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# Microstructure modification and mechanical property improvement of reduced activation ferritic/martensitic steel by severe plastic deformation

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ABSTRACT

9Cr2WVTa ferritic/martensitic steel was processed by cold-swaging and post-annealing in order to investigate the microstructure evolution and its effect on the mechanical properties. Optical microscopy, scanning electron microscopy with electron backscatter diffraction, and transmission electron microscopy were utilized for the microstructural characterization during the cold-swaging and post-annealing process, and the mechanical properties were determined by microhardness, tensile and creep tests. The results revealed that, nearly equiaxed ultrafine grains with the average size of ~ 330 nm and fine dispersed carbides with the average length of ~ 50 nm, were obtained after cold-swaging and post-annealing. In comparison with the initial normalized-tempered sample, effective grain boundary strengthening and dispersion strengthening in the post-annealed sample enhanced the strength at room and elevated temperature. The presence of fine dispersed carbides could retard the initiation and propagation of the cracks, leading to a better ductility in the post-annealed sample. During creep test, fine dispersed carbides in the post-annealed sample effectively slowed down grain boundary migration. The pinned grain boundaries and fine dispersed carbides in the post-annealed sample acted as obstacles to the motion of mobile dislocations, which resulted in the enhanced creep properties.

## 1. Introduction

In comparison with the world-wide operating commercial nuclear reactors, higher operating temperatures and higher neutron doses are expected in the advanced nuclear systems, which pose significant challenges to the material selection. Reduced activation ferritic/martensitic (RAFM) steels, such as CLAM, F82H, Eurofer-97 and 9Cr2WVTa, are considered as an important category of structural materials for the advanced Generation VI fission and fusion energy systems, due to their superior thermo-physical properties and higher resistance to irradiation swelling in comparison with austenitic stainless steels [1,2]. However, significant changes in the microstructure would occur in the RAFM steels after long-term service at high temperature up to 823 K, such as martensitic lath widening, recovery of tempered lath martensite structure, coarsening of $M_{23}C_6$ carbides, and precipitation of Laves phase, which would impair the strength, toughness and creep properties [3–6]. Therefore, great efforts have been focused on improving the high temperature mechanical properties of RAFM steels.

After decades of research, high temperature mechanical properties of RAFM steels have been substantially improved through optimization of alloy compositions [7–11]. For example, addition of ~ 1.5 wt% W to RAFM steels would provide enough solid solution strengthening, and decrease the possibility of Laves phase precipitation [7]. The Ta content in the range of 0.06–0.15 wt% would promote the formation of small TaC carbides and refine the prior-austenite grain size [9]. In addition, the oxide dispersion strengthened (ODS) ferritic steels, especially those hardened by dispersed nano-sized oxide particles can further improve the high temperature mechanical properties [12–14]. These dispersed nano-sized oxide particles not only serve as the obstacles to dislocation motion, but also act as trapping sites for point defects to retard irradiation swelling [14,15]. However, ODS steels are produced by expensive mechanical-alloying and powder metallurgy techniques, which is the main limitation for its wide application [16]. Similarly, an increased density of fine M(C, N) precipitates through thermomechanical treatment (TMT) is also proposed to improve the high temperature mechanical properties, because TMT could significantly increase the nucleation sites for the fine M(C, N) precipitates [17,18]. According to the above approaches, the introduction of fine dispersed precipitates is an effective method to improve the high temperature mechanical properties of RAFM steels.

Recently, it is reported that the grain size, carbide size and distribution in the ferritic/martensitic steels could be adjusted by severe

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plastic deformation (SPD) such as surface mechanical attrition treatment (SMAT) [19,20], equal channel angular pressing (ECAP) [21,22] and cold-swaging [23]. Wang et.al [19] found that ferrite grains/cells and $M_{23}C_6$ carbides in P92 steel could be separately refined to the size of ~ 35 and ~ 20 nm after SMAT. However, SMAT is a SPD method to synthesize a nanostructured surface layer on metallic materials and cannot fabricate samples with large dimensions to investigate the mechanical properties. By contrast, ECAP and cold-swaging are considered to be an effective technique to fabricate the bulk ultrafine-grained materials. Song et al. [21] reported that ultrafine grains with the size of ~ 304 nm and fine carbides with the size of ~ 56 nm were obtained in T91 steel after ECAP at 700 °C, which resulted in an increased room temperature strength but a decrease in ductility. Nevertheless, our recent investigation found that ultrafine-grained 9Cr2WVTa steel with a uniform fine dispersion of carbides was fabricated through cold-swaging and post-annealing, leading to higher strength and better ductility than the coarse-grained counterpart at room temperature [23]. Until now, most efforts are focused on the room temperature mechanical properties of ultrafine-grained steel with fine dispersed carbides fabricated by SPD, while less attention has been paid to its high temperature mechanical properties. Furthermore, although ultrafine-grained/nano-grained ferritic/martensitic steels fabricated by SPD were also reported to exhibit enhanced irradiation tolerance [24,25] and high temperature oxidation resistance [20,26,27] compared with the coarse-grained materials, stability and mechanical properties of ultrafine-grained/nanograined ferritic/martensitic steels at high temperature were needed to be carefully investigated in view of the service environment in the future advanced nuclear reactor. Hence, the aim of this work is to fabricate ultrafine-grained structure with fine dispersed carbides and investigate its high temperature mechanical properties.

In the present work, the 9Cr2WVTa steel was subjected to various strains by multi-pass rotary-swaging to study the microstructure evolution as a function of cumulative strain. Subsequently, an annealing treatment was studied and applied to optimize the microstructure. Meanwhile, tensile and creep-rupture properties of the ultrafine-grained ferritic/martensitic steels processed by cold-swaging and post-annealing were measured and compared with those of the coarse-grained ferritic/martensitic steels.

## 2. Experimental

### 2.1. Material

The chemical composition of 9Cr2WVTa steel used in this work was Fe-0.14C-8.78Cr-1.74W-0.24V-0.05Ta-0.47Mn-0.03Si-0.006P-0.004S (in wt%). The ingot was produced by vacuum induction melting, followed by forging into two 30 mm thick plates after holding at 1373 K for 2 h. The plate was normalized at 1323 K for 1 h and water quenched, then tempered at 1023 K for 2 h followed by air cooling, which was denoted as the N&T sample. Cylindrical samples with the size of ~ 22 mm in diameter and ~ 190 mm in length were cut from this plate by using electrospark discharge method. After grinding the surface, cylindrical samples were separately cold-swaged at room temperature to a diameter of ~ 13.3, ~ 9.2 and ~ 5.2 mm, with a total reduction in area (R) of ~ 63%, ~ 83% and ~ 94%. The cumulative strain $\varepsilon = -\ln(1 - R)$ for the above cold-swaged samples was 0.99, 1.78 and 2.81, respectively. After cold-swaging, the cold-swaged sample with the cumulative strain of 2.81 was annealed at 873–1023 K for 15–120 min to modify the microstructure.

### 2.2. Mechanical testing

The Vickers microhardness (HV) of the samples was measured by Buehler model micromet 5103 microindentation tester under a load of 980 g for 15 s. The microhardness values were taken from the cross-section of the cold-swaged sample and the post-annealed sample. And the final microhardness values in the figure were all the average of 6–8 points. Tensile and creep-rupture specimens were cut from the samples by electrospark discharge method, then machined with a gauge section of Φ3 mm × 18 mm. Tensile tests were carried out on a MTS-880 mechanical testing machine at room temperature with the tensile rate of 2 mm/min, and at 823–923 K with the tensile rate of 1 mm/min. Each data was an average of three tensile tested values. Uniaxial creep-rupture tests were carried out at 823 K with the applied stress of 230, 250 and 270 MPa on a RDL-50 creep test machine. One or two creep specimens were tested at each condition.

### 2.3. Characterization analysis

Microstructures of the samples before and after tensile tests were characterized by optical microscopy and FEI Inspect F50 scanning electron microscopy (SEM). Thin foils for transmission electron microscopy (TEM) observations were cut from the cross section of the cold-swaged samples and prepared by twin-jet electro-chemical polishing in a 10 vol% perchlorate alcohol solution at 20 V and −20 °C, then examined on Technai G220 TEM operated at 200 kV. The martensitic lath width and subgrain/cell/grain size were measured by linear intercept method in vast TEM images. The carbide size along the long axis was averaged from a few hundred carbides selected randomly from TEM images. For electron backscatter diffraction (EBSD) test, the samples were mechanical polished, then polished using ion milling technique on a Leica RES101 machine to remove the deformation layer on the top surface. EBSD maps were obtained using a Zeiss FE-SEM with the step size of 0.01 μm and EBSD data were analyzed with HKL Channel-5 software.

## 3. Results

### 3.1. Microhardness evolution

The cumulative strain dependent microhardness of the cold-swaged 9Cr2WVTa steel is shown in Fig. 1a. A significant increase in the microhardness is observed with an increase in the cumulative strain. The microhardness of the cold-swaged sample with the cumulative strain of 2.81 reaches ~ 348 HV, which is ~ 1.5 times as much as that of the N&T sample. The effect of annealing temperature and time on the microhardness of the cold-swaged sample is shown in Fig. 1b. It is found that the microhardness of the cold-swaged sample decreases as the annealing temperature and time increase. The microhardness drops quickly at the early annealing time of ~ 15 min and tends to a saturation value with prolonged annealing time. In addition, the microhardness of the cold-swaged sample after annealing at 1023 K for 2 h is ~ 197 HV, which is lower than that of the N&T sample (~ 224 HV), as shown in Fig. 1a and b.

### 3.2. Microstructure evolution

Fig. 2 shows the microstructure of 9Cr2WVTa steel before cold-swaging. It can be found that a typical tempered martensitic structure is observed in the N&T sample and the average prior austenite grain size is ~ 35 μm (Fig. 2a). High-magnification SEM image shows that numerous carbides precipitate on the prior austenite grain boundaries and martensitic lath boundaries, as shown in Fig. 2b. TEM observations show that the average width of martensitic laths is ~ 350 nm, and a high density of dislocations is present in the martensite laths. Meanwhile, numerous rod-like carbides with the average length of ~ 170 nm precipitate along the martensitic lath boundaries (Fig. 2c). These carbides are typical of $M_{23}C_6$-type carbide according to the corresponding selected area electron diffraction (SAED) pattern (see the inset in Fig. 2c).

Fig. 3 shows the microstructure of 9Cr2WVTa steel after cold-swaging with various cumulative strains. When the cumulative strain is