fracture toughness that comes with the array of more attractive properties of monolithic ceramics. This problem was effectively dealt with by the introduction of continuous-fiber ceramic composites. However, the toughness of composites was found to be dependent on controlled low-strength fiber-matrix interfaces, which was in turn dependent on fiber coatings that unfortunately also imparted environmental resistance problems. There were also problems with the stability of the fibers themselves; however, while there is still room for improvement, significant progress on improved fibers has been made. The Ceramics Group chose to focus effort on the problem of oxidation resistant interface control. This was both the most difficult problem -- probably requiring a long term, comprehensive attack -- and the one receiving the least attention.

There are numerous interrelated issues that must be addressed as part of the composite interface control effort. The crack deflection event has a complex dependence on the fracture properties of the constituents. Hence, in addition to designed oxide coatings for both oxide and non-oxide fibers, substantial activity has pursued understanding and controlling fiber degradation, isolating and understanding aspects of the failure process, designing processes and tests that provide definitive criteria for evaluation. We strive to focus our attack on basic issues that make a permanent contribution to the literature in ways that are more broadly meaningful than simply composite development. We have built the capabilities to meaningfully address these issues and have become the world leaders in this effort. Recently, progress has become quite rapid and we hope to see significant transition to practice in the near future.

## OBJECTIVE

The overall objective of the Ceramics Research Group is to provide the key missing scientific links necessary to enable the development of new materials systems for applications of major importance to the Air Force. Our current objective is the establishment of the foundation of knowledge and understanding to enable the development of fibrous ceramic composites with properties that sum to no less than a revolution high temperature materials.

### 3. APPROACH

- (a) Deduce and evaluate approaches to oxidation resistant control of the fiber/matrix interface.
- (b) Determine factors controlling mechanical response of brittle-matrix composites through interactive theoretical and experimental studies in mechanics.
- (c) Determine strategies for the design and practical fabrication of high temperature composites.
- (d) Design and evaluate innovative microstructures for tough "in-situ" composites.

### 4. RESULTS

#### 4.1 Summary

There are numerous interrelated issues that are being addressed as part of the composite interface control effort. Crack deflection is a complex function of the fracture properties of the constituents. Hence, in addition to designed oxide coatings for both oxide and non-oxide fibers, substantial activity has pursued understanding and controlling fiber degradation, isolating and understanding aspects of the failure process, designing processes and tests that provide definitive criteria for evaluation.

These efforts have resulted in the identification and solution of a persistent and pervasive problem with fiber degradation during coating. This has finally enabled evaluation of the coatings in real composites made both in-house and as part of numerous collaborations with industry, academe and other government laboratories. This in turn led to the recent definitive proof of the viability of oxide coating approaches in real composites.

With this recent definitive demonstration of oxide coating viability, the specific focus is shifting towards understanding how best to design the overall composite system to best employ the new coatings. The word "design" is used in the sense of the choice of the basic constituents of the composites and the choice and execution of the nano and microstructures of the constituents, and the micro and macrostructures of the composites.

Collaborative research with the Combustion Research Group of PRSF has indicated that oxide-oxide composites are viable candidates, with respect to the most basic requirements, for an

innovative new turbine engine combustor, but will require careful design to control thermal stresses. This work has been considering current technology coatingless composites, but coated-fiber composites will be introduced as appropriate. This work has led to several innovations (including one patent, one patent pending) on materials approaches to thermal stress control.

#### 4.2 Narrative

The following narrative attempts to touch on a few high points and give a flavor for the nature and overall flow of the work done. It is by no means comprehensive, sacrifices rigor for brevity and is meant to provide context for the accompanying bibliography and the review.

Our early research in this area was concentrated in three areas: identification of the required coating properties and associated governing mechanics, identification of viable coating schemes, and coating processing by solution chemistry and CVD. This work was successful in establishing criteria that coatings must meet and in developing the science base to measure them. It was also successful in identifying and screening numerous concepts and in establishing potential viability of several. Furthermore, it was successful in developing a new, practical, solution-based process for coating fiber tows (subject of several patents).

The first of these areas, measurement and interpretation of interface properties, provides an example of the synergistic interaction between analytical and experimental work. It is also a fine example of the power of asking the right question. We developed an analytical model for fiber pushout/pullout behavior while simultaneously devising an experimental protocol. When we compared friction values obtained from two different regions of the experiment with one derived using the model, they were significantly different. We initially suspected the model, but eventually asked the right question: Suppose both values are correct? That led us to postulate a progressive contribution of roughness to the friction, which was immediately borne out by an inspired experiment devised by Dr. Jero; again growing out of the discussions comparing experiments and models.

A second model addressing progressive roughness produced yet another fine example of the power of concurrent modeling and experiment. This analysis predicted significantly different pushout load-deflection curves for some cases, but such curves had never been reported or observed in our lab. Subsequently collaborators at the University of Bordeaux were found to be enthusiastic about a new family of composites with higher than usual interfacial friction and very good composite properties, but perplexing pushout curves. A variation of our analysis fully explained the curves and enabled reduction of data from them. Recent work on Nextel-reinforced Blackglas composites with monazite, BN, or porous  $Zr_xSi_yO$  interfacial coatings demonstrated behavior atypical of conventional composites, but completely consistent with the progressive roughness model. It appears that nearly all of the latest oxide coatings require the progressive roughness analysis to reduce the data to materials properties; hence, the investment in modeling of a postulated phenomenon ultimately paid off well.

A related aspect of this work centered on anticipating issues that would be raised by the substitution of new fiber coatings in composites. We introduced the entire topic of composite design in the sense of adjusting microstructural-level geometry and constituent elastic properties in a complimentary fashion to achieve the desired composite behavior. We also applied these insights to

existing studies and devised simple means to introduce previously overlooked factors into existing models.

As noted above, a number of different approaches to interface control have been evaluated to varying degrees. The fugitive coating concept was demonstrated to be a viable approach for oxide composites, though not without limitations. The sacrifice in off-axis properties was felt to limit possible application and placing the results in the literature was felt to be sufficient transition. Several other approaches had shown promise, but there were lingering questions or significant challenges remaining for most of them. For example, hexaluminates were found to cleave easily enough on basal planes to deflect cracks, and processes to obtain the correct microstructure were developed. However, despite remarkable progress in identifying phase stabilizers to reduce process temperatures, they remain a little too high for the current generation of polycrystalline fibers. By late FY98, it was decided to focus additional effort on a single coating approach and move it to a definitive determination.  $\text{LaPO}_4$  monazite, originally proposed by researchers at the Rockwell Science Center, was chosen as being somewhat ahead of other approaches in terms of all aspects of the problem, in part due to our own efforts on the coating process in support of an ML managed DARPA funded activity.

During the course of experimental work on coatings, it was quickly discovered that both oxide and non-oxide ceramic fibers are much less robust to the seemingly relatively benign coating processes than previously thought. This led to a significant research activity investigating the mechanisms of degradation, an inherently interesting and important question, and to the elimination of the problem in coating. Research to identify the source of fiber degradation identified stress-corrosion cracking from vapor phase intermediates during coating as a probable cause. A set of experiments to test this hypothesis was designed. The vapor phase components were determined by mass spectrometry. Fibers were then exposed in a manner mimicking the coating process but with isolated combinations of the gases. Significant differences were observed, but the results do not correspond to straightforward models. Nevertheless, this work led to a new approach involving a refined process leading to a much purer sol, and a new variation of the chemistry. This approach yielded remarkably improved results. The substantial degradation previously observed was completely absent for both oxide and SiC based fibers. The processing/fiber degradation breakthrough was confirmed for  $\text{LaPO}_4$  monazite coatings on a wide variety of fibers. Multiple coating passes were found to be effective in obtaining a variety of thicknesses of coating over a realistic range for composite use, and coverage was found to be essentially complete. As noted above, rather than each coating cycle severely degrading the fiber, each pass actually increases the strength. This result is surprising because investigation confirms that it is extremely unlikely that the coating carries significant load. Evaluation of coated-fiber composite performance implies that the coatings may play a significant role in protecting the fibers from forms of environmental degradation that were not even known to exist, and this may be the source of the effect. Research on mechanisms is briefly addressed in a paragraph below.

Surprising room-temperature fiber pushout results reported by RSC researchers led to TEM investigations at ML that uncovered substantial plasticity in  $\text{LaPO}_4$  (monazite) coatings. Related results and general questions on the details of debonding and sliding led to an experiment that produced a remarkable sequence of TEM images detailing the entire length of the debonding crack in a partially debonded pushout specimen. This specimen has exploded assumptions about the basic

character of the debonding process in monazite coatings and is providing considerable new insight into the process. Moreover, it is providing abundant evidence for the surprising role of true plasticity via dislocations and deformation twinning. Details of the twinning and dislocation processes have been determined. The analysis of deformation twinning is groundbreaking in that it is the first on such a complex compound and shows that the governing energetic factor is indeed different than in simpler materials.

Dense Nextel™ 720/monazite/Blackglas™ minicomposites were fabricated and tensile tested, revealing improved strength over control samples with no interfacial coatings. Although little fiber pullout was seen on the fracture surfaces of these minicomposites, push-in testing demonstrated that the monazite did debond readily. Applying the progressive roughness model to the push-in data revealed a low debond energy ( $1 \text{ J/m}^2$ ), a friction coefficient of 0.2, and an interfacial shear stress in the sliding regime of 18 MPa. The absence of significant pullout lengths may be attributed, in part, to the roughness of the monazite coatings. Nevertheless, the results for this coating continue to be very promising. By the end of FY99 we believed proof-of-principal to be in-hand, though there were no results on true tow-reinforced composites with thin fiber coatings.

In FY00, we began focussing on exploiting the breakthrough in fiber coating to pursue definitive evaluation of new coatings in true composites. Initial focus remained on monazite as the fiber coating. The solution of the fiber degradation problem has proven to be the expected watershed development in that the proper evaluation of oxide coatings is now proceeding at a faster pace. Coated fibers of six different types, two oxides and four SiC based, have been provided to industry, academia and other government labs for incorporation into composites and mini-composites for evaluation of coating performance (Appendix C). This hands-on experience also provides a mechanism for rapid and effective transition of technology. Composites and micro/mini composites have been fabricated in-house to the same end. Internal composite processing research has very recently generated a new system better suited for the evaluation and use of the new coatings. Preliminary results of both internally produced and collaborative composites imply an immediate 100C improvement in composite use temperature, though much refinement will be required to fully exploit the new coatings. Most importantly, these composites have finally provided definitive proof that oxide coatings can perform the vital role of deflecting cracks and activating distributed damage failure in real, yarn reinforced composites.

While the tow coating process is straightforward and relatively economical, cloth/preform coating could bring down the cost of processing durable ceramic composites, and reduce the possibility of coating damage, particularly in complex architectures. We have undertaken several modest exploratory efforts to evaluate approaches to cloth coating. These have included investigations of manipulation of the surface charges on fibers and colloidal particles, and freeze-gelation and sublimation: an innovative process studied in earlier work. However, while there were some promising aspects of the results, they have failed to achieve even modest levels of uniformity and coverage of fibers.

As part of the evaluation of coated fibers, both uncoated and monazite coated Tyranno-SA SiC fibers were exposed to oxidizing conditions. Monazite coated fibers apparently oxidized at a much lower rate than uncoated fibers. The scales were far thinner than accountable by diffusivity in the monazite coating, and far more uniform than the coatings, implying some other mechanism. More rigorous investigation including thermogravimetric analysis (TGA), analytical microscopy and



surface doping has been conducted to confirm the effect and investigate kinetics mechanisms. The evidence points to effects of very dilute fiber dopants that are negated by the coating, but definitive proof of the mechanisms has not yet been obtained. This finding could also be of great importance in areas other than fibrous composites. We have initiated an invention disclosure.

For the last three years we have been engaged in an AFOSR Entrepreneurial Research collaborative program with the AFRL Propulsion and Power Directorate (PRSF) entitled "Ultra Compact Ceramic Combustor (UC<sup>3</sup>)". The ML half has been collaborative with the Behavior & Life Prediction Group and the Mechanics Group. This investigation of an innovative composite combustor and enabling materials led to three innovative ideas for thermal stress control in oxide composites. Two of these led to invention disclosures and, so far, one patent. The activity has moved forward on several parallel fronts. A critical subelement and suitable boundary conditions were identified, and modeled by RG 2.3, in order to evaluate the viability of oxide composites for such applications. For example, it was determined that a single piece shell design would have excessive thermal stresses, but relatively simple design approaches can manage the stresses. For example, a multi-piece approach that breaks the component into three regions spanning smaller temperature differentials reduces the stresses to quite low levels.

The UC<sup>3</sup> activity will finish devising a strategy for the evaluation and introduction of a substantially new material – oxide composites – for a turbine engine part using a much more integrated approach than is convention. It will identify the critical issues and best subelements for benchmark tests. In addition, it will have determined a preliminary conclusion on viability of oxides and a strategy for introduction of suitable composite components.

Related studies have been performing experimental evaluation of the barrier layer and mixed fiber composite approaches to thermal stress management. A patent application was filed on the latter, along with a subsequent one on a broader interpretation of the idea (see hybrid composites below). We have also been active in the task to integrate the simulation activities in the two Directorates. This will greatly facilitate a more integrated approach to materials evaluation and application. Microstructural and mechanistic evaluations have progressed on a continuing basis in support of all the UC<sup>3</sup> tasks.

A spin-off of a porous oxide coating study has demonstrated an improved porous-matrix composite system. Preliminary testing has shown a 100 C advantage over current systems using the same fiber. Transition discussions are underway.

We will continue to pursue applications for near-term composites, such as improved porous-matrix materials. We will develop such ideas on an opportunistic basis, but will continue to focus on determining the viability of ceramic composites for the more aggressive, broadest payoff applications.

In summary, our substantial effort in this area is beginning to pay off in rapid progress toward multifaceted evaluation of the technology in multiple organizations. We will continue to aggressively pursue that end, and to simultaneously pursue transition-to-practice and anticipation of problems associated with actual application. If progress continues in the current positive direction, real impact of our research on Air Force systems may not be far away.

## 6. Publications, Invited Presentations and Inventions

### Publications

- <sup>1</sup>M. K. Cinibulk, T. A. Parthasarathy, K. Keller, and T. Mah, "Porous Yttrium-Aluminum Garnet Fiber Coatings for Oxide Composites," *J. Am. Ceram. Soc.* (in review).
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- <sup>4</sup>P. Mogilevsky, R. S. Hay, E. E. Boakye, and K. A. Keller, "Texture of Rhabdophane Derived Monazite Coatings," *J. Am. Ceram. Soc.* (In review).
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- 175 R. Ruh and A. Zangvil, "Phase Relationships in the Silicon Carbide-Aluminum Nitride System," *J. Am. Ceram. Soc.* **71** (10) 884-890 (1988).
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- 177 K. Cho, R. J. Kerans, and K. A. Jepsen (Keller), "Selection, Fabrication, and Failure Behavior of SiC Monofilament-Reinforced Glass Composites," *Ceram. Eng. Sci. Proc.* **9** (7-8) 815-824 (1988).
- 178 R. J. Kerans, "Dependence of Composite Properties Upon Statistical Variations in Fiber Length," *Ceram. Eng. Sci. Proc.* **9** (7-8) 613-620 (1988).

### Invited Presentations

- R.J. Kerans, R.S. Hay, E.E. Boakye, M.K. Cinibulk and T.A. Parthasarathy, "Oxide Fiber-Coatings for Controlled Fracture in Robust Ceramic Composites", International Congress of Fracture (ICF), Honolulu, HI, 3-7 December 2001
- R.J. Kerans, "Progress in Oxidation Resistant Interface Control in Ceramic Composites" High Temperature Ceramic Matrix Composites (HT-CMC 4), Munich, Germany, 1-3 October 2001
- R.J. Kerans, "Oxidation Resistant Interface Control in Fiber Reinforced Ceramics" Annual Meeting, American Ceramic Society, Indianapolis, IN, 1-4 May 2001
- R.J. Kerans, "Oxidation Resistant Ceramic Composites: How 'Weak Interfaces' Became 'Designed Failure Processes,'" Materials Research Society Annual Meeting, Boston, MA, 1 December 2000
- M. K. Cinibulk and T.A. Parthasarathy, "Characterization of Oxidized Bayer SiBCN Fiber," Ultra High-Temperature Ceramics Workshop, University of Colorado, Boulder, CO, August 2000.
- M.K. Cinibulk, "AFRL's Fiber-Matrix Interface Activities for All-Oxide CMCs," Ultra High-Temperature Ceramics Workshop, University of Colorado, Boulder, CO, August 2000.
- R.J. Kerans, "Designed Failure Processes and the Development of Oxidation Resistant Ceramic Composites," 10<sup>th</sup> Iketani Conference on Materials Research, Karuizawa, Japan, 29 June 2000
- R.J. Kerans, "Lightweight Power and Selected Materials Innovations At the Beginning and End of the Century," Graduate Seminar, University of Dayton, 23 March 2000
- R.J. Kerans, "Lightweight Power and Selected Materials Innovations At the Beginning and End of the Century," Graduate Seminar, Case Western Reserve University, 22 February 2000

- R.J. Kerans, "High Temperature Applications of Ceramic Composites", 1999 International Mechanical Engineering Congress and Exposition (ASME), Nashville, TN, 14-19 Nov. 1999
- R. S. Hay, 2nd Acta Materialia Workshop on "Ceramic and Bimaterial Interfaces: Designing for Properties," Seville, Spain, Sep 1999
- R. S. Hay, "Solid State Studied in Ceramics," 1999 Gordon Conference, Aug 1-6, Kimball Union Academy, NH
- T.A.Parthasarathy and R.J.Kerans, "Modeling Cracks for Material Design and Life Prediction in Ceramic Matrix Composites", NIST workshop on Reliability of Ceramics, Gaithersberg, MD, August 31, 1999.
- M. K. Cinibulk, "Synthesis of Hexaluminates for Coatings on Polycrystalline Oxide Fibers," IMA Seminar, University of Bayreuth, Germany, July 1999.
- R. J. Kerans, "Crack Deflection in Ceramic Composites and Fiber Coating Design Criteria," JFCC International Workshop on Fine Ceramics '98, Nagoya, Japan, Mar 12-13 1999
- R. J. Kerans, "Damage Tolerant Ceramic Composites," Gordon Research Conference on Composites, Ventura, California, Jan 10-15 1999
- R. J. Kerans, "Design for Damage Accumulation and the Development of Ceramic Composites," International Meeting on Thermo-Mechanical and Electrical Properties of High-Temperature Materials, Renaissance Wailea Beach Resort, Maui, Hawaii, Jan 4-9 1999.
- R. S. Hay, "Fiber Coatings and Fiber Strength", Recent Advances in Ceramic Matrix Composites - Structural Design, Fabrication, and Long Term Use, MRS Fall 1998, Boston, MA
- T. A. Parthasarathy, "Successes and Challenges in Ceramic Matrix Composites", Graduate Seminar, University of Dayton, October 1998
- R. J. Kerans, "Interface Control For Durable Ceramic Composites", TMS-AIME Fall Meeting, Chicago IL, 11-15 October 1998
- R. J. Kerans, "Issues in the Design of Selected Advanced Ceramic Systems," Advanced Materials for Extreme Environments: New Experimental Opportunities in Neutron Scattering, Argonne National Laboratory, Argonne IL, September 11-12 1998.
- R. S. Hay, "Fiber Coating Issues for Oxide-Oxide Matrix Composites," Oxide/Oxide Workshop 98, June 22-24, Irmsee, Germany
- T. A. Parthasarathy, R. J. Kerans and R.S.Hay, "Challenges in Design and Evaluation of Advanced Interface Control Strategies in CMCs, Mardi Gras Conference on 'Challenges in Materials Design for Extreme Environments', Baton Rouge, LA, February 1998
- T. A. Parthasarathy, R. J. Kerans, T. Mah, " The Outer Limits : Selection of Materials for Structural Use in Combustion Environments at V.High Temperatures", International Workshop on Ultrahigh Temperature Ceramics, Boulder CO, July 1998
- T. A. Parthasarathy, M. G. Mendiratta, D. M. Dimiduk, " Creep Behavior of PST and Fully Lamellar Polycrystals of Ti-48Al", TMS Annual Meeting, San Antonio, TX, 1998
- R. J. Kerans, "Damage Development, Toughening, and the Design and Performance of Ceramic Composite Systems (Keynote Presentation)," ASME Annual Meeting, Dallas TX, Nov 16-21 1997.

R. J. Kerans, T. A. Parthasarathy and R. S. Hay, "Crack Deflection in Ceramic Composites and Fiber Coating Design Criteria," 5<sup>th</sup> Japan International SAMPE Symposium & Exhibition (JISSE-5), Tokyo Big Sight, Tokyo, Japan, October 28-31 1997.

R. S. Hay, J. R. Welch, and M. K. Cinibulk, "Characterization of Ceramic Coatings on Ceramic Fiber Tows," ICMCTF, April, 1997

M. K. Cinibulk, J. R. Welch, and R. S. Hay, "Preparation of TEM Thin Sections of Coated Ceramic Fibers," MRS Spring, SF 1997

R.J. Kerans and T.A. Parthasarathy, "Debond Crack Roughness, Interface Properties and Fiber Coating Design in Ceramic Composites", International Conference on Ceramic and Metal Matrix Composites CMMC-96, San Sebastian, Spain, 9-12 September 1996

R. S. Hay, "Characteristics of Sol and Solution Derived Ceramic Fiber Coatings," 2nd Int. Mtg. Pacific Rim Cer. Soc., July 15-17, 1996

R.J Kerans, "Fiber Coating Design Parameters for Ceramic Composites as Implied by Considerations of Debond Crack Roughness" Ceramic Microstructures '96: Control at the Atomic Level, Berkeley, CA 24-27 June

R. J. Kerans and R. S. Hay, "Oxidation Resistant Fiber/Matrix Interface Control for Ceramic Composites," AEROMAT '96, June 5, 1996.

R.J. Kerans, "Fiber/Matrix Interface Properties and the Design of Fiber Coatings for Ceramic Composites" ICCI-V Microphenomena in Advanced Interfaces, Zichron Yaacov, Israel, 5-8 May 1996

R. S. Hay, "Fiber-Matrix Interface Concepts for CMCs," Materials Science Seminar, U. Dayton, Feb. 1, 1996

R. S. Hay, "Fiber-Matrix Interfaces for CMCs," National Materials Advisory Board, Committee on Advanced Fibers for High-Temperature Ceramic Composites, Cocoa Beach, FL, Jan 1996

R. S. Hay, "Progress in Oxidation Resistant Fiber Coatings," Materials Science Seminar, U. Illinois, Oct. 26, 1995.

R. J. Kerans, "The Role of Fiber/Matrix Interfacial Topography in Determining Composite Design Parameters", Keynote Lecture, 4th International Conference on Interfacial Phenomena in Composite Materials, Eindhoven, The Netherlands, 10-13 September 1995.

R.J. Kerans, "Design of Fiber Coatings for Control of Interface properties in Ceramic Composites", ASME 1995 Summer Mechanics and Materials Joint Meeting, Univ. Calif. Los Angeles, 28-30 June 1995

R. J. Kerans, "Design of Fiber Coatings for Control of Interface Properties in Ceramic Composites," 97th Annual Meeting of the American Ceramic Society", Cincinnati, OH, 29 April-4 May 1995

R. J. Kerans, "Design of Fiber Coatings for Control of Interface Properties in Ceramic Composites," Graduate Seminar, Carnegie Mellon University, 14 March 1995

R. J. Kerans, "Design of Fiber Coatings for Control of Interface Properties in Ceramic Composites," Graduate Seminar, University of Cincinnati, 3 Feb 1995

R. J. Kerans, "Progress and Challenges in Fiber/Matrix Interface Control in Ceramic Composites," Graduate Seminar, Pennsylvania State Univ., 17 Nov 1994

R. J. Kerans, "Progress and Challenges in Fiber/Matrix Interface Control in Ceramic Composites," Keynote Lecture, Basic Sciences Div. Annual Meeting, Am. Ceram. Soc., Louisville, KY, 26-29 September 1994.

R. J. Kerans, "Control of Fiber-Matrix Interface Properties in Ceramic Composites," First International Conference on Composite Engineering, New Orleans, LA, USA, 28 - 31 August 1994.

M.K. Cinibulk, "Magnetoplumbite-Type Compounds as a Fiber Coating in Oxide/Oxide Composites," Structural Ceramics Group Seminar, Rockwell Science Center, Thousand Oaks, CA, May 1994.

R. J. Kerans, "Control of Fiber-Matrix Interface Properties in Ceramic Composites," Graduate Seminar, University of Illinois, 18 March 1994.

R. J. Kerans, "Effects of Fiber/Matrix Interface Topography and Debonding in Ceramic Composites", ARPA University Research Initiative Review, University of California at Santa Barbara, 7 January 1994.

R. J. Kerans, "Control of Fiber Matrix Interface Properties in Ceramic Composites", ARPA, University Research Initiative Review, University of California at Santa Barbara, 6 January 1994.

R. S. Hay, "Deformation During Solid-State Reactions with Volume Expansion," Materials Science Colloquium, Case Western Reserve University, Aug. 5 (1993).

T.A. Parthasarathy, R. J. Kerans, P.D. Jero and D.B. Marshall, "Friction in Fiber/Matrix Interfaces in Ceramic Composites", PAC Rim Meeting, Am. Ceram. Soc., Honolulu, HI, 7-10 November 1993.

P.D. Jero, R.J. Kerans and T.A. Parthasarathy, "Interface Evaluation in Ceramic Composites", Symposium on Mechanics and Ultrasonic Characterization of Interfaces", ASME Winter Meeting, New Orleans, LA, 28 November

R.J. Kerans, "Control of Fiber-Matrix Interface Properties in Ceramic Composites," International Conference on High Temperature Ceramic Matrix Composites, 6th European Conference on Composite Materials, Bordeaux, France, 21 September 1993.

R. J. Kerans, "Issues in the Control of Fiber/Matrix Interfaces in Ceramic Composites", 2nd International Workshop on Interfaces, Santiago, Galicia, Spain, 27-29 September 1993.

R. S. Hay, "Deformation During Solid-State Reactions with Volume Expansion," Materials Science Colloquium, Case Western Reserve University, Aug. 5 (1993).

R.J. Kerans, "Effects of the Fiber-Matrix Interface on Measured Interface Properties and the Behavior of Ceramic Composites," Gordon Research Conference on Solid State Studies in Ceramics, August 1993.

R.J. Kerans, "Fiber-Matrix Interface Control in Ceramic Composites," 95th Annual Meeting of the American Ceramic Society, Cincinnati, OH, 24 April 1993.

M. Ruhle, H.-J. Kleebe, M.K. Cinibulk, and I. Tanaka, "High-Resolution Electron Microscopy of Grain-Boundary Films in Silicon Nitride Ceramics," Fall Meeting of Materials Research Society, December 1992.

M.K. Cinibulk, "Intergranular Phases in Silicon Nitride Ceramics and Their High-Temperature Stability," Institut für Keramik im Maschinenbau Seminar, Universität Karlsruhe, Germany, December 1992.

M.K. Cinibulk, "Intergranular Phases and Their Influence on the High-Temperature Behavior of Silicon Nitride Ceramics," Materials Science Division Seminar, Lawrence Livermore National Laboratory, October 1992.

R.J. Kerans, "Issues in the Control of Fiber-Matrix Interface Properties in Ceramic Composites," International Workshop on Advances in Inorganic Fiber Technology, Melbourne, Australia, 13 August 1992.

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface in Composites," Graduate Seminar, The University of Dayton, 19 October 1992.

M.K. Cinibulk, "Experimental Studies on Intergranular Films in Silicon Nitride Ceramics," Advanced Ceramics Research Laboratory, Dow Chemical Co., Midland, MI, April 1992.

M.K. Cinibulk, "Effects of Oxidation on Microstructure and Properties of Silicon Nitride Ceramics," Center for Materials Science Seminar, Los Alamos National Laboratory, Los Alamos, NM, April 1992

P.D. Jero, T.A. Parthasarathy and R.J. Kerans, "Measurement of Interface Properties from Fiber Pushout Experiments," Graduate Seminar, University of Illinois, 20 March 1992.

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface in Composites," Seminar, National Institute of Standards and Technology, 2 December 1991

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface in Composites," Graduate Seminar, The Pennsylvania State University, 20 November 1991.

R. S. Hay, "Phase and Microstructure Evolution in Sol-Gel Derived Yttrium-Aluminum Garnet Thin Films," Materials Science Colloquium, Case Western Reserve University, Nov. 5 (1991).

R.J. Kerans, "Issues in the Development of Structural Ceramic Composites," Centennial Symposium on Materials Science and Engineering, Centennial Annual Meeting of the Ohio Academy of Sciences, The Ohio State University, 28 April 1991.

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface," Seminar, Laboratoire des Composites Thermostructuraux (University of Bordeaux), Bordeaux, France, 18 April 1991.

R.J. Kerans, "Oxidation of Non-Oxide Reinforcements in Oxide-Matrix Composites," Seminar, Laboratoire des Composites Thermostructuraux (University of Bordeaux), Bordeaux, France, 18 April 1991.

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface," Seminar, LERMAT (University of Caen), Caen, France, 17 April 1991.

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface," Seminar, Rolls-Royce Advanced Ceramics Laboratory, (University of Warwick), Warwick, England, 13 April 1991.

R.J. Kerans, "Oxidation of Non-Oxide Reinforcements in Oxide-Matrix Composites," Seminar, Rolls-Royce Advanced Ceramics Laboratory, (University of Warwick), Warwick, England, 13 April 1991.

R.J. Kerans, "Fracture and Sliding in the Fiber-Matrix Interface and Failure Processes in Ceramic Composites," Plenary Lecture, Annual Convention of The Institute of Ceramics (UK), Oxford University, Oxford England, 10 April 1991.

R. S. Hay, "Chemically Induced Grain Boundary Migration," Materials Science Colloquium, Ohio State University, May 10 (1990).

R.J. Kerans, "The Role of the Fiber-Matrix Interface in Brittle-Matrix Composites," Graduate Seminar, University of Cincinnati, 25 May 1990.

R.J. Kerans, R. S. Hay, N. J. Pagano, and T. A. Parthasarathy, "The Role of the Fiber-Matrix Interface in Ceramic Composites," Short Course/Workshop on High Temperature Ceramic and Metallic Matrix Composites", Michigan State University, 12 December 1989.

R.J. Kerans, "Processing of Ceramic Composites: The State of the Art and the Challenges," Symposium on The State of the Art in Materials Processing, ASM Materials Week, 5 October 1989.

## Inventions and Patents

- <sup>1</sup>R. Kerans and T. A. Parthasarathy, Hybrid Composite Materials, Patent No. Air Force Invention No. AFD 552, USA Patent Filed (2002).
- <sup>2</sup>D. M. Dimiduk, T. A. Parthasarathy, M. Uchic, and S. Rao, Comprehensive Design Tool Methodology for Rapid Assessment of Structural Capability of Materials, Patent No. AF Invention Disclosure AFD 594, USA - Patent Filed (2002).
- <sup>3</sup>T. A. Parthasarathy and R. J. Kerans, Thermal Gradient Resistant Ceramic Composite, Patent No. Patent No. 6,251,815, USA (2001).
- <sup>4</sup>T. Mah, T. A. Parthasarathy, and M. K. Cinibulk, Ceramic Matrix Composite Cutting Tool, Patent No. US Patent, USA (2001).
- <sup>5</sup>M. K. Cinibulk and R. S. Hay, Method of Forming Tubular Inclusions in Single-Crystal Alumina, Patent No. US Patent No. 5894035, USA (1999).
- <sup>6</sup>R. S. Hay and M. D. Petry, Coating Apparatus for Continuous Tow, Patent No. U. S. Patent #5,951,764, USA (1999).
- <sup>7</sup>E. Boakye, R. S. Hay, and M. D. Petry, Ceramic Composite, Patent No. U. S. Patent #5,759,632, USA (1998).
- <sup>8</sup>R. S. Hay, Method for Coating Continuous Tows, Patent No. U. S. Patent #5,217,533, USA (1993).
- <sup>9</sup>R. S. Hay and E. E. Hermes, Coating Apparatus and Method for Continuous Fibers, Patent No. U. S. Patent #5,217,533, USA (1993).

## Selected Professional Activities and Awards

Associate Editors, JI of Amer. Ceram. Soc., T.A.Parthasarathy and R.J.Kerans

Fellow of the American Ceramic Society, R.J. Kerans

Fellow of the Air Force Research Laboratory, R.J. Kerans

AFRL/ML John J. Cleary Awards for Excellence in Research, R.S. Hay 1996, P.D. Jero and R.J. Kerans, 1993, (2 Finalists)

AFRL/ML Robert T. Schwartz Award for Excellence in Engineering, L.E. Matson, 1994

Affiliate Societies Council Scientist of the Year, R.J. Kerans

Air Force Basic Research Award Runner-Up

Organized and Co-Sponsored with NASA/GRC and DoE/ORNL the Interfaces Working Group

Gordon Conference Presentations (3), Gordon Conference Moderator (2)

Feature Article in J. Am. Ceram. Soc. (in press)

Invited Article in *Current Opinion in Solid State and Materials Science*, R.J. Kerans, R.S. Hay and T.A. Parthasarathy

Organizing or Advisory Boards for 3 International Conferences