STANDARDIZATION EFFORTS FOR MECHANICAL TESTING AND DESIGN OF ADVANCED CERAMIC MATERIALS AND COMPONENTS

Jonathan A. Salem Life Prediction Branch NASA Glenn Research Center Cleveland, Ohio (216) 433-3313 jsalem@grc.nasa.gov Michael G. Jenkins Department of Mechanical Engineering University of Washington Seattle, Washington (206) 685-7061 jenkinsm@u.washington.edu

ABSTRACT

Advanced aerospace systems occasionally require the use of very brittle materials such as sapphire and ultra-high temperature ceramics. Although great progress has been made in the development of methods and standards for machining, testing and design of component from these materials, additional development and dissemination of standard practices is needed. ASTM Committee C28 on Advanced Ceramics and ISO TC 206 have taken a lead role in the standardization of testing for ceramics, and recent efforts and needs in standards development by Committee C28 on Advanced Ceramics will be summarized. In some cases, the engineers, etc. involved are unaware of the latest developments, and traditional approaches applicable to other material systems are applied. Two examples of flight hardware failures that might have been prevented via education and standardization will be presented.

INTRODUCTION

Environmental and energy concerns (often bolstered by governmental regulations) have placed increasingly greater demands on materials used in advanced engineering designs such as aerospace systems. For example, as greater efficiencies are sought and achieved in the design of gas turbine engines, so too have the temperature, strength and weight requirements of their components changed to "push the limit" of the mechanical properties of the various materials (generally metallic alloys). Large amounts of time and effort have been devoted to the search for structural materials that will keep pace with these engineering demands. Often these searches identify underutilized materials that can be classified as "brittle." In some cases, such as sapphire for windows, processing successes (both primary and secondary) have led to the successful use of an advanced ceramic in demanding applications [1]. In other cases, in spite of tremendous strides in understanding and processing materials, only recently have structural ceramic materials, such as silicon nitrides or silicon carbide fiber-reinforced silicon carbide matrix composites, reached the developmental stage required to receive focused attention as plausible successors to the more traditional metallic alloys [2]. The applications contemplated require optimum material behavior with physical and mechanical property reproducibility, component reliability, and well-defined methods of data treatment and materials analysis. As advanced ceramics are contemplated for introduction into advanced heat engine, these issues are best dealt with via standard methods [2].

A variety of organizations, such as ASTM, ISO, and the NASA TSP, are involved with the development of standards, and the standards developed by one organization often feed into the development of standards in another organization. The American Society for Testing and Materials (ASTM) [a.k.a., ASTM International] is the primary standards writing establishment in the United States. As a private, nonprofit corporation, ASTM relies upon the voluntary cooperation of industry, government, and academe to develop standards by full consensus. ASTM Committee C28 "Advanced Ceramics" was formed in 1986 when it became apparent that ceramics were being considered for "high-tech" applications. More background on ASTM and its committees can be found at the organization's website.¹

Advanced ceramics are defined [3] as "highly-engineered, high-performance, predominantly non-metallic, inorganic ceramic material having specific functional attributes." A standard is defined by ASTM [4] as "a rule for an orderly approach to a specific activity, formulated and applied for the benefit and cooperation of all." The implication of the term "standards" is manifold. "Standards" may mean fundamental test methodologies and units of

¹ www.astm.org

measure to the researcher and the technical community. However, to the manufacturer or end-product user, "standards" are materials specifications and tests to meet requirements. Amongst designers, manufacturers and product users, commercial "standards" equate to the rules and terms of information transfer [5]. Because the term "standard" can be connoted differently depending on the user, it is the role of standards development organizations to assist in bringing together seemingly divergent interests of industry, government and academe by developing voluntary consensus "standards".

However, it is important to note that ASTM's organizational role is as a facilitator to the "real" standards writers, the task group members. The flow of the standardization process is from the task groups to the ASTM Committee on Standards (COS). An ASTM standard may take the form of a guide (a series of options or instructions that do not recommend a specific course of action), a <u>practice</u> (a definitive procedure for performing one or more specific operations or functions that does not produce a test result), a <u>terminology</u> standard (a document comprising definitions of terms; descriptions of terms; explanations of symbols, abbreviations, or acronyms), a <u>test method</u> (a definitive procedure for the identification, measurement, and evaluation of one or more qualities, characteristics, or properties of a material, product, system, or service that produces a test result). In addition, a standard may also be in the form of a <u>classification</u> (a systematic arrangement or division of materials, products, systems, or services into groups based on similar characteristics such as origin, composition, properties, or use) or a <u>specification</u> (a precise statement that indicates the procedures for determining whether each of the requirements of a material, product, system, or service is satisfied).

ASTM Committee C28 is organized into various non-administrative subcommittees including C28.01 Properties and Performance, C28.02 Design and Evaluation, C28.05 Characterization and Processing, C28.07 Ceramic Matrix Composites, C28.91 Nomenclature and Education, and C28.94 ISO TAG, with task groups addressing specific technical topics under each subcommittee. Leadership and membership of the committee and various subcommittees are distributed over approximately 100 representatives from industry, government, and academe. Currently (August 2002) there are 40 standards for advanced ceramics under the jurisdiction of Committee C28 as shown in Table 1, and 3 new standards in the balloting process as also shown in Table 1.

Outside the United States, the International Organization for Standardization (ISO) is recognized as the international forum for normalization that crosscuts regions and nations. ISO standards are often not intended to be new standards but instead are intended to harmonize existing standards to provide consensus documents that promote compliance by agreement and "buy-in." Because of this, ISO standards developed by three guiding principles (consensus, industry-wide, and voluntary) are widely recognized, giving clear benefits to industry and consumers. Some examples of successful ISO standards include ISO film speed codes, ISO standardized telephone and banking card formats, standardized freight containers, standardized symbols for the SI systems of units, and standardized paper sizes. More background on ISO and its committees can be found at the organization's website.²

ISO defines standards as documented agreements containing technical specifications or other precise criteria to be used consistently as rules, guidelines, or definitions of characteristics to ensure that materials, products, processes and services are fit for their purposes. Thus, the term "ISO standard" includes all types of standards from test method to specification. Over 140 member nations participate in ISO activities. A single standards writing organization from each member nation (e.g., ANSI for the USA) provides the technical and administrative expertise for ISO efforts. In the USA, because ANSI is an association of many different standards writing organizations (SDOs) within the USA, actual technical work for an ISO committee is often carried out by a technical advisory group (TAG) within one of these SDOs (e.g., ASTM). Just as in ASTM, the actual development of standards within ISO is decentralized, and carried out by the technical experts, the Central Secretariat in Geneva acts to ensure the flow of documents, clarifies administrative details, coordinates balloting on draft international standards, and convenes meetings of committees. The scope of ISO not limited to any particular topic (except electrical and electronic engineering standards). Over 224 different technical committees address topics ranging from information technology to threaded fasteners, to paper to glass containers to nuclear energy to earth moving equipment to environmental management to civil defense.

² www.iso.ch

ISO Technical Committee (TC) 206 was established in 1993 to address issues of harmonizing and advancing standards in the area of fine (advanced, technical) ceramics. Japan is the committee secretariat. Currently, TC206 is comprised of 14 participating (P) members and 19 observer (O) members, each representing a different nation that provides technical experts for its 28 working groups. While the primary focus of the committee to date has been structural applications of ceramics (e.g., mechanical properties), non-structural applications such as coatings, insulators, etc are well within its scope. The standards process within TC206 is initiated by a P member who submits a new work item proposal. This proposal can be accepted after first meeting several criteria: at least two national/regional standards must currently exist in the topical area, a market need exists for the proposed standard (minimum rating score of 15 out of a possible 25 points), a working group. Once a new work item is accepted, a working group is established and the technical experts refine the working draft (WD) to a committee draft (CD). When the document has reached sufficient maturity it is submitted for a TC vote to elevate it to a draft international standard (DIS). If no technical objections are raised, the document finally advances to international standard (IS). Table 2 is a list of IS and DIS documents generated by TC206 to date. Numerous other WD and CD documents developed by the 28 working groups are not shown.

At NASA, the NASA Technical Standards Program (TSP) is charged with not only developing an integrated NASA Preferred Technical Standards System that improves the availability of technical standards for design, development and operation of NASA's Programs and Projects, but also to increase use and development of voluntary, non-government standards by enhancing the awareness of standardization in NASA. Just as with ASTM and ISO, the NASA TSP recognizes certain types of standards products. These include technical standards (uniform engineering and technical requirements for processes, procedures, practices and methods); specifications (in support of acquisition by clearly and accurately describing technical requirements); handbooks (authoritative engineering technical, or design information and data relating to processes, procedures, recommended practices and methods); guidelines (technical information in support of standards, specifications and handbooks); regulations (standards accepted and enforced by the Government); and codes (a group of standards dealing with one subject). More background on NASA TSP can be found at the organization's website.³

A very visible aspect of NASA TSP is its publicly accessible website and its link to "Lessons learned" database [6]. This comprehensive database has the potential of being a boon to technical personnel because it links relevant technical standards to lessons learned in both developing and using standards. Such lessons in the past have often been anecdotal and as such were not documented. The NASA TSP provides an archival resource to not only document past experience but also a mechanism for extrapolated future efforts as part of long range planning for future standards development based on past needs.

This introduction has provided a brief background on the status of some of the higher profile standardization efforts for advanced ceramics (ASTM and ISO) as well as a forum in which standards and lesson learned have been combined (NASA TSP) to provide an archival data base for current usage and future development of standards. In the next section, some examples of advanced ceramics applications are discussed as a background for potential standardization. These examples are followed by discussion and conclusions sections in which some future directions for standards on advanced ceramics are posed.

EXAMPLES OF NEEDS FOR STANDARDS

Example 1 - Failure of a Leading Edge

<u>Background</u>: In order to improve the maneuverability and aerodynamics of future generations of re-entry vehicles and airframes, sharper leading edges are required. Sharper leading edges result in higher edge temperatures and the need for more oxidation and temperature resistant leading edge materials. One class of materials for such applications is commonly referred to as UHTC's (ultra-high temperature ceramics) that are refractory metal diborides containing additives such as SiC and possibly carbon. Recent testing of three candidate UHTC materials resulted in catastrophic failure of a number of the segments, as shown in Figure 1.

³ http://standards.nasa.gov

Responsible	Designation	Title
Subcommittee	(Year adopted)	
C28.01	C1161-01 (1990)	Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperatures
Properties and	C1198-01 (1991)	Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance
Performance	C1211-02 (1992)	Test Method for Flexural Strength of Advanced Ceramics at Elevated Temperatures
	C1259-01 (1994)	Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration
	C1273-95 (1994)	Test Method for Tensile Strength of Monolithic Advanced Ceramics at Ambient Temperatures
	C1291-95 (1995)	Test Method for Elevated Temperature Tensile Creep Strain, Creep Strain Rate, and Creep Time to Failure for Advanced Monolithic Ceramics
	C1323-96 (1996)	Test Method for Ultimate Strength of Advanced Ceramics with Diametrally Compressed C-Ring Specimens at Ambient Temperatures
	C1326-99 (1996)	Test Method for Knoop Indentation Hardness of Advanced Ceramics
	C1327-97 (1996)	Test Method for Vickers Indentation Hardness of Advanced Ceramics
	C1361-01 (1996)	Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Advanced Ceramics at Ambient Temperatures
C28.02	C1366-97 (1997)	Test Method for Tensile Strength of Monolithic Advanced Ceramics at Elevated Temperatures
Design and Evaluation	C1368-01 (1997)	Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Ambient Temperature
	C1421-01 (1999(Test Methods for the Determination of Fracture Toughness of Advanced Ceramics
	C1424-99 (1999)	Test Method for Compressive Strength of Monolithic Advanced Ceramics at Ambient Temperatures
	C1465-00 (2000)	Test Method for Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress-Rate Flexural Testing at Elevated Temperature
	C1470-00 (2000)	Guide for Testing the Thermal Properties of Advanced Ceramics
	C1499-01 (2002)	Test Method for Monotonic Equibiaxial Flexural Strength Testing Of Advanced Ceramics At Ambient Temperature
	In ballot (2002)	Test Method for Ultimate Strength of Advanced Ceramics with Diametrally Compressed O-Ring Specimens at Ambient Temperatures
	In ballot (2002)	Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Flexural Testing (Stress Rupture) at Ambient Temperature
	C1175-99 (1991)	Guide to Test Methods for Nondestructive Testing of Advanced Ceramics
	C1212-98 (1992)	Practice of Fabricating Ceramic Reference Specimens Containing Seeded Voids
	C1239-95 (1993)	Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
	C1331-96 (1996)	Practice for Measuring Ultrasonic Velocity in Advanced Ceramics with the Broadband Pulse-Echo Cross-Correlation Method
	C1332-96 (1996)	Test Method for Measurement of Ultrasonic Attenuation Coefficients of Advanced Ceramics by the Pulse-Echo Contact Technique
	C1336-96 (1996)	Practice for Fabricating Non-Oxide Ceramic Reference Specimens Containing Seeded Inclusions
C28.05	C1251-95 (1993)	Guide for Determination of Specific Area (of Advanced Ceramics) by Gas Adsorption
Characterization	C1274-95 (1994)	Test Method for Advanced Ceramic Specific Area by Physical Adsorption
and Processing	C1282-96 (1995)	Test Method for Determining Particle Size Distribution of Advanced Ceramics by Centrifugal Photo Sedimentation
	C1322-96 (1996)	Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
	C1494-01 (2001)	Test Method for Determination of Mass Fraction of Carbon, Nitrogen, and Oxygen in Silicon Nitride Powder
	C1495-01 (2001)	Test Method for Effect of Surface Grinding on Flexure Strength of Advanced Ceramics
	In-ballot (2001)	Test Method for Particle Size Distribution of Silicon Nitride or Silicon Carbide by X-ray Monitoring of Gravity Sedimentation

Table 1 Summary of Completed and In-ballot Standards of ASTM Committee C28 "Advanced Ceramics"

C28.07	C1275-00 (1994)	Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross
Ceramic Matrix		Sections at Ambient Temperatures
Composites	C1292-95 (1995)	Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures
	1337-96 (1996)	Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Advanced Ceramics under Tensile Loading at Elevated
		Temperatures
	C1341-00 (1996)	Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics
	C1358-96 (1996)	Test Method for Monotonic Compressive Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular
		Cross Sections at Ambient Temperatures
	C1359-96 (1996)	Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross
		Sections at Elevated Temperatures
	C1360-01 (1996)	Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at
		Ambient Temperatures
	C1425-99 (1999)	Test Method for Test Method for Interlaminar Shear Strength of 1-D and 2-D CFCCs at Elevated Temperatures
	C1458-00 (2000)	Test Method for Transthickness Tensile Strength of Continuous Fiber- Reinforced Advanced Ceramics with at Ambient Temperatures
	In-ballot (2002)	Test Method for Tensile Strength and Young's Modulus for High-Modulus Single Filament Advanced Ceramics
C28.91	C1145-94 (1989)	Definition of Terms Relating to Advanced Ceramics
Nomenclature	C1286-95 (1995)	System for Classification of Advanced Ceramics

Table 1 Summary of Completed and In-ballot Standards of ASTM Committee C28 "Advanced Ceramics" (cont'd)

* As of August 2002 Note: CXXXX is the permanent designation, -XX is the year of the most recent modification

Table 2 Summary of Completed and Draft Standards of ISO Technical Committee TC206 "Fine (Advanced, Technical) Ceramics"

Designation (Year adopted)	Title
ISO 14703 (2000)	Fine ceramics (advanced technical ceramics) - Sample preparation for the determination of particle size distribution of ceramic powders
ISO 14704 (2000)	Fine ceramics (advanced, technical ceramics) - Test method for flexural strength of monolithic ceramics at room temperature
ISO 14705 (2000)	Fine ceramics (advanced technical ceramics) - Test method for hardness of monolithic ceramics at room temperature
ISO 15165 (2001)	Fine ceramics (advanced technical ceramics) – Classification system
ISO 15490 (2010)	Fine ceramics (advanced technical ceramics) - Test method for tensile strength monolithic ceramics at room temperature
ISO 15733 (2001)	Fine ceramics (advanced technical ceramics) - Test method for tensile stress-strain behavior of continuous fiber reinforced composites room temperature
ISO 15761 (2002)	Fine ceramics (advanced technical ceramics) - Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance
ISO 15762 (2001)	Fine ceramics (advanced technical ceramics) - Test method for linear thermal expansion of monolithic ceramics by push rod technique
ISO/DIS 15732	Fine ceramics (advanced technical ceramics) - Test method for fracture toughness of monolithic ceramics at room temperature by SEPB method
ISO/DIS 17565	Fine ceramics (advanced technical ceramics) - Test method for flexural strength of monolithic ceramics at elevated temperature
ISO/DIS 18754	Fine ceramics (advanced technical ceramics) – Determination of density and apparent porosity
ISO/DIS 18756	Fine ceramics (advanced technical ceramics) - Determination of fracture toughness of monolithic ceramics at room temperature by SCF method
ISO/DIS 18757	Fine ceramics (advanced technical ceramics) - Determination of specific surface area of ceramic powders by the gas adsorption using the BET method
ISO/DIS 20501	Fine ceramics (advanced technical ceramics) – Weibull statistics for strength data
ISO/DIS 20507	Fine ceramics (advanced technical ceramics) – Terminology
ISO/DIS 20508	Fine ceramics (advanced technical ceramics) – Determination of light transmittance of ceramic thin films with transparent substrates.

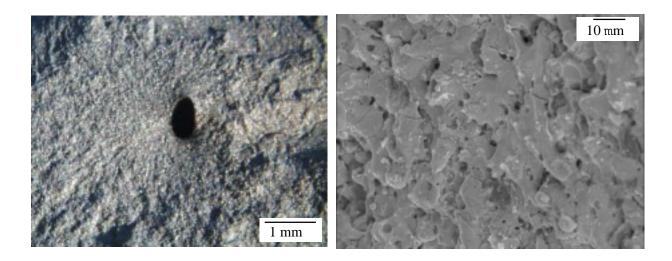


Figure 1. Failed test specimen showing thermocouple hole.

Figure 2. Crack network inside of the thermocouple hole.

<u>Observations:</u> Investigations [7, 8] indicated that the edge segments frequently failed from machining damage associated with the thermocouple holes in the edge segments. The holes were generated via EDM (electrodischarge machining), which lead to the severe cracking showing in Figure 2.

<u>Potential for Standards</u>: The use of better EDM machining procedures or the choice of a different technique that minimizes damage might have prevented the failures. Should standards have played a role? Currently no machining standard exists for ceramics, however, several standards do give guidance on how to machine specimens for a specific standard method, and some general literature exists. The current standards for test specimens generally prevent failure from machining damage. Unfortunately, the generalization of standards-specific techniques can lead to problems for the multiaxial stress states that are encountered in real-word components. Thus a guideline on how to machine a variety of test specimens and components is needed and likely could minimize failures do to machining damage.

In addition, during the failure investigation, it was noted that fracture mirror constants were lacking for estimating the failure stress of components made from advanced materials such as such as whisker, particulate, or *in-situ* reinforced ceramics. Also, a concise methodology to measure ill-defined mirror boundaries and thereby minimize subjectivity was noted. In addition, a function to place confidence levels on the estimated fracture stress was not available. A standardized procedure for measuring mirror boundaries and estimating the fracture stress and associated standard deviation might also benefit the ceramics community. Such a procedure could be added to existing fractography standards such as ASTM Practice C1322 [9].

Example 2 - Test Specimens for a Combustion Facility Window

<u>Background:</u> Design and life prediction of sapphire windows for use in the International Space Station Fluids and Combustion Facility (ISSFCF) required generation of strength and slow crack growth data under the conditions of interest. At the time, the only standardized test specimen that was practical was the uniaxial flexure test specimen. As a result, two sets of test specimens were machined according to typical scratch-dig [10] specifications.

<u>Observations:</u> During testing, one data set exhibited substantially greater scatter than the other data set [11]. Investigations using x-ray topography indicated that although both sets met scratch-dig specifications and appeared optically adequate, the sets contained substantially different amounts of subsurface machining damage, as shown in Figure 3. In follow-up discussions with the vendor [12], it was indicated that polishing of beams, rather than flat plates, is more difficult, and that they have less experience in estimating the appropriate forces and rates to use. A second procurement of circular plates of sapphire indicated this to be the case, as shown in Figure 4.

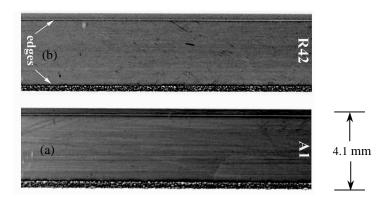


Figure 3. X-ray topographs of the tensile face of (a) an *a*-plane flexure test specimen and (b) an *r*-plane flexure test specimen. Note the asymmetric bevel finish on both test specimens and the remnant, longitudinal grinding marks on the *a*-plane test specimen (a).

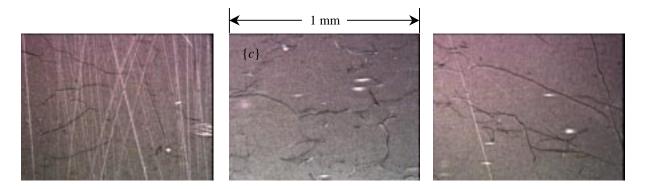


Figure 4. X-ray topographs of the faces of three, 50.4 mm disk test specimens showing a near dislocation level finish. The face of the disks is the *c*-plane.

<u>Potential for Standardization:</u> How could standards have improved the data quality? As in the previous example, a machining guideline may have eliminated the subsurface damage. In addition, had a specification for biaxial testing of plate-like specimens, rather than beams, been available, then multiple improvements in the test results and design could have been made: (1) plates likely would have had less subsurface damage, even without a machining guideline; (2) plates would have been a better representation of the component (a plates-like window), thereby minimizing the degree of extrapolation required in the design; (3) plates are less sensitive to edge chips than beams, and plates thus would have better represented the flaw distribution actually encountered in the windows.

Example 3 - Measurement of Inert Strength

<u>Background:</u> Ceramics and glasses exhibit stress corrosion or "slow crack growth" when subjected to stress in a corrosive environment such as water. Estimation of slow crack growth design parameters for glasses and ceramics via "dynamic" and "static" loading requires measurement of the materials strength both in the environment of interest and in the absence of the corrosive environment. The strength measured in the absence of the corrosive species is known as "inert strength."

<u>Observations:</u> A variety of methods, such as vacuum, low temperature, dry nitrogen, mineral oil, and silicone oil have been used, and several ASTM and ISO standards [13, 14] allude to these techniques.

Unfortunately, no systematic verification of the techniques has been performed, and the techniques do not produce statistically equivalent results to the ideal case of a vacuum, as shown in Figure 5. This occurs because the different techniques eliminate the environment to differing degrees. The use of "silicone oil" as an environmental barrier ignores the availability of several grades of silicone oil for use in diffusion pumps and transformers. The diffusion pump oils have differing degrees of permeability, and thus may allow differing rates of diffusion of the corrosive media. The use of dry nitrogen is also not without complication. A sufficient time for the nitrogen to dry the test specimen is required, especially if the material is porous. For the Dry N₂ Rate A tests, a flow rate of 2400 ml/min for 2 minutes was used, whereas for Dry N₂ Rate B tests, a flow rate of 3200 ml/min for 3 minutes was used. The flow rates and times corresponded to replacing the chamber volume 3 times and 6 times, respectively.

<u>Potential for Standardization:</u> Further investigation and the publication of a standard or guide for the measurement of inert strength is needed to insure consistent, accurate results.

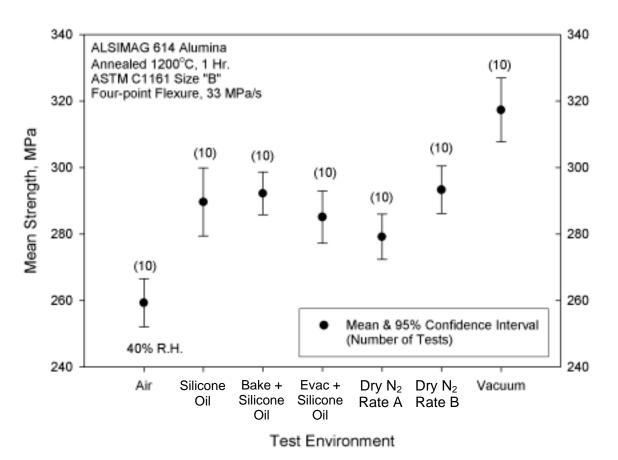


Figure 5. Inert strength of an alumina as a function of test environment.

Example 4 - Failure of a Sapphire Solar Collector

<u>Background</u>: Solar concentrator systems harness the sun's energy and concentrate it so that useful work can be extracted. The use of a system with both primary and refractive secondary concentrators (RSC's) provides higher solar concentrations ratios, efficiency, and heat receiver cavity flux tailoring as compare to conventional hollow refractive parabolic concentrator systems [15, 16]. The materials considered for RSC's are generally single crystal oxides such as sapphire (Al₂O₃), yttria-stabilized zirconia (Y_2O_3 -ZrO₂), yttrium-alumina-garnet ($Y_3Al_5O_{12}$ or YAG), and magnesium oxide (MgO). These materials are relatively brittle, and the reliability of such RSC's under the thermal shock conditions encountered during space mission sun-shade transitions is of great concern. Not only will the concentrator material experience thermal shock, but also large temperature gradients may be sustained at elevated temperature.

<u>Observations:</u> Recent testing of a sapphire RSC [17] resulted in severe cracking of the lens and transition sections, as shown in Figure 6. Failure analysis of the RSC indicated a large "bruise" on the face of the lens, as shown in Figure 7. Coarse machining marks within the bruise implied that it was made during machining and polishing, rather than during rig setup and testing. Failure likely occurred from either the bruise or a sharp transition that contain a steep temperature gradient. Elimination of the bruise and better design of the transition to minimize temperature gradients and thermal stresses would have improved the survivability of the RSC.

<u>Potential for Standardization:</u> As with the leading edge previously discussed, a standard for machining, polishing and handling might be beneficial. In addition, mirror constants for single crystal materials such as sapphire, were lacking. Once again, a standardized procedure for measuring mirror boundaries and estimating fracture stresses might benefit the ceramics community.

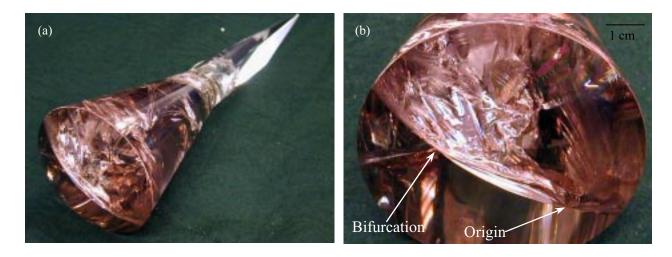


Figure 6. Solar refractive secondary concentrator after testing: (a) Overall view, and (b) Lens face.

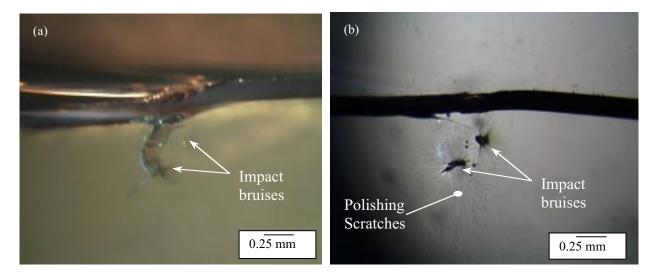


Figure 7. Lens face of a solar RSC observed under (a) transmitted and (b) reflective lighting conditions. Note the scratch marks and cracks within and emanating from the bruises, respectively.

Example 5 - Design Guides for Failure Critical Optical Components

<u>Background</u>: One other aspect of designing components such as the solar concentrator and leading edge segments is the choice of design methodology. Currently two approaches are generally considered: a deterministic (safe-life), fracture mechanics approach via the FLAGRO¹ computer code, and a probabilistic, Weibull strength based approach via the CARES² computer code. The FLAGRO analysis is required for all NASA fracture critical hardware.

<u>Observations:</u> The FLAGRO approach has the advantage of defining a flaw of inspectable size that can be insured via proof testing and inspection of the actual component, thereby lending confidence to the predictions. Unfortunately, proof testing is not easy for components such as the RSC. The probabilistic approach is convenient because it requires only strength data as an input to the code. However, it has the disadvantage that complete similitude is required: the flaw distribution causing failure in the test specimens must be identical in behavior to those in the component for the analysis to be accurate. Another disadvantage of the Weibull approach is that test specimen strength data, which typically has large statistical variability, is extrapolated in both scale and time, thereby resulting in low confidence in component predictions. Although the probabilistic approach incorporates fracture mechanic failure criteria, the analysis is based on strength statistics and does not use the fracture toughness of the material or relate it to a flaw size.

<u>Potential for Standardization</u>: For critical situations, a window must be sufficiently thick so that a critical flaw can readily be detected. Thus the fracture toughness of the material is the necessary basis of the design. The use of strength based design, probabilistic or otherwise, may be somewhat misleading for such situations because the "strength" of very well polished test samples and components can be quickly degraded in the service by small scratches, etc. Thus, designs base purely on strength statistics should only be used with caution. Some guideline for choosing the appropriate design method might be beneficial.

DISCUSSION AND CONCLUSIONS

The preceding examples provide evidence of lessons that could be learned for developing standards for advanced ceramics. Each example gives a real world scenario with attended problems of using ceramics in structural applications. In each case, observations were made that lead to conclusions that either existing standards had not fully addressed the problem encountered or no standard existed to address the problem. The two SDOs (ASTM and ISO) of the authors direct involvement, have developed about 50 highly useful standards for advanced ceramics. However, as useful as these standards may be, they only address the measurement of fundamental properties of advanced ceramics, and even then only within the limited experience of the technical experts who write the standards (e.g., silicon nitrides for heat engine applications). Additional development of standards is needed to promote the continued safe use and future introduction of advanced ceramics in demanding applications.

Lessons learned from the examples outside the experience base of the technical experts who populate the current SDOs for ceramics could be used to either extend existing standards or develop new standards. The growing web-based database of lessons learning and technical standards supported by NASA TSP provides a worldwide and publicly accessible means of documenting and archiving such examples and resulting standards.

ACKNOWLEDGEMENTS

The authors acknowledge Paul Gill of the NASA Standards Program for funding work on several standards currently being developed.

¹ NASA/FLAGRO 3.0.11," JSC 22267A, NASA Johnson Space Flight Center, May 1994.

² CARES Users and Programmers Manual, NASA Technical Paper TP2916, 1990

References

[1] Harris, D.C. Materials for Infrared Windows and Domes, SPIE Optical Engineering Press, Bellingham, WA, pp 173-177 (1999)

[2] VanRoode, M and Ferber, M. K, <u>Progress in Advanced Ceramics for Gas Turbine Applications</u>, ASME International, New York, NY 2002.

[3] American Society for Testing and Materials, C1145-95 "Standard Terminology of Advanced Ceramics," 1995 Annual Book of ASTM Standards, Vol. 15.01, ASTM International, West Conshohocken, PA (2001)

[4] American Society for Testing and Materials, <u>Standards Make the Pieces Fit</u>, ASTM International, West Conshohocken, PA (1994).

[5] Schneider, S.J. and Bradley, D.R., "The Standardization of Advanced Ceramics," Advanced Ceramic Materials, Vol. 3, No. 5, pp. 442-448 (1988)

[6] Gill, P., Vaughan, W.W., Garcia, D, "Lessons Learned and Technical Standards: A Logical Marriage," ASTM Standardization News, pp. 24-27, November (2001)

[7] Salem, J.A., "Failure Analysis and Failure Stress Estimation of a UHTC HfB₂-SiC Segment used in the SHARP B2 Flight Experiment" NASA Technical Memorandum 2002-211480, NASA Glenn Research Center, Cleveland, Ohio, March (2002).

[8] Mecholsky, J.J., "Reliability of UHTC Materials for Next Generation Space Vehicles," Sandia Report SAND2001-2896, Sandia National Laboratories, October 2001.

[9] ASTM C 1322-96a "Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics, "ASTM *Annual Book of Standards*, Vol. **15.01**, ASTM International, West Conshohocken, PA (2001)

[10] MIL-PRF-13830B, "Optical Components for Fire Control Instruments, General Specifications Governing the Manufacturing, Assembly and Inspection of," Defense Quality and Standardization Office, Falls Church, VA, (1997)

[11] Salem, J.A., Powers, L., Allen, R., and Calomino, A., "Slow Crack Growth and Fracture Toughness of Sapphire For a Window Application," pp. 41-52 in Window and Dome Technologies and Materials VII, Vol. 4375, R.W. Tustison, ed., Proceedings of the SPIE, SPIE, Bellingham, WA, 2001.

[12] Private Communication, Klaus Burckhardt, ISACO, Quakertown, PA, August (2000).

[13] ASTM C 1368-01 "Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Rate Testing," Annual Book of ASTM Standards, V. 15.01, American Society for Testing and Materials, West Conshohocken, Pennsylvania, (2001).

[14] ISO 14704, "Fine ceramics (advanced, technical ceramics) – Test method for flexural strength of monolithic ceramics at room temperature," Geneva, Switzerland (2000)

[15] Soules, J.A., Buchele, D.R., Castle, C.H., and Macosko, R.P., "Design and Fabrication of a Dielectric Total Internal Reflecting Solar Concentrator and Associated Flux Extractor for Extreme High Temperature (2500K) Applications," NASA CR 204145, NASA Glen Research Center, November 1997.

[16] Ning, X., Winston, R., and O'Gallagher, J., "Dielectric Totally Internally Reflecting Concentrators," Applied Optics, 26 [2], 300-305, 1987.

[17] Wong, W.A., Castle, C.H., "High Temperature Solar Vacuum Testing of a Sapphire Refractive Secondary Concentrator," in proceedings of the Space Technology and Applications International Forum--STAIF 2002, Mohamed S. El-Genk, ed., Albuquerque, New Mexico, 3-6 February 2002.