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Review
Cavitation in creep

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Metals often fail in service under creep conditions because of the formation of cavities on the grain boundaries which are approximately normal to the applied stress. This phenomenon of creep cavitation is becoming of increasing technological importance. As a result a complete understanding of it is desirable so that alloys with improved cavitation resistance can be designed. This paper reviews the development of our present understanding of the phenomenon which is one of nucleation, growth and linkage leading to failure.

Several mechanisms of nucleation, such as at grain-boundary ledges or precipitates, have been postulated and experimental evidence in support of each has been cited. Similarly, deformation- or vacancy-controlled growth mechanisms have been discussed. It is apparent from the literature that *no single mechanism is applicable*, indeed, the work discussed here suggests that several mechanisms may operate and each may become dominant at different stages of the creep life.

Finally, the status of research into nickel-base superalloys is reviewed with reference being made to such effects as regenerative heat-treatment.

1. Introduction

A number of metals which are normally ductile at their operating temperature fail after a relatively limited extension when held in tension for a prolonged period in the temperature range of 0.3 to 0.9 of their absolute melting points. It has been found that this limited ductility, which can be as little as 1%, is related to either cracking at grain-boundary triple junctions or to the formation of cavities (or voids) on grain boundaries which are approximately normal to the applied stress. These two types of defect are often referred to [1] as w-type cracks and r-type voids respectively. It is the development of this latter type leading to premature failure with limited ductility which is termed creep cavitation.

Cavitation seems to be favoured by lower stresses whereas triple-point cracking is the preferred mode at higher stresses [2]. The transition is not distinct [2, 3], both types are frequently found together [2] and the detailed microstructure is also significant [4]. The change-over in dominant mode is often referred to as the Stroh-McLean transition as McLean [2] invoked the equation for the fracture of a sliding interface due to Stroh [5] in an attempt to analyse it. The occurrence of triple-point cracking has been treated by the theory of Zener [6] and confirmed experimentally by Chang and Grant [7]. An estimate of creep life based on this type of failure mode has been given by Williams [8].

The full significance of the cavities in limiting life under creep conditions was first realized about 20 years ago by Greenwood [9-12], although the voids had been observed previously [13-15]. Since that time much work has gone into understanding their nucleation and growth, and also the mechanism by which failure is initiated. The initial impetus to study cavitation came from failures in the magnesium alloy cladding of nuclear reactor fuel rods and has been continued because of turbine blading problems and low creep rupture ductility of power plant components. The general features [11] of cavitation failure are an increasing loss of ductility with increasing temperature and decreasing strain-rate. These changes are directly associated [16] with cavity development and with the inter-cavity spacing which increases with temperature. The voids show a distinct preference to form on the grain boundaries approximately perpendi-

cular to the applied stress but do not form on twin boundaries. The onset of cavitation coincides with the onset of grain-boundary sliding but not all boundaries develop cavities. Low temperature cavitation has also been reported, for example at 50° C in copper alloys by Sergeant [17].

It is the purpose of this review to assemble published research in the field of cavitation creep with a final reference to the work done on nickel-base alloys.

2. The phenomenon of creep

A metal under tension will extend slowly as a function of time. The rate of extension is not constant but is usually made up of three distinct stages under the conditions of interest here. A typical creep curve is shown schematically in Fig. 1. The division of the curve follows from the

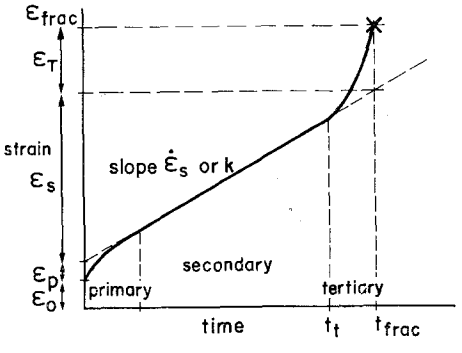

Figure 1.

classical work of Andrade [18, 19]. Expressing the length change in terms of strain, the equation becomes [20, 21]:

$$\epsilon = \epsilon_0 + \beta t^{1/3} + Kt\,, \qquad (1)$$

where ϵ_0 is the initial extension on loading, β is the coefficient of primary or transient creep and K is the coefficient of secondary creep (alternatively termed constant, steady state or minimum rate creep or flow). This stage is often followed by an accelerating tertiary stage leading to fracture. Equation 1 has been shown to describe the behaviour of a large number of materials particularly in the primary range [22]. The secondary creep rate defined by it cannot be truly constant in contrast to observation. An alternative extended form has been developed [23, 24] from the Garofalo equation [1]:

$$\epsilon = \epsilon_0 + \epsilon_T\,[1 - \exp(-mt)] + \dot{\epsilon}_s t + \epsilon_L \exp[p(t - t_t)]\,. \qquad (2)$$

The four terms on the RHS of Equation 2 relate to the various stages of the creep curve in Fig. 1: ϵ_0 is the initial extension as before, ϵ_T is the primary creep strain and m is a constant describing the decay of this stage. $\dot{\epsilon}_s$ is the secondary creep rate. Finally, t_t is the time of onset of tertiary creep and ϵ_L and p are constants.

The processes involved in recovery creep are discussed as follows. The creep rate in the secondary stage is thought to be controlled [25-27] by the exact balance between the strain hardening of the metal caused by deformation on the one hand and thermal softening or recovery on the other:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = \frac{r}{h}\,, \qquad (3)$$

where r is the recovery rate and h is the coefficient of strain hardening. This is the Bailey-Orowan theory of secondary creep. The first test of this proposal was made by Cottrell and Aytekin [27] on single and polycrystals of zinc as part of a more general study. They carried out recovery experiments and observed the induction period of zero flow following load reduction as was predicted by Orowan [26].

Subsequently, Davies *et al* [28] studied the effect of stacking fault energy (SFE) on secondary creep in nickel-cobalt alloys. They argued that lowering the SFE should reduce the recovery rate and also increase the flow stress, thereby reducing the overall creep rate. This was observed and tends to support the Bailey-Orowan model.

An extensive investigation and confirmation of the general validity of Equation 3 has been made by Mitra and McLean [29, 30]. They studied [29] the rate of recovery in nickel and aluminium at several temperatures through step-wise stress relaxation. From the known creep rate and their evaluated recovery rate, the strain hardening coefficient was derived and found to be in accordance with that found in pre-creep-tested tensile samples. They were also able to show that the stress sensitivity of the creep rate follows from the stress sensitivity of the rate of recovery.

Subsequently, Mitra and McLean [30] investigated the apparent contradiction between the observation that after each stress reduction there is some recovery, whilst there is no apparent concomitant change in the dislocation density. They explained this by assuming that during this

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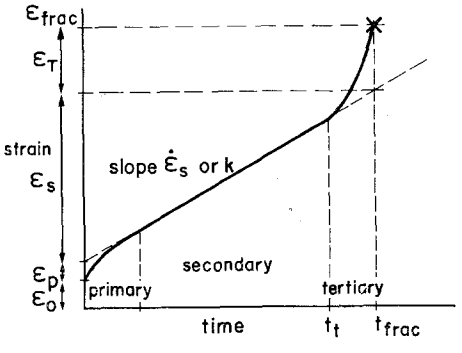

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copper stopped both cavitation and sliding by inducing grain-boundary serrations. A number of models based on sliding have been proposed but the amount of direct experimental evidence is limited according to Cottrell [44].

The significance of grain-boundary sliding was demonstrated in detail by Intrater and Machlin [45, 46]. They studied copper bicrystals stressed in shear along the boundary in the range 680 to 900°C. They found that the number of voids generated was proportional to the amount of sliding independent of temperature (again ruling out a vacancy mechanism); also the void spacing was less than the slip line spacing [47]. Two types of grain-boundary sliding were distinguished [36, 48]: true sliding and grain-boundary zone shear. The sliding is spasmodic and voids can be generated by either type of sliding. They concluded that cavity nucleation is associated with grain-boundary jogs which must themselves deform plastically [45] before separation occurs. Grant [49] pointed out in a 1959 review article, that sliding alone is not a sufficient condition because pure aluminium does not cavitate. Also, as Boettner and Robertson [50] found, the presence of grain boundaries alone does not necessarily mean that cavitation will occur even in a metal such as copper which normally does cavitate. Hence a further condition is also required to be satisfied.

The idea that grain-boundary jogs are significant (and that there is a link between slip lines and cavitation) goes back to the original proposals for heterogenous nucleation. The association of voids with slip lines was observed by Greenwood *et al* [11] and led Gifkins [51] to propose that an intersecting slip line trace would cause a jog in the grain boundary which could be opened up into a void by grain-boundary sliding. Chen and Machlin [40, 47] pointed out that any discontinuity in the grain boundary could act as such a nucleus. They remarked that the sliding of a grain boundary over a discontinuity could itself generate sufficient concentration of stress to cause a void to nucleate. They emphasized that not all jogs would open under the action of sliding because some would be of the compressive type shown in Fig. 2, an effect which is of importance in understanding the orientation of void-containing boundaries relative to the applied stress, as discussed below. Intrater and Machlin [45] commented on the need for local plastic deformation before a jog would fracture. The influences of such local

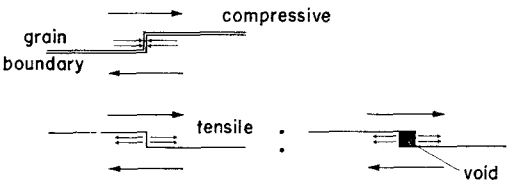

Figure 2.

effects are significant because nucleation of cavities is continuous during creep.

Davies and Dennison [52] argued against the model of Gifkins [51] that grain-boundary jogs would be flattened and removed by grain-boundary migration faster than they could open up by sliding. The significance of grain-boundary migration, which was also realized both by Nield and Quarrell [53] and by Crussard and Tamhankar [54], was subsequently demonstrated by Chen and Machlin [55] using copper polycrystals. They found that migration extended creep ductility and reasoned that this was because the migration destroyed boundary jogs thereby preventing nucleation. Similar observations were made later in nickel by Davies and Wilshire [56] and Dennison and Wilshire [57]. They observed that enhanced elongation, mainly in the tertiary stage, accompanied grain-boundary migration on recrystallization. Migration could be prevented by solute atom segregation at the boundaries, this, in preventing migration, enhanced cavitation. The effect of grain-boundary migration seems to be quite general. Inhibition of cavitation has been reported in copper by Intrater and Machlin [58], in magnesium by Harris [59] and, more recently, in zinc and its alloys by Singh, Rao and Taplin [60]. These latter also remarked that migration may alter the orientation of grain boundaries during creep, an effect which should be studied further.

Davies and Dennison [52] proposed an alternative jog formation mechanism in which a ledge is produced in a grain boundary by intersecting screw dislocations that have a screen component normal to the plane of the boundary. Such a ledge cannot be removed by grain-boundary migration. The model was criticized in his 1959 review by Gifkins [16] on the grounds that such a ledge would turn parallel to the sliding direction. A variation on the model was put forward by Crussard and Tamhankar [54] who suggested that the edge components of

intersecting dislocations create a step whilst the screw components enter the boundary.

Gifkins [16] reviewed all the mechanisms which had been put forward up to the time of his review. These included the proposal of Nield and Quarrell[53] which does not involve a direct concentration of the applied stress, but envisages the setting up of tensile and compressive forces at irregularities due to relaxation in the grain-boundary zone. Sliding will produce pores if these forces are not removed by grain-boundary migration. Gifkins [16] regarded their model as a generalization of earlier ones. A further model in which tension and compression regions are invoked, is that due to Ivanova and Oding [61]. According to their proposal, such regions cause the stress-directed diffusion of vacancies to microvoids. The existence of such microvoids, which was also envisaged both by Jenkins *et al* [13] and by Machlin [39], seems doubtful as they would probably sinter up [44] before growth could occur.

Gifkins [16] also considered the problem of nucleation quantitatively and came to the conclusion that the grain-boundary ledge idea was viable. The importance of grain-boundary sliding had been clearly demonstrated [45, 46]. Further, a possible role for grain-boundary inclusions is as anchors against sliding. This would explain the increased ductility of brass [62] through removing oxide inclusions by remelting it. The effect of removing impurities (through directional solidification) was demonstrated again later in copper by Boettner and Robertson [50].

Since that time, work has been carried out in a number of areas: the distribution of cavitated boundaries with respect to the applied stress, the continued nucleation of cavities throughout the creep test and finally the various attempts to incorporate observations into an acceptable model.

Historically, the next observation of importance occurred in 1961 when Boettner and Robertson [50] studied cavitation in copper, principally using density measurements. They demonstrated that cavity nucleation began at the very first stage of creep and continued throughout a creep test. They also concluded that heterogeneous nuclei, possibly some insoluble particles, are necessary. Optical microscopy is unable to resolve cavities at short times. Heslop [63] reported observing cavities in Nimonic 80 A and 90 only after some deformation had occurred. Tyler and Whittaker [64] observed cavities only in the secondary stage of creep in Magnox A 12. Using the greater resolving power of the electron microscope with a shadowgraphic method, Taplin and Barker [65] observed cavities in brass deformed to 0.1% strain.

The density observations of Boettner and Robertson [50] for copper were confirmed in magnesium by Ratcliffe and Greenwood [66] using high precision density measurements [67]. Cavitation begins at the beginning of the creep test and continues throughout it as shown by the cavity size distribution study of Brookes *et al* [68]. The nucleation and growth rates of cavities in copper in the primary creep range were measured by Gittins [69], again using density measurements. He found that they varied as $t^{1/2}$ and t respectively and remarked on the importance of grain-boundary sliding in nucleation. Greenwood [70] then pointed out that the rate of increase of void population as a function of time was not the same in all reported work: in contrast to Gittins [69], Ratcliffe and Greenwood [66] found a linear dependence in *time* which was in accord with the observations of Intrater and Machlin [45] and Price [71]. Greenwood [70] was able to show that all the data were in agreement with a model in which the number of cavities depends linearly on creep *strain*. The intercept of the curve is, however, off-set from the origin; this implies that a degree of strain is necessary before nucleation begins and also that nucleation is a strain-linked phenomenon. This was also noted by Johannesson and Thölén [72] in copper and steel.

Another dimension was added to cavitation studies by Hull and Rimmer [73]. They showed that cavitation in copper could be prevented by the presence of a hydrostatic stress equal in magnitude to the applied tensile stress. These results parallel the diffusion experiments of Barnes and Mazey [74] on copper-nickel couples where void formation can also be affected by hydrostatic stress. Taking advantage of these observations, Ratcliffe and Greenwood [66], experimenting with magnesium, confirmed the effect, and showed that preformed cavities do not continue to grow in the presence of hydrostatic pressure. They also found that, in a test begun under such a pressure which was subsequently removed, cavitation is unaffected by the primary deformation. Thus the rate of

cavity nucleation is unaffected by existing deformation. In later work, Ratcliffe and Greenwood [75] carried out tests, again with magnesium, under different stresses (strain-rates) with a hydrostatic pressure applied such that the difference between the applied and the hydrostatic stresses was constant. They could thus study the effect of deformation rate at a constant cavitation rate. The results showed that nucleation was not strongly sensitive to deformation rate and they concluded that either the sintering together of cavities was ineffective or, alternatively, that the voids reached a stable critical size very rapidly.

Transmission electron microscopy studies aimed at locating the nuclei for cavitation in magnesium have been presented by Presland and Hutchinson [76, 77]. They found that the majority (some 75%) of voids nucleated on cusps in grain boundaries resulting from the intersection of a sub-boundary. They envisage a combination of grain-boundary sliding and transcrystalline slip accompanied by rapid polygonization to form the sub-boundaries. They found [78] no evidence for nucleation on particles and claimed a qualitative agreement between prediction and observation, e.g. a temperature of maximum cavitation rate and a strain-rate dependence which also passes through a maximum. The proposals have been criticized by Harris [59], who favoured particle nucleation in magnesium, on the grounds that the spacing of the sub-boundaries is too short to allow an effective local stress build-up through grain-boundary sliding. He also noted, on the question of sub-grain-boundary nucleation in general, that copper which cavitates does not polygonize whilst aluminium which does not suffer from cavitation nevertheless polygonizes extensively during creep.

A further aspect of cavitation is the distribution of cavity-bearing grain boundaries with respect to the applied tensile stress which reflects both nucleation and growth. Greenwood *et al* [11] remarked that the cavities tended to form preferentially on boundaries approximately perpendicular to the applied stress. This is true in almost all cases reported since. The problem involved here is why the cavities should appear there instead of on boundaries at 45° to the tensile axis where it might be expected that grain-boundary sliding would be a maximum. An approach to this aspect was first made by Davies and Wilshire [79] who argued that the majority of ledges formed on the boundaries in the 30 to 60° bracket under tension should be of the compressive type (Fig. 2) envisaged by Chen and Machlin [40]. Referring back to their earlier proposal [80] that the ledges form mainly during primary creep by the intersection of slip line bundles with grain boundaries, Davies and Wilshire produced [79] ledges by pre-deforming in compression as shown in Fig. 3. Subsequent creep testing in tension caused voids to develop on the tension-type ledges which had been created by the initial compression. Quite a remarkable increase in cavities on boundaries in the 30 to 60° bracket was found in the dilute nickel alloy which they tested.

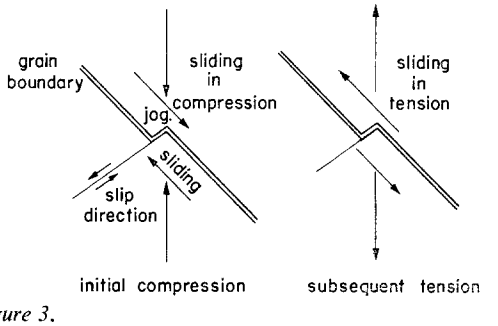

Figure 3.

The same type of experiment, with the same result, was carried out on Magnox AL 80 by Bowring *et al.* [81]. Prestressing also alters the distribution of cavities according to Dyson and Henn [82]. A maximum in cavitation on 60 to 90° boundaries in Cr—Mo steel was further reported by Day [83] and discussed in similar terms. However, these pre-compression experiments are not unambiguous because proven nucleation on ledges under such conditions does not prove that ledge nucleation is dominant in the absence of a pre-compression.

At about the same time as Bowring *et al* [81], Gittins and Williams [84] studied the relationship between cavitated boundary distribution and creep rate in copper. Their aim was to reconcile the growth models based on sliding and vacancy condensation which will be discussed below. Their results show, however, that the cavities tend to occur on the 60 to 90° boundaries only at slow creep rates. At higher rates, a shift to the 30 to 60° boundaries was seen. Included in their analysis was a correction of the apparent distribution measured on specimens sectioned parallel to the stress axis, to

the true distribution in three dimensions using a method developed by Scriven and Williams [85]. The result was contested by Davies *et al* [86], also working with copper, who found no such change in distribution over a wide range of strain-rates (the distribution is independent of stress level according to Davies and Wilshire [87]) using their own [79] method of correcting data to the true distribution. A shift in distribution following a compressive pre-strain was confirmed at the same time, as found before in nickel [79] and magnesium [81]. They claim [86] that their results still support the ledge mechanism for nucleation. As a result of the conflicting evidence [84, 86], Taplin [88] studied the distribution of cavitated boundaries in brass as a function of grain size, remarking that whilst Gittins and Williams [84] used copper of 0.13 mm grain size, that of Davies *et al* [86] was 6 mm. Using brass of 0.055, 0.24 and 0.43 mm, Taplin [88] showed a shift in distribution from 45° boundaries at the smaller grain size to 90° boundaries in the large grained material. The data were corrected following Scriven and Williams [85], but the correction did not change the apparent distribution very much and the conclusion not at all, as already noted by Davies *et al* [86]. Taplin concludes that a change in cavity growth mechanism is responsible for the shift in distribution. A comment on the estimating of void numbers and distributions was made by Gittins [89] who pointed out that a section through the several arms of a dendritic cavity of the type found by Taplin and Wingrove [90] may cause errors in counting.

Much of the experimental work presented above could be taken as evidence for nucleation by jogs, of some description, in the grain boundaries. As a result an attempt was made by McLean [91] to analyse a model for jog nucleation. He showed that a step 4 atom diameters in height could open up by grain-boundary sliding and be stable against the closing up imposed by thermal diffusion. It was envisaged that such a process could take place during a sliding burst of the type reported previously [36]. Harris [59] later showed the model to be unacceptable because an incorrect value for the diffusion coefficient was used in the numerical calculation. Introducing the correct value shows the step model to be untenable, a conclusion in which McLean [92] later concurred.

The alternative to the grain-boundary jog model in its various forms, is nucleation on particles or precipitates in the grain boundaries; this proposal was made very early on by Jenkins *et al* [13]. Early evidence [50, 62], mentioned above, rests on the reduction in cavitation which occurs when a material is purified by remelting, an observation which also negates gas pocket nucleation [39]. This reduction in void formation by remelting has been observed in brass [62] and copper [93], for example. The proposal was supported in the early review by Cottrell [44] who envisaged vacancy condensation on particles which had no lattice cohesion (i.e. non-wetting) as had Resnick and Seigle [62].

The experimental evidence for nucleation on particles is not completely unambiguous. For example, Hyam [94] found evidence for association of a large percentage of voids with particles in electron microscope studies of samples of a magnesium alloy fractured by impact before creep failure. He later concluded (in a private communication to Gifkins [95]) that the association was simply due to the voids enveloping the particles during growth. Harris [59] considered the nucleation problem for magnesium alloys and concluded that both sub-boundary/boundary [76-78] and grain-boundary ledges [91] were unlikely mechanisms on the basis of calculations which he presented. By a process of elimination he supported the particle model and also presented good arguments in its favour. Finally he showed that more than sufficient particles were present in his alloys to account for nucleation. In an earlier paper [96], he had found that approximately 5% of cavities in Magnox alloys were indeed associated with particles and surrounded by a network of new grains [97]. He concluded that particle nucleation and vacancy growth models could account for the behaviour of Magnox AL 80.

Association between voids and particles has also been seen in thoriated nickel by Wilcox and Clauer [98], the particles being regarded as probable nucleation sites. In fractographs of iron, Taplin and Wingrove [3] observed an association between γ—Fe_2O_3 particles and voids, a careful preparation technique [99] being employed in this study. Association of pores with large carbide particles on prior austenitic grain boundaries has been noted in bainitic steel by Tipler *et al* [100], and by Boniszewski and Eaton [101]. Johannesson and

Thölén [72] found voids associated with carbide particles but not with non-metallic inclusions. Finally, Williams and Lindley]102] have suggested an association between voids and cracked precipitate particles lying on grain boundaries.

The concept of a non-wetting particle was examined by Greenwood and Harris [103]. They showed that adhesion between matrix and particle must indeed be very poor for nucleation by vacancy condensation to occur. They suggest that the initial breaking of adhesion is most likely to occur by deformation. This view was first raised by Greenwood [104] and was repeated by him [105] in a 1969 review. Particles seem to offer a method of nucleating a void which will not tend to be closed up by sintering. He concluded that cavities are nucleated on fine particles as a consequence of deformation which may involve shear both within the grains and the boundaries.

The theory of nucleation was taken up by Smith and Barnby [106] who studied a Stroh-type model in which the stress generated by a dislocation pile-up is now supported by a *finite* thickness of material. The consequence of the model is that nucleation can occur at discontinuities such as particles at considerably lower stresses than had previously been thought. They concluded that the conditions for nucleation may frequently be met in practice.

A different role for grain-boundary precipitates was proposed in a 1966 review by McLean [42]. The presence of such particles should slow down grain-boundary sliding [16] and, hence, reduce the onset of cavitation. This reduction of sliding distance has also been discussed by Gittins [107]. Grain-boundary precipitates have been shown to reduce void formation in Nimonic 80 A by Weaver [4] and in other nickel alloys [108], including the recent work on Inconel X 750 by Venkiteswaran, *et al.* [109]. Marked changes in grain-boundary sliding in silver caused by the inclusion of inert oxide particles have been found by Raj and Ashby [110]. An alternative role for particles was advanced by Taplin and Whittaker [111] who proposed that the second phase may restrict void *growth* in aluminium-manganese alloys.

In summary, experiments have shown that grain-boundary sliding with a net stress across the grain boundary is an essential feature in the nucleation of cavities during creep. The cavities form on grain boundaries which are approximately normal to the tensile stress. They begin to form very early in the test and continue to form at a rate which is proportional to the creep strain. The sliding of the grain boundaries, which occurs in bursts, causes stress concentrations to be set up at irregularities in the boundaries. These local stress intensities cause boundary separation to occur and cavities to initiate. Indeed (as based on discussion with C. W. Corti) grain-boundary sliding will cause cavities to develop at irregularities whenever plastic flow or diffusion is not fast enough to prevent it. The role of the applied stress is to cause sliding and the development of local stress concentrations.

Two general types of mechanism have been invoked. First, steps or ledges in grain boundaries have been supposed to result from the intersection of slip traces or sub-grain boundaries with the grain boundary. Alternatively, any boundary irregularity may be significant. Second, local break-down of adhesion associated with precipitates or impurity particles, possibly of low adhesion with the matrix, has been suggested. Local plastic deformation seems to be a requisite in both cases, i.e. the creep strain is significant. The evidence in the first case rests on metallographic observations of cavitation and also on the ability to induce cavitation to occur on the 45° boundaries by pre-deforming to produce suitable steps. In the second case, association of cavities with particles in the grain boundaries has been observed by a number of authors. Removal of such particles increases ductility. This could, however, be due to the grain-boundary migration which increases ductility by preventing cavitation. An alternative effect, seen mainly in nickel alloys, is the reduction of grain-boundary sliding which also reduces cavitation.

The evidence for both mechanisms is persuasive, but does not indicate any particular mechanism as being universal. Indeed, it appears that several mechanisms are possible and that one or more may dominate [75, 84] in a particular instance. Cottrell and Aytekin [27] have pointed out that a number of mechanisms may operate during creep and that it may not always be the same process which dominates. Finally, the work of Smith and Barnby [106] may well imply that nucleation is not the problem theoretically which it had previously been thought [59, 91].

3.2. Cavity growth

In his original work Greenwood [9-11] had

envisaged both nucleation and growth of cavities to be occurring by vacancy accretion, at the high temperatures and low strain-rates involved. Growth should also be aided [11, 12]. by local stress concentrations at the void periphery. Support for vacancy agglomeration came immediately from the work of Balluffi and Seigle [38] who were able to correlate pore growth by a vacancy mechanism in brass where zinc atoms, diffusing out of the metal, form a counterflow. Later work by Resnick and Seigle [62] was in accord with these results.

The general problem of the growth of a spherical cavity located *away* from a grain boundary was considered by Machlin [39]. The model consists of draining a spherical volume of the surrounding lattice of its vacancies which are generated from dislocation jogs. Extending the model to growth *on* a grain boundary, then, for a given number of nuclei, enhanced creep leading to intercrystalline failure would follow. He was able to predict that increasing grain size should decrease elongation and that the product of creep life and secondary creep rate should be constant – a result which is reviewed in the next section.

A number of pieces of work then appeared which were cited as evidence in support of the vacancy growth model. Gifkins' model [51] for nucleation assumed vacancy growth. Hopkin [112] found antimony increased cavitation in brass and McLean [2] associated void growth in nickel alloys with a vacancy flow. However, Crussard and Friedel [113] pointed out that the vacancy flow here would be the reverse of that in Nabarro-Herring [114] creep.

Further work on brass by Chen and Machlin [47] demonstrated cavitation on grain boundaries perpendicular to the applied stress in tension and also parallel to it in compression. These cavities were not formed by dezincification. Chen and Machlin were unable to distinguish between the vacancy model and a growth mechanism based on local plastic flow under a local stress field, either was consistent with their observations. That the presence of stress markedly affects the distribution of voids during brass dezincification was shown by Hopkin [115]. A model for void growth by vacancy accretion was then advanced by Balluffi and Siegle [116] where vacancies, which originate in the transverse grain boundaries and flow to the voids, create a diffusion controlled system. The model predicts that no transverse void growth should occur under compressive stress; the applied stress, when in tension, plays the role of simply preventing sintering. No cavitation at all was indeed found to occur during compressive creep of magnesium, according to Stacey [117] or a steel by Day [83] but, as already remarked, longitudinal cavitation was found in brass by Chen and Machlin [47] and also in the same material by Bonesteel and Sherby [118]. The Balluffi-Seigle model [116] also predicts that twin boundaries are poor sources of vacancies because voids are not found on them [11]. Support comes from Barnes *et al* [119] who studied vacancy-aided helium precipitation in irradiated copper. However, a difficulty with vacancy growth had already been raised by Nield and Quarrell [53]. They found that the volume fraction of voids was not consistent with this type of growth. This observation was followed by the Chen and Machlin [47] study with the inability to distinguish vacancy from a local plastic deformation style of growth. Since that time evidence has accumulated to support both vacancy- and strain-controlled growth.

The vacancy growth model was pursued by Hull and Rimmer [73]. They studied creep in copper applying both tensile and hydrostatic stresses, σ and P respectively. As their initial results tended to support the model proposed by Balluffi and Seigle [116] of growth by grain-boundary vacancy diffusion, they developed an analytical model. They showed that, for the condition where failure was controlled by diffusional growth of a fixed number of voids, the creep life is related to the difference $(\sigma - P)$. They found firstly that no cavitation occurred when $\sigma = P$, secondly a creep life dependence on $(\sigma - P)$ was observed, but thirdly a σ dependence was seen which was not predicted. This last they attributed to a stress-dependent number of void nuclei which could be the result of grain-boundary sliding as suggested by Gifkins [51] and Chen and Machlin [40].

This general model found support in the work of Oliver and Girifalco [120]; a constant number of voids was reported to grow during the creep of silver. They also found the void size to increase linearly with creep strain. They concluded that growth occurs by the lattice diffusion of defects – probably divacancies.

However, in view of the evidence discussed in the previous section (that the number of voids is not constant during creep) the theory of Hull and Rimmer [73] was not acceptable in its

original form and was extended. Greenwood [104] set the increase in void fraction proportional to the rate of change of the population of voids of a given volume. The result is a void fraction which is proportional to creep time and strain. Such a relationship in terms of strain is required in the interpretation of later experimental studies [66, 69, 71] as subsequently remarked by Ratcliffe and Greenwood [70, 75].

A further extension of the Hull-Rimmer model [73] in the same direction is due to Skelton [121]. The increase of void number was included and a relationship of the form $t_f . \dot{\epsilon}^u$ = constant (t_f and $\dot{\epsilon}$ are time-to-fracture and secondary creep rate respectively) was derived. The value of the constant u is less than unity. It approaches unity as m, in the experimentally observed relation [122] $t_f . \dot{\epsilon}^m$ = constant, also approaches unity.

Experimental support for the vacancy growth model comes from a number of pieces of work. In some cases, however, the vacancy model was assumed and the experimental data were shown to be consistent with it. This assumption was made, for example, by Feltham and Meakin [123] who studied recovery creep in copper. Boettner and Robertson [50] found, in their studies of the same metal, that void growth began near the specimen surface and worked its way inwards. This observation tended to support the grain-boundary vacancy diffusion model of Balluffi and Seigle [116] with the surface acting as the principal vacancy source. The study tends to show that lattice diffusion can be neglected. They also found that void volume is linearly related to strain at a given temperature and stress level.

The voids found in magnesium by Presland and Hutchinson [76] had surfaces formed along the low index planes suggesting thermal equilibrium and a vacancy condensation mechanism. They found later [77, 78], partly using microradiography techniques, that two distinct types of cavity existed: polygonal as before and also wedge-shaped ones. The former were attributed to vacancy diffusion whilst the latter were associated with the occurrence of grain-boundary sliding. Differences in cavity type in magnesium alloys were also reported by Stacey [117] and discussed in terms of a change in growth mechanism.

Returning now to the Hull-Rimmer model [73] and its developments [104, 121], the observation that the condition $\sigma = P$ inhibits cavitation in copper has been confirmed for magnesium by Ratcliffe and Greenwood [66]. They showed that hydrostatic stress would suppress (further) cavitation no matter at what stage the creep was. These results are strong support for the vacancy mechanism, but the rate of cavity growth is too large to be vacancy-controlled, unless the number of voids increases during the test as was subsequently shown to be the case.

These hydrostatic pressure experiments are not universally accepted as critical evidence for vacancy growth. Machlin [46] remarks that the experiments only show that a net stress difference across the boundaries is necessary to prevent re-cohesion. The results simply agree with those of Chen and Machlin [55].

There is thus an incomplete amount of evidence (in the opinion of Gifkins [95] in a 1963 review) in favour of the vacancy condensation model, where grain-boundary diffusion [116] is the principal path. A number of objections have been raised to the model to favour strain-controlled growth. Apart from the early comments of Nield and Quarrell [53] and Chen and Machlin [47], Kramer and Machlin [124] found that creep strain and void area are linearly related in nickel. This rules out vacancy controlled void growth, although the creep process itself is reported [125, 126] to be dominated in this metal by a vacancy-producing mechanism.

Another objection to vacancy growth originates in the study of Intrater and Machlin [45]. Working in quite a wide temperature range of 650 to 900°C, they found that the area of visible voids was independent of temperature. In dilute alloys a linear relation between void volume and strain has been reported by Bowring *et al* [127], although in Magnox AL 80 the linearity was only achieved late in the creep life. Linearity with creep strain was also noted by Hensler and Cullen [128] in a series of magnesium alloys. They found the effect to decrease with decreasing grain size.

The effect of pre-existing voids was studied by Davies and Dennison [129] who compared samples produced by powder-metallurgical techniques (purposely to contain voids) with fully dense vacuum-cast samples of the same material. They showed the $t_f \propto \dot{\epsilon}$ relation to hold independently of stress and temperature. They went on to assume that the creep life had been reduced through an increased creep rate. This

results from the presence of voids which drain off any excess vacancies. The results also support those of Kramer and Machlin [124] on the void fraction/creep strain relationship and reflect the independence of creep rate from vacancy-controlled growth.

In a later variation on this experiment, Davies and Williams [130] studied the growth of dislocation loops in aluminium in the presence of grain-boundary cavities. They concluded again that the voids absorb vacancies thus preventing loop growth. The cavities are considered to become thermodynamically stable at the onset of tertiary creep.

The occurrence of angular cavities in magnesium, reported by Presland and Hutchinson [77, 78], has been confirmed also for Magnox by Harris *et al* [96]. This group emphasized that great care must be taken in the preparation of samples in order to ensure a correct image of the voids. This point has also been made by others [112, 120]. In addition, it has been found by Gittins (cited by Gifkins [95]) that the largest cavities occur on grain boundaries which have slid the most.

The shape of voids in α-brass and copper was studied by Taplin and Barker [65] using a shadowgraphic technique in the electron microscope. They found the cavities to be very irregular in shape and do not accept that diffusional processes can be responsible. They put forward a number of other mechanisms (a) crack propagation from void apices (b) tearing (c) internal necking with enhanced inter-cavity sliding, and (d) grain-boundary sliding.

Continuing the study of void shapes, Taplin and Wingrove [3] then looked at iron by electron microscope fractography, concluding that any discussion in terms of either vacancy- or deformation-controlled growth oversimplified the question of cavity enlargement. They found that grain-boundary sliding made a significant contribution to creep, but that precipitates retarded local cavity growth. They observed a number of types of cavities; crystallographic voids resulting from diffusional growth (the voids often contained the striations reported previously), irregular oriented cavities due to sliding and, on occasions, both types together. This mixture of types was also reported by Cocks and Taplin [99]. An interesting change in growth mode from fingered to rounded was also given [3].

In a further study of cavity morphology in iron, Wingrove and Taplin [90] found that a ductility minimum was associated with grain-boundary sliding and the dendritic finger-like type of cavities. Planar and faceted growths were also observed. The terracing reported previously [3] is thought to develop into facets. Dendritic growth is associated with high stress and high grain boundary sliding rates which enhance vacancy diffusion. Planar growth is favoured by a low temperature/high strain-rate or a high temperature/low strain-rate combination.

Cavities in fractures and sections of samples of both iron and copper were investigated by Davies and Williams [131], using the preparation method of Taplin *et al* [3, 99]. They found similar results and presented a mechanism for the formation of the dendritic type void: they envisate nucleation at a slip line/grain-boundary intersection [51] followed by a combined grain-boundary sliding and triple-point cracking mechanism where one of the boundaries forming the triple point is now the slip band which was associated with the nucleation process. A succession of such effects leads to a dendritic type of cavity as shown in Fig. 4. The faceting of voids has also been reported in CrMoV steel welds and their heat-affected zones by Boniszewski and Eaton [101]. The voids were formed on prior austenite boundaries and appeared to be associated with V_4C_3 particles. Working with the same material, Tipler *et al* [100, 132] found no void faceting. However, they reported that, although the lines of cavities are frequently normal to the applied stress, few of the voids are so-oriented.

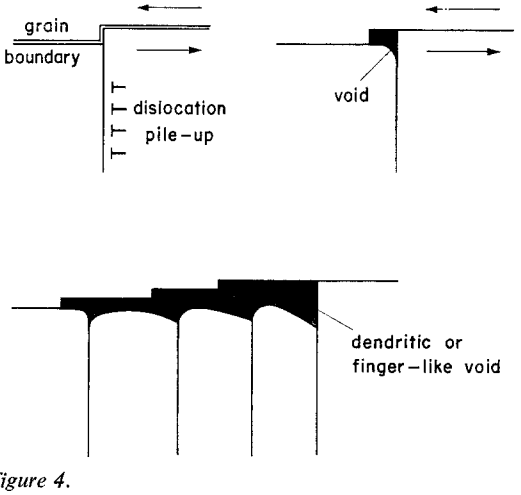

Figure 4.

The significance of the distribution of cavities with respect to the applied stress axis was discussed in the previous section with reference to nucleation. Differing results were obtained as function of strain-rate [84, 86], grain size [88] and prior compression [79, 81]. Taplin [88] has also drawn conclusions regarding growth mode. Referring back to his earlier work [133], he attributes the various observations to changes in growth mode. In fine grained material, sliding of grain boundaries tends to encourage growth on those which are oriented in the region of 45° to the applied stress. In contrast, coarse grained material tends to cause the voids to tear at their apices at boundaries approximately normal to the applied stress; thus the growth mechanism is not unique even for a given metal. He remarks in a later article [134] that sliding and tearing become more important at later stages of cavity growth.

Creep behaviour under the influence of a stress reversal has also been adduced for the mode of cavity growth. Davies and Wilshire [135] creep-tested dilute nickel alloys to the tertiary stage, a stress reversal produced compressive creep which was followed by a second stress reversal to recommence tensile creep. This latter stage was found to be tertiary. They argued that, if growth is by vacancy condensation, then compressive creep should cause void shrinkage. As the final creep stage was still tertiary, they concluded that void shrinkage had not occurred and hence void growth was not vacancy- but deformation-controlled.

A development of this type of experiment was carried out by Davies and Dutton [136] using constant stress equipment [137] to minimize barrelling. They reasoned that an intermediate compressive stress may only stop growth and not necessarily cause shrinkage. If, however, sliding is indeed the dominant process then stress reversal simply reverses the sliding direction and causes void shrinkage; in contrast, compression at 90° to the initial applied stress should maintain the initial sliding direction, thus causing the voids to continue to grow. These effects were observed by them in a copper-aluminium alloy.

Criticism of this type of experiment has come from two sources. Taplin and Gifkins [138] commented that reversed flow does not necessarily cause void closure but could cause continued growth. Void closure due to sintering governed by vacancy release can explain the Davies-Dutton [136] results but does not prove that growth had occurred by vacancy absorption or grain-boundary sliding. They regard the case as non-proven and reiterate their view [3, 65] that several modes of growth are possible, of which one or more may dominate under a given set of conditions.

A second criticism has come from Gittins [89] who conceded that the cavitation rate at high strain-rates was probably controlled by grain-boundary sliding [72] and at low strain-rates by a combination of sliding and vacancy accumulation [139]. He pointed out that sintering can occur on reversed stressing at room temperature [69] hence grain-boundary sliding is not a prerequisite. Also, sintering occurs under stress reversal during fatigue tests. He had shown previously [140] that voids can be made to grow and shrink through vacancy diffusion. In conclusion, all the work taken together points to a combination of tearing, vacancy accretion and grain-boundary sliding.

In response to these comments, Davies and Williams [141] reiterated their view that the effect of reversed sliding is that further growth by sliding is unlikely to occur; they had not stated that this was a sintering mechanism. They had shown that vacancy loss can be superseded by deformation-controlled growth. They do not accept that a vacancy contribution is significant in cavity growth.

Based on grain-boundary sliding, Spark and Taplin [139] have presented a general model for cavity growth. By analysing the grain shape in their brass samples they concluded that grain-boundary sliding is the dominant mode. They propose that the grain-boundary area occupied by voids is proportional to the degree of sliding. The rate of growth is strongly influenced by the grain-boundary area occupied, although growth is a diffusional process.

In view of the increasing amount of discussion in terms of a dominant controlling process, attempts have been made to find a transition from vacancy- to deformation-controlled growth. Day [83] was unable to distinguish between them in steel, nor were Chen and Machlin [47] in their early work with brass. The strain-rate dependent void distribution (with respect to the tensile axis) found by Gittins and Williams [84] was cited as evidence for such a transition but was criticized, as discussed above, and referred to a change in the mode of deformation-control by Taplin [88].

A change in control of growth has also been sought by Evans [142]. Working with a dilute magnesium-aluminium alloy, he concluded that growth was dominated by grain-boundary sliding. The rate of increase of void length is directly related to the sliding rate. The minimum in ductility is attributed to a maximum void growth rate and its parallel maximum sliding rate. The sliding rate at higher temperatures is decreased by the formation of grain-boundary cusps.

A minimum in the relation between creep rate and grain size was reported by Wilshire [34]. He found that it increased as the temperature increased and again discussed the effect in terms of the increasing importance of grain-boundary sliding with increasing grain size.

A further criticism [92, 127] of vacancy-control comes from the observation that the diffusion coefficient is hardly affected by alloying whereas creep rates can change markedly; indeed this is the basis of the design of creep-resistant alloys. Strain and degree of cavitation are always related as discussed in the previous section.

An interesting attempt to produce a model in which both deformation within the grains and vacancy accretion play a role has been made by Ishida and McLean [92]. They envisage dislocations moving down slip lines within the grains to intersect a grain boundary. Under the action of the tensile stress they migrate along the boundary which can only be done by climb (still retaining their Burgers vector [143]). The vacancies emitted during the climb process migrate to a growing cavity. The dislocation also exerts a force on this cavity, tending to open it, which is proportional to the distance between the cavities. The model is of particular interest because it relates growth to grain-boundary sliding.

This model has been invoked to explain experimental observations in dilute nickel and magnesium alloys by Bowring *et al* [127] and in copper and a steel by Johannesson and Thölén [72].

From the foregoing it has become apparent that there is no longer a question of deciding between vacancy- or strain-controlled growth. It is also clear that the same mechanism does not necessarily apply in a given material at all times. Indeed, a change in dominant mechanism in a single test can be envisaged.

The early review of Gifkins [95] in 1963 justifiably quotes the support for the vacancy model as incomplete. The mechanism may well control in some instances and would justify the dicussion of creep in magnesium in such terms by Harris [97]. The review of Greenwood [105] in 1969 also tends to support vacancy accretion in the light of the experiments involving hydrostatic pressure. However, these experiments are not without critics. As remarked above, Machlin [46] has given an alternative explanation. Taplin [134] gives support to the possible role of vacancies at low strain-rates. In contrast Wilshire [144], writing in 1973, tended to negate the evidence for vacancy growth because of the results of reversed stressing experiments. If it is accepted that several mechanisms are possible, then evidence that vacancy absorption by voids and their resultant growth is nevertheless persuasive. Evidence in favour of deformation-controlled growth is far stronger. The non-sphericity of cavities, the need for tension across the boundaries to avoid sintering, the temperature-independence of void growth, the marked effect of alloying which does not alter the diffusion coefficient, all can be cited [92, 95, 105, 134, 144] in support. In addition, the relationship between cavitation and creep strain, and between the rate of change of both and creep life is also indicative [144].

Finally, there is a good case to be made for several types of deformation-controlled growth [134].

3.3. Tertiary creep and fracture

In earlier studies, fracture was discussed simply in terms of the linking together of cavities to form a crack. Chen [41] viewed the presence of voids as a prerequisite of crack propagation whilst the linkage of voids by cracks was studied experimentally in brass by Chen and Machlin [47]. Weaver [145] observed though that such an intercrystalline crack would be stopped on intersecting a twinned region in one of the adjacent grains. As a result he envisaged twinning as a mechanism for delaying fracture, although he pointed out that such a process would stress the grain-boundary triple points. The propagation of such cracks was attributed to stress concentration at the crack tip by Greenwood [10-12]. Finally, a theory of propagation was presented by Fujita [146] in which a crack a few atoms wide and of the order of 100 to 1000 Å long was shown able to grow as the result of

the stress field generated by piling up dislocations.

Later work has tended to highlight the later stages of cavitation creep in rather more detail. The aspects may conveniently be divided into the studies concerned with the onset of tertiary creep and its relationship to secondary, the various types of failure mechanism and, finally, the prediction of creep life as based on the secondary creep rate.

The study of tertiary creep as a problem in itself was begun by Davies and Dennison [129]. They experimented with sintered nickel base alloy powders in comparison with the bulk metal. They found that the pre-existing voids in the sintered samples drained off excess vacancies thereby causing an increased creep rate. As an outcome of their experiments, they concluded that the onset of tertiary creep is a direct consequence of the existence of the grain-boundary voids. They went on to suggest that the voids reach a critical size at this point as related to the degree of grain-boundary sliding. Following Kramer and Machlin [124], the void area corresponds to a specific elongation. Thus, when the voids have achieved the critical size, tertiary creep begins and fracture processes become significant.

A number of further pieces of work was carried out to characterize tertiary creep in the dilute nickel-base type of alloy used in the above-discussed work. Davies and Wilshire [56] found that the effect of purity on primary and secondary creep was slight, whilst the tertiary elongation was affected very much. They concluded that low elongation is associated with the lack of a facility to recover. Dennison and Wilshire [57] showed further that the onset of tertiary creep is hastened by higher purity which also leads to higher ductility at fracture because of grain boundary migration. Additions of gold and silver increase creep life according to a later paper of theirs [147].

Some further observations by Davies and Wilshire [135] on tertiary creep showed that a period of compressive creep (when a sample is in tertiary tensile creep) does not stop the sample continuing in tertiary when the stress is returned to a tensile state.

Ratcliffe and Greenwood [66] found the rate of change of density in magnesium increased just before fracture. This change reflects an increased rate of cavity growth which was discussed in vacancy-aided growth terms by them.

The association between grain-boundary cavities and the onset of tertiary creep was studied in gold samples by Davies and Evans [148]. They followed the creep curve until the tertiary stage began and then annealed the samples before continuing creep testing. In this way they were able to demonstrate that tertiary creep only occurs when the voids are actually on the grain boundaries, voids within the grains have no effect. They also showed, incidentally, that the creep properties could be recovered by suitable annealing.

Davies and Dutton [149] showed that tertiary creep is associated with an increased rate of recovery and not a change in work hardening. In compression, tertiary creep is related to specimen barrelling and is not related to the existence of voids even if they continue to grow during this stage. In contrast, in tensile creep, the presence of cavities is a necessary condition for the onset of tertiary creep [148] and, further, their stability is also significant.

This latter aspect was taken up by Davies and Williams [130] who also studied the effect of cavities on vacancy diffusion following Davies and Dennison [129]. They proposed that the onset of tertiary occurs when the cavities become thermodynamically stable. In a later article, Davies and Williams [33] showed that the beginning of true tertiary creep occupied a transitional period during which the time dependence was $Kt^{4/3}$ until true tertiary and an exponential $A\ \exp(\beta t)$-type of behaviour was achieved. They attributed the transition to the achievement of the critical size for thermodynamic stability by the array of cavity sizes present.

The effect of grain size on the onset of tertiary creep was studied by Wilshire [34]. He found that the parameters m, p and $\dot{\epsilon}_s$ in the generalized creep Equation 2 all vary in the same manner with grain size. He concluded that the same basic mechanism of creep controls all stages. Cavity growth governed by grain-boundary sliding can explain these results where failure is controlled by the deformation rate.

In summary, the onset of tertiary creep is the direct result of the existence of voids on the grain boundaries. The tertiary stage of creep is controlled by the same mechanism which controls the earlier part of the creep curve, namely deformation and recovery. The growth of the voids is controlled by grain-boundary sliding.

The mechanism by which the cavities link up has also received attention though, historically speaking, relatively later in the study of cavitation. Evidence for tearing was offered by Seigle [150] who showed that the fracture edges match in deoxidized copper with little grain-boundary sliding. At about the same time a different type of fracture in copper, as associated with voids, was discussed by Rogers [151, 152]. He found that the area between the voids necked and fractured locally in a ductile manner, provided that the metal did not work harden too much. This is termed the void-sheet mechanism.

The implication, that the final mode of fracture may not be unique, was taken up by Taplin [133] a few years later. He attributed the onset of failure to the ease with which voids are able to interlink to form a crack capable of spontaneous propagation. Factors which are significant here include grain boundary smoothness which favours interlinkage and also the presence of precipitates which restrict linkage. Also significant is increasing precipitate refinement as found by Hensler and Cullen [128]. Taplin [133] also discussed the void-sheet mechanism of Rogers where the reduction of specimen cross-section by the voids has a dominant effect as in aluminium-manganese for example. In contrast, voids in brass linked until the grain boundaries were filled to permit crack propagation by the Griffith mechanism. The possibility that void shape might also influence the tendency to form cracks was pointed out by Krishnan [153].

The work on brass and also on copper was pursued by Taplin and Barker [65] in their shadowgraphic studies. They concluded that tearing due to the concentration of stress at cavity apices and concomitant grain-boundary sliding, control crack propagation. They also made the interesting comment that a vacancy condensation mechanism will affect the crack tip by blunting it, thereby possibly increasing ductility.

The fracture of iron, in which dendritic-type cavities were found by Taplin and Wingrove [3, 90], has an associated ductility minimum. This minimum is related to the condition of maximum grain-boundary sliding which supports the influence of this latter parameter as has been discussed above. The importance of grain-boundary sliding in combination with grain deformation was emphasized by Davies and Williams [131] also studying iron.

An aspect of crack propagation by cavity linkage, which is also significant, is the effect of grain size. In some materials cavities link until the grain boundary is filled. If the critical Griffith crack length is exceeded in the process then failure follows. If not, then an interaction between cavity groups on different boundaries is necessary for crack propagation. This aspect was studied by Cocks and Taplin [154] who followed this feature of the grain size effect. Small grain sizes do require such an interlinkage between grain edges for failure by plastic tearing. At very small grain sizes a different mechanism is preferred, this is the ductile sheet effect where failure is dominantly related to loss in specimen cross-section rather than to cavity linkage. However, at these small grain sizes, the microstructure is not completely stable and boundary migration may occur to hold off failure.

These features of the grain size effect were also found in copper by Fleck *et al* [155]. In coarse grained material, failure is a question of critical crack length. In fine grained material in which such a critical crack cannot be formed on the given grain edges because they are too short, then failure occurs by the ductile sheet mode.

Cracking between cavities to lead to failure (at least in rather coarser grained metals) is not confined to the simple systems. Boniszewski and Eaton [101] showed that maze-like cracking between cavities was responsible for failure in a CrMoV steel. Working with the same material, Tipler *et al* [132] found crack linkage with very little cavity coalescence. The resulting fracture resembled a honeycomb structure.

In a later article [134] where his grain size work was reiterated, Taplin remarked on the fact that the interlinkage of cavities, and the propagation crack thereby produced, was stopped at a triple junction. The contrast with failure by triple point cracking at stresses above the Stroh-McLean transition clearly requires investigating.

The question of deriving an estimate of life-time, when cavitation is the failure-controlling mechanism, has received attention. As early in the studies of creep cavitation as 1956 Machlin [39] developed the first model for rupture life. He assumed that voids grew by vacancy accretion and was able to predict the relation mentioned above, namely that the product of secondary creep rate $\dot{\epsilon}_s$ and rupture life t_f is invariant. He pointed out that this relation is in accord with

observations given in the literature [156, 157]. It should be repeated [158] in passing that some of the data considered by Machlin were for aluminium, which does not fail by cavitation. Inherent in the Machlin model [39] is the assumption that secondary creep rate and the vacancy production rate are the same, where the secondary creep rate is being used here as an approximation to the overall average creep rate [158]. This inherent assumption implies the identity of creep rate and cavitation rate. Putting the model in a more general frame (which is necessary because much evidence points to deformation-controlled void growth) Machlin's [39] conclusion holds true whenever the mechanism controlling both the creep rate and the cavitation rate is the same, be it vacancy- or deformation-control. This is apparent in an alternative explanation of the $\dot{\epsilon}_s.t_f$ product advanced by Machlin [48] some years later. Attributing fracture to a critical void area in the cross section, then this is controlled by a critical degree of grain-boundary sliding [45] and hence of total elongation. This last is approximately given by $\dot{\epsilon}_s.t_f$ and hence the relation follows.

The constancy of the product $\dot{\epsilon}_s.t_f$ has since been demonstrated in many systems. Examples are the work of Monkman and Grant [122], and Davies and Dennison [129]. The value of the product is affected by alloying additions in a given metal, for example in nickel, according to Dennison and Wilshire [147]. The applicability to superalloys and copper has been discussed by Feltham and Meakin [123, 158]. Not all systems follow the relation, however, the case of gold where a large primary creep stage occurs, has been discussed by Davies *et al* [159].

Hull and Rimmer [73], also working with a vacancy model, predicted lifetimes dependent upon the difference between an applied tensile stress and a hydrostatic pressure. They find a dependence on void fraction as well, which itself is stress dependent. The Hull-Rimmer model [73] has been criticized as discussed in previous sections. The derivation of a rupture model in particular, was put in doubt by Greenwood [104] on the basis that continuous void nucleation is the rule rather than the assumed constant void fraction. A further criticism comes from Speight and Harris [160] who point out that one other assumption may not always be valid, namely that fracture will occur when the growing voids meet.

An attempt has been made by Skelton [121] to correct the Hull-Rimmer model by incorporating continuous void nucleation. Following the earlier models where growth and creep rate are vacancy-controlled, he derives an equation of the form $(t_f.\dot{\epsilon}_s)^u$ is constant, where the constant u tends to unity as the value of m in $(t_f.\dot{\epsilon}_s)^m$, the constant of Feltham and Meakin [123], and Oding and Burdukski [161], also tends to unity.

A somewhat different aspect of rupture life estimation comes out of a study by Davies *et al* [24]. Following the well established approach that failure is directly attributable to the presence of voids [148], they studied the parameters of the creep process through the constants in the generalized form of the creep Equation 2. That is, the process of cavitation and creep are taken to have the same controlling mechanism. They find the parameters of Equation 2 all to be related to the steady creep rate, as was found earlier [23] for primary and secondary, also that the contribution from the tertiary stage is eight times the steady creep rate. Extending this work to grain size effects, Wilshire [34] confirmed the dependence of all stages of creep on the same mechanism because they all have the same grain-size dependence. He concludes that cavity growth by grain-boundary sliding can explain the observations where failure is controlled by the deformation rate. Failure occurs when the critical crack length for spontaneous growth is achieved.

3.4. Impurity effects

Impurity effects tend to occur either as a result of the inherent purity level of a metal under examination or as a result of gaseous pick-up during testing.

There is evidence that the gaseous envelope employed during tests does have an effect on cavitation. Apart from the stabilizing of cavities by gas envisaged by Greenwood *et al* [11], an indirect effect was reported by Hopkin [115] who showed that brass creep-tested in vacuum failed by cavitation but that no cavities were observed in an atmosphere of nitrogen. This observation is related to the prevention of zinc loss and its concomitant vacancy generation. In copper bicrystals the atmosphere played a more direct role. Intrater and Machlin [45] found cracking in a poor vacuum but only isolated void formation in a hydrogen atmosphere. The oxygen partial pressure in the

vacuum (5×10^{-4} Torr) was considered responsible for the development of pores into cracks. They later found [36] the activation energy for grain-boundary sliding to be 40 kcal mol^{-1} in vacuum but only half that value in hydrogen. The difference was attributed to a change in grain-boundary migration. Price [71] found that oxygen inhibits grain-boundary sliding in silver.

In contrast, Hensler and Cullen [128] found that with either a vacuum or carbon dioxide atmosphere, the results for magnesium alloys were the same. Gas stabilization as foreseen by Greenwood *et al* [11] has been investigated by Gittins [69, 140]. He found that cavities which had been formed during creep of copper were reduced in size by annealing under hydrostatic pressure. They regrew when subsequently isothermally annealed in the absence of stress or pressure. All testing was carried out at the same temperature of 400°C.

Effects of impurities in tested samples have been reported by a number of authors. Hopkin presented evidence that antimony encourages vacancy condensation on grain boundaries in both brass [112, 115] and copper [162]. McLean [2] also reported a harmful effect of grain boundary-segregating impurities on interfacial energy in superalloys. This effect of lowering cohesion at grain boundaries was brought up by Gifkins [16] in his 1959 review. He came to the conclusion that the concept of crack initiation through such a reduction in cohesion shows no dependence on the deformation mechanism nor can it explain the observed crack types. Nevertheless the concept of interfacial energy reduction was again used to explain the effects of antimony on copper by Tipler and McLean [163] where the reduced ductility with increasing creep rate was associated with a change in the rate and degree of cavitation.

Other evidence of impurity effects comes from vacuum remelting studies; it is however conflicting. Reid and Greenwood [43] found such treatment to have no effect on cavitation in Cu—Ni alloys. In contrast, Boettner and Robertson [50] could not get cavitation in copper samples from which insoluble phases had been removed. They found that the degree of purity played a large part in cavitation kinetics; a variation of up to a factor of two in the degree of cavitation could be observed in nominally the same material tested under the same nominal conditions.

A direct influence or purity level in nickel has been established by Wilshire [164]. He used it to study creep mechanisms as based on a dislocation jog model and concluded that creep in this metal may not be controlled by dislocation climb. The major influence of purity in nickel is in the tertiary range. Davies and Wilshire [56] attributed the marked variation in tertiary creep engendered by changing the purity level to changes in the facility for grain-boundary migration and recovery. Decreasing purity increases creep resistance and rupture life, but the product $\dot{\epsilon}_s . t_f$ is unaffected according to Dennison and Wilshire [57]. Such effects may depend upon the impurity level because Wood and Cook [165] found a number of elements Pt, Bi, Tl and Te to be particularly harmful at the 5 to 20 ppm level. In contrast, additions of 1% of gold or silver made by Dennison and Wilshire [147] improved creep resistance and affected the product $\dot{\epsilon}_s . t_f$. Silver caused a decrease whilst gold increased both the value of this product and also the degree of primary creep. Again Gibbons [166] found the removal of lead by vacuum remelting improved the creep ductility of Nimonic 80 A.

In his study Price [71] found that impurities at the grain boundaries reduced sliding. He also reported that both types of impurity effect can occur together and may indeed interact. Specifically he found the grain-boundary sliding reduction, which is caused by impurities, to be enhanced when tested in air. This he attributed to oxidation.

4. Nickel-base superalloys

The so-called superalloys of nickel are high strength, highly creep, hot corrosion and oxidation resistant materials which are used extensively for turbine components operating at elevated temperatures. The materials comprise some 50% by weight of current engines. Their metallurgy is complex but, in general, well understood. They normally contain at least a dozen principal alloying elements together with numerous trace additions. As a result, several phases may be present; each of which may appear in a number of morphologies. Superalloys have been and still are under continuous development. A number of excellent reviews have been given e.g. by Betteridge [108] in 1959 and by Decker and Sims [167] in 1972.

For the purposes of reviewing cavitation creep in the nickel-base superalloys, a somewhat

simplified description of the microstructure is sufficient. The superalloy matrix is a face-centred cubic austenitic phase (γ). The most important precipitate occurring in it is based on the intermetallic Ni_3Al (γ'). This phase is possibly unique in that it is strengthened by antiphase boundaries and gets stronger with increasing temperature. The γ' phase is coherent with the matrix of a rather spherical morphology and is of the order of 10 nm in size. In advanced alloys it may be larger (0.5 μm) and of a cubic morphology. Both the γ and γ' phases are strengthened by solid solution additions which also increase their stacking fault energy, reducing cross-slip. Other additions such as cobalt increase the volume fraction of the γ' phase. Elements which partition mainly to the γ phase are cobalt, chromium, molybdenum and tungsten, whilst titanium, tantalum, niobium and aluminium are found in the γ'. This latter phase also appears at grain boundaries and grain-boundary carbides in some alloys and is also found as "γ-γ' eutectic" nodules in cast alloys. γ' size and morphology can usually be controlled by a suitable heat-treatment to achieve the desired mechanical properties.

A second important family of phases are the various carbides. Primary MC carbides occur in a blocky spherical form distributed heterogeneously throughout the alloy both within grains and at their boundaries. Also formed are the $M_{23}C_6$ carbides either as a result of heat-treatment or a reaction of the following type: $MC + \gamma \rightarrow M_{23}C_6 + \gamma'$ during service. $M_{23}C_6$ occurs frequently along grain boundaries which are thereby inhibited from sliding so long as the carbide is in the form of discrete particles. The third type of carbide is M_6C which exists over a wider range of composition than this formula suggests. It forms at higher temperatures than $M_{23}C_6$ but, like it, often through MC decomposition. It is more stable than $M_{23}C_6$ at high temperatures and is also found at grain boundaries where its stability leads it to be used in controlling grain size in wrought alloys.

Major alloying additions made to superalloys are designed to alter the composition, morphology and distribution of the phases present. A further category of alloying elements comprises those which, because of atomic size, tend to segregate to grain boundaries. Examples are boron, magnesium and zirconium. They improve creep life and elongation quite markedly, although the mechanism is not understood.

The majority of earlier alloys was of the wrought type, but many of the more recent developments are casting alloys. Both types can be subjected to heat-treatment to control and optimize the distribution of γ' and carbides, grain size etc. In terms of grain size control this has to be set to the optimum for a suitable compromise between improved creep strength and decreased tensile and fatigue properties which follow as the grain size is increased.

4.1. Creep cavitation in superalloys

The mechanism of creep in superalloys is the Bailey-Orowan recovery type. This was demonstrated in Nimonic 80 A by Sidey and Wilshire [168], who found the creep resistance to be controlled partly by γ' acting as dislocation obstacles but mainly by its effect on the rate of recovery. Recovery creep was also found to operate in a similar but carbon-free alloy by Hopkins and Gibbons [169] where the recovery rate was controlled by the grain size and γ' volume fraction. They also found the dislocation structure to be consistent with recovery occurring at the grain boundaries – an observation also made earlier by Gibbons and Hopkins [170]. Grain-boundary sliding and extensive recovery, below the recrystallization temperature, has been found in Nimonic 108 by Mayes and Hancock [171].

Failure in creep occurs by one of two distinct mechanisms. As discussed previously, cavitation is responsible for failure only at low stress levels. At higher stresses beyond the so-called Stroh-McLean transition [2, 5], cracking begins at grain-boundary triple points. This is the Zener mechanism [6] and was firmly established in nickel-chromium alloys by Chang and Grant [7]. Heslop [63] has shown that two distinct types of triple point failure occur. In one there is cracking in the regions neighbouring the final failure whilst in the other type, no such adjacent cracking occurs. He ascribes the change to the lack of ability for the matrix to deform so that a crack, once formed, propagates catastrophically. Working with samples in which some ductility existed, McLean [2] noticed that the propagation of a triple point crack could be stopped at an intersecting twin. This observation was pursued by Weaver [145] and associated with the lower free energy of a twin boundary. He found that a crack could be held up for a while or stopped completely by such an intersection. He concluded that whilst twinning may slow down

creep, the loading of the triple points would be enhanced so that the situation would be comparable with reducing the grain size.

The transition from triple point to cavitation failure was studied in a number of superalloys by McLean [2]. He found that, with the exception of Nimonic 80 where very few voids were seen, the transition was quite sharp but occurred at a different stress for each alloy. He analysed the change in fracture mode in terms of Stroh's equation [5] for the fracture of a sliding interface. Weaver [4] found that the transition is not unique for a given alloy but that microstructural details such as grain-boundary carbides had a distinct effect. He commented [172] that the effect of the carbides is to reduce the possible sliding distance and hence the tendency to triple point cracking. The transition in superalloys has also been studied by Heslop [63]. The Stroh equation has been reconsidered and corrected for use in samples of finite thickness by Smith and Barnby [106] who showed that the transition would occur at lower stresses than predicted by the original equation.

The occurrence of cavitation in Nimonic 80 A was shown by Weaver [4] to depend on grain-boundary structure and the presence of carbide particles. Both the number and the size of cavities increased with fracture strain and much of the elongation is due to the development of the cavities. The voids form preferentially on the transverse boundaries between carbide particles. They do not form on the particles which can also stop crack propagation. In a later paper [173] he reported that the optimum micro-structure consisted of coarse grain-boundary carbides with an associated γ' duplex structure. The size of the carbide particles is important apparently because a fine dispersion leads to low creep ductility. The absence of γ' is also deleterious. The significance of coarse carbides has been questioned by Gibbons and Hopkins [170] studying carbon-free alloys. These had poor ductility but it was found possible to correct this by adding niobium. They showed further that the effects of grain size and γ' volume fraction are not independent: the grain-size dependence of the creep rate developed progressively as the γ' volume fraction was raised.

The number of voids was shown to increase linearly with strain at a number of stress levels in Nimonic 80 A by Dyson and McLean [174]. They proposed the use of the state of cavitation as a guide to the creep life remaining to a part in service. This proposal was covered in his 1973 review by Wilshire [144] who commented that cavities are often only visible towards the end of creep tests so that predictions during the early part of the creep curve are not possible. However, at later stages where cavitation becomes well developed it may prove to be possible to recognize a critical situation. A further aspect is the effect of purity and variations in composition between heats of the same alloy. The former was discussed above whilst the effect of the latter can be seen in the data given by Dyson and McLean [174] where the utilization of samples from a different heat had quite an effect on the cavitation characteristics.

Three further aspects of cavitation have been studied, namely: the prediction of creep life by extrapolation, the effect of prestraining and the possibility of regenerating creep properties by annealing treatment.

Prediction of time-to-failure has received a certain amount of attention although one major problem is structural changes in service which were touched on in the preceding section. Monkman and Grant [122] fitted an equation of the type $\log t_r + m \log \epsilon_s = \text{constant}$. They found values of m to be of the order of 0.8 in Nimonics 80 and 90 and Inconel 700. Feltham and Meakin [158] applied the same type of equation to data from Nimonic 90 and found $m = 1$ in contrast to the earlier work. Betteridge [175] compared a number of extrapolation methods and concluded that the form due to Manson and Haferd [176] was the best:

$$\frac{T - T_a}{\log t - \log t_a},$$

where T_a and t_a are constants to be determined. This equation is a development of the oft-used Larson-Miller [177] parameter.

The influence of pre-strain at room temperature on the cavitation characteristics in Nimonic 80 A has been studied by Dyson and Henn [82]. They were interested in finding out whether pre-straining in itself could give rise to cavitation or whether a period of stress under creep conditions was also necessary. Therefore, they heat-treated samples with and without stress and concluded that non-propagating cavitation is a direct consequence of grain-boundary decohesion during pre-strain. They also concluded that creep processes such as grain-boundary sliding are not a necessary condition for void formation. In a later article, Dyson [178]

reiterated this view point and noted that the degree of cavitation was considerably greater following a pre-stress. He proposed that nucleation due to pile-ups was operative and that the release of residual stress from the pre-loading was responsible for growth. These effects would not exist under normal creep conditions in the absence of pre-strain, and leads to some doubt being cast on the validity of the earlier conclusion that grain-boundary sliding is not necessary.

The possibility that creep properties could be regenerated by a heat-treatment was demonstrated in gold by Davies and Evans [148]. They showed that separating the grain boundaries from the cavities formed during creep (and which are responsible for the onset of the tertiary stage and failure) resulted in renewed creep resistance. Davies *et al* [179] studied Nimonic 80 A from this point of view and carried out repetitive annealing treatments following the onset of tertiary creep. They found that properties could be recovered in this way because the voids involved were small and probably annealed out. The number of possible repetitive regeneration treatments was found to be limited by a number of factors. High temperature annealing led to over-ageing and carbide coarsening resulting in a reduced creep life (as already reported by Betteridge and Franklin [180]). Oxidation of cavity surfaces and the development of an internal gas pressure can stop the pores from sintering up.

At about the same time Friedl [181] reported the results of a regeneration study of a number of alloys including a nickel-base superalloy. He found material to become progressively more ductile and notch sensitive. Coupling these results with the microstructure, he concluded that not all the changes induced by creep can be recovered; some non-microstructural effects are not affected by recovery heat-treatments. These seem to result in some form of slipblocking.

In a later article, Davies *et al* [182] studied recovery in samples taken to different degrees of tertiary creep in Nimonic 80 A. The sintering out of voids is controlled by lattice self-diffusion. The length of annealing treatment which is necessary to permit recovery increases with the creep strain. However, there is a limit beyond which regeneration is no longer possible. In the alloy studied it was found that a secondary creep stage could not be reinstituted after the initial creep had been allowed to attain a rate of eight times the secondary creep rate.

Regeneration was studied in a number of superalloys, namely Nimonics 75, 90 and 105 and Incoloy 800, by Hart and Gayter [183]. They found regeneration to be viable but that some grain growth occurred. They propose that the heat-treatment, in addition to returning the microstructure to its original condition, also causes a redistribution of the dislocation network. Grain-boundary migration removed the cavities from damaging sites. The problem still remains extant of how to determine the extent of creep damage in service parts before the voids become apparent.

The general problems associated with regeneration, namely the separation of grain boundaries and cavities and the effects of over-ageing, internal oxidation and gas build-up were emphasized by Wilshire [144] in his review.

4.2. Related effects

Cavitation as a subject in itself has only been studied relatively sparsely. The presence of grain-boundary carbides and precipitate-free and chromium-denuded zones complicate the problems of void nucleation and growth mechanisms. However, conditions which lead to extensive ductility must also be those where cavitation is reduced. These aspects are now discussed briefly.

The nature of grain-boundary microstructure, as affected by heat-treatment, and its effect on creep properties has been investigated. Betteridge and Smith [184], working with Nimonics 80 A and 90 (and some compositional variations of the latter), showed that not only are grain size and precipitation important but also the condition of the grain boundaries themselves. Specifically, carbide precipitates are desirable for good creep properties. In a later article, Betteridge and Franklin [180] confirmed as beneficial the presence of chromium carbide in the boundaries. Indeed, whereas the time and temperature of heat-treatment largely determine the creep rate, the total elongation is controlled by the grain-boundary carbides. The importance of carbides and also their size was emphasized by Weaver [4, 173] in work on Nimonic 80 A. However, some doubt about the role of carbides has been cast by Gibbons and Hopkins [170] in their work on carbon-free alloys where niobium has a beneficial effect on creep ductility.

Betteridge and Franklin [180] also suggested

in their study that the formation of the chromium carbides at the grain boundaries was accompanied by the development of a chromium denuded zone. This zone has a lower stress-carrying capacity but would confer the facility of accommodating local stressing effects thus counteracting any tendency to boundary decohesion and cavity formation.

The existence of such a zone was investigated by Fleetwood [185] in Nimonic 80 A which he heat-treated in various ways and then subjected to a microprobe analysis. He found that such a chromium denuded zone did exist in samples which had been heat-treated to give the optimum of creep properties; other treatments did not always generate such a zone. The local chromium loss was compensated by a higher nickel level, such that the sum of the two elements was invariant across the boundary width.

A similar type of zone was observed in some nickel superalloys by Fell [186], who found γ' precipitate-free zones adjacent to the grain boundaries when chromium carbides were present in them.

Further work has been done by Gibbons [187] on carbon-free alloys of nickel-chromium-γ' type [169, 170]. He also found a denuded zone in Nimonic 80 A but it was considerably narrower than that reported by Fleetwood [185]. The work also indicated extensive polygonization within the zone, which supports the stress relaxation ideas advanced by Betteridge and Franklin [180].

Effects due to these zones have been discussed by Heslop [63] with reference to his work on fracture type. He follows the arguments advanced above that the denuded zone can accommodate stress concentrations. Heslop points out that the weaker zones could also lead to too much local deformation and premature failure. This would occur if the zones were too wide and/or too weak. Neither condition is satisfied so the material remains ductile although somewhat less creep resistant.

Another effect was pointed out by Raymond [188] studying Inconel X-750. The precipitation of $M_{23}C_6$ carbides and the associated precipitate-free zone caused poor notch-rupture properties which can, however, be corrected by an appropriate heat-treatment.

Finally, the study of cavitation and its relationship to microstructural features has been pursued in Inconel X-750 by Venkiteswaran *et al.* [109]. They showed that the optimum creep life followed from a combination of a relatively wide precipitate-free zone, discrete carbide particles on the boundaries and fine γ' within the grains.

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