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Diffusion creep in oxide ceramics

Atul H. Chokshi

Department of Metallurgy, Indian Institute of Science, Bangalore 560 012, India

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Abstract

Although the models for diffusion creep were developed over 50 years ago, recently there has been considerable debate in the literature regarding the experimental validity of the models. This report summarizes recent theoretical developments in diffusion creep, with specific emphasis on ceramics. Concomitant microstructural changes during high temperature deformation, such as grain growth and cavitation, frequently interfere with the identification of the rate controlling process. Recent careful experiments coupled with appropriate microstructural characterization will be reviewed in structural oxide ceramics such as alumina and tetragonal zirconia; it will be demonstrated that these oxides deform by diffusion creep. Detailed analysis of the data in tetragonal zirconia will also be utilized to demonstrate the significance of diffusion creep in superplastic ceramics.

Keywords: Diffusion creep; Grain boundary sliding; Grain growth; Grain boundary diffusion; Superplasticity

1. Introduction

Ceramics are being considered increasingly for structural applications at high temperatures. Under these conditions, the time dependent plastic deformation of materials, termed creep, is an important design criterion. High temperature plastic deformation in materials may occur by intragranular dislocation motion, stress directed diffusion creep or grain boundary sliding.[1] In some ceramics containing a significant amount of glassy phase, deformation may also occur by solution–precipitation[2] or void formation.[3] The rate controlling deformation process is generally determined from a comparison of the experimental mechanical characteristics with theoretical models and appropriate microstructural characterization.

The high temperature mechanical behavior of materials is usually expressed in the form of the following equation:

$$\dot{\varepsilon} = \frac{ADGb}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n \qquad (1)$$

where $\dot{\varepsilon}$ is the steady state strain rate, A is a dimensionless constant, G is the shear modulus, b is the magnitude of the Burgers vector, k is Boltzmann's constant, T is the absolute temperature, d is the grain size, p is the inverse grain size exponent, σ is the imposed stress, and n is the stress exponent. The diffusion coefficient may be expressed as $D=D_0\exp(-Q/RT)$, where D_0 is the frequency factor, Q is the activation energy, and R is the gas constant. For intragranular dislocation creep processes, $p=0$; for diffusion and grain boundary sliding processes which depend on the grain size, $p>0$.

Although the models for diffusion creep were established more than 50 years ago,[4–6] and they have been sufficiently well accepted to be part of textbooks,[7,8] over the last 15 years there has been a vibrant debate in the literature regarding the existence of diffusion creep because of apparent discrepancies in the theoretical and experimental creep rates and microstructures.[9–12] In contrast to metallic alloys, there are many reports of diffusion creep in ceramics due to the difficulty in enabling dislocation motion and the finer grain sizes in ceramics.[1,13]

This report briefly reviews the mechanical characteristics and microstructural aspects of diffusion creep in ceramics, and then discusses critically some recent data on diffusion creep in oxide ceramics. It is shown that there is good experimental evidence for diffusion creep in oxide ceramics, and there is also good support for the important role of diffusion creep in large strain superplastic deformation.

E-mail address: achokshi@metalrg.iisc.ernet.in (A.H. Chokshi).

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2. Models for diffusion creep

The standard model for diffusion creep consider some grain boundaries subject to tension and others subject to compression. The tensile stress assists in vacancy creation so that there is an increase in the vacancy concentration on boundaries experiencing tension, and a corresponding decrease in the vacancy concentration on the boundaries experiencing compression; the vacancy concentration gradient sets up a flux of vacancies. When vacancy flow occurs through the matrix, the process is referred to as Nabarro–Herring[4,5] diffusion creep with the creep parameters of $n=1$, $p=2$ and $Q=Q_l$, where Q_l is the activation energy for lattice diffusion. When vacancy flow occurs along grain boundaries, the process is referred to as Coble[6] creep with the creep parameters of $n=1$, $p=3$ and $Q=Q_{gb}$, where Q_{gb} is the activation energy for grain boundary diffusion. In ultra-fine grained nanocrystalline materials,[13] there is an additional possibility of diffusion along triple lines, with the creep parameters of $n=1$, $p=4$ and $Q=Q_{tp}$, where Q_{tp} is the activation energy for creep along triple lines. As noted elsewhere,[13] it is possible to rationalize the grain size dependence for the various diffusion paths as follows. The diffusion creep rate $\dot{\varepsilon}_d$ can be expressed as

$$\dot{\varepsilon}_d \propto A_v/d^4 \tag{2}$$

where A_v is the cross-sectional area available for diffusion flow. Since Nabarro–Herring, Coble and Triple-Point diffusion are associated with diffusional flux cross-sectional areas of $\sim d^2$, δd and δ^2, the corresponding values of p are 2, 3 and 4. It is to be noted that triple-point diffusion creep is likely to be important only in materials with a grain size of a few nanometers, and it is not considered in the remainder of this paper.

Since lattice and grain boundaries are independent paths for vacancy diffusion, creep is controlled by the faster mechanism. Fig. 1 illustrates schematically, for a fixed grain size, the variation in creep rate with T/T_m where T_m is the absolute melting temperature, for Nabarro–Herring and Coble creep. Since the activation energy for grain boundary diffusion is about half the value for lattice diffusion in metals, Fig. 1 indicates that Nabarro–Herring creep will be important at higher temperatures whereas Coble creep will be dominant at lower temperatures. In a similar manner, at a fixed temperature, Coble creep will be more important at finer grain sizes.

2.1. The coupling of diffusion fluxes in ceramics

In ceramics, it is necessary to modify the above picture to take into account the transport of the anions and cations during diffusion creep. The conventional means of coupling the diffusion fluxes, suggested originally by Gordon,[14] requires the total transport of vacancies from the horizontal to the vertical boundaries (Fig. 2) to be in the appropriate stoichiometric ratio. This leads to the prediction that creep in this case is controlled by the

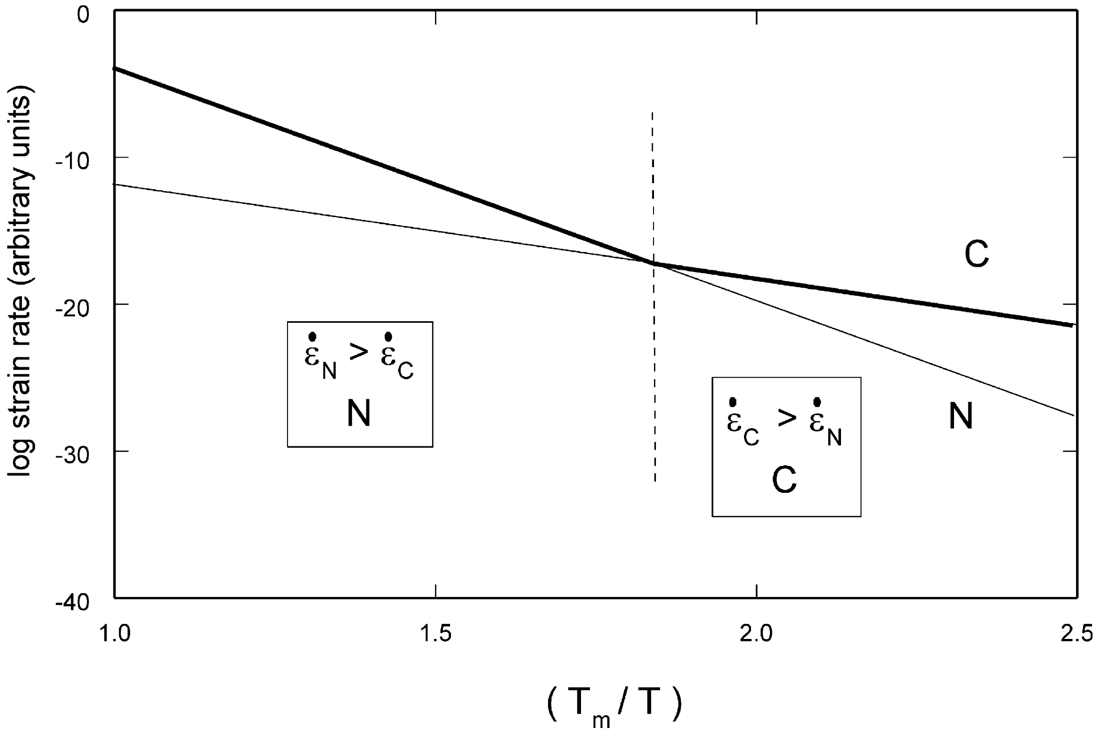

Fig. 1. Schematic illustration of a transition from Coble creep to Nabarro–Herring creep with an increase in temperature.

diffusion of the slower moving species along the faster diffusion path. In this scenario, it is possible for the cations and anions to be transported predominantly along different paths as depicted in Fig. 2a.

However, as noted by Dominguez-Rodriguez and Castaing,[15] the above scenario will lead to local non-stoichiometry which is not observed. Consequently, it may be more appropriate to constrain diffusion fluxes along each path to be in the appropriate stoichiometric ratio, as depicted schematically in Fig. 2b. In this scenario, it is necessary to find the slower moving species along each path, and the rate controlling process is then determined from the faster diffusion path.

Fig. 3 illustrates schematically for a ceramic $A_\alpha B_\beta$ the divergence in predictions by the above two different coupling procedures, with plots of the variation in strain rate with T_m/T, for a fixed grain size.[16] In these figures the symbols C and N represent Coble and Nabarro–Herring creep, and the superscript $^+$ and $^-$ represent cation and anion, respectively. Thus, Fig. 3a, corresponding to Fig. 2a, indicates that there will be transitions with an increase in temperature from diffusion creep controlled by cation grain boundary diffusion (C^+) to cation lattice diffusion (N^+) to anion grain boundary diffusion (C^-) to anion lattice diffusion (N^-). On the other hand Fig. 3b, corresponding to Fig. 2b, indicates that over the same temperature range, there will only be a single transition from Coble creep controlled by cation grain boundary diffusion to Nabarro–Herring creep controlled by anion lattice diffusion.

Clearly, the two different procedures for coupling diffusion fluxes lead to substantially different predictions. Unfortunately, the lack of reliable and complete diffusion data precludes a critical experimental validation of the appropriate diffusion coupling.

2.2. Microstructural aspects of diffusion creep

The standard model for diffusion creep considers the plating of matter to boundaries experiencing tension, which will lead to an elongation of grain along the tensile axis. Analyses of changes in grain shape during diffusion creep indicate that grain boundary sliding and grain elongation are both an integral part of diffusion creep. As noted by Raj and Ashby[17] and others,[18] the overall deformation process can be construed as diffusion creep accommodated by grain boundary sliding or grain boundary sliding accommodated by diffusion creep.

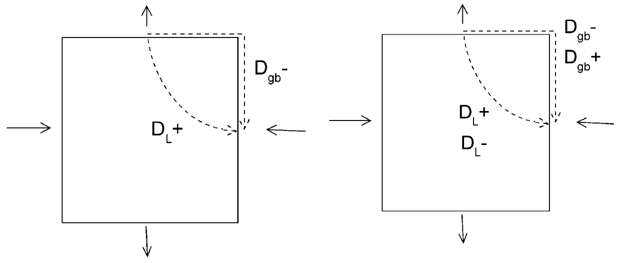

Fig. 2. Schematic illustration of two alternate procedures for coupling diffusion fluxes in ceramics: (a) the total flux from the horizontal boundaries to the vertical boundaries is in the appropriate stoichiometric ratio, (b) the flux along each transport path is in the appropriate stoichiometric ratio.

In a material containing particles along grain boundaries and the matrix, the diffusion of matrix elements from the compression to the tension boundaries will lead to a dispersion free zone along the tensile boundaries. Although there have been reports of the formation of such zones in a Mg alloy,[19] recent analysis has challenged the validity of these observations since the experimental conditions apparently corresponded to a dislocation creep rather than a diffusion creep regime.[20] There have not been such observations in ceramics, owing largely perhaps to the fine grain sizes of these materials so that second phase particles frequently have similar dimensions as the matrix grains.

2.3. Limitations of diffusion creep

The overall process of diffusion creep involves the creation of excess vacancies at the horizontal boundaries, the transport of the vacancies to the vertical boundaries, and their annihilation at the vertical boundaries (Fig. 2). The standard models for diffusion creep considered above assume that vacancy creation and annihilation are relatively easy processes that occur readily, so that creep is controlled by the diffusion of vacancies. Clearly, vacancy creation, transport and annihilation are sequential processes, in which the slower one will be rate controlling. When interfaces do not act as perfect sources and sinks for vacancies, the process then becomes interface controlled,[21–23] and the creep rate will be slower than that predicted by the standard Nabarro–Herring and Coble models. There are various models available for interface controlled diffusion creep, and they generally predict a stress exponent greater than one, and an inverse grain size exponent <3. In general, because of the sequential nature of the process, interface controlled diffusion creep becomes important in finer-grained materials being deformed at low stresses.

3. Experimental data on oxide ceramics

Diffusion creep has been reported in a large number of oxide ceramics such as alumina,[24] cubic zirconia,[25] magnesium oxide,[26] iron oxide,[27] uranium dioxide[28] and nickel oxide.[29] However, inspection of the available data reveals several discrepancies. Thus, for example, while the earlier report by Dimos and Kohlstedt[25] on 25 mol%

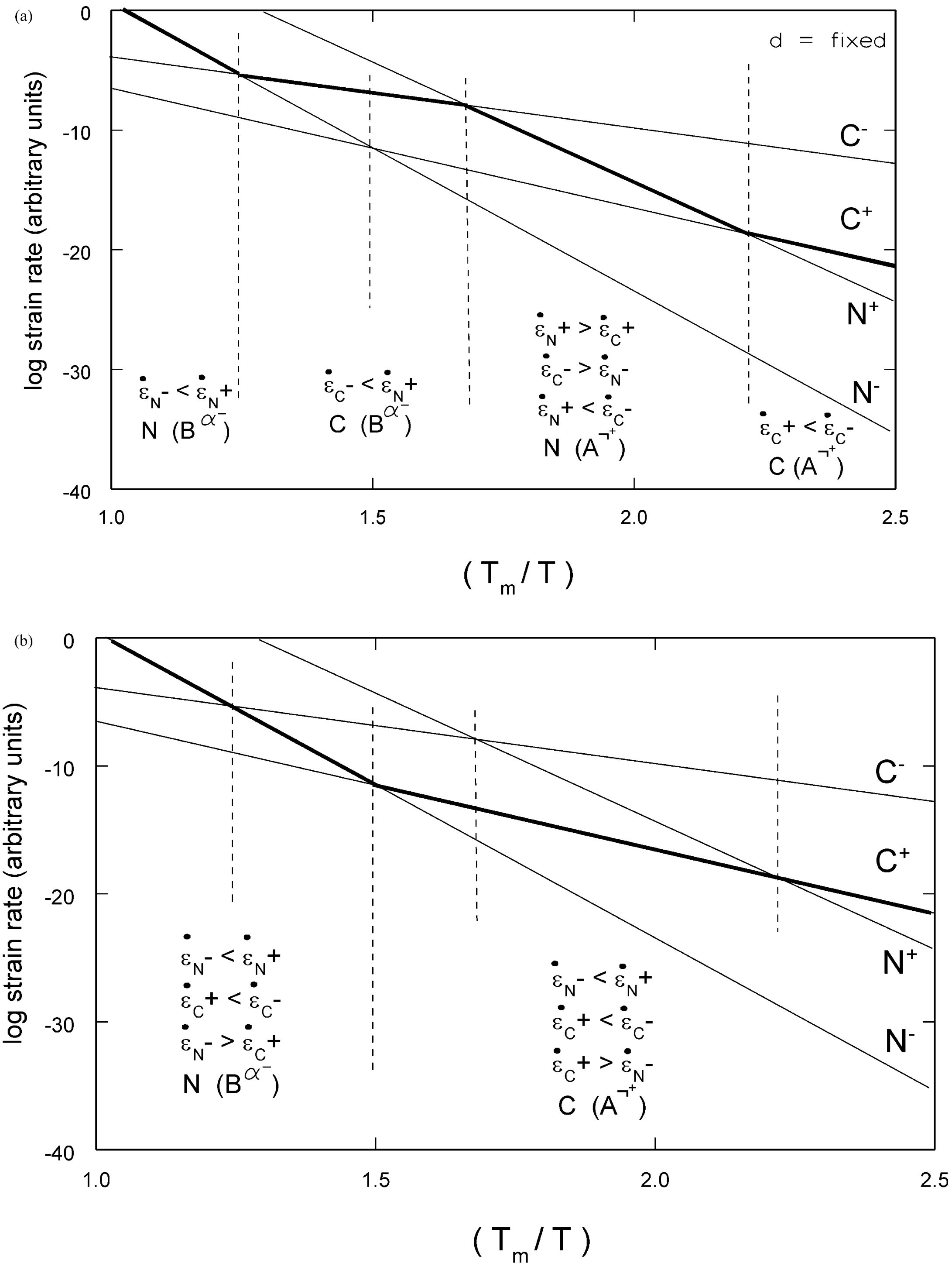

Fig. 3. Schematic illustration showing possible transitions in deformation mechanisms for (a) situation corresponding to Fig. 2a and (b) situation corresponding to Fig. 2b.

yttria stabilized cubic zirconia yielded a stress exponent of ∼1, the later study by Wakai et al.[30] on 8 mol% yttria stabilized cubic zirconia yielded a stress exponent of ∼2. In order to keep this report tractable, the discussion in this section is limited to recent experimental data on (a) tensile deformation in alumina, and (b) role of diffusion creep in superplastic tetragonal zirconia.

3.1. Tensile deformation in alumina

Although alumina has been a popular model material for studying high temperature deformation in ceramics, with the first report being as early as in 1950,[31] a critical review of the literature indicates that most of the early studies on alumina were performed in compression and bending. It has been demonstrated that there are several complications associated with tests in bending,[32] so that the analysis of the results is not always clear. The detailed review by Cannon and Coble[24] concluded that creep occurred by an interface controlled diffusion process, with some evidence of a transition to diffusion creep at higher stresses.

Although the first study on tensile creep in alumina was conducted about 30 years ago by Davies and Sinha Ray,[33] there was not much interest in pursuing this aspect due to the apparently limited ductility of alumina and the difficulty in conducting tensile tests. However, the recent reports of superplasticity in tetragonal zirconia have revived interest in examining the tensile deformation characteristics of alumina and there have been several studies on this topic since 1988. A review of the literature indicates that there are several inconsistencies in the data reported from tensile deformation.[34] Thus, for example, while some studies reported a value of n close to one other studies reported a value close to 2. The grain size dependence of deformation has not been studied in much detail. Microstructural examination following tensile deformation has been rather limited; except in cases where the initial grain size is very large (> 10 μm) or the testing temperature has been low, grain growth has been observed whenever such measurements have been undertaken. Although the influence of grain growth on the stress exponent and activation energy has been recognized, the appropriate correction for concurrent grain growth has not been included in most studies.

Recently, a detailed study was undertaken[34] for the tensile and compressive deformation in polycrystalline alumina doped with magnesia with grain sizes $\geqslant 2$ μm; in tensile deformation, a maximum elongation of 44% was obtained under optimum conditions. Microstructural examination of the specimens after deformation revealed the occurrence of significant grain growth and some cavitation; however, the grains retained their equiaxed shapes, as shown in Fig. 4.

A complete analysis of the data incorporating the influence of grain growth revealed that the experimental results in tension and compression, were consistent with the true value of the creep parameters as $n \sim 1$, $p \sim 3$ and $Q \sim 520$ kJ mol^{-1}, as illustrated in the normalized plot in Fig. 5. It is to be noted that in the absence of correction for grain growth, the raw data yielded stress exponents of up to ~2 both in polycrystalline alumina and a companion study on a 8 mol% yttria stabilized cubic zirconia.[35] There is a slight difference of a factor of ~2 in the mechanical data in tension and compression which could not be accounted for by variations in concurrent cavitation and friction. A similar difference of a factor of ~2 in tension and compression data was also observed in alumina tested earlier by Robertson et al.[36] and tetragonal zirconia tested by Wakai et al.[37]

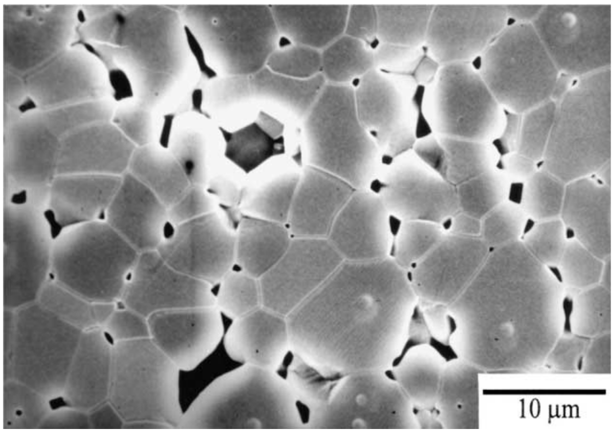

Fig. 4. Scanning electron micrograph showing the retention of an equiaxed microstructure and concurrent cavitation after significant tensile deformation (21%) in polycrystalline alumina.

The mechanical characteristics of alumina are generally consistent with the occurrence of Coble diffusion creep. Unfortunately, as noted elsewhere,[34] there is not sufficient information available on diffusion in alumina; the available information exhibits substantial variation over several orders of magnitude. Fig. 6 shows the experimental data obtained at 1823 K together with the theoretical predictions for Coble creep using the activation energy for Al grain boundary difffusion from an earlier analysis by Cannon and Coble[24] and the oxygen grain boundary diffusion value reported by Prot et al.[38]

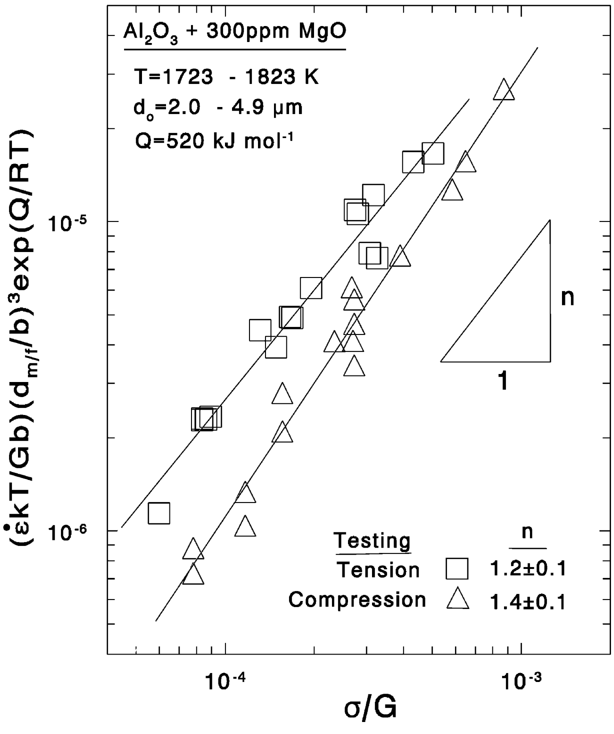

Fig. 5. Normalized plot of the variation in strain rate with stress for polycrystalline alumina tested in tension and compression.

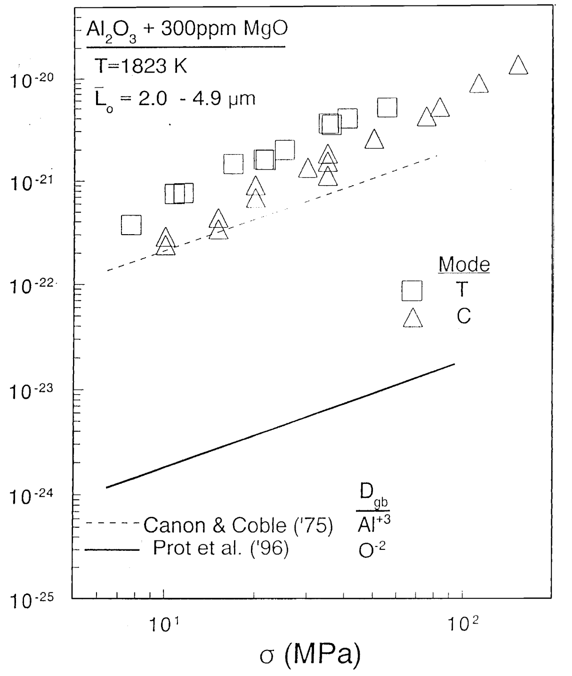

Fig. 6. Comparison of the tensile and compressive experimental data in alumina with theoretical predictions of Coble creep using reported data for Al and O diffusion.

Since the activation energy for Al grain boundary diffusion was obtained from early creep data by Cannon and Coble, Fig. 6 essentially indicates that the present experimental results are in good agreement with the earlier data on alumina.

Although the mechanical data are consistent with Coble diffusion creep, the microstructural observation of the retention of an equiaxed grain size after substantial plastic deformation is apparently in conflict with the expectation of grain elongation accompanying diffusion creep. This conflict is resolved when it is noted that there is significant grain growth, and as shown schematically in Fig. 7, grain growth will lead to grain switching which is a process that enables the maintenance of an equiaxed grain size during diffusion creep.[39,40]

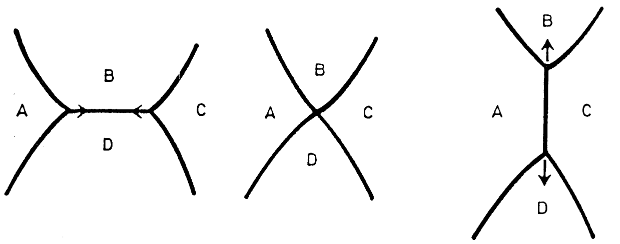

Fig. 7. Schematic illustration of grain switching accompanying grain growth; the grain boundary curvatures shown in (a) drive the triple points towards each other, forming a quadruple junction (b) which splits into two triple junctions (c).

3.2. *Role of diffusion creep in the superplastic deformation on tetragonal zirconia*

Following the initial report of large ductility in the 3 mol% yttria stabilized tetragonal zirconia by Wakai et al.,[41] there have been numerous studies on the material[42–44] and deformation has been attributed variously to mechanisms such as grain boundary sliding,[41] dislocation creep,[45] interface reaction controlled diffusion creep,[46,47] diffusion creep,[48] and grain boundary sliding with a threshold stress.[44] One of the key observations on the material is that the mechanical characteristics are very sensitive to the presence of trace impurities.[42–44,49] Although early studies speculated on the variations in deformation characteristics arising from the presence of amorphous phases at grain boundaries, recent detailed studies have shown that there are variations in deformation behavior even in the absence of an amorphous grain boundary phase.[50–52]

Fig. 8 illustrates the variation in strain rate with stress from some studies on 3YTZ. It is interesting to note that when experimental data are obtained over a sufficiently wide range of experimental conditions, there appears to be a transition from a stress exponent of ~2 at high stresses to a value of ~3 at low stresses.[53] Furthermore, the earlier data exhibiting $n \sim 2$ fall on an extrapolation of the recent data, suggesting that the transition from $n \sim 2$ to $n \sim 3$ is sensitive to impurity content such that transition is shifted to lower stresses with an increase in the impurity content.[41,45,53–55] Later studies have demonstrated that variations in trace levels

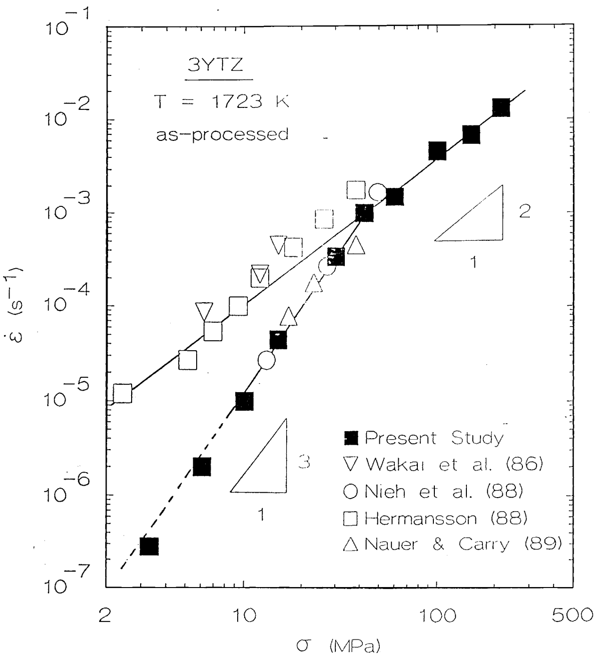

Fig. 8. Variation in the strain rate with stress from several investigation on 3YTZ depicting a transition in stress exponent from $n \sim 2$ at high stresses to $n \sim 3$ at low stresses.

of Al (from 10 to 100 ppm range) are responsible for the shift in the transition from $n\sim2$ to $n\sim3$.[50,51]

As shown schematically in Fig. 9, there are four possible causes for the observed transition in stress exponents: (a) grain growth, (b) operation of two sequential processes, (c) grain boundary sliding with a threshold stress, and (d) interface controlled diffusion creep.

Since $\dot{\varepsilon} \propto d^{-p}$, where $p>1$ for processes involving grain boundaries, it is clear that concurrent grain growth will lead to a decrease in strain rate (at a constant stress) and an apparent increase in the stress exponent at lower stresses where grain growth is more pronounced. However, analysis of the available data indicates that there is a transition to a higher stress exponent even in the absence of grain growth.

The operation of two sequential processes such as grain boundary sliding ($n=2$) and interface controlled grain boundary sliding ($n=3$), such that the slower process is rate controlling, would be consistent with the experimental observations.[52] However, in view of a lack of any theoretical models, it is not possible to critically evaluate this approach.

3.2.1. Threshold stress approach

Jimenez-Melendo and colleagues[44,56] have examined superplasticity in tetragonal zirconia in terms of grain boundary sliding ($n=2$) with a threshold stress, in a manner similar to superplastic metallic alloys. The threshold stress is usually evaluated from a linear plot of $(\dot{\varepsilon})^{1/2}$ vs σ; the extrapolation of the data to zero strain rate gives the value of the threshold stress. However, there are some inconsistencies with this approach. First, analysis yields a negative threshold stress,[53,57] which is meaningless. Although it may be possible to obtain a positive threshold stress by neglecting some higher stress data, the process of selecting which data to neglect becomes arbitrary and subjective. Secondly, as shown schematically in Fig. 10, the influence of impurity content on the transition from $n=2$ to a higher value is different for superplastic metals and ceramics.[58] In superplastic metals, an increase in the impurity content leads to an increase in the transition stress; this is related to a possible solute drag process for movement of grain boundary dislocations. In contrast, for superplastic ceramics, an increase in the impurity content leads to a decrease in the transition stress such that a region with a high stress exponent is not observed in material with a sufficiently high impurity content. This analysis suggests that it is not possible to adopt directly the approach for superplastic metals in superplastic ceramics, although both groups of materials exhibit a similar phenomenological transition from $n\sim2$ to a higher value at low stress.

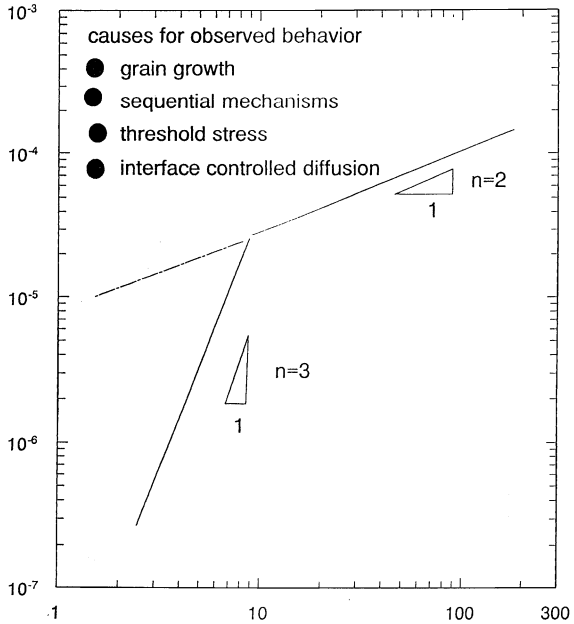

Fig. 9. Schematic illustration of the possible causes for the observed transition in stress exponents.

3.2.2. Diffusion creep approach

Wakai and colleagues[46,47] had proposed interface controlled diffusion creep as a rate controlling process for deformation in 3YTZ. A recent report by Berbon and Langdon[59] has revived interest in this possibility.

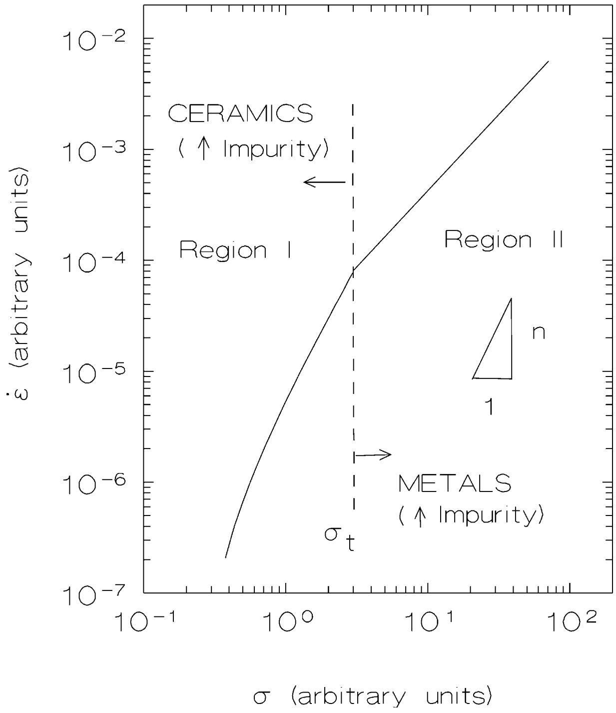

Fig. 10. Schematic illustration depicting the phenomenological similarity in the transitions in stress exponent in superplastic metals and ceramics; note, however, that with an increase in impurity content, the transition is shifted to higher stresses in metals and lower stresses in ceramics.

In contrast to the continuum model shown schematically in Fig. 2, the model by Arzt et al.[60] allows for the creation and annihilation of vacancies only at discrete sites such as grain boundary dislocations. The analysis by Arzt et al.[60] indicates that the creep rate may be expressed by the following expression:

$$\dot{\varepsilon}_{\mathrm{A}} \propto \frac{\sigma D_{\mathrm{gb}}}{d^3}\left(\frac{N^2}{N^2+1/2}\right) \tag{3}$$

where N is the number of grain boundary dislocations in a grain boundary.

For a coarse grained material, where N is large, the model by Arzt et al.[60] reduces essentially to the predictions of the Coble[6] creep rate. However, for fine grained materials, at sufficiently low stresses, the model predicts a reduction in creep rate below the Coble creep rate, and $\dot{\varepsilon} \propto \sigma^3 D_{\mathrm{gb}}/d$. Berbon and Langdon[59] demonstrated that such an approach was consistent with experimental data of the form shown in Fig. 8.

However, there is considerable uncertainty on the appropriate value for grain boundary diffusion in 3YTZ. Therefore, it is desirable to develop an alternative critical means of evaluating the possibility of interface controlled diffusion creep. An analysis of the model by Arzt et al.[60] indicated that diffusion creep should be favored at high stresses in materials with coarse grain sizes.[39]

A study was completed recently to critically evaluate interface controlled diffusion creep in 3YTZ.[61] Some of the experimental data from this study are shown in Fig. 11 in the form of the variation in strain rate with stress for specimens tested with an initial linear intercept grain sizes of ~0.37, 0.61, 0.81 and 1.2 μm, respectively. The experimental results show clearly for the first time transitions with a decrease in stress from a grain size independent region with n~7 to a region with n~1 and finally a region with n~2. A region with n~3 was not observed clearly due either to the higher stresses utilized in the study or the larger impurity content of the material. The unambiguous observation of a region with n~1 at high stresses provides clear evidence for interface controlled diffusion creep as the rate controlling process for superplasticity in 3YTZ.

Fig. 12 shows a comparison of the present experimental results for the n~1 region with the theoretical Coble creep rate using the grain boundary diffusion coefficient for CeO_2 stabilized tetragonal zirconia. The experimental data are in very good agreement with the theoretical model, again confirming the validity of the present approach.

In spite of the good agreement between theory and predictions, it is necessary to note some of the limitations of the model by Arzt et al.[60] The model predicts that there is an apparent region with n~2 only as a transition from n=1 to n=3; this is in contrast with the experimental observations of extended regions with

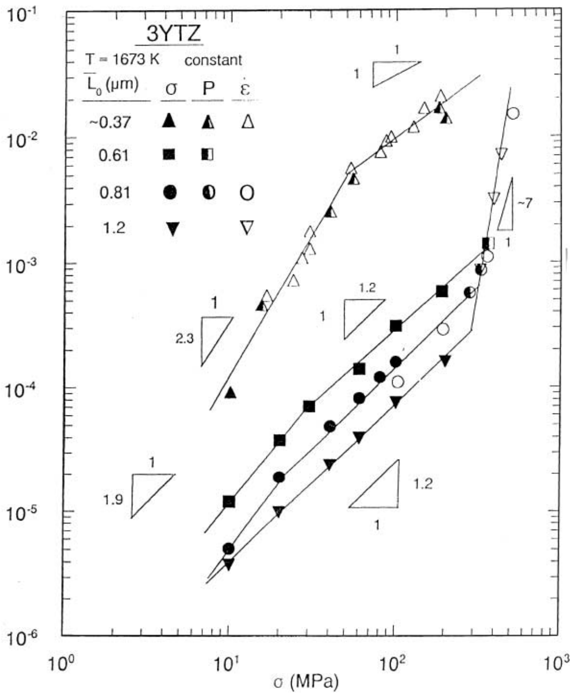

Fig. 11. Variation in strain rate with stress for 3YTZ tested over a wide range of experimental conditions, depicting transitions with a decrease in stress from n~7 to n~1 to n~2.

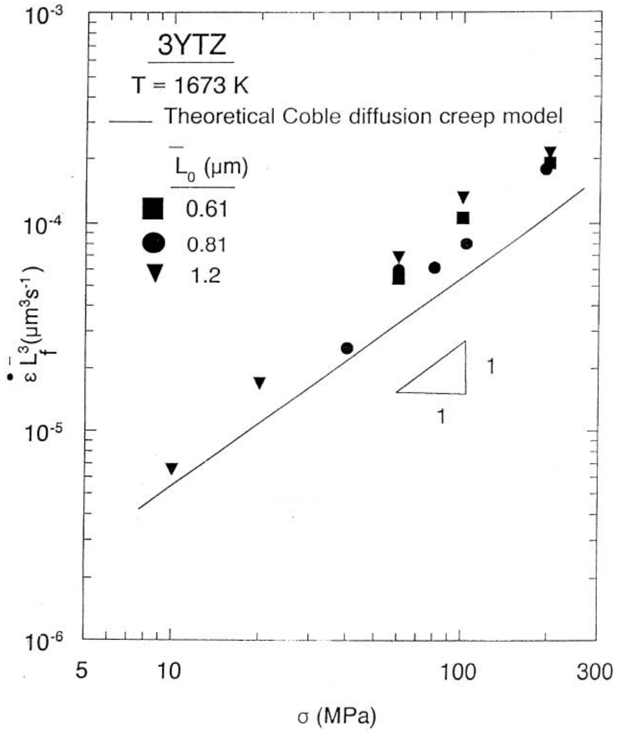

Fig. 12. Comparison of the experimental data in the n~1 region with theoretical predictions of Coble diffusion creep.

n~2 in 3YTZ. It is also noted that there are no terms in the creep rate expression reported by Arzt et al.[60] that consider the role of impurities in modifying the transitions in stress exponent. Consequently, although the present analysis is consistent generally with superplasticity being controlled by interface controlled diffusion creep, it is necessary to develop additional models for interface controlled diffusion creep with reference to 3YTZ.

The experimental data shown in Fig. 11 demonstrate for the first time clear evidence for a transition at high stresses to a grain size independent region, in a manner similar to superplastic metallic alloys. In superplastic metallic alloys, the transition is usually attributed to the advent of intragranular dislocation slip.[62] Fig. 13 shows some of the experimental data from the present study together with data from dislocation flow controlled deformation of single crystalline cubic zirconia.[63] Also shown in Fig. 13 is the steady state flow stress reported recently for the deformation of single crystalline tetragonal zirconia deformed at 1667 K.[64] There is good agreement between the observed transition to a region with $n \sim 7$ and an extrapolation of the data from single crystalline zirconia, suggesting thereby that the high stress region arises from intragranular dislocation flow. Rapid fracture in the high stress experiments precluded the examination the microstructures of deformed samples for intragranular dislocation activity.

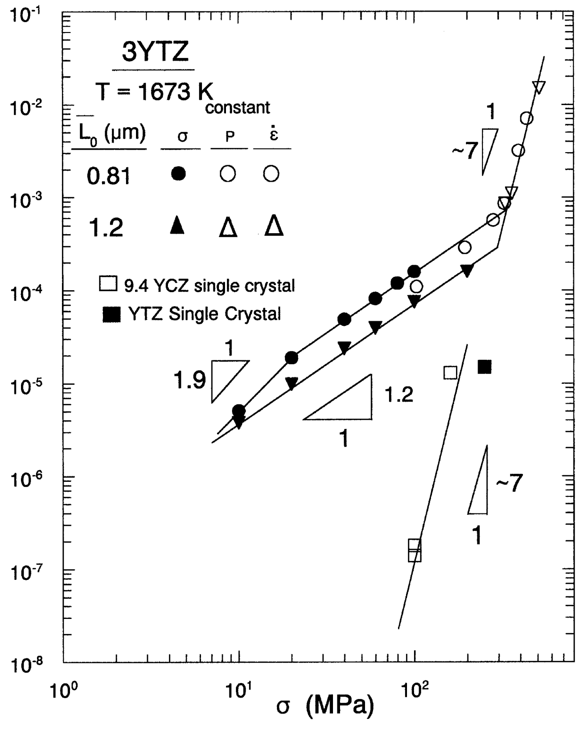

Fig. 13. Variation in strain rate with stress for polycrystalline 3YTZ and single crystalline cubic zirconia suggesting that the $n \sim 7$ region arises from a grain size independent intragranular dislocation creep process.

4. Summary and conclusions

There is good experimental evidence for the diffusion creep in oxide ceramics. Concurrent grain growth frequently accompanies deformation of fine grained materials at high temperatures, and this will enable grain switching together with a retention of an equiaxed grain size. Interface controlled diffusion creep is the rate controlling process for superplasticity in tetragonal zirconia. It is necessary to develop additional models for interface controlled diffusion creep to rationalize the observed influence of impurity content on transitions in stress exponents. The lack of diffusion data is an important limitation curtailing critical comparison of experimental and theoretical models.

Acknowledgements

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