Materials Science & Engineering A 658 (2016) 123–134

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Study of the deformation and damage mechanisms of a 9Cr-ODS steel: Microstructure evolution and fracture characteristics

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ARTICLE INFO

Article history:
Received 1 December 2015
Received in revised form
28 January 2016
Accepted 29 January 2016
Available online 30 January 2016

Keywords:
ODS steel
Tempered martensite
Tensile properties
TEM
Deformation
Damage

ABSTRACT

In this study, the tensile properties of a tempered martensitic 9Cr oxide dispersion strengthened (ODS) steel are investigated. The tensile tests were performed in the temperature range of 25 °C to 800 °C at a nominal strain rate of $10^{-3}\,s^{-1}$. At room temperature, the material exhibited high yield strength and ultimate tensile strength of 929 and 1052 MPa, respectively. A decrease in strength was observed with increase in temperature down up to 156 MPa of ultimate tensile strength at 800 °C. The total elongation-to-failure that was 7.6% at 25 °C increased sharply with increase in temperature and reached a maximum of 38.4% at 700 °C. In order to compare the influence of heat treatment the tensile tests were also performed on specimens when in a ferritic state. Interestingly, at elevated temperatures both states presented a similar trend of strength and elongation. Transmission electron microscopy after deformation revealed a modification of the deformation mechanism with the temperature. The dislocation activity that was homogeneously distributed at room temperature was localized close to grain boundaries at elevated temperatures. A strong particle–dislocation interaction was observed at all testing temperatures. Orowan mechanism is supposed to govern particle–dislocation interaction at moderate temperatures. At elevated temperatures, an attractive particle–dislocation interaction phenomenon called interfacial pinning was identified. The additional microstructural evolution that includes reduced dislocation density, the transformation of lath structure into coarse equiaxed grains and the $M_{23}C_6$ carbide coarsening, resulted in a loss of strength at elevated temperatures. Fracture surface investigation at room temperature revealed intragranular fracture with dimples. As the temperature increased, the fracture surface formed with a shear-lip zone at the outer periphery and dimples that were larger and deeper in comparison to the ones formed at lower temperatures. These observations are associated with the increased ductility. A change in fracture mechanism from intragranular to intergranular fracture was observed at 650 °C. This change became fully apparent at 800 °C where it is associated with a reduced ductility. The change in damage mechanism is due to the modification of the deformation mechanism. In comparison to other commercial, as well as experimental, ODS steels, the material offers an excellent compromise between strength and ductility.

1. Introduction

The development of innovative nuclear reactors requires a breakthrough in the materials technology. The aggressive operating conditions for future nuclear plants including generation IV (GEN IV) fission and fusion reactors will be beyond those experienced in current nuclear power plants in term of higher temperature, higher neutron dose and compatibility with coolants. Nowadays, Fe–Cr binary alloys are the most promising base for fabricating reduced activation ferritic/martensitic (RAFM) and ferritic (RAF) steels for structural applications of current nuclear reactors [1]. However, these steels rapidly lose their strength above 650 °C, as at higher temperature re-crystallization, particle coarsening and dissolution lead to a reduced creep resistance [2,3]. Oxide dispersion strengthening appears to have the capability to improve high-temperature strength of the high-chromium steels, thus would allow in principle for a higher operating temperature which improves reactor's thermal efficiency [4–6]. The nano-sized oxide particles not only act as a barrier for dislocation motion, which controls the high-temperature tensile properties and creep resistance [7,8], but also act as an effective sink for irradiation-induced defects that provide higher irradiation resistance [9–12]. Considering associated advantages, there has been a resurgence in the fabrication and study of oxide dispersion strengthened (ODS)

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http://dx.doi.org/10.1016/j.msea.2016.01.109
0921-5093/

steels under various national and international projects for their further improvement, see for example [13].

The present work involves another effort towards the search of new cladding and blanket materials for nuclear reactors. The 9Cr-ODS steel was produced under the framework of the MATISSE FP7 project in which the characterization and the mechanical testing of several Fe–Cr alloys are found between the main topics. The good thermal conductivity, swelling resistance and low radiation damage accumulation (up to a dose of 150 displacement per atom (dpa)) of the tempered martensitic steel, are further enhanced by the presence of a fine dispersion of oxide particles. The work presents the results of high-temperature tensile tests and the microstructural analysis which was characterized before and after deformation using electron microscopy. Fracture surface investigations are performed in order to understand the damage mechanism.

2. Experimental procedure

2.1. Material

The 9Cr-ODS steel (designated herein as 9YWT-MATISSE) was developed and produced by CEA, France within the framework of MATISSE (Material's innovations for safe and sustainable nuclear), a project dealing with materials for transmutation technologies and advanced reactors. The pre-alloyed metal powder, obtained from Aubert & Duval, was mechanical alloyed with 0.25 wt% Y_2O_3 in a dry-type attrition ball mill. The milling was performed by Plansee in a hydrogen atmosphere for 24 h. Thereafter, the powder was sealed in a soft steel can and hot extruded at around 1100 °C. The air cool bars, now in a ferritic state, were semi-finished in the form of cylinders with 30 mm length and 4.2 mm diameter. These cylinders were then austenized at 1050 °C for 10 min, followed by quenching with helium gas where the cooling rate was carefully chosen (6–7 °C/s). Then, a tempering treatment was performed at 750 °C for 20 min, which was followed by the air cooling. The chemical composition of the steel is shown in Table 1.

2.2. Mechanical characterization

Although the focus of this research is on the investigation of a tempered martensitic 9YWT-MATISSE, tensile tests were also performed on specimens when in a ferritic state. The universal testing machine (Instron 4505) equipped with a high-temperature furnace and an extensometer was used for this purpose. Owing to the material quantity limitations small tensile test specimens with the gauge length of 13 mm and gauge diameter of 2 mm were used. All specimens were machined parallel to the extrusion direction. The tests were carried out in air at temperatures ranging from room temperature (25 °C) up to 800 °C with a nominal strain rate of $10^{-3}\ s^{-1}$. At elevated temperatures, the holding time prior to the test start was at least 30 min. The temperature was monitored by a (Ni–Cr)–Ni thermocouple placed in contact with the specimen and was controlled to within ± 2 °C.

2.3. Microstructural characterization

Microstructural characterization was carried out for tempered martensitic 9YWT-MATISSE in both as-received as well as in

Table 1
Chemical composition (H* in ppm, others in wt%) of as-received 9YWT-MATISSE.

Element	Cr	W	Ti	Si	Ni	Mn	C	H*	Y_2O_3	Fe
Amount	9.1	1.1	0.3	0.3	0.5	0.3	0.1	10	0.25	Bal.

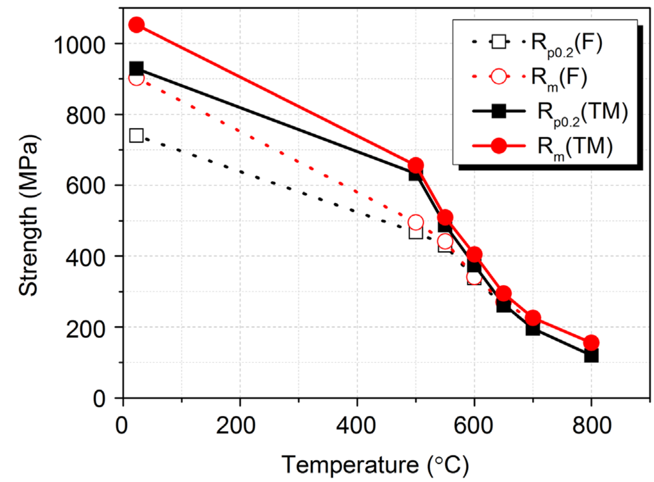

Fig. 1. Comparison of yield strength ($R_{p0.2}$) and tensile strength (R_m) of 9YWT-MATISSE in ferritic (F) and tempered martensitic (TM) states at various testing temperatures.

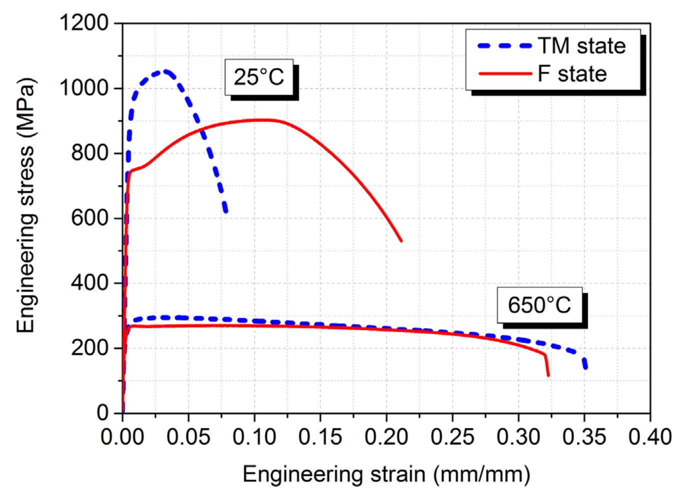

Fig. 2. Engineering stress–strain curves of 9YWT-MATISSE in both ferritic (F) and tempered martensitic (TM) states.

Table 2
Tensile data for tempered martensitic 9YWT-MATISSE at various temperatures.

Temperature (°C)	$R_{p0.2}$ (MPa)	R_m (MPa)	A_g (%)	A (%)	N_C (%)
25	929	1052	2.7	7.6	67.7
500	633	656	0.9	16.5	60.5
550	487	509	1.0	22.6	60.3
600	374	405	1.6	30.7	61.2
650	264	295	2.6	35.0	58.6
700	196	226	6.1	38.4	50.0
800	120	156	8.9	16.7	37.5

Table 3
Tensile data for ferritic 9YWT-MATISSE at various temperatures.

Temperature (°C)	$R_{p0.2}$ (MPa)	R_m (MPa)	A_g (%)	A (%)	N_C (%)
25	740	903	10.3	20.9	71.4
500	468	495	5.3	19.9	66.0
550	431	442	3.9	22.1	63.4
600	338	341	5.6	30.6	61.9
650	261	271	7.5	32	60.5
700	196	218	8.7	25.6	52.7