

REPORT

Measuring Elastic Properties of Advanced Technical Ceramics - A Review

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Measuring Elastic Properties of Advanced Technical Ceramics

- A Review

by

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Abstract

A variety of methods have been proposed for standardisation of the measurement of elastic properties of advanced ceramic materials in CEN ENV 843-3. This report reviews practical considerations involved in the various techniques, ascertains the levels of error potentially involved, and discusses limitations for the extension of the methods to elevated temperatures.

It is concluded that:

- i) the impact excitation method is the simplest for determining Young's modulus, and is readily extended to high temperatures by using small ceramic spheres as the impacting body, although the reliability of determining shear modulus is less good than the resonant beam method;
- ii) the resonance method is experimentally more difficult to extend to high temperatures in air when testing small test-pieces because of problems in obtaining effective suspension methods; use of cotton thread carbonised in situ appears to be the most successful route, but this requires the test to be performed in vacuum or a protective gas environment;
- iii) the ultrasonic time of flight method is ideal for determining the degree of anisotropy of elastic properties, but is difficult to extend to high-temperature use. Alternative laser acoustic methods may be more reliable, but expensive to establish;
- iv) the traditional beam flexure method is the most error-prone of all the methods, but is simple to set up and to extend to elevated temperatures. Strain gauges can be used to improve accuracy, but are limited in temperature capability.

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LIST OF SYMBOLS

a	Distance between inner and outer rollers in four-point bending
A_i	Correction factor, where $i = f$ for flexure and t for torsion
b	Test-piece width perpendicular to direction of flexure
B	Mathematical function
d	Test-piece diameter
E	Young's modulus
f_l	Longitudinal resonance frequency
f_f	Flexural resonance frequency
f_t	Torsional resonance frequency
F	Force applied to test-piece
G	Shear modulus
h	Test-piece height in direction of flexure
H	Mathematical function
l	Test-piece length (free resonance), or span length (flexural method)
m	Test-piece mass
n	Mode of vibration (= 1 for fundamental mode)
V_l	Velocity of sound for longitudinal (P) waves
V_s	Velocity of sound for shear (S) waves
x	h/l
X	Correction factor for longitudinal resonance due to Love
y	h/b
γ	$d/2\lambda$
δ	flexural displacement
Δi	Error in variable i
λ	Wavelength of vibration
ν	Poisson's ratio
ρ	Density of test-piece

1. INTRODUCTION

Elastic moduli are important parameters, especially for the design of components and assessment of performance. They permit the calculation of strains and distortions when a component is subjected to stress. Accurate data are often required, but commonly are not readily available for many ceramic products, particularly if they have been obtained by the traditional beam-bending method. This report covers issues evaluated during DTI Project AM8 Task 3 on modulus determination of advanced ceramics and cermets.

There is a large number of methods that can be used for the determination of modulus. Traditionally, the modulus of metallic materials is measured as the slope of the tensile stress/strain curve at the zero stress origin, but this is inappropriate for ceramics since tensile testing is complex and difficult to perform reliably, and thus not routinely performed. In particular, as has been shown in recent work on tensile modulus determination in metal matrix composites (Roebuck et al. (1992)), a large scatter in results can be obtained between laboratories depending on the precautions taken and the method of data analysis.

Alternative, more-appropriate methods of measuring elastic properties are therefore needed. For both glass and ceramic materials, methods related to resonant frequency methods are well established in ASTM standards, and have been subjected to substantial research during the 1930s and 1940s (see Annex A). Others, such as impulse excitation (Lemmens, Heritage et al. (1988)), are of more recent origin, but basically follow the same principles as resonant methods. Static flexure has also traditionally been used for ceramics on the grounds that modulus can be determined as a part of strength measurement. Its value can be enhanced by the use of strain gauges, a technique which eliminates possible errors due to inaccurate correction of jig stiffness, and is almost essential for ceramic matrix composites (Morrell (1995a)). Ultrasonic velocity methods have been available for about 30 years, but are more complex than the others. For these alternative methods, of particular concern are:

- (i) the potential accuracy of the various methods;
- (ii) practical limitations in performing the tests;
- (iii) limitations to particular material types.

This report evaluates some of the issues reviewed during experimental work on a variety of methods at NPL when applied to small advanced technical ceramic test-pieces.

2. RESONANCE METHODS

2.1 Principle

The principle of the resonance method is to suspend a test-piece by thin wires which can be used also to excite the test-piece and to detect the vibration. The frequency of excitation is scanned and the resonances are detected. The relative frequencies of the resonances are used to identify the vibration mode (flexural, torsional, longitudinal, etc.), and hence the

fundamental frequency, which is then used to calculate the modulus from standard equations. These equations have been reviewed in Morrell (1995b) and Annex A gives details. It should be noted that in order to obtain Poisson's ratio, both flexural and torsional determinations are necessary.

2.2 Practical considerations

2.2.1 Suspension systems for flexural mode

The key factor in setting up this method is the suspension of the test-piece at points close to but not at its nodal points for fundamental flexural vibration (the nodal points are considered to be at 0.224 of the length from each end), see Figure 1. The suspension material must be of as low mass as possible, yet still transmit the vibrations from the source and to the detector. Thread or thin wires are recommended by ASTM C623 and C848. Rubber bands are also reported to work effectively. Thin metal wires have been found to be too springy for use with small ceramic test-pieces (mass $\sim 1-3$ g), whereas they may work more satisfactorily for heavier test-pieces (mass > 20 g). Carbon fibres are also a possibility, although we were unable to find a completely satisfactory method of knotting them around the test-piece. Thread and rubber bands are clearly limited to room temperature. However, in inert conditions it has been reported (Kromp (1995), Ponton (1996)) that carbonisation of cotton thread permits the retention of sufficient resilience to maintain the suspension function to high temperatures, at least 1000 °C. However, this does restrict operation to a vacuum chamber enveloping the suspension, detection and furnace system. For high-temperature use in air, the only practical solution for small test-pieces is to use a fine metal wire, such as platinum, but the wire diameter needs to be extremely small (much less than 0.1 mm) for an effective test.

2.2.2 Excitation

For excitation purposes, a frequency generator connected to a loudspeaker served well as a simple source, and for detection, a record player stylus worked well since the important resonant frequencies are within the audio range. Such arrangements have been reported elsewhere, and are included in ASTM standards. The frequency is swept slowly and resonances determined.

An alternative method is to rest the test-piece on supports at the nodal positions and to excite vibration electrostatically by bringing into proximity with the test-piece at the antinodal position an electrode which is then subjected to high sinusoidal voltage. The test-piece needs to have a conducting metallic coating on the underside which is in electrical earth contact with the support system. The coating should not affect the test-piece mass significantly, yet should be adequate to function as an electrode which is mechanically attracted by the exciting electrode. This technique is said to work well for fundamental mode flexural vibrations¹.

¹ The "Erudite" equipment (CNS Electronics Ltd,) is a commercial system for undertaking transverse, longitudinal and torsional frequency measurements at room temperature in air using an electrostatic driver and a separate detector (information from R Stannard, Morgan Materials Technology Ltd). CNR-IRTEC (Faenza, Italy) has a vacuum apparatus with a single driver/detector exciting a common electrode (information from G De Portu).

2.2.3 Torsional vibration

The excitation of torsional vibration requires the suspension points on the test-piece to be different from those used for the fundamental flexural mode. The suspension points should be near to the ends, and over opposite corners of the test-piece to achieve torsional vibration in preference to flexural. This is more difficult to achieve than flexural vibration, and a wider, thinner test-piece is preferred.

2.2.4 Longitudinal resonance

Longitudinal resonance can be excited using a mechanical driver contacting the end of a bar test-piece, setting up a standing longitudinal wave. The test-piece is usually sitting horizontally on a mount which does not excessively damp the vibration, e.g. rubber, cork or ceramic fibre. Corrections need to be made if the test-piece shape is not slender. In principle this method can be extended to elevated temperatures by using a refractory support, and an excitation rod and a measurement detector probe made from a ceramic material such as alumina. The technique was exploited and reviewed in detail by Davies (1968).

An important general point is that with this technique, the detector will tend to detect all excited vibration modes, and thus torsional, flexural and longitudinal vibrations may be concurrent over a given frequency range with a risk of false identification. For a given expected modulus value it is possible to dimension the test-piece such that the expected frequencies are well separated and clearly identifiable as fundamentals and series of overtones. Some initial estimates of moduli are needed in order to do this. This may overcome some of the identification problems that can arise with the technique.

2.2.5 Anisotropy

The theory is based on homogeneous isotropic elastic properties. If there is anisotropy, then to a first approximation, the longitudinal and flexural vibration modes measure the lengthwise value of Young's modulus. It is rather less clear what effect anisotropy has on torsional behaviour. If the material is not homogeneous, then this has an unknown and indeterminate effect of apparent results.

A recent paper (Sakata et al. (1992)) has examined the methodology for deducing anisotropic moduli in a uniformly anisotropic material from a series of three orthogonally-cut test-pieces orientated in the principal directions of the anisotropy but using the impact excitation method to determine natural frequencies (see below). For each test-piece it is necessary to determine fundamental and first harmonics for flexural vibrations in the orthogonal directions, plus fundamental and first harmonic for torsional excitation. The diagonal elements of the stiffness tensor can then be determined. The other elements require additional test-pieces cut at different known orientations relative to the axes of anisotropy. In principle, the same technique could be employed in conjunction with resonance, but it is not a simple exercise. For cost-effectiveness, the ultrasonic time-of-flight method using shear waves and longitudinal waves on a single cuboidal test-piece of side 10-20 mm is to be preferred.

2.2.6 Resonance versus static modulus

The modulus data generated by this technique are essentially the adiabatic values, which are slightly different from the isothermal ones determined by static loading. However, because of the high frequency and low strains involved, meaningful data can be recorded to temperatures above the macroscopic softening point of the material, which limits static modulus determinations. Damping slowly increases as the temperature is raised towards the softening point of the material, and then increases greatly. This results in significantly broadened resonance peaks, indicating a reduction in Q-factor and increasing internal mechanical losses. This aspect of materials behaviour may be important in vibrating structures, and so is often characterised using the resonance technique, either from the width of the resonance peak or the decay rate when excitation is halted (Ponton (1996)).

2.3 Calibration

The calibration requirements are limited to a micrometer, a laboratory balance and a frequency counter, none of which pose problems in providing traceability of measurement.

2.4 Measurement errors

The equations developed to describe free-free vibration were based on simple vibration analysis with correction factors for end effects. These end effects were originally determined experimentally using different shapes of test-piece, and then the correction factor is curve-fitted to the data. It has to be assumed that these equations are fundamentally correct to better than 1% since this is typical of the accuracy of the constants used in them. The original authors claim their accuracy is much better than 1% for slender test-pieces (Spinner et al. (1960)).

Longitudinal resonance:

The basic equation for a long thin round-section rod of diameter d , length l , mass m , Young's modulus E , Poisson's ratio ν , and fundamental resonant frequency f_l is:

$$E = \left\{ \frac{16 m l f_l^2}{\pi d^2} \right\} \quad (1)$$

A correction factor (see Annex A) is required if the rod if $l/d \leq$ about 10.

Fundamental flexural mode:

For a rectangular section bar height h , width b , shear modulus G , (subscript f = flexural, t = torsional)

$$E = 0.9465 \left(\frac{m f_f^2 l^3}{b h^3} \right) A_f \quad (2)$$

where:

$$A_f = 1 + 6.5850(1+0.752\nu+0.8109\nu^2) - \frac{100.083(1+0.203\nu+2.173\nu^2)x^4}{12+76.06(1+0.14088\nu+1.536\nu^2)x^2} - \frac{0.86806x^4}{(2A)} \quad (2A)$$

The expression for A_f may be truncated if $x = h/l$ is small as in slender bars.

Torsional mode:

$$G = \left(\frac{4lmf_t^2}{bh} \right) A_t \quad (3)$$

where:

$$A_t = \frac{(h/b) + (b/h)}{4(h/b) - 2.52(h/b)^2 + 0.21(h/b)^6} \quad (3A)$$

The choice of equation or, more explicitly, the choice of the correction factor employed in terms of the effect on the calculated result, has been evaluated and is attached as Annex A. Generally, it appears that experimental errors contribute more significantly to the accuracy of the result than the choice of correction factor, at least for thin bars, particularly the size and dimensional accuracy of test-piece, and the assumption of its elastic homogeneity. For small test-pieces, the thickness in the direction of flexure, which may be as small as 1 mm, is raised to the third power in the basic equation, which means that an overall error in E of $\pm 3\%$ is incurred for a measurement and/or parallelism accuracy of ± 0.01 mm. Since dimensions are measured on the highest points of the surface, a systematic overestimate of the true elastic thickness is likely to occur, leading to a small underestimate of modulus. Other parameters, i.e. the mass of the test-piece, the test-piece length and the resonant frequency, can all be determined to rather less than 1% error, and these potential error sources can thus be ignored.

In torsion, the potential thickness error is less consequential, but remains the largest likely contributor. The overall error of measurement of G is considered to be approximately $\pm 1\%$.

Poisson's ratio can be determined from E and G through the relationship:

$$\nu = \frac{E}{2G} - 1 \quad (4)$$

Since an accurate determination of E requires knowledge of ν , it has been proposed (ASTM draft, Committee C28) that both E and ν are calculated through an iterative process, commencing with an assumed value for ν , calculating E , then using the measured G recalculate ν , repeating this cycle until a stable value for ν is obtained. For slender bars, inspection of Equation 2A above shows that the error in ignoring the correction factor for E becomes insignificant for $x = h/l < 0.04$ in comparison with errors of thickness measurement.

The error in ν determined from Equation 4 is substantial because a small difference is being taken. As an example, if $E = 300$ GPa, $G = 120$ GPa, $\nu = 0.25$, and the potential errors in E and G are $\pm 3\%$ and $\pm 1\%$ respectively from the uncertainty in thickness, the combined error in $E/2G$ is typically $\pm 4\%$, and hence that in $E/2G - 1$ is typically ± 0.05 , or $\pm 20\%$. This is why there is great variation in reported Poisson's ratio data, even though the moduli data may be acceptably accurate. In fact, it may be more accurate to determine Poisson's ratio directly using longitudinally and transversely operating strain gauges on elastically deformed test-pieces than attempting to determine it indirectly.

2.5 Conclusions

The resonance method employing test-piece suspension requires some skill to set up and use, especially for elevated temperature testing in air with small test-pieces. It has advantages over other methods in permitting also the determination of damping characteristics as a function of temperature. In comparison with the impulse excitation method (see 3. below), the latter is much simpler to set up and use.

3. IMPULSE EXCITATION OR NATURAL FREQUENCY METHOD

3.1 Principle

This method is based on the wheel-tapper approach. It has many similarities to the resonance method in that it uses the same equations, but relies on the natural ringing of a struck object. If the object is struck at a particular position, a given variety of natural frequencies will be generated. The overtones tend to decay faster than the fundamental, so detecting and measuring this longer-lived frequency is equivalent to determining the fundamental resonance. The technique was pioneered by Lemmens Elektronika BV who provide an instrument (the "Grindosonic", from its value to the grinding wheel industry as an NDE tool) specifically for the purposes of detecting the decaying fundamental. Alternatively, a frequency analyser can be used to obtain a Fourier transform of the spectrum of vibrations, permitting the direct identification of the fundamental and its overtones (Figure 1). In the present project the two instruments have been employed simultaneously to examine the value of the technique as applied to ceramics and metallic materials.

Conventionally the test has been used on round or rectangular section bar test-pieces, but recently there has been a proposal in ASTM Committee C28 to use the technique on disc test-pieces, which are often the form in which developmental materials are made. By supporting and striking the disc at particular positions it is said that both E and G can be determined from the single test-piece. This technique was not evaluated within the timescale of the present project.

3.2 Practical considerations

3.2.1 Support and impact technique

The same principles apply as for the resonance method in that an accurately shaped regular geometry test-piece is required. It is suspended or supported at its nodes for flexural mode,

on a central support for torsional mode, or on a soft non-damping base for longitudinal mode vibrations.

The method and position of impact excitation has been found to be critical. Unless the fundamental frequency is clearly interpreted by the Grindosonic, its output response can be in error. It appears imperative that the amplitude of unwanted vibrations is kept to a minimum, and therefore in striking the object, care must be taken to ensure that, for example, for flexural impact the impact is normal to the test-piece surface, so that longitudinal vibrations do not dominate. The best method is to employ a flat, compliant elastic strip (e.g. a large cable tie), with a ceramic or metal ball fixed to one end with an adhesive ("Blu-Tak" works well). A clean single, normally-approaching strike can be developed using this, avoiding multiple bounce and spurious sideways movement.

In experimental evaluations, it has been found that the consistency of indicated frequency or two-cycle time on the "Grindosonic" for clean impacts is typically better than 1 part in 1000, and this is reinforced by the consistency of indicated frequency on the frequency analyser used in parallel. This applies even at elevated temperature (see below).

3.2.2 Torsional excitation

Experimentally it has proved particularly difficult to excite torsional vibrations without excessive flexural mode being concurrent. Wide, thin test-pieces generally pose least problems, but a typical flexural strength test-piece with cross-section 3 x 4 mm, which is ideal for flexural mode vibration cannot be used reliably for the torsional mode.

3.2.3 Natural frequency detection

The natural frequencies are detected either acoustically by a microphone in close proximity, or by a contacting piezoelectric probe, both provided by the instrument maker. Acoustically, the general sound emitted upon excitation is detected, whereas the piezoelectric probe can be used to sample the vibration at specific positions in the test-piece. This has the disadvantage of possibly introducing some damping into the test-piece, but a distinct advantage in being able to select particular modes of vibration. For example, in the flexural mode with the test-piece being supported at its nodes and struck centrally, contacting the probe just outside the node position results in avoidance of most of the longitudinal and torsional modes of vibration.

Both detectors can be used at elevated test-piece temperatures, the microphone assisted by a directional ceramic tube waveguide, and the piezoelectric detector assisted by a thin ceramic or silica glass rod passing into the furnace system. These extension components have their own natural frequencies which must not be allowed to interfere with the measurements. We have found that the use of a frequency analyser helps to identify such spurious vibrations.

3.2.4 Impact at elevated temperature

A serious issue is how to provide a controlled impact at elevated temperatures inside a furnace system. It has been found that dropping balls 1-2 mm diameter down a refractory guide-tube onto the hot test-piece is the simplest way of achieving this. For oxidation

resistance, ceramic balls are recommended, e.g. attrition milling media or small ruby bearings can be used. An important factor is that the balls do not come to rest on the test-piece. If they do, the effective frequency will change, or could be damped out completely. The risk of this happening is minimised by using narrow test-pieces. The technique has been used effectively to 1000 °C on an alumina ceramic, and to 600 °C on a variety of metal alloys. It has even been used to examine the nature of coatings and oxide films on metallic materials. For this work, the test-piece support comprised woven glass fibre sleeving.

3.2.5 Torsional measurement at elevated temperature

In principle the technique could work for torsional measurement at high temperatures, although with only central support for the test-piece, problems were found with the position of the test-piece changing because it is repeatedly hit on one end. The technique might be more successful with heavier test-pieces. Again, it is important that the balls do not come to rest on the test-piece, but bounce off to one side.

3.3 Calculation of elastic moduli

The same equations as for the resonance method are employed for impact excitation. The "Grindosonic" manufacturer provides software for calculations, and this appeared to give numerically correct results. If the torsional excitation data are not available, Poisson's ratio is unknown, and an assumed value is requested by the software. For long thin test-pieces excited in flexural mode, the effect of varying the input Poisson's ratio is very small, as also indicated for the resonance method in Annex A.

3.4 Calibration

Accurate calculations of moduli assume that the "Grindosonic" is accurately indicating the true frequency, but the manufacturers suggest no recommended method for assuring this. The "Grindosonic" instrument relies on a quartz oscillator to energise a frequency counter. This is claimed by the manufacturer to be particularly accurate and stable over long time periods, as demonstrated by testing with a reference test-piece, but this clearly does not constitute traceable calibration. In order to provide traceability, this project has worked with the NPL Acoustics Branch to develop a calibration routine referenced back to NAMAS calibrated instruments. A frequency generator was used to supply a known frequency to a hi-fi loudspeaker and the frequency was determined using a NAMAS calibrated counter. In order to provide the required decaying signal needed by the Grindosonic, a sharply rising and slowly decaying sound intensity was provided by manually operating a "volume" control on the loudspeaker. In this mode, the "Grindosonic" microphone could be used, and the indicated result (time for two complete sinusoidal cycles) was recorded and compared with the frequency from the calibrated counter. This was done at three different frequencies covering the range typically used for impact excitation testing, 1, 4 and 10 kHz. To check the function of the piezoelectric detector, a metal plate was set up in front of the loudspeaker to act as a transducer, and the probe was then directly contacted onto the metal plate. This method could also be used to calibrate the frequency analyser over a wider range than its own internal 50 Hz calibration. This technique was simple and effective, and needed only a traceably calibrated frequency counter. It was in fact established that both instruments identified a given single frequency with an accuracy of better than 0.1% (Morrell (1996)).

3.5 Measurement Errors

These are essentially the same as for the resonance method, since the equations are the same, but there is also the potential error of a false reading being displayed on the "Grindosonic" instrument as a result of it detecting a spurious vibration. Normally the experimental repeatability is good, successive indicated values differing by less than 0.1%, but occasionally there is a value differing by 5% or more. There are two possible views: to include it as a valid "result", or to ignore it as being potentially spurious. By using the frequency analyser simultaneously with the "Grindosonic", the potentially spurious values often coincide with poor frequency spectra. Although the analyser shows the correct frequency to be present, it may be of low intensity relative to noise or other frequencies, one of which the "Grindosonic" has identified as being the longest-lived, but incorrectly associated with the fundamental. In the author's opinion, such results should be ignored as spurious. The frequency analyser output is a useful guide to the actual behaviour, removing reliance on a single computed value from a "black box". It is noteworthy that the incidence of spurious results increases with:

- (i) decreasing modulus
- (ii) increasing porosity
- (iii) increasing damping (becoming acoustically "dead"), typically occurring with increasing temperature
- (iv) increasing intensity of impact

3.6 Disc test-pieces

As mentioned above, ASTM committee C28 have proposed an annex to ASTM standard C1259 to cover the use of the technique for disc test-pieces, based on original work by Glandus (1981). This has potential advantages since by exciting two different modes on the same test-piece, Poisson's ratio can be directly determined. The disc test-piece is supported either on four points equally arranged around the nodal circle, i.e. a circle of radius 0.681 of the disc radius at which there is no displacement for fundamental flexure with maximum amplitude at the disc centre, or on soft foam that does not influence the vibration. For the flexural mode vibration, it is struck at the disc centre, and the vibration detected at the disc periphery (at 45 ° to the line joining a pair of opposed support points). For the torsional mode, it is struck on the nodal circle (at 45° to the line joining a pair of support points, and the vibration is detected on the nodal circle 90° from the striking point. This minimises the excitation of the flexural mode.

Poisson's ratio is calculated directly from the ratio of the flexural to torsional frequency and the ratio of thickness to radius of the disc according to a table. Young's modulus is taken as the mean of the flexural and torsional values using the obtained Poisson's ratio, requiring correction factors taken from tables. Shear modulus is similarly obtained from Young's modulus and Poisson's ratio.

Although untried in the present work, this technique has some promise for material available only in disc form. It may be possible to extend its use to high temperatures using the ball

drop method, although the risk of balls coming to rest on the test-piece surface are higher than with a test bar. The accuracy of the method, like that for bars, is still principally dependent on parallelism of the disc surfaces and accuracy of thickness measurement, and so is unlikely to be accurate for as-fired discs. Caution is also advised on the disc thickness to radius ratio, and the calibrations are likely to be less accurate for thickness to radius ratios greater than about 0.15.

3.7 Conclusions

The impact excitation technique is a very convenient one for modulus determination. A variety of regular shapes can be used with standard calculation routines (software is supplied with the instrument). In particular it is quick and reliable. It does not require exceptional skill in setting up, unlike the resonance method, but gives similar accuracies. We have shown that it can be readily extended to elevated temperatures by use of contacting or non-contacting probes. Its use in conjunction with a frequency analyser permits the exclusion of spurious results based on the clarity of the frequency spectrum detected. A simple technique for maintaining NAMAS traceable calibration has been developed.

4. STATIC FLEXURAL LOADING

4.1 Principle

A traditional method of determining modulus is to record the flexural displacement of a loaded beam. This has been incorporated, albeit in a rather unsatisfactory form, in IEC 672-2 for some years. CEN TC184/WG3 has included this in its published standard ENV 843-2 in a revised form where the accuracy of displacement measurement is improved by requiring long thin test-pieces to be used. The displacement can be recorded by a variety of methods, including:

- 1) relative movement of the loading and support rollers in either three or four-point bending using a direct measuring device, or based on machine crosshead displacement;
- 2) relative movement of two or more points on the convex side of the test-piece using a displacement transducer, in order to determine the curvature;
- 3) in four-point bending, the output from strain gauges fixed to the central part of the loading span.

4.2 Machine displacement method

Previous work at NPL has shown that very reproducible results can be achieved on long thin parallel-faced test-pieces by using a low load range saw-tooth cycle on a conventional mechanical testing machine, recording the apparent machine displacement, and correcting the results for the machine stiffness. Because testing machines tend to be somewhat non-linear on initial loading and on load reversal, it is important to use a part of the loading curve away from these end effects. Normally the first cycle should be ignored since the repeatability of machine behaviour seems to improve with continuous cycling.

The machine stiffness can be determined by substituting a thick stiff bar for the test-piece and repeating the loading cycle. Since the deflection of the test-piece is inversely proportional to the third power of the thickness, a bar ten times the thickness of the test-piece will show 0.1% of the deflection, which is negligible.

An alternative approach to determining the machine stiffness correction is to rotate the test-bar through 90° and retest it by flexing it in the direction of greater thickness, which appears raised to the third power in the formula (see Annex B). By taking the difference in deflection for the two geometries, the machine displacement can be cancelled out.

4.3 Transducer methods for test-piece displacement

Direct measurement of displacement using a transducer positioned to record relative movement of two points on the test-piece, e.g. the centre and under the loading position in four-point bending, requires no correction for machine stiffness. The difficulties that arise with this method are in ensuring that the contact forces are small compared with the loads being applied to the test-piece to bend it, and in making precise measurement of the positions of contact relative to the loading geometry.

Attempts to instrument simply a standard size (CEN EN 843-1 size B) flexural test-jig met with poor reproducibility of results. It is thought that this originated partly in the design of the transducer, which needs to have reproducible displacement characteristics, and partly because the displacements being measured were small, less than 10 µm.

4.4 Strain gauge methods

Work at NPL using strain gauges has been applied particularly to metal matrix and ceramic matrix composite materials tested in four-point bending. Provided that the source of strain gauges is reputable, and skill is used in applying them correctly, reproducible measurements can be made not only of the direct axial strain, but also of the corresponding Poisson strain, permitting Poisson's ratio to be determined also. Strain gauges can be used on both the tensile and compressive faces of the test-piece, allowing a comparison of apparent behaviours. In a monolithic ceramic these should be equal and opposite. In a composite ceramic, it has been shown that it is possible to deconvolute separate tensile and compressive stress strain behaviours (Morrell (1995a)), which may be different as a result of matrix cracking effects in tension.

An advantage of the strain gauge method is that it does not require a thin test-piece. It can be applied to any normal flexural strength test-piece. A disadvantage is the time and expense involved in preparing individual test-pieces and in ensuring the calibration of the strain gauges. Most gauges are calibrated for a given type of material, i.e. a given Poisson's ratio, so a potential source of error results from use on materials with a different Poisson's ratio.

Performing the test in flexure gives a well-controlled geometry and accurate knowledge of the stresses. For direct tensile or compressive stressing, issues of alignment are more complex, several gauges have to be used. Normally one would expect greater deviations between the outputs than in the flexural case, and the potential errors in averaging the output from them are rather greater than for the flexural case.

4.5 Error assessment

The principal errors involved in static flexure measurement derive from:

- i) lack of parallelism of the test-piece faces, especially in the direction of flexure;
- ii) frictional effects at support and loading points, which can be sensibly eliminated by use of freely rotating rollers;
- iii) non-linearity and a lack of repeatability of load/displacement behaviour in the test machine (when using machine displacement as the measure of deflection);
- iv) repeatability of contact positions of the linear displacement transducer on the test-piece (when using a transducer);
- v) the accuracy of calibration of strain gauges (strain gauges only);
- vi) resolution and repeatability of movement of the transducer;
- vii) precision of span measurement.

An analysis of the role of these factors with special regard to the recent revision work for the standard IEC 672-2 is given in Annex B. It should be noted that this standard permits a variety of shapes of test-pieces appropriate to a range of electrotechnical ceramics, including traditional porcelains. For advanced technical ceramics, which in many cases are significantly stiffer than traditional ceramics, long thin accurately machined test-pieces are preferred in order to maximise the displacement and/or reduce the forces applied or stress levels in the test-piece, but even then the minimum level of error is likely to be about $\pm 3\%$, which is poor compared with other methods, such as impact excitation where the principal error is in the uniformity of test-bar thickness and its measurement.

5. ULTRASONIC TIME-OF-FLIGHT METHODS

5.1 Principle

An ultrasonic pulse from a piezoelectric transducer driven at typically 5-20 MHz is transmitted along the length of a test-piece, and the time is recorded for it to reach the opposite face (Markham (1957)). This gives the ultrasonic velocity. Normally both longitudinal (P-waves) and shear waves (S-waves) are employed in order to determine Poisson's ratio. The moduli values for an isotropic material are given by:

$$G = \rho V_s^2 \quad (5)$$

$$\nu = \frac{(V_l/V_s)^2 - 1}{2(V_l/V_s)^2 - 1} \quad (6)$$

$$E = \frac{\rho V_l^2(1 + \nu)(1 - 2\nu)}{(1 - \nu)} \quad (7)$$

5.2 Practical considerations

5.2.1 Test-pieces

Flat parallel-faced test-pieces are preferred with lateral dimensions which are greater than the size of the transducer. Long rod samples, for example, are inappropriate due to reflection effects, especially using shear waves. The reason for this is that scatter from the side walls leads to the generation of secondary longitudinal waves, which generally travel faster than shear waves, confusing the shear wave response. A cuboidal test-piece (side 10-20 mm) is ideal since the isotropy of moduli can also be determined by making measurements in three orthogonal directions to yield the elements of the stiffness tensor.

For materials which show significant anisotropy, attempts need to be made to align the axes of the test-piece with those of the anisotropy, otherwise deconvolution of the results is complex.

5.2.2 Equipment

The principal requirements are for an electronic system which produces and receives appropriate pulse types, and determines the time of transmission. Normally a high frequency (5-20 MHz is typical) is used with a pulse length of about 10 μ s. The time of pulse traverse is taken from equivalent positions in the sent and received pulses, typically either the first cross-over of the pulse (where the intensity crosses the baseline, Markham (1957)) or the first arrival of the pulse (ASTM C769 for graphite). Using appropriate amplifiers and an oscilloscope, the time-of-flight can usually be determined to within a fraction of a microsecond. Multiple echoes can also be examined provided that attenuation is not excessive. A single sender/receiver transducer can also be used, relying on reflection from the opposite face.

5.2.3 Coupling compound

A key part of the experimental technique is to ensure that the pulse is cleanly transmitted from the emitting transducer and clearly detected by the receiving transducer. A coupling compound is usually required to provide reliable results. This is either a gel or medium hardness rubber, with the transducer forced into contact with the test-piece, or the transducer can be temporarily glued to the test-piece.

5.2.4 Limitations

The technique does not work particularly well on materials which highly scatter or attenuate the sound pulse, either because of porosity, acoustically mismatched second phases, or microcracks.

The technique is also difficult to apply at high temperatures. One solution proposed by Grigg et al. (1986) is to use the one-ended technique on a long rod heated at one end. This end has a step reduction in radius enabling part of the pulse to be reflected from the step and the other from the back face. The time difference between the two echoes corresponds to twice the length of the reduced section. The authors suggest that owing to sidewall scattering,

examination of the detail of the echoes permits both P-wave and S-wave velocities to be determined. A problem in generalising the application of this technique is that it has been demonstrated only on a dense, low thermal conductivity material, zirconia, in which a reasonably uniform temperature can be generated in the reduced radius section. Any temperature inhomogeneity will be worse with a material of higher thermal conductivity.

A recently developed alternative approach to high temperature measurement is the use of laser acoustic microscopy or variations of this (Oishi et al. (1990)). Since the source of excitation is remote from the test-piece, this can be heated. In one version available at AEA Technology, Harwell, a laser is used to detect the time difference between the incident pulse on the front face of the test-piece and the response at the sample back face detected by sensitive laser interferometry. The arrival of both compressional (P) waves and shear (S) waves can be detected, the latter by detecting displacement away from the axis of the incident pulse. It is said² that the greatest uncertainty is in accurate knowledge of the test-piece thickness, which requires accurate knowledge of expansion coefficient.

5.3 Error analysis

A detailed error analysis has not been conducted for this method. However, it is understood that the principal sources of error arise from four factors:

- i) Dimensional accuracy of the test-piece. It is essential to have accurately parallel faces measured to typically 0.01 mm to make dimensional errors negligible.
- ii) Correct detection of equivalent wave positions on the incident and detected pulses such that the determined traverse time is accurate. Depending on the nature of the material, pulse forms can become distorted due to attenuation effects such that there is uncertainty of this equivalence. The error is thus material dependent. For a typical traverse time of 10 μs using pulses of 20 MHz frequency ($= 0.05 \mu\text{s}/\text{cycle}$), the start of the arrival pulse, or its cross-over positions, may become uncertain by as much as a complete cycle, giving an error of at least 0.5%. Assuming the timebase of the instrumentation is significantly more accurate than this, this error converts to about $\pm 0.7\%$ in determining modulus from the square of the calculated pulse velocity.
- iii) The density of the test piece. This can normally be measured to better than 0.1%, but the technique clearly relies on this property also being homogeneous.
- iv) Errors in coupling the pulse between transducers and the test-piece. This uncertainty is probably equivalent to a distance of traverse error of typically 0.02 mm or less, but potentially unknown and variable.

Overall, this suggests a reliability of typically better than $\pm 1\%$ for shear modulus (Equation 5), better than $\pm 2\%$ for Poisson's ratio (Equation 6), and better than $\pm 3\%$ for Young's modulus (Equation 7) by propagation of errors, when the method is applied to small cuboidal test-pieces. These values are similar to those for the other methods.

² C Scruby, AEA Technology Ltd, Harwell Laboratory, 1995.

6. CONCLUDING REMARKS

The various methods of modulus measurement applicable to small ceramic test-bars have been reviewed theoretically and experimentally with particular attention to experimental limitations and expected levels of error. As a general tool for Young's modulus measurement to elevated temperatures, the impact excitation method appears to be the simplest and most convenient to employ. It is less convenient for shear modulus determination, and provides no information on damping characteristics. In contrast, the resonance method provides all this information, although for high temperature use it appears necessary to use vacuum systems to achieve effective suspension for small test-pieces. Ultrasonic methods provide information on the anisotropy of elastic properties, but are difficult to apply effectively at elevated temperatures. The traditional quasistatic beam flexure method can be applied to advanced technical ceramics. Reliable results can be obtained provided that attention is paid to the experimental set-up to minimise the errors, which can be large if machine cross-head displacement is used to record deflection. Long thin flat test-pieces are preferred. Strain gauges provide a reliable alternative means of measuring strain and hence modulus for beams in four-point bending.

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Resonance Frequency Test System

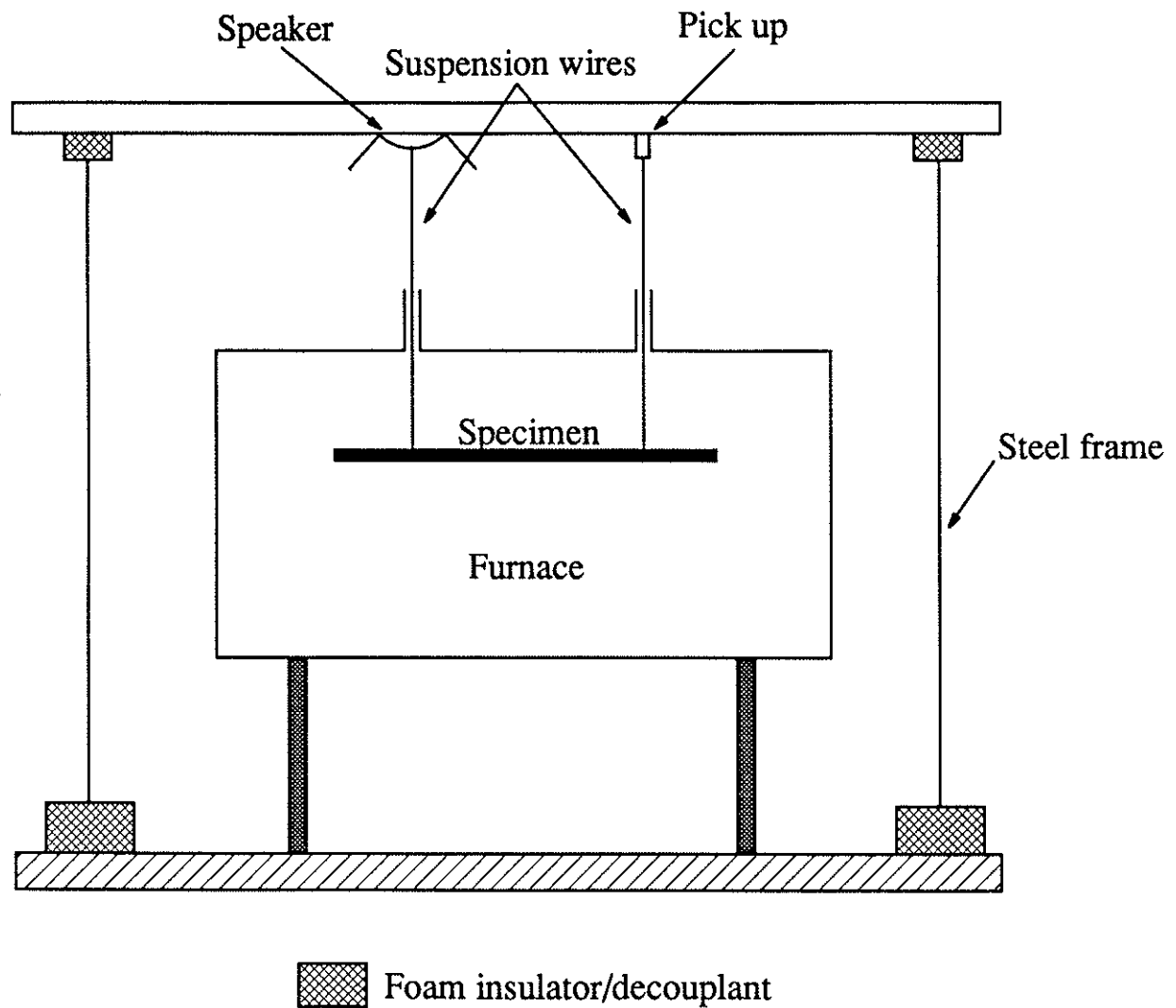


Figure 1 Flexural resonance method set up for use at elevated temperatures using metal wire suspension and a split furnace.

Trac Coord Data A: CH1 Lin Spec Data B: CH1 Lin Spec
[FFT] Coord A: LogMag Coord B: LinMag

Date: 16/03/96 Time: 20:23:00

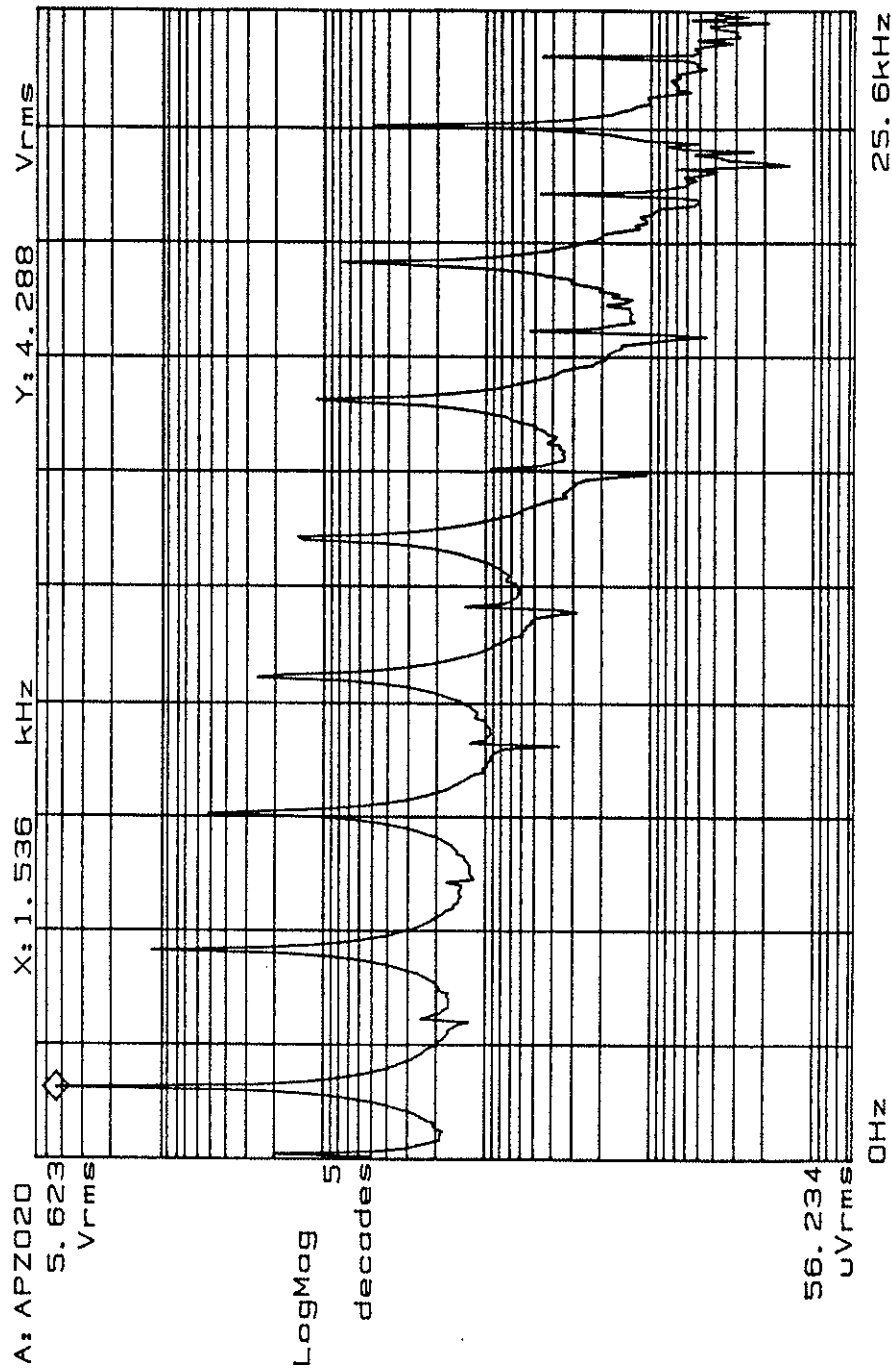


Figure 2 Typical frequency analyser response for impact excitation of a metallic test-bar showing the fundamental natural frequency at 1.536 kHz and the subsequent odd-numbered harmonics. Note that the response is on a logarithmic scale.

ANNEX A

SELECTION OF EQUATIONS FOR ELASTIC MODULUS MEASUREMENT OF ADVANCED TECHNICAL CERAMICS BY RESONANCE AND IMPULSE EXCITATION METHODS (Morrell (1995b))

A.1 INTRODUCTION

The objective of this annex is to define the accuracy limitations when using various equations for calculating elastic properties from resonant frequency or impulse excitation frequency of test bars. In reviewing these equations for incorporation into a CEN standard for testing advanced ceramics, the question of the most appropriate choice of equation arose. A study of levels of error was made, and from this, justification for the choice of equation form was obtained.

A.2 EQUATIONS

Different literature sources give various equations relating elastic properties to modes of vibration in bar test-pieces. In each of these equations, various simplifying assumptions or correction factors have been made, giving varying levels of accuracy in calculating elastic moduli. The error levels in ignoring or truncating correction factors are calculated for each equation as functions of geometry and Poisson's ratio, and are compared with possible errors due to the production and dimensional measurement of test-pieces.

It should be noted that in the original derivation of the correction factors there is inevitably some error. The magnitude of the error is sometimes given in the original references, and in some cases upper limits are as large as the differences in truncation errors that are considered below. In this annex these potential errors are ignored.

A.2.1 Longitudinal resonance

The basic equation for longitudinal vibration of a cylindrical test-piece is:

$$E = \left\{ \frac{16 m l f_{ln}^2}{\pi d^2 n^2} \right\} \quad (A.1)$$

for the n^{th} mode of vibration. For a rectangular section bar, $\pi d^2/4$ is replaced by bh , the cross-sectional area. Strictly this equation is valid only for a long thin rod or bar, and a correction term has been proposed (Love (1964), Glandus (1981)) for the case where the rod cannot be considered long compared with its diameter. This factor is given by:

$$X = 1 - \pi^2 v^2 \gamma^2 \quad (A.2)$$

where $\gamma = d/2\lambda = dn/4l$ for the n^{th} mode, and modifies Equation 1 to:

$$E = \left\{ \frac{16 m l f_{\text{ln}}^2}{\pi d^2 n^2 X^2} \right\} \quad (\text{A.3})$$

Inspection of the correction factor shows that the value of X^2 increases with increasing diameter to length ratio, and with increasing Poisson's ratio. The magnitude of this correction is given in Table A.1 assuming Poisson's ratio is 0.3, a value higher than for most advanced technical ceramics in order to obtain a maximum likely value for the correction factor.

Table A.1 - Correction factor for longitudinal vibration

d/l		0.01	0.02	0.05	0.10	0.20
X^2	$n = 1$	1.00001	1.00004	1.00028	1.00111	1.00446
	$n = 2$	1.00004	1.00028	1.00111	1.00446	1.01802

The results of the evaluation show that failure to use the correction results in underestimation of Young's modulus by a small amount. If it is assumed that the accuracy target for a standard test is 0.5%, it is desirable to keep the calculation error below 0.1%. The correction factor can therefore be ignored for the fundamental frequency if $d/l \leq 0.09$, or for the second mode, if $d/l \leq 0.04$. For $n = 1$, this condition can be readily met and is an appropriate figure to use for a standard.

In comparison, the errors of measurement of dimensions can be calculated as shown in Table A.2 for two cases (a) when the equation is used in the form of Equation A.1 based on mass and dimensions, and (b) when the mass and dimensions are replaced by density measured independently by substituting ρ for $4m/\pi d^2 l$ (the equivalent substitution can be made for rectangular section bars). The minimum sizes chosen for the test-piece give a ratio $d/l = 0.05$. Non-uniformity of diameter is assumed to be less than $d/100$ for rod test-pieces, but this is only important if calculation (a) is used.

Table A.2 demonstrates that the largest error is in measuring rod diameter (or the corresponding parameters for a rectangular section bar), and that for the d/h ratio chosen, an independent measure of density to 0.01 Mg m^{-3} will give a more accurate result for small diameter bars, but remains the largest error. Thus the density form of the equation might be used with advantage, especially for test-pieces which do not have particularly uniform diameter along their lengths, or which do not have accurately parallel faces. These results vindicate ignoring the Love correction unless the test-piece has $d/l > 0.1$ and the density is measured more accurately than to 0.01 Mg m^{-3} (assuming it is practically uniform to this level within the test-piece). Errors in determining the frequency are usually considered negligible compared with other parameters, although this is true only if the suspension system plays a negligible role, and for this reason its mass must be kept to a minimum.

Table A.2 - Dimensional errors in longitudinal resonance.

Source of error	Accuracy of measurement proposed	Magnitude of parameter	Effect of error using Equation 1 (Case (a))	Effect of error using separate density value (b)
Mass, g	Weigh to 1 mg	10 g	< 0.01%	-
Length, mm	Measure to 0.05 mm (callipers)	>60 mm	< 0.1%	< 0.2%
Diameter, mm	Measure to 0.01 mm (micrometer)	>3 mm	< 0.7%	-
Frequency, Hz	Measure to fourth place	1000 to 50000 Hz	< 0.2%	< 0.2%
Density, Mg m ⁻³	Measure to 0.01 Mg m ⁻³ by immersion	>2.5 Mg m ⁻³	-	< 0.4%

A.2.2 Flexural resonance

There is a variety of equations used in the literature, with varying complexity depending on the correction functions employed. For a rectangular section bar using SI units, they all have the form:

$$E = 0.9465 \left\{ \frac{m f^2 l^3}{b h^3} \right\} A_f \quad (\text{A.4})$$

where A_f is a correction factor to account for inertia effects and shearing. This correction factor has terms in Poisson's ratio, and the depth to length ratio $h/l = x$. Goens (1931) was the first to evaluate this fully, followed by modification by Pickett (1945), and consolidation into a single form was later done by Spinner, Reichard and Tefft (1960), which is considered to be accurate to better than 1% for wide range of specimen shapes, but much more accurate for slender bars:

$$A_f = 1 + 6.5850(1 + 0.752\nu + 0.8109\nu^2)x^2 - \frac{100.083(1 + 0.203\nu + 2.173\nu^2)x^4}{12 + 76.06(1 + 0.1408\nu + 1.536\nu^2)x^2} - 0.86806x^4 \quad (\text{A.5})$$

where $x = h/l$. In the draft ASTM standard for ceramics (Committee C28), the numerical terms of this equation are rounded to give:

$$A_f = 1 + 6.58(1 + 0.08\nu + 0.8\nu^2)x^2 - \frac{100(1 + 0.02\nu + 2.2\nu^2)x^4}{12 + 76(1 + 0.14\nu + 1.54\nu^2)x^2} - 0.87x^4 \quad (\text{A.6})$$

Inspection of this correction factor shows that its value is highly sensitive to the h/l ratio, being negligible for h/l small, and increasing with increasing h/l .

In the analysis of an international round-robin Wolfenden et al. (1989) ignored this term completely. In existing ASTM standards, C623 for glasses and C848 for ceramic whitewares, it is tabulated as a function h/l for various values of Poisson's ratio. In the proposed new ASTM Committee C28 standard for advanced ceramics, the correction factor is used in the form shown in Equation 6 to adjust iteratively the values of E and ν from measurements of flexural and torsional vibration, although its use has now been dropped for bars of $h/l \leq 0.05$ when only the first two terms are required. In the Japanese standard for ceramics, JIS R1602, it is truncated to the first two terms. There is therefore a considerable variety of approaches already employed in various standards, and in order to determine the most appropriate form to employ for the CEN draft standard, it was necessary to evaluate the correction function. It is clearly desirable to avoid having to employ the full Equation A.6 if a valid approximation can be developed that is accurate to within, say, 0.1%, which is smaller than the errors due to measurement of geometrical parameters.

Rewriting Equation 6 in the form:

$$A_f = 1 + A_1x^2 + \frac{A_2x^4}{A_3 + A_4x^2} + A_5x^4 \quad (\text{A.7})$$

the relative values of the terms of the sum for $\nu = 0.3$ are evaluated in Table A.3, This value of Poisson's ratio is possibly the highest likely to be determined for ceramics at ambient temperature.

Table A.3 - Evaluation of the terms of Equation A.7 for $\nu = 0.3$

$x = h/l$	0.01	0.02	0.05	0.1	0.2	0.5
Term 1	1	1	1	1	1	1
Term 2	7.212e-4	2.885e-3	1.803e-2	0.0721	0.288	1.803
Term 3	1.047e-7	1.672e-6	6.432e-5	9.75e-4	0.0166	0.283
Term 4	8.7e-9	1.392e-7	5.43e-6	8.7e-5	1.3e-3	0.054
Total, A_f	1.000722	1.00289	1.0181	1.0733	1.306	3.14

If the correction factor were to be completely ignored, in order to keep the accuracy of the of Young's modulus calculation to better than 0.1% it would be necessary to have h/l less than 0.01. Such a slender specimen would be practically difficult to control and would need rather low frequencies for driving. It is therefore necessary to employ the correction factor to at least the second term, and this would require keeping h/l to less than about 0.05, which is much more reasonable. Under this condition, the third and fourth terms can be dropped since their maximum contribution is only 7×10^{-5} for $h/l = 0.05$. For bars with greater h/l , the full correction factor becomes necessary to retain 0.1% accuracy.

The evaluation of A_f in Table A.3 is for $\nu = 0.3$. To check the evaluation for other values of Poisson's ratio, the value of A_f to the first two terms only is given in Table A.4.

Table A.4 - Evaluation of A_f for varying Poisson's ratio and $h/l = 0.05$

ν	0.1	0.2	0.3	0.4	0.5
Total, A_f	1.01671	1.01724	1.01803	1.01908	1.02040

It can be seen that the value of A_f (and hence the value of Young's modulus) is sensitive to Poisson's ratio to a level of about 0.1% over the range of expected values for the majority of advanced technical ceramics. The level of error might thus be significant if a Poisson's ratio factor is not included in the correction.

In conclusion, without loss of accuracy greater than 0.1%, we can write:

$$A_f = 1 + 6.58(1 + 0.08\nu + 0.8\nu^2)(h/l)^2 = 1 + B(h/l)^2 \quad (\text{A.8})$$

with an upper limit of error of 0.1% for h/l less than or equal to 0.05. For $\nu = 0.3$ this reduces to:

$$A_f = 1 + 7.21(h/l)^2 \quad (\text{A.9})$$

with an error of the order 0.1% for values of Poisson's ratio higher or lower by 0.1. The formula given in JIS R1602 is slightly different. It assumes no particular value of Poisson's ratio, and is given as:

$$A_f = 1 + 6.59(h/l)^2 \quad (\text{A.10})$$

In other words, this formula has used the assumption that $\nu = 0$, giving a systematic error underestimating E by about 0.15%, as shown by Table A.5. (Note that the factor 6.59 in Equation A.10 is obtained by upward rounding of Equation A.5, whereas Equation A.8 results from downward rounding.)

These errors must be compared with errors from other sources in making the measurement. These are summarised in Table A.6, where it can be seen that by far the largest potential error

is in the measurement of thickness, and in the parallelism of these faces. The physical limits of parallelism from surface grinding the test-pieces is probably similar to the measurement accuracy, so it is difficult to reduce the error by setting the parallelism to better than $h/100$ for $h = 1$ mm. Ideally, the error can only be reduced by increasing the beam thickness to, say, 5 mm, giving 0.6% error in E . To reduce this error further towards the same significance as other errors, would require a beam thickness of 10 mm, and with a ratio h/l of < 0.05 , this would require a test-piece of the somewhat impractical length of 200 mm (for advanced ceramics).

Table A.5 - Evaluation of B in $A_f = 1 + B(h/l)^2$ (Equation A.8)

ν	0	0.1	0.2	0.3	0.4	0.5
B (Eqn. 8)	6.580	6.685	6.689	7.212	7.633	8.159
A_f for $h/l = 0.05$	1.0165	1.0167	1.0172	1.0180	1.0191	1.0204
Difference: $A_f(\nu = 0.3) - A_f$	0.15%	0.13%	0.08%	0	-0.11%	-0.24%

Table A.6 - Measurement errors in flexural resonance

Source of error	Accuracy of measurement proposed	Magnitude of parameter	Percentage error In measurement In formula	
Mass, m	Weigh to 0.001 g	> 2 g	$< 0.05\%$	$< 0.05\%$
Length, l	Measure to 0.05 mm (callipers)	> 50 mm	$< 0.1\%$	$< 0.3\%$
Thickness, h	Measure to 0.01 mm (micrometer)	1 to 2 mm	$< 1\%$	$< 3\%$
Width, b	Measure to 0.01 mm (micrometer)	3 to 10 mm	$< 0.3\%$	$< 0.3\%$
Frequency, f_f	Measure to fourth figure in Hz	100 to 20000 Hz	$< 0.1\%$	$< 0.2\%$

In conclusion, therefore, if Equation A.8 were to be employed, the errors in the correction factor are insignificant, and the area for most attention is the precision of machining of the test-piece, its roughness and parallelism, and the measurement of its thickness in the direction of flexure.³

³ It has been agreed by Committee CEN TC184/WG3 that for CEN standard purposes, Equation A.10 will be used to maintain agreement with ASTM and JIS standards, but subject to the restriction that the length to thickness ratio is ≥ 20 .

A.2.3 Torsional resonance

There is closer agreement in the literature over the form of the equations to be employed for torsional resonance than for flexural resonance. In this case the relationship for a rectangular section is given by:

$$G = \left(\frac{4 l m f_t^2}{b h} \right) A_t \quad (\text{B.11})$$

where the geometrical factor A_t has been calculated by Thompson (1939) and by Spinner and Tefft (1961), and is reviewed by Pickett (1945) and Spinner and Valore (1958). The Thompson version as used in the draft ASTM C28 standard is:

$$A_t = \frac{(h/b) + (b/h)}{4(h/b) - 2.52(h/b)^2 + 0.21(h/b)^6} \quad (\text{B.12})$$

assuming $h < b$. Inspection of Equation A.12 shows that it is not insignificant for any value of h/b , and thus must always be employed. Table A.7 shows the effect of the possible deletion of the sixth power term.

This illustrates that the error is greater than 0.1% when h/b is greater than about 0.3. Although from the point of view of encouraging torsional resonance it is desirable to have h/b less than 0.2, to restrict the validity of the equation in this way may be inappropriate, especially for the impulse excitation technique. In addition, the term does not add greatly to the complexity of the correction term, unlike the flexural case.

Table A.7 - Torsional resonance correction term, deletion of sixth power term

h/b	0.05	0.1	0.2	0.5	1.0
A_t	103.51	26.948	7.437	1.8204	1.1834
A_t ignoring sixth power term	103.51	26.948	7.437	1.8248	1.3514
Error, %	0	0	0	+0.24	+14.2

The errors due to inaccuracy of dimensional measurements can be treated in a similar way to the longitudinal method. In this case, to estimate the errors in A_t due to errors in h and b , it is necessary to differentiate Equation A.12 with respect to h and b . Using the substitution $y = h/b$, Equation A.12 becomes:

$$A_t = \frac{y + 1/y}{4y - 2.52y^2 + 0.21y^6} \quad (\text{A.13})$$

Differentiating with respect to y gives:

$$\Delta \frac{A_t}{A_t} = \left\{ \frac{1 - 1/y^2}{1 + 1/y} - \frac{4 - 5.04y + 1.26y^5}{4y - 2.52y^2 + 0.21y^6} \right\} y \left\{ \frac{\Delta h}{h} - \frac{\Delta b}{b} \right\} \quad (\text{A.14})$$

in which ΔA_t is the error in A_t derived from errors Δh in h and Δb in b . This can be rewritten as:

$$\frac{\Delta A_t}{A_t} = H(y) \left\{ \frac{\Delta h}{h} - \frac{\Delta b}{b} \right\} \quad (\text{A.15})$$

In Table A.8, values of $H(y)$ and $\Delta A_t/A_t$ are tabulated in the second and third lines respectively for various h/b ratios for $\Delta h/h = \pm 0.01$, the value set as the required accuracy of measurement of the test bar thickness in the draft CEN standard. If the error $\Delta b/b$ is set to the same level, the total rms error (line 4 of Table A.8) will be $\sqrt{2}$ times the error due to $\Delta h/h$ alone. In practice, the error in $\Delta b/b$ is likely to be significantly less than $\Delta h/h$ in proportion to the shape of the test-piece, and for h/b less than 0.03 becomes insignificant. The data for the total error should thus be treated as pessimistic, and those in line 3 are probably more realistic.

It is clear from this Table that the error in A_t is strongly dependent on the ratio h/b , ranging from 2.6% for $h/b = 0.1$ to 0.1% at $h/b = 1$. It should be noted that the errors scale linearly with choice of $\Delta h/h$ and $\Delta b/b$. In Table A.9, the magnitude of this error is compared with other errors.

Table A.8 - Torsion resonance - determination of errors due to dimensions

h/b	0.1	0.2	0.3	0.4	0.5	0.8	1.0
$H(y) = H(h/b)$	-1.832	-1.656	-1.467	-1.266	-1.051	-0.384	-0.103
Error, $\Delta A_t/A_t$ ($\Delta h/h = 0.01$, $\Delta b/b = 0$)	-0.0183	-0.0166	-0.0147	-0.0127	-0.0105	-0.0038	-0.0010
Total rms error *	0.0259	0.0234	0.0207	0.0179	0.0149	0.0054	0.0014

* Assuming the measurement error is random and the total is the root of the sum of the squares of the individual equal contributions from $\Delta h/h$ and $\Delta b/b$.

Unlike the longitudinal case, Table A.9 shows that the consequent error in G will depend principally on the measurement of h (and b if not significantly greater than h) whether or not dimensions or density are used in Equation A.11, and will be of the order 1 to 2% for accuracies of measurement $h/100$ and $b/100$, but increasing with decreasing h/b . Improvement in the accuracy of G will depend on improving the accuracy of measurement of h and b (and

probably also the uniformity of h and b). If a conventional micrometer is specified for measurement of thickness, the accuracy will be limited to 0.01 mm, giving a minimum specimen thickness of 1 mm ($100 \times \Delta h$). Since the error in A_t falls in proportion with that of h , the error can only be made insignificant relative to other errors if the specimen thickness increases to 5 mm or more. As with the longitudinal and flexural cases above, the limitation on accuracy for small ceramic test bars remains controlled by specimen dimensions.

Table A.9 - Errors in measurement of torsional modulus

Source of error	Accuracy of measurement proposed	Magnitude of parameter	Percentage error	
			In measurement	In formula
Mass, m	Weigh to 0.001 g	> 2 g	< 0.05%	< 0.05%
Length, l	Measure to 0.05 mm (callipers)	> 50 mm	< 0.1%	< 0.1%
Thickness, h	Measure to 0.01 mm (micrometer)	1 to 2 mm	< 1%	< 1%
Width, b	Measure to 0.01 mm (micrometer)	3 to 10 mm	< 0.3%	< 0.3%
Frequency, f_t	Measure to fourth figure	200 to 50000 Hz	< 0.1%	< 0.2%
A_t	See Table A.8	1 to 100	up to 2.6 %	up to 2.6 %

A.3 CONCLUSIONS

From the above discussion of the factors affecting the accuracy of measurement and calculation equations for elastic moduli, it can be concluded that:

1. In general, the achieved parallelism and measurement accuracy of the test-piece are the principal sources of inaccuracy in making measurements of elastic moduli by resonance or impulse excitation techniques. Unless dimensions are very accurate and surface quality is excellent, the magnitude of these errors on small test bars generally outweighs any questions of the accuracy of the calculation equations.
2. If a target accuracy of the formula to be used is set at 0.1%, which is substantially smaller than most other errors of measurement, then the following conclusions concerning the equations to be employed are:
 - a) In longitudinal resonance, the correction term is unnecessary for length to diameter ratios of less than 20.
 - b) In flexural resonance, the complex correction factors cited in various standards or drafts can be safely truncated or ignored for simplicity of calculation. This situation is achieved when the length to thickness ratio is greater than 20. It is recommended that Equation 8 is used.

- c) In torsional resonance, while it is desirable to have a wide thin specimen for easy driving of resonance, curtailing the high power correction term is not especially useful, and would restrict the validity of the equations to wide thin beams. This may be inconvenient for the impulse excitation method.
- 3. This analysis allows the safe use of selected forms of the equations, and indicates preferred ranges for the geometrical shape of test-pieces.

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ANNEX B

ERROR ANALYSIS OF THE STATIC FLEXURE METHOD OF YOUNG'S MODULUS MEASUREMENT

[NOTE: This Annex was originally prepared as a discussion/demonstration document to answer questions arising in international voting on IEC 672-2 Specification for ceramic and glass insulating materials, Part 2 Methods of test during 1995, and is included here without significant modification because of its general nature.]

B1. Background

Following the discussion during IEC voting about this method, there is some confusion concerning the test-piece dimensional requirements. In particular, there is confusion concerning the proposed change from dimensions equal to those of strength test-pieces, to those of long, thin parallel-sided strips as defined in CEN ENV 843-2. It is considered that the existing 1980 version of IEC 672-2 using normal flexural strength test-pieces is likely to be inaccurate to a commercially significant level. Thus the redraft tightens the method to use thin test-pieces, which provide greater levels of flexural displacement that can be measured more accurately by direct test-piece deflection (proposed method (i)) or indirect machine displacement (method (ii)) techniques. In contrast, as is demonstrated below, errors associated with strain gauge methods are essentially independent of test-piece geometry.

B2. Error analysis

The three-point flexure equation is:

$$E = \frac{F l^3}{4 \delta b h^3} \quad (\text{B.1})$$

where F is the applied force, l is the test span, b is the width of a rectangular section test-piece, h is the height in the direction of flexure, and δ is the recorded displacement.

A root sum square error assessment can be defined by partial differentiation as:

$$\frac{\Delta E}{E} = \sqrt{\left\{ \left(\frac{\Delta F}{F} \right)^2 + 3 \left(\frac{\Delta l}{l} \right)^2 + \left(\frac{\Delta \delta}{\delta} \right)^2 + \left(\frac{\Delta b}{b} \right)^2 + 3 \left(\frac{\Delta h}{h} \right)^2 \right\}} \quad (\text{B.2})$$

In this expression:

$\Delta F/F$ is about 2×10^{-2} at best, based on the accuracy of calibration of loads applied by testing machines (typically 1%), plus errors involved in recording and reading off two load levels in the test;

$\Delta l/l$ is about 10^{-3} or 2.5×10^{-3} , respectively, for 100 or 40 mm span jigs, resulting from a span measurement accuracy of about 0.1 mm at best using a travelling microscope;

by comparison, the error in a (support to loading roller distance) is four times larger in quarter-point bending;

$\Delta b/b$ is about 10^{-3} or 2.5×10^{-3} in, respectively, 10 mm or 4 mm width test-pieces measured to an accuracy of 0.01 mm;

$\Delta h/h$ is about 10^{-2} for an accurately parallel-faced test-piece of thickness 1.0 mm, measured to an accuracy of 0.01 mm; this assumes the test-piece is uniform to this level, and this would normally require machining.

The largest errors are therefore those in F , and in h , especially when this latter parameter is raised to the power 3 in the calculation, and these act as a lower limit on the level of accuracy achievable, i.e. about $\pm 2\%$ with all other errors insignificant.

The error in $\Delta\delta/\delta$ is a result of the accuracy of measuring displacement. The error levels of $\Delta\delta/\delta$ and other parameter are interlinked, however, since reducing the thickness of the test-piece to increase the deflection and reduce the error in δ increases the error in h . Clearly, in order to retain the proposed accuracy target of $\pm 4\%$, the error in deflection measurement should be similar to, or preferably significantly less than, that contributed by $\Delta h/h$. Thus, a target figure for $\Delta\delta/\delta$ is $\leq 1\%$. We can calculate the accuracy requirements of each factor contributing to this error as follows. Turning equation 1 round gives:

$$\delta = \frac{F l^3}{4 E b h^3} \quad (\text{B.3})$$

The test method suggests that the maximum force applied to the test-piece should not exceed half the fracture stress, i.e. $0.5\sigma_f$ to avoid risk of breakage. The manual assessment method requires measurement of the displacement over the range 10% to 90% of the maximum force applied, so under such conditions, the maximum displacement achievable is given when the stress differential is equivalent to $0.4\sigma_f$. For three-point bending:

$$\sigma = \frac{3 F l}{2 b h^2} \leq 0.4\sigma_f \quad \text{or} \quad \frac{F l}{b h^2} \leq \frac{0.8\sigma_f}{3} \quad (\text{B.4})$$

Substituting this in Equation B.3 above gives:

$$\delta \leq \frac{0.067 \sigma_f l^2}{E b} \quad (\text{B.5})$$

Since $\sigma_f/E \sim 0.001$ for many ceramic materials, this gives:

$$\delta \leq 6.7 \times 10^{-5} \left(\frac{l^2}{h} \right) \quad (\text{B.6})$$

Evaluating this expression gives the maximum possible deflection given by any ratio of l^2/h . Long, thin test-pieces are therefore going to give much larger deflections than shorter, thicker

test-pieces. If $l = 100$ mm, and $h = 1$ mm, $\delta \leq 0.7$ mm, which is readily measurable with reasonable accuracy, say 1%, by transducer methods or by calibrating the machine displacement/chart-recorder displacement ratio and measuring from the chart. If $l = 40$ mm and $h = 1$ mm, $\delta \leq 0.11$ mm, thus requiring the sensitivity of measurement to be a factor 6.25 more sensitive. Similarly, if $l = 100$ mm and $h = 10$ mm for a standard porcelain-type bar, $\delta \leq 0.07$ mm, requiring yet more sensitivity and accuracy. Table B1 illustrates this for the various methods involved.

The implications of these levels for the three methods in the standard are now considered:

Method (i), displacement transducer recording directly the displacement between the centre of the test-piece and the support point.

Unless a particularly expensive and accurate device is employed, the resolution of displacement is typically $1 \mu\text{m}$, limited by mechanical stability, noise, temperature fluctuations, repeatability of positioning, etc. Assume that a voltage output is recorded on a chart recorder or other device. The linearity of the transducer is assumed to be 0.1% over its working range. Assume also that it is calibrated to an accuracy of 0.1% over its nominal working range, which is greater than the displacements measured on test-pieces, such that any given difference in voltages corresponding to a displacement can be converted to a displacement to this accuracy. This means that the total error in any one measurement of displacement is given by a combination of the calibration error, plus the reading errors at each position defining the displacement. Assume the reading error is likely to be similar to the resolution limit of $\pm 1 \mu\text{m}$. The total error is thus likely to be at least equivalent to $\pm 3 \mu\text{m}$ on any displacement measured, and this assumes that the systems is perfectly stable and reproducible mechanically. This minimum value has been used in Table B1. If the mechanical stability of the transducer/test-piece system is worse than this, the error will be larger. When this transducer error is divided by the displacement likely to be achieved, it can be seen that the error $\Delta\delta/\delta$ for thick test-pieces is the controlling part of the overall error. Thinner test-pieces as proposed by the redrafted standard have errors less than $\pm 4\%$. To eliminate the role of the transducer error for thick test-pieces, it is necessary to reduce it to typically 1% overall or better by use of a very high quality instrument and good engineering.

Therefore, thinner test-pieces with uniformity of thickness to better than 0.01 mm give greater deflections which are more readily measured to an accuracy of better than $\pm 4\%$. This is clearly better than attempting to perform the test on normal-thickness strength-test bars. Note also that the analysis for the thick bars has assumed that they are accurately flat and parallel faced to 0.01 mm. For porcelain materials this is unlikely to be achieved in the as-fired condition. If, as has been suggested, ± 0.1 mm on a 10 mm bar suffices, the error in thickness becomes a significant contribution to the overall error, and will increase it compared with the values in the Table.

Method (ii): recording the load displacement curve and correcting for test-machine stiffness.

The load/displacement curve is obtained by running a chart recorder at a known speed while the cross-head is moving at a much slower speed. In order to permit the actual displacement to be determined from the chart recording, the two rates need to be calibrated to obtain the

ratio. Let us assume that a dial gauge is used to measure machine cross-head displacement in the unloaded condition as a function of time. Assuming that the machine runs smoothly at its slowest rate, e.g. 0.05 mm/min, and that the displacement is recorded over a 20 minute period using a stopwatch of fractional accuracy better than 10^{-3} , the displacement achieved is 1.0 mm. This can be read to the nearest 0.01 mm, giving an accuracy of 1%. Similarly, the chart recorder can be timed running, say, 500 times faster. The distance in 20 minutes is 500 mm, which can be measured to an accuracy of 0.5 mm, or 0.1%. The magnification factor is thus known to an overall accuracy of about 1%. This assumes that both test machine and chart recorder behave in a consistent fashion. The practice requires several load cycles, and consistency must be achieved to minimise other potential errors due to machine factors.

Assume that load cycling is performed as specified in the standard, and positions on the chart traces are measured for the test-piece and for a large steel bar, the latter to determine jig and machine stiffness. The procedure requires an upper and a lower applied load to be selected, and the displacement over this range is read off. Assuming that the visual reading error at any one position on the chart is limited by chart pen width and noise to about ± 0.3 mm, the root sum of squares error for the four positions on the two traces (test-piece and steel bar) is ± 0.6 mm. The total error in converting the chart displacement to actual displacement is therefore this error plus the 1% error from the magnification factor. This is used to calculate $\Delta\delta$ in Table B.1 assuming a magnification of 500, from which $\Delta\delta/\delta$ is calculated. The overall error is then calculated using Equation B.2 for both three-point and four-point ($a = l/4$, quarter-point) bending. Increasing the magnification could be beneficial in reducing the size of the reading error in relation to the accuracy of magnification calibration.

It can be seen that in **three-point bending**, all cases except the 40 mm span x 3 mm thick test-piece have overall error levels of less than 4%, with the determining factor being measurement of thickness for the thin test-pieces, and displacement measurement in the thick test-piece. Relaxing the tolerances on the thickness of the thick test-piece to ± 0.1 mm makes this a significant error.

In **four-point bending**, the thicker test-pieces both give errors greater than 4%, with the accuracy of displacement measurement being the limitation.

Method (iii), strain gauges

In general, strain gauges are calibrated for use on certain types of material, e.g. steel or aluminium, and usually not for ceramics of rather different elastic properties. Claimed accuracies of measurement of strain are typically 1%, but an additional allowance of at least 1% should be made to compensate for use on other materials. In Table B1, an overall error of 2% has been assumed. The equation to be employed is:

$$E = \frac{3Fa}{bh^2\varepsilon} \quad (\text{B.7})$$

The total error is thus given by:

$$\frac{\Delta E}{E} = \sqrt{\left\{ \left(\frac{\Delta F}{F} \right)^2 + \left(\frac{\Delta a}{a} \right)^2 + \left(\frac{\Delta b}{b} \right)^2 + 2 \left(\frac{\Delta h}{h} \right)^2 + \left(\frac{\Delta \epsilon}{\epsilon} \right)^2 \right\}} \quad (\text{B.7})$$

which is different from Equation B.2 because different factors are involved. It will be noted from Table B.1 that the total error obtained is not sensitive to test-piece geometry, and the principal sources of the error are the applied load, the jig dimensions, and the strain gauge reliability. This is understandable, since geometrical errors are small. Built into this is the assumption that the strain gauge bridge system makes a negligible error contribution to the result.

B3. Round test-pieces

The same type of analysis may be performed for round bar test-pieces. The diameter of the test-pieces appears to the fourth power in the equation. Assuming that the bars are cylindrically ground to uniform diameter, and measured to 0.01 mm, then a 10 mm diameter bar will have an error $\Delta d/d$ which is the same as that for $\Delta b/b$ and $\Delta h/h$ for a square bar. The contribution to the overall error is the same. However, a round bar is a little less stiff than a square bar of the same lateral dimension, so the error in measuring deflection is slightly less. The overall error is thus similar to, but slightly less than that on square bars.

The strain gauge method cannot reliably be applied to round bars because of potential curvature errors.

B4. Conclusions

With regard to the issues raised during the international voting of IEC 672-2, the following conclusions can be drawn, using the basis of an estimate of minimum error contributions and combining these using the root sum square method:

1. Thin test-pieces with uniform thickness have smaller errors associated with mechanical measurement of their deflection than thicker test-pieces.
2. For the strain gauge method, the errors are essentially independent of test-piece shape or span, but are controlled primarily by the accuracy of load and strain gauge calibration.
3. Round bars give similar error levels to those for square bars, permitting similar conclusions to be drawn for these.
4. If an upper limit of 4% is placed on the acceptable total level of error in each method, thicker bars are generally ruled out with the exception of the strain gauge method.
5. It is considered that a relatively optimistic view has been taken of the potential errors associated with each parameter in the calculation. Error levels will increase significantly with:

- 5.1 Increasing the error in load measurement above $\pm 2\%$;
- 5.2 Increasing the error in loading arm (dimension a) measurement above ± 0.1 mm for 40 mm span, four-point bending as needed for the strain gauge method;
- 5.3 Measuring the test-piece thickness less accurately than to better than $\pm 1\%$, which also implies that the uniformity of thickness must be significantly better than this. Measuring the width less accurately has minor significance;
- 5.4 For the strain gauge method, increasing the overall error above $\pm 2\%$;
- 5.5 For the transducer method, increasing the overall error of measurement of a displacement (i.e. the difference between two positions) above ± 0.003 mm for all cases except the 100 mm span, 1.0 mm thickness test-piece, as a result of lack of mechanical stability and repeatability, non-linearity, and calibration errors;
- 5.6 For the chart displacement method, reducing the distance corresponding with the deflection on the chart, increasing the chart-reading uncertainty through mechanical or electrical noise, increasing the inaccuracies of measurement of the magnification factor, and having significant lack of reproducibility between load cycles as a result of machine bedding-down or lead screw effects.

Table B.1 - Minimum error analysis for static flexure testing

(Figures in bold are the controlling limits in reducing errors, except in the last column where they represent those figures > 4%)

Method	Dimensions				Minimum errors, %								Total error, $\sqrt{\sum(\Delta^2)_*}$	
	l	a	b	h	$\Delta F/F$	$\Delta l/l$	$\Delta a/a$	$\Delta \varepsilon/\varepsilon$	$\Delta b/b$	$\Delta h/h$	δ_{max} , mm	$\Delta \delta$, mm		$\Delta \delta/\delta$
3-pt, transducer method	100	-	10	10	2	0.1			0.1	0.1	0.067	0.003	4.5	4.9
				1.0	2	0.1			0.1	1	0.67	0.003	0.45	2.7
	40	-	4	3.0	2	0.25			0.25	0.3	0.036	0.003	8.33	8.6
				1.0	2	0.25			0.25	1	0.107	0.003	2.80	4.0
4-pt (1/4 point), strain gauges	100	25	10	10	2		0.8	2	0.1	0.1				2.9
				1.0	2		0.8	2	0.1	1				3.1
	40	10	4	3.0	2		2	2	0.25	0.3				3.5
				1.0	2		2	2	0.25	1				3.9
3-pt, chart method	100	-	10	10	2	0.1			0.1	0.1	0.067	0.00155 ¹	2.3	2.9
				1.0	2	0.1			0.1	1	0.67	0.00675 ¹	1.02	2.8
	40	-	4	3.0	2	0.25			0.25	0.3	0.036	0.00146 ¹	3.9	4.4
				1.0	2	0.25			0.25	1	0.107	0.00176 ¹	1.16	3.0
4-pt, chart method	100	25	10	10	2	0.1			0.1	0.1	0.033	0.00144 ¹	4.2	4.7
				1.0	2	0.1			0.1	1	0.33	0.00358 ¹	1.85	3.2
	40	10	4	3.0	2	0.25			0.25	0.3	0.018	0.00142 ¹	7.8	8.1
				1.0	2	0.25			0.25	1	0.057	0.00150 ¹	2.6	3.7

Key: *

1 Appropriate to the equation used to calculate the result. Figures in bold in this column are those > 4%.

Based on assumption that the error on the chart is a root sum of squares combination of ± 0.7 mm combined reading error, plus a $\pm 1\%$ error in chart displacement due to uncertainty in magnification factor, based on a magnification factor of chart speed : crosshead speed of 500.

