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Investigation of ion irradiation hardening behaviors of tempered and long-term thermal aged T92 steel

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ABSTRACT

9Cr ferritic/martensitic steels are promising materials for in-core components in advanced Gen-IV reactors. In these applications, their long-term microstructural stability under thermal exposure and resistance to neutron irradiation are essential. Tempered (unaged) and long-term thermal aged T92 samples were used to evaluate the effects of thermal aging and ion irradiation on the microstructure and micromechanical properties of the steel. Both the tempered and aged samples were irradiated with 3 MeV Fe^{11+} ions to 0.25, 0.50, 1.00 and 5.00 dpa at room temperature. Using the nanoindentation technique, the irradiation hardening behaviors of T92 steel were investigated. The irradiation hardening effect was observed in both the tempered and aged T92 samples. To eliminate the soft substrate effect, the critical indentation depth was determined using the ratio of the average hardness of irradiated and unirradiated samples at the same depth. Under the same irradiation conditions, the macroscopic hardness values of the aged T92 samples after irradiation were lower than those of the tempered samples. The irradiation hardening effect was more significant in the aged T92 due to the decreased dislocation density and the coarsened martensitic lath after long-term thermal aging.

1. Introduction

As one type of Generation IV reactors, the sodium-cooled fast reactor (SFR) has significant advantages over Generation III reactors in sustainability, economics, safety and reliability. In Generation IV fast neutron reactors, swelling of alloys for fuel cladding leads to geometrical changes and loss of mechanical strength, which can threaten the safety and lower the efficiency of nuclear fuel. Compared to austenitic stainless steels such as 316L and 15/15Ti (a Ti-modified austenitic steel), 9Cr ferritic/martensitic (F/M) steels exhibit a remarkably lower irradiation swelling rate, even at high damage levels [1]. Thus, 9Cr F/M steels are considered the primary candidate materials for in-core (cladding and ducts) components in SFRs [2]. These components are exposed to higher operating temperatures and higher neutron irradiation than those in Generation III reactors [1–5]. Therefore, irradiation-induced degradation of the microstructure and mechanical properties in 9Cr F/M steels has attracted intensive research interest recently.

T92 F/M steel was developed for fossil fuel thermal power plants in the 1980s, with a good combination of mechanical, thermal, and corrosion properties at elevated temperatures. Since some of the components are expected to serve for decades without replacement, degradation resistance during long-term service is one of the most critical issues. Studies on microstructure and mechanical properties were performed on T92 steels in both tempered [6–8] and long-term thermal aged [9–12] states. Different families of precipitates and broken martensitic laths were observed after aging, which have detrimental effects on their mechanical performance. The irradiation resistance of T92 steel is another import issue. Heavy ion irradiation is widely used to simulate neutron irradiation and study radiation damage. Particularly, self-ion irradiation, e.g., Fe ion for steels, has the following advantages: rapid damage production, no induced-radioactivity, and easy control of the irradiation conditions [13]. Despite the various ion types and energy ranges used, the penetration depth of the ions is often limited to 10 μm or less. Therefore, characterization of the evolution

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in the microstructure and mechanical properties induced by ion-irradiation requires testing and analysis methods on the nano or microscale, such as nanoindentation and transmission electron microscopy (TEM).

High-energy displacement cascades produced by irradiation induce the formation of various sizes of vacancies and interstitial defect clusters in materials. This process plays a vital role in the mechanical properties of the irradiated materials. The irradiation-induced microstructural evolution in Fe-Cr alloys has been extensively studied previously [14–16]. Defect clusters and dislocation loops were observed in the irradiated samples. Cem et al. [14] performed in situ irradiation on NF616 (P92) and 9Cr model alloy with 1 MeV Kr ions between 50 K and 673 K. They found that defect cluster density increased with the dose and became saturated in both alloys. The strong interaction between the dislocation loops and the preexisting defects was observed in T91 F/M steel between 573 and 773 K. This interaction was different at different temperatures [15]. Irradiation-induced dislocation loops and defect clusters led to hardening behavior in the materials. Nanoindentation has been widely used to evaluate the hardening effects resulting from ion irradiation [13,17–20]. Stoller and Rice [17] carried out nanoindentation experiments on a series of Fe-based alloys; they successfully established the relationship between the evolution of irradiation-induced defects and irradiation hardening. Heintze et al. [19] investigated the influence of the irradiation dose, Cr-level, and irradiation temperature on Fe-ion irradiation hardening in Fe-Cr alloys; they found that the hardness of 9Cr model steels exposed to 1 dpa increased significantly at room temperature and 300 °C, but not at 500 °C. Previous studies [19,20] also indicated that the hardness of 9Cr steels increased with the damage dose but became saturated over certain damage doses.

9Cr steels are considered to be used as structural materials for the in-core and out-of-core components in SFRs, where the input temperature and output temperature of the liquid sodium are approximately 370 °C and 550 °C [21], respectively. In the present work, thermal aging was conducted at 650 °C to accelerate the aging process for up to 15,000 h. The ion irradiation hardening behaviors of both the tempered and long-term thermal aged T92 samples were investigated by the nanoindentation technique. The microstructural evolution during thermal aging as well as the irradiation-induced defects was studied by TEM.

2. Material and experimental procedure

The chemical composition of the investigated T92 F/M steel is listed in Table 1. The hot-rolled T92 tubes were obtained from Baoshan Iron & Steel, Shanghai, China, in the normalized and tempered condition (i.e., austenitized at 1080 °C for 20 min followed by air cooling and then tempered at 780 °C for 150 min). The as-received tubes were Φ 50.80 mm × 11.50 mm × 6.00 m in size. All of the samples were cut from the tube along the same direction to eliminate the influence of the anisotropic microstructure.

To evaluate the effect of long-term thermal aging on the ion irradiation behaviors, some of the tempered T92 F/M samples were exposed to 650 °C for 15,000 h in air. The microstructures and micromechanical properties of both the tempered (unaged state) and the aged T92 samples were investigated before and after irradiation.

Specimens with a dimension of 5 × 10 × 5 mm were cut from the tempered and aged T92 tubes for the microstructure study as well as ion irradiation experiments. The microstructures of the steels at different states were characterized by an optical microscope (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM). All the specimens were mechanically polished: first, they were ground with silicon carbide sandpapers down to grade P2000, and then they were polished using diamond solutions and colloidal silica suspension with a grain size of 0.5 μm. OM specimens were etched using a solution of 5 g ferric chloride +100 ml hydrochloric acid +100 ml ethanol +100 ml deionized water. After mechanical polishing, the SEM and ion-irradiation specimens were electrochemically polished using a solution of 20% perchloric acid in ethanol to remove the mechanically deformed layer. The TEM specimens were first cut as discs of 3 mm in diameter then mechanically ground to a thickness of 50 μm, and finally electrochemically polished in a solution of 10% perchloric acid +90% ethanol using a twin-jet polishing apparatus at −30 °C.

Fig. 1 (a) and (b) show the typical lath martensite structure. The precipitates with different sizes are distributed mainly along the prior austenite grain boundaries and martensitic lath boundaries in the tempered T92.

Ion irradiation experiments were conducted on the 320 kV multidiscipline research platform for highly charged ions at the Institute of Modern Physics (IMP) in Lanzhou, China. Prior to irradiation, the surfaces to be irradiated were mechanically polished, and then finished by electro-polishing to remove any deformation layer. All of the samples were carefully checked in SEM to ensure a good quality surface. The samples were irradiated with 3.0 MeV Fe^{11+} ions at a beam flux of 3.34×10^{15} ions/m^2/s at room temperature. For TEM observation of the irradiated samples, the specimens were milled perpendicular to the irradiated surface using a focused ion beam (FIB) system. The procedure was finished by polishing the TEM specimen at a low accelerating voltage of 10 kV. A detailed description can be found in Ref. [22].

The radiation damage was calculated by the SRIM-2008 [23] Monte Carlo simulation code [24], using the quick damage mode with the input for the average displacement energy at 40 eV. The depth profiles of the displacement damage of 3 MeV Fe-ion irradiation in the T92 F/M steel are shown in Fig. 2. According to the calculated results, the highest damage level in the specimen appeared at about a depth of 800–1000 nm from the surface. The peak damage levels were taken as their nominal displacement damages, which were 0.25, 0.50, 1.00 and 5.00 dpa.

TEM observations were conducted in a Tecnai G2 F30 TEM. The changes of martensitic laths, dislocations, and different families of precipitates (identified by electron diffraction and energy dispersive X-ray spectroscopy (EDS)) were investigated. Ion irradiation-induced defects were also observed in the TEM to study the irradiation damage differences between the tempered and aged T92 samples.

The irradiation hardening behaviors of T92 F/M steel were measured using a nanoindenter (Nano Indenter DCM) with a Berkovich type indentation tip. The instrument was calibrated against a fused silica standard sample. The continuous stiffness measurement mode was used with an indentation depth of 1800 nm, amplitude of 2 nm, and frequency of 45 Hz. More than five indents were measured for each sample.

Table 1
Chemical composition of the investigated T92 F/M steel (wt.%).

C	Si	Mn	P	S	Cr	W	Mo	Ni	V	Nb	B	N	Fe
0.12	0.2	0.53.	0.017	0.0008	8.86	1.65	0.38	0.20	0.20	0.048	0.0024	0.046	Balance