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Microstructure of HIPed and SPSed 9Cr-ODS steel and its effect on helium bubble formation

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HIGHLIGHTS

- The microstructure changes of two ODS steels before and after helium ion implantation have been elucidated.
- The mechanism of the microstructures of ODS steels under varied thermal mechanical processing paths have been explored.
- The dependence of the size, density and distribution of helium bubbles on the specific microstructure features are explored.

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ABSTRACT

Two 9Cr-ODS steels with the same nominal composition were consolidated by hot isostatic pressing (HIP, named COS-1) and spark plasma sintering (SPS, named COS-2). Helium ions were implanted into COS-1, COS-2 and non-ODS Eurofer 97 steels up at 673 K. Microstructures before and after helium ion implantations were carefully characterized. The results show a bimodal grain size distribution in COS-2 and a more uniform grain size distribution in COS-1. Nanoscale clusters of GP-zone type Y–Ti–O and $Y_2Ti_2O_7$ pyrochlore as well as large spinel $Mn(Ti)Cr_2O_4$ particles are all observed in the two ODS steels. The Y–Ti-enriched nano-oxides in COS-1 exhibit higher number density and smaller size than in COS-2. The Y–Ti-enriched nano-oxides in fine grains of COS-2 show higher number density and smaller size than that in coarse grains of COS-2. Nano-oxides effectively trap helium atoms and lead to the formation of high density and ultra-fine helium bubbles.

1. Introduction

First wall/blanket is one of the key structural components in future fusion reactors and is directly bombarded by 14 MeV neutrons at high temperatures, that generates high level of radiation-induced displacement damage in the form of vacancy and self-interstitial atom (SIA) defect. Even more, large amount of helium atoms produced by (n, α) transmutation reactions are introduced into the materials up to several thousands atom parts per million (appm) [1–3]. Helium atoms will be trapped at vacancies produced by irradiation, to form He-vacancy clusters. High concentration helium atoms will precipitate out as gas bubbles due to the extremely low solubility (<<1 at. appm in metals) and low migration energy via interstitial sites (about 0.078 eV) in steels [4]. Helium bubbles are easily trapped at the microstructure defects, such as dislocations, grain boundaries and the surface of precipitates. Helium accumulation under high stress at elevated temperatures may cause formation and growth of voids and grain boundary creep cavities, resulting in degradation of fatigue, thermal creep, creep rupture and creep fatigue properties, especially the tensile ductility due to helium embrittlement [5,6].

Nano-structured oxide dispersion strengthened (ODS) reduced activation ferritic/martensitic (RAFM) steels are promising candidate materials for the first wall/blanket of the future fusion reactors. Ultra-high density nano-oxides, for example, Y–Ti–O nanoclusters (NCs), are dispersed homogenously in the steel matrix and grain boundaries in ODS steels. The interface between matrix and nano-oxides provide a great number of trap sites for helium atoms and bubbles, preventing the helium agglomerating to coarse

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bubbles and minimizing the risk of helium embrittlement due to helium atoms diffusing to the grain boundaries [7]. Moreover, nano-sized helium bubbles themselves could act as stable sinks for enhancing the recombination of vacancies and interstitials survived from cascade collision, effectively suppressing void swelling in irradiated materials. On the other hand, nano-oxides may also act as thermodynamically stable obstacles to impede dislocation and grain boundary movement, enhancing the mechanical properties of the steels even under high temperature [8,9]. Many comparative studies on swelling resistance or helium bubble distribution in conventional RAFM steels and ODS steels have been carried out and reported in the literature [10,11]. Dual heavy ion-He irradiation experiments were performed by Kimura et al. Their results show that the helium bubbles were refined, void swelling was suppressed in ODS steels than RAFM steels [12,13]. Furthermore, high concentration helium ions were implanted into the Eurofer97 steel and a 14Cr-ODS steel developed by author's group. Our results show that helium bubbles formed in 14Cr-ODS steel exhibited smaller size and lower volume fraction than in Eurofer 97 steel [14]. Analyses conducted by transmission electron microscope (TEM) and atom probe tomography (APT) have indicated that dislocations, grain boundaries and the surface of precipitates were the preferential trap sites for absorbing helium atoms [5,6,15]. Similarly to the nano-oxides in ODS steel, grain and phase boundaries can also effectively absorb the radiation-induced defects and trap helium atoms as revealed in nanocrystalline metals and multilayers [16–18].

The specific characteristics, for example, size, density, composition and structure of nano-oxides are highly depending on the alloy composition and production process. The microstructures of ODS steels are still not well controllable and understood because of the variables in composition and producing methods, even characterization methods. However, some common observations and understanding regarding the nano-oxides include the following: (1) Stoichiometric and near-stoichiometric pyrochlore structure $Y_2Ti_2O_7$ have been found in most ODS steels. The size of $Y_2Ti_2O_7$ ranges from a few nanometers to tens of nanometers [14,19–22]. Orthorhombic and hexagonal Y_2TiO_5 has also been reported but they are not very common [19]; (2) Very small size Y–Ti–O oxides, called nanoclusters (NCs) or nano-oxide features (NFs), have also been reported in many references [1,23,24]. Composition analyses of NCs normally have performed by APT. The results show that many of the NCs are highly nonstoichiometric GP-zone like transition sub-oxide phases with high Ti/Y and low O/(Ti + Y) ratios. Comparing with the generally accepted proofs of the chemical composition, the structures of NCs are still in debate. The NCs pose a big challenge for microstructure characterization because of the ultra-fine particle size. Comprehensive high-resolution STEM analysis and computer simulation have been done by Hirata and his co-workers. They stated that NCs were the NaCl rock salt structure, which was coherent with the ferritic matrix [25]. Brandes claimed that the amorphous NCs were found in 14YWT [24].

To further optimize the microstructure and properties of ODS steels as a radiation tolerant material, some important relationships between the microstructure and properties need to be understood, for example, how the natures and distributions of nano-oxides affect the helium behaviors in ODS steels, and what kind of microstructure will lead to a stronger suppression for helium bubble formation. In this study, two kinds of 9Cr-ODS steels solidified by either HIP (hot isostatic pressing) or SPS (spark plasma sintering), along with a non-ODS Eurofer 97 steel have been studied. High fluence helium ions were implanted into these three materials. Microstructure characterizations on the steels before and after helium implantation were performed by TEM, STEM and APT. The microstructures of ODS steels under varied thermal mechanical processing paths have been explored. The changes of the size, density and distribution of helium bubbles in three materials with respect to the specific microstructure features are examined and compared.

2. Experimental

Two 9Cr-ODS steels with the same nominal composition of Fe–9Cr–1.5W–0.4Mn–0.1Ta–0.2V–0.3Ti–0.3Y_2O_3 (mass%) were fabricated at Northeastern University in China. High-purity elemental powders and nanoscale yttrium powders were mixed. Mechanical alloying (MA) was conducted in a planetary high-energy ball mill (Fritsch P5) at room temperature for 50 h in an argon-gas atmosphere. One batch of as-milled powders was consolidated by hot isostatic pressing (HIP) at 1373 K under a pressure of 200 MPa for 2 h, named COS (China ODS Steel)-1. Another batch was consolidated by spark plasma sintering (SPS) at 1223 K under a pressure of 40 MPa for 5 min, named COS-2. A non-ODS Eurofer 97 steel [26] with a nominal composition of Fe–8.93Cr–1.07W–0.49Mn–0.15Ta–0.28V–0.12C (mass%) was also employed in this study for comparison purpose.

For helium ion implantation, the samples with a size at 4×4 mm^2 and a thickness approximately 2 mm were prepared. The surfaces were mechanically polished using 0.05 μm alumina polishing solutions. The polished surfaces of COS-1, COS-2 and Eurofer 97 steels were then implanted with 400 keV He^+ ions to fluences up to 4×10^{17} He^+/cm^2 at a temperature of 673 K at the Michigan Ion Beam Laboratory (MIBL), University of Michigan. Raster beam was used in this case for applying a homogeneous implantation. Predictions of local dose and helium concentration in steels were calculated by the Stopping and Range of Ions in Matter 08 (SRIM08) code in Kinchin-Pease mode with a displacement threshold energy of 40 eV. The result is shown in Fig. 1. The predicted helium peak was at a depth of 850–900 nm stretching from the sample surface, with a He concentration of ~19% and damage of ~9 dpa.

TEM and STEM samples used in this study were all prepared by focused ion beam (FIB) lift-out techniques by using FEI Helios Nanolab Dualbeam workstation in order to obtain the cross-section specimens and to minimize the effect of ferromagnetism. The standard “lift-out” procedure for the cross-sectional TEM sample

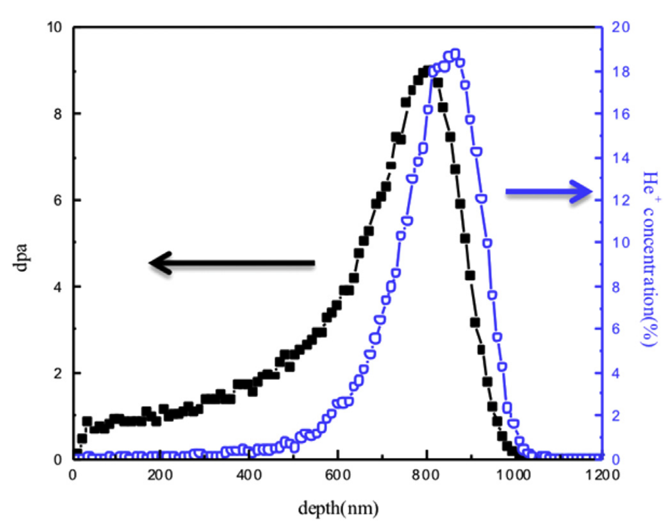

Fig. 1. Depth profiles of displacement damage (dpa) and helium concentration predicted by SRIM code for steels of this study implanted with 400 keV He^+ to 4×10^{17} He^+/cm^2.