Microstructure and thermomechanical pretreatment effects on creep behaviour of helium-implanted DIN 1·4970 austenitic stainless steel

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Abstract. Microstructure investigations were carried out on unimplanted and 150 at.ppm helium implanted foil specimens of DIN 1·4970 austenitic stainless steel after various thermomechanical pretreatments. Creep tests were also carried out for both helium-implanted and unimplanted specimens at 700°C and 800°C. The strength, ductility and rupture time are correlated with the dislocation and precipitate distributions. Helium embrittlement can be reduced in these experiments when dispersive TiC precipitate distributions are produced by proper pretreatments or allowed to form during creep test.

Keywords. Helium implantation; austenitic stainless steel; thermomechanical pretreatments; helium embrittlement; radiation effects; nucleation sites; precipitate interfaces; helium embrittlement; creep tests.

1. Introduction

The subject of radiation effects in materials is a vital area of relevance of reactor engineering as well as a large field for basic materials research (Rodriguez *et al* 1984). During irradiation of metals with high energy neutrons, in addition to other effects—such as increases in yield strength and ultimate tensile strength and decreases in the rate of work hardening and the uniform and total elongation—helium is generated by nuclear $B^{10}(n, \alpha)Li^7$ reactions. Because of the very low solubility (Schroeder 1983) of He in metals it has a strong tendency to precipitate out into bubbles. These He bubbles then grow under the influence of high temperature, continuous He generation and stress. Preferred nucleation sites for the He bubbles are the inhomogeneities of the material like precipitate interfaces, dislocations and grain boundaries. The growing bubbles first weaken the grain boundaries and finally perforate them leading to embrittlement i.e. early brittle intergranular failure. The effect of helium on mechanical properties using foil samples into which helium was introduced by α -particle implantation has been studied by many workers (Kramer *et al* 1968, 1972; Smidth *et al* 1974; Matlock and Nix 1975; Matsumoto *et al* 1977). This simulation technique provides a fast and convenient way of introducing helium with a minimum of side-effects and it was found that a few hours of cyclotron irradiation results in uniform helium concentrations equal to those produced by several years of reactor exposure. It has been found that (Böhm and Heß 1970; Bloom *et al* 1976; Kesternich 1979; Kesternich and Rothaut 1981) Ti addition in austenitic steels reduces the deleterious effects of helium and this is due to the trapping of helium at TiC precipitates and that effective suppression of helium embrittlement can be achieved by using finely dispersed distributions of TiC precipitates in the matrix.

The present work is aimed at finding out the relationship between the microstructure and thermomechanical pretreatments of austenitic stainless steel by studying its creep

behaviour at 700°C and 800°C and by using transmission electron microscopy. The effect of helium implantation of the strength, ductility and rupture time of this material is also investigated.

2. Experimental

2.1 Sample preparations

The starting material for the samples consisted of SNR-300 Mark made of DIN 1-4970 stainless steel. Two fuel pin tubes (7.6 mm diameter and 0.5 mm wall thickness) were split longitudinally, cold-rolled and solution-annealed at 1100°C for 1 h under high vacuum 10^{-7} mm Hg. They were subsequently cold-rolled again to their final thickness of 105 μ m ($\approx$ 50% cold work, c.w.). From these strips creep specimens were sparked out by spark erosion. These specimens were of 11 mm gauge length, 200 μ width and 105 μ thickness. The final heat treatments given were (i) 1-4970—50% c.w. + 900°C for 20 h and (ii) 1-4970—50% c.w. + 1050°C for 1 h. The chemical composition as provided by the manufacturer (Sandvik Sweden) is given in table 1.

2.2 Helium implantation

Creep specimens were α -implanted at room temperature with homogenous He-deposition throughout the specimen thickness using 28 MeV α -beam of compact cyclotron at KFA-IFF Jülich, West Germany. Helium implantation rate was 50 at.ppm/hr.

2.3 Creep tests

Creep tests on specimens with 150 at.ppm helium and without were performed in high vacuum better than 10^{-6} torr at 700°C and 800°C test temperatures. The creep test facility consists of a high vacuum system, a tubular furnace and a creep machine made of stainless steel. The creep machine is built in a frame attached to a vacuum flange. The sample is clamped between two blocks. The lower block is connected to the frame by a fixed rod and the upper block to a spring. This spring loading system permits constant stress conditions to be achieved during the test. The sample

Table 1. Chemical analysis of DIN 1-4970 wt.%

C	Si	Mn	Cr	Ni	Mo	Ti	N
0.095	0.31	1.81	15.1	15.0	1.3	0.030	0.01
B	Co	Ta	Cu	V	Fe		
0.005	0.02	0.003	0.02	0.02	Bal.		

elongation was measured by linear distance voltage transducers and recorded continuously using two-channel chart recorder (model BBC Servogor 120-11). A Ni-Cr, Ni thermocouple is used to measure the temperature at the centre of the gauge length of the specimen. A control system is used to maintain constant test temperature with a maximum change $\pm 2^\circ\text{C}$. The elongation-time curves at constant test temperature and stress were recorded continuously for the tested specimens.

2.4 Microstructure investigations

The microstructures of creep specimens before and after helium implantation and creep tests were studied using a 120 kV Philips E M 400 and a transmission electron microscope (TEM). The TEM samples were thinned by electrolytic polishing in a dual jet machine using a 10% perchloric acid and 90% acetic acid solution operated at 11°C and 70 V. Pulsing the current at about 1 Hz with a duty cycle of 50% prevented overheating of the polishing cell. Further details of the experimental procedures were published earlier (Sagües *et al* 1978; Kesternich *et al* 1984).

3. Results

The typical microstructure of DIN 1.4970 stainless steel annealed at 1100°C for 1 h under a vacuum 10^{-7} mm Hg and before further cold work is shown in the transmission electron micrograph of figure 1. It shows TiC precipitates of 1000 Å diameter. After giving 50% cold work in the form of reduction in thickness, highly deformed bands are also observed (figure 2). The transmission electron micrographs of figure 3a, b show the effect of annealing at 900°C for 20 h under high vacuum. Figure 3a shows smaller TiC precipitates (≈ 200 Å diameter) and a somewhat inhomogenous particle distribution while figure 3b shows part of the grains containing dislocations network. Creep tests were carried out under high vacuum and under constant stress at test temperatures of 700 and $800^\circ\text{C} \pm 20^\circ\text{C}$ for the above annealed specimens at 900°C for 20 h for unimplanted and helium-implanted specimens. Figure 4 shows new dislocation networks formed during creep test at 700°C for unimplanted specimens while for 150 at.ppm He-implanted samples crept at 700°C , figure 5 shows He-bubbles at grain boundary and dislocations at a larger TiC particles.

Solution-treated samples at 1100°C for 1 h followed by 50% cold work and annealing at 1050°C for 1 h in vacuum show larger grains with larger and less dense TiC particles as seen in figure 6. Figure 7 for unimplanted specimens crept at 700°C shows that dislocation networks formed newly and dispersive TiC particles with 100 Å diameter were created. For 150 at.ppm helium-implanted specimens crept at 700°C and 800°C respectively, figure 8 shows He-bubbles at TiC particles which are at dislocations or at grain boundaries while figure 9 for He-implanted specimen crept at 800°C shows larger He-bubbles. Figures 10 and 11 show the time-to-rupture values as a function of applied stress at 700°C and 800°C for both unimplanted and He-implanted steel-50% c.w. + 900°C solution-annealed for 20 h and 50% c.w. + 1050°C solution-annealed for 1 h.

Figure 1. Large TiC precipitates in a specimen annealed at 1100°C for 1 h.

Figure 2. Highly deformed bands after 50% cold work.

Figure 3a. Microstructure of sample annealed for 20 h at 900°C.

Figure 3b. Dislocation networks within the grains.

Figure 4. Microstructure of unimplanted specimen shows new dislocation networks formed during creep at 700°C.

Figure 5. Helium bubbles at grain boundaries and dislocations at a large TiC precipitate in the centre of helium implanted specimen crept at 700°C.

Figure 6. Microstructure after 1100°C (1 h) + 50% c.w. + 1050°C (1 h) showing large grains with large and less dense TiC particles.

Figure 7. Dislocations networks newly formed and dispersive TiC particles with 100 Å diameter in unimplanted specimen crept at 700°C.

Figure 8. Microstructure of helium-implanted specimen crept at 700°C shows He-bubbles at TiC particles at dislocations or at grain boundaries.

Figure 9. Same as figure 8 but the specimen crept at 800°C shows larger helium bubbles.

4. Discussion

Figures 3–5 show that TiC precipitation occurs and the precipitate population is coarser and more inhomogeneously distributed. This microstructure reflects on the mechanical test data as shown in figure 10 and causes a decrease in precipitation strengthening combined with higher ductility. It also shows less effective suppression of He-embrittlement. From creep test data it was also found that elongation-to-fracture values at stress equals 200 MPa decreased from 45% to 18.5% for 150 at.ppm helium implanted specimens crept at 700°C.

For DIN 1-4970 type steel 50% c.w. and solution annealed at 1050°C for 1 h crept at 700°C dislocations are created during creep tests and TiC can precipitate by a

Figure 10. Creep rupture time as a function of stress at 700°C.

Figure 11. Creep rupture time as a function of stress at 800°C.

dislocation precipitation interaction (figures 6, 9). Mechanical data (figure 10) for 1 h at 1050°C show that creep rupture strength is not reduced after helium implantation within the accuracy of experimental data, while elongation-to-fracture values were independent of stress; the average value decreased from 26 to 17.2 after implantation and crept at stress equals MPa. The presently accepted mechanism for explaining high temperature helium embrittlement of He-implanted samples (Sagües *et al* 1978) is that helium-filled cavities located in grain boundaries weakens the boundaries of the material resulting in intergranular fracture. As against helium bubbles which weakens the grain boundaries it has a strengthening effect of the grains which shows up in many ways (Thompson 1975) e.g. higher yield stress which increases with increasing helium content and a creep rate which is lower than in unimplanted specimens. These macroscopic results are confirmed by microstructural investigations which often show dislocations pinned by helium bubbles within the grains (figures 3–9). Creep tests at 800°C (figure 11) for samples having the same thermomechanical pretreatments, do not show any peculiar behaviour other than those crept at 700°C.

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