Life Prediction Methodologies and Data for CERAMIC MATERIALS

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Foreword

This publication, *Life Prediction Methodologies and Data for Ceramic Materials,* contains papers presented at the symposium of the same name, held in Cocoa Beach, FL on 11–13 Jan. 1993. The symposium was sponsored by ASTM Commitee C-28 on Advanced Ceramics and the American Ceramics Society. C. R. Brinkman of Oak Ridge National Laboratories in Oak Ridge, TN and S. F. Duffy of Cleveland State University in Cleveland, OH presided as symposium chairmen and are editors of the resulting publication.

Overview

ASTM Committee C-28 on Advanced Ceramics was organized in 1986 when it became apparent that ceramics were under consideration for many new high technology applications. Proposed applications in the aerospace, biomedical, military, power generation, processing, and automotive industries were viewed as being particularly demanding in terms of property requirements and subsequently required an abundance of experimental data to guide emerging design and fabrication technologies. Hence, it became apparent that industry-oriented standards were needed for production, inspection, testing, data analysis, and probabilistic design of components in order to use the attractive features of these emerging materials, as well as minimize any shortcomings. Accordingly, Committee C-28 was organized into various subcommittees (including C-28.02 on Design and Evaluation) whose goals and objectives are reflected in the needs just mentioned. Specific responsibilities include: development of appropriate standards that address the topics of nondestructive evaluation (NDE), statistical analysis, and design of components fabricated from advanced ceramics. Early in 1990, members of this committee determined that it was appropriate to organize an international symposium aimed at presenting a state-of-the-art review. The review would focus on requisite design data and methods of generating this data, failure modeling, statistical techniques for the analysis and interpretation of this data, and probabilistic design methodologies that are a necessity in the analysis of components used in high technology applications, such as advanced heat engines. The anticipation was that the information presented at this symposium would serve as a basis in developing future standards. Time will tell whether this anticipation is fulfilled.

Twenty-seven papers were presented at the symposium, and 24 were subsequently published in this volume. The papers contained herein were grouped into three general subject areas. This selection was somewhat arbitrary, and several papers could easily be placed in more than one category. The categories include data and model development, life prediction methodologies, and prediction of the behavior of structural components. We expect that these subjects will not only be of interest to authors of future ASTM standards, but also to those interested in data generation requirements and model development. We also expect that the information contained in this publication will be pertinent for brittle monolithic ceramics as well as ceramic matrix composite materials. We note that ceramic-based material systems will be used in many advanced technologies where performance at elevated temperatures and in environments where strength degradation due to slow crack growth is of concern, or both. Thus, the articles that address this issue may be of particular interest to individuals who are involved in the development of lifing methods for advanced ceramics.

Data and Model Development

The papers in this section broadly describe generation of mechanical properties data. These data are used to identify optimized fabrication processes that minimize defects in advanced ceramics. Advanced NDE techniques such as acoustic microscopy and microfocus X-ray for pretest examination of specimens are discussed and results presented. The articles presented in this section also describe specific types of test data including: experimental procedure and equipment for determining tensile fast fracture, tensile creep, tensile cyclic fatigue, and flexure behavior used in life prediction methodology development. The influence of loading wave form, time, and temperature on the behavior of silicon nitride is described. Examples of representation and interpretation of data are given in various ways. These include: the use of fracture maps that can be used to generate stress allowables for a given application, competing Weibull analyses that delineate the probability of failure by surface or volume flaws, and constitutive equations for predicting creep and creep-rupture behavior under uniaxial and multiaxial loading conditions. Note that the presented creep loading regimes were both constant with time and also varied in a step-wise fashion. The validity and problems associated with use of flexural data for determining creep parameters are discussed. Finally, results and analyses of monotonic tensile fast fracture are presented and compared with several kinds of flexural test data. Results presented in papers found in this section should be of particular interest to the experimentalist whose focus is characterizing ceramic materials using tensile and flexural test techniques. As an example, the paper by Foley et al. contains results of over 100 tensile tests conducted at room temperature on a single material. Tensile specimens and the equipment required to successfully conduct this type of test are often prohibitively expensive. Testing in sufficient numbers to fully characterize a material with multiple flaw (or strength) distributions further increases the cost. Hence, reducing the expense in obtaining an optimum, high-quality data base was identified as being a major challenge to future experimentalists.

Life Prediction Methodologies

Metal alloys, such as those currently used for pressure vessels and gas turbines, have mechanical and physical properties that are readily available and easily implemented in an analysis of a component's response to applied boundary conditions. With metal alloys, the engineer often associates a high degree of confidence in the resulting component analysis. Factors of safety are applied to define exact stress allowables as a part of a deterministic design methodology. Furthermore, materials with high ductilities are selected for an extra margin of safety. In contrast, components fabricated from ceramic materials require new design methodologies for predicting component life that account for uncertainty in safe life expectancy. These materials are brittle by nature with an inherent scatter in strength. Life expectancies are not only controlled by the distribution and evolution of defects present after a component has been fabricated, but also by defects that may nucleate under load. Use of these materials fundamentally requires probabilistic design techniques that account for this behavior. Several articles in this section review life prediction methodologies for monolithic materials and ceramic matrix composites (CMC) and applications are provided in most instances. Various failure models are employed that are embedded in the framework of weakest-link theory and Weibull statistics. These models are exercised with a number of simple component/specimen geometries such as uniaxial three- and four-point unnotched bars. In addition, component/test specimens with more complex biaxial states of stress, such as notched beams and bars, ball-on-ring, ring-on-ring, and uniform pressure-on-disk specimens are highlighted.

In a paper by Scholten et al. a question is posed as to whether or not it is necessary to track the defect from which a brittle fracture originates when predicting multiaxial strength of ceramics. In order to examine this, a data set was developed from uniaxial and biaxial test specimens fabricated from several materials. Mixed-mode fracture criteria were compared with experimental results. Deviations from weakest-link theory were found in some instances when different fracture criteria were applied. These deviations were greatest in the more dense materials where the defect density was small. Microcracks were found nucleating during the test that the authors maintain violates the weakest-link principles. It was concluded that strength could be predicted with the introduction of a "size-independent" strength parameter.

It was further concluded that if consideration is given to experimental errors, then differences in multiaxial strength predictions for several specific loading conditions can readily be attributed to lack of precision. This underscores the importance of experimental accuracy in conducting multiaxial tests.

A methodology that predicts creep life using continuum damage mechanics is outlined by Chuang and Duffy for continuous fiber reinforced ceramic matrix composites (CFCMC). A number of potential creep-damage mechanisms in advanced ceramics are examined and the corresponding constitutive laws are outlined. The authors demonstrated that this methodology has potential for establishing estimates of creep life when stresses, temperatures, volume fractions of the constituents, and material properties are known. The work also points to the need for additional theories that allow extrapolation of short-term laboratory data to long-term service conditions.

Fracture data representing uniaxial and biaxial bend specimens fabricated from sintered alumina were compared by Chao and Shetty. Both environment (inert dry N_2 and deionized water) and strain rate were varied to determine if strength degradation due to slow crack growth in biaxial flexure can be predicted from simple uniaxial tests conducted in an inert environment. The authors concluded that this predictive approach was feasible, so long as the statistical uncertainties in both Weibull parameters (modulus and characteristic strength) and slow crack growth parameters (crack growth exponent and crack growth velocity) are properly taken into account.

Johnson and Tucker pointed to the variations observed in estimates of the Weibull parameters when two different estimation procedures are used. A data base composed of specimens with different applied boundary conditions and multiple specimen sizes was employed. A pooled sample of 137 test specimens fabricated from sintered silicon carbide were tested in six different combinations of specimen size and bending configuration. Comparisons are made with results using maximum likelihood and linear regression estimators after the estimators were applied to the uniaxial specimens in the pooled sample. A general consensus has emerged, which the authors support by their analysis, that maximum likelihood estimators are preferred since this approach offers the ability to unbias parameter estimates and establish confidence bounds on the estimated Weibull values. Furthermore, the authors emphasized the importance that high-quality fractography has on parameter estimates in the presence of multiple flaw populations.

Tucker and Johnson demonstrated that two multiaxial stochastic models recently reported in the literature (that is, the Batdorf-Heinisch and the Lamon-Evans models) yield equivalent probability of failure predictions. This allowed the authors to define a generalized size factor that accounts for geometry, loading conditions, and multiaxials stress states. The factor facilitates parameter estimation when the data base contains multiple specimen geometries and applied boundary conditions.

A method is presented by Margetson for analyzing component strength in the presence of both surface and volume flaws for a number of probabilistic models based on the principles of fracture mechanics. The methodology presented is applicable to multiaxial test configurations.

Prediction of the Behavior of Structural Components

Progress as well as difficulties encountered in predicting behavior of specific ceramic components and subcomponents are outlined in the articles found in this section. Component geometries include: notched bars, C- and O-ring specimens, ring-on-ring square plates and internally pressurized tubes. Initially, modeling a component typically requires the design engineer to resort to finite element methods to obtain accurate stress distributions and identify regions with high-stress gradients. Once gradients have been minimized and the stress state has been ascertained, component life is determined using a number of models that address different failure modes. Design concepts using principles from continuum damage mechanics, fracture mechanics, and Weibull statistics are often incorporated in life prediction codes such as the NASA computer program CARES/LIFE. Many authors emphasized the need for an adequate materials data base to properly implement this type of design approach. The data base must be constructed using carefully selected test-specimen geometries that establish requisite design parameters. Once these data are established, the results from structural component tests can be used to challenge the predictive capabilities of the models incorporated in various design codes. In order for this information to be of use to the design engineer, component level tests must represent the various service conditions encountered in real-life applications. Moreover, component tests must promote and isolate failure modes such as: fast fracture, slow crack growth, and creep.

Jadaan examined internally pressurized SiC tubes that were tested at temperatures and pressures in order to promote failure by slow crack growth and creep rupture, or both. Methodologies are presented that allow prediction of failure by either of these two mechanisms. Supporting data from standard creep-rupture, O-ring, and compressed C-ring tests are used to develop the methodology. Complications in use of these types of specimens for characterization of highly porous tubes are subsequently discussed in a paper by Krankendonk and Sinnema.

Estimating the stochastic parameters that characterize the inherent strength of a ceramic material is fundamentally important to any type of probabilistic design approach. Cuccio et al. provided an extensive treatment on estimating Weibull parameters and component reliability, providing methods to establish confidence intervals on both. The authors provide methodology for the following:

- censored analysis of competing strength distributions,
- analysis of data from specimens with multiple sizes,
- analysis of data from specimens with multiple-loading conditions,
- analysis of data from multiple temperature tests,
- calculation of confidence intervals on parameter estimates, and
- calculation of confidence intervals on reliability estimates.

The methodology is exercised using a data base from uniaxial fast-fracture tests conducted on test specimens fabricated from silicon carbide and silicon nitride.

Fabricators of ceramic components can dramatically improve their product reliability by removing prior to service components with gross or unusual defects. This is accomplished through NDE programs or proof-testing components. Highly sophisticated methods have been recently developed to perform NDE inspection. However, Brückner-Foit et al. point out that the NDE techniques suffer from several drawbacks, including cost and resolution. The authors discuss the advantages, disadvantages, and outline an approach for multiaxial proof testing. In addition, two examples are presented that illustrate the approach and typical problems associated with proof testing.

Finally, an overview of the integrated design code CARES/LIFE is presented in a paper by Nemeth et al. This public domain computer algorithm allows the design engineer to predict the time-dependent reliability of a component if the dominant failure mode is slow crack growth. The authors outline the supporting theoretical development, and two examples provide the reader with insight regarding the capabilities of the code.

Based on comments and feedback following the symposium, the chairmen felt the symposium was most successful in meeting the goals and objectives originally set forth during initial organization. Much of the information given in these papers is currently being used in formulating new ASTM standards and by the designers who are implementing advanced ceramics in many demanding applications. Furthermore, the chairs wish to express their gratitude to the authors for their efforts in preparing their manuscripts, putting on well-orchestrated and professional presentations at the symposium, and responding to reviewers comments in a thoughtful manner. In addition, the chairs are deeply indebted to the reviewers for their timely efforts and scholarly assessment of the manuscripts.

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Contents

Data and Model Development

Michael R. Foley[1], Vimal K. Pujari[1], Lenny C. Sales[1], and Dennis M. Tracey[1]

SILICON NITRIDE TENSILE STRENGTH DATABASE FROM CERAMIC TECHNOLOGY PROGRAM PROCESSING FOR RELIABILITY PROJECT

REFERENCE: Foley, M. R., Pujari, V. K., Sales, L. C., and Tracey, D. M., **"Silicon Nitride Tensile Strength Database from Ceramic Technology Program Processing for Reliability Project,"** Life Prediction Methodologies and Data for Ceramic Materials, ASTM STP 1201, C. R. Brinkman and S. F. Duffy, Eds., American Society for Testing and Materials, Philadelphia, 1994.

ABSTRACT: Tensile strength data generated in Norton's Ceramic Technology Program (CTP) Processing for Reliability Project is presented for a hot isostatically pressed (HIP'ed) 4 wt% yttria-silicon nitride (designation NCX-5102). This database represents the result of an extensive multi-variable experimental matrix designed to identify an optimized process directed at eliminating or minimizing critical flaws. The strength data follow from room temperature fast fracture tests of net-shaped-formed, pressure cast cylindrical buttonhead tensile bars. Results of over one hundred tensile tests coupled with detailed fractography are summarized using Weibull statistics including competing risk analyses. Specimen fabrication and mechanical testing issues (e.g. machining, strain gaging) which were addressed to ensure the integrity of the strength database will also be discussed.

KEYWORDS: silicon nitride, reliability, tensile strength, Weibull analysis, competing risk, machining, strain gaging

A variety of strength-degrading flaws introduced during the initial stages of traditional processing methods can produce unacceptable mechanical reliability of structural ceramics. Impurities in the starting material components (powder, sintering aids, surfactants, binders) and agglomerates formed during powder processing are but two examples of strength-degrading flaws. Forming related cracks, voids and metallic impurities introduced during forming and grain growth during densification are further examples of failure originating flaws. Even if all of these intrinsic flaw types can be minimized or eliminated, the final step of fabrication, machining, can leave various extrinsic flaws on the surface of the component.

Silicon nitride-based ceramics are continually being evaluated for

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room and elevated temperature structural applications [1-8]. Flaws such as the ones described above not only limit the ultimate strength of these materials but increase the scatter in the strength data giving rise to low reliabilities (low Weibull modulus). Mechanical testing coupled with detailed fractography can identify specific flaw types and provide valuable feedback [9] to the powder processing, forming, densification as well as the machining unit operations. The data discussed in this paper are from the second phase of a three stage Department Of Energy (DOE)/Oak Ridge National Laboratory (ORNL) CTP program on processing for reliability [10]. This report addresses the issues of machining, bending during tensile testing and reliability statistics. Room-temperature tensile strength data are used in conjunction with detailed fractography data to perform competing risk Weibull analyses.

EXPERIMENTAL PROCEDURE

Materials

A 4 wt% yttria-doped Si_3N_4 (NCX-5102) was selected to be tested at room temperature. The net shaped formed (NSF) buttonhead tensile rod is the model component being used to evaluate all process improvements. The material is processed and pressure cast in a closed loop, aqueous powder processing operation in a clean room environment. After casting, the specimens were dried, presintered and HIP'ed to >99.5% theoretical density (ρ_{THEO} = 3.23 g/cm^3). After densification, the tensile specimens underwent a pre-machining inspection including density and runout, see Fig. 1. The individual HIP runs were qualified using flexure strength in accordance with ASTM Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature (C 1161 type B) and K_{Ic} [11] as controls. The entire flow diagram for the tensile specimen history from densification to post-mortem analysis is also shown on Fig. 1.

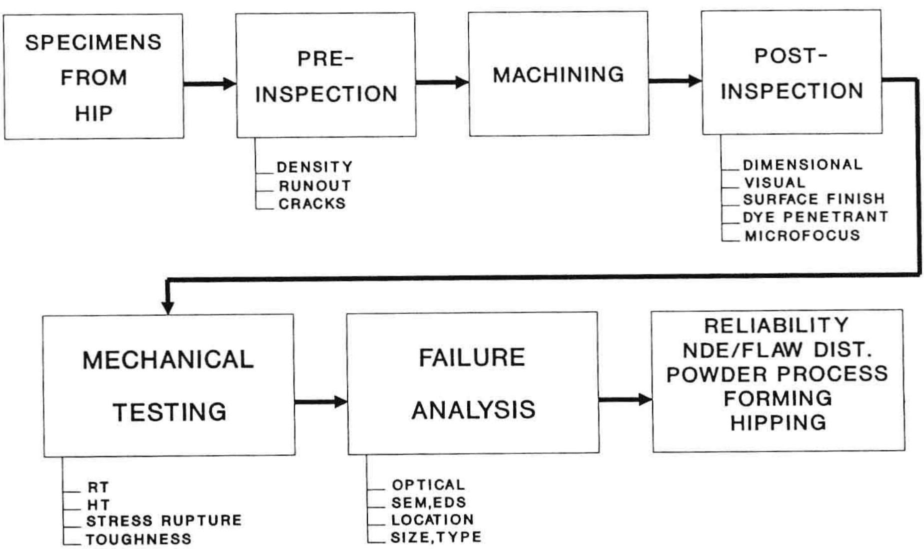

FIG. 1--Specimen flow diagram for testing and failure analysis.

Experimental Design

Several variables were examined by way of an L8 x L4 experiment. The experimental plan [10] involved 16 processed batches of silicon nitride and each of the experimental blocks was evaluated through room temperature tensile tests on approximately thirty tensile rods amounting to over 500 rods tested. The unit operation control variables evaluated in the experimental plan were:

Slurry conditions

* Binder. (2)
* Surfactant. (2)
* Casting rate. (2)
* Solids loading. (2)
* Pre-HIP treatment. (2)

HIP conditions

* HIP cycle. (2)
* Fixturing. (2)

The results of the experimental plan defined a set of optimum slurry and HIP conditions. The following additional variables were subsequently evaluated separately:

Post HIP conditions

* Machining conditions.(4)
* Post machining conditions.(2)

The four machining conditions are described in detail below and are part of a separate experiment. The two post-machining conditions involved either performing a thermal treatment aimed at annealing surface and subsurface damage created by machining or leaving the surface as-machined. Previous work [12] showed a 10% increase in room-temperature mean tensile strength by application of the surface oxidation heat treatment.

Finally, the fully optimized process was repeated for a subset of tensile specimens. The process included a 2-step HIP process, a specified procedure for machining, and thermal surface treatment. The statistical analysis, described below, was performed on this subset of tensile specimen data.

Machining

The final machined[2] cylindrical buttonhead tensile specimen is the ORNL design [8] except that the gage diameter is 6.0 ± 0.1mm and 35 mm gage length. All specimens for the main body of strength data were machined using a specifically designed standard operating procedure (SOP) (procedure #1) as shown in Table 1. It should be noted that the gage section is longitudinally ground during both the roughing and final steps.

After machining, all specimens underwent an extensive inspection including: dimensional tolerance, surface finish, microfocus X-ray and liquid dye penetrant. Customary practice expresses surface finish specification in terms of the average roughness R_a. Earlier work [9] resulted in a reduction of the R_a specification from 0.4 μm (16 μin) to 0.25 μm (10 μin) at the buttonhead radius and to 0.2 μm (8 μin) along the gage length.

Four distinct machining procedures (including the original SOP) were evaluated for their influence on tensile strength as an additional experiment. The four machining procedures differed according to

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[2]Chand Associates, Inc., 2 Coppage Dr., Worcester, MA 01603-1252

intermediate grinding steps, diamond wheel grit size and depths of cut as outlined in Table 1. This experiment maintained the above R_a specification while altering the rough and intermediate grinding steps with the intent being to reduce subsurface damage due to the prior step. On the basis of the grinding parameters employed, the machining procedures were ranked according to operational precision from #1 to #4, with #4 being the most precise procedure. Consistent with the focus on machining damage effects, the specimens were not given post-machining heat treatments for the purpose of this study. A total of 72 specimens from 3 HIP runs were machined and tested for this study. The procedures were evaluated by tensile strength and statistical analyses.

TABLE 1--Procedure steps for machining tensile rods.

Procedure	Roughing Step	Intermediate Step(s)	Finishing Step
#1 Original SOP	180 grit	-	320 grit (0.51mm)*
#2 Experimental	320 grit	320 grit	320 grit
#3 Experimental	180 grit	320 grit	800 grit (0.15mm)
#4 Experimental	180 grit	320, 400, 600 grit - (0.05mm) (0.05mm)	800 grit (0.05mm)

*The number in parenthesis refers to amount of stock removed by that step.

Testing

The cylindrical buttonhead tensile specimens were tested at room-temperature on a commercial electro-mechanical test machine[3] utilizing commercial ,self-aligning self-contained hydraulic load train couplers[4] and straight tri-split copper collets. A double ramp loading procedure was used to test all specimens. The specimen is initially loaded to 6668 N at 39 MPa/min. This allowed time for the fully annealed copper collets to deform to match the radius of the buttonhead. After the initial ramp to 6668 N, the specimen was then loaded to failure at a stressing rate of 600 MPa/min.

The load train was checked before testing with an alignment tool for actuator/load cell alignment and a strain gaged tensile specimen for coupler alignment.

Strain Gaging of Tensile Specimens

A total of 68 specimens were strain gaged prior to testing, to study bending during testing and to determine the effect of bending on tensile strength and reliability data. Each specimen had four gages equispaced and circumferentially attached at the longitudinal center of the gage section. The percent bending (PB) was calculated in accordance with ASTM Test Method for Sharp-Notch Testing with Cylindrical Specimens (E 602) such that

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[3]Instron Model 8562, Canton, MA

[4]Instron "Super-grip", Canton, MA