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Rejuvenation procedures to recover creep properties of nickel-base superalloys by heat treatment and hot isostatic pressing techniques

A review

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The rejuvenation procedures to recover the creep properties of nickel-base superalloys by atmospheric pressure heat treatment and hot isostatic pressing techniques have been reviewed in detail. It is very important that such treatments be applied at an optimum stage in the service life of a turbine blade. In other words, the rejuvenation procedures must be applied early enough to prevent catastrophic failures or irreparable damage and late enough to give a cost-effective benefit. The optimum stage at which to undertake a rejuvenation procedure to extend the creep lives of superalloys is immediately prior to the tertiary stage. By using these techniques it is not possible to extend the creep lives of superalloys indefinitely because of the accumulation of some "permanent" damage incurred during service conditions.

Nomenclature

ERF	Economic repair factor
P_r	Price of repaired and rejuvenated part
P_n	Price of new part
L_n	Potential operational life of new part
L_r	Potential operational life of repaired/rejuvenated part
N	Cavity density or number of cavities per unit area (mm^{-2})
n_v	Number of cavities per unit volume (mm^{-3})
ε	Creep strain
σ_1	Maximum principal stress (MPa)
$\bar{\sigma}$	von Mises effective shear stress (MPa)
t_f	Time to failure
t_t	Time to commencement of tertiary creep
λ	Creep damage tolerance parameter
ε_f	Strain at fracture (or failure)
T_m	Absolute melting temperature
σ_0	Friction stress
r	Spherical radius of cavities
2χ	Intercavity spacing
δ	Grain boundary width
P_I	Cavity gas pressure
P_H	External hydrostatic pressure
Ω	Atomic volume
k	Boltzmann constant
T	Absolute temperature
γ	Surface energy of the cavity
D_b	Grain boundary diffusion coefficient
ϕ_d	Ductility recovery parameter
ε'	Strain to reach the same acceleration after recovery annealing
ε_0	Strain necessary for standard material to reach a given acceleration of the secondary-creep rate in the tertiary region
ε_t	Strain needed to have produced the reduced cavity volume after rejuvenation annealing
$\dot{\varepsilon}$	Creep rate
$\dot{\varepsilon}_s$	Secondary or minimum creep rate
ε_1	Strain previous to the regenerative annealing period
n	Total number of strain/regenerative anneal cycles
ϕ_v	Recovery parameter for cavity volume
V_0	Original total cavity volume at the start of the recovery
V_t	Cavity volume after recovery annealing for a time t

1. Introduction

The repair of industrial gas turbine components has developed into a significant after-sales market over the last ten years or so. Until the mid-1970s most turbines were overhauled and components "repaired" by replacement. As a result, many expensive components were being removed from service which were potentially repairable with proper application of existing metallurgical and mechanical engineering technology. However, the necessary technology for repair of gas turbine components have existed in the flight turbine market since the 1960s [1].

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The high cost of modern nickel-base superalloys and the potential problem of shortage of critical constituent elements has led to the increased interest in extending the operation lives of superalloy components by various repair and rejuvenation (restoration) procedures. It is very important that such treatments be applied at an optimum stage in the service life of a turbine blade [2]; they must be applied early enough to prevent catastrophic failures or irreparable damage and late enough to give a substantial cost-effective benefit. Therefore, the nature and accumulation of damage that limits the life of superalloy components for various applications must be quantitatively characterized in order to provide a baseline from which the remaining life can be assessed.

Three broad categories of refurbishing procedures were proposed [2] for superalloy gas turbine components:

(a) Non-structural methods, which attempt to recover the original form or microstructure of the component or alloy by rejuvenation heat treatment, recoating or hot-isostatic pressing (HIPing).

(b) Shaping, which adjusts small distortion of the components by, for example, grinding or straightening.

(c) Structural methods, which make radial changes to a seriously damaged blade, by, for example, welding, or brazing of surface cracks or by replacement of damaged regions of the component.

The present review is concerned with assessing the potential of increasing the operational life of components by rejuvenating procedures including recovery heat treatment and HIPing, rather than by repairing obvious defects.

The turbine blades in aircraft gas turbine engines are subjected to a complex combination of stresses. With hot-gas temperatures of ~ 1700 K, centrifugal accelerations of around 200 000 g, and outside-to-inside temperature gradients of the order of $150\,\mathrm{K\,mm^{-1}}$ in blades cooled efficiently, superimposed life-consuming mechanisms come into play [3]. Aerodynamic or speed-related excitation at the natural frequency can lead [4] to high-cycle-fatigue (HCF) damage; alternating mechanical and thermal stresses during take-off and landing cycles can cause low-cycle-fatigue (LCF) or thermal-fatigue damage; and creep mechanisms, accompanied by superimposed hot-gas corrosion, are activated during steady-state conditions. Particular mechanism(s) may dominate and interactions are possible. The damage in turbine blades may be classified [5] as follows:

(a) Physical alterations such as corrosion, distortion (or dimensional change), erosion and internal cracking.

(b) Metallurgical degradations involving $\gamma'[Ni_3(Al, Ti)]$ coarsening, partial carbide solutioning, transformation or incipient melting, etc.

The metallurgical degradations or microstructural instability can affect the development of the physical damage features, and impair the component's capability to resist its normal service stresses. Therefore, aeroengines are regularly overhauled, and some of their components are then retired for repair, while others are damaged to such an extent that they can only be scrapped or stored until some appropriate rejuvenation procedure is worked out. Metallurgical rejuvenation implies particularly treatments which, although sometimes rather costly, cannot be dispensed with for critical parts upon which many human lives and high-value equipment depend. Notwithstanding this additional cost, repair and rejuvenation are often cheaper than refurbishing the engine with new parts. The generally accepted rule [6, 7] is that they are viable whenever their economic repair factor (ERF), taking remaining operation life into account, does not exceed 0.75:

$$\mathrm{ERF} = \frac{P_r}{P_n} \times \frac{L_n}{L_r} \qquad (1)$$

2. Creep deformation in nickel-base superalloys

Primary importance for a blading material is its creep strength. The creep behaviour of γ–γ' alloys strengthened by the coherent $\gamma'[Ni_3(Al, Ti)]$ phase primarily depends on the following factors: (i) the volume fraction of γ' phase [8]; (ii) the precipitate particle spacing, shape, size and distribution [9–15]; (iii) the coherency strains due to the lattice misfit between the γ and γ' phases [16]; (iv) the $M_{23}C_6/M_6C$ carbides at the grain boundaries [17]; (v) the formation of a serrated grain boundary structure [18–20], and (vi) the grain size [8, 15, 21].

Nickel-base superalloys are strengthened through carbide precipitation at the grain boundaries [17, 22]. Discrete grain-boundary carbides are generally considered beneficial since they reduce the extend of grain boundary sliding and therefore the onset of creep cavitation and rupture [17, 23]. Some workers [24, 25] have tried to correlate the creep rate with carbide particle size in complex superalloys such as Nimonic 105.

The γ' dispersion is established as hindering the dislocation movement within the grain interiors, and the minimum creep has been shown to vary as the cube root of the γ' volume fraction [26]. It was also observed [15] that in Inconel 700, creep strength is predominantly governed by the γ' particle size and its volume fraction within the matrix. It is therefore very important to correlate the creep strength parameters and microstructural features, to provide guidelines for the development of rejuvenation technology.

It is well known that creep is the dominant mode of deformation in turbine blades at operating stresses and temperatures. Therefore, the life of turbine blades is commonly consumed by creep, determined by centrifugal force and the temperature of the material. At service temperatures, microstructural changes occur in blade airfoils which decrease their creep resistance and promote airfoil distortion. These time-dependent microstructural changes include

(i) coarsening of the γ' phase;

(ii) changes in the grain boundary and grain-boundary carbide morphologies;

(iii) precipitation of brittle intermetallic phases such as σ-phase; and

(iv) formation of cavities or voids.

The loss of creep strength eventually promotes temperature- and stress-assisted creep cavitation, which leads to internal cracking and ultimately to failure; the formation of creep cavities, and their condensation as the grain boundary lowers the grain-boundary cohesion [23]. While indefinite creep lives cannot be obtained, the rejuvenation procedure has been shown to result in significant life improvements, although it is necessary to device specific heat treatment and/or HIPing technique schedules for individual superalloys.

3. Creep cavitation

It is known, based on the pioneering work of Greenwood [27, 28], that creep fracture usually takes place by the nucleation, growth and eventual coalescence of grain-boundary cavities, primarily on those grain boundaries that are nearly perpendicular to the tensile stress axis [29]. In general both diffusion [30] and plasticity [31, 32] play a role in the cavitation process, with one or the other dominating under different experimental conditions or during different stages of growth. Temperature, strain rate, stress state, impurity content, microstructure and strength are some of the variables that influence creep cavitation [33, 34].

The cavity population in nickel-base superalloys is dependent on the grain boundary chemistry [35, 36], stress, strain [37] (Fig. 1), and time [38]. In addition, in more complex loading, the number of cavities per unit strain varies with stress state [39] (Fig. 2). Generally, cavity nucleation [40] is continuous during the test life and the form of the strain–time dependence, together with the stress and stress-state sensitivity, is dependent on the nucleation sites present. The general features of cavitation failure are an increasing loss of ductility with increasing temperature and decreasing strain rate [41]. These changes are directly associated [42] with cavity development and with the intercavity spacing which increases with temperature.

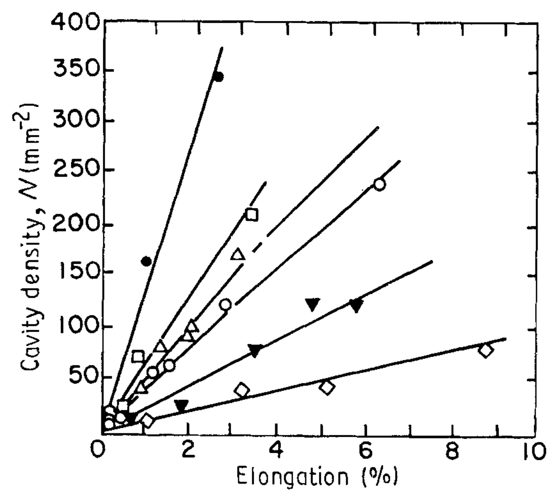

Figure 1 Linear relationship between cavity density N and creep elongation in Nimonic 80A [37]: (●) 92 MN m^{-2}, (○) 154 MN m^{-2}, (□) 185 MN m^{-2}, (△) 216 MN m^{-2}, (▼) 308 MN m^{-2}, (◇) 385 MN m^{-2}. Creep temperature = 1023 K.

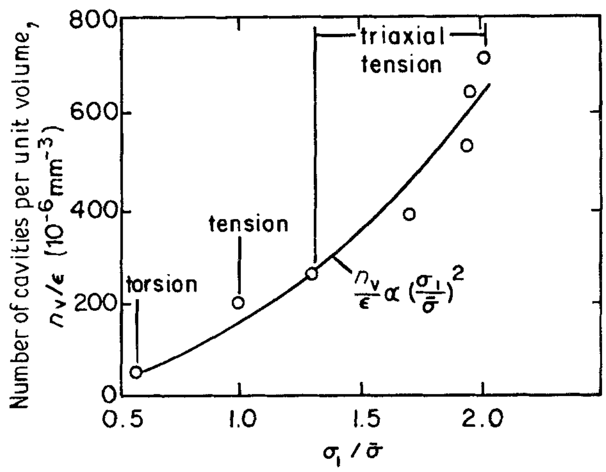

Figure 2 Effect of stress state on cavity nucleation in Nimonic 80A [39].

The accumulation of cavities is a dominant life-limiting factor in many superalloys. In such alloys the rejuvenation heat-treatment procedures are undertaken at an intermediate stage in a high-temperature component's life to recover the original creep properties and thereby extend the useful creep life. The basis of such regenerative heat treatments are either to inhibit further growth (by grain boundary diffusion of pre-existing cavities by isolating them within grain interiors through recrystallization or grain growth [41]), or to remove cavitation completely by means of sintering heat treatment [43].

It is extremely important to assess the rejuvenation potential of used turbine engine components. In some alloys cavitation kinetics may be used to monitor the remaining creep life time. For example, in Nimonic 80A, as in several pure metals and alloys [44], Dyson and Mclean [37] found that the plot of cavity density N against strain is linear for a range of stresses, taken over approximately 75% of creep life (Fig. 1). The linear relationship means that a complete creep test is not necessary before $N(\varepsilon)$ can be determined for a particular stress. Therefore a fairly short-term test fixes the N versus ε curve, and a subsequent measurement of cavity density N in service yields ε or the remaining creep life. The same workers [37] also found that cavity density data at all stresses could be rationalized within a reasonable scatter band on to one curve if N is plotted against t/t_f, the fractional time to fracture (Fig. 3). Advances in knowledge of cavitation failure mechanics under multi-axial stressing suggests that the slope of N against ε is dependent on stress state. For example, Fig. 2 suggests [45, 46] that N/ε is proportional to $(\sigma_1/\bar{\sigma})^2$. Using such information, it is possible also to predict the remaining life for complex engineering loadings. It is important, however, to note that the relationships between the remaining life and stress state may vary from material to material [45, 47, 48].

To relate creep cavitation to remaining creep life, an approach advocated by Neubauer and Wedel [49] has also been used in practice. In this approach, damage is classified into different stages and, for each classification, remedial action is prescribed, as illustrated in

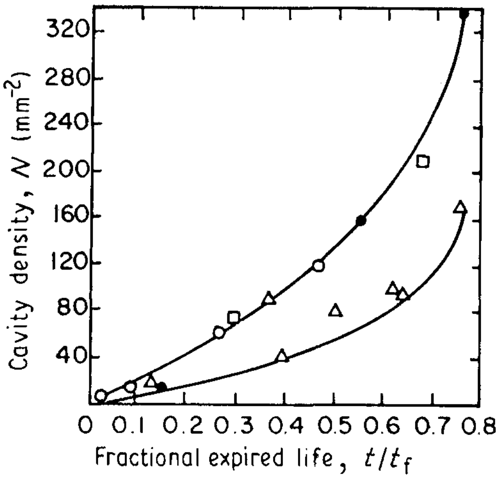

Figure 3 Relationship between the cavity density N and the fractional expired life t/t_f for Nimonic 80A [37]: (●) 92 MPa, (○) 154 MPa, (□) 185 MPa, (△) 216 MPa. Creep temperature = 1023 K.

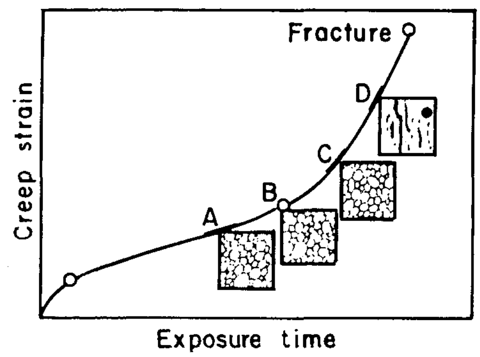

Figure 4 Classification of creep damage [4]. Observations on replica require action as follows: (A) isolated cavities – observe; (B) oriented cavities – observe, fix inspection intervals; (C) cavity linkage/microcracks – limited service until repair; (D) macrocracks – immediate repair.

Fig. 4. For Class A damage, no remedial action would be required. For Class B damage consisting of oriented cavities, re-inspection within a period of $1\frac{1}{2}$ years would be recommended. For Class C damage, repair or replacement would be required within six months. For Class D damage, immediate repair or replacement would be required.

4. Tertiary creep damage mechanisms

Regeneration of the properties of blades subjected to lifetime-consuming operating conditions calls for knowledge of the damage mechanisms involved and of the specific structural parameters, as the re-establishment of the components to full serviceability requires that they be recovered to an as-new condition [4]. The fracture of metals after high-temperature creep results from the progressive accumulation of damage through the creep life. The first indication of eventual fracture is usually the acceleration in creep rate at the onset of the tertiary stage of creep. Dennison and co-workers [50, 51] have concluded that the creep damage is generally due to (i) the development of grain-boundary cavities and cracks to a size sufficient to affect the deformation processes [23, 52] or (ii) microstructural instability, such as grain growth or recrystallization in single-phase materials [53, 54] or changes in particle dispersion during creep of two-phase alloys [55]. Also the other possible sources of damage [24] would occur as an accumulation of lattice defects and changes in the arrangement of such defects, or development of inhomogeneous structures due to segregation of solute atoms to grain boundaries (or the formation of denuded zones).

Because of this wide range of possible sources of damage it is important to ascertain the exact nature of the creep damage, since the rejuvenation procedure applied depends on the characteristics of the damage. A method of distinguishing between these causes of tertiary creep or creep damage has been suggested [50, 51, 54, 55] depending on the time to the commencement of tertiary creep, t_t, and the time to fracture, t_f. For many single-phase materials which are microstructurally stable during creep, when tertiary creep begins as a result of cavity development (in the absence of grain growth and recrystallization), both t_t and t_f are inversely proportional to the steady-state creep rate [54] so that the ratio t_f/t_t is a constant ($\simeq 1.5$) over wide ranges of stress and temperature. When tertiary creep is associated with grain growth [53] or over-ageing [50, 55], considerably larger t_f/t_t ratios are recorded. The relationships between t_t and t_f for different materials are shown in Fig. 5. Whilst $t_f/t_t \simeq 1.5$ for pure gold [41] and Nimonic 80A [43], larger values of this ratio are found for Nimonic 105 [24], Nimonic 115 [50] and IN-100 [51, 56] ($t_f/t_t \simeq 2.5$), which suggests that the onset of tertiary creep is a consequence of a progressive change in microstructure.

Another way of distinguishing the creep damage mechanisms has been proposed by Ashby and Dyson [57]. They suggested that the creep damage tolerance

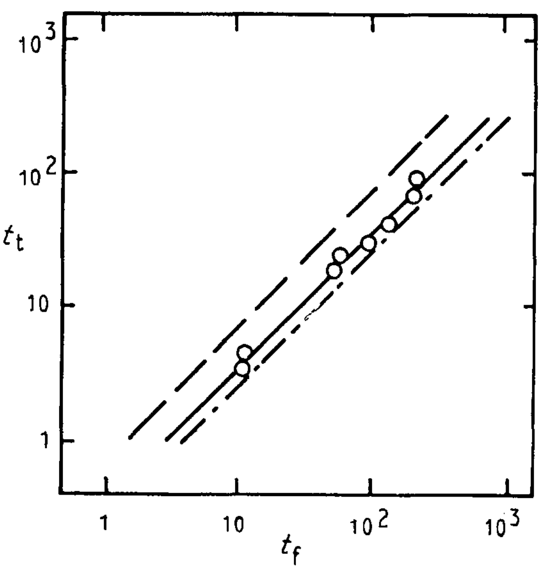

Figure 5 The relationship between the time to the onset of tertiary creep, t_t, and the time to fracture, t_f, for (——) pure gold and Nimonic 80A, (○) IN-100 and (—·—) Nimonic 105 and 115 [50, 51].

parameter λ, defined by $\lambda = \varepsilon_f/(\dot{\varepsilon}_s t_f)$, originally introduced by Leckie and Hayhurst [58], could be used to identify the dominant creep damage mechanism(s) responsible for the tertiary creep. For this purpose they constructed a "diagnostic" diagram on the basis of ε_f versus $\dot{\varepsilon}_s t_f$, and they have used this diagram to identify the dominant creep failure mechanism. Thus, failure dominated by grain-boundary cavitation tends to occur in the range $1 < \lambda < 2.5$, and failure dominated by microstructural degradation occurs at values of λ around 10 or higher.

5. Design of rejuvenation cycles

Because of the cost and complexity of many components serving at elevated temperatures, a method which permits extension of their useful lives may offer considerable economic advantages [59]. For this reason numerous studies [20, 24, 41, 43, 51, 55, 56, 58, 60–67] have been undertaken in an attempt to improve the creep lives of nickel-base superalloys by employing repetitive creep/recovery heat treatment (and/or HIPing) cycles, i.e. a test piece is kept for a time t_i, usually to the late secondary or early tertiary stage, re-heated and the test continued for a further period.

In early work on creep-life recovery involving pure gold, Davies and Evans [41] showed that softening of the grain interiors was required to recover the primary creep stage but that steady-state creep and the rupture properties could be regenerated only if the cavities were isolated within the grain interiors by boundary migration. Similar experiments on pure copper [68] later confirmed these observations. The significance of these results is that they demonstrated that cavitation need not be considered "permanent damage" and that cavity sintering was not essential to obtain full recovery. The work also raised the possibility that the success of rejuvenative re-heat treatment procedures in relatively complex alloys may similarly be due to cavity isolation rather than to their sintering.

Selection of the optimum rejuvenation cycle is based upon the following considerations [69]: (i) rupture life recovery, (ii) time to specified creep strain (say 1% strain), (iii) prior creep strain recovery, (iv) creep resistance or minimum creep rate, and (v) creep curve behaviour. In the following sections the techniques applied to restore the creep properties of alloys, namely rejuvenation heat treatment and HIPing procedures, will be discussed in detail.

5.1. Rejuvenation heat-treatment procedure

Many attempts have been made to recover or at least extend the useful creep life of operating components by procedures which involve the regeneration of the grain microstructure [24, 51, 56, 58, 60–62, 64, 65] or the "removal" of the grain-boundary cavitation [41, 55, 61, 70] or both, by regenerative annealing in the absence of stress. In general, the ability of rejuvenation heat treatment to re-establish creep properties relies on several factors:

(i) First, the heat treatment should dissolve the $\gamma'[Ni_3(Al, Ti)]$ phase coarsened and elongated during the service (Fig. 6) and reprecipitate it in a normal cuboidal form (Fig. 7).

(ii) Second, undesirable carbide phases should be dissolved and, during the ageing cycle, reformed into more desirable carbide, e.g. as discrete particles along the serrated grain boundaries.

(iii) Third, cavitation damage, which generally occurs at brittle grain boundaries and by vacancy diffusion, must be sintered closed by thermally activated diffusion.

An important requirement for the regenerative solution treatment temperature is that the grain size in service-exposed blades be held constant, i.e. it should not be allowed to increase during rejuvenation heat treatment [21, 71, 72]. Fig. 8 illustrates the effect of temperature on the grain size of Nimonic 108 [21]. Therefore, any regenerative heat treatment should take into account the grain-coarsening temperature of the service-exposed material, which in effect is a function of various precipitate–complex alloy systems.

As pointed out by Dennison and Wilshire [50] many results obtained during past years have suggested that the improved creep lives obtained by repetitive creep/regenerative heat-treatment programmes can be accounted for in terms of two types of material behaviour:

(i) When tertiary creep begins as a result of cavity development (i.e. with materials having t_f/t_t ratios of ~ 1.5, see Fig. 5), improved creep lives can be obtained if the cavities can be sintered out periodically, the sintering behaviour depending on gas or oxide stabilization – for example, the recovery of creep properties of pure gold [41, 43].

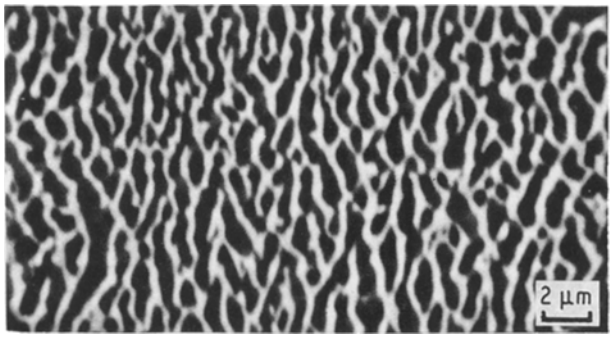

Figure 6 Coarsened and elongated (or rafted) γ' precipitate particles during the creep test at 1173 K/276 MPa for cast IN-100 alloy.

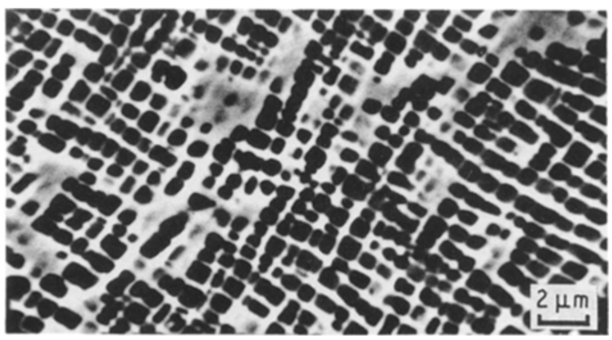

Figure 7 Cuboidal type γ' precipitate particles after regenerative heat treatment of crept (up to $\sim 1\%$ strain) IN-100 alloy.

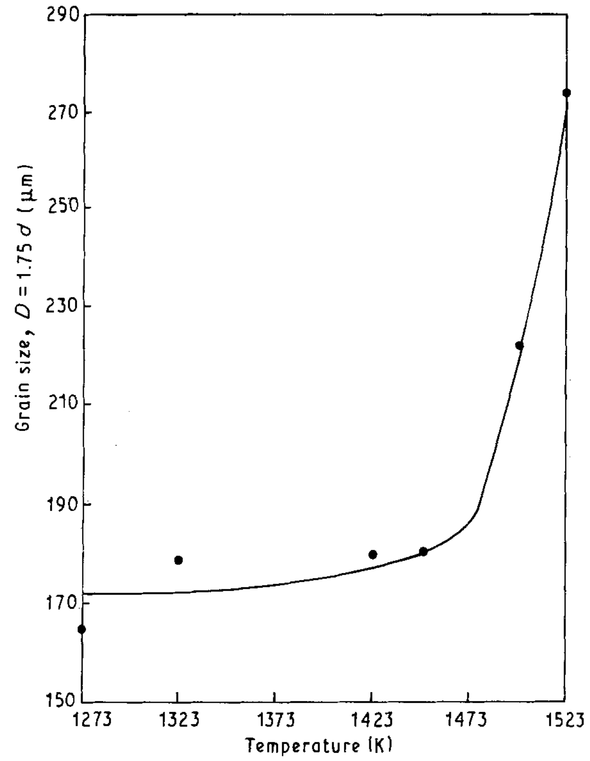

Figure 8 Grain-coarsening characteristics of service-exposed Nimonic 115 turbine blades after 1.5 h holding time at various temperatures [21].

(ii) When tertiary creep begins as a result of microstructural instability, such as over-ageing (i.e. when $t_f/t_t > 1, 5$, see Fig. 5), improved creep lives can be obtained, even without sintering out the cavities, by periodic heat-treatment schedules designed to minimize cavity growth rates by maintaining the original particle dispersion, for example, the recovery of creep lives of Nimonic 115 [56] and IN-100 [51] alloys. Therefore, any regenerative heat treatment for extending the useful creep life of engineering components must take account of the microstructure of the material as well as cavitation and voiding.

The work on pure gold by Davies and Evans [41] showed that in regenerative-annealed specimens which had been creep-tested to the onset of tertiary creep, grain growth and sintering of grain-boundary cavities took place. On retesting, the creep properties were completely regenerated and it was established therefore that tertiary creep occurs only when cavities are sited on grain boundaries. The re-establishment of creep life by regenerative heat treatment was achieved either by sintering the cavities or causing grain growth, isolating the cavities within the grain and thereby inhibiting their growth by eliminating the grain-boundary vacancy diffusion paths.

A relatively simple alloy, Nimonic 80A, which consists essentially of 15 vol % γ' precipitates and occasional carbide particles in a Ni–20% Cr matrix, was used to carry out similar work [43]. Complete recovery of the creep properties was achieved by rejuvenation heat treatment at the creep temperature (1023 K) or the ageing temperature (1093 K) after creep testing early in the tertiary stage. Creep life was extended by a factor of ~ 4 by repetitive creep/regenerative heat-treatment cycles, i.e. a sample was crept at 1023 K for a time t_a (about 20 min), late in the secondary region, rejuvenation heat-treated at 1023 or 1093 K and the test continued for a further period t_a, and so on. Indefinite creep life could not be achieved because of the loss of creep and fracture resistance due to extensive over-ageing [43]. However, to obtain indefinitely long creep life for a given specimen Davies *et al.* [43] pointed out that the following conditions are essential:

(i) Rejuvenation heat treatment must be carried out before the onset of tertiary creep, to prevent penetration of intergranular oxygen and to get a smaller cavity-size distribution;

(ii) Rejuvenation heat treatment would have to include complete re-heat treatment of the metal, to avoid the gradual over-ageing of the specimen during its prolonged life.

In conclusion, the life-extension of Nimonic 80A by rejuvenation heat-treatment is attributed either to isolation of the cavities by grain growth or to the sintering of the cavities (for cavities of approximately the critical size [30]), despite the fact that no direct observations of cavities were made.

In relatively more complex superalloys such as Nimonic 75, Nimonic 90, Nimonic 105, Nimonic 115 and IN-100 there are additional types of creep damage, apart from cavitation, which could cause tertiary creep, leading to fracture [51, 56, 65]. These are as follows [24]:

(i) over-ageing of the γ' precipitate may occur owing to the accelerated ageing under stress during creep; and

(ii) changes in composition of the precipitate could occur throughout a creep test. Thus, variations in distributions of elements between γ matrix and γ' precipitate could affect both the particle/matrix misfit and the extent of solute-hardening of the matrix.

In these types of superalloy, successful creep life recovery is only achieved by rejuvenation heat treatment which dissolves and reprecipitate the γ' phase in a distribution and size similar to those of the original microstructure. Therefore, for more complex alloys, prolonged creep lives are only obtained in repetitive creep/rejuvenation heat-treatment cycles, by using more sophisticated re-heat treatments, the detail procedure depending on the solvus temperature of the γ' and carbide phases [24, 56, 65]. In these alloys the onset of tertiary creep was identified [73] with decrease in the "friction stress" σ_0 as a result of the easier dislocation motion, which is permitted by the γ' precipitate coarsening. In the same study it was shown that the recovery of the friction stress was associated with regeneration of the original γ' precipitate dispersion. Although the γ' phase must be dissolved to achieve recovery, growth of the carbide phase is undesirable [24]. This growth can be reduced by using shorter regenerative annealing times or lower temperatures [24].

Creep-life recovery heat-treatments involving resolution of the γ' phase have been performed on nickel-base superalloys such as Nimonic 75 and Nimonic 90 [65], Nimonic 105 [24], Nimonic 115

[56] and IN-100 [51]. For example, with Nimonic 105 (having ~ 40 vol% main hardening constituent γ' and some carbide precipitates) rejuvenation heat treatment at or just above the creep temperature did not restore the creep properties, and more complex heat-treatment procedures were found necessary to obtain improved creep lives [24]. The regenerative heat treatment of this alloy was carried out at 1323 K/57.6 ks, air cooling (AC) plus 1123 K/57.6 ks, AC. Using repetitive creep/recovery heat-treatment cycles, increased creep lives at 1123 K (creep temperature) were obtained by employing a recovery heat-treatment schedule which dissolved the γ' phase (leaving the carbide out of solution to prevent grain growth [24]) followed by ageing to re-establish the initial particle dispersion. The times to the commencement of tertiary creep decreased, which can be attributed to the gradual coarsening of the grain-boundary carbide particles during rejuvenation heat-treatment, which has been shown to affect both creep and rupture properties of Nimonic alloys [74–76]. Again, indefinite creep lives could not be achieved, suggesting that some permanent damage was being accumulated, such as stabilization of cavities by surface oxidation or internal gas pressure. Complete restoration heat treatment would be expected to remove all types of damage except for oxidized cracks and cavities [24].

Another example in creep-life extension obtained by periodic rejuvenation heat-treatment to restore the original γ' dispersion is shown [51] in Fig. 9 for the cast IN-100 alloy. Clearly, after each rejuvenation heat-treatment (1493 K for 3.6 ks plus 1373 K for 3.6 ks), the original primary and secondary creep curves were re-established and, by repetitive creep/rejuvenation heat-treatment procedures, the creep lives were extended by a factor of ~ 3.

The difference between so-called simple alloys such as pure gold and Nimonic 80A and complex alloys such as Nimonic 115 and IN-100 in rejuvenation behaviour appears to be a consequence of gas stabilization [43, 62]. Oxygen does not dissolve in gold and

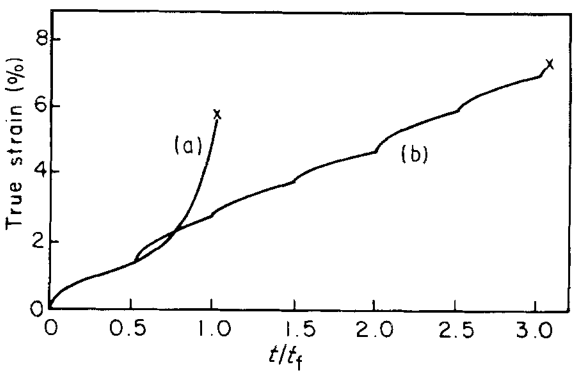

Figure 9 Illustration of the extension in creep life attainable for IN-100 by periodic re-heat treatment [51]. Creep curve (a) illustrates the uninterrupted creep curve at 185 MN mm^{-2} and 1223 K. Curve (b) shows the life improvement which can be achieved by periodic re-heat treatment involving rejuvenation heat treatment at 1493 K for 3.6 ks and cooling at 0.1 K s^{-1} to 1173 K, followed by a further heat treatment at 1373 K for 3.6 ks after which the samples were cooled at 4 K s^{-1}.

the oxidation resistance of Nimonic 80A is extremely good. Conversely, the relatively poorer oxidation resistance of alloys such as Nimonic 105 and 115, together with the higher creep temperatures used with these higher-strength alloys, may produce gas-filled cavities which cannot be sintered out [50].

5.2. Rejuvenation hot isostatic pressing cycle procedure

HIPing is now a well-established stage in the manufacture of certain cast and powder superalloy components. The HIP process leads to improved structural integrity, manifested as increased or more consistent mechanical properties, through accelerated consolidation of powders and elimination of porosity in casting [2]. HIP is also being increasingly advocated as a method of rejuvenation for service-exposed parts in order to extend their operating or useful lives [20, 63, 64, 66, 67, 77–79]. Regeneration by HIPing has been shown to be usable for certain applications, provided that the life consumption due to service is caused by cavities or voids which are surface-connected [4]. This means that any application of a regenerative treatment by HIPing must be investigated closely for the mechanisms acting during creep life. The principle advantages of this process for material rejuvenation are claimed [2, 80] to be: (i) almost all internal defects (casting porosity, creep cracks or cavities) are sintered; (ii) stress-rupture properties are restored to a high degree; (iii) stress-rupture ductility can be increased in part; and (iv) the stress-rupture scatter-band is narrower. HIP accomplishes this by exerting a high argon pressure at an elevated temperature to encourage creep and diffusion-closure of voids and diffusion-bonding of the interfaces. If a procedure for healing all pre-existing or creep-induced surface cracks could be devised, the allowable lifetime of turbine blades could probably be extended substantially with attendant savings in cost and materials. The conventional HIPing procedure [79] is: (i) HIPing followed by relatively slow cooling; (ii) solution heat-treatment followed by rapid quenching; and (iii) ageing. The full procedure includes, in addition to HIPing, restoration to the original dimensions by tip welding and forming [79]. HIPing is a process whereby a high isostatic pressure is applied at a high temperature in an autoclave to a component containing internal voids or cavitation. Solid-state diffusion of the elements across the interface at these temperatures metallurgically eliminates these internal voids or cavities [1].

5.2.1. Theory of sintering of grain boundary cavities by HIPing and its application to nickel-base superalloys

Although various microstructural changes occur in engineering components when operating under service conditions of low stress and high temperature ($\sim 0.5 T_m$), it is the nucleation and growth of grain-boundary cavities which contribute commonly to the creep-life consumption [23, 81]. Although cavities may not in general be resolved optically until the late stages of creep life [82], precision density measurements [30, 83–85] show that cavity nucleation and growth occur continuously through the creep life.

The diffusional growth of cavities under the action of an externally applied stress was described by Hull and Rimmer [86]. Ashby [87] has applied this model to the sintering of a spherical cavity of radius r sited on a grain boundary. Each cavity is considered to be surrounded by a concentric sink situated midway between cavities, to which vacancies diffuse in the plane of the grain boundary so that cylindrical geometry is obeyed [66] (Fig. 10). The contribution of hydrostatic pressure to the sintering characteristics of creep-induced cavities using a basis of the treatment of sintering described by Ashby [87] was studied by Stevens and Flewitt [63, 66]. They determined the sintering rate as follows:

$$\frac{dr}{dt} = \frac{D_b \Omega \delta}{2kTr^2}\left(\frac{2\gamma}{r} - P_I + P_H\right) \Bigg/ \left[\ln\left(\frac{\chi}{r}\right) - \frac{3}{4}\right] \qquad (2)$$

Equation 2 gives the condition that the cavity contains gas at a pressure P_I, and is subjected to an external hydrostatic compressive strength P_H. It was suggested [88] that creep cavities in copper contains gas at pressures of $\sim$ 10 MN m^{-2}. The gas pressure develops during creep by diffusion of external gas along grain boundaries and bulk diffusion of interstitial gas atoms [63].

Using Equation 2, Stevens and Flewitt [66] predicted the variation of cavity radius in pure nickel with sintering time at a temperature of 1073 K (800 °C), for an initial cavity radius $r_i = 10\ \mu$m (Fig. 11). In this figure it is assumed that the cavities contain no internal gas, so that increasing hydrostatic pressure enhances the sintering rate. For example, the application of a hydrostatic pressure of 70 MPa causes a decrease of two orders of magnitude in closure time compared to the situation where no hydrostatic pressure is applied such that the only driving force for sintering is the surface tension of the cavities, the term $2\gamma/r$ in Equation 2.

Fig. 12 illustrates [66] the predicted variation of the cavity size as a function of hydrostatic pressure, indicating little benefit in using a hydrostatic pressure above about 150 MPa. The closure times as a function of initial cavity size for a range of hydrostatic pressures in the range of 70 to 150 MPa give effective sintering [66].

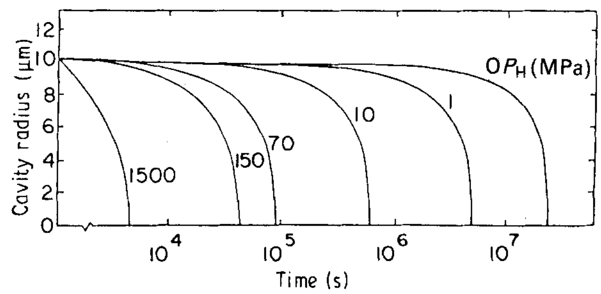

Figure 11 Predicted sintering behaviour of creep cavities in pure nickel at a temperature of 1073 K. Initial cavity radius = 10 µm, initial spacing = 40 µm [66].

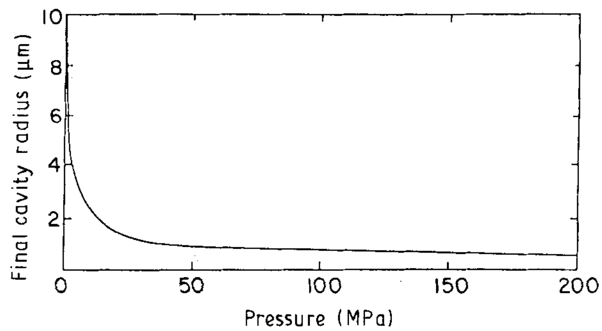

Figure 12 Final cavity radius as a function of hydrostatic pressure, for cavities in pure nickel of initial radius 10 µm and initial internal pressure of 0.1 MPa (1 atm) [66]. Temperature = 1073 K.

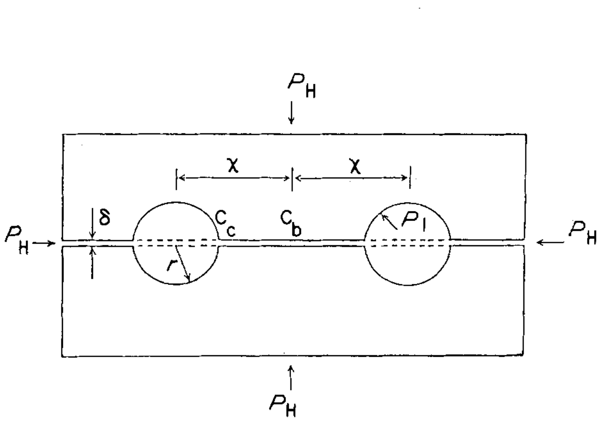

Figure 10 Model for cavity shrinkage by grain-boundary diffusion [66].

Therefore, the necessary parameters (pressure, temperature, dwell time) in Equation 2 may be used as a guide to optimize the HIPing conditions for sintering various size distributions of grain-boundary cavities. For example, for the case of the dilute nickel alloy Ni–2 % Cr, temperatures of $\sim$ 1000 K and pressures of $\sim$ 50 MPa were selected [63] to sinter completely in times up to $\sim 10^5$ s. Therefore, Equation 2 points to the need to carefully select the sintering conditions, pressure, temperature and dwell time, to remove creep-induced cavities in polycrystalline materials [63].

However, in reality the HIPing parameters (pressure, temperature, dwell time) are decided on the basis of a series of experiments and are influenced by available high-temperature creep and other data. In HIP cycles for castings the restrictions on the temperature range are influenced by the strength of the material (minimum level) and the occurrence of incipient melting (maximum level) [67]. The range may be as little as 30 °C [67].

Fig. 13 illustrates a comparison between heat-treated and HIPed IN-738 alloy after creep deformation at approximately three-quarters of the rupture life [66]. Rejuvenation heat-treatment at atmospheric pressure resulted in an increase of 28% overall rupture life, whilst the HIP heat treatment increased the life by 47%. In addition the rupture ductility increased by more than 100% compared to only 2.7% gained by atmospheric pressure heat-treatment.

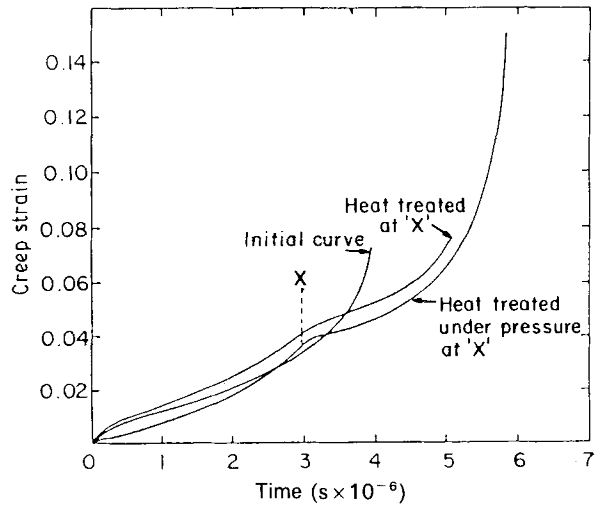

Figure 13 Creep curves for IN-738 at a stress of 244 MPa and a temperature of 1123 K [66]. At point X, tests were interrupted and samples heat-treated (7.2 ks at 1393 K, 57.6 ks at 1118 K) both at atmospheric pressure and under a pressure of 138 MPa.

The following metallurgical factors should be considered [21, 71, 72] prior to the selection of the HIP temperature: (i) grain coarsening temperature including the temperature dependence of grain size; (ii) γ', $M_{23}C_6$ and MC solvus temperatures; and (iii) extent of primary MC carbide degeneration that occurred during service. Past experiences in selection of the rejuvenation HIPing conditions, for example for the rejuvenation of IN-738 [89], IN-738LC [20], Inconel 700 [15], Inconel X-750 [90], Nimonic 105 [91] and Nimonic 115 [15] have led Koul and co-workers [20, 21, 92] to arrive at the following general conclusions:

(i) The HIPing temperature should lie above the γ' and $M_{23}C_6/M_6C$ solvus temperature but preferably below the MC solvus temperature of superalloy, since primary MC carbides govern the grain-coarsening characteristics of a number of nickel-base superalloys, and it also should be selected to avoid incipient melting. HIPing above the γ' and $M_{23}C_6$ solvus temperatures decreases resistance to plastic flow and ensures complete closure of shrinkage and/or creep cavities, while keeping the HIP temperature below the MC solvus temperature prevents rapid grain growth. Grain growth reduces the grain boundary area available for carbide precipitation, which in turn can lead to continuous carbide film formation along the grain boundaries during post-HIP ageing treatments. This in turn decreases the creep-rupture properties of a nickel-base superalloy [3, 15, 93]. In addition, HIPing in the MC solvus temperature range can dissolve sulphocarbides and the free sulphur can segregate at the grain boundaries [91], which can considerably decrease the creep resistance [94].

(ii) As the existence [19] of an inherent serrated grain boundary structure is controlled [95, 96] by the grain-boundary γ' precipitation, HIP application above the γ' solvus usually destroys the serrated grain boundary and produces a straight grain-boundary structure, which considerably reduces the creep resistance of the alloy (see for example Fig. 14). The existence of serrated grain-boundary structure depends on controlled cooling from the HIPing or post-HIPing solution-treatment temperature through the γ' precipitation range [19, 89].

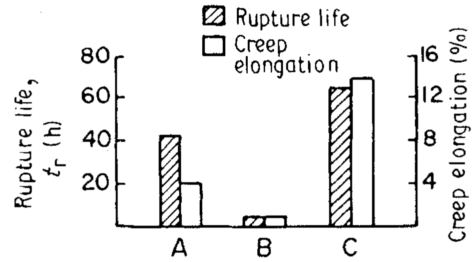

Figure 14 Effect of serrated grain boundaries on stress-rupture properties of IN-738 at 586 MPa and 1033 K in the standard heat-treated conditions. Serrated grain boundaries are formed by controlling the cooling rate from the solution-treatment temperature [97]. (A) As-cast + standard heat treated + serrated grain boundary; (B) as-cast + HIPed + standard heat treated (no serrated grain boundary); (C) as-cast + HIPed + standard heat treated with serrated grain boundary.

(iii) The post-HIPing ageing treatments for controlling the precipitation of primary and secondary γ' precipitates are usually, but not necessarily, the same as those designed for the virgin alloy.

Based on the past experience summarized above, Koul *et al.* [92] have used three HIPing rejuvenation cycles for eliminating the shrinkage cavities and rejuvenating the damaged microstructure of service-exposed IN-738 turbine blades. The three cycles were as follows:

Cycle 1: HIPing plus the standard high-temperature/low-activity aluminide coating cycle.

Cycle 2: HIPing plus a silicon-modified low-temperature/high-activity aluminide coating cycle.

Cycle 3: HIPing at 1473 K/2 h/105 MPa/furnace cooling plus solution treatment at 1473 K/2 h/controlled cooling to 1403 K/AC plus a slurry-coating heat-treatment cycle.

5.3. Kinetics of creep recovery

The sintering kinetics of cavities were studied by Brett and Siegle [98], who concluded that cavities resulting from dezincification in brass closed during annealing only if on or near to a grain boundary. On the other hand, intragranular cavities in aluminium, introduced by quenching, have been observed to anneal at a rate consistent with a volume diffusion process [99–101]. There is a considerable amount of knowledge to support a diffusional sintering mechanism for cavities in a range of materials including copper [78], nickel [102], tungsten [103] and stainless steel [62, 104]. Walker and Evans [62] have demonstrated that sintering of grain-boundary cavities, produced in a stainless steel by creep deformation, occurred with an activation energy equal to that for grain-boundary diffusion. Initially these results were described using a model which assumed vacancies to be annihilated only at the midpoint between cavities. Subsequently they considered [104] gas stabilization of cavities to explain the decrease of sintering rate with time.

The kinetics of the recovery process can be investigated by the use of a fractional recovery parameter ϕ_d

defined [60, 61] as

$$\phi_d = \varepsilon'/\varepsilon_0 \quad (3)$$

For complete recovery, $\phi_d = 1$ and for no recovery, $\phi_d = 0$.

The kinetics of creep recovery were studied by Davies *et al.* [61] for stress-free anneals in the temperature range 873–1023 K (600–750 °C) using Nimonic 80A. The temperature dependence of the recovery process was established on alloy specimens taken to early tertiary creep ($\dot{\varepsilon} = 1.5\dot{\varepsilon}_s$). At each temperature, the rate of recovery decreased continuously with annealing time, but the recovery parameter ϕ_d increased with increasing time as shown in Fig. 15a. The results of annealing at 1023 K after creep to different stages of the tertiary creep are given in Fig. 15b, in which the values of ϕ_d are plotted as a function of annealing time for specimens strained to 1.5, 3 and 8 times the secondary creep rate ($\dot{\varepsilon}_s$). The curves are similar in shape, but it is evident that considerably longer times of rejuvenation annealing are required to anneal out the creep damage present at the latter stages of tertiary creep. These results underline the importance of regenerative heat treatment at relatively early stages in the creep deformation. Davies *et al.* [43, 61] identified permanent creep damage with the formation of gas-filled or oxidized cavities and cracks in the later stages of the creep.

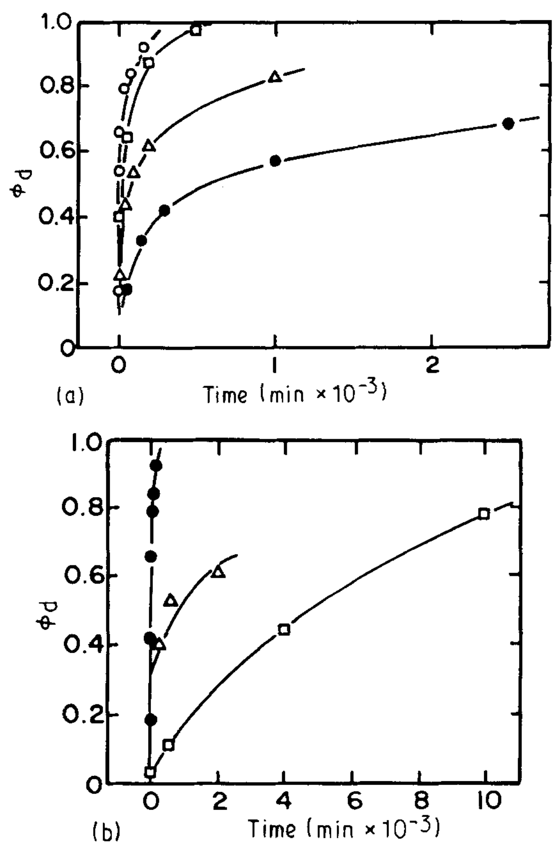

Figure 15 The kinetics of recovery of creep properties for Nimonic 80A crept at 1023 K [61]. (a) Time-dependence of the recovery parameter, ϕ_d, at various temperatures. All specimens were strained to early tertiary creep ($1.5\dot{\varepsilon}_s$) and rejuvenated at various temperatures: (●) 600 °C, (△) 675 °C, (□) 725 °C, (○) 750 °C. (b) Time-dependence of ϕ_d, for specimens strained to various stages of tertiary creep and recovery-annealed at 1023 K: (●) strained to $1.5\dot{\varepsilon}_s$, (△) strained to $3\dot{\varepsilon}_s$, (□) strained to $8\dot{\varepsilon}_s$.

As it is difficult to resolve the creep cavities either optically or by density changes, Evans and co-workers [43, 60] used a simpler material, 20% Cr–25% Ni stainless steel, to perform similar recovery heat-treatment experiments, and characterize fully the rate of change of cavity volume with strain. The recovery of fractional rupture parameters was determined through the use of a "mean" ductility recovery parameter, $\bar{\phi}_d$:

$$\bar{\phi}_d = \sum_{i=1}^{n} \frac{\varepsilon_i}{n\varepsilon_1} = \frac{n\varepsilon_1 - \varepsilon_t}{n\varepsilon_1} \quad (4)$$

Generally, ε_1 was taken as a small fraction ($\leqslant 0.25$) of the rupture ductility and a maximum number of six recovery-annealing periods was imposed on a given specimen. In addition, another recovery parameter for cavity volume ϕ_v was defined [62] as

$$\phi_v = \frac{V_0 - V_t}{V_0} \quad (5)$$

The kinetics of the recovery process for a specimen strained repetitively to 4% at 1023 K and given stress-free recovery anneals at various temperatures before re-testing to failure at 1023 K is shown [62] in Fig. 16.

The similarity in behaviour between ϕ_v and ϕ_d (Figs 16 and 17) strongly suggests that the recovery of ductility is due to the annealing out of grain-boundary cavities, although the limiting value of ϕ_d is different and decreases with decreasing temperature. It was shown [62] that both parameters were equal since there is a linear relationship between the cavity volume and strain. Thus, by definition (Equation 4)

$$\phi_d = \frac{n\varepsilon_1 - \varepsilon_t}{n\varepsilon_1} = \frac{V_0 - V_t}{V_0} = \phi_v \quad (6)$$

Therefore, the limiting values of the ductility recovery parameter ϕ_d are associated with permanent fracture damage such as the stabilization of cavities by entrapped gas.

As the activation energy obtained from the initial recovery rates of Fig. 16 is comparable with that for grain-boundary diffusion of the major alloying elements in the alloy, this could readily be associated with cavity sintering by transport along grain boundaries [62, 105]. The activation energy for Nimonic

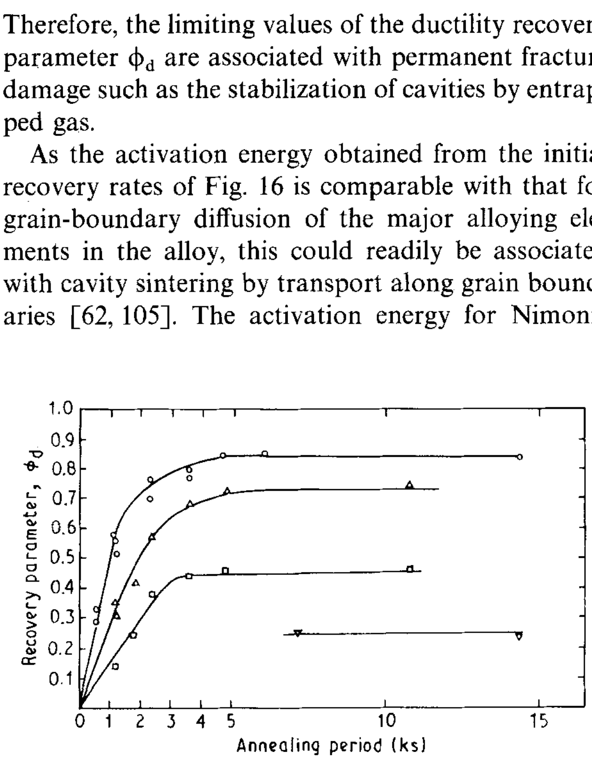

Figure 16 Variation in the recovery parameter ϕ_d with annealing time and temperature for a 20% Cr–25% Ni steel given a prior strain of 4% at 1023 K [62]: (▽) 650 °C, (□) 700 °C, (△) 725 °C, (○) 750 °C.

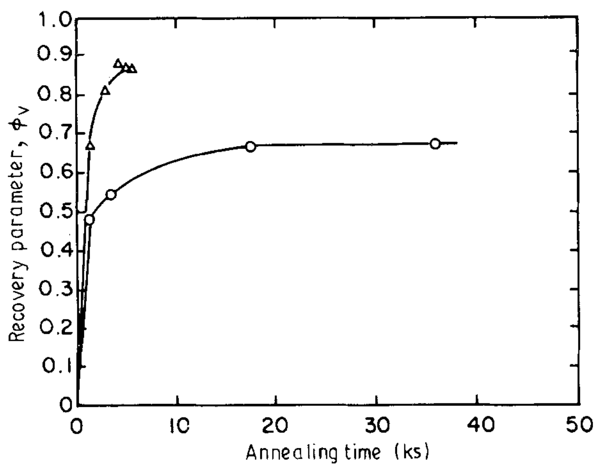

Figure 17 Variation in the recovery parameter ϕ_v with annealing time at 1025 K for a 20% Cr–25% Ni steel given prior strains of (△) 4% and (○) 8% at 1023 K [62].

80A (Fig. 15a) is different; here the recovery process is controlled by lattice diffusion, indicating either that grain-boundary transport or sinks are inhibited. It is possible that this effect could be associated with the presence of the dense network of intergranular carbides in this alloy.

6. Concluding remarks

The recovery of the creep properties of nickel-base superalloys by rejuvenation heat-treatment and HIPing techniques has been reviewed in detail. To recover or extend the useful creep life of nickel-base superalloys is firstly to re-establish the original material's microstructure, and secondly either to inhibit further growth by grain-boundary diffusion of pre-existing cavities (by isolating them within grain interiors through recrystallization or grain growth), or to remove the cavitation completely by means of atmospheric pressure heat-treatment or HIPing techniques. The application of HIPing technique is essentially concerned with the removal of "holes" in alloys. In designing an appropriate HIPing cycle for rejuvenation purposes, a number of metallurgical parameters need to be considered for obtaining optimum microstructures and creep properties, and the optimum results are obtained by proper control of HIPing temperature, solution-treatment temperature, cooling rate, partial solution-treatment temperature, and ageing treatments [20]. It is also very important to optimize the cooling rate for a given alloy since a slow cooling rate can produce over-aged primary γ' precipitates, whereas a fast cooling rate can produce very fine serrations and either of these features may not regenerate the desired creep resistance [15, 89]. Serrated grain boundaries prevent grain-boundary sliding [106], the deformation mechanism that predominates at service stresses and temperatures [97].

In rejuvenation heat-treatment it is the surface tension of the cavity alone that is the driving force behind the sintering out of the cavities, whereas in HIPing an additional external stress is superimposed, and slip processes occur as well as diffusion.

Although in some materials, as discussed in Section 5.1, atmospheric pressure rejuvenation heat-treatment may be enough for recovering the creep lives in realistic times, the application of HIPing will always accelerate the recovery process and enhance the probability of complete cavity removal. Inclusion of HIPing into the regenerative heat-treatment cycle to extend the creep life of components such as air-foil blades can increase performance by eliminating also the casting porosity apart from the creep cavities, as well as giving an extra precaution [59]. Using the HIPing technique will obviously increase the additional costs, but for many superalloys HIPing is essential to reduce the time-scale of the rejuvenation process to make it commercially viable and reliable.

In addition to the basic metallurgical aspects of the base alloy (including whether the alloy is cast or wrought), as discussed in previous sections, the service conditions of blades to be repaired must also be considered. Repair procedures must take into account, among other things [1]: (i) whether or not the blade has been coated with a corrosion protection system which must be removed prior to repair, but which is often critical in order to prevent corrosion thinning of the walls which could render a part unrepairable; (ii) the extent, if any, of surface penetration by corrosion or oxidation products on uncoated parts; (iii) whether the part has been previously repaired, and by what method and material; (iv) the nature and extent of mechanical property degradation due to service life; (v) the loss of material by corrosion and/or erosion, and (vi) the nature and extent of physical damage to the blade.

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