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High-temperature creep resistance and effects on the austenite reversion and precipitation of 18 Ni (300) maraging steel

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ABSTRACT

In this paper, the high-temperature creep resistance and effects on the austenite reversion and the dynamic evolution of precipitates of maraging 300 steel were investigated. The main strengthening mechanism in a solution treated and aged material is the fine needle shaped $Ni_3(Ti,Mo)$ precipitates densely dispersed in a single martensitic phase. The specimens were submitted to creep tests at temperatures of 550, 600 and 650 °C and stress conditions of 200, 300 and 500 MPa. Stress exponent (n) varied from 6.0 to 7.2 and activation energy for creep (Q_c) from 364 to 448 kJ/mol, associated to the tangled and cells arrangements of the dislocations, show that the dominant creep mechanism is controlled by dislocations climb and slip. The experimentally determined threshold stresses are about 25 MPa at 550 °C and close to 4 MPa at 600 and 650 °C. Due to high-temperature creep exposure, part of martensite was reverted to austenite in a range of 17.2% to 48.5%, depending upon the time, temperature and applied stress. At the same time, the $Ni_3(Ti,Mo)$ precipitates were coarsened and Fe_2Mo precipitated, leading to undesirable alloy's strength reduction. Volume fraction of reverted austenite showed strong negative correlation with hardness. Fracture surfaces of specimens presented ductile failure consisting of equiaxed and bi-modal dimples in the fibrous zone surrounded by 45° shear lip.

1. Introduction

The superior properties of maraging steels, such as ultra-high strength, high ductility, good hardenability, good weldability, simple heat treatment without deformation steps have led to widespread application of maraging steels for demanding applications. Hence, it has been considered to be an excellent material not only for aerospace, military and nuclear industries, but also for transportation, manufacturing, tooling, die making and electromechanical components. The high strength and high toughness comes from the precipitation strengthening of martensitic microstructure during aging heat treatment. Maraging 300 steel is a member of iron–nickel based alloy family [1–4].

Creep behavior studies on a variety of different materials and metals systems have increased significantly the understanding of the deformation mechanisms responsible for the evolution of the microstructure during plastic flow. Major improvements in creep resistance can be achieved by introducing a dispersion of fine precipitates, such as the intermetallic precipitates in maraging steels, which provide effective obstacles to dislocation movement [5]. By the other hand, the intermetallic precipitates formed during aging of maraging steels are not the stable equilibrium phases in the sense that prolonged high temperature exposure would lead to the formation of equilibrium austenite and ferrite. Since nickel, one of the major alloying elements, is an austenite stabilizer, the reversion tendency to austenite depends on whether the alloying elements enrich or deplete the matrix with respect to nickel [1–4,6]. Data available on the mechanical properties of maraging steel at elevated temperature under creep are scarce [3,7–10]. Specialized applications of the steel occasionally demand short-time exposures to high temperatures and it is desirable to have data on the creep behavior of the material during such service conditions. Although there are some research on the identification of precipitates in maraging steels [1–4,6,8,9,11–13], there is no study related to the studying of microstructure, morphology of precipitates and reverted austenite after exposure to high temperature and stress during creep tests and their relation to mechanical properties. Therefore, an investigation of microstructure and its correlation with creep behavior at high temperature in maraging 300 steel is a subject of interest and is discussed in this paper.

2. Experimental procedures

The maraging steel used in this study was a 300 grade solution treated at 820 °C — 1 h and then air cooled followed by aging at 480 °C — 3 h and then air cooled in a Brasimet Koe 40/25/65 furnace. The adopted heat treatment is the industrial practice which presents an optimal strength, ductility and toughness combination [2,3]. The chemical

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Table 1
Chemical composition (wt.%) of the maraging 300.

Ti	Co	Mo	Ni	Al	C	S	P	Si	Mn	Fe
0.63	9.37	4.94	19.00	0.08	0.008	0.002	0.004	0.06	0.01	Balance

composition of the material is given in Table 1. The specimens with a 18.5 mm gauge length and a 3.0 mm in diameter were submitted to constant load creep tests at temperatures of 550, 600 and 650 °C and stress conditions of 200, 300 and 500 MPa in a standard Mayes creep machine, according to ASTM E139 standard [14]. In order to characterize in terms of microstructure and mechanical properties before and after the creep tests the following tests were performed: dilatometry (Linseis model L75V 1400 RT), microhardness (FutureTech model FM-700), X-ray diffraction (Panalytical model X'Pert Powder), optical microscopy (Carl Zeiss model Axio Imager 2), scanning electron microscopy (TESCAN model VEGA 3) and transmission electron microscopy/energy dispersive X-ray micro-analysis (Philips TECNAI model G2F20 TEM).

3. Results and discussion

3.1. Characteristic of the material before creep tests

Table 2 shows transformations temperatures obtained from dilatometry test for the maraging 300 and represented as P_s (Precipitation start), P_f (Precipitation finish), A_s (Austenite formation start), A_f (Austenite formation finish), M_s (Martensite formation start) and M_f (Martensite formation finish). The results indicate that the material should have a single martensitic phase structure upon cooling to room temperature as far as the M_f is 62 °C.

The X-ray diffraction (XRD) pattern in the 2θ ranging from 35–105° is shown in Fig. 1. The XRD spectra confirm that the microstructure of the solution treated and aged specimen is completely martensitic at room temperature corroborating with dilatometry result. The light micrograph shown in Fig. 2 indicates that the microstructure consists essentially of lath martensite. Transmission electron microscopy of the sample is shown in Fig. 3. The bright-field (BF) micrograph, Fig. 3a, shows the basic microstructure comprising of aligned laths martensite. The Fig. 3b shows the BF image of tangled dislocations within lath martensites and densely dispersed and very fine needle shaped $Ni_3(Ti,Mo)$ precipitates. Dislocation density for solution treated and aged sample is of the order of 10^{10} mm^{-2}. Higher magnification in Fig. 3c shows the BF image of $Ni_3(Ti,Mo)$ precipitates with average diameter of 1.6 nm and average length of 13 nm. Fig. 3d is the corresponding SAD pattern using the $[1\bar{1}3]$ zone axis and specific spots. Table 3 shows the chemical composition by EDS/TEM of matrix and $Ni_3(Ti,Mo)$ precipitate showing that the precipitate is rich in Mo, Ti and Ni. $Ni_3(Ti,Mo)$ precipitates at this aging temperature are the main strengthening phase since they are coherent with the martensitic matrix and provide effective resistance to the motion of dislocation during deformation. The average hardness increased from 331 ± 5 HV in the solution treated condition to 604 ± 18 HV after aging. The precipitation behavior and precipitation hardening mechanism at the aging temperature in this study are supported by other reports [2,4,6, 8,9,11–13].

Table 2
Transformation temperatures of the maraging 300 steel.

	P_s	P_f	A_s	A_f	M_s	M_f
Temperature (°C)	500	595	623	801	194	62

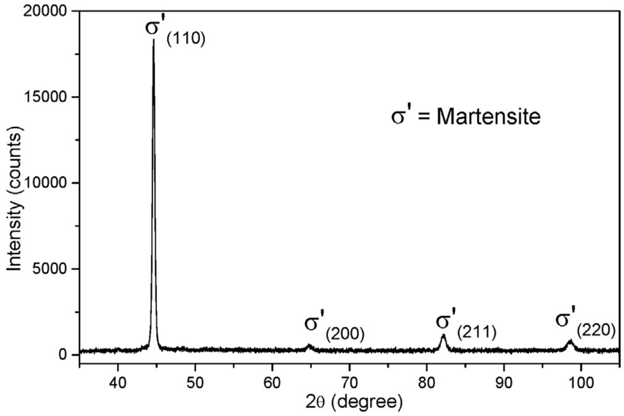

Fig. 1. XRD pattern for the solution treated and aged maraging 300 showing peaks corresponding to the martensite phase.

3.2. Creep behavior

Fig. 4 displays a representative creep curve of strain rate ($d\varepsilon/dt$) versus strain (ε) at 600 °C and 300 MPa. Maraging 300 steel exhibits typical creep curves consisting of well-defined primary (I), secondary (II) and, when submitted to the rupture, the tertiary (III) stage. A relatively short initial period in which the primary creep rate decreases is probably associated with hardening due to the accumulation of dislocations. However, most of the creep life is dominated by a constant creep rate that is thought to be associated with a stable dislocation configuration due to the recovery and hardening process [5]. The results from creep tests are summarized in Table 4, which shows the values of the secondary creep rate ($\dot{\varepsilon}_s$) and, when submitted to the rupture, the time to rupture (t_r), the percent elongation (EL) and percent reduction in area (RA). From the Table 4, it is possible to notice that $\dot{\varepsilon}_s$ increase as the temperature or stress increase and t_r presents reverse behavior accordingly. At 650 °C the increase of secondary creep rate is more pronounced since this temperature is higher than A_s.

For most metal and alloys, the relationship between the strain rate ($\dot{\varepsilon}_s$), stress (σ) and temperature (T) can be expressed by the power-law creep equation:

$$\dot{\varepsilon}_s = A\sigma^n exp(-Q_c/RT) \tag{1}$$

where Q_c is the activation energy for creep, A is a constant that depends on the microstructure, temperature and applied stress (σ), n is the

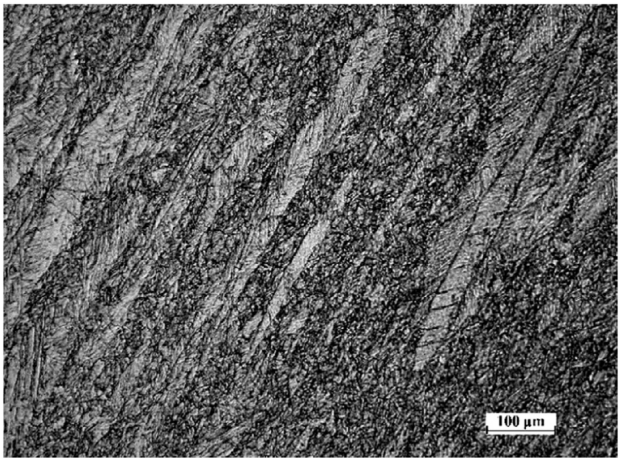

Fig. 2. Optical micrograph of the solution treated and aged maraging 300 showing lath martensites.