High Temperature Mechanical Behavior of a 30%Ni–19%Cr Steel

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The deformation behavior at high temperature of a Type 800 Incoloy alloy reinforced by TiC particles was investigated by tensile and torsion tests at temperatures ranging from 800 to 1 200°C. The as-received material exhibited a microstructure of coarse equiaxied grains with subgrains decorated by small TiC particles. The material showed an activation energy for plastic flow of 400kJ/mol similar to the activation energy for lattice diffusion in austenitic stainless steels. The stress exponents varied strongly with testing conditions. After testing at temperatures up to 1 000°C, the initial austenitic grains were elongated in the tensile direction, but the subgrain structure did not change with stress. In contrast, a microstructural refinement was observed after tensile testing at 1 100°C, which was associated with a process of dynamic recrystallization that occurs during deformation. At this temperature the deformation behavior of the material can be described by a slip creep mechanism. At the rest of the test temperatures the controlling mechanism is that of constant-structure slip creep.

KEY WORDS: iron-base superalloy; microstructure; high temperature mechanical behaviour; constant structure creep.

1. Introduction

Fe–Cr–Ni alloys have been widely used over the last half-century as structural material in a great variety of applications demanding high corrosion and heat resistance as for jet engines, petrochemical plants or power plants. Many different aspects of the mechanical properties and forming behavior of the Cr–Ni stainless steels have been widely investigated.1,2 These steels exhibit good combination of ductility and toughness even at high-strength levels when an austenitic microstructure is stabilized. However, standard austenitic stainless steels suffer from the drawback of a low proof stress value, which falls even more with increasing the temperature.3 As a result, these materials are not satisfactory for elevated temperature applications.4

Many attempts have been performed to improve the creep strength of simple austenitic stainless steels to operate in the temperature range from 600 to 700°C. At this service temperature, the strengthening mechanism used for these materials (based on cold working and martensitic transformations) loses its effectiveness during long exposure.5 In this case, an effective way to increase the high temperature strength is by introduction of a fine distribution of second phase particles homogeneously distributed in the matrix. Second phase particles precipitation in Fe–Cr–Ni alloys includes the addition of some amount of interstitial elements, carbon and/or nitrogen, and carbide-stabilizing elements.1,2 M23C6 carbides are the most favorite strengthening particles.4 Since these particles are incoherent with the matrix, they can act as barriers to dislocation movement and force dislocation by-passing of the particle. The specific kinetics of precipitation depends on the chemistry of the alloy, heat treatment and precipitation sites. At temperatures of higher than 700°C intergranular precipitation becomes predominant.5 Intergranular precipitates are effective in suppressing the nucleation of mechanical damages like voids and cracks and also their propagation.6,7 In addition, intergranular dispersoids may increase the creep resistance.8,9 However, strengthening due to intergranular precipitation is much less effective than intragranular precipitation.

Iron-base superalloys evolved from austenitic steel, type AISI 304, to reach better properties at higher temperatures. These iron rich alloys have large amounts of Ni (13 to 43 wt%) and Cr (13 to 20 wt%). Elements with high affinity for carbon and nitrogen, like titanium, niobium, tantalum and zirconium, are introduced to promote intragranular precipitates of MC carbides, carbonitrides or nitrides.10,11

This investigation is aimed at the study of the elevated temperature mechanical behavior of 30Ni–20Cr stainless steel strengthened by Ti carbide and nitride. Based on the consideration of the relative atomic weight of titanium and carbon, the titanium content required to stabilize TiC carbides is about four times the carbon content. Thus, for a Ti content of 0.5 wt% a carbon content of about 0.12 wt% was added. The deformation behavior at high temperature of this alloy was investigated by tensile and torsion tests at temperatures ranging from 800 to 1 200°C. In order to elu-
cidate the controlling deformation mechanism, the investigation was completed with a detailed microstructural analysis of the as-received and deformed materials.

2. Experimental Procedure

The as-received material was supplied as a bar of 400 mm, which was prepared by hot rolling a bar of 600 mm in diameter heated to 1200°C for 45 min and then air cooled. The chemical composition of the alloy used in the present study was determined by Inductively Coupled Plasma, except for nitrogen and carbon that were determined by infrared absorption after combustion in an induction furnace. The composition was the following (wt%): 30Ni–19Cr–0.8Mn–0.5Al–0.5Si–0.2C–0.01N, balance Fe.

The microstructures of the as-received material and after deformation were studied by optical microscopy, and scanning electron microscopy (SEM) equipped with energy dispersive X-ray (EDX) microanalysis. Metallographic preparation included mounting the samples in gauelette and polishing by the conventional method. The microstructure was revealed by etching at room temperature with a solution of 45 mL of HCl, 15 mL of HNO₃ and 20 mL of distilled water.

Elevated temperature mechanical properties were characterized by tensile and torsion tests. The tensile tests were performed at strain rates ranging from $3 \times 10^{-6}$ to $2 \times 10^{-3}$ s⁻¹ in the temperature range 800 to 1100°C. Tensile samples with a rectangular cross section of 3 × 5 mm and a gage length of 20 mm were machined in the direction parallel to the bar axis. In order to minimize the effect of oxidation, tensile tests were performed under a protective argon atmosphere. True tensile strains, stresses and strain rates were calculated from the load–deformation curves with the assumption of uniform deformation and conservation of volume.

Hot torsion tests were performed at equivalent strain rates ranging from 1 to 20 s⁻¹ in the temperature range from 1100 to 1275°C. The torsion samples, machined in the direction parallel to the tube axis, had a circular cross section of 6 mm in diameter and a gauge length of 17 mm. The samples were introduced in a silica tube with a helium inlet to ensure protection against oxidation and to minimize adiabatic heating. All samples were held at temperature during 20 min for homogenization. The equivalent true flow stress and strain were calculated from the experimental torque-number of revolution curves using Fields and Backofen relation.[12]

3. Results

3.1. Microstructure

The as-received material is characterized by a coarse microstructure with equiaxial austenitic grains with an average grain size of about 300 μm, Fig. 1(a). An analysis at higher magnification of these grains showed the presence of a structure of subgrains of about 10 μm in size decorated by second phase particles, Fig. 1(b). As these particles are smaller than the excitation area of the electron beam, only a qualitative microanalysis from the EDX was possible. As the microanalysis showed the presence of a high level of Ti and carbon, they were identified as TiC. The origin of this substructure has been associated with the intragranular precipitation of these carbides on dislocations and/or stacking faults during solidification. In addition to TiC, SEM images showed also the presence of other second phase particles of about 10 μm in size with a well defined cubic morphology, as shown in Fig. 2. These particles have been identified as TiN by EDX microanalysis.

Microstructure observations after tensile strain rate change tests were conducted. In the samples tested at temperatures up to 1000°C, the initial austenitic grains were...
elongated in the tensile direction, as shown at 950°C in Fig. 3(a). A detail at higher magnification of this microstructure, Fig. 3(b), shows that these deformed grains have similar subgrain structure as that of the as-received material, Fig. 2. On the other hand, a microstructural refinement was observed after testing at 1100°C, Fig. 4. This figure shows a finer microstructure than that of Figs. 1(a) and 3(a). This result was associated with a process of dynamic recrystallization that occurs during deformation. The presence of subgrains was not revealed even at high magnification.

3.2. High Temperature Mechanical Behavior

The high temperature mechanical behavior was studied by means of strain-rate-change tension tests and torsion tests at various strain rates from which the stress and temperature dependencies of the strain rate were obtained. The true strain–true stress curve of the tensile tests presents a well-defined steady state at all strain rates in the temperature range from 800 to 1100°C. On the other hand, the equivalent stress–strain curves obtained from the torsion test had the characteristic peak stress. All samples failed just after the peak.

The results of the high-temperature deformation tests are presented as a plot of the steady-state strain-rate, $\dot{\varepsilon}$, against the Young's modulus compensated flow stress, $\sigma/E$, on log-log scales in Figs. 5(a) and 5(b) for the tensile and torsion tests respectively. The values used for the Young's

![Fig. 3. Microstructure after tensile strain rate test at 950°C at (a) low magnifications and (b) high magnifications.](image)

![Fig. 4. Microstructure after tensile strain rate test at 1100°C.](image)

![Fig. 5. Logarithm steady state strain rate vs. logarithm Young's modulus compensated flow stress from (a) tension and (b) torsion tests.](image)
modulus at the various temperatures were taken from the data available for stainless steels of the 300 series.\textsuperscript{13} On these plots, the slope of the curves corresponds to the stress exponent, \( n \). Fig. 5(a) shows \( n \) values of about 7 at testing temperatures up to 1 050°C and about 5 at 1 100°C. On the other hand, the torsion data given in Fig. 5(b) show high stress exponents. The activation energy for creep derived from both sets of data given in Figs. 5(a) and 5(b) is about 400 kJ/mol. This value is similar to that found in stainless steels and high alloy steels.\textsuperscript{14}

4. Discussion

Available data for 304 and 316 stainless steels show a stress exponent of about five.\textsuperscript{15,16} It is generally accepted that the rate-controlling mechanism for 5-power law creep is associated with slip creep where the controlled step is due to dislocation climb at pile-ups.\textsuperscript{17} Subgrains have been reported to form during deformation under this mechanism at temperatures above 700°C.\textsuperscript{18,19} The size of these subgrains, \( \lambda_s \), resulting from this type of creep, is strongly dependent on stress and is independent of the temperature and the steady state-creep strain. It is well established that the subgrain size varies with stress according to the relation:

\[
\lambda_s = A_s b \left( \frac{\sigma}{E} \right)^{\frac{1}{2}} \quad \text{(1)}
\]

where \( b \) is Burgers vector and \( A_s \) is a constant for a given material.\textsuperscript{20,21}

In the type 800 Incoloy of this investigation, a stress exponent of about five was obtained only at 1 100°C where the material underwent a recrystallization process during deformation. This is revealed in the micrograph of a sample deformed at 1 100°C, Fig. 4, showing a microstructure refinement during deformation due to dynamic recrystallization. Recrystallization will cause softening during testing which may be responsible for the low stress exponent.

In contrast, stress exponents in the range 7 to 9 were deduced from the tensile tests carried out in the temperature range 800 to 1 000°C. This high stress exponent suggests that deformation is governed by a constant structure slip creep mechanism, \textit{i.e.} a creep mechanism where the subgrain size would not vary with stress. The particle strengthened material of this investigation contains a fine subgrain structure that is stabilized mainly by the presence of carbides. Such microstructure should remain invariant during creep. The microstructure observed after tensile testing at temperatures below 1 050°C (Fig. 3(a)) indicates that the initially equiaxic austenitic grains are elongated in the tensile direction. It has been also shown that the subgrain size remain unaltered in the course of deformation indicating that the Incoloy material creeps at constant structure. Under this condition, \( \lambda_s \) is related with the strain rate as \( \dot{\varepsilon} \propto \lambda_s \) and the constitutive equation that describes the creep behavior is the following:\textsuperscript{21}:

\[
\dot{\varepsilon} = \left( \frac{D_s}{b^2} \right) \left( \frac{\lambda_s}{b} \right)^3 \left( \frac{\sigma}{E} \right)^8 \quad \text{(2)}
\]

The stress exponent of about 7 found in this work is close to that given by this equation. This result and the constant microstructure observed after creep attest to the validity of Eq. (2) to describe the mechanical behavior of the Incoloy material at most temperatures.

The activation energy for deformation for the tensile and torsion sets of data was determined as 400 kJ/mol and is typical of many stainless steels. Figure 6 shows a Zener–Hollomon parameter (\( Z = \dot{\varepsilon} / \exp (-Q/RT) \)) vs. \( \sigma/E \) plot. In this figure, the data obtained at various temperature fit well into a single curve for each type of tests. It is to be noted that for the same value of the Zener–Hollomon parameter, the torsion stress is stronger than the tensile stress, and therefore both sets do not fit into a single curve. This unusual creep behavior has been related to the interaction between matrix dislocations and second-phase particles and the lack of a steady state. This interaction promotes work hardening at high strain rates since the climbing rate for the corresponding mobile dislocation density is not sufficiently fast and the elimination of mobile dislocation can not be achieved at the required rate relative to their production. In addition, limited ductility is attained in the torsion tests which makes even more difficult to reach a steady state of deformation.

5. Conclusions

(1) The microstructure of the as-received material consists of coarse austenite grains containing subgrains and fine particles of TiC. The grains are elongated after tensile testing at temperatures below 1 050°C, but the subgrain size remain constant.

(2) Analysis of the creep data at temperatures below 1 050°C according to a conventional power-law creep relation led to stress exponent values of about 7 and an activation energy for plastic deformation similar to that for lattice self-diffusion in austenitic stainless steels. A constant struc-
ture creep mechanism is attributed to control deformation.

(3) Recrystallization occurs during tensile tests at 1100°C and a stress exponent of about 5 is obtained. Deformation at this temperature is controlled by a conventional slip creep mechanism.

(4) The material tested in torsion is stronger than that tested in tension at the same temperature. This is attributed to the lack of a steady state during the torsion tests.

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