

**A NATIONAL MEASUREMENT  
GOOD PRACTICE GUIDE**

**No. 19**

Tests for advanced  
technical ceramic  
materials performance

# **NPL Measurement Good Practice Guide No. 19**

## **Tests for advanced technical ceramic materials performance**

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### **Abstract:**

The purpose of this guide is to give a contemporary account of the status of methods for determining the various performance characteristics of advanced technical ceramics, particularly those for mechanical applications, or where mechanical properties play an important ancillary role. Also covered are density/porosity determination, surface roughness measurement, thermal properties and corrosion testing. The background to the selected standard methods is briefly given, together with information on the relevance of the methods to end uses. Where appropriate, the material classes for which the standardised methods need some modification or adaptation are discussed. Advice is given on typical sets of testing methods for quality assurance, materials selection and design data collection purposes. The Bibliography lists the current status of relevant standards.

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# Tests for advanced technical ceramic materials performance

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## Executive summary

Over the last decade there has been much activity in Europe, USA and Japan, and recently also internationally, to define a suite of standardised procedures for evaluating advanced technical ceramic products and materials. This industry-led activity is driven by the commercial need to have well-designed documented procedures to back up performance claims, and to help the exploitation of new materials or to encourage the use of ceramics in substitution for other materials for existing applications. In many cases there have been extensive research and interlaboratory testing programmes to evaluate some of the testing issues, and to provide data from which levels of confidence in testing can be established. To a large extent, involvement in this drive has been limited to the research and manufacturing communities, but the results have been spectacular in terms of the numbers of procedures available now or to become available in the near future in the ongoing development programmes.

This guide is aimed at those who research, manufacture or procure ceramic components. It provides a current picture of the availability of standardised procedures for testing or making property measurements on advanced technical ceramic products which are intended primarily for mechanical purposes, or for purposes in which mechanical properties are key attributes. It covers tests for quality assurance, materials comparison and design data collection, including:

- density and porosity
- surface roughness
- strength
- elastic properties
- hardness
- fracture toughness
- high temperature mechanical properties (deformation, creep, fatigue)
- thermal properties (thermal expansion, thermal conductivity/diffusivity, specific heat, thermal shock resistance)
- chemical corrosion and oxidation testing

Emphasis is placed on identification of standardised procedures, especially those developed within Europe (CEN), USA (ASTM) and Japan (JIS), as well as new international activities in ISO. Methods are briefly described, and some of the important aspects of the testing and its relevance to material specification and quality assurance are highlighted.

This guide does not cover certain aspects of testing advanced technical ceramics, such as electrical, magnetic and optical properties. Some other aspects are dealt with in other NPL guides and reports, including wear testing (*'Wear testing standards database'*, S. Owen-Jones and M.G. Gee, NPL Report CMMT (A) 89, December 1997), and microstructural evaluation (*'Microstructural measurements on ceramics and hardmetals'*, NPL Measurement Good Practice Guide No. 21).

# 1. Objectives

In recent decades, testing of advanced technical ceramics has been somewhat unstructured, with individual researchers and developers using whatever techniques or facilities that are available or seem appropriate at the time. This led to a very unsatisfactory situation in which it became impossible to compare data from different sources in a meaningful way. Performance was difficult to judge correctly, and excessive and misleading claims were often developed as a result. It became recognised that unification of testing practices was necessary in order to inject realism into understanding of performance, and to give the materials something of a pedigree. Over the past decade there has been much activity internationally concerning the development of standardised procedures for evaluating the properties of advanced technical ceramics (ATCs), and many of these are appearing as formalised standards, published by CEN (Comité Européene de Normalisation), ASTM (American Society for Testing and Materials), JIS (Japan Industrial Standards) and very soon in ISO (International Standards Organisation). The main driving force for this activity has been to put in place for the first time a series of unified testing methods which are wholly appropriate for advanced technical ceramics, and which will yield consistent and verifiable results for a number of purposes:

- Materials formulation and testing
- Comparative materials evaluation
- Testing of materials to specifications
- Compilation of databases.

Behind this international effort there has been much research to understand issues in testing, and method verification exercises have also been conducted, such as through the VAMAS (Versailles Agreement on Advanced Materials and Standards) programme in Technical Working Area 3.

The purpose of the present guide is to give a contemporary account of the status of methods for determining the various performance characteristics of advanced technical ceramics, particularly those for mechanical applications. The background to the selected standard methods is briefly given together with information on the relevance of the methods to end uses, and the materials classes for which the standardised methods need some modification.

The guide covers:

- **physical properties**, including density, porosity and surface roughness
- **room-temperature mechanical properties**, including strength, elastic moduli, hardness and toughness
- **high-temperature mechanical properties**, including strength, elastic moduli, thermal shock resistance, and anelastic properties such as deformation and creep

- **thermal properties**, including thermal expansion, thermal conductivity, thermal diffusivity and specific heat
- **chemical resistance properties**, including corrosive attack and oxidation/reduction characteristics

The guide does not cover determination of microstructural parameters or wear characteristics, which are dealt with in other guides in this series.

## 2. Physical properties

### 2.1 Density and porosity

#### 2.1.1 Introduction

Unlike many other materials, density measurements assume high importance for the majority of ceramic materials as a measure of the efficacy of the powder route processing employed. Densities are seldom close to the theoretical value because the materials usually contain residual porosity, which may either consist of entirely closed holes within the body, or may be interconnected, or partly interconnected with the external environment. In many cases the theoretical value is not known with accuracy because of uncertainties in knowing the precise mixture of phases present or their individual densities.

Measurement of density thus reveals the consistency with which the processing has been made, and the closeness of the articles' densities to a specified level commensurate with achieving minimum mechanical characteristics which are sensitive to the level of porosity. For example, properties such as elastic moduli and strength are strong functions of porosity. Measurement of porosity, particularly open porosity, reveals the likely level of liquid absorption, which can affect leak-tightness, pick-up of dirt and corrosion resistance. It should of course be noted that many applications for ceramic materials in filtration and catalysis arise because porosity can be developed and controlled in final products, in which case not only total open porosity but also pore channel diameter and permeability may be critical measures of performance.

#### 2.1.2 Measuring density and open porosity

The most common technique is the Archimedes displacement method. The test sample is dried, allowed to cool, and weighed in air. It is then immersed in water (or an alternative organic liquid if water is chemically unacceptable) and weighed whilst suspended. It is then removed, excess surface moisture is dabbed off, and re-weighed in air. These three weighings, corrected for the mass of suspensions and the density of the immersion liquid at the measurement temperature, are used to derive the **bulk density** (the mass per unit bulk volume including porosity), the **apparent solid density** (the mass per unit volume excluding open pores) and the **apparent or open porosity** (the fraction of the bulk volume occupied by

accessible pores). Details of the immersion technique and some useful points of test practice can be found in EN 623-2 (EN = Euronorme).

The technique has limited accuracy for open porosity levels if these are less than about 2% by volume. The limitation is the accuracy of weighing the dabbed but wet test-piece. Low levels of porosity are better checked using dye penetration and fracture tests (see EN 623-1).

The level of open porosity detected may be influenced by the manner in which the liquid gains access to the open pores. Simple soaking for a limited period may not be sufficient to penetrate the finest pores and for trapped air to be removed. Evacuation and re-pressurisation under liquid is a favoured method for improving penetration. Alternatively, a raised pressure can be used. A method for doing this is shown in IEC 60672-2.

### 2.1.3 Alternative methods of measuring density

**Bulk density** can be determined from mass and dimensions provided that the shape is regular such that its volume can be calculated. The technique works well for test-pieces with masses of more than a few grams provided that dimensional regularity is better than about 0.01 mm. This generally means it can be used on machined parts, but not unmachined, as-fired parts with distortions which are larger than this level. The method has been incorporated within EN 623-2.

**Apparent solid density** can be measured using a pycnometer. The volume of a glass tube is computed from the volume of a suitable gas required to fill it. The tube is then filled with a known mass of ceramic components or fragments, and the volume of gas required to fill it again determined. The apparent solid density of the test material can thus be calculated. A standard has been developed for granular materials such as soils (ASTM D5550) which can also be used for highly porous ceramic materials such as catalyst supports and filters.

## 2.2 Surface roughness

### 2.2.1 Introduction

Surface roughness is important in many applications of ceramic materials. Because ceramics are hard and abrasive to contacting materials, the quality of surfaces can be crucial in determining the pick-up of foreign material (*e.g.* metal marking). In addition, because ceramics are hard, they are used for seals and pistons which need not only defined roughness but also defined surface liquid retention. Roughness thus commonly appears in specifications for ceramic products, and is routinely checked by both manufacturer and customer as part of quality assurance procedures.

The usual measures of surface roughness, such as *Ra*, *Rt*, *Rz*, *etc.*, have been devised for describing the output of roughness sensing devices in which a diamond stylus is drawn across

the surface. The methods have been well established for and primarily used on metallic materials. Standards have been prepared for machine behaviour/performance (BS ISO 3274:1996), parameter definition (BS ISO 4287:1997), metrological characterisation of profile filters (ISO 11562:1996), computational procedures (BS ISO 4288:1996), and machine calibration using known artefacts (ISO 5436-1:2000 (~ BS 6393:1987)). No standards exist for alternative instruments which operate using different principles, including scanning laser profilometry and various types of scanning probe microscopies.

The application of stylus techniques to the short-range roughness of machined or abraded ceramic surfaces can be seriously questioned. Stylus tips are designed to follow gently undulating roughness, not the steep-sided short-range roughness typically encountered with ceramic materials. Except on very smooth surfaces, the recorded profile is thus only a measure of the interaction of a radiused probe with the surface, and not a true physical measurement.

Developments in CEN have focused on creating a single procedure for ATCs (EN 623-4) in which the machine parameters are much more closely restricted than for general engineering applications. In a recent round-robin study it has been found that the restrictions are not adequate, and that further restrictions need to be made in order to ensure that the results obtained on different instruments at different sites can be brought closer together.

### 2.2.2 Measuring surface roughness

The test-piece needs to be rigidly mounted on the test machine platen, and the stylus positioned to make the profile recording. The surface must be reasonably flat and near parallel to the machine measurement plane, although some out-of-flatness can be tolerated. The ability to set the machine parameters to specified values given in the standard will depend on the machine employed. At least 10 profiles of length of typically 5 mm each are then recorded to yield at least 4 mm of **evaluation length** in each profile. In most modern machines the profiles are digitally processed to yield the relevant parameters.

The basic steps in data processing are:

- Removal of tilt: the general trend of the profile is computed and used to ‘level-off’ the traces;
- Application of a long-range filter (low frequency cut-off): this removes waviness of the surface which has a wavelength greater than the so-called **cut-off length**, normally set to 0.8 mm;
- Application of a short range filter (high-frequency cut-off): this removes excessive noise from the trace;
- Computation of relevant parameters: the **evaluation length** is normally divided into five **sampling lengths** each 0.8 mm in length. Parameters defined over the sampling length, such as *Ra* and *Rz*, are then computed for each sampling length of each trace and the

averages are determined. Parameters defined over the evaluation length, *e.g.*  $R_t$ , are also computed.

### 2.2.3 Problems with stylus measurements

Differences in result between different machines can be caused by several factors:

- Different stylus tip radii: styli wear or can become chipped, and even new styli may differ from the specified values by a significant amount. The requirement in EN 623-4 is to use a 2.5  $\mu\text{m}$  tip radius. Running the stylus tip over an upright new razor blade tip has been found to give quite good checks on the actual radius.
- Different levels of stylus force: although there is a narrow range specified in ISO 3274, and a specific value of 0.75 mN is cited at the mid height of its range, this is difficult to check without specialised equipment.
- Different dynamic response of the stylus: the mechanical response of the stylus (inertia, damping) will affect its ability to follow the surface correctly without bouncing. This factor seems unquantified at present, but could be important for applications involving ATC surfaces.
- Different traverse speeds: it seems that few machines have the option to change speeds, but no checks are standard. The ‘standard’ speed of 0.5 mm/s may in any case be too fast for reliable profile recording on ATC surfaces.
- Different set filter levels: while the low frequency cut-off is recommended in the standards, high frequency filter settings are not, but perhaps should be.

If after checking that measurement settings on respective machines are selected equivalently results between laboratories still do not agree acceptably, it is recommended that:

- Checks are made on the stylus tip radius, or new styli are used;
- Reference samples are exchanged and results are compared;
- The level of disagreement is agreed.

### 2.2.4 Alternative surface roughness measurement techniques

As mentioned earlier, laser scanning profilometry offers an alternative non-contact method for determining both two-dimensional and three-dimensional roughness parameters. However, its application on ceramic materials has shown it to produce results which are very significantly different from those of stylus contact methods. The origin of this difference appears to be caused by the greater ability of the light beam to penetrate into the depths of the true profile, even inside the material, because on the scale of a few micrometres most ceramics are transparent or translucent. Computed 2-D roughnesses are generally significantly greater than those from stylus instruments.

Scanning probe microscopies have developed in recent years. The vertical resolution of such instruments is remarkable, but they are still limited by having a finite effective probe tip

radius. It remains unclear whether profiles determined using such instruments are closer to the true roughness than either stylus or scanning laser instruments. In any event, results from any form of probe instrument, including optical ones, are likely to be different from those from conventional stylus measurements.

### 3. Room-temperature mechanical properties

#### 3.1 Elastic properties

##### 3.1.1 Introduction

The majority of ceramic materials show linear elastic behaviour to fracture, but the strain at failure is typically rather small, less than 0.1%. This means that elastic properties are well defined, but difficult to measure by conventional metallurgical methods, i.e. by strain measurement during a tensile test.

Elastic properties for many products are isotropic because powder route methods lead to random grain orientations. However, certain processing routes produce elastically anisotropic materials, including extrusion, slip or tape casting, electrophoretic deposition and hot pressing. In these methods, crystallographic alignments tend to occur as a result of particle shape or behaviour, and the resulting elastic properties then have an orientation dependence which generally aligns with the direction of processing. Data for ceramic materials is typically cited assuming elastic isotropy.

Some ceramics do not show linear elastic behaviour, because they typically contain microcracks which develop progressively with increasing stress, the material losing stiffness. In such cases, elastic properties can be measured only using low stress methods (see below).

##### 3.1.2 Test methods

There is a variety of methods in common usage which are summarised in Table 1. Some of these are well-established, and others are of more recent provenance.

**Table 1 - Measurement methods for elastic properties**

| Method                    | Test-pieces     | Measures    | Standards                                   |
|---------------------------|-----------------|-------------|---------------------------------------------|
| Beam flexure              | Long thin beams | $E$         | IEC 672-2, EN 843-2                         |
| Resonance                 | Beam(s)         | $E, G, \nu$ | EN 843-2, ASTM C623, C848, C1198, JIS R1602 |
| Impact excitation         | Beams           | $E$         | EN 843-2, ASTM C1259                        |
|                           | Discs           | $\nu, E, G$ | ASTM C1259 (Annex)                          |
| Ultrasonic time-of-flight | Rods            | $E$         | EN 843-2, ASTM 494, E494                    |
|                           | Blocks          | $E, G, \nu$ | EN 843-2                                    |

The **beam flexure method** is a quasistatic, relatively high-stress method for determining only Young's modulus,  $E$ , conducted on thin beam test-pieces, most commonly using a universal mechanical testing machine and a suitable flexure jig. Either the displacement of the bent specimen is recorded (three or four point bending), or the faces of the test-piece are strain-gauged (four-point bending only).

If test machine displacement is used as a measure of the flexural displacement, it must be corrected for machine compliance. If transducers are used in a differential mode for determining displacements at two points, care needs to be taken concerning the metrology of contact positions on the test-piece relative to the loading and support positions, and on the transducer calibrations. Long, thin test-pieces give more displacement for a given level of stress in the test-piece. In recent interlaboratory research using standard flexural strength test-pieces 3 x 4 x 50 mm loaded in the 3 mm direction, it was found that this geometry did not give particularly reproducible results because the displacements are very small. Longer and thinner test-pieces are to be preferred.

With strain gauges, the thickness of the test-pieces is of less importance since the surface strain is directly measured. However, if transverse as well as longitudinal strain gauges are used, Poisson's ratio,  $\nu$ , can also be determined.

In either case, it is advisable to make a series of loading and unloading cycles to ensure repeatable behaviour, and to evaluate the results after the system has mechanically settled. With standard flexural strength test beams, the level of accuracy is not likely to be better than 5%, but with long thin beams (*e.g.* 1 x 10 x 100 mm) of accurate geometry and good surface finish, the results can be accurate to better than 1%.

The **resonance** and **impact excitation** methods use the same mathematical basis for the fundamental frequency of vibration of a regular beam shape, and are used to determine dynamic, low-stress moduli. In the resonance method, the beam is continuously vibrated using a driving source, the frequency of which is scanned. The intensity of the vibration is sensed by a separate sensing device, and the peaks correspond to various fundamental and harmonic modes of vibration. The standard methods recommend separate determinations of flexural and torsional modes, ideally with test-pieces designed to optimise responses of those modes. In the impact excitation method, the test-piece is struck rather than continuously vibrated, and the set of natural frequencies is detected by a piezoelectric transducer or microphone. Using an instrument such as the 'Grindosonic'<sup>1</sup> or its equivalents, or a Fourier transform frequency analyser, the fundamental modes, corresponding with the longest-lived natural frequencies, can be determined. With appropriate equipment, these methods offer the simplest and quickest means of determining moduli, which are typically accurate to better than 1%.

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<sup>1</sup> Lemmens Elektronika BV, Leuven, Belgium

An extension of the impact excitation method has been approved as an annex to ASTM C1259 (and has been included in EN 843-2), and involves the use of disc test-pieces. A disc struck at its centre will vibrate in a ‘diaphragm’ mode with circular symmetry and a ring node at about 0.6 of the disc radius. If the disc is supported on four points on the nodal circle and is struck on the nodal circle at a position between the support points, a lower frequency, ‘saddle’ mode of vibration is preferentially induced. The ratio of these two frequencies, coupled with the thickness to diameter ratio of the disc gives Poisson’s ratio,  $\nu$ , from a look-up table. The two frequencies individually together with disc geometry and  $\nu$  give values of Young’s modulus,  $E$ . Shear modulus,  $G$ , is computed from  $E$  and  $\nu$ . Experimental work has shown that results are most reliably determined using a frequency analyser, that the smallest discs that can be measured are about 25 mm diameter (otherwise frequencies are too high and too short-lived for conventional instruments to record), and that the discs must be round and flat to close tolerances.

In the **ultrasonic time-of-flight** method a stress pulse is applied to one surface of a test-piece, and is detected at the opposite surface (two-transducer, pulse transmission method) or is reflected back from the opposite surface and detected at the emitting point (single transducer/gated signal, pulse-echo method). Emitting/detecting transducers can operate either in the longitudinal mode (P-waves) or transverse mode (S-waves).  $E$ ,  $G$ , and subsequently  $\nu$  can be computed from the two wave velocities. By making the measurement in a set of defined orientations on the test-piece, anisotropic elastic behaviour can be readily determined.

The methods involving the use of high frequencies work well on materials which are dense and ‘ring’ when struck. With increasing porosity or tendency to microcrack, ringing responses become less well-defined, resonance peaks become broader, and damping of vibration is quicker. Under these conditions, results become less reliable, or even impossible to obtain. There is insufficient information available at the present time to provide clear guidance as to what can be tested and what cannot by a particular method, but difficulties may be encountered when total porosity levels are greater than about 20-30% depending on the size of distribution of the porosity. In principle the quasistatic methods would still work on such materials.

### 3.1.3 Factors influencing elastic moduli data

For a given material type, the principal influence on elastic moduli is the level of porosity. Increasing porosity markedly reduces Young’s and shear moduli, typically according to a relationship of the form:

$$E, G = E_0, G_0 \exp\{-aV\}$$

where the subscript 0 refers to the fully dense form,  $a$  is a constant and  $V$  is the volume fraction of porosity. A 5% level of closed porosity typically leads to a 10% drop in modulus.

Another important factor is the phase assemblage. Continuous glassy phases generally have low elastic moduli, while many crystalline phases of interest in ceramics have rather higher moduli. To a first approximation, a volumetric rule of mixtures can be used for multiphase ceramics:

$$E = E_1V_1 + E_2V_2 + E_3V_3 + \dots$$

As mentioned earlier, internal microcracking causes losses in strength and stiffness. Microcracking is caused by anisotropic internal stresses of thermal expansion mismatch origin which are sufficiently high that grain boundaries spontaneously crack. Examples of materials in which the effect is marked are magnesium and aluminium titanates. Microcracking, and hence loss of modulus, is reduced by reducing the grain size.

## 3.2 Strength

### 3.2.1 Brittle behaviour

The strength of brittle materials such as ceramics is determined by the resistance to propagation of cracks from microstructural imperfections or external sources of microcrack damage. The smaller the imperfections or microcracks, the stronger is the material. The greater the microscale toughness, the greater is the strength. The strength of a component is thus determined by the perfection of processing. Because the imperfections controlling strength are typically of the order 10 µm to 100 µm in size, it is virtually impossible to detect them non-destructively or to eliminate them in conventional processing, and this leads to a variation in strength within a group of nominally identical components. The largest imperfection with the highest stress concentration on it ultimately dictates whether fracture occurs. Thus the strength of any individual component is indeterminate until tested to failure. Further, it follows that the larger the item being tested, the lower the strength is likely to be on average because the largest imperfection is likely to be bigger.

Caution therefore has to be exercised over the definition and interpretation of 'strength'. The objectives of undertaking strength tests on test-pieces of geometry, processing route and surface finishing not directly related to the final components being manufactured and used has to be seriously questioned. It is considered that tests on specially prepared test-pieces have the following particular uses:

- To compare potential strength behaviours of differing materials prepared in the same way;
- To evaluate the effect of processing route (or batch-to-batch consistency) of a given material type;
- To evaluate the influence of surface machining procedures on likely subsequent mechanical behaviour;
- To evaluate the effects of stressing rate (*e.g.* ENV 843-3), time under stress (known as 'static fatigue' or 'delayed failure'), or environment on strength properties.

Although much has been written in the technical literature concerning using test-piece data to predict statistically the strength behaviour of more complex shaped components, such extrapolations are invalidated unless all factors associated with the distributions of flaw sizes are equivalent for the test-pieces and the components, and often this is impossible to achieve.

### 3.2.2 Strength test methods

Strength testing can be performed in a wide variety of geometries, the common feature being that they are all readily analysable in terms of simple equations. The most widely used is the flexural strength test on a beam test-piece (see *NPL Measurement Good Practice Guide no. 7*), but other geometries may be easier to use depending on the availability of test-pieces. For example, if the material is produced as thin discs, it may be easier to perform a disc test piece (see *NPL Measurement Good Practice Guide no.11*) than to prepare beam test-pieces from the discs and to make a beam test. Similarly tubular shapes are most readily tested using O-ring or C-ring tests. Table 1 lists the possibilities and the availability of standardised procedures.

**Table 2 - Standardised uniaxial/biaxial strength tests for ceramics**

| Test                                          | Test-piece                       | Standards                                                                                                |
|-----------------------------------------------|----------------------------------|----------------------------------------------------------------------------------------------------------|
| Beam flexure (three-point, four-point)        | Rectangular section beam         | IEC 672-2, EN 843-1, ASTM C1161, JIS R-1601, ISO 14704                                                   |
|                                               | Round or flattened round section | IEC 672-2                                                                                                |
| Disc flexure (biaxial)                        | Thin disc                        | ISO 6474 (ring-on-ring loading)<br>ASTM F394 (punch/3-ball loading)<br>ASTM C1499 (ring-on-ring loading) |
| Disc diametral compression ('Brazilian disc') | Thick disc, small diameter       | -                                                                                                        |
| Tension                                       | Rod with gaugelength             | ASTM C1273, JIS R-1606, ISO 15490                                                                        |
| O-ring diametral compression                  | Ring cut from tube               | -                                                                                                        |
| C-ring diametral tension or compression       | Slotted ring cut from tube       | ASTM C1323, underdevelopment in CEN                                                                      |
| Tube/ring pressurisation                      | Tube or ring                     | ASTM draft                                                                                               |
| Compression                                   | Cylindrical with similar anvils  | ASTM draft, JIS R1608                                                                                    |

The key issues to be addressed in undertaking tests are as follows:

- Select a method which is most appropriate for the test material available and capable of generating the required information;
- Prepare test-pieces with due attention to the geometrical issues for each method (shape, flatness/parallelism of surfaces, cylindricity, edge chamfers) using appropriate machining procedures; at least 10 test-pieces should be used to obtain a mean strength;
- Ensure that surface finishes applied are consistent, appropriate and documented as far possible;
- Ensure that the test jigging employed operates in a manner appropriate to the test such that a close approach to the assumed stress distribution for the analysis is achieved; features such as appropriate local distribution of applied forces, self-alignment, elimination of frictional constraints are very important in achieving consistent testing.

Strength testing can be expensive in terms of test-piece preparation, and cost may predicate the choice of approach. For example, uniaxial tensile testing is seldom performed routinely because of the cost of carefully machined test-pieces and self-aligning jigging, as well as the time involved in setting up the tests and checking alignment.

### 3.2.3 Consistency of strength testing

It is important to distinguish between the natural spread of strength results between test-pieces, which is typically  $\pm 10\%$ , often greater, and the accuracy with which the test can be performed. With attention to experimental detail of the test arrangement, it should be possible to reduce known sources of error to less than  $\pm 2\%$  in total in the case of the beam flexure test. With the other tests, uncertainties may be rather higher. In tensile testing the precision of alignment in reducing the bending contribution is the key factor. In biaxial disc flexure testing, circular symmetry of loading is crucial, and the use of compliant layers may not be adequate to ensure this. In diametral disc compression, compliance at load contact and cylindrical surface/platen alignment are crucial. In O-ring and C-ring testing, test-piece symmetry and surface/platen alignment are crucial.

For determining a mean strength to a confidence level of about 5%, typically 10 valid test results are normally required. For characterising the distribution of strengths, for any sensible analysis, results from 25 to 30 test-pieces are required to have any reasonable level of confidence in the resulting parameters describing the distribution. The method of analysis favoured in standards is the Weibull two-parameter method fitted using the maximum likelihood method, and full details will be found in the texts of standards (ISO 20501, EN 843-5, ASTM C1239, JIS R-1625). The results from such an analysis should be computed with the appropriate confidence intervals on the computed parameters.

Increasingly the merits of undertaking post-test fractography on fractured test-pieces are being emphasised. Fractography can assist in the identification of the position and nature of fracture origins as a means not only of explaining unusually weak test-pieces and hence guiding improved materials processing, but also of checking that the test method is operating effectively. See *NPL Measurement Good Practice Guide no. 15*, EN 843-5 or ASTM C1322 for further information on techniques of examination and appearances of fracture origins.

### 3.3 Fracture toughness testing

#### 3.3.1 Introduction, methods and definitions

Although ceramics are brittle there are significant differences in toughness between the different types which affect performance, particularly abrasive wear resistance, impact resistance and thermal shock resistance, and thus may require characterisation. However, undertaking such tests is not a trivial matter because obtaining correct and reproducible results depends very much on the use of appropriate techniques, as well as a recognition that different techniques may produce genuinely different results.

For metallic materials fracture toughness is conventionally determined by methods well documented in ASTM E399 and its derivatives. Many of these methods need adaptation for use with ATCs because test-pieces tend to be impractically large, or pre-cracking more difficult than in metallic materials. Over the last decade there have been useful developments in methods of testing specifically for ATCs, as well as a number of round-robins to evaluate the reproducibility of the methods between laboratories. Some standards are now appearing dealing with specific favoured methods, while other methods have been deemed inappropriate because of uncertainties in calibrations or the meaning of results.

Toughness is essentially defined as the resistance to propagation of a pre-existing crack of known dimensions, and is nowadays usually expressed in terms of a critical crack tip stress intensity factor,  $K_{Ic}$ :

$$K_{Ic} = Yc^{1/2}\sigma_c$$

where  $Y$  is a crack geometry parameter,  $c$  is the crack length, and  $\sigma_c$  is the applied stress normal to the crack plane at fracture for the so-called ‘opening’ mode (mode I). Most fine grained materials have clearly defined values of  $K_{Ic}$ , but with increasing grain size, or with deliberately engineered microstructures to improve toughness, the crack faces do not completely separate as the crack front propagates. The remaining ligaments exert an additional drag across the crack faces impeding their separation, and giving rise to increasing crack propagation resistance with increasing crack length, or a so-called ‘R-curve’. Because different fracture toughness test methods employ different pre-cursor crack lengths, tests on R-curve exhibiting materials will give method-dependent results, which can be confusing and can lead to overestimates of toughness for small cracks. These effects appear particularly in

materials with a grain size greater than about 8  $\mu\text{m}$ , and with whisker or platelet reinforcement.

Recent standardisation moves are designed to produce minimum toughness values for short, well-defined cracks, and modifications to these methods are needed if R-curve behaviour is to be determined.

### 3.3.2 Test methods

There is a wide variety of methods in the technical literature, some of which produce good results, while others are strictly for comparative purposes only. Table 4 (extracted primarily from an introductory guide published in ENV 14425-1) lists these methods and provides comments on their suitability, and figure 1 shows them schematically. Many of them are based on flexural strength test-pieces, while others can with advantage use larger test-pieces, especially if the material has a moderate grain size or texture. In terms of preferred methods, the following are recommended:

|                    |                                                      |
|--------------------|------------------------------------------------------|
| For short cracks:  | single-edge vee-notch beam, surface crack in flexure |
| For medium cracks: | single-edge pre-cracked beam, chevron notched beam   |
| For long cracks:   | double cantilever beam                               |

Tests which are not recommended for reference data determination, and are suitable only for comparative use are those involving assumptions concerning the stress fields around indentations.

Test-piece preparation follows normal practices for strength test-pieces with appropriate attention to dimensional tolerances. The key step is the introduction of the precursor crack or notch, which is different for each method. A summary of pre-cracking methods can be found in *NPL Measurement Note CMMT (MN) 035*. Some important points are as follows:

**Single-edge pre-cracked beam:** a bridge compression jig is required. The test-piece with starter indentations is laid on an anvil with the indentations over a gap. A second anvil is placed on top of the test-piece and a force is applied to ‘pop’ a planar crack in from the indentation. This operation requires good compression jig design, careful alignment, and skilled operation to give successful pre-cracks with good geometry.

**Chevron notch beam:** two narrow co-planar saw cuts must be made to form the chevron notch. These cuts must be accurately positioned so that a symmetrical planar crack can be produced when it propagates from the sharp tip of the notch. Ideally, the roots of the cuts should be as sharp as possible so that the crack can start easily from the notch tip. In some cases this does not occur in a stable manner, in which case the test is deemed invalid. The crack should be generated smoothly, or ‘pop-in’ a short distance and then propagate smoothly for a valid test according to the method of computation of toughness.

**Table 4 - Fracture toughness test method summary**

| Method/Fig. 1                                   | Description                                                                                                                                             | Calibrations                                                                                                                                              | Uncertainties or difficulties                                                                                                                                                                                   | Standards (see Note)                       |
|-------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------|
| Single-edge pre-cracked beam (SEPB)<br>Fig 1(a) | Flexural beam test into the tensile side of which a short straight sharp crack has been introduced                                                      | Accurate calibrations available for wide range of pre-crack lengths; toughness at a defined crack length                                                  | Pre-cracking requires some skill to obtain straight-fronted cracks. Results are influenced by rising crack resistance behaviour                                                                                 | ASTM C1421<br>JIS R1607-97<br>EN ISO 15732 |
| Chevron notched beam (CNB)<br>Fig 1(b)          | Flexural beam test with two coplanar angled notches leaving a sharp-tipped triangular shaped region to fracture                                         | Accurate calibrations                                                                                                                                     | Toughness at an ill-defined crack length; crack initiation difficult in some materials; result influenced by rising crack resistance behaviour                                                                  | ENV 14425-3<br>ASTM C1421                  |
| Surface crack in flexure (SCF)<br>Fig. 1(c)     | Flexural beam test in which a small semi-circular flaw has been introduced by indentation on the tensile side, and with the indentation damage removed. | Accurate calibrations, assuming the pre-crack shape approximates to an ellipse after removal of surface damaged region.                                   | Requires observation and measurement of pre-crack dimensions, which may not always be clearly visible; limited to materials in which indentation produces good quality cracks. Result typical for small cracks. | ASTM C1422<br>EN ISO 18756                 |
| Single-edge Vee-notch beam (SEVNB)<br>Fig. 1(d) | Flexural beam test in which a narrow notch made on the tensile side has its tip sharpened by honing with diamond paste.                                 | Accurate calibrations assuming a sharp crack (same as SEPB).                                                                                              | Assumes that if the tip radius of the sharp notch is of the order the grain size the notch is equivalent to a sharp crack and the SEPB calibration can be used.                                                 | CEN TS 14425-5<br>ISO DIS 23146            |
| Indentation fracture (IF)<br>Fig. 1(e)          | A test in which the length of cracks emanating from the corners of a Vickers hardness indentation is measured                                           | Poor calibrations owing to uncertainties in stress fields developed by indentation methods of cracking. Wide variety of different calibrations available. | Effective only in materials which do not chip or flake when indented. Inappropriate for tough materials. Result typical for small cracks.                                                                       | JIS R1607-95                               |
| Indentation strength (IS)<br>Fig. 1(f)          | A flexural test on a beam into which has been placed an indentation on the tensile side.                                                                | Poor calibrations owing to uncertainties in residual stress fields developed by indentation which are not removed.                                        | Reproducible test if well-defined cracks are developed by indentation. Result typical for small cracks, but may depend on the indentation force.                                                                | -                                          |
| Double cantilever beam<br>Fig 1(g)              | A plate test in which a through crack in one edge is pulled open                                                                                        | Good calibration                                                                                                                                          | Useful for R-curve and slow crack growth behaviour. Best pulled horizontally to avoid gravity effects. May need side grooves to control crack path                                                              | -                                          |
| Double torsion<br>Fig.1(h)                      | A plate test in which a through crack in one edge is extended by oppositely twisting the two halves of the plate.                                       | Calibration unclear owing to possible mixed mode effects and the crack front not being straight                                                           | Useful for slow crack growth behaviour. Crack front not straight, and obtaining the effective length may require a compliance calibration                                                                       | -                                          |

Key: (CEN TS (= ENV) = provisional standard number not yet allocated, DIS = draft international standard (ISO))

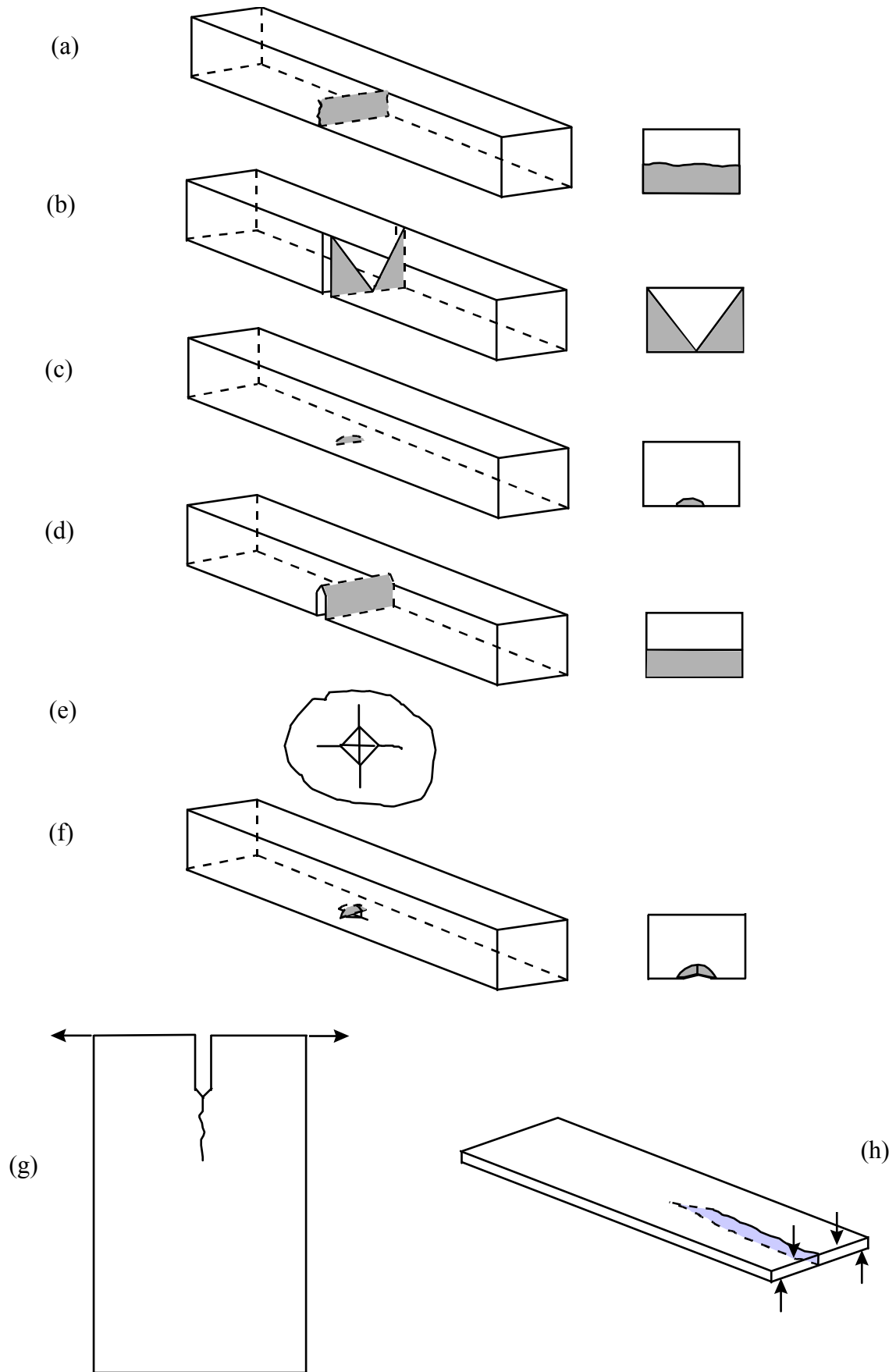


Figure 1 Schematic representation of the various fracture toughness test methods in Table 1.

**Surface flaw in flexure:** an indentation is placed in a beam test-piece, and the half-penny crack produced is used as the starter crack after removal of indentation damage by grinding off the immediate surface of the test-piece. This method is quite reliable, but requires the measurement of the starter crack after fracture, which in turn requires the operator to have fractographic skills. The technique does not work well on coarse-grained materials in which fracture topography does not allow the pre-crack boundary to be clearly distinguished. It does not work well on materials which do not produce good half-penny shaped cracks, but double-lobed surface (or 'Palmqvist') cracks instead. Tricks have to be performed, such as angling the test-piece under the indenter so that one of the lobes is exaggerated at the expense of the other. This information is included in the standards.

**Single-edge vee-notched beam:** a simple saw-cut (the traditional single-edge notched beam' (SENB)) is considered to be too blunt to give reliable results for many materials. Recent developments and a round robin evaluation have suggested that more reliable and realistic data, in most cases equivalent to sharp crack data, can be obtained if the notch tip is sharpened using a razor blade and diamond paste. This operation can be done by hand, but is somewhat tedious on hard materials. It is more efficient to make a small reciprocating machine to guide the razor blade during this operation. In fact, it is frequently not necessary to use a saw-cut starter notch at all, it having been proved possible to hone the notch entirely using the razor blade. Notch tip radii similar to the material's grain size can be produced, down to about 2  $\mu\text{m}$ . This method has been standardised in CEN and is proceeding through ISO.

**Indentation fracture:** although superficially the simplest method, it requires operator visual perception of crack lengths, and so can be a subjective measurement. There are also considerable uncertainties concerning the calibration equations employed, of which there are many to choose from, all with different underlying assumptions or regions of proposed validity. **It is strongly recommended that this method is not used for definitive data, and used only for comparative purposes.** The technique relies on measuring the lengths of surface cracks, which can be somewhat subjective, after indentation of a polished surface using a Vickers indenter at a suitable force, typically 50 N to 100 N. The technique works best in fine-grained materials, but with increasing grain size, cracks tend to meander, fork, or be poorly defined, with consequent large scatter in results.

**Indentation strength:** this method has the advantage of not requiring a crack length measurement, the crack length being inferred from the indentation force and the strength after indentation. However, it relies on the production of a suitable planar half-penny crack from an indentation, and on various assumptions such as that the theory fully takes into account the plastic behaviour of the material during indentation. Experimental evidence suggests that these assumptions are not always completely valid, and that results can be indentation force dependent. Again, **this method should not be used for obtaining definitive data, only as a comparative method.**

**Double cantilever beam:** This plate method has several variants, and has not been standardised except for metallic test-pieces (ASTM E399). Test-pieces need to be carefully machined, and an appropriate means of pulling them introduced; examples include holes for the attachment of pin yokes, or recesses to permit hooks to be inserted into the crack mouth. In addition, it can be difficult to maintain symmetry during crack propagation, the crack tending to veer to one side as the side arms bend outwards. Guide grooves may be needed to keep the crack straight, but these need to be sharp otherwise the cracks can climb out of the grooves. The method is not widely used because of the large quantity of material that is required for a single determination, but is quite useful for slow crack growth studies.

**Double torsion:** This plate method is simpler to undertake than the double cantilever beam method, and relies on twisting in opposite directions the arms on either side of a starter crack or notch. It has the advantage of a constant stress intensity, independent of crack length, but a disadvantage that the crack front is curved and its length thus not well-defined, and that the crack separation is small, and thus may pose problems for coarser grained materials. It is useful for crack growth studies on plate material, such as substrates or electrolyte materials but is not recommended for definitive fracture toughness determinations.

### 3.3.3 Application of the methods

The suitability of these methods for use on particular types has not been extensively evaluated, but some provisional guidance is included in Table 5 for some of the more commonly available material types.

## 3.4 Hardness

### 3.4.1 Principles

Because many ATCs are hard and have applications which exploit this property, there is a strong use made of hardness testing, even though the normal methods employed for metallic testing are not ideal. Hardness is basically measured using the width (or depth) of an indentation made in the material under a defined force. Recent international developments have concentrated on optimising the conditions under which tests on ATCs are performed, and this optimisation is being fed through into formal standardisation. There are several commonly employed test procedures:

**Vickers method (HV):** A square-based pyramidal indenter is loaded into the material under a defined force, removed, and the diagonal lengths of the residual indentation are measured optically. A test force of typically 9.81 N (1 kgf) is recommended (EN 843-4, ASTM C1326, ISO 14705) because for many materials this represents a compromise between cracking and chipping often experienced at higher forces, and increasing errors of measurement and the onset of indentation size effects at lower forces. However, it is

recognised that both higher and lower forces need to be permitted if appropriate to the material under test.

**Table 5 – Appropriateness of fracture toughness test methods**

| Material type                  |                           | Appropriateness of methods |                |                |                |    |    |     |    |
|--------------------------------|---------------------------|----------------------------|----------------|----------------|----------------|----|----|-----|----|
| Type                           | Grain size, $\mu\text{m}$ | SEPB                       | CNB            | SCF            | SEVNB          | IF | IS | DCB | DT |
| <b>Dense types:</b>            |                           |                            |                |                |                |    |    |     |    |
| Alumina                        | <5                        | ✓✓                         | ✓✓             | ✓✓             | ✓ <sup>3</sup> | ✓  | ✓  | ✓✓  | ✓  |
| Alumina                        | >5                        | ?                          | ✓              | ×              | ✓ <sup>3</sup> | ×  |    | ✓   | ✓  |
| SiC                            | <10                       | ✓✓                         | ✓✓             | ✓✓             | ✓              | ✓  | ✓  | ✓✓  | ✓  |
| Si <sub>3</sub> N <sub>4</sub> | <5                        | ✓✓                         | ✓ <sup>1</sup> | ✓✓             | ✓✓             | ✓  | ✓  | ✓✓  | ✓  |
| ZrO <sub>2</sub> (cubic)       | <50                       | ✓                          | ✓✓             | ?              | ✓✓             | ×  | ×  | ✓✓  | ✓  |
| ZrO <sub>2</sub> (tough PSZ)   | <50                       | ✓✓                         | ✓ <sup>1</sup> | ?              | ✓              | ×  | ×  | ✓✓  | ✓  |
| ZrO <sub>2</sub> (Y-TZP)       | <1                        | ✓✓                         | ✓ <sup>1</sup> | ✓ <sup>2</sup> | ✓ <sup>4</sup> | ×  | ×  | ✓   | ✓  |
| ZrO <sub>2</sub> (Ce-TZP)      |                           | ?                          | ✓              | ×              | ?              | ×  | ×  | ✓   | ?  |
| BN (hex)                       |                           | ×                          | ✓              | ?              | ✓              | ×  | ×  | ✓   | ✓  |
| BN (cubic)                     |                           | ?                          | ✓              | ?              | ✓✓             | ×  | ×  | ✓   | ✓  |
| TiB <sub>2</sub>               | <5                        | ✓✓                         | ✓✓             | ✓✓             | ✓✓             | ✓  | ✓  | ✓✓  | ✓  |
| AlN                            | <5                        | ✓✓                         | ✓✓             | ✓✓             | ✓✓             | ✓  | ✓  | ✓✓  | ✓  |
| Steatite                       |                           | ✓                          | ✓✓             | ?              | ✓✓             | ×  | ×  | ✓✓  | ✓  |
| Cordierite                     |                           | ✓                          | ✓✓             | ?              | ✓✓             | ×  | ×  | ✓✓  | ✓  |
| Porcelains                     | <20                       | ?                          | ✓✓             | ?              | ✓✓             | ×  | ×  | ✓✓  | ✓  |
| Carbon/graphite                | <20                       | ?                          | ✓              | ✓              | ✓✓             | ×  | ×  | ✓✓  | ✓  |
| Glasses                        |                           | ?                          | ✓              | ?              | ✓ <sup>3</sup> | ×  | ×  | ✓✓  | ✓  |
| Glass-ceramics                 | <3                        | ✓✓                         | ✓✓             | ✓✓             | ✓ <sup>3</sup> | ✓  | ✓  | ✓✓  | ✓  |
| Machinable glass-ceramic       | 10-50                     | ?                          | ✓✓             | ×              | ✓              | ×  | ×  | ✓✓  | ✓  |
| <b>Open porous:</b>            |                           |                            |                |                |                |    |    |     |    |
| RBSN                           | <1                        | ✓✓                         | ✓✓             | ✓ <sup>2</sup> | ✓✓             | ×  | ×  | ✓✓  | ✓  |
| SiC                            | 20-200                    | ×                          | ✓              | ×              | ✓              | ×  | ×  | ✓   | ×  |
| Refractory porcelains          | <50                       | ?                          | ✓              | ×              | ✓              | ×  | ×  | ✓   | ?  |

**Key:** ✓✓ = fully appropriate, ✓ = possible, × = unlikely, ? = not known

Notes:

- 1: May not give valid results owing to uncontrolled fracture – pre-cracking of the notch tip may be required.
- 2: May not produce good pre-cracks by indentation. Higher load, or angled indentation may be needed.
- 3: Caution advised: crack may advance significantly from the crack tip due to subcritical crack growth before fracture.
- 4: grain size may be too small for accurate result – toughness overestimated compared with sharp crack value.

**Knoop method (HK):** A rhomboidal based pyramidal indenter is loaded into the material under a defined force, removed, and the long diagonal of the residual indentation is measured optically, as for the Vickers indentation. Assessment of reading errors and scatter has shown that there is practically no advantage gained at a given test force in measuring the longer but shallower indentation than in the HV case. However, there is a distinct advantage in that there tends to be less chipping and cracking, permitting higher forces to be used. This has led to the recent recommendation that a test force of 19.62 N (2 kgf) should be used (ASTM C1327, ISO 14705, EN 843-4).

**Rockwell hardness (HR):** In this test, a ball-ended indenter is loaded onto a surface under a minor load, the axial displacement zeroed, a major load is applied and removed, and the net displacement under the minor load recorded. Hardness is recorded on a scale related to the indentation depth. There are a number of different Rockwell scales, the A-scale (60 kgf total force) is seldom used for ceramic materials because of the risk of gross test-piece fracture, but the superficial (N-scale) method with 45 kgf total force is more commonly employed. It is covered by EN 843-4.

Other methods are coming into vogue, particularly depth-sensing micro- and nano-hardness, but techniques are not standardised, and recent work has shown that in terms of reducing scatter of results there is sensibly no experimental advantage compared with the conventional methods described above. It should also be noted that no two methods produce the same numerical result, in other words, hardness is not strictly a material property (*e.g.* in metals it is often taken as a measure of yield stress), but the response of the material to a given type of indentation.

### 3.4.2 Test procedures

The conventional metallic materials methods are well documented and standardised. The key requirement for ceramic test-pieces for HV and HK testing is the production of a good polished surface finish and a well-supported test-piece. The surface should be perpendicular to the axis of the diamond indenter so that symmetrical indentations are produced. The recent standards include acceptance/rejection criteria for the quality of indentations, so that ones with poor definition of indentation corners or excessive spalling are rejected. Other aspects which are expected to develop in the near future as a result of recent interlaboratory work include the advice to use a high-hardness reference block rather than a conventional metallic block prior to undertaking tests on a ceramic test-piece. This is a considerable value, because it not only ensures that the machine is operating correctly and the indenter tip is undamaged, but also provides a means of checking the visual criteria being used by the operator on materials of similar reflectivity. High hardness reference blocks have been developed. An HK2/HK1 reference material (a HIPed silicon nitride, SRM 2830) and an HV1 reference material (a hipped tungsten carbide, SRM 2831) are available from the National Institute for Standards and Technology, USA.

In the HRN case, surface finish of the test-piece is less important, but flatness and stability more so. Again, high hardness reference blocks (normally metallic) should be employed to ensure that the test machine is functioning correctly.

### 3.4.3 Uses of hardness data

The value of hardness data for ceramics is principally in material selection and quality assurance. Materials can be ranked, or compared with a reference material. Increasing levels of porosity in a given type of material correlate with declining hardness. The different methods have been shown to rank materials in the same order, provided that excessive cracking is not induced. In some types of abrasive wear situation, hardness anticorrelates with abrasive wear rate, although fracture toughness is also involved.

It is generally agreed that conversion between hardness scales should never be attempted for ceramic materials. Vickers and Knoop values at the same force are different, and there are different load dependences of hardness in the microhardness range.

## 3.5 Fatigue testing

### 3.5.1 Fatigue mechanisms

ATCs can suffer from fatigue like other materials, but being essentially elastic solids, the mechanisms tend to be rather different. Under constant stress, a small pre-existing crack may grow naturally, especially if the stress intensity at its tip is close to its critical value. The crack growth rate depends on the material type and the environment at the crack tip. One of the principal rate-determining factors is moisture, which accelerates the process of crack growth in most non-oxide and some non-oxide based materials, especially those containing silica, either as a component of a major crystalline phase or as an impurity phase at grain boundaries. This mechanism is often known as '**static fatigue**' or '**delayed failure**' (the equivalent in metals of 'stress-rupture'). This behaviour is often reflected in a stressing rate or environment dependence of strength in flexural strength tests.

Under cyclic stress, similar effects occur under the tensile part of a loading cycle. It has been shown that for tensile only loading, the effect on crack growth is essentially the integrated static fatigue effect over the stress cycles. If the stress has a compressive part (*e.g.* push-pull or reverse bending), additional crack extension effects can be seen as a result of levering effects on the crack front when subject to compression, thought to come about because crack faces cannot completely close. Lifetimes tend to be shorter than in all-tensile fatigue to the same peak stress level

### 3.5.2 Methods for fatigue testing

Methods for determining the tendency for subcritical crack growth from conventional monotonically loaded flexural tests are given in EN 843-3 (room temperature), ASTM C1368 (room temperature) and ASTM 1465 (elevated temperature). Of particular interest in such tests is the ‘slow crack growth exponent’,  $n$ , which relates crack velocity,  $v$ , to the stress intensity factor,  $K_I$ , at the crack tip:

$$v = AK_I^n$$

where  $A$  is a constant, which can be derived from the slope of a log (stress) vs. log (time to failure) plot.

For ‘static fatigue’ testing, dead-weight, long-term loading of test-pieces in conventional strength testing jigs is feasible, and provides information on time to failure (ASTM C1576). The data from such testing is widely scattered in time for a given stress, and some research has been conducted to attempt to reduce the scatter and obtain a more reliable value for the mechanistic parameters. Pre-cracking the test-piece using techniques similar to the SCF fracture toughness method provides a well-defined starter crack, as opposed to the random sizes of natural imperfections in test-pieces.

For cyclic loading in conventional push-pull testing, the only standard in existence is ASTM C1361, which is a general practice for uniaxial or tension/compression testing. This standard does not fix test-piece sizes or test conditions which must be chosen according to availability of gripping systems, and provides guidance on alignment methods. Obviously, such testing is similar to well-established metals testing practice.

For cyclic fatigue in flexure, tensile-only stress conditions are readily achieved by cycling loading of a test-piece in a simple flexural jig (a standard is being developed in ISO TC206). However, for tension/compression loading it is necessary to subject the test-piece to reverse bending. This is not so easy with conventional four-point bending, and alternative methods can be found in the literature, including cantilever vibration of thin bars, rotating/bending of round test-pieces, and pure bending between contra-rotating pivots.

In all cases, lifetimes are critical dependent on the condition of the surface of the test-piece, particularly any damage put in by machining damage.

## 4. High-temperature mechanical properties

### 4.1 Short-term strength properties

#### 4.1.1 Introduction

Testing the strength of ATCs at raised temperature is particularly important for a range of applications, many of them developmental, where reliability is crucial. In general terms, most

ATCs for mechanical purposes retain strength to a temperature at which one of the phases, usually the amorphous grain boundary phase begins to soften. Thus typically, most glass-phase containing high-alumina materials start to lose short-term strength above about 850-900 °C, and even those with no glass phase are beginning to weaken above 1000 °C. The complex phase transformation behaviour in zirconias, particularly those based on the tetragonal phase, causes strength to decline quite steeply with increasing temperature, and two-thirds of their room temperature strength may be lost by 600 °C. The strength behaviour of non-oxide ceramics depends very much on the effects of oxidation, which can cause scale formation, pitting or surface crazing. Sintered silicon carbides, for example, will retain strength to very high temperatures because they contain no glassy phases, but may lose strength as a result of oxidation, which occurs to a significant extent above about 1000 °C. Silicon nitrides are more prone to oxidation effects, and many of them contain glassy phases. Strength losses occur in open-porous reaction bonded silicon nitride above about 900 °C in air (higher in inert conditions) as a consequence of oxidation, and most dense silicon nitrides with secondary phases start to lose strength above about 1000 °C. Silicon nitrides with carefully formulated compositions containing minimal glassy phase and have excellent resistance to oxidation have been reported to retain room-temperature levels of strength to temperatures above 1400 °C.

Exposure to high temperatures can reduce subsequent room temperature short-term strength by changing the nature of strength limiting surface flaws, even developing new flaw types, such as oxidation pits.

Longer-term properties are also of importance. One of the principal room-temperature attributes of ATCs is dimensional stability, but the softening of secondary phases at elevated temperature means that they can creep and distort, perhaps with limited life under stress.

Considerable effort has been placed internationally in evaluating methods of making high temperature tests on ATCs, the principal problem being the low strain to fracture. However, methods of overcoming testing problems have been developed, and some of these are being standardised. A summary appears in Table 6.

**Table 6 - Methods for high-temperature testing**

| Characteristic        | Method                           | Standards                                  |
|-----------------------|----------------------------------|--------------------------------------------|
| Elastic properties    | Beam resonance                   | EN 820-5, ASTM C1198, JIS R1602            |
|                       | Impact excitation                | EN 820-5, ASTM C1259                       |
| Short-term strength   | Flexural strength (3 or 4-point) | ISO 17565, EN 820-1, ASTM C1211, JIS R1604 |
|                       | Tensile strength                 | ASTM C1366, JIS R1606                      |
| Dimensional stability | ‘Sag’ test                       | EN 820-2, JIS R1612                        |
| Creep/stress rupture  | Flexural creep                   | EN 820-4                                   |
|                       | Tensile creep                    | ASTM C1291, JIS R1631                      |
| Fatigue               | Flexural                         | none                                       |
|                       | Tension/compression              | none                                       |

## 4.2 Short-term strength properties

### 4.2.1 Elastic properties

Measuring elastic properties at elevated temperature is more difficult than at room temperature because means have to be found to remotely sense deformations. Of the four methods readily usable at room temperature, only two are simple to extend to elevated temperatures, the resonance and impact excitation methods. The other two, beam flexure and ultrasonic velocity measurement are more problematical.

In the case of **resonance**, the simplest method of extending its use to high temperatures is to use a carbon fibre suspension system, although this does require that the tests are performed in vacuum or inert gas. Loops of carbon fibre are suspended from driving and detecting transducers, and the test-piece is suspended with the loops positioned just outside the flexural node positions. The test procedure is otherwise the same as for room temperature testing.

In the case of **impact excitation**, an arrangement has to be devised in which the test-piece situated inside a furnace enclosure is impacted, typically either from below with a small spring-loaded hammer, or from above using small ceramic balls aimed at the test-piece. Detecting the natural frequencies can be done either using a microphone with a ceramic tube as a sound waveguide through the furnace wall, or by using a long thin ceramic rod passing freely through the furnace wall and connected to a piezoelectric transducer. These arrangements clearly work best with an air atmosphere; inert atmosphere use takes more ingenuity. At NPL, tests have been made on both bars and discs to high temperatures using

the long thin connecting rod method, although with this method care has to be taken to ensure that any resonances in the sensing rod are known and rejected.

These two methods are usable to a temperature at which the test-piece can no longer ‘ring’ freely. As secondary phases begin to soften, there is a rapid increase in the degree of damping of vibrations. Peaks in the resonance spectrum become broader and less intense, and it may no longer be possible to find a natural frequency in the impact excitation method. Data at higher temperatures requires alternative methods, but these are not particularly accurate.

The **beam flexure** method can in principle be used at any temperature and in principle can give a measure of Young’s modulus as the low-stress slope of the load/displacement curve, even if some deformation occurs at higher loads. However, because the test has to be performed inside a furnace jacket the load train is long, as are extensometer rods for detecting deflections. Careful engineering is required to ensure that the deflections measured can be properly corrected for displacements in the measuring system, which may be rather larger than that of the test-piece.

The **ultrasonic velocity** method using the pulse echo (single send/receive transducers, for both S- (shear) and P-waves (compressional)) suffers from problems of the number of cemented interfaces usually necessary between the hot test-piece and the cold transducers. The effect of these interfaces can be minimised by acoustic impedance matching, or alternatively the interfaces can be eliminated by using a long rod test-piece with a reduced diameter of short but defined length in the hot zone (Grigg, M.W., *et al.*, *J. Phys. E.*, 1986, **19**, 1059-63). The pulse entering the rod at the cold end is reflected from the step in diameter and from the end, and the time delay between the two return pulses is determined. Provided that clean pulses are detected, measurements can be made to higher temperatures than the resonant or impact excitation method. However, the drawback of this method is the size of test-piece that is required, which must pass through the furnace wall with its stepped end in the uniform hot zone.

#### 4.2.2 Flexural strength

Flexural strength testing requires the use of a test jig that can withstand the required temperature of operation, yet still perform its function of minimising misalignment and friction. For temperatures beyond the capability of hard and oxidation resistant metal alloys, a ceramic test jig is normally required. The most popular material is sintered silicon carbide which, although brittle, has the advantage of being usable to temperatures in excess of 1300 °C. When used in air, the oxidation that takes place at such temperatures results in a thin silica film being formed, but it is generally thought that this does not greatly influence the function of the jig, and can be readily removed by soaking for a short period in HF.

Achieving high temperatures requires the use of a furnace arrangement in which the test jig sits, with support and loading rams passing through the furnace insulation. Since the test-piece generally needs to be positioned in the test jig while at room temperature, a low thermal

mass, fast heating/cooling furnace design is desirable to minimise testing time. Care is needed in the design of the facility to ensure that the ceramic parts are not thermally shocked, that the temperature distribution along the test-piece length after temperature stabilisation does not exceed limits placed in the standards (typically no more than 10 °C), and that heat is not transmitted to load cells or mechanical parts of the test machine into which this unit is connected.

The test procedure is then very similar to the room temperature testing, with the exception that at high temperatures where materials may no longer show linear force/displacement behaviour, the stress distribution within the test-piece is no longer equivalent to the purely elastic case upon which the simple strength calculations rely. Instead, the true surface stress in the test-piece is less than the elastic stress by an indeterminate amount, and the result is normally considered to be nominal. There are methods of acquiring true stress/strain data from flexural testing, but these are complicated and rely on obtaining a large data set.

Normally, if true strengths are required under non-linear conditions, it is necessary to use tensile testing. This is an expensive test, and the value of the results must be seriously considered before undertaking it. Techniques for gripping need to be carefully designed in relation to the furnace system being employed, and alignment is crucial (see 4.3.3) Standards exist (see Table 6) to guide the user.

#### **4.2.3 Fracture toughness testing**

The room temperature methods preferred for ceramics involving most beam flexure can readily be conducted at elevated temperatures if necessary, but there are some important issues associated with crack healing that need to be considered. Any method which involves pre-cracking the test-piece may give suspect results at high temperature if either the crack tends to heal by sintering of contacting surfaces or its surfaces oxidise and the crack becomes filled with oxidation products. A VAMAS round-robin showed that fracture toughness tests on a silicon nitride material in air at 1200 °C gave  $K_{Ic}$  values considerably higher than when the tests were performed in an inert atmosphere. Methods such as CNB or SEVNB which do not require pre-cracking are probably the most convenient and reliable.

#### **4.2.4 Hardness**

Hardness tests can be made at elevated temperature in specially designed systems. A diamond indenter is usable to about 1100 °C in inert atmospheres without excessive degradation, but at higher temperatures other hard indenters (normally non-oxides) need to be used. The results of such tests have some value for materials comparison purposes, and for determining limiting temperatures for contact loading in service by showing, for example, the temperature at which a major loss in hardness occurs.

## 4.3 Longer-term testing

### 4.3.1 Deformation testing

For purposes of simply determining the low-stress usability of an ATC at elevated temperature, a simple self-loaded sag test can be used. A beam of defined dimensions is measured for flatness, and then supported at its ends on refractory supports and heated to a given temperature. After cooling, the flatness of the beam is re-measured. By making a series of such tests at a sequence of temperatures, the temperature of the onset of ‘self-loaded deformation’ can be determined. This is essentially a pragmatic way of defining an upper use temperature for materials such as furnace tubes, thermocouple insulators, probes and ceramic heating elements.

### 4.3.2 Flexural creep

For use at higher loads, a flexural creep test can be performed. However, this method has to be considered as providing only nominal information compared with conventional tensile creep testing because of the difficulties of analysing the data correctly, as already highlighted in flexural strength testing. The stress to be applied and the deformations obtained are normally computed assuming equations for the elastic case. However, the effect of doing this is that the true stress is overestimated, and the true strains are slightly underestimated once creep deformation occurs. There is an exception in the case that the creep is viscous, i.e. the creep rate is directly proportional to stress, and not to some higher power of stress. In most ceramic materials, the latter is the case. In fact, often the creep behaviour is different in tension and compression, so in a bent beam, there is additionally a shift in the neutral axis (the line along the test-piece where the stress is zero) towards the compression side.

The scientific literature contains a number of examples of attempts to deconvolute flexural creep into equivalent tensile and compressive creep behaviours. However, this is complicated, requires a number of tests at different temperatures and stresses, and is probably only worthwhile performing if true engineering creep data are needed for design purposes. For most materials comparison purposes, the nominal creep behaviour revealed in flexure, including any tendency for failure to occur, is adequate for decision-making. A procedure for flexural creep testing will shortly be published as EN 820-4.

### 4.3.3 Tensile creep

If true engineering creep performance is required, there is no real substitute for undertaking tensile creep testing. However, this is a specialist test requiring careful design of gripping systems and an alignment procedure which minimises bending. It is thus an expensive test to undertake. Considerable effort has been expended, especially in the USA and Japan, on this topic. Test method development has centred around three methods of gripping dog-bone shaped test-pieces:

1. Cold grip, external to a short furnace, with strain measured either external to the furnace and corrected for the effects of creep in the shoulder regions, or via extensometry directly on the gaugelength.
2. Warm grip, typically of button-head test-pieces, using high-temperature metal alloy split or collet grip systems, usually with strain measured using extensometry directly on the gaugelength.
3. Hot grip, typically of button-head or flat shouldered test-pieces located in heavy duty ceramic pull devices, usually with strain measured using extensometry directly on the gaugelength.

Alignment is crucial, and the maximum permitted bending strain should be less than 5% of the axial strain. To achieve alignment, a series of strain gauges are applied to a dummy test-piece, which is then load cycled in order to determine the nature of the bending. Its near elimination is achieved by adjusting the load train positioning (depending on design of the system).

The procedures for alignment and testing have been standardised in ASTM C1291 and JIS R1631, but there is currently no standards action for ceramics in Europe. Alignment procedures have been developed, and are included as an Annex in ASTM C1291, and more general procedures have been published by NPL (*NPL Measurement Good Practice Guide No. 1, Bending in uniaxial low cycle fatigue testing*, ISSN 1368-6550).

#### **4.3.4 Fatigue testing**

Although there are a number of reports in the scientific literature concerning fatigue testing at elevated temperature, there are no standardised procedures yet published. In principle, testing can be performed by a variety of methods depending on the frequency required, and whether compressive as well as tensile stresses need to be used. Tensile-only fatigue can be accomplished by using the same set-up as for short-term tensile testing, but for push-pull fatigue, a rigid warm grip system using special button-head test-pieces is needed. Fatigue testing in flexure is possible, but currently limited to unidirectional flexure in three or four-point bending. Reverse bending to achieve compression/tension stress cycles is difficult unless a means is found for gripping specimens and using cantilever or pure bending via end grips.

## **5. Thermal properties**

### **5.1 Introduction**

Thermal properties of ATCs are of considerable significance in many applications, and are often directly exploited:

- Thermal expansion: Many ceramic materials have lower thermal expansion coefficients than most metals, so if dimensional matching is required for rigid joints,

properties need to be known quite accurately so that materials can be appropriately selected and designs can be prepared which will overcome problems of mismatch.

- Thermal conductivity: Thermal conductivity values cover a wide spectrum, from thermal insulators (*e.g.* silicates,  $\text{ZrO}_2$ ) to good thermal conductors (*e.g.*  $\text{AlN}$ ,  $\text{Si/SiC}$ ). Component design can take advantage of this range, allowing appropriate materials to be selected.
- Thermal shock resistance: Unlike metals, the lack of ductility in ceramics means that under adverse conditions they can suffer from thermal shock. Resistance to thermal shock in fully dense fine-grained materials is generally determined by a combination of thermal expansion coefficient (lower is better), thermal conductivity (higher is better) and fracture toughness (higher is better). Coarser grained or microcracked materials which do not have high strengths may have better thermal shock resistance in terms of less catastrophic loss in strength.

Thermal property test or measurement methods for ceramic materials are fundamentally the same as for any other material, with the exception that higher temperatures are often needed.

## 5.2 Test methods

### 5.2.1 Thermal expansion

Thermal expansion measurement is typically carried out using mechanical dilatometry (*e.g.* EN 821-1, ASTM E228, JIS R-1618). A small rod or bar test-piece is placed in a test-piece holder constructed from fused silica, recrystallised alumina, or other dimensionally stable refractory material, with one end up against a rigid stop. The other end is contacted by a separate expansion measuring device, normally a push-rod of the same material as the testpiece holder. As the system is heated or cooled, the relative movement of the push-rod and holder is recorded. Corrections are required for the thermal expansion of the holder, and any baseline movement resulting from temperature differences between holder and push-rod.

Thermal expansion data are normally expressed either as the fractional change in length referenced to a starting point, *e.g.*  $25^\circ\text{C}$ , or as a coefficient of thermal expansion, the fractional change per unit temperature change. If the latter is cited at a given temperature it is often termed expansivity, while if cited over a temperature range, it is usually termed mean linear expansion coefficient. The numerical values may be significantly different, so care is needed in interpretation of data.

With particular regard to ceramic materials, thermal expansion behaviour may reveal several important features about a material's behaviour:

- **Internal stresses:** Thermal cycling in the apparatus may cause relaxation of thermal stresses which causes length changes. Softening effects at high temperatures can also be detected, *e.g.* in glassy phase containing materials.
- **Hysteresis:** Materials which contain microcracks or high levels of crystallographic thermal expansion anisotropy may show different behaviours on heating and cooling. Data derived may be a function of the maximum temperature of the test cycle.
- **Phase changes:** If the ceramic contains phases which are thermodynamically unstable, depending on the amounts of the phases and their specific volume changes, the phase transformations may be seen as non-linearities in the thermal expansion/contraction curves. Classical examples are  $\alpha/\beta$  transformations in quartz and cristobalite, and monoclinic/tetragonal transformations in unstabilised or partially stabilised zirconia.

### 5.2.2 Thermal conductivity/thermal diffusivity

For materials with moderate to high thermal conductivities, typically greater than  $5 \text{ W m}^{-1} \text{ K}^{-1}$ , direct measurement under a temperature gradient is usually performed using a comparator method, in which the test-piece is sandwiched between two reference materials of known conductivity, and the relative thermal gradients are measured. For materials with low thermal conductivity, typically less than  $5 \text{ W m}^{-1} \text{ K}^{-1}$ , larger thinner disc test-pieces are needed for guarded hot-plate type measurements.

For many applications requiring data to high temperatures, it may be simpler and more cost-effective to measure thermal diffusivity,  $a$ , which is related to thermal conductivity,  $\lambda$ , via:

$$\lambda = a\rho c_p$$

where  $\rho$  is the density and  $c_p$  is the specific heat capacity. This measurement can be readily and more cheaply undertaken at a series of temperatures using the laser flash technique in which a laser pulse is incident on one face of the thin disc test-piece, and the temperature response curve on the other face is recorded. The thermal diffusivity is related to the time interval for the temperature transient to reach half its final height, with appropriate corrections for finite pulse time effects and radiation losses. The technique does, however, have some limitations. The thickness of the test-piece has to be related to the thermal diffusivity in order that the temperature transient is achieved within a reasonable time period, typically 50 - 200 ms to minimise heat losses. Typically, thicknesses of 1 mm for low- $a$  materials to 3 mm or thicker for high- $a$  materials need to be used. The former requirement makes the method unsuitable for materials which have a microstructure on a scale of 200  $\mu\text{m}$  or greater. Further, most ceramics are optically translucent to some degree, so direct transmission of part of the laser pulse is feasible. This has to be eliminated by coating both faces of the test-piece with an opaque medium, typically graphite, so that the laser pulse is completely absorbed in the surface and converted to heat. Even so, materials with good infrared transmitting properties may still pose problems, and give results which are a function

of test-piece thickness. The method has been standardised in EN 821-2, ASTM E1461-92 (for carbon and graphite), and JIS R1611-97.

### 5.2.3 Specific heat capacity

Specific heat capacity can often be calculated approximately from the individual components in a material, where these are known, by a rule of mixtures approach. Further, for many ceramic materials, particularly those comprising principally oxides, carbides or nitrides of magnesium, aluminium and silicon, specific heat capacity does not vary greatly with composition. However, it should be noted that data can be strong functions of temperature, so direct measurement is often necessary for data for thermal modelling purposes.

There are two basic methods available for determining specific heat capacity, differential scanning calorimetry (DSC) and drop calorimetry, the former being most commonly available. In DSC, a small sample is placed in a crucible and the temperature difference between it and a similar crucible with a reference material (normally  $\alpha$ -alumina) is monitored as the pair is simultaneously heated or cooled. This temperature difference is related to differences in thermal mass, from which specific heat capacity at the test-piece temperature can be computed. The maximum temperature for satisfactory measurements depends on the design of the system, but the technique appears to be usable to 1600 °C.

Drop calorimetry uses larger samples, often in a lidded crucible of known mass which is dropped from a hot environment into a cold one, the temperature rise of the latter being monitored accurately. It provides mean specific heat data over the temperature range of the drop, which has to be latter deconvoluted to produce data equivalent to that produced by the DSC.

Both techniques have been standardised for ATCs in EN 821-3, and DSC is also standardised in ASTM E1269-95.

## 5.3 Thermal shock

### 5.3.1 Introduction

A nominally stress-free ceramic article suffers from internal stresses when subjected to a temperature gradient. These can be particularly severe when the surface of the article is suddenly changed by, for example, quenching or exposure to hot gas jets. The resistance to fracture under the influence of such internal stresses is known as thermal shock resistance, but this parameter is particularly difficult to quantify as a material ‘property’ in the accepted sense. Consequently a pragmatic approach of testing the actual components in a simulated or actual environment is the most reliable, rather than trying to predict performance limits from a first principles basis. Thermal shock damage is most likely under conditions of downward quenching when the surface is placed into severe tension. The level of tension depends on:

- **Heat transfer coefficient:** The rate at which heat can be transferred from the article into the medium through the interface depends on the thermal properties, viscosity and velocity of the medium and the nature of the interface.
- **Article wall thickness:** In thinner articles, cooling of the bulk is quicker, and this reduces the surface stress transient.
- **Article edge curvature:** The sharper any corners, the faster that heat can be removed, and the more severe is the stress transient, so corners tend to suffer more than flat faces.
- **Article properties:** Low thermal expansion coefficient and high thermal conductivity are conducive to improved thermal shock resistance. High strength and high toughness also improve reliability.

Quantifying all these issues can be difficult, so for the purposes of material selection, ‘thermal shock parameters’ (see, for example, Morrell, R., Handbook of properties of engineering and technical ceramics, Part 1, Introduction for the engineer and designer, HMSO, 1985, pp. 152-8) can be computed using basic thermophysical and mechanical properties. However, for design purposes there are some useful rules of thumb which include:

- Choose a material with the best combination of properties commensurate with price and availability;
- Make the article as thin-walled as practical for the application;
- Use a generous radius of curvature on all internal and external corners;
- Design the application to reduce imposed thermal transients and to minimise exposure of the ceramic article to them; use thermal shielding where possible to reduce direct shock effects;
- Design the component so that any joints to metallic materials are remote from direct thermal shock; and ensure that metal components are as thin-walled as possible so that mechanical constraints are reduced.

### 5.3.2 Thermal shock testing

There is a fully standardised test for thermal shock resistance of electrotechnical ceramics incorporated into IEC 60672-2. This is a water quench test on flexural strength test-pieces, which has the function of providing data for comparison with the minimum specification given in IEC 60672-3. EN 820-3 gives some advice and guidance, including some general procedures for undertaking materials ranking tests using water quenching, which are adaptable to other conditions. Two main criteria for detecting the effect of thermal shock are suggested, loss in strength of bar test-pieces (but note that results may depend on the size and geometry of the test-pieces), and dye penetration testing for detecting the appearance of significant surface cracks. The latter is particularly useful for testing components of irregular shape that cannot be subjected to a strength test. There are no standardised tests for other conditions of thermal shock, so it is necessary to simulate an appropriate set of conditions.

## **6. Chemical corrosion testing**

### **6.1 Introduction**

Increasingly, ceramics are considered for engineering applications in which their resistance to chemical corrosion is a principal characteristic employed. However, materials must be correctly selected for corrosion resistance, because compositions and microstructures are often not designed specifically for such applications. Optimum solutions may need to be found for particular chemical environments, and there is no substitute for testing candidate materials in those environments.

Most ceramics are electrical insulators, and do not corrode by electrochemical effects in the same way as most metal alloys. Development of thick corrosion layers and pitting tend not to occur, except during the oxidation of non-oxides at elevated temperatures. Because ceramic materials often contain several phases, one or more of which may be continuous throughout the microstructure, it is possible to obtain penetration of the material without any overt sign of material removal. This is one of the most common modes of attack, particularly in aqueous-based corrosion. Methods of assessing the extent of corrosion therefore have to be devised in an appropriate manner.

### **6.2 Test methods**

These can be very varied, and depend on the medium and on the temperature/pressure/time conditions required. For laboratory materials screening work, chemical digestion vessels offer a very convenient method of assessing the significance of attack. The PTFE liner means that they can be used to 200 °C, and at pressures of up to 10 atmospheres, permitting accelerated tests to be performed. The draw-back is that only a limited volume of non-refreshed corrosive medium can be employed.

Care needs to be taken in aiming for accelerated testing simply by increasing temperatures, pressures or corrodent concentrations. Different reactions may occur than those under service conditions. It is often necessary to weigh up the pros and cons of accelerating the tests.

ISO 17092 provides for short-term testing in specific environments of boiling 6N HCl and 6N NaOH.

### **6.3 Evaluation criteria**

A number of different criteria can be employed in appropriate combinations. Table 7 indicates the main ones, of which dye penetration and strength tests are the most pragmatic ways of quantifying the results for many aqueous based environments. EN 12923-1 provides guidance on the selection of method and the processing of results.

**Table 7 - Evaluation methods for chemical corrosion tests**

| Test type                       | Aqueous based corrosion                     | Corrosion by melts                                 | Corrosion by gases and vapours                                                                |
|---------------------------------|---------------------------------------------|----------------------------------------------------|-----------------------------------------------------------------------------------------------|
| Penetration                     | Appropriate                                 | May be appropriate                                 | Appropriate in some cases                                                                     |
| Change of mass                  | Appropriate, but sometimes inadequate       | Inappropriate                                      | Appropriate if non-slagging                                                                   |
| Change of cross-section         | Not always appropriate when change is small | Appropriate                                        | Appropriate                                                                                   |
| Change of strength              | Appropriate                                 | May be appropriate if adherent material is removed | Appropriate if non-slagging; may also be appropriate if adherent slagging material is removed |
| Change of surface roughness     | Appropriate for slight corrosion            | Usually inappropriate                              | Usually inappropriate                                                                         |
| Change of hardness              | Appropriate                                 | Usually inappropriate                              | May be appropriate if non-slagging                                                            |
| Change of corrodant composition | Appropriate                                 | Usually inappropriate                              | Appropriate                                                                                   |

## 7. Testing methodology

### 7.1 For quality assurance

Testing for quality assurance (QA) purposes can be limited to those characteristics which are essential markers of consistency in the product in question. The extent to which testing is undertaken should depend on the criticality of the application, and the relative financial values applied to rejection of parts and failure of parts to fulfil their function in assembly or in service. Sampling schemes may be applied to large batches to ensure adequacy of density, dimensions, surface quality, *etc.*, if the design specification is such that tolerances are not critical. In some cases, every item needs to be tested. Some examples of simple QA procedures which might be applied to all manufactured items are given in Table 8. QA procedures that might be performed on a sampling basis as required are given in Table 9.

**Table 8 - Some simple QA testing procedures**

| Test             | Function                                                     | Standard procedures |
|------------------|--------------------------------------------------------------|---------------------|
| Visual           | Chips, discolorations, inclusions                            | -                   |
| Dye testing      | Searches for surface breaking cracks, pores, pits, scratches | EN 623-1            |
| Dimensions       | Checks that dimensions are maintained during production run  | -                   |
| Mass             | Consistency of die fill in pressing                          | -                   |
| Vacuum tightness | Item performance for such applications                       | -                   |

These testing regimes do not, of course, prove that every individual component has high strength. If a guarantee is required that every component has a minimum strength, then it is necessary to make individual inspections. Non-destructive methods, *e.g.* ultrasonic scanning, X-ray microradiography, scanning acoustic microscopy, *etc.*, can reveal internal defects such as large pores and delaminations, sometimes density inhomogeneities. However, since the small imperfections in advanced technical ceramics remain difficult to locate, identify and interpret with reliability using such methods, in critical cases it is necessary to consider proof-testing. This procedure is based on the premise that a short loading/unloading cycle applied to the component to stress it in the manner in which it will see stress in service will test for the presence of defects which might cause later failure. There are two ways in which proof-testing regimes can be established:

1. Based on an understanding of the long-term mechanical behaviour of the material at stress levels likely to be seen in the application, a prediction of a proof-test stress level can be made which will guarantee that proof-test survivors will go on to survive a given period under load in service.

**Table 9 - Some QA testing procedures used on a sampling basis**

| Test                  | Function                                                                                                                                                                          | Standard procedures                                                 |
|-----------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------|
| Density/open porosity | Checks for effectiveness of firing                                                                                                                                                | EN 623-2                                                            |
| Microstructure        | Grain size or phase volume fraction to check on consistency of manufacture                                                                                                        | EN 623-3, EN 623-5                                                  |
| Strength              | Checks for mechanical quality of the item - should be done on the final product, so the test needs to be carefully designed, and may only be feasible for certain shapes of item. | None- likely to need to be custom-designed                          |
| Chemical composition  | Generally used only for raw material batches                                                                                                                                      | Custom designed for type of material - some standards are available |
| Surface roughness     | Checks general surface quality and profile, particularly for seals, sliding parts, <i>etc.</i>                                                                                    | EN 623-4                                                            |

2. A test force can be applied which broadly represents, say, twice the force likely to be seen in practice.

The first option requires extensive research, but is capable of predicting the likely losses in proof-testing, and thus is useful in conjunction with a design tool which looks at the probabilistic nature of failure. The second option is more pragmatic, and in not relying on an understanding of material behaviour, cannot provide any long-term guarantees in service. One has to accept the number of failures that result under the selected conditions. In either case it is important to try to understand what proof-testing has done to the distribution of survivor strengths. The simplest way is to test a batch of survivors to destruction, and to compare the strength distribution with that of a batch not subjected to the proof test. If the low strength tail of the distribution has been removed without significantly weakening the survivors, the proof-test has been set up in such a way that it will identify and remove from the population those items with the worst defects of manufacture. A standard providing guidance on proof-testing is under development in CEN (to be published as TS 843-7).

## 7.2 Testing for materials comparison

The schedule of testing that needs to be adopted depends on the performance parameters perceived to be critical to the application. For most applications, any of the test procedures described in this guide may need to be used, but as a general rule, the properties to be compared are those which might limit performance. For higher temperature applications, or those in corrosive environments, it is important to make the measurements under simulated

in-service conditions. Table 10 gives some examples of property measurements that might be made for various circumstances of use.

**Table 10 - Most relevant properties for materials comparison**

| Application:                                                     | Properties:                                                                                                                               |
|------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------|
| Seals, pump parts, sliding wear parts, chemical plant components | Hardness, strength, corrosion resistance                                                                                                  |
| Electrical insulators                                            | Electrical characteristics, strength, thermal conductivity/diffusivity (heat sink characteristics), thermal expansion                     |
| Furnace parts, high-temperature components                       | High temperature strength, deformation or creep resistance, thermal shock resistance, thermal expansion, thermal conductivity/diffusivity |
| Seals, joints to other materials                                 | Thermal expansion, elastic properties                                                                                                     |
| Cutting tools                                                    | Hardness, toughness, strength                                                                                                             |
| Components for abrasive materials handling                       | Hardness, toughness, abrasive wear resistance                                                                                             |
| Guides, rollers, pick-ups, die parts                             | Hardness, toughness, strength                                                                                                             |
| Machine parts                                                    | Strength, elastic properties, thermal expansion                                                                                           |
| Moulds, parts for molten metal handling, crucibles               | Corrosion resistance, high-temperature strength                                                                                           |
| Orthopaedics                                                     | Strength, corrosion resistance (including biocompatibility)                                                                               |

### 7.3 Testing for data design acquisition

With increasing use made of modelling methods for design, there has to be an emphasis on using accurate numerical data, rather than comparative data. Many of the newer test methods being produced are designed to provide good quality data, backed up with targeted levels of accuracy and demonstrations of reproducibility between laboratories. Considerable effort has been placed in the last decade in trying to provide such information. For the designers, knowing the uncertainty levels is helpful, allowing them to predict confidence levels for the device performance.

In this respect it is important for designers to recognise that ATCs vary widely from source to source, and that the detailed characteristics of one material may well be different from those which are superficially similar. The technology of processing ceramics via powder routes, although often well established and stabilised, leads to difficulties for manufacturers to

provide precise control of parameters such as composition, homogeneity, grain size and density. Different manufacturing routes, or different sizes of part, may have different properties. The designer also needs to recognise that properties are also very often non-linear functions of temperature, so it is important to ensure that properties, such as thermal expansion behaviour, are acquired over a relevant temperature range.

## Bibliography - Listing of standard test methods for advanced technical ceramics

The listing given below is correct at the time of writing, but will change as new standards are developed and old ones are modified or substituted. For up-to-date listings, refer to the catalogues of the individual standards organisations or their websites (see end of Bibliography). The listing is not complete for all types of ATC, but provides titles for the major property measurement and testing standards discussed in this guide for fabricated 'monolithic' ceramics (*i.e.* not powders or granular materials, long-fibre composites, or coatings).

### Notes:

1. English language versions of CEN ENs are available in the UK from BSI as BS ENs, and CEN TSs (provisional standards, formerly ENVs) are available as BS DD TSs (DD = draft for development). French language versions are available from the Association Francaise de Normalisation (AFNOR), Paris, and German versions from the Deutsches Institut für Normung (DIN), Berlin. There are now no separate BS standards for ATCs.
2. ISO and IEC standards are available direct from ISO or IEC respectively as dual language (French/English) documents, or through national standards bodies. In the UK, where an ISO standard has been adopted as a BS, it will be identified as BS ISO xxxx, or if additionally adopted in Europe, as BS EN ISO xxxx.
3. ASTM standards are available in annually printed volumes direct from ASTM, which has a UK Office. Most standards related to advanced technical ceramics appear in Volume 15.01, while some for more traditional ceramics appear in Volume 15.02. More general measurement methods appear in other volumes.
4. JIS standards are available from Japan Industrial Standards of Tokyo. Many of those related to advanced technical ceramics are available in official English translation.
5. Details of standards still under development are provisional, and titles may change. Where no number has yet been allocated, they are denoted by 'xxxxx'.

### European standards

|          |                                                                                                                                                               |
|----------|---------------------------------------------------------------------------------------------------------------------------------------------------------------|
| EN 623-1 | Advanced technical ceramics - Monolithic ceramics - General and textural properties Part 1: Determination of the presence of defects by dye penetration tests |
| EN 623-2 | <i>ibid</i> , Part 2: Determination of density and porosity <i>ibid</i> ,                                                                                     |
| EN 623-3 | <i>ibid</i> , Part 3: Determination of grain size (under revision)                                                                                            |

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|---------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| ENV 623-4                 | <i>ibid</i> , Part 4: Determination of surface roughness                                                                                                                      |
| ENV 623-5                 | <i>ibid</i> , Part 5: Determination of phase volume fraction                                                                                                                  |
| EN 820-1                  | Advanced technical ceramics - Methods of testing monolithic ceramics - Thermomechanical properties, Part 1, Determination of flexural strength at elevated temperatures       |
| EN 820-2                  | <i>ibid</i> , Part 2: Determination of self-loaded deformation                                                                                                                |
| EN 820-3                  | <i>ibid</i> , Part 3: Determination of resistance to thermal shock by water quenching                                                                                         |
| EN 820-4                  | <i>ibid</i> , Part 4: Determination of flexural creep                                                                                                                         |
| TS 820-5                  | <i>ibid</i> , Part 5: Determination of elastic moduli at elevated temperatures                                                                                                |
| EN 821-1                  | Advanced technical ceramics - Monolithic ceramics - Thermophysical properties - Part 1: Determination of thermal expansion                                                    |
| EN 821-2                  | <i>ibid</i> , Part 2: Determination of thermal diffusivity by the laser flash (or heat pulse) method                                                                          |
| EN 821-3                  | <i>ibid</i> , Part 3: Determination of specific heat capacity                                                                                                                 |
| EN 843-1                  | Advanced technical ceramics - Mechanical tests at room temperature, Part 1: Determination of flexural strength                                                                |
| EN 843-2                  | <i>ibid</i> , Part 2: Determination of elastic moduli (under revision)                                                                                                        |
| EN 843-3                  | <i>ibid</i> , Part 3: Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests                                               |
| EN 843-4                  | <i>ibid</i> , Part 4: Determination of hardness                                                                                                                               |
| EN 843-5                  | <i>ibid</i> , Part 5: Statistical analysis                                                                                                                                    |
| EN 843-6                  | <i>ibid</i> , Part 6: Guidelines for fractographic investigation                                                                                                              |
| TS 843-7 (in preparation) | <i>ibid</i> , Part 7: C-ring tests                                                                                                                                            |
| TS 843-8 (in preparation) | <i>ibid</i> , Part 8: Guidelines for conducting proof tests                                                                                                                   |
| TS 843-9 (in preparation) | <i>ibid</i> , Part 9: Method of test for edge chip resistance                                                                                                                 |
| EN 1006                   | Advanced technical ceramics - Methods of testing monolithic ceramics - Guidance on the sampling and selection of test-pieces (under revision)                                 |
| EN 12923-1                | Advanced technical ceramics - Monolithic ceramics - Part 1: General laboratory procedures for chemical corrosion testing                                                      |
| EN 12923-2                | <i>ibid</i> , Part 2: Oxidation test for non-oxides (under development)                                                                                                       |
| TS 14425-1                | Advanced technical ceramics - Monolithic ceramics - Test methods for determination of apparent fracture toughness: Part 1: Guide to test method selection (under development) |
| EN ISO 15723              | <i>ibid</i> , Part 2: Single-edge pre-cracked beam method                                                                                                                     |
| EN 14425-3                | <i>ibid</i> , Part 3: Chevron notch method                                                                                                                                    |
| EN ISO 18756              | <i>ibid</i> , Part 4: Surface crack in flexure method                                                                                                                         |
| TS 14425-5                | <i>ibid</i> , Part 5: Single-edge vee-notched beam method                                                                                                                     |

## ASTM standards

|                     |                                                                                                                                                                |
|---------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------|
| ASTM C623-92        | Standard test method for Young's modulus, shear modulus and Poisson's ratio of glass and glass-ceramics by resonance                                           |
| ASTM C848-88 (1994) | Standard test method for Young's modulus, shear modulus and Poisson's ratio of ceramic whitewares by resonance                                                 |
| ASTM C1161-94       | Standard test method for flexural strength of advanced ceramics at ambient temperature                                                                         |
| ASTM C1175-97       | Standard guide to test methods and standards for non-destructive testing of advanced ceramics                                                                  |
| ASTM C1198-96       | Standard test method for dynamic Young's modulus, shear modulus and Poisson's ratio for advanced ceramics by sonic resonance                                   |
| ASTM C1211-98       | Standard test method for flexural strength of advanced ceramics at elevated temperatures                                                                       |
| ASTM C1239-95       | Standard practice for reporting uniaxial strength data and estimating Weibull distribution parameters for advanced ceramics                                    |
| ASTM C1259-98       | Standard test method for dynamic Young's modulus, shear modulus and Poisson's ratio for advanced ceramics by impulse excitation of vibration                   |
| ASTM C1273-95       | Standard test method for tensile strength of monolithic advanced ceramics at ambient temperatures                                                              |
| ASTM C1291-95       | Standard test method for elevated temperature tensile creep strain, creep strain rate, and creep time-to-failure for advanced monolithic ceramics              |
| ASTM C1322-96       | Standard practice for fractography and characterisation of fracture origins in advanced ceramics                                                               |
| ASTM C1323-96       | Standard test method for ultimate strength of advanced ceramics with diametrically compressed C-ring specimens at ambient temperature                          |
| ASTM C1326-96       | Standard test method for Knoop indentation hardness of advanced ceramics                                                                                       |
| ASTM C1327-96       | Standard test method for Vickers indentation hardness of advanced ceramics                                                                                     |
| ASTM C1331-96       | Standard test method for measuring ultrasonic velocity in advanced ceramics with broadband pulse-echo cross-correlation method                                 |
| ASTM C1332-96       | Standard test method for measuring ultrasonic attenuation coefficients of advanced ceramics by pulse-echo contact technique                                    |
| ASTM C1361-96       | Standard practice for constant amplitude, axial, tension-tension cyclic fatigue of advanced ceramics at ambient temperature                                    |
| ASTM C1366-97       | Standard test method for tensile strength of monolithic advanced ceramics at elevated temperatures                                                             |
| ASTM C1368-97       | Standard test method for determination of slow crack growth parameters of advanced ceramics by constant stressing rate flexural testing at ambient temperature |
| ASTM C1419-99       | Standard test method for sonic velocity in refractory materials and its use in obtaining an approximate modulus                                                |
| ASTM C1421-99       | Standard test method for determination of fracture toughness of advanced ceramics                                                                              |

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|------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| ASTM C1424-04          | Standard test method for monotonic compressive strength of advanced ceramics at ambient temperature                                                                     |
| ASTM C1465-00          | Standard test method for determination of slow crack growth parameters of advanced ceramics by constant stress-rate flexural testing at elevated temperature            |
| ASTM C1470-00          | Guide for testing the thermal properties of advanced ceramics                                                                                                           |
| ASTM C1495-01          | Standard test method for effect of surface grinding on flexural strength of advanced ceramics                                                                           |
| ASTM C1499-05          | Standard test method for monotonic equibiaxial flexural strength of advanced ceramics at ambient temperature                                                            |
| ASTM C1525-04          | Standard test method for determination of thermal shock resistance of advanced ceramics by water quenching                                                              |
| ASTM C1576-05          | Standard test method for determination of slow crack growth parameters of advanced ceramics by constant stress flexural testing (stress rupture) at ambient temperature |
| ASTM D5550-94          | Standard test method for specific gravity of soil solids by gas pycnometry                                                                                              |
| ASTM E228-85<br>(1989) | Test method for linear thermal expansion of solid materials with a vitreous silica dilatometer                                                                          |
| ASTM E399-90           | Standard test method for plane-strain fracture toughness of metallic materials                                                                                          |
| ASTM E494-94           | Standard practice for measuring ultrasonic velocity in materials                                                                                                        |
| ASTM E1269-95          | Standard test method for determining specific heat capacity by differential scanning calorimetry                                                                        |
| ASTM E1461-92          | Standard test method for thermal diffusivity of solids by the flash method                                                                                              |
| ASTM F394-78<br>(1991) | Standard test method for biaxial flexure strength of (modulus of rupture) of ceramic substrates                                                                         |

## IEC Standards

|                   |                                                                                 |
|-------------------|---------------------------------------------------------------------------------|
| IEC 60672-1: 1995 | Ceramic and glass insulating materials - Part I: Definitions and classification |
| IEC 60672-2: 1999 | ibid, Part 2: Methods of test                                                   |
| IEC 60672-3: 1998 | ibid, Part 3: Specification for individual materials                            |

## ISO standards

|                |                                                                                                                                         |
|----------------|-----------------------------------------------------------------------------------------------------------------------------------------|
| ISO 3274:1996  | Geometrical product specifications (GPS) - Surface texture: profile method - Nominal characteristics of contact (stylus) instruments    |
| ISO 4287: 1997 | Geometrical product specifications (GPS) - Surface texture: profile method - Terms, definitions and surface texture parameters          |
| ISO 4288:1996  | Geometrical product specifications (GPS) - Surface texture: profile method - Rules and procedures for the assessment of surface texture |

|                                  |                                                                                                                                       |
|----------------------------------|---------------------------------------------------------------------------------------------------------------------------------------|
| ISO 5436:1985 (= BS 6393:1997)   | Calibration specimens - stylus instruments - types, calibration and use of specimens                                                  |
| ISO 6474:1994 (= BS 7253-2:1997) | Implants for surgery - ceramic materials based on high-purity alumina                                                                 |
| ISO 11562:1996                   | Geometrical product specifications (GPS) - Surface texture: profile method - Metrological characterisation of phase correct filters   |
| ISO 14704                        | Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for flexural strength at room temperature                |
| ISO 14705                        | <i>ibid</i> , - Test method for hardness of monolithic ceramics at room temperature                                                   |
| ISO 15165                        | <i>ibid</i> – Classification system                                                                                                   |
| ISO 15490                        | <i>ibid</i> - Test method for tensile strength of monolithic ceramics at room temperature,                                            |
| ISO 15732                        | <i>ibid</i> - Test methods for fracture toughness by single-edge pre-cracked beam (SEPB) method                                       |
| ISO 17092                        | <i>ibid</i> - Determination of corrosion resistance of monolithic ceramics in acid and alkaline solutions                             |
| ISO 17561                        | <i>ibid</i> - Test method for elastic moduli of monolithic ceramics by sonic resonance                                                |
| ISO 17562                        | <i>ibid</i> - Test method for determination of linear thermal expansion of monolithic ceramics by push rod technique                  |
| ISO 17565                        | <i>ibid</i> - Test method for flexural strength of monolithic ceramics at elevated temperatures                                       |
| ISO 18754                        | <i>ibid</i> - Determination of density and apparent porosity                                                                          |
| ISO 18755                        | <i>ibid</i> - Determination of thermal diffusivity of monolithic ceramics by laser flash method                                       |
| ISO 20501                        | <i>ibid</i> - Weibull statistics for strength data                                                                                    |
| ISO 20509                        | <i>ibid</i> - Determination of oxidation resistance of non-oxide monolithic ceramics                                                  |
| ISO 20808                        | <i>ibid</i> – Determination of friction and wear characteristics of monolithic ceramics by ball-on-disk method                        |
| ISO 22214                        | <i>ibid</i> - Test method for cyclic bending fatigue of monolithic ceramics at room temperature                                       |
| ISO 22215                        | <i>ibid</i> - Test method for tensile creep of monolithic ceramics                                                                    |
| ISO/DIS 23146                    | <i>ibid</i> - Test methods for determination of fracture toughness of monolithic ceramics – Single edge vee-notch beam (SEVNB) method |
| ISO 24370                        | <i>ibid</i> - Test method for fracture toughness of monolithic ceramics at room temperature by chevron notched beam (CNB)             |
| ISO/CD 26602                     | <i>ibid</i> – Silicon nitride materials for rolling bearing balls                                                                     |
| ISO xxxxx<br>(TC206 NP0610)      | <i>ibid</i> – Test method for thermal shock resistance of porous ceramics                                                             |

## Japanese Industrial Standards

Note: Many of those listed below are available in Japanese only, identified by (J). Some of the English language ones are equivalent to the ISO versions.

|              |                                                                                                                                             |
|--------------|---------------------------------------------------------------------------------------------------------------------------------------------|
| JIS R1601-95 | Testing method for flexural strength (modulus of rupture) of high performance ceramics                                                      |
| JIS R1602-95 | Testing methods for elastic modulus of high performance ceramics (J)                                                                        |
| JIS R1604-95 | Testing method for flexural strength (modulus of rupture) of high performance ceramics at elevated temperature (J)                          |
| JIS R1605-95 | Testing methods for elastic modulus of high performance ceramics at elevated temperature (J)                                                |
| JIS R1606-95 | Testing method for tensile strength of high performance ceramics at room and elevated temperature (J)                                       |
| JIS R1607-95 | Testing methods for fracture toughness of high performance ceramics (J)                                                                     |
| JIS R1608-90 | Testing methods for compressive strength of high-performance ceramics (J)                                                                   |
| JIS R1609-90 | Testing methods for oxidation resistance of non-oxide high-performance ceramics (J)                                                         |
| JIS R1610-91 | Testing method for Vickers hardness of high performance ceramics (J)                                                                        |
| JIS R1611-97 | Testing method for thermal diffusivity, specific heat capacity and thermal conductivity for high performance ceramics by laser flash method |
| JIS R1612-93 | Testing method for bending creep of high performance ceramics (J)                                                                           |
| JIS R1613-93 | Testing method for wear resistance of high performance ceramics by ball on disk method (J)                                                  |
| JIS R1614-93 | Testing method for corrosion of high-performance ceramics in acid and alkaline solutions (J)                                                |
| JIS R1617-94 | Testing method for fracture toughness of high performance ceramics at elevated temperature (J)                                              |
| JIS R1618-94 | Measuring method of thermal expansion of fine ceramics by thermomechanical analysis (J)                                                     |
| JIS R1621-95 | Testing methods for bending fatigue of fine ceramics at room temperature                                                                    |
| JIS R1623-95 | Testing method for Vickers hardness of fine ceramics at elevated temperatures                                                               |
| JIS R1624-96 | Testing method for bending strength of fine ceramic joint                                                                                   |
| JIS R1625-96 | Weibull statistics of strength data for fine ceramics                                                                                       |
| JIS R1630-97 | Test method for tensile strength of fine ceramic joint                                                                                      |
| JIS R1632-98 | Test method for static bending fatigue of fine ceramics                                                                                     |
| JIS R1634-99 | Test methods for density and apparent porosity of fine ceramics                                                                             |
| JIS R1640-02 | Methods for the quantitative phase analysis of silicon nitride (J)                                                                          |

|                |                                                                                                                               |
|----------------|-------------------------------------------------------------------------------------------------------------------------------|
| JIS R1642-1:02 | Test method for internal friction test of fine ceramics at elevated temperatures – Part 1: free decay by torsion pendulum (J) |
| JIS R1642-2:02 | Test method for internal friction test of fine ceramics at elevated temperatures – Part 2: Flexural resonance test (J)        |
| JIS R1645:02   | Testing method for water jet erosion of fine ceramics (J)                                                                     |
| JIS R1646:02   | Testing method for cavitation erosion of fine ceramics (J)                                                                    |
| JIS R1648:02   | Testing method for thermal shock resistance of fine ceramics (J)                                                              |
| JIS R1654:03   | Testing method for wear resistance of fine ceramics by ball-on-disk method at elevated temperatures (J)                       |
| JIS R1658:03   | Testing method for bending fatigue of fine ceramics at elevated temperatures                                                  |
| JIS R1659:03   | Testing method for elastic modulus of porous fine ceramics                                                                    |
| JIS R1664:04   | Testing method for bending strength of porous fine ceramics (J)                                                               |
| JIS R1666:05   | Testing method for macro-heterogeneity in microstructure of fine ceramics (J)                                                 |
| JIS R1668:05   | Testing method for fracture toughness of porous fine ceramics                                                                 |
| JIS R1669:06   | Fine ceramics – fundamental characteristics and classification of silicon nitride materials for rolling bearing balls (J)     |
| JIS R1670:06   | Testing method for grain size in fine ceramics (J)                                                                            |
| JIS R1671:06   | Testing method for water permeability and hydraulic equivalent diameter of porous fine ceramics (J)                           |
| JIS R1674:07   | Statistical analysis for influence of machining damage on strength of fine ceramics (J)                                       |
| JIS R1676:07   | Testing method for thermal shock resistance of porous fine ceramics (J)                                                       |
| JIS R1677:07   | Testing method for bending fatigue of porous fine ceramics at room temperature (J)                                            |

## Addresses of standards organisations

### **British Standards Institution (BSI):**

389 Chiswick High Road, London UK, W4 4AL

Phone: (+44) 208 9969000, Fax: (+44) 208 9967400

URL: [www.bsi.-global.com](http://www.bsi.-global.com)

### **Deutsches Institut für Normung (DIN):**

Burggrafenstrasse 6, Postfach 1107, 10787 Berlin, Germany

Phone: (+49) 30 26012344, Fax: (+49) 3026011231

URL: [www.din.de](http://www.din.de)

### **Association Française de Normalisation (AFNOR):**

Tour Europe, 33 place des Corolles, La Defense 2, 92400 Courbovie, France

Phone: (+33) 14291 5555, Fax: (+33) 14291 5656

URL: [www.afnor.fr](http://www.afnor.fr)

### **International Standards Organisation (ISO):**

1, rue de Varembe, Case postale 56, CH-1211 Geneve 20, Switzerland Phone: (+41) 227490111, Fax: (+41) 227333430

URL: [www.iso.ch](http://www.iso.ch)

### **International Electrotechnical Commission (IEC):**

IEC Central Office, 3 rue de Varembe, CP 131, CH-1211 Geneva 20. Switzerland Phone: (+41) 22 919 0211, Fax: (+41) 22 919 0300

URL: [www.iec.ch](http://www.iec.ch)

### **American Society for Testing and Materials (ASTM):**

100 Barr Harbour Drive, West Conshohocken, PA 19428-2959. USA

Phone: (+1) 610 832 9500, Fax: (+1) 610 832 9555

UK Office: 27/29 Knowl Piece, Wilbury Way, Hitichin, Herts. SG4 OSX.

UK Phone: (+44) 1462 437933, Fax: (+44) 1462 433678

URL: [www.astm.org](http://www.astm.org)

### **Japan Industrial Standards Committee (JIS):**

Standards Department, AIST, MITI, 1-3-1 Kasumigaseki, Chiyoda-ku, Tokyo 100-8921, Japan.

URL: [www.jisc.org](http://www.jisc.org)

Sales: Japan Standards Association: 4-1-24, Akasaka, Minatoku, Tokyo 107-8440, Japan

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URL: [www.jsa.or.jp](http://www.jsa.or.jp)

