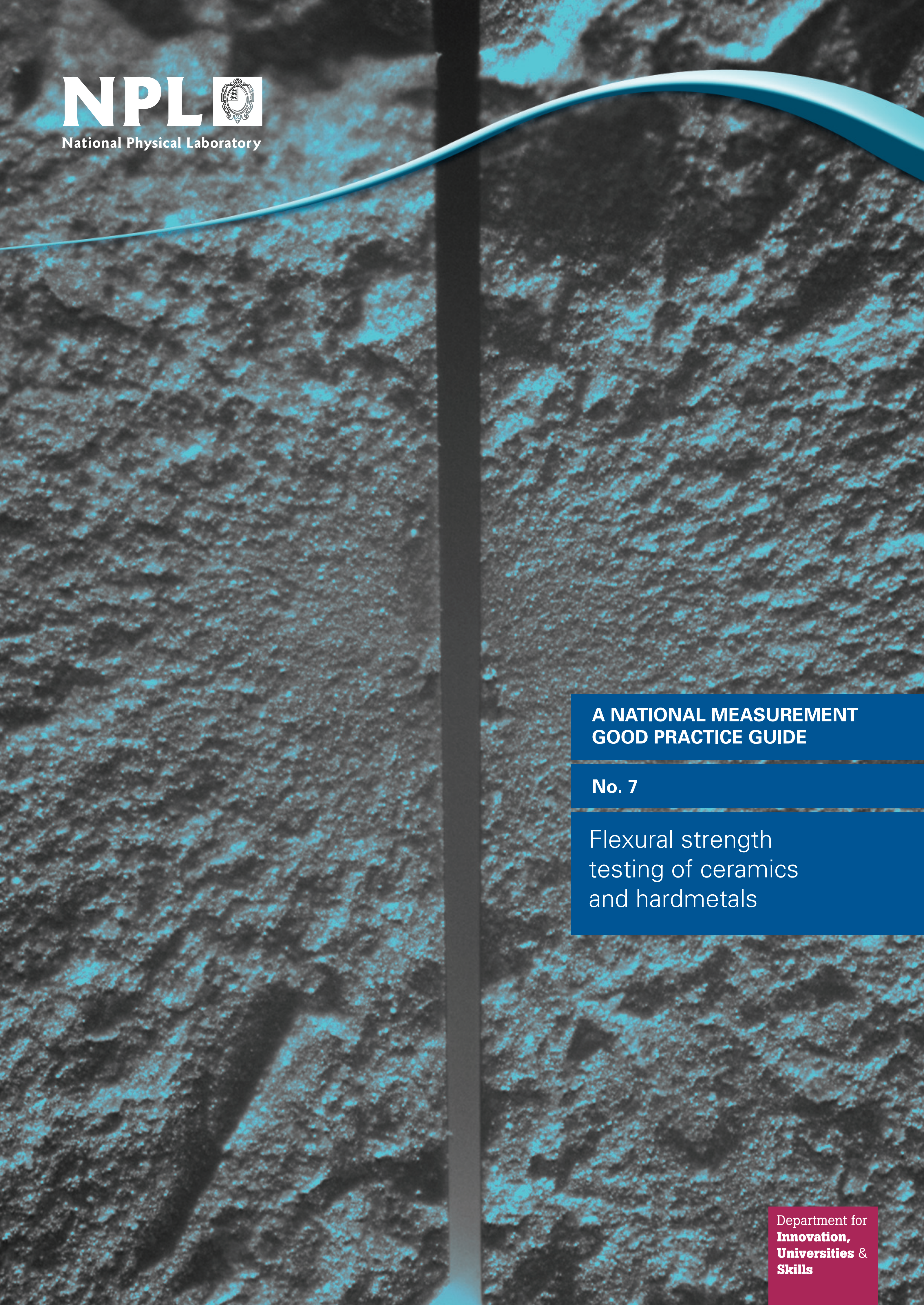


NPL

National Physical Laboratory

A NATIONAL MEASUREMENT GOOD PRACTICE GUIDE

No. 7

Flexural strength testing of ceramics and hardmetals

Department for **Innovation, Universities** & **Skills**

Measurement Good Practice Guide No. 7

Flexural Strength Testing of Ceramics and Hardmetals

Roger Morrell

Centre for Materials Measurement and Technology
National Physical Laboratory

Abstract: This guide is intended to aid the establishment of good practice in flexural testing of beam test-pieces of non-ductile materials, especially ceramics and hardmetals, whether or not it is performed in accordance with standardised procedures. There is discussion of the different purposes behind undertaking flexural testing and the influence this may have on the choice of procedure. Background information is provided to explain why the practices and test-piece geometries and tolerances have been adopted in standards. There is guidance on what the important factors are in interpreting the results of flexural testing, including fractographic investigation. The mathematical basis for analysing the stress errors in non-standard tests when assuming simple thin-beam bending equations is presented. A summary of Weibull statistical analysis is given, together with some of the pitfalls in using the derived information for design purposes. This guide will have value to those considering commissioning, setting up to perform, or undertaking flexural testing, as well as to those using the results from series of tests.

ISSN 1386–6550

September 1997
Updated with new standards references, November 2007

National Physical Laboratory
Teddington, Middlesex, UK, TW11 0LW

Acknowledgements

This guide has been produced in a *Characterisation of Advanced Materials* project, part of the *Materials Measurement* programme sponsored by the EAM Division of the Department of Trade and Industry. The advice and steer from the Ceramics and Hardmetals Industrial Advisory Group in reviewing earlier versions of this Guide are gratefully acknowledged.

This document reflects the discussions held in CEN Technical Committee 184, Working Group 3 during its development of EN 843-1 for advanced technical ceramics, and also the ongoing debate on test methods in the British Hardmetal Association Research Group which has prompted the VAMAS initiative in flexural strength testing as well as recent proposals to ISO for changes to the current standard.

The author would also like to acknowledge the long-standing collaboration and philosophical argument with George D. Quinn of the Ceramics Division, National Institute of Standards and Technology, Gaithersburg, Maryland, USA, and formerly of US Army Materials Laboratory, Watertown, Mass., USA, without whose inspiration and attention to detail the compilation of this Guide would never have been accomplished. Several NPL colleagues are also thanked: John Cox and Liam Byrne for test-jig design and operation experience, and Lewis Lay and Bryan Roebuck for reviewing the manuscript and offering helpful suggestions.

For further information on *Materials Measurement* contact the Materials Enquiry Point at the National Physical Laboratory:
Tel: 0208-943 6701
Fax: 0208-943 7160
E-mail: materials@npl.co.uk

Flexural Strength Testing of Ceramics and Hardmetals

Contents

Figures

Executive summary

This Guide documents as completely as practicable all the currently understood issues relating to flexural strength testing of hard materials, particularly advanced technical ceramics and hardmetals. It has been prepared to act as an underpinning document to the establishment of standardised testing methods, and as a source of advice when the use of standardised methods is impractical, a situation which often arises when the available material geometry is inappropriate for standard tests. A range of purposes in undertaking testing is considered, such as material development, quality assurance, and scientific investigation.

The Guide explains the underpinning mechanics involved in flexural testing, including the assumptions in the mathematical analysis when using simple evaluations based on thin-beam theory. It covers issues of how best to design testing jigs which most closely meet the ideal thin, frictionless loaded beam arrangement on which the analysis is normally based. It explains how the shapes of standardised test-pieces for some of the newer tests have been arrived at, and the accuracies and tolerances that are needed to keep the total errors involved in testing to less than 2%.

The testing practice is described, discussing the precautions needed in sampling and preparing test-pieces, including issues of surface finish and geometrical perfection. Precautions in testing and handling the test-pieces are recommended, especially if fractographic assessment is to be conducted later to identify fracture origins.

The statistical aspects of brittle fracture are covered in general terms, and the importance of understanding the significance of the results, whether a mean strength, a scatter, or a more detailed analysis of the strength distribution using Weibull statistics.

A series of Annexes covers the mathematical analysis of stresses in beams and the potential errors in testing, particularly from the view-point of necessary deviation from the ideal (best compromise) position adopted in modern standardised testing, e.g. for thick beams, curved beams, beams with large corner chamfers. Finally, the basis for two and three-parameter Weibull analysis is described, together with the preferred methods of data assessment now adopted in standards, and some warnings concerning the limited value that can be ascribed to such parameters for design purposes.

List of symbols

A = variable, representing area or constant in the crack velocity/stress intensity factor relationship
b = width of test-piece perpendicular to direction of loading
c = width of 45° chamfer measured on faces of beam test-piece
d = diameter of round-section test-piece
d_1 = distance between inner and outer support points in symmetrical four-point bending
d_1' = distance between inner and outer support points in four-point bending for eccentric loading
E = Young's modulus
F = normal force applied to test-piece
F_m = maximum force applied at fracture
g = probability density function
G = cumulative distribution function
h = thickness or height of test-piece in direction of loading
i = rank
I = cross-sectional moment of inertia
k_1, k_2 = constants in twist error equation
K_I = stress intensity factor, mode I, opening mode
K_{Ic} = plane strain critical stress intensity factor
l = span between support points in flexural testing
L_T = total test-piece length
m = Weibull modulus
M = bending moment
n = subcritical crack growth exponent
N = number of test-pieces in a batch
P_f = probability of failure
r = radius of chamfer
u = variable
v = crack tip velocity
V = shearing force
w = variable
x = variable, in direction of length of test-piece
y = variable, through thickness of test-piece
β = scale parameter in Weibull distribution
ε = error
ε_x = strain in *x*-direction
θ = angle
μ = coefficient of friction
ρ = radius of curvature of a bent beam
$\bar{\sigma}$ = mean strength
$\dot{\sigma}$ = stressing rate
σ_e = maximum stress in beam with eccentric loading
$\sigma_{f,3}$ = nominal three-point bending fracture stress
$\sigma_{f,4}$ = nominal four-point bending fracture stress
σ_{max} = maximum nominal stress in symmetrically loaded beam
σ_u = lower strength limit in three-parameter Weibull analysis
σ_x = stress in *x*-direction
σ_0 = characteristic strength (scale parameter in the Weibull equation)
τ = shear stress
φ_s = angular twist of test-piece along its full length
φ_f = angular twist of test-piece along between inner and outer load point

1 Introduction

Usually, the question raised is:

Is material/batch A stronger than material/batch B?

Because the strength of a brittle material, unlike a yield stress in a ductile metal, is indeterminate until a given type of test-piece is fractured in a given manner, such a question covers a multitude of underlying issues, often not recognised by those who make decisions using the results of tests.

This Guide is intended to cover all aspects of flexural strength testing of brittle materials using bar-shaped test-pieces, the most convenient method of stressing when gripping for tensile testing is difficult. The objective of this Guide is to provide, in one place, the presently scattered items of information that are needed to perform effective testing for any purpose. Particular attention is paid to test-jig arrangements, to preparation of test-pieces, and to the interpretation of test results.

The main text of this Guide goes through all the principal issues in setting up to perform tests, including test-jig design, test-piece preparation and analysis and interpretation of results. It details the similarities and differences between different test method standards, and discusses the options available to the tester, and the reasons for the conditions prescribed in standards. The principal intention is to alert the reader to the many issues behind strength testing, which if ignored can limit the value of testing programmes.

One of the messages in this Guide is that if possible, a standardised test should be used. The standards provide procedures designed generally to minimise systematic errors. However, there may be many circumstances in which a non-standard test has to be performed, for example, if the test-pieces have a non-standard geometry. The series of Annexes to this Guide provide the details of the systematic errors and other corrections needed for some of the more common deviations from standard geometries. These details have hitherto been widely scattered in the literature, and an objective of this Guide is to bring this information together.

The Guide can be used either as a cover-to-cover procedure for setting up and performing strength testing, or as a reference manual for individual aspects of testing.

2 Objectives of strength testing

2.1 What is 'strength' of a brittle material?

Non-ductile materials lack any method of flowing or yielding significantly to accommodate the stresses being applied. This means that any small cracks or flaws[1] in the material act as stress concentrators, but the material around them cannot flow to blunt them and reduce the local stresses. Instead, as soon as the tensile or shear stress is high enough, the most highly stressed crack or flaw tip starts to propagate. Once this happens, the crack is usually difficult to stop, and even if it does stop, the presence of the crack means that the component is much weaker and usually cannot perform its function as effectively.

The strength of a given article is thus controlled by the combination of the largest flaw subject to the highest stress. Because the flaws that control strength are usually very small, typically less than 100 μm in a dense fine-grained material, they are difficult to see and eliminate from the material. The result is that within a given number of nominally identical articles there will be a spread of sizes of the 'worst' flaws, leading to a spread in strengths.

This has a number of consequences:

- the strength of any one test article is indeterminate until it is broken;
- it usually requires several test results to ensure some reliability in the average results;
- there will always be nominally identical articles which are significantly weaker, or conversely, significantly stronger, than those subjected to the limited number of tests;
- changing the methods of manufacturing or machining may change the average strength result;
- changing the size of article or the manner of stressing may change the average strength result;
- in some materials, changing the rate of stressing and/or the environment of the test can change the strength result.

A completely different philosophy has to be adopted concerning the value ascribed to strength test results on non-ductile materials compared with yield characteristics in ductile ones.

[1] The use of the term 'flaw' is traditional in discussing strength of brittle materials. The meaning is distinct from that in normal everyday use. The use of the term should in no way imply that the product is 'flawed' and therefore unfit for service. In this Guide, the term is used to describe microstructural sources which control strength. They mostly arise naturally as a result of microstructural type, processing route or machining procedure.

2.2 Determining strength using flexural testing

As a bending force is progressively applied to a bar-shaped test-piece of a brittle material, the stresses inside the bar increase in an elastic fashion. Those on the convex side of the curvature are in tension, while those on the concave side are in compression, in both cases directed along the axis of the beam. Since brittle materials are more susceptible to fracture under tensile stresses than under compressive stresses, we are concerned most with the convex side, particularly the near-surface regions which are subject to the highest stress levels. This in turn means that flaws at or near to the surface of the bar are more likely to act as fracture origins than those closer towards the bar centre, while those in the compressive half can usually be ignored.

Compared with tensile testing, the flexural strength test has many advantages:

- no problems with gripping;
- easy to align;
- small and hence fairly cheap-to-make test-pieces;
- quick to test;

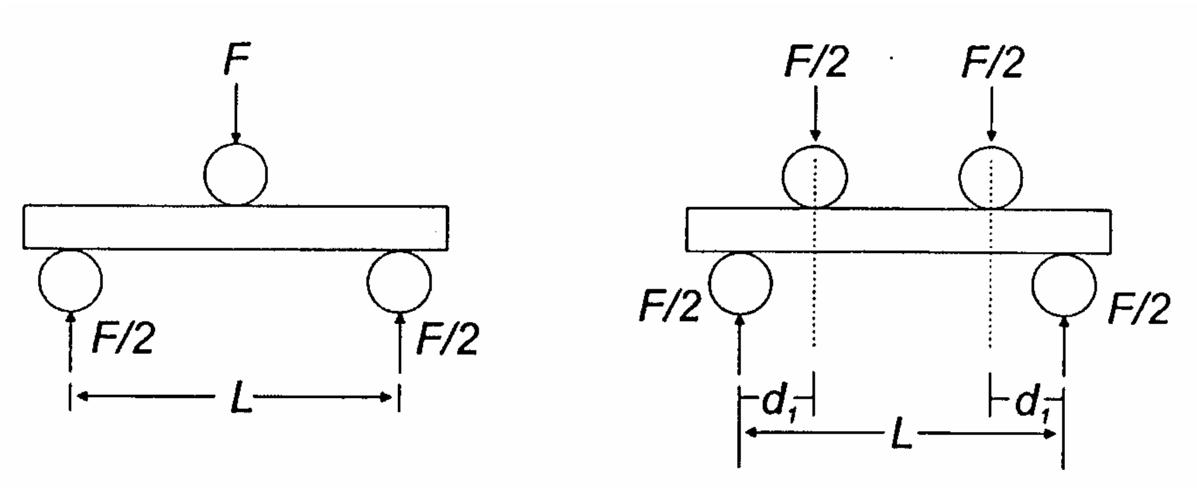
and some potential disadvantages:

- a tendency for surface flaws to dominate;
- care is required with test-piece edges, especially in ensuring that edge failure is minimised.

The last of these has led to increasing use being made of disc flexure testing, in which edges can be remote from the highly stressed regions. However, this alternative has a number of other problems associated with it which will be dealt with in a separate review.

There are two convenient ways of applying a bending force to a bar-shape: three-point bending and four-point bending (Figure 1). Pure bending, generated by applying a bending torque to the ends of the bar, is generally not used because of the likelihood of stress concentrations at the points of gripping.

In both cases, the fracture stress in the test-piece is calculated from the force applied at the moment of fracture and the dimensions of the test-jig and the test-piece, using a simple formula based on thin-beam bending (see Annex 1). The nominal flexural strength in three-point ($\sigma_{f,3}$) and four-point ($\sigma_{f,4}$) bending is calculated using the appropriate simple thin-beam bending equation for rectangular section beams:



(a) (b)

Figure 1 *Conventional geometries for beam flexural testing: (a) three-point bending and (b) four-point bending.*

$$\sigma_{f,3} = \frac{3F_m l}{2bh^2}; \qquad \sigma_{f,4} = \frac{3F_m d_1}{bh^2} \qquad (1)$$

where F_m = maximum force applied

l = support span

d_1 = the distance between the outer support and inner loading rollers (the 'loading arm') for symmetrical loading

b = width of test-piece

h = thickness of test-piece in direction of bending

For round-section beams the equivalent formulae are:

$$\sigma_{f,3} = \frac{8F_m l}{\pi d^3}; \qquad \sigma_{f,4} = \frac{16F_m d_1}{\pi d^3} \qquad (1A)$$

where d = diameter of test-piece

Typically, 10 test-pieces are tested for determining a mean strength, but 30 or more test-pieces are usually recommended to obtain reliable information on the statistics of strength. See section 8 for a more detailed discussion of the significance of test-piece numbers. Typical strengths obtained with standard geometry tests are 50 to 1000 MPa for ceramics and 1500 to 3500 MPa for hardmetals, depending on factors such as porosity and grain size.

2.3 Choosing between three and four-point bending

Three-point bending has advantages of being easier to undertake with a simpler test-jig arrangement, and tends to be favoured by industrial laboratories who use it, notably for QA purposes. It is also less prone to errors of test-jig geometry and alignment. The standard hardmetal tests use the three-point arrangement, as do tests for traditional clay-based technical ceramics. However, four-point bending, despite requiring more attention to detail, is increasingly preferred because a larger volume of the test-piece is placed under a more-uniform stress, resulting in a test that is more searching for occasional large flaws. This test is particularly appropriate for obtaining design data. A consequence of this is that strengths determined using three-point bending are usually greater than those for four-point bending when testing over the same span, and the two types of result cannot reliably be directly compared without a detailed statistical analysis (see Annex C, section C4.2).

Standard test procedures usually specify one of the two methods, or permit both. In the latter case, the user is generally left to exercise his discretion concerning the more appropriate geometry.

Three and four-point bending tests over the same span produce different strength results on the same batch of test-pieces. Choose the more-appropriate geometry for the circumstances and keep to it to provide inter-material comparisons.

2.4 Uses of strength testing

Despite the strength of brittle materials not being an inherent unvarying mechanical property, flexural strength testing is widespread, and is undertaken for a variety of reasons:

- **Quality assurance:** ensuring **consistency** of mechanical properties from batch to batch in a given test configuration.
- **Materials specifications:** flexural strength commonly appears in commercial brochures and data sheets, but suppliers are usually very reluctant to supply to a given strength level. Further discussion of this appears in section 8.
- **Materials development:** comparing a **new development** with a previous one.

- **Materials behaviour:** determining the role of **composition, process or environmental variables** on strength; determining the effect of **stressing rate**, or **time under load** (static fatigue or stress rupture).
- **Design data:** using strength data **to design components** for use in critical situations where the risk of mechanical failure is significant.

Whatever the purpose of the testing, one of the key issues is **sampling** (see also section 5). Strength can be affected by so many parameters that it is essential to ensure that the basis for sampling is understood, and that the batch of test-pieces truly reflects the condition which is to be examined. In many cases, the test-pieces will have to be specially prepared for testing, and thus the results may reflect the preparation procedure, rather than the factors being examined.

Consistent procedures for the production of test-pieces and their traceable identification are critical for reliable comparison of results from series of tests.

2.5 Standardised versus non-standardised test arrangements

For in-house quality assurance (QA) or materials development use, or for a particular component or test-piece shape, strength testing can be performed in any appropriate manner. Provided that the stressing geometry is consistent, the results on a series of batches, trials or experiments yields useful information, irrespective of the test geometry. However, for purposes in which data are changing hands, or being used in any quantitative sense, it is essential to use a standardised method when possible.

As a result primarily of the increasing demand for comparative and useful engineering data, as opposed to QA testing, over the past decade there have been strong efforts to produce and implement standardised tests. The advantage of this is that there is a consistent basis for measurement, permitting information from different sources to be directly compared. The disadvantages are principally the reduction in flexibility and occasionally questions of the relevance of testing using an inappropriate size of test-piece relative to the end use. Nonetheless, good testing practice and attention to testing detail incorporated in modern standards has undoubtedly made strength data more reliable.

Standardised procedures should be used where possible.

3 Standardised testing geometries and requirements

3.1 Definitions

Bend strength, bending strength, flexural strength, transverse rupture strength, modulus of rupture (equivalent terms): the maximum calculated stress at the instant of fracture in a transversely, elastically loaded beam test-piece.

NOTE: Transverse rupture strength (often abbreviated to TRS) is used primarily in the hardmetal field. Modulus of rupture (MOR) is used primarily in the traditional ceramic industry, but will also be found in the advanced technical ceramics literature.

The adjective **nominal** often precedes the term. This is because the maximum stress calculated, which is normally the so-called ‘outer-fibre’ or surface stress, may be greater than that at the flaw which acts as the fracture origin.

Three-point bending: in which the bar is placed on supports near its ends, and a central force is applied; the maximum axial tensile stress is localised on the convex side under the loading point.

Four-point bending: in which the bar is placed on supports near its ends, and two equal forces are applied at two symmetrical positions between the supports; the maximum axial tensile stress is approximately uniform on the convex side between the two loading points.

Wedging stresses: in thick test-pieces, the stress shielding that occurs as a result of shear effects between the support and loading rollers, countering the bending. Such effects are negligible in long thin beams.

Articulation: the ability of support and loading rollers to rotate about an axis parallel to the length of the test-piece to permit misalignments to be taken up, and thus improve the uniformity of stressing.

Anticlastic bending: the tendency of a beam to develop an opposite curvature in a direction orthogonal to the principal (intended) direction of bending as a result of Poisson strain effects. This is negligible in standardised geometries but increases with increasing beam width.

As-sintered, as-fired, as-moulded: the geometrical and surface conditions of a test-piece as it is produced without any post-firing operations to prepare flat surfaces for testing.

Chamfer: a rounded edge or a 45^{o} flat machined along the long edges of the test-piece to reduce the incidence of edge-originating flaws.

3.2 Standards

Table 1 lists a variety of current standard test methods which contain beam-flexure tests. In recent years there has been a move to try to eliminate the former wide variety of spans and test-piece cross-sections, notably for ceramic materials, and to consolidate towards sizes and shapes which are:

- convenient to use;
- give minimal errors when using the thin-beam equation;
- of a single type appropriate for most materials in a class.

It should be noted, however, that there are significant differences of approach for different classes of material.

For **clay-based materials**, larger test-pieces are employed which are easy to make by extrusion or dust pressing, and easy to test in the as-fired condition. Material cost is not normally a problem for such materials.

For **glasses**, rod test-pieces are preferred, because glass becomes readily damaged by preparing rectangular sections. Alternatively, disc flexure testing is common.

For **hardmetals**, the standards have been long established (ISO 3327, BS EN 23327), although a revision is expected shortly to cover the use of round-section test-pieces. Although it can be shown that the Type B test-pieces (see Table 1) unfortunately are too thick for accuracy of use of thin-beam bending equations these tests continue to be widely used for convenience, and **a systematic overestimate of true fracture stress of 10 to 15% is incurred**. There are also technical issues associated with the forces required to break hardmetal test-pieces which can lead to problems of jig reliability, and indentation of rollers into test-pieces. Work has recently been conducted in VAMAS to identify possible alternative test methods to the standards listed in Table 1 [1–3]. An outstanding issue is whether test-pieces should be annealed. Normally this is not the case. Machined test-pieces usually have much higher strengths than annealed ones because of residual compressive stresses in the surface.

For **advanced technical ceramics**, the thinner rectangular section test-pieces are designed to give negligible errors when using thin-beam equations to calculate the stress. The 40 mm span varieties emanated from international collaborative work between the USA and Europe in the late 1970s and 1980s, while the 30 mm span was contemporarily developed in East Asia, notably in Japan [2].

[2] ISO 14704 contains both 30 mm and 40 mm spans, with the latter preferred.

Amongst the various standards there are also differences of approach to loading jig requirements. Friction effects are generated between the test-piece and the support points (and loading points in four-point bending) leading to errors in the thin-beam bending assumption. These are most readily removed by permitting the supports to roll (see section 4.1). Articulation (see section 4.2) is essential for as-fired test-pieces, and desirable even for machined test-pieces, to ensure even application of the applied forces across the test-piece width, but articulation is not a general requirement except in EN 843-1.

3.3 Demonstrating that standardised geometries are effective

The best way of demonstrating the effectiveness of a standard is to conduct a round-robin and to examine the consistency of test results. A number of round-robins, both national and international, have been conducted.

- In the Technical Cooperation Program (Australia, Canada, New Zealand, UK and USA), 1500 test-pieces of 99.9% Al_2O_3 and reaction-bonded silicon nitride were tested in batches of 30 to 35 in a number of test configurations. In general, consistent results between test laboratories were obtained only when the US MIL-STD 1942 procedure (the forerunner of ASTM C1161 and EN 843-1) was employed [4]. This required rotating or rolling rollers to eliminate friction effects.

- In the UK, BSI Committee RPM13 conducted a round-robin in 1989. Using 96% Al_2O_3 test-pieces in the US MIL-STD 1942 geometry, participants using test-jigs with fixed loading and support points obtained higher apparent strengths (by 10 to 15%) than participants using rotating or rolling rollers [10].

- As part of the IEA programme between the USA, Sweden and Germany concerning ceramics for advanced heat engines, nearly 2600 test-pieces (cross-section 3.5 x 4.5 mm as defined in a former German pre-standard) of silicon nitride and silicon carbide were employed among 23 laboratories [5]. Results generally showed good consistency, but some laboratories were a long way out of line. A follow-on programme of testing loading jigs with strain-gauged test-pieces resulted.

Table 1 - Standardised flexural testing geometries

Standard	Test-piece width (*b*) x height (*h*) x length (*L*), and outer/inner span ($l/l\text{-}2d_1$), mm: 3-point bending	4-point bending	Other controlled parameters
ISO 14704 (ISO 17565 for high temperatures) (ATCs*, internatl.)	4 x 3 x >35, 30 span 4 x 3 x >45, 40 span	4 x 3 x >35, 30/10 spans 4 x 3 x >45, 40/20 spans	Selection of surface finishing procedure; rolling support and loading rollers
CEN EN 843-1 (EN 820-1 for high temperatures) (ATCs*, Europe)	A: 2.5 x 2 x >25, 20 span B: 4 x 3 x >45, 40 span	A: 2.5 x 2 x >25, 20/10 spans B: 4 x 3 x >45, 40/20 spans	Selection of surface finishing procedure; rolling support and loading rollers
ASTM C1161 (C1211 for high temperatures) (ATCs, USA)	A: 2 x 1.5 x >25, 20 span B: 4 x 3 x >45, 40 span C: 8 x 6 x >85, 80 span	A: 2 x 1.5 x >25, 20/10 spans B: 4 x 3 x >45, 40/20 spans C: 8 x 6 x >85, 80/40 spans	Selection of surface finishing procedure; rolling support and loading rollers
JIS R1601 (ATCs, Japan)	4 x 3 x >35, 30 span	4 x 3 x >35, 30/10 spans	Fixed surface finish, fixed support and loading rods
GB 6569-86 (ATCs, PR China)	4 x 3 x >35, 30 span	4 x 3 x >35, 30/10 spans	As per JIS R1601
IEC 60672-2 (1996 revision, various electrotechnical ceramics)	10 ∅ x 120, 100 span 10 x 10 x 120, 100 span 10 x 8 x 120 ('flatted round'), 100 span	-	As moulded bars; rolling loading rollers
IEC 60672-2 (1996 revision, various electrotechnical ceramics)	4 x 3 x >45, 40 span	4 x 3 x >45, 40/20 spans	Normally machined; rolling loading rollers
IEC 60672-2 (1996 revision, glasses)	-	10 ∅ x 120, 100 mm span	For as-drawn glass and also toughened glass; rolling loading rollers
ISO 3327 (1982), hardmetals	A: 5 x 5 x >35, 30 span B: 6.5 x 5.25 x 20, 14.5 span C: 3.3 ± 0.5 ∅, 25 ± 5 long, 14.5 ± 0.5 span	-	'Freely lying' hardmetal support rollers, hardmetal loading roller or centralised 10 mm hardmetal ball
ASTM B406-90, hardmetals	6.25 x 5 x >19, 14.3 span (≈ ISO B-type)	-	Hardmetal support rollers, centralised 10 mm hardmetal ball
JIS CIS026-1983 hardmetals	A: 5 x 5 x >35, 30 span (= ISO A-type) J: 8 x 4 x 24, 20 span	-	Fixed hardmetal support and loading rollers or knife edge, 1.6-3.0 mm radius

* ATC = Advanced Technical Ceramics

- A VAMAS project was conducted to study environmental effects in strength testing of an alumina ceramic [6]. Agreement between laboratories on the environmental effects was good, provided that a sufficiently wide range of stressing rates was used (see Annex C, section C.4.3), but there remained some differences in strength levels between laboratories. In part this might be attributed to using small numbers of test-pieces, and in part to test-jigs with inadequate loading characteristics.

- In an International Congress on Glass round-robin, soda-lime glass microscope slides were subjected to a similar study to that in VAMAS, with similar results [7].

- A VAMAS round robin has been conducted using various hardmetals in a variety of test configurations. Results [1, 2, 3] show reasonable consistency for a given type of test-jig and test-piece geometry, but large differences between different geometries which relate to different test geometries and lack of correction for thickness effects.

These round-robins have shown that if care is taken with test-piece preparation to ensure that the test material itself and its preparation into test-pieces are consistent, consistent strength results can be obtained when the test-jig functions correctly. This probably needs a skilled operator aware of the pitfalls of testing.

If either the test-piece machining is poor, or the test jig does not function correctly and eliminate misalignments and friction, inconsistent results arise and agreement between laboratories is poor.

3.4 Non-standard geometries

As outlined in section 2.4, non-standardised testing sometimes cannot be avoided because of the nature of the available material, or there may be special reasons for supplying data to a different test specification. Generally it will not be possible to compare the results obtained with those from standardised tests. There should be no attempt to use the data for purposes other than the immediate one, unless it is a properly organised design exercise.

Some key points in non-standardised testing are:

- **very thin test-pieces** are more compliant, and the large deflections before failure lead to significant calculation errors (see Annex A, section A.7); if beam span:height ratios (l/h) do not exceed about 80, the errors are insignificant;
- **wide test-pieces** suffer from significant anticlastic bending (see section 3.1, and

Annex A, section A.3.3), which is partially restrained by the loading rollers, leading to inaccuracies in stress calculated;

- **round section test-pieces** have the maximum stress only along a line, and give higher strength than a rectangular section test-piece of equivalent cross-sectional area (see Annex A);
- the stress field in **thick test-pieces** is complicated by shearing effects. These make the test-piece more rigid than estimated by the thin-beam analysis, leading to an overestimate of strength (see Annex A);
- **large chamfers** change the effective cross-section, and increase the stresses near the surface (see Annex B);
- **asymmetric cross-sections** tend to twist in loading, leading to stress concentrations. Caution is advised in analysing the stress field;
- some designs of test-jig use **non-articulating support or loading rollers**. Even if the test-piece is perfectly machined, the test-jig may not be, and the result is uneven loading across the test-piece faces, leading to stress concentrations and predominance of corner failures. Fractography can help to identify this problem (see section 6);
- use of **non-rotating support or loading points** can lead to friction errors which can be as high as a 15% overestimate of flexural strength (see Annex B). Friction opposes the flexure of the test-piece. It depends on the surface finish of both test-piece and roller, and thus can be test variable.

When testing non-standard geometries, ensure that conditions are optimised towards those of standard procedures and that the correct mathematical analysis is used.

4 Test-jig design

None of the standards attempts to give more than a schematic of the design of a test-jig because different test machines may require different types of fixture. ISO 14704, EN 843-1 and ASTM C1161 prescribe the **function** of the test-jig only. Other standards merely describe the important dimensions of component parts. Designing test-jigs to operate efficiently and effectively is not a trivial task. This section lists some of the main features required and means of achieving them.

4.1 Reducing friction effects

The origins of, and principles of reducing, friction are now well understood see Annex B, section B1). The support and loading rollers (in four-point bending) should be free to rotate to accommodate movements and eliminate friction. Rollers can be:

- captive cylinders supported by freely rotating roller bearings, or
- loose cylinders rolling on a flat surface.

They should not be:

- cylinders sitting in a Vee-groove or notch, nor
- profiled fixed blocks.

Captive cylinders in roller bearings have the advantage of having fixed pivot positions, which define a load-independent span, but the rollers and the bearings may become damaged by repeated use due to high forces being applied or to the shock of breaking test-pieces, and thus eventually may not work effectively. In addition, when the cylinder surfaces become damaged by the fracturing test-pieces, renewing captive components may be more difficult than using loose cylinders.

Loose cylinders have the advantage that they are easy to remove and inspect for damage, and economical to replace if necessary. The disadvantage is that they normally have to be gently restrained into known start positions in the test-jig in order to define the spans and the symmetry of the loading roller(s) to the support rollers. There has to be an arrangement such that they are held against stops (e.g. Figure 2), but free to roll, the outer support rollers rolling outwards, and the inner loading rollers in four-point bending rolling inwards. Possible methods of restraint suitable for room temperature use are:

- rubber bands (Figure 3; these may require frequent replacement);
- compliant phosphor-bronze leaf springs (Figure 3);
- positioning jigs which move rollers to correct positions while the test-piece is under a small pre-load;
- a slight angle of the support plate such that they roll to stops at the correct positions (Figure 3).

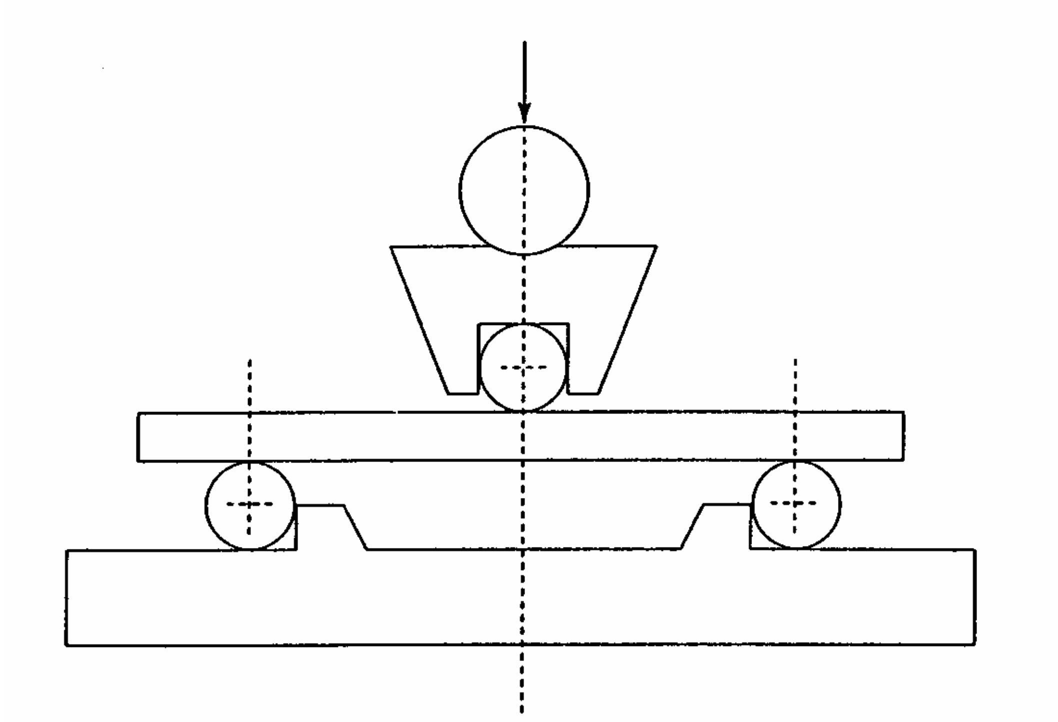


Figure 2 *Schematic of a three-point test facility using rolling rollers and provided with articulation of the loading roller only. Note that the support rollers are free to roll outwards.*

(a) *(b)*

(c)

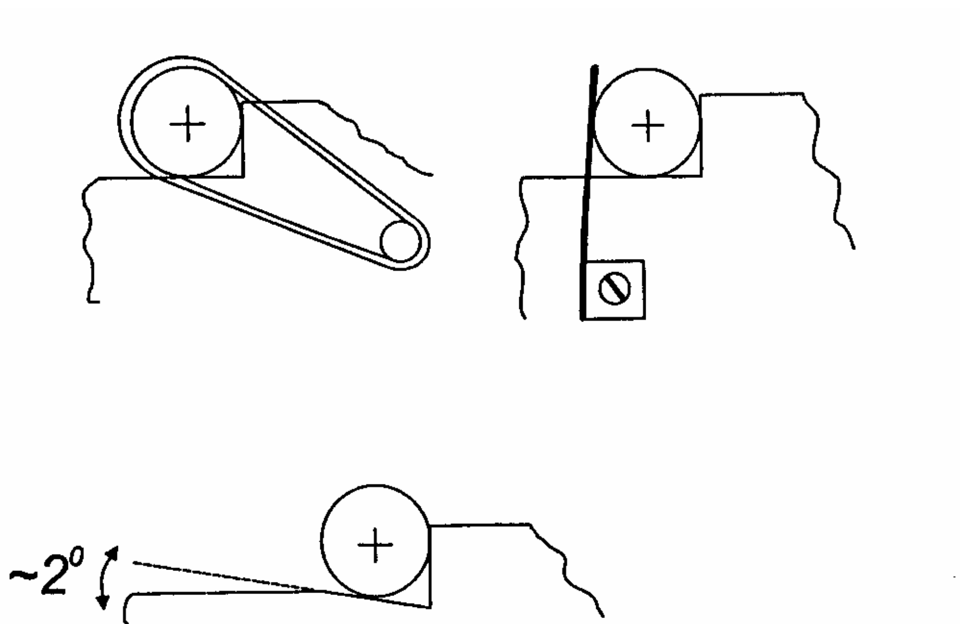


Figure 3 *Methods of retaining the rollers in their start positions: (a) using rubber bands, (b) using a thin compliant leaf spring, (c) using a slight slope on the support surface.*

For high-temperature use, the choice is more restricted:

- positioning jigs which move rollers to the correct positions while the test-piece is under a small pre-load;
- temporary combustible organic adhesive gluing the rollers in position during set-up, but disappearing at the test temperature.

Questions have been raised concerning the utility of rolling rollers under very high loads required to break materials having flexural strengths in excess of 1000 MPa, including some strong ceramics and most hardmetals. For such materials, hard-faced components need to be used in test-jigs to resist indentation, either of the roller into the test-piece or of the roller into the bearing surface. The use of thinner test-pieces or longer spans is a means of reducing the forces involved.

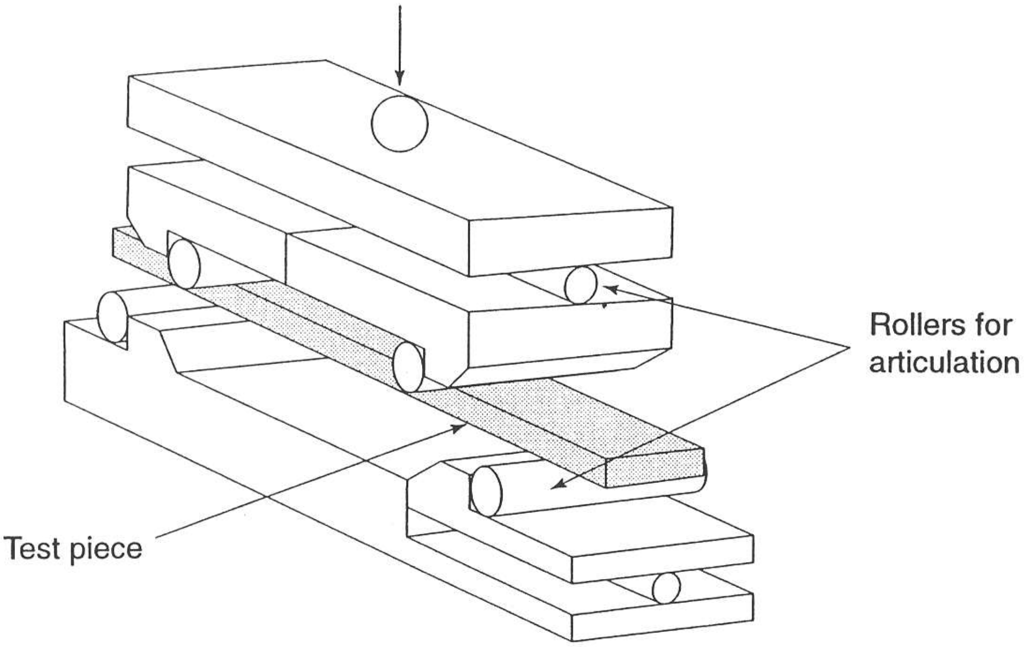
4.2 Achieving articulation

Because it is not always possible to machine test-piece surfaces flat and parallel, nor to make a test-jig in which the loading and support rollers are all in perfect parallelism, some degree of articulation, typically up to about ± 2°, is usually needed to make the distribution of load across the test-piece face as uniform as possible and thus to avoid twisting the test-piece and developing edge stress concentrations. F or as-fired or as-moulded test-pieces, articulation is essential.

For well-machined test-pieces and test-jigs, articulation is needed only for the loading unit relative to the support unit (e.g. Figure 2, based on ASTM C1161). However, to cover all eventualities, it is convenient to have a jig which is fully articulated, as required by CEN EN 843-1. Usually, one support roller needs to articulate relative to a non-articulating one to provide stability to the jig. The loading roller unit needs to articulate relative to the support rollers, and for four-point bending, one loading roller needs to be able to articulate relative to the other. To achieve such motion, various designs have been used:

- tilting the rolling surface on a bearing, the axis of which lies parallel to the test-piece length and preferably along the test-piece surface to minimise sideways movement; Figure 4(a) is based on an example given in ASTM C1161, although this does introduce sideways movement on rotation;
- placing a curvature (axis parallel to the test-piece length) on the rolling surface, e.g. ASTM C1211; Figure 4(b) is an example made in silicon carbide at NPL;
- using crossed rollers.

(a)



(b)

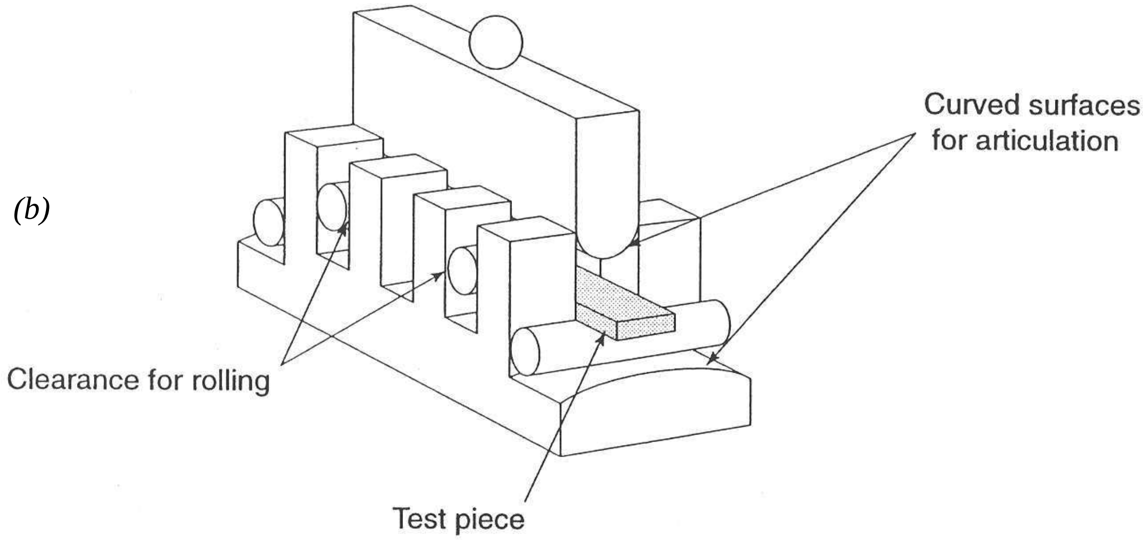


Figure 4 *Schematics of two fully articulating four-point flexural strength jigs: (a) with multiple tilting parts similar to that shown in ASTM C1161; the loading parts must additionally have means of alignment relative to the support parts; (b) made from a monolithic guide system suitable for use at room and high temperatures. Note that the slots in which rollers are positioned are typically 0.2 mm wider than the roller diameter to permit sufficient rolling action to eliminate friction. Care must be taken with this design to ensure that the rollers start in contact with the appropriate side of the slot.*

The first of these has the largest support area, and thus minimises indentation at contacts. In the case of loose rollers, the roller maintains contact with the flat surface, which rotates in a plain or roller bearing. However, the bearing surfaces permitting the tilting must be kept clean and free from fracture debris. In the case of captive bearings, the engineering design is more complicated, but the bearings can be protected from debris ingestion. In both cases, ideally, the axis of rotation should intersect the line of contact between test-piece and roller. This minimises sideways movement and hence a risk of uneven loading through lack of axiality.

The second method has some advantages for use at high temperatures where sliding bearings cannot readily be lubricated. Ceramic rolling surfaces have been used. However, there is a significant stress concentration at the effective line contact, which may considerably limit the force that can be applied compared with the first method. The third method has advantages of simplicity, but it too suffers from risk of indentation, and should only be used for low applied forces.

It should be noted that ASTM C1161 (and C1211 for elevated temperature testing) permit the use of semi-articulating jigs for carefully machined test-pieces in which the pairs of support and loading rollers are individually non-articulating, but which articulate relative to each other. This presumes accuracy in the geometry of the jig to produce coplanar lines of contact on the test-piece.

4.3 Achieving alignment

There is a requirement in most standards for the loading roller in three-point bending to be centrally positioned between the support rollers, or in four-point bending for the loading rollers to be symmetrically positioned with respect to the support rollers, to a precision that is typically 0.1 mm. To achieve this requires means of adjusting and fixing the location of the loading system relative to the support system. Failure to achieve the required tolerances leads to an asymmetry in the stress distribution in the test-piece, and a tendency to underestimate the strength in four-point bending (see Annex B, section B.3).

In a typical mechanical testing machine, one possibility is rigidly to clamp the support system to the lower part of the machine and the loading system to the upper part. However, limitations are imposed by the need to permit articulation of the loading system to take up misalignments due to imperfections in geometry of test-pieces. Another method is to provide a sliding guide for the loading system within the support system (e.g. Figure 4(b)). This has advantages in permitting the test-jig to be free standing and simply loaded between flat platens in the test machine. In any case, the line of loading must be centralised between the support rollers, and in the case of four-point bending, the load must be equally divided between the two loading rollers. This is most easily achieved by ensuring jig symmetry.

In addition, loading must be symmetrical across the width of the test-piece by ensuring the line of loading passes through the centre line of the test-piece. It is convenient to provide stops against which to push the test-piece, which can either be permanent but adjustable to the test-piece width, or removable, e.g. a positioning jig.

Measurement of the support and loading roller spacings is required by all standards. The simplest way of doing this is to use a travelling microscope set up to view the jig orthogonally to the test-piece length. Another is to place a thin layer of ’engineer's blue’ on the surface of the rollers and to place a test-piece on the rollers to transfer the dye to the test-piece, then to measure the apparent spacing of the contact lines on the flat test-piece.

4.4 Test-jig materials

The test-jig body itself can be made of any material which is sufficiently resilient to perform the function required. Typically, mild steel can be used for low applied forces, although to avoid atmospheric or handling corrosion, a stainless steel is advisable. For use in aqueous liquids, 316 grade is recommended as having a minimum risk of crevice corrosion. For use with high applied forces, or at temperatures above about 300 °C, metals tend to soften and deform under the rollers, and consideration needs to be given to using hardmetal or ceramic materials. Hardmetal parts will be usable to typically 400 °C (500 °C in vacuum), but the most commonly employed material is SiC, primarily for its capability of operating to at least 1400 °C in air with minimal oxidation and little if any loss of elasticity.

The test-jig rollers and the articulating surfaces on which they rest must be hard to remain round and to withstand risks of damage when test-pieces break. For many general applications, hardened tool steel is adequate for strengths up to about 200 MPa, but for higher strength test-pieces it is necessary to consider using hardmetal (e.g. fine-grained 6% Co/WC) or ceramic (high-alumina, silicon nitride or silicon carbide) rollers and contacting surfaces to ensure resistance to flattening or embedding. Roller surfaces need to be accurate smooth-ground uniform cylinders at least twice the width of the test-piece, preferably polished, and with no protruding burrs. Rollers need to be readily replaced if damaged, and should be considered a consumable. The roller diameters are usually prescribed in standards and are a balance between minimising the local stress concentration of a line contact while retaining a reasonably well defined position of contact.

4.5 Unacceptable design features

Some examples are as follows:

- rollers intended to rotate but held in round or Vee grooves which inhibit free rotation under load;

- no self-alignment capability between support and loading rollers; there should be an ability to articulate the loading rollers (three and four-point bending), and to pivot the loading block relative to the support block (four-point bending);
- rollers in slots which do not permit sufficient lateral movement to eliminate frictional effects, or if there is clearance, when the rollers do not start against the appropriate sides of the slots;
- no lateral alignment capability to ensure symmetry of loading.

5 Test-piece preparation

5.1 Sampling

Often the material for testing is supplied as specially prepared rough bar shapes, or needs to be prepared from a block. Occasionally, test-pieces will need to be machined from available components. In all cases, the issue of 'sampling' arises. Whether consciously or otherwise, some selection/rejection procedure is always involved in preparing the test-piece set. Normally, this is not prescribed in standards because circumstances vary, notably the purpose of undertaking the testing. It is strongly recommended that all test-pieces have, when available, their history of preparation, *e.g.* batch numbers, processing parameters, selection criteria, *etc.*, which permit traceability of the strength results obtained.

Some general factors that need to be considered are:

- homogeneity of the material in the form of large blocks;
- reproducibility of individual as-fired test-pieces;
- the presence of surface skins and the extent to which these will be removed by machining procedures in preparing test-pieces.

5.2 As-moulded or as-fired test-pieces

In the as-moulded or as-fired condition test-pieces may be warped or twisted, or of non-uniform cross-section, or have sharp edges and rough surfaces. If test-pieces are used in such a condition, it is clear that results could be widely scattered. It is highly desirable to place some restraints on the dimensional variation between test-pieces, on the degree of warpage allowed, and the surface quality. Most standards permitting as-fired test-pieces place some restrictions. In order to keep the potential errors to a minimum it is recommended that:

- Warpage: the curvature of the test-piece should be equivalent to less than 0.5 mm of flexure over the supported span.

- Twist: the twist in the test-piece should be less than 0.001 mm per mm of test-piece width between the supports unless there is articulation in the test-jig. An upper limit of 0.035 mm per mm of test-piece width is desirable even if there is jig articulation[3].

- Non-uniform cross-section: any taper along the length of the test-piece may result in a biased fracture position. In three-point bending in particular, this will lead to an overestimate of nominal flexural strength. If fracture occurs within the central loading span in four-point bending, the result is still acceptable. In either case, the cross-sectional dimensions at the plane of fracture should be determined after the test and used for calculating nominal strength. Ideally, dimensions should be uniform to better than 1% along the span length.

- Lack of chamfers: the edges of unchamfered rectangular section as-fired test-pieces are prone to damage in handling, and fracture is often initiated from this damage. If test-pieces are made by die-pressing there may be press flash along the edges. If the test-pieces are made by green machining from pressed blocks, edge chipping may be prevalent. In both cases, a chamfer, typically 0.1 to 0.15 mm (measured along the main test-piece surfaces) should be used. If the chamfer is larger than this, a significant error is introduced in not correcting the thin-beam equations for the change in geometry[4]. Any flashing can be removed, and the chamfer can be created by hand on the green shape by careful 'fettling' using a cloth or fine-grit abrasive paper. If chamfering is done after firing, this will usually require machining or abrading the edges, which are not then representative of the as-fired condition.

---

[3] CEN EN 843-1 specifies a maximum 2^{o} twist over the span length.

[4] ISO 3227 and CIS 026-1983 for hardmetals permit the use of a correction factor for large chamfer sizes. No correction is necessary for 45^{o} chamfers $\leq$ 0.2 mm on 4 mm and 5 mm thickness test-pieces, but the nominal thin beam equation needs to be multiplied by a factor of 1.03 for chamfers of 0.4–0.5 mm on type A (5 x 5 mm cross-section) test-piece. The errors due to not correcting for chamfer size are discussed in Annex B, section B.4.

5.3 Machined test-pieces

For most material types, the machining procedure used for manufacturing test-pieces is a critical factor in determining the result of a strength test. The coarser the grit size employed, the larger the cuts taken and the lower the fracture toughness of the material, the greater is the depth of damage (Figure 5). Then, generally, the more the subsequently determined strength is lowered. Grinding leaves behind an anisotropic surface stress, which may be advantageous or disadvantageous from the strength point of view. Thus, it is highly desirable to control in a known way the machining sequence employed for test-pieces. However, it is generally the case that no two combinations of grinding machines and grinding wheels give the same degree of damage [8]. This has created a dilemma for the preparation of standards. Various attempts have been made to advise care in grinding, but none is totally successful.

The best approach is probably to consider first the purpose for which strength testing is to be conducted:

- **Quality assurance:** for internal batch checking the key criterion should be consistency from batch to batch. An internal specification should be developed which goes much further than most standards, setting wheel and machine operating parameters:

  - wheel type, grit size and concentration, frequency of dressing
  - specific grinding machine, coolant type, concentration (if water-based) and flow rate
  - wheel speed, traverse speed (X-direction), lateral step size (Y-direction)
  - sequence of depths of cut (Z-direction)

  Any departures from such a procedure required because of difficulties encountered with a particular batch should be fully recorded.

- **Materials evaluation:** if the objective is to evaluate scientifically the role of the real microstructure of the material in controlling strength, machining damage must be minimised. This requires a sequence of different grinding grit sizes and accompanying depths of cut designed to remove traces of damage from previous grinding steps such that grinding damage is completely removed. Final stages may require lapping and polishing (e.g. CEN EN 843-1, para. 6.3.3.3, condition III.2).

  If the objective is to compare materials after a given machining procedure, then an approach similar to that for QA should be adopted. Alternatively, a prescribed procedure can be used. CEN EN 843-1 and ASTM C1161 for advanced technical ceramics recommend similar prescribed procedures (CEN EN 843-1, para 6.3.3.2, condition III.1; ASTM C1161, para 7.2.4, standard procedure), although there are small differences in the surface roughness specifications.

- **Materials design data:** if the end objective is the determination of strength characteristics which can subsequently be used for evaluating component designs, it is imperative that the strength tests are performed on test-pieces which mimic as closely as possible the expected microstructure and surface finish of the components. A component may be subjected to a variety of machining procedures on some or all parts of its surface, and this may need to be taken into account in the preparation of a number of batches of test-pieces with relevant surface finishes. Advanced technical ceramic standards cover this eventuality (ISO 14704, para 6.2.4, CEN EN 843-1, para. 6.3.2, condition II; ASTM C1161, para 7.2.2, application matched machining).

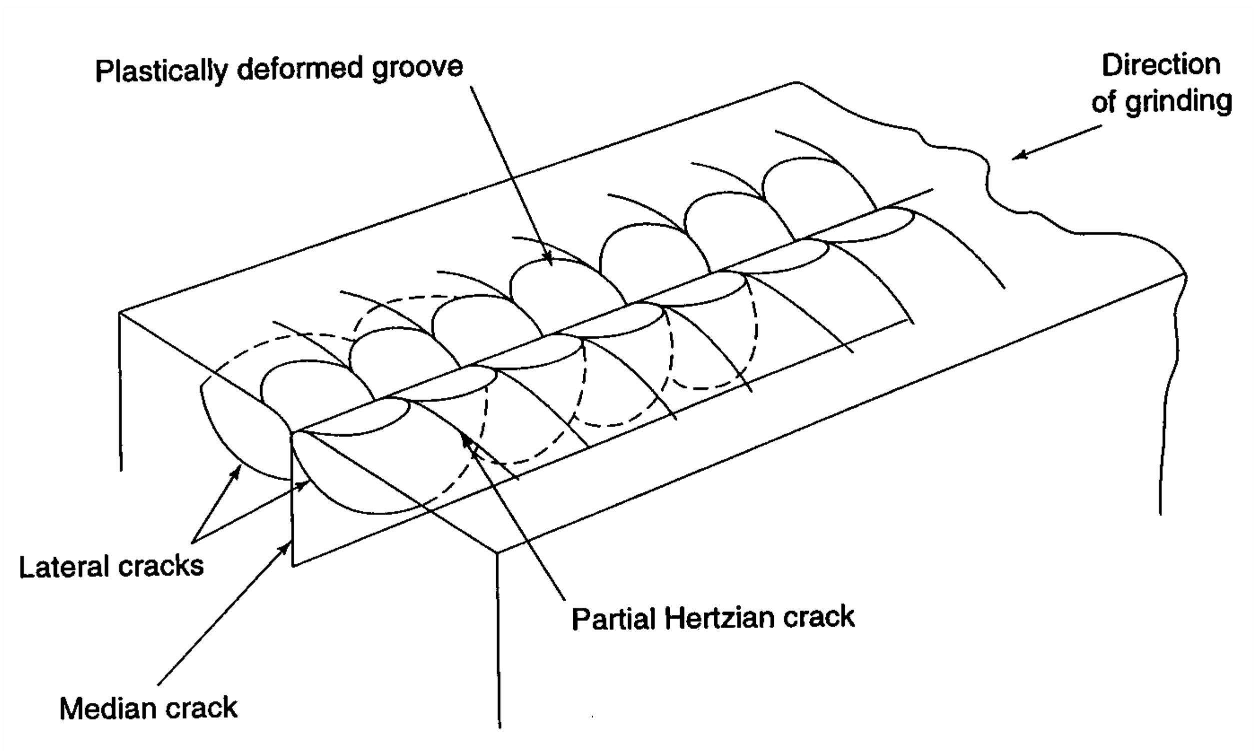


Figure 5 *Schematic of damage patterns that can be introduced by coarse grinding. In addition to ploughed grooves, cracks can be driven in vertically along the groove, and shallow Hertzian partial ring cracking can run roughly perpendicular to the groove. Subsurface lateral cracks can also be generated. To remove this type of damage requires adequate subsequent machining with finer grits which induce less damage.*

The grinding direction is also an important parameter. Most standards specify that the abrasion direction should be along the length of the test-piece (ISO 3327 or BS EN 23327 for hardmetals, EN 843-1 and ASTM C1161 for advanced ceramics). The reason is primarily to avoid the development of grinding grooves and subsurface (median) cracks running perpendicular to the direction in which stress is applied (Figure 5). However, most grinding is also accompanied by the development of lateral cracks running roughly perpendicular to the direction of grinding, but the damage is usually less severe. Differences of 20% in strength between the directions are not uncommon. Similarly, it is recommended that chamfers are introduced by machining with the same type of procedure along the test-piece length (see below).

This specification can be unrealistic, especially for design data collection, for which the anisotropy of strength may be required, or in which multidirectional grinding or lapping procedures may be involved. Nevertheless the general cautionary tone stated in such procedures should be observed for all grinding operations.

A good product can be ruined by bad grinding. Grinding needs to be done to minimise damage while achieving the appropriate dimensions.

5.4 Machining test-pieces from components

If it is required to strength test material from a fabricated component, it must be recognised that the act of cutting into the component may change the dominance of the flaw type examined in the strength test compared with that determining the strength of components. Precautions must be adopted to ensure as far as possible that test-piece preparation damage produces less severe flaws than those in the component itself. In cases where the material in the component is suspected of having anisotropic microstructure, the orientation of test-pieces and the proposed direction of fracture relative to the component axes must be considered. Individual test-piece orientations need to be fully documented.

5.5 Test-piece dimensions and tolerances

Most standards lay down test-piece dimensions and tolerances. Overall size tolerances are typically ± 0.2 mm on cross-sectional dimensions as the dimensional variation within the batch, but within each test-piece, the flatness and parallelism requirements for machined advanced technical ceramic test-pieces are typically ± 0.02 mm, or ± 0.1 mm along the length and ± 0.05 mm across the width of as-fired test-pieces (CEN EN 843-1, ISO and ASTM requires slightly tighter tolerances). For the larger cross-section hardmetal test-pieces, 0.01 mm per 10 mm length for machined test-pieces and 0.05 mm per 10 mm length for as-fired test-pieces are specified (ISO 3327).

For most purposes, in order to quote the standard validly as part of the result, the dimensions specified should be adhered to as far as possible to provide a means of comparing test results with those from other sources. Furthermore, significant departures from thickness to width or thickness to span ratios may introduce significant errors in assuming the thin-beam equations are still valid. When necessarily test-pieces of other dimensions are used, a check should be made using Annex A as to the magnitude of any corrections that should be used.

5.6 Machining chamfers

The purpose of chamfers is to reduce stress concentrations caused by damage along the edges of rectangular section test-pieces. In this regard, the grinding damage introduced by chamfering **should be similar to or less than** that involved in face grinding.

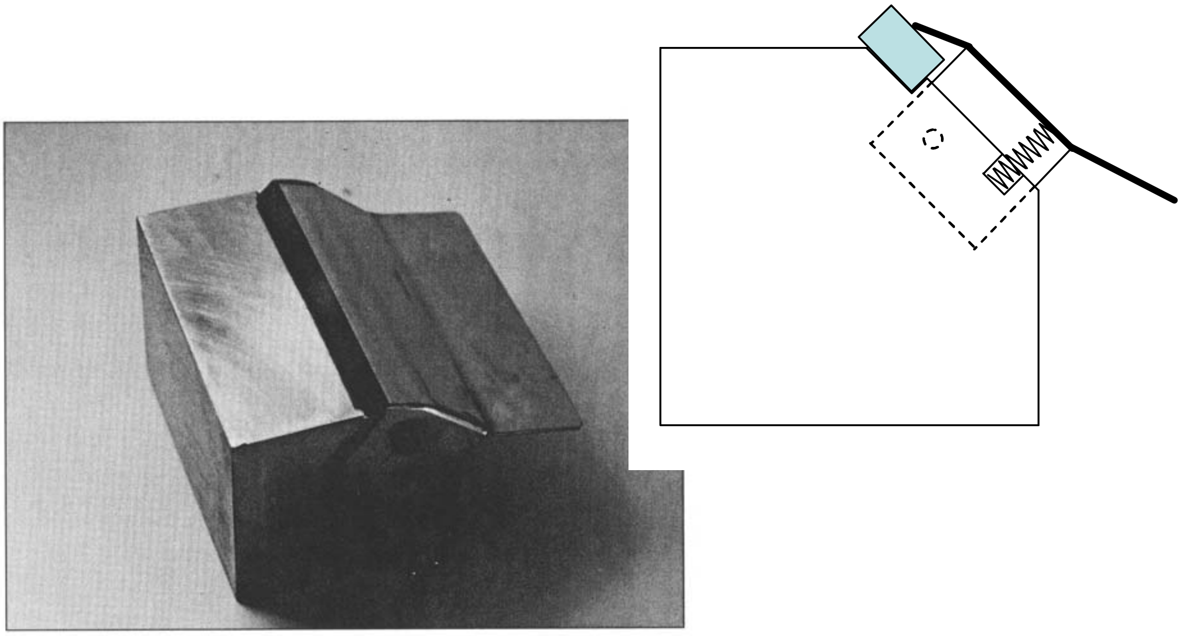
At least two, and ideally all four, long edges of rectangular-section test-pieces should be chamfered. The size of the chamfer must be quite small to avoid having to modify the flexural strength equations (equation 1, see more detail in Annex B, section B.4), and the standards recommend 0.1 to 0.15 mm as a typical width measured along the flat test-piece surface. Alternatively, the corners can be rounded to a similar radius of curvature. The errors in calculation of fracture stress introduced by these sizes are negligible.

To minimise damage introduced by chamfering, the grinding direction should be parallel to the length of the test-piece unless there is a specific requirement to test the damage introduced by grinding perpendicular to the length. Unfortunately, machining chamfers is not straight-forward in an engineering sense. Each edge usually has to be done individually. Flat chamfers can be produced by gripping each test-piece in turn in a Vee-block or suitable jig at the 45^{o} angle, and by running the diamond wheel along the length. Rounded chamfers, also permitted in most standards, are more difficult to produce by normal diamond grinding, but can be made using profiled wheels or manually on a flat lap.

Test-pieces waxed down as a batch onto a plate for face grinding may not have exactly equal dimensions, nor necessarily be perfectly square-edged[5], and for the subsequent chamfering operation this requires re-setting the starting height of the grinding wheel for each edge. This can be very tedious, especially as detecting touch-down of the grinding wheel on an edge can be difficult, particularly on softer materials.

[5] CEN EN 843-1 places a maximum out-of-squareness of 5^{o}, tested using a vernier protractor or engineering shadowgraph. ISO 14704 and ASTM C1161 specify no more than 0.015 mm off square.

(a



(b)

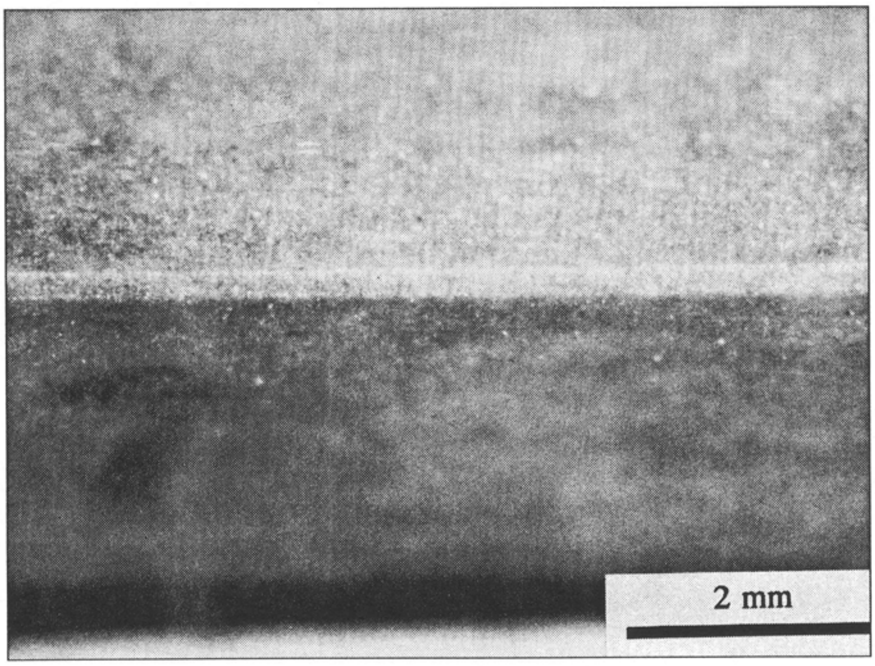


Figure 6 *(a) A simple Vee-block jig for gripping test-pieces for hand or machine chamfering; (b) optical micrograph of a chamfer introduced into an alumina ceramic using a diamond lap and hand-held jig.*

Chamfering manually may in many cases be easier. A simple spring-grip Vee block jig for 4 x 3 mm test-pieces is shown in Figure 6. The test-piece is placed in the groove, the jig inverted, and the test-piece contacted onto a flat diamond rotary lap for a defined short period of time. If the lap has a grit size similar to or less than that of the grinding wheel, the outer region of the lap is used, and the test-piece is orientated tangentially to the lap, a good approximation to machine chamfering can be obtained. The mass of the Vee block will supply sufficient pressure to create the small chamfer on most materials, and the time of chamfering, typically a few seconds per edge, can be adjusted by inspecting the chamfer size. Alternatively, reciprocating hand grinding on abrasive paper strips can be used, although there is a greater risk of obtaining an uneven size of chamfer because of rocking.

Chamfering reduces, but does not eliminate, risk of damage accumulation at free edges. It must be done with care, and to a sufficient size to eliminate chips and cracks. Large chamfers need a stress calculation correction.

5.7 Annealing test-pieces

Grinding or lapping damage can be annealed out in some cases. Annealing can have two effects, depending on the temperature at which it is performed. Residual stresses due to grinding, which tend to be principally compressive at the immediate surface, and thus lessen the significance of surface flaws or surface intersected bulk-type flaws, are relaxed, and this may result in a reduction in strength. Annealing of this type takes place when the softest phases start to become slightly plastic to permit stress relaxation. In hardmetals, this is typically 600 to 800 ^{o}C (in vacuum to avoid oxidation effects). In glass-phase containing ceramics, it is typically 700 to 800 ^{o}C. However, this type of annealing does not necessarily heal gross damage, such as cracks. It is necessary to take the materials to higher temperatures to permit sufficient material migration to allow cracks and chips to seal and thus eliminate the flaws. Information on this topic is sparse, but it can be anticipated that treatment at 100 to 150 ^{o}C below the original sintering temperature might be required.

Whatever type of annealing has been performed, it can be anticipated that the strength distribution obtained will be different from that of machined test-pieces, and often the mean strength is reduced by annealing.

5.8 Handling precautions

Having spent effort to obtain a high-quality machined test-piece, additional accidental damage should be avoided. One of the main sources is allowing test-pieces to jostle each other in a bag. It is desirable to keep test-pieces separate, and individually identified by numbering with pencil. Any suitable protective medium can be used, from tissue paper to individual compartments in multicomponent plastic trays. The same guideline applies to storage after fracture, especially if fractographic examination is contemplated (see section 6).

5.9 Good practice for surface finish

This section of the Guide will have demonstrated to the reader the importance ascribed to correctly specifying and obtaining appropriate surface finishes on test-pieces. To summarise the key points in arriving at the test with acceptable test-pieces:

- consider the relevance of surface finish to the proposed use of the test data;
- select a test-piece manufacturing route which incorporates all the processing and surface finishing procedures relevant to the proposed use of the test data, and create a machining specification;
- check that the achieved finish meets the machining specification in terms of dimensional precision and surface quality, including chamfers;
- document all the relevant details with the test results.

6 Test procedure

6.1 Equipment

Standards lay down procedures in various degrees of detail. The main points are as follows.

6.1.1 Test machine

Any type of compression-mode lead screw or actuator driven mechanical testing machine with appropriate load capacity can be used, either under ramped load control or ramped displacement control. Machine compliance is not an issue for ordinary fracture testing.

6.1.2 Machine load calibration

It is critical that load measurement is accurate. Calibration of the load cell should be undertaken regularly. If necessary, check the calibration before and after each series of tests. A 1% error in calibration leads to a 1% error in determined strength. Most standards specify this level of accuracy.

6.1.3 Data recording

Many modern machines have an electronic peak load detector. This tends to have a much faster sampling and response time than a chart recorder or some computer-based slow data logging systems, and thus is likely to be inherently more accurate in determining peak load in a short-term fracture test. However, to accommodate the older types of machine with chart-recorders, the loading rate suggested in standards is normally restricted to achieve fracture in 10 to 15 s. Typically, cross-head speeds of 0.5 mm/min or loading rates of 50 to 200 N/s are recommended to achieve this target. Some adjustment of speed may be needed depending on the stiffness of the load train or the strength of the test-pieces. Conducting the test faster than this is likely to lead to an underestimate of strength because the chart recorder cannot respond fast enough.

6.1.4 Alignment

The alignment of jigs rigidly fixed to the static and moving parts of the test machine should be checked to ensure that when a test-piece is in place, loads are transmitted to the test-piece axially (and symmetrically in four-point bending). The spans and symmetry should be checked and adjusted if necessary once the jig parts are assembled. For free-standing test-jigs with self-aligning elements, there are no special requirements on axiality of the test machine. Placing the test-jig between flat platens normally suffices.

6.1.5 Environment

For ceramic and glass materials it is recognised that strength may be affected by the humidity of the surrounding environment. Humidity should be checked and recorded, together with ambient temperature. If a controlled humidity environment is required, this will normally involve using an environmental chamber about the test-jig[6]. Humidity does not appear to be an issue in testing hardmetals and some non-oxide ceramics, such as silicon carbides.

6.2 Measuring test-piece dimensions

Measurements with an engineering micrometer are usually specified, and are normally perfectly adequate for the task. Ball ended anvils on micrometers should not be used, especially for glasses and weaker ceramics, because of the risk of cracking due to localised loading during measurement. Measurements of thickness and width should be made at several places along the length, checking for adherence to test specifications at the same time. It is easier to make the dimensional measurements before testing than after fracture, although as a check, measurements at the fracture position can be made after testing, particularly if there is any unevenness in as-fired test-pieces.

[6] If testing is required in dry conditions, enclosing the test-jig in a plastic bag and blowing dry gas in for 30 s before testing is quite effective, as revealed in a VAMAS round robin. For testing in liquid environments, immersing the test jig into a small flat-bottomed stainless steel container of the test liquid works well.

6.3 Undertaking the test

6.3.1 Test-piece alignment

The test-piece should be carefully aligned in the test-jig, preferably against positioning stops which can later be withdrawn. It is particularly useful to ensure that the test-piece is marked with a unique number at both ends and with its orientation in the test-jig. The positions of the loading and support rollers should also be marked (e.g. in pencil, but use caution in high-temperature tests). This is a useful aid to later examination.

If necessary, re-measure the support and loading spans using a travelling microscope or similar device, and adjust the positions such that loading is symmetrical to better than 0.1 mm, especially in the case of four-point bending.

6.3.2 Test rate

For conventional short-term strength testing, standards tend to lay down testing rate ranges within which to operate to give fracture in a given time range. For machines under load control, this is normally set as a load ramp rate in N/s, while for displacement control, a cross-head or actuator displacement rate in mm/min is set. In both cases, with a material of unknown strength, some experimentation may be needed to achieve fracture within this time range. Significant departures from the recommended rates can lead to changes in the apparent strength as a result of subcritical crack growth effects (see Annex C, section C.4.3), but testing over a wide range of rates ('dynamic fatigue'), or indeed under static load ('static fatigue'), can give numerical information on long-term strength potential of a material.

6.3.3 Safety

The higher the strength of the test-piece, the greater the stored elastic energy at the point of fracture, and the greater the kinetic energy associated with the fragments. Test-jigs should be screened as far as possible to minimise the risk of flying fragments. Cushioning the fragments from impact with jig parts is also useful to minimise the risk of secondary damage which might confuse subsequent fractographic evaluation.

6.3.4 Test validity

With the exception of accidents or the use of test-pieces containing obvious scratches or chips or other defects which are non-representative, all individual tests produce a result and should be reported and used to calculate the mean nominal flexural strength. No test result should be rejected since it reveals something about the character of the material. The important aspect to note is that the result of the test gives a nominal strength because of the assumption that failure has occurred from the region of maximum stress at the test-piece surface. In practice, failure may occur away from this region, *i.e.* significantly away from the loading roller in three-point bending, or outside the inner loading span in four-point bending, purely as a result of the presence of a significant flaw at that point.

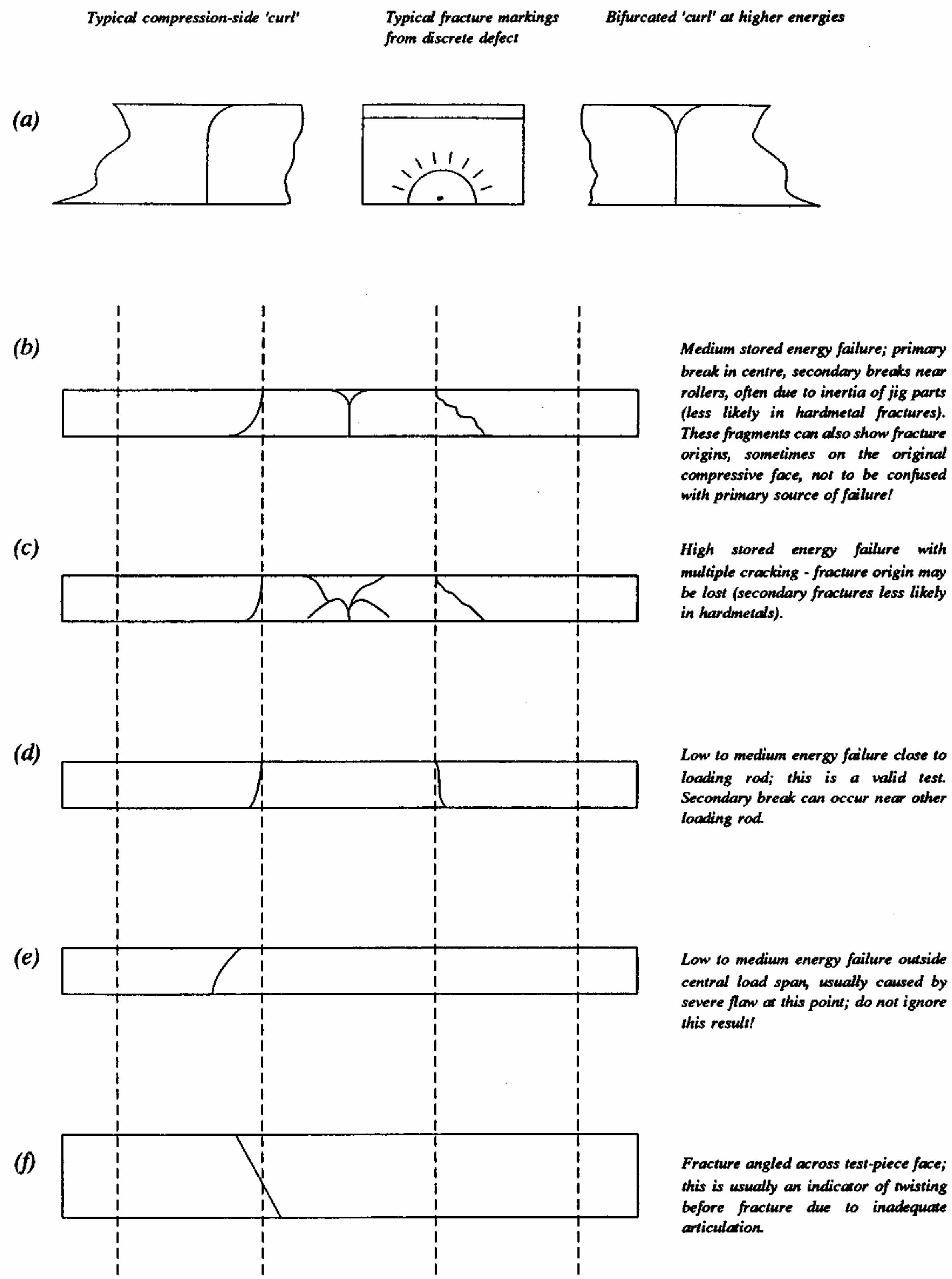


Figure 7 *Macro-fractography of test-pieces as indicators of fracture mode and failure origin.*

The true stress at the point of failure would be rather less than the nominal stress calculated. The same is true if the fracture origin, *e.g.* a pore, is positioned some distance from the immediate surface of the test-piece. It is possible to correct the nominal result to the true result (see EN 843-1), but this is not normally done in calculating 'nominal' mean strength, although it can be for fracture mechanical investigations.

Tests can be considered suspect if fractures tend to occur frequently under one of the pair of loading rollers in four-point bending. In such a case there is a strong likelihood that the loading roller pair is not symmetrical between the support rollers, and some adjustment to the roller positions should be made. Similarly, if failures always occur from one edge rather than the other, the axiality of loading must be checked. In both cases, the degree of error can be significant, but is essentially indeterminate without careful investigation using a strain-gauged test-piece.

Figure 7 gives some examples of macro-scale fractography of types of failure that commonly occur. Note that except in low energy fractures there is usually a 'curl' to the primary fracture as it enters the zone of high compressive stress. This is a normal feature and can be used to check on the direction of fracture. At higher energy levels, the crack may bifurcate giving a wedge-shaped fragment. Secondary fractures can also occur as a result of the shock of the primary fracture, or the impact of fragments with the test-jig. In very high strength materials, multiple fragmentation occurs, and even if all the fragments can be retained, it is then a difficult task to reassemble the test-piece for identification of the origin.

Evaluation of the appearance of the fragments can tell something about the test as well as the material, notably incorrect jig function such as twisting (Figure 7(f)), or inadequacy of chamfering procedures, and is worthwhile undertaking.

6.3.5 Determining fracture mechanisms

Fractography of the fracture origin on a micro-scale is a very useful adjunct to strength testing because it acts as a diagnostic tool for determining their size and nature, and thus to assign the fracture to a particular class, or to determine what processing factors are limiting the strength of the product.

Effective fractography can be carried out only if all fracture fragments can be captured and kept clean and free from contamination. The main sources of contamination are:

- grease from fingers, jig parts and other engineering devices;
- metal marks from contact with the test-jig or handling with tweezers;
- skin fragments;
- adhesive from adhesive tape;

- smears from mounting clays (e.g. ‘Plasticene’);
- dust, lint, plastic scrapings from containers;
- chipping by mutual contact after the test.

It is strongly advised to take precautions to avoid contamination as far as possible, since most of the above cannot be remedied by post-test cleaning. The main recommendations are:

- wear disposable gloves when handling fragments to avoid pick-up of skin and grease;
- mark the positions of the support and loading roller contacts on the side of the test-piece using a pencil for later identification of fracture position relative to the stress field;
- pack cotton wool around the test-piece; in particular, place cotton wool or a rubber strip under the centre of the test-piece to prevent the edges of the primary fracture from hitting the test-jig and suffering secondary damage; this may be less important for tougher hardmetals;
- handle the test-piece and all fragments with plastic-tipped tweezers to avoid contamination; steel tweezers may leave metal marks;
- keep all fragments separate and do not allow them to jostle each other (e.g. loose in a bag) or the container in which they are kept; do not try to ’reassemble’ the test-piece because fracture surfaces can be damaged and valuable evidence destroyed.

Some skill is required to use fractography effectively. An understanding of likely fracture features in different material types is needed to be able to interpret visual, and microscope observations correctly. An ASTM standard guide on fractography, C1322, and a similar European standard (EN 843-6) have been published, both relevant to flexural strength testing. More general documents on fractography are NPL Good Practice Guide no. 15[7], and NIST SP 960-16 (2007)[8].

To find the approximate location of the origin, it is necessary to follow fracture features which tend to radiate from origin. Failure of these features to focus down to a discrete point may be due to an extended origin, such as a pre-existing crack, or could simply be that the fragments with the origin are missing! However, in most cases, the feature is distinct and identifiable. Fracture origins can be of a number of types:

- single large pores (often due to incomplete compaction of the powder, or burn-out of large organic particles, or vaporisation of impurities, and may be associated with an inclusion);

[7] Available from National Physical Laboratory, Teddington, UK
[8] Available from National Institute for Standards and Technology, Gaithersburg, MD 20828, USA, and as a download file from the NIST website www.nist.gov.

- groups of small pores (also incomplete compaction);
- elongated groups of pores (can be due to delaminations or to burn-out of organic contamination such as lint, hair, skin), sometimes called a porous seam or line;
- single large grains (large grain seeds in batch);
- dense agglomerates surrounded by continuous or discontinuous pores;
- inorganic contamination spots changing the local microstructure;
- normal microstructure (limiting strength);
- surface contamination, pick-up from firing, etc.;
- microcracks, surface chips, scratches;
- machining damage.

All the types associated with bulk microstructure can appear at or close to the surface and can be confused with surface originating defects. Careful microscopical examination, usually with a scanning electron microscope equipped with element analysis facilities, can often give a very clear indication of the precise nature of origin. Make sure that both halves of the fracture are examined since the combined interpretation may be different from the individual ones.

In cases where there is no feature at an apparent origin, it could be a natural microcrack, machining damage or genuine microstructure-limited failure. These often occur in very strong materials where large features, for example, pores have been eliminated by hot isostatic pressing. Strictly, such cases should be annotated as ’unidentified’.

Fractography is a post-mortem examination - keep everything clean - do not rush to conclusions - gain all the evidence first.

6.3.6 Reporting

It is strongly recommended that, in view of the number of variables involved, results of tests are reported in detail sufficient to permit reproduction of all the conditions of test-piece preparation. An example report pro-forma appears in Table 2.

Table 2 Example proforma for reporting

Laboratory:		Testing date:	Job no.:
Operator:		Signature:	Date:
Test material supply code:		Date of receipt:	
Details of material preparation (if relevant):		Customer details: Address: Order no.:	
Test-piece preparation site:		Job/batch no.:	
As-fired:	Yes/No	Ground/lapped/polished :	Yes/No
Grinding procedure: wheel types: removal rates: coolant:	Rough grinding:	Finish grinding:	Lapping/polishing:
Chamfering:	Manual/machined	Chamfer procedure: Grit size: Machine type: Depth of chamfer:	
Test-piece nominal dimensions:	Depth:	Width:	Length:
Strength test type:	Three-point/four-point	Jig type:	Fully/partially articulating
Test machine type:		Load calibration date:	
Parameters set on machine:	Displacement rate:	Loading rate:	Peak load detection method:

Test-piece no.:	Dimensions, $b \times h$, mm	Fracture force, N:	Nominal flexural strength, MPa:	Fractographic ident. of failure site:
1				
2				
3				
4				
.				
.				
.				
.				
.				
.				
.				
.				
.				
Number of valid tests:			Mean strength, MPa: Standard deviation, MPa:	
Comments about test or fractured test-pieces:				
Report authorisation:		Name:	Signature:	Date:

7 Test method accuracy and error budget

7.1 Error budget for standard tests

Adherence to the prescribed geometries and precision of measurement given in EN 843-1 and ASTM C1161 for advanced technical ceramics should lead to individual nominal flexural values accurate to typically ± 2%. Typical error budgets for size B test-pieces (4 x 3 x 40 mm span) are:

Load	± 1%
Span	± 0.1 mm in 40 mm = ± 0.25%
Inner span (4-point)	± 0.1 mm in 20 mm = ± 0.5%
Thickness	± 0.01 mm in 3 mm = ± 0.33%
Width	± 0.01 mm in 4 mm = ± 0.25%
Total root mean square error	± 1.2% for three-point bending
	± 1.6% for four-point bending

7.2 Systematic errors

Systematic errors increase with:

- increasing departure from thin-beam geometry;
- increasing size of chamfer;
- increasing unevenness of geometry;
- friction effects;
- misalignments;
- lack of articulation.

Systematic errors certainly exist in hardmetal geometries in assuming thin-beam behaviour. ISO 3327 (BS EN 23327) acknowledges that size B test-pieces give typically 10 to 20% higher apparent nominal flexural strengths than size A provided that they have the same surface finish, principally because the low span-to-thickness ratio gives rise to so-called 'wedging' stresses.

Annex B deals with some of these factors, and may assist with creating error budgets for alternative non-standard geometries.

7.3 Discrimination between data sets

The error budgets given above indicate that the error associated with the result of a given test is fairly small, and much smaller than the typical scatter of results from a batch of say 10 test-pieces which is usually of the order 10% or more (see section 8). Thus for the purposes of discriminating between data sets, or comparing an average strength with a given specified level, the contribution to errors from test method inaccuracy in standard test procedures can essentially be ignored, but may be significant for non-standard geometries.

8 Statistical analysis

8.1 Mean and standard deviation

Most test standard procedures require a minimum specified number of tests: at least 10 for ceramics in ISO 14704, CEN EN 843-1, ASTM C1161 and IEC 60672, at least five for hardmetals in ISO 3327, CIS 026-1983, and five only in ASTM B406. These numbers of tests are sufficient to give a reasonable idea of mean strength, but insufficient to determine the strength distribution parameters with any accuracy. If the mean and standard deviation are calculated, and the coefficient of variation (standard deviation/mean) obtained is divided by $\sqrt{n}$ where n is the number of valid tests, the standard error of the mean is obtained. This parameter is a good guide to the likely level of confidence in the mean result. Thus for a mean strength of 400 MPa and a standard deviation of ± 40 MPa on ten test-pieces, the standard error of the mean is ± 3.2 MPa. Thus, repeating the experiment several times could lead to a spread in mean result of typically twice this amount in either direction, so that mean strengths in the range 394 to 406 MPa are not significantly different.

Similarly, if a minimum mean strength is set in a specification to be 400 MPa, and the experimental result on ten test-pieces is 394 MPa with a coefficient of variation of ± 10%, the batch has to be deemed to have passed the specification at a confidence level equivalent to about two standard deviations, or about 95%.

Confidence in the mean value, the standard deviation and other measures of confidence in the result clearly improve with increasing numbers of test-pieces. However there are still limitations as outlined below.

8.2 Weibull analysis

It has been found that for many purposes, strength test results can be described by the Weibull distribution. This is explained in detail in Annex C. There are many approaches to using the Weibull distribution function, but the simplest method for an initial analysis is to use the so-called two-parameter method. If a probability of failure, P_f, is assigned to each strength test result, σ_f, when ranked in ascending order of strength, the Weibull distribution is:

$$P_f = 1 - \exp\left[- \left(\frac{\sigma_f}{\sigma_0} \right)^m \right] \qquad (2)$$

where: σ_0 = scaling factor, sometimes known as the **characteristic strength**
m = the so-called **Weibull modulus**

These two parameters describe respectively the position and width of the distribution, i.e. are measures of the scatter of strength important for predicting reliability.

There is a wide range of approaches to determining these parameters from a data set. The standards ISO 20501, CEN ENV 843-5 and ASTM C1239 give preferred procedures for analysing sets of data to obtain best fit values for the Weibull parameters based on the principles of maximum likelihood, which is explained in Annex C, section C.2.

Alternative methods are statistically a little less efficient, and may give slightly different answers. For example, it is popular to apply a linear regression fit to the ranked strength data when plotted as a double logarithmic function of the cumulative probability distribution, but this graphical approach leads to biased estimates of Weibull parameters.

Since one of the objectives of using Weibull analysis is to make some prediction of the risk of failure, especially at low failure probabilities, the reliability of such predictions depends on the confidence with which the parameters can be determined. Statistical analysis shows that the reliability of Weibull parameters is poor unless at least 30 valid test results are employed.

Weibull statistics have poor reliability on less than 30 test-pieces.

It is important to recognise that although the Weibull distribution is a special case of the exponential distribution, its application to any physical phenomenon is empirical. It assumes that a single type of largest-flaw distribution is present in the batch of test-pieces. This is often not the case. There may be several types of flaw, each with its own distribution, and each with a chance of initiating fracture in a strength test. For example, a material may have concurrently, inclusions, large pores, large grains, and machining damage. Their respective distributions may be such that some are present only in a small proportion of the test-pieces. The dominance of one flaw type over another can change with size of test-piece or component. Consequently, it becomes important to understand whether the two-parameter

Weibull distribution is an appropriate method of analysing the results. There are two stages to this process:

1. Make a Weibull plot (ln ln $(1/(1-P_f))$ *vs.* ln σ_f) and inspect it for linearity. If the points lie close to a straight line, then there is probably some validity in using the two-parameter Weibull analysis. If there is a distinct curvature or kink in the distribution of points, then there is a good chance that the two-parameter fit is inappropriate.[9]

2. Check each test-piece fractographically, and determine the type of fracture origin. Annotate the Weibull distribution with the flaw type. Examine the result and seek any evidence that one type of flaw is preferentially at one or other end of the distribution. If so, then there is a good argument for seeking an alternative means of analysis.

8.3 Value of Weibull analysis

Weibull parameters are **not necessarily** material characteristics. They can be a function of many variables that go into preparing and testing a set of test-pieces. They therefore have no general applicability to a given material, but may have value under clearly defined conditions. There are a number of cases in the scientific literature where it has been demonstrated that results from different sizes and shapes of test-piece are consistently represented by a single Weibull distribution (as reviewed in [9]), but mostly these are for well-defined materials subjected to close control of preparation methods, and where the critical flaws are of a distinct type and volume distributed such that preparing test-pieces does not alter the distribution. More generally, this will not be the case, because powder batches and processing conditions are not always sufficiently consistent, and variable surface preparation procedures used for test-pieces may variably influence the critical flaw distribution.

To deal with all the issues relating to the statistics of fracture is beyond the scope of this Guide, but the following key points should be noted:

- It is often the case that components contain occasional flaws which are not represented in test-bars, and typically, the scatter of strengths is wider than in a set of carefully prepared test-bars. The sampled volume of the test-bars ideally needs to be as close as possible to that of the component, *e.g.* by scaling the test-bar size, either up or down as appropriate.

[9] CEN ENV 843-5 contains a simple procedure for comparing the distribution of data points with defined limits of the combined confidence intervals on m and σ_0 to decide whether the two-parameter fit is valid for the purposes of the standard. ASTM C1239 gives an example of extending the same type of analysis to a bimodal distribution.

- It may be cheaper and more realistic to experiment with components than with test-bars combined with a risky extrapolation.

- Extrapolation of Weibull statistics significantly beyond the dimensional scale of the test-piece, or from uniaxial to multiaxial stressing, or to low probabilities of failure beyond those used to determine the Weibull distribution should not be contemplated without analysis of all potential factors.

- A Weibull extrapolation to low probabilities of failure is normally based on the expectation that the frequency of occurrence of other flaw types is zero. This cannot always be guaranteed unless components or test-pieces containing such potential flaws are eliminated by inspection.

- The basis of comparing two materials through the use of Weibull parameters requires careful thought. A high value of Weibull modulus coupled with a low mean strength may give better overall reliability to a population of components than a low Weibull modulus with a high mean strength. Much depends on the reliability level being sought.

- The experimental confidence interval widths on Weibull parameters are disappointingly wide, even for 30 test-pieces. The confidence interval needs to be taken into account when estimating the reliability of any extrapolated assessments.

9 Bibliography

[1] Roebuck, B.; Effect of test-piece geometry on the flexure strength of representative hardmetals and cermets, *14th Int. Plansee Seminar, May 1997, Reutte, Austria* **2**, 352–65.

[2] Roebuck, B.; Bend strength measurements for hardmetals, Part 1: Rationale and results, VAMAS Technical Report No. 22, June 1996, National Physical Laboratory.

[3] Roebuck, B.; Bend strength measurements for hardmetals, Part 2: Analysis of results, VAMAS Technical Report No. 31, October 1997, National Physical Laboratory.

[4] Quinn, G.D.; Flexure strength of advanced structural ceramics - a round robin, *J. Amer. Ceram. Soc.* 1990, **73**(8), 2374–84; see also US Army Report TR 89–62 [1989], US Army Materials Technology Laboratory, Watertown, Mass. USA.

[5] Tennery, V.; Ferber, M.K.; Fracture strength analysis of silicon nitride and silicon carbide ceramics from an international cooperation research program, Proc. 27th Contractors Coordination Meeting (23–26 October 1989), Society of Automotive

Engineers, Warrendale, PA, USA, pp. 265–74.

[6] Boch, P.; Freiman, S.W.; Fuller, E.R., Jr.; Interlaboratory round robin on environmental crack growth parameters, VAMAS Bulletin no. 8, July 1988, National Physical Laboratory, Teddington, Middlesex, UK, pp. 10–11.

[7] Ritter, J.E.; Service, T.H.; Guillemet, C.; Strength and fatigue parameters for soda-lime glass, *Glass Technol.* 1985, 26(6), 273–8.

[8] Giovan, M.N.; Sines, G.; Biaxial and uniaxial data for statistical comparisons of a ceramic's strength, *J. Amer. Ceram. Soc.* 1979, 62(9–10), 510–15.

[9] Quinn, G.D.; Morrell, R.; Design data for engineering ceramics: a review of the flexure test, *J. Amer. Ceram. Soc.* 1991, 74(9), 2037–66.

[10] Byrne, W.P., Morrell, R.; Results of the UK Interlaboratory strength test exercise, NPL Report CMMT (A) 296, November 2000.

Annex A - Stresses in beams and derivation of bending equations

A.1 Bending moments and curvature

Following Timoshenko's simple explanation [A.1], a thin beam bent to a small curvature ρ develops internal strains which are tensile on the convex side and compressive on the concave side, and which vary linearly through the thickness. Thus, in an element of beam of length *dx* at distance *y* from the neutral axis or plane of zero stress, there is an increase in length $y d\theta$ where $d\theta$ is the angle subtended by *dx* at the radius of curvature ρ. The axial stress developed is therefore:

$$\sigma_x = \varepsilon_x E = Ey \frac{d\theta}{dx} = \frac{E\,y}{\rho} \qquad \text{(A.1)}$$

assuming the material follows Hooke's law. Clearly there must be a balance of forces between the tensile and compressive sides of the beam, so the following integral must be zero:

$$\int_A \sigma_x \, dA = \int_A \frac{E}{\rho} y \, dA = 0 \qquad \text{(A.2)}$$

from which it is concluded that the neutral axis must pass through the centroid of the beam.

The moment of force on the area element *dA* at distance *y* from the neutral axis is $dM = y\sigma_x dA$, and the sum of these moments must equal the applied moment *M* resulting from external forces:

$$M = \int_A y \sigma_x \, dA = \frac{E}{\rho} \int_A y^2 \, dA \qquad \text{(A.3)}$$

The integral in this equation is called the **cross-sectional moment of inertia**, *I*. The basic relationship between beam curvature and the applied moments is therefore:

$$\frac{1}{\rho} = \frac{M}{EI} \quad where \quad I = \int_A y^2 \, dA \qquad \text{(A.4)}$$

From equation (A.1), the stress in the beam is given by:

$$\sigma_x = \frac{My}{I} \qquad \text{(A.5)}$$

i.e. is a maximum at the extremities from the neutral axis. For a rectangular beam of height *h* and width *b*, the cross-sectional moment of inertia can be calculated to be:

$$I = \frac{bh^2}{12} \qquad \text{(A.6)}$$

or for a circular rod:

$$I = \frac{\pi d^4}{64} \qquad \text{(A.7)}$$

The value for other cross-sections can be calculated by suitably evaluating the integral.

A.2 Stresses in thin beams

The bending moment applied to a beam is a result of the forces applied and the reactions at supports. In Figure A1 for symmetrical **three-point bending**, at a distance *x* from a support point at the origin, there are forces *F* applied at the centre and *F/*2 at each support. The bending moment at *x* is the sum of the moments of the forces to the right of this point, *i.e.*

$$M(x) = F(\frac{l}{2} - x) - \frac{F}{2}(l - x) = -\frac{Fx}{2} \qquad \text{(A.8)}$$

using the convention of clockwise moments being positive. When *x* is greater than *l* /2, the bending moment declines as -*F*(*l* - *x*)/2. Inserting equation (A.8) in equation (A.5) for the left-hand side of the beam yields:

$$\sigma_x = \frac{My}{I} = \frac{12}{bh^3}.\frac{Fx}{2}.y \qquad \text{(A.9)}$$

and the right hand side is symmetrical with this. The stress reaches a maximum at the centre of the beam, *i.e.* at *x* = *l* /2, and at the outer surfaces of the beam, *i.e.* at *y* = ± *h*/2. The nominal maximum stress in the beam is thus given by:

$$\sigma_{\max,3} = \pm\frac{3Fl}{2bh^2} \qquad \text{(A.10)}$$

which is treated as the maximum nominal fracture stress $\sigma_{f,3}$ when the beam breaks. Similarly for **four-point bending**, for the point *x* the bending moment *M* rises when *x* lies between the outer support point and the nearest loading roller, then remains constant at $-Fd_1/2$ where d_1 is the inner to outer roller distance to the second loading roller, and declines between the second loading roller and the second support roller. In the central region the maximum stress is therefore given by:

$$\sigma_{\max,4} = \pm \frac{3Fd_1}{bh^2} \qquad \text{(A.11)}$$

which, as for three-point bending, is treated as the maximum nominal fracture stress $\sigma_{f,4}$ when the beam breaks.

This analysis can also be used to determine the stress at any point in the beam, *e.g.* at a fracture origin which is away from the point of maximum stress, or inside the test-piece rather than at the surface.

Similar formulae can be developed for other types of cross-section simply by calculating the appropriate form of *I*.

A.3 Assumptions behind the thin-beam analysis

A.3.1 Assumptions

This analysis assumes a number of simplifying assumptions:

- a plane stress solution, *i.e.* there are no lateral stresses developed in the beam, and no restrictions on Poisson strains developing;
- the beam is thin, but the deflection is small compared with the thickness of the beam;
- shearing effects are ignored, which leads to errors in thick beams;
- no friction effects at supports or loading points.

In selecting the most appropriate geometry for test-pieces which permits the use of thin-beam equations without significant errors (< 2% overall), analyses have been done to optimise the geometry [A.1]. The most significant effects have been shown to be due to friction, twisting and thickness. The geometrical effects are considered here, and the other aspects related to undertaking the test are in Annex B.

A.3.2 Thickness effects

As the beam thickness increases, the forces required to produce a given level of thin-beam flexural stress increase, and the local stresses under the loading points begin to have a significant influence on the stress on the tensile face. Timoshenko [A.1] has show that a concentrated load on a beam produces so-called wedging stresses under the load point which modify the stress distribution on the opposite side. In the analysis a formula is derived:

$$\sigma_{\max,3} = \frac{3Fl}{2bh^2}\left(1 - \frac{4h}{3\pi l}\right) \qquad \text{(A.12)}$$

which shows that as the height of the beam h increases relative to the span l, the maximum true tensile stress decreases sharply. This deviation rapidly diminishes on moving away from the line of loading. For ISO 3327 Type B test-pieces 5.25 mm thick tested over a 14.5 mm span, this formula estimates a 16% decrease in real stress compared with the thin-beam assumption.

In the case of four-point bending, the situation is less severe because the individual loads at the loading points are halved and, unlike the sharp drop-off in stress away from the loading point in three-point bending, there is a nominally constant stress between the loading points. This can be perturbed significantly by wedging stresses. It becomes necessary to evaluate this as a function of position.

Reference [A.2] gives a more-detailed analysis for both three and four-point bending which similarly shows that immediately under the loading roller, there is a **reduction** in tensile surface stress, which is very significant in three-point bending. For ISO 3327 Type B test-pieces 5.25 mm thick tested over a 20 mm span, l/h is 2.8, and the error calculated is 6.4%, *i.e.* less than that given by Timoshenko. In four-point bending, there is a smaller reduction at this position than in the equivalent three-point bend situation, but a non-uniform **enhancement** of the thin-beam tensile surface stress between the loading rollers. For a ratio of $d_1/h < 3$ the enhancement exceeds 1% and for $d_1/h = 1$, is nearly 3%. For this reason, the span or loading arm to thickness ratio in the ceramic tests has been kept greater than 3 so that the potential error can be ignored.

For thick test-pieces, care needs to be taken concerning the corrections for thick-beam effects; they are different in three and four-point bending and are fracture location dependent.

A.3.3 Width effects

Anticlastic bending, or reverse curvature developed orthogonal to the main direction of bending, can occur because of Poisson contraction effects. In a thin, wide beam there is a risk that the natural anticlastic curvature will be flattened by the loading rollers, and thus introduce biaxial stresses influencing the net tensile stress experienced by the beam. An analysis of this effect has been undertaken [A.3], in which it is shown that the effect is negligible in beams of aspect ratio $b/h < 20$. For most purposes, therefore, the effect can be ignored.

A.3.4 Test-piece overhang

There must be some overhang of the test-piece beyond the support points otherwise there could be an interaction of the local stresses associated with the support contact with the free surface, which in a worst case could lead to the shearing of the test-piece end. Reference [A.1] states that such effects disappear if the overhang is of the order of the test-piece height, *i.e.* for a 40 mm span, 3 mm thick test-piece, there should be typically a 3 mm overhang at each end making the minimum recommended length 46 mm.

Make sure the beam is long enough. It should overhang the support rollers at each end by the beam thickness or there is a risk of the ends shearing.

A.3.5 Elastic anisotropy

The analysis assumes that the material has homogeneous elastic properties. When this is true, the tensile stresses developed are independent of the actual value of Young's modulus, even if this is anisotropic. Problems clearly arise if the material does not have spatial elastic homogeneity, producing inhomogeneous strains and deflections, and the resulting errors are essentially indeterminate.

Another situation is when the elastic behaviour is different on the tensile and on the compressive side of the beam. In this case, the neutral axis does not coincide with the geometrical centroid, but is shifted towards the higher modulus side. This situation can exist in fibre composite materials where the overall stiffness is governed by a microcracked matrix which has no stiffness in tension, but a significant contribution in compression. The microcracking can develop during the flexure test, even if it is not present before testing. Normally the effect is indeterminate on a beam, but if strain gauges are used on the tensile and compressive faces, and the surface strain is monitored, it is possible to deconvolute the separate tensile and compressive behaviours and thus identify the respective elastic moduli as a function of load [A.4]. This is outside the scope of this Guide.

Thin beam equations assume elastic isotropy and homogeneity. Special care needs to be taken with inhomogeneous materials.

A.4 Finite element analysis (FEA) of non-thin beams

Analytical studies of stress distributions have some limitations, and a clearer visualisation can be achieved using FEA. As part of the research at NPL, a study of the stress distributions within beam test-pieces was conducted under subcontract[10].

Using the standard four-point flexural geometry for Size B test-pieces in CEN EN 843-1 and ASTM C1161 (40 mm span, 3 mm thick, 4 mm wide test-pieces, 40 mm span, 20 mm loading span), the stress distribution in the central span was found to match exactly the thin-beam analytical solution except near the loading rollers. Here, there was a small axial tensile stress enhancement of about 1% peaking 1.5 mm inside the loading roller. This is because of finite so-called 'wedging' stresses.

As the test-piece thickness to span ratio increases, the true tensile surface stress at the mid-point of the tensile surface increases above the thin-beam value but the stress enhancement near the loading roller becomes less localised, and under the loading roller declines in accordance with the Timoshenko analysis. For the particular case of quarter-point bending ($d_1 = l/4$), the maximum enhancement in the central span is 3% for l/h = 5, and 8% for l/h = 2.

A.5 Shearing effects

Timoshenko [A.5] has shown that the shear stress acting inside the beam is parabolic in nature, and of magnitude:

$$\tau = \frac{V}{2I}\left(\frac{h^2}{4} - y^2\right) \qquad \text{(A.13)}$$

where y is the distance from the neutral axis and V is the shearing force at the section in question. The shearing force at any section x is built up as the sum of all the external forces applied perpendicular to the beam to one side of that point, being balanced by the internal shearing forces. Thus in a beam in three-point bending, the net shearing force is a constant $F/2$, with the result that the maximum shear stress at the neutral axis is given by:

$$\tau_{\max} = \frac{Fh^2}{16I} = \frac{3F}{4bh} \qquad \text{(A.14)}$$

independent of position along the beam length. The ratio of maximum shear stress to maximum tensile stress:

[10] Dr J Margetson, Defence Research Consultancy, using 2D FEA analysis.

$$\frac{\tau_{\max}}{\sigma_{\max,3}} = \frac{h}{2l} \qquad \text{(A.15)}$$

is normally quite small since $l >> h$.

In the case of four-point bending, the shear effects are solely between support and loading rollers, and there is no shear in the central region between the loading points.

FEA of shear stresses demonstrates the parabolic distribution through the thickness, and an increase in maximum shear stress with increasing beam thickness for a given maximum tensile stress on the outer surface. However, as the beam thickness increases an additional factor becomes significant. The forces applied to the beam increase, and local stresses under the points of contact increase. When the loading arm to height ratio d_l/h rises above 0.5, the maximum shear stresses appear associated with the loading points, and are of greater magnitude than those at the neutral axis.

In a monolithic ceramic or hardmetal material the shear strength is typically one third of the tensile strength, so shear stresses are therefore of little importance, and can be ignored. However, if the shear strength of the material is low, such as in fibre composites with the fibre plane aligned parallel to the length of a beam test-piece, it can readily be seen that shear stresses can be of significance and lead to shear delamination as the primary source of failure, rather than tensile fracture. For this reason, the depth to span ratio in fibre composites needs to be less than the shear strength to tensile strength ratio if the flexural test is to give a result reflecting tensile failure. Since the shear strength is often not known at the outset of a series of tests, a ratio of 1:40 or less is recommended in such cases.

A.6 Curved beams

The above analysis assumes a straight beam. When a beam is curved (assuming downward curvature in the direction of bending) but the deflections caused by loading remain small, the stress on the tensile side is enhanced relative to the value for a straight beam. Following an analysis by Timoshenko for a pure bending situation, Reference [A.2] gives an evaluation which indicates that a significant (> 1% error) arises only when the radius of curvature to beam height ratio is less than about 40, reaching 10% when this ratio is about 3. Consequently, an ENV 843-1 size B or ASTM C1161 type B test-piece can be out-of-straight over a 40 mm span by as much as 1.7 mm without significant influence on the test result.

A.7 Large deflections

This is a complex situation that tends to arise with very thin test-pieces, or materials with a very high strength to modulus ratio. Ref. [A.2] reviews existing literature and comments that several factors can occur in principle. Firstly, the test-piece rolls round the support rollers and gives an effective support span change, also referred to as the ’tangency point shift’. Secondly, the system cannot then be totally frictionless because the forces to be supported are at an angle to the surfaces on which rollers are supported. It is concluded that in a worst-case scenario of a brittle material with a strength : modulus ratio of 0.002 the large-deflection error rises above 1% when the beam span to height ratio reaches about 80 for four-point bending and 100 for three-point bending. Normally this aspect ratio is never achieved, (values are typically about 15 or less) except in unusual circumstances.

A.8 Bibliography

[A.1] Timoshenko, S.; *Strength of Materials, Part II - Advanced theory and problems*, Robert Krieger, Huntington, NY, 1958 (reprint 1976).

[A.2] Baratta, F.I.; Quinn, G.D.; Matthews, W.T.; Errors associated with flexure testing of brittle materials, Technical report TR 87-35, July 1987, US Army Materials Technology Laboratory, Watertown, Mass. USA.

[A.3] Baratta, F.I.; When is a beam a plate? *J. Amer. Ceram. Soc.* 1981, **65**(5), C-86.

[A.4] Lord, J.D., Roebuck, B.; McCartney, L.N.; Cooper, P.M.; *Proc. BNF 7th Intnl. Conf., The materials revolution through the 90s - powders, metal matrix composites, magnetics, Oxford University, July 1989,* Vol. 1, Paper 26. Wantage, UK: BNF Ltd.

[A.5] Timoshenko, S.; Young, D.H.; *Elements of strength of materials*, 4th edition, Van Nostrand, New York, 1962.

Annex B - Errors in flexural testing

B.1 Friction effects

It has been known for some time that bending a test-piece between fixed points introduces a potential systematic error due to frictional forces. For a very thin test-piece in which the flexural displacement is large, the arc length of the test-piece is longer than the span, and this tends to drag on the supports and to introduce a net tensile force on the test-piece. Conversely, if the test-piece is thicker, and does not deflect greatly, as is usually the case for typical flexural strength test-pieces, the reverse happens. The arc length of the test-piece increases because of the elastic strain developed, and tends to push outwards on the supports (and inwards on the loading points in four-point bending). If the rollers supporting (and loading (four-point bending)) the test-piece are not free to roll to accommodate this change, a lateral frictional force is developed at the contact points which is proportional to the applied force. This is equivalent to placing an additional bending moment on the test-piece [B.1, B.2]:

$$M = \frac{Fd_1}{2} - \frac{\mu Fh}{2} = \frac{F}{2}(d_1 - \mu h) \qquad \text{(B.1)}$$

where d_1 = loading arm
h = test-piece thickness
F = total applied force on test-piece
μ = coefficient of friction

Since the stress in the test-piece is proportional to the bending moment, there will be a proportional error, ε, in the stress:

$$\varepsilon = 100 \times \left[\frac{\mu}{(d_1/h) - \mu} \right] \qquad \text{(B.2)}$$

expressed as a percentage. For a typical size B test-piece with d_1 = 10 mm, h = 3 mm, a low coefficient of friction of 0.1 gives an error of 3%. A high coefficient of friction of 1 gives an error in excess of 40%! This is equivalent to a rigid connection. A more typical value of μ for dry contact between a ceramic and a hard, unlubricated metal is about 0.3, for which an error of 10% would be predicted.

FEA of the four-point situation has confirmed that the stress distribution in the tensile face of the beam is modified, with 6% and 12% errors for μ = 0.2 and 0.4 respectively, exactly in line with the above analysis.

The magnitude of the sliding and rolling friction coefficients between various curved and flat ceramic surfaces has recently been established by NPL [B.3, B.4] up to high temperatures.

Friction effects can be force dependent, and can be very variable with temperature and with surface finish on both the test-piece and the supports. In contrast, there is negligible friction associated with rolling. This demonstrates that the best route to avoiding errors is to use rolling rollers.

B.2 Twist errors

If the test-piece is twisted, or the rollers not aligned, the test-piece is not loaded uniformly along its contact with the roller. If the rollers cannot articulate to accommodate the misalignment, a torsional force is applied to the test-piece as it is loaded, limited by whether the test-piece twists sufficiently to contact the loading points across the full width. Error estimation is complex [B.1]. The addition of a torsional stress to a tensile stress on the convex side of the test-piece twists the angle of maximum principal stress from parallel to the test-piece axis to an angle θ. Following Baratta *et al.* [B.1], the maximum principal stress, σ_1 is given by:

$$\sigma_1 = \frac{\sigma_x}{2}\left(1 + (1/3k_2)\,[(n_1 b/d_1)^2 + 9k_2^2]\right)^{1/2} \qquad \text{(B.3)}$$

where:
- σ_x = maximum flexural stress in the absence of twist
- k_2 = a dimensionless numerical constant depending on the ratio of b to h
  = 0.23 for 4 × 3 mm cross-section test-pieces
- n_1 = 1 if the test-piece does not fully contact the rollers, and
  < 1 if the test-piece fully contacts part way through the test, given by:

$$n_1 = [3k_1(E/\sigma_f)/(1+\nu)][(h/L_T)\,\phi_s + (h/d_1)\,\phi_f](d_1/b) \qquad \text{(B.4)}$$

where:
- σ_f = flexural strength of test-piece
- k_1 = second dimensional constant depending on the ratio of b to d
  = 0.18 for 4 × 3 mm cross-section test-pieces
- L_T = total test-piece length
- E = Young's modulus
- ν = Poisson's ratio
- φ_s = angular twist of test-piece along its whole length, in radians
- φ_f = angular twist of test-piece between an inner and an outer load point, in radians

For an alumina with E = 386 GPa, υ = 0.22, in an EN 843-1 or ASTM C1161 size B geometry test-piece, it can be shown that angular twists of as little as 0.2° between the support rollers can result in the maximum effect of twisting (no conforming of the test-piece), with an error in the maximum stress of 7.5%. The maximum principal stress is higher than the nominal stress, leading to an underestimate of strength [B.2]. In addition, the plane of fracture tends to be perpendicular to the maximum principal stress, rather than straight across the test-piece. This fact can be used to diagnose that a twist error exists in practice, and the angle of the plane to the normal direction is a measure of the magnitude of the error.

There are therefore considerable advantages in using an articulating test-jig. The effects of small imperfections in either jig or test-piece manufacture can be eliminated using articulation. If articulation is impractical for any reason, the required perfection of co-planarity of support and loading rollers and of the parallelism of test-piece surfaces can be estimated.

B.3 Eccentric or uneven loading

If the load is not applied centrally in three-point bending, or symmetrically in four-point bending, the maximum stress in the beam is changed. This situation can arise particularly if the loading points are rigidly connected to the cross-head of the testing machine and the support points to the support platen such that there is no automatic centralisation. The problem is worsened if the loading head in four-point bending cannot articulate to apply even forces to the two loading rollers (Figure B.1). This situation is fairly straightforward to evaluate based on simple beam theory by using an asymmetrical geometry, and can be found in Reference [B.1], and a summary of the percentage errors appears in Table B.1.

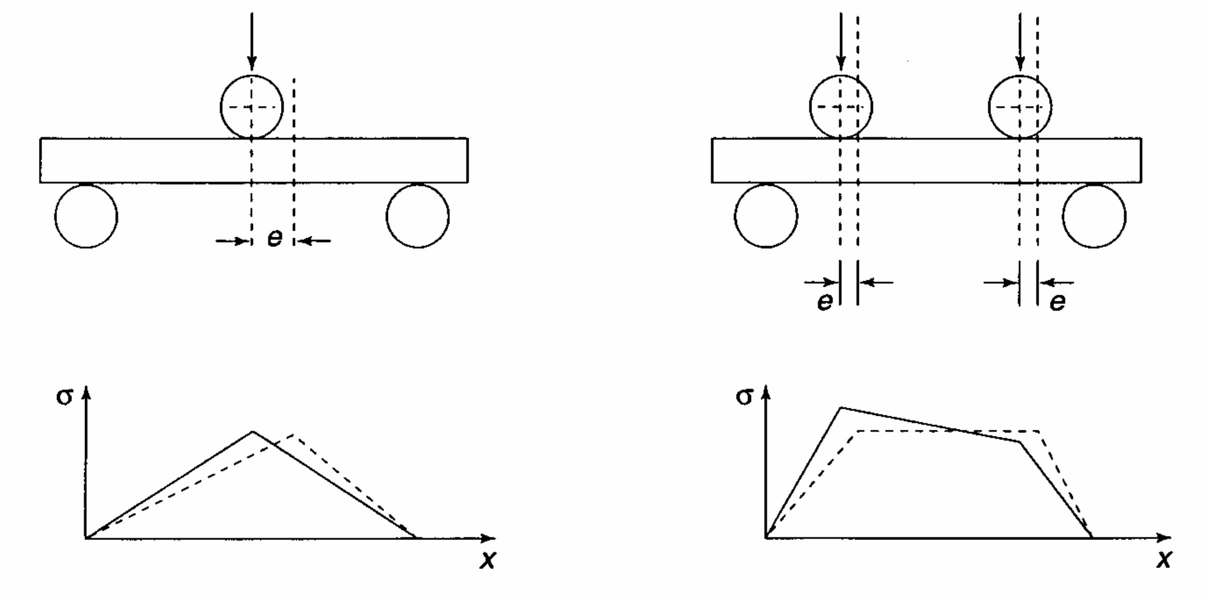


Figure B.1 *Changes in tensile surface stress distribution due to lateral misalignment, e, in three and four-point bending. The effect is more severe in four-point bending.*

For three-point bending, inspection of Table B.1 will show that for a given level of eccentricity, e.g. 1 mm in a 40 mm span giving e/l = 0.025, the stress under the loading roller declines, but negligibly, and the change only becomes significant if the eccentricity exceeds 2 mm.

For four-point bending, Table B.1 shows that there is a substantial difference between rigidly aligned loading heads and heads able to pivot to take up asymmetrical displacements. This arises because the loading applied by the two loading rollers is different in the two cases. In both cases, the stress under the loading roller nearer the support roller increases, and that under the other decreases. The changes are test-piece geometry dependent, and more significant for quarter-point bending than for third-point bending. Large errors very rapidly accumulate for non-pivoting heads, and hence they should never be used for flexural strength testing. For eccentricities of 1 mm, the error in assuming the thin-beam equation can approach 20%, and lead to a severe underestimate of strength. The errors for pivoting heads are much smaller than for non-pivoting heads, but still rather larger, and of opposite sign, compared with three-point bending. For the 40/20 mm geometry, an eccentricity greater than 0.2 mm results in a stress error of more than 1%, hence the requirement for accurate centralisation.

Table B.1 - Errors due to eccentric loading [estimated from reference B.1]

Lateral displacement error to span ratio, $e/l = (d_1' - d_1)/l$	% error in maximum stress, $(((\sigma_e - \sigma_{max})/\sigma_e) - 1) \times 100$				
	Three-point bending	Four-point bending, third-point ($d_1 = l/3$)		Four-point bending, quarter-point, ($d_1 = l/4$)	
		Non-pivoting	Pivoting	Non-pivoting	Pivoting
0	0.0	0.0	0.0	0.0	0.0
-0.005	-0.05	3.4	0.50	5.0	1.0
-0.010	-0.10	6.3	0.95	8.8	1.9
-0.015	-0.15	9.0	1.4	12.0	2.8
-0.020	-0.20	11.3	1.8	14.7	3.7
-0.025	-0.25	13.3	2.15	17.0	4.5
-0.030	-0.30	15.1	2.45	~20	5.2
-0.050	-1.0	~20	~3.5	~25	7.2

Note: e/l is negative as the loading roller moves towards the support roller from the symmetrical position. In four-point bending the stress increase occurs for the roller for which e/l is negative, and decreases under the roller for which e/l is positive. Third-point loading is used in JIS R1601 and ISO 14704, and quarter-point loading in ISO 14704, EN 843-1 and ASTM C1161.

B.4 Errors due to chamfer size

The basic bending equations ignore the presence of edge chamfers and assume a true rectangular section. The effect of making chamfers on the bars to minimise the effects of edge damage is to change the cross-sectional moment of inertia, *I*. Reference [B.1] illustrates the procedure and gives equations for estimating the error for ignoring chamfers, either of radius *r* or of width *c* (measured across test-piece face) machined at 45° to the faces (see Figure B.2). The cross-sectional moment of inertia, *I*, in equation A.9 is modified by the effect of the chamfer:

$$I(c) = \left(\frac{bh^3}{12}\right) - \left(\frac{c^2}{9}\right)\left[c^2 + \frac{(3h-2c)^2}{2}\right] \qquad \text{(B.5)}$$

The true surface stress is enhanced by the presence of chamfers because *I* is reduced, and hence the strength of a test-piece is underestimated. Table B.2 shows the error, which is a function of test-piece width *b*.

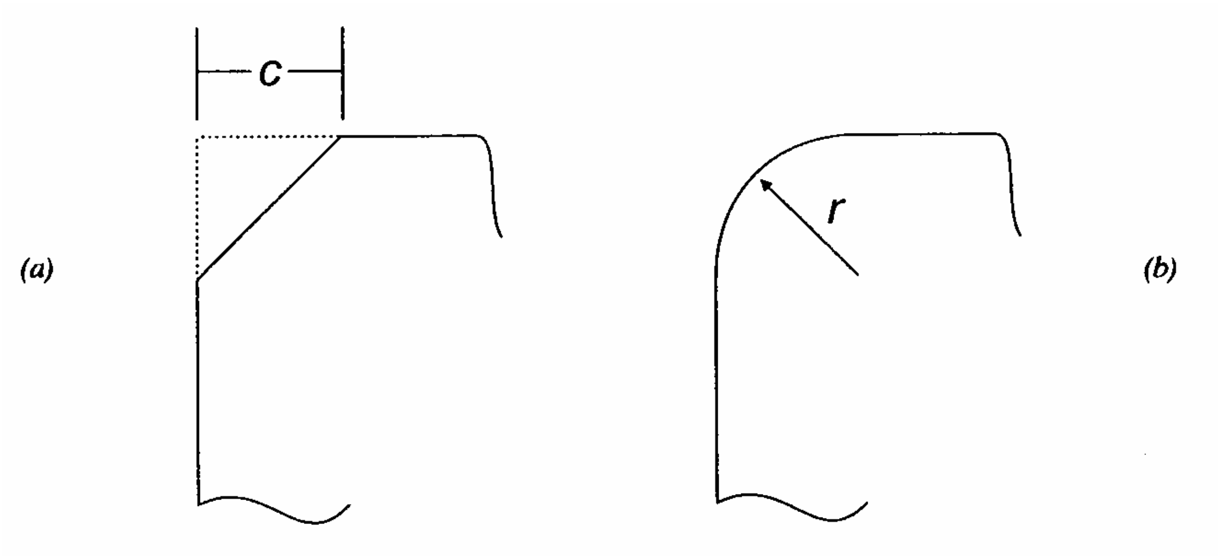


Figure B.2 *(a) 45° flat chamfer (size c) or (b) rounded chamfer (radius r) on test-pieces. If only two edges are chamfered, these should be the edges subjected to tensile stress.*

In order to keep the stress error below 1% for effective use of the uncorrected beam equations, this table indicates that the chamfers should not be of size greater than r/h = 0.07, and c/h = 0.048 for b/h = 1.33. For the standard size ceramic test-bars in ENV 843-1 and ASTM C1161 size B test-pieces, these translate to $r \leq 0.2$ mm and $c \leq 0.15$ mm. If the chamfers are larger than these dimensions, a correction should be made.

In ISO 3327 (BS EN 23327) for hardmetals, chamfers of 0.4 to 0.5 mm placed on bars before firing require strength correction factors of 1.03 (*i.e.* 3% underestimate) on 5 x 5 mm cross-section type A test-pieces and 1.02 (*i.e.* 2% underestimate) on 6.5 x 5.5 mm cross-section type B test-pieces. [Note that there is no definition of how a 45^o chamfer is measured in this standard. These corrections correlate with Table B.2 if the chamfer is measured across the 45^o angle.]

Table B.2 - Strength errors resulting from ignoring edge chamfers (all four edges)

Chamfer size relative to height of test-piece, *r/h* or *c/h*	% error in assuming no chamfer							
	Radiused chamfers, for *b/h* =				45^o chamfers for *b/h* =			
	1.00	1.33	2.00	4.00	1.00	1.33	2.00	4.00
0.00	0.00	0.00	0.00	0.00	0.0	0.0	0.0	0.0
0.01	-0.03	-0.02	-0.01	-0.01	-0.1	-0.0	-0.0	-0.0
0.02	-0.10	-0.08	-0.05	-0.03	-0.2	-0.2	-0.1	-0.1
0.03	-0.23	-0.17	-0.11	-0.06	-0.5	-0.4	-0.2	-0.1
0.04	-0.40	-0.30	-0.20	-0.10	-0.9	-0.7	-0.5	-0.2
0.05	-0.52	-0.46	-0.31	-0.15	-1.4	-1.1	-0.7	-0.4
0.06	-0.88	-0.66	-0.44	-0.22	-2.0	-1.5	-1.0	-0.5
0.07	-1.19	-0.89	-0.59	-0.30	-2.7	-2.0	-1.3	-0.7
0.08	-1.53	-1.15	-0.77	-0.38	-3.4	-2.6	-1.7	-0.9
0.10	-2.35	-1.77	-1.18	-0.59	-5.2	-3.9	-2.6	-1.3
0.15	-5.06	-3.80	-2.53	-1.27				
0.20	-8.60	-6.45	-4.30	-2.15				

B.5 Non-parallel faces on test-pieces

If the production method for test-pieces yields flat but non-parallel faces, this introduces non-uniformity to the stress field.

In three-point bending, assuming an articulating test-jig which takes out misalignment, the principal error involved is in the non-uniformity of stress field across the width on the tensile side at, or close to, the loading roller. To a first approximation, the maximum in the stress field will occur at the thinnest point of the test-piece. If the thickness here is 1% less than the average in the centre of the width (0.03 mm on a 3 mm thick test-piece), this incurs a 2% enhancement of strength above the expected mean.

If the test-jig does not permit articulation of the loading roller relative to the support rollers, the situation is changed to more like the twisting problem, with a concentration of stress on the thicker side of the test-piece until contact is achieved across the full width.

In four-point bending, in addition to variations in thickness over the width, variations in thickness over the length are more important than in three-point bending. The stress field changes along the central span, and there will be a tendency for fracture to occur under the loading roller where the thickness is least and the stresses therefore highest.

For reliable testing, make sure the test-pieces have parallel faces to within the tolerances given in the standards.

B.6 Small test-pieces

As the size of test-pieces and the associated test-jig is reduced, it is practically difficult to scale the tolerances on dimensions and positional accuracy in the same way. For example, in order to maintain an overall accuracy of 2% in the strength result, the precision of parallelism and dimensional measurement must be improved to retain the same fractional error as for larger test-pieces. Since it can be difficult and expensive to achieve higher levels of precision because of handling and manufacturing limitations, the accuracy of the strength result tends to get poorer. A recent study [B.5] has reviewed the build-up of errors, and concluded that they approach 10% (totalling all sources) for a test-piece of 2 x 1.5 x 13 mm span using 1 mm diameter rollers in four-point bending. Faced with the situation of having to use such test-piece sizes, accurate results can come only with considerable attention to the detail of test-piece preparation and test-jig design.

B.7 Bibliography

[B.1] Baratta, F.I.; Quinn, G.D.; Matthews, W.T.; Errors associated with flexure testing of brittle materials, Technical report TR 87-35, July 1987, US Army Materials Technology Laboratory, Watertown, Mass. USA (reprinted March 1997).

[B.2] Quinn, G.D.; Twisting and friction errors in flexure testing, *Cer. Eng. Sci. Proc.* 1992, **13**(7/8), 319–30.

[B.3] Cox, J.M.; Gee, M.G.; Hot friction testing of ceramics, *Wear* 1997, **203–204**, 404–17.

[B.4] Cox, J.M.; Morrell, R; Gee, M.G.; Direct measurement of friction applicable to hot strength measurements, in *Euroceramics 5*, 1997, published as *Key Engineering Materials*, 1997, **132–6**, 476–9.

[B.5] Lube, T.; Manner, M; Development of a bending test device for small samples, in *Euroceramics 5*, 1997, published as *Key Engineering Materials*, 1997, **132–6**, 488–91.

Annex C - Statistical analysis of fracture data

C.1 Basics

The variation in flaw type, size, shape and orientation in a brittle material leads to a scatter in strength, and it is generally the case that the strength of any one component or test-piece is indeterminate until broken. The average strength of a batch is therefore a simple measure of roughly how strong the material can be expected to be, but this says nothing of the scatter in strengths, and hence there is no information on the risk that one or more components in a batch has an unacceptably low strength. Statistical analysis of the distribution of strengths may therefore be required, and there needs to be an assumption made concerning the characteristics of the population of 'worst' flaws in the batch. In the absence of information to the contrary, it is usual to start with the assumption that all the critical flaws are of one type. While in principle a number of different distribution types might be used to characterise the population, experimentally it has been found that the Weibull distribution gives empirically a useful flexible fit to many data sets, and is therefore the most commonly used descriptor of the scatter of strengths.

C.2 Weibull statistics

The function $g(x)$ is a probability density function for the continuous random variable x if:

$$g(x) \geq 0 \qquad \text{(C.1)}$$

and:

$$\int_{-\infty}^{\infty} g(x)\,dx = 1 \qquad \text{(C.2)}$$

The probability, P, that the random variable x assumes a value between u and w is given by:

$$P\,(u < x \leq w) = \int_{u}^{w} g(x)\,dx = G(w) - G(u) \qquad \text{(C.3)}$$

where G is the cumulative distribution function. The continuous random variable x has a two-parameter Weibull distribution if the probability density function is given by:

$$g(x) = \left(\frac{m}{\beta}\right)\left(\frac{x}{\beta}\right)^{m-1} \exp\left[-\left(\frac{x}{\beta}\right)^{m}\right] \qquad x > 0 \qquad \text{(C.4)}$$

$$g(x) = 0 \qquad x \leq 0 \qquad \text{(C.5)}$$

These correspond with a cumulative distribution function:

$$G(x) = 1 - \exp\left[-\left(\frac{x}{\beta}\right)^m\right] \qquad x > 0 \tag{C.6}$$

$$G(x) = 0 \qquad x \le 0 \tag{C.7}$$

where: m is the Weibull modulus or shape parameter (> 0)
β is the scale parameter (> 0)

The random variable representing the fracture strength of a ceramic test-piece will assume only positive values, and the distribution is asymmetric about the mean. These characteristics rule out the use of the normal distribution amongst others, and point to the use of the Weibull distribution or similar skewed distributions. The assumption usually made is that the Weibull distribution will approximate to the true distribution of strengths observed.

A more common representation of this equation is:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma_f}{\sigma_0}\right)^m\right] \tag{C.8}$$

where:
σ_0 = scaling factor, sometimes known as the **characteristic strength**
m = the so-called **Weibull modulus**
P_f = a probability of failure
σ_f = the fracture strength as a variable

Determining m and σ_0 from a set of data can be done in a number of ways, but the most efficient method is considered to be the maximum likelihood method. Standards now exist for this method which include computer algorithms for iteratively establishing the best fit values: ISO 20501, EN 843-5 and ASTM C1229.

The likelihood function for a single critical flaw distribution is given by the expression:

$$\Lambda = \prod_{j=1}^{N}\left(\frac{m}{\sigma_0}\right)\left(\frac{\sigma_{fj}}{\sigma_0}\right)^{m-1}\exp\left[-\left(\frac{\sigma_{fj}}{\sigma_0}\right)^m\right] \tag{C.9}$$

where N is the number of fracture data.

This function is maximised by differentiating the log likelihood (ln (Λ)) with respect to m and σ_0, and setting these functions to zero yielding, respectively, estimates $\hat{m}$ and $\hat{\sigma}_0$, for m and σ_0:

$$\frac{\sum_{j=1}^{N} \sigma_{fj}^{\hat{m}} \ln \sigma_{fj}}{\sum_{j=1}^{N} \sigma_{fj}^{\hat{m}}} - \frac{1}{N} \sum_{j=1}^{N} \ln \sigma_{fj} - \frac{1}{\hat{m}} = 0 \qquad \text{(C.10)}$$

and

$$\hat{\sigma}_0 = \left[\left(\sum_{j=1}^{N} \sigma_{fj}^{\hat{m}} \right) \frac{1}{N} \right]^{1/\hat{m}} \qquad \text{(C.11)}$$

Equation C.10 must be solved numerically to obtain a solution for $\hat{m}$, which can then be used to solve for $\hat{\sigma}_0$ through Equation C.11. Computer routines for doing this to a given level of accuracy are available in the standards.

C.3 Weibull distributions

The maximum likelihood method does not produce a visualisation of the fitted distribution. To accomplish this, recourse has to be made to alternative methods of assigning a value of P_f to a given fracture strength σ_f. The standards recommend that if the N strength data are ranked in ascending order, the ith value is assigned a probability of failure $P_{fi} = (i - 0.5)/N$. Equation (C.8) can be rearranged as:

$$\ln \ln(1/(1-P_f)) = m \ln \sigma_f - m \ln \sigma_0 \qquad \text{(C.12)}$$

Plotting the left-hand side of this equation against ln σ_f should give a straight-line fit to the data with slope m and intercept $-m$ ln σ_0. The standards (e.g. ENV 843-5, ASTM C1239) recommend that the maximum likelihood derived optimised values are used to plot the best fit line. Although it is possible to use a least-squares fit to the plotted data, this is not recommended because unless a weighting function is used, the outlying points at the ends of the distribution gain undue influence in determining the results.

The standards also go on to require the confidence intervals on the results to be determined. These can be surprisingly large at, say, the 95% level, even for 30 strength data points, and is illustrative of the uncertainties in dealing with the statistics of failure.

Deviations from a straight line can occur for a number of reasons:

- The distribution of strengths is not of Weibull type for some reason.
- There is more than one distribution of flaw types causing failure, characterised by different values of *m* and/or σ_0.
- There is a minimum strength amongst the population as a result of previous elimination of obviously defective test-pieces or components by inspection or by mechanical proof-testing.

If it is thought that a minimum strength level σ_u exists, typically when there is a downward curve to the plot at low stress levels, a fit can be made by inserting this third parameter into the Weibull distribution equation:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma_f - \sigma_u}{\sigma_0}\right)^m\right] \quad \text{(C.13)}$$

However, it should be noted that:

When using the three-parameter Weibull distribution, different values of *m* and σ_0 will be obtained which are not comparable with the two-parameter Equation C.8.

Commonly the plot may appear bimodal. If fractographic evidence suggests that there is justification for separating the set of data into two groups failing from different fracture origin types, *e.g.* machining flaws and inclusions, then this can validly be done and each set separately analysed. (It should **not** be done simply by inspecting the data and cutting them into two groups near a significant change in slope in the plot, since both parts may contain both types of fracture origin.) The total distribution is then given by:

$$P_{f,A+B} = 1 - [1 - P_{f,A}][1 - P_{f,B}] \quad \text{(C.14)}$$

where A and B represent the two separated populations. The separated distributions can be analysed to obtain values of m_A, m_B, $\sigma_{0,A}$, $\sigma_{0,B}$, which are then characteristic of the two types of flaw population.

C.4 Using Weibull data for design purposes

This is a complex mathematical field and is best left to the statistical experts! Some key points will be made here.

C.4.1 Extrapolation to low probabilities of failure

A Weibull plot can be extrapolated to low probabilities of failure to seek a safe stress level for design purposes. However, this has to be done cautiously because usually the confidence in *m* is poor. Normally this is not done without additional factors being taken into account, including those described briefly below.

C.4.2 Volume and area effects

The critical flaws may be volume distributed, such as pores and inclusions, or may be surface related, such as machining damage, or even a combination of several types. If the size of the test-piece or component is changed while retaining the same flaw distributions, the probabilities of failure will change, reflecting a change in the chances of meeting flaws of a given size. There is thus a stressed-area dependence of strength for surface flaws, and a stressed-volume dependence for volume flaws. There is a number of approaches to analysing this, a common one being the stress-volume integral and the unit volume tensile strength concept (and their surface area equivalents for surface distributed flaws). Details of manipulations using these relationships will be found in the literature [C.1 to C.4]. One of the significant ones is the relationship between three and four-point flexural strengths obtained from the same type of test-piece over the same span *l*:

$$\frac{\sigma_{f,3}}{\sigma_{f,4}} = (m+2)^{1/m} \qquad \text{(C.15)}$$

For $m = 10$, this ratio has the value 1.28, i.e. the three-point flexural strength can be expected to be 28% greater than the four-point flexural strength. The ratio is sensitive to the value of *m*, declining with increasing *m*.

In principle, such relationships can be used to extrapolate from test-bars to components, but in doing so there is a presumption that the controlling flaw population remains the same. In practice, large pieces may have occasional defects which are not found to a significant level in small test-pieces, so a guideline is to make sure that the test-pieces fully reflect the occurrence of flaws appearing in components, perhaps by making them in a size similar to the components, or processing them in exactly the same way, or cutting them from components provided the surface preparation does not influence the result. The greater the level of extrapolation the less reliable the prediction is likely to be.

C.4.3 Stressing rate effects

The dynamic test completed in a few seconds does not give an indication of how the material will withstand stress over a longer period of time. In most ceramic materials, but not to any extent in hardmetals or cermets, the small strength controlling flaws can grow under stress, especially enhanced by the presence of atmospheric moisture. This means that over longer timescales, the strength is reduced. The critical crack growth parameter *n* in the relation:

$$v = A\left(\frac{K_I}{K_{Ic}}\right)^n \tag{C.16}$$

where K_I = the stress intensity factor at a flaw tip
K_{Ic} = the critical stress intensity factor for fast fracture
A = constant

typically has values in the range 10 to several hundred, with low values being obtained for materials which are more prone to this effect. It can be shown that assuming the above empirical relationship to be valid, the mean strengths $\bar{\sigma}$ of two batches of test-pieces tested at different stressing rates $\dot{\sigma}$ can be related by:

$$\frac{\bar{\sigma}_1}{\bar{\sigma}_2} = \left(\frac{\dot{\sigma}_1}{\dot{\sigma}_2}\right)^{1/n} \tag{C.17}$$

Similar relationships can be derived for static loading (stress-rupture) flexural testing. The effect of a finite value of *n* has to be taken into account in design where long-term loading is envisaged.

C.4.4 Multiaxial stressing

The stress distribution in a flexural strength test-piece is essentially uniaxial, but in components, the stress distribution may be multiaxial, and the design methodology needs to take this into account. There are a number of ways of treating the flaw distribution information to take multiaxiality into account, but usually a criterion has to be adopted for doing so, and therein lie a number of assumptions about how the effects of stresses acting in different directions are combined. This is an area which is not well understood, and the criteria best adopted may be material dependent.

C.5 Bibliography

[C.1] Quinn, G.D.; Morrell, R.; Design data for engineering ceramics: a review of the flexure test, *J. Amer. Ceram. Soc.* 1991, **74**(9), 2037–66.

[C.2] Creyke, W.E.C.; Sainsbury, I.E.J.; Morrell, R.; *Design with non-ductile materials*, Applied Science Publishers, London, 1982.

[C.3] Jayatilaka, A. de S.; *Fracture of engineering brittle materials*, Applied Science Publishers, London, 1979.

[C.4] Stanley, P.; Sivill, A.D.; Fessler, H.; The unit strength concept in the interpretation of beam test results on brittle materials, *Proc. Inst. Mech. Eng.* 1976, **190**(49/76), 585–95.