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NDE Standards for High Temperature Materials

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NDE STANDARDS FOR HIGH TEMPERATURE MATERIALS

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Introduction

There is a need for a new generation of structural materials suitable for high performance, high temperature heat engines. The materials must exhibit sufficient strength, toughness, and durability to resist mechanical damage and thermal degradation while operating at extreme temperatures, i. e., at maxima of approximately 1300 or 1600 degrees Centigrade depending, respectively, on whether metallic or ceramic materials are used [1]. In addition, the materials must permit being formed into light-weight, efficient heat engine components. These requirements can be met by toughened monolithic ceramics and by ceramic fiber reinforced refractory composites with ceramic, metallic, and intermetallic matrixes [1,2]. Monolithic silicon carbide and silicon nitride are leading candidates for hot section components in terrestrial automotive heat engines [3]. Ceramic fiber reinforced composites with ceramic, metallic, and intermetallic matrixes are contemplated for aerospace power and propulsion applications and associated high temperature structures.

It has been estimated that quality control and inspection of advanced composites may represent as much as 35 percent of the cost of manufacture [4]. This suggests the degree of thoroughness and sophistication of inspection technology that will be necessary not only for final products but also for monitoring and controlling incoming raw materials and for component processing and fabrication. New refractory materials will tax the capabilities of current nondestructive evaluation (NDE) and inspection technology [5]. Some totally new NDE and standardization approaches will be needed. Existing inspection techniques and standards will require augmentation.

The situation demands that present inspection standards be upgraded and that new standards be developed in concert with the advancement and development of inspection methods. This should be done concurrently with the evolution of processing and fabrication methods for the new generation of high temperature materials and structural components. Appropriate inspection standards should be considered for use (a) during raw material processing to ensure purity and quality, (b) during component fabrication to screen out defective pieces, and (c) during service to assess mechanical damage and thermo-chemical degradation [6,7].

Without suitable inspection methods and standards the quality, integrity, reliability, and serviceability of new high temperature structures will remain uncertain. High temperature materials and structures that fail to meet internationally developed and accepted inspection standards are unlikely to successfully compete in high technology markets [8]. This report reviews prevailing needs and recommends approaches and activities required to ensure that appropriate and necessary inspection methods and standards are developed.

Situation Assessment

Advanced structural ceramics and refractory composites for space power and propulsion systems present inspection difficulties that exceed those encountered with conventional engineering materials. Nondestructive evaluation methods and standards that will ultimately be applicable to production and quality control of the new refractory materials and structures are still emerging. The problems being addressed range from flaw detection well below 100 micron levels in monolithic ceramics to global imaging of fiber architecture and matrix anomalies in composites. The inspection needs are different depending on the processing stage, the fabrication method, and the nature of the finished product. For example, specific methods are needed for inspecting powders and green compacts before monolithic ceramics are densified by hot pressing or sintering.

For fully densified monolithic ceramic components inspection techniques must certainly detect and characterize various types of discrete defects like cracks, voids, and other overt discontinuities. It is also important to discern and characterize microstructural conditions and diffuse flaws that govern overall strength, fracture toughness, impact resistance, and resistance to thermal-mechanical-chemical degradation [9,10]. Dispersed micro-flaws and morphological anomalies can reduce reliability and service life just as much as individual macro-flaws. McCauley [11] has pointed out that "hidden 'defects' like subtle differences in porosity, phase composition, microstructure retained strain (residual stress), and sub-critical cracks can result in properties well below acceptable levels, even though traditional nondestructive interrogation reveals no gross inhomogeneities, cracks, or voids."

Although monolithic ceramics have fairly good high temperature strength and superior oxidation resistance, their brittle nature and sensitivity to minute defects lead to wide variations in mechanical properties and a low fracture toughness [12]. The fracture toughness of monolithic ceramics can be improved by transformation or whisker toughening. Further improvements in strength, toughness, and durability can be achieved by ceramic fiber reinforcement in ceramic and intermetallic matrix composites. While the strength of monolithic ceramics is governed by the size and population density and distribution of minute defects, fiber reinforced composites are insensitive to minor matrix flaws [13]. Strength, toughness, and fracture resistance

of composites depend primarily on intrinsic fiber strength, fiber-matrix bond strength, and ability of the matrix to absorb fracture energy via micro-cracking [14].

Advanced heat engine components are likely to consist of fiber strengthened composite structures. Strengthening will include reinforcement with a variety of intermixed ceramic fibers that are three-dimensionally interwoven. The extrinsic thermomechanical properties of these composite structures are literally created *in-place* during processing and fabrication stages [15]. Their complex nature creates the need for new approaches and standards that allow unambiguous evaluations of defect states, internal structural anomalies, and subtle morphological factors that govern their mechanical and load response properties.

Because high temperature materials are still under study and development, they are moving targets for materials characterization and inspection technology. This situation calls for parallel development of nondestructive evaluation technology alongside processing and fabrication research advancements. By parallel development it becomes possible to assure that inspection methods and standards mature simultaneously with advancements in refractory materials.

Approaches to Standards Development

ASTM Activities - The formulation of reference and calibration standards for inspecting ceramics and refractory composites was formally initiated during 1988 by Committee C-28 of ASTM. A task force for devising ceramic NDE standards was formed. The task force began by surveying all pertinent extant documents with the idea of modifying them if necessary to cover advanced ceramics. Since early 1990, over twenty-five ASTM E-07 standards were reviewed and changes were recommended to make them applicable to advanced ceramics and refractory composites. The recommendations were forwarded to cognizant subcommittees and are in various stages of becoming incorporated into appropriate ASTM documents.

One result of the previously mentioned ASTM activities is a proposed new document entitled *Test Methods and Standards for Nondestructive Testing of Advanced Ceramics*. The purpose of the document is to serve as a standard guide that identifies radiological, ultrasonic, and liquid penetrant inspection methods and procedures for advanced ceramics and refractory composites. The guide identifies current ASTM standards that are directly applicable to the examination of ceramics and refractory composites. The guide also covers ASTM standards that have been modified by mutual agreement between Committees E-07 and C-28.

A second result of ASTM activities is the development of a new document entitled *Fabricating Ceramic Reference Specimens Containing Seeded Voids*. This document provides an ASTM Standard Practice for fabricating green and sintered bars of silicon carbide and silicon nitride containing internal and surface-connected voids

at prescribed locations. The test bars will contain intentionally introduced discontinuities with known sizes and shapes. The purpose is to provide calibration standards for determining the relative detection sensitivity and spatial resolution of ultrasonic and radiographic techniques. Bars of this type have been used to establish probability-of-detection statistics and inspection parameters and procedures for a range of material conditions in monolithic ceramics [16,17].

A third result of ASTM activities is a new tabulation of densities and ultrasonic velocities for advanced ceramics and high temperature composites. These are essential engineering data that are currently unavailable in the ASTM Standard Practice E 494, *Measuring Ultrasonic Velocity in Materials*. This is a continuing effort to ensure that accurate, comprehensive density and velocity data are available for a broad range of ceramics and refractory composites.

Unique sets of characteristic ultrasonic velocities are exhibited by fully dense, monolithic materials, e.g., polycrystalline metals and glasses. However, ceramics that have porosity and fiber reinforced composites that have both texture and porosity will exhibit a range of velocities according to the degree of porosity and anisotropy [18,19]. Examination and evaluation of ceramics and refractory composites are ultimately dependent on compilations of data connecting velocity with texture and porosity. These data are needed because of the interrelations among velocity, texture, density, elastic moduli, and mechanical properties.

Current ASTM activities will certainly help assure that needed inspection techniques and standards are established for high temperature materials. In some instances it appears that modifications of existing documents will suffice. These modifications are necessary but insufficient because the documents were originally developed for conventional materials and methods. The proper inspection of advanced materials and structures will require some totally new standards based on innovative NDE methods.

Monolithic and Toughened Ceramics - For monolithic ceramics the chief problem is to detect distinct flaws such as cracks, voids, grain clusters, and foreign inclusions having sizes to 100 micron levels and often down to 10 micron levels [20]. Appropriate flaw detection methods are needed to deal with surface, sub-surface, and volume flaws. Dispersed micro-porosity, diffuse flaw populations, texture and density variations also need to be found for their potentially deleterious effects on the strength and fracture resistance of monolithic and toughened ceramics.

Among the most important requirements for the specification of inspection methods for ceramics is the establishment of probability-of-detection (POD) data for a variety of flaw types. Probability-of-detection data must be accompanied by a description of exact material conditions (surface finish, thickness, shape, grain structure/coarseness, etc.) under which they were determined for specific inspection

procedures and instrument settings. Only with this approach can a basis be established for selecting appropriate inspection parameters and for determining their potential effectiveness.

Fractography conducted on monolithic ceramic bend specimens has shown that principal fracture origins were subsurface and surface pores or voids [21]. These were followed, in approximate order of frequency by narrow crack-like voids, columnar grains, large grains, clusters of grains, metallic inclusions, and surface machining scars. The principal fracture origins just mentioned are common to the MOR (modulus of rupture) bars used, i. e., bend specimens that are sensitive to surface flaws. Volume flaws rather than machining scars and superficial flaws would dominate in other cases, depending on stress patterns. For each type of potential fracture origin, surface or volume, external or internal, it is necessary to establish POD statistics for each individual inspection technique.

In addition to detecting dominant individual flaws such as inclusions, voids, and cracks, it is essential to characterize monolithic ceramics relative to dispersed porosity patterns, density gradients, and grain size fluctuations. These latter factors form the environments of discrete flaws and govern susceptibility to crack growth and fracture. In the case of particulate, transformation, and whisker toughened ceramics it is necessary to detect and characterize microstructural anomalies, density variations, adverse textures, and anomalous whisker alignments [22].

Appropriate nondestructive evaluation techniques are required to quantitatively characterize the above-mentioned microstructural and morphological features in monolithic and toughened ceramics. These techniques should provide imaging and mapping methods that reveal global variations of porosity, texture, and diffuse flaw populations. The imaging need not resolve each individual micro-flaw in diffuse populations. In this case resolution of the individual micro-flaws is usually impractical and unnecessary. Instead, what is needed is a quantitative assessment of the extent and distribution of these aberrations. This materials characterization approach is useful for comparing parts before they are placed in service and assessing changes due to thermomechanical degradation from exposure to service environments.

Ceramic and Intermetallic Matrix Composites - Composites must be inspected for constituent integrity, delaminations, disbonds, and other overt discontinuities as well as for harmful local and global variations in matrix densification, fiber distribution, fiber architecture, intralaminar integrity, and fiber-matrix bond quality [23]. It is relatively easy to create artificial disbonds in composite laminates by inserting foreign materials having various sizes and shapes, e. g., plastic wafers, metal foils, or debonding agents. These are contrivances used to simulate real discontinuities in calibration samples. They have been used as means for establishing detectability data, instrument settings, and inspection parameters.

Composites can be approached with the attitude that the detection of individual *micro*-flaws is unnecessary. This does not mean that distinct *macro*-flaws such as delaminations, cracks, and similar discontinuities can be ignored. It should simply be recognized that composites may contain a profusion of minute defects that have no discernable effect on reliability or performance unless they are in close proximity and interact massively or permit environmental degradation at high temperatures.

What must be detected in composites are associations of flaws that can collectively degrade reliability and performance. Sparsely distributed, occasional matrix cracks, broken fibers, or misaligned fibers need be of little concern. Improper bonding between fiber and matrix must be of high concern [13]. In ceramic matrix composites the fiber-matrix bond should be neither too strong nor too weak while in metallic and intermetallic matrix composites the bond may be quite strong. Generally, a key factor is the quality of fiber-matrix interfaces and interphases that, in turn, determine overall strength, fracture toughness, and impact resistance. In refractory composites, therefore, a major challenge is to characterize the collective effect of improper fiber-matrix and interlaminar bonds on the mechanical integrity and strength. This is in addition to the need to detect any overt, dominant discontinuities or global aberrations that would have an overriding effect on structural integrity under particular loading conditions.

Probably, the greatest challenge to the inspection of composites is the difficulty of generating reference and calibration standards that possess subtle microstructural aberrations that nevertheless can have significant effects on mechanical properties and load response, e. g., fiber-matrix interface bond irregularities. The calibration standards should be in the form of material samples that possess representative structural aberrations and corresponding mechanical property variations while duplicating the anisotropies and geometric properties of real parts.

Materials Characterization - McCauley [11] has argued that advanced refractory materials represent enormous challenges and that it is necessary to "recognize the importance of materials characterization concepts for controlling and monitoring a material's full unique signature" and that "this will require the extension of traditional NDT into chemical and microstructural interrogation, transitioning sophisticated materials characterization techniques out of the research laboratory."

It is difficult enough even in the case of monolithic, polycrystalline solids, e.g., metals, ceramics, to generate reference standards for quantitative ranking of microstructure-dependent properties (that is, strength, toughness, impact resistance). The difficulty is compounded for composites with complex, heterogeneous, anisotropic microstructures. These complications need to be overcome in developing representative materials and benchmark structures that can be used as comparative reference standards for materials characterization and instrument calibration.

A specific challenge to inspection standardization and calibration technology is the need to fabricate reference samples that exhibit microstructures and morphologies that represent a realistic range of material conditions and mechanical properties from poor to ideal. This is to ensure that nondestructive materials characterization techniques will be able to differentiate rejectable from desirable parts. Underlying this approach is the fact that nondestructive methods are indirect and depend on signal interpretations and empirical correlations to assess the quality and mechanical characteristics of a material or structure.

The simplest approach is to comparatively characterize a set of test samples that have been subjected to different levels of thermal or mechanical degradation. Each sample in the set would initially have been identical to all the others, based on careful verification by suitable NDE methods. After thermal or mechanical conditioning, each sample exhibits different physical-chemical-mechanical properties, e. g., modified fiber-matrix interface properties. Although each sample in the set constitutes an important reference, the sample with optimum properties is taken as a benchmark. This assumes that the benchmark sample is either in a pristine condition or otherwise represents an ideal, preferred condition of the material or structure.

Because the quality and strength of monolithic, composite, and composite-like material are subject to numerous processing variables, it is useful to feed back nondestructive evaluations to process development research. This concomitantly aids in creating temporary "application" standards for identifying the most successful production conditions and the best resultant materials and parts. Nondestructive monitoring during processing research and fabrication development can help identify and refine the best ultimate inspection standards and property characterization procedures.

The structural integrity of monolithic ceramics and refractory composites depends on avoiding fabrication flaws and maintaining high quality during processing [24]. An approach for consistently producing high quality ceramics is to utilize nondestructive evaluation techniques during materials research and processing development to help determine stages when harmful flaws are likely to be introduced. Steps can then be taken to minimize their occurrence through improvements in processing. This can be done at various stages of processing to save the cost of finishing parts that contain defects from an earlier stage. The least efficient approach, usually avoidable, is to use nondestructive evaluation after the last stage of fabrication to reject parts that contain harmful flaws. This can result in costly high rejection rates because one cannot "inspect in" quality!

Signal Analysis and Evaluation - The peculiarities and complexities of advanced materials, especially composites and composite structures, will require approaches that go beyond simple calibration pieces. For advanced materials, calibration samples and elementary procedures may not suffice. Indeed, simple "universal" calibration

standards can be invalid and illusory. This observation is based on the fact that many individual factors can simultaneously influence probe media used to interrogate materials for assessing their microstructural, morphological, and mechanical property variations.

In the case of computed tomography, the effects of x-ray beam hardening and geometric shadowing can undermine image reconstruction algorithms. In the case of ultrasonics, multiple reflections, mode conversions, and boundary conditions can hinder correct measurements. In either previous case, and in general, clear correlations may be obscured by a host of incidental geometric and microstructural factors. Sophisticated interpretational methods will then be required to extract from images and signals the desired information regarding particular material characteristics or properties. In addition to advanced signal analysis approaches, multiparametric probing using several nondestructive evaluation techniques may be mandatory to extract and separate complementary and corroborative data. This will help remove ambiguities that would arise if only one technique were relied upon and where the effects of several material variables overlap and need to be isolated.

Material calibration standards certainly need to be augmented with advanced multiparametric signal analysis software and computerized evaluation methods. The appropriate foundations for these advanced methods are expert systems based on adaptive learning methods and neural networks that are, in turn, based on carefully devised learning sets. The learning sets should consist of extensive series of material samples that exhibit all combinations of factors that influence probe media and factors that are likely to exist in the materials and structures to be interrogated. Nondestructive evaluation approaches evolved from this data base may very well consist of standardized signal processing and interpretation software packages. The packages would contain algorithms for signal transformation, image enhancement, signature analysis, feature extraction, pattern recognition, and classification [25,26].

NDE Technology

General - The primary nondestructive evaluation techniques applicable to ceramics and refractory composites are visual-optical examination, liquid penetrant inspection, radiography, and ultrasonics [5,6,9]. Specialized techniques include fluorescent penetrants, microfocus x-radiography, computed tomography, analytical ultrasonics, and acoustic microscopy for monolithic and particulate and whisker toughened ceramics. Computed tomography, film and digital radiography, scanning ultrasonics, and acousto-ultrasonics are among the specialized techniques suitable for inspecting ceramic fiber reinforced ceramic and intermetallic matrix composites.

Methods for Raw Materials - The screening and characterization of ceramic powders and ceramic toughening agents (crystallites, whiskers) are the first step in assuring the quality of monolithic structural ceramics. Particle size and size distribu-

tion, chemical purity, crystalline phase, morphology, contaminants, and physical properties are among the attributes that require assessment and close control. Inspection methods include light scattering, gas absorption, microscopy, x-ray diffraction, Auger and mass spectroscopy, and chemical analysis [27]. These are primarily physical-chemical analysis methods that nevertheless fall under the purview of nondestructive characterization and require appropriate standards. Similar methods are needed to assess continuous ceramic fibers, fiber tows/bundles, and fiber preforms used to fabricate composites.

Additional raw materials involved in fabricating monolithic ceramic structures and refractory composites are processing aids such as organic binders, dispersants, lubricants and also carrier vehicles such as water, solvents, vapors, and gasses. These latter ingredients must be characterized for purity, contamination, molecular weight, viscosity, and their relative effectiveness during processing stages such as forming, injection molding, slip casting, infiltration, and chemical vapor deposition.

Methods for Green Compacts - The formation of bisques and green state bodies is an intermediate step in the fabrication of structural ceramics and refractory composites. The overall shapes of structural components are created at this stage, followed by sintering or hot pressing to form densified near net or final shapes. This is a crucial stage during which flaws can be introduced or substandard materials can be accidentally produced. Binder mal-distribution, density fluctuations, porosity, inclusions, and similar volume discrepancies must be assessed and controlled [28]. Also, green compact dimensions and surface roughness are factors that can attest to the goodness or poorness of processing conditions and controls.

X-ray absorption and nuclear magnetic resonance are sensitive to binder distribution anomalies in green compacts [29]. Laboratory studies have shown that porosity and other volume flaws in these compacts can be detected using film and digital radiography, computed tomography, and nuclear magnetic resonance methods. Green state compacts and bisques are quite fragile so that inspections are best accomplished with techniques that avoid forceful physical contact.

Metrology methods using noncontacting laser optical techniques provide fast and sensitive means for monitoring and verifying correct green compact shapes and dimensions. Ultrasonics usually requires contact but can be accomplished without damage to green state forms under certain conditions, e. g., by use of air coupled probes. Light scattering and laser optical techniques lend themselves to surface roughness measurements for green state and also for fully densified sintered structures. All these nondestructive evaluation methods can provide valuable feedback for perfecting processing parameters and then for monitoring various fabrication steps.

Methods for Densified Materials - Conventional, appropriate, and mandatory techniques for surface-connected flaws are optically aided visual and liquid penetrant inspections [30]. They should be used routinely to screen out articles that are cracked, pitted, marred, spalled, or have poorly finished surfaces. Immersion scan ultrasonics, film radiography, and computed tomography detect subsurface and volume flaws. If the flaws are isolated and fairly large, i.e., of the order of 500 microns or more, then conventional ultrasonic scanning and film radiographic methods are suitable. However, the spatial and image density resolution of these conventional methods becomes taxed in the "grey area" represented by flaws in the 500 to 50 micron size range.

High Resolution Flaw Detection Methods - For discrete flaws below 100 microns in size it is necessary to consider high resolution methods like acoustic microscopy, microfocus radiography, and micro-tomography. Acoustic microscopy and microfocus radiography can detect flaws down to the 20 micron level in monolithic silicon carbide and silicon nitride. These methods are successful and have high resolution and high probability-of-detection only under the most stringent conditions of material thickness, part shape, surface finish, etc. [16,17]. Even under the best conditions and with high resolution methods, some flaws remain very difficult to detect, e.g., tight cracks, mega-grains, and grain clusters having densities or acoustic impedances that match their surroundings.

Recent laboratory results with new high strength monolithic ceramics have shown that many failures are initiated by surface and near-surface defects between 20 and 40 microns in size. Acoustic microscopy affords the potential for detection of flaws of this nature, given the right conditions. Surface preparation by polishing or fine grinding is needed to enhance the detectability even of exposed surface voids on the order of 50 microns diameter and less. Surface roughness affects the signal-to-noise ratio in acoustic microscope images. Moreover, sintered samples with as-fired surfaces show decreased volume flaw detectability with increased thickness and flaw depth. Flaw detectability also depends on the relative coarseness of the material's grain structure. In coarse grained (silicon carbide) samples flaw detectability was found to be significantly less than in (silicon nitride) samples that had a much finer grain structure [31].

Scanning acoustic microscopy can image flaws in monolithic ceramics with a resolution of about 20 microns or better. Scanning acoustic microscopes usually operate at 50 to 200 megahertz and can be focused up to several millimeters into fine grained monolithic ceramics. A scanning acoustic microscope operating at a center frequency of 50 megahertz is readily able to image voids 20 microns in diameter at a depth of 1 millimeter in silicon nitride [32].

Near surface, i. e., subsurface, flaws may require examination by the ultrasonic surface wave method [33]. A focused ultrasonic transducer operating at frequencies

up to 100 megahertz is used to launch and collect Rayleigh waves that can interact with and resolve minute cracks and other defects down to the 10 micron level. The surface wave method overcomes difficulties encountered by the pulse reflection or scanning acoustic microscopy method primarily because the waves travel parallel rather than normal to the surface.

Microfocus radiography provides a high resolution imaging tool with the potential of being readily applied in production as well as laboratory environments. Film and real-time video versions are available for inspecting a variety of test objects for flaws distributed throughout a volume. Recent research has shown the combined spatial and image density resolution of microfocus radiography to be at least twice that of conventional film radiography [34]. Like other projection radiographic methods, microfocus radiography is only suitable for detecting flaws that have three-dimensional extent, e. g., voids, inclusions, as opposed to two-dimensional or planar flaws like cracks.

Computed tomography systems can produce the high resolution images required for characterization of structural ceramics and their composites [35]. Unlike film and projection radiography, computed tomography produces cross-sectional and three-dimensional reconstructions of both discrete and diffuse flaw populations in an examined volume. High speed computed tomographic systems readily provide image resolutions on the order of 250 microns. Using microfocus x-ray sources, advanced tomographic systems are being developed for resolving down to 25 microns [36].

Materials Characterization Methods - Fairly large flaws are frequently encountered in components such as turbine rotors. But, in monolithic ceramics flaws less than 10 microns in size have been routinely found to be fracture origins. Such flaws tend to be quite numerous in fine-grained ceramics and this situation will overburden the capabilities of any high resolution technique. High resolution imaging is inherently time-consuming. It is important to decide whether there is sufficient pay-off to examine each and every cubic millimeter of a monolithic ceramic article for each 10 micron flaw. Of course, there will be critical zones where high resolution examination is justified.

Below the 50 micron level it may be impractical and even unnecessary to image and characterize individual flaws in non-critical zones of monolithic ceramic structures and certainly unnecessary in refractory composite structures. The alternative is to use low resolution methods to characterize the global environment in which flaws reside. This is the primary goal of analytical ultrasonics and macroscopic computed tomography [9,37]. These two technologies can quantitatively characterize and image diffuse flaw populations, dispersed micro-porosity, adverse whisker alignments, sintering anomalies, fiber misalignment, etc.

The term analytical ultrasonics denotes a methodology for quantitative characterization of the microstructure and mechanical properties of engineering materials. Ultrasonic velocity and attenuation are analytical methods for assessing bulk density, grain size, and other extrinsic factors that govern strength and toughness. Models explaining and predicting the empirical correlations found between ultrasound and mechanical properties have been advanced [10,38]. These correlations depend heavily on the experimental conditions and the nature of the material sample, e. g., size, shape. Factors that influence ultrasonic attenuation and velocity measurements include surface finish, pore fraction, pore size and shape, grain size, grain size distribution, texture, and elastic anisotropy. Of course, these same factors also govern mechanical properties, load response, and thermal and mechanical degradation.

Low ultrasonic attenuation is characteristic of nearly fully dense monolithic ceramics with fine microstructures, i.e., samples with a mean grain size of less than ten microns and densities greater than 95 percent of theoretical. For monolithic and toughened ceramics significant attenuation differences are evident only at frequencies greater than approximately 100 megahertz. Fairly high frequencies are needed to correctly assess subtle microstructural aberrations such as excess detrimental granularity and porosity.

Ultrasonic attenuation is influenced by bulk density and the combined effects of pore size and grain size and, therefore, is a sensitive indicator of microstructural variations in structural ceramics when measurements are made at the appropriate frequencies [39]. However, meaningful attenuation measurements require not only fairly smooth surfaces but also constraints on sample size, shape, and thickness. When accurate attenuation measurements are needed, the surface roughness should be minimized [40]. Nevertheless, it is possible to make comparative attenuation measurements on as-fired or unpolished machined specimens provided that the surface roughness is the same on all samples and the signal-to-noise ratio is sufficiently high.

Ultrasonic velocity is a monotonically increasing function of density in porous solids [19]. Variations in pore size and grain size have little effect on this relation. Although poor surface finish and overall sample thickness can reduce accuracy somewhat, velocity measurements are not as vulnerable to surface roughness as are attenuation measurements. Since velocity measurements are not strongly affected by pore or grain size, they are convenient for estimating bulk density of monolithic and toughened ceramics. Experimental results show that velocity measurements can be used to estimate bulk density within approximately one percent. Velocity measurements can be used to screen out low density monolithic ceramic components and refractory composite structures.

Ideally, both attenuation and velocity measurements require essentially flat, parallel opposing surfaces or geometric simplicity. Actual part shapes do not always

permit precision attenuation or velocity measurements. An alternative approach is the ultrasonic backscatter method for ultrasonic determination of porosity, grain, and similar microstructural variables [41,42]. Backscattered, and under some conditions forward scattered, ultrasound radiations can be used to characterize volume properties of parts having complex shapes[43].

The acousto-ultrasonic technique was developed specifically for characterizing defect states and mechanical property variations of composites [44,45]. The acousto-ultrasonic technique has been applied to fiber reinforced composite laminates to detect local and global anomalies such as matrix crazing and porosity, modulus or stiffness variations, interlaminar bond and fiber-matrix bond strength variations, and fatigue and impact damage. Acousto-ultrasonics is similar to coin tap, sonic vibration, and dynamic resonance methods for assessing the overall global condition of fabricated shapes [46,47]. The acousto-ultrasonic technique is a comparative analytical ultrasonic method that does not impose the stringent constraints on material surface conditions required for the attenuation measurements mentioned previously.

Conventional film radiography and projection radiography are important imaging methods for macro-flaw detection, for assessing global density variations, and for locating porosity in monolithic and toughened ceramics. Digital radiography provides an excellent quantitative means for comparing degrees of densification in a volume of material. Computed tomography applied at lower resolutions can produce three-dimensional images of density variations, fiber architecture, dispersed flaw populations, and any global aberrations in refractory composite structures [48].

Auxiliary Methods - The previously mentioned nondestructive evaluation methods are prominent among the ones currently being considered and applied to high temperature materials. This does not preclude various other methods that can be equally viable and appropriate. For example, eddy-current testing has been applied to polymer matrix composites and may prove particularly useful for characterizing intermetallic matrix composites [49]. There are numerous thermal wave techniques that already have been applied to monolithic ceramics and that may readily apply to refractory composites [50]. Electric and magnetic testing, dielectrometry, and microwave techniques have been demonstrated for polymeric composites and should also be considered for monolithic ceramics and refractory composites [51].

Acoustic emission techniques have applications ranging from materials research to component proof testing [52]. In materials research, acoustic emission can be used to monitor fracture processes and to help identify factors that govern or contribute to material failure. Acoustic emission monitoring during proof testing can aid in assessing the infirmity or integrity of high temperature components.

Conclusion

Current activities under the leadership of ASTM committees will help assure that nondestructive evaluation and inspection standards are established for high temperature materials. In some instances it appears that modifications of existing documents will suffice. There are other instances where new inspection methods and associated standards will be required. These depend on the development of sophisticated inspection strategies demanded by advanced ceramic and refractory composite structures. The technological needs are described in this report and suitable approaches are suggested. The major observation is that pivotal roles will be played by advanced techniques for high resolution flaw detection and innovative techniques for nondestructive materials characterization.

Materials characterization and high resolution flaw detection are currently primarily laboratory techniques that require further investigation, development, and adaptation before they can be applied in materials processing, fabrication, and field environments. Practical implementation of these methods in production and field uses awaits the development of suitable calibration standards and standards of practice. Flaw detection techniques for monolithic and toughened ceramics depend on investigations that will establish statistical foundations for probability-of-detection of various types of defects over a range of material and component conditions. Emerging approaches for nondestructive materials characterization of ceramics and refractory composites require thorough investigation and development before they can be relied on to assess initial quality, mechanical properties, diffuse defect states, or thermo-mechanical degradation in high temperature structures. Computerized interpretational procedures using expert systems will undoubtedly be needed to assure unambiguous nondestructive characterizations of specific material properties.

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16. Abstract High temperature materials include monolithic ceramics for automotive gas turbine engines and also metallic/intermetallic and ceramic matrix composites for a range of aerospace applications. These are materials that can withstand extreme operating temperatures that will prevail in advanced high-efficiency gas turbine engines. High temperature engine components are very likely to consist of complex composite structures with three-dimensionally interwoven and various intermixed ceramic fibers. The thermo-mechanical properties of components made of these materials are actually created in-place during processing and fabrication stages. The complex nature of these new materials creates strong incentives for exact standards for unambiguous evaluations of defects and microstructural characteristics. NDE techniques and standards that will ultimately be applicable to production and quality control of high temperature materials and structures are still emerging. The needs range from flaw detection to below 100 micron levels in monolithic ceramics to global imaging of fiber architecture and matrix densification anomalies in composites. The needs are different depending on the processing stage, fabrication method, and nature of the finished product. This report discusses the standards that must be developed in concert with advances in NDE technology, materials processing research, and fabrication development. High temperature materials and structures that fail to meet stringent specifications and standards are unlikely to compete successfully either technologically or in international markets.			
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