AN ASSESSMENT OF HOT ISOSTATIC PRESSING AND REHEAT TREATMENT FOR THE REGENERATION OF CREEP PROPERTIES OF SUPERALLOYS

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The creep lives of superalloys can be improved considerably by periodic reheat treatments. Further live improvements are unlikely to be achieved by incorporation of hot isostatic pressing operations into the rejuvenative procedures when failure occurs by the development of oxidized surface cracks.

In view of the high cost of superalloy components, considerable attention has been devoted to the development of reheat treatment schedules which eliminate creep damage (1). Significant life improvements have been achieved for wrought Nimonic* alloys and the cast material IN100 using periodic reheat treatment programmes. Reheat treatment had to be carried out during late secondary or early tertiary creep (Fig 1) since restorative procedures applied later in the creep life proved to be ineffective.

Fig 1. Schematic representation of repetitive creep/heat treatment cycles giving improved creep lives.

*Trade Mark of Henry Wiggin & Co. Ltd.

With Nimonic 80A, the creep lives could be increased by a factor of about four simply by periodically annealing at the creep temperature under zero stress. In contrast, with Nimonic 105 and 115, a comparable life improvement was achieved only with reheat treatments which involved redissolving the γ' or carbide particles (or both) followed by ageing to reproduce the original precipitate dispersion. Similarly, with IN100, rejuvenation was accomplished by solution treatment above the γ' solvus temperature and restoration of the original microstructure by controlled cooling which simulated the initial casting conditions (2).

The type of reheat treatment schedule needed for rejuvenation appears to depend upon whether tertiary creep is caused by the development of grain boundary cavities and cracks to a size sufficient to affect the deformation rate or by changes in particle dispersion (1). With Nimonic 80A, cavities were present early in the creep life. Since annealing at the creep temperature delayed rather than promoted the start of tertiary creep, the initial acceleration in creep rate during the tertiary stage is a result of cavity development rather than overageing. Although periodic sintering of cavities then extended the creep life, indefinite lives were not obtained because continued creep and periodic anneals at the creep temperature caused overageing and a gradual loss of creep and fracture resistance. With alloys such as Nimonic 105 and IN100, cavities were not discernible at the onset of tertiary creep whereas significant changes in particle dispersion were evident at this stage (2, 3) suggesting that tertiary creep is due to microstructural instability. Although restoration of the initial particle dispersion postponed the onset of tertiary creep, fracture eventually occurred by the development of intergranular cracks which were not eliminated even at the high temperatures used with the restorative heat treatments needed for these alloys. The improved lives resulting from periodic restoration of the microstructure are then a consequence of cracks developing only at the rate expected for the original structure rather than at the enhanced rates associated with the overaged structures present during the tertiary stages of conventional creep tests.

In order to obtain complete restoration of the creep properties of nickel-base superalloys, it therefore appears necessary for the rejuvenative procedures

- i) to eliminate any cavities and cracks which may be present and
- ii) to redevelop the original particle dispersion to avoid any deleterious effects associated with microstructural instability.

REJUVENATIVE PROCEDURES INVOLVING HOT ISOSTATIC PRESSING

The creep properties of pure metals can be fully recovered by sintering out cavities or even by restricting their growth by moving the grain boundaries away from the cavities present (4). However, with stainless steel, it has been shown that sintering was inhibited by gas stabilization and cavity removal was achieved only by annealing under a hydrostatic pressure (5). With nickel-base superalloys, creep cavities and the porosity which may be present in cast materials can be eliminated by hot isostatic pressing (6). In order to examine the extent to which hot isostatic pressing can result in recovery of creep damage, a series of experiments was carried out using the wrought alloy, Nimonic 105, and the cast material, IN100 (2, 3).

Testpieces of Nimonic 105 were initially solution treated in vacuo for 14.4ks at 1423K and air cooled, then held for 57.6ks at 1303K and air cooled, followed by a final ageing treatment of 57.6ks at 973K. This heat treatment resulted in an average grain diameter of $\sim 50\mu\text{m}$ and an initial γ' particle diameter of $\sim 40\text{nm}$. Constant stress creep tests at 201 MN/m^2 and 1123K (3) were interrupted and the specimens cooled under load, late during the secondary stage and also during the tertiary stage when the creep rate had accelerated to 3, 6 and 12 times the secondary rate. The samples were then annealed for 14.4ks at 1173K under a pressure of 103 MN/m^2 by Messrs. Henry Wiggin and Co. Ltd. This relatively low sintering temperature was selected so that the γ' distribution present when the test was discontinued should not be modified by the HIP operation. The results presented in Fig 2a illustrates that, on recommencing the tests under the original conditions, the creep properties were not affected significantly by the hot isostatic pressing treatment used.

A similar series of experiments was undertaken for IN100. The samples were taken from a single cast for which the cooling conditions, in particular the cooling rate of 0.1Ks^{-1} to below 1300K, were controlled to ensure production of a microstructure equivalent to that of commercial blade blanks (2). Creep tests carried out at 185 MN/m^2 and 1223K were again interrupted after various fractions of the creep life and the samples annealed for 14.4ks at 1173K under a pressure of 103 MN/m^2 . The results obtained on retesting also demonstrated that the HIP operation employed did not result in improved creep lives, and the creep rates after the HIP treatment were faster than the value recorded for the uninterrupted test (Fig 2b).

Fig 2. Curves for uninterrupted creep tests compared with those obtained for specimens which were retested to failure following a HIP treatment after varying fractions of the rupture life.

a) Curves for Nimonic 105 recorded at 201 MN/m^2 and 1123K and

b) Curves for IN100 obtained at 185 MN/m^2 and 1223K .

The present observations are therefore consistent with the results of earlier work which suggested that the application of an hydrostatic pressure during annealing operations does not lead to an improvement in the creep lives of nickel-base superalloys (6). This failure to achieve increased lives (Fig 2) may be attributable to the HIP conditions selected being unsuitable for cavity sintering. On this basis, complete restoration of the creep and fracture properties of superalloys may require

- i) a high temperature HIP operation to eliminate intergranular damage and
- ii) a subsequent reheat treatment to reproduce the original microstructure.

FRACTURE PROCESSES DURING CREEP OF SUPERALLOYS

With Nimonic 80A, intergranular cavities present during early tertiary creep could be eliminated simply by annealing at the creep temperature under zero stress (1). Considerably higher sintering rates would then be expected by annealing under hydrostatic pressure (5). However, with alloys such as Nimonic 105 and IN100, it appears that creep lives significantly above those obtainable by conventional reheat treatment procedures are unlikely to be achieved even by use of combined HIP/reheat treatment schedules. Metallographic examination of longitudinal sections of specimens of Nimonic 105 and IN100 which had been cooled under load after varying fractions of their creep lives suggests that, with these alloys, fracture occurred primarily by the formation of oxidized surface cracks rather than by cavity development on boundaries well away from the specimen surface. Only rarely were internal cavities detectable even when the creep rate during the tertiary stage had accelerated to ~ 12 times the secondary rate i.e. after about 75% of the rupture life in conventional creep tests carried out with Nimonic 105 (Fig 3a). The internal cracking evident behind the fracture surface of failed specimens (Fig 3b) therefore appeared to form only when the creep rate became very large just prior to rupture. In contrast, the oxidized surface cracks were clearly discernible during the tertiary stage when the creep rate had accelerated to ~ 6 times the secondary value (Fig 3c).

Fig 3. Microstructures of Nimonic 105 tested at 201 MN/m^2 and 1123K showing

- a) the virtual absence of internal cavities during late tertiary creep ($\times 150$)
- b) the voids evident behind the fracture surface of failed specimens ($\times 150$) and
- c) oxidized surface cracks developing during tertiary creep ($\times 320$).

The metallographic features of the surface cracks developed during the creep of Nimonic 105 and IN100 were consistent with initial nucleation and growth taking place by stress-assisted intergranular oxidation (7). The growth rates of these cracks however, appear to be determined by the deformation characteristics of the alloys. This view is supported by the observation that, for both materials, the rupture life is inversely proportional to the steady-state creep rate (Fig 4). On this basis, a consistent interpretation can be provided for the behaviour observed for the repetitive creep/heat treatment cycles and the results obtained following hot isostatic pressing (Figs 1 and 2). Since growth rates of the oxidized surface cracks are controlled by the deformation rates, periodic restoration of the original microstructure leads to improved creep lives by avoiding the acceleration in creep rate associated with overageing (Fig 1). However, with this failure mode (Fig 3c) hot isostatic pressing will not lead to increased rupture lives since this operation will not remove oxidized surface cracks. Only if the crack surfaces could be thoroughly cleaned and the sample coated to isolate the cracks from the specimen surface would hot isostatic pressing be expected to eliminate fracture damage with the failure mode observed under the testing conditions considered.

Fig 4. The relationship between the time to fracture and the steady-state creep rate for Nimonic 105 (O) tested over the stress range 200-400 MN/m² at 1123K and the cast alloy IN100 (Δ) over the range 140-250 MN/m² at 1223K.

In the present programme, the maximum rupture lives examined were 1600ks so that intergranular crack development by the formation growth and link-up of internal cavities may be more relevant with long term service conditions. Yet the non-steady stress and temperature conditions experienced by blades in aeroengine gas turbines may result in fracture damage being accumulated primarily in the peak stress/temperature periods of the operating cycle. Failure by the development of oxidized surface cracks could then typify service behaviour (7). The present work therefore suggests that

i) only with materials and creep conditions whereby fracture is a consequence of internal cavitation would combined HIP/ reheat treatment schedules appear to be advantageous and

ii) when rupture occurs by oxidized surface cracking the incorporation of hot isostatic pressing operations into the rejuvenation procedures is unlikely to result in an improvement in creep lives above those recorded for high strength superalloys by periodic reheat treatments aimed at restoring the original microstructure.

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