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### The direct correlation between precipitate-size-dependent strain at the interface and the irradiation hardening in V-4Cr-4Ti



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#### A R T I C L E I N F O

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#### ABSTRACT

V-4Cr-4Ti alloy, one of the promising candidate structural materials for fusion first wall/blanket applications, was annealed in different temperatures (800 °C and 1000 °C) followed by hydrogen and subsequent helium ions irradiation. It is designed for ion irradiations by forming different size of precipitates, intending to investigate the effect of interfacial coherency characteristic on the irradiation damage behavior at an atomic scale. The microstructure was studied by transmission electron microscopy and the effect of irradiation hardening was evaluated by nanoindentation technique. The results show that TiC precipitate with different size and density was formed during the heat treatment. The lattice matching and the resulting strain state at the interface between the precipitate and the matrix were also investigated quantitatively. The samples with large precipitate exhibited a relatively lower irradiation hardening rate than the one with smaller precipitate and vanadium alloy matrix. It suggests that the strain state at the interface between the precipitate and subdistinct aggregation of He atoms at the interface between the precipitate and the matrix plays a key role in determining the distribution state of irradiation defects thus affect the irradiation damage behavior significantly.

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#### 1. Introduction

The V-4Cr-4Ti alloy has been identified as one of the promising candidate structural materials for fusion first wall/blanket applications, due to low induced radioactivity, good resistance to neutron radiation damage and good elevated-temperature strength [1–4]. Neutron irradiation can result in helium generation by transmutation reactions, involving the host atoms and neutrons. Therefore, it's very important to investigate the interaction of helium with vanadium alloy [5]. Vanadium, chromium and titanium are mutually soluble in each other at elevated temperatures in high purity V-4Cr-4Ti alloy, where is homogeneous and no phase transition. Ti-rich precipitate may usually form due to the existence of impurities in alloys such as carbon, nitrogen and oxygen [6–8], which would affect the mechanical properties strongly. Therefore, vanadium alloys can be optimized by designing size, density, distribution of the precipitate.

The interface issue has been one of the key points affecting the

\* Corresponding author. E-mail address: qzhan@mater.ustb.edu.cn (Q. Zhan). irradiation damage behavior that researchers pay more and more attention recently. Interfaces, acting as unbiased defects sinks, can absorb irradiation-induced point defects and complex thus cause the decrease of the defects concentration in the matrix then improve the ability of resistance to irradiation damage. Computation results revealed that on a multilayer heterointerface, implanted helium could be trapped in a discrete site known as misfit dislocation intersection (MDI) [9]. And the artificially designed heterophase interfaces were considered to present unique characteristics compared to bulk metals under irradiation, including several orders of magnitude higher helium solid solubility, dramatic reduction of bubble density, interface confined growth of helium bubbles, and much lower radiation hardening [10]. In the situation of austenitic steel with M<sub>23</sub>C<sub>6</sub> precipitate, bubbles in higher number density and smaller sizes were found at the semicoherent boundaries, and are located preferentially at the interfacial mismatch dislocations. Meanwhile, larger bubbles were observed at the intersection of M<sub>23</sub>C<sub>6</sub> and grain boundaries because of the reduction of interfacial energies in this place [11]. Investigation by using atom probe tomography (APT) on the interface of nanoclusters or precipitates in oxide dispersion strengthened (ODS) steel showed that the trapping of He atoms at the interface



between the secondary phase and the alloy matrix could reduce the diffusion and coarsening of free He atoms at the grain boundaries, then reduce the degree of He embrittlement [12].

A preliminary study about Ti-rich precipitate behavior in V-4Cr-4Ti under helium irradiation was carried out, showing helium bubbles gathered on the interfaces between precipitate and matrix [13]. While this study puts particular emphasis on heat treatment and micro morphology, the study at an atomic scale needs to be deepened. Which kind of interface structure is conducive to improve the resistance to irradiation damage is still inconclusive. The influence of the coherency degree due to lattice mismatch between alloy matrix and precipitates on the irradiation damage behavior is not clearly understood yet. The resulting stress field range and the strain state around precipitates also need to be discussed in detail. Moreover, property evolution due to irradiation is meaningful for engineering application which can be derived from studies focused on mechanical performance of V-4Cr-4Ti [14]. In the present study, the nucleation and growth of helium bubbles on interface and the size effect of precipitate were investigated systematically to clarify the role of interface structure in affecting the irradiation damage behavior at an atomic scale. Combining the nanoindentation data, we tried to understand the relationship between microstructure and irradiation hardening. This study is expected to be helpful for effective designing the interface structure in the processing of materials.

#### 2. Experiment

The investigated V-4Cr-4Ti alloy was fabricated by vacuum arc remelting of consumable electrode (VAR). The chemical compositions are shown in Table 1.

The  $30 \times 15 \times 15$  mm<sup>3</sup> specimens were cut from the V-4Cr-4Ti alloy ingot. All of those specimens were wrapped by Zr foils [15] and sealed in quartz tubes with high vacuum ( $<10^{-5}$  Pa). Then, specimens were isothermally held at 800 °C and 1000 °C for 10 h respectively to obtain precipitates with different size. Following the heat treatment, specimens were quenched in water at room temperature. Bulk specimens were cut out from two kinds of heat treatments for ion irradiation and nano-indentation (NI) test.

The surface of the NI specimens was mechanically polished carefully by 0.05 µm diamond polishing paste. Nano-indentation hardness measurements were performed at room temperature by an MTS Nano Indenter XP system with a load resolution of 50 nN and displacement resolution of 0.01 nm. The indenter was a threesided pyramid Berkovich-type, and the tip truncation was calibrated using fused silica as a reference specimen. The angle between the normal to the bottom surface and the surface of the conical indenter is 65.3°. The indentation depth limit was set to 1500 nm (in consideration of irradiation depth) under a strain  $0.05 \text{ s}^{-1}$  rate target. The nano-hardness data were analyzed and calculated from at least 6 single hardness-depth profiles by the Oliver and Pharr method [16]. The continuous stiffness measurement (CSM) was conducted to acquire continuously the nanoindentation hardness as a function of depth [17]. The continuous stiffness was measured with a harmonic displacement of 2 nm and 45 Hz frequency.

The Transmission Electron Microscope (TEM) specimens were polished using a twin-jet electro-polisher with a polishing solution

 Table 1

 Chemical compositions of V-4Cr-4Ti alloy (wt.%).

Elements	V	Cr	Ti	С	Ν	0
Content	Balanced	3.88	4.70	0.012	0.038	0.038

of sulfuric acid: methanol: ethylene glycol butyl ether = 1:5:1 at 253 K. Structural investigation has been carried out by using an FEI Tecnai F20 TEM at 200 kV.

Hydrogen and helium ion irradiation was performed at room temperature in Beijing Radiation Center (BRC). The implantation energy is 70 keV for H<sup>+</sup> and 110 keV for He<sup>+</sup>. Sequential-ion implantation referring  $5 \times 10^{16}$  ions/cm<sup>2</sup> H<sup>+</sup> followed by  $5 \times 10^{17}$  ions/cm<sup>2</sup> He<sup>+</sup>. Fig. 1 shows the depth distribution of irradiation in the vanadium alloy calculated in terms of displacement damage using the SRIM2013 code, according to the NRT formula [18]:

$$\mathsf{DPA} = \frac{0.8}{2\mathsf{E}_{\mathsf{d}}} \times \left(\frac{\mathsf{dE}}{\mathsf{dx}}\right)_{\mathsf{n}} \times 10^8 \times \frac{\mathsf{\Phi}}{\mathsf{N}}$$

where the displacement threshold energy  $E_d$  is usually set to be 40 eV;  $(dE/dx)_n$  is the linear energy transferred (LET) per ion to the target by nuclear processes in eV/Å. In SRIM,  $(dE/dx)_n$  equals the sum of phonon and binding energy distribution profiles in which the binding energy profile can be obtained from the SRIM vacancy profile by multiplying by the lattice binding energy;  $\phi$  is ion dose in ions/cm<sup>2</sup>; N is the atomic density in atom/cm<sup>3</sup>. The depths of sequential (H<sup>+</sup> plus He<sup>+</sup> subsequently) ion irradiation were approximately 600 nm.

#### 3. Results and discussion

#### 3.1. TiC precipitate

Morphology and electron diffraction pattern (EDP) of V-4Cr-4Ti alloy are shown in Fig. 2. The original sample without ion irradiation is very clean and the needle-like precipitates can be observed that distribute homogeneously in the matrix in both 800 °C and 1000 °C samples, as shown in Fig. 2(a) and (c) respectively. Fig. 2(b) and (d) give the corresponding magnified images with the inset showing the related electron diffraction patterns (EDPs). It is obvious that different heat treatment temperature leads to distinct differences of precipitates in size and density. The number density of the precipitate is about  $42 \times 10^{19} \text{ m}^{-3}$  with the size of 50 nm  $\times$  5 nm for 800 °C samples, while  $1 \times 10^{19} \text{ m}^{-3}$  in density and 500 nm  $\times$  10 nm in size for 1000  $^\circ C$  ones. The same orientation relationship:  $(010)_m || (0\overline{1}1)_p$ ,  $(100)_m || (100)_p$ ,  $[001]_m || [011]_p$ , was obtained between the matrix and the precipitates in both samples, which means precipitates regardless of the size have the same structure (subscripts m and p represent matrix and precipitate respectively). To confirm the composition of precipitate, electron energy loss spectroscopy (EELS) analysis was carried out. Fig. 3(a) shows a typical EELS spectrum of the precipitate. Compared with vanadium matrix (Fig. 3(b)), the existence of carbon in the precipitate can be observed clearly without distinct evidence of nitrogen ( $N_k = 401 \text{ eV}$ ) and oxygen ( $O_k = 532 \text{ eV}$ ). Combined with the index of EDPs, the precipitate was determined to be TiC phase. It should be note that the peak of V present in the precipitate derived mainly from the influence of vanadium matrix since the precipitate is quite small.

Note that needle-like precipitate tends to elongate along the  $<0\overline{1}1>_p$  direction. The morphology of a precipitate, which is formed by a normal quench and aging treatment, is generally determined by its crystal structure, misfit and the interface structure between the precipitate and the matrix. When the crystal structures of the precipitate and matrix are basically the same and the misfit is small, the precipitate takes the form of a sphere. With increasing the misfit, the shape of precipitate would change from spherical to needle-like. When the crystal structures of precipitate and the

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