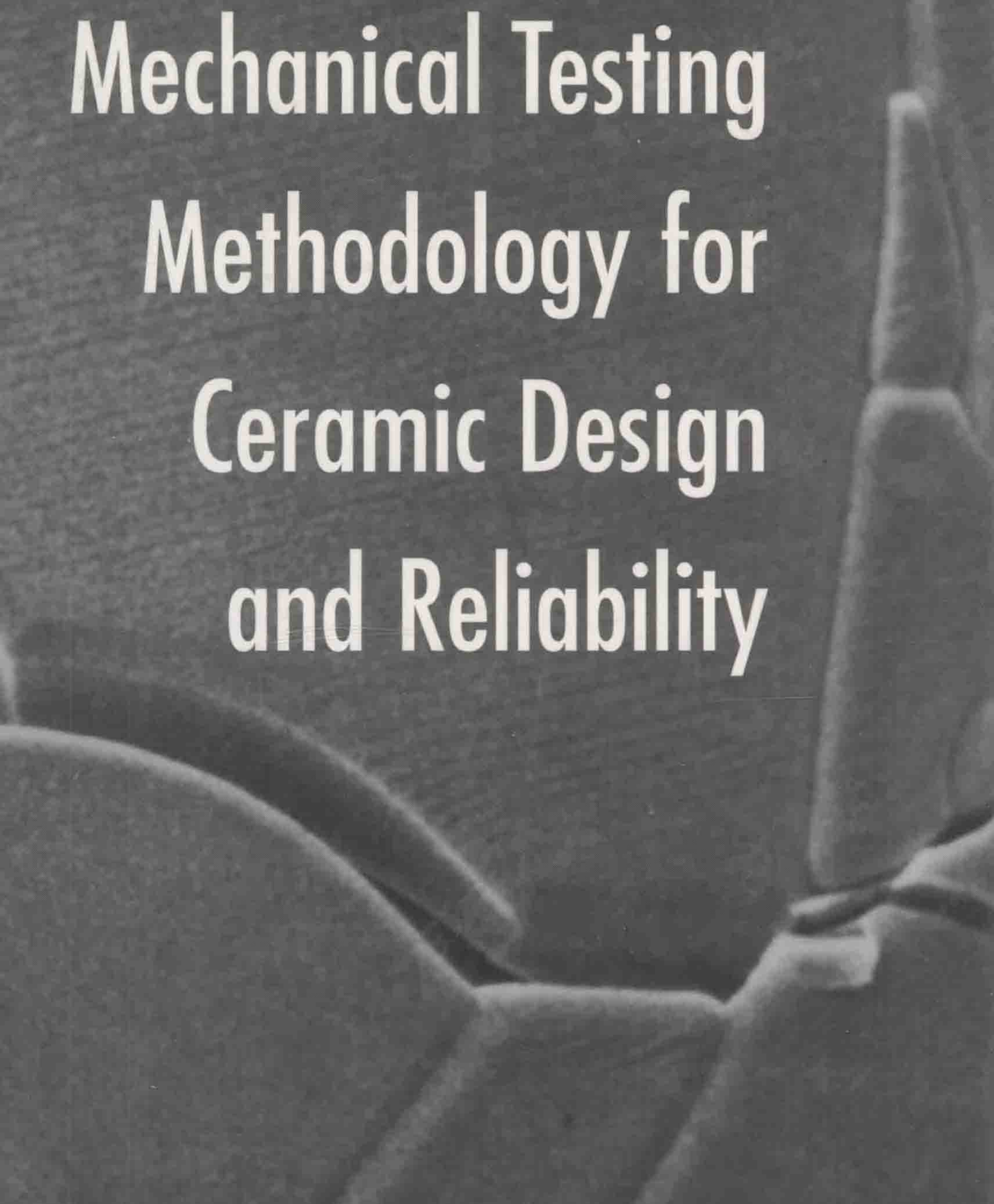

Mechanical Testing Methodology for Ceramic Design and Reliability

edited by

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Preface

Mechanical properties testing has been a significant part of ceramic technology since the discovery of ceramics and glasses. Over the years, substantial progress has been made in our understanding of the behavior of ceramics as well as the test methods needed to determine those properties. In this volume, we have tried to cover as broad a range of mechanical testing methods and methodology as possible. In addition to the practical information on test methods for creep, strength, fatigue, and slow crack growth, we have included the philosophy of why we need to test what we test, descriptions of the effects and theoretical basis of environment and microstructure on crack growth and material behavior, and the linkages of the properties of ceramics to the design of components.

The philosophical underpinnings of why we test what we test serve as the basic introductory material for the book and are set forth by Stephen Freiman. He discusses the use of test procedures as they relate to the end use of the data for scientific understanding, quality assurance, design of systems and components, or estimations of service life. He explains some of the reasoning behind the selection and use of particular test methodologies for strength, fracture toughness, and environmentally assisted crack growth. The specific test procedures used as examples focus on determination of fracture parameters important to the design and use of ceramics. Finally, he discusses test procedures in terms of the need to predict the reliable, safe use of ceramics in critical applications using the most up-to-date statistical techniques. Dr. Freiman argues that we must weigh the economic, technical, and psychological benefits to be gained in collecting particular data against the cost in time and materials needed to perform the test.

Having developed a philosophy of testing, we turn to some of the theory and processes that affect the mechanical behavior of ceramics. The most important ones are the influences of environment and those of microstructure. In Chapter 2 Grady S. White focuses his discussion on the understanding of the mechanisms of environmentally assisted crack growth and how the theoretical basis for understanding it is shifting from interpretations of experimental crack growth studies to calculations based on environmental-molecule/strained-crack-tip-bond systems.

Stephen J. Bennison (Chapter 3) discusses the crack resistance behavior of ceramics in terms of the microstructure of these materials and how manipulation of the microstructural features can significantly change the behavior. The realization that the brittleness and unreliability of ceramics may be mitigated by exploitation of microstructure-sensitive toughening mechanisms has led to global research efforts aimed at designing ceramic microstructures for specified structural performance. He reviews several shielding mechanisms that may be exploited in the design of structural ceramics. He discusses the role of microstructure and gives examples of its successful tailoring for specified mechanical performance. Dr. Bennison examines the primary methods of R-curve determination, with particular emphasis on relatively new methods of R-curve determination at crack-length scales approaching those of the microstructural elements responsible for shielding. Bennison examines mechanisms in some detail and conceptually classifies them into frontal-wake and bridged-interface, depending on whether the microstructural-toughening elements are activated in the near-tip field ahead of the primary crack or in the trailing wake of the primary crack. He presents a brief discussion concerning the newly emerging field of "quasi-brittle" fracture and damage mechanics as applied to ceramics, and closes with an examination of R-curve influence on reliability and engineering design requirements.

These initial chapters set the stage for a series of discussions of specific sets of test techniques—strength, creep, and fatigue and slow crack growth. Fracture strength, discussed by Mattison K. Ferber, Andrew A. Wereszczak, and Michael G. Jenkins in Chapter 4, is one of the most commonly cited properties for structural ceramics. They trace the importance of fracture strength as a characterization descriptor to several factors. First, fracture strength and fracture distribution are often used as critical indicators of the success of materials development programs. They provide specific examples from recent research related to processing advanced ceramics for automotive gas turbine applications. Second, the design of ceramic components for advanced structural applications should be based on probabilistic failure methodology. This methodology requires the characterization of the fracture strength distribution and its dependence on specimen volume. Finally, the measurement of the time, load-rate, or stress dependencies of fracture strength is often used in fundamental studies of slow crack growth. Chapter 4 also reviews a number of techniques and methodologies developed for the measurement of fracture strength. Most of these techniques equate fracture strength to the maximum stress (tensile or compressive) at fracture. Consequently, in order for a particular load and specimen geometry to be useful for the determination of fracture strength, the stress distribution must be well established. A complicating factor in the determination of fracture strength is that the strength of ceramic materials is quite sensitive to the size, shape, and surface finish of the test sample. This sensitivity is largely responsible for the wide variation in strength values often reported by different investigators for a given material. To be viable, the test methodology must therefore account for these effects.

In Chapter 5, the long-term deformation characteristics and test methods are discussed by Michael G. Jenkins, Robert K. Shiffer, and Sheldon M. Wiederhorn. As the authors point out, the need for this type of information is driven by requirements for greater energy efficiency, which in turn have placed increased thermal demands on materials used in advanced heat engines and other energy-consuming devices. Substantial research effort has been devoted to the search for high-temperature structural materials that can keep pace with these engineering demands. The determination of mechanical properties at elevated temperature is inherently more difficult than at room temperature because it is necessary to control stress and temperature simultaneously. Extended periods of testing are required to simulate operating conditions in turbines (>10,000 h for aircraft turbines and >25,000 h for industrial gas turbines). Interest in creep of materials dates back at least 90 years and includes work on metals, ceramics, polymers, and rocks. In this chapter, the authors review modern methods of conducting creep tests, and discuss the advantages and limitations of each.

The remaining set of properties and property measurement techniques focus on fatigue and slow crack growth, and are discussed in Chapter 6 by Kristin Breder and Andrew A. Wereszczak. The term *fatigue* in reference to ceramics traditionally covers the phenomena of cyclic fatigue, static fatigue, and dynamic fatigue. Cyclic fatigue involves cyclic loading of a component or specimen until failure occurs, and can include various minimum-to-maximum stress ratios (e.g., tension-compression, tension-tension) and numerous different wave forms. Static fatigue describes the stable growth of a crack under a static (tensile) loading at a stress level lower than that required to cause sudden fracture, and dynamic fatigue is the term used for subcritical stable crack growth under a monotonically increasing stress or load. The terms *environmentally enhanced crack growth*, *delayed failure*, and *slow crack growth* (SCG) are also frequently used, and the authors define them and discuss their implications on test techniques and measurements. Time-dependent failure are found in almost all structural ceramics and glasses, in some cases in humid air and water at room temperature (e.g., glass and Al_2O_3) and in other cases at elevated temperatures in ambient air or an oxidizing environment (e.g., Si_3N_4).

According to the authors of Chapter 6, the need for measuring the SCG properties for structural ceramics becomes evident when the reliability and lifetime need to be determined for design. Design with ceramics involves the determination of strength and reliability. They argue that a statistical design approach is necessary because of the high variability of the strength of structural ceramics which, due to their relatively low toughness, fail at a combination of critical applied stress and critical flaw size. The statistical nature of the strength of ceramics is currently well recognized, and is usually accounted for by utilizing Weibull or similar statistical distributions. Design tools using a combination of strength measurements, stress analysis, and statistics are available and reasonably well developed. These design codes also incorporate material data such as elastic constants as well as flaw distributions and time-dependent properties. The fast fracture reliability for ceramics may be different from their time-dependent reliability, and, further confounding the design complexity, the time-dependent reliability varies with the environment/temperature/stress combination. Therefore, it becomes important to be able to accurately determine the behavior of ceramics under the conditions in question in order to provide a prediction of the lifetime and reliability for a given component.

The types of fatigue and slow crack growth tests that are described by Breder and Wereszczak are grouped into one of two classifications: (1) fatigue crack propagation testing with short cracks and (2) fatigue crack propagation with long cracks. They discuss specific methods for determining fatigue crack propagation relationship including specimen requirements, fixtures, and measurements, for structural ceramics having natural or artificial flaws as short cracks or for structural ceramics having long cracks.

The preceding series of chapters on strength, creep, fatigue, and slow crack growth cover the basics of the test techniques. Osama Jadaan's treatment, in Chapter 7, shows how these basic techniques and analyses can be used to examine the behavior of a specific set of geometries in real applications involving heat exchangers, radiant tubes, power generators, and other thermal applications. Using the various test and corresponding analytical methods reviewed in earlier chapters to evaluate the reliability of ceramic tubular components, he describes methods varying from testing small ring specimens to testing close to standard size cylindrical components at room and high temperatures. In his treatment, he emphasizes testing of these specimens in fast fracture, slow crack growth, creep rupture, steady state and transient thermal loading modes.

The test methods and procedures used allow an experimenter to measure a wide range of mechanical properties over a wide range of temperature and other conditions, but can have innumerable variations in the details of specimen size, geometry, and test conditions. Crucial to the increased use of ceramic materials and standard test methods that allow us to truly compare one material's behavior to another. The processes by which standards are written and agreed upon, as well as discussion of the current state of standardization in the United States and other countries, are provided in an excellent chapter by Charles R. Brinkman and George Quinn. In the United States, a group of users or representatives from various sectors including material producers, test laboratories, academia, government, potential customers, and other interested parties select by consensus the best or standard methodology so as to produce the greatest good to the largest number. Standards accomplish many things including establishment of a common language, thereby promoting exchange of required information between buyer and seller in commerce. The authors present an overview of progress made to date in the areas directly involved with mechanical property determination of monolithic and ceramic matrix composite materials, including domestic and foreign standards.

Finally, methods of utilizing the theoretical and practical information are demonstrated in Chapter 9 by David W. Richerson, where the principles are applied to real world manufacturing applications. The preceding chapters discussed types of testing used to determine mechanical characteristics of ceramic materials and predict reliability. These tests are necessary to provide a general database for the material, but have limitations regarding component reliability assurance: (1) the tests are generally conducted on representative test samples rather than the actual hardware for the application and (2) the conditions of the application (e.g., stress distribution, heat transfer boundary conditions, and environment exposure) are generally not precisely simulated by the materials characterization testing. To minimize these limitations, Richerson points out that the mechanical characterization testing must be carefully integrated with the design of the ceramic component, with the development of the fabrication process for the component, and with the transition of the component into the application (prototype testing, redesign/material modification iterations, and scale-up to the full requirements of the application). He discusses the methodology and techniques for achieving this integration of mechanical property testing with component design, component fabrication development, and reliable component application.

The methodology described in Chapter 9 for integrating mechanical testing into material, component, and application development depends significantly on the overall methodology of the organization for R&D and commercialization. It includes such factors as how R&D, prototype component development, and manufacturing development are conducted in the company, either in distinct steps (by separate groups within the organization) or in parallel (by an integrated team as part of a strategic plan). Substantial reduction in time-to-market-entry has been demonstrated by this approach.

The challenge of the transition development methodology is to minimize the time-to-market-entry and still achieve high reliability. The reduced time places a substantial burden of responsibility on the mechanical property test team. The remainder of the chapter addresses the specific role of the mechanical properties team for each of the key activities that the team must conduct. The case studies presented are associated with the development of heat engine components such as turbocharger rotors.

A set of techniques that are not addressed in detail pertain to the measurement of fracture toughness. These measurement methodologies are still evolving and there is still debate on the precise meaning of fracture toughness in ceramics, let alone how to reliably measure it. In principle, the fracture toughness, K_{Ic}, of a material is a measure of its resistance to the extension of a sharp crack. It can be used in different ways. In design applications, K_{Ic} can be used to determine allowable stresses and crack sizes to ensure structural integrity of a component. In materials development, K_{Ic} is used as an indicator of damage tolerance, and a high fracture toughness value is desirable. These two applications are connected in that the second (i.e., the quest for a material with higher fracture toughness) is a result of the first. However, there is also a basic difference. In design applications, the experimentally determined fracture toughness value is used in quantitative relationships determining design stresses. In materials development, the fracture toughness values are used to compare and rank materials or processes. Here, the absolute value is less important. Given the murky situation in ceramics—whether long crack or short crack, differences in values obtained using different techniques, and an inability to resolve or explain these differences—we suggest that the reader review the current literature when choosing to pursue fracture toughness testing.

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The Reasons Behind Mechanical Test Procedures for Ceramics or Why We Measure What We Measure

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1.1. INTRODUCTION

Because of the potential for catastrophic brittle failure, designers of glass and ceramic components must be concerned with their fracture behavior. The focus of this chapter is a discussion of the test procedures which are used to determine the fracture parameters, e.g., fracture toughness, strength, and susceptibility to moisture-enhanced crack extension, that are important to the design and reliable use of ceramics in almost every conceivable application. In each of these topic areas we will discuss test procedures relative to the end use of the data, i.e., scientific understanding, quality control, design, etc. The advantages and disadvantages of certain test procedures will be outlined. Finally, test procedures will be discussed in terms of a need to predict the reliable, safe use of ceramics in critical applications using the most up-to-date statistical techniques.

1.2. FRACTURE MECHANICS

We choose to use fracture mechanics to describe a material's resistance to fracture because this provides a way of separating the intrinsic resistance of the material to crack extension from the size of defects which can be more or less severe depending on processing, handling, etc. The use of fracture mechanics leads to the well-known relationship between fracture stress, σ_f and K_{IC}:

$$\sigma_f = \frac{YK_{IC}}{a^{1/2}} \tag{1.1}$$

where K_{ic} is the critical stress intensity factor, i.e., fracture toughness, a is the flaw radius, and Y is a geometric factor that depends on flaw shape and location.

In general terms we can define K_{IC} as the value of stress intensity factor at the critical point. But, what do we mean by ''critical'' and how do we measure it? Stress intensity factor is defined in terms of the stress in the vicinity of a crack tip, as shown in Fig. 1.1. By definition [1]:

$$K_I = \text{limit}\ \sigma_y \cdot (2\pi r)^{1/2} \tag{1.2}$$

where σ_y is the stress perpendicular to the crack plane at some distance r from the crack tip. The crack tip stress can only be defined at distances r greater than d from the tip (Fig. 1.1); at $d < r$, deformation is nonlinear, and nonelastic, so that fracture mechanics is no longer is applicable. Fortunately, this nonlinear zone does not interfere with our determination of fracture toughness.

For metals, K_{IC} is defined through ASTM method E399 [2]: ". . . in mode I for slow rates of loading . . . fracture toughness is the value of stress intensity factor designated K_{IC} as measured using the operational procedure specified in this method E399, which centers attention on the start of crack extension. . . ."

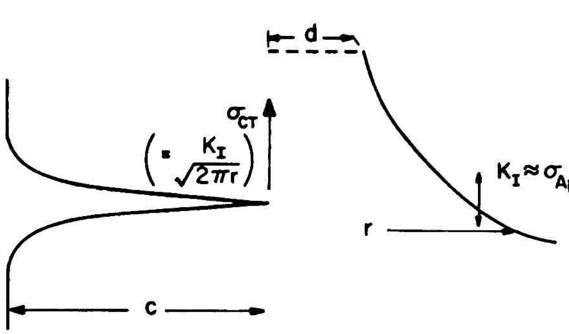

Figure 1.1 Schematic of stress system at a crack tip demonstrating the definition of stress intensity factor K_I. The stresses become undefined at distances from the tip smaller than d.

The problem with applying this definition to ceramics is twofold: (1) Unlike most metals, subcritical crack growth in ceramics due to environmental effects is extremely difficult to avoid so that the start of crack extension will depend heavily on the test conditions. (2) In large part because of these environmental effects, K_{IC} for ceramics has historically been defined as a point at which *rapid* crack extension occurs, usually as determined by a sudden load drop on a test machine. The question is, how rapid is rapid? In examining a typical plot of crack velocity as a function of stress intensity factor (Fig. 1.2), one sees that even at high velocities there is a finite positive slope to the V-K_I curve. Since there are no discontinuities in the curve, it is not clear where a critical point would be. Working backward from published values of K_{IC} would place a critical velocity at about 0.1 m/sec, but this number is dependent on test geometry, loading rate, etc. We are left with the unsettling conclusion that one of the key parameters in our determination

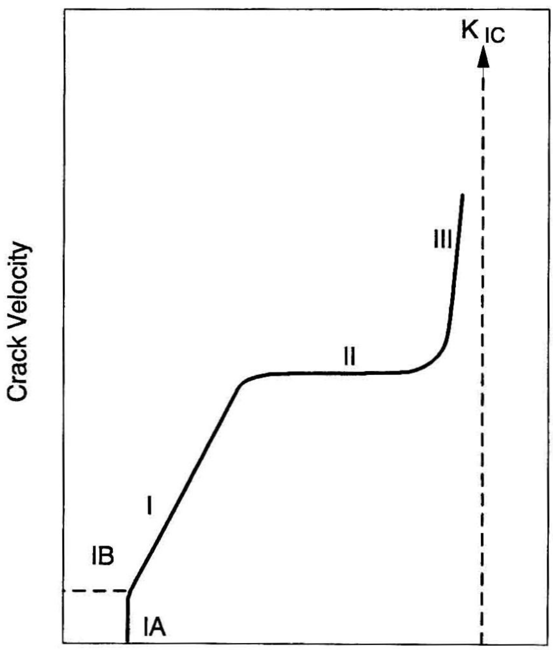

Figure 1.2 Schematic of a general crack velocity V-K_I curve showing the differing mechanistically controlled regions.

of structural reliability is very poorly defined. Unfortunately, there does not seem to be any easy solution to this dilemma.

The situation with respect to the definition of K_{IC} is made worse by the fact that there are a significant number of test procedures which purport to yield fracture toughness. At this time there is no one procedure which is accepted as a "standard." Three tests are currently being balloted in ASTM for inclusion as standard test methods: surface crack in flexure, single edge precracked beam, and chevron notch (Fig. 1.3) [3]. However, their acceptance as standards does not mean that the value of K_{Ic} obtained by each test will be the same. Other tests can be used as well; each has its advantages and disadvantages [4]. The choice of tests will be based on a number of factors, e.g., the form and quantity of material, and the end use of the data. Elaborating on this last point, fracture toughness data can be used to rank materials, assure reproducibility, predict other properties (e.g., erosion), or develop a basic understanding of the fracture process. **Because toughness is not a parameter used directly in design, an absolute value of K_{IC} may not be a necessity**. What is required is a reproducible test procedure that can be easily carried out, at times by the nonexpert.

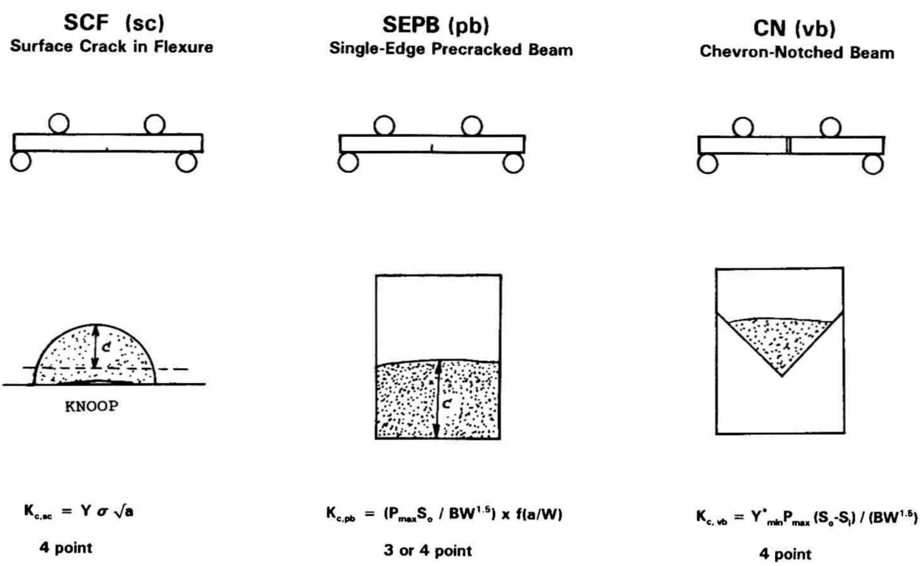

Figure 1.3 Potential fracture toughness standards now under consideration by ASTM Committee C-28.