Effect of cold work deformation on irradiation hardening of vanadium alloys

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Abstract
Vanadium alloys are regarded as promising candidate structural materials for the advanced blanket concept in fusion reactors due to their low activation, good high-temperature strength and, in particular, their compatibility with liquid lithium. In the present work, six kinds of V–5Cr–5Ti alloys under heavy cold work with deformation amounts of 40\%, 60\% and 80\%, and/or subsequent annealing were investigated. Irradiation damage of 0.1, 0.3 and 0.5 dpa was introduced in both specimens using 352.8 MeV Fe ions at 100° C. Electron backscattered diffraction and transmission electron microscopy (TEM) were used to investigate pre-irradiation microstructures such as grains, dislocations, precipitates and bubbles. X-ray diffraction was used to evaluate the pre-existing dislocation density and TEM was used to image the irradiation defects. The change in hardness was evaluated using micro-hardness tests. Before irradiation, the hardness increased with the increasing deformation amount but decreased after subsequent annealing. Dislocation cells turning into sub-grains with low-angle boundaries were observed, while the deformation amount reached 80\% in cold-worked specimens. After irradiation, hardening was observed in all specimens and at all irradiation doses, and a power-law relation was observed in dose-dependent hardening. The effect of the initial microstructure on irradiation hardening was discussed in terms of the sink strength while ignoring grains and precipitates due to their large size. Pre-existing bubbles could effectively reduce irradiation hardening compared with previous results. Meanwhile, with the increasing sink strength of dislocations, hardening decreased in a different manner in cold-worked and annealed specimens. The irradiation defects in some specimens were investigated to clarify the inherent mechanism in the relationship between the initial microstructures and irradiation hardening.

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1. Introduction

Nuclear energy, especially fusion energy, has raised research heat worldwide in the search for reliable, cost-effective and sustainable energy to decarbonize energy sources [1, 2]. However, the degradation of structural materials, such as plasma-facing materials and blanket materials, which sustain intense neutron flux and transmutation products, is a key issue that constrains the development of fusion reactors, as well as the fusion energy [3, 4]. Energetic neutrons will cause defects such as dislocation loops, irradiation-induced precipitates and voids in materials, deteriorating the mechanical properties and shortening the service life of the structural materials [5].

Despite the helium embrittlement at elevated temperature, which restricts the upper limit of the temperature window of service and void swelling as well as thermal creep, irradiation hardening/embrittlement at low temperature \( T < 0.3 \ T_m \), \( T_m \) is the melting temperature of materials, which restricts the lower limit of the temperature window of service, is a major concern for structural materials due to the low dose threshold of its occurrence (about 0.1 dpa) [4, 5]. Irradiation-induced defects and accompanying property degradation has been widely investigated in fusion-relevant structural materials such as tungsten, reduced-activation ferritic/martensite steels and oxide dispersion-strengthened steels (ODS), SiC and vanadium alloys [6–10].

Due to their low activation, good high-temperature strength and compatibility with liquid lithium, vanadium alloys have been regarded as prime candidate structural materials for the advanced blanket concept in fusion reactors [11–14]. The addition of V and Ti in vanadium alloys was comprehensively investigated due to the enhanced strength and improved ductility [15, 16]. In addition, \( V-(4–5)Cr-(4–5)Ti \) alloys were fabricated because of their improved high-temperature strength, good ductility and not higher ductile–brittle transition temperature [17]. The addition of impurities such as Si and Y in \( V-(4–5)Cr-(4–5)Ti \) alloys to decrease irradiation damage was also reported previously [18, 19]. Despite the composition modification, the introduction of sinks serving as point defect recombination centers in Fe-base alloys has been intensively investigated [5, 20, 21]. Fine precipitates through the mechanical alloy process, ultra-fine-grain steels and engineered dislocations by additive manufacture (AM) are expected to reinforce the radiation tolerance of materials [22–24]. In recent years, severe plastic deformation (SPD) and AM were used to produce dislocations in materials [25, 26]. In addition to that, traditional method, like cold work process in 316 stainless steel (deformation amounts of 20%), is proved to have better void swelling resistance due to the introduction of dislocations [27]. Meanwhile, the effect of cold and subsequent annealing on materials was reported in previous papers [28–30]. However, in vanadium alloys, engineered sinks to enhance radiation tolerance have been poorly investigated. In the present work, the effect of heavy cold work deformation with different amounts and subsequent annealing on the hardening resistance of materials was investigated in terms of the changing dislocations. Also, fine-scale bubbles were introduced into the vanadium alloys to investigate the effectiveness of sinks originating from bubbles.

Due to the high expense and complicated post-irradiation examination of neutron irradiation experiments, energetic heavy-ion beams, which have a similar median recoil energy \( T_{1/2} \) to that of fast neutrons in materials, have long been used as surrogate irradiation sources to investigate radiation effects in candidate structural materials [31–35]. Due to the limited damage depth of ions with fixed kinetic energy in materials, high-energy heavy ions were used to cause a deep enough damage layer in materials, facilitating the evaluation of the change in hardness using micro-indentation tests.

2. Experimental methods

2.1. Materials

The materials used in the present work were \( V–5Cr–5Ti \) alloys (\( V–4.86Cr–4.98Ti–0.01C–0.004N–0.038O, \text{ wt.}\% \)). The materials were first fabricated by vacuum melting, homogenizing annealing and hot forging. Then the materials were processed by cold rolling with deformation amounts of 40%, 60% and 80% to modify the microstructure. Meanwhile, parts of the alloys were annealed at 1273 K for 1 h in a vacuum condition for recrystallization. For convenience, the \( V–5Cr–5Ti \) alloys under different fabrication processes were denoted as 40% CW, 60% CW and 80% CW for cold-worked samples and 40% CW + annl, 60% CW + annl and 80% CW + annl for annealed samples with different deformation amounts, respectively.

Then, specimens with a size of \( 5 \times 15 \text{ mm}^2 \) and a thickness of 1 mm were cut from the plates with the normal direction perpendicular to the rolling direction. Before tests, all of the samples were firstly mechanically grounded with SiC papers (from 600# to 2400#) and then polished with colloidal silica suspension (0.1 μm) to diminish small scratches.
2.2. Irradiation experiments

The samples were irradiated simultaneously using $^{56}$Fe$^{17+}$ ions with a kinetic energy of 352.8 MeV in a sector-focused cyclotron (SFC) chamber in the Heavy Ion Research Facility in Lanzhou (HIRFL) located at the Institute of Modern Physics, Chinese Academy of Sciences (IMPCAS). The six specimens were mounted on a copper disk fixed on the sample stage with the temperature controlled by a cooling/heating dual system unit. An aperture/Al-foil assembly and a Faraday cup were placed in the beamline to monitor the beam current. Because the damage caused by the ions with a given kinetic energy varies with the depth and reaches a maximum at the end of the projective range of the ions in materials, a two-wheeled energy degrader equipped with a series of Al foils with different thicknesses was placed in front of the sample stage to obtain quasi-uniform damage distribution in the specimens. A quasi-uniform distribution of defects from the near-surface to a maximum depth of 30 $\mu$m was introduced in the specimens after successively degrading the kinetic energy of the incident ions. An image of the chamber together with a schematic diagram are shown in figure 1. A detailed description of the irradiation chamber and irradiation experiments was given in our previous paper [36]. The depth distribution of irradiation damage, displacement per atom (dpa), calculated using SRIM 2013 [37] (using the Kinchin–Peace model with $E_{d-V/Cr} = 40$ eV and $E_{d-Ti} = 30$ eV [38]) is shown in figure 2 with the thicknesses of the used Al foils given in the right column. According to figure 2, total damage of about 0.5 dpa with a corresponding ion fluence of $8.5 \times 10^{15}$ ions/cm$^2$ was attained in alloys, analogous to 0.1 and 0.3 dpa with a corresponding ion fluence of $1.7 \times 10^{15}$ and $5.1 \times 10^{15}$ ions/cm$^2$, respectively. During irradiation, the temperature was maintained at a nominal 100 $^\circ$C with a thermocouple to monitor the temperature. The vacuum of the irradiation chamber was kept at $2 \times 10^{-5}$ Pa in the steady state.

2.3. Hardness test

A micro-hardness tester with a Vickers indenter (Falcon 600, Innovatest Corp) was used to evaluate the hardness of materials. A calibration was performed on a standard sample before the tests to diminish the system error. Indentations with nine loads were applied (0.01, 0.015, 0.02, 0.025, 0.05, 0.1, 0.2, 0.5 and 1.0 kgf) to each specimen to obtain the hardness at varied indentation depth. To eliminate the random error, the indentation tests were repeated at least six times at a fixed load to obtain an average result. During tests, the indenter remained 10 s at the maximum load.

2.4. Microstructure investigation

A scanning electron microscope (SEM, JSM-7900F) equipped with electron backscatter diffraction (EBSD, Symmetry, Oxford Instruments) was used to characterize the grains. Specimens for EBSD tests were prepared by vibratory polishing (VibroMet 2, Buehler) to obtain a smooth and flat surface and relieve the deformed layer in the surface. Grain size distribution was statistically measured by considering grains with a fixed minimum misorientation angle of 15$^\circ$ using OIM (orientation imaging microscopy) analysis.
Figure 3. The grain maps obtained by EBSD for all samples with (a)–(c) representing the 40%, 60% and 80% CW samples, and (d)–(f) representing the 40%, 60% and 80% CW + annl samples. The white arrows denote the fine or coarse regions and the yellow arrow denotes the rolling direction.

A transmission electron microscope (TEM, Tecnai F20) was used to investigate the dislocations, precipitates and bubbles in the virgin specimens and irradiation defects in the irradiated specimens. Two kinds of TEM specimens were prepared. One was prepared using a standard double-jet technique (Struers Tenupol 5). Small disks (3 mm in diameter) were cut off from the materials and then mechanically ground down to a thickness of 50–100 μm. As an electrolyte, a mixture of 95% CH₃OH and 5% H₂SO₄ was used at −10 °C, 10 V. Other specimens were prepared by the focused ion beam (FIB) lift-out technique (FEI, Helios nanolab 600). Considering that the pre-existing dislocations in unirradiated samples are of high density and large size, the dislocations were investigated using Φ3 specimens in the region with a thickness greater than 150 nm. In addition, the kinematic bright field (KBF) condition was used to image the initial dislocations. Considering that irradiation defects are of small size (<10 nm), to weaken the interference from the initial dislocations and the overlap of irradiation defects, the weak-beam condition was conducted using the FIB specimens.

After the plastic deformation, the initial dislocations usually tend to tangle and cluster, which makes it tough to clearly visualize individual dislocations and properly estimate the dislocation density. Thus, x-ray diffraction (XRD) analysis as a complementary method was used in the present work. Before XRD analysis, the materials were vibratory polished (see details in the EBSD part) to relieve the deformed layer in the surfaces. Cu Kα radiation with 40 kV, 100 mA was used. Diffraction patterns were recorded at 2θ with a step size of 0.02. The time intervals of the measurements were 10 s/step. The XRD intensity profiles were analyzed using Jade software. The lattice parameters were determined through the FullProf fitting of the XRD profiles. XRD line broadening was characterized by the integral width β. The instrumental contribution to the overall line broadening was corrected using a LaB₆ standard material.

3. Results

3.1. Pre-existing microstructures

The grain results obtained by EBSD are shown in figure 3. Grains with the size of dozens of micro-meters are observed in all alloys. In cold-worked samples, the grains are distributed non-uniformly and are divided into two parts: a fine-grain region with small equiaxial grains and a coarsening-grain region with large elongated grains (as denoted with the white arrow in figure 3). With the increasing deformation amount, the extent of elongation of the large grains is more obvious. Also, with the deformation amount reaching 80%, the proportion of fine grains increases rapidly. Similar cases were observed in hot extruded steels [39]. In annealed samples, small grains grow up as the large grains disappear, which results in a quasi-uniform distribution of grains. The size distribution of grains was depicted in our previous paper [40]. The average diameter of grains is statistically measured by considering the amounts of grains, and the result is shown in table 1.

As shown in table 1, the average grain diameter of all alloys varies from 25 to about 40 μm and decreases with the increasing deformation amount. After annealing, the average diameter increases slightly.

The dislocations imaged by TEM using the KBF condition with g = ⟨200⟩ are shown as follows. As shown in figure 4,
Table 1. Parameters obtained by EBSD and XRD and the calculated sink strength of all samples.

| Specimens     | Dislocation density \( \rho \) \( (1 \times 10^{15} \text{ m}^{-2}) \) | Grain size (\( \mu \text{m} \)) | Lattice parameter (Å) | Sink strength of dislocations: \( S_d \) \( (1 \times 10^{15} \text{ m}^{-2}) \) | Total sink strength: \( S_{\text{total}} \) \( (1 \times 10^{15} \text{ m}^{-2}) \) |
|---------------|-----------------------------------|-------------------------------|------------------------|--------------------------|--------------------------|
| 40% CW        | 5.1 ± 3.4                         | 39.8                          | 3.06                   | 15.5 ± 4.1               | 11.5 ± 1.3               |
| 60% CW        | 5.4 ± 0.5                         | 26.2                          | 3.02                   | 15.7 ± 0.6               | 9.1 ± 1.2                |
| 80% CW        | 8.0 ± 2.7                         | 25.5                          | 3.03                   | 18.9 ± 3.2               | 11.6 ± 1.3               |
| 40% CW + anml | 5.2 ± 0.5                         | 32.7                          | 3.02                   | 2.9 ± 0.7                | 3.04                 |
| 60% CW + anml | 8.0 ± 1.0                         | 32.7                          | 3.02                   | 2.4 ± 0.6                | 3.04                 |
| 80% CW + anml | 4.0 ± 1.1                         | 27.7                          | 3.02                   | 1.9 ± 1.0                | 3.04                 |

Dislocations with high density are produced in the 40% CW samples, which formed into band-like structures (dislocation cells) with a direction parallel to the cold rolling direction (as denoted by yellow arrow). The high magnification result indicates that scattered dislocations with a length of dozens of nanometers make up the dislocation cells.

With the increasing deformation amount, the boundaries of the dislocation cells are more specific and the dislocation cells are much denser. In 80% CW samples, sub-grains with low-angle boundaries parallel to the rolling direction were observed, as shown in figure 5. The result indicates that dislocation cells transform into sub-grains. Combined the results of figures 4 and 5, the cases that elongated sub-grains with high dislocation density co-exist with sub-grains with few dislocations (as denoted by black dotted arrow) were observed in the cold-worked samples.

In the annealed samples, the dislocation morphology and arrangement were obviously different. The dislocation cells parallel to the rolling direction in cold-worked samples are no more specific. When the deformation amount reaches 60%, the dislocation cells become narrow and tend to form grain boundaries (GBs), as shown in figure 6(a). Parts of the dislocation lines no longer show distributed scattering, but are instead tangling with each other, as marked with the rectangular dotted lines in figure 6(a), while other dislocation lines increase to hundreds of nanometers and form dislocation networks as marked by the circular dotted lines in figure 6(b). When the deformation amount reaches 80%, the cases where the dislocation walls transfer into GBs are more obvious, as shown in figure 7. The grains next to the GB exhibit different dislocation features: grains with few dislocations (on the left of the GB) co-exist with grains with high-density dislocations (on the right of the GB). The case is similar to that shown in figure 5, and is also reported in a previous paper [41]. Also, the dislocations in the right-hand grain become sparse in the region away from the GB, which provides evidence that the dislocations migrate to the GB during annealing.

From the above TEM results, the dislocations overlap, cluster or tangle with each other, making it difficult to distinguish each single dislocation line clearly and evaluate the dislocation density. So quantitative analysis of dislocation density using TEM is a tough challenge. XRD line profile analysis was used as a complementary method to obtain the dislocation density. The obtained XRD results are shown in figure 8. The XRD line profiles of all specimens are broadening to some extent. Compared with cold-worked samples, the line broadening of the annealed specimens obviously decreased. In general, the line broadening is mainly ascribed to two factors: fine grains and dislocations. The Williamson–Hall (WH) method has been widely used to qualitatively estimate the two components [42].

However, for traditional WH plots, the line broadening does not monotonously increase with the order of reflections in the deformed alloys. The discrimination originated from the anisotropies in materials. In previous papers, the anisotropies were mainly ascribed to the line broadening induced by dislocations and the corresponding dislocation contrast factors.
Figure 4. TEM results of dislocations in 40% CW samples obtained by KBF condition with \( g = \langle 200 \rangle \) using \( \varphi 3 \) specimens. The yellow arrow denotes the rolling direction.

Figure 5. TEM results of dislocations in 80% CW samples obtained by KBF condition with \( g = \langle 200 \rangle \) using \( \varphi 3 \) TEM specimens. The yellow dotted arrows denote the GBs and the black dotted arrow denotes grains with few dislocations.

Thus, a modified model which correlates the integral width \( \beta \) and the dislocation density \( \rho \) was used to obtain the dislocation density [45],

\[
\beta = \frac{k}{D} + b_0 W(g) + B_0 \sqrt{\rho} \sqrt{\chi} \sin \frac{\theta}{\lambda} + O^2 \quad (1)
\]

where \( k \) is the Scherrer constant, \( b_0 \) is a factor related to the effect of stacking faults, \( W(g) \) is an orientation factor, \( B_0 = A_0 b \sqrt{2} \ln P \) (with \( A_0 \sim 1 \), \( b \)-Burgers vector, \( P \)-factor), \( \chi \) is the dislocation contrast factor, \( O^2 \) is the higher-order term (not considered) and \( D \) is the mean grain size. The \( P \)-factor can be estimated from the line profiles [45, 46]. The \( \chi \) is estimated based on the method reported by Ungar [44] with the relating elastic parameters from the V–5Cr alloy [47]. The method has been widely used to evaluate the dislocation density in deformed metals. TEM has also been used to validate the accuracy of the results from XRD [41]. In the present work, the stacking faults are not considered. However, the anisotropies still exist at some data points in the present work. The reason may be the severe deformation. The plots based on equation (1) are shown in figure 9, where a quasi-monotonous increment was demonstrated. By linear fitting of the data points, the obtained dislocation density with standard error from the linear fitting is shown in table 1. The results of the present work lay in the values reported in other deformed alloys [48, 49].

In cold-worked samples, the dislocation density increases with the increasing deformation amount, and a sharp increment occurs when the deformation amount reaches 80%. After annealing, the dislocation density decreases at a fixed deformation amount. The TEM results indicate that annealing, growing or migrating to the GBs of dislocations results in a decrease in the dislocation density. A non-monotonous increase but firstly a slight decrease and then a sharp increase of dislocation density was observed in the annealed samples. The abnormal phenomenon is discussed as follows. Firstly, in cold-worked samples, no obvious increase in dislocation density occurs when the deformation amount reaches 60%. Thus, no apparent amount of dislocation was produced. Secondly, in TEM results, sub-grains and dislocations migrating to GBs were observed in cold-worked and annealed samples with higher deformation amounts, respectively, indicating that the transformation of dislocation to GBs results in the decrease of dislocation density in annealed samples when the amount of deformation reaches 60%.

Meanwhile, during fabrication, fine-scale bubbles with high number density were introduced in all specimens, as shown in figures 10 and S1 (see the supplementary materials). The bubbles in figure 10 were characterized by the Fresnel contrast that white contrast in the under-focused image or black contrast in the over-focused image. The method has been widely used in many works. Also, the existence of bubbles was checked in both specimens prepared by electropolishing and FIB to exclude the possible disturbance from artifacts. The same results were observed in both specimens, as shown in figure S1. Meanwhile, as expected, bubbles were also observed in the post-irradiation specimens, as shown in figure S1. After statistically counting, the bubbles do not obviously change following the changing deformation amount. In cold-worked specimens, the average size of bubbles is 1.37 nm with a number density of \( 5.39 \times 10^{23} \) m\(^{-3}\). After annealing, the average size slightly decreases to 1.17 nm with the number density decreasing to \( 4.63 \times 10^{23} \) m\(^{-3}\).

TEM equipped with a spherical aberration corrector with a high voltage of 300 keV (Titan Themis G2 300) was attempted to investigate the composition of the bubbles. The
Figure 6. TEM results of dislocations in 60% CW + anl samples obtained by KBF condition with \( g = \langle 200 \rangle \) using \( \varphi 3 \) TEM specimens. The yellow dotted arrows denote the GBs and the rectangular and circular dotted lines denote the tangling dislocations and dislocation networks, respectively.

Figure 7. TEM results of dislocations in 80% CW + anl samples obtained by KBF condition with \( g = \langle 200 \rangle \) using \( \varphi 3 \) TEM specimens. The yellow dotted arrow denotes the GB.

Figure 8. XRD line profiles of all samples.

Figure 9. Linear fitting results of XRD line broadening according to equation (1).

high-angle circular dark field image obtained using the STEM model (HADDF-STEM) is shown in figure 11. The contrast in the HADDF-STEM image comes from the atomic number \( Z \) contrast [50]. The black contrast zone with a spherical shape as denoted by the yellow dotted arrows shows a similar diameter to the bubbles determined in figure 10(b). Hence, the defects are bubbles, possibly. Furthermore, the EDS (energy dispersive spectroscopy) spectra obtained from the yellow rectangular area are shown in the right image. Due to the limited resolution, precise determination of the composition failed in the present work, but the sub-peak as denoted with the dotted yellow arrows seemingly indicates the possibility of the appearance of O-rich or N-rich bubbles.

As shown in previous papers, Ar-filled bubbles were observed in ODS steels after mechanical alloy in Ar-atmosphere [51]. Also, it was reported by Fukumoto that small voids were observed in vanadium alloys after irradiation with Cu ions at 400 °C, and after reducing the interstitial impurity by the Zr-treatment method, void formation could be inhibited, indicating interstitial impurity-assisted vacancy clustering [52]. However, due to the limitation of the TEM resolution and low atom concentration in the bubbles, the precise determination of the nature of the bubbles is a tough challenge. The detailed investigation already exceeds the scope of the present work and will be discussed in our next works.

Typical precipitates in 80% CW + anl samples characterized by TEM are shown in figure 12. As shown in figure 12(a),
Figure 10. Under-focused TEM results of (a) 40% CW and (b) 40% CW + annl specimens with the asterisk representing the corresponding over-focused results obtained using ϕ3 TEM specimens.

Figure 11. HADDF-STEM results obtained by Cs-corrected TEM in 80% CW + annl specimen using ϕ3 TEM specimens.

the precipitates are elliptical-shaped and attached to GBs. As shown in figure 12(c), selected area electron diffraction (SAED) indicates that the precipitate has a face-centered cubic (fcc) structure while the matrix has a body-centered cubic (bcc) structure (as shown in figure 12(b)). Except for the precipitates in GBs, the precipitates within grains are surrounded by dislocations, as shown in figure 12(d). The size of the precipitates is hundreds of nanometers. One of the co-authors, Zengde Li, has already investigated the evolution of precipitates in alloys during the fabrication process [53]. The precipitates in the cold-worked samples have a hexagonal close-packed (hcp) or fcc structure, while precipitates in the annealed samples only have an fcc structure. The EDS results indicate that precipitates are mainly TiV(CON). Both of our works revealed that precipitates were mainly of a size of hundreds of nanometers, and no dispersion phase with a size less than 20 nm was observed.

3.2. Hardness

The typical Vickers hardness results are shown in figure 13(a). For a fixed load, the hardness of the irradiated specimens is apparently larger than the unirradiated counterpart, which indicates that after irradiation, a clear hardness increment occurred. As shown in the profiles, the hardness decreases with the increase in the applied load or the indentation depth, and tends to reach a steady state. This is ascribed to the indentation
size effect, as reported in a previous paper [54]. Geometrical necessary dislocation was introduced to extrapolate the equivalent bulk hardness, which is also well-known as the Nix–Gao model,

\[
\frac{HV}{HV_0} = \sqrt{1 + \frac{h_0}{h}}
\]  

(2)

where \(HV\) is the measured hardness at a fixed load, \(HV_0\) is the hardness at an infinite depth, and also represents the equivalent bulk hardness, \(h_0\) is the character length, which depends on the indenter and material, and \(h\) is the indentation depth [55]. Hence, the hardness profiles are replotted and linear-fitted based on equation (2), as shown in figure 13(b). A good linear relation in the entire indentation depth regime for the unirradiated samples was observed. However, a bilinear manner for the irradiated specimens with a turning point at about 0.25 \(\mu m\) (corresponding to the sixth load, 0.1 kgf) was observed. In the indentation test, the deformation region beneath the indenter is usually 5–10 times deeper than the indentation depth. During indentation, the deformation region transfers from the irradiated region into a region that covers part of the unirradiated region.
Table 2. The evaluated hardness $HV_0$ and the calculated irradiation hardening $\Delta HV_0$ of samples in units of kgf/mm$^2$.

| Specimens  | Unirr. $HV_0$ | 0.1 dpa $HV_0$ | $\Delta HV_0$ | 0.3 dpa $HV_0$ | $\Delta HV_0$ | 0.5 dpa $HV_0$ | $\Delta HV_0$ |
|-----------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| 40% CW    | 234.5 ± 1.2   | 246.6 ± 1.7   | 12.1 ± 2.7    | 251.9 ± 2.7   | 17.4 ± 3.9    | 254.4 ± 1.7   | 19.9 ± 2.9    |
| 60% CW    | 241.7 ± 0.8   | 247.2 ± 1.5   | 5.5 ± 2.3     | 252.6 ± 0.9   | 10.9 ± 1.7    | 257.0 ± 0.8   | 15.3 ± 1.6    |
| 80% CW    | 271.8 ± 1.3   | 275.5 ± 1.2   | 3.7 ± 2.5     | 278.4 ± 0.8   | 6.6 ± 2.1     | 280.6 ± 1.2   | 8.8 ± 2.5     |
| 40% CW + annl | 177.4 ± 0.8   | 198.5 ± 0.7   | 21.1 ± 1.5    | 204.9 ± 1.5   | 27.5 ± 2.3    | 210.0 ± 1.9   | 32.6 ± 2.7    |
| 60% CW + annl | 176.7 ± 1.0   | 202.3 ± 2.8   | 25.6 ± 3.8    | 207.1 ± 1.8   | 30.4 ± 2.8    | 213.1 ± 3.5   | 36.4 ± 4.5    |
| 80% CW + annl | 214.3 ± 1.8   | 221.7 ± 1.0   | 7.4 ± 2.8     | 230.3 ± 0.8   | 16.0 ± 2.6    | 235.6 ± 0.5   | 21.3 ± 2.3    |

Figure 14. The evaluated hardness $HV_0$ (represented with bar chart) and the elongation from [56] (represented with points) of the pristine specimens.

Figure 15. Dose dependence of all samples. The dotted lines represent the non-linear fitting results based on the power-law relation, and the fitted power-law relation of each sample is shown in the lower right corner.
Figure 16. Bright-field images of (a) 40% CW and (b) 40% CW + annl specimens irradiated to 0.5 dpa with the asterisk representing the corresponding dark field images obtained by TEM under the same condition (WBDF, \(g/4g, g = (200), B = [001]\)) as shown by the SAED patterns inserted in (a) and (b). The arrows denote the typical dislocation loops with color having no meaning.

In the present work, a simplified but commonly used power-law relation,

\[
\Delta HV = a \times (\text{dpa})^n
\]  

(3)

where \(a\) and \(n\) are the parameters, was used to describe the dose dependence [57]. As shown in figure 15, the power-law relation is suitable for all the samples. The fitted parameter, \(n\), varies from 0.21 to 0.65, which is consistent with previous papers [58].

3.3. Irradiation defects

Normally, a higher dpa (damage level) means higher defect production, and a specimen with a lower deformation amount means lower strain in the matrix, making the investigation of irradiation defects easier. Hence, in the present work, 40% CW and 40% CW + annl specimens irradiated to 0.5 dpa were selected to characterize the irradiation defects. The results obtained by TEM under the weak beam dark field (WBDF) condition (\(g/4g, g = (200), B = [001]\)) using FIB specimens are shown in figure 16. Small defects with a loop or dot shape, as denoted by the dotted arrows, were observed in both specimens and are regarded as dislocation loops (black dots are usually regarded as small dislocation loops). As reported, the formation of radiation-induced precipitates (RIPs) relies on the irradiation temperature, which affects the diffusion of the impurity atom. In irradiated vanadium alloys, RIPs, especially Ti-enriched precipitates, were usually observed at a temperature above 250 °C and have different characteristics with dislocation loops [59–63]. Meanwhile, after a careful check, no additional speckles were observed in SAED, which on some occasions accompanies the formation of RIPs [61]. Hence, the irradiation dislocation loops are the main defects in the present work. Images of two selected areas were used to obtain the average diameter and number density of irradiation defects. The average size of the irradiation defects in the 40% CW specimen is 5.1 nm with a number density of \(5.1 \times 10^{21} \text{m}^{-3}\), while in the 40% CW + annl specimen the size is 3.1 nm with a number density of \(10.1 \times 10^{21} \text{m}^{-3}\). A decrease in the average size and
an increase in the number density of irradiation defects were observed.

4. Discussion

The material design will lead to a change in mechanical properties through the microstructure evolution. In the degradation of materials after irradiation, the hardness increment strongly relates to the surviving irradiation defects, which depend on the irradiation tolerance of materials, as well as the sink strength of materials. Hence, a discussion will be conducted concerning the relationship between hardening and sink strength.

As reported, the initial microstructure, including grains, dislocations, precipitates and voids, can serve as sinks for irradiation defects [5, 64, 65]. The influence of small bubble-son defect evolution has not been clearly investigated. In the present work, multi-factors in initial microstructure such as precipitates, grains, dislocations and bubbles puzzle the analysis. For precipitates, in our previous papers, dispersed nano-feature precipitates (a few nanometers) with a high number density (above $1 \times 10^{21} \text{m}^{-3}$), such as oxides in ODS steels, will provide sufficient sink strength for damage evolution [39, 66]. Because the precipitates in the present work are of large size and low number density, they will not be considered here. The sink strength of dislocations and grains analyzed using kinetic rate theory [64] is described in equations (4)–(6):

\begin{align*}
S_d &= Z_d \rho_d \\
S_g &= 6(S_{\text{total}})^{1/2} d_g, \quad (S_{\text{total}})^{1/2} d_g \gg 1 \\
S_g &= 60/d_g^2, \quad (S_{\text{total}})^{1/2} d_g \ll 1 \\
S_b &= 4\pi rC \\
S_{\text{total}} &= S_g + S_d + S_b
\end{align*}

where $Z_d$ is the dislocation capture efficiency, $\rho_d$ is the dislocation density, $d_g$ is the grain size, $S_{\text{total}}$ is the total sink strength, and $S_d$ and $S_g$ are the sink strength of dislocations and grains, respectively. For SIA, in previous papers, $Z_d$ was taken as 1.2 [28]. Also, the sink strength of cavities was given in reference [64]. In the present work, while hardening is mainly considered, a simplified model considering a spherical sink was used, as shown in equation (7) [39, 67], where $r$ and $C$ are the average diameter and number density of bubbles, respectively.

The calculated sink strength is shown in table 1. The calculated $S_g$ is of the order of $1 \times 10^{13} \text{m}^{-2}$, which is significantly lower than $S_{\text{total}}$, and is regarded to contribute little to hardening, according to our previous work [39]. Meanwhile, the calculated $S_b$ is $9.3 \times 10^{13} \text{m}^{-2}$ and $6.8 \times 10^{15} \text{m}^{-2}$ for the cold-worked and annealed specimens, respectively, indicating that bubbles may act as effective sinks. The calculated $S_d$ and $S_{\text{total}}$, as shown in table 1, follow a similar trend to the dislocation density as the deformation amount changes.

Despite the kinetic rate theory analysis, experiments were attempted to give direct evidence of the effect of the initial microstructure on irradiation effects [39, 66, 68]. In our previous work, sinks could change the defect production, as well as the irradiation hardening, and a linear relation between sink strength and hardening was observed [39]. Hence, in the present work, the change in hardening following the changing sink strength was demonstrated to verify the effectiveness of the investigated sinks. However, despite the grains and precipitates, two variants including bubbles and dislocations dominate the total sink strength, making the investigation difficult.

Fortunately, irradiation hardening of vanadium alloys without pre-existing bubbles has been investigated in many works, in which the materials are usually under a certain extent of deformation with subsequent annealing, similar to the 40% CW + annl specimens. Hence, the hardening of the 40%
when the dislocation density reached 1 elseif the irradiation hardening. As shown in reference [82], indicating that pre-existing dislocations could evidently influence in both cold-worked and annealed specimens at all doses, the increasing sink strength, irradiation hardening decreased in log–log plots was observed in the present work and the materials located in the shaded region are of similar size but microstructures, irradiation defects in some post-irradiation effecting data in previous papers cover multiple conditions, such as varied irradiation temperatures and different irradiation sources (neutrons and ions). As shown in figure 17, the hardening of vanadium alloys without pre-existing bubbles is always located in the shaded region regardless of the irradiation temperature and irradiation sources, and is obviously greater than the materials investigated in the present work. The results strongly demonstrate that pre-existing bubbles significantly reduce irradiation hardening, and verify the effectiveness of sinks originating from bubbles. As for the microstructures, irradiation defects in some post-irradiation materials located in the shaded region are of similar size but a higher number density (with a magnitude of $1 \times 10^{23}$ m$^{-3}$) when compared with 40% CW + annl specimens [61]. Hence, pre-existing bubbles will significantly reduce the number density of irradiation defects due to their role as point defect recombination centers, which will enhance the annihilation of point defects during irradiation. As reported, fine-scale precipitates (size less than 2 nm) in nanofatigue alloys are considered to make materials of excellent radiation tolerance [21].

For figure 17, it is also worth noting that a linear relation in log–log plots was observed in the present work and the shaded region, especially for the specimens irradiated at low temperature ($T \leq 300 \, ^\circ C$). A similar relation was observed in reference [58], where multiple materials with bcc, fcc and hcp structures were irradiated with neutrons or protons at a temperature below 200 $^\circ C$. The discrete points in the shaded region are mainly divided into two parts: a high-temperature irradiation condition leads to the lower limit, and ion irradiation makes up the upper limit. Irradiation at a high temperature will lead to the annihilation of irradiation defects, resulting in a lower irradiation hardening. Ion irradiation lead to a higher dose rate, resulting in a higher irradiation hardening, as shown in reference [71]. Meanwhile, bubbles can also appear in post-irradiation materials, especially after neutron, helium plasma or helium ion irradiation, which were well-known as helium bubbles in many works [72–74].

The effect of sink strength of dislocation on irradiation hardening was investigated using materials with different heat treatments and the results are shown in figure 18. With the increasing sink strength, irradiation hardening decreased in both cold-worked and annealed specimens at all doses, indicating that pre-existing dislocations could evidently influence the irradiation hardening. As shown in reference [82], when the dislocation density reached $1 \times 10^{15}$ m$^{-2}$, pre-existing dislocations could effectively reduce the irradiation defects. Also, as calculated, pre-existing dislocations could influence the radiation cascades and hence reduce irradiation defects [24, 83]. In situ irradiation experiments have also revealed that regions with a high dislocation density have a lower defect level than those with few or no dislocations [82, 84].

It is worth noting that the descending trend following the increasing sink strength shows a slight difference for the different doses. For the lower dose (0.1 dpa), the decrease in irradiation hardening is slightly greater than for the higher dose (0.5 dpa), indicating that at a lower dose, pre-existing dislocations have a higher capability of reducing irradiation hardening. As shown in [84], pre-existing dislocations will resolve into dislocation segments and even diminish during irradiation. Also, as reported in reference [24], dislocation cells acting as sinks will degrade due to the radiation-enhanced diffusion and lead to a decreasing sink strength. Hence, with the increasing dose, irradiation-induced pre-existing dislocations or dislocation wall degradation will lead to a decrease in the sink strength, virtually, resulting in a weaker capacity for reducing irradiation hardening.

Also, the descending trend following the increasing sink strength is different in specimens before and after annealing. This phenomenon may be ascribed to the different dislocation configurations in specimens that dense dislocation cells consist of scattered small dislocations in cold-worked specimens and dislocation networks with a size of 100 nanometers in annealed specimens, as shown in section 3.1. It needs further investigation. The statistical results regarding irradiation defects show that the size of irradiation defect specimens decreases but the number density increases in cold-worked compared with annealed specimens. The results are obviously different with the evolution of irradiation defects when bubbles act as sinks.

The relationship between irradiation hardening and irradiation defects is depicted in the well-known dispersion hardening model, $\Delta H_v = \alpha \mu v \sqrt{ND}$, where $\alpha$ is the strength factor, which is dependent on the type and size of irradiation defects, $\mu$ is the shear modulus, $b$ is the Burgers vector of dislocation, and $N$ and $D$ are the average size and number density of irradiation defects, respectively [39, 57]. Also, in a previous paper, $\alpha$ showed strong size dependence, whereby it increased with the increasing size of irradiation defects [85]. Despite that, as reported, irradiation defects interact with dislocations in different ways, such as the pinning of or reaction with dislocations [86, 87], resulting in different irradiation hardening. Hence, the effect of dislocation on irradiation hardening is hard to investigate not only due to the complicated defect evolution but also the complicated means of interaction between dislocations and irradiation defects. The details have not yet been clarified, although many works have contributed. Hence, the effect of dislocations, especially those with different configurations, on the irradiation effect should be considered, if necessary or possible, in the following work. The effect of the total sink strength on hardening in all doses is shown in figure S4 (see the supplementary materials). At both doses, irradiation hardening decreases following a power-law relation with the increasing sink strength, although large deviations occurred at some points.
5. Conclusion

In the present work, irradiation hardening of V–5Cr–5Ti alloys was investigated. Fine-scale bubbles with high number density and dislocations were introduced in pristine specimens. The effectiveness of the calculated sink strength of bubbles and dislocations was investigated by comparing irradiation hardening. Pre-existing bubbles could effectively reduce irradiation hardening by decreasing the number density of irradiation defects. With the increasing sink strength of dislocations, irradiation hardening decreased. Despite the introduction of bubbles, materials under 80% cold work could reinforce the radiation tolerance accompanied by a slight loss of elongation.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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