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Composites and Advanced Ceramic Materials

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Program Chair

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Each issue of *Ceramic Engineering and Science Proceedings* includes a collection of technical articles in a general area of interest, such as glass, engineering ceramics, and refractories. These articles are of practical value for the ceramic industries. The issues are based on the proceedings of a conference. Both The American Ceramic Society, Inc., and non-Society conferences provide these technical articles. Each issue is organized by an editor who selects and edits material from the conference. Some issues may not be complete representations of the conference proceedings. There is no other review prior to publication.

Preface

The Fourteenth Annual Conference on Composites and Advanced Ceramics, held 14–17 January 1990 in Cocoa Beach, FL, was a great success. One hundred three papers and 23 posters were presented during the meeting. Ninety of the total are published in these two issues of *Ceramic Engineering and Science Proceedings.* The technical sessions covered all aspects of ceramic matrix composites as well as processing/microstructure/property relationships in monolithic ceramics.

The James I. Mueller lecture, "Is There Anything of Practical Value Hidden Amongst the Composite Toughening Theories? A Jim Mueller Perspective," was presented by Frank Gac. This lecture was followed by the Plenary session that included presentations from DOD, DARPA, DOE, NASA, AND NIST, followed by a talk on the status of the Engineering Ceramics Division by Bill Payne, the president of ACerS. The panel discussion, organized by Ron Barks, "The Business of Technology: Accelerating Development with Acquired Technology," was well attended. Summaries of the plenary and panel discussion presentations are included in these Proceedings. The papers given by the invited speakers are also included.

A one-day special seminar on "Reaction-Based Processing" was organized by Dick Spriggs that included 16 papers on these exciting processing techniques.

For the first time, ECD presented awards for the Best Overall Presentation in Student and Non-Student Categories. The winner in the Student Category was "Mechanical Behavior of Nicalon-Reinforced Calcium Aluminosilicate Composites," presented by S. -W. Wang, a graduate student at the University of Delaware, and co-authored by A. Parvizi-Majidi. In the Non-Student Category, the winner was "Room-Temperature Tensile and Fatigue Properties of Silicon Carbide Fiber-Reinforced Aluminosilicate Glass," presented by Larry Zawada of the Air Force Materials Lab and co-authored by Larry Butkus of AFML and George Hartman of University of Dayton Research Institute.

Awards for Best Poster were also presented. The winners were: 1st Prize Student Category for "Finite Element Studies of Crack Deflection in Ceramics," by J. S. Lyons, T. L. Starr, and C. W. Meyers of the Georgia Institute of Technology; 2nd Prize Student Category for "Effect of Green Microstructure on Microwave Processing of Alumina," by A. S. De, I. Ahmad, D. E. Clark, and E. D. Whitney of the University of Florida; 1st Prize Non-Student Category for "Microstructural Characterization of a New VLS TiN Whisker Product," by T. A. Nolan,

L. F. Allard, and D. W. Coffey of Oak Ridge National Lab; and 2nd Prize Non-Student Category for "Creep and Oxidation of SiC Whisker-Reinforced Alumina," by H. Hubner and O. K. Lorenz of Technische Universität Hamburg-Hamburg. All the award-winning presentations and posters are presented in these Proceedings.

Kaycee Logan, who organized the poster session and chaired the awards committee, deserves a special thanks for a job well done. I would also like to thank the session chairs who performed an invaluable service and also reviewed all the manuscripts, and the student pages who are so essential to the smooth operation of the meeting. Finally, I would like to thank the authors, without whom the meeting would never have happened.

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(Editor's Note: Proceedings of the 14th Annual Conference on Composites and Advanced Ceramic Materials appear in both this issue and in *Ceramic Engineering and Science Proceedings*, volume 11, number 9–10.)

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John B. Wachtman

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Is There Anything of Practical Value Hidden Amongst the Composite-Toughening Theories?!-A Jim Mueller Perspective

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Numerous theories have been developed over the last three decades for explaining the toughening behavior of discontinuous fiber-reinforced brittle matrix composites. The issue is the practical engineering utility of these theories. Upon compiling a table of fiber parameters that are identified in the predominant toughening mechanisms, a number of important features becomes evident for achieving high toughnesses. First, all of the mechanisms indicate that a high-fiber volume fraction is desirable. Second, residual stresses appear to influence all of the composite-toughening mechanisms. Third, the highest fiber tensile strength is preferred. Finally, fiber diameter and fiber-matrix interfacial shear strength are also important, but both are composite system- and toughening mechanism-specific.

Introduction

It is a tremendous honor to present the 1990 James I. Mueller Memorial lecture. It is especially flattering to be the first student of Jim's to do such.

Nearly four years have passed since his death. It is a certainty that many of you knew Jim, and it is also likely that some of you have little or no idea who this man was. The following brief biography will stir up memories for some and provide new information for others.

James I. Mueller was born to August L. and Lydia (Heyn) Mueller on June 26, 1916 (Fig. 1).* Jim received his B.S. in 1939 from The Ohio State University and Ph.D. in 1949 from the University of Missouri-Rolla. Both degrees were in Ceramic Engineering.

*The author did not realize until preparing this lecture that his son, Benjamin Thomas Gac, and James I. Mueller share the same birthday. Benjamin, however, arrived on the scene 64 years later.

Jim's accomplishments and awards were many. His career was centered around the University of Washington, which he joined in 1949 as an assistant professor, with the assignment of developing a ceramic engineering curriculum (Fig. 2). As a result of his efforts, the Ceramic Engineering Division was created within what was then the Department of Mining, Metallurgical, and Ceramic Engineering, and is now termed the Department of Materials Science and Engineering. Many a renowned student has been produced by that department over the years, and "Doc," as his students affectionately called him, no doubt influenced all of them. One example is astronaut Dr. Bonnie J. Dunbar, who during the presentation of this lecture was orbiting the earth aboard the space shuttle Columbia! Bonnie was part of a five-member crew who had two primary objectives: (1) the deployment of SYNCOM, a communications satellite, and (2) most importantly from the standpoint of materials types, the long-awaited retrieval of the Long Duration Exposure Facility (LDEF).

Space—what an exciting frontier! Doc was best known for his interest in the space program (Fig. 3). From 1963–1987, he received continuous funding from NASA totalling nearly $8 million. That translates to roughly $240 000 a year. One especially noteworthy outcome from that research was the development of an innovative, interdisciplinary program entitled Brittle Materials Design. In fact, it was that program that attracted the author to the University of Washington to pursue a Ph.D. Yet another noteworthy development was the research Jim directed on addressing the attachment problems associated with the space shuttle's thermal protection tiles. This work resulted in Jim's being awarded NASA's Public Service Medal in 1981.

Jim was also a devoted member of the American Ceramic Society. One cannot begin to review all of the committees and programs he participated in. Jim's efforts did not go unnoticed. He was recognized both as a Fellow and a Distinguished Life Member of the American Ceramic Society, received numerous other awards, and served as the President of the Society in 1981-1982.

Jim had a propensity for the practical. He displayed an impressive ability to identify national and worldwide trends in materials and boil these trends down to practical issues. These issues might be technical or administrative in nature, which could then be addressed by an individual *or preferably a team of individuals.* Today's Cocoa Beach meeting, the Engineering Ceramics Division of the American Ceramic Society, and the United States Advanced Ceramics Association (Jim, incidentally, was USACA's first president in 1985) were no doubt heavily influenced by Jim Mueller's foresight and practicality.

The next section will demonstrate how Jim Mueller's practical perspective pervades the author's approach to ceramic engineering, and, more specifically, influenced a topic of particular interest to the attendees of the Fourteenth Annual Meeting on Composites and Advanced Ceramic Materials, namely: ceramic composite development. The question at hand is, "Is there anything of practical value hidden amongst the composite-toughening theories?"

Composite-Toughening Mechanisms

Numerous concepts have been proposed for toughening and, to a certain extent, for strengthening ceramic matrix composites. All of the concepts can be generalized into one or more of three basic mechanisms (Fig. 4). One mechanism embodies increasing the local driving force necessary to propagate a crack to failure. This could be accomplished by imposing a compressive stress state on the ceramic, for example, by shrink fitting a metallic sleeve around a ceramic rod. A second mechanism involves increasing the mechanical energy consumed per unit area of crack propagation. The incorporation of a ductile phase in a ceramic would satisfy this criteria because mechanical energy would be consumed in plastically deforming the ductile phase during crack propagation. The third mechanism involved decreasing the local strain by cracking, which reduces the crack-tip stress concentration. Matrix microcracking without catastrophic fracture obviously satisfies this criteria. The issue, of course, is what is the engineering utility of the microcracked composite?

The predominant toughening concepts are enumerated in Fig. 5. The remainder of this paper will elaborate on the practical fundamentals of most of these concepts and culminate with a table that one can use as a guide for the development of fiber- or whisker-reinforced ceramic matrix composites.

Modulus Transfer

Modulus transfer operates on the basis of transferring the applied load from a lower elastic modulus matrix to the higher elastic modulus fibers, to achieve strain uniformity within the structure (i.e., strain in composite = fiber strain = matrix strain). A strong, nonslipping fiber-matrix interface is required for this mechanism to operate effectively.

For a composite reinforced with continuous, unidirectionally aligned fibers, the corresponding stress in the composite is given by:

$$\sigma_c = \sigma_m \left[(1 - V_f) + V_f \frac{E_f}{E_m} \right] \tag{1}$$

where σ_c is the stress in the composite, σ_m is the stress in the matrix, V_f is the volume fraction of fibers, and E_f, and E_m are the modulus of elasticity of the fiber and matrix, respectively. Examination of this expression reveals that the greatest improvement in the composite strength would occur when the elastic modulus of the fiber is much greater than the elastic modulus of the matrix. In addition, the higher the fiber volume fraction the better (up to a practical limit, of course).

A potential composite that could benefit from the modulus transfer concept is the silicon carbide (SiC) whisker-reaction bonded silicon nitride (RBSN) matrix system. The elastic modulus of VLS SiC whiskers has been reported to be 581 GPa (84.3 Mpsi)[2] and that of RBSN is reported to vary over the range of 97–221 GPa (14.1–32.1 Mpsi).[3] Thus, the elastic modulus of the SiC whiskers is 3–6 times greater than that of the RBSN. Hence, strengthening by the modulus transfer concept is conceivable. The difficulty arises in maintaining the SiC whisker integrity during RBSN fabrication.

Toughening is accomplished through this concept simply because an increase in local driving force is required to propagate a crack through the composite system, as increased load is being accommodated by the fiber. When dealing with discontinuous fiber- or whisker-reinforced composites, one must also address the load that fiber of a given length can bear and the stress concentrations that occur at the fiber ends. This concept was nicely illustrated by Schuster[4] in a composite consisting of a 25-μm diameter by 3 000-μm-long sapphire whisker in a photoelastic resin matrix (Fig. 6). The elastic modulus ratio of the whisker to the matrix is approximately 125. The matrix stress reduction along the length of the whisker is roughly a factor of two, whereas the stress is intensified at the whisker tips by a factor of three.

Fiber Pullout

An expression has been developed for the fiber pullout theory which defines the maximum work-of-fracture (WOF) one can achieve in a composite.[5–10] This is important because the WOF is an indication of the toughness (or more specifically, the *R*-curve behavior) of the composite. The expression is:

$$WOF_p = \frac{V_f \sigma_f^2 r_f}{12\tau} \tag{2}$$

where WOF_p is the maximum work-of-fracture for fiber pullout, r_f is the fiber radius, τ is the fiber-matrix interfacial shear strength, and all other terms are as previously devined. This expression states that the composite "toughness" will be enhanced with large values of fiber volume fraction, high fiber strength, a small interfacial shear strength, and, interestingly enough, a large fiber radius.

Crack Bridging

Another important toughening concept that is receiving a lot of attention is crack bridging. The crack bridging theory[11–13] postulates that intact reinforcements behind the primary crack front will bridge the crack surfaces in the following wake region, thus inhibiting further crack opening and reducing the stress intensity at the crack tip. Evans and McMeeking[12] have proposed three bounding solutions for this model: (1) frictional bridging resulting from an unbonded fiber, (2) strong particle bridging, and (3) ductile particle bridging.

Frictional Bridging: As shown in Fig. 7, when the reinforcing fibers are unbonded and fiber motion is restrained by friction, then the critical stress intensity factor (toughness), K_c, for frictional bridging can be approximated by:

$$K_c \approx (\sigma_f^3 G/3E_f\tau)^{1/2}\sqrt{r_f A_f} \tag{3}$$

where G is the elastic shear modulus of the composite, A_f is the areal fraction of reinforcements on the crack plane (an indication of the volume fraction of reinforcements) and all other terms are as previously defined.

Strong Particle Bridging: Figure 8 shows that the critical stress intensity factor for the strong particle bridging case is approximated by:

$$k_c \approx 1.1\sigma_f\sqrt{r_f A_f(1 - \sqrt{A_f})(1 - A_f)} \tag{4}$$

where now the "f" subscript simply refers to the reinforcement, be it a whisker or a particle. This expression represents the situation where the interfacial shear strength goes to infinity, such as for whiskers that are strongly bonded to a matrix. It should be noted that the bridging particle does not need to be tougher than the matrix for this mechanism to operate. It must only be stronger.

Ductile Particle Bridging: When the particle is "tougher" than the matrix, the situation can be described by a ductile-particle-extension bridging mechanism (Fig. 9). The expression for that critical stress intensity factor is approximated by:

$$K_c \approx \sqrt{\Omega C A_f \sigma_y G r_f (0.5 + \exp(\epsilon_f))} \tag{5}$$

where Ω is an empirical factor related to the ductility and size of the ductile particle, C is a constraint factor that is believed to be of the order of 6–8 for crack pinning by ductile particle reinforcement, σ_y is the yield stress of the particle, ϵ_f is the particle failure strain, and all other terms are as previously defined.

One central result emerges from three crack-bridging mechanisms: toughening will increase with increasing reinforcement strength, increasing reinforcement size or diameter, and increasing reinforcement volume fraction.

Crack Arresting/Blunting

Yet another toughening mechanism is crack arresting or blunting. On one hand, this can be viewed as a generalized form of crack bridging in that it represents the extreme case where the particles have sufficient strength and toughness to completely resist fracture. On the other hand, it represents a situation where crack propagation is eliminated by removing the stress concentration at the crack tip, such as by introducing a hole (e.g., a circular or spherical pore) ahead of a propagating crack. The issue is one of composite performance criteria. A particle which displays sufficient toughness to arrest cracking at ambient temperature, e.g., a metal, may be much too plastic and/or lack environmental resistance at elevated temperatures. Conversely, refractory reinforcements are typically brittle at ambient temperature, and thus, lack the fracture toughness necessary to completely arrest fracture in that temperature regime. Finally, the presence of porosity may be advantageous from the standpoint of toughness but can have an undesirable effect on strength.

Crack Bowing

The crack bowing concept[14–16] is related to crack bridging in that it is also a crack impediment process. As described by Faber and Evans:[17]

> "Crack bowing originates from resistant second phase particles in the path of a propagating crack. The crack tends to bow between the particles, causing the stress intensity along the bowed segment of the crack to decrease (while resulting in a

corresponding increase in the stress intensity at the particle). The degree of bowing increases until the fracture toughness of the particle is reached, whereupon crack advance ensues."

The crack bowing concept is illustrated in Fig. 10, with a sketch taken from a publication by Lange.[14] In this work, Lange proposed a model for crack bowing that was based upon the hypothesis that a crack front possesses a line energy similar to a dislocation. The result of this work is contained in the following equation:

$$G_c = 2\left[\gamma_o + \frac{T}{D}\right] \tag{6}$$

where G_c is the amount of energy required to extend a crack a unit length (i.e., the fracture energy), γ_o is the surface energy of the matrix material, T is the line energy per unit length of crack front, and D is the distance between the second-phase particles. This expression indicates that a brittle material's resistance to fracture, i.e., toughness, increases with a decreasing mean free path between the particles, and hence, an increasing volume fraction of particles. This implies a particle-size dependence in that at a given volume fraction, smaller particles will provide for more crack-pinning sites per unit volume. Lange also concluded that significant toughening does not occur unless the particle spacing is significantly less than the flaw size.

Crack Deflection

The crack-deflection concept[17–20] is related to the crack-bowing concept in that it similarly addresses the interaction of a propagating crack with a second-phase inclusion or particle. The distinction lies in the fact that crack deflection produces a nonplanar (twisted) crack, as illustrated in Fig. 11, whereas crack bowing only produces a nonlinear crack front. The nonplanar crack arises either from residual stresses present in the material and/or from the existence of weakened interfaces.

Faber and Evans[17,18] conducted a systematic study of crack deflection in an attempt to develop a model for the phenomenon. The model is based on fracture mechanic principles and consists of two parts, a crack tilt function and a crack twist function. They assumed that when a crack approaches or intercepts a microstructural inhomogeneity, it will tilt at an angle that depends on the orientation and position of the particle with respect to the advancing crack, as well as upon the nature of the residual stress that may be present between the particle and the matrix. The tilted crack is subject to mixed-mode local loading, characterized by Mode I (opening) and Mode II (sliding) contributions to the stress intensity. Faber and Evans then assumed that subsequent advance of the crack may result in crack front twist and additional tilt, depending on the orientation, position, and stress state of adjacent particles. The twisted crack contains both Mode I and Mode III (tearing) stress intensity components. They proposed that the increase in fracture toughness imparted by crack deflection could then be determined by evaluating the local stress intensities at the tilted and twisted portions of the crack front. Faber and Evans proceeded with this analysis and also incorporated particle morphology effects by investigating three dominant morphologies: a sphere, a rod, and a disk.

As a result of their analysis of crack deflection, Faber and Evans[17] presented a number of important conclusions. The increase in toughness imparted by crack deflection depends on particle shape and the volume fraction of the second phase. The most effective particle morphology for deflecting cracks is the rod of "high" aspect ratio. However, the toughening increase tends to saturate at aspect ratios greater than about 12. Similarly, toughening saturates at fiber volume fractions of about 20%. The Faber-Evans analysis also predicted that toughening will be independent of fiber size. However, some question exists on this prediction, since at a given volume fraction, smaller particles will provide for more crack interaction sites. Finally, the Faber-Evans analysis made no prediction as to the effect of interfacial bond strength on toughening, although it is certainly understood that weakened interfaces directly influence the propensity for crack deflection.

Matrix Microcracking

The phenomenon of matrix microcracking occurs because of thermal expansion differences, phase transformations, and differences in elastic moduli between the matrix and a second phase.[21–24] The refractories industry has capitalized on microcracking for years to achieve thermal shock-resistant bodies. The microcracks in these systems are often preexisting and large in size, resulting in relatively weak bodies. In more recent years, the generation of small microcracks at, or near, a main crack tip has become a topic of interest. Microcracks occur from the superposition of the high tensile stresses concentrated near the crack tip with the intrinsic mismatch stresses, resulting in a microcracked "process" zone around the crack tip, as illustrated in Fig. 12.

A first order estimate of the upper bound of fracture energy increase, ΔG_{mc}, due to microcracking (per unit area of advance of the main crack), was determined by Rice.[21] The result is:

$$\Delta G_{mc} = \frac{2\pi a \gamma_{mc} V_f L}{d} \tag{7}$$

where L and aL are the major and minor axes of an elliptical microcrack process zone (a is <1), γ_{mc} is the microcrack interfacial fracture energy, V_f is the volume fraction of second-phase particles, and d is the diameter of the second-phase particles. Thus, ΔG_{mc} is directly proportional to the boundary feature energy, the volume fraction of particles which induce the microcracks, and the process zone size. It is inversely proportional to the diameter of the particles.

Residual Stress Effects

The importance of elastic modulus differences have already been discussed, and the influence of phase transformations and thermal expansion differences have been alluded to. Residual stresses resulting from thermal expansion differences merit additional discussion.

Thermal expansion differences can result in residual stresses which may directly influence the load-bearing and toughening characteristics of a composite. For a two-dimensional problem in plane strain consisting of a circular reinforcement particle (fiber) embedded in an infinite matrix, the fiber experiences a radial stress, σ_r, which can be approximated by:[25]

$$\sigma_r = \frac{(\alpha_m - \alpha_f)\Delta T E_m}{(1 + \nu_m) + (1 + \nu_f)(E_m/E_f)} \tag{8}$$

where α_m is the thermal expansion coefficient of the matrix, α_f is the thermal expansion coefficient of the fiber, ΔT is the change in temperature, ν_m is the Poisson's ration of the matrix, ν_f is the Poisson's ratio of the fiber, and E_m and E_f are the same as previously defined.

When $\alpha_f > \alpha_m$, cooling from the processing temperature places the fiber in tension and the matrix in compression. Silicon carbide whiskers in a silicon nitride or cordierite matrix are examples of this situation. In this scenario, an increased local driving force would be necessary for crack propagation to occur through the matrix. If the fiber-matrix mismatch is too large and/or the interfacial bond is too weak, then cracks may develop around the fiber, essentially debonding the fiber from the matrix. This could be beneficial to toughening by a fiber pullout mechanism, but it would probably inhibit significant strengthening. Another point to consider is that if the interfacial bond strength is so large that the matrix is maintained in compression, then the fiber is "pre-loaded" in tension. This may reduce the overall tensile load that the fiber can sustain within the composite and the strength of the composite may not achieve its optimal level.

If $\alpha_f < \alpha_m$, cooling from the processing temperature places the fiber in compression and the matrix in tension. This is the situation for SiC whiskers reinforcing alumina, mullite, molybdenum disilicide, magnesium aluminate spinel, or zirconia matrices. This scenario is believed to enhance toughening by matrix microcracking and crack multiplication. It could also enhance toughening by promoting crack bridging by the fiber, since greater applied stresses must be imposed on the fibers to attain their fracture stresses.

Summary of Toughening Theory Predictions

Table I summarizes the toughening predictions for discontinuous fiber-reinforced ceramic matrix composites. From the practical materials engineering viewpoint, a number of features are evident in this tabular summary. First, all mechanisms dictate that a high-fiber volume fraction is desirable for achieving maximum toughness, i.e., "more is better." Hence, processing research should be directed at effectively incorporating high-volume loadings (at least 10 and preferably >20 vol%) of fibers within ceramic matrices. Second, residual stresses appear to influence all of the composite toughening mechanisms, although few relationships have been developed for quantitatively predicting the residual stresses or their effects on composite toughness. Thus, research is warranted in this area. In spite of this lack of predictive capabilities, the competent ceramic matrix composite designer should at least be cognizant of the influence that residual stresses may have on his composite system. Third, it appears that the highest fiber tensile strength is advantageous, i.e., "stronger is better." Thus, a high-strength, single-crystal SiC whisker would be preferred over a chopped, polycrystalline SiC fiber, whose strength is inherently lower. Finally, the fiber diameter and fiber-matrix interfacial shear strength also appear to be important, but both are composite system and toughening mechanism specific.

Table I. Influence of Fiber Parameters on the Toughness of Ceramic Matrix Composites

Mechanisms	Fiber Parameters							Residual Stress Parameters		Influence on Composite K_c
	ℓ/d	V_f	$d = 2r_f$	σ_f	τ	f-spac.	K_f	E_f/E_m	α_f/α_m	
Modulus Transfer		↑		↑	↑			≥2		↑
Fiber Pull-Out	max. K_c@ $(\ell/d)_{cp} = \sigma_f/2\tau$	↑	↑	↑	↓			important		↑
Crack Bridging		↑	↑	↑	strong (but limited)	[illegible]	*	important		↑
Crack Arresting/Blunting		"↑"				"↓"	"↑"	important		↑
Crack Bowing		↑	"↓"			↓	"↑"	important		↑
Crack Deflection	↑ (sat. @12)	↑ (sat @20)	independent (controversial)		no prediction (but important)			important		↑
Matrix Microcracking		↑	↓					"<1"	"<1"	↑

*The importance of the fiber toughness depends on the type of crack bridging mechanism that is operating.

Note 1: ℓ/d = fiber length-to-diameter ratio, V_f = fiber volume fraction, d = fiber diameter, r_f = fiber radius, σ_f = fiber tensile strength, τ = fiber-matrix interfacial shear strength, f-spac. = distance between fibers, K_f = fiber fracture toughness, E_f = fiber Young's modulus, E_m = matrix Young's modulus, α_f = fiber coefficient of thermal expansion, α_m = matrix coefficient of thermal expansion, K_c = critical stress intensity (toughness) of composite.

Note 2: Symbols surrounded by quotations represent intuitive predictions; all other predictions are the result of theoretical models.