

## Hot tensile properties of 9Cr-2WVTa reduced-activation ferritic/martensitic steel

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### Abstract

Reduced-activation ferritic/martensitic steels have been developed by replacing molybdenum by tungsten and niobium by tantalum in commercial elevated-temperature Cr–Mo steels to reduce half-life of radioactive isotopes. The objective of this study was to investigate tensile properties of a reduced-activation ferritic/martensitic steel (9Cr-2WVTa) in normalized-tempered (NT) and quenched-tempered (QT) conditions over the temperature range of room temperature to 600 °C. The increase in temperature led to reduction in yield and ultimate tensile strengths in both conditions. Furthermore, the strengths of NT samples were similar to those of QT samples. Variations of elongation and reduction of area with temperature revealed that both properties initially decreased up to 400 °C but thereafter increased as the temperature increased. Since fracture surfaces of all tensile samples were dominated by dimples, it was presumed that the decrease in elongation and reduction of area in the intermediate temperature range was related to Dynamic Strain Ageing (DSA). In addition, NT samples exhibited superior elongation at all temperatures except 500 °C.

**Keywords:** Ferritic/Martensitic Steel; Reduced Activation; Dynamic Strain Ageing.

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## 1. Introduction

Development of reduced-activation ferritic/martensitic steels for first wall and blanket structures of future fusion reactors began in the mid-1980s. The objective was that when reduced-activation steel was irradiated in a fusion neutron environment, nuclear transmutation of alloying elements in the steel produces relatively short-lived radioactive isotopes. This rapid radioactivity decay allows the reactor components constructed from reduced-activation steels to be disposed of by shallow land burial when removed from service, instead of by the much more expensive deep geologic storage. According to the early investigations, reduced-activation status could be achieved if the typical alloying elements Ni, Mo, Nb, Cu, and N were eliminated or restricted [1-5].

Before the development of reduced-activation steels, commercial elevated-

temperature Cr–Mo steels were considered for fusion applications. The first commercial steels considered in the international fusion materials programs beginning in the late 1970s were tempered martensitic steels that had been investigated in the previous decade in the international fission fast breeder reactor programs [1, 3]. These steels, which were developed by the steel industry for the conventional power-generation and petrochemical industries, had a range of compositions that included 2.25–12% Cr, 1–2% Mo, 0–0.3% V, 0–0.5% W, 0–0.5% Ni, 0–0.2% Nb, 0.1–0.15% C, 0–0.07% N (all compositions are in wt.%). Steels chosen for the international fusion programs were EM-12 in France, FV448 in the United Kingdom, DIN 1.4914 in Germany, JFMS in Japan, and Sandvik HT9, modified 9Cr–Mo, and  $2\frac{1}{4}$ Cr–1Mo in the United States (Table 1).

**Table 1. Compositions of some commercial steels (wt %) [3]**

Steel	C	Cr	Mo	V	Nb	W	Mn	Si	Ni	N	B
Mod 9Cr-1Mo	0.086	8.44	0.89	0.24	0.08		0.37	0.16	0.11	0.054	-
Sandvik HT9	0.21	12.11	1.03	0.33		0.53	0.50	0.21	0.58	0.004	-
EM12	0.086	9.58	1.91	0.28	0.41		0.92	0.37	0.12		-
FV448	0.10	10.7	0.60	0.14	0.26		0.86	0.46	0.65	0.050	-
DIN 1.4914	0.15	11.0	0.50	0.30	0.25		0.40	0.50	0.75	0.030	-
E911	0.11	9.0	1.0	0.20	0.08	1.0	0.45	0.20	0.20	0.06	-
NF616	0.07	9.0	0.50	0.20	0.05	1.8	0.45	0.06		0.06	0.005

Reduced-activation steels were developed in Europe, Japan, and the United States. Generally, the development strategy involved replacing molybdenum by tungsten, niobium by tantalum, and nickel by manganese in the commercial steels. Finally, the development programs

settled on 7–9% Cr steels including F82H, EUROFER 97, and ORNL 9Cr–2WVTa [2-7] developed in Japan, Europe, and the United States, respectively. Nominal compositions of these reduced-activation steels are given in Table 2

**Table2. Compositions of reduced-activation steels (wt.%) [3]**

Steel	C	Cr	V	Ta	W	Mn	Si	Ni	N	B
F82H	0.093	7.50	0.14	0.02	2.01	0.17	0.16	0.02	0.006	0.003
EUROFER 97	0.10	8.82	0.19	0.07	1.10	0.37	0.01	0.02	0.021	0.005
9Cr-2WVTa	0.11	8.90	0.23	0.06	2.01	0.44	0.21	<0.01	0.022	-

The objective of present investigation was to evaluate the tensile properties of reduced-activation ferritic/martensitic steel in normalized-tempered (NT) and quenched-tempered (QT) conditions in temperature range of 25 to 600°C. For this purpose, ORNL 9Cr-2WVTa was selected since among the considered steels it is the most recent alloy and exhibits superior properties.

## 2. Experimental procedure

A 12 kg capacity, 125 kW, 3 kHz basic lined induction furnace under protective atmosphere (Ar) was used for preparing the 9Cr-2WVTa heat. The melt were poured into a metal mould in the shape of strip with dimensions of 25×80×300mm<sup>3</sup>. The chemical composition of the heat is given in Table 3.

**Table3. Chemical composition of the heat (wt %)**

C	Cr	V	Ta	W	Mn	Si	Ni	N	B
0.11	8.90	0.23	0.06	2.01	0.44	0.21	<0.01	0.022	-

The compositional inhomogeneities due to the as-cast structure were eliminated by hot rolling with a soaking time and temperature of 5 h and 1100 °C, respectively. As a result, the thickness of the slab was reduced from 25 to 15 mm.

Normalizing-tempering (NT) and quenching-tempering (QT) heat treatments with the same austenitizing and tempering conditions were considered in this study. After austenitizing at 1050 °C for 1 h, NT and QT specimens were cooled in air and water, respectively. Tempering was carried out at 700 °C for 2 h.

Tensile tests were conducted at room temperature, 200, 300, 400, 500 and 600 °C on specimens with a reduced gage section of 32.0 mm long by 6.25 mm wide by 3.0 mm thick. All specimens were machined with gage length perpendicular to the rolling direction. The tests were made in a 120 kN-capacity universal testing machine at a cross-head speed of 1 mm/min (at a nominal strain rate of 5×10<sup>-4</sup> s<sup>-1</sup>). The results reported in this paper

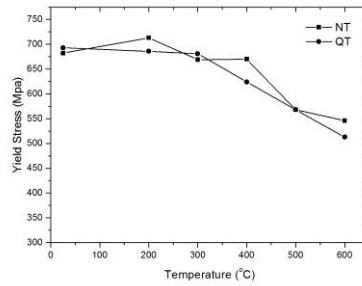
include 0.2% yield strength, ultimate tensile strength (UTS), total elongation, and reduction of area; and are the average of two tests.

Optical microscope and Scanning Electron Microscope (SEM) were used for observing the general microstructures and fractography. After standard metallographic techniques, the samples were etched by Marble's etching solution including 20 cc HCl, 4 gr CuSO<sub>4</sub> and 20 cc H<sub>2</sub>O.

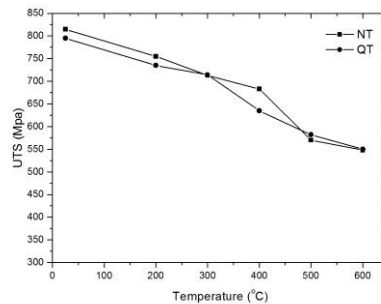
## 3. Results and discussion

### 3.1. Tensile Properties

Figures 1 and 2 show yield strength and UTS change in QT and NT specimens with temperature. The figures reveal that the strength of alloy in NT and QT conditions are approximately equal. Although at two temperatures (200 and 400 °C) the strength of NT specimens are higher than QT samples, their difference are less than 50 MPa.



**Fig1. Variation of yield strengths with temperature**



**Fig2. Variation of ultimate tensile strengths with temperature**

As expected, increasing the temperature has led to deterioration of yield strength and UTS of both QT and NT samples. Previous studies have revealed that two effective strengthening mechanisms in these alloys consist of solid solution and precipitation hardening. The presence of tungsten and chromium is the major cause of solid solution hardening. Precipitation hardening stems from attendance of strong carbide and nitride formers including Cr, W, V, and Ta in the chemical composition. While Cr and W only form carbide precipitates of  $Cr_{23}C_6$  and WC, respectively, Ta and V form carbonitrides [8-14]. Based on the solubility products of the precipitates, increasing the test temperature cannot reduce contribution of precipitates in the strengthening; since even  $Cr_{23}C_6$  which is the least stable precipitate in this alloy, is not affected in

the time length of tensile test in the considered temperature range. Hence, the observed decrease in the strengths can be related to the weakening of solid solution hardening due to the reduction in Peierls-Nabarro stress of matrix.

Elongation and reduction of area variations with test temperature are shown in Figures 3 and 4. As can be seen, both properties initially decrease up to 400 °C but thereafter increase as the temperature increases. Although Klueh et al [4, 15] have reported the same behavior in the similar alloys, they offered no reason to illuminate the microstructural stimulus of this behavior. At first glance, secondary hardening may be thought as the reason for decrease in ductility. However, Figures 1 and 2 show that the strengths have also been reduced with temperature.

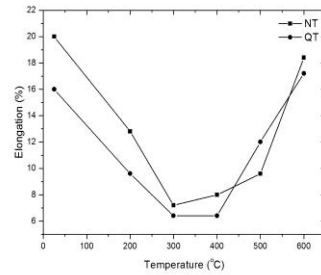


Fig3. Variation of elongations with temperature

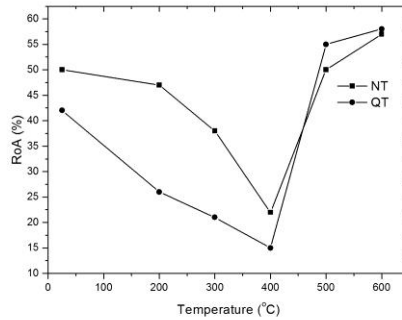


Fig4. Variation of reduction of areas with temperature

Reed-Hill and Abbaschian [16] have suggested that competition between transgranular and intergranular fracture plays a pivotal role in the variation of ductility with temperature. The reduction in ductility at intermediate temperatures stems from grain boundary fracture. Rhines and Wray [17] have designated this phenomenon as “*intermediate temperature ductility minimum*”. However, fractographies conducted on the fractured samples revealed that fracture surfaces were dominated by ductile rupture. Figures 5(a) and (b) shows presence of dimples on the fracture surfaces of tensile NT samples

tested at 400 and 600 °C, respectively. On the other hand, Choudhary et al [18-19] have attributed the reduction in ductility in intermediate temperatures to “*Dynamic Strain Ageing*” (DSA). They suggested that diffusion of interstitial solute such as carbon in 9Cr-1Mo ferritic steels led to DSA. It has been reported that DSA causes an increased rate of dislocation multiplication and delay in recovery of dislocation structures for many alloy systems [20]. Also, the increased tendency toward uniform distribution rather than cell structures has been reported in the DSA regime for different alloy systems [21].

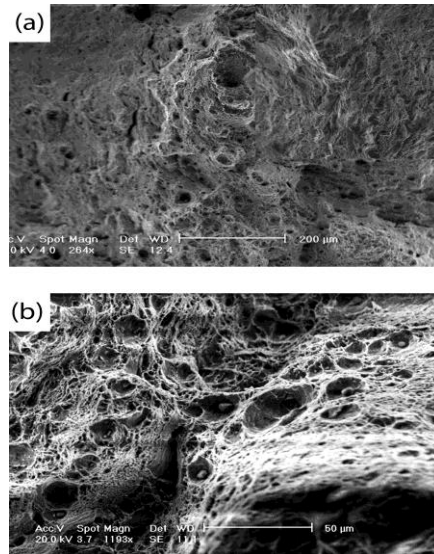


Fig5. Fracture surface of tensile specimen tested at: (a) 400 and (b) 600 °C

Another point inferred from Figures 3 and 4 is the superior ductility of NT samples in all temperatures except 500 °C.

Based on optical microscopy shown in Figure 6, both heat treatment cycles have resulted in similar microstructures consisting of extremely small and uniformly dispersed precipitates embedded within a **ferritic** matrix due to the tempering of martensite. Hence, the observed discrepancy in ductility can be attributed to characteristics of the

precipitates, including their size and distribution, formed in the microstructure of this alloy after each heat treatment cycle. Nevertheless, since these precipitates are very fine and cannot be observed by optical and scanning electron microscopes, verification of this suggestion needs a comprehensive transmission electron microscopy which can be the subject of another investigation.

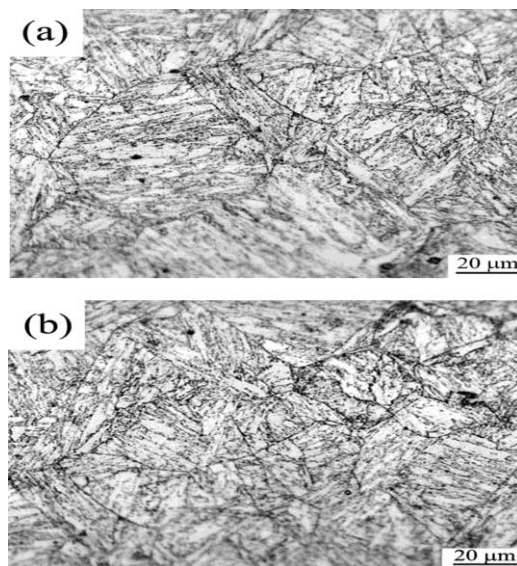


Fig6. Optical microstructure of: (a) NT sample and (b) QT sample

#### 4. Conclusions

Tensile properties of 9Cr-2WVTa ferritic/martensitic steel in normalized-tempered (NT) and quenched-tempered (QT) conditions have been studied over the temperature range of room temperature to 600 °C. observations and conclusions can be summarized as follows:

1. NT and QT samples showed similar strengths and their yield and ultimate tensile strengths decreased with increasing temperature. The decrease in the strengths can be related to the weakening of solid solution hardening due to the reduction in Peierls-Nabarro stress of matrix.

2. NT samples exhibited superior elongation and reduction of area in all temperatures except 500 °C. In addition, variations of elongation and reduction of area with temperature for both conditions revealed that both properties initially decreased up to 400 °C but thereafter increased as the temperature increased. It is presumed that this behavior relates to dynamic strain ageing.

3. Both heat treatment cycles have resulted in similar tempered martensite microstructures consisting of extremely small and uniformly dispersed precipitates embedded within a **ferritic** matrix. Nevertheless; since different kinds of carbides and carbonitrides present in the microstructures, the characterization of fine-scale precipitates including their composition, size, and distribution needs a comprehensive transmission electron microscopy which can be the subject of another investigation.

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