THE CREEP AND FAILURE OF ENGINEERING CERAMICS
UNDER MULTIAXIAL STATES OF STRESS

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by

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ABSTRACT: The effort dedicated to developing the material properties of engineering ceramics has not been accompanied by a similar effort in developing design methods that would allow engineers to make full use of these materials. In particular the high temperature creep behaviour of engineering ceramics has received little attention. In this thesis two parallel approaches, one theoretical and one practical, have been taken towards the final aim of constructing design codes for the creep of ceramic materials.

In the theoretical work the principles developed for modelling creep and failure in metals were employed and adapted where necessary to provide new models that describe the behaviour of ceramics under multiaxial stresses. Important changes were made to account for differences in microstructure between these two classes of materials.

In the practical work equipment was developed to provide suitable multiaxial creep test data with which to verify and further construct models. This involved the construction of a tension/torsion creep testing machine featuring a radio-frequency heating furnace, cooled grip heads, extensometry equipment, biaxial loading system and a temperature measurement and control system. The machine was capable of operating for at least 300 hours at a temperature of at least 1400 °C.

Nine creep tests were conducted on reaction bonded silicon nitride specimens including two unique tests under pure torsion and combined tension/torsion. Four tests were conducted on aluminium oxide specimens including a unique test under combined tension/torsion. Tensile test results showed good agreement with previously published data for both materials confirming the equipment accuracy. Results from the multiaxial tests indicated that reaction bonded silicon nitride fails in response to the value of the effective stress. In addition reasonable agreement was obtained between the test data and predictions from the new models.
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NOMENCLATURE

A  Dimensionless constant.
A_\text{L}, B_\text{L}  Constants of integration.
b  Burger's vector (m).
d  Grain diameter (m).
D  General diffusion coefficient (m^2/s).
D_\text{q}  Frequency factor (1/s).
D_\text{b}  Grain boundary diffusion coefficient (m^2/s).
D_\text{L}  Lattice diffusion coefficient (m^2/s).
D_\text{s}  Surface diffusion coefficient (m^2/s).
D_\text{i}  Solute interdiffusion coefficient (m^2/s).
E  Young's modulus (N/m^2).
f_\text{C}  Area fraction of a grain boundary consumed by a crack.
f_\text{C}'  Growth rate of crack area fraction (1/s).
f_\text{h}  Area fraction of a grain boundary consumed by a void.
f_\text{h}'  Growth rate of void area fraction (1/s).
f_\text{V}  Void volume fraction.
G  Shear modulus (N/m^2).
j_\text{i}  Volumetric flux across a unit length of the i'th grain boundary (m^3/s).
k  Boltzman's constant (1.38 x 10^{-23} J.K^{-1}).
L  Creep damage tolerance.
l_\text{0}  Initial void spacing (m).
l  Void spacing (m).
N  Number of cavities per grain boundary facet.
n  Creep constant.
p  Creep constant relating to grain size.
Q  Activation energy (J mole^{-1}).
R  Gas constant (8.31 J mole\(^{-1}\) K\(^{-1}\)).

r  Void radius (m).

r\(_c\)  Crack tip radius (m).

T  Temperature (K).

t  Time (s).

\(t_f\)  Time to failure (s).

\(U_i\)  Local velocity of separation of the \(i^{th}\) grain boundary (m/s).

\(U_i^s\)  Sliding velocity of the \(i^{th}\) grain boundary (m/s).

\(U_x, U_y\)  Remote elongation in the x and y directions (m).

v  Volume of a given element (m\(^3\)).

\(a\)  Atomic volume (m\(^3\)).

\(v\)  Volume of a void (m\(^3\)).

\(\dot{v}\)  Volume growth rate of a void (m\(^3\)/s).

\(V_i^b\)  Volume flow by boundary diffusion onto the \(i^{th}\) boundary (m\(^3\)).

\(V_i^s\)  Volume flow by surface diffusion away from a growing void (m\(^3\)).

\(X_i\)  Distance from the centre of the \(i^{th}\) grain boundary (m).

\(\alpha\)  Constant.

\(\delta\)  Parameter measuring the effect of stress state on void growth.

\(D_a, D_b\).

\(e_b\)  Grain boundary width (m).

\(e_s\)  Thickness of the layer of material in which surface diffusion occurs (m).

\(\dot{e}\)  Strain rate (1/s).

\(\dot{e}_i\)  Remote strain rate (1/s).

\(\dot{e}_s\)  Secondary creep strain rate (1/s).
\( \varepsilon_0 \)  Creep constant.
\( \varepsilon_f \)  Strain to failure.
\( \varepsilon_e \)  Effective creep strain.
\( \tau_s \)  Surface free energy (\( J m^{-2} \)).
\( \lambda \)  Mean void spacing (\( m \)).
\( \nu \)  Creep constant.
\( \sigma \)  Uniaxial stress (\( Nm^{-2} \)).
\( \sigma_o \)  Creep constant.
\( \sigma_i \)  Principal stress (\( i = 1, 2 \) or \( 3 \)) (\( Nm^{-2} \)).
\( \sigma_e \)  Effective stress (\( Nm^{-2} \)).
\( \sigma_f \)  Stress local to a grain boundary facet (\( Nm^{-2} \)).
\( \sigma_e^* \)  Remote effective stress (\( Nm^{-2} \)).
\( \sigma_C \)  Capillarity stress (\( Nm^{-2} \)).
\( \sigma_i^* \)  Remote principal stress (\( i = 1, 2 \) or \( 3 \)) (\( Nm^{-2} \)).
\( \sigma_i^m \)  Mean stress on the \( i \)'th grain boundary (\( Nm^{-2} \)).
\( \sigma_m^* \)  Remote in-plane mean stress (\( Nm^{-2} \)).
\( \sigma_t \)  Stress normal to a grain boundary at a crack tip (\( Nm^{-2} \)).
\( \sigma_i \)  Excess chemical potential of an atom in the \( i \)'th grain boundary (\( J \)).
\( \omega \)  Measure of creep damage.
\( \dot{\omega} \)  Creep damage rate (\( 1/s \)).
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CHAPTER 1: INTRODUCTION

The phenomenon of creep has been recognised in materials for a long time. It is well established that a knowledge of the time dependent deformation and failure of metallic materials under given load conditions is essential in the design of any component intended for long life service at elevated temperature. A considerable volume of work has been dedicated to a full experimental and theoretical examination of creep in metals. This has lead to the establishment of design codes to allow design engineers to design components based on a knowledge of well characterized material limitations.

Developments in the field of materials science have now considerably widened the range of structural materials available to the design engineer. Of the new breed of materials, the monolithic ceramics are now well accepted as showing potential for improved properties when compared to metals, particularly at elevated temperatures. It should thus follow that if these promising materials are to be used for long life, high temperature components under continuous or cyclic load, they should be subject to the same type of theoretical and experimental characterization as metals. Only then can the option to use such new materials be truly available to the designer.

It is with this in mind that this work was conceived. Work to study the behaviour of monolithic ceramic materials under creep conditions has so far been done under strictly limited conditions, largely determined by the difficulties imposed by fabricating, gripping and heating specimens. This
work describes the construction and testing of equipment that allows the examination of ceramic specimens under conditions of multiaxial stress. It is only through such test conditions that an understanding of the material behaviour in real components can be obtained, and useful design information thus derived.

In addition to this experimental work, a number of theoretical approaches taken for the modelling of multiaxial creep in metals have been evaluated in terms of their applicability in describing the creep of ceramics. As the underlying atomic processes that lead to creep are similar in both ceramics and metals it seemed possible to adopt the same basic principles established for metals to model the behaviour of ceramics. This has been done and has lead to the development of analytical and computational models to describe the microstructural phenomena that occur during the creep and failure of idealised ceramic structures.

With it's completion this work has resulted in the construction of a combined tension/torsion creep test facility capable of use up to at least 1400 °C, from which a limited amount of data for the creep of both reaction bonded silicon nitride and alumina has been obtained. It has also resulted in the development of micromechanical models that predict the type of behaviour that may be expected for the failure of ceramics under creep deformation. These separate developments are described in the following chapters, beginning with a background to the modelling of creep phenomena and the introduction of new models developed to describe the behaviour of ceramics.
PART 1
THEORETICAL ASPECTS OF THE CREEP BEHAVIOUR OF ENGINEERING CERAMICS
CHAPTER 2 : BACKGROUND ON THE CREEP BEHAVIOUR OF ENGINEERING MATERIALS.

2.1 General creep behaviour of materials

The generalised form of creep behaviour in uniaxial tension is shown in FIGURE 1. This general form is observed for all types of materials and is usually characterized in terms of three distinct stages; primary creep, secondary or steady state creep and tertiary creep that leads to failure of the material. Some authors take the view that creep behaviour can not so easily be split into distinct regions (e.g. [1]). The traditional viewpoint of creep as a three stage process is still commonly held and from the standpoint of this work the micromechanical processes that underlie the physical phenomena, rather than generalised creep curve shape are of concern, and the more standard three stage general description will be used where necessary.

It is the purpose of the theoretical part of this study to examine the secondary, and most importantly, the tertiary stages of creep with specific respect to the behaviour of engineering ceramics. In this and the following chapter the existing models for both stages of creep will be outlined and compared. Models that have been developed for metals will be evaluated in terms of their applicability to ceramic materials and existing models for ceramics will be discussed. In chapter 4 new models based on the principles
FIGURE 1: THE GENERAL FORM OF CREEP BEHAVIOUR IN UNIAXIAL TENSION.
discussed here but of direct relevance to ceramic materials are described.

2.2 Secondary creep of materials

The process of secondary or steady state creep in both metals and ceramics has been modelled in terms of phenomenological and mechanistic equations for some considerable time. The relevant equations describing the behaviour of polycrystalline materials have been extensively reviewed by Cannon and Langdon [2] and Frost and Ashby [3]. The mechanisms controlling this process are generally well understood and equations to model the process are well established. The secondary creep strain rate can be expressed in terms of the standard Norton creep law. For uniaxial stress states the Norton law defines:

\[ \dot{\varepsilon}_s = \frac{A D G b}{k T} \left( \frac{b}{d} \right)^p \left( \frac{\sigma}{G} \right)^n \]

(1)

[ \dot{\varepsilon}_s = \text{secondary creep strain rate}, A = \text{dimensionless constant}, D = \text{diffusion coefficient}, G = \text{shear modulus}, b = \text{Burger's vector}, k = \text{Boltzman's constant}, T = \text{temperature}, d = \text{grain diameter}, \sigma = \text{tensile stress}, n \text{ and } p = \text{creep constants} ]

The value of diffusion coefficient being given by:
\[ D = D_0 e^{-Q/RT} \]  

(2)

\[ D = D_0 e^{Q_e} \]  

(2)

\[ D = D_0 e^{-Q/RT} \]  

[ \text{\(D_0\) = frequency factor, \(Q\) = activation energy, \(R\) = gas constant} ]

The parameter \(A\) in the Norton law is a constant derived from the need to take into account the effects of structural variables such as grain shape. Throughout this thesis such structural parameters in models and equations that describe creep and failure will be shown to have a significant importance when making comparisons between the behaviour of metallic and ceramic materials.

The Norton law can also be expressed in the following abbreviated form:

\[ \dot{\varepsilon} = \dot{\varepsilon}_0 (\sigma/\sigma_0)^n \]  

(3)

This form will be used in later chapters where it is more convenient to incorporate a general form of the Norton law. In addition by replacing stress by the effective stress and strain rate by the effective strain rate the Norton equation becomes applicable to multiaxial stresses. This also applies to the following expressions for the different controlling mechanisms.

The mechanisms by which secondary creep occurs fall into two basic types; those where material flows by
diffusion and those where creep occurs by the movement of dislocations. In the case of diffusional mechanisms the creep exponent, \( n \), is close to 1.0 and the constant \( p \) is greater than zero i.e. there is a grain size dependence in the process. When diffusion occurs through the bulk of the grains (Nabarro-Herring creep [4]) the creep strain rate, using the form of equation (1), is given by:

\[
\dot{\varepsilon} = A_{\text{NH}} \frac{D_1 G b}{kT} \left( \frac{b}{d} \right)^2 \left( \frac{a}{\delta} \right)
\]  

(4)

[ \( A_{\text{NH}} \) and \( D_1 \) relate to Nabarro - Herring creep and lattice diffusion respectively ]

and when diffusion occurs primarily along grain boundaries (Coble creep [5])

\[
\dot{\varepsilon} = A_c \frac{D_b G b}{kT} \left( \frac{b}{d} \right)^3 \left( \frac{a}{\delta b} \right) \left( \frac{\delta_b}{b} \right)
\]  

(5)

[ \( A_c \) and \( D_b \) relate to Coble creep and boundary diffusion respectively, \( \delta_b \) is the grain boundary width. ]

The value of \( A_{\text{NH}} \) has been found experimentally to be close to 10 and the value of \( A_c \) has been found to be close to 30 [2]. As stated earlier these values, largely derived from experiment, are included in the model to account for geometric variations of the grains. Simple models do not have the scope to include descriptions of the complex variation in shape and size of real grain structures. It should also be noted that in real cases both diffusional creep mechanisms may occur simultaneously, and the situation may be further complicated by the presence of two or more
diffusing species, where the rate controlling diffusion coefficient must be identified.

Secondary creep may also arise from the motion of dislocations within the bulk of grains. In this situation the process is independent of grain size \((p=0)\) and the creep exponent has a value between 3 and 5. When the value is close to 5, a process of dislocation glide and climb is said to occur, with the climb of dislocations over discontinuities in the lattice being the rate controlling process. For this process the steady state creep rate is given by:

\[
\dot{\varepsilon}_s = A_d c \frac{D G b}{k T} \left( \frac{\sigma}{G} \right)^{4.5}
\]

\([A_d c \text{ relates to dislocation creep where climb processes are rate determining. In this case this constant will also include a value of the active dislocation concentration.}]\]

When the creep exponent is close to 3, the same dislocation climb and glide process is the mechanism of deformation, but with the glide process as rate determining as a result of drag caused by solute atoms in the lattice. For this process the steady state creep rate is given by:
\[ \dot{\varepsilon}_s = A_{d_g} \left( \frac{KT}{GB_s} \right)^2 \frac{D_{Gb}}{KT} \left( \frac{\sigma}{G} \right)^3 \]  

[ \( A_{d_g} \) relates to the process of dislocation creep where dislocation glide processes are rate determining. This constant also includes terms relating to solute atom concentration and the size difference between solute and solvent atoms.] 

In the dislocation creep equations the A constants again reflect material microstructure. In these cases A incorporates a measure of the dislocation structure as the constant is proportional to \((L/b)^{1/2}\) where L is the mean dislocation spacing. This length scale is a characteristic of the microstructure. Similar characteristic spacings relating to damage in tertiary creep will be encountered later and again these will be shown to have a significant part to play in differentiating between ceramic and metallic material models.

Use of all the equations given above to predict creep rates depends on having accurate data for diffusion coefficients and a knowledge of which diffusion process and which atomic species is dominant under the conditions being examined. In most cases prediction of experimental data from these equations is accurate only to an order of magnitude. However, as is the case for many material models, the equations describe accurately the trends in observed response.

The mechanisms described above are generally applicable to both ceramic and metallic materials. Langdon and Cannon
[6] compare creep data for mechanisms where $n = 5$ and $n = 3$
(i.e. power law creep processes) for ceramics and metals and
produce the results shown in FIGURE 2 which point to the
overlap in behaviour that occurs within the two classes of
material. This similarity arises from a similarity in the
dislocation density observed under conditions where power
law creep dominates i.e. the materials have a similar
characteristic length scale (dislocation spacing) under
these conditions. The faster observed creep rate in metals,
for a given set of experimental conditions, reflects the
fact that ceramics generally have higher activation energies
for diffusion and lower intrinsic diffusion rates at a given
ratio of melting temperature to test temperature.

Although the mechanisms that can occur in metals are
the same that can occur in ceramics, there is a difference
in the extent to which a particular mechanism will dominate.
For metals, unlike ceramics, there are relatively few
examples where pure diffusional creep is the principal
mechanism of creep deformation, and in power law creep pure
metals show a tendency to have a stress exponent close to 5
(glide and climb dislocation movement with the climb process
rate determining [6]) while ceramics show a greater range of
behaviour where $n$ may be close to 5 or close to 3. The
concept of deformation maps developed by Ashby [7] to show
dominant mechanisms for deformation over a range of test
variables can highlight such differences. For example the
maps for magnesium oxide and nickel of comparative grain
size in FIGURE 3 (taken from [3]) show the enhancement of
diffusional processes in the ceramic material.
FIGURE 2: THE OVERLAP OF CREEP VS. STRESS DATA FOR METALS AND CERAMICS.
FIGURE 3: DEFORMATION MECHANISM MAPS FOR MAGNESIUM OXIDE AND NICKEL OF EQUAL GRAIN SIZE (FROM [3]).
It has been stated [6] that the enhancement of diffusional creep in ceramics is a result of two factors. The average grain size of most ceramics is an order of magnitude less than that for metals (approximately 10 microns for ceramics, approximately 100 microns for metals). This results in a larger proportion of grain boundary material for Coble creep and smaller diffusion distances for Nabarro-Herring creep in ceramics. Also if grain sizes are comparable, as is the case in FIGURE 3, the di- or poly-ionic nature of ceramics often implies the presence of a small cation capable of rapid grain boundary diffusion favouring Coble creep mechanisms, and the presence of a large anion which slows down the dislocation type processes occurring in power law creep. The prevalence of power law creep with $n = 3$ rather than $n = 5$ is not explained in terms of a solute drag type of process, which is rare in ceramics, but by a process of dislocation climb where crystallographic slip is restricted.

2.3 Tertiary creep and creep failure

The phenomenon of failure in both ceramic and metallic components at high temperature will occur by a range of mechanisms dependent on the exact conditions of temperature and loading. FIGURE 4 shows a generalised relationship between time to failure and applied stress for a typical ceramic material. Three regions of different failure mechanisms have been identified. For ceramic materials high stresses result in short times to failure and this failure is by brittle fracture. One of the major differences in
FIGURE 4: GENERALISED TIME TO FAILURE VS. STRESS RELATIONSHIP FOR A CERAMIC MATERIAL SHOWING FAILURE MECHANISM BOUNDARIES.
behaviour between metals and ceramics is the extent to which the brittle fracture process dominates in ceramics with respect to such processes in metals. Intermediate values of stress result in longer times to failure and the failure mode is by creep crack growth i.e. the slow growth of a dominant flaw in the material. The region of low stress and resulting long times to failure is the area of interest of this work.

Considerable work has been done to understand the mechanisms and model the phenomena associated with these three processes, however the mechanism of failure at low stress that results in long times to failure is the main concern of this work. The relevant mechanisms are generally termed continuum damage processes and this term will be used when discussing tertiary creep and failure.

The influence of temperature on these processes is to enhance the tendency towards creep damage and creep crack growth mechanisms as temperatures increase and diffusion processes become more prevalent in the material. For example the combined influence of stress and temperature on the fracture mechanism for hot pressed silicon nitride has been mapped by Quin [8] giving the diagram shown in FIGURE 5.

As has just been discussed, under conditions of low stress and long periods of time at load, failure will occur by a continuum damage mechanism. It is this process that is responsible for the accelerated creep strain rate observed in the tertiary stage of the creep curve.

The continuum damage failure mechanism is characterized by three stages. Firstly voids will nucleate at preferred sites within the material microstructure. These voids,
FIGURE 5: THE INFLUENCE OF STRESS AND TEMPERATURE ON THE TIME TO FAILURE OF HOT PRESSED SILICON NITRIDE (FROM [8]).
typically at triple grain junctions, will grow along grain boundaries to form facet size cracks. These cracks may then continue to grow and coalesce with other cracks within the material leading to a critical size flaw that causes rupture of the whole body.

Considerable evidence exists for the presence of such damage mechanisms in both metals and ceramics. Whilst such evidence has been long established for metals, evidence that the continuum damage process occurs to produce failure under creep conditions in a wide range of ceramics has emerged more recently. For example FIGURE 6, taken from [9], shows voids, full facet cracks and linked chains of cracks in a high purity sintered alumina examined after creep fracture. Similarly evidence for the same mechanism in silicon carbide [10] and hot pressed silicon nitride [11] has been published.

The build up of damage within a material microstructure, as it is the source of the accelerated tertiary creep rate, is an important measure of the progress of creep failure and highly relevant in design of components experiencing creep conditions. In this thesis two ways of quantifying the process of damage growth are examined. The first of these is by the creep damage tolerance (L) as proposed by Ashby and Dyson [12]. The damage tolerance is related to steady state creep rate ($\dot{\varepsilon}_S$), strain at failure ($\varepsilon_f$) and time to failure ($t_f$) such that:

$$L = \frac{\varepsilon_f}{\dot{\varepsilon}_S t_f}$$  \hspace{1cm} (8)
FIGURE 6: THE MICROSTRUCTURE OF HIGH PURITY ALUMINA AFTER CREEP FAILURE (FROM [9])
This parameter is one experimental measure of creep performance that any model of creep failure should be able to predict. This measure is applicable to both uniaxial and multiaxial creep testing and changes with respect to stress state can be predicted from multiaxial modelling.

The more design orientated testing work done on metals under multiaxial stress states has produced a second characterising feature of the process of creep damage and failure. This is the isochronous surface. This is a graphical depiction of a surface in stress space that connects stress states that lead to equal times to failure in the material. To derive such a surface experimentally a number of tests are required under both uniaxial and more complex stress conditions. The volume of such tests for metallic materials has lead to the discovery of general trends in material behaviour. For example the isochronous surfaces of copper and aluminium (taken from [13]) are given in FIGURE 7. These two materials show two types of typical isochronous surface. Copper fails in response to the value of the maximum principal stress, whilst aluminium fails in response to the value of the effective stress. This conclusion was earlier observed in the experimental work of Johnson et. al. [14]. As yet no data exists to establish the shapes of isochronous surfaces applicable to ceramics, and it is this gap in the data that has prompted the development of a suitable multiaxial creep testing machine. This development forms the bulk of the experimental work undertaken in this study and this is described in part 2 of this thesis.
Maximum principal stress material (Copper)

Effective stress material (Aluminium)

FIGURE 7: THE ISOCHRONOUS SURFACES OF COPPER AND ALUMINIUM (FROM [13]).
CHAPTER 3 : BACKGROUND ON THE MODELLING OF CREEP FAILURE IN ENGINEERING MATERIALS

Work done on the theoretical modelling of the continuum damage process and the resulting creep failure has been to a very great extent concentrated on modelling phenomena observed in metals. Modelling of the same phenomena in ceramic materials has been quite limited. In the following sections existing models for both metals and ceramics are reviewed. It was on the basis of this background, and in particular with a view to the modification of models established for metals so that they can be used in describing the behaviour of ceramics, that the new theoretical modelling described in chapter 4 was undertaken.

3.1 Existing models in metals

A large amount of test data has been produced for metallic materials and a wide range of different test conditions have been used (e.g. biaxial tension, simple torsion, tension-torsion). This practical work has been accompanied by a considerable depth of theoretical modelling. The experimental data and the models have been combined in order to gain an understanding of the high temperature failure behaviour of this class of materials. The mechanisms that lead to the accumulation of damage and eventual failure of metals have been summarised by Ashby and Dyson [12]. As well as void growth and creep crack growth processes that generally can occur in both metals and ceramics, they describe three other possible damage
producing processes. The first is by loss of external section, where a constant volume of material loses width as it creeps. If no other failure mechanism occurs rupture will happen when the material necks down to a point. This process does occur in some pure metals at very high temperatures (above 0.8 of the melting point) but not in ceramics where it is extremely unlikely that necking down will occur to such an extent without failure initiating at an internal flaw.

The second additional process is that where material microstructure changes, either coarsening of second phase particles or changes in the dislocation structure (e.g. dislocation density change or dislocation network formation) influence the creep damage process. Second phase material in engineering ceramics tends to be largely of an amorphous nature and unreactive to coarsening (except by oxidation) however microstructural change can occur in ceramic grains, for example in alumina at temperatures close to the sintering temperature where grain growth may often be observed. This will slow grain size dependent secondary creep processes (Coble and Nabarro - Herring creep) with a resulting effect on localised build up of damage. However for the purposes of this work these effects have been assumed to be secondary to mechanisms involving void formation and growth. This assumption may need to be checked at a later date by experimental observation.

The final damage mechanism identified for metals is that where damage grows by a process of oxidation. This process is obviously not relevant to oxide ceramics with little or no grain boundary phase, however oxidation as a
cause of high temperature failure has been observed in non-oxide ceramics such as silicon nitride (e.g. [15]) and a number of mechanistic studies have been undertaken to enable the process to be modelled empirically [16].

Of the above mechanisms those most relevant to the creep of both metals and ceramics under the low stress\long time to failure conditions examined in this work are failure by oxidation damage and failure by continuum growth of voids. Modelling of oxidation induced failure has not been included within the scope of this work, however such models may be required at a later date to describe the failure of the reaction bonded silicon nitride samples tested. The continuum damage process is that of most concern to this work and the remainder of this section concentrates on models derived to describe this particular mechanism.

Two essentially different approaches have been taken to the modelling of the continuum damage process. A mechanistic approach has been taken where the macroscopic processes observed in creep failure are described in terms of the micromechanical processes occurring within the structure of the material. The nature of this type of modelling may well differ between ceramic and metallic materials as their microstructures have important differences. Alternatively creep failure has been modelled in terms of a macroscopic measure of the damage within the bulk of the material. This second technique is generally termed as a phenomenological model. It is not necessary to include descriptions of the nature of material in this model, and provided experimental data is available it should apply to any type of material. The following two sections look at these two different
approaches. Each one derives expressions for strain rate that take account of the state of damage within a material. Generally these expression take the form:

\[ \dot{\varepsilon} = f(\sigma, \omega) \quad \dot{\omega} = f'(\sigma, \omega) \]  

(9)

where \( \omega \) is a measure of the damage in the material. The expression of \( \omega \) may be specific, for example it may be the area fraction of voids in the mechanistic models, or it may be more general as is the case in the phenomenological models.

3.1.1 Mechanistic models for continuum damage failure of metals

The purpose of mechanistic models is to describe the process of void nucleation, growth and coalescence that characterises continuum damage failure by describing the micromechanical events that lead to these features.

A number of efforts have been made to model the initial stage of void nucleation. Theoretical analysis [17] indicates that a tensile stress of the order of \( E/100 \), where \( E \) = Young’s modulus, is required for nucleation to take place. Such a high stress, being an order of magnitude greater than typical creep test stress, implies some form of local stress concentration is required. Such stress concentrations may be at triple grain points, particulate inclusions, ledges or at the intersection of slip bands and grain boundary particles. Such mechanisms have been described and reviewed in a number of papers (e.g. [18], [19], [20]). These stress concentrations arise from the
driving force of intermittent, i.e. stochastic, grain boundary sliding processes [21]. This process of periodic stress peaks caused by stochastic grain boundary sliding has been modelled by Chan and Page by making use of standard probability expressions for stochastic processes [20]. They show a linear relationship between cavity density, grain boundary sliding displacement and creep strain. They also observe that these results appear to apply equally to both ceramic and metallic materials.

Whilst these models give a good indication of the type of processes that give rise to cavity nucleation, they only predict the relationship between such values as cavity nucleation rate and observed creep strain. They do not directly predict the magnitude of these values. It is also worth noting that, as is pointed out in a number of earlier papers (e.g. [18]) the presence of existing cavities in both engineering ceramics and metallic materials as a result of processing conditions will automatically provided damage sites from which subsequent growth and coalescence of voids can occur.

Taking into account the previous discussion, it has been the case that to incorporate an expression for void nucleation rate into general micromechanical models authors have relied on experimentally observed relationships between void population and strain rates. For example Tvergaard [22] uses the following experimentally derived expressions to relate the number of cavities per grain facet, \( N \), to effective strain, \( \varepsilon_e \).
The process of void growth has been modelled more precisely than the process of void nucleation. In such modelling the growth of voids on an isolated boundary can be considered as an initial starting point. Cocks and Ashby [23] list a number of possible controlling mechanisms by which this void growth may occur. These are by surface diffusion, by grain boundary diffusion, by power law creep of the surrounding matrix material or by any combination of these mechanisms. The basic mechanisms are summarised in FIGURE 8 taken from [23].

Expressions for void growth rate can be derived in terms of the principal facet stress, $\sigma_f$. This term is used rather than maximum principal stress to take into account the fact that voids grow in response to local facet stresses. These stresses will differ from remote stresses as a result of grain boundary sliding effects. In the equations that follow the simplified form of the Norton law given in equation (3) has been followed.

The value of void growth rate where grain diffusion alone is the mechanism of growth is given by the following equation:
FIGURE 8: VOID GROWTH MECHANISMS (FROM [23]).

a) VIA GRAIN BOUNDARY DIFFUSION.

b) VIA SURFACE DIFFUSION.

c) VIA POWER-LAW CREEP OF MATRIX MATERIAL.
\[
\dot{f}_h = \frac{\xi_0 \tau}{f_h^{1/2} \ln(1/f_h)} \left( \frac{\sigma}{\sigma_0} \right) (l_0/l)^3 \quad (11)
\]

where \( \tau = \frac{2D_s \xi_o \theta}{kT l_o^2} \frac{\sigma_o/l_o}{1} \)

[ \( f_h \) = area fraction of a grain boundary consumed by a void, 
\( \theta \) = atomic volume, \( 2l_0 \) = initial void spacing, \( 2l \) = void spacing ]

Whilst for surface diffusion alone:

\[
\dot{f}_h = \frac{\xi_0 \psi f_h^{1/2}}{(1/f_h)^{3/2}} \left( \frac{\sigma}{\sigma_0} \right)^3 (l_0/l)^4 \quad (12)
\]

where \( \psi = \frac{D_s \xi_s}{kT l_0} \frac{\sigma_o^2/l_o}{\gamma_s^2} \)

[ \( D_s \) = surface diffusion coefficient, \( \xi_s \) = thickness of the layer of material through which surface diffusion occurs, 
\( \gamma_s \) = surface free energy. ]

In addition to the creep constant, \( \xi_0 \), these expressions contain terms relating to void area fraction, \( f_h \). This quantity is the measure of damage, \( \omega \), in the general form given in equation (9). The quantities \( \tau \) and \( \psi \) also have considerable physical significance in that they represent an initial measure of damage within the material \( \omega_0 \). They contain a length scale relating to the initial spacing of voids which is a characteristic of the material being analysed. This value itself will also relate to the nucleation process that determines the number of cavities on a grain boundary. One important consequence of this length scale is that the analysis for metals assumes that the material behaves as a continuum, that is that the zone of
material over which damage occurs is large compared to the characteristic length scale. This is not always the case for ceramic materials. The consequence of making such an assumption for ceramic materials, particularly early on in tertiary creep, is investigated in the newly developed models described in chapter 4.

Manipulation of the above equations can lead to expressions for the strain rate that occurs as a result of void growth. They may also be evaluated in stress space to give surfaces of constant void growth rate. However the value of the local facet stress must be taken into account rather than the simple macroscopic stresses.

In the third type of void growth mechanism a void may grow as a result of power law creep occurring in the surrounding matrix. The area of material shaded in FIGURE 8(c) creeps according to the magnitude of the local stress field. The remaining matrix material will creep according to the bulk stress field. The result is a difference in the creep rates as shown and a net dilation in the shaded zone i.e. a net growth of the void. Again an expression for the void growth rate is given in terms of void area fraction and effective stress, $\sigma_e$:

$$\dot{f}_h = \dot{f}_0 \beta \left[ \frac{1}{(1-f_h)^n} - (1-f_h) \right] \left( \frac{\sigma_e}{\sigma_0} \right)^n \tag{13}$$

The term $\beta$ in this expression is a parameter that measures the effect of the stress state in the material on the void growth rate. For the case of simple tension $\beta$ has a value of approximately 0.6. For more complex stress states the value of $\beta$ is more difficult to obtain, however Cocks
and Ashby do give an expression for this term under combined axial and hydrostatic tension [23]. In general \( \theta \) is a function of \( \sigma_m/\sigma_e \) where \( \sigma_m \) is the mean or hydrostatic stress.

The three mechanisms discussed above may act simultaneously resulting in overall growth rate being determined by the coupling of mechanisms as shown in FIGURE 9. The dominant mechanism may also change during the lifetime of the material as local stresses build up and relax.

In addition to the processes that cause voids to grow on an isolated boundary the effect of the more general microstructure must be considered if void growth in a realistic engineering solid is to be modelled. The grain structure of such solids allows for the possibility of sliding behaviour of the grain boundaries and this sliding is essential to accommodate void growth by diffusion. The result of this behaviour is the relaxing of shear stresses on sliding boundaries and the subsequent build up of stresses on adjacent areas. This can have a very significant effect on the overall growth rate of voids. Tvergaard [24] concluded that the effect of including free grain boundary sliding in overall models for void growth was to dramatically increase the growth rate. In his model the predicted time to coalescence of voids on a single boundary was between 0.15 and 0.4 of the value predicted if the grain boundaries were rigid. Cocks and Ashby [23] provide an analysis for a sliding boundary in a two-dimensional array of hexagonal grains. The orientation of the plane of analysis can be at any angle to the remote stress, so by
Figure 9: The coupling of void growth mechanisms with respect to stress (σ) and area fraction of voids.
averaging the results over all values of the angle of orientation they derive the following expression for local stress on the sliding grain boundary in terms of the remote stresses.

$$\sigma_x = \frac{4}{3} \sigma_1 - \frac{1}{6} (\sigma_2 + \sigma_3)$$  \hspace{1cm} (14)

More generally Cocks and Leckie [25] have used:

$$\sigma_x = C \sigma_1 + \sigma_m$$

where \( \sigma_1 \) is the maximum principal deviatoric stress. The constant \( C \) is a measure of the amount of grain boundary sliding. In general \( C \) will be greater than 1. As \( C \) approaches 1.0 grain boundary sliding ceases to have an effect.

Incorporating grain boundary sliding alone is not sufficient to fully model the void growth. A further modification to the modelling is also required to take into account another feature of real engineering solids. This modification is necessary as simplified models for void growth assume a uniform distribution of voids throughout the material. In practice the nucleation of voids uniformly on all boundaries is not always observed for metals. In such cases cavitated boundaries can be surrounded by areas of undamaged material that may be creeping at a different rate to that in the damaged area. The growth of voids is then limited by the rate at which the matrix material can deform. Such a mechanism has been termed constrained cavity growth [26]. This mechanism was first analysed by Rice [27] where a grain facet containing a chain of penny shaped cracks was
surrounded by a power-law creeping matrix. Cocks and Ashby [23] also modelled such a situation and include cases where void growth is by boundary diffusion and matrix creep is either by diffusional, power-law or a coupling of the two mechanisms. When both processes are diffusion controlled and the separation between cavitated boundaries, again a characteristic length scale of the material, is much larger than the grain size void growth rate is controlled completely by the rate at which the matrix creeps. This result is of considerable consequence when considering creep models derived for metals that are suitable for ceramic materials. It is precisely these conditions that prevail in ceramic materials and the new modelling discussed in the following chapter takes this into account. When matrix creep is by power-law processes however these authors concluded that void growth rate is dependent only on the void area fraction and independent of the spacing between cavitated boundaries. This case is more applicable to metals where creep is generally dominated by dislocation mechanisms.

The models developed to describe void growth can be summarised as follows. For isolated voids growing on a grain boundary facet in a uniform material expressions for growth by diffusional, power-law and combined processes can be derived. To account for the granular structure of engineering materials it is necessary to incorporate expressions that allow for the redistribution of stress by grain boundary sliding. Finally if not all the boundaries are cavitated the resulting constraint on void growth imposed by the surrounding undamaged matrix material must also be included in the models.
The final process in the sequence of damage growth that leads to failure is cavity coalescence. This is the least studied of the three phases of the continuum damage mechanism. However this process does not strongly influence the time to failure provided a reasonable criterion for coalescence is chosen. In most models it is generally assumed that a grain boundary fails when $f_h > 0.25$ for that boundary.

The aim of deriving the types of models described above must be to provide a means of predicting the response of engineering materials and components in realistic stress states. An example of the combination of different models covering the three stages of continuum damage creep failure is given by Tvergaard [28] for conditions of multiaxial stress. Nucleation is incorporated by the use of an experimentally observed relationship between cavity nucleation rate and effective strain rate. Growth of damage is by diffusion combined with matrix creep and the effects of both grain boundary sliding and constraint are considered. Different distributions of cavities, nucleation conditions and loading histories are modelled and the results presented in terms of isochronous surfaces. For most of the conditions considered the results indicate material response close to the surface of an effective stress material such as aluminium as discussed in chapter 2.3. The model however did not easily produce results close to the maximum principal stress response of pure copper. As with all micromechanical models a number of assumptions are required to allow the highly complex structure and behaviour of real materials to be described. For example the Tvergaard model assumes that
the density of cavitated boundaries remains constant throughout the tertiary creep process, which is not the case in practice. Other variations of this model have examined where tertiary creep only occurs as a result of fully formed cracks, as is approximately the case in practice [25]. In this case cracks perpendicular to the maximum principal stress are fully constrained. Tertiary creep is then dependent on the magnitude of this principal stress and on how cracks that do not lie perpendicular to the maximum principal stress behave.

The use of the mechanistic type of approach to creep analysis can be extended to simple components by the incorporation of models into finite element methods. The diffusional and geometric constants that are needed to determine precise behaviour can be obtained from experiment. In general however these values, particularly in the case of diffusion coefficients, are only known to a poor degree of accuracy from experiment, if at all. However this approach does allow a detailed analysis of important trends in behaviour to be performed.

The adaptation of this type of mechanistic analysis developed exclusively for metals to modelling ceramics is considered in chapter 4. A successful approach depends on recognising the way in which differences in the microstructures between the two classes of materials influence the exact nature of the void nucleation and growth processes, and developing methods to incorporate these differences in a mechanistic model.
3.1.2 Phenomenological models for continuum damage failure of metals

In these models instead of examining the microstructural development of damage a simple measure of damage, $\omega$, is assumed as in the general form of equation (9). However no direct relationship to a physical characteristic such as area fraction of voids is attempted. Instead the value of $\omega$ is assumed to be zero at the start of the test and one at failure. This type of approach was introduced by Kachanov [29] and extended to multiaxial stress states by Hayhurst [30]. Using the form of equation (9) the basic relationships between stress, strain and damage are given by:

\[ \dot{\varepsilon} = \dot{\varepsilon}_0 + \dot{\varepsilon}_0 \left( \frac{\sigma}{\sigma_0 (1-\omega)} \right)^n \]  \hspace{1cm} (15)

and

\[ \dot{\omega} = \dot{\omega}_0 \left( \frac{\sigma}{\sigma_0 (1-\omega)} \right)^\nu \]  \hspace{1cm} (16)

$\dot{\omega}_0$, $n$ and $\nu$ are creep parameters determined experimentally.

By integrating these equations between $\omega = 0$ and $\omega = 1$ the following equation for time to failure can be obtained:

\[ t_f = \frac{1}{(\nu+1) \dot{\omega}_0} \left( \frac{\sigma}{\sigma_0} \right)^\nu \]  \hspace{1cm} (17)

The above equations, combined with a simple experiment to determine the value of $\dot{\omega}_0$, can accurately predict times to failure and creep curves for simple material tests. When
applied to multiaxial stress states [31] equation (16) can be generalised to:

\[ \dot{\sigma} = \dot{\sigma}_0 \left( \frac{X(\sigma_{ij})}{\sigma_0 (1-\omega)} \right)^\nu \]  

(18)

The function \(X(\sigma_{ij})\) describes the shape of the isochronous surface and generally has the form:

\[ X(\sigma_{ij}) = \alpha \sigma_{ij} + (1-\alpha)\sigma_e \]  

(19)

In the case of a principal stress type material \(\alpha = 1\) and in the case of an effective stress type material \(\alpha = 0\). Isochronous surfaces with any intermediate value of \(\alpha\) will result from an intermediate type of behaviour. These equations have the advantage of being relatively simple to use in practice, as they only require a knowledge of the isochronous surface shape and experimental values of creep constants to predict creep curves and time to failure. However they do not give the insight into the controlling mechanisms and microstructural features observed in tertiary creep that micromechanical modelling provides.

As with the mechanistic models this approach is capable of being extended from the analysis of a basic test geometry to cover the analysis of simple engineering structures [32]. Finite element methods can be employed as before however the simpler form of the phenomenological model allows for a more direct approach. This approach introduces a new parameter to represent the global damage in a structure in the same way that \(\omega\) represents damage in a simple test specimen. This parameter can be obtained by integrating over the structure volume a function for damage for each element of the
structure in the simplest case, or over a specific damaging area of the structure as is the case in, for example, a plate containing a central hole. For the second case it is necessary to identify a failure mechanism for the damaging material, and from the stress distribution within the damaging region the time to failure of the component can be determined.

These models contain no assumptions concerning events occurring at the microstructural level, except that the formation of damage is the cause of tertiary creep and failure. Thus there is no fundamental reason to suppose that phenomenological models should not also describe the failure of ceramics. It is only the lack of suitable data from which to derive the shape of the isochronous surface that is needed before this type of model can be attempted for ceramic test specimens and components. As has been stated earlier it is the aim of this work to construct equipment capable of obtaining this data.

Before proceeding to look at the types of models that have been proposed for ceramic materials it should be noted that Cocks and Ashby [23] have made direct comparisons between the results produced by mechanistic and phenomenological models. As has been noted in the discussion of both types of modelling a general form of expression applies. Thus equations assuming a direct physical interpretation of damage can be written in the more general phenomenological form. For example taking the general form of equation (9) the expression for power-law creep type void growth (equation (13)) can be written thus:
\[ \dot{\omega} = \dot{\omega}_0 \theta \left[ \frac{1}{(1-\omega)^\nu} - (1-\omega) \right] \left[ \frac{\sigma}{\sigma_0} \right]^\nu \] (20)

Here \( f_h \) has been replaced by \( \omega \), \( \dot{\epsilon}_0 \) by \( \dot{\omega}_0 \) and \( n \) by \( \nu \). This form is close to that of equation (16) and becomes identical for the case of simple tension where \( \theta \) is approximately 1 and when \( f_h \) is large (i.e. the second \( 1-f_h \) term can be neglected). This indicates that the two approaches are in good agreement generally, particularly when the area fraction of voids, in the micromechanical case, or the damage, in the phenomenological case, is large. However as the phenomenological approach assumes the material behaves as a continuum some significant differences do appear at low degrees of void area fraction (damage) where characteristic length scales are large. These differences result from a need in the continuum analysis for a finite damage rate even when there is no damage, and for the damage rate to be always accelerating with increasing damage.

3.2 Existing models for creep failure in ceramics

The volume of work dedicated to modelling creep failure in ceramic materials is considerably less than that for metals. Those models that do exist tend to be of the micromechanical type where diffusional processes within the structure are modelled under the simple cases of uniaxial stress. Before discussing these models it is necessary to point out some of the microstructural features that occur in ceramics that can considerably effect properties such as resistance to creep failure.
With a few specialised exceptions, it is generally true that polycrystalline ceramic materials are formed by a process of heating powdered raw material at a temperature below the bulk material melting point in order to fuse together the powder particles to form a dense monolithic material. This process is known as sintering and, whilst used to form some metallic materials, is the method most commonly associated with the forming of engineering ceramics. The sintered microstructures that result are dominant in determining the mechanical, thermal and electrical properties of the final material. The creep properties are no exception and before beginning to consider any models for tertiary creep and failure a distinction has first to be made between two basic types of ceramic material.

In ceramics where a liquid phase forming sintering aid has been added during the fabrication process, usually to lower the sintering temperature or improve the sintered density, this phase typically remains at grain boundaries or at grain triple points after cooling as an amorphous material of relatively low viscosity. For example it is typical to fabricate hot pressed silicon nitride with the addition of MgO, Al₂O₃ or Y₂O₃ to facilitate sintering [33]. The viscous flow of the amorphous material that results from such additives after sintering plays an important part in the creep deformation and failure of the material.

It is possible however to sinter some materials, for example aluminium oxide (alumina) to high density with little or no additives. It is also common to remove the need to add sintering aids by applying both pressure and heat
during the sintering in the processes of hot pressing or hot isostatic pressing. Such materials have their creep properties more directly determined by diffusional rather than viscous flow processes.

3.2.1 Modelling failure in ceramics containing boundary phases

In the case of materials having a significant amount of grain boundary material as a result of processing creep failure has been modelled using a liquid phase sintered silicon nitride as a typical material [34]. After creep testing of this material triangular voids are observed to be present in pockets of amorphous grain boundary phase at triple grain junctions. Very occasionally oblate voids at two grain junctions and small spherical voids within the triple point phase are observed.

A model for this process was proposed [35] whereby a threshold stress is required to nucleate voids at triple points, below which deformation occurs by a solution/reprecipitation process [36] and above which full facet size cavities develop from hole nuclei. Depending on the magnitude of the stress normal to the grain boundary, this development can be from oblate holes on two grain junctions (high normal stress) via viscous flow as shown in FIGURE 10(a-c), or from spherical triple point voids (lower normal stress) by viscous flow combined with solution/reprecipitation of the viscous phase as shown in FIGURE 10(d-f). Models have been proposed for both of these processes. Viscous flow is modelled on the basis of
FIGURE 10: THE FORMATION OF FULL FACET CAVITIES FROM OBLATE OR SPHERICAL HOLES (FROM [36]).
equations that describe the flow of liquid through narrow channels and the behaviour of a liquid meniscus. Instabilities in the meniscus behaviour were shown to result in a finger-like surface of the growing cavity. Such growth features have been observed in crept liquid phase containing ceramics [34]. The solution/reprecipitation process was also modelled, either as a diffusion or an interface controlled process, and equations were obtained for both mechanisms.

Neither of the materials used for testing in this work contain significant quantities of grain boundary phase. As a result the type of models discussed here based on viscous flow have not been required. The following section discusses models more suited to the materials actually tested.

3.2.2 Modelling failure in boundary phase free ceramics

In materials that are sintered with no additives or a very small percentage of additive, for example high purity alumina, creep can generally be assumed to be entirely a diffusion controlled processes.

The most comprehensive existing models for the continuum growth mechanism in grain boundary phase free ceramics have been produced by Evans and co-workers ([9], [37], [39]). They take a micro-mechanical approach where the growth of voids is described by basic diffusion laws using an established method developed for cavity growth in a generalised material [38]. They also recognise that the viscous sliding of grain boundaries is required for the accommodation of void growth. They model the formation of equilibrium shaped cavities at preferred sites within the
microstructure e.g. areas of high grain contact angle, and predict the growth of these cavities according to the sequence shown in FIGURE 11: Equations were obtained to predict the deformation rates attributable to each type of cavity with respect to the undamaged deformation of the material by Coble creep. However these equations were obtained by the simple addition of void growth models on top of Coble creep. No account was taken of the interaction of these two events as would be occurring in a constrained type of void growth. This is a major failing of this model in that it is highly likely that the microstructure developed in a ceramic during tertiary creep, particularly at the early stages of damage formation, will have growing voids surrounded by large areas of undamaged material that will constrain such growth.

In a second related analysis taking the same approach [39] the influence of cavitated zones on the surrounding material was considered, with three processes being identified. When deviations of the parameters that influence cavity nucleation and growth (dihedral angle and surface diffusivity) are small, the damaged zone spreads rapidly, the voids remain small and the time to failure is long. If appreciable variation in these parameters occurs full facet cracks are predicted to form rapidly and such damage zones are isolated. Such isolated full facet cracks have been observed in alumina [9]. In zones where the dihedral angle varies considerably but diffusion rates are constant, void formation occurs within a limited zone and growth and coalescence are rapid leading to short times to failure.
FIGURE 11: THE SEQUENCE OF FULL FACET CAVITY GROWTH FROM EQUILIBRIUM CAVITIES (FROM [9]).
For set damage zone conditions expressions for cavity growth velocity and local stresses in the cavity region are obtained from the model and the behaviour predicted is shown in FIGURE 12 where cavity growth velocity and local stresses are plotted in terms of the ratio of cavity length to grain facet length. By integrating the cavity growth equations over time, values of time to propagate cracks of a given length could be predicted.

The recognition of zones of localised damage that exist in an undamaged material has incorporated the ideas of constrained growth in this model. This analysis is thus a more reasonable description of a ceramic material. However the type of analysis is still the continuum one used for metallic materials. Such a model is appropriate only when the damage zone is large compared to the characteristic length scales of the microstructure, and hence not suited to a ceramic material unless the damage formed is very extensive.

3.3 Summary of theoretical background material

The above sections provide a brief overview of the theoretical work done so far in the field of creep and creep failure, with particular emphasis on modelling the failure process. The underlying similarity in creep and creep failure mechanisms between metals and ceramics is not reflected in the quantity of work covering modelling of the failure process in both materials, with little attention being paid so far to applying the design orientated methods developed for metals to ceramics. This is partly explained
FIGURE 12: CERAMIC MATERIAL MODEL PREDICTIONS (FROM [9]) OF:

a) CAVITY GROWTH VELOCITY ($\dot{a}$) vs. AREA FRACTION OF CAVITY ($f_h$).
b) LOCAL STRESS ($\sigma_f$) vs. AREA FRACTION OF CAVITY ($f_h$).
by the relative novelty of engineering ceramics as potential materials for practical components, and partly by a lack of suitable data to base models on and to test out predictions.

The micromechanical models that have been proposed for ceramics are much more limited than those for metals, although their basis is generally the same. Two areas of deficiency in the existing models can be highlighted. Firstly early models take no account of the interaction between the processes of void growth and the deformation of bulk material. This is a key feature of models that describe the failure of metals and is essential in describing the build up of local stresses and the resulting formation and growth of damage. In addition no full account is taken in any of the models of the potential influence of grain structure on damage mechanisms. The situation in ceramic materials where voids form at triple grain junctions and the spacing between voids may be several or very many grain boundary lengths is considerably different to that observed in metals where numerous voids may occur on a single boundary. This makes assumptions made in continuum models invalid as the characteristic length scales, such as void spacing, adopted for modelling will be much larger than the size of the damaged region.

The following chapter looks at taking the micromechanical approach typically used to model void growth in metals, and considers the adaptations necessary to develop new models for ceramics by taking into account the effects of constraint and the microstructural differences between the two classes of materials.
CHAPTER 4 : NEW MODELS FOR THE GROWTH OF DAMAGE IN CERAMIC MATERIALS.

4.1 Introduction

The previous section has detailed the types of models that exist to describe the process of continuum damage failure in metals, and outlined the basis on which a similar approach to modelling ceramic materials would be thought suitable. The need to introduce into models for ceramics a consideration of the effect of grain structure and of the effect of interactions between void growth and matrix creep was highlighted. The following sections detail the development of such mechanistic models for the diffusional growth of damage in a ceramic.

Two models have been derived that examine the growth of damage at both an early stage and at a stage where damage is more widespread. The first model looks at situations where the formation of damage is well established and the distance between voids and cracks in the microstructure is of the order of a few grain boundary lengths. This situation is relatively simple from a modelling point of view in that the diffusion occurs over only a few boundaries and the possible paths for diffusion are limited. This allows the modelling to be done analytically. The results from this model are used to point out the effects of constraint on the various stages of damage growth in ceramic materials.

The second model looks at the early stages of damage growth where voids are separated by many grain boundary lengths. In this case the extent of diffusion and the number
of possible pathways are considerable and a computation based model is required. The results from this model are used to point out the influence of characteristic length scales and the role played by the bulk of material in determining deformation behaviour. The results are also compared with those obtained by models of the type used for metallic microstructures.

In both models predictions of the damage growth rate are made. Discussions of the physical significance of these predictions is included in the relevant sections. In addition the way in which these models could be used to predict physical data and actual comparisons with the data obtained from the test equipment are given in chapter 15.

4.2 The analytical model.

When microstructural models of metals have been constructed the presence of a large number of voids per grain boundary during tertiary creep allows the assumption that void growth and deformation can be analysed independently, giving information about void growth rates and creep rate in terms of local average stress. When these expressions are combined results are produced describing the behaviour of the whole material [40].

In contrast the microstructure developed during tertiary creep of fine grained engineering ceramics may typically only contain a small number of cavitated grain boundaries, with a significant distance (several grain boundary lengths) between voids. Thus the basic unit for all modelling cases is a single void located at a grain boundary.
shape which, for simplicity, is assumed to be circular in two dimensions. As the void grows by diffusional processes surface diffusion must be at least as fast as boundary diffusion if the equilibrium shape is maintained. In fact as stresses increase or the voids become large boundary diffusion begins to dominate and a 'nose' begins to grow along the most highly stressed boundary. Finally this process results in the formation of a full facet crack running the length of the grain boundary. These situations are illustrated in FIGURE 13. Such boundary cracks can themselves continue to grow by surface and boundary diffusion. Evidence for such structures in crept ceramic materials exists in the literature [41].

To perform the analysis a number of repeating units are identified containing voids, crack-like cavities or full facet cracks. These repeating units are shown in FIGURE 14. The full analysis used to derive remote strain rates and void growth rates is given in APPENDIX A. The simplest of these repeating units contains no voids at all (FIGURE 14(a)), i.e. the material will deform by boundary diffusion alone. For this case the model predicts remote strain rates as follows.

\[
\varepsilon_1 = -\varepsilon_2 = \frac{D_b \delta_b R}{kT \delta^3} \left(\sigma_1 - \sigma_2\right) \quad (20)
\]

This equation is the standard one for Coble creep under biaxial stress. From it can be seen that, in two dimensions, deformation is driven by in-plane shear stress. For a simple case such as this in-plane shear stress can be related to
FIGURE 13: THE DIFFERENT TYPES OF CAVITY CONSIDERED FOR ANALYTICAL MODELLING.

a) TRIPLE POINT EQUILIBRIUM SHAPED CAVITY.
b) TRIPLE POINT CAVITY WITH A CRACK-LIKE EXTENSION.
c) FULL FACET CRACK.
FIGURE 14: REPEATING UNITS SHOWING THE CAVITY AND CRACK DISTRIBUTIONS EXAMINED BY THE ANALYTICAL MODEL.
the 3-D deviatoric components of stress [42] with
deformation being independent of hydrostatic stress.

The introduction of voids at all triple points is the
next stage in the modelling. This is illustrated by the
repeating unit in FIGURE 14(b). As this unit contains a
uniform distribution of voids no constraint can occur as all
areas will deform at the same rate. The following
expressions for remote strain rate are obtained:

\[
\dot{\gamma}_1 = 36 \frac{D_b \delta_b 0}{kT d^3} \frac{(D_b \delta_b 0)}{(1-f_h)^3} + 18 \frac{D_b \delta_b 0}{kT d^3} \left[ \frac{D_b \delta_b 0 (1-f_h)}{(1-f_h)^3} \right] \\
\dot{\gamma}_2 = 36 \frac{D_b \delta_b 0}{kT d^3} \frac{(D_b \delta_b 0)}{(1-f_h)^3} + 18 \frac{D_b \delta_b 0}{kT d^3} \left[ \frac{D_b \delta_b 0 (1-f_h)}{(1-f_h)^3} \right]
\]

\[\underline{\sigma}_C = \text{capillarity stress}\]

where \(f_h = \frac{2 r}{l}\) is the area fraction of boundary consumed by
the growing void. When void size is small the first term in
the equations reduces to the Coble creep expression. The
second terms relate to dilational deformation. If

\[
\frac{\sigma_1 + \sigma_2}{2} \leq \sigma_c (1-f_h)
\]

where the term on the left represents the macroscopic mean
stress voids will not grow but sinter. As the mean stress
increases voids begin to grow, and when \(\sigma_C\) becomes small
compared to the applied stress dilational strain becomes a
significant part of the overall deformation.
Void growth rates can be derived from the expressions above. Details of the procedure required are given in APPENDIX A. The expression obtained for void growth rate is:

$$f_n = \frac{108/3}{k} \frac{D_b \sigma_n \left(\sigma_m - \sigma_n (1-f_n)\right)}{kTd^3 f_n (1-f_n)^3}$$

(24)

where $\sigma_m$ is the remote in-plane mean stress ($\sigma_1 + \sigma_2)/2$. This allows the calculation of a surface in stress space linking stress states that give a constant void growth rate. Such a surface is shown in FIGURE 15 for the 2-D plane. Stresses have been normalised with respect to the stress $\sigma_0$ required to give the same void growth rate under uniaxial stress.

By assuming the mean stress corresponds directly to the hydrostatic stress in a 3-D case the same surface also applies, however the 2-D plane considered may actually lie in any of the x-y, y-z or x-z planes with the overall response being an average of all the possible orientations. A critical orientation will exist that produces maximum void growth and leads to the failure of the material. In this case this maximum growth of damage is determined by the average of the maximum and intermediate principal stresses. FIGURE 16 shows the resulting surface of constant void growth rate. The result, whilst being identical to the 2-D case in the positive quadrant, is significantly different in the second and fourth quadrants.

By removing one of the voids from the uniform distribution it is possible to introduce the effects of
FIGURE 15: CONSTANT VOID GROWTH RATE SURFACE FOR THE UNCONSTRAINED GROWTH OF EQUILIBRIUM VOIDS.
FIGURE 16: TWO POSSIBLE GENERALISATIONS OF FIGURE 15 TO 3-D. THE SOLID LINE ASSUMES THAT HYDROSTATIC STRESS GOVERNS THE GROWTH OF DAMAGE. THE BROKEN LINE ASSUMES THAT THE PLANE OF THE REPEATING UNIT IS ORIENTATED IN THE MOST DAMAGING PLANE WITH RESPECT TO THE REMOTE STRESS FIELD.
constraint into the analysis. Such constraint arises from the presence of an undamaged area in close proximity to the growing void. The lower rate of deformation of the undamaged region will restrict void growth and redistribution of local stresses is expected to occur. The repeating unit analysed is that of FIGURE 14(c) and the following expressions are obtained for remote strain rates and void growth rate.

\[ \dot{\epsilon}_1 = \frac{36 D_{b} \sigma_0}{k T d^3} \left[ \frac{3\sigma^* - \frac{5}{2}\sigma^*}{\left(1-f_h/2\right)^2} \right] - \frac{\sigma^*}{2} \left(1-2f_h\right) \left(\frac{3}{4} \left[ \frac{7}{3} - \frac{5}{2} f_h \right] + \left(1-f_h\right)^3 \right) \]  

\[ \dot{\epsilon}_2 = \frac{12 D_{b} \sigma_0}{k T d^3} \left[ \frac{\sigma^* - \sigma^*}{(1-f_h/2)} \right] - \frac{3(10-2f_h)}{(1-f_h/2)} D_{b} \sigma_0 \left[ \frac{3\sigma^* - \frac{5}{2}\sigma^*}{\left(1-f_h/2\right)^2} \right] - \frac{\sigma^*}{2} \left(1-2f_h\right) \left(\frac{3}{4} \left[ \frac{7}{3} - \frac{5}{2} f_h \right] + \left(1-f_h\right)^3 \right) \]  

\[ \dot{\epsilon}_h = \frac{36 D_{b} \sigma_0}{f_h k T d^3} \left[ \frac{\sigma^* - \sigma^*}{(1-f_h/2)} \right] + \frac{18(1-2f_h)}{(1-f_h/2)f_h} D_{b} \sigma_0 \left[ \frac{3\sigma^* - \frac{5}{2}\sigma^*}{\left(1-f_h/2\right)^2} \right] - \frac{\sigma^*}{2} \left(1-2f_h\right) \left(\frac{3}{4} \left[ \frac{7}{3} - \frac{5}{2} f_h \right] + \left(1-f_h\right)^3 \right) \]  

The introduction of constraint has resulted in more complex expressions for strain and growth rates and these expressions are best examined graphically. An interesting feature of the growth rate expression can be seen in FIGURE 17. When \( f_h \) is small void growth rate is inversely proportional to \( f_h \), but as \( f_h \) increases the growth rate decreases and reaches zero at \( f_h = 0.5 \). Above this value the growth rate becomes negative, i.e. the void would begin to
Figure 17: Variation of void growth rate with void area fraction. The constrained growth of voids subjected to uniaxial stress.
shrink. This can be explained by examining the stress distribution along the three boundaries. FIGURE 18 shows this stress distribution for $f_h = 0.2$ and $\sigma_C = 0$. The net compressive stress on boundary two results in material flowing from this boundary. This combined with material flowing as a result of sliding of boundary two can result in more material being fed into the void than is flowing out along boundary three.

It should be noted that although this analysis predicts that a situation of zero void growth can be obtained, in real materials the nucleation of further voids is likely to occur. Such nucleation would most readily occur in the regions of high stress shown in FIGURE 18 where unloading of stress from boundary 1 to boundary 3 leads to a three fold increase in local stress.

The next stage in the development of damage is the formation of crack-like voids. This starts to occur when the rate of boundary diffusion becomes faster than the rate of surface diffusion. As a result a 'nose' grows along the grain boundary. A repeating unit for a uniform distribution of crack-like voids is shown in FIGURE 14(d). The expression for void growth rate derived is:

$$\dot{f}_h = \frac{54/3}{\sqrt{2}} \frac{D_b \delta_b \rho}{kT \delta_a^3} \frac{[(\sigma_1 - \sigma_2) + (\sigma_1 - \sigma_2)/2]^{3/2}}{(1-f_h)^3 \sigma_a^{1/2}}$$

The two dimensional constant void growth rate surface obtained is shown in FIGURE 19. Here shear stress is
FIGURE 18: DISTRIBUTION OF STRESS ALONG GRAIN BOUNDARIES 1, 2 AND 3 OF THE REPEATING UNIT OF FIGURE 14(c) UNDER A REMOTE UNIAXIAL STRESS WHEN $f_h = 0.2$. 
FIGURE 19: CONSTANT VOID GROWTH RATE SURFACE FOR THE UNCONSTRAINED GROWTH OF CRACK-LIKE VOIDS.
playing a much more important role in determining void growth rate. In three dimensions the shear stress can be interpreted as the von Mises effective stress and the in plane mean stress can be interpreted as the multiaxial mean stress, giving a surface described by:

\[ \frac{2}{\sqrt{3}} \sigma_e^* + \sigma_n^* = \text{constant} \quad (29) \]

and shown in FIGURE 20. Alternatively the two-dimensional plane could be oriented such that \( \sigma_1^* \) is the maximum principal stress and \( \sigma_2^* \) is the minimum principal stress. Again this interpretation is shown on FIGURE 20.

Again by removing one of the crack-like voids a constrained growth situation can be analysed. The repeating unit becomes that in FIGURE 14(e) and the void growth rate is given by:

\[ t_c = \frac{54}{\sqrt{2}} \frac{D_b f_{b_0} \sqrt{(1-e_h)^3/2}}{kT \Delta s_{1/2}^2 \sigma_s^{1/2}} \left[ \frac{11(\delta_1 - \delta_2) + (\delta_1 + \delta_2)}{7 + 4(1-f_h)^3} \right]^{3/2} \quad (30) \]

The void growth rate surface derived from the above equation is shown in FIGURE 21. The same three dimensional interpretations given in the previous section are possible. If the in plane shear stress is interpreted as the von Mises effective stress and the mean stress as the hydrostatic stress then the constant void growth rate surface is given by:
FIGURE 20: TWO POSSIBLE GENERALISATIONS OF FIGURE 19 TO 3-D. THE SOLID LINE IS GIVEN BY EQUATION (29) AND THE BROKEN LINE ASSUMES THAT THE PLANE OF THE REPEATING UNIT IS ORIENTATED IN THE MOST DAMAGING PLANE WITH RESPECT TO THE REMOTE STRESS FIELD.
FIGURE 21: CONSTANT VOID GROWTH RATE SURFACE FOR THE CONSTRAINED GROWTH OF CRACK-LIKE VOIDS.
Alternatively the maximum and minimum principal stress interpretation can be made. Both cases are shown in FIGURE 22 and both give quite similar results.

The final stage in the growth of damage on a single boundary is the formation of a facet size crack. The repeating unit analysed is shown in FIGURE 14(f) and the analysis required is again detailed in APPENDIX A. Crack opening rate is given by the following expression:

\[
\frac{1}{\sqrt{3}} \sigma_{1} + \sigma_{m} = \text{constant}
\]  

(31)

\[
i = \frac{48D_{b} \delta_{A}}{(1-f_{c})^{2}} \left[ \frac{3}{4} \left(1-\frac{3}{8}f_{c}\right) \sigma_{2}^{\gamma} \right.
\]

\[
+ \frac{3}{4} \left(1-\frac{3}{4}f_{c}\right) \frac{1}{(1-8f_{c})} (\sigma_{1}-\sigma_{2})(\sigma_{1}+\sigma_{2}) \left(1-4f_{c}\right)(\sigma_{1}+\sigma_{2})\right]
\]

(32)

The surfaces of constant crack opening rate given by this equation for a range of values of crack area fraction are shown in FIGURE 23. When the crack is small the void growth rate surface is given by:

\[
\frac{-\sigma_{1}}{\sigma_{0}} = 1, \quad \frac{\sigma_{2}}{\sigma_{0}} = 1
\]

(33)

This indicates that growth is dominated by the value of normal stress. As the crack opens the surface flattens out, going through a stage where it is dependent only on \(\sigma_{1}^{\gamma}\). Finally at \(f_{c} = 0.25\) the growth rate becomes zero. This situation is analogous to the situation described for a non-uniform distribution of equilibrium voids described in earlier.
FIGURE 22: TWO POSSIBLE GENERALISATIONS OF FIGURE 21 TO 3-D. THE SOLID LINE IS GIVEN BY EQUATION (31) AND THE BROKEN LINE ASSUMES THAT THE PLANE OF THE REPEATING UNIT IS ORIENTATED IN THE MOST DAMAGING PLANE WITH RESPECT TO THE REMOTE STRESS FIELD.
FIGURE 23: SURFACES OF CONSTANT CRACK OPENING RATE FOR A RANGE OF CRACK AREA FRACTIONS.
Before moving on to consider the computational model that is suited to analysis of the early stages of damage growth the above models can be used to illustrate the effects of constraint on the later stages of damage growth. FIGURE 24 shows the predicted variation of growth rate with area fraction of damage for both equilibrium voids (from equations (24) and (27)) and crack-like voids (from equations (28) and (30)) in uniaxial tension. To plot these curves the value of capillarity stress, being much less than applied stress, has been assumed to be effectively zero. In addition the value of $\sigma_s$ has been assumed to be 1.0 as discussed in appendix 1. From these curves it can be seen that constraint slows the growth of damage over the whole range of void sizes from $\log(f_h) = -3.3$ ($f_h = 0.0005$) up to $\log(f_h) = -0.4$ ($f_h = 0.4$). In addition as the voids become larger damage grows more rapidly in the unconstrained case, whilst in the constrained case the redistribution of stress associated causes growth to slow down as the void size increases.

A direct indication of the practical significance of constrained growth can be seen in FIGURE 25 where the growth rates of voids and crack-like cavities under uniaxial tension are compared for constrained and unconstrained conditions. The same approximations are made as previously, with the addition that a value of the term $\sigma_s$ that appears in the equations for crack-like void growth must be obtained. As defined in appendix 1 this term is proportional to surface free energy divided by grain facet length. For direct comparison of crack-like void growth under constrained and unconstrained conditions it was not
FIGURE 24: VOID GROWTH RATE vs. AREA FRACTION OF DAMAGE FOR EQUILIBRIUM VOIDS AND CRACK-LIKE VOIDS.
UNCONSTRAINED GROWTH OF DAMAGE

CONSTRAINED GROWTH OF DAMAGE

FIGURE 25: DAMAGE GROWTH RATE VS. AREA FRACTION OF DAMAGE
FOR UNCONSTRAINED AND CONSTRAINED CONDITIONS.
necessary to give a value to this term, however for comparison to growth expressions for equilibrium voids a value must be assigned. In most materials the value of surface free energy is close to 1.0 Jm$^{-2}$. For an engineering ceramic a typical grain facet length would be 10 microns. This gives a value of $\sigma_s$ of $1 \times 10^5$ Pa. A typical tensile stress for creep testing is $1 \times 10^8$ Pa. Hence for comparison to equilibrium voids $\sigma_s$ has been set as $1 \times 10^{-3}$ $\sigma_t$.

These graphs give a practical indication of which stage of damage growth will determine the overall rate of damage formation. For unconstrained growth below a value of log ($f_H$) = -1.8 ($f_H = 0.016$) it is the growth of crack-like voids that is rate determining, whilst above this figure the growth of equilibrium voids becomes rate determining. The effect of constraint is to shift this transition point to log($f_H$) = -2.4 ($f_H = 0.004$). As constrained growth more accurately reflects the non-uniform distribution of damage in real ceramic materials it is the predictions of this analysis that are most likely to accurately model experimental data.

4.3 Analysis by computational methods

In parallel to the analytical methods described above, which are capable of analysing simple situations in regular arrays of hexagonal grains, a computational technique was developed by Cocks [43] and later applied to more general situations by Cocks and Searle [44]. Details of the exact techniques used are given in these references, however the results obtained can be summarised here.
As discussed earlier the computation technique is well suited for the description of the early stages of void growth where the separation between voids is very large. To simulate this a large array of hexagonal grains containing a single void was analysed. The situation is shown in FIGURE 26. This microstructure is very different to that typically analysed for metals where the continuum methods used assume that damage occurs over an area much larger than the void spacing and it is not necessary to consider the effects of the bulk of undamaged material on deformation. To identify differences that can arise as a result of such modelling assumptions the growth of the void in FIGURE 26 was simplified in two different ways. In the first case the void is assumed to grow as if the grain structure did not exist, but rather the void was situated in uniform linear visco-elastic material. This would only approximate to reality if the void radius was very much larger than the grain size. A standard solution for this type of growth has been given by Tracey [45]:

\[
\frac{\dot{V}}{V \epsilon_0} = \frac{4 \sigma_0}{(1-f_V) \sigma_0} \]  

[ f_V = void volume fraction ]

A second simplification was made by assuming that the void grows only by grain boundary diffusion along an adjacent boundary with growth being uncoupled to deformation and hence no account of the influence of surrounding material on void growth is included. Such a situation is analogous to a linear array of voids on a single boundary. Void separation is small compared to the length over which
FIGURE 26: THE REPEATING UNIT FOR COMPUTATIONAL ANALYSIS CONTAINING A SINGLE VOID. THE SIDES AB, BC, CD, AND DA ARE ALL AXES OF SYMMETRY.
damage is spread, i.e. this is the continuum approach taken for metals. The situation can be analysed in the same way as the problems described in the previous sections to give a volumetric void growth rate:

\[
\frac{\dot{V}}{V_i} = \frac{0.33\sigma_0/\sigma}{(\lambda/d)^{3}f_v(1-f_v^{1/2})^{3}}
\]  

(35)

[\(\lambda = \text{mean void spacing}, f_v = \text{void volume fraction}\)]

[\(\dot{V} = \text{volume growth rate of a void}, V = \text{volume of a void}\)]

Computational methods were used to analyse the full array shown in FIGURE 26 for a range of values of \(\lambda/d\) and \(r/d\), where \(r\) is the void radius. As with the analytical approach detailed in the previous sections the diffusive growth of voids, the Coble creep of the matrix material and free sliding of grain boundaries are accounted for in the formulation of the analysis. The results obtained for a given set of conditions and under hydrostatic stress are compared to those for the simplified analyses in FIGURE 27. The computational analysis predicts much higher volumetric growth rates than either of the other methods. The shaded areas indicate the variation in growth rate as the size of the arrays analysed increases, i.e. the diffusional distance increases. The very large difference in growth rate between the simple diffusional analysis and the computational analysis can not simply be explained by the larger number of possible diffusion pathways considered in the latter. In fact the distance material diffuses away from the void is
FIGURE 27: PREDICTED VOLUMETRIC GROWTH RATE vs. MEAN STRESS FOR THREE TYPES OF ANALYSIS. THE SHADED BANDS REPRESENT THE RANGE OF PREDICTED GROWTH RATES AS THE MEAN VOID SPACING INCREASES.
smaller in the computation than in the simplified analysis. The difference in growth rates can only be explained by a coupling between diffusional flow of material from the void and accommodation of the resulting deformation by coble creep of the large quantity of surrounding matrix. The coupling mechanisms that occur lead to a relaxation of stress locally around the void with the result that the matrix sees the void as larger than the actual defect.

By addition to the existing program it was possible to calculate the magnitude of these local stresses for a given chain of grain boundaries. Such a stress distribution is shown in FIGURE 28 and comparison is made to the distribution for Coble creep. Relaxation of stress on boundaries close to the void can be seen.

The effect of increasing void size was investigated by increasing the value of r/d in the computational analysis. The slope of the lines shown in FIGURE 27 was used as a comparison tool. An equation to approximate to this slope for the computational analysis was derived such that:

\[ C = \frac{4.6}{(r/d)^{1.36}} \] (36)

where C = the slope of the graph. This parameter is plotted against r/d in FIGURE 29 for r/d up to 0.25 and shows a good agreement with the computation. However as r/d increases this approximation becomes less applicable. When r/d = 0.5, i.e. a whole grain is removed from the analysis the value of C varies from 3.6 to 4.0 over the range of array sizes. By removing further whole grains from the analysis the values
FIGURE 28: VARIATION OF STRESS NORMAL TO THE GRAIN BOUNDARIES ACROSS A CHAIN OF BOUNDARIES COMPARED WITH COBLE CREEP.
of r\d can be further increased. The value of C approaches 4.0 for all arrays on removal of two grains. With any more grains removed it becomes more appropriate to treat the situation as for the growth of a void in a linear viscous material. An analysis for large voids in a fine grain material has been published by Chokshi and Langdon [46]. They model the growth by grain boundary diffusion of a circular void in a superplastic material with a grain size less than five microns and a void size much larger than the grain size. They predict that in this limit growth rate is independent of cavity radius and inversely proportional to grain size. The results of the computer modelling indicate that this conclusion is incorrect as it places too much emphasis on the boundary diffusion processes. In fact for even quite small voids the changes in local stress are confined to a few grain boundary lengths. When the void has reached the size of several grains diffusional processes are very limited in extent and it is deformation of the bulk of the matrix that is predominant.

4.4 Summary of new models

The work described above has served to point out the type of analysis required when examining the deformation of fine grained materials such as engineering ceramics in both the early and advanced stages of the creep failure process. Analytical methods were applied to simple situations and were able to point to a range of possible void and crack growth determining stress states and indicate the importance of incorporating constrained growth into models for ceramic
materials. The void growth rate surfaces produced are a step on the way to full descriptions of isochronous surfaces for failure. As in the case of the work by Tvergaard on metals [28] expressions to incorporate the full failure process could be included. Models for void nucleation rate based on experimental observations may provide a good route for incorporation of this stage of the failure process into an overall model. Similarly the point at which facet sized cracks have grown and linked within the situations analysed could be taken as the point of failure of the bulk material. An example of how this may be done is given in chapter 15.3.

Whilst the situations analysed by the analytical model are simplified and represented ‘snap-shots’ in time of a continuous process it was possible to derive a number of important insights into the stress triaxiality that can determine damage growth. Having said this if one of the processes that have been modelled, for example the growth of the crack-like cavities, is dominant for most of the lifetime of the material, and the initial growth of equilibrium voids and the subsequent growth of facet sized cracks is rapid the models given here should allow the prediction of isochronous failure surfaces directly. Such a simplification could only be verified by comparison with experimental data which as yet is not available in sufficient quantity.

As a step towards analysing more complex situations a computational technique was developed and tested to look at much larger arrays of ceramic grains. This allows situations early on in the creep damage process where the spacing between voids is large to be analysed. Use of this technique
illustrated the way in which the deformation of fine grained materials occurs by a coupling of diffusional and deformation processes. The unloading of stress associated with such a mechanism could be calculated for a chain of grain boundaries close to a cavity. In addition the computational method of analysis was compared to the methods typically used for metals and that for a void growing in a uniform visco-elastic material. The predicted void growth rate from a continuum model was shown to be considerably lower than that for the computation where a full account is taken of the effects of a large void spacing, the effect of the large bulk of matrix material and the resulting widespread stress redistribution. The uniform visco-elastic continuum model was only suitable for describing a ceramic grain structure when void size was of the order of several grain sizes. Models by other authors for such large voids would appear to overestimate the importance of diffusion in such cases as they take no account of the influence of the bulk of the microstructure.

Both of these models illustrate that by taking into account the considerations found to be important when deriving models to describe creep failure of metallic materials, and then introducing suitable adaptations, the more diverse features of ceramic materials can be described. In particular the more complex relationship between void size, diffusion distance and void spacing, and the resulting effects on growth in a ceramic matrix have been considered. These models can be looked at as steps towards the goal of full constitutive descriptions of the growth of damage during creep of ceramic materials, with the ultimate aim of
preparing design rules based on predicted isochronous surfaces and creep damage tolerance.

In part 2 of this thesis the models developed will be further discussed in terms of how they can be verified by practical data.
PART TWO

THE CONSTRUCTION AND OPERATION OF MULTIAXIAL CREEP TESTING EQUIPMENT FOR CERAMIC MATERIALS
CHAPTER 5 : BACKGROUND LITERATURE

Part one of this thesis has dealt with the theoretical aspects of the modelling of creep failure in both metallic and ceramic materials. In this second part the background to the testing of engineering ceramics is introduced by way of literature surveys on the fabrication and resulting microstructural characteristics of the two materials used to fabricate test specimens, the current extent of creep testing on ceramics and on the equipment developed to perform tensile creep tests. This chapter is followed by a detailed report of the construction of the test apparatus built for the combined tension-torsion creep testing of ceramic tubes. The work described in these chapters forms the bulk of the effort undertaken in this project.

At the end of the report results obtained from tests in simple tension, simple torsion and combined tension-torsion are presented as proof of the suitability of the apparatus as a multiaxial creep testing machine.

5.1 Background literature on ceramic materials

Prior to construction of the equipment it was decided to choose a suitable material from which to prepare test specimens. Initially reaction bonded silicon nitride was chosen as the only test material. However towards the end of the work it became clear that further test specimens were needed. The reaction bonded silicon nitride tubes were
supplied free of charge by T&N Ltd, Rugby, England but only in a limited quantity. This meant that if at all possible a second more readily available material should be selected. Alumina was chosen as it could be obtained readily, in this case from Degussa Ltd, Germany, and machined to profile in-house. Reaction bonded silicon nitride was chosen as it is known to have very good high temperature creep resistance, good thermal shock resistance, is relatively cheap and easy to manufacture and shows promise as an engineering material as, for example, hot zone components such as combustion chambers and piston crowns in gas turbine and diesel engines respectively [47]. In contrast alumina is of limited application as an engineering material but has often been used as a typical model polycrystalline ceramic material.

The purpose of the remainder of this section is to provide a brief description of the means used to fabricate both of these materials, and to describe the resulting microstructural features that are formed. As such this section is not an exhaustive literature survey, but rather a representative selection of review articles that best summarise these materials.

5.1.1 Sintered aluminium oxide

Polycrystalline aluminium oxide, commonly known as alumina, is probably the most studied of all ceramic materials and shows all of the features typical of a ceramic. An introductory review is given in [48]. The material may be fabricated by a range of standard ceramic forming techniques. The most basic of these techniques is
the sintering of calcined and milled powder. The addition of sintering aids, for example silicon dioxide, calcium oxide or magnesium oxide is often employed. These additives reduce the required forming temperature but result in amorphous phases being formed at grain boundaries.

Commercial aluminas formed by solid state sintering have a composition in the range 85% - 99.9% aluminium oxide. The higher purity materials such as were used in this work are generally cream\white in colour and opaque as a result of residual porosity from the sintering process.

The features described above are typical of many ceramic materials formed by solid state sintering. The presence of boundary phase glassy material and the pore content are usually the limiting factors in the mechanical properties of the material. Typical commercial aluminas have a room temperature failure strength of 300-400 MPa and an elastic modulus of 400 GPa. Grain size is of the order of 1-20 microns. The material is very hard-wearing and electrically insulating. In terms of application as a high temperature engineering material however, it is very limited in application due to it's high susceptibility to thermal shock and relatively poor creep resistance.

Although a number of other forming techniques are available for the preparation of very small grain size, high density and even transparent alumina, the details given above summarise the type of material used for testing in this work. Material data supplied by the manufacturer and optical and electron micrographs of the samples used in this work are given in APPENDIX B.
5.1.2 Reaction bonded silicon nitride

Reaction bonded silicon nitride is considerably different to alumina in both the forming technique used and the resulting microstructure of the material. A number of comprehensive reviews of the microstructure and properties of the material have been published ([49],[50],[51]). The following is a summary of the main features.

The reaction bonding forming process consists of heating compacts of silicon powder in a controlled nitrogen atmosphere. Two crystalline phases of silicon nitride are formed. Below 1410 °C (the melting point of silicon) fine, fibrous alpha-phase material is formed producing a network of material known as the matte. Above 1410 °C the beta-phase is formed rapidly as large, equi-axed grains. However the process does not yield a fully dense microstructure and a commercial material may have residual porosity in the region of 18-26%. Thus the final microstructure is a three ‘phase’ system of alpha silicon nitride, beta silicon nitride and porosity. Impurities present in the original silicon metal powder may also be present, particularly in the case of iron contamination.

The microstructure described above leads to a moderately low room temperature strength of 200-300 MPa as a result of the high porosity, however the material is of interest for engineering purposes as there is very little drop off in this strength even up to temperatures of 1400 °C. This is also reflected in the good creep resistance of the material.
In addition to the high temperature properties the forming process is relatively cheap and is capable of producing complex shapes with little distortion from the shape of the original compact.

A number of other forming methods, such as solid state sintering or hot pressing have been developed for silicon nitride. These materials have been produced in near theoretical density, however the presence of grain boundary phases leads to significantly reduced strength above 1000 °C. It is for this reason that this work has concentrated largely on the testing of reaction bonded silicon nitride.

Material data supplied by the manufacturer and optical and electron micrographs of the samples used in this work are given in APPENDIX C.

5.2 Background literature on the tensile creep testing of engineering ceramics

Although the creep testing of metallic materials has been in progress since the early part of this century, work on the testing of ceramic materials only began in the mid-1950’s with the work of Kingery and Coble [52] on polycrystalline aluminium oxide. Although the volume of published work has significantly increased, it is noticeable that the type of data obtained is limited largely to that from uniaxial compression or bend tests. These test configurations tend to be easier to set up practically, however from an engineering point of view information on
component behaviour under tensile, torsional and combined stress states are of more practical interest.

Cannon and Langdon [2] provided an extensive review of all ceramic creep testing up to 1981. Examining their review shows that for a total of 253 reported experiments on polycrystalline ceramic materials, 58% of tests were done in compression, 32% in bend, and only 10% in uniaxial tension. None of the authors attempted multiaxial stress state testing.

Looking more closely at those experiments of most relevance to this work i.e. those done in tension, the early work of Davies and Sinha Ray [53] describes basic equipment for tensile testing and determines creep curve shapes for alumina. The earliest work published on the tensile creep of silicon nitrides, either sintered, hot pressed or reaction bonded, was that of Kossowsky [54], [55] where data on the creep of sintered silicon nitride under uniaxial tension was obtained, giving time to failure, strain to failure and steady state creep rate. Kossowsky also observed extensive cavitation and grain boundary separation after creep. The creep behaviour was interpreted as arising from grain boundary sliding enhanced by the low viscosity glassy grain boundary phase, leading to wedge shaped cracks opening at grain junctions which finally coalesce to give failure.

A later study by Arons and Tien [56] again examined this mechanism using data obtained from uniaxial tensile tests on sintered silicon nitride rods. They determined creep activation energy, stress exponent and again proposed a model for creep based on relative grain
motion accommodated by fluid grain boundary glass flow giving cavitation and wedge shaped cracks.

The work of Birch et al. [57] on the creep of both hot pressed and reaction bonded silicon nitrides under tension discusses the observation that creep rates in tension are of the order of ten times those found under compression. The authors account for this difference by noting that micro-crack opening accommodates grain boundary sliding in tension but not in compression.

The work of Govilla [58] - [61] initially examined the creep failure of hot pressed silicon nitride under uniaxial tension [58]. Although later references were all concerned with bend tests, this work points out the importance of atmospheric oxidation in enhancing creep rate and forming initiating sites for failure. As more attention has been drawn to the importance of construction of tensile testing equipment during the last few years a number of recent references have dwelt on this subject. As a result a number of trials tests for their chosen material have been reported. For example Kandil and Dyson [62] report tensile creep data at 1450 °C for RBSN, Carroll and Wiederhorn [63], [64] report tensile and compressive creep data for reaction bonded silicon carbide at 1300 °C and Ohji [65] reports tensile creep data for hot pressed silicon nitride at 1220 °C.

The above references form the bulk of the published work on the tensile testing of alumina, reaction bonded silicon nitride and other ceramics. More precise details of the results obtained by other workers for the creep of both reaction bonded silicon nitride and alumina
are given in chapter 14. Although the bulk of these results are from bend tests they provide a basis for both setting up test conditions and comparing results obtained. No references could be found that deal with tests under multiaxial stress. It is this lack of published work that has encouraged the construction of a suitable test machine, the construction and use of which is described fully in chapters 6 - 13.

5.3 Background literature on creep testing equipment

As has been discussed previously the predominant method for creep testing in the past has involved the use of either four point bend test equipment or compression testing equipment. These test geometries are simple in terms of specimen requirement, being typically beams or solid cylinders, hence test pieces are cheap and easy to fabricate. Loading systems are also relatively simple to construct e.g. ceramic knife edges for bend tests, and the whole loading system can be readily enclosed within a furnace.

In contrast the construction of tensile testing equipment poses three major problems. Firstly ceramic specimens of controlled profile must be machined at reasonable cost to give suitable ends to enable secure, bending moment free gripping. The brittle nature of ceramic materials precludes the use of machined holes and fixing pins as would be used for metallic test pieces. This problem leads to a second major difficulty, that of constructing a suitable gripping system. This problem is compounded by the
need to perform tests at temperatures in excess of 1500 °C, well above the service temperature of metallic grip heads in air. Finally the usual method of strain measurement for metal specimens, involving attaching strain measuring equipment directly to the specimen, is prohibited by the high test temperatures and the need to avoid introducing any flaws to the ceramic surface that may induce brittle or crack growth failure mechanisms.

The above problems have been addressed by a number of authors. The following is a comprehensive summary of the literature.

Although earlier machines had been constructed [66], [67] the earliest equipment of note for tensile testing of ceramics was described by Davies and Sinha Ray [53]. Their machine consisted of a tube furnace heated by platinum windings capable of 1800 °C, through which long rod shaped ceramic specimens with a gauge section of reduced diameter were placed. These rods were gripped outside the furnace using a collet system similar to a self centring drill chuck, and alignment was maintained by universal joints. Bending percentage was quoted as less than 4% of the tensile load. Strain is measured outside the furnace by use of displacement transducers.

A very similar machine was used two years later by Morrell [68] and results reported [69] for the testing of glass tensile specimens at 1000 °C. This machine had advantages over the Davies equipment in that pre-machined specimens were easier to grip, and in that the use of knife-edge universal joints reduced friction in the grip movement.
A different approach to the above was taken by Birch et al. [57] whereby similar tensile specimens were held by molybdenum grips and the whole system is enclosed within the furnace. This type of equipment required that the furnace atmosphere be non-oxidising to protect the metal grips. A value of 4% variation in strain due to off axis loading components was quoted.

Kossowsky et al. [55] also used molybdenum or graphite grips mounted inside a furnace containing an inert atmosphere of helium. Creep strain was measured by recording displacement of the load train outside the furnace and agreement with measured strains to failure on specimens was quoted as always within 10%.

To enable external gripping as close to a furnace as possible Lange et al. [70] employed water cooled steel grips. These grips incorporated powder cushions to minimise bending. Strain measurement was by recording the displacement of alumina rods attached directly to the specimen, but well away from the gauge length. This system was limited to operating temperatures below 1300 °C and stresses below 100 MPa, and strain measurement inaccuracies and bending forces were not quantified.

Arons and Tien [56] employed grips mounted on universal couplings at both ends of the specimen to ensure 'no bending moment' and made of nickel based alloy for heat and oxidation resistance. A split furnace comprising two electrically heated molybdenum disilicide elements was used and at the operating temperature (1260 °C) control was to +/- 2 °C with a specimen gauge section gradient of 2 °C. Strain measurement was via alumina rods spring loaded
against the grip faces, displacements of which were recorded by LVDT. Agreement to within 5% was achieved for the strain measurement.

The equipment used by Govilla [58] had a gripping system based on the use of silicon carbide pins to hold 'dog-bone' shaped specimens with suitable fixing holes machined into the ends. The use of water cooling allowed the grips to be used close to the furnace body even at 1400 °C. Specimens were aligned before test by using strain gauges attached to the specimen surface which were disconnected prior to heating and 'burnt out' at high temperature. This pre-alignment gave below 3% bending at full load.

Work done by Heathwood [71] used an unusual furnace system based on focussed infra red energy from tungsten lamps with furnace ends sealed with ceramic wool to prevent excessive grip heating. A complex laser beam extensometry system was used to record deformation. The system was capable of operating at 1450 °C (lamp life was limited to a maximum of 300 hours) giving a gauge length temperature gradient of 10 °C and strain measurement had a resolution of no better than 0.2 % strain.

The equipment of Kandil and Dyson [62] employs a short length split furnace to allow cold gripping of RBSN specimens and allows a maximum operating temperature of 1500 °C. Bending was reduced by a knife edge system and by arranging axial extensometers in pairs on perpendicular planes actual bending moments could be measured during the test. The system was claimed to operate with less than 2% bending stress without the need for extensive specimen machining for accuracy.
Most recently work done by the Oak Ridge national laboratory (ORNL) and the national institute of standards and technology (NIST) has resulted in more commercial rather than laboratory based systems.

The system of NIST [72] uses flat tensile specimens gripped by silicon carbide pins similar to the system used by Govilla described above, however they have gone to considerable length to eliminate bending stresses by very accurate machining of tapered pin holes. This requires complex and accurate grinding or electrical discharge machining (if the ceramic is electrically conducting), thus making specimen preparation expensive ($65 - $100). They claim less than 1% bending at full load. They also stress the use of a complex laser based extensometry system whereby the position of silicon carbide 'flags' attached to the specimen is monitored through a window cut in the furnace wall. This system is sensitive to variations in air density in and around the furnace, and considerable efforts were made to reduce convection currents that give rise to such variations. This was achieved by improved furnace insulation. The final system had an accuracy of +/- 2 microns.

The ORNL system [73], [74] is based around a self-aligning grip system and has been adapted to either resistance furnaces or RF heating furnace operation. The key feature of the grip system is the use of radially arranged hydraulic pistons that sit around the specimen ends and keep bending forces in balance. To operate this system at high temperature ceramic pull rods are required to connect the grip system to the actual specimen. Bending stresses in this
system are said to be less than 0.5% of the tensile load. The RF heating system used was based on silicon carbide susceptors in two tubular half shells supported by fire brick and fitted within the RF coil. The induction heating system had the advantage of efficiently focussing the heat input to the mid-section of the specimen.

The ORNL grip system has been licensed to the INSTRON company who sell this system complete with a short length resistance furnace and their own loading and strain measurement equipment.

5.4 Summary of existing ceramic tensile creep testing equipment

The above section has detailed the ceramic tensile testing equipment described in the literature. A number of points can be made concerning these systems.

1) Griping systems can be divided into two basic types. Those that sit inside the furnace and require protective atmosphere ('hot' grips) and those that sit outside the furnace and often require water cooling ('cold' grips).

2) Alignment of griping is of great importance. Systems to eliminate bending have taken a number of forms, however in general no system has succeeded in guaranteeing bend stresses much less than 1-2 %.
3) Extensometry of ceramic specimens is made difficult by the requirement that surface flaws are not introduced to the specimen, thus precluding the attachment of strain measuring equipment directly to the sample gauge section surface. Solutions to this problem have either involved remote strain measurement usually based on the displacement of grip heads or load train, or by complex and expensive laser based techniques that require reference marks on the specimen and need to view in to the furnace.

4) Temperature measurement is made difficult by the requirement that temperature probes do not contact the sample surface. Where thermocouples have been used it is the atmosphere temperature within the furnace rather than the specimen temperature that is being measured. This can lead to problems, particularly in knowing the exact nature of temperature profiles that exist both along and outside the specimen gauge length. Non contacting methods of temperature measurement such as I.R. pyrometry avoid the above problem as they measure spot temperatures on the specimen. However the required apparatus must be mounted outside the furnace and thus viewing windows must be cut into the furnace. This results in disturbance to the furnace temperature profile, and optical losses associated with the viewing window itself. Further it is clear that the technique can also very sensitive to thermal currents in the air contained within the furnace chamber. Finally these non-contact temperature measurements are subject to errors resulting from any uncertainty in the specimen surface emissivity. This is even more important where the nature of the specimen surface may
be changing during an experiment as a result of sample oxidation.

These four basic problems of high specimen temperature, bend stress free gripping and accurate strain and temperature measurement are further discussed in the following chapters.
Before commencing the construction of a combined tension-torsion creep testing machine suitable for engineering ceramics the required operating conditions needed to be set out. The following criteria needed to be met:

1) The heating system should be capable of providing a temperature of at least 1500 °C to allow testing of ceramics in the creep range.

2) The heating system should be capable of heating a gauge section of the specimen to uniform temperature at all points along the gauge section. This gauge section should be at least \(2(Rh)^{1/2}\) in length where R is the specimen tube radius and h is the tube thickness.

3) The heating system should function over long periods of time at temperature and over numerous heating and cooling cycles without significant degradation.

4) The temperature measurement and control system should be capable of maintaining an accurately monitored set temperature for time periods in excess of 1000 hours. Accuracy should be at the least comparable to British standards for creep testing of metals e.g. +/- 6 °C at 1000 °C (B.S. 3500).
5) The specimen design should allow for relatively easy fabrication and be profiled to assist in creating a uniform temperature over a central gauge section.

6) The specimen grips should be capable of providing slip free, and bending moment free gripping. To reduce specimen size the grips should be capable of operating in close proximity to the gauge section without suffering thermally induced damage.

7) The specimen loading system should be capable of applying uniform orthogonal loading of suitable magnitude to perform tests in the region of creep behaviour and ensuring no off-axis load components exist.

8) The strain measurement equipment should be capable of precise measurement of displacement in orthogonal axes over periods in excess of 1000 hours. The equipment must also be capable of recording displacement without contact being made with ceramic specimen surfaces.

All of these requirements are discussed in the relevant sections that follow. It is worth noting at this stage that no formal standards have yet been established for the required accuracy of ceramic materials creep testing. As the history of creep testing in metals is considerably longer and testing is more widespread than in ceramics, metal testing standards have been in existence for some time. The requirements set down in British standards for
creep and stress rupture tests on metals are given in the following table.

<table>
<thead>
<tr>
<th>STANDARD NUMBER</th>
<th>PROPERTY</th>
<th>FORCE</th>
<th>% STRAIN</th>
<th>TEMPERATURE</th>
</tr>
</thead>
<tbody>
<tr>
<td>BS 3500</td>
<td>STRESS RUPTURE</td>
<td>1%</td>
<td>NOT DEFINED</td>
<td>+/- 6 DEGREES C (AT 1000 DEGREES)</td>
</tr>
<tr>
<td>PART 1&amp;2 1962</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BS 3500</td>
<td>CREEP</td>
<td>0.50%</td>
<td>2</td>
<td>+/- 6 DEGREES C (AT 1000 DEGREES C)</td>
</tr>
<tr>
<td>PART 3&amp;4 1962</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BS 444</td>
<td>CREEP</td>
<td>0.50%</td>
<td>0.005</td>
<td>+/- 6 DEGREES C (AT 1000 DEGREES C)</td>
</tr>
<tr>
<td>PART 1 1967</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As the temperatures used are higher and the strains measured are lower when ceramics are being tested, it is to be expected that such figures would represent an upper limit for any standards to cover ceramic creep testing. This creates a number of technical problems which are discussed in depth in the following chapters.

At the end of the project a successful machine had been constructed that could be used for routine creep testing. Photographs in FIGURE 30 were taken during a combined tension\ torsion test and show all the features of the test rig. The figures that follow on are schematic drawings indicating the scale, layout and details of the equipment. In the chapters that follow details of the exact construction of this rig are given and these figures will be referred to.
FIGURE 30: TENSION/TORSION CREEP TEST EQUIPMENT

a) LOADING FRAME, FURNACE, GRIPS AND RF SET

b) CLOSE UP OF a) SHOWING FURNACE, GRIPS AND EXTENSOMETER
FIGURE 30c: TENSION/TORSION CREEP TEST EQUIPMENT
FRONT VIEW (NOT TO SCALE)
FIGURE 30d
TENSION/TORSION CREEP TEST EQUIPMENT
SIDE VIEW (GRIPS AND FURNACE AREA NOT SHOWN)
(NOT TO SCALE)
FIGURE 30e: DETAIL OF FURNACE, GRIP AND EXTENSOMETER AREA
An initial basic design was chosen for all test pieces. The constraints imposed by manufacturers of reaction bonded silicon nitride, silicon carbide and alumina resulted in this design being based around a tubular specimen approximately 150 mm long x 20 mm ID x 25 mm OD. These dimensions give a diameter to thickness ratio of 16:1 as recommended by Rees [75]. To allow testing up to 100 MPa in tension or shear a central gauge section of 1.5 mm wall thickness is required. The transition region between this central gauge length and the gripped tube ends was the most difficult area to define for the final specimen design. The profile and position of this region should be defined in order to provide negligible stress concentration due to both mechanical and thermal loading, while ensuring that the stress remains uniform, with negligible thermal stresses, over at least 10 mm of the central gauge length. In order to get some basic guidelines as to the relationship between temperature profile along the tube length, transition region position and thermal stresses a simple finite element analysis program was used. This program was the commercially available MSHELL package from Engineering Software Co Ltd which can be run on IBM compatible PCs. MSHELL is capable of analysing a range of thermal and mechanical loadings applied to axi-symmetric structures. However the data input system used in the package is limited to defining only linear changes in temperature and sample shape.

The analysis was performed on a tube structure with a nominal length of 200 mm, as at this stage of the
work the constraints on the final tube length had not been provided for us by the manufacturer. Tube thickness was set at 25 mm at the ends and a reduced centre gauge section of 50 mm length was created by a linear reduction in diameter to 23 mm over a length of 25 mm between 50 mm and 75 mm from either end of the tube. The tube was constrained such that rotational and radial displacements were zero at the tube ends and the only loading applied was as a result of defined thermal gradients along the tube outer surface. Data for thermal and mechanical properties were those of the actual experimental reaction bonded silicon nitride specimens as detailed in appendix C.

A series of analyses were performed using the single set of fixed tube dimensions given above with the aim of determining the interaction between thermal gradients and dimensional changes. Thermal gradients were defined such that the temperature rose from 20 °C at the tube end to a peak temperature of 1400 °C at a defined point along the tube length and remained at the peak temperature over a defined central region. Initially the data input system meant that it was only possible to look at linear temperature gradients. However it was found possible to access and directly edit the temperature data input file generated by the program. In this way it was possible by a lengthy manual process to input smoothly varying temperature profiles which would be closer to the actual conditions likely to be experienced under thermal loading. No attempt was made at this stage to accurately define the shape of a temperature profile likely to be seen under test conditions. Instead for smooth profiles a simple curve was generated.
such that temperature, $T$, at a point along the tube length, $R$, was given by $T = M + cR^2$ where $M$ and $c$ are constants that define the magnitude and length over which the temperature rise occurs.

The results obtained from both linear and smooth temperature profiles are given in the following table.

<table>
<thead>
<tr>
<th>TEMPERATURE PROFILE TYPE</th>
<th>PEAK TEMPERATURE POSITION **</th>
<th>POSITION ** OF PEAK HOOP STRESS</th>
<th>MAGNITUDE OF PEAK HOOP STRESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>LINEAR</td>
<td>100</td>
<td>100</td>
<td>80 MPa</td>
</tr>
<tr>
<td>LINEAR</td>
<td>85</td>
<td>85</td>
<td>45 MPa</td>
</tr>
<tr>
<td>LINEAR</td>
<td>75</td>
<td>75</td>
<td>30 MPa</td>
</tr>
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<td>70 MPa</td>
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<tr>
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<tr>
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<td>18 MPa</td>
</tr>
<tr>
<td>QUADRATIC</td>
<td>75</td>
<td>18</td>
<td>12 MPa</td>
</tr>
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</table>

** RELATIVE DISTANCE ALONG TUBE : TUBE END = 0 , TUBE CENTRE = 100

THERMAL LOADING ONLY.

ROTATION AND AXIAL DISPLACEMENT CONSTRAINED AT TUBE ENDS.

LINEAR REDUCTION IN TUBE DIAMETER FROM 25 mm TO 23 mm BETWEEN POSITION 50 AND 75.

These results showed that very high hoop stresses could be generated by the sharp changes in thermal gradient that occur when a linearly increasing temperature changes to a constant temperature. These stresses were even larger if the change in thermal gradient coincided with a sharp change in the tube profile. The magnitude of the stresses generated are significantly high and of the order of the likely applied creep test stresses (50 - 100 MPa). However when smoothly changing temperature gradients were analysed no significant peaks in hoop stress were observed over the central gauge section or in the transition region where diameter is changing. In these cases peak stresses lay very
close to the tube ends where displacement is constrained. In addition the magnitude of these stresses was much less, being only of the order of 1 MPa or less in the central and transition regions.

The results obtained from this simple analysis were useful in the initial design of both the specimen and the furnace. It was clearly indicated that sharp changes in temperature gradient should be avoided if at all possible. In addition if sharp changes in temperature are likely, such as when the tube passes out of the furnace and into the grip system, any changes in tube profile should occur away from this section. At the same time, from a purely mechanical loading point of view it was desirable to make the transition region as long as possible to minimize the stress concentration resulting.

Finally before fixing the gauge section and transition region the length of tube fixed in the grip heads needed to be known. This was set at 25 mm in order to give a reasonable length of tube over which the gripping forces could act, leaving 50 mm to the tube centre.

With all these criteria and limitations in mind the final design of specimen tube chosen was that shown in FIGURE 31. This incorporates a central section of 23 mm o.d. and 15 mm length. The transition regions extend up to points 50 mm from the tube ends and are smooth curves of radius 75 mm. This was as long a length of transition as was possible to comply with the requirement that the regions should end within the area of fairly constant temperature in the furnace. Of the remaining 50 mm length of 25 mm o.d. tube at each end, 25 mm sits within the grip head, 19 mm
FIGURE 31: SPECIMEN DESIGN SHOWING DRAWING AND PHOTOGRAPH OF AN EARLY PROFILED TUBE IN SILICON CARBIDE
sits within the region of insulation and cooling plates where temperatures decrease sharply and 6 mm sits within the central area of the furnace.

An initial study to determine the practicality of producing this profile was performed on tubes of reaction bonded silicon nitride, purchased from Morgan Materials, and alumina, supplied by Degussa. The as supplied tubes were of varying dimensions and ovality and further machining operations needed to be performed in-house to form the gauge section and ensure concentricity of inner and outer surfaces so that the tubes did not fracture on gripping.

The first stage of producing a specimen involved internal and external grinding of one of the as received tubes. The bore was honed to size using diamond impregnated shoes. The size of the bore was accurately controlled to 20-30 microns and the surface finish was better than 5 microns. The bore was then used to locate the specimen on a mandrill and the external surface profile ground over the gauge length and parallel ground at the grip ends using diamond impregnated wheels. The specimen was polished over the gauge length to achieve a surface finish of better than 1 micron and inspected using a Talysurf.

In-house manufacture of the specimens by the above method was highly time consuming. In addition machining of ceramic materials can introduce unwanted damage into the component. For the final series of tests T&N Technology produced a number of samples of reaction bonded silicon nitride which were machined to specification in the green state. Since these materials remain dimensionally stable during firing no further machining of the specimens is
required. During nitriding the alpha phase can form preferentially on the surface, which might affect the fracture strength of the material. Examination of sectioned samples under the SEM, however, showed little or no difference between the structure of the material in the bulk of the specimen and that near the surface. Machining of the specimens could potentially introduce more damage in the material and have a more drastic effect on the failure characteristics of the material. It was therefore decided to test the specimens in the as received state. Samples of alumina could, however, only be obtained in the form of sintered tubes, thus requiring the machining described to produce the finished components.

The suitability of the final specimen design was partly confirmed by the observation that all failures occurring during the testing program occurred very close to the centre of the gauge section, thus indicating that the design successfully concentrated peak stresses over that area.
CHAPTER 8: FURNACE EQUIPMENT

8.1 Radio frequency system

One of the novel aspects of the approach taken in this work was the use of a radio-frequency (RF) induction heating system. Such a system allows for flexible, 'custom built' furnace designs to be produced that are capable of rapid heating to very high temperatures provided suitable susceptor materials are used (neither reaction bonded silicon nitride nor alumina are capable of self heating via induction of RF).

The unit used for this project consisted of a Solid State Induction Ltd. 30 kHz generator providing up to 8 kW of power feeding into a two or three turn water cooled copper coil. The coil used for the bulk of the testing had 3 turns and an overall length of 50 mm, a bore of 70 mm and the tube was of 8 mm square section.

The generator is of a reasonably low frequency rating for an RF set and was chosen to eliminate the 'skin' only heating effect that occurs in higher frequency systems. The only alterations made to the set as received from the manufacturers was to tune down the minimum operating power (done by service engineers) to accommodate efficient operation at the low power requirements of the final furnace design. A standard Eurotherm controller was used to regulate power supplied from the generator in order to maintain constant specimen temperatures, and this control was capable of being switched between either thermocouple or pyrometer based measurement.
8.2 Susceptor materials

As the ceramic test materials themselves do not inductively heat by RF to any great degree, it was necessary to find a suitable material that would heat sufficiently to produce specimen temperatures in excess of 1500 °C by radiative heat transfer. This implied susceptor temperatures possibly well in excess of 1500 °C, depending on the efficiency of the furnace design. The material to be used must also be capable of withstanding long periods of time at temperature and be capable of sustained use through a large number of heating and cooling cycles.

Early trials using silicon carbide rod susceptors showed that at the low operating frequency of the RF set it was not possible to heat this material to more than a few tens of degrees above ambient even at full power.

Possibly the cheapest susceptor material would have been graphite. This is available in a large range of densities (1.7–2.2 Mg m⁻³) and purities and is easily machined and relatively strong. However oxidation occurs at temperatures above 400 °C and prolonged survival at temperatures in excess of 1000 °C is only possible in vacuum. With high density and low porosity graphites highly efficient susception could be obtained with oxidation rates of less than 0.25% per hour at 1200 °C (e.g. 1346 extra-fine grain structure grade produced by Le Carbonne) however for the long term very high temperature performance required by the creep test equipment pure graphite materials had to be rejected.
One possible way to progress with the use of graphite was via protecting the material surface from oxidation. Two protected graphite materials were investigated. Firstly, siliconised susceptors of graphite were tested. A further reduction in oxidation rate was observed, but at a lower efficiency due to the loss of graphite volume and the surface layer of non-suscepting silicon carbide. Secondly, protection can be afforded by coating the surface of the graphite with a non-oxidising material such as alumina or zirconia. Samples of Metco 115 super-fine-spray alumina coated graphite were investigated. The coating porosity of this material was of the order of 1%. It was found that with such a level of porosity significant weight loss from oxidation was still measured. Eventually this resulted in a ceramic outer shell with a reduced volume susceptor contained within. Cracking of the coating was also observed at sectional transitions even though a radius was intentionally generated to reduce stresses.

Having largely eliminated graphite materials other susceptor materials had to be investigated. The very high temperatures required eliminated all known ferrous and non-ferrous metals. However metal alloys have been developed with some protection against oxidation at very high temperature. Such a material, Incoloy MA956, was located. This alloy develops it’s own surface oxide layer that acts as an effective barrier to further oxidation. This alloy is basically a single-phase material of 74% iron which suscepts very efficiently and it was possible to run a susceptor of this material at temperatures of up to 1400 °C, however
sudden meltdown was found to occur above 1450 °C exposing unprotected material. Whilst being promising for applications below around 1200 °C, again this material had to be rejected.

The material finally used to fabricate susceptors was molybdenum disilicide ($\text{Mo}\text{Si}_2$). This is manufactured commercially and sold as a series of ‘Kanthal’ grades. This material is conventionally used to manufacture heating elements for electrical resistance furnaces and is fabricated into pre-shaped rod suitable for installation into furnaces. By the use of additives capable of creating an inert protective surface layer of oxide, this material can be used in oxidising atmospheres for prolonged periods of time at temperatures in excess of 1800 °C. It was found that in the RF field generated by the set being used, this material could be heated to temperatures in excess of 1500 °C in a few minutes. Rods of a few millimeters thickness could be subjected to a large number of rapid heating and cooling cycles without suffering thermal shock failure.

The molybdenum disilicide material met the susceptor requirements in all respects and was used as the heating element in all designs of furnace described in the following sections.
CHAPTER 9: FURNACE DESIGN

Having established a suitable susceptor material to act as a heat source, it was necessary to incorporate this material into a furnace in such a way as to meet the heating requirements of the test equipment. This process involved a number of practical design iterations and some theoretical heat distribution modelling of the final design. As the ceramic specimens to be used were tubular in shape the possibility of heating from within and externally to the tube existed. Designs for both systems are discussed in the following sections.

9.1 Internal susceptor design.

The components of the final design of internal susceptor are shown in the photograph in FIGURE 32. Initial supplies of molybdenum disilicide material were in the form of rod sections cut from long resistance furnace heating elements. Pieces were available in both 10 mm and 5 mm diameters and the larger size was well suited to use as an internal susceptor. By use of mounting holes (machined centrally by spark erosion) and silicon carbide pins, it was possible to mount the 10 mm diameter rods inside the ceramic specimen tubes. To position the susceptor either centrally along the specimen length, or in any position off centre with respect to the gauge section, ceramic fibre board plugs were machined to fit the specimen tube ends. These plugs also served as highly effective thermal insulation keeping down power requirements. The exact composition of the fibre
FIGURE 32: PHOTOGRAPH AND SKETCH OF THE FINAL INTERNAL SUSCEPTOR ASSEMBLY SHOWING ROD SHAPED CENTRAL SUSCEPTOR, CERAMIC FIBRE BOARD INSULATION PLUGS AND SILICON CARBIDE LOCATING PINS
material was not known but was likely to be alumina based. As the effective sintering temperature of alumina is close to 1500 °C it was not surprising that the fibre plugs became powdery and lost strength after prolonged service at high temperature. The need to replace these plugs after a few heat/cool cycles was a draw-back in the design, however other more expensive materials tried, such as zirconia fibre board, showed a similar reduction in strength at high temperature.

The above practical design process provided a serviceable internal susceptor system provided the end plugs were considered as a consumable and replaced often. The experience gained in this part of the design pointed out the continual problem of obtaining thermal insulation materials capable of withstanding temperatures in excess of 1500 °C and still retaining mechanical strength. No entirely suitable materials were found for this purpose.

9.2 External susceptor design

The design of an external heating system followed two independent routes. The two designs differed as a result of the nature of the molybdenum disilicide material available. Initially the same rod shaped material as used for the internal susceptor was the only type available and a cradle design was produced to hold these susceptors. Later a tubular form of the material was obtained and a compact furnace could be constructed around the central element. This second design was favoured and all later work was carried out with this unit.
Briefly, the cradle design consisted of zirconia fibre board end caps with holes machined to retain the rod shaped susceptors. As early versions of this design suffered from thermal stress cracks at the holes, the rod ends were lined with zirconia felt and relocated within the holes. This allowed the full cradle of 10 susceptors to operate. A photograph of the assembled cradle is shown in FIGURE 33. After initial trials it was found that this unit was difficult to assemble, suffered from degradation of the zirconia felt and needed more insulation to produce sufficiently high sample temperatures. This design was abandoned in favour of the tubular susceptor design.

Samples of tubular molybdenum disilicide of 50 mm length and either 36 mm or 48 mm o.d. were used to construct the furnace. This material was fabricated for us by T&N Technology, Rugby. Prior to use a sight hole for pyrometer measurement had to be machined in the tube wall. This was achieved by spark erosion machining.

In order to support the susceptor and to protect the insulation material from direct contact with the high temperatures of the susceptor, end plates were machined from siliconised silicon carbide. This material was found to be the only one capable of withstanding direct contact with hot susceptors over a large number of heating and cooling cycles without suffering thermal shock fracture or undergoing substantial reaction with the molybdenum disilicide.

The susceptor and end plate assembly was enclosed in a machined insulation housing made from alumina fibre board and capable of withstanding temperatures up to 1700 °C. To allow removal of the susceptor assembly this housing
FIGURE 33: CRADLE DESIGN OF EXTERNAL SUSCEPTOR
consisted of a split shell with one half further divided into two pieces. One of these pieces contained a machined hole into which a sapphire window and alumina tube were located to act as a sight hole for the pyrometer. On assembly the susceptor and ceramic shell holes were carefully lined to provide a continuous line of sight.

After a number of runs the insulation material became quite brittle and showed several fractures, however a more suitable material could not be located and the fractured shells could still be quite easily assembled to house the susceptor.

The complete insulation housing was retained within a large tube made from an easily machinable ceramic (pyrophyllite) to provide a solid furnace body, again with a suitably placed sight hole machined and aligned with the line of sight. To allow for differential thermal expansions a layer of zirconia felt was incorporated between the fibre board shells and the outer casing. Small aluminium tabs were located at the bottom edge of the outer casing and positioned to support the inner section of the furnace. Despite the proximity to the RF field when in operation no excessive heating of these tabs was detected.

To complete the furnace assembly a lip was machined at the top of the outer casing to allow the whole furnace assembly to rest on the top of the three coils of the RF generator. The coil had been previously cast in Durapot 809, a high temperature polymer to provide a level cylindrical holder for the furnace. The assembly was carefully adjusted to ensure the furnace lay central with respect to the centre axis of the load train.
A sketch drawing of the final construction of the furnace is given in FIGURE 34a. In addition a photograph of the disassembled parts of the furnace is shown in FIGURE 34b. This furnace was used for all the tests done with the equipment. It was capable of generating specimen temperatures of at least 1400 °C whilst using less than one quarter of the available generator power. It is relatively small in size and has only a few simple components. With the possible exception of the insulation board, the components showed no sign of major degradation during repeated and extended operation. As such this furnace design represents an important part of the equipment developed during this work.

9.3 Modelling of furnace designs

The final design of furnace has the option of including both an internal and an external susceptor, and in addition there is the possibility of adjusting the shape of either or both of these susceptors. To gain an understanding of how the arrangement of susceptors was likely to influence temperature gradients obtained along the gauge section of the specimen tube it was decided that a finite element analysis of the whole furnace construction should be attempted. The finite element package used for this modelling was the commercially available ABAQUS program (Hebbitt, Karlsson and Sorensen Ltd, Providence R.I. USA).

To model the whole furnace a mesh was generated that matched the shape and dimensions of the actual furnace and with the addition of a layer of air 200 mm thick. This
FIGURE 34a: SKETCH DRAWING OF FURNACE DESIGN
FIGURE 34b: COMPONENTS OF THE FINAL DESIGN OF FURNACE SHOWING SILICON CARBIDE END PLATES (1), MOLYBDENUM DISILICIDE SUSCEPTOR (2), SAPPHIRE WINDOW (3), RETAINING RING (4), FURNACE BODY (5) AND UPPER HALF OF THE INSULATION SHELL (6)
layer allowed the temperature of the remote atmosphere to be specified as a boundary condition. The outline of this mesh is shown in FIGURE 35. Nodes were placed at regular intervals along the lines indicated and quadrilateral elements were generated. These elements were of a type specifically used by the program for the analysis of thermal diffusion problems (type DCAX4). It was also necessary to specify the thermal conductivity, heat capacity and density of the materials used in the furnace construction. The material modelled was reaction bonded silicon nitride and the figures supplied by the manufacturer, as listed in APPENDIX C, were used. For the susceptor and the insulation material figures were those supplied by the manufacturers. For the commercial KANTHAL 1800 grade susceptor material density was given as 5.6 g/cm\(^3\), thermal conductivity as 15.0 W/mK and specific heat as 0.42 J/g per °C. For the insulation material density was 0.7 g/cm\(^3\), thermal conductivity was 0.4 W/mK and specific heat 0.9 J/g per °C. For the air surrounding the furnace data reference values were used, these being a density of 0.001 g/cm\(^3\), a thermal conductivity of 0.94 W/mK and a specific heat of 0.73 J/g per °C.

To look at the influence of susceptor position and shape three different basic susceptor shapes were studied for both the external and internal pieces. In the first case a simple tubular external susceptor of wall thickness 5mm and a rod shaped internal susceptor of diameter 10 mm were examined. The other two cases involved stepped designs of susceptor. In the first of these the diameter of the internal susceptor and the wall thickness of the external
FIGURE 35: MESH USED FOR ABAQUS FINITE ELEMENT ANALYSIS
OF FURNACE DESIGN SHOWING NODE LOCATIONS (N) AND
ELEMENT LOCATIONS (E). DIMENSIONS MATCH THOSE
GIVEN IN FIGURE 34a. 200 mm OF SURROUNDING AIR
IS ALSO INCLUDED IN ORDER TO DEFINE REMOTE
ATMOSPHERE TEMPERATURE.
susceptor are reduced by a factor of two. This reduction in dimension occurred linearly over 10 mm of length starting from points 10 mm from the top and bottom of the susceptors. In the final case a similar reduction in dimension is made but this time to values of one tenth of the original. These cases were examined over a range of combinations of internal and external shape and surface temperatures. As no direct measurements could be made of susceptor surface temperature profiles the assumption was made that surface temperature was constant throughout the susceptors. Whilst undoubtedly this is not the case in practice the simplification allows the intended examination of trends in behaviour. Details of the analyses performed and the temperature differences predicted between the centre point of the tube and a point +/- 10 mm from the centre (20 mm gauge length) and a point +/- 20 mm from the centre (40 mm gauge length) are given in TABLE 1. The factor of two reduction in dimension is denoted as the STEP (SMALL) type of susceptor and the factor of 10 reduction as the STEP (LARGE) type.

These results show that for a single external susceptor at 1400 °C a temperature difference of 29 °C is predicted over the central 20 mm, i.e. points plus and minus 10 mm away from the centre point are 29 °C cooler than the centre point. This value drops to 12 °C with the introduction of an internal susceptor at 1400 °C indicating that an internal susceptor can significantly improve the temperature profile in the gauge section. The temperature difference reduces slightly over a 20 mm gauge length and significantly over a 40 mm gauge length when the temperature of the inner susceptor is lowered. In practical terms
### Table 1: Results of the Finite Element Analysis of the Effect of Susceptor Position and Shape on Gauge Temperature Profiles

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<th>External Susceptor Type</th>
<th>Internal Susceptor Type</th>
<th>Temperature Variation (20 mm Gauge Length) (DEGREES C)</th>
<th>Temperature Variation (40 mm Gauge Length) (DEGREES C)</th>
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<td>1300</td>
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<td>SMOOTH</td>
<td>SMOOTH</td>
<td>1980</td>
<td>81</td>
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<tr>
<td>SMOOTH</td>
<td>SMOOTH</td>
<td>2000</td>
<td>81</td>
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</tbody>
</table>
however it is unlikely that any control over the inner
susceptor temperature could be achieved independently of the
external susceptor, both temperatures being effectively
determined by the extent of RF heating.

The introduction of step profiles to both external
and internal susceptors generally reduces the magnitude of
the temperature differences further. Effectively heating is
being directed towards the points further from the tube
centre, although this always remains the hottest point. The
full range of results are again given in TABLE 1. The
smallest temperature difference is recorded when both
susceptors have a large reduction in diameter in the centre
and both are at 1400 °C, this temperature difference being 7
°C over the 20 mm gauge length.

It was clear from these results that definite
improvements in gauge section temperature profile could be
achieved by combining internal and external susceptors. In
addition by profiling these susceptors the temperature
differences could be further improved.

It was possible to check these predictions against
actual temperature measurements. This work is detailed in
chapter 10.2 and studies the introduction of an internal
susceptor. From a practical point of view the lengthy
machining required and the limited supply of susceptor
material meant that the option of profiled internal and
external susceptors could not be investigated. This remains
as an option that can be considered when refining the test
equipment.
Before commencing creep tests it was necessary to be able to establish accurate control of the temperatures produced on the specimen surface, and to understand the actual temperature distributions over the length of the specimen. The work described in the following sections covers the control system and temperature measurements made.

10.1 Temperature control

At the outset of the project it was assumed that the presence of radio-frequency fields in the hot zone of the furnace precluded the use of thermocouple measurement as a means of temperature control. This was confirmed by experiments on a higher frequency machine performed prior to the beginning of this work. As a result a system based on an Ircon radiation pyrometer was devised. This type of temperature measurement requires no direct contact with the specimen surface as it is based on recording the level of thermal radiation emitted from the material. However it is difficult to calibrate such a system against standards. In addition the nature of the material surface can effect the temperature recorded by variations in it’s emissivity. This second problem was not serious as in the essentially closed conditions of the furnace chamber it is safe to assume that black body conditions are created and an emissivity of 1.0 was chosen for all materials. As confirmation of this assumption practical tests were made. Two pyrometer heads were used, one focused on a specimen surface and another
focused through a hole cut in the specimen onto the back wall of the furnace. Good agreement was obtained between the two pyrometers at 1400 °C indicating that the environment is indeed at the same temperature as the sample. However the problem of knowing the absolute accuracy of the temperature recorded by the pyrometer remained. The problems involved in getting a calibrated thermocouple into a suitable position in the surface of a material whereby the pyrometer and thermocouple readings could be compared in a valid way were great. In most situations the thermocouple will only give an average temperature of the material and the surroundings unless it is placed exactly on the surface. In order to directly measure tube temperature profiles such a system based on embedded thermocouples was constructed. This is described in the following section and the results obtained, although far from perfect and not amounting to a full calibration, do give some indication as to the accuracy of the pyrometer.

When setting up the equipment it was essential that a clear line of sight was established to allow the pyrometer to view the specimen surface. Thus the components of the furnace all had holes cut to allow pyrometry. This would normally allow heat loss and thermal air currents to build up around the hole in the furnace, resulting in a cold spot on the specimen. Thus it was necessary to incorporate a window into the sight line to eliminate thermal currents. The material chosen was single crystal sapphire in the form of a 2 mm thick, 10 mm diameter disc. The arrangement of the sight hole, window and supporting ring (to fix the window in position) is shown in the detail included in FIGURE 34a. The
sapphire material was chosen for the window because it has a high transmittance in the region of pyrometer operation [76], being around 97%. The quartz window originally used was said by Ircor to produce up to 6.5% error in temperature reading. However it must be recognised that sapphire, despite being the best material we could obtain for the window will cause problems in that the small absorbance will result in errors in the actual temperature reading. The work reported later on thermocouple measurement (chapter 10.2) will discuss this discrepancy further.

The temperature signal from the pyrometer was fed to a Eurotherm controller unit which provided a continuous digital read-out of temperature and gave output to act as a control signal to the RF generator. The generator used this signal to adjust power output thus maintaining a constant specimen temperature. This system was capable of controlling the specimen surface temperature to an accuracy of +/− 2 °C or better at 1400 °C over periods in excess of 100 hours.

10.2 Temperature mapping

To establish the accuracy of the equipment it was necessary to characterise the temperature distribution along the specimen gauge length. This serves the dual objective of obtaining a practical measurement of the gauge section temperature profile and giving an indication of how the temperature measured by pyrometer compares to measurements made by calibrated thermocouples. To fulfil the first objective the use of a pyrometer to scan the length of the gauge section was originally considered. However this would
require the cutting of a large slot in the furnace wall, inner shell and susceptor, thus creating serious disturbances to the temperature profile. To fulfil both objectives it was necessary to find another temperature measurement method. It was found that the use of very fine wire (0.2 mm wire diameter, 0.5 mm bead size) Pt/Rh thermocouples was possible even within the RF field. This was determined by placing the thermocouple within the furnace and establishing that a steady reading could be obtained, then briefly switching off the RF field and looking for drastic changes in recorded temperature. No change could be observed in this experiment and all further measurements were made with these thermocouples. The satisfactory performance of these thermocouples may be due to one of the following factors, or a combination of them. The very fine bead size may limit effective induction, the low frequency of the RF field may not be suitable to couple well to the Pt/Rh beads and the field may be very efficiently absorbed by the susceptor material, thus effectively shielding the thermocouple.

As the Pt/Rh thermocouples were said to be reactive with silicon nitride, all temperature profile measurements were made using alumina tubes. The tubes were profiled to the standard gauge section then thin grooves were cut along the length of the gauge section using a 1mm diameter diamond impregnated saw blade to approximately half the depth of the gauge section. Thermocouples contained within alumina sheaths were embedded into these grooves such that the beads were positioned at the centre of the gauge section and at +/- 5mm and +/- 10 mm away from the centre.
The sheaths and the beads were then cemented into place using an alumina ceramic cement to leave a smooth surface flush with the gauge section. Photographs of the assembled tube are shown in FIGURE 36a.

The completed tube was mounted in the grips as for a standard test. This involved the machining of a special mounting plate to hold thermocouple connections (shown in FIGURE 36b). All visible gaps were initially sealed from the outside with either insulating felt or loose insulation fibre. The final assembly is also shown in FIGURE 36b.

Four tests were performed to determine temperature profiles using various arrangements of susceptors and insulation. The results obtained are given in TABLE 2.

Test 1 had an external susceptor, an internal insulation plug instead of a susceptor and limited external insulation around visible external gaps. Test 2 was as for test 1 with the addition of insulation on the top and bottom of the furnace and in the gaps between the cooling plate and furnace. In addition the insulation was more tightly packed such that all free space was completely filled with either felt or loose fibre material. Test 3 was as test 2 but with the tube rotated 180 degrees within the furnace. This test was to determine if there was any radial temperature distribution caused by heat loss through the pyrometer sight hole. Test 4 was as test 2 with the addition of an internal susceptor fitted in the manner shown in the previous FIGURE 32.

A considerable drop in temperature variation was observed when the insulation of the furnace area was
FIGURE 36a: ALUMINA TUBE WITH FINE WIRE THERMOCOUPLES CEMENTED IN PLACE ALONG THE GAUGE SECTION
FIGURE 36b: FINAL ASSEMBLY FOR TEMPERATURE PROFILE TESTS INCORPORATING HOLDING PLATE (1) AND FULL CERAMIC FIBRE INSULATION (2)
### Table 2: Results of the Experimental Determination of Gauge Section Temperatures Obtained Using the Equipment Shown in Figures 36a and 36b

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Conditions</th>
<th>Recorded Profile (°C)</th>
<th>Temperature Variation (±5 mm)</th>
<th>Temperature Variation (±10 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>External Susceptor, Internal Insulation Plug, Limited External Insulation.</td>
<td>1424.6, 1453.5, 1471.8, 1474.2, 1457.3</td>
<td>20.7 Degrees</td>
<td>49.6 Degrees</td>
</tr>
<tr>
<td>2</td>
<td>External Susceptor, Internal Insulation Plug, Additional External Insulation.</td>
<td>1470.4, 1480.9, 1484.6, 1479.7, 1453.2</td>
<td>4.9 Degrees C</td>
<td>31.4 Degrees C</td>
</tr>
<tr>
<td>3</td>
<td>As Test 2 but with tube rotated 180 degrees so that thermocouples were next to sight hole</td>
<td>1481.8, 1486.1, 1490.9, 1485.1, 1455.1</td>
<td>5.8 Degrees C</td>
<td>35.8 Degrees C</td>
</tr>
<tr>
<td>4</td>
<td>As in Test 2 but with an internal susceptor</td>
<td>1477.6, 1494.4, 1497.3, 1496.3, 1470.1</td>
<td>2.9 Degrees C</td>
<td>27.2 Degrees C</td>
</tr>
</tbody>
</table>
improved and this variation was improved further when a combination of external and internal susceptors was used.

The results obtained confirm the predictions made by finite element analysis, i.e. that the presence of an internal susceptor is advantageous in reducing temperature variation. This provides good evidence that the predicted additional improvements in temperature variation gained by altering susceptor profile would be worth considering in future practical improvements to the equipment.

Test 3 seemed to indicated that a small increase in temperature variation occurs on the sight hole side of the tube. However it is noticeable that the peak temperature for this test is higher (by 6 degrees) than for the test with the thermocouples furthest away from the sight hole, i.e. the expected loss of heat through the sight hole did not occur. As these were the only two tests that could be performed it is not clear whether the variation represents random experimental error or if they represent a genuine effect. However the magnitude of the effect is quite small and for the purposes of the initial use of the furnace these temperature differences are not significant.

A significant difference was noted in all tests between the magnitude of the pyrometer reading and that recorded by the thermocouples. For all tests the RF generator control system was set to maintain a constant pyrometer reading of 1400 °C. However the thermocouple measurements made with the calibrated thermocouples indicated a temperature 70 - 100 °C higher, depending on the nature of susceptor and insulation arrangement. This represents an error in the order of 6%. Some lowering of the
temperature as recorded by the pyrometer is unavoidable as a result of having to view through the sapphire window. It was hoped that the magnitude of this error would be significantly less than that quoted by Ircon for a quartz window (6.5%). Clearly either this is not the case, or other factors were influencing the results. One possible source of error within the test was indicated when re-examining the test set-up. A small alumina ring had been used to locate the window into the furnace wall as indicated in FIGURE 34a. The internal diameter of this ring was 5 mm, whereas the required diameter of site hole for the pyrometer at that distance from the sample was 7 mm. As a result it is likely that the pyrometer site line was partly obscured. This would account for some of the discrepancy in temperature values. Whilst by the time this flaw in the test procedure had been discovered it was no longer possible to repeat the tests, a larger ring was used for the final creep testing. Hence the value of these measurements as a calibration procedure has been significantly reduced. It can only be stated that the pyrometer reading is, at worst, 100 °C below the actual tube temperature. This is a very large variation and not acceptable for creep testing. This problem is considered further in chapter 19.

One reason why repeat tests could not be performed was that after 6 hours at temperature during the fourth test it was noticed that the thermocouple readings were beginning to drift. Finally signal from one of the couples was lost. This loss of thermocouple performance was attributed to gradual reaction between the Pt\Rh bead and the ceramic cement. Whilst the degradation of thermocouple performance
means that the ultimate accuracy of the temperature values is not clear, it was considered that the values obtained are a good reflection of the actual surface temperature of an alumina tube within the furnace. The problem of precise temperature measurement, particularly within a RF furnace is a difficult one. The solution attempted above, whilst far from ideal gives an indication of the true test conditions. As such the total variation across a central 10 mm section of the specimen of +/- 1.5 °C is achievable, with this dropping off to +/- 14 °C over a 20 mm section.
CHAPTER 11 : GRIP DEVELOPMENT

11.1 Grip design.

The design for the gripping system was produced by Mr. C. Morrison, chief experimental officer at the engineering department of Leicester university. The ultimate design is similar to the grip system designed and developed at ORNL by Lui and Brinkman [73]. The specimen used in their system is button-headed with a solid section. Each griphead is hydraulically operated using eight built-in independent hydraulic pistons equally spaced in a circular arrangement. Bending tests have indicated that variations of less than 1% in the axial stress around the circumference of the specimen are readily obtainable. These are now commercially available and widely used for short-term tensile testing.

The use of tubular specimens however requires a considerable modification of this basic design. Gripping tubular specimens requires the use of a friction type grip, button head type gripping being impossible on the tube ends. However due to the hardness of the ceramic materials no indentation of the surface could be tolerated without inducing local failure or cracking. Gripping the ceramic tube laterally produces undesirable crushing of the tube and it was necessary to balance the external grip force with an equal and opposite force produced by another set of grips acting internally.

The fine balance was achieved by using equal area floating pistons to apply the normal force to the split tapered collets; one on the outside and the other on the
inside of the tube. The items involved in the grip construction are shown in both assembled and disassembled state in the photograph in FIGURE 37. The components are identified by an item number and details of the function of each item is given in table beneath. Detailed sketch drawings of each item are included in Appendix D.

The grips operate when gas pressure is applied simultaneously to the bottom face of the inner piston and the top face of the outer piston. The pressure source for the grips is nitrogen at 1000 psi provided from a standard cylinder. A valve system was used to first close the grips. This was achieved by introducing gas pressure to force the inner piston (item 1) upwards against the outer collet (item 9) and simultaneously force the equal area outer piston (item 2) backwards pulling down on the pull rod (item 10) which is fixed by a retaining nut to the back of this piston. The result of this is that the tapered outer surface of the outer collet is forced up into the tapered hole in the outer retaining ring (item 6), thus causing it to apply an even compressive force onto the outside surface of the sample tube, whilst the pull rod pulls down into the tapered inner collet (item 8) again causing it to apply a compressive force, this time to the inner surface of the tube. Having the inner and outer pistons of equal area ensures that the internal and external gripping forces are equal. It was initially thought necessary to apply gas pressure in the reverse direction such that the inner piston is forced down and the outer piston forced up in order to release the gripping action. However, although the gas system and valves required to do this were incorporated into
FIGURE 37: ASSEMBLED GRIP (TOP LEFT OF PHOTOGRAPH) AND DISASSEMBLED COMPONENTS OF THE GRIP PLUS ASSOCIATED EQUIPMENT.

<table>
<thead>
<tr>
<th>ITEM NUMBER</th>
<th>ITEM NAME</th>
<th>FUNCTION OF ITEM</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>INNER PISTON</td>
<td>TO PUSH UP ONTO THE OUTER COLLET CAUSING IT TO CONTRACT</td>
</tr>
<tr>
<td>2</td>
<td>OUTER PISTON</td>
<td>TO PROVIDE DOWNWARDS FORCE ON THE PULL ROD</td>
</tr>
<tr>
<td>3</td>
<td>GRIP BODY</td>
<td>TO CONTAIN INNER AND OUTER PISTONS AND PROVIDE A GAS TIGHT CHAMBER FOR THEM TO OPERATE IN.</td>
</tr>
<tr>
<td>4</td>
<td>GRIP BODY CAP</td>
<td>LOCATE THE OUTER RETAINING RING</td>
</tr>
<tr>
<td>5</td>
<td>INNER RETAINING RING</td>
<td>TO PROVIDE A LIMIT FOR THE UPWARD TRAVEL OF THE INNER PISTON</td>
</tr>
<tr>
<td>6</td>
<td>OUTER RETAINING RING</td>
<td>TO LOCATE THE OUTER COLLET</td>
</tr>
<tr>
<td>7</td>
<td>BACK PLATE</td>
<td>TO ENABLE MANUAL OPENING AND CLOSING OF GRIPS</td>
</tr>
<tr>
<td>8</td>
<td>INNER COLLET</td>
<td>TO APPLY AN EVEN INTERNAL COMPRESSIVE FORCE TO THE TUBE</td>
</tr>
<tr>
<td>9</td>
<td>OUTER COLLET</td>
<td>TO APPLY AN EVEN EXTERNAL COMPRESSIVE FORCE TO THE TUBE</td>
</tr>
<tr>
<td>10</td>
<td>PULL ROD</td>
<td>TO PULL DOWN INTO THE INNER COLLET CAUSING IT TO EXPAND</td>
</tr>
</tbody>
</table>
the design, it was found more convenient to lever the back plate (item 7) upwards to manually release the grips.

In order to allow these grips to operate in close proximity to the RF coils, water cooled copper plates were fabricated and fixed to the front faces of the grips. Again a detailed sketch drawing of these plates and the fixing brackets is given in APPENDIX D. These plates had the dual function of cooling by cold water flow and absorbing stray RF field from the coils. Their use ensured that even at operating temperatures in excess of 1400 °C the grip temperature never exceeded 130 °C at the collets and 70 °C in the bulk of the grip head. No signs of heat damage were ever detected on any part of the gripping assembly. One consequence of the incorporation of these plates into the design however was air gaps were introduced. These gaps were between the grip face and the cooling plate face, and in the central hole of the cooling plate. It was these gaps that had to be thoroughly insulated in order to obtain the best performance from the furnace.

In the grip testing that is described in the following section it was not found necessary to alter this design. The original design also incorporated slots in the collets, pull rod and retaining ring that were intended to provide extra support, by means of plate inserts, to prevent slip during the application of torsional load. These plates are shown in the photograph in FIGURE 37. However it was found that the grip without the plate inserts was slip free at all torsional loads applied during testing and they were not employed during testing.
11.2 Grip testing.

In order to establish that the grips could function to provide a bend stress free loading at suitable stresses a steel tube with a thinned down gauge section was used as a test piece. This tube was conventionally strain gauged at the thinned down section at positions 90 degrees around the circumference.

The operation of the grips both on closure and under load was checked by monitoring the gauge outputs on an oscilloscope. It was found that rapid application of the gas pressure to the grips caused a 'snapping on' of the collets and a severe peak in bend stress. This peak in stress was also observed on opening of the grips by gas pressure. As a result a technique whereby gas pressure was increased slowly from a cylinder, rather than rapidly from a valve was tried. Again the grips 'snapped on', although to a lesser degree. Finally by closing the grips by hand pressure on the back plate, prior to gradually increasing the gas pressure provided a bend stress free grip operation. This procedure needed to be followed closely, as a number of ceramic tube fractures did occur at an early stage in the test programme on application of the grip pressure.

Once the grips had been closed it was possible to apply loads up to the limit needed for ceramic creep testing with no sign of slip of the metal tube. However it was not possible to strain gauge a ceramic tube and repeat these measurements due to the severe limits on the number of ceramic specimens available.
Tests to measure the magnitude of the bend stresses generated by the system under static load were performed during the evaluation of the extensometer system and are reported in chapter 12.2.

11.3 Grip operation

As discussed earlier a procedure for operation of the grips was established for a tubular metal specimen. In addition to this a procedure had to be established in order to load ceramic specimens into the grips and within the furnace. A number of early failures occurred due to particle contamination in the collets causing brittle failure during grip application. As a result all collet components were ultrasonically cleaned and degreased prior to assembly. A thin layer of particle free high temperature grease and flake graphite was applied to the sliding faces to aid release. The bottom grip was assembled off the rig, with care being taken to align collet slots, the specimen inserted and the grip closed by hand pressure on the back plate. The grip was then fixed to the rig via a pin joint and the furnace placed over the tube to rest on the generator coils. The top grip, already assembled but not tightened, was then fixed to the load train and lowered gently to locate the specimen. The grip was then tightened by hand.

By use of this procedure it was possible to load a specimen and fully prepare the rig in 10 to 15 minutes.
CHAPTER 12 : EXTENSOMETRY

12.1 System design

As has been discussed earlier, a number of ways exist by which displacement of a ceramic specimen in creep can be measured. It was decided at the outset that using any kind of contact device on the specimen surface, or making any kind of mark on the surface as a reference point would involve introducing unwanted damage to the ceramic material and introduce extra machining costs. Despite this, early attempts were made to construct a contacting extensometer based on alumina rods contacting the specimen at the end of the gauge section, but still within the furnace. Considerable difficulties were encountered, largely related to the lack of space within the furnace for such a system and the need for extra holes to be cut in the furnace walls. Non-contact methods such as laser based extensometry also require extra sight holes to be cut in the furnace and in addition are expensive to set up.

To accommodate the above conditions the chosen method for extensometry was to measure the displacement of the grip head faces with respect to each other. Although not ideal, the measurement of strain outside the furnace has become common for high temperature creep test machines (e.g. [62]). Provided the collet system is slip free such a 'remote' strain measurement system is acceptable and by far the most convenient to use.

The location of the equipment developed with respect to the grips is shown in the photographs and sketch
drawing contained in the earlier FIGURES 30a, 30b, 30e. A more detailed drawing of the extensometer system is given in FIGURE 38. The construction was based on the use of two alumina rods located in an aluminium fixing ring attached to the top grip body. The lower ends of these two rods were fixed by means of a stiff ball joint to an aluminium reference piece containing two flat perpendicular faces. On the lower grip face, again located within a fixing ring, were four non-contacting gap sensors arranged such that displacement of the "target" reference pieces could be monitored in both the vertical and rotational directions. The sensors provided a voltage output that was proportional to the gap between the probe face and the perpendicular face of the reference target piece. By continuously monitoring the voltage output a continuous measure of the displacement between the grip heads could be achieved. However the voltage output from the gap sensors was not linear with respect to displacement. A calibration procedure was required and this procedure is detailed in the following section.

The pieces that comprise the extensometer equipment could be assembled rapidly after the sample had been located within the grip/furnace area. As will be detailed later it was important that the probe/reference target gap was initially set in the range where the probes are most accurate. To achieve this small metal shims of known thickness were used to set an initial gap.

During testing the output from the sensor heads was recorded as a voltage signal via a computer controlled data collection system. This comprised a "MERLIN" data
FIGURE 38: EXTENSOMETER ASSEMBLY
logging unit supplied by Solatron Instrumentation Group, Farnborough, England and an APPLE II PC. This system was used to monitor all creep testing in the laboratory and monitored up to 32 channels of voltage output. Data was collected at predefined time intervals, in the case of these tests every hour, and stored on the PC floppy disc. It was necessary to replace the floppy disk at regular intervals to avoid the disc becoming full and loosing test data. From the floppy disc data could be transferred to the university main frame computer system for subsequent tabulation and plotting. In addition to the data logger a multichannel digital volt meter with an accuracy of +/- 1 millivolt was used to monitor visually the probe output voltages. This meter could also produce a simple chart paper output of voltage for up to 4 channels, again at a specified time period, to provide an additional record of the testing. Several practical problems were experienced with the operation of this data logging equipment. These are discussed in detail in chapter 14.

12.2 System calibration

Prior to use on the test equipment it was necessary to calibrate the sensor heads used against a known series of gaps. This was necessary as the voltage response of the sensors was not linear with respect to distance, and was not identical for each head. The heads were calibrated on a jig consisting of a head locator with a plate suspended above. This plate was attached to a micrometer allowing the gap between the plate and the head to be adjusted and
measured to an accuracy of +/- 1 micron. The heads were calibrated for gaps from zero to 6 millimeters, in steps of 0.1 mm. The raw data obtained from this experiment is shown in TABLE 3. This data was analysed by a polynomial curve fitting procedure contained in the commercial PC software package GRAPHER (Golden Software Inc., USA) in order to obtain equations that would yield accurate values of displacement for any recorded voltage. The polynomials generated for each probe are listed in TABLE 4. These polynomial functions were generally of a high order, being between 7th and 10th order equations. The order of polynomial depended on the number of terms required to give an acceptable fit as defined by the curve fit analysis package. It was found that very significant errors were introduced if all the polynomial terms were not included in the curve fitting process.

It was recognised that this procedure of curve fitting introduced its own source of error into the data collecting system. To quantify this error the raw voltage data was fed back into the polynomials to generate predicted values of gap setting. These values could then be compared to the actual measured gaps. The data obtained from this exercise and the errors resulting from the curve fitting are presented graphically in FIGURE 39. From this data it can be seen that in general there was a working range over which good agreement between the measured and predicted gap was achieved. Both above and below this range agreement was significantly worse. This had important consequences for the setting up of the probe gaps prior to testing as it was
TABLE 3: CALIBRATION DATA FOR EXTENSOMETER PROBES

<table>
<thead>
<tr>
<th>GAP SET / mm</th>
<th>PROBE 1 VOLTAGE</th>
<th>PROBE 2 VOLTAGE</th>
<th>PROBE 3 VOLTAGE</th>
<th>PROBE 4 VOLTAGE</th>
</tr>
</thead>
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<tr>
<td>0</td>
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<td>4.995</td>
<td>5.172</td>
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<tr>
<td>0.3</td>
<td>4.709</td>
<td>4.181</td>
<td>4.508</td>
<td>4.738</td>
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<td>4.109</td>
<td>4.36</td>
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<td>3.463</td>
<td>3.758</td>
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<td>3.445</td>
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<td>2.893</td>
<td>3.157</td>
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<td>2.646</td>
<td>2.896</td>
<td>3.179</td>
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<td>2.869</td>
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<td>1.324</td>
<td>1.504</td>
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</tr>
<tr>
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<td>1.108</td>
<td>1.276</td>
<td>1.571</td>
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<td>0.658</td>
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<td>0.313</td>
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<td>0.196</td>
<td>0.338</td>
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<td>4.5</td>
<td>0.202</td>
<td>0.114</td>
<td>0.123</td>
<td>0.219</td>
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<td>0.07</td>
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<td>0.027</td>
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<td>-0.002</td>
<td>-0.001</td>
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### Table 4: Polynomial Equations Derived to Describe the Extensometer Probe Calibration Data

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<th>DEGREE</th>
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<th>2</th>
<th>3</th>
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<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>COEFFICIENT</td>
<td>5.31533</td>
<td>-5.32866</td>
<td>3.70548</td>
<td>-1.73109</td>
<td>0.504821</td>
<td>-0.0876903</td>
<td>0.00827196</td>
<td>-0.000325382</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
<td>10</td>
</tr>
<tr>
<td>COEFFICIENT</td>
<td>5.33996</td>
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<td>13.4404</td>
<td>-12.0457</td>
<td>6.84007</td>
<td>-2.49817</td>
<td>0.584795</td>
<td>-0.0846971</td>
<td>0.00690318</td>
<td>-0.000241887</td>
<td></td>
</tr>
<tr>
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<td>0</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
<td>10</td>
</tr>
<tr>
<td>COEFFICIENT</td>
<td>5.85949</td>
<td>-13.7491</td>
<td>27.6916</td>
<td>-35.2792</td>
<td>27.9662</td>
<td>-14.1905</td>
<td>4.679</td>
<td>-0.996014</td>
<td>0.131846</td>
<td>-0.00985865</td>
<td>3.17846E-4</td>
</tr>
<tr>
<td>DEGREE</td>
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<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
<td>10</td>
</tr>
</tbody>
</table>
essential that the gaps remained in the working range during the total time of a test.

Following this analysis an overall assessment of the accuracy of each probe was made and the results are shown in TABLE 5. From the curve fitting data it was possible to work out the mean and maximum error for each probe due to this procedure. In addition, to calculate an overall expected accuracy, the error of +/- 1 micron in the calibrating equipment has to be included and it was assumed that the voltage readings as recorded by the data logger were accurate to no better than +/- 1 millivolt over the appropriate ranges. This voltage accuracy limit resulted in an additional error of around +/- 1 micron in the probe readings. The resulting overall maximum and mean errors associated with each probe are given in TABLE 5. The maximum expected error is between 5.3 and 18.4 microns depending on which probe is being used. For a 15mm gauge length this corresponds to an error in recorded strain of between 0.04% up to 0.12%. The mean error in all cases is less than 0.05% strain.

Comparing these figures to the existing standards quoted for creep testing of metals shown in chapter 6, it can be seen that the mean error is still greater than that required by British standard BS4A4 (0.005%) by a factor of between 4 and 9 times. However taking into account the requirement that the method of extensometry must be a non-contacting one, it was considered that this was an acceptable level of accuracy. It can be noted that the system is slightly more accurate than the laser based system of Heathwood [71] (strain resolution no better than 0.2%)
# Table 5: Calculated Accuracy Levels of Extensometer Probes

<table>
<thead>
<tr>
<th>PROBE NUMBER</th>
<th>WORKING RANGE</th>
<th>CURVE FIT ERROR</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mm</td>
<td>VOLTS</td>
</tr>
<tr>
<td>1</td>
<td>0.1 - 3.5</td>
<td>5.7 - 0.5</td>
</tr>
<tr>
<td>2</td>
<td>0.3 - 3.5</td>
<td>4.1 - 0.3</td>
</tr>
<tr>
<td>3</td>
<td>0.5 - 3.0</td>
<td>4.1 - 0.5</td>
</tr>
<tr>
<td>4</td>
<td>0.5 - 4.5</td>
<td>4.0 - 0.2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>PROBE NUMBER</th>
<th>+/- 0.001 VOLTS ERROR</th>
<th>ERROR IN CALIBRATION UNIT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>+/- 0.95 MICRONS</td>
<td>+/- 1 MICRON</td>
</tr>
<tr>
<td>2</td>
<td>+/- 1.1 MICRONS</td>
<td>+/- 1 MICRON</td>
</tr>
<tr>
<td>3</td>
<td>+/- 1.0 MICRONS</td>
<td>+/- 1 MICRON</td>
</tr>
<tr>
<td>4</td>
<td>+/- 0.95 MICRONS</td>
<td>+/- 1 MICRON</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>PROBE NUMBER</th>
<th>MAXIMUM TOTAL ERROR / MICRONS</th>
<th>MEAN TOTAL ERROR / MICRONS</th>
<th>MAXIMUM TOTAL ERROR / % STRAIN</th>
<th>MEAN TOTAL ERROR / % STRAIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.65</td>
<td>+/- 2.65</td>
<td>0.044</td>
<td>+/- 0.018</td>
</tr>
<tr>
<td>2</td>
<td>5.32</td>
<td>+/- 2.9</td>
<td>0.035</td>
<td>+/- 0.019</td>
</tr>
<tr>
<td>3</td>
<td>18.36</td>
<td>+/- 6.6</td>
<td>0.122</td>
<td>+/- 0.044</td>
</tr>
<tr>
<td>4</td>
<td>10.41</td>
<td>+/- 4.35</td>
<td>0.069</td>
<td>+/- 0.029</td>
</tr>
</tbody>
</table>
even taking the maximum error. However the maximum error is
well in excess of that quoted for the NIST laser based
equipment [72] (+/- 2 microns).

Having calibrated and established the accuracy of
the extensometer probes trials were performed with the
system on a metal tube mounted in the grips and loaded using
the actual loading equipment used for creep testing. In this
way probe results could be directly compared with strain
gauge measurements by attaching gauges to the tube. The
results of this testing are shown in TABLE 6. These results
show a good agreement between strain gauge readings and
probe readings. The mean values of displacement per unit
tensile load for Probes 1 and 4 agreed with gauge values to
within 1.5 microns. However the scatter of values for the
probes was larger than that for the gauges, reflecting the
errors that are to be expected from the calibration
procedure. Agreement was not so good for the torsional
measurement, the mean values for displacement per unit
torsional loading recorded with probes 2 and 3 being within
5 - 6 microns of the gauges. This is just within the region
of maximum calculated error for probe 2 but within the
region of mean error for probe 3. As such these results are
still acceptable. Again it can be noted that the scatter of
readings is higher for the probes than for the gauges,
particularly in the case of probe 3 which had the highest
associated calibration error.

This analysis could not be repeated for a ceramic
tube as all the available tubes were retained for actual
creep testing. It was considered that the attachment of
gauges for low temperature tests would significantly damage
<table>
<thead>
<tr>
<th>TENSILE LOAD /kg</th>
<th>TORSIONAL LOAD /kg</th>
<th>CHANGE IN DISPLACEMENT / MICRONS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>GAUGE 1</td>
<td>PROBE 1</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
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</tr>
<tr>
<td>20</td>
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<td>9</td>
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<tr>
<td>40</td>
<td>0</td>
<td>8.9</td>
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<tr>
<td>60</td>
<td>0</td>
<td>8.8</td>
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<td>0</td>
<td>8.7</td>
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<td>-8.5</td>
</tr>
<tr>
<td>40</td>
<td>0</td>
<td>-8.9</td>
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<tr>
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<td>10</td>
<td>0</td>
</tr>
<tr>
<td>20</td>
<td>0</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Mean change per 20 kg tensile step (loading):

- Tensile load: 8.85 9.40 9.40 7.63 0.00 -0.95 -0.03 -0.43
- Torsional load: -8.70 -8.23 -9.53 -8.53 0.00 -0.07 0.03 -0.43
- Mean change per 10 kg torsion step (loading): -0.43 -0.13 0.67 -0.03 67.00 72.67 66.00 76.00

Mean change per 10 kg torsion step (unloading):

- Tensile load: 0.37 0.07 0.37 -0.50 67.33 -69.33 -65.67 73.33

Table 6: Comparison of the data recorded by extensometer probes and strain gauges during the loading and unloading of a metal tube.
the surface of the ceramic tubes and make the specimens unsuitable for later high temperature tests.

By doing a further series of trials using the strain gauged metal tube it was possible to look at the extent to which gauges on either side of the tube agreed and in this way to obtain a measure of an bending created by the loading system. The results from such an experiment using the strain gauged tube are given in TABLE 7 for both tensile and torsional loading. These results show a maximum difference in displacement of 4 - 5% for tensile gauges and 5 - 6% for torsional gauges. Kandil and Dyson [62] discuss this type of measurement and state that quoted values in the literature for this bending strain ratio vary between +/- 3% up to +/- 5%. They recommend that the maximum value in ceramic materials testing should not exceed +/- 3%. As the measured values fall within this range the equipment would appear to be performing acceptably.

Attempts to repeat this experiment using a ceramic tube with the probes alone were abandoned for two reasons. Firstly only very low elastic displacements occurred at loads that were considered safe enough to use without risking fracturing the samples, and secondly the uncertainty in the probe readings was high compared to the displacements being measured. In the absence of figures for ceramic tubes the values for the metallic specimen should indicate the maximum displacement due to off axis loading i.e. bending. The actual displacements for ceramic materials of considerably higher stiffness will be much less than those observed in the metal specimen. However, rather than make assumptions regarding bending in a ceramic tube, it is
TENSILE LOADING

GAUGE 1 AND 2 IN THE SAME PLANE AS THE PROBES
GAUGE 4 AND 7 IN PLANE AT 90 DEGREES TO PROBES

<table>
<thead>
<tr>
<th>TENSILE LOADING /kg</th>
<th>DISPLACEMENT GAUGE 1 /MICRONS</th>
<th>DISPLACEMENT GAUGE 2 /MICRONS</th>
<th>DIFFERENCE GAUGE 1:GAUGE 2 /MICRONS</th>
<th>DIFFERENCE GAUGE 1:GAUGE 2 %</th>
<th>DISPLACEMENT GAUGE 4 /MICRONS</th>
<th>DISPLACEMENT GAUGE 7 /MICRONS</th>
<th>DIFFERENCE GAUGE 4:GAUGE 7 /MICRONS</th>
<th>DIFFERENCE GAUGE 4:GAUGE 7 %</th>
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<td>0</td>
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<td>8.9</td>
<td>0.5</td>
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<td>2.5</td>
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<tr>
<td>40</td>
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<td>18.3</td>
<td>1.2</td>
<td>4.3</td>
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<td>2.9</td>
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<tr>
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<td>0.4</td>
<td>4.7</td>
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TORSION LOADING

GAUGE 3 AND 6 IN THE SAME PLANE AS THE PROBES
GAUGE 5 AND 8 IN PLANE AT 90 DEGREES TO PROBES

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<td>0</td>
<td>2</td>
<td>3</td>
<td>1</td>
<td>--</td>
</tr>
</tbody>
</table>

Table 7: Comparison of strain gauge readings taken on opposite sides of a metal tube during tensile and torsional loading at room temperature.
considered that a true evaluation of the bending at room
temperature should be made as one of the next steps in
verifying the accuracy of the test equipment.
CHAPTER 13 : LOAD SYSTEM

As the equipment was intended to act as a combined tension/torsion testing machine it was necessary to design loading mechanisms to apply load in orthogonal directions with no off axis loading. A photograph of the final loading system in operation during a combined tension/torsion test and sketches of the equipment are shown in FIGURE 30a - 30d. Details of the two systems are given below.

13.1 Tensile load system

For simplicity a lever arm dead weight tension load system was employed with hardened steel knife-edge pivots. A sketch of the lever arm is shown in FIGURE 40, this sketch being a detailed view of the overall sketch shown in FIGURES 30c and 30d. Load is applied in the form of metal weights that are placed on the load hanger. This hanger is attached to one end of the lever arm by a free moving bearing joint. The weight of the hanger itself is counter balanced by applying a suitable weight to the opposite end of the arm. Load is transmitted to the grips via a second free moving bearing which connects to the pin jointed main load train. To maintain the lever arm in a position perpendicular to the rig a LVDT was employed to monitor the arm position. The signal from the LVDT was used to drive a worm gear motor located at the bottom of the rig. This drive gear was attached to the bottom platen of the rig onto which was attached the universal joint of the bottom grip. Displacement of the specimen causes the lever arm to
FIGURE 40
DETAIL OF LEVER ARM CONSTRUCTION

SIDE VIEW

TOP VIEW

LEVER ARM

KNIFE EDGE PIVOT

Ø 30 mm

100 mm

10 mm

Ø 30 mm

50 mm

BEARING FOR ATTACHMENT TO TENSILE LOAD HANGER

BEARING FOR ATTACHMENT TO LOAD TRAIN

Ø 20 mm

250 mm

100 mm
drop and hence activate the drive motor via the LVDT. The result is a downward motion of the whole load train and consequent levelling of the lever arm, at which point the LVDT switches off the motor. This system ensures that the lever arm stays perpendicular to the load train during the test and that the specimen centre section remains fixed with respect to the furnace centre.

At the end of a test that runs to failure the system above will activate when the tube breaks and result in a continuous drive of the bottom platen downwards. To ensure this did not happen a limit switch was set such that the motor power would be cut once a certain distance had been moved. A similar limit switch was also incorporated at the top limit of the drive to protect against possible damage by over driving the bottom platen upwards during initial set up. The position of all these components is indicated on the photographs and sketches in FIGURES 30a-d.

It should be stressed that the load system was not calibrated prior to use in this work. As a result a degree of uncertainty must exist as to the exact nature of the loads being applied. This uncertainty was not considered to be a major fault in the preliminary set of tests conducted during the period of this work. However for further work the errors associated with this load system should be fully quantified by calibration against a standardised load cell.

13.2 Torsional load system.

The torsional loading system used was that described previously by Trampczynski [77] for
tension\ torsion testing of metal specimens. The system is based around an air bearing mounted within the load train and again is shown in the sketches in FIGURES 30c and 30d and in a more detailed sketch in FIGURE 41. The air bearing consists of upper and lower discs separated by an air cushion provided by a compressed air supply. The upper of the two discs was fixed via a central spindle to the lower part of the load train by a pin jointed universal coupling. The lower disc was mounted on a rigid metal cradle that enclosed both discs. This cradle was linked by a pin jointed universal coupling to the top part of the load train. Two grooves were cut in the edge of the top disc to contain a length of steel cable, which was attached firmly so as to run half way around the disc, around a system of pulleys and around the other half of the disc where it was again firmly fixed. A weight hanger was constructed from a free running wheel and weight support and this was mounted on the wire running between the two discs. Again the arrangement is shown in FIGURES 30c and 30d. On application of load the wire was tensioned such that the upper disc rotates applying the torque directly to the bottom section of the load train. On failure the weight hanger was allowed to fall to a support positioned just below the base of the hanger and provided the extra rotational displacement was smaller than 180 degrees no damage could be done to the extensometry equipment.

As with the tensile loading system no attempt was made to calibrate this loading system prior to the tests reported in this work. Prior to undertaking a detailed study
of tension/torsion creep testing such a calibration should be made.
CHAPTER 14: CREEP TESTING

At the outset of this work it was the intention to construct test equipment for combined tension-torsion creep testing. As such a number of tests were required to establish the performance of the equipment. As the construction of equipment formed the bulk of the work the number of tests was limited to 13.

Initially it was intended that only reaction bonded silicon nitride specimens be examined. However as these were in short supply, there being only nine remaining for a testing program, four alumina specimens were included. Before testing samples of either material it was necessary to identify suitable testing conditions such that this testing program could be viewed in the context of previous work.

On examining the literature on reaction bonded silicon nitride, an initial surge in interest in the creep properties can be seen in the 1970's. The results obtained during this time were summarised by Grathwohl and Thummler [81]. The graphical summary they prepared is shown in FIGURE 42. This shows a range of experimental temperatures between 1100 °C and 1400 °C and a range of stresses between 10 and 300 MPa. In addition these authors note that under similar test conditions the steady state creep rate can vary by a factor of more than 300 as a result of variations in material properties. These variations may arise from different fabrication methods, impurity levels, pore size and size distribution, and alpha/beta phase ratio. This variation makes direct comparison between these results and
FIGURE 42: STEADY STATE CREEP STRAIN RATE AGAINST UNIAXIAL TENSILE STRESS FOR A RANGE OF REACTION BONDED SILICON NITRIDE MATERIALS (FROM REF [81])
our own material difficult. However it was discovered just prior to the start of the test program that the data reported recently by Kandil and Dyson [62] had been obtained using test pieces machined from the same manufacturers material as our own test specimens. The material data they published matches that listed in APPENDIX C. This allows for a direct comparison and an easier identification of appropriate test conditions. The summary of the results obtained by these authors is given in FIGURE 43 along with data obtained by Lee [82] on a similar material. For Kandil and Dyson's tests the temperature was fixed at 1450 °C and the stress was varied between 5 and 40 MPa. The sample tested at 5 MPa did not fail even after 1000 hours, whilst that tested at 40 MPa failed in under two hours. The data obtained by Lee was at 1370 °C and tests were performed at between 40 and 100 MPa. Failure times were between 1.5 and 250 hours under these conditions.

It was decided that the new equipment should be tested over a reasonable range of stresses but at the same time that the times to failure should, if possible, be kept to below 100 hours due to the need to complete the testing in the time remaining on the project. The first test done on the machine was a short step loading test to check the performance of the loading and strain measuring equipment. After this it was decided to initially investigate a range of stresses in pure tension between 30 and 50 MPa, but, in view of the times to failure indicated by Kandil and Dyson's work, to fix the test temperature for all tests to 1400 °C. In order to first verify that the equipment was capable of producing reproducible valid data comparable with previously
FIGURE 43: UNIAXIAL TENSILE STRESS AGAINST TIME TO FAILURE FOR REACTION BONDED SILICON NITRIDE (FROM REF [62])
published work, it was decided to conduct a number of these pure tensile tests before investigating tension/torsion behaviour. As no data was available on torsional or combined stress state testing of reaction bonded silicon nitride test conditions were chosen to be at the low end of the stress range in the hope that very short times to failure did not occur. As a result one experiment in pure torsion was performed at 30 MPa stress and a combined tension/torsion test was performed at 15 MPa tension/15 MPa torsion. Both tests were again performed at 1400 °C. A list of all the tests performed and conditions used is given in TABLE 8 at the start of chapter 15.

When considering appropriate conditions for the creep testing of alumina a very large volume of testing exists with which data could be compared. Cannon and Langdon in their extensive review [2] list over 40 papers on testing of high purity alumina alone. This large volume of data has allowed the construction of deformation mechanism maps for alumina. Such maps have been compiled by Frost and Ashby [3]. Maps showing temperature contours on a stress versus strain rate graph, and strain rate contours on a stress versus temperature graph for alumina of similar grain size to that used in this work are shown in FIGURE 44. As the models discussed in the first part of this thesis are based on diffusional flow it was decided that testing should be done well within the boundaries of the diffusional region of the maps. Initially tensile testing was done at 1200 °C and at 30 MPa stress. After instantaneous failure of the first sample loaded under these conditions a second sample ran to failure but with a failure time in excess of 300 hours. As a
FIGURE 44: DEFORMATION MECHANISM MAPS FOR ALUMINA WITH A GRAIN SIZE OF 10 MICRONS (FROM REF [3])
result it was decided to raise the test temperature to 1300°C. To try to ensure that at least one combined tension/torsion test was performed on alumina the next test was performed with a small tensile stress of 5 MPa and a torsional stress of 30 MPa. With this test running to failure without a problem a final pure tension test at 30 MPa was conducted. Again a summary of the conditions used is given in TABLE 8.

It was decided that all tests should be performed with the furnace running with the external susceptor only, the inside of the tubes being filled with a non-contacting insulation plug. As these tests were intended only as a proof of the equipment, rather than for the collection of highly accurate data this configuration was chosen, being easier to assemble. As has been shown by the finite element modelling and the practical measurements, the best temperature profiles are only obtained from the use of a combined internal and external susceptor arrangement.

In the testing program problems were encountered with the operation of the data logging equipment. Initially these problems were due to a fault with the computer used to collect and store data. This fault took some time to locate and as a result all tests performed on reaction bonded silicon nitride had data collected on the paper print-out from the volt meter. For all tests on alumina tubes however it was thought that the computer problem had been solved and the volt meter recorder was turned off. On later examination it was found that no data could be retrieved. It is unclear as to why this was so. Human error due to an unfamiliarity with the system is the most likely explanation.
Unfortunately the error was only picked up at the end of the test program and as a result the only records of the tests on alumina tubes are the manual volt meter readings recorded in a laboratory note book at random times. These were taken only as a check on the progress of the test. However in most cases they were taken often enough to give a reasonable description of the creep behaviour.

At the end of each test fracture surfaces were examined by scanning electron microscope (SEM) for evidence of damage within the microstructure. To aid this examination samples from the fracture surface and from the grip ends of both materials were etched to reveal the undamaged grain structure. In the case of alumina samples a thermal etch at 1450 °C for 20 minutes was used as described in standard procedures [78]. In the case of reaction bonded silicon nitride, whilst chemical etching is reported as being possible (e.g. [79]) it was decided to attempt thermal etching in vacuum as described in [80]. To perform this small samples of fracture surface and tube ends were sealed in evacuated silica glass tubes, then heated at 1400 °C for 2 hours. To further assess damage and simplify microstructural investigation sections close to the fracture surface were taken and mounted in epoxy resin. The mounted samples were ground flat and polished on a lapping wheel with successive grades of diamond grinding paste down to 1 micron size. These samples were also examined by SEM and thermally etched. Results obtained from the microstructural examinations are given in chapter 15.4.
### SUMMARY OF TENSILE PROBE RESPONSE DURING CREEP TESTING

<table>
<thead>
<tr>
<th>TEST NUMBER</th>
<th>MATERIAL</th>
<th>TEST TEMPERATURE /DEGREES C</th>
<th>TENSILE LOAD (MPa)</th>
<th>TORSIONAL LOAD (MPa)</th>
<th>TIME TO FAIL (HOURS)</th>
<th>STRAIN AT FAILURE (%) (PROBE 1 OR 3)</th>
<th>STRAIN AT FAILURE (%) (PROBE 4)</th>
<th>CREEP STRAIN RATE (1/HR) (PROBE 1 OR 3)</th>
<th>CREEP STRAIN RATE (1/HR) (PROBE 4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CT1</td>
<td>RBSN</td>
<td>1400</td>
<td>10 - 100</td>
<td>0</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>CT2</td>
<td>RBSN</td>
<td>1400</td>
<td>50</td>
<td>0</td>
<td>15.75</td>
<td>0.44 (1)</td>
<td>0.11</td>
<td>25.3 x 10 E-5 (1)</td>
<td>7.43 x 10E-5</td>
</tr>
<tr>
<td>CT3</td>
<td>RBSN</td>
<td>1400</td>
<td>50</td>
<td>0</td>
<td>4</td>
<td>0.31(1)</td>
<td>0.25</td>
<td>76.4 x 10 E-5 (1)</td>
<td>62.3 x 10E-5</td>
</tr>
<tr>
<td>CT4</td>
<td>RBSN</td>
<td>1400</td>
<td>50</td>
<td>0</td>
<td>13</td>
<td>0.37 (1)</td>
<td>0.23</td>
<td>26.3 x 10 E-5 (1)</td>
<td>15.5 x 10E-5</td>
</tr>
<tr>
<td>CT5</td>
<td>RBSN</td>
<td>1400</td>
<td>40</td>
<td>0</td>
<td>21.1</td>
<td>0.65 (1)</td>
<td>0.53</td>
<td>27.8 x 10E-5 (1)</td>
<td>24.6 x 10E-5</td>
</tr>
<tr>
<td>CT6</td>
<td>RBSN</td>
<td>1400</td>
<td>30</td>
<td>0</td>
<td>17</td>
<td>0.77 (1)</td>
<td>0.64</td>
<td>39.1 x 10E-5 (1)</td>
<td>27.1 x 10E-5</td>
</tr>
<tr>
<td>CT7</td>
<td>RBSN</td>
<td>1400</td>
<td>30</td>
<td>0</td>
<td>22</td>
<td>0.97 (1)</td>
<td>0.61</td>
<td>31.8 x 10E-5 (1)</td>
<td>18.6 x 10E-5</td>
</tr>
<tr>
<td>CT8</td>
<td>RBSN</td>
<td>1400</td>
<td>0</td>
<td>30</td>
<td>16</td>
<td>0.36 (1)</td>
<td>0.28</td>
<td>17.5 x 10E-5 (1)</td>
<td>17.2 x 10E-5</td>
</tr>
<tr>
<td>CT9</td>
<td>RBSN</td>
<td>1400</td>
<td>15</td>
<td>15</td>
<td>37</td>
<td>UNKNOWN(*)</td>
<td>UNKNOWN(*)</td>
<td>26.7 x 10E-5 (1)</td>
<td>3.68 x 10E-5</td>
</tr>
<tr>
<td>CT10</td>
<td>ALUMINA</td>
<td>1200</td>
<td>30</td>
<td>0</td>
<td>FAILED INSTANTLY</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>CT11</td>
<td>ALUMINA</td>
<td>1200</td>
<td>30</td>
<td>0</td>
<td>312 (APPROX.)(***)</td>
<td>0.75%(APPROX.)(***) (3)</td>
<td>1%(APPROX.)(***)</td>
<td>3.72 x 10E-5 (3)</td>
<td>3.27 x 10E-5</td>
</tr>
<tr>
<td>CT12</td>
<td>ALUMINA</td>
<td>1300</td>
<td>5</td>
<td>30</td>
<td>22 (APPROX.)(***)</td>
<td>&gt;1.25% (***) (3)</td>
<td>&gt;1.1% (***</td>
<td>105 x 10E-5 (3)</td>
<td>96 x 10E-5</td>
</tr>
<tr>
<td>CT13</td>
<td>ALUMINA</td>
<td>1300</td>
<td>5</td>
<td>30</td>
<td>DID NOT FAIL(***)</td>
<td>---</td>
<td>---</td>
<td>140 x 10E-5 (3)</td>
<td>126 x 10E-5</td>
</tr>
</tbody>
</table>

### SUMMARY OF TORSIONAL PROBE RESPONSE DURING CREEP TESTING

<table>
<thead>
<tr>
<th>TEST NUMBER</th>
<th>MATERIAL</th>
<th>TEST TEMPERATURE /DEGREES C</th>
<th>TENSILE LOAD (MPa)</th>
<th>TORSIONAL LOAD (MPa)</th>
<th>TIME TO FAIL (HOURS)</th>
<th>STRAIN AT FAILURE (%) (PROBE 1 OR 3)</th>
<th>STRAIN AT FAILURE (%) (PROBE 2)</th>
<th>CREEP STRAIN RATE (1/HR) (PROBE 1 OR 3)</th>
<th>CREEP STRAIN RATE (1/HR) (PROBE 2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CT8</td>
<td>RBSN</td>
<td>1400</td>
<td>0</td>
<td>30</td>
<td>16</td>
<td>0.68 (3)</td>
<td>0.67</td>
<td>35.3 x 10 E-5 (3)</td>
<td>35.4 x 10E-5</td>
</tr>
<tr>
<td>CT9</td>
<td>RBSN</td>
<td>1400</td>
<td>15</td>
<td>15</td>
<td>37</td>
<td>UNKNOWN(*)</td>
<td>UNKNOWN(*)</td>
<td>16.9 x 10 E-5 (3)</td>
<td>19.0 x 10E-5</td>
</tr>
<tr>
<td>CT12</td>
<td>ALUMINA</td>
<td>1300</td>
<td>5</td>
<td>30</td>
<td>22 (APPROX.)(***)</td>
<td>&gt;0.53% (***) (1)</td>
<td>&gt;0.50% (***)</td>
<td>444 x 10E-5 (1)</td>
<td>422 x 10E-5</td>
</tr>
</tbody>
</table>

* RF TRIP BLEW DURING TEST. RESTARTED AFTER TEMPERATURE HAD FALLEN TO APPROXIMATELY 1000 DEGREES C

** RF TRIP BLEW DURING TEST AFTER 35 HOURS. SAMPLE COOLED TO ROOM TEMPERATURE. TEST NOT RESTARTED.

*** DATA LOGGER NOT FUNCTIONING. MANUAL READINGS ONLY. EXACT TIME OF FAILURE AND STRAIN AT FAILURE UNCERTAIN.

TABLE 8 : SUMMARY OF CREEP TEST CONDITIONS AND RESULTS
CHAPTER 15 : TEST RESULTS

The results obtained from each test are listed along with the experimental conditions used in TABLE 8. For tests CT2 to CT8 a full set of data was available from the print-out of the volt meter. This allowed the determination of strain to failure, time to failure and steady state creep strain rate for each probe in these tests. Test CT9 had the RF over power trip blow during the test. This problem is discussed later in chapter 16. The resulting creep curve is discussed in more detail in the following section. As discussed earlier test CT10 failed instantaneously on application of load. Again this is discussed further in chapter 16. The final three tests did not have a full set of data due to the previously discussed problems with the data logging equipment. As a result both the values for time to failure and strain to failure are uncertain for these tests.

These 13 tests constitute a relatively small volume of data. However they do contain two unique tests, these being the first reported tests of the creep behaviour of reaction bonded silicon nitride and alumina under combined tension/torsion loading. As such they are of considerable relevance to the field of creep testing of ceramic materials. From the overall set of results it is possible to make important evaluations of the equipment, the models proposed in the first part of this thesis, and an initial assessment of the multiaxial creep behaviour of the two materials. In addition interpretation of failure modes and fracture surface analysis gives some insight into the
material behaviour. The following sections examine each of these aspects of the results.

15.1 Creep curves

The process by which raw voltage data was gathered from the extensometers, the way this data is converted into actual displacements and the associated errors have all been detailed in chapter 12. Conversion of the displacement data obtained from the extensometers to provide strain values required the identification of specimen gauge lengths for both tensile and torsional loading. These gauge lengths depend on the specimen geometry and temperature distribution along the length of the tube. The creep gauge length for tensile loading \((L_C)\) was determined from the following standard equation:

\[
L_C = \int_{0}^{L} \left[ \frac{A}{A_0} \exp \left( \frac{-Q}{R \cdot (T - T_0)} \right) \right] \, dL
\]

[ \(L = \) sample length, \(A_0 = \) initial sample cross sectional area, \(A = \) sample cross sectional area at \(L\), \(T_0 = \) initial temperature, \(T = \) temperature at \(L\).]

Temperature profiles measured as described in chapter 10.2 give values of \(T \) and \(T_0\), and an activation energy for creep deformation of 600 kJ/mol obtained from the literature [81] were used. Since the temperature drops off steeply in the thicker section of the tube the computation
of the gauge length is insensitive to the creep exponent (n) for the material being tested. The resulting gauge length was determined to be 15 mm for tensile loading. Provided it is assumed that the tube radius remains constant during the test and that the tube dimensions allow thin wall calculation assumptions to be made, it can be proved that the gauge length for torsion is the same as that for tension [83]. However account has to be taken of the fact that the rotational strain is not measured at the tube surface but at a point some distance away from the surface. Hence the observed rotation strain, \( \frac{D_g}{L} \) where \( D_g \) is the strain gauge displacement and \( L \) is the torsional gauge length, must be adjusted by a factor of \( \frac{r}{r_g} \), where \( r \) is the tube radius and \( r_g \) is the distance between the tube centre and the centre point of the gauges.

The creep curves produced are shown in FIGURES 45 to 59 on the following pages. Each test is considered in order in the following paragraphs with interpretations of the data obtained in terms of how well the equipment has performed. The data obtained is compared to other authors work where appropriate in section 15.3.

**CTL - Step loading test** (reaction bonded silicon nitride tube): This test was set up in order to establish that the loading, griping and extensometer systems functioned correctly under test conditions. Loading was increased in steps of 10 MPa up to 100 MPa and temperature was set at 1400 degrees C. The creep curve obtained is shown in FIGURE 45. Good agreement is obtained between probes 1 and 4 recording tensile strain at opposite sides of the specimen.
STEP LOADING CREEP TEST : CT1 (10 MPa - 100 MPa, 1400°C,
MATERIAL : REACTION BONDED SILICON NITRIDE)

% STRAIN

TIME / MINUTES

FIGURE 45
until the end of the fifth loading step. For the last three points of this step a drop in strain occurs (at approximately 310 - 315 minutes). The magnitude of the drop is around the same for both probes, being approximately 0.01% strain. It is thought that this may be due to a local cooling of the equipment caused by air currents in the laboratory. The equipment happened to be located next to the entrance door to the laboratory. At the time of the test program the effects of small temperature variations in the atmosphere were not considered and access into and out of the laboratory was not restricted. This is not likely to noticeably affect the temperature within the well insulated furnace. However the faces of the grips, to which the fixing rings which contain the mountings for the probes and probe targets are attached, are at around 70 °C. The rings are 15 mm thick and made from aluminium which has a thermal expansion coefficient of approximately 25 x 10^-5 K^-1. Cool air entering into the warm atmosphere of the laboratory may be as much as 10 °C cooler. Assuming this causes a temperature drop in the extensometry equipment of only 2 degrees the resulting contraction could cause an error in recorded strain of up to 0.01%. This is approximately the magnitude seen in the data. The fact that probe 4 has dropped further than probe 1 may reflect the fact that probe 1 was located closer to the source of cold air, although it can be seen that probe 4 was reading slightly lower prior to the obvious drop. Possible further evidence of similar effects in later creep tests are pointed out in the relevant sections. However the data sampling rate in all of the
following tests was less and this type of effect is less obvious.

A more serious problem is indicated in the curves at the transition between the 70 and 80 MPa loading step (approximately 415 minutes). Whilst probe 1 records the expected increase in strain probe 4 shows hardly any increase. The difference between the two probes is 0.06% strain and remains close to that value throughout the 80 MPa step. This represents a 15% variation in recorded value of strain. The most likely explanation of this is that some bending has occurred due to off axis loading at this relatively high stress. It is possible that at high stress the pin joints of the load frame have locked-up and can no longer accommodate off axis loading components. Further evidence of this can be seen at the 90 and 100 MPa loading steps where the strain rate indicated by probe 4 is less than that for probe 1. This step loading test would seem to indicate that some improvement should be made to the pin joint system if testing is to be performed at stresses much higher than 60 MPa.

Creep test CT2 (reaction bonded silicon nitride tube): This test was conducted under 50 MPa tensile stress and at 1400 °C. The creep curve obtained is shown in FIGURE 46. It is immediately apparent that very poor agreement has been obtained between the two probes. Probe 1 indicates a strain rate over three times that indicated by probe 4. By comparison with other authors results, as discussed in the following section, it appeared that probe 1 was indicating the expected magnitude of strain rate. The behaviour of
CREEP TEST CT2 (50 MPa tension, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)

FIGURE 46
probe 4 can only be explained by a very large degree of bending. At the end of the test it was noted that the pin joints at various stages along the load column were randomly orientated and did not lie in mutually perpendicular planes. This alignment had not been checked prior to the test and for all later tests it was ensured that a check of this was made prior to applying a load. For the results analysis the strain rate value indicated by probe 4 has been discarded as clearly erroneous. This has been done rather than taking the average value of the two probes because, as is discussed in the following tests, it is apparent that significant errors exist in the data obtained from probe 4.

**Creep test CT1** (reaction bonded silicon nitride tube): This test was again at 50 MPa tensile stress and at 1400 °C. The creep curve obtained is shown in FIGURE 47. Having initially ensured the pin joints were correctly alined, this test gave an improved agreement between probes 1 and 4. However again probe 4 records a significantly lower strain value. The average difference is around 0.05% strain. This is close to the value of the mean error calculated for these probes (± 0.02% for probe 1, ± 0.03% for probe 4, as detailed in chapter 12) however the error is not random and is quite consistent. It should be noted here that in almost all of the remaining tests a similar effect is noted where probe 4 reads lower than probe 1. Throughout most of test CT1 very good agreement was obtained between probes 1 and 4. It is possible that the calibration of either probe has drifted or changed in some way, or that one of the probes has been damaged physically in some way. This effect only became
Creep Test C13 (50 MPa tension, 1400 C, Material: Reaction Bonded Silicon Nitride)

Figure 47
apparent some considerable time after the testing program ended and the calibration could not be checked. Before further work is carried out on the equipment the calibration procedure should be repeated to check if an error has occurred.

In addition to the general disagreement between the probes, at 1.5 hours into the test probe 1 shows a drop in strain whilst probe 4 shows a sharp increase. The effect is reversed over the next recording interval. This would appear to be due to a bending effect which is later recovered.

Creep test CT4 (reaction bonded silicon nitride tube): This test was the third conducted under 50 MPa tension and at 1400 °C. The creep curve obtained is shown in FIGURE 48. Again there is a large discrepancy between the readings of probe 1 and probe 4. In addition a large scatter can be seen in the data recorded from probe 4. This scatter is still within the calculated average error of +/− 0.03% as detailed in chapter 12, however it is unclear as to why it is so obvious in this test and no other. The data recorded from probe 1 is quite smooth with only a small degree of variation. A small "hump" in the strain recorded by probe 1 can be seen between approximately 4 and 5.5 hours into the test. This may be due to the type of temperature variation effects discussed earlier. It would appear this can still be identified with a data sampling rate of once every 15 minutes.
CREEP TEST CT4 (50 MPa tension, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)

FIGURE 48
Creep test CT5 (reaction bonded silicon nitride tube): This test was conducted under 40 MPa tensile stress and at a temperature of 1400 °C. The creep curve obtained is shown in FIGURE 49. After the first 5 hours of this test probe 4 again begins to read lower than probe 1. At 4.75 hours a relatively large jump in the strain recorded by probe 4 occurs. This is not accompanied by a drop in probe 1 as was seen in the bending event recorded in test CT3. It is not clear what has caused this effect, however afterwards the gap between the strain values recorded by probes 1 and 4 emerges again. Close to the end of this test (19.75 hours) a jump in recorded strain appears for both probes. This jump of approximately 0.03 % strain is higher than that expected from the type of transient temperature variation discussed earlier which would be in the order of 0.01%. Data for several time intervals following this event was not recorded due to the chart paper running out on the volt meter output device. For the final two points the value of strain for probe 4 has dropped, whilst that for probe 1 has risen. This may again represent a bending process although this would not fully explain the jump in strain recorded by both probes.

Creep tests CT6 (reaction bonded silicon nitride tube): For this test the tensile load was further reduced to 30 MPa whilst the temperature was kept at 1400 °C. The data sampling rate was reduced to once an hour to reduce on the amount of data processing required for what was expected to be a longer test. The creep curve obtained is shown in FIGURE 50. The difference between the two probes is again
CREEP TEST CT5 (40 MPa tension, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)

% STRAIN (PROBE 4) — % STRAIN (PROBE 1)

Figure 49
CREEP TEST CT6 (30 MPa tension, 1400 C. MATERIAL:
REACTION BONDED SILICON NITRIDE)

\% STRAIN (PROBE 1)  \% STRAIN (PROBE 4)

TIME / HOURS

STRAIN \%
apparent after the first hour of the test. As a result of the lower data sampling frequency random temperature effects are less noticeable and the curves from both probes are smooth. Towards the end of the test at 15 hours a bending type process can again be seen where strain recorded by probe 4 increases more than expected whilst that at probe 1 decreases.

**Creep test CT7** (reaction bonded silicon nitride tube): this test was conducted under the same conditions as test CT6 (30 MPa tensile stress at 1400 °C). The creep curve obtained is shown in FIGURE 51. In general the same pattern as for the previous tests is followed with the agreement between probes 1 and 4 being quite good up to approximately 0.2% strain and becoming gradually worse as the test progresses. This test shows no signs of bending and the curves are smooth as a result of using the hourly sampling rate. In addition this test shows the largest strain to failure recorded in any of the tests and also shows evidence of accelerated creep rate in both curves after 17 hours under load.

**Creep test CT8** (reaction bonded silicon nitride tube): this test was conducted with 30 MPa of torsional stress, zero tensile stress and at 1400 °C. Separate curves are presented for the tensile response (recorded by probes 1 and 4) in FIGURE 52 and the torsional response (recorded by probes 2 and 3) in FIGURE 53. The tensile probe results show fairly smooth curves and, as with previous tests show a good agreement between probes up to approximately 0.2% strain
CREEP TEST CT7 (30 MPa tension, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)

% STRAIN (PROBE 1)  % STRAIN (PROBE 4)

FIGURE 51
CREEP TEST CT8: TENSILE BEHAVIOUR (0 MPa tension, 30 MPa torsion, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)

FIGURE 52
CREEP TEST CT8 : TORSIONAL BEHAVIOUR (0 MPa tension, 30 MPa torsion, 1400°C, MATERIAL: REACTION BONDED SILICON NITRIDE)

FIGURE 53
after which the value recorded by probe 4 drops below that recorded by probe 1.

The curves recorded by probes 2 and 3 for torsional strain are in good agreement throughout the test. A small difference of approximately 0.02% strain can be seen over the full length of the test with probe 2 recording slightly higher strain values than probe 3. This may represent a slight error in calibration although the magnitude of the difference is close to that of the mean error calculated for these probes and is not considered to be a significant difference.

Creep Test CT9 (reaction bonded silicon nitride tube): This test was the first conducted under conditions of combined tensile and torsional loading. Both tensile and torsional stresses were set at 15 MPa and the temperature was again set at 1400 °C. Unfortunately during this test the RF power supply trip blew at some time between 23 and 24 hours into the test. It was observed that this had happened and the trip was reset, however by this time the specimen temperature had dropped to around 1000 °C. The temperature was returned to 1400 °C and the test ran to failure. Possible reasons why the trip should have been activated are considered in the following chapter. Again curves for tensile probe data (probes 1 and 4) and torsional probe data (probes 2 and 3) are presented separately in FIGURES 54 and 55 respectively.

The tensile creep curves obtained again show a considerable difference between probes 1 and 4. In this case probe 4 does not even show reasonable agreement with probe 1.
CREEP TEST CT9: TENSILE BEHAVIOUR (15 MPa tension, 15 MPa torsion, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)
CREEP TEST CT9: TORSIONAL BEHAVIOUR (15 MPa tension, 15 MPa torsion, 1400 C, MATERIAL: REACTION BONDED SILICON NITRIDE)

% strain (probe 2) ■ % strain (probe 3) □

FIGURE 55
over the first 0.2% strain as it does in the previous tests. In fact during the first two hours of the test probe 4 records almost no displacement. The reason for this is not clear as no evidence of a bending process can be seen in the response of probe 1. Again however it is clear that probe 4 is not responding correctly.

Both tensile probes clearly show evidence of the cooling event that took place between 23 and 24 hours into the test. The result in terms of displacement of the specimen appears to be a large bending where strain at probe 1 increases by around 0.5% and strain at probe 4 decreases by a similar amount. This may be due to different cooling rates, particularly in the furnace where cooling will be greatest on the side opposite the sight hole, causing large thermal stresses. After the temperature has returned to 1400 °C the curves recorded show smooth creep type behaviour up to failure. However the creep rate for both probes is faster than that recorded prior to cooling and reheating. This would seem to provide evidence that the material has been damaged in some way by the temperature cycle.

For the purpose of analysis the steady state creep rate is taken as that recorded up to 23 hours of the test. As a result of the accelerated creep rate observed after the cooling event the time to failure can only be taken as a minimum value under these conditions. Similarly strain at failure is uncertain due to the large bending event and no value can be stated with any degree of certainty.

The torsional probes show a moderately good agreement up to the trip failure. As in test CT8 probe 3 records a lower value of strain than probe 2. In this case
the difference increases during the test and reaches a peak value of approximately 0.05 % strain. This is slightly larger than the mean calculated error for probe 3 and is still considered to be reasonable. The effect of the cooling event is less marked than in the tensile probes, however after the temperature has returned to 1400 °C both the gap between the probe readings and the overall creep rate has increased. This would again seem to provide evidence that the cooling and reheating process has in some way damaged the specimen.

**Creep test CT10 (alumina tube):** This was the first test conducted using an alumina tube. The intended test conditions were a stress of 30 MPa and a test temperature of 1200 °C. However instantaneously on the application of the full load the specimen fractured within the section of tube enclosed in the bottom grip. The possible causes of this failure are discussed in the following chapter.

**Creep test CT11 (alumina tube):** This test used the same conditions as the unsuccessful test, CT10. No problems were experienced and the test ran to failure. The creep curve obtained is shown in FIGURE 56. As discussed earlier this and the following tests did not have data logged by the volt meter but had voltage readings noted by hand at random intervals. Due to the long duration of this test however a significant number of points were obtained and the creep curve is well defined.

By chance the probes had been switched in location prior to this test. Thus the tensile displacement for this
CREEP TEST CT11 (30 MPa TENSION, 1200 C, MATERIAL: ALUMINA)

% strain (probe 3)  % strain (probe 4)

TIME / HOURS

0  50  100  150  200  250  300  350

S T R A N

0  0.2  0.4  0.6  0.8  1.0
test was recorded by probes 3 and 4 rather than by probes 1 and 4. The data obtained showed again that probe 4 was in good agreement with probe 3 up to around 0.2% strain, after which the readings from probe 4 were significantly lower. This would seem to be strong evidence that probe 4 is not recording data accurately but reads low regardless of which of the probes it is compared against. Evidence is also seen of a bending type event between 140 and 150 hours into the test.

**Creep test CT12 (alumina tube):** This test was the only one to be conducted under combined tensile and torsional loading on the alumina specimens. The applied stresses were 5 MPa tensile stress and 30 MPa torsional stress. Again the only results available were those recorded by hand. The tensile creep curve is shown in FIGURE 57 and the torsional creep curve is shown in FIGURE 58. Only four data points were obtained during the first 11 hours of the test. After this time the equipment was running overnight. The following day a person was present in the laboratory at the moment of failure and the time was noted, although no strain values were recorded just prior to failure. The resulting time to failure was noted as 22 hours although data was only available for the first 11 hours of the test.

As with test CT11 tensile data was recorded using probes 3 and 4 and again the difference between the gauges is seen. In addition there would appear to be a significant acceleration in creep rate between approximately 10 and 11 hours into the test. This is only half way towards the eventual time to failure and it is uncertain if this
CREEP TEST CT12: TENSILE BEHAVIOUR (5 MPA tension, 30 MPa torsion, 1300 C, MATERIAL: ALUMINA)

%strain (probe 3) — %strain (probe 4)

TIME / HOURS

FIGURE 57
CREEP TEST CT12: TORSIONAL BEHAVIOUR (5 MPa tension, 30 MPa torsion, 1300°C, MATERIAL: ALUMINA)

FIGURE 58
acceleration truly reflects the beginning of tertiary creep or if it is some error in the measuring equipment.

The data for torsional strain was obtained using probes 1 and 2. These gauges show very good agreement throughout the test up to a strain of 0.5%. As probes 2 and 3 were in good agreement when recording torsional strain in tests CT8 and CT9, this would also seem to indicate that probe 4 is in error.

Creep test CT13 (alumina tube): This test was conducted under the same tensile stress as test CT11 (30 MPa) but with the temperature increased to 1300 °C. The creep curve obtained is shown in FIGURE 59. During this test the same type of trip failure as experienced in test CT9 occurred. However in this case the furnace temperature had cooled to almost room temperature before the problem was discovered. As a result the test was not restarted but was abandoned at that point with the tube remaining intact. It was estimated that the trip failure occurred at around 35 hours into the test. However data had only been noted by hand up to 28 hours into the test. As a result the steady state creep rate is the only parameter that can be determined from the results. The data collected again shows probe 4 recording a lower strain than probe 3. The magnitude of the difference is comparable to that recorded in test CT11. In both cases a difference of between 0.1 - 0.2 % strain is recorded at around 0.5% total strain. In addition much higher values of strain are recorded in C13 than in CT11. The difference between the probe values can be seen to continue to increase at these higher strains.
CREEP TEST CT13 (30 MPa tension, 1300 C, MATERIAL: ALUMNIA)

FIGURE 59
The above detailed examination of the 13 tests conducted using the new combined tension/torsion testing equipment reveals three important features. Firstly it is clear that one of the probes (probe 4) consistently gives low values of strain when compared to the other probes. It seems highly likely that this is due to some calibration error as the probe functions well in all other respects. The calibration procedure for this probe should be repeated before any further testing is done.

Secondly it is clear that a number of bending events have occurred during testing. Major bending problems were eliminated by ensuring that the pin joints in the load train were correctly positioned in orthogonal planes. However minor bending events still persisted. Lubricating the pin joints may be one possible way of reducing this effect particularly at high loads. Alternatively some type of system based on knife edges, such as that used by Kandil and Dyson [62] may have to be employed. However in general it can be said that the effects of bending in the existing equipment appear to be small.

Finally there is some evidence to suggest that transient cooling processes caused by air currents in the laboratory can influence the values of strain recorded. Although the effect is small it is recommended that access to the equipment and the surrounding laboratory area is restricted during testing.
15.2 Failure modes

One of the simplest observations from the tests is the modes of failure observed under different stress states. FIGURE 60 shows three failed components of reaction bonded silicon nitride. The torsional and combined stress state specimens are the first recorded examples of such failures in reaction bonded silicon nitride. In terms of failure surface profile, the uniaxial tension test specimens all exhibited a flat failure surface perpendicular to the direction of loading, as shown in the example taken from test CT7 in FIGURE 60a. The pure shear test (CT8) failed along a line which makes an angle of 45° with respect to the axis of loading, FIGURE 60b. Part of the final failure surface for this component lies outside the gauge section, but examination reveals a spiral crack which extends over a long length of tube and through the gauge section. It was not possible to determine from this experiment whether the spiral crack formed before or at the same time as the crack that resulted in the final failure of the component, but it is likely that the crack first formed in the gauge section and the final failure resulted from the dynamic response of the specimen due to the rapid unloading which followed the formation and propagation of the gauge section crack. It was considered highly unlikely that the crack origin would be in the cool thick section of the specimen. The specimen tested under combined tension/torsion (CT9), such that the axial and shear stresses were equal in magnitude failed along a line which makes an angle of about 30° with respect to the axis of loading (FIGURE 60c). These results would initially
FIGURE 60: FAILED SAMPLES OF REACTION BONDED SILICON NITRIDE UNDER:

a) UNIAXIAL TENSION
b) PURE TORSION
c) COMBINED TENSION/TORSION
appear to be consistent with a maximum principal stress failure criterion. This purely observational result is compared with the analysis of data obtained in the tests in the following section.

Of the three tests conducted on alumina specimens, one did not fail, tensile test (CT11) had a flat failure surface as with the tensile tests on reaction bonded silicon nitride, and the test conducted under 5 MPa tension and 30 MPa torsional stress was badly shattered. In this final case no actual failure surface could be identified, although spiral cracks similar to those seen in the pure torsion test on reaction bonded silicon nitride could be identified.
15.3 Results analysis.

The results detailed in section 15.1 can be analysed in two important ways. Firstly it is important to compare the results obtained under pure tensile loading with those obtained from tests on similar materials by other authors. Only in this way can the validity of the data be confirmed and hence the equipment be proven as suitable for creep testing. Secondly the results obtained can be compared to the behaviour predicted by the new models described in the first part of this thesis.

In chapter 14 a summary of comparable tests carried out on reaction bonded silicon nitride and alumina is given. In the case of reaction bonded silicon nitride the data obtained from tests CT2 to CT8 can be plotted directly onto the graphs reported by Grathwohl and Thummler and by Kandil and Dyson. This has been done and the resulting graphs are given in FIGURES 61 and 62.

For the steady state strain rate against stress plot the average value of creep rate was obtained using the values from both probes. The values of strain rate recorded by probe 4 were included. This was done because, at the time it was discovered, the error in the calibration of probe 4 discussed in section 15.1 could not be confirmed. Only the value for probe 4 in test CT2 was omitted as clearly erroneous. Given that the range of behaviour shown on the figure reflects the large variation that can be seen as a result of variations in the microstructure and composition of different grades of reaction bonded silicon nitride, the results from this series of tests lie quite close to the other results recorded at 1400 °C. This seems to be good
FIGURE 61: COMPARISON OF DATA OBTAINED ON REACTION BONDED SILICON NITRIDE IN THIS WORK WITH DATA COMPILED BY GRATHWOHL AND THUMMLER [81]
FIGURE 62: COMPARISON OF DATA OBTAINED ON REACTION BONDED SILICON NITRIDE IN THIS WORK WITH DATA OBTAINED BY KANDIL AND DYSON [62] AND LEE [82]
evidence that the strain rate measurements made with the new equipment are reasonable.

A closer comparison can be made with the data obtained by Lee and in particular that obtained by Kandil and Dyson who tested specimens made from an identical reaction bonded silicon nitride to that used in the current tests. This data is presented in terms of a plot of stress against time to failure. The plot with the current results added is shown in FIGURE 62. As would be hoped the results from tests CT2 to CT7 at 1400 °C lie between the values obtained by Lee at 1370 °C and those obtained by Kandil and Dyson at 1450 °C. The results also show a similar scatter to those of Lee but much higher scatter than that reported by Kandil and Dyson whose results lie very close to a straight line. The reason for this higher scatter when compared to tests done on an identical material is uncertain. It would seem reasonable to expect some degree of experimental variation and this would be expected to be reasonably large in the case of tests on ceramic specimens that can show quite large microstructural variation from specimen to specimen. However in general the results obtained from the new equipment would appear to be in good agreement with those obtained from tests on the same material but using different equipment.

These comparisons with three different sets of data for reaction bonded silicon nitride seem to confirm that the data obtained from the tests using the new equipment can be regarded as valid. As a result it would seem to be reasonable to also regard the results obtained from the torsional and combined stress tests as an accurate
description of behaviour under these previously unreported test conditions.

A similar comparison can be made between the results obtained on alumina specimens and the deformation mechanism maps discussed in chapter 14. The results obtained in tests CT11 and CT13 are plotted on a strain rate against stress map in FIGURE 63a and stress against temperature map in FIGURE 63b. The strain rate against stress plot in FIGURE 63b indicates that the tests results would be expected to have been obtained at temperatures of around 1230 °C for CT11 and 1400 °C for CT13. In fact the tests were carried out at 1200 °C and 1300 °C. This may be a reflection of a problem that occurred towards the end of the testing of the reaction bonded silicon nitride specimens. It was observed that the sapphire window through which pyrometer temperature measurement were made had developed a brown deposit on the inner surface. It was found to be very difficult to remove this deposit from the window. The presence of the deposit could well result in the pyrometer recording an artificially low temperature. The true specimen temperature could indeed have been close to that predicted from the map. This problem is discussed further in chapter 16.

In terms of the stress against temperature mechanism map shown in FIGURE 63a the position of the test data would indicate a strain rate of around $7 \times 10^{-8}$ 1/s for CT13 and $7 \times 10^{-9}$ 1/s for CT11. The actual strain rates recorded were $4 \times 10^{-7}$ 1/s and $1 \times 10^{-8}$ 1/s respectively. Again the values predicted could be seen to reflect that the tests were done at a slightly higher temperature than that set. However in the case of both temperature and the strain
FIGURE 63a: COMPARISON OF DATA OBTAINED ON ALUMINA WITH STRESS VS. TEMPERATURE DEFORMATION MECHANISM MAP COMPILED BY FROST AND ASHBY [3]
FIGURE 63b: COMPARISON OF DATA OBTAINED ON ALUMINA WITH STRAIN RATE Vs. STRESS DEFORMATION MECHANISM MAP COMPILED BY FROST AND ASHBY [3]
rate comparison it should also be noted that this type of map has a degree of error in the position of contours, and that the grain size of the test specimens is slightly less than that quoted for the map. Given these uncertainties it can again be stated that the results obtained with the new equipment are in quite good agreement with results reported in the literature.

Having confirmed that the results obtained are in reasonable agreement with previously reported data, where such data exists, it is possible to use the results to evaluate the models developed in the first part of this thesis.

As detailed in section 1.3 of chapter 1 the creep damage tolerance \( L \), defined in equation (8), is a parameter that describes the extent to which the damage mechanisms occur in a material. The expression quoted was:

\[
L = \frac{\epsilon_f}{\epsilon_s t_f}
\]

To verify this type of creep parameter from the models requires evaluation of the quantities in the above equation. By assuming that steady state creep is by boundary diffusion alone evaluation of the steady state creep rate can be made simply by use of the standard expressions used in chapter 4. It has already been verified in chapter 4.4 that the micromechanical models derived in this work reduce to the expression for Coble creep when damage is small. To evaluate the strain to failure and the time to failure extension of the models to incorporate void nucleation
expressions and a failure criterion is required. Cocks and Ashby [23] simplified this process by assuming that all voids nucleate at a set time, $t_n$, and that failure occurs at a critical value of $f_c$. For most cases a value of 0.25 is appropriate. Integration of the equations for strain rate between the time limits $t = t_n$ (when $f_c$ is very small) and $t = t_f$ (where $f_c = 0.25$) will yield expressions for strain to failure and time to failure. This has been done for the equations describing the constrained and unconstrained growth of equilibrium voids derived from the analytical model (equations (21) and (24) in chapter 4 for unconstrained, equations (25) and (27) for constrained). The value of $f_c$ is assumed to be very much less than the applied stress and the calculated ratio of $\epsilon / \epsilon_f$ is plotted against the ratio of $t / t_f$ for uniaxial tension in FIGURES 64a - 64i. These same ratios have been calculated from the creep data for all tests where all the relevant data is available. The problems encountered during the testing meant that data for tests CT9, CT12 and CT13 was incomplete and the curves could not be calculated. The resulting curves are also plotted in FIGURES 64a - 64i. In each case any primary creep has been removed from the data prior to calculation as the models do not take this into account.

Certain trends can be picked out from these graphs. For the tests on reaction bonded silicon nitride, tests CT2, CT3 and CT4 (FIGURES 64a, 64b, 64c) all conducted at 50 MPa tensile stress show the measured data to lie above the curve predicted for constrained growth of voids and well above that for unconstrained growth of voids. As the magnitude of the tensile stress is reduced to 40 MPa in test
STRAIN/STRAIN AT FAILURE Vs. TIME/TIME AT FAILURE FOR CT3

- ■ strain/strain at failure (probe 1)
- ○ strain/strain at failure (probe 4)
- ● MODEL (unconstrained voids)
- ○ MODEL (constrained voids)

FIGURE 64b
STRAIN/STRAIN AT FAILURE Vs. TIME/TIME AT FAILURE FOR CT4

- strain/strain at failure (probe 1)
- strain/strain at failure (probe 4)
- MODEL (unconstrained voids)
- MODEL (constrained voids)

FIGURE 64c
STRAIN/STRAIN AT FAILURE Vs. TIME/TIME AT FAILURE FOR CT5

- ■ strain/strain at failure (probe 4)
- ○ strain/strain at failure (probe 1)
- • MODEL (unconstrained voids)
- O MODEL (constrained voids)

STRAIN / STRAIN AT FAILURE

TIME / TIME AT FAILURE

FIGURE 64d
STRAIN/STRAIN AT FAILURE Vs. TIME/TIME AT FAILURE FOR CT6

- ■ strain/strain at failure (probe 1)
- ○ strain/strain at failure (probe 4)
- ● MODEL (unconstrained voids)
- □ MODEL (constrained voids)

FIGURE 64e
Figure 64f

Strain/Strain at Failure vs. Time/Time at Failure for CT7

- Strain/Strain at failure (probe 1)
- Strain/Strain at failure (probe 4)

MODEL (constrained voids)
MODEL (unconstrained voids)
STRAIN/STRAIN AT FAILURE VS. TIME/TIME AT FAILURE FOR TENSILE PROBES IN CT8

MODEL (constrained voids)

MODEL (unconstrained voids)

strain/strain at failure (probe 1)

strain/strain at failure (probe 4)

FIGURE 64g
TORSIONAL PROBES FROM CT8

MODEL (constrained voids)

MODEL (unconstrained voids)

strain/strain at failure (probe 3)

strain/strain at failure (probe 2)

FIGURE 64h

STRAIN/STRAIN AT FAILURE VS. TIME/TIME AT FAILURE FOR
STRAIN/STRAIN AT FAILURE Vs. TIME/TIME AT FAILURE FOR CT11

- ■ strain/strain to failure (probe 3)
- □ strain/strain to failure (probe 4)
- ● MODEL (unconstrained voids)
- ▪ MODEL (constrained voids)

FIGURE 64i
CT5 (FIGURE 64d), and to 30 MPa in tests CT6 and CT7 (FIGURES 64e and 64f) the measured data falls much closer to the predicted curves. For test CT5 the constrained growth model provides a good fit throughout the test. For tests CT6 and CT7 the measured data fits closely to the constrained model in the early part of the test but fits more closely to the unconstrained model towards the end of the test. A similar trend in behaviour is observed in both the tensile and torsional strain response recorded during the pure torsional test CT8 (FIGURES 64g and 64h) conducted under 30 MPa torsional stress.

It is clear that the predicted curves do provide a reasonable prediction of experimental data. However from this set of results it is difficult to draw any clear conclusions in terms of the validity of the new models described in the first part of this thesis. The models were based on material flow due to the stress driven diffusion of atoms. However this mechanism is unlikely to be the principal one involved in the creep of a covalently bonded non-oxide material such as reaction bonded silicon nitride. Oxidation has been recognised and modelled as the principal mechanism of creep [15,16] for this material.

The mechanisms on which the modelling was based are however those which occur during the creep of alumina. Of the three tests conducted on alumina specimens only CT11 had sufficient data for the relevant plot to be obtained and this is shown in FIGURE 64i. CT13 did not fail and in CT12 neither the strain at failure nor the time at failure were known with any degree of accuracy. The data obtained from test CT11 lies above the predicted curve for constrained
growth of voids and significantly above the curve for unconstrained growth. Clearly the constrained model provides a better description of observed behaviour. This result would appear encouraging in that it would seem most likely that the irregular distribution of damage and the large spacing between areas of damage that is observed in ceramics such as alumina would be best described by the constrained growth model.

It would not be expected that a model based purely on growth of equilibrium voids, and containing additional simplifying assumptions required in integration, could provide a highly accurate description of real material behaviour. However the fact that the constrained model provides the best description of real data confirms an important basis of the modelling. That is that it is highly important to consider effects arising from the microstructural nature of ceramic materials, such as constraint on the growth of damage, if the models are to be in any way accurate.

A second feature that can be used to describe creep failure is the isochronus surface. The models described in the first part of this thesis presented predicted behaviour in terms of iso-void growth rate surfaces which predicted a range of stress dependency of the time to failure. Effective stress, mean stress and maximum principal stress type behaviour could be predicted depending on which stage of the damage formation mechanism dominates. Three tests were performed under either torsional or combined stress states. Of these the only one was performed on alumina, the material to which the models best apply.
With the lack of comparable tensile test data from the two tensile tests and the uncertainty in time to failure in the combined stress test (CT12), it is not yet possible to draw conclusions as to the likely stress dependency of failure under multiaxial loading. However it is possible to do this for reaction bonded silicon nitride. A plot of stress against time to failure for all the tests conducted on reaction bonded silicon nitride is shown in FIGURE 65. For purely tensile tests the effective stresses and the maximum principal stresses are equivalent. For the tests conducted under pure torsion (CT8) and combined tension/torsion (CT9) values of both the effective stress and the maximum principal stress are plotted. As the mean stress in test CT8 (0 MPa tensile stress, 30 MPa torsional stress) was zero it was clear that this could not be the criterion for failure and these values were not plotted. At a time to failure of 36 hours both stress values lie reasonably close to the approximate straight line fitted to the tensile test data. The values are within the range of scatter shown during the purely tensile tests. However at a failure time of 16 hours the effective stress value lies very close to the tensile stress test value, whilst the maximum principal stress value is significantly lower, being approximately twice the range of scatter below the straight line fit. This would seem to provide good evidence, albeit on the basis of only two tests, that when tested under multiaxial stress conditions reaction bonded silicon nitride fails in response to the value of the effective stress. This, if confirmed by further testing, is an important conclusion with respect to the design of real components made from this material. As the
STRESS vs. TIME TO FAILURE FOR ALL TESTS ON REACTION BONDED SILICON NITRIDE

- UNIAXIAL TENSILE STRESS (CT2 - CT7)
- EFFECTIVE STRESS (CT8 & CT9)
- MAXIMUM PRINCIPAL STRESS (CT8 & CT9)

FIGURE 65
examination of failure surface shape, as detailed in section 15.2, seemed to indicate a maximum principal stress criterion the generation of further data under combined loading is essential. However the failure time data rather than the observations of fracture shape would appear more conclusive.

Again the comments made above with regard to the suitability of the models to describe the creep behaviour of reaction bonded silicon nitride apply. Some of the models do predict an effective stress type behaviour in the iso void growth surfaces. However the modelling can only truly be assessed when further data is available on the multiaxial testing of alumina.

In addition to micromechanical modelling a phenomenological approach to creep failure could be taken. However this approach which has been taken to derive equations to describe creep failure in metals can only be attempted once a relatively large volume of isochronus surface data has been obtained. This data is needed as the method requires the derivation of creep constants from experimental data and the derivation of a function that describes the full shape of the isochronous surface. When this has been done evaluation of the accuracy of this type of approach for ceramic materials can be made and it can be verified whether such a modelling technique is indeed independent of the nature of the material being analysed. This approach would seem to be a better way of describing the failure of reaction bonded silicon nitride where oxidation is the principal cause of creep deformation and
failure and no micromechanical models of the creep process are available.

15.4 Microstructural studies

As described in chapter 14, failed specimens were subjected to a detailed microstructural examination, both at the failure surface and at polished sections taken close to and away from the gauge section.

An example of the failure surface of a silicon nitride specimen is shown in the micrograph in FIGURE 66a. This surface is very unlike the polished section shown in FIGURE 66b. It is almost certain that the smooth, cracked surface with signs of surface crystal growth is an oxide film that has grown on the newly exposed fracture face after failure. As such this can tell us nothing about the failure process occurring, other than oxidation is obviously occurring. Sections taken just away from the fracture as typified by FIGURE 66b were again uninformative as they closely resembled the undamaged microstructure as shown in APPENDIX C. Any growth of porosity or other damage is unidentifiable due to the already very high (approximately 25%) level of porosity in the starting material. The attempts made to thermally etch the polished sections were unsuccessful revealing no extra detail of the grain structure. The reason for this is unknown, however as the principal mechanism of creep damage and failure in this material is likely to be the growth of oxide material, further significant effort was not expended on attempting to refine the rather complex etching technique.
FIGURE 66a: SEM MICROGRAPH OF THE FRACTURE SURFACE OF A REACTION BONDED SILICON NITRIDE TUBE SHOWING SURFACE OXIDE LAYER (MAGNIFICATION x2900, SCALE BAR = 3.45 MICRONS)

FIGURE 66b: SEM MICROGRAPH OF A POLISHED SECTION OF A REACTION BONDED SILICON NITRIDE TUBE FROM AN AREA CLOSE TO THE FAILURE SURFACE (MAGNIFICATION x253, SCALE BAR = 39.5 MICRONS)
FIGURE 66c: SEM MICROGRAPHS OF THE FAILURE SURFACE OF AN ALUMINA TUBE. THERMAL ETCHING HAS OCCURRED AFTER FAILURE CAUSING GRAIN BOUNDARIES TO BE VISIBLE (MAGNIFICATION x679, SCALE BAR = 14.7 MICRONS)
Microstructural examination of the failure surfaces of the alumina samples was much more instructive than was the case for the reaction bonded silicon nitride material. The most obvious evidence for the growth of damage and the failure process came from direct observation of the failed surfaces. As these surfaces had been exposed to test temperature for some time before the furnace could be turned off and cooled down a thermal etch treatment had effectively being applied. This can be seen in the micrographs in FIGURE 66c where the grain boundaries are clearly shown. It can be seen that fracture has occurred by an intergranular process where the grain boundaries have failed and the grains have been left intact. Evidence can be seen of the occurrence of triple point voids, crack like voids and full facet cracks, particularly around the smaller grains. This evidence is typical of the type of damage reported earlier by Evans et. al. [41].
CHAPTER 16 : TEST PROBLEMS

Of the 13 tests performed, 3 did not run satisfactorily to completion. One test resulted in the failure of an alumina tube on application of the grip pressure. As all other tests on these tubes ran acceptably this failure was put down to one of two reasons. Either the tube itself was flawed in some way to cause it to be too weak to stand the grip pressure, possibly as a result of machining damage, or a particle of hard material had become trapped between the grip face and the tube causing an indentation failure. Within the surroundings of a general laboratory dealing with hard ceramic materials the possibility of such contamination is always present. After this failure the cleaning and assembly of equipment was moved to as clean an area as possible and the ultrasonic bath used to clean down the equipment was drained and re-filled with clean solvent prior to each cleaning.

The two remaining failures occurred during test as a result of the power limit trip activating on the RF set. As typical power levels for running the furnace were only around 25% of full power the reason for these failures is not obvious. The general power supply for the machine is that used for the rest of the laboratory. It is possible that 'spikes' in the supply have caused the machine to blow a trip. This could only be remedied by having an independent power supply for this equipment.

A more serious problem was encountered during the testing of the reaction bonded silicon nitride tubes. During the course of the tests a brown deposit occurred at the cool
ends of the tubes and on the top of the furnace. On examination by SEM elemental analysis this deposit was shown to contain iron and was most likely iron oxide. Iron is a typical impurity within the silicon powder used to form the nitride. This was partly confirmed by the lack of such deposits during the testing of the alumina tubes. The major problem with this deposit was that it settled on the surface of the sapphire window. Thus the optical properties of the window changed during the tests, resulting in errors in the temperature recorded by the pyrometer. Whilst attempts were made to clean this window by polishing off the deposit the brown layer built up each time a test was run. It must be considered that, as well as the uncertainties introduced by having to view the specimen through the window, the gradual build up of this deposit will have resulted in a gradual rise in temperature during long term tests as the temperature the pyrometer 'sees' appears to fall and the generator responds by increasing power. This must be seen as a major problem in the testing of reaction bonded silicon nitride tubes by this method. The iron contamination is typical of commercial grades of RBSN, and as such will inevitably produce oxide during creep testing at high temperature. Attempts could be made to ensure the window is hot enough such that deposition does not occur, although this would most likely involve inserting the window into the susceptor wall. The technical problems involved in doing this have not been investigated and the possibility of reaction with the susceptor and subsequent degradation of optical performance of the window must be considered.
CHAPTER 17 : SUMMARY OF TESTING

The capability of the equipment described in detail in the previous chapters 6 - 13 to perform creep tests under multiaxial stress states at temperatures up to 1400 °C has been proved. The system would appear capable of running at temperatures well in excess of this value as only 25% of the available power is typically used to maintain 1400 °C. No degradation of the equipment was observed other than the cracking of insulation block as discussed in section 9.2, however deposits formed as a result of impurities within the reaction bonded silicon nitride material may have hindered the performance of tests by obscuring the pyrometer site hole.

The results obtained using the new equipment under conditions of uniaxial tension could be compared to tests on similar materials reported by other authors. This comparison generally showed good agreement indicating that valid results were being obtained. The later tests on alumina did indicate the possibility that the test temperature was slightly higher than indicated by the pyrometer. This may be due to the contaminated window in the furnace.

The examination of fracture modes initially indicated that the reaction bonded material fails in response to the value of maximum principal stress. However data analysis provided more conclusive evidence that failure occurs in response to the value of effective stress. Microscopic examination of failure surfaces was not instructive in the case of reaction bonded silicon nitride where oxidation is a principal factor in initiating high
temperature failure. However, in the case of alumina specimens, evidence for an intergranular failure mode and the presence of damage at grain boundaries was obtained.

Attempts were made to compare the data obtained from this equipment with the modelling work presented in part 1 of this thesis. This was difficult to do with the results obtained on reaction bonded silicon nitride, as the creep and failure mechanisms for this material do not match those used for the models. However for the one test on alumina for which suitable data was available, a reasonable agreement was obtained between the test data and the constrained growth of damage type model. This was taken as evidence that models should take into account microstructural variations in order to accurately describe the creep of ceramic materials.
In chapter 6 of this thesis a set of required operating conditions were set out for the test equipment. The features of the equipment developed to meet those conditions are summarised in the following paragraphs.

A RF heating furnace has been designed and constructed that is capable of sustained operation at temperatures of at least 1400 °C for long periods of time without significant degradation. Temperature profiles were achieved such that temperature variation was within ±1.5 °C of the set temperature over a central 10 mm section and ±14 °C over a central 20 mm section.

Temperature measurement and control has been achieved by use of an optical pyrometer focused through a sapphire window onto the specimen surface. The accuracy of the temperature measurement was not well defined as a result of the need to view through the sapphire window. Temperature control was achieved by a feed back loop to the RF generator and gave a stable temperature to ±2 °C over periods up to 300 hours.

Specimen gripping has been achieved by a novel system of linked internal and external collets providing even grip to the inside and outside surfaces of tubular specimens. Slip of this gripping system was not observed at any time under tension, torsion or combined loading conditions. Activation of these grips was achieved in such a way as to produce minimum bending and transient stresses in the specimen.
Displacements occurring during testing have been measured outside the furnace area by means of a rigid extensometer mechanism fixed to the grip faces. Examination of this system with both metallic and ceramic specimens indicated an accuracy of +/- 4 microns was obtainable.

Loading has been achieved by a pin jointed load train linked to a standard lever arm system of dead loading for tension, and a previously reported disc and cable system for torsion. Tests on a directly strain gauged metal tube indicated bending strains were of the order of 5% under maximum load. This value was confirmed by tests with the extensometry system and a ceramic tube.

Specimens designed using finite element analysis have been used for test, either in the as received state in the case of reaction bonded silicon nitride, or as machined in house in the case of alumina. A number of tests in pure tension, pure torsion or combined states were performed. One of the extensometer probes showed evidence of calibration problems, otherwise the tests were generally satisfactory, with the exception that deposition of iron oxide impurity from the reaction bonded silicon nitride tubes on the sapphire window meant that further inaccuracy had been introduced into the temperature measurement system.

Comparing these achievements with the requirements listed in chapter 6 shows that almost all of the requirements have been satisfied. The only area of concern lies in the accuracy of the temperature measurement. Actions that should be taken to resolve this final problem are discussed in the following chapter.
PART THREE

CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER WORK
At the outset this work was conceived with the eventual aim of applying to ceramics the type of design methods previously applied to metallic components at high temperatures. This would enable the design engineer to have a true capability to use ceramics in real applications with a full knowledge of how these materials would perform, thus enabling him to exploit the excellent potential they offer for improved performance. It was not the object of this three year study to fulfil this aim in its entirety, nor indeed would it have been possible in such a time period. However significant steps have been taken along two converging pathways.

In the first part of this thesis new theoretical models have been proposed to describe the behaviour of idealised ceramic materials under multiaxial states of stress. These models had to take into account the features proven to be important when modelling the behaviour of metals. As a result models describing diffusional growth of cavities have been used that incorporate grain boundary sliding and constrained growth of creep damage. An analytical model has been used to examine a number of simple situations corresponding to the stages in damage growth where damage is widespread. This model has yielded descriptions of the type of behaviour possible in these conditions. The analytical model has been taken as far as would seem sensible for the description of creep deformation and failure. A second modelling technique was required to describe more complex situations where voids in a ceramic
microstructure are widely spaced and the number of possible diffusion pathways is large. This situation is close to that seen at an early stage of the formation of damage. This modelling was achieved with the development of computer simulations of the deformation of large arrays of ceramic grains containing damage in the form of voids, cracks and even large holes. Through this type of modelling important conclusions were drawn as to the type of assumptions that could be taken in describing the deformation of fine grain materials. Only by taking into consideration the full response of a large number of grains, as is the case in the new model, can the relationship between void growth and matrix creep be described accurately.

The computer based modelling technique may have further applications outside the field of creep damage modelling. For example the sintering, rather than growth of voids may be modelled, particularly in the case of pressure driven sintering. In addition by the removal of chains of grain boundaries to form a long crack the growth and local stress fields around a dominant flaw can be analysed providing a model for the creep crack growth failure mechanism.

In the second part of this thesis the construction and operation of a creep testing machine to operate under multiaxial states of stress has been described. This work is important in providing the experimental data required to verify the new micromechanical models derived in this work, and to provide a data base from which phenomenological models of the type already used in metals can be constructed. Full details of the construction and operation
of this equipment have been given. The data obtained from the equipment under uniaxial tension showed good agreement with other authors data. Importantly, analysis of the multiaxial tests provided evidence that reaction bonded silicon nitride fails in response to the value of the effective stress. Physical evidence from the shape of fracture surfaces seemed to run slightly contrary to this however the experimental data, although limited in quantity, appeared more conclusive. In addition some preliminary analysis was possible and this analysis gave promising agreement with the predictions of the analytical model of constrained damage growth.

As is to be expected during the early stages of testing a number of problems and potential improvements to the system were indicated by the results obtained. It seemed clear from a detailed examination of the creep curves obtained that one of the extensometer probes had a calibration error and this probe requires recalibration prior to further testing. However without doubt the major problem experienced with the equipment is the accurate measurement of specimen temperature. The pyrometer system chosen has a considerable drawback in that a window is required to enable observation of the specimen without allowing heat to escape to any great degree. Although the chosen window material was of the lowest optical absorbance readily available, results indicate that a 6% error between actual and recorded temperature may still be expected. Bearing in mind the sensitivity of creep rates to temperature this error is not acceptable. Future developments must be considered. Some success has been
experienced in the use of fine wire thermocouples to monitor temperature within the RF field. The presence of the field is somewhat worrying as it has been known to effect the readings of thermocouples, although experiments carried out in this work did not seem to indicate this was a problem for such fine wire thermocouples at the frequency generated by the chosen machine. This system would be a compromise in the sense that only the furnace atmosphere temperature could be recorded rather than the specimen surface temperature. Direct application of thermocouples to a specimen surface would almost certainly result in damage or contamination of the specimen surface and hence the formation of a likely failure initiation site. However it is suggested that further investigations are made as to the accuracy of such a thermocouple system, with the aim of either replacing or calibrating the pyrometer with a thermocouple measuring the furnace chamber temperature. In view of the problems experienced with volatile components escaping from the reaction bonded silicon nitride samples, replacement rather than calibration may seem the preferred option, however the possibility of contamination of the thermocouple bead should also be born in mind. Although this solution would be a compromise the work done on temperature profiling a sample tube, if repeated, could provide a suitable correlation between chamber and actual specimen temperatures.

In addition to work on the temperature monitoring equipment, a further assessment of the bending stresses present in the equipment should be attempted. It was clear from the data that some instances of bending occurred during creep tests. In the work completed prior to the operation of
the equipment direct evaluation of bending was confined to tests on metal tubes. It is suggested that a full evaluation of a strain gauged ceramic specimen be made at both room temperature and at elevated temperatures, although this is very unlikely to be at test temperature, under relevant loads to truly quantify bend stress. This work may need to be followed by improvements to the pin joint system of the load train.

As indicated by both finite element heat models and by practical measurement, the temperature profile of the specimen within the furnace may be improved by the correct placement of internal susceptors. Firstly a convenient assembly of such an internal susceptor should be produced such that the current complex arrangement is replaced. In addition practical and modelling work could still be done on the shape of the external susceptor, and the position of internal susceptors to further improve temperature profiles.

The above recommendations are the most urgent in terms of improving the accuracy and reliability of the equipment. Having pointed out these problems however, the preliminary tests have shown evidence that the general performance of the machine is very promising. Thirteen tests were performed, ten of which ran to failure with a maximum time at temperature and under load of over 300 hours being recorded. Already the examination of multiaxial test data has revealed that reaction bonded silicon nitride seems to fail in response to the value of the effective stress. Bearing in mind the lack of published information on combined stress state testing of ceramic materials, these
tests are the first real indication of how a ceramic will respond under such conditions.

It has been possible to point out the way in which test results from this equipment can be used to verify modelling procedures. In the case of micromechanical models, if simplifying assumptions are made regarding void nucleation and crack coalescence it is possible to integrate the equations derived over time. The predictions made thus could be checked and the validity of the model and the assumptions proved by comparison to accurately known creep test results. This has already been partly achieved in the analysis of uniaxial test data on alumina and the results show acceptable agreement with modelling. In the case of phenomenological models, once the multiaxial response of materials is more fully known the experimentally derived parameters for the model can be incorporated into the equations and predictions checked against actual results.

If the recommendations for modifications and improvements to the equipment detailed above are followed, an important volume of data on the response of engineering ceramics under multiaxial stress states can be produced and the predictions of the computer, analytical and phenomenological models can be evaluated with respect to real observations. By this process the two branches of research detailed in this work can be brought together. With modifications as required to the models and the collection of enough reliable data, design codes could then be constructed to fulfil the eventual aim of providing the design engineer with a reliable tool with which to further the use of engineering ceramics.
REFERENCES


APPENDIX A: AN ANALYTICAL MODEL FOR THE GROWTH OF CREEP DAMAGE IN CERAMIC MATERIALS.

1) Principles of modelling

To analyse the structures formed when creep damage occurs a regular two dimensional array of idealised ceramic grains is assumed such as in FIGURE A1. As sides AB, BC, CD and DA are all axes of symmetry the grains contained are unable to rotate, thus any deposition of matter along a given boundary as a result of void growth and creep must be uniform. Prior to analysing the repeating unit shown in this figure a number of local parameters must first be defined. These are laid out in FIGURE A2.

As creep and void growth occurs grain boundaries move apart with a velocity $u_i$, where $i = 1, 2$ or $3$ as relevant to the particular boundary in FIGURE A2. Grains lying on a line of symmetry (1 and 3) have only half the standard boundary thickness, $s$, and move relative to the line of symmetry. Boundaries on either side of boundary 2 are free to slide resulting in a sliding velocity $u_2^S$. The total velocities of the boundaries in the unit must be compatible, thus:

$$u_1 = u_3$$

and

$$u_2^S = \frac{(2u_1 - u_2)}{\sqrt{3}} \quad (1)$$

Elongation rates are then given by

$$u_y = u_1$$
FIGURE A1 : A REGULAR TWO DIMENSIONAL ARRAY OF HEXAGONAL GRAINS SUBJECTED TO REMOTE STRESSES. THE SIDES AB, BC, CD AND DA OF THE RECTANGULAR ELEMENT ABCD ARE ALL AXES OF SYMMETRY.
FIGURE A2: THE REPEATING UNIT OF FIGURE A1 SHOWING:
(a) THE LOCAL GRAIN BOUNDARY VELOCITIES.
(b) THE LOCAL STRESSES.
(c) THE LOCAL CO-ORDINATE SYSTEMS.
and

$$u_x = \frac{2u_2 - u_1}{\sqrt{3}}$$

providing remote strain rates:

$$\epsilon_1 = \frac{2u_y}{d} = \frac{2u_1}{d}$$

and

$$\epsilon_2 = \frac{2u_x}{\sqrt{3d}} = \frac{4u_2 - u_1/2}{3d}$$

where \(d\) is the grain size measured between two parallel faces.

Since the grain boundaries are able to slide freely they are unable to support any shear stress. The remaining mean stresses are in equilibrium such that:

$$\sigma_3 = 3\sigma_1 - \sigma_2 - \sigma_2$$

and

$$\sigma_2 = \sigma_2$$

In order to analyse the diffusional processes that cause void growth equations that describe the flux of matter are required. Flux of atoms in a potential gradient can be described by Fick's first law [A1]:

$$j_i = \frac{\alpha D_b \kappa_b}{kT} \frac{d\mu_i}{dx_i}$$
Where $\mu_i$ is the excess chemical potential of an atom in the boundary plane,

$$\mu_i = -\sigma_i \Omega$$  \hspace{1cm} (vi)$$

and $\sigma$ is $1/2$ for a boundary lying on an axis of symmetry and 1 otherwise. The alpha term arises from the axis of symmetry along certain boundaries that means only half of such boundaries are associated with a given element.

For the flow of atoms along a unit length of boundary conservation of matter requires that [A2]:

$$\frac{dj_i}{dx_1} = -u_i$$  \hspace{1cm} (vii)$$

Equations (v) and (vii) can be combined to give an expression for chemical potential, and hence stress along a given boundary:

$$\sigma_i = -\frac{u_i}{2\alpha\Omega(D_{ij}\delta_b/kT)} x_1^2 + A_i x_1 + B_i$$  \hspace{1cm} (viii)$$

Where $A_i$ and $B_i$ are constants of integration.

The mean facet stress can be determined from the distribution of equation (viii) using:

$$\sigma_{i_{\text{mean}}} = \frac{1}{\ell} \int_{-\ell/2}^{\ell/2} \sigma_i dx_1$$  \hspace{1cm} (ix)$$

Where $\ell = d\sqrt{3}$ is the length of a grain boundary facet.
The basic principles detailed above were used to analyse a number of situations. Figure A3 shows the repeating units for each case. Details of the results for each case are given in the following sections.

2) Analysis of Coble creep

Whilst equations describing Coble creep are well established, use of the above approach to derive the known relationships is useful as a check. For this problem (shown in FIGURE A3(a)) stresses and flows are symmetric and \( \sigma_3 = \sigma_1 \). Boundary conditions apply such that

\[
\begin{align*}
  j_1 & = 0 \text{ at } x_1 = 0 \\
  j_2 & = 0 \text{ at } x_2 = 0
\end{align*}
\]

(a consequence of there being zero flux across a line of symmetry), and

\[
\begin{align*}
  j_1 & = -j_2 \\
  \sigma_1 & = \sigma_2 \\
  \text{at } x_1 & = \frac{1}{2} \text{ and } x_2 = -\frac{1}{2}
\end{align*}
\]

(a consequence of flux and potential being continuous across a grain junction).

Combining these boundary conditions with equations (viii), (ix), and (iv) and then substituting into equations (ii) and (iii) gives the standard expression for Coble creep in terms of remote strain rates:

\[
\dot{\varepsilon}_1 = -\dot{\varepsilon}_2 = \frac{36}{kT} \frac{D_b \varepsilon_1 \varepsilon_2}{D_b \varepsilon_1 \varepsilon_2} (\sigma_1 - \sigma_2) \quad (X)
\]
FIGURE A3: REPEATING UNITS SHOWING THE CAVITY AND CRACK DISTRIBUTIONS EXAMINED BY THE ANALYTICAL MODEL.
3) Analysis of uniformly distributed triple point voids

Making the assumption that voids are circular in 2-D, i.e. surface diffusion is much faster than boundary diffusion, the next simplest situation to analyse is a uniform distribution of voids at triple points as shown in FIGURE A3(b).

Here again the problem is symmetric. As before a number of boundary conditions relating flux and potential apply, namely:

\[ \begin{align*}
  j_1 &= 0 \quad \text{at} \quad x_1 = 0 \\
  j_2 &= 0 \quad \text{at} \quad x_2 = 0
\end{align*} \]

and

\[ \begin{align*}
  \sigma_1 &= \sigma_c \quad \text{at} \quad x_1 = (l/2-r) \\
  \sigma_2 &= \sigma_c \quad \text{at} \quad x_2 = -(l/2-r)
\end{align*} \]

(where \( r \) is the void radius and \( \sigma_c \) the capillary stress where
\[ \sigma_c = \gamma_s/r \]
with \( \gamma_s \) representing the surface energy).

Combining these conditions with equation (viii) and then equations (ii), (iii) and (iv) remote strain rates are given by:

\[ \begin{align*}
  \dot{\epsilon}_1^* &= 36D_b f_b \sigma \left( \frac{\sigma_t^2 - \sigma_t^2}{(1-f_b)^3} \right) + 18D_b f_b \sigma \left[ \frac{\sigma_t^2 + \sigma_t^2 - 2\sigma_c (1-f_b)}{(1-f_b)^3} \right] \\
  \dot{\epsilon}_2^* &= 36D_b f_b \sigma \left( \frac{\sigma_t^2 - \sigma_t^2}{(1-f_b)^3} \right) + 18D_b f_b \sigma \left[ \frac{\sigma_t^2 + \sigma_t^2 - 2\sigma_c (1-f_b)}{(1-f_b)^3} \right]
\end{align*} \] (xii)

where \( f_b = 2r/l \) is the area fraction of boundary consumed by the growing void.
Void growth rates can be derived from the expressions above as follows: the volume of the repeating unit is

\[ V = \sqrt{3/4} \, d^2 \]  

(xi)

The volumetric growth rate can be given by

\[ \dot{V}/V = \dot{\epsilon}_1 + \dot{\epsilon}_2 \]  

(xiv)

and

\[ \dot{V} = 2\pi r \dot{r} = -\frac{\pi}{2} f_h \dot{f}_h = \frac{\pi d^2}{6} \dot{f}_h \]  

(xv)

Combining with the strain rate expressions in equations (xi) and (xii) gives

\[ f_h = \frac{108/3}{\pi} \frac{D_\text{eff}}{kTQ} \frac{(\sigma - \sigma_c(1-f_h))}{(1-f_h)^3} \]  

(xvi)

where \( \sigma_m \) is the remote in-plane mean stress \((\sigma_1 + \sigma_2)/2\).

4) Analysis of non-uniformly distributed triple point voids

The situation shown in FIGURE A3(c) corresponds to a non-uniform distribution of voids. As a result the repeating unit is no longer symmetric about \( x_2 = 0 \) and flow of material along all three boundaries must be considered. Boundary conditions are now:

\[ f_1 = 0 \quad \text{at} \quad x_1 = 0 \]
\[ f_3 = 0 \quad \text{at} \quad x_3 = 0 \]
\[ \sigma_1 = \sigma_c \quad \text{at} \quad x_1 = (1/2-r) \]
\[ \sigma_2 = \sigma_c \quad \text{at} \quad x_2 = -(1/2-r) \]
\[ \dot{f}_1 = \dot{f}_3 \quad \text{at} \quad x_2 = 1/2 \quad \text{and} \quad x_3 = 1/2 \]
\[
\sigma_2 = \sigma_3 \quad \text{at} \quad x_2 = \frac{1}{2} \quad \text{and} \quad x_3 = \frac{1}{2}
\]

When combined as previously with the equilibrium equations the following expressions for remote strain rates are produced:

\[
\dot{\varepsilon}_1 = \frac{3e^{-D_b^0}}{kT d^3} \left[ \frac{3 \sigma_1^+ - \frac{5}{2} \sigma_2^- \left( \frac{1-0.2f_h}{1-f_h/2} \right) - \frac{\sigma_5}{2} \left( 1-2f_h \right)}{\frac{3}{4} \left( \frac{7}{3} - \frac{f_h}{2} \right) + (1-f_h)^3} \right] \quad (xvii)
\]

\[
\dot{\varepsilon}_2 = \frac{12 e^{-D_b^0}}{kT d^3} \left[ \frac{3 \sigma_1^+ - \frac{5}{2} \sigma_2^- \left( \frac{1-0.2f_h}{1-f_h/2} \right) - \frac{\sigma_5}{2} \left( 1-2f_h \right)}{\frac{3}{4} \left( \frac{7}{3} - \frac{f_h}{2} \right) + (1-f_h)^3} \right] \quad (xviii)
\]

Again expressions for the rate of increase of void area fraction can be obtained (with the introduction of a factor of 0.5 into equation (xv) to take into account the fact that the repeating unit only contains half a void):

\[
\dot{\theta}_h = \frac{36/3D_b^0}{kT d^3} \left[ \frac{\sigma_1^+ - \sigma_2^- (1-\frac{f_h}{2})}{(1-\frac{f_h}{2})^3} \right] + \quad (xix)
\]

\[
\frac{18/3 (1-2f_h)}{(1-\frac{f_h}{2}) x_f^0} \frac{D_b^0}{kT d^3} \left[ \frac{3 \sigma_1^+ - \frac{5}{2} \sigma_2^- \left( \frac{1-0.2f_h}{1-f_h/2} \right) - \frac{\sigma_5}{2} \left( 1-2f_h \right)}{\frac{3}{4} \left( \frac{7}{3} - \frac{f_h}{2} \right) + (1-f_h)^3} \right]
\]
5) Analysis of crack-like cavities on an isolated grain boundary.

The result of void growth where surface diffusion is not fast enough to maintain an equilibrium shape is the formation of crack-like cavities. Uniform and non-uniform distributions of such cavities are shown in FIGURE A3(d) and A3(e). Before analysing such distributions the type of analysis can be demonstrated for an isolated grain boundary.

It is assumed that growth of cavities only occurs on boundaries perpendicular to the maximum principal stress \( \sigma_1 \) and that the end of the cavity adjoining boundary 2 remains in the equilibrium shape. As crack growth only occurs at high stress and high void fractions capillarity stress is set at zero for boundary 2 and the crack is assumed to have minimal extension along boundary 2.

To perform the analysis we require expressions for both grain boundary and surface diffusion as both are significant in crack-like cavity growth. Expressions for boundary diffusion have already been given. Expressions for the rate of flow of material along the surface of a cavity have been given by Chuang and Rice [A3]. Volumetric flow of material away from the cavity is:

\[
\frac{dV_f}{dt} = \frac{3D_b \delta_b n}{kT} \frac{\sigma_f - \sigma_f (1-f_b)}{(1-f_b)^2}
\]

(XX)

(where \( \sigma_f \) is the stress normal to the boundary at the tip of the cavity)

and the flow rate towards the tip along the surface is:
\[
\frac{dV}{dt} = \frac{D_s \dot{e}_s \sigma_s^2}{kT/2 \gamma_s} \quad (xxi)
\]

Matter conservation requires that these two expressions are equal, giving:

\[
\sigma_t = \frac{\sigma_s}{2(1-f_h) \Delta_s} \left[ \left(1 + \frac{4\sigma_t}{\sigma_s} \Delta_s \right)^{1/2} - 1 \right]
\]

where \( \sigma_s = \frac{2/3 \gamma_s}{l} \) and \( \Delta_s = \frac{D_s \dot{e}_s}{D_b \dot{e}_b} \) \( (xxii) \)

The velocity of the boundary with respect to the line of symmetry can be derived from these last three equations, giving:

\[
u = \frac{3}{2} \frac{D_s \dot{e}_s \sigma_o}{kT/2 \Delta_s^2 (1-f_h)^2} \left[ \left(1 + \frac{4\sigma_t}{\sigma_s} \Delta_s \right)^{1/2} - 1 \right]^2 \quad (xxiii)
\]

and the rate of growth of the void area fraction is given by:

\[
\frac{dV}{dt} = \frac{1}{r_o} \frac{\dot{V}}{} \quad (xxiv)
\]

where \( r_o \), the crack tip radius, \( = \gamma_s/\sigma_t \) [A3]. This leads to a final expression for the growth rate of voids:

\[
\dot{f}_h = \frac{9}{472} \frac{D_s \dot{e}_s \sigma_o}{kT} \left[ \left(1 + \frac{4\sigma_t}{\sigma_s} \Delta_s \right)^{1/2} - 1 \right]^3 \quad (xxv)
\]

Whilst these equations apply in general, for the following examples it is most realistic to examine the case where \( 4\sigma_t \Delta_s / \sigma_s \) is much larger than 1. This is most realistic in the early stages of growth where \( f_h \) is still small and \( \sigma_o \) is similar in magnitude to the applied stress. In this case \( \Delta_s \),
is generally of the order of 1 [A4] and in general $\sigma_1^m$ is always much larger than $\sigma_2$.

6) Analysis of a uniform distribution of crack-like cavities.

The situation analysed here and shown in FIGURE A3(d) only requires analysis for the section of boundary between $x_1 = 0$ and $x_2 = 0$. For boundary 1 the solution obtained for the local stress and velocity in the previous section applies (equation (xxiii) in the limit where $4\sigma_1^mA_g/\sigma_2$ is much larger than 1). For boundary 2 boundary conditions apply such that:

\[ j_2 = 0 \text{ at } x_2 = 0 \]
\[ \sigma_2 = 0 \text{ at } x_2 = -1/2 \]

Combining these expressions with compatibility and equilibrium relationships gives the relevant expressions for the remote strain rates:

\[ \dot{\epsilon}_1 = \frac{36D_b\delta b\Omega}{kT_d(1-f_h)^3} (\sigma_1 - \sigma_2) + \frac{18D_b\delta b\Omega}{kT_d(1-f_h)^3} (\sigma_1^m + \sigma_2^m) \]  (xxvi)

\[ \dot{\epsilon}_2 = \frac{36D_b\delta b\Omega}{kT_d(1-f_h)^3} (\sigma_1 - \sigma_2) + \frac{18D_b\delta b\Omega}{kT_d(1-f_h)^3} (\sigma_1^m + \sigma_2^m) \]
\[ - \frac{48D_b\delta b\Omega}{kT_d^3} \left[ \frac{1}{(1-f_h)^3} - 1 \right] \sigma_1^m \]  (xxvii)

Combination with the cavity growth rate for an isolated void given in the previous section gives an expression for total cavity growth rate:

\[ \dot{f}_h = \frac{54/3}{/2} \frac{D_b\delta b\Omega [(\sigma_1 - \sigma_2) + (\sigma_1 - \sigma_2)/2]^{1/2}}{kT_d^3\Delta_s^{1/2} (1-f_h)^3 \sigma_s^{1/2}} \]  (xxviii)
7) Analysis of a non-uniform distribution of crack-like cavities.

As before boundary conditions for boundaries 2 and 3 are:

\[ j_3 = 0 \quad \text{at} \quad x_3 = 0 \]
\[ j_2 = -j_3 \quad \text{at} \quad x_2 = \frac{1}{2} \quad \text{and} \quad x_3 = \frac{1}{2} \]
\[ \sigma_2 = \sigma_3 \quad \text{at} \quad x_2 = \frac{1}{2} \quad \text{and} \quad x_3 = \frac{1}{2} \]
\[ \sigma_2 = 0 \quad \text{at} \quad x_2 = -\frac{1}{2} \]

By combination with equation (xxiii), in the limit where \( \sigma_{\text{H}} \gg 1 \), gives strain rate and void growth rate expressions:

\[
\dot{\epsilon}^\text{ex} = \frac{36D_b \delta_k \sigma_0}{kT_d^3} \left[ \frac{11(\sigma_1^2 - \sigma_2^2) + (\sigma_1^2 + \sigma_2^2)}{7 + 4(1-\epsilon_h)^3} \right] \quad \text{(xxix)}
\]

\[
\dot{\epsilon}^\text{ex} = \frac{12D_b \delta_k \sigma_0}{kT_d^3} \sigma_1^2 - \frac{30D_b \delta_k \sigma_0}{kT_d^3} \left[ \frac{11(\sigma_1^2 - \sigma_2^2) + (\sigma_1^2 + \sigma_2^2)}{7 + 4(1-\epsilon_h)^3} \right] \quad \text{(xxx)}
\]

\[
\dot{\epsilon}_h = \frac{54/3}{kT_d^3 \delta_k^{1/2} \sigma_0^{1/2}} \left[ \frac{11(\sigma_1^2 - \sigma_2^2) + (\sigma_1^2 + \sigma_2^2)}{7 + 4(1-\epsilon_h)^3} \right]^{3/2} \quad \text{(xxxi)}
\]

8) Analysis of the growth of a facet sized crack.

The final result of growth of crack like cavities is the formation of full facet cracks. The situation analysed here is shown in FIGURE A3(1) where the cavity maintains an equilibrium profile where it meets boundary 2. The growing crack extends a fraction \( \epsilon_C = r_C/l = \delta/d \) (\( r_C \) = radius of
curvature at the crack end, \( \delta \) is the crack width) of the way along the boundary

The following boundary conditions apply:

\[
\begin{align*}
\sigma_2 &= 0 \quad \text{at} \quad x_2 = -(1/2 - r_c) \\
\sigma_2 &= \sigma_c \quad \text{at} \quad x_2 = x_3 = 1/2 \\
\eta_2 &= -\eta_3 \quad \text{at} \quad x_2 = x_3 = 1/2 \\
\eta_3 &= 0 \quad \text{at} \quad x_3 = 0
\end{align*}
\]

The following strain rate expressions are obtained as before:

\[
\begin{align*}
\dot{\gamma} &= \frac{12D_0}{KTd^3(1-f_c)(7/3 - f_c)} [(11-8f_c)(\sigma \dot{T}-\dot{\sigma})+(1-4f_c)(\sigma \dot{T}+\dot{\sigma})] \\
\dot{\eta} &= \frac{12D_0}{KTd^3(1-f_c)} \frac{\sigma \dot{T}}{kT^2d^2(1-f_c)(7/3 - f_c)} \\
& \quad \times [(11-8f_c)(\sigma \dot{T}-\dot{\sigma})+(1-4f_c)(\sigma \dot{T}+\dot{\sigma})]
\end{align*}
\]

and an expression for the crack opening rate is also obtained:

\[
\dot{\delta} = \frac{48D_0}{(1-f_c)^2KTd^2(1+4.2f_c)} \left[ \frac{3}{4} (1-\sqrt{3/8}f_c) \sigma \dot{T} \\
+ \frac{1/4-f_c}{(7/3-f_c)} ((11-8f_c)(\sigma \dot{T}-\dot{\sigma})+(1-4f_c)(\sigma \dot{T}+\dot{\sigma})) \right]
\]
REFERENCES TO APPENDIX A


APPENDIX B : PHYSICAL AND MECHANICAL PROPERTIES OF ALUMINA

The manufacturers of the alumina tube used for test specimens (Degussa Gmbh, Germany) supply a full listing of mechanical and thermal properties for the grade of alumina used. The values quoted are given in TABLE B1. No attempt was made to verify any of these values. Sections were cut from the ends of failed test specimens and polished prior to being photographed under both the optical and the scanning electron microscope. In addition the thermal etch procedure detailed in chapter 15.4 was used to reveal the grain structure of the alumina under the scanning electron microscope. The micrographs obtained from the optical microscope are shown in FIGURE B2. Magnifications of X50 and X200 were used and appropriate scale markers are shown on the micrographs. Both photographs were taken prior to the thermal etch treatment and show the extent of porosity in the material. The largest porosity is of the order of 30 microns. After the thermal etch treatment the samples were examined in the scanning electron microscope. The micrographs obtained at magnifications of X1050 and X2100 are shown in FIGURE B3. These clearly show the grain structure of the material and the location of pores within this structure. The grain size varies between approximately 3 and 20 microns, with the average size being approximately 8 microns, as determined by the direct measurement of the long axis of the grains. The porosity is most commonly located at triple grain junctions. In addition some pores trapped within the actual grains can be seen. This latter
## TABLE B1: PROPERTIES OF ALUMINA

### PHYSICAL PROPERTIES

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>DENSITY</th>
<th>POISSONS RATIO</th>
</tr>
</thead>
<tbody>
<tr>
<td>UNIT(S)</td>
<td>g/cm³</td>
<td>DIMENSIONLESS</td>
</tr>
<tr>
<td>VALUE</td>
<td>3.9</td>
<td>0.22</td>
</tr>
</tbody>
</table>

### MECHANICAL PROPERTIES

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>4 POINT BEND STRENGTH</th>
<th>TENSILE MODULUS</th>
<th>COMRESSIVE STRENGTH</th>
<th>FRACTURE TOUGHNESS (K1C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEMPERATURE</td>
<td>20 DEGREES C</td>
<td>20 DEGREES C</td>
<td>20 DEGREES C</td>
<td>20 DEGREES C</td>
</tr>
<tr>
<td>UNIT(S)</td>
<td>MPa</td>
<td>GPa</td>
<td>MPa</td>
<td>MPam1/2</td>
</tr>
<tr>
<td>VALUE</td>
<td>290</td>
<td>350</td>
<td>2000</td>
<td>4.9</td>
</tr>
</tbody>
</table>

### THERMAL PROPERTIES

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>THERMAL CONDUCTIVITY</th>
<th>SPECIFIC HEAT</th>
<th>THERMAL EXPANSION COEFFICIENT</th>
<th>THERMAL EXPANSION COEFFICIENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEMPERATURE</td>
<td>20 DEGREES C</td>
<td>N/A</td>
<td>20 - 800 DEGREES C</td>
<td>800 - 1000 DEGREES C</td>
</tr>
<tr>
<td>UNIT(S)</td>
<td>W/m.K</td>
<td>J/kg.DEGREE C</td>
<td>10 E-6/ DEGREES C</td>
<td>10 E-6/ DEGREES C</td>
</tr>
<tr>
<td>VALUE</td>
<td>24</td>
<td>900</td>
<td>7.3</td>
<td>8.9</td>
</tr>
</tbody>
</table>
FIGURE B2: OPTICAL MICROGRAPHS OF POLISHED SECTION OF ALUMINA
FIGURE B3: SCANNING ELECTRON MICROGRAPHS OF POLISHED AND ETCHED SECTION OF ALUMINA
type of porosity is a typical feature of sintered microstructures.
APPENDIX C: PHYSICAL AND MECHANICAL PROPERTIES OF REACTION BONDED SILICON NITRIDE

The reaction bonded silicon nitride tubes supplied by Turner & Newall Ltd were made from their REFEL grade material. They were supplied with a full listing of mechanical and thermal properties. This is given in TABLE C1. These properties were not checked but were taken as correct. This material is the same as that used by Kandil and Dyson [62] however they used long rod shaped test pieces. They quote the values given in TABLE C1 and in addition they evaluated the room temperature strength of their samples using their creep apparatus. They quote a value of 123 MPa failure stress.

Samples from the ends of tested tubes were polished and examined under both optical and scanning electron microscopes to show the undamaged microstructure of the material. FIGURE C2 shows the optical micrographs obtained at two magnifications (X50 and X200). Both photographs indicate the high degree of porosity in the material. Some areas of higher density can also be seen. These features are shown more clearly in the scanning electron microscope (SEM) micrographs contained in FIGURE C3. These were taken at magnifications of X210, X700 and X2100. Again dense areas of material can be seen surrounded by a large amount of porosity. As discussed in chapter 5 this type of microstructure is typical of reaction bonded silicon nitride. The dense areas are the beta phase nitride material. No obvious signs of the typical fine needle like
TABLE C1: PROPERTIES OF REACTION BONDED SILICON NITRIDE

**PHYSICAL PROPERTIES**

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>DENSITY</th>
<th>OPEN POROSITY</th>
<th>POISSONS RATIO</th>
</tr>
</thead>
<tbody>
<tr>
<td>UNIT(S)</td>
<td>g/cm³</td>
<td>%</td>
<td>DIMENSIONLESS</td>
</tr>
<tr>
<td>VALUE</td>
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<td>25</td>
<td>0.27</td>
</tr>
</tbody>
</table>

**MECHANICAL PROPERTIES**

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>3 POINT BEND STRENGTH</th>
<th>3 POINT BEND STRENGTH</th>
<th>TENSILE MODULUS</th>
<th>COMRESSIVE STRENGTH</th>
<th>FRACTURE TOUGHNESS (K1C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEMPERATURE</td>
<td>20 DEGREES C</td>
<td>1000 DEGREES C</td>
<td>20 DEGREES C</td>
<td>20 DEGREES C</td>
<td>20 DEGREES C</td>
</tr>
<tr>
<td>UNIT(S)</td>
<td>MPa</td>
<td>MPa</td>
<td>GPa</td>
<td>MPa</td>
<td>MPam1/2</td>
</tr>
<tr>
<td>VALUE</td>
<td>190</td>
<td>190</td>
<td>170</td>
<td>550</td>
<td>3</td>
</tr>
</tbody>
</table>

**THERMAL PROPERTIES**

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>THERMAL CONDUCTIVITY</th>
<th>SPECIFIC HEAT</th>
<th>THERMAL EXPANSION COEFFICIENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEMPERATURE</td>
<td>20 DEGREES C</td>
<td>N/A</td>
<td>20 - 800 DEGREES C</td>
</tr>
<tr>
<td>UNIT(S)</td>
<td>W/m.K</td>
<td>J/kg.DEGREE C</td>
<td>10 E-6/ DEGREES C</td>
</tr>
<tr>
<td>VALUE</td>
<td>16</td>
<td>800</td>
<td>3</td>
</tr>
</tbody>
</table>
FIGURE C2: OPTICAL MICROGRAPHS OF POLISHED SECTION OF REACTION BONDED SILICON NITRIDE
FIGURE C3: SCANNING ELECTRON MICROGRAPHS OF POLISHED SECTION OF REACTION BONDED SILICON NITRIDE
alpha phase could be seen at these magnifications. This would indicate that if it is present the grain size of the alpha material is less than 1 micron. No information as to the ratio of alpha and beta phases in the material was supplied by the manufacturer and a determination of this ratio was not attempted.
APPENDIX D : DRAWINGS OF GRIP SYSTEM COMPONENTS

The following pages contain drawings of the components of the grip developed to hold ceramic tubes in the tension torsion test equipment. A drawing of the cooling plate that is attached to the grip face is also included. The drawings are not to scale. Each drawing is labelled with an item number. A description of the function of each item, along with a photograph of the parts in assembled and disassembled form are given in FIGURE 37.
ITEM 3: GRIP BODY  MATERIAL: EN24

ITEM 4: GRIP BODY CAP  MATERIAL: EN24
ITEM 5: INNER RETAINING RING MATERIAL: EN24
ITEM 6: OUTER RETAINING RING MATERIAL: EN24
ITEM 10 | PULL ROD MATERIAL | EN24
COOLING PLATE: MATERIAL = COPPER

COOLING PLATE FIXING PIECE (x4)
MATERIAL = TUFFNEL