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Machine Design Requirements for Uniaxial Testing of Ceramics Materials

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1 INTRODUCTION

Ceramics as a group of materials challenge us with unique problems in material testing. As new materials and better processing are developed the need for conventional engineering design data is growing. If these new materials are to reach their full potential, solutions to these testing problems must be found so that a database of information can be established and trusted by designers.

Unlike most metals, ceramics tend to be very 'unforgiving' towards imperfect test methods. As a result, data scatter can be large and test results questionable. A literature search of work done during the 1960s and 1970s quickly reveals some of the problems encountered, especially with respect to tensile testing. The key to successful testing of these materials is to design the system around the special requirements of a particular test mode. Hence, flexure, compression, tension, tension/compression or fibre testing may all require slightly different systems to perform the test properly. Often with metal testing, selection of the appropriate grip and a Universal Testing Machine was all that was necessary to produce good test results. This is seldom the case with ceramics, however, where all aspects of the testing system must be chosen carefully to obtain valid results.

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2 UNIDIRECTIONAL FLEXURE TESTING

2.1 General

The flexure (3- and 4-point bending) test has been used for many years as a method of assessing the mechanical properties of brittle materials. The main advantages of the technique are as follows:

— Simple specimen geometry
— Simple fixture design
— Suitable for a wide range of testing machines
— Easily adapted for high temperature tests
— Deflection measurement is straightforward
— Easily automated for multispecimen test rigs
— Flexibility (strength, creep and cyclic fatigue properties can be studied)

2.2 Testing system requirements

There are no special problems involved in using entirely standard, commercially available testing machines of the well-known 'universal' variety. Testing speeds of 0·5 mm/min to 5 mm/min are typical of those used for flexure tests and this is well within the range of both electro-mechanical and servo-hydraulic systems. Modern electro-mechanical machines are available to cover position controlled speed ranges of 0·001 mm/min to 1000 mm/min in one unit so it can be seen that the ceramics flexure test is not demanding in this respect. It is important that a closed loop system of crosshead speed control is used in order to ensure a known and constant speed. The resolution of the feedback transducer can limit the drive smoothness at low speeds and incremental motions of the crosshead should be avoided.

Peak loads of up to 200 N are typical for the small (80 × 4 × 3 mm) flexure specimens characteristic of technical ceramics. Loads may be considerably lower than 200 N, particularly at high temperatures, and a sensible choice of load transducer is essential (particularly if used as the feedback device for controlling the machine) since electronic noise and transducer non-linearity will affect 'over-amplified' systems.

Centre point deflections of 1–2 mm are typical and easily catered for by LVDT or strain gauge measurement devices. Errors can be introduced into results generated by indirect measurements of specimen deflection and so direct measurement is preferred.

The stiffness of the testing machine, flexure fixture and pushrods should be as high as is practical in order to minimise the effect of stored energy on transient material behaviour. The comparatively low loads of the flexure test are a help in this respect and it is not usually necessary to be over-concerned with system stiffness. However, it should be noted that load frame stiffnesses can cover an enormous range (from as low as 10 kN/mm to 400 kN/mm and higher).

2.3 Flexure fixture requirements

Mechanical tests on ceramics produce results that are characterised by data scatter. This is because of the extreme sensitivity of these materials to defects within their structure. However it is possible to process the data in a statistical manner in order to allow interpretation and conclusions to be drawn about the mechanical behaviour.

It is a critical requirement, therefore, that extraneous influences due to testing technique (experimental errors) are minimised so that the inherent scatter can be analysed correctly. The main sources of experimental error that are important in the flexure test are:

(1) misalignment of the loading and support axes;
(2) poor specimen preparation or design.

It is a much easier proposition to ensure good loading axiality by prudent fixture design than to concentrate on the testing machine construction itself.

Since flexure tests are often carried out at high temperatures with the load being appplied through long pushrods, the possibility of misalignment of the loading axis is considerably increased. One approach is to incorporate special alignment fixtures at either end of the loadstring but this may require laborious setting up with dial gauges. Another way is to incorporate self-aligning features into the test fixture itself. This can be done by employing articulating rollers which ensure that the load is applied uniformly across the specimen even in the presence of misalignment or with twisted specimens (Fig. 1).

In order to ensure that the load is applied on the central axis of the specimen, it is necessary to take great care in the location of rollers and in assembly of the fixture. Top quality fixturing requires very close machining tolerances (often in very hard materials such as SiC) in order to ensure that the best alignment can be met repeatedly. Misalignment of the fixture with the upper pushrod axis can be overcome by using a ball or roller arrangement on the upper part of the fixture (Fig. 2).

The specimen deflection can be estimated from crosshead or actuator movement but this is not entirely satisfactory. This is particularly true for 4-point tests where assumptions have to be made about the shape of the

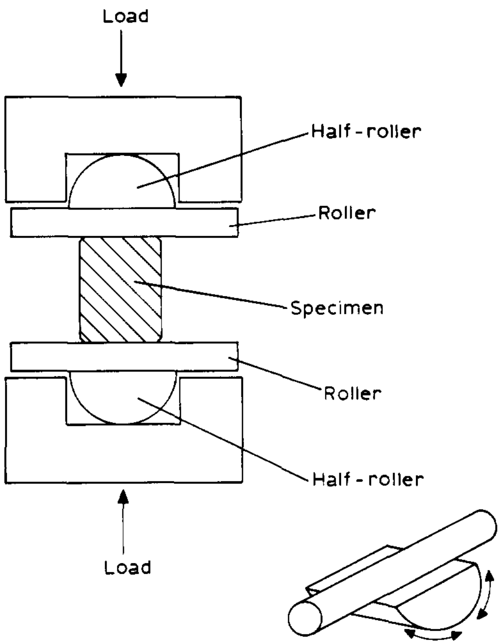


Fig. 1. Articulating roller design for minimising effects due to loading misalignment and specimen distortion.

deformed specimen between the loading points. Direct measurement is therefore favoured and this can be achieved by using a probe in contact with the specimen mid-point. The probe transmits the specimen deflection to a suitable transducer. Such a method has been used successfully even at high temperatures and opens up the possibility of performing creep investigations (Fig. 3).

Any high temperature mechanical test on ceramic materials carries with it the possibility of material compatibility problems between the specimen and the flexure fixture. It is therefore important to allow for this in the design of the flexure fixture and allow easily interchangeable components wherever necessary to allow damaged parts to be replaced. Figure 4 shows the component parts of a 1500°C bend fixture. All the rollers and articulating components can be replaced individually as required.

The high hardness of the specimens requires that the contact rollers should be as hard as possible to minimise wear and damage. Tool steel, WC or alumina may be used at room temperature with SiC being widely used at temperatures up to 1500°C in air or 1650°C in vacuum.

2.3.1 *Flexure tests at high temperatures*

High temperature flexure tests up to 1500°C in air can be achieved with comparative ease. Whilst furnaces are available for higher temperatures, the

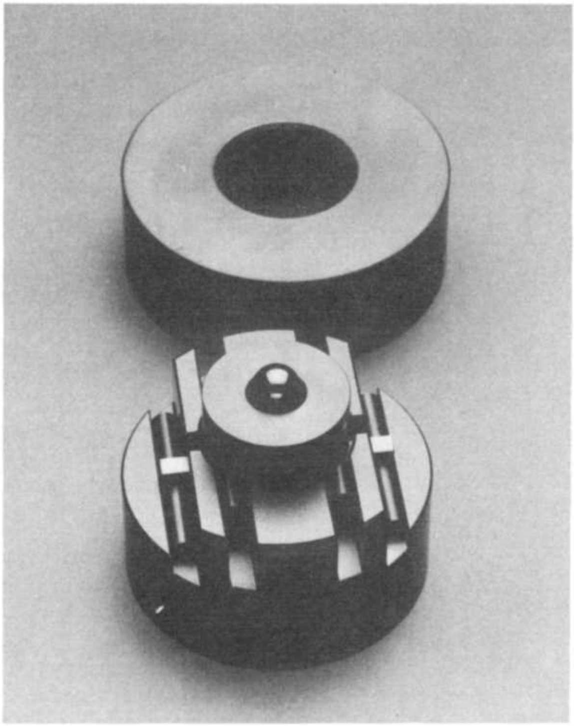


Fig. 2. High temperature bend fixture showing ball seating for upper pushrod.

limiting factor becomes the life of the flexure fixture and pushrod systems. Excessive oxidation of SiC occurs above 1500°C and alumina starts to soften.

Inert atmospheres or vacuum conditions overcome the oxidation problem with SiC and also allow the use of graphite fixturing which is both easier to make and has a higher operating temperature range. Much higher temperatures can be achieved with vacuum systems, and routine flexure testing at 1650°C is perfectly practical. For many classes of ceramic, the working conditions of the final component are oxidising and for this reason there is a clear requirement for increasing the temperature capability of in-air flexure testing. The solution to this problem appears to be in materials development and therefore it may be some time before routine flexure or creep-flexure testing above 1500°C in air becomes an economic proposition.

2.3.2 Multiple specimen flexure testing

The statistical nature of the ceramic flexure failure means that large numbers of specimens need to be tested in order to draw valid conclusions. This has led to the development of systems capable of testing many specimens in an uninterrupted test sequence.

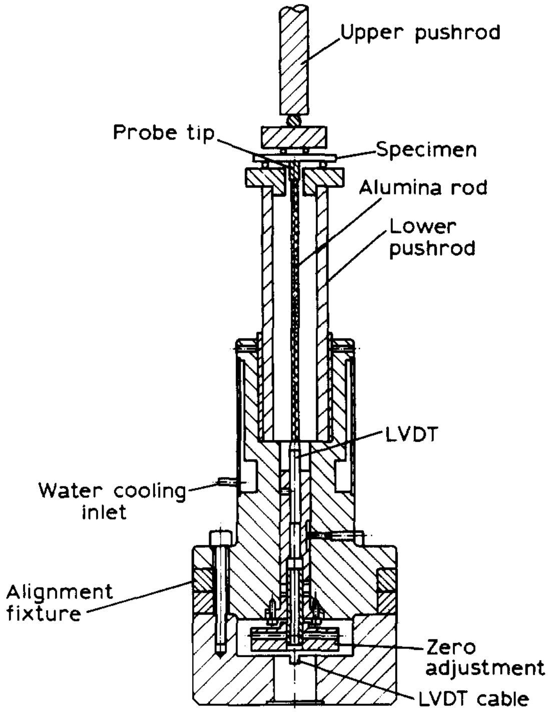


Fig. 3. Deflection measurement system for high temperature bend fixture.

The advantages of multi-specimen testing systems become particularly apparent when considering high temperature tests. The aim becomes to eliminate the lengthy cool-down time between tests that characterise 'single-shot' flexure tests.

Approaches to the problem vary from simple fixtures containing a number of specimens to fully automated 12-station test systems. The simple approach of loading all the specimens into the furnace and testing them sequentially suffers from the fact that each specimen has a different thermal history. This may not be a problem if each test is comparatively short-term or the furnace atmosphere considered non-deleterious. It is certainly the simplest and cheapest way, since the fixturing can be incorporated into an existing 'single-shot' system without extensive modification.

A more sophisticated approach that overcomes problems caused by different thermal histories utilises separate bend fixtures which are introduced individually into the furnace hot zone and removed following the test. The test sequence can be controlled entirely automatically including soak-times for each fixture. This not only speeds up the testing but allows personnel to perform other duties. An example of a fully automated test system is shown in Fig. 5. The system is based around a suitable testing

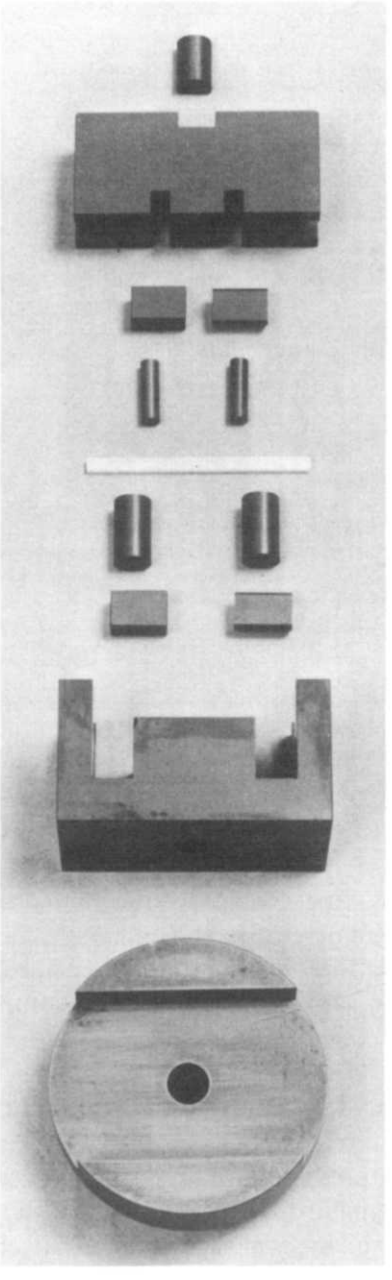


Fig. 4. Component parts of a typical articulating roller high temperature bend fixture.

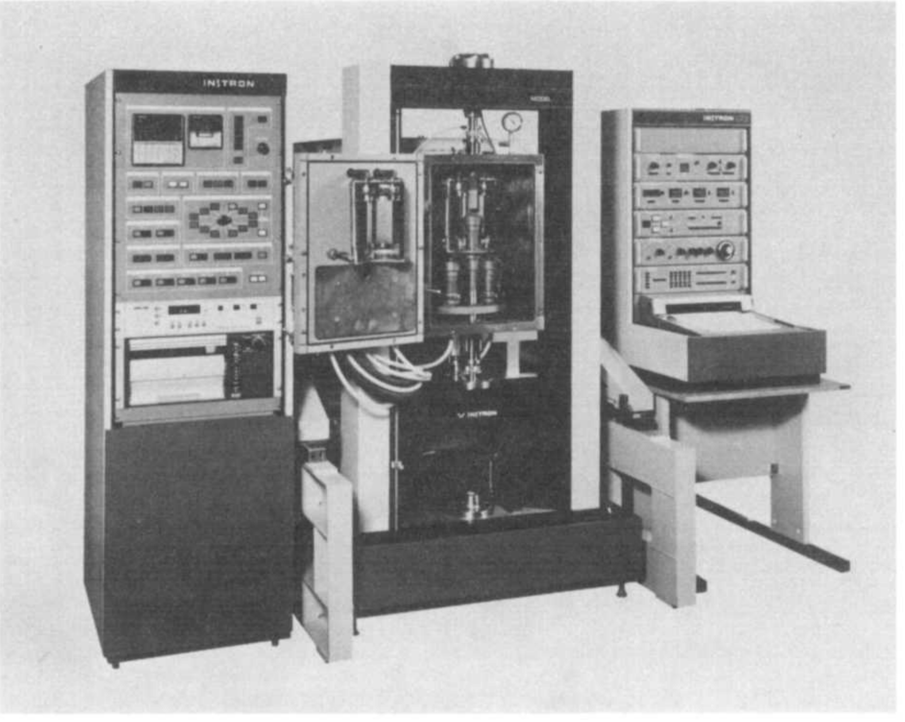


Fig. 5. Fully automated multi-specimen 12 station high temperature bend test system.

machine and consists of a vacuum furnace plus associated equipment, turntable containing 12 fixtures, the sequence controller and the testing machine console.

The system shown is capable of fully automated testing at temperatures up to 1650°C (in vacuum or inert gas). Similar systems are also available for use in air at temperatures up to 1500°C. Improvements in specimen throughput of at least 400% are achievable compared to conventional 'single shot' systems.

2.4 Future requirements for flexure testing apparatus

Higher in-air temperatures are already in demand and the advantages of multiple-specimen testing are slowly gaining acceptance. It is unlikely that there will be any major changes in the flexure test itself. The current struggle is to establish standardised test procedures which will increase the usefulness of the results.

The flexure test is a simple and comparatively cheap way of screening new ceramic materials for further study and it is likely that demand for straightforward multi-testing systems will increase along with higher test temperatures.

3 TENSION OR TENSION/TENSION FATIGUE TESTING OF CERAMICS

The requirement is straightforward. There is a need for tensile data on new ceramic materials to be used as design input for the various programmes requiring the use of high-temperature materials as structural components. The temperature objectives vary but are almost always above 1000°C and may go as high as 3500°C. Since most of the applications are for use in air, the design data input requires tests performed at high temperature in an air environment. Measuring strain on the specimen is desirable since temperature effects and specimen geometry make it difficult to estimate strain at the gauge length from crosshead motion. Hence, the three problem areas to be addressed are as follows: (1) applying uniaxial stress to the specimen (i.e. gripping without excessive bending); (2) heating the specimen to the temperature of interest without affecting alignment; and (3) measuring strain on the specimen at temperature. Although this paper is mostly concerned with (1) and its influence over the test machine design, the importance of an integrated system cannot be overemphasised. All components of the test system must be designed to work together at these extreme testing conditions or degradation of the test results may occur.

3.1 Alignment

Any discussion of tension testing of ceramics ultimately turns into a discussion of bending in the specimen. Although the definition of bending varies slightly between authors who have analysed their load strings, it can be defined as the percentage difference in the strain as compared to the average strain at any given cross-section of a specimen. Since it is a function of average strain or axial load, it usually becomes less at higher loads.

As an example, consider the typical specimen geometry shown in Fig. 6. If we assume a modulus of $E = 400$ GPa, then at an axial load of 2·5 kN, the average axial strain will be 197 $\mu\varepsilon$ (micro strain). If the objective is to have 1% bending at this load, then the tolerance is only $\pm 1{\cdot}97\,\mu\varepsilon$. For the specimen geometry given, the moment required at the end of the specimen is $M = I/c = 0{\cdot}02$ N m.

Not only is it difficult to measure 2 $\mu\varepsilon$ accurately with strain gauges, but to achieve alignment to the tolerances necessary is impractical for the average testing machine.

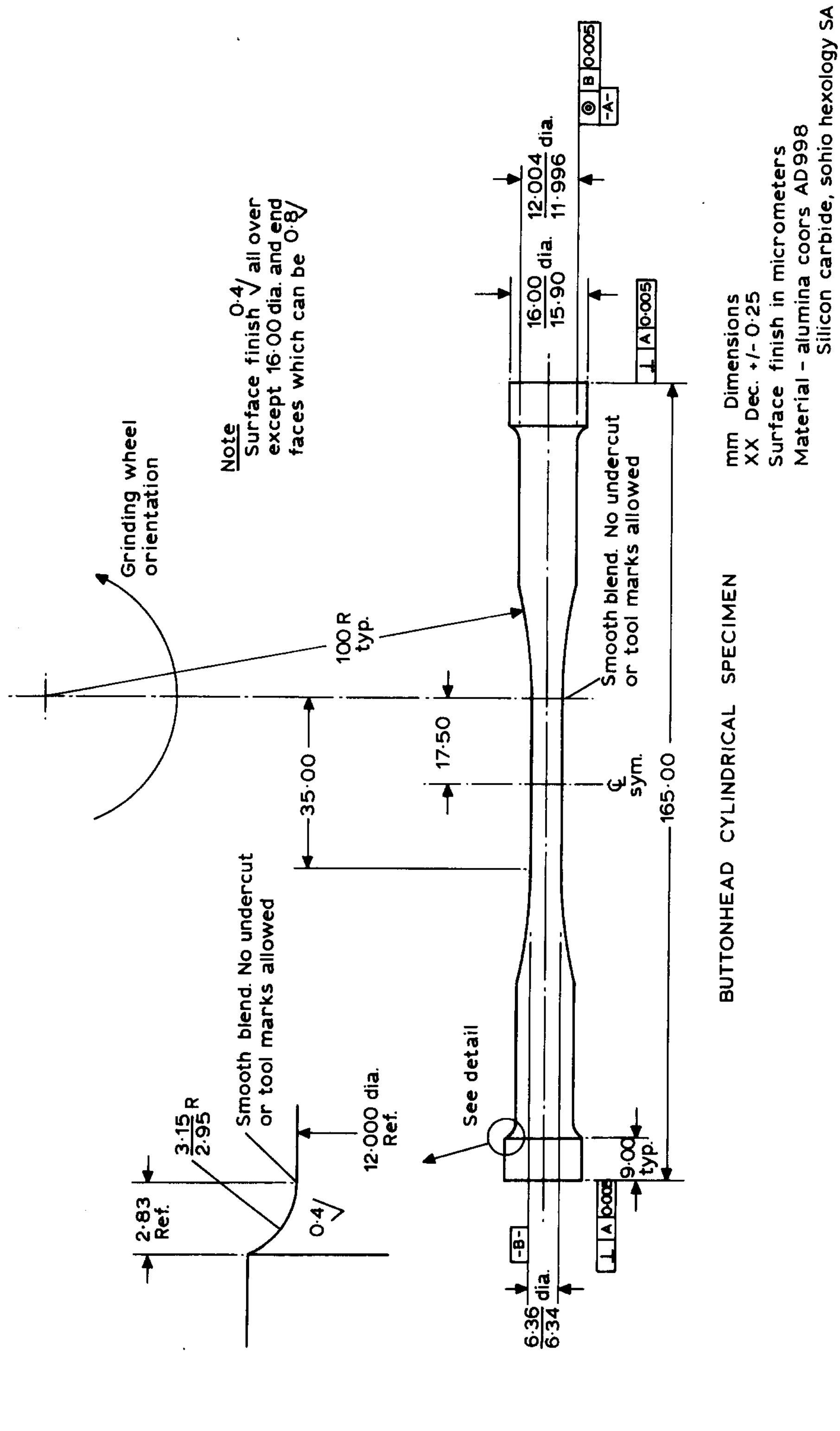


Fig. 6. Tensile test specimen geometry.

Figure 7 shows a more general approach to determine the criteria for 1% bending in the specimen. The result $d = 0{\cdot}0025\,r$ is the general condition necessary to give 1% bending due to a nonaxial load.

What does all this mean with respect to a testing machine? There really are only two things one can do to ensure good alignment in a load frame for tension testing. One can have a rigid load string that is meticulously pre-aligned to the above criteria or one can provide adjustments in the load string to compensate for any intrinsic misalignment of the machine. Since all testing machines require a moving crosshead or ram of some sort to apply the load, even perfect pre-alignment will be changed once this moving component changes location. To keep a machine within the 0·0075 mm tolerance calculated in this example is extremely difficult and in most cases impractical. Therefore, the most realistic approach is to take the burden off the load frame and put it onto specially designed components of the load string.

The load string must be designed in a way not to transmit any bending moments to the specimen while loading the specimen in a pure uniaxial mode. This turns out to be a two-part problem. The first requirement is to

Axial $\sigma = F/\pi r^2$

Bending $\sigma = \dfrac{Mc}{I} = \dfrac{F \cdot d \cdot r}{I}$

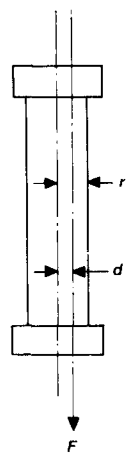
$$I = \frac{\pi r^4}{4}$$

For bending = 1% axial σ

$$\frac{MC}{I} = 0{\cdot}01 \frac{F}{\pi r^2}$$

$$\frac{4\,F \cdot d \cdot r}{\pi r^4} = \frac{0{\cdot}01\,F}{\pi r^2}$$

$$\frac{4\,d}{r^3} = \frac{0{\cdot}01}{r^2} \quad \therefore \quad \boxed{d = 0{\cdot}0025 r}$$



e.g. for 6·35 mm diameter specimen

—required concentricity = (0·0025) (3·18 mm)
= 0·0079 mm

– to achieve 1% at 2·5 kN
Moment = $F \cdot d$ = (2·5 kN) (0·0679 mm)
= 0·02 Nm

Fig. 7. Criteria for 1% Bending.

Attempts to Overcome
Tension Testing Problems

1.	Crushable shims (Bortz, 1962)	10%
2.	Gas bearing (Digesu & Pears, 1963)	<0·11 Nm torque
3.	Precision alignment (Penny *et al.*, 1966)	
4.	Powder cushion (Lange & Diaz, 1978)	5%
5.	ORNL coupling (Liu & Brinkman, 1985)	1%

Fig. 8. Tension testing grip designs.

achieve flexibility in the pull rods independent of load. That is, a flexible coupling is needed that will not transmit moments and does not lock up due to friction at higher loads. The second condition requires the specimen to be held in a way that all the load is transmitted through the geometric centre. It has already been shown that very small eccentricities can cause significant bending. Flexible couplings in the load string above and below the specimen ensure no bending moments can be applied to the gripping mechanism as a result of machine misalignment. The grips themselves, however, must still meet the criteria of loading concentricity described above. The flexible couplings then reduce the requirements of the test frame and transfer it to the gripping devices where it is more practical to achieve tight tolerances.

Figure 8 lists several techniques that have been employed to overcome the limitations of testing machine alignment and produce pure uniaxial stress.

4 TENSION/COMPRESSION TESTING

Although the demand for reversed cyclic stress testing is somewhat less than that for tension-only work, there are some people in the world with active programmes in this area (see Soma *et al.*, Paper 15). Test conditions in terms of temperature and strain measurement are similar to those for tensile testing. Two factors make this type of test much more difficult to achieve over that of uniaxial tension. The first and most obvious is the need to make the load string laterally stiff in the compression cycle to minimise compressive bending that could cause erroneous results in a way similar to the tensile problem. This requirement precludes the use of flexible couplings and, in fact, suggests stiffening the machine and load string even further. The second problem to overcome is pre-loading the actual gripping mechanism so that reverse stress testing (through zero load) can be achieved without nonlinear load string stiffness effects. In simple terms, the buttonhead of the specimen, shown in Fig. 6, must be pre-loaded into its grip with a force greater than the one it will experience during the test cycle. This must be done in a way not to fail the specimen at the end as well as not to cause bending at the gauge length. For the specimen shown in Fig. 6, approximately 15 kN of pre-load is required on the buttonheads to exceed the highest possible test loads.

Figure 9 shows one way of achieving more stiffness from a conventional servohydraulic-type of load frame. First of all, added stiffness is achieved by using a load frame of higher load capacity than the test requirement. The frame illustrated is a 250 kN dynamically rated frame that is only being used to 100 kN. The larger load cell of the sandwich shear-element design provides high lateral stiffness at the top end of the load string. On the bottom end, an oversized actuator (250 kN capacity) is used with a single-stage servo valve. This gives better control at low strain rates at the expense of dynamic performance and reduced pressure. Therefore, the actuator

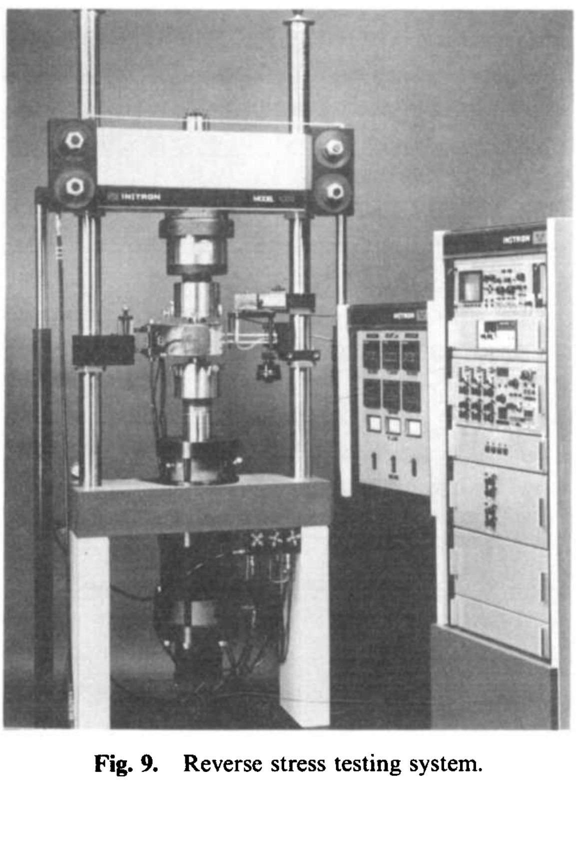


Fig. 9. Reverse stress testing system.

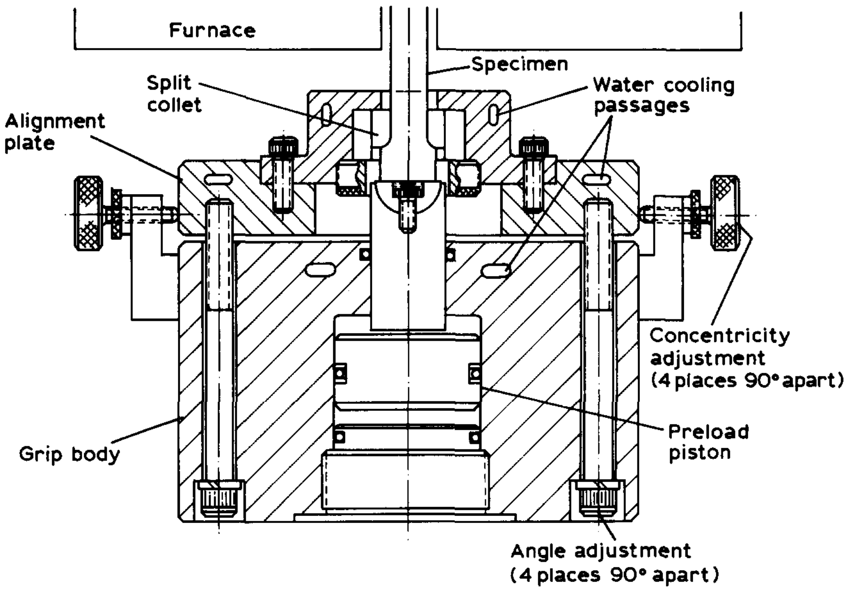


Fig. 10. Cross-section of reverse stress grips.

capacity is reduced to approximately 100 kN in this configuration. Finally, to increase the lateral stiffness of the actuator, a hydrostatic bearing is put on top of the table, thus reducing the free length of piston rod and increasing lateral stiffness. This type of bearing, unlike labyrinth or plastic bearings, has centring forces that resist side loads and returns the piston to the same location with respect to the frame.

Even though the utmost care is taken in the alignment of the load cell with the actuator, acceptable bending can only be achieved when further adjustments are made with the grips. Figure 10 shows a cross-section of how the grips are adjusted for translation and angle. This procedure requires that every specimen be strain-gauged so that feedback from the actual test specimen is used to achieve the optimum test setup. At a 2·5 kN axial load, less than $\pm 1\%$ bending in the tension direction and $\pm 3\%$ in the compression mode can be achieved. An experienced operator can set up and adjust this system in about one hour.

5 HOT VERSUS COLD GRIPS

One of the key decisions that must be made when designing a high-temperature test system is whether to use hot or cold grips. Although hot grips—that is, grips that are at the specimen temperature—make it easier to achieve a good temperature gradient with standard furnaces and smaller specimens, there are some significant technical problems. First of all, the materials that can tolerate temperatures as high as 1500°C are the materials that are being tested, they are difficult to machine and, hence, expensive. They are also prone to failure from thermal cycling to the test temperature, so the lifetime is likely to be low. Chemical interaction may occur between the grip material and the specimen, so different grip materials may be necessary for different test specimens. Finally, long extension rods reaching into a furnace make alignment difficult and are impractical for loading in the compression mode where lateral stiffness is important.

Cold (i.e. water-cooled) grips that remain outside the furnace can be made of standard metallic materials and are easier to align and pre-load for tension/compression work. They also ensure no chemical interaction between the specimen and grip ends. The two main disadvantages of cold grips are that the specimen must be longer and that the furnace design becomes critical to achieve an acceptable temperature gradient on the specimen. Also, for those materials that may exhibit increased strength at higher temperatures, specimen failure could occur in the transition between the hot zone and the cooled ends of the specimen. In some cases, this problem can be overcome with suitable specimen design.

6 FIBRE TESTING

As the work progresses on ceramic composites so does the interest in measuring the properties of ceramic fibres. With diameters in the $10\,\mu m$ range or less, gripping and handling become the major obstacles to overcome. Figure 11 shows a system designed to measure the tensile strength of ceramic fibres at temperatures as high as 1500°C. Since the thermal mass is small, a furnace with a slotted front can be left at a temperature in the retracted position while new fibres are being loaded into the grip. On-specimen extensometry is out of the question, so either non-contact optical methods or total elongation outside the furnace must be used. In the system illustrated, two LVDTs are used to average the grip translation during the test. Since ultimate loads are in the 100 gram range, machine stiffness is not a factor in using total elongation to compute specimen strain. Although the optimum gripping faces may depend on material, everything from double-sided tape to soft metals can be used with some success.

7 TENSION/TORSION TESTING

When a specimen starts to fail, it can often exert a lateral load onto the load string and, hence, onto the test frame. A classical example is materials that

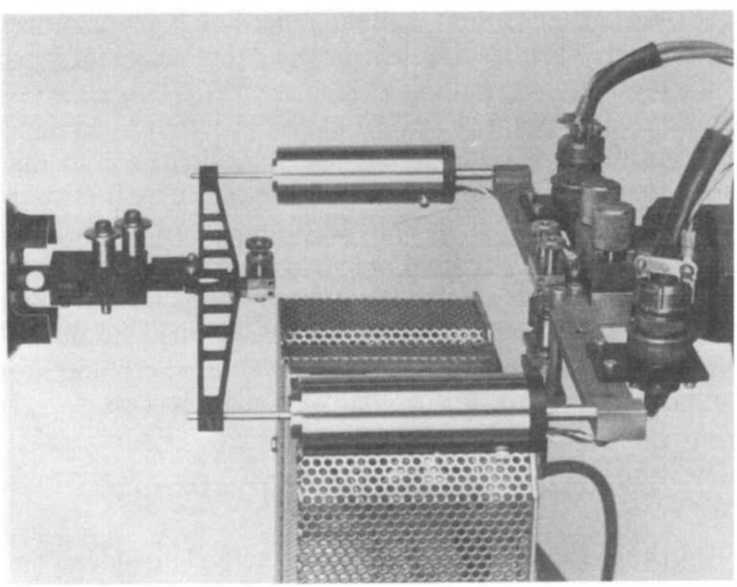


Fig. 11. Ceramic fibre testing system.

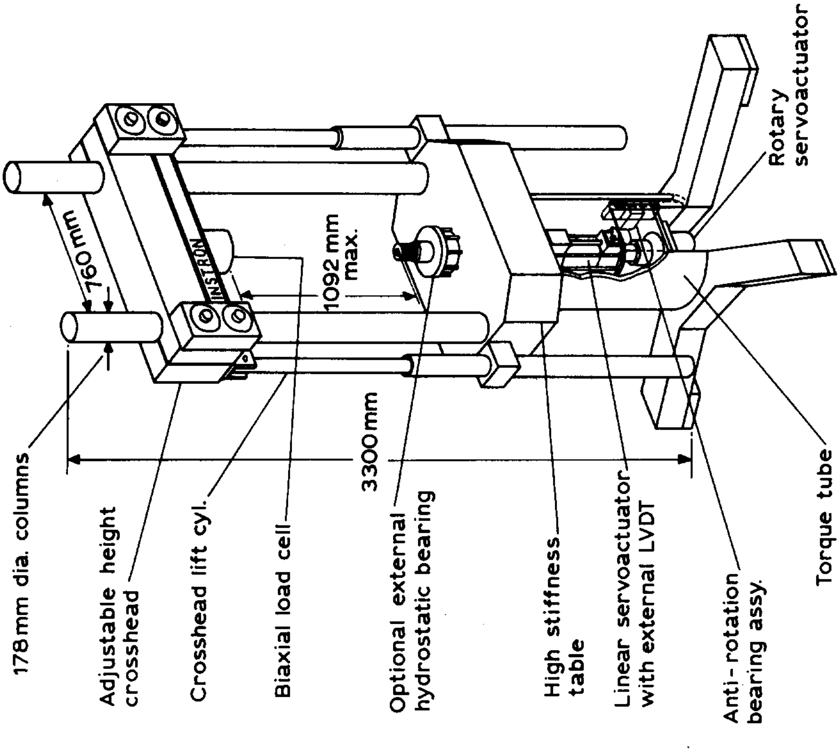


Fig. 12. Tension torsion testing system.

fail along a 45° plane during a compression test. Ceramic composites with various fibre geometries can give a similar effect. For example, if a flat, wide ceramic started to fail at one edge, the crack might propagate horizontally across the specimen. If the machine were compliant in the lateral direction, it would deflect, causing a tearing action rather than a uniaxial stress on the remaining material. For more complex specimens—ceramic composite tubes for example—under biaxial loading conditions, the test machine stiffness is important to understand the failure mechanisms. That is, the machine must maintain its original alignment during the whole failure process. Standard servohydraulic frames are compliant in the torsional mode and, hence, require 'stiffening' to meet the stiffness requirements of ceramics. Figure 12 shows a high-stiffness tension/torsion system designed for ceramics composites work. The axial stiffness of the frame is $1{\cdot}05 \times 10^{9}$ N/m and the torsional stiffness with a crosshead to table separation of 1 m is $1{\cdot}13 \times 10^{6}$ N m/degree.

8 SUMMARY

The successful mechanical testing of ceramic materials depends on the application of truly axial loads and the elimination of extraneous forces which can result from the fixtures and grips used to hold the specimens. Whilst the testing machine itself must combine high stiffness with smooth and controlled application of load it is generally impractical to try and achieve the exceptional axiality of loading required by concentrating on the machine design itself. A much more practical approach is to design grips and test fixtures that can be aligned or are self-aligning.

Particular aspects of the test machine and fixture designs are more important in some applications than in others. Flexure tests can be performed using almost any testing machine providing attention is paid to the flexure jig design. On the other hand, a reversed cyclic stress test requires special consideration of the testing machine characteristics as well. The statistical nature of the ceramic specimen failure means that larger numbers of specimens are required compared to tests on more conventional materials. This has led to the development of automated systems capable of running a number of tests unattended.

Systems are now available that allow routine tensile tests on ceramic materials without time-consuming manual adjustments of loading axiality being required. However developments in machine design are continuing in an attempt to cater for the special needs of creep, creep-fatigue and high cycle fatigue applications at elevated temperatures.

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