Microstructural analysis of creep exposed IN617 alloy

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Ni base alloys such as IN617 are one of the preferred choices for steam turbine components used by fossil fuelled power generation plants. IN617 is a solid solution strengthened Ni based superalloy containing ~23%Cr, 12%Co and 9%Mo with low content of precipitation strengthening elements Al, Ti and Nb. In the ‘as received’ (solution annealed condition), the microstructure consists of primary carbides (M23C6) and occasional TiN particles dispersed in a single phase austenitic matrix. Owing to high temperature exposure and the creep deformation processes that occur in service, evolution of the microstructure occurs. This results in secondary precipitation and precipitate coarsening, both on grain boundaries and intragranularly in areas of high dislocation density. The influence of creep deformation on the solution treated IN617 alloy at an operating condition of 650°C/574 h, with emphasis on the morphology and distribution of carbide/nitride precipitation is discussed. The applied stress was at an intermediate level.

Keywords: IN617, Solid solution strengthening, Hardness, Creep, Tin, Cr23C6

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Introduction

The fundamental requirement to increase the efficiency of fossil fuelled power plants is to raise the working thermal efficiency. Higher thermal efficiencies can be achieved by raising the steam operating pressure and temperature. The current thermal efficiencies of power plants are around 45%, typically operating at 30 MPa steam pressure and 600°C steam temperature.1 Recently, ultrasupercritical steam conditions up to 700°C and 37-5 MPa have been under consideration to increase the thermal efficiencies to ~50% or more.2-5 This has prompted the selection and identification of stronger high temperature materials, capable of operating under higher stresses at ever increasing temperatures. The required high temperature material properties for longer life are good creep rupture strength, long term microstructural and metallurgical stability, good fatigue resistance, fracture toughness and excellent corrosion and oxidation resistance in aggressive operating environments.6 Ni based superalloys have been under investigation for steam turbine components to meet these stringent material targets from the early 1970s. Among the Ni based superalloy family, IN617 was originally developed as a solid solution strengthened alloy and is now one of the preferred structural materials for future generation steam turbines and their components.7-11 In the present work, microstructural changes in creep exposed IN617 alloy, relevant to a plant operating temperature of 650°C, were evaluated.

IN617 is a Ni based austenitic superalloy having a face centred cubic crystal structure and primarily strengthened by solid solution hardening. Additional hardening is provided by the fine intermetallic precipitates, embedded in the disordered austenitic matrix phase.12 Alloying elements such as Cr, Co, Mo, Al, Ti, Fe, etc. are responsible for elevated temperature structural and mechanical performance. Solutes such as Cr, Co and Mo provide solid solution strengthening to the austenitic matrix and contribute to secondary hardening by localised carbide and nitride precipitation after aging heat treatment and during exposure at service temperatures. Solutes such as Al, Ti and Nb provide additional precipitation strengthening by coherent intermetallic precipitation.13,14

The precipitation of carbides, such as M23C6 (M=Cr, Co, Mo), MC and MN (M=Ti), within the grains and at grain boundaries, during service exposure, is a common phenomenon in Ni based superalloys.15,16 On exposure to high temperatures under mechanical stresses in aggressive environmental conditions with time, the mechanical properties of the alloys degrade, owing to changes that occur in their microstructures. The degradation of properties can ultimately lead to failure of components in service.17

This paper presents the effect of intermediate applied stress, time and temperature (574 h and 650°C) on IN617. These conditions are relevant to future power plant. Precipitate evolution and morphology have been studied with SEM and TEM. The evolution of mechanical properties has been investigated using hardness testing. Finally, the creep failure mechanism of the failed sample has been investigated by observation of the fracture surfaces and cross-sections through the fracture by SEM and optical microscopy (OM) respectively.
Experimental

Material chemical composition and heat treatment

The complete nominal chemical composition of as received IN617 is Ni–22.8Cr–9.0Mo–11.15Al–0.48Ti–0.35Fe–0.06C–0.05Si–0.02W–0.02Mn–0.013Cu–0.005Zr–0.005P–0.005V–0.004N–0.003Nb–0.002B (wt-%). A standard creep rupture specimen was prepared from a solution annealed forged rod of IN617. The ASTM grain size number and hardness of the as received specimen were 5 and 186 ± 6 HV20 respectively.

The solution annealed forged rods (1100 °C/3 h/WQ) were given a heat treatment (670 °C/10 h/AC), which usually precipitates primary carbides only on grain boundaries (Fig. 1). Creep test specimens were then machined from the rods. The creep rupture test was conducted at 650 °C under an applied intermediate stress in air by Alstom Power Ltd (Rugby, UK). The creep test was continued until fracture occurred.

Hardness and microstructural observations

Vickers hardness tests were conducted using a Vickers hardness testing machine, with a 20 kgf load on ground and polished longitudinal cross-section of the creep fractured specimen. The failure mechanism of the creep exposed sample was evaluated using environmental SEM. The metallography was conducted on the specimen after etching in glyceregia, which is a mixture of hydrochloric acid, glycerol and nitric acid in a 3:2:1 ratio, using OM and field emission gun SEM. The Heyn lineal intercept method was used to measure the average grain size on optical micrographs of as received and creep exposed samples.18

The area fractions of phases were determined on different fields of view, at magnifications of ×5000 to ×10 000, on backscattered electron field emission gun SEM images. These magnifications were chosen because they were high enough to resolve the phase morphology. The representative area fractions are the mean of individual area fractions of phases. The standard errors were in the range from 0.1 to 3.4%.

Thin foils having thickness ranging from 0.1 to 0.12 mm and a diameter of 3 mm were prepared from the gauge length of the creep exposed specimen for TEM analysis. These foils were mechanically polished and subsequently subjected to twin jet electropolishing in a solution of methanol with 20 vol.-% perchloric acid at −22 °C under an applied potential of 14 V.

X-ray diffractometry and peak separation

X-ray diffractometry (XRD) was performed using a Philips PW 1716 diffractometer, with Cu Kα1 radiation (λ=1.54 Å) in the angular range of 2θ=20°–125°, Δ2θ=0.0096° and t=0.5 s/step, to characterise the phases present in the creep failed sample. Some potential precipitate peaks overlap with the austenitic γ matrix peaks. The PeakFit19,20 software was used to separate the peaks.

Results and discussion

Microstructural stability of creep exposed alloy

Figure 2 shows the optical microstructures of the as received (solution annealed) and IN617 sample creep exposed to 650 °C for 574 h at an intermediate stress. The as received sample shows an equiaxed austenitic grain structure (Fig. 2a). The grain orientation has previously been shown to be random.21 Twins and twinning boundaries are observed in a number of grains. The grain size varied from a minimum of 70 μm to a maximum of 440 μm with an average size of 120 μm. The austenitic grain structure of the creep exposed IN617 sample is shown in Fig. 2b. The grain size of the

1 Heat treatment cycle

2 Images (OM) of IN617 a as received and b creep exposed at 650 °C for 574 h at intermediate stress, from longitudinal gauge section, 2 cm behind fracture surface
creep exposed sample varied from a minimum of 46 μm to a maximum of 544 μm with an average size of ~158 μm.

Figures 3 and 4 show montages of SEM images of as received and creep exposed IN617 samples respectively. Carbides are present in the matrix and at grain boundaries. The grain boundary precipitates are mostly rich in chromium, when analysed by energy dispersive spectroscopy (EDS). The blocky particles M(C,N) are mainly composed of titanium, when analysed by EDS and are randomly distributed in the matrix and at twin boundaries. They will be referred to in the paper as Ti (C,N). The XRD result (see later) shows the precipitates to be Cr rich M23C6 carbides and Ti (C,N). The Cr rich M23C6 carbides will be referred to as Cr23C6 in the present paper. The as received SEM images (Fig. 3) indicates that precipitates of size <2 μm are situated in intergranular regions and some larger particles within the grains. The morphology of the precipitates was found to vary considerably from irregular geometries to a more symmetrical cubic geometry. Cr rich precipitates (M23C6) varied in size from 1 to 6 μm and occupied an area fraction of 1-4%. Ti rich precipitates (TiC/TiN) were observed to range in size from 2 to 10 μm, and the area fraction was 0-3%.

Figure 4 shows that precipitates were found both intra- and intergranularly in the creep exposed specimen. The precipitate morphology varied from a symmetric to a totally asymmetric morphology. The size of Cr based precipitates ranged from 1 to 17 μm, and the area fraction was 6-3%. The size of Ti based precipitates was 3–5 μm, and the area fraction was 0-8%. The area fraction of Cr precipitates increased from 1-4% in the ‘as received’ to 6-3% in the creep exposed.

The major precipitates found in as received IN617 alloy and in the gauge length after creep exposure are shown in Table 1.

### Fractography of creep exposed sample
The failure mechanism of the creep fractured specimen was analysed using SEM and OM. A fractograph of the creep failed sample is shown in Fig. 5. The failure mechanism was intergranular fracture by decohesion of the grain boundaries. Intergranular creep fracture involves the nucleation, growth and subsequent linking of voids at grain boundaries to form two differing types of cavities: wedge type cavities and isolated type cavities. Wedge type cavities are associated with cracking at grain boundary triple points and form by grain boundary sliding. The formation of cavities is controlled by diffusion controlled processes. Triple point wedge cracking (marked) and creep cavitation (arrowed) are shown in Fig. 5.

The failure mechanism of the sample was further confirmed by OM and SEM of a polished cross-section of the fractured surface. Figure 6 shows further confirmation of wedge cracking, decohesion and crack propagation along the grain boundaries in the creep exposed sample (650°C for 574 h creep duration). The

| Table 1 Precipitate evolution after creep exposure |
|---------------------------------------------------|
| **Precipitate morphology**                       |
| **Specimen ID** | Precipitate location | Size, μm | Shape | Area fraction, % |
|---------------|----------------------|----------|-------|-----------------|
| As received   | Intra- and intergranularly | Ti(C,N)  | 2–10  | Symmetrical to totally | 0-3 |
|               |                      | Cr23C6   | 1–6   | asymmetrical geometry | 1-4 |
| Creep exposed | Extensive precipitation of Cr enriched particles on grain boundaries and twin boundaries | Ti(C,N)  | 3–5   | | 0-8 |
|               |                      | Cr23C6   | 1–17  | | 6-3 |

3 Image (SEM) of as received IN617: blocky shape (darker contrast) particle (marked ‘Ti’) is Ti (C,N) and grey contrast particles (marked ‘Cr’) are Cr23C6, distributed intragranularly; Cr23C6 are also present on grain boundary

4 Image (SEM) of creep exposed sample: blocky shape (darker contrast, marked ‘Ti’) near twin boundary (marked with white arrow) is Ti rich phase, TiN; facettted particles (marked ‘Cr’) are distributed intragranularly, at triple junctions, and also, decorating grain boundary (black arrow) and twin boundaries (white arrow) are Cr rich phase, Cr23C6
direction of loading is shown by the arrows in Fig. 6. The open cracks intruded inside the sample to a depth of 100–500 µm.

**Hardness profile for creep exposed IN617**

The hardness was measured at 2 mm intervals along the head and gauge length of the longitudinally sectioned specimen of the creep exposed sample. The results of the hardness testing are shown in Fig. 8. Each hardness value is an average of three indentations across the diameter of the specimen. The error bars show the 90% confidence limits.

In Fig. 8, it can be seen that the hardness in the head region of the specimen, where it may be assumed that the major effect on the sample from creep exposures is from the thermal effects with minimal effect from stress, the hardness is considerably lower than the hardness measured along the gauge length. The head hardness has however increased substantially in the creep exposed sample above that of the 'as received' sample, which had a hardness of 186.6 HV. The average value of Vickers hardness in the gauge length was 334 HV. This sample showed little reduction in area at failure (17-6%). When the microstructure was analysed, the sample showed that extensive precipitation of Cr and Ti enriched precipitates had occurred throughout the matrix. The rise in hardness observed in the head and gauge of the specimen is attributed to further development of the precipitates (namely the area fraction of Cr₂₃C₆ has increased from 1-4% in the as received sample to 6-3% in the creep exposed sample).

The internal damage has occurred from coarsening of the carbide phase, grain growth and grain boundary creep voidage. Internal damage of this nature does not lead to any dimensional changes in the specimen but results in a decrease in high temperature mechanical properties such as creep rupture strength.²²,²³

**Transmission electron microscopy**

The microstructure of creep exposed IN617 alloy has been analysed using analytical TEM. Phase identification was performed using electron diffraction patterns and X-ray spectrometry (EDS).

Figure 8 shows a montage of TEM images of the creep exposed sample. The incident direction of the electron beam is [001]. The selected area electron diffraction pattern indicates that the precipitates are Cr₂₃C₆ phase. The dislocation activity near the carbide particles is arrowed. Grain boundaries are decorated with varying sizes of Cr₂₃C₆ carbide particles. Their large variation in size might indicate that they formed both at different stages of the thermal treatment and during creep exposure.

Figure 9a depicts a region at relatively low magnification. Along the grain boundary, fine scale precipitates have formed. A magnified image of the region (marked) is shown in Fig. 9b. These tiny precipitates (<0-1 µm) are potentially second generation carbides formed in the material during the application of stress. These carbides were found on the grain boundaries and near the larger precipitates.

**X-ray diffraction**

X-ray measurement was performed on the as received and creep exposed samples to characterise the precipitates formed under the application of intermediate stress and high temperature. The resultant spectrum for the creep exposed sample is shown in Fig. 10.

One of the difficulties of identifying precipitates in IN617 is that the peak positions for a number of the carbides and nitrides overlap. Following the approach of Mukherji et al.,¹⁹²⁰ peak separation has been performed using PeakFit software.¹⁹ Thus, the precipitates in the sample were identified to be Cr₂₃C₆ and TiN/TiC by analysis of the XRD trace.
Conclusions

The experimental observations on phase precipitation and microstructural evolution in creep exposed IN617 (650°C/574 h/intermediate stress) are summarised as follows.

1. The average grain size of the as received IN617 was 120 μm, but for the creep exposed IN617 specimen, the average grain size was increased to 158 μm.

2. The rise in hardness observed in the creep exposed sample is thought to be associated with additional Cr23C6 precipitation during creep exposure.

3. Precipitates in as received and in the creep exposed samples were identified as Cr enriched (M23C6) and Ti enriched (TiN). In the creep exposed sample, the precipitates were found to precipitate in large proportions at twin and grain boundaries, but otherwise, precipitates were randomly distributed throughout the matrix.

4. Precipitates were found to have varying morphology from a symmetric to an asymmetric morphology.

5. The creep fracture mode was found to be predominantly intergranular in nature.

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9 Images (TEM) illustrating different scales of second phase carbides in creep exposed sample

10 X-ray diffractograph (intensity versus 2θ) of creep exposed sample

Creep-exposed IN617 (800°C/554 h/intermediate stress). Aged: 10 h at 650°C/CAC

Cu Kα Wavelength = 1.54 Å

- matrix
- Cr23C6
- TiNi(3C)

Intensity [Counts]

2θ (Degrees)

20 40 60 80 100

29 59 89 119 149

X-ray diffractograph (intensity versus 2θ) of creep exposed sample