INTERNATIONAL STANDARDS FOR PROPERTIES AND PERFORMANCE OF ADVANCED CERAMICS – 30 YEARS OF EXCELLENCE

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ABSTRACT

Mechanical and physical properties/performance of brittle bodies (e.g., advanced ceramics and glasses) can be difficult to measure correctly unless the proper techniques are used. For three decades, ASTM Committee C28 on Advanced Ceramics, has developed numerous full-consensus standards (e.g., test methods, practices, guides, terminology) to measure various properties and performance of a monolithic and composite ceramics and coatings that, in some cases, may be applicable to glasses. These standards give the "what, how, how not, why, why not, etc." for many mechanical, physical, thermal, properties and performance of advanced ceramics. Use of these standards provides accurate, reliable, repeatable and complete data. Involvement in ASTM Committee C28 has included users, producers, researchers, designers, academicians, etc. who write, continually update, and validate through round robin test programmes, more than 45 standards in the 30 years since the Committee's inception in 1986. Included in this paper is a pictogram of the ASTM Committee C28 standards and how to obtain them either as i) individual copies with full details or ii) a complete collection in one volume. A listing of other ASTM committees of interest is included. In addition, some examples of the tangible benefits of standards for advanced ceramics are employed to demonstrate their practical application.

KEYWORDS – ceramics, composites, coatings, standards, characterizations, properties, measurements.

INTRODUCTION AND BACKGROUND

It is noteworthy that the 30th anniversary year of ASTM Committee C28 "Advanced Ceramics," coincides with 40th International Conference and Expo on Advanced Ceramics and Composites. This is not mere happenstance but instead reflects the driving forces and visionaries of the early to mid 1980's who recognized that the time had come for ceramics to become commonly recognized, used, and fabricated among the four commonly accepted classes of engineering materials: Metals; Ceramics/Glasses; Polymers; Composites.

Advanced ceramics is the accepted term in the United States for what are also known as engineering ceramics, structural ceramics, fine ceramics, and technical ceramics. By definition [1] an advanced ceramics is: *a highly engineered, high performance, predominately non-metallic, inorganic, ceramic material having specific functional attributes.* Historically, technological evolution has been the driver to push material performance requirements far beyond those normally satisfied by common engineering materials. As engineering demands for advanced technology applications increased, materials (including ceramics along with modern composites) were the enabling technology and, as it turned out, standards and design codes, were the enabling supporting technologies.

Until about 1980, most uses for ceramics in engineering applications were for situations involving wear-resistance, low stress, electrical insulation or some combination of these. Although many advanced technology applications of ceramics may seem common today, in the mid 1980's they were potential, not actual applications: microprocessor substrates, gas turbine vanes and blades, ball bearings, hip joints, ballistic armor, window panes, cutting blades, sensors, and electrolytes, to name a few. Indeed, the market prognosticators were keen on high-temperature applications such as future heat engines, both reciprocating and turbines

In light of the growing and projected applications of advanced ceramics, in 1985 the Engineering Ceramics Division of the American Ceramic Society evolved out of the long-standing Ceramic-Metal Systems Division. For similar reasons, in 1986 Committee C28 Advanced Ceramics of American Society for Testing and Materials (now ASTM International) was formed as a new stand-alone committee separate from a subcommittee within Committee C08 Refractories.

The scope of Committee C28 reads as follows: the promotion of knowledge, stimulation of research and development of standards (classifications, specifications, nomenclature, test methods, guides, and practices) relating to processing, properties, characterization, and performance of advanced ceramic materials. Committee C28 works in concert with other technical committees (e.g., D30 "Composite Materials," E07 "Non Destructive Testing," E08 "Fatigue and Fracture," E28 "Mechanical Testing," F04 "Medical and Surgical Materials and Devices", and G02 "Wear and Erosion") and other national and international organizations having mutual or related interests.

Committee C28 develops and maintains standards for monolithic and composite advanced ceramics. Standards of Committee C28 cover methods for testing bulk and constituent (powders, fibers, etc.) properties, thermal and physical properties, strengths and strength distributions, and performance under varying environmental, thermal, and mechanical conditions. The breadth of applications of the methods ranges from quality control through design data generation. The Committee's primary objective is the development of technically rigorous standards that are accessible to the general industrial laboratory and, consequently, are widely-accepted and used in the design, production, and utilization of advanced ceramics.

It is useful to know that ASTM International is the primary standards writing organization (SWO) for testing materials in the United States and is a private nonprofit corporation for the development of voluntary, full-consensus standards on the characteristics and performance of materials, products, systems, and services and for the promotion of related knowledge. These efforts are accomplished through the work of various ASTM committees consisting of volunteer experts, who, following previously established regulations generate a product (i.e., a standard) that is widely recognized, high quality, well accepted and generally used. ASTM standards are classified as test methods, practices, nomenclature and guides.

In this paper, details are provided for various periods the 30 years of existence of Committee C28: Early Years – Direction and Growth (1986-93); Transition Years – International Harmonization (1993-2006); Present Years – Applications and Validation (2006-Today). Finally, some examples of the tangible benefits of standards for advanced ceramics are employed to demonstrate their practical applications.

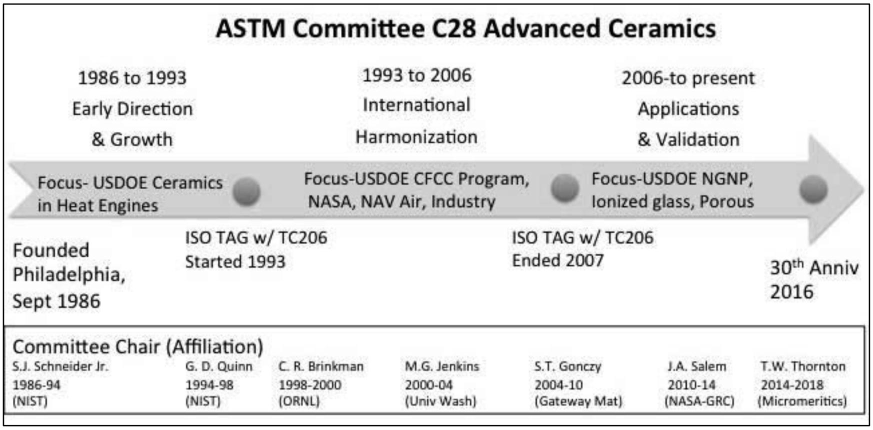

Figure 1 – Timeline for three decades of ASTM Committee C28

Committee C28: Early Years – Direction and Growth (1986-93)

In August 1986 at a meeting held at ASTM headquarters in Philadelphia and attended by over 150 interested parties, ASTM Committee C28 Advanced Ceramics was formed. Members included a wide range of interested parties from industry, government facilities, and universities. These members were classified as producers (those who made ceramic materials or products), users (those who used ceramic materials or products in components or devices) and general interest (those did not have a commercial interest in ceramics or ceramic components/devices). As it turned out the peak membership of Committee was in 1986 when interest was the greatest but the hard work of writing standards and bringing them to publication through a full-consensus balloting and approval process had not yet begun. Figure 1 illustrates the membership of Committee C28 from the early years through the transition years to the application years.

Early leadership of Committee C28 reflected organizations that had strong interest in the commercial and technological success of advanced ceramics. For example, the inaugural chair (Samuel Schneider, Jr) and vice chair (George Quinn) were affiliated with the Ceramics Division of National Institute for Standards and Technology (NIST) within the US Department of Commerce. In addition, Charles Brinkman and Robert McClung of the Metals and Ceramics Division at Oak Ridge National Laboratory (US Department of Energy) along with David Cranmer of NIST were the chairs of subcommittees C28.01 Properties and Performance, C28.02 Design and Evaluation, and C28.07 Ceramic Composites, respectively.

The direction of Committee C28 was dictated primarily by the perceived needs of programs for insertion of advanced ceramics in heat engines. These programs were primarily driven and funded by US federal government agencies and involved issues relevant to processing and characterization of ceramics as well as design with ceramics, including reliability.

The organization of Committee C28 in the early years consisted of three administrative committees (C28.90 Executive, C28.91 Nomenclature and C28.93 Awards) and four technical subcommittees (C28.01 Properties and Performance, C28.02 Design and Evaluation, C28.05 Characterization and Processing, and C28.07 Ceramic Composites).

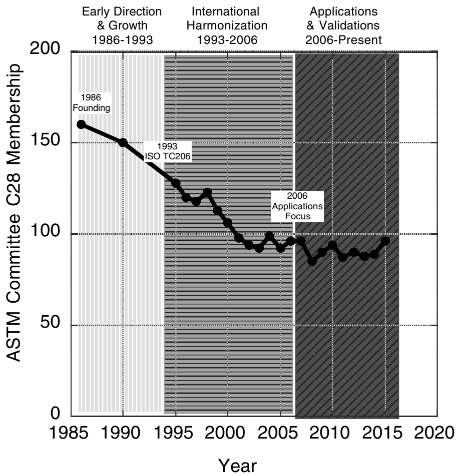

Figure 2-Membership of ASTM Committee C28 from 1986 to Present

Although Committee C28 began in 1986, the first standard developed and approved under its jurisdiction was Test Method C1161 on flexure testing at room temperature, first published in 1990. Test Method C1161 evolved from MIL STD 1942 (MR), "Flexural Strength of Advanced Ceramics at Ambient Temperature," which had been adopted in November 1983. Many years of effort had already gone into MIL STD 1942 including error analysis, procedures for test specimen preparation, round robin testing, etc. However, the development and subsequent publication of Test Method C1161 still required much effort in refining information contained in MIL STD 1942 and reconfiguring it into an ASTM standard.

It is important to note that the general process of developing a standard involves the following steps:

1) Establish of task group of experts (both within and outside ASTM);
2) Create a work item and prepare a draft standard;
3) Ballot the draft standard at the subcommittee level;
4) If approved with no negative ballots, ballot the draft standard at the committee and society, levels.
5) If approved at the committee levels, the standard is published as a separate or within volume in the ASTM Annual Book of Standards.

These steps may take two years or more to complete. If any negative ballots are received at any level these must be resolved before the balloting can proceed to the next level, thus ensuring the full-consensus approval process. It is important to note that all ASTM standards must undergo a mandatory review process every five years during which they must be either reapproved or revised, so as to maintain the relevancy and currency of published standards. Standards under the jurisdiction of Committee C28 are published in Volume 15.01 in the ASTM Annual Book of Standards. Obtain a complete listing of Committee C28 standards at http://www.astm.org/COMMITTEE/C28.htm.

Standards under the jurisdiction of Committee C28 that were approved and published during the early years of the committee numbered seven with one each under the jurisdictions of subcommittees C28.91 and C28.05, two under the jurisdiction of subcommittee C28.02 and three under the jurisdiction of subcommittee C28.01.

Committee C28: Transition Years – International Harmonization (1993-2006)

In 1993, two developments occurred that would dramatically alter the next decade of Committee C28: 1) The rise of ISO TC206 and 2) The growth of the CFCC Program and continued support of ceramics in heat engines projects in US DOE.

The first development reflected a natural evolution in standards writing organizations (SWOs). To wit the internationalization of standards that had evolved from organizational to national to regional to, finally, international standards. This evolution should, in theory, lead to the harmonization of the various organizational/national/regional standards into one international standard that reflects best practices, insights, methodologies and interpretations contained in these other standards. ISO TC206 Fine (technical, advanced) Ceramics was formally established in late 1992 with its first plenary meeting in 1994 at which Samuel Schneider of NIST (the first chair of Committee C28) presided as chair of ISO TC206, a position he held until 2003. As points of reference, several major national and regional SWOs from which ISO TC206 harmonized its standards included CEN TC184 on Technical Ceramics (est. 1989), ASTM C28 on Advanced Ceramics (est. 1986), JIS R on Fine Ceramics (est. 1979), BSI RPI/13 on Technical Ceramics (est. 1984), DIN NMP291 on Technical Ceramics (est. 1987) to name a few. Early membership of ISO TC206 included 10 participating (P) and 22 observer (O) countries.

Committee C28 created a new subcommittee, C28.94 ISO TC206 Technical Advisory Group (TAG) to work with ISO TC206. Since the official US representation to ISO is through ANSI, the C28.04 TAG acted only in an advisory role to ANSI for official ISO TC206 matters. In its evolution from its formation in 1993 to its dissolution in 2006, Subcommittee C28.94 developed bylaws in which technical experts were identified for various ISO TC206 efforts, subcommittees balloted on various stages of ISO TC206 activities and delegates for ISO TC206 plenary meetings were selected. One of the challenges of maintaining an ANSI/ISO TAG is financial because i) ANSI demands an annual fee to maintain each ISO TC affiliation and ii) delegates to international meetings incur travel costs that must somehow be met. Another challenge is the time commitment because in addition to advancing and maintaining its own ASTM standards with a primarily national membership, Committee members must advance and maintain ISO standards with an international membership. Although participation of Committee C28 members in ISO TC206 was initially vigorous and productive, the financial and time burdens along with politics within ASTM International, led to Committee C28's decision to officially discontinue its direct participation in ISO TC206 and to dissolve Subcommittee C28.94 in 2006. Note that ISO TC206 continues to this day with 18 P and 13 O member countries.

The second development was a well-funded broad-based program funded by US DOE and driven by industrial and aerospace applications of continuous fiber ceramic composites (CFCCs). It was recognized that CFCCs were the enabling technology for many types of advanced technologies that included chemical and petroleum refineries, next generation nuclear power, aerospace planes, gas turbines and scramjets. Because CFCCs were fabricated quite differently than monolithic ceramics and behaved quite differently unique test methods were required. Eventually, eleven standards for CFCCs were developed and published during this period. In addition, two symposia related to CFCCs were organized during this period resulting in two STPs (STP 1309 and 1392). Membership and participation in Subcommittee C28.07 Ceramic Composites grew during this time as well.

During this same period, efforts funded by US DOE in the area of insertion of ceramics in advanced heat engines also drove development of standards for monolithic advanced ceramics. In particular, contracts for fabrication and processing of ceramics included requirements for uniaxial, uniform testing (e.g., tension and compression). Long-term performance requirements for ceramic engine components required standards for creep, slow crack growth. Many years of development finally led to a comprehensive standard for fracture toughness testing of monolithic that included three different techniques that give remarkably consistent results for a NIST-provided standard reference material (SRM 2100). These efforts also lead to two other symposia that resulted in two additional STPs for monolithic and composite ceramics (STP 1201 on life prediction/data and STP 1409 on fracture resistance).

Also, occurring during this period was the development by Committee C28 of an ASTM-required, long-range plan (LRP). As part of this plan, a new permanent subcommittee on long range planning (Subcommittee C28.95) was established. This LRP also included details of an operating plan that addressed development and maintenance of national and international standards for advanced ceramics as well as organization, leadership, membership, outreach and funding of Committee C28. As part of outreach, a summary and compilation of Committee C28 standards was created as a faux newsletter, called “Advanced Ceramic Sentinel.” This publication was distributed on the web, at meetings and for a time on a CD as a hypertext linked interactive document. Similarly, a pictorial compilation of C28 standards was developed and distributed in poster form (see Figure 3).

While the number of new standards under Committee C28 grew rapidly during this period, the Committee also engaged in its first mandatory review of existing standards per ASTM requirements. That is, individual standards must be re-approved or revised every five years or they will be subject to mandatory withdrawal as active standards. This requirement assures that standards remain relevant and up to date. Figure 4 graphically represents the number of standards under jurisdiction of Committee C28 from its inception to the present as well as the number of standards requiring review in any given year.

Organizationally, the subcommittee structure of Committee C28 changed during this period as well with addition of two administrative subcommittees and the reorganization and renaming of several technical subcommittees. Part of this reorganization was the result of developing a long-range plan that including a refocus on user-specific standards (i.e., applications). Some areas that were impacting applications including liaisons with Mil-Hdbk-17 on Composites, ASTM Boiler and Pressure Vessel Code and Gas Research Institute.

Another change that was implemented for a short time was the semi-annual meeting location and schedule. Since its inception, Committee C28 had sought to develop a close relationship with ACerS by scheduling one of its twice-a-year meetings in conjunction one of the major meetings of ACerS. Up until about 1999, Committee C28 had been meeting in January at the “Cocoa Beach” conference of ACerS in January and then wherever ASTM was meeting in May/June in order to interact with fellow ASTM committees such as E08 on Fatigue and Fracture or D30 on Composites. Starting in with 2000, Committee C28 changed its meeting schedule such that its April/May meeting was in conjunction with the ACerS annual meeting and its November meeting was during an ASTM committee week. Part of the motivation for this change was to interact with a broader ceramics community. In addition, Committee C28 organized sessions on standards at the ACerS annual meeting in order to educated and reach out the broader ceramics community.

Standards under the jurisdiction of Committee C28 that were approved and published during the transition years of the committee numbered 37 with one each under the jurisdictions of subcommittees C28.91 and C28.05, two under the jurisdiction of subcommittee C28.04, six under the jurisdiction of subcommittee C28.03, 11 under the jurisdiction of subcommittee C28.07, and 16 under the jurisdiction of subcommittee C28.01

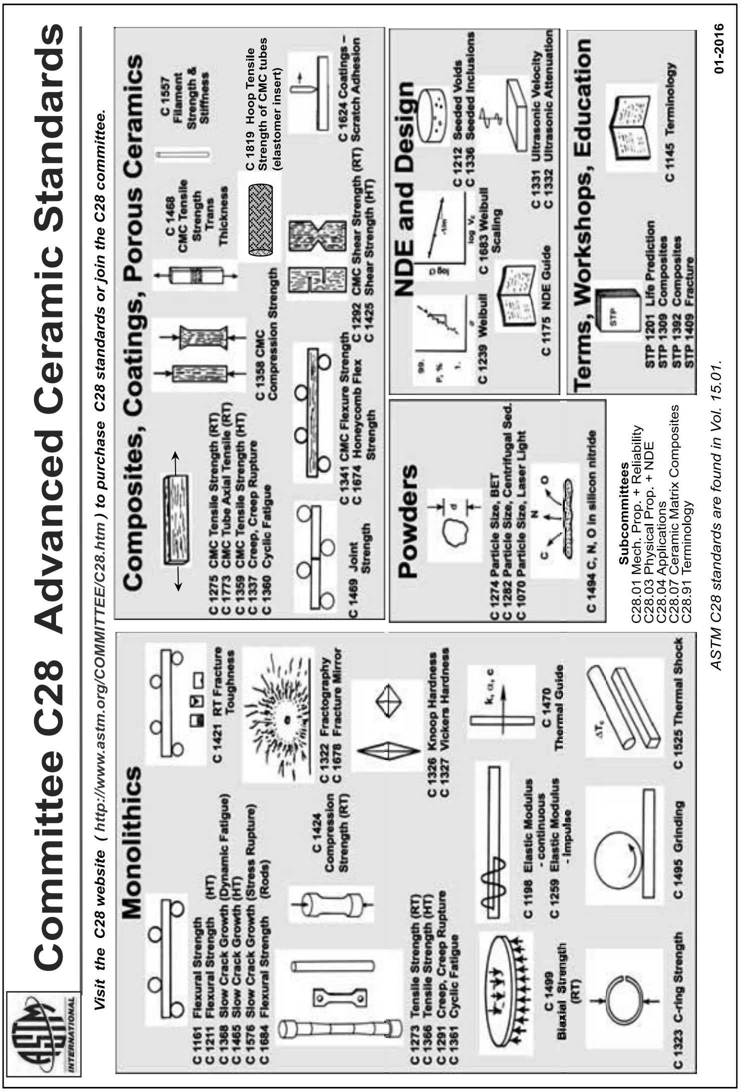

Figure 3-Pictorial illustration of Committee C28 standards

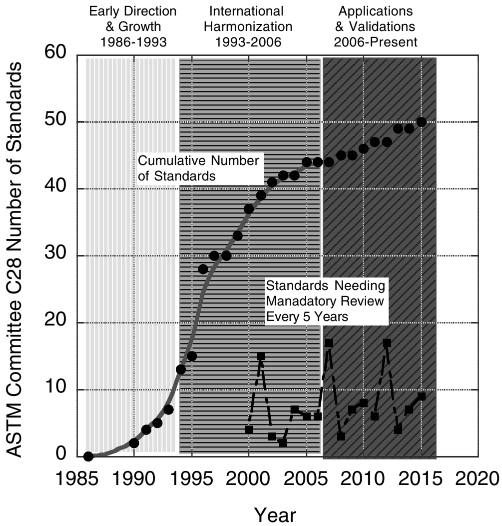

Figure 4 – Cumulative number of Committee C28 standards and number of standard needing mandatory review in any given year

Committee C28: Present Years – Applications and Validations (2006-Present)

Reflecting on the long-range plan and the increasing time-commitment to the duality of development/upkeep of ASTM standards and the development/upkeep of ISO TC206 standards, Committee C28 made a decision to reassess its role in standards development. Concurrently in industry more emphasis was being placed on applications of such as ionized glass for touch screens, electrolytes for solid oxide fuel cells (SOFCs), windows in aerospace applications, porous ceramics for filters, traps and substrates, and joining materials for ceramic joints. In addition, a new initiative from US-DOE called Next Generation Nuclear Power (NGNP) was calling for new standards to assess the mechanical, physical, and thermal behaivour of CFCC materials in unique shapes such as tubes.

Committee C28 was once again reorganized to reflect changes in direction. A major change was the decision to withdraw official participation ISO TC206. In addition, Committee C28 decided to focus more effort on education and outreach in order the “get the word out” on C28 standards and their applications.

In 2006, Committee C28 once again changed its meeting schedule back to the original one of meeting in January at the “Cocoa Beach” conference of ACerS and then wherever ASTM was meeting in May/June in order to interact with fellow ASTM committees. However, Committee C28 soon decided that for economy of both time and finances, a teleconference meeting might be better investment for its midyear meeting. As a result, the current, and relatively successful twice-a-year meeting schedule for Committee C28 is as follows: Late January in conjunction with ACerS International Conference on Advanced Ceramics and Composites (ICACC) in Daytona Beach, Florida and in mid July as a WebX teleconference.

Reflecting its emphasis on education and outreach, Committee C28 has presented a poster standard for ceramics at the Poster Session of the ICACC for the last half decade. Recently, the Committee has created a Linked In presence. Other initiatives include regular articles in ASTM's *Standardization News* and ACerS's *Ceramic Technology* burst e-mail and *Ceramic Bulletin.*

Note that one of the hallmarks of ASTM standards that contributes to their technical rigor and quality is the Precision and Bias (P&B) statement. The P&B statement is required by ASTM in all test methods and provides the user with insight on the repeatability (precision) of the procedures and their accuracy (i.e., bias) to some known reference material. Typically round robin test programs per ASTM E691-99 "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method" are used to produce inter- and intra-laboratory repeatability values. Over the years members of Committee C28 have been instrumental in organizing, participating, interpreting and applying round robins for validating not just C28 standards but ISO and other SWO standards as well.

The current subcommittee structure and their functions are summarized as follows.

C28.90 Executive This subcommittee manages administrative matters of main Committee C28 through its membership comprised of the committee and subcommittee officers of C28.

C28.91 Nomenclature and Editorial This subcommittee compiles nomenclature and terminology used in the various standards of Committee C28.

C28.92 Education and Outreach This subcommittee develops and supports efforts for education and outreach for the C28 committee.

C28.93 Awards This subcommittee accepts/acts on nominations for various awards

C28.95 Long Range Planning This subcommittee proposes, facilitates and promotes long range planning activities consistent with the mission.

C28.01 Mechanical Properties & Reliability This subcommittee develops standards for mechanical properties and reliability (short term and long term) of monolithic advanced ceramics in a number of areas including flexural strength, tensile strength, compressive strength, cyclic fatigue, creep and creep rupture, hardness, and fracture toughness.

C28.03 Physical Properties & NDE This subcommittee develops standards for physical, chemical, micro-structural, and non-destructive characterization of powder and bulk advanced ceramics.

C28.04 Applications This subcommittee develops standards (including guides, specifications, practices, test methods) for various engineering applications of advanced ceramics, such as nanoceramics, coatings, electrodes, porous ceramics, fuel cells, armor, sensors/actuators, and thermal systems.

C28.07 Ceramic Matrix Composites This subcommittee develops standards for determination of the thermo-mechanical properties and performance of ceramic matrix composites including tension, compression, shear, flexure, cyclic fatigue, creep/creep rupture, ceramic fibers, interfacial properties, thermo-mechanical fatigue, environmental effects, and structural/component testing.

Standards under the jurisdiction of Committee C28 that were approved and published during the present years of the committee numbered seven with one each under the jurisdiction of subcommittee C28.05, two under the jurisdiction of subcommittee C28.07 and three under the jurisdiction of subcommittee C28.01.

Tangible Benefits of Standards

Although many examples of tangible benefits of ASTM C28 standards could be cited only a few are given here in the interests of brevity.

F2393 Standard Specification for High-Purity Dense Magnesia Partially Stabilized Zirconia (Mg-PSZ) for Surgical Implant Applications

ASTM Committee F04 on Surgical and Medical Devices and the U.S. Food and Drug Administration used generic standards from Committee C28 for their standard specification, F2393. Some specifics are as follows:

- "The average room temperature flexural strength shall be 600 MPa (87 000 psi) or greater by 4 point bend testing in accordance with Test Method C1161, test configuration B. A minimum of 10 samples are to be tested."
- "If Weibull modulus is determined, test results shall be evaluated in accordance with Practice C1239. The minimum number of test specimens shall be 30 and the minimum acceptable uncensored, unbiased Weibull modulus shall be 10."
- "The minimum room temperature elastic modulus shall be 180 GPa (26 200 ksi) in accordance with Test Method C1198.Arectangular specimen with dimensions of 60 by 10 by 3 mm is recommended. An acceptable alternative test method for elastic modulus is Test Method C1259."
- The minimum Vickers hardness value shall be 1000 HV in accordance with Test Method C1327. The load shall be 9.8 N (1kg) and the dwell time shall be 15 s.

F2094/F2094M Standard Specification for Silicon Nitride Bearing Balls

ASTM Committee F34 on Rolling Element Bearings used generic standards from Committee C28 for their standard specification, F2094. Some specifics are as follows:

- "Either 3-point or 4-point test methods may be used for flexural strength, which should be measured in accordance with Test Method C1161 (size B)..."
- "Fracture resistance shall be measured by either …… (see Annex A1) or by a standard fracture toughness test method." (C1421)
-

Transparent Armor Ceramics as Spacecraft Windows

Standards from Committee C28 allowed comparisons among authors and helped to interpret data. In particular:

- Standardized fracture toughness tests using Test Method C1421 ensured correct comparisons of different authors' results
- Standard-sized circular disks could be used to determine Poisson's ratio and Young's modulus via Test Method C1259 and biaxial strength via Test Method C1499 as well as the slow crack growth parameters, n and A, via Test Method C1368. This allowed efficient understanding of the behavior of the material.

Standard Reference Materials

Standard Reference Materials (SRMs) certified by NIST are available for users to verify test procedures including user techniques and test apparati. In particular:

- SRM 2100 was developed to improve fracture toughness testing of ceramics. It may be used with conventional testing machines and flexure (bend bar) test configurations. The SRM is a set of five hot-isostatically pressed silicon nitride test specimens. The fracture toughness is certified by billet of the SRM material. For example, Billet C is certified to have a mean K_{Ic}=4.572 MPa•m$^{1/2}$ and uncertainty of 0.228 MPa•m$^{1/2}$ (5% of mean) for a single test specimen and 0.106 MPa•m$^{1/2}$ (2.3% of mean) for all five test specimens.

- SRM 2830 was developed to improve Knoop hardness testing of ceramics. It may be used with conventional hardness testing machines that make indentations that are measured with an optical microscope. The SRM is prepared from a silicon nitride ceramic bearing ball in which five indentations have been made at a load of 19.6 N (2 kgf). Each SRM is individually certified for the size of each of the 5 indentations with average diagonal length (≈ 142.0 m), and average hardness HK2. The HK2 is nominally 13.86 GPa or 1,414 HK2. (Test Method C1326)
- SRM 2831 was developed to improve Vickers hardness testing of ceramics and hardmetals. It may be used with conventional hardness testing machines that make indentations that are measured with an optical microscope. The SRM is a hot-isostatically pressed tungsten carbide with 12% cobalt disk which has five indentations made at a load of 9.8 N (1 kgf). Each SRM is individually certified for the size of each of the 5 indentations, the average diagonal length (≈ 35.0 m), and the average hardness HV1. The HV 1 is nominally 15 GPa which is in middle of the hardness range for most ceramics and cutting tool carbides (Test Method C1327)

CONCLUSIONS

Demand for advanced ceramics and ceramic matrix composites in the market place is expected to continue to grow as these materials improve in consistency and reliability, and reduced cost. Standardized test methods are expected to accelerate use of these materials as they become available and are used nationally and internationally. ASTM Committee C28 on Advanced Ceramics has produced, as of this writing, fifty high-quality, technically-rigorous consensus standards for processing, characterization, design, and evaluation of this class of materials. These activities have accelerated in recent years and many more standards are expected to be completed in the near future.

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