

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

CONF-890310--5

CONF-890310--5

DE89 010420

TRANSIENT EFFECTS IN CREEP CAVITY NUCLEATION AND EARLY GROWTH IN CERAMICS

R.A. Page and K.S. Chan

Southwest Research Institute
6220 Culebra Road
San Antonio, TX 78284

ABSTRACT

Creep fracture of ceramic materials frequently occurs by the nucleation, growth, and coalescence of grain boundary cavities. Results of recent experimental studies of cavitation kinetics in compression crept ceramics are presented to illustrate the transient nature of cavity nucleation and early growth. The transient character of cavitation arises primarily due to the dependence of both cavity nucleation and early cavity growth on the stochastic process of grain boundary sliding. Possible mechanisms for the observed transient behavior will be presented and implications discussed.

KEYWORDS

Creep cavitation, nucleation, growth, ceramics, grain boundary sliding, transient behavior.

INTRODUCTION

The operative cavitation mechanism in ceramics is thought to vary with the grain boundary microstructure (Evans and Rana, 1980); diffusive cavity growth occurring when glass-free grain boundaries are present and viscous cavity growth occurring in the presence of a glassy grain boundary phase. The initial treatment of diffusive cavity growth was provided by Hull and Rimmer (1959). Many corrections and modifications have since been made to Hull and Rimmer's analysis; these include numerical corrections of the original model (Weertman, 1973; Speight and Harris, 1967; Speight and Beere, 1975; Skelton, 1966) as well as extension of the model to include cyclic loading (Skelton, 1966; Weertman, 1974), matrix constraint (Raj and Gosh, 1981; Rice, 1981; Hsueh and Evans, 1981), surface diffusion (Chuang and Rice, 1973; Chuang et al., 1979), power law creep (Beere and Speight, 1978; Needleman and Rice, 1980; Chen and Argon, 1981), and grain boundary sliding (Chen, 1983) effects. The growth of cavities in a viscous film was first analyzed by Raj and Dang (1975). Subsequent modifications have included matrix constraint effects for viscous cavity growth under both tensile (Marion et al., 1983) and compressive (Chan et al., 1984) loading.

dk
DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

7th International Conference on Fracture
March 1989
Houston, TX

FG05-84ER45063

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

One commonality throughout most of the cavity growth models is the use of steady-state or equilibrium stress conditions in the growth rate derivations. Cavity nucleation models, on the other hand, have generally had to include large stress transients, induced by grain boundary sliding, in order to obtain realistic prediction of nucleation kinetics (Nix, 1983). The apparent dependence of cavity nucleation on the highly stochastic process of grain boundary sliding has led to the consideration of cavity nucleation as a stochastic process as well (Argon, 1983; Page and Chan, 1987). It is obvious that if one assumes that cavity nucleation occurs as a result of grain boundary sliding transients, then the initial growth kinetics of a cavity should be dictated by the stress and sliding rate transients present at nucleation and not by the equilibrium stress conditions employed in most of the growth models. Unfortunately, although transient growth effects have been examined (Raj, 1975; Weertman, 1979; Argon *et al.*, 1980), these models do not provide an adequate treatment of the conditions present immediately following a nucleation event. It could easily be argued that the short durations of the transient conditions would make any growth during these periods relatively unimportant. It is the purpose of the paper to refute this argument, at least for ceramic systems, by demonstrating, through the use of both experimental results and theoretical treatments, that the transient conditions can have an important and measurable effect on cavity growth rates, and in so doing demonstrate a need for the inclusion of nonequilibrium stress conditions in cavity growth rate derivations.

CAVITY NUCLEATION

Prior to any discussion of the possible effects of transient stress and sliding conditions on cavity growth in ceramics it is necessary to justify any such discussion by demonstrating that transient conditions are likely responsible for cavity nucleation in ceramics. Fortunately, ample evidence supporting this hypothesis is now available. Previous studies of compressive creep in silicon carbide (Page *et al.*, 1984; Lankford *et al.*, 1986) and alumina (Lankford *et al.*, 1986; Page *et al.*, 1987; Page and Lankford, 1983; Page *et al.*, 1984) have demonstrated that significant cavity nucleation takes place during creep, even in ceramics that contain residual porosity left over from processing. As illustrated in Figure 1, cavity nucleation in these ceramic systems is frequently continuous and can be expressed in the form

$$\frac{N_c}{V} = at^b \quad (1)$$

where N_c/V is the number of cavities per unit volume, t is the creep time and, for a given applied stress, a and b are constants. Values of b ranging from 0.19 to 1.0 have been observed in the ceramic systems for which nucleation data are available (Page *et al.*, 1984a, b; Lankford *et al.*, 1986; Page *et al.*, 1987; Page and Lankford, 1983). These results are quite similar to the b values of 0.38 to 1.0 that have been observed in a number of metallic materials (Greenwood, 1969; Gittens, 1967; Chen and Argon, 1981) and they are also similar to the probable time dependency of grain boundary sliding. This latter observation is significant since a recent sliding induced cavity nucleation model (Page and Chan, 1987) relates the cavity density directly to the number of stochastic grain sliding events, as given by

$$\frac{N_c}{V} = C_0 u'(t) \quad (2)$$

where C_0 is a constant that depends on temperature but not on time and $u'(t)$ represents the mean value of the number of grain boundary sliding events at time t .

The cavity siting and morphology are also consistent with sliding induced cavity nucleation. Observed cavity densities are often relatively high (Page *et al.*, 1984a, b; Lankford *et al.*, 1986; Page *et al.*, 1987; Page and Lankford, 1983), suggesting that cavitation must occur on two grain facets, as well as along three grain junctions.

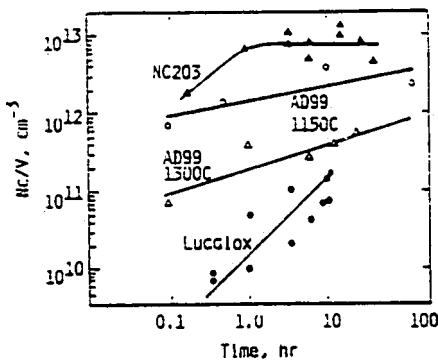


Fig. 1. Number of cavities per unit volume vs creep time for a hot-pressed silicon carbide, NC203, and two sintered aluminas, AD99 and Lucalox (Lankford et al., 1986).

The presence of cavities on two grain facets has been confirmed by direct observation with both transmission electron microscopy (Lankford et al., 1986; Page et al., 1987; Page and Lankford, 1983; Page et al., 1984) and scanning electron microscopy (Lankford et al., 1986; Page et al., 1987). These observations have also shown that the cavities are predominantly present in closely spaced clusters and that the cavities in a cluster are frequently all of equal or nearly equal size. The presence of clusters of cavities of nearly equal size seems to support Argon's hypothesis (Argon, 1983, 1982) that cavity nucleation can only occur when a grain boundary segment containing potential nucleation sites slides rapidly. Additionally, Jupp (1985) and Wiederhorn et al. (1986) have observed clusters of creep cavities situated at grain boundary ledges; these ledges would presumably act as stress concentrators during sliding events.

The observed cavity nucleation rates are quite high, sometimes exceeding 10^{13} nuclei/m²/s (Page et al., 1984a, b; Lankford et al., 1986; Page et al., 1987). In the absence of stress concentrations, thermal nucleation theory predicts nucleation rates which are tens of orders of magnitude lower than the observed values (Page et al., 1984). However, due to the strong stress dependence of the nucleation equation (Argon et al., 1980; Raj and Ashby, 1974; Raj, 1978; Evans et al., 1980) it is possible to obtain the observed nucleation rates with stress concentrations ranging from 3 to 20 (Page et al., 1984; Chan et al., 1986), depending on the shape of the nucleated cavity. Chan et al. (1986), by considering the time dependencies of both the shear stress relaxation along the boundary and the relaxation of the resulting stress concentration at the ledge by grain boundary diffusion, were able to describe the stress concentration due to a ledge as

$$\frac{\sigma(x,t)}{\sigma_a} = \sin \theta \cos \theta \left\{ \frac{\lambda}{h} [1 - \exp(-t/t_{BR})] \right\} \\ \times \exp(-t/t_c) \exp(-2x/h) - \cot \theta \quad (3)$$

where σ_a is the applied stress, h is the ledge height, λ is the ledge spacing, θ defines the angle between the boundary and the applied stress, x is the distance from the ledge, t_{BR} is the characteristic time for boundary relaxation, and t_c is the

characteristic time for diffusional relaxation of the elastic stress concentration. Realizing that for the concentrated stress to be effective it must be present for a time at least equivalent to the cavity incubation time, Chan *et al.* (1986) evaluated Eq. (3) for $t=t_i$, where t_i is the incubation time for cavity nucleation. As demonstrated by Figure 2, the result of this evaluation indicated that large stress concentrations could remain until t_i , provided the proper ledge height to spacing ratio (h/λ) was present.

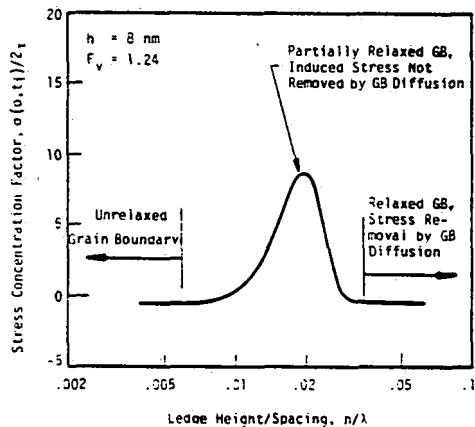


Fig. 2 Stress concentration factor at an 8 nm high grain boundary ledge as a function of ledge height to spacing ratio evaluated at the incubation time for cavity nucleation (Chan *et al.*, 1986).

Although not providing definitive proof, the results presented above clearly support the contention that creep cavity nucleation in ceramics requires the development of a stress concentration at a specific microstructural feature, such as a triple point, ledge, or particle, through grain boundary sliding. It would thus be expected that the initial growth of a nucleated cavity would be determined by the transient stress and sliding conditions that are present immediately following nucleation. It will be shown in the following section that the transient conditions, although often of very short duration, can contribute significantly to the overall growth of the cavities.

CAVITY GROWTH

Estimates of cavitation kinetics based on small-angle neutron scattering measurements of a number of different ceramics crept under compressive loading (Page *et al.*, 1984a, b; Lankford *et al.*, 1986; Page *et al.*, 1987; Page and Lankford, 1983) have shown that the volume of an individual cavity can be expressed as

$$V = \alpha t_n^{\beta} \quad (4)$$

where t_n is the time from nucleation, and α and β are constants, with β ranging from 0.0 to 0.62. Previous measurements of cavity growth rates in ceramics have, thus, resulted in either a zero growth rate (Lankford *et al.*, 1986; Page *et al.*, 1987; Page *et al.*, 1984), corresponding to $\beta=0$, or a growth rate that decreases with time (Page *et al.*, 1984; Lankford *et al.*, 1986; Page *et al.*, 1987), corresponding to $0 < \beta < 1.0$.

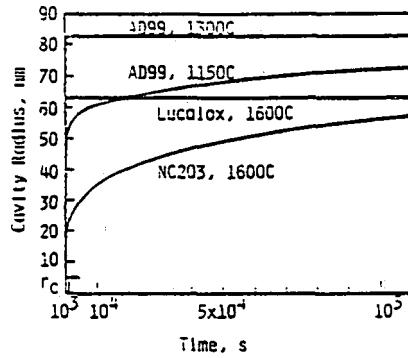


Fig. 3 Evolution of the individual cavity radius with time (Lankford et al., 1986). Time denotes the elapsed time since cavity nucleation and r_c is the critical cavity radius.

The observed cavity growth behaviors are illustrated in the plot of cavity radius vs time after nucleation provided in Figure 3. It should be noted that in the systems for which no apparent cavity growth was observed, i.e., $\beta=0$, the constant cavity radius was much larger than the estimated critical cavity radius. Since the estimated critical cavity radius was thought to be accurate, the condition of $\beta=0$ has been interpreted (Page and Chan, 1987) as indicating the presence of a very rapid growth transient of short duration immediately following nucleation. It was further concluded that the length of the growth transient must have been so short that the experimental measurements could not detect it.

The experimental measurements presented above clearly show cavity growth in these ceramics systems as a transient, rather than a steady-state process. This can be further demonstrated by comparing the experimentally measured cavity growth behavior with model predictions. Chan et al. (1984), treating the constrained growth of cavities in a material containing a continuous glassy grain boundary phase (as in the AD 99 and NC 203 materials of Figure 3), envisaged cavities growing on boundaries oriented parallel to the applied compressive stress in response to a local boundary normal stress that arose due to grain boundary sliding. In their analysis Chan et al. (1984) were able to relate the average normal stress, σ_n , acting on the cavitating boundary to the grain boundary sliding rate, \dot{u} , as

$$\bar{\sigma}_n = \frac{33\dot{u}n}{2d} \quad (5)$$

where n is the viscosity of the glassy phase and d is the grain diameter. By combining Eq. (5) with an earlier treatment of viscous hole growth by Raj and Dang (1975) it was possible to relate the cavity growth rate, \dot{R} , to the sliding rate as

$$\dot{R} = \frac{h^2(2\sqrt{3}\dot{u}^2 - 8\pi R^2)[\frac{33\dot{u}n}{2d} - 2\gamma K(1-0.9\alpha^2)]}{12\pi R^2 n^2 [0.96\alpha^2 - 1n\alpha^2 - 0.23\alpha^4 - 0.72]} \quad (6)$$

where h is the thickness of the glassy phase, l is the cavity spacing, s' is a cavity shape factor, γ is the surface energy, K is a constant related to the ratio of the grain boundary, surface and interfacial energies, and a' is the ratio of the cavity radius to the cavity spacing. When viewed in this manner, it is clear that the grain boundary sliding rate provides the driving force for cavity growth during compressive creep. It is evident from Figure 4 that if one considers only steady-state sliding, then agreement between the measured cavity growth rates and those predicted by Eq. (6) is observed only in the latter stages of the growth process. The constrained growth model (Chan *et al.*, 1984), from which Eq. (6) was derived, thus does not do an adequate job of modeling the time dependence of the experimentally measured growth rate in the initial transient region. Similarly, diffusion based steady-state models (Hull and Rimmer, 1959; Weertman, 1973; Speight and Harris, 1967; Speight and Beere, 1975) also fail to describe the observed growth behavior.

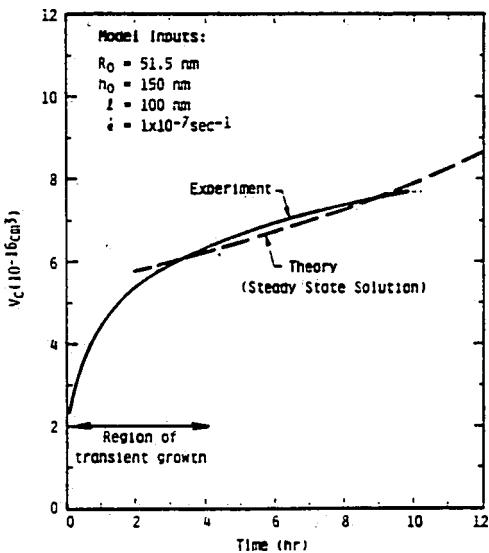


Fig. 4 Comparison of the calculated and measured cavity volume (Page and Chan, 1987). Both the experimental and theoretical curves are for AD99 alumina crept at 1150°C and 220 MPa.

The observations of continuous cavity nucleation accompanied by limited growth from which the transient growth behavior is obtained are neither an artifact of the measurement technique nor unique to the particular systems or conditions investigated. Studies of cavitation in a number of metallic systems using techniques other than small-angle scattering have shown similar behavior (Needham and Gladman, 1980; Chen and Argon, 1981). It must, therefore, be concluded that stress and/or sliding transients do indeed influence initial cavity growth. Furthermore, the transient growth regime can be responsible for a substantial portion of the total observed growth. Predictions based solely on steady-state growth could thus be expected to underpredict cavity size and overpredict creep lifetime.

The experimental measurements presented above clearly show cavity growth in these ceramic systems as a transient process. The results also indicate that a very wide spread in the length of the transient growth event is possible, e.g., in some systems

transient periods lasting on the order of hours have been observed while in others the transient period is so short that the cavities appear to nucleate at their final size. If it is assumed that the transient growth results from the presence of transient stresses, then the characteristic time, t_c , for the relaxation of these stresses through grain boundary diffusion is given by (Raj, 1975; Chan *et al.*, 1986)

$$t_c = \frac{(1-v)kT L^3}{4\Omega D_b \delta_b G} \quad (7)$$

where v is Poisson's ratio, k is Boltzman's constant, T is temperature, Ω is the atomic volume, $D_b \delta_b$ is the grain boundary diffusivity, G is the shear modulus, and L is the characteristic diffusion length.

Because of the complex nature of cavity nucleation and growth, a minimum of three different stress concentration mechanisms, each with its own characteristic diffusion length, could contribute to transient cavity growth. As discussed previously, cavity nucleation in the ceramic systems for which the transient growth behavior has been observed is thought to occur at stress concentrations produced at grain boundary ledges during boundary sliding. The characteristic length for the relaxation of the stress at the ledge is the ledge height, which has been estimated (Chan *et al.*, 1986) to be approximately 10 nm. Raj (1975) has suggested that transient stresses also can result from the nucleation of the cavities and from the sliding of nonplanar grain boundaries. The characteristic length for relaxation of the stress concentration due to nucleation of the cavities is simply one-half of the cavity spacing, which has been estimated to be approximately 100 nm (Page *et al.*, 1984a, b; Lankford *et al.*, 1986; Page *et al.*, 1987; Page and Lankford, 1983), while that for the grain boundary sliding transient is one half of the grain size (Raj, 1975), which can range from 2 to 30 μm . Due to the L^3 dependence of t_c in Eq. (7), a very wide range for the transient duration could be expected depending upon which relaxation mechanisms were contributing. Order of magnitude estimates of t_c for the ceramic systems being discussed range from approximately 10^{-7} sec to 10^{-5} sec and 10^3 sec for relaxation of the stresses due to grain boundary ledges, cavity nucleation, and grain boundary sliding, respectively. Either of the former two relaxation mechanisms could thus be responsible for the extremely short transients that resulted in cavities that appeared to nucleate at their final size, while the latter relaxation mechanism could be responsible for the longer duration transients that lasted up to a few hours prior to the onset of steady-state growth.

SUMMARY

The presence of transient cavity growth has been demonstrated for a number of ceramic systems. It has been argued that the transient growth is a direct consequence of the transient stress and sliding conditions that are present immediately following cavity nucleation. Since transient growth can account for a significant portion of the overall growth, accurate treatments of transient behavior must be developed if we hope to fully understand damage development in ceramic systems. Furthermore, more complete analytical treatments of transient growth behavior may provide an explanation of the creep-fatigue effects that have recently been observed in ceramics (Fett *et al.*, 1986; Page and Lankford, 1987). Although complete analytical treatments of transient stress and sliding effects are expected to be quite difficult, their importance cannot be overemphasized for, as Nix (1983) pointed out, the intimate link that exists between cavity nucleation and early growth and the creep process itself is probably responsible for the correlation that has been observed between fracture time and minimum creep rate in many structural materials.

ACKNOWLEDGMENT

This work was supported by the United States Department of Energy, Office of Basic Energy Research, under Grant No. DE-FG05-84ER45063.

REFERENCES

Argon A.S., I-W. Chen and C-W. Lau (1980). Creep-fatigue-environment interactions. (R.M. Pelloux and N.S. Stoloff, eds.) AIME, New York, p. 46.

Argon A.S. (1982). Recent advances in creep and fracture of engineering materials and structures. (B. Wilshire and D.R.J. Owen, eds.) Pineridge Press, Swansea, U.K., p. 1.

Argon A.S. (1983). Scripta Metall. 17, 5.

Beere W. and M.V. Speight (1978). Met. Sci. 12, 172.

Chan K.S., J. Lankford and R.A. Page (1984). Acta Metall. 32, 1907.

Chan K.S., R.A. Page and J. Lankford (1986). Acta Metall. 34, 2361.

Chen, I-W. and A.S. Argon (1981). Creep and fracture of engineering materials and structures. (B. Wilshire and D.R.J. Owen, eds.) Pineridge Press, Swansea, U.K., p. 289.

Chen I-W. and A.S. Argon (1981). Acta Metall. 29, 1321.

Chen I-W. and A.S. Argon (1981). Acta Metall. 29, 1759.

Chen I-W. (1983). Metall. Trans. 14A, 2289.

Chuang, T.J. and J.R. Rice (1973). Acta Metall. 21, 1625.

Chuang, T.J., K.T. Kagawa, J.R. Rice and L.B. Sills (1979). Acta Metall. 27, 265.

Evans, A.G. and A. Rana (1980). Acta Metall. 28, 129.

Evans A.G., J.R. Rice and J.P. Hirth (1986). J. Am. Ceram. Soc. 63, 368.

Fett T., G. Himsolt and D. Munz (1986). Adv. Ceram. Mater. 1, 179.

Gittens A. (1967). Met. Sci. J. 1, 214.

Greenwood G.W. (1969). Phil. Mag. 19, 423.

Hsueh C.H. and A.G. Evans (1981). Acta Metall. 29, 1907.

Hull, D. and D.E. Rimmer (1959). Phil. Mag. 4, 673.

Jupp R.S. (1985). McMaster Univ., Hamilton, Ontario, Canada, unpublished research see paper by Chan K.S., R.A. Page and J. Lankford (1986).

Lankford J., K.S. Chan, and R.A. Page (1986). Fracture mechanics of ceramics. (R.C. Bradt, A.G. Evans, D.P.H. Hasselman and F.F. Lange, eds.) Plenum Press, New York, p. 327.

Marion J.E., A.G. Evans, M.D. Drory and D.R. Clarke (1983). Acta Metall. 31, 1445.

Needlam N.G. and T. Gladman (1980). Met. Sci. J. 14, 64.

Needleman A. and J.R. Rice (1980). Acta Metall. 28, 1315.

Nix W.D. (1983). Scripta Metall. 17, 1.

Page R.A. and J. Lankford (1983). J. Am. Ceram. Soc. 66, C-146.

Page R.A., J. Lankford and S. Spooner (1984). J. Mater. Sci. 19, 3360.

Page R.A., J. Lankford and S. Spooner (1984). Acta Metall. 32, 1275.

Page R.A. and J. Lankford (1987). J. Mater. Sci. 22, 2521.

Page R.A. and K.S. Chan (1987). Metall. Trans. 18A, 1843.

Page R.A., J. Lankford, K.S. Chan, K. Hardman-Rhine and S. Spooner (1987). J. Am. Ceram. Soc. 70, 137.

Page R.A. and K.S. Chan (1987). Metall. Trans. 18A, 1843.

Raj R. (1974). Metall. Trans. 6A, 1499.

Raj R. and M.F. Ashby (1975). Acta Metall. 23, 653.

Raj R. and C.H. Dang (1975). Phil. Mag. 32, 909.

Raj R. (1978). Acta Metall. 26, 995.

Raj R. and A.K. Gosh (1981). Metall. Trans. 12A, 1291.

Rice J.R. (1981). Acta Metall. 29, 675.

Skelton R.P. (1966). Phil. Mag. 14, 163.

Speight, M.V. and J.E. Harris (1967). Met. Sci. 1, 83.

Speight, M.V. and W. Beere (1975). Met. Sci. 9, 190.

Weertman J. (1973). Scripta Metall. 7, 1129.

Weertman J. (1974). Metall. Trans. 5, 1743.

Weertman J. R. (1979). Can. Metall. Quart. 18, 73.

Wiederhorn S.M., B.J. Hockey, R.F. Krause, Jr. and K. Jakus (1986). J. Mater. Sci. 21, 810.