Materials Science & Engineering A 621 (2015) 39–51

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Dynamic strain ageing, deformation, and fracture behavior of modified 9Cr–1Mo steel

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ARTICLE INFO

Article history:
Received 5 January 2014
Received in revised form
2 October 2014
Accepted 7 October 2014
Available online 15 October 2014

Keywords:
Modified 9Cr–1Mo steel
Serrated plastic flow
Internal friction
Dislocations
Dynamic strain ageing
Rosette fracture

ABSTRACT

Tensile behavior of modified 9Cr–1Mo steel was studied over the temperature range from room temperature (RT) to 600 °C at three strain rates 10^{-5}, 10^{-4} and 10^{-3} s^{-1}. Serrated plastic flow was observed from 250 to 350 °C at strain rate of 10^{-4} s^{-1}, signifying occurrence of dynamic strain ageing (DSA). Characteristic features of DSA such as plateau/peak in yield and tensile strength, minima in ductility, negative strain rate sensitivity, and peak in internal friction were also observed. Activation energy for DSA was found equivalent to that for diffusion of nitrogen, hence nitrogen was considered to cause DSA. Dislocation substructure in the region of DSA revealed dislocation debris, kinks, bowing of dislocations and high density of dislocations. Irrespective of temperature from 200 to 450 °C there was formation of dislocation cell structure; however, cell size in the region of DSA was smallest. Fractographic analysis showed rosette type fracture at RT resulting from longitudinal splitting. It was associated with decohesion of interface of carbide particles and prior austenite grain boundaries. Non uniform and shallow dimples were observed in fibrous zones in the region of DSA. Contours of dual shear lip zone were observed at elevated temperatures from 200 to 450 °C.

1. Introduction

Ferritic/martensitic 9Cr–1Mo steel is an important structural material in several applications like tubing in superheater, reheater and steam generator components in fossil-fired thermal and liquid metal cooled fast breeder reactors (LMFBR). It possesses lower coefficient of thermal expansion and higher resistance against stress corrosion cracking in water-steam systems and higher stability of microstructure on long exposures at elevated temperatures compared to austenitic stainless steel [1]. It has also emerged as an important candidate material for wrapper of metallic fuel in sodium cooled fast breeder reactor because of its high resistance against void swelling and high creep strength [2–4]. Modified 9Cr–1Mo steel (with small addition of Nb & V) possesses higher strength and better stability of microstructure at elevated temperatures. This has been attributed to enhanced stability of martensite lath from vanadium carbide precipitates along the lath interfaces and precipitation of fine $(Fe, Cr)_3C$ carbide particles within the martensite laths [5,6]. Baek et al. [7] found that modified 9Cr–1Mo steel maintained its microstructural stability at 600 °C during long-term ageing of 50,000 h, equivalent to in-service life of the sodium cooled fast breeder reactors (SFR) fuel cladding. One major issue with ferritic steels is transition in their behavior from ductile to brittle from fast neutron irradiation [2].

Both conventional [1,8–11] as well as the modified 9Cr–1Mo [12–17] steel have been reported to exhibit DSA over the temperature range from 225 to 420 °C. However, slight difference has been observed in temperature range of DSA. The phenomenon of DSA in the modified 9Cr–1Mo steel has been manifested by serrated plastic flow, plateau in yield and tensile strength, negative strain rate sensitivity and ductility minima [12–16]. DSA has been attributed to interaction of dislocations with carbon atoms in the conventional 9Cr–1Mo steel [1,8–10], and to interaction of dislocations with nitrogen atoms in the modified 9Cr–1Mo steel [12,13,16]. Keller et al. [15] have hypothesized occurrence of DSA to carbo-nitrides. DSA has also been characterized by internal friction in several structural materials like austenitic stainless steel [18], nickel base superalloy [19], and zirconium–hydrogen alloy [20]. Snoek peak in the region of DSA signifies enhanced interaction between dislocations and interstitial atoms. DSA has been found deleterious to tensile ductility, low cycle fatigue (LCF) life and creep resistance of structural components [21].

Different aspects of DSA in the modified 9Cr–1Mo steel have been studied by several investigators [12–16]. However, little

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http://dx.doi.org/10.1016/j.msea.2014.10.011
0921-5093/

attention has been paid to characterization of its deformation in the region of DSA and the fracture behavior.

The present study was undertaken to establish the range of temperature and strain rate for DSA in the modified 9Cr–1Mo steel and in particular to characterize dislocation substructures in the specimens tested at temperatures below the region of DSA, in the region of DSA (250–400 °C), and above the range of DSA. Also fracture behavior of this steel was characterized from room temperature (RT) to 600 °C.

2. Material and methods

The modified 9Cr–1Mo steel was procured from the Indira Gandhi Center for Atomic Research (IGCAR) Kalpakkam, India, in the form of plate of $24 \times 700 \times 700\ mm^3$ size in normalized and tempered (N&T) condition. Normalizing was carried out at 1060 °C for 1 h and cooling to RT in air. Normalized plates were tempered at 780 °C for 1 h and cooled in air to RT. The elemental composition of the steel is presented in Table 1. Optical microstructure in the N&T condition was examined following mechanical polishing and etching with Villela's Reagent (1 g picric acid, 5 ml conc. HCl & 100 ml ethyl alcohol). Blanks of $40 \times 15 \times 10\ mm^3$ size were sectioned from the as received plate and cylindrical tensile specimens were machined with gage length and diameter of 15 mm and 4.5 mm respectively. Gage section of the tensile specimens was mechanically polished to remove machining marks, if any. Tensile tests were performed on a 50 kN screw-driven Instron™ Universal Testing Machine (Model: 4206) over the temperature range from RT to 600 °C at nominal strain rates of 10^{-5}, 10^{-4} and $10^{-3}\ s^{-1}$. Stress–strain curves were established from the load-extension data and 0.2% offset yield stress was determined. In situ sample heating to desired test temperature was achieved by a split electric resistance heating furnace. Before starting the tensile tests, samples were soaked at test temperatures for 15 min to stabilize and homogenize the temperature of the specimen. Vickers microhardness of tensile tested samples was determined using microhardness tester (Model: Shimadzu HMV-AD). Internal friction measurements were made over the temperature range from RT to 450 °C using torsion pendulum, for the N&T, 5% & 10% cold worked conditions. Fracture surfaces of the tensile tested specimens were examined by field emission scanning electron microscope (FEI Quanta 200F). TEM foils were prepared from transverse sections of the tensile tested specimens by electro polishing in an electrolyte containing 10% perchloric acid in methanol, at 20 V and −35 °C, using a twinjet electropolisher (Fishione Model:110). TEM examination was carried out by FEI Tecnai $20G^2$, operated at 200 kV.

3. Results

3.1. Microstructure

Microstructure of the modified 9Cr–1Mo steel in N&T condition was examined by optical, SEM & TEM and is shown in Fig. 1. The optical micrograph (Fig. 1a) shows microstructure with prior austenite grain boundaries (PAGbs). These grain boundaries decorated with carbide precipitates are revealed more clearly in the SEM micrograph (Fig. 1b). Second phase particles along the PAGbs and lath boundaries may be seen in TEM micrograph (Fig. 1c). Fine second phase particles may be seen also within the laths. There were different morphologies of the second phase particles. Crystal structures of the matrix and precipitates were analyzed from their SAD patterns. Crystal structure of the matrix and precipitates were characterized as *bcc* and *fcc* respectively (Fig. 1d and e). Lattice parameters of different types of precipitates were calculated from their SAD patterns and compared with JCPDS. The cuboidal particles were identified as $M_{23}C_6$ carbide with lattice parameter $a \approx 10.65$ AU and the round precipitates as niobium carbide (NbC) with lattice parameter $a \approx 4.47$ AU. The rod like precipitates were identified as vanadium carbide (VC). These observations are consistent with earlier findings for this steel in N&T condition [5,6].

3.2. Tensile properties and microhardness

Tensile properties of the modified 9Cr–1Mo steel at different temperatures and strain rates are presented in Table 2. Stress–plastic strain curves corresponding to different temperatures, from RT to 600 °C, at the strain rate of $10^{-4}\ s^{-1}$, are shown in Fig. 2a. Serrated plastic flow may be seen in the curves corresponding to test temperatures from 250 to 350 °C at the strain rate of $10^{-4}\ s^{-1}$ (Fig. 2b), however, there was no serrated flow either at the higher strain rate of $10^{-3}\ s^{-1}$ or at the lower strain rate of $10^{-5}\ s^{-1}$. Serrations at low temperature were predominantly of type A; type A+B and E at 300 °C; and type A, E at 350 °C. Further, serrations in the stress–strain curves appeared almost from beginning of the plastic deformation and disappeared before the attainment of ultimate tensile strength over the temperature range from 250 to 350 °C. The amplitude of serrations increased with increase in strain and the critical strain for the onset of serration also increased with temperature.

The variation of 0.2% offset yield strength with temperature at strain rates of 10^{-5}, 10^{-4} & $10^{-3}\ s^{-1}$ is shown in Fig. 3a. The yield strength continuously decreased with rise in temperature at high strain rate of $10^{-3}\ s^{-1}$ and the rate of fall was relatively lower from 200 to 450 °C. There was a mild peak at 300 °C at the low strain rate of $10^{-5}\ s^{-1}$. On the other hand there was plateau in the plot from 200 to 300 °C and a pronounced peak at 350 °C at the strain rate of $10^{-4}\ s^{-1}$. The effect of temperature and strain rate on tensile strength is presented in Fig. 3b. Tensile strength continuously decreased with increase in temperature at the high stain rate of $10^{-3}\ s^{-1}$. There was flattening of the plot over the temperature range from 250 to 400 °C at the strain rate of $10^{-5}\ s^{-1}$. On the other hand there was distinct hump at 350 °C at the strain rate of $10^{-4}\ s^{-1}$.

The variation of plastic strain to fracture (e_{pf}) with temperature at different strain rates is shown in Fig. 4a. The e_{pf} may be seen to undergo minima in the temperature range 250–400 °C. Elongation minimum was found to shift towards the lower test temperature with decrease in strain rate. The uniform plastic strain (e_{pu}), plastic strain up to ultimate tensile strength, did not show minima rather showed slight increase over the above range of temperature. On the other hand necking plastic strain (e_{pn}), strain from maximum tensile stress to fracture stress, showed noticeable minima (Fig. 4b).

Table 1
Chemical composition of the modified 9Cr–1Mo steel.

Elements	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Nb	N	V	Fe
wt%	0.1	0.26	0.41	0.018	0.002	9.27	0.95	0.33	0.013	0.074	0.044	0.21	Balance