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Standardisation of Mechanical Testing and Quality Control

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1 INTRODUCTION

Testing in compliance with agreed standards is essential for trustworthy product release quality assurance (QA) and for providing design engineers and plant operators with a reliable database on which both the initial design of high-technology plant and its subsequent life-monitoring procedures can be based. Laboratory accreditation schemes, such as that organised in the UK by NAMAS (National Measurement Accreditation Service), go a long way to giving confidence in test data provided that such accreditation is based on National or International Standards. In the metals field, such Standards are well established for the majority of tests generally viewed as being necessary for characterising material properties, as shown in Table 1. However, in the ceramics field, nationally and internationally accepted standards for many of the fundamental material properties are yet to be agreed. The notable exception is bend testing where there appears to be a plethora of national standards which unfortunately are not mutually compatible (Table 2). Bend test methods have evolved to meet the requirements of various industrial sectors, such as ceramic whitewares, electrotechnical, electronic or orthopaedic ceramics, and were based on traditional practices, resulting in little commonality of approach. Only very recently has sufficient research been performed to analyse the critical features of testing such that test specifications can be considerably tightened.

The aim of this paper is to review briefly some of the standards used for metallic materials for simple tensile testing and uniaxial creep tests with a view to evaluating their suitability for extrapolation to the

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TABLE 1
Summary of High-Temperature Testing and Associated Standards for Metallic Materials

High temperature test description	*Material property*	*Symbol*	*Current testing standards*	*Associated calibration standards*	*Comments*
Tensile	Young's modulus	E	BS 3688	BS 1610, load	Compare with
	Upper and lower	R_{eH} & R_{eL}	ASTM E21	ISO	room temp.
	Yield strengths	R_e	DIN 50145	BS 3846 extensometers	standards:
	Proof strength	R_p	ISO DP783		BS 18
	Tensile strength	R_m			ISO 6892
	Fracture elongation (ductility)	A_t			
	Reduction in area	Z			
Creep (Uniaxial)	Rupture lifetime,	t_f	BS 3500	BS 3846 extensometers	Also shape of
	Creep rate,		ISO R203		curve,
	Ductility	ε	ISO R206		primary,
	Creep crack growth		ASTM E139		second and
			DIN 50118		tertiary
High cycle fatigue	No. of cycles to failure	N_f	BS 3518	BSI DD2	Load control
	S–N curve	$\pm$	ISO R373		
	Fatigue crack growth		ASTM E647		
Low cycle fatigue	No. of cycles to failure	H_f, T, IN or P	ASTM E606	BS 1610, load	Strain or load
	S–N curve	e	HTMT committee	BS 3846 extensometry	control
			Code of practice (NPL)	BS 3500 temperature limits	
Fracture toughness	Plane strain fracture toughness	K_{1C}	BS 5447		Tests based on
			ASTM E399		RT standards
	Stress intensity	K			
	Contour integral	C^*			
	Crack growth measurement	–			

Impact	Absorbed energy Transition temperature	J T_T	BS 131 ASTM E23	BS 1610 load	Charpy Isod New precision impact testing standard now drafted (BS)
Instrumented impact	Absorbed energy (area under load versus displacement trace)	J	No standards	BS 1610 load E23	Charpy
Hardness	Brinell	HB	BS 240 Pt 1 ISO R191	BS 240 Pt 2 ISO R156	
	Rockwell	HR	BS 891 ISO R80	BS 891 Pt 2	
	Rockwell (N&T)	HR	BS 4175 ISO R1079	BS 4175 Pt 2 ISO R1355	
	Vickers	HV	BS 427 ISO R192	BS 427 Pt 2	
Wear	Pin on wheel method, depth of groove for given number of revolutions		ASTM B611		
Thermal expansion	Coefficient of expansion		No standards		
Young's modulus		E	ASTM E231		Static methods only

TABLE 2
Summary of Existing Flexural Strength Tests for Ceramic Materials

Standard	*Ceramic type*	*Geometry*	*Specimen dimensions (mm)*	*Span (mm) 3-pt., 4-pt.*	*Loading rod diameter (mm)*	*Loading rod features*	*Loading conditions*	*Alignability of jig*
BS 4789	Microwave	Flexure, 3-pt.	120 × 10 ϕ extruded 120 × 10 × 10 pressed	100 100	10	Unspecified	> 30 s fail time	Unspecified
DIN 40685	Electrical insulators	Flexure, 3-pt.	A: 120 × 10 ϕ extruded B: 120 × 10 ϕ × 7 pressed, flatted circle	100 100	10	Unspecified	10–30 s fail time	Unspecified
IEC 672	Electrical insulators	Flexure, 3-pt. or 4-pt.	120 × 10 ϕ extruded 120 × 10 ϕ × 6–8 100 × 10 × 10 pressed 65 × 5 ϕ 35 × 3·5 × 3–5	100, 100/33 100 — 50, 50/17 25	10	Fixed	20–50 N s^{-1} loading rate	Unspecified
ASTM C674	Whitewares[a]	Flexure, 3-pt.	153 × 28·6, 19·2, 12·7 ϕ 95 × 6·4 ϕ 114 × 25·4 × 12·7	127 76 102	6·4	Rotatable	≃ 60 s fail time	Yes
ASTM F417	Electronic[b]	Flexure, 3-pt.	28·6 × 1·8 × 1·8 min.	25·4	6·4	Unspecified	19·4–26·4 MPa s^{-1} stressing rate	Yes
ASTM F394	Electronic[b]	Biaxial flexure, 3-ball and punch	30·2 ϕ × 1·5–3	25·4 ϕ	1·6 (punch) 3·2 (balls)	Unspecified	19·4–26·4 MPa s^{-1} stressing rate	No, uses pad under punch
JIS 1601[c]	Engineering	Flexure, 3-pt., 4-pt.	36 × 4 × 3 chamfered	30, 30/10	4–6	Unspecified	0·5 mm min^{-1} cross-head speed	No
MIL-STD-1942	Engineering [a] or [b]	Flexure, 3-pt., 4-pt.	A: 25 × 2 × 1·5 B: 45 × 4 × 3 C: 90 × 8 × 6 (all chamfered)	20 40, 40/20 80, 80/40	2·5 4·5 9·0	Rotatable	0·2 mm min^{-1} 0·5 mm min^{-1} 1·0 mm min^{-1} cross-head speed	Partial for machined yes for as-fired
ISO 6474	Orthopaedic alumina	Flexure, 3-pt.	30 × 4·5 × 4·5 (waisted shape allowed)	25	2–4	Rotatable	10 MPa s^{-1} stressing rate	Yes

[a] Specimen finish to be 'application-matched'.
[b] Specimen finish specified.
[c] JIS 1604 under development for use at elevated temperatures using same basis.

stress/temperature regimes required for testing engineeering ceramics and ceramic composites. In addition, the existing tests used for characterising ceramics are reviewed, and recommendations are given for future standards suitable for acquiring reliable design data or for QA testing.

2 APPLICABILITY OF METALS STANDARDS TO CERAMICS

Accepting the premiss that measurement and testing standards are necessary to enable the comparison of data from different sources, the question arises as to what is the minimum permitted tolerance on the specified testing parameters that can be regarded as being acceptable?

It must be recognised that for any materials database, scatter in the results is invariably attributable to two sources: inherent material scatter due to batch-to-batch variations in the production process of the material, whether it be a metal alloy, monolithic ceramic or composite; and that due to tolerances in the specified testing parameters, which may also include human errors. From a pragmatic point of view, there is little merit in striving for the ultimate accuracy in testing parameters if the inherent scatter in the material being tested is an order of magnitude greater. Therefore, it is important to choose appropriate achievable accuracy for the tolerances on testing parameters whilst still ensuring that the cost of carrying out any specific test is kept to a minimum. It should be noted that any test should be designed with the 'fitness-for-purpose' concept in mind; hence the accuracy of a test developed to produce design data may be considerably greater than that required for a pass/fail QA test.

In most tests, the worst scatter attributable to errors in the testing method is the cumulative sum of the errors due to the individual parameters contributing to the desired measurement. It should be noted that the theoretical treatment of errors is a complex subject which has been dealt with more fully elsewhere (Campion *et al.*, 1980). However, for the purposes of this contribution, it is instructive to examine the tolerances accepted in the present standards for tensile and creep testing of metallic materials.

2.1 Tensile test standards

The permitted strain rate ranges for ambient and elevated temperature tensile testing of metals are shown in Fig. 1. Also shown is the typical strain rate range normally used for testing ceramics. This range is only slightly wider than that specified in ISO DP 783 for elevated temperature testing of metals. For the sake of harmonisation it would perhaps be sensible in future

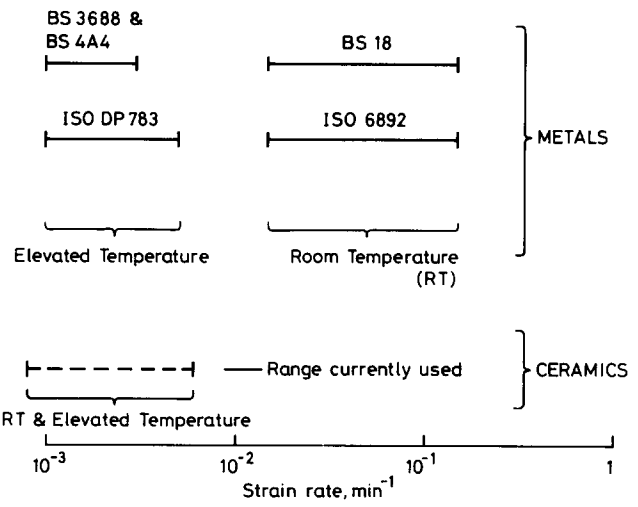

Fig. 1. Strain rates for proof tests at ambient and elevated temperatures for metals compared with conventional practice for flexure testing of ceramics.

to restrict the strain rate range for ceramics testing, whether in tension or flexure, to that specified in ISO DP 783.

The importance of harmonisation with the ISO Standards should not be underestimated, since where appropriate ISO Standards exist they are being adopted as drafts for the new European Standards. When the latter are published they will supersede the equivalent national standards of the member nations of the European Community.

The permitted errors in temperature measurement from all sources for the British and ISO Standards for elevated temperature testing are illustrated in Fig. 2. It may be seen that for temperatures above 1000°C there are no specified tolerances on temperatures other than those agreed between the contractor and the customer. It is difficult to monitor accurately the temperature of a testpiece above 1000°C, particularly on ceramic specimens where physical contact of noble metal thermocouples may cause premature failure (Loveday & Evans, 1988). Thus, despite improvements in furnace temperature controllers over the past few years, it is probably not practical to expect the testpiece temperature to be maintained accurately over its entire gauge-length to better than ± 8 K or even ± 10 K at temperatures between 1000°C and 2000°C, compared with the desirable upper limit of ± 6 K discussed in section 2.2.3 below. (These accuracy levels included possible errors from all sources.) This topic is considered in detail elsewhere (Buddery, 1988).

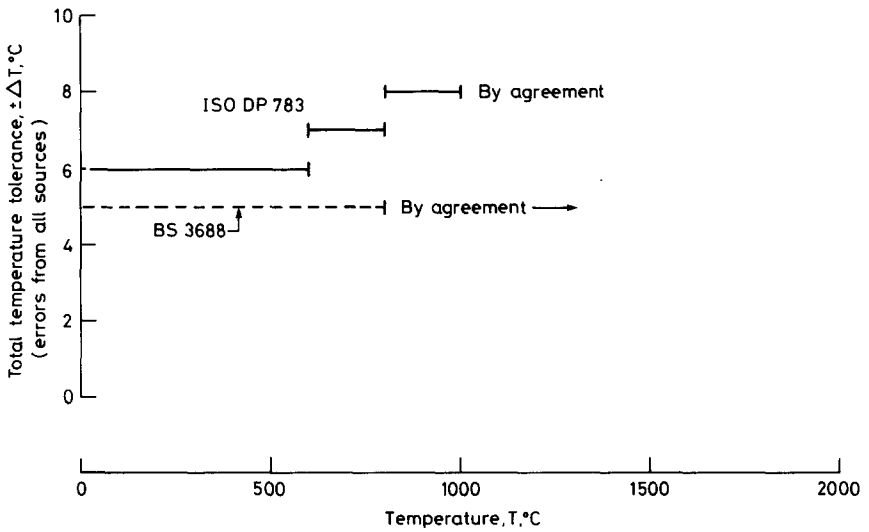

Fig. 2. Total temperature tolerance from all sources as a function of test temperature in existing standards for testing metals.

2.2 Creep standards

In the case of creep of most metallic materials, the minimum creep rate $\dot{\varepsilon}_{min}$ may be related to the applied stress σ and the temperature T over limited stress ranges by a relationship having the form:

$$\dot{\varepsilon}_{min} = A\,\sigma^n \exp(-Q/RT) \tag{1}$$

where n is the stress index, Q is the activation energy and R is the gas constant. Assuming for a given material that the fracture life t_f is directly proportional to the inverse of the minimum creep rate, it can be seen that errors in t_f or $\dot{\varepsilon}_{min}$ are due to errors in σ and in T in the two separate components of eqn (1).

It is necessary to specify some arbitrary criteria for desired accuracy, and over the years in the field of metals, experience at NPL has shown that the determination of minimum creep rate to within a factor of two (i.e. $\pm 50\%$) is as good as can be achieved under normal advanced laboratory testing conditions. If half of this scatter is assumed to be due to batch-to-batch variations in the material, then the total scatter due to variations in the experimental test parameters may not exceed $\pm 25\%$.

2.2.1 Stress tolerance

The allowable tolerances in BS 3500 and the proposed ISO Creep Standard for measurement of area and force are shown in Fig. 3(A and B) respectively.

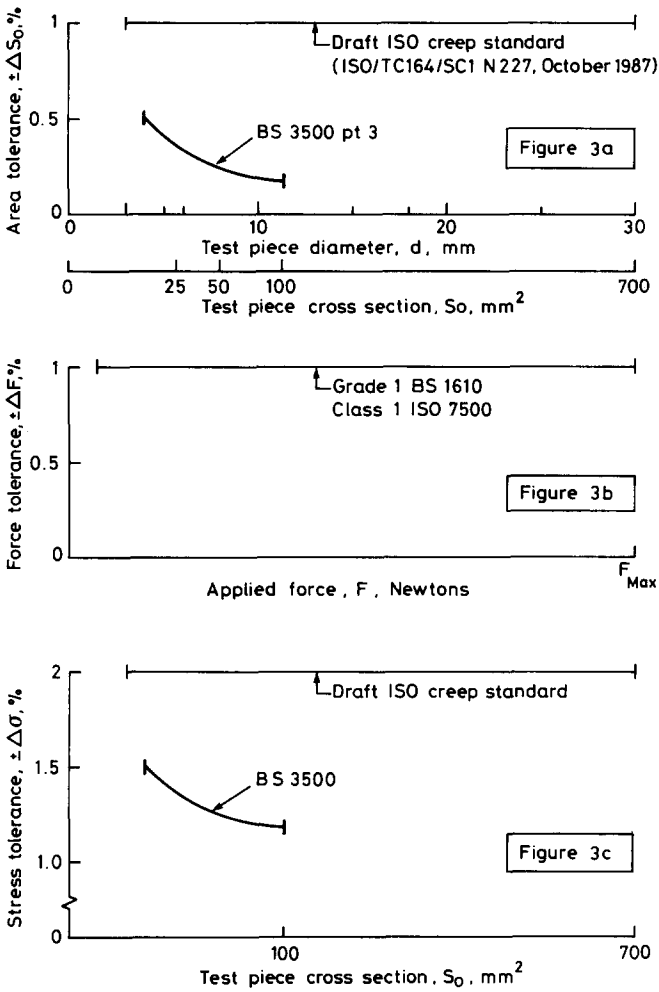

Fig. 3. The requirements in BS 3500 and the Draft ISO standard for creep testing of metals on (A) area measurement, (B) force measurement, and (C) stress measurement (combining (A) and (B)).

These two measurement tolerances combine to give the permitted maximum tolerance on stress as shown in Fig. 3(C).

In eqn (1) it was seen that the applied stress σ was raised to the power n. The error tolerance $\Delta\sigma$ should therefore be multiplied by n. The expected scatter in creep rate or fracture lifetime for various values of $\Delta\sigma$ plotted as functions of n are shown in Fig. 4. For materials with a low stress index, e.g. ceramics with $n \simeq 3$ or less, it can be seen that whilst complying with $\Delta\sigma = \pm 2\%$ the expected error in creep rate is only $\pm 6\%$ or less, whereas for metals n may be considerably larger and, for example, if $n = 10$, the error will be $\pm 20\%$.

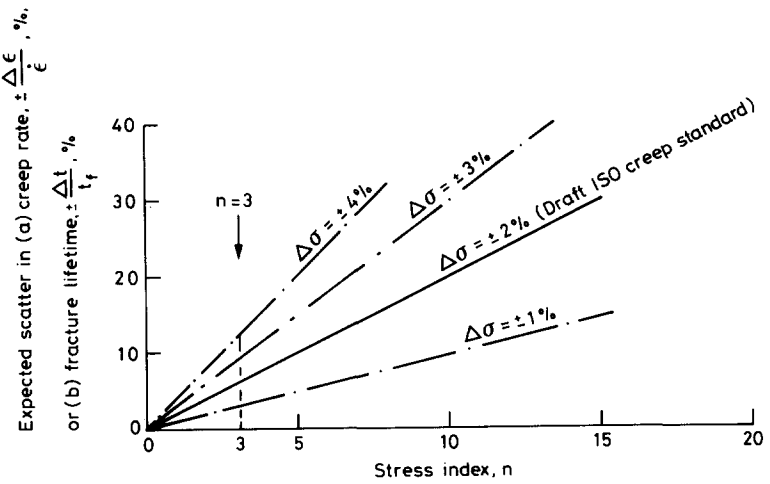

Fig. 4. Scatter in creep rate or time to failure as a function of creep stress index for various levels of error in applied stress.

In practice it is fairly easy to maintain the applied load to within $\pm 0{\cdot}5\%$ and also to determine the testpiece cross-sectional area to an accuracy of better than $\pm 0{\cdot}5\%$ using properly calibrated toolmaker's micrometers. Thus, even if n is as high as 10 it should be possible to restrict the expected uncertainty in $\dot{\varepsilon}_{min}$ of t_f to less than $\pm 10\%$, while for ceramics with $n = 3$, the uncertainty should be less than $\pm 3\%$.

2.2.2 Temperature tolerance

Variations in test temperature T influence the accuracy of the creep rate as given in the exponential term of eqn (1). For a variety of high-temperature materials, including hot-pressed silicon nitride (HPSN), Fig. 5 shows the expected error in minimum creep rate if tests are carried out above or below the nominal temperature by different values of ΔT, assuming representative values of creep activation energies. It can be seen that the higher the service temperature the less the effect of a given value of ΔT.

It is instructive now to consider the permitted temperature tolerances in the relevant creep standards. For the sake of clarity only two standards have been illustrated in Fig. 6(A), namely those in BS 3500 for creep tests lasting more than 100 h, and those in the proposed ISO Creep Standard. A full comparison of the testing parameters for all creep testing standards is given elsewhere (Guest, 1982). It should be noted that the tabulated values in the British Standard include errors from all sources whereas in the draft ISO Standard, allowance has been made for the specified resolution and accuracy of the measurement system. The effect of these permitted temperature tolerances as a function of test temperature is shown in

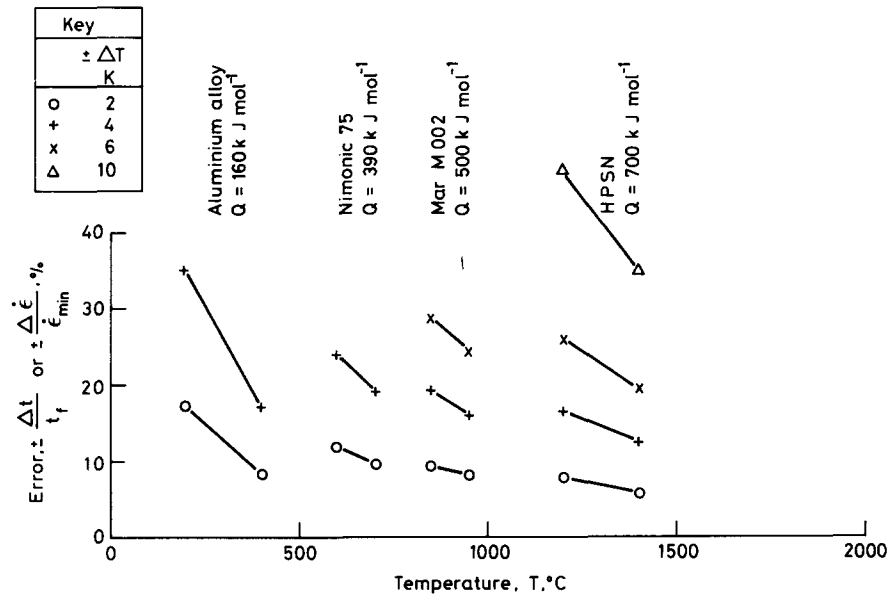

Fig. 5. Scatter in creep rate or time to failure as a function of temperature due to various levels of error of temperature for three types of metal alloy and hot-pressed silicon nitride, assuming typical creep behaviour.

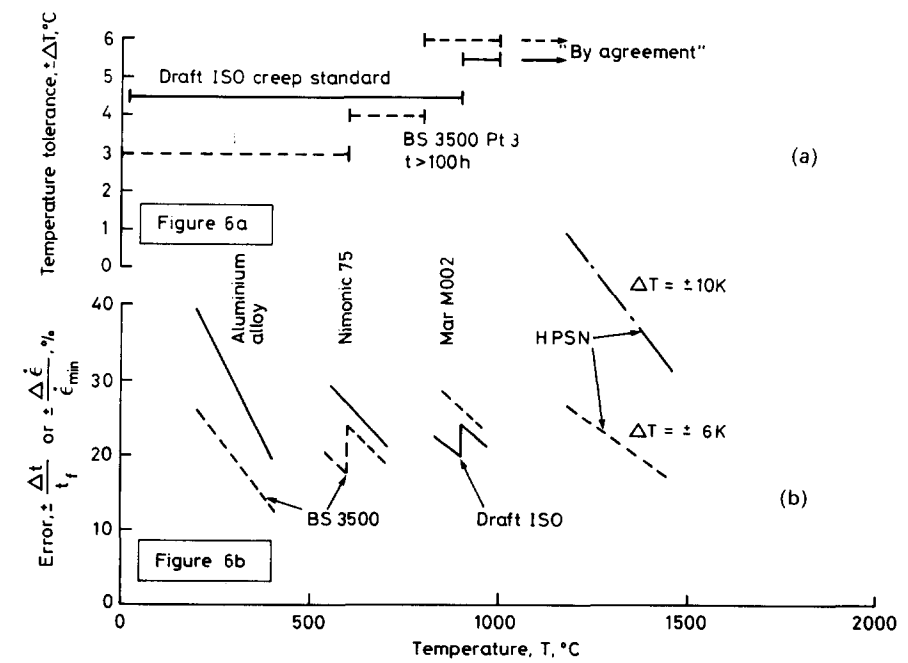

Fig. 6. Acceptable scatter in creep rate or time to failure (B) given in standards for metals in terms of temperature tolerances (A), compared with that suggested for HPSN.

Fig. 6(B), where it can be seen that scatter of approximately $\pm 25\%$ is expected whilst testing within the tolerances specified.

2.2.3 Total uncertainty in $\dot{\varepsilon}_{min}$ or t_f

The total uncertainty in $\dot{\varepsilon}_{min}$ or t_f is the sum of the uncertainties in stress and temperature. In the case of ceramics with $n \simeq 3$ it was shown in Fig. 4 that if the ISO criterion of $\Delta\sigma = \pm 2\%$ was met, then the expected scatter in $\dot{\varepsilon}_{min}$ or t_f would be $\pm 6\%$, and if the greater precision of $\Delta\sigma = \pm 1\%$ was achieved, then the scatter would reduce to $\pm 3\%$. In the worst case, this stress uncertainty should simply be added to that contributed by temperature tolerance ΔT. For a ceramic material, it can be seen in Fig. 6(B) that the uncertainty in $\dot{\varepsilon}_{min}$ or t_f is about $\pm 20\%$ $\Delta T = \pm 6$ K, or about $\pm 40\%$ if ΔT is ± 10 K. Thus in order to come within the limits currently allowable by metal standards, temperature uncertainties due to factors such as temperature inhomogeneity (especially gradients due to the use of cold gripping), temperature control fluctuations, and the position, calibration stability and reading errors of thermocouples must be limited to about ± 6 K up to say 1500°C. This could pose practical difficulties, especially for long-duration tests. However, for short-duration tests, tighter temperature accuracies have been reported (Kandil & Dyson, 1988). Note that this analysis has ignored additional difficulties associated with failure at very small strains due to lack of ductility or with alignment problems during testing.

2.3 Strain measurement

The problems associated with strain measurement are discussed elsewhere in this book (Liu, 1988), but it is essential that extensometry should be capable of achieving the appropriate accuracy for the desired measurement, and should be verified by calibration in accordance with the national and international standards. Since ceramics will, in general, have low ductilities, it is recommended that extensometry should have an accuracy better than 0·5% corresponding to a 0·5 grade device in the ISO Standard (or Grade B in the British Standard). The differences in the methods of calibrating and grading extensometers between the British and the ISO Standards have been reviewed elsewhere (Loveday, 1986).

3 MECHANICAL TEST STANDARDS FOR CERAMICS

Most laboratories involved in the testing of ceramics have adapted test methods of a somewhat empirical nature established for clay-based materials such as porcelains and whitewares. Only in recent years has there

been a move towards tests specially devised for high-strength structural ceramics. Because of the inherent lack of ductility of ceramics, it has been the norm to test them in flexure or in indirect tension (e.g. internal pressurisation of a ring) rather than direct tension in order to avoid uncertainties associated with poor alignment. However, flexure testing has severe limitations in terms of producing design data. Failure at or near surfaces or edges is emphasised, so the preparation of the testpiece becomes a controlling feature of the result. Volumes under stress are usually small, and therefore do not necessarily encompass a scattered population of serious defects or flaws that might define the performance of a component. Four-point bending is clearly an improvement over three-point bending, but a tension test would be much more searching for more serious internal defects. As a result, mean tensile strengths are typically 50–70% of the nominal flexural strength of a testpiece of the same size. Such lower figures are usually considered much more appropriate for design purposes. Nevertheless, it is considered that well-designed flexure tests are a very convenient tool for QA, especially as a large number of testpieces usually need to be examined in order to establish the statistical data often required with ceramics.

3.1 Short-term strength tests

Short-term strength tests (Table 2) are based exclusively on flexure, and this is usually in uniaxial bending. Only ASTM F394 is concerned with biaxial testing. The objective of these tests is to give some indication of basic material quality. The lack of definition of a number of test parameters, such as load-train alignment, elimination of friction and surface preparation, implies that the test methods are open to interpretation, and that the results from different sources are not directly comparable. A number of improvements in test methodology are required, but it is becoming clear that with attention to jig design, more-consistent results can be obtained than is usually the case. In this respect, US MIL-STD-1942 for flexure testing is a major advance in test practice.

The data derived from flexural tests cannot be used in a statistical sense to make performance predictions for components that are of significantly larger dimensions, or made in a different way, or with a different surface finish. A clearer idea of component performance may be obtained from tension tests, but at present the difficulties associated with testing reliability have precluded standardisation of any tensile methods. Recent work by Ohji (Paper 8) indicates, however, that standardisation may be possible.

Taking a pragmatic view, one might argue that even if tensile test data were to be obtained, there could be sufficient differences between the defect populations in tension specimens (including both bulk and surface defects)

and components so that short-term fracture data would be of little value. It is probably more important to evaluate the longer-term behaviour in service environments and to design the component to minimise peak tensile stresses and stress concentrations. Direct tensile tests at ambient temperature may therefore be an expensive way of testing specially prepared testpieces. It is felt that emphasis is better placed on ensuring that components incorporating shape-dependent processing defects are adequately strong. It may be appropriate to test for component quality by cutting out and testing small but representative flexural testpieces. This argument may not be true for fibre composites because such materials are much less defect sensitive and generally behave rather differently in tension and flexure.

3.2 Short-term tests at elevated temperatures

The simplest test to employ is a bend test, which can be used as a QA tool in the same way as at room temperature. JIS 1604 (currently in draft form) is the first procedure to appear, and this is basically an extension of JIS 1601 (Table 2) designed for use at room temperature. Clearly it is advantageous to employ the same jig geometry, testpiece dimensions and alignment techniques as employed for room-temperature testing. Simple test jigs constructed from silicon carbide are in fact available commercially to meet the design requirements of JIS 1601 and MIL-STD-1942 for room-temperature testing.

If the material behaves in an essentially elastic fashion, the usual equations used to calculate stress and strain in flexure specimens are valid. If testing is performed under the same conditions of loading rate as for room temperature tests, the effect of temperature on short-term strength as defined by the basic material and/or its surface finish is clearly demonstrated. For QA purposes, such tests would appear perfectly adequate. However, if quantitative data are required on inelastic behaviour at high temperatures, this requires tensile testing in order to obtain accurate information. The exact stress distribution in a flexure specimen becomes unknown and can result in the actual surface stress being significantly less than the nominal 'elastic' stress, and the performance becomes seriously overestimated. For QA purposes this may not be a serious problem because if significant deformation occurs in a short-term test, the material becomes unsuitable for most stressed applications at that temperature.

3.3 Delayed failure tests

Statically loaded ceramic specimens may show some form of delayed failure in the absence of inelastic deformation processes (commonly referred to as

static fatigue) at all temperatures. The external environment may exert a strong influence on performance, and at high temperatures the rate of oxidation of non-oxides can control the propagation of surface defects. The effect may be negligible in some materials, notably silicon carbide at low temperatures. To characterise behaviour, it will become essential to develop a means of quantifying the effects of surface finish, environment and temperature on tendency to delayed failure for both QA and design data purposes.

In most high-quality ceramics, internal defects are likely to be less susceptible to subcritical crack growth processes than surface or near-surface defects which may be strongly influenced by the environment. It is therefore felt that flexure testing will be a sufficient indicator of performance for QA purposes, since an adequate surface area can be tested. However, for design data, this may not be satisfactory because of differences in defect populations between test bars and components. Tension tests have similar shortcomings, so one concludes that, as with short-term strength tests, the choice of test will be dictated by cost and simplicity, i.e. flexure will be preferred, but lifetime analysis will require component tests.

3.4 Creep tests

There have been a number of attempts in the technical literature to evaluate flexural creep in terms of separate tensile and compressive parts. Mostly, some assumptions of a consistent mathematical relationship between tension and compression creep are made, but in the general case, and especially during the early stages of a flexural creep test, the neutral axis of the test bar moves towards the compressive side in an indeterminate way. From the design point of view, the separate tension and compression creep performances must be determined, and thus direct uniaxial, perhaps even biaxial, creep tests are needed. However, from a QA standpoint, flexure testing will give some guidance as to material properties (Ernstberger *et al.*, 1986; Quinn, 1986, etc.) in terms of temperatures at which creep occurs under given loading conditions, even if creep rates, stresses and estimated stress dependences of creep rate are in error by a considerable margin.

Stress-rupture under creep conditions similarly requires tension testing for the acquisition of design data. Cracks are likely to initiate from both internal defects and from surfaces, requiring the testing of significant volumes of material under uniform tensile stress.

3.5 Fatigue tests

Like metallic materials, the performance of a ceramic in cyclic fatigue is influenced by the cycle type and the external environment. It has been shown

(Evans & Fuller, 1974; Fujita *et al.*, 1986) that low-cycle, tension–tension fatigue approximates to the integrated effect of the tensile stress in a reasonably predictable manner. However, high-cycle tension–tension fatigue and reverse-cycle fatigue have been studied very little. There are some indications of frequency dependence, and that the integrated effect is no longer valid (e.g. Krohn & Hasselman, 1974). Crack growth may occur even in the compressive part of the cycle (Ewart & Suresh, 1987), and this has been proposed as a method of propagating controlled cracks for fracture toughness testing. At present there is a lack of understanding of the limitations of material behaviour, no standardised tests, and very few data. Cyclic flexure tests and rotating-bending tests have been attempted, but often the number of test points are insufficient to give a clear picture of the fatigue process, which in these cases will be defined predominantly by surface defects and their interaction with the test environment. Uniaxial push–pull tests may be preferable, and a few systems for loading in this way have now been developed and tested (Amaral & Pollock, Paper 4; Soma *et al.*, Paper 15, this volume). Clearly this is an area where more reserach should be directed towards understanding mechanical behaviour before any move is made towards standardisation.

3.6 Elastic moduli

There already exist a number of standards for the determination of elastic properties, based on flexure, ultrasonic time of flight or resonance (Table 3).

TABLE 3
Summary of Existing Tests for Determination of Elastic Moduli of Ceramics

Standard	*Ceramic type*	*Test type*	*Specimen dimensions (mm) and other factors*
ASTM C848	Whitewares	Resonance (flexural & torsion)	125 × 15 × 6 or 125 × 10–12 ϕ
DIN 40685	Electrical insulators	Flexure, 3-pt.	Long rods or tubes (dimensions not fixed)
		Ultrasonic	None specified
		Resonance	None specified, reference to scientific paper only
IEC 672	Electrical insulators	Flexure	As for strength testing (see Table 2)
JIS 1602	Engineering ceramics	Flexure, 3-pt. or 4-pt.	35–85 × 4 × 1 minimum, span 30–80, loading rods 4–6 ϕ fixed
		Resonance	40 × 5 × 1 minimum
		Ultrasonic	10 × 10 ϕ

These generally appear to produce quite reliable results, suitable both for design data and for QA. Care must be taken when testing at elevated temperatures that the ultrasonic or resonance methods do not overestimate the elastic stiffnesses because of the very short stressing period compared with static loading tests under conditions where plastic deformation can occur in the time-scale of loading. Care is also required to ensure that anisotropic materials are correctly characterised.

4 CONCLUSIONS

4.1 Tests for quality assurance

4.1.1 Basic material properties

Mechanical quality of a ceramic can be assured by flexure testing under standardised conditions. The effects of particular environments, machining procedures, fabrication routes and test temperature can all be assessed adequately to compare performance against a specification. This is particularly the case if the testpieces are machined from components, or are made in the same way as components. However, the data so obtained may not reliably be used for design. In addition, flexure tests will not always ensure that a component is fit for service in terms of survival under design loads, because for many materials it may be almost impossible to match adequately the defect distribution of a component, even if the testpiece is machined from a component.

4.1.2 Component quality

Apart from simple checks such as visual inspection, density measurement and dye penetration tests, which can be considered as the final stages of the manufacturer's quality control procedures, only defects which are larger than about 50 μm can presently be detected with any certainty by non-destructive methods. Even when this is done there are no guarantees that the component is of adequate mechanical quality. Mechanical proof tests are the only means currently available to eliminate unacceptably weak members of a batch. However, considerable care must be taken to ensure that the test is meaningful, and genuinely eliminates weak components without unacceptably weakening those that pass. For example, proof tests for thermally-loaded components are difficult to perform because temperature distributions are not readily modelled. The same applies to many mechanically-loaded situations. Proof tests are totally inadequate if the service conditions modify the defect population, such as occurs during oxidation, corrosion, impact and wear. All the test would demonstrate is that the component as

supplied has a degree of initial quality. This may be enough for contractual purposes, but is not a guarantee of service life.

Sampling for destructive testing is the only alternative, and may allow for assessment of the statistics of component quality, but it gives only a probability of failure for the remaining population. Under service conditions it cannot guarantee each component.

4.1.3 Ceramic fibre composites

The above discussion applies to monolithic ceramic components, but ceramic fibre and to a lesser extent ceramic/whisker composites present different problems (Davidge & Davies, Paper 19). The strength characteristics are determined by factors associated with the fibre/matrix distribution and the nature of the fibre/matrix interface. Destructive testing of a sample component population may be the only method of assessing average batch quality. Test methods, especially for long fibre composites will need to be radically different from those for monolithic ceramics to take into account the failure modes, such as shear failure in flexure testing. It may be necessary initially to base testing on experience with organic matrix materials such as glass- or carbon-fibre reinforced plastics, and it remains to be seen whether testing at very high temperatures poses problems of grip design for wide strip specimens or panels.

4.2 Tests for acquisition of design data

From the above discussion, it is not clear what constitutes design data for brittle materials. Neither direct tension, nor biaxial nor uniaxial flexure tests can at present adequately describe the performance of components under elastic conditions because of practical factors affecting the consistency of defect populations in test specimens compared with components. Therefore, there seems little point in attempting to develop short-term tensile strength tests that will inevitably be expensive and time-consuming to perform on an adequate and standardisable basis. Similarly, for long-term testing under static load where environment is a major factor, a tension test will probably reveal no more than a flexure test. However, uniaxial tension and/or tension/compression testing *is* required for fatigue and creep data in order to avoid uncertainties associated with stress distributions in flexure. Such tests need careful experimental control, as the discussion in section 2.2 demonstrates, particularly with regard to temperature distribution, control and measurement, and to axial alignment to avoid stress inhomogeneity. In order to attain the accuracy typically defined in accepted standards for metals it will be necessary to be particularly vigilant with furnace design. For example, the trend towards using short specimens (because of size availability limitations) with cold grips will pose increasing problems of

TABLE 4
Relative Importance of Creep Testing Parameters on Test Reproducibility for Metals and Ceramics

Factors influencing measurements	*Relative importance[a] for:*	
	Metals	*Ceramics*
Alignment	M	H
Surface machining	L	H
Internal defects	L	H
Oxidation (material dependent)	L–M	M–H
Temperature accuracy	H	H
Load accuracy	M	M
Loading rate	L	M (avoid shock loading)

[a] L = low, M = medium, H = high.

temperature inhomogeneity as temperatures are raised. In addition, the alternative method of hot-gripping may pose severe limitations in obtaining and retaining good alignment (Amaral & Pollock, Paper 4). These and other factors, summarised in Table 4, will pose considerable demands on the quality of testing that must not be ignored.

5 RECOMMENDATIONS

From the discussion in this paper, the authors make the following recommendations for test practices which should be standardised for testing of ceramic materials.

(1) For QA purposes, flexural testing jig design, specimen preparation conditions and test practice should be fully standardised with a limited number of options on specimen size. These factors are to be kept the same for testing over long time periods and at higher temperatures.
(2) For acquisition of design data, direct uniaxial tension testing procedures should be developed for creep testing and possibly for fatigue testing with limitations on the temperature distributions to give data accurate in strain rate or time-to-failure terms that are currently accepted in standards for testing metals. A summary of recommended parameters for testing are given in Table 5.
(3) Testing short-term strength or delayed failure of monolithic ceramics for design data is unlikely to be cost-effective if performed in uniaxial tension, nor will it necessarily produce a result more valid than

TABLE 5
Recommended Parameters for Testing Ceramics

Parameter	*Recommended values*	*Applications*				
		Room temp.		*High temp.*		*Uniaxial creep*
		Tension	*Bend*	*Tension*	*Bend*	
Load accuracy	Better than ±1% (BS 1610, grade 1)	X	X	X	X	X
Axial alignment	Off-axis bending <1%	X	[a]	X	[a]	X
Strain rate	0·001–0·005 min^{-1}	X	X	X	X	—
Temperature tolerance from all sources	$\Delta T = \pm 6$ K, $1000 < T < 1500$°C			X	X	X
	$\Delta T = \pm 8$ K, $1500 < T < 2000$°C	—	—	X	X	X
	$\Delta T = \pm 10$ K, $T < 2000$°C	—	—	X	X	X
Specimen dimensions[b]						
Uniaxial	$l/d > 5$	X	—	X	—	X
Bend, 3-pt	40 mm span, $a \simeq 1{\cdot}5b$, $b < L/8$	—	X	—	X	—
Bend, 4-pt	40 mm span, 20 mm load span $a \simeq 1{\cdot}5b$, $b < L/16$	—	X	—	X	—
Surface finish	To be specified, or application matched. Edges of bend specimens to be chamfered.	X	X	X	X	X

[a] There are self-aligning jig geometries which should be employed to minimise errors in alignment which are otherwise difficult to measure and specify.
[b] Key: l, d = length and diameter of gaugelength in tension specimens; L = support span in 3- or 4-pt bending; a = width; b = depth. See Table 2 for details of dimensions for existing tests.

flexure testing on ceramics in which the strength is limited by the quality of the surface finish.

(4) Flexure testing will give an adequate indication of the influence of factors such as grinding procedures, oxidation, corrosion, fretting and wear on strength as defined by surface finish.

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