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The effect of prior monotonic creep on the resultant fracture toughness and tensile properties of Inconel X-750

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Precipitation-hardened polycrystalline nickel-base superalloy type Inconel X-750 was loaded at 700° C and crept to increasing fractions of its creep life. Secondary small fracture-toughness specimens and standard tensile specimens were machined from the initial rather large creep specimens. The secondary specimens were used to study the resultant resistance of the crept alloy to the extension of sharp cracks and its resultant tensile properties as functions of the amount of prior creep. Intergranular formation and coalescence of voids, which took place during the creep process, were responsible for a drastic reduction in the fracture toughness of the alloy long before it reached the end of its creep life. This behaviour indicates that possible deleterious changes in fracture toughness may be the governing factor which determines the useful and safe lives of materials which serve while loaded at high temperatures.

1. Introduction

An approach was put forward, some time ago, whereby the useful life of materials which are loaded at high temperatures may have to be determined by possible harmful changes in their fracture toughness while in service rather than by their creep or fatigue lives [1]. An exploratory investigation of the feasibility of the approach was carried out on a nickel-base solid–solution type alloy [1]. A drastic reduction in resistance to the extension of sharp cracks was observed after creep deformation at 650° C, while an increase in toughness was possible after similar deformation at slightly higher temperatures. The reduction in toughness was mainly due to the formation and coalescence of voids along grain boundaries, and partially due to an increase in dislocation density caused by the level at which the tests were completed. No voids were observed in specimens which were deformed to the same extent at temperatures slightly lower or higher than 650° C. The resultant tensile properties and impact energy of Charpy keyhole specimens made of crept and aged Type 316 stainless steel were also reported [2]. Intergranular and intragranular carbide, which formed during the early stages of exposure to high-temperature soak and creep, were considered as the main contributors to the observed reduction in uniform and total tensile elongations and to the decrease in impact energy to fracture of the Charpy keyhole specimens [2].

Several microstructural events may take place during high-temperature loading of crystalline materials:

(i) change in dislocation density, configuration and distribution;
(ii) grain boundary sliding and migration;
(iii) formation of subgrains;
(iv) nucleation, growth and coalescence of voids;
(v) nucleation, migration and growth of new phases;
(vi) dimensional and morphological changes, migration and disappearance of old phases;
(vii) migration of undesirable elements to grain boundaries.

Some of these events are interdependent, some will occur while the material is exposed to high temperatures without being loaded mechanically, while some can be accelerated due to loading and others will not take place at all in the absence of external loads. These microstrucutal events may greatly alter the fracture toughness of materials during high-temperature loading. Thus, a situation may develop whereby a material with an initially acceptable fracture toughness value may become dangerously sensitive to the presence of sharp cracks long before its final rupture at the end of controlled creep or cyclic loading tests. In such a case, the useful and safe life of the material will be shorter than its actual creep of fatigue life.

The previously reported investigation was carried out on a fairly simple solid-solution strengthened nickel-base alloy so as not to have too many of the aforementioned microstructural changes taking place at the same time [1]. The purpose of this paper is to report additional work on the effect of prior monotonic (constant load) creep on the resultant fracture toughness and tensile properties of a precipitation-hardened polycrystalline nickel-base superalloy.

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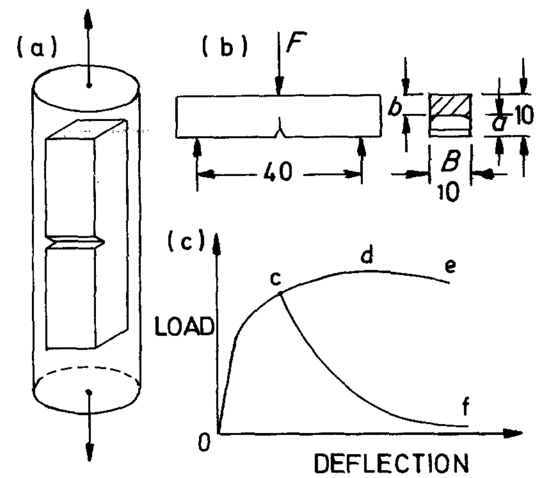

Figure 1 (a) Orientation of the three-point pre-cracked bend-specimens with respect to the loading axis of the original creep specimen. (b) Dimensions in millimetres of the bend specimens. (c) Two typical load against mid-section deflection curves of bend specimens; curve 0cde is typical of the as-heat-treated alloy and 0cf is typical of the crept alloy.

2. Experimental procedure

Inconel X-750 was selected as the precipitation-hardened alloy. Round 25.4 mm diameter bars were solutionized at 1150° C for 2 h and air-cooled; stabilized at 843° C for 24 h (1st age) and air-cooled; aged at 704° C for 20 h and air-cooled. Large creep specimens of 134 mm gauge length and 16 mm diameter were machined from the bars, and creep-loaded at 700° C and at an engineering tensile stress of 468.6 MN m^{-2}. The creep machine had especially designed and constructed six-zone furnaces facilitating uniformity of temperature within $\pm$ 1° C along a vertical distance of 0.22 m which included the specimen and part of the loading train. Some specimens crept until rupture took place, while testing of others was stopped after various fractions of their creep life were expended. Stoppage of tests was done by rapid cooling of the still loaded specimens. Three secondary near-Charpy-size three-point bend specimens or two three-point bend specimens and a tensile specimen were machined out of each creep specimen. A sharp fatigue pre-crack was introduced into each of the bend specimens by a constant displacement precracker. The pre-cracked specimens were three-point loaded at toom temperature and at a constant mid-section deflection rate of 1 mm min^{-1} while both the load and the mid-section deflection were continuously recorded. The orientation of the bend specimens with respect to the original large creep specimen, the dimensions of the bend specimens in millimetres and typical load–deflection curves are shown in Figs 1a, b and c, respectively. The curve Ocde in Fig. 1c is typical of the as-heat-treated alloy while the curve Ocf is typical of bend pre-cracked specimens machined out of the crept alloy.

The small dimensions of the pre-cracked, three-point bend specimens did not qualify them even for valid J_{IC} determination according to existing standards [3]. Consequently, the amount of specific energy A/Bb to reach the peak of the load–deflection curve served as a measure of the resistance of the alloy to extension of sharp cracks. However, we will use this last term interchangeably with "fracture toughness". A is the energy spent to reach the peak of the load–deflection curve of a specific specimen. B and b in Fig. 1b are the width and the remaining ligament of the precracked specimens, respectively. The value of b is a calculated average of 21 readings taken at equally spaced distances along the width of the specimen. If the specimens were valid, the specific energy A/Bb would be equal to one-half of the J-integral at the maximum load, namely J_{max}.

The tensile specimens machined out of the creep specimens were of 25.4 mm gauge length and 6.35 mm diameter, and they were loaded at room temperature and at a nominal strain rate of 0.04 min^{-1}.

Transmission and scanning electron microscopies (TEM, SEM) were utilized to observe the microstructural changes which took place during the various stages of the creep process.

3. Results and discussion

The results of the mechanical testing are presented in Fig. 2. Curve (a) in Fig. 2 is the creep curve of the alloy indicating a continuously increasing creep rate from the moment of loading. Curve (b) represents the fracture toughness (FT) of the alloy as a function of creep time. The initial values in this and in the following curves are those of the as-heat-treated alloy. The fracture toughness of the alloy at the end of its creep life was determined from ruptured creep specimens, with the sharp pre-crack of the bend specimens being located slightly less than 25 mm away from the zone where final rupture took place at the end of the creep process. It is apparent from Curves (a) and (b) that about two-thirds of the resistance to extension of sharp cracks was lost by the time the alloy expended only slightly over one-half of its creep-life and elongated only one-fifth of its total deformation to rupture. Curves (c), (d) and (e) in Fig. 2 represent respectively the resultant UTS, yield point and elongation to fracture of tensile specimens prepared from the large creep specimens as a function of prior creep time. The creep process had little effect on the resultant UTS of the alloy. An increase in yield point and a reduction in resultant elongation to fracture were observed as the alloy expended about one-half its creep life.

Cracks advanced continuously and the load–deflection curve was smooth (like Curve 0cde in Fig. 1c) during bending of the pre-cracked three-point-bend specimens made of the alloy in the as-heated condition. Cracks became unstable and a sudden drop in load was observed upon reaching the peak of the load–deflection curve of bend specimens made of the crept alloy. The load–deflection curves of such specimens are represented by the curve 0cf in Fig. 1c. The advancement of cracks in pre-crept bend specimens was intermittent and it was accompanied by intermittent audible noise as the loading reached and continued beyond the peak of the load–deflection curve. This phenomenon will be discussed later. No audible noise was detected at any stage of bending of specimens made of the alloy in the as-heat-treated condition.

Fig. 3 comprises two SEM micrographs of a polished and etched specimen which crept for slightly longer

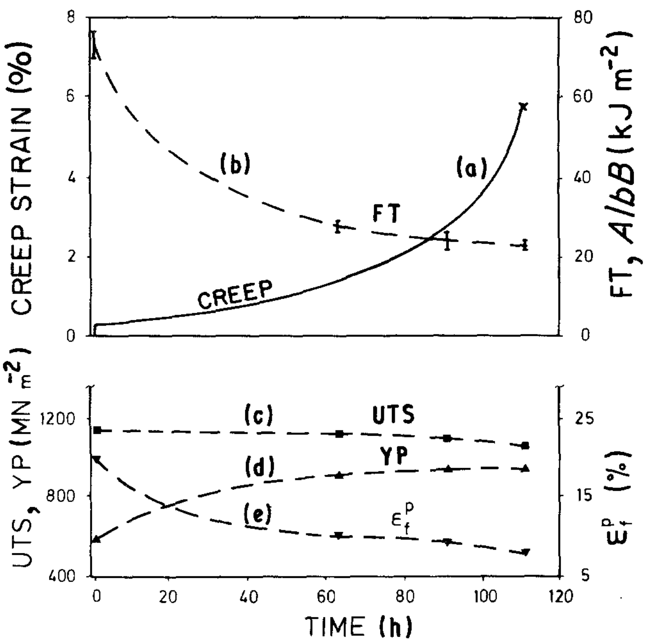

Figure 2 The dependence of mechanical properties of the alloy on creep time: (a) creep strain, (b) resistance to extension of sharp cracks or fracture toughness (FT), (c) ultimate tensile strength (UTS), (d) yield point (YP), (e) tensile elongation to fracture (ε_f^p).

than one-half of it creep-life. The direction of loading during the creep process is vertical. Voids and carbides are clearly observed along the diagonally oriented grain boundaries. The light cuboidal spots in the grains, with the exception of the denuded zones on both sides of the grain boundaries, are the coarse γ' precipitates which formed during the first ageing at 843° C. The fine γ' precipitates, which formed during the second ageing, are evenly distributed throughout the grains, including the zone denuded of the coarse γ', and they were observed by TEM at larger magnification than that of Fig. 3. Apparently, the formation of voids and their coalescence along grain boundaries during the creep process are the main cause for the drastic reduction of fracture toughness of the alloy.

SEM observations of fracture surfaces of ruptured creep specimens and of the three-point-bend specimens further clarify the cause for the reduction in fracture toughness of the alloy during the creep process. Figs 4 and 5 represent two extreme morphologies of fracture surfaces. Fig. 4 depicts the fracture surface of a ruptured creep specimen at the end of a creep test. The fracture surface of the specimens is primarily intergranular, representing a continuous process of weakening due to the formation and coalescence of voids along grain boundaries during creep. Fig. 5 depicts the fracture surface of a slowly bent pre-cracked three-point specimen made of the virgin, as-heat-treated alloy. In this case the fracture was dimpled, transgranular over more than one-half of the fracture surface and intergranular over the remainder of the fracture surface. This morphology is different from the primarily dimpled intergranular propagation observed in another study of Inconel X-750 which received similar heat treatment as in our work [4]. However, others observed the formation of intergranular voids during the early stages of creep of

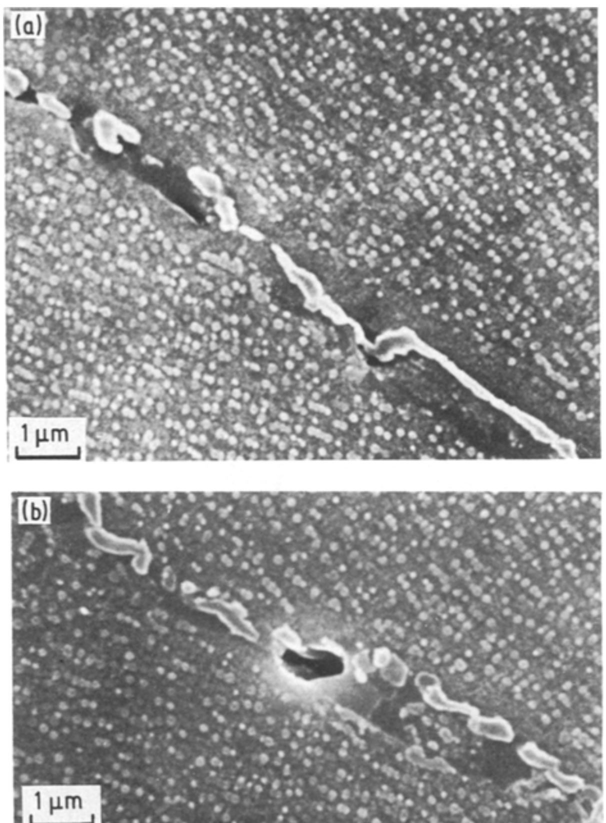

Figure 3 (a, b) carbides and voids along a grain boundary of a crept specimen. Both micrographs are of the same grain boundary.

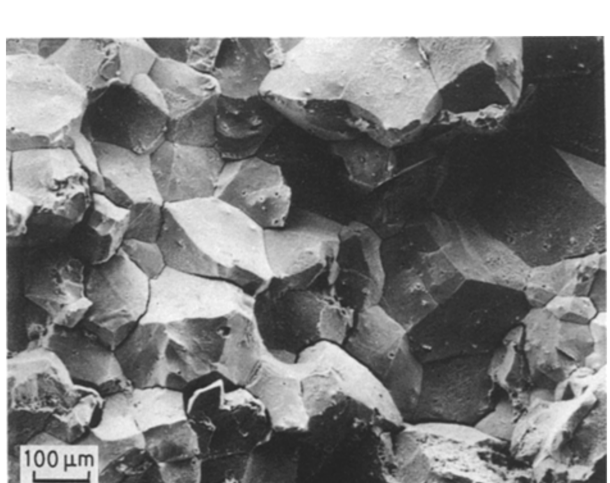

Figure 4 Mostly intergranular fracture surface of a ruptured creep specimen at the end of a creep test.

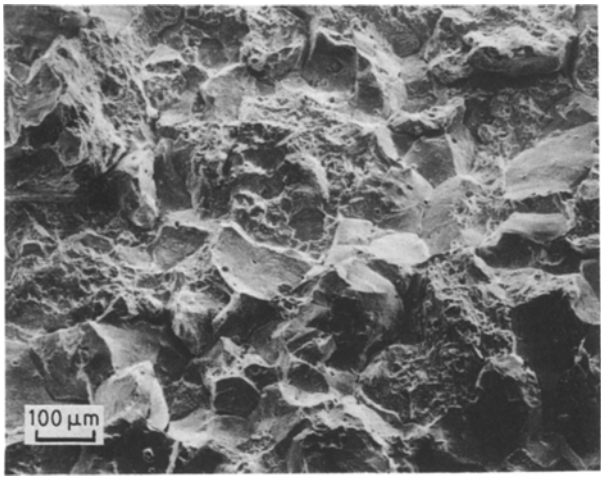

Figure 5 Mixed transgranular and intergranular fracture surface of a bend pre-cracked Charpy-sized specimen made of the as-heat-treated virgin alloy.

Inconel X-750 [5]. Dimples were also observed, in our work, to be superimposed on grain boundaries which were exposed during bending and fracturing of the pre-cracked bend-specimens made of the as-heat-treated alloy. However, these dimples were smaller and shallower than the transgranular dimples.

The appearance of the fracture surface of the pre-cracked bend-specimens changed with the extent of prior creep. Increasing the amount of prior creep strain resulted in an increase of the amount of intergranular fracture surface at the expense of the transgranular fracture. The dimpled transgranular fracture consumes more energy as compared to fracture along grain boundaries which had been already partially separated by the formation and coalescence of voids during prior creep. The intermittent noise which accompanied the bending of the fracture toughness specimens made of the crept alloy was apparently caused by intermittent, sudden advancing of the cracks through discontinuities which had been created by coalescence of grain-boundary voids during the creep process.

TEM observations revealed the generation and multiplication of dislocations due to the high-temperature deformation process and due to the need to compensate for the lower net cross-sectional area of the discontinuity-containing creep specimens. The TEM observations did not reveal meaningful coarsening in the γ' precipitates which, if it happened, might have contributed to changes in fracture toughness during creep.

The formation and growth of intergranular voids during creep of polycrystalline metals and alloys have been studied extensively and received detailed reviews [6–8]. Several empirical approaches were made to predict creep life [9–11]. Others proposed a method of predicting remaining creep-life which is based on observing void density and creep strain [12]. Several theoretical approaches have been proposed for predicting time to rupture, at the end of the creep test, due to the growth of cavities [8, 13–17]. Constructors of fracture maps were also concerned with the time to rupture at the end of well-controlled creep and tensile tests [18, 19]. We observed in this work that during monotonic creep, a precipitation-hardened nickel-base alloy experienced a drastic reduction in its fracture toughness long before it exhausted its creep life. Thus, a situation may develop whereby a material with an initially accepted fracture toughness value may become extremely sensitive to the presence and/or introduction of sharp cracks or to a sudden overload, long before reaching the end of its creep-life or even the 1 to 5% creep strain allowed by some modern design practices [20].

4. Conclusions

Out of seven categories of microstructural change which may take place during high-temperature loading, the formation and coalescence of intergranular voids were the main causes of a drastic reduction in the fracture toughness of polycrystalline Inconel X-750 long before the alloy had reached the end of its creep life. The observations made in this and in previous work [1] point out the possibility that deleterious changes in fracture toughness, which may take place long before materials exhaust their creep lives, may be the governing factor which will determine the useful and safe life of materials which serve while loaded at high temperatures.

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