Effect of thermal and thermomechanical ageing on the microstructure and mechanical properties of a Ni-Cr alloy

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Abstract. Thermal ageing experiments at 850 and 950°C for 1000 h with and without an applied stress have been applied to INCONEL 617 alloy. The consequences of these treatments characterized in terms of microstructure evolution and the tensile behavior have been studied at room and elevated temperatures. It is shown that thermal ageing strongly reduces the ductility of the alloy at room temperature, whereas the saturation stress measured at elevated temperature is only slightly affected. Applying a small stress of 7MPa during thermal ageing has negligible effects on the tensile behavior.

1. Introduction

In Very High Temperature Reactors (VHTR), the Intermediate Heat eXchanger (IHX) imposes severe conditions (coolant temperature up to 1000°C) for long time (target: 20000h). Currently, the solid-solution nickel based alloy INCONEL 617 is a candidate material for the IHX and the hot ducts of the VHTR thanks to its oxidation and corrosion resistance and its high temperature mechanical properties. In the range of ageing temperature [700°C-1000°C], alloy 617 shows a peak of hardness within hundred first hours of ageing followed by hardness stabilization [1,2]. This peak has been related to the precipitation of intragranular Cr rich M₂₃C₆ carbides that appear at the beginning of thermal treatment, localized on dislocations in the slip lines [1,3,4]. For longer ageing time, these intragranular carbides are dissolved or coalesce. The consequences of such microstructural evolution on the mechanical properties of the INCONEL 617 have been widely studied in the literature for various types of mechanical testing: tensile testing [3,5-8], creep [1,4,9,10] and fatigue [11].

In the present paper, we report the results of tensile testing at room and elevated temperatures (850 and 950°C) and we compare the properties of annealed and heat treated alloy. Heat treatments were realised at 850 and 950°C and were complemented by thermomechanical treatment, consisting of ageing at the same temperatures under an applied stress of 7 MPa. This latter treatment is believed to be more representative of the life operation conditions of the alloy in the IHX. The effect of the imposed strain rate is also examined at high temperature for the three states of the alloy.
2. Experimental
A bar of INCONEL 617 with a diameter of 25.4 mm was purchased from Special Metals (chemical composition is given in Table 1). The material was hot forged and then solution annealed at 1175°C for 36 min followed by air cooling; this annealed state is further referred to “as received” (AR).

| Table 1. Chemical composition of the Inconel 617 supplied by Special Metals (wt %) |
| Ni | Cr | Co | Mo | C | Ti | Al | Si | Mn | Fe | Cu |
|---|---|---|---|---|---|---|---|---|---|---|
| 55.35 | 21.49 | 12.00 | 9.23 | 0.060 | 0.36 | 0.95 | 0.12 | 0.06 | 1.24 | 0.06 |

Parts of the initial bar were cut for thermal ageing (TA) treatments of 1000 h at 850°C or 950°C. Those TA sample were introduced in cold furnace working in air, the furnace was then warmed-up to ageing temperature, held for 1000 h and finally the samples were all furnace cooled. The samples for thermomechanical (TMA) treatments (part of the initial bar) were aged under a constant load at 850°C or 950°C for 1000 h corresponding to a stress of 7 MPa (normal service conditions in the IHX). The ageing duration of 1000 h has been chosen because all the major macrostructural evolutions occur for shorter treatments. After thermal and thermomechanical ageings, part of the treated rods are mechanically polished and electrochemically etched for optical and scanning electron microscopy.

Cylindrical specimens with a gauge diameter of 4 mm and 20 mm gauge length were machined from the as received and aged alloy 617 rods. Tensile tests were performed in laboratory at room temperature and ageing temperature (850°C or 950°C). For high temperature testing, the sample was first heated and held for 1 h at the testing temperature in order to obtain good thermal equilibrium of the whole load train. Tensile tests were conducted on screw driven electromechanical testing machines, using a displacement rate of the crosshead of 0.6 and 6 mm min⁻¹ corresponding to an imposed strain rate of 5.10⁻³ and 5.10⁻⁴ s⁻¹, respectively. Deformation was measured directly on the gauge length using a MTS extensometer. The tensile curves are shown in engineering stress and strain coordinates.

3. Results and discussion
3.1. Microstructural evolution during thermal and thermomechanical treatments
The initial microstructure reveals γ austenitic grains (ASTM 2) containing homogeneously dispersed Ti(C,N) precipitates and alignment of Mo rich M₆C carbides parallel to the axis of the bar. Few intergranular Cr rich M₃₂C₆ secondary carbides are also present, principally at grain boundaries. After ageing at 850°C, numerous fine Cr rich carbides are observed on slip lines, while they have totally disappeared after 1000 h at 950°C. The decomposition of primary carbides (Ti(C,N) and M₆C) that transform to M₃₂C₆ appears in aged samples, more developed at 950°C [1]. M₃₂C₆ carbides are also observed at grain boundaries with a morphology which depends on the treatment temperature (dendritic and globular at 850°C, globular at 950°C [1,4]). At the scale of the optical and scanning electron microscope one cannot distinguish any difference of microstructure for the samples treated with and without stress. However, it is difficult to quantify and compare the amount of small M₃₂C₆ intragranular carbides in samples aged with and without stress at 850°C since their distribution depends strongly on the observed grain.

3.2. Tensile properties
3.2.1. Room temperature tensile tests. The curves of tensile tests at room temperature on the as received Inconel 617 and aged materials are presented in figure 1. Comparing the AR and TA material, the ageing treatment at 850°C slightly reduces the yield strength (YS) but it also increases the work hardening rate so that the ultimate tensile strength (UTS) of the aged sample is nearly the same as the AR one while the elongation to rupture and reduction in area have been strongly reduced. These differences can be related to the precipitation of the M₃₂C₆ carbides on slip lines and at grain boundaries. During ageing at 950°C, the intragranular carbides have dissolved, inducing a more
important reduction of the YS and the UTS. According to figure 1, applying low stress during the ageing treatment at 850°C has little influence on the tensile response of the alloy. Only a slight reduction of tensile strength and ductility is observed. This phenomenon might be related to the coarsening of $M_23C_6$ carbides which could be enhanced by a promoted diffusion thanks to the stress during heat treatment.

At 950°C the diffusion is more efficient. So, the reduction of the tensile response of the INCONEL 617 due to the stress during ageing treatment at 950°C is a bit more significant for YS and UTS values and further reduces the ductility, while no difference is observed on work hardening rate, as after treatments at 850°C.

3.2.2. High temperature tensile tests. Figure 2 shows the ensemble of results obtained by high temperature tensile testing of the as received and treated Inconel 617 at two strain rates: $5 \times 10^{-3}$ s$^{-1}$ and $5 \times 10^{-4}$ s$^{-1}$. First of all, one notices that all the tensile curves obtained at high temperature are very similar for all the conditions that have been tested, and the material behaves at high temperature very differently from room temperature. Indeed, some strain hardening is only observed at 850°C for the higher imposed strain rate, $5 \times 10^{-3}$ s$^{-1}$, at the beginning of the tensile curve. After the UTS, it has been verified that the engineering stress decrease does not correspond to the necking of the specimen but instead to a homogenous strain and a constant true stress, revealing a steady state deformation. For the lower strain rate at 850°C and for 950°C, this steady state deformation starts nearly at the plastic transition, sometimes associated to a more or yielding. Such behaviour has been reported in the literature [12]. It clearly appears in Figure 2 that this steady state stress level strongly depends on both temperature and strain rate. We have verified that these stress levels fit very well with the Norton plot obtained during constant load creep experiments performed at the same temperature [1], showing that similar mechanism operates over a wide plastic strain rate range (from $5 \times 10^{-3}$ s$^{-1}$ as shown in this paper down to $10^{-8}$ s$^{-1}$ for creep tests [13]).

![Figure 1. Room temperature engineering stress vs. strain curves of as received and treated Inconel 617 at a strain rate of $5 \times 10^{-4}$ s$^{-1}$.](image1)

![Figure 2. High temperature engineering stress vs. strain curves of as received and treated Inconel 617 at $5 \times 10^{-3}$ s$^{-1}$ and $5 \times 10^{-4}$ s$^{-1}$ imposed strain rate for two temperatures: (a) 850°C and (b) 950°C.](image2)
One can also notice the occurrence of plastic instabilities on tensile curves, especially for the lower imposed strain rates. The role of solutes atoms may be important in the high temperature regime.

For both 850 and 950°C testing temperature, the general effects of thermal ageing are similar: TA and TMA tend to slightly decrease the stress level, but in moderate way. A more marked effect is visible on the ductility of the alloy, especially for the higher strain rate (5 × 10^{-3} s^{-1}). Finally, comparing TA and TMA, it appears that applying a stress does not affect the tensile behavior as compared to samples that were aged by thermal treatment only.

4. Conclusions
This paper presents one of the first studies on the effect of stress ageing on tensile properties of the INCONEL 617. The low stress imposed during 1000 h heat treatments at 850°C and 950°C to simulate normal service conditions in the IHX does not induce major microstructural differences and tensile properties compared to alloy aged without stress under the same conditions. Ageing treatments are therefore sufficient to study the alloy for high temperature applications.

Precipitation of secondary M₂₃C₆ carbides occurred during ageing treatments in INCONEL 617; they promote damage evolution and reduce ductility at room temperature. The RT work hardening is increased after treatments at 850°C as compared to the as received and 950°C treated material due to different intragranular precipitation.

For high temperature tensile tests, deformation reaches a saturation stage which is only slightly affected by TA or TMA treatments.

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