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Current Testing Methods—a Critical Assessment

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1 INTRODUCTION

Ceramics offer a fascinating wide field of applications in such different areas as microelectronics, sensors and transducers, superconductors, bioceramics, cutting tools, refractories and as structural parts for many components which are often used in severe environments. A large proportion of ceramic research activities during the last three decades was spent on the challenge to develop ceramics as high-temperature materials. The target to replace classical high-temperature materials, such as superalloys, in gas turbines has not been reached so far. However, it is unquestionable that ceramics, especially the non-oxides based on Si_3N_4 and SiC, possess strong potential for such high-temperature applications because of their inherent properties. Further progress in ceramic technology is still needed in order to realise this potential. Thus one particular objective is in providing a ceramic database relevant to design and application in the proposed high-temperature field. Table 1 lists the properties of structural ceramics that have to be considered for high-temperature applications.

The state-of-the-art in testing ceramics at high temperatures will be reviewed, including some actual results. Emphasis is given to mechanical properties of advanced ceramics. Unresolved problems in current test techniques are identified, and the paper also gives a perspective on new developments.

The current situation in mechanical testing of advanced ceramics is characterised by a large deficit of standardisation as far as testing methods at high temperatures or long-term conditions are concerned. At ambient

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TABLE 1
Properties at High Temperatures Required for Engineering Ceramics

Elastic properties
Yield strength
Fracture strength and statistical analysis of failure
Impact strength
Fracture toughness, crack resistance curve
Slow crack growth
Creep
Creep damage and rupture
Relaxation
Life time
Fatigue
Hardness
Wear resistance
Friction coefficient
Thermal shock resistance
Thermal fatigue
Corrosion resistance
Microstructural instability

temperature some standardisation work has been started but there is still a long way to go until a satisfying level of acceptance for the proposed methods is achieved. Because of this and other reasons, today's advanced ceramic products are often poorly characterised. However, a wide variety of testing procedures have been developed and applied in research and development in order to investigate fundamental properties of these new materials. Many of them are discussed elsewhere in this book. The present paper focuses on the problems in testing which are relevant with respect to fundamental understanding and reliable design of ceramic components. The properties at high temperatures—i.e. strength and fracture toughness, slow crack growth and creep, rupture lifetime and cyclic fatigue—will be discussed with their problems in testing and evaluation, in more detail.

2 STRENGTH

The development of high-performance ceramics is orientated in directions to optimise maximum allowable values of both temperature and stress as shown in Fig. 1. Great success has already been achieved with respect to each of these objectives individually; however, the progress considering both objectives simultaneously has been much less. A principal property that

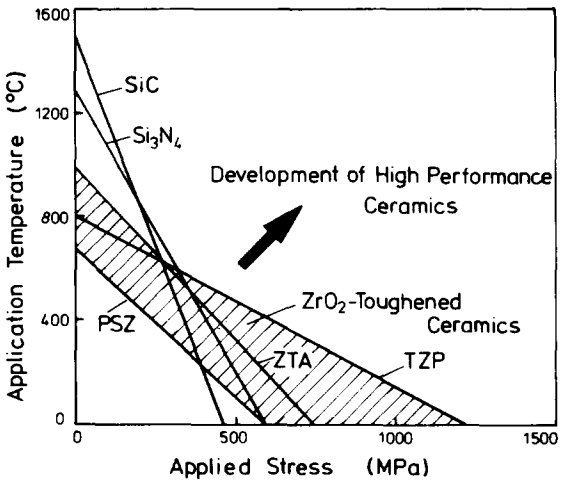

Fig. 1. Schematic diagram indicating the potential application range of some engineering ceramics (Claussen, 1985).

characterises this behaviour is the fracture strength measured at high temperatures. For most applications, fracture due to applied tensile stresses has to be considered; thus fracture strength should be measured under tensile stress. While, normally, fracture strength data are obtained in conventional flexural (bend) strength tests, the required homogeneous stress distribution is not attained in such specimens.

Figure 2 gives the configuration of a four-point bending fixture, made for example from silicon carbide, which can be used up to 1500°C. This fixture provides an effective solution for many testing problems; complexity of the configuration is reduced as far as possible with respect to the typical sources

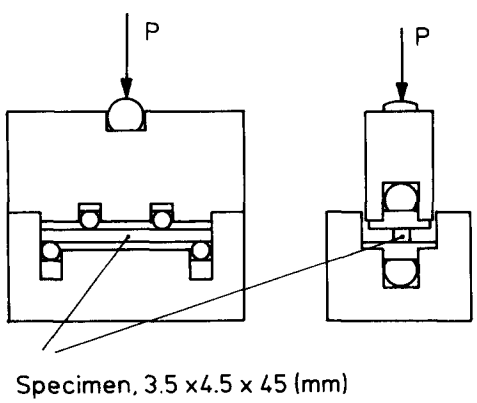

Fig. 2. Four-point bending fixture (material: SiC) for testing ceramics at high temperatures.

TABLE 2
Strength of Ceramics at High Temperatures: Limitations of the Current Testing Procedures

—Data commonly available from flexural strength tests
—Principal limits of bending tests:
  —Small volume (surface) under nominal stress
  —Surface phenomena predominate (chemical and mechanical effects)
  —Inhomogeneous stress distribution
—Typical sources of error in evaluation of bending tests:
  —Friction between specimen and loading edges
  —Twisting due to improper loading
  —Assumption of linear-elastic behaviour
—Low reliability due to insufficient data base
—Erratic Weibull parameters due to scattering of test conditions
—'Multitester' with serial capacity under identical conditions required
—Modification of critical flaws due to slow crack growth, chemical attack and plastic flow
—Direct tension tests not generally attainable (accuracy, costs)

of error in bending tests (Table 2). Improper loading leading to an unsymmetrical stress distribution and to twisting of the specimen is avoided by allowing three of the four rollers to pivot about the axis parallel to the specimen's (long) axis. The general requirements for flexural tests were discussed by Marschall & Rudnick (1974), Newnham (1975) and Baratta (1980) and should be considered in the urgently needed work of standardisation for strength tests (Loveday & Morrell, Paper 2).

Specific problems in high-temperature flexural testing of ceramics are scarcely covered by the early publications. The related experimental difficulties (see Table 2) arise mainly due to the following reasons. At high temperatures, chemical reactions can take place at the surface and the interface of specimen and fixture, influencing the (unknown) friction coefficients and impeding the required mobility of the fixture members against each other. This and other reasons concerning degradation effects of specimen and fixture during periods of soaking at temperature can lead to poorly defined test conditions and to a lowered reliability of the available data. The dramatic decline of the Weibull modulus, m, with increasing test temperature was manifested, for example in the work of Hartsock & McLean (1984), where its effect on the allowable design stress was also demonstrated.

The concentration of the relevant fracture processes in the surface region of the bending specimen brings about a further drawback of the flexural test. In this test, a relatively small effective volume (surface) is being tested; since the apparent strength depends on the specimen volume under stress, the flexural strength data have to be weighted by the related factor, e.g. on the basis of Weibull statistics. Additionally, the small stressed volume (surface)

is particularly exposed to microstructural alterations due to environmentally induced reactions. These problems can be partly overcome by the realisation of tensile strength tests, as discussed, for example, by Ohji (1988).

3 CRACK RESISTANCE AND SLOW CRACK GROWTH

While crack growth in ceramics at room temperature is a classical subject of linear-elastic fracture mechanics, the techniques and concepts used to characterise crack growth at high temperatures have to encompass additional problems. The determination of fracture toughness, crack resistance and crack velocity can be influenced by phenomena such as high-temperature plasticity, slow crack growth and microstructural instabilities. Figure 3 gives an example of the determination of stress intensity factors (K_I-values) with the commonly used SENB-specimen (single edge-notch bend). If slow crack growth can be excluded, the initial value, $K_{I\max}$, is calculated on the basis of maximum load and initial crack length (notch depth); however, if slow crack growth is occurring at lower loads, the actual crack length has to be taken into account in order to determine meaningful K_I-values. Depending on the thermal activation of slow crack growth processes, the values K_{IO} (at the fatigue limit) and $K_{I\max}$ can differ considerably from each other as K_{IO} has to be determined on the basis of the initial crack length and the threshold load to start slow crack growth. Thus, the main problem in

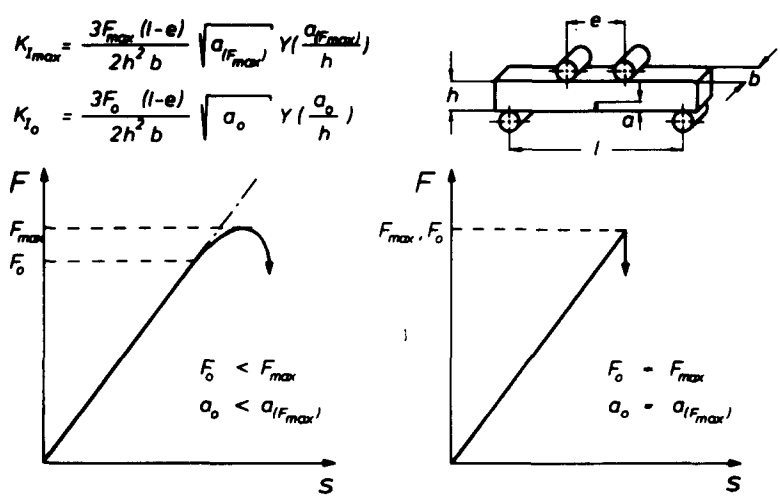

Fig. 3. Schematic load-displacement diagrams with (a) and without (b) slow crack growth at $F < F_{max}$ (Kriz, 1983).

many fracture mechanics experiments at high temperatures is related to the determination of the actual crack length or crack growth rates as a function of the loading parameters.

There are basically three types of method to determine crack growth data at high temperatures as listed in Table 3. A direct method was developed by Bornhauser (1983) where a travelling microscope was used focusing on the moving crack tip at the surface of a SENB-specimen. Limits of this method are given by the maximum magnification of the optical system and by the fact that the crack growth is only recorded as observed at the surface. Differences between the crack behaviour at the surface and in the inner parts of the specimen may arise, and can be explained in terms of fracture mechanics and materials properties. However, illuminating results can be achieved by this technique, as shown in Fig. 4. Even under the restriction of surface observation, the crack processes in the surface of Si-SiC were seen to be strongly rate-dependent. Thus, although the specimen exhibits a smooth load–deflection curve at higher loading rates, it shows a discontinuous nature at lower deflection rates, e.g. 0·024 μm/min. This behaviour was only detectable by this direct visual technique and can be explained in terms of the large differences in the high-temperature properties of the two phases of Si-SiC. All other methods provide information on crack growth behaviour not from small parts of the specimen volumes, but after summation of the crack growth processes over the total specimen width or even the total fracture surface.

These indirect methods may be divided into two groups. First, double-torsion (DT) or bend specimens can be used for the determination of crack elongation on the basis of compliance testing techniques. Due to this fact, these methods are basically limited to linear-elastic behaviour of the

TABLE 3
Crack Growth Evaluation Concepts for Ceramics at High Temperatures

—Double torsion test
  —indirect method, based on specimen compliance (linear-elastic behaviour)
  —restricted for long, macroscopic cracks
  —for crack velocities $> 10^{-9}$ m/s
  —instationary curvature of crack front
—Bending of beams with various types of notches or precracks
  (a) indirect method: partial unloading technique based on specimen compliance
  (b) direct method: crack observation on the surface using travelling microscope
—Bending of beams with natural cracks
  (a) indirect method: probabilistic approach based on correlation of two distributions, i.e. inert strength and lifetime, range of crack velocities extended up to 10^{-12} m/s
  (b) indirect method: based on the relation between strength versus loading rate

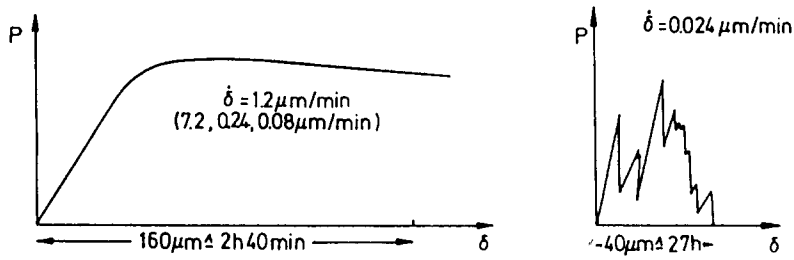

Fig. 4. Schematic load-deflection curves at various strain rates for Si-SiC at high temperatures (Pabst, 1985).

specimens. Double-torsion tests at elevated temperatures have been widely used, as proposed by Evans & Wiederhorn (1974); deviation from pure elastic behaviour due to creep processes leads to a necessary modification of the original technique, demonstrated by Kriz (1983), with intermediate unloading/reloading sequences. A similar procedure with compact tension specimens is commonly used for metals and has also been proposed for ceramics using SENB-specimens by Berweiler *et al.* (1987).

In the second group of indirect methods used to determine slow crack growth parameters, relationships between strength and loading rate or strength and lifetime were used. As noted in Table 3, the DT-technique is restricted to crack velocities, v, higher than about 10^{-9} m/s. This limit, and also the fact that DT-specimens only concern the elongation of large macroscopic cracks, have been overcome by a new concept proposed by Fett *et al.* (1988). By measuring the distributions of inert strength, σ_c, and lifetime, t_f, of two 'identical' samples of at least 15 specimens, v–K-curves can be determined after correlation of the two ranked distributions of σ_c, and t_f. The v–K-curves can be extended by this method into the region of crack velocity of about 10^{-12} m/s, which is particularly important with respect to lifetimes of technical components.

4 CREEP AND CREEP DAMAGE

Creep of ceramics is often measured in bending. Reasons for this are based on the simplicity of specimen geometry and the loading configuration; in addition, the evaluation of the time-dependent outer fibre strain appears to be quite simple since it is measured as beam deflection. Typical flexural creep curves of a multiphase Si_3N_4-material (SRBSN) are shown in Fig. 5 over a wide range of temperatures. Most phenomena to be discussed in flexural creep of these ceramics can be seen in these curves. A very long time period of transient creep behaviour is observed and it was shown by Ernstberger *et al.*

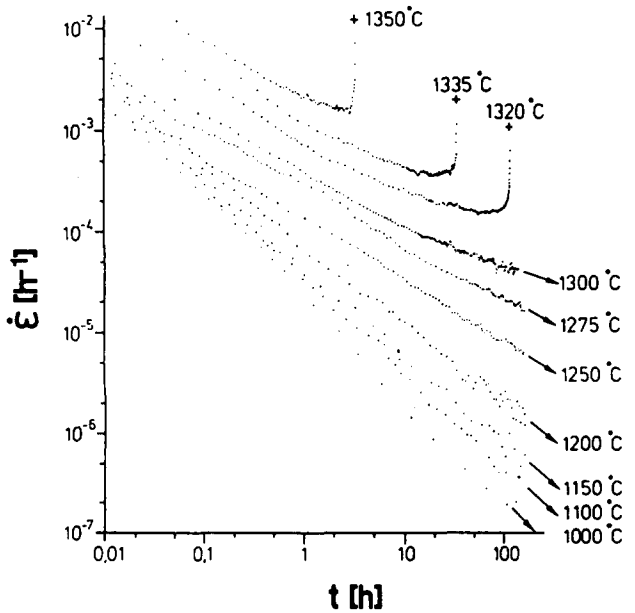

Fig. 5. Influence of temperature on the flexural creep curves of SRBSN at 160 MPa (initial outer fibre stress).

(1987) that the related time exponent c ($\varepsilon \sim t^{-c}$) is lowered from $c = 1$ at moderate temperatures down to $c = 0{\cdot}5$ at a temperature level where the creep curve leads to fracture after passing a minimum of the creep rate. At these higher temperatures, creep damaging processes counteract to a certain extent the microstructural processes leading to increased creep resistance.

The disadvantages of the flexural creep technique are manifested when creep analysis is taken into account. The limitations of the technique as discussed in Table 2 are still valid. In addition, the apparent creep curve normally does not reflect the true nature of the creep behaviour for the following reasons:

- —the inhomogeneous stress distribution in the flexural beam will be changed due to non-linear creep. The resulting time-dependent stress distribution has been calculated by several authors; examples are shown in Fig. 6 as determined by Cohrt *et al.* (1984). The stresses in the outer fibres are reduced, with a consequent increase in the inner fibres;
- —the neutral axis of the flexural beam, shifts towards the compressive side of the specimen, as also shown in Fig. 6. This effect is due to the higher creep resistance of these types of ceramics under compression compared with tension. This difference can be as high as three orders

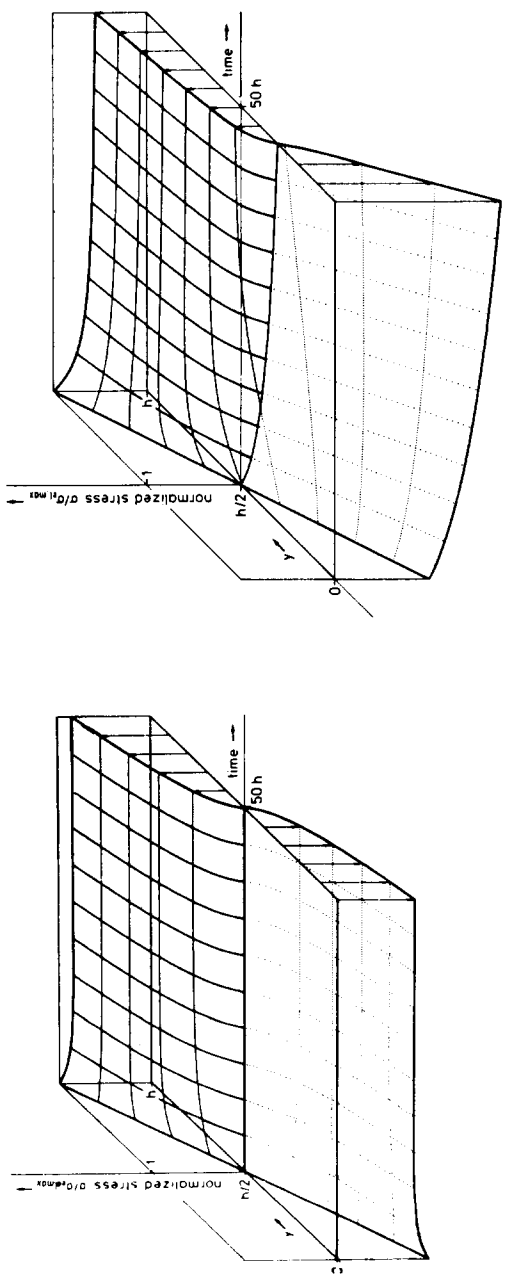

Fig. 6. Stress distribution during creep of a bending beam as a function of time for (a) symmetrical and (b) unsymmetrical material behaviour.

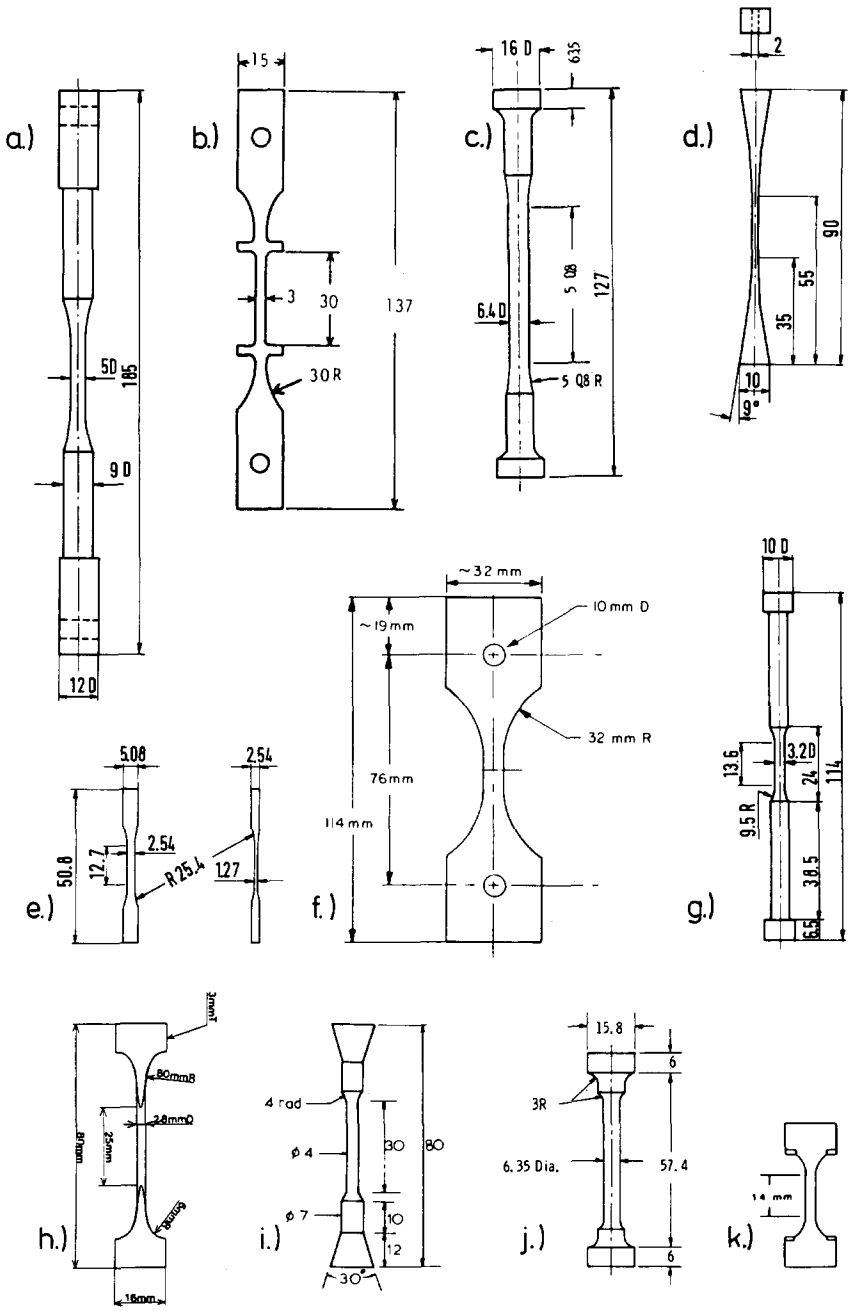

Fig. 7. Synoptical diagram of various ceramic specimens for tensile tests as proposed by (a) Morrell (1972), (b) Kawai *et al.* (1984), (c) Lange *et al.* (1979), (d) this work, (e) Mah *et al.* (1985), (f) Govila (1982), (g) Boussuge *et al.* (1982), (h) Ohji (Paper 8), (i) Birch *et al.* (1978), (j) Kossowsky (1974), (k) Wiederhorn *et al.* (1988).

of magnitude as shown already by Birch *et al.* (1978). The shift has also been experimentally confirmed by Wiederhorn *et al.* (1986) in terms of the position of the axis for zero strain;

— the calculation of the outer tensile fibre strain from the measurement of the creep deflection is not as straightforward as it is in the linear-elastic regime. Deviations from this ideal solution occur in the parts of the specimen where the curvature of the beam is not constant. Jakus & Wiederhorn (1988) only recently showed that the radius of curvature, as measured on the tensile surface, reaches extreme (minimal) values under the inner loading points of a four-point bending specimens under creep conditions;

— since the parts of the specimen under tension and compression react differently, not only in their creep rate, but also in their damaging behaviour and generally in their microstructural changes during creep, the elastic response of the bending beam due to unloading/reloading cannot provide meaningful results. It was shown by Grathwohl (1984*a*) that a strong change in the elastic response appears during creep of HPSN. However, due to the gradients of creep porosity in flexural beams, as shown also by Robertson & Wilkinson (1986), the elastic modulus will also exhibit gradients over the specimen thickness. Thus, the overall stiffness measured in flexure does not give valuable results.

For analysis of creep and creep damage a test technique is required which uses a specimen with a uniform, stationary stress distribution in its gauge length. This is best achieved in a tensile creep test which is considered

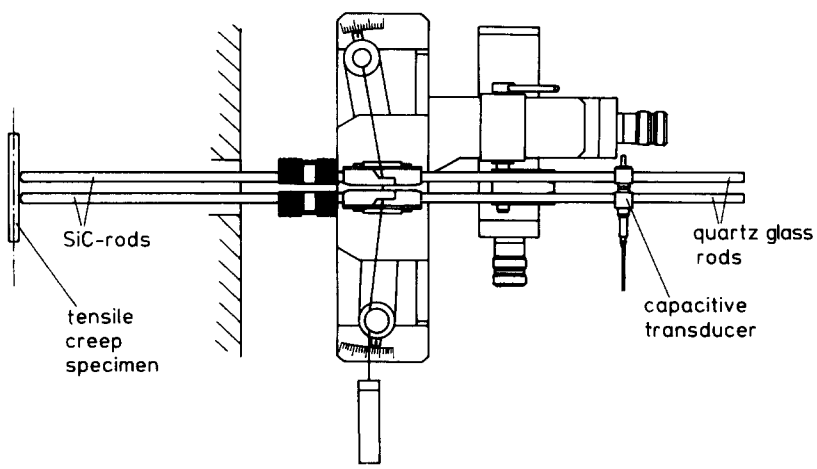

Fig. 8. Schematic diagram of a high-temperature extensometer with SiC-rods and a capacitive transducer.

elsewhere in this volume (Papers 9–11). In Fig. 7, a selection of 11 ceramic tensile specimens as suggested by various authors is presented. Some of them are designed for creep tests, some others for strength and rupture tests. Tensile specimens have been proposed with circular or with rectangular cross-sections, one specimen (h) being flat in the gripping region and round in the gauge length. Hot, warm or cold grippings are used, manufactured from ceramics or metals. In addition to precise alignment, tensile creep tests require a high-resolution device for measuring the elongation of the specimen gauge length: optical and various mechanical systems are being used for this purpose. In Fig. 8, a new high-temperature extensometer, which basically works without any friction and reaction forces during the measurement, is presented. Vertical forces due to gravity can be balanced to zero, while horizontal forces for providing contact between measuring rods and specimen can be minimised and kept to constant values by controlling the angle between the suspension wires and a vertical datum line.

5 LIFETIME

Lifetime of ceramic components at high temperatures can be governed by different mechanisms, e.g. slow crack growth and stress corrosion, creep and microstructural degradation processes. In most lifetime studies published so far, only slow crack growth was considered as the rate controlling process and the data are commonly interpreted on the basis of velocity as a function of stress intensity (v–K) curves. However, as argued, for example, by Katz (1985) the various test methods to determine slow crack growth data exhibit very different potentials to predict the real lifetime of actual components. As discussed already in section 3, this may be mainly due to the different types of cracks being responsible for the particular processes in each individual type of test.

If slow crack growth is controlling the lifetime of a ceramic component at high temperature, it has to be decided whether the linear-elastic fracture mechanics concept can be applied in order to describe slow crack growth. When crack growth occurs under creep conditions, the elastic stress field at the crack tip will be changed and a modified fracture mechanics concept considering also time-dependent deformation has to be applied. Riedel & Rice (1980) have proposed a critical time to decide whether the related K—or the C^*—concept, should be applied. Following a suggestion of Fett *et al.* (1988), the K-concept can be applied in a straightforward manner if the creep strain is small compared with the elastic deformation ($\varepsilon_c < 0{\cdot}05\,\varepsilon_e$).

Besides these measurements and interpretation of lifetime as a slow crack growth dominated process, high-temperature failure has also to be taken

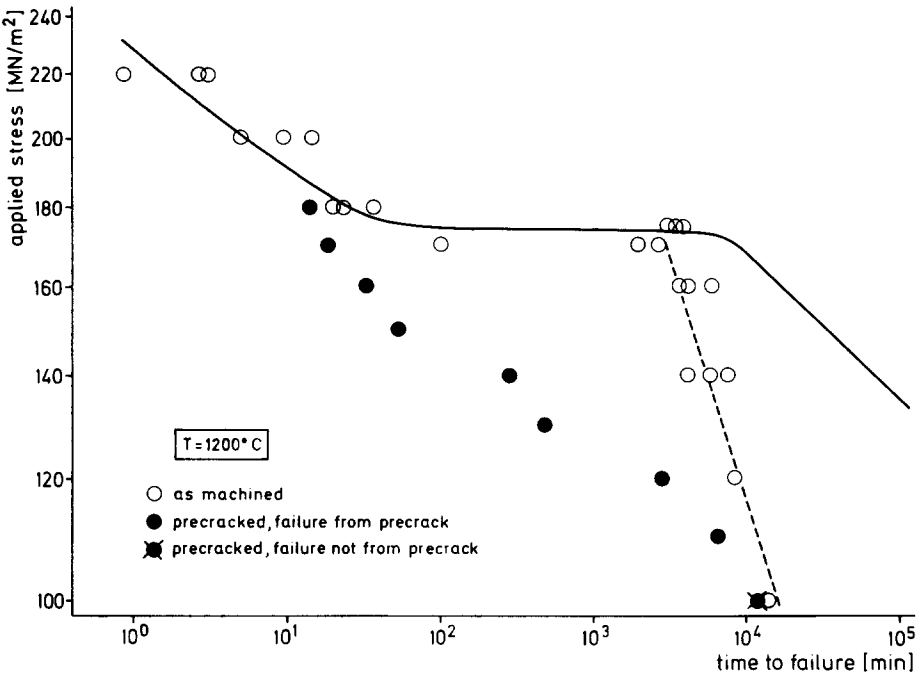

Fig. 9. Stress rupture diagram of HPSN specimens in the as-machined and precracked state.

into account as the result of creep rupture. Grathwohl (1984*b*) reported the fact that growing cracks in HPSN can be stopped due to creep and stress relaxation if a stress is applied on the test specimen that is below a critical value; under these conditions true creep rupture is observed after the critical creep strain is reached. The situation is illustrated in Fig. 9 showing the dependence of lifetime of HPSN on the applied stress for a particular temperature. The lifetime curve deviates from the straight line (expected according to linear-elastic behaviour) at the higher stresses in the direction of extended lifetimes. This and the threshold value for slow crack growth is mainly due to the stress redistribution in the bending beam. If specimens can reach this threshold, no further failure will occur until creep is progressed to the critical levels of strain and microstructural damage. The same phenomena have been observed by Quinn (1984) in other Si_3N_4 materials. Fracture maps have then been developed exhibiting the typical areas of failure.

Since all these experiments were performed in bending, it has again to be emphasised that different lifetime curves are to be expected if the data are measured with specimens with homogeneous and stationary stress distribution. Very few data on tensile stress rupture behaviour have been

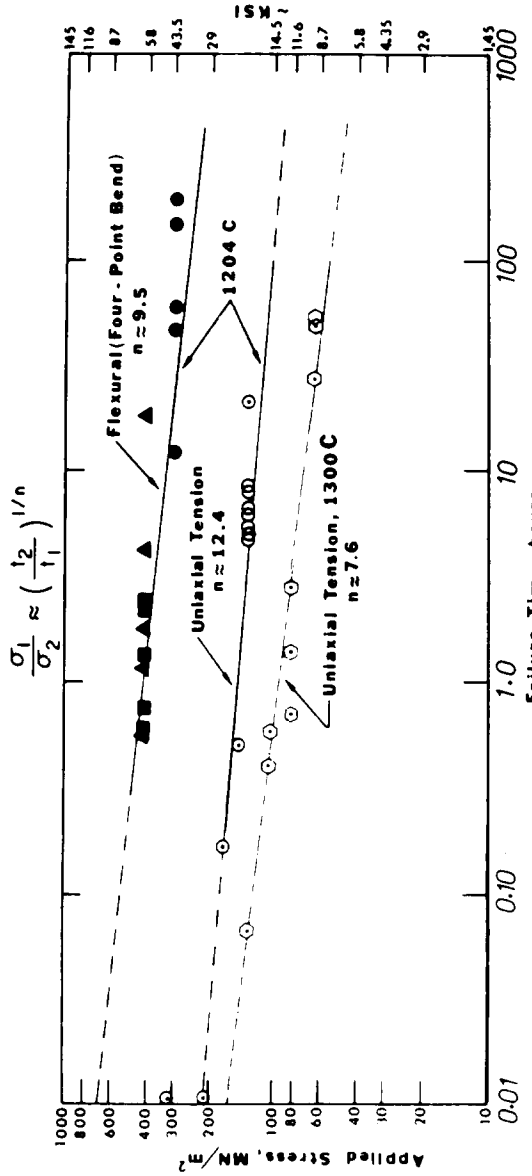

Fig. 10. Flexural and uniaxial tensile stress rupture results for HPSN (Govila, 1982).

published so far. As seen from Fig. 10, large differences are manifested between flexural and tensile stress rupture results not only in the level of the applied stresses but also in the slope of the apparent lifetime curves. In order to compare these results, a further treatment of the data is certainly necessary; a quantitative evaluation requires recalculation of the stresses and the knowledge of the dominating types of flaws in flexure and under tension. While these flaws are typically found in the near-surface region of flexural specimens, the situation cannot be generalised in the case of tension. However, it was shown by Kawai *et al.* (1984) that typical stages in the lifetime curves correlate with typical types of flaws, i.e. internal flaws dominate the case of very short lifetimes and dynamic failure, while large parts of the fracture surfaces are marked by slow crack growth from surface flaws in the case of longer lifetimes at high temperatures and low stresses. It may be learned from this situation that the individual lifetime data have to be analysed in each single case with respect to the acting stresses including the stress distribution in tensile specimens during unsymmetrical slow crack growth and the type of failure and controlling flaw.

6 FATIGUE

Fatigue is defined here as being microstructural and a lifetime-limiting phenomenon due to cyclic stresses. The knowledge of fatigue properties of engineering ceramics at high temperatures is very limited so far; however, with their projected applications in high-temperature components in sight, the understanding of specific cyclic fatigue behaviour of these high-temperature materials becomes very important. After discussion of the other high temperature failure phenomena in the previous sections, it is obvious that loading of a ceramic component under cyclic mechanical stresses at high temperatures induces several degradation phenomena, not just cyclic fatigue. Therefore, the first objective of a cyclic fatigue study would be to clarify whether a specific cyclic fatigue effect exists in these ceramics at high temperatures.

In order to answer this question, all static degradation phenomena due to slow crack growth, creep, environmental attack and microstructural instabilities at high temperatures should be known. Only those types of fatigue phenomena are considered which would not be present if the type of loading were not cyclic. As learned from the previous sections, the database for conventional (static) lifetime phenomena is often not very clear; quantitative and reliable results are missing with respect to their dependencies on actual stresses or other loading parameters. In order to achieve a valuable fatigue database and to quantify the relevant functions

between fatigue and loading parameters, it is necessary that specimens with uniaxial homogeneous stress distributions are used for fatigue tests. Otherwise, stress redistribution effects as discussed before will alter the effective state of loading of the specimen during fatigue testing. In addition, the cyclic stresses have not only to change in magnitude but also in sign during fatigue tests. It may be supposed that fatigue effects exist which are only manifest if reverse stresses are applied to the specimen.

Only a very few fatigue studies on ceramics at high temperatures have been published so far, mostly on the basis of flexural tests applying a range of stressing rates (often referred to as 'dynamic' fatigue). Some further studies employed tensile specimens but stresses remained in tension only during cyclic loading. Kossowsky (1974) has performed reverse cyclic tests at high temperatures, but again these were realised with flexural loading with a cantilever type specimen. More recently, Soma *et al.* (Paper 15) have performed some tests of direct type with reverse stressing. It is suggested that the fundamental question about the existence of specific cyclic fatigue effects can only be answered on the basis of reliable results gained from tension/compression loading of specimens with homogeneous stress distribution.

The actual discussion of this question can best be demonstrated in the light of the fatigue study on HPSN by Fett *et al.* (1986). The quintessence of this work is concentrated in Fig. 11, where cyclic fatigue results (open circles)

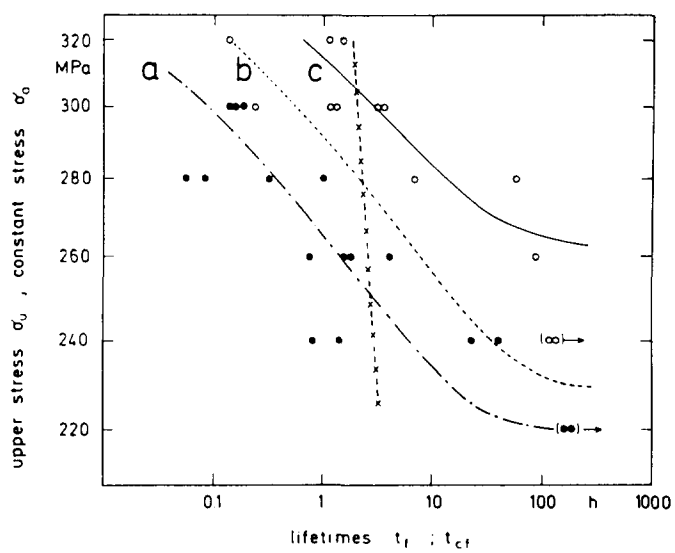

Fig. 11. Comparison of predicted cyclic lifetimes with experimental data of HPSN at 1200°C; predictions (b, c) on the basis of measured static lifetimes (a) considering creep effects (b) and viscous glassy bridges (c) (Fett *et al.*, 1986).

are compared with lifetimes under static conditions (closed circles, fitting curve (a)). As discussed before, the part of the cyclic lifetime which can be explained by static effects has to be identified, and is conveniently done by using a stress function as formulated by Evans and Fuller (1974). Doing this and considering also stress redistribution effects in the flexural specimens due to creep, a cyclic lifetime curve can be predicted as shown in Fig. 11, as dashed curve (b). This prediction underestimates the experimental cyclic lifetimes (c). Thus, cyclic loading provides a beneficial effect on lifetime compared with static loading. This result was explained as an effect of the viscous glassy bridges between the crack edges carrying a part of the applied load in the cyclic but not in the static loading case.

It may be concluded that cyclic fatigue of ceramics containing viscous intergranular phases is not such a severe problem at high temperatures as it can be at ambient temperatures as shown by Grathwohl (1988) and other authors. However, it is again emphasised that reversed stresses should be applied for conclusive results. Since these materials can undergo specific cyclic fatigue at room temperature, it should be investigated how this effect varies with temperature and how it correlates with the brittle character of the failure processes.

7 CLOSING REMARKS

The current testing methods for mechanical properties of engineering ceramics at high temperatures provide materials assessment information rather than design relevant data. This is mainly due to the commonly used flexural test techniques with inhomogeneous and also often unsatisfactory stress distributions in the specimens. Thus, bending is very unfavourable with respect to the objective of evaluation of constitutive laws, e.g. for creep. Although considerably 'easier' in the experimental technique, compared for example with the theoretically favoured tensile test, there are a number of principal limitations and also common errors in performing high-temperature flexural tests.

Tensile tests have been proposed in many laboratories and fracture strength and creep test techniques have evolved using flat or circular tensile specimens with hot, warm or cold grips. In comparison, relatively few tensile data sets have been published so far, possibly due to the high costs and the extreme difficulties of performing high accuracy tests. Standardisation of these techniques would help the scientific community to provide the required data with increased efficiency.

Techniques for evaluation of crack elongation and also creep induced

degradation often rely on compliance measurements with bending, double-torsion or other specimens. This is unlikely to be successful using bending specimens under conditions causing microstructural creep damage, and all other compliance techniques also have to envisage plastic deformation processes eventually taking place and modifying the original compliance approach. In addition, crack growth data as measured by various techniques result in tremendous differences if used for lifetime prediction for real components. In view of the lack of valid extrapolation concepts which take into account all relevant degradation and failure phenomena, lifetime measurement techniques should approximate the real loading conditions as far as possible. The same is true for fatigue testing; fatigue data are scarcely available so far and it can be conjectured that the cyclic fatigue tests do not disclose the whole truth about fatigue of ceramics if only low-cycle tension–tension stresses are employed instead of alternating tensile/compressive stresses.

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