THE SIGNIFICANCE OF INTERGRANULAR CAVITATION IN CREEP-FATIGUE

V. SKLENIČKA
Institute of Physics of Materials, Academy of Sciences of the Czech Republic,
CZ-616 62 Brno, Czech Republic

ABSTRACT

This work deals with the significance of intergranular cavitation with respect to the advanced stages of crack formation, propagation, and fracture, and its influence on lifetime and ductility in copper and nickel during high temperature creep and creep/high-cycle fatigue. A semi-quantitative and quantitative investigation of the extent of cavitation has been carried out on fractured specimens subjected to a broad range of the loading conditions. The results show that the final intergranular creep fracture occurs when all the stressed volume of a creeping body is critically damaged by integranular cavitation. In contrast, fatigue fracture occurs by single crack propagation and the fracture path is not primarily determined by the amount of cavitation. The role of cavitation thus increases with decrease of the cyclic stress component, as demonstrated by the fracture behaviour under creep/fatigue conditions.

KEYWORDS

Intergranular cavitation, creep-fatigue interaction, high temperature fracture, quantitative metallography, single crack propagation, copper, nickel.

INTRODUCTION

High temperature fracture behaviour under creep/fatigue loading has been studied quite intensively over the past two decades both experimentally and through theoretical modelling. A comprehensive survey of this subject area is available in several reviews and volumes (e.g., Sadananda and Shahinian, 1981; Skelton, 1983; Riedel, 1987; Suresh, 1991; Webster, 1995). It should be stressed, that most models and characterization methodologies for crack initiation and growth are essentially phenomenological, since understanding of the synergetic effect under combined creep/fatigue loading conditions is limited. Crack growth under these complex loads is classified as cycle- or time-dependent, or a combination of the two processes. Cycle-dependent crack growth is due to fatigue and time-dependent crack growth is due to creep cavitation, environmental interactions, and/or a combination of the two. Thus, it is not clear that formulations of the type given by a micromechanistic approach can adequately define the interaction of creep and fatigue, as they usually do not separate the competing mechanisms in a physically realistic manner.

While the dominant crack nucleation and propagation mode of fracture is often very sensitive to the chemical environment (Ericsson, 1979), the cavitational mode is relatively insensitive

(Maiya, 1981). The transition between the cavitational mode and microcrack propagation depends strongly on the material and testing conditions, and is very sensitive to environment.

Similarly to the case of creep, creep/fatigue failure at high temperatures may also occur by more or less homogeneous cavitation of grain boundaries over the whole cross section of specimen. A particularly interesting aspect of cavitation under cyclic loads is that the extent of cavitation is strongly affected by the shape of the stress wave. Slow tension and fast compression strain rates, i.e., loading wave shapes with long hold times at tensile stresses, exhibit a greater propensity for cavitation than balanced load cycles where the loading and compression periods are identical (Riedel, 1987; Suresh, 1991; Baik and Raj, 1982). In some cases, however, symmetric loading cycles with zero mean stress may also cause cavitation; for example, copper cavitates readily under balanced cyclic loading, both at high loading frequencies and low amplitudes (Saegusa and Weertman, 1978; Skleničkа *et al.*, 1993a) and at low frequencies and high amplitudes (Sidey and Coffin, 1978; Baik and Raj, 1983).

The present work deals with the significance of intergranular cavitation with respect to the advanced stages of crack formation, propagation, and final fracture and its influence on lifetime and ductility in copper and nickel during high temperature creep and creep/high-cycle fatigue. The semi-quantitative and quantitative investigation of the critical role of intergranular cavities has been carried out on fractured specimens under a broad range of loading conditions.

EXPERIMENTAL MATERIALS AND PROCEDURES

Copper

Copper of commercial purity (99,96 percent) was annealed at 873 K for 10 h in vacuum to give a uniform grain size of 0.05 mm. Creep, high-cycle fatigue, and high-cycle fatigue/creep tests were carried out in air at 773 K using a modified resonant Rumul machine. Cyclic loading was performed at a frequency of about 120 Hz. Both the mean load and the load amplitude were kept constant. In the following, each test will be characterized by the load ratio, R, defined as the ratio of the minimum load to the maximum load. Thus, R = -1 for pure fatigue and R = 1 for pure creep; R ratios for cyclic creep lie between these two extreme values. All the specimens were run to final fracture. Fractography revealed (Sklenička *et al.*, 1993a) that the optimum stress interval for the study of the creep/high-cycle fatigue interaction leading to complete intergranular fracture is the stress range from 20 to 50 MPa.

Nickel

Nickel of commercial purity (99,9 percent) was annealed at 1373 K for 6 h in vacuum to give uniform grain size of 0.18 mm. The combined creep/fatigue tests were carried out in hydrogen at 1073 K using a specially designed testing machine (Sklenička *et al.*, 1993b) in which axial loads could be held for long periods of time with accuracy ±0,5 percent. The lateral vibratory stress was applied by means of an electromagnetic shaker. Cyclic loading was performed at the frequency of about 22 Hz. Again, all specimens were run to final fracture.

Assessment of Cavitation

By means of linear analysis along the grain boundary lines, several quantitative parameters of intergranular cavitation were evaluated (Sklenička *et al.*, 1991). For each specimen the total number of cavities per unit area of the microsection, N_A, was counted. The cavity number per unit length of grain boundary lines, N_c, was calculated using the relationship $N_c = 2N_A/\pi N_L$, where N_L is the number of intersections of grain boudaries per unit length of microsection. In order to assess cavity size, the length of cavity chords on grain boundary lines, l_c, was measured. Measurements of N_A and l_c were performed at a constant magnification of 1000 x, thus limiting the detectable cavity size to approximately 1 μm. The extent of cavitation is quantitatively described by the fraction of grain boundary area occupied by cavities,

$$A_c = \left(2\sum_{A} l_c\right) / (\pi A N_L)$$

where A is the studied tested area of the microsection. The inhomogeneity of cavitation at various grain boundaries can be characterized by the parameter κ_c, expressing the fraction of grain boundaries with observable cavitation. Knowing A_c and κ_c, the most important parameter of the local extent of cavitation can be introduced, namely the parameter $\widetilde{A}_c = A_c/\kappa_c$ representing the areal fraction of cavities in cavitated grain boundaries.

RESULTS

Cavitation in Creep-Fatigue of Copper

The effect of the cyclic stress component on the time to fracture,t_f, can be expressed in terms of the dependence of t_f on the stress cycle asymmetry characterized by stress ratio R. Figure 1 shows the dependence of the time to fracture on the maximum stress in the stress cycle for different R ratios in the maximum stress range from 20 to 50 MPa. For a given stress, pure creep exhibits the longest time to fracture, pure fatigue exhibits the shortest time to fracture. The lives of cyclically loaded specimens ($-1 < R < 1$) lie between them. Thus, the results in Fig. 1 suggest that the detrimental effect of the cyclic stress components on creep life is substantial even for relatively low stress amplitudes. Figure 2 displays the dependence of the fracture strain on the R ratio. It can be seen that the fracture strain increases roughly linearly with the R value.

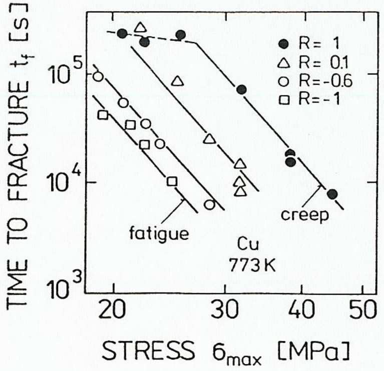

Fig. 1. Dependence of time to fracture on maximum stress for different R ratios.

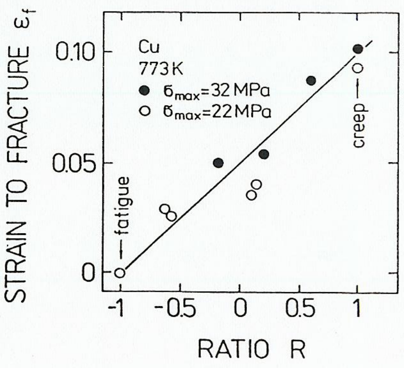

Fig. 2. Dependence of fracture strain on R ratio.

In order to investigate more thoroughly the amount of intergranular cavitation on the fracture, quantitative metallography was employed in assessing cavitation in the creep, fatigue, and creep/fatigue fractured speciments tested at 22 MPa. From Fig.3a it can be seen that the

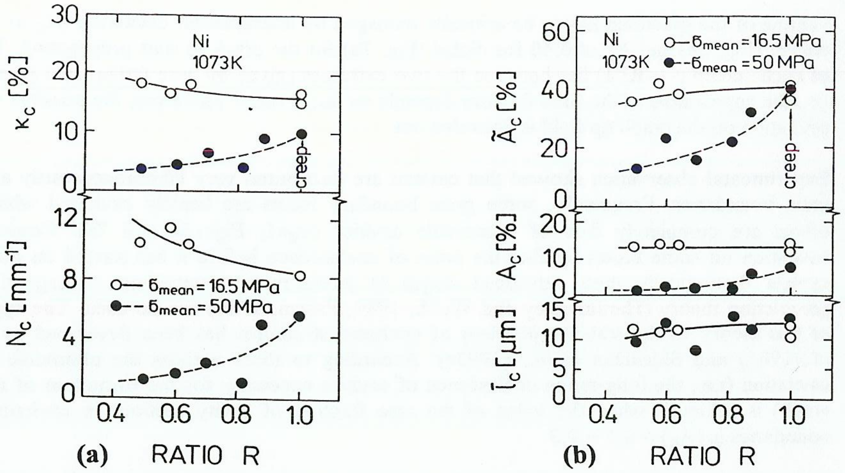

Fig. 3. Dependence of (a) topological parameters of cavitation and (b) metric parameters of cavitation on R ratio fractured specimens.

topologic quantities of cavitation (cavity density, N_C, and the fraction of cavitated boundaries κ_C), exhibit a systematic decrease with increasing R value: clearly, the values of N_C and κ_C for pure fatigue are higher than those for pure creep. On the other hand, the variation of the metric parameters of cavitation (cavity size l_C, cavitated areas of grain boundary A_C and $\widetilde{A}_C$) with the R value are more complicated (Fig. 3b). Increasing the R value results in larger cavities, while the values of A_C are nearly constant ($A_C = f(N_C, l_C)$). Nevertheless, the area fraction of cavities in cavitated grain boundaries, $\widetilde{A}_C$, increases considerably with the R value, indicating that the local extent of cavitation is essentially dependent on the cyclic stress component (Fig. 3b). Thus, the value $\widetilde{A}_C$ of 27 percent for pure creep is approximately four times higher than those for pure fatigue.

Cavitation in Creep-Fatigue of Nickel

Figure 4 shows the dependences of the minimum creep rate and the time to fracture, on the applied stress for pure creep tests performed under constant load at 1073 K. A variation of the strain to fracture with stress is shown in Fig. 5. In Fig. 5 two regions are apparent: a low stress region ($\sigma \leq 30$ MPa), where the fracture strain is relatively low, and a high stress region ($\sigma > 30$ MPa), where the strain increases gradually with increasing stress. Fractography has revealed that in the low stress region completely intergranular fracture takes place by the nucleation and growth of cavities at grain boundaries (Sklenička *et al.*, 1993b). As the stress is raised in the high stress region, the fracture mode changes into a mixture of intergranular and transgranular type. This results in gradual increase of the strain to fracture (Fig.5).

Thus, the two constant values of the initial mean stress were chosen, namely 16.5 MPa and 50 MPa, for range of the stress ratio $R \in (0.4, 1.0)$.

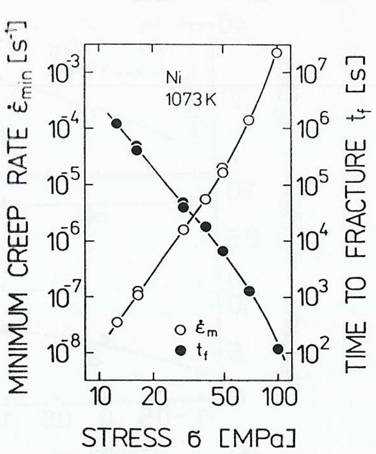

Fig. 4. Dependence of minimum creep rate and time to fracture on applied stress for pure creep tests.

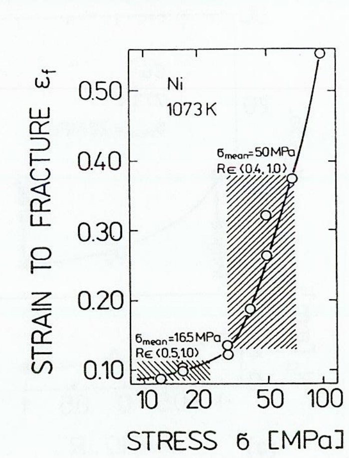

Fig. 5. Dependence of fracture strain on applied stress for pure creep tests. Hatched areas indicate the stress regions for creep/fatigue at 16.5 MPa and 50 MPa.

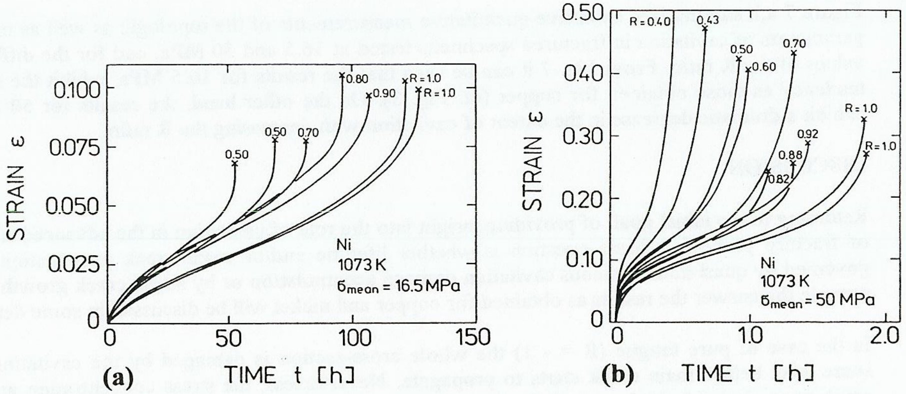

Fig. 6 a,b. Time dependence of strain for pure creep tests (R = 1) and for creep/fatigue tests (R < 1) at mean stresses: (a) 16.5. MPa; (b) 50 MPa.

The deformation behaviour under pure creep (R = 1.0) and creep/fatigue loading (0.4<R<1.0) is shown in Fig. 6 a, b. It can be seen that for both mean stresses the time to fracture has decreased under cyclic loading. The effect of small cyclic stress (for R > 0.4) is, thus always detrimental. On the other hand, the effect of cycling on the strain to fracture is more complicated. While no clear effect is found for 16.5 MPa (Fig. 6a), the results for 50 MPa (Fig.6b) exhibit strong dependence of the fracture strain on R. In this case a decrease in R is accompanied by an increase in ductility. This phenomenon is closely related to the occurrence of dynamic recrystallization. Metallographic observations have revealed, that during the creep/fatigue exposures at 50 MPa, extensive recrystallization occurred in the whole specimen volume. The lower the value of the R ratio, the higher the volume fraction of recrystallized grains. No such recrystallization or change in grain size have been detected for specimens

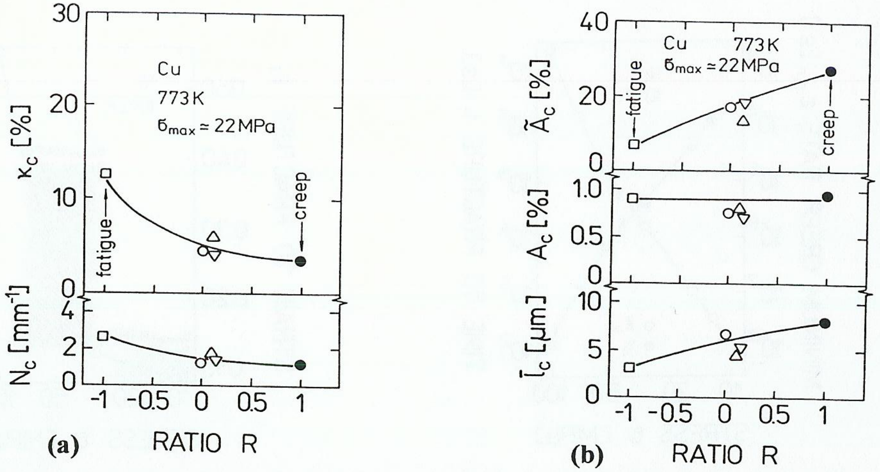

Fig. 7a, b. Dependence of (a) topological parameters of cavitation and (b) metric parameters of cavitation on R ratio for specimens fractured at 16.5 MPa and 50 MPa, respectively.

which underwent pure creep loading at 50 MPa. It should be pointed out that for 16.5 MPa only very local recrystallization was found in close proximity to the fracture path for cyclic creep specimens.

Figure 7 a,b summarizes extensive quantitative measurements of the topologic as well as metric parameters of cavitation in fractured specimens tested at 16.5 and 50 MPa, and for the different values of the R ratio. From Fig. 7 it can be seen that the results for 16.5 MPa exhibit the same tendency as those obtained for copper (cf. Fig. 3). On the other hand, the results for 50 MPa exhibit a dramatic decrease in the extent of cavitation with decreasing the R ratio.

DISCUSSION

Returning to the initial goal, of providing insight into the role of cavitation in the advanced stages of fracture process, a basic question is whether lifetime and/or main crack propagation are governed by quasi-homogeneous cavitation damage accumulation or by single crack growth. To provide the answer the results as obtained for copper and nickel will be discussed in some detail.

In the case of pure fatigue (R = - 1) the whole cross-section is damaged by the cavitation to some level before main crack starts to propagate. Nevertheless, the stress concentration at the crack tip is obviously high enough (i.e., not effectively relaxed) for a single crack to propagate across the whole cross-section, and the process of main crack growth by intergranular cavitation is not of primary importance. However, cavitation may enhance the fracture process, and main crack propagation can be accelerated by the presence of intergranular cavities or microcracks.

In the case of pure creep (R = 1) for a long period of time, the stress concentrations at the tips of the surface cracks are probably sufficiently relaxed, so that no single crack can propagate across the whole cross-section. The advanced stage of the creep fracture process is characterized by extensive microcrack formation due to long-range coalescence of damage inside the creeping body; these microcracks may join surface cracks. The final creep fracture then takes place by a relatively fast propagation of one of the long surface cracks. This suggests that all the stressed volume of the specimen has to be critically damaged by intergranular cavitation (A_c of 0.27 for copper (Fig. 3b) and A_c of 0.40 for nickel (Fig. 7b) for the crack to start propagating. The case of cyclic creep (-1<R<1) lies between the two extremes (given by pure fatigue and pure creep), i.e., the appearance of the final fracture depends on the R ratio. Therefore, the possible effect of cavitation on the crack tip field is not ruled out.

Experimental observation showed that cavities are distributed very inhomogeneously along the grain boundaries. Frequently, some grain boundary facets are heavily cavitated while many others are completely free of observable cavities ($\kappa_c \ll 1$, Figs. 3a and 7a). Consequently, cavitation on some facets reaches the point of coalescence before it has started on others. To explain theoretically these advanced stages of intergranular cavitational damage the bond percolation theory (Hammersley and Welsh, 1980; Essam, 1980) can be used. The application of this theory to the stability problem of cavitated structures has been developed by Saxl *et al.*,(1985) and Sklenička *et al.*, (1993a). According to these authors the ultimative state of cavitation (i.e., the long-range coalescence of cavities necessary for the formation of magistral crack) is achieved when the value of the area fraction of cavity sections in cavitating grain boundaries is $(\widehat{A}_c) > 0.1 \div 0.3$.

The agreement of this theoretical estimate with the previous experimental results (Sklenička *et al.*, 1991) found for several materials crept under a broad range of experimental conditions is surprisingly good. The same applies for results of this work (Figs.3b and 7b). The higher values of $\widehat{A}_c$ for nickel than those obtained for copper could be explained most probably by different mechanisms of cavitation. While in copper cavitation damage occurs by the formation of several discrete cavities known as r type cavities, in grain boundaries aligned mostly normal to the tensile axis, in nickel we see wedge shaped microcracks as a result of preferential cavitation at the triple grain junctions.

It should be pointed out that percolation model assumes spontaneous propagation of a main crack as soon as a sufficiently cavitated fracture path is formed in the whole specimen transverse cross-section. However, a gradual propagation of the main crack due to successive growth and interlinkage of cavities nucleated ahead of its tip requires a considerably lower value of $\widehat{A}_c$. Since the percolation theory assumes an infinitive graph (i.e., given process passing trough the modelled medium), it can be applied only in the case of a crack passing at the same time through numerous clusters of cavities distributed along the direction of propagation; hence, the limited applicability of the percolation theory in the case of fatigue failure. Nevertheless, the developing theory of time-dependent percolation processes seems to be a promising tool in solving this problem.

Fracture behaviour of nickel under creep/fatigue at 50 MPa is strongly infuenced by extensive dynamic recrystallization. White and Rossard (1968) first pointed out that the migration of grain boundaries during recrystallization leads to blunt growing cracks, and results in the development of more irregular cavities. Further, recrystallization can quickly isolate some grain boundary cavities and microcracks within the grain interiors and prevent their growth (Pahutová and Sklenička, 1992). Accordingly, these intragranular cavities do not take part in the formation of the intergranular fracture path in the final stage of fracture process. The contention, then, is that the final fracture is governed by single crack propagation rather than guasi-homogeneous cavitation damage.

CONCLUSIONS

The results obtained from this study indicate that the creep life is strongly affected by the cyclic stress component. Pure creep exhibits the longest time to fracture. Time to fracture for creep/fatigue depends on the R-ratio. The final intergranular creep fracture occurs when all the stressed volume of creeping specimen is critically damaged by intergranular cavitation. On the other hand, creep/fatigue fracture occurs by single crack propagation and the fracture path is not primarily determined by the amount of cavitation. At high value of mean stress even a small cyclic stress component can initiate dynamic recrystalization which, in turn, can retard the development of cavitation damage.

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