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Irradiation Behaviors in BCC Multi-Component Alloys with Different Lattice Distortions

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Abstract: Recently, the irradiation behaviors of multi-component alloys have stimulated an increasing interest due to their ability to suppress the growth of irradiation defects, though the mostly studied alloys are limited to face centered cubic (fcc) structured multi-component alloys. In this work, two single-phase body centered cubic (bcc) structured multi-component alloys (CrFeV, AlCrFeV) with different lattice distortions were prepared by vacuum arc melting, and the reference of α -Fe was also prepared. After 6 MeV Au ions irradiation to over 100 dpa (displacement per atom) at 500 °C, the bcc structured CrFeV and AlCrFeV exhibited significantly improved irradiation swelling resistance compared to α -Fe, especially AlCrFeV. The AlCrFeV alloy possesses superior swelling resistance, showing no voids compared to α -Fe and CrFeV alloy, and scarce irradiation softening appears in AlCrFeV. Owing to their chemical complexity, it is believed that the multi-component alloys under irradiation have more defect recombination and less damage accumulation. Accordingly, we discuss the origin of irradiation resistance and the Al effect in the studied bcc structured multi-component alloys.

Keywords: multi-component alloys; body centered cubic (bcc); lattice distortion; irradiation swelling resistance; irradiation softening; chemical complexity

1. Introduction

Metals and alloys have been used for several thousands of years, since the Bronze Age [1]. Alloys are typically composed of one or two principal elements and several small quantities of other elements (minor elements) aimed at modifying microstructures and properties [2,3]. The conventional alloys are located in the corner of phase diagrams, and this greatly limits new alloy design. In 2004, Yeh et al. [4] and Cantor et al. [5] designed novel multi-principal element alloys with simple crystal structures called high entropy alloys (HEAs). HEAs are defined by Yeh as alloys consisting of five or more metal elements with equimolar or near-equimolar ratios. HEAs have been extensively researched for about two decades, and there are some derivatives from the initial definition, e.g., Ni-containing equiatomic alloys (fewer than five elements), which are called single-phase concentrated solid-solution alloys (SP-CSAs). SP-CSAs have been systematically researched, and they have distinct properties, such as slow energy dissipation stemming from low thermal and electrical conductivity and less irradiation-induced damage accumulation [6,7]. Owing to their random atomic arrangements without complex intermetallic phases, HEAs exhibit superior mechanical properties at cryogenic temperatures [8,9], stability at elevated temperatures [10,11], and good irradiation resistance under ion irradiation [12–14]. In this paper, we refer to the above alloys as multi-component alloys to prevent confusion.

The irradiation behavior of multi-component alloys is a relatively new and important topic in nuclear materials, and only a few irradiation studies on bcc multi-component alloys have been performed, e.g., Al_{1.5}CoCrFeNi [14], Ti₂ZrHfV_{0.5}Mo_{0.2} [15], W_{0.5}(TaTiVCr)_{0.5} [16,17]



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). under gas-ion irradiation, and TiVXTa (X = Nb, Zr, Cr) [18] and $W_{38}Ta_{36}Cr_{15}V_{11}$ [19] at low dose (<10 dpa).

Here we report a study of the irradiation behaviors in CrFeV and AlCrFeV multicomponent alloys with different lattice distortions. α -Fe was used as the reference sample. The two alloys can be classified as medium entropy alloys based on Yeh's report [20]. The AlCrFeV alloy with large lattice distortion shows superior swelling resistance compared to α -Fe and CrFeV. The AlCrFeV alloy exhibits scarce irradiation softening after Au ions irradiation to high dose at 500 °C.

2. Materials and Methods

In the present study, α -Fe and its alloys CrFeV and AlCrFeV with equiatomic compositions were synthesized as ingots with sizes of approximately Φ 30 × 10 mm via the arc-melting method starting from pure elements of high chemical purity (>99.99%) on a water-cooled copper hearth under an inert high-purity argon atmosphere. The ingots were flipped over and re-melted at least five times in order to obtain high homogeneity. The ingots were then cut into samples with sizes of 5 × 5 × 1.5 mm using electrical discharge machining. Thereafter, each sample was firstly grinded by abrasive paper, then mechanically polished with 0.05 µm colloidal silica suspension to obtain mirror-like surfaces.

Following polishing, the crystal structures of the samples were characterized by X-ray diffraction (XRD). The XRD measurements were performed on a Panalytical Empyrean instrument operated at 45 kV and 40 mA, with a scanning rate of 4° per minute from 35° to 90°. Imaging of the surface morphology and chemical analysis on the samples before irradiation were also performed on a FEI Quanta 200F field emission environmental scanning electron microscope (SEM) equipped with energy dispersive X-ray spectrometer (EDS).

The irradiation was performed at the 2×1.7 MV tandem accelerator facility, located in State Key Laboratory of Nuclear Physics and Technology at Peking University. Samples were irradiated by 6 MeV Au ions to a fluence of 1.5×10^{16} cm⁻² at 500 °C. The irradiation lasted for about 6 h, and the samples were cooled naturally to 60 °C in a vacuum target chamber after irradiation. The beam flux of Au ions was high (~ 7.5×10^{11} cm⁻²·s⁻¹), and the damage rate was sufficient owing to its large atomic weight, so we chose Au ions to obtain a high dose for the sake of time and cost. The temperature of 500 °C was selected as it is located in the medium temperature regime (0.3–0.6T_m) of the studied Fe-containing materials, and voids are the predominant visible features for irradiated bcc materials [21]. After irradiation, cross-section transmission electron microscopy (TEM) samples were prepared using focused-ion beam (FIB) milling down to tens of nm on a Helios G4 UX. TEM observations were carried out on a Tecnai F20 microscope operated at 200 kV with 0.205 nm point resolution. The SEM-EDS, FIB milling, and TEM observation were all performed at Electron Microscopy Laboratory of Peking University.

Nanoindentation tests were employed in this study to investigate the mechanical properties of α -Fe, CrFeV, and AlCrFeV. The tests were performed using an Agilent G200 (KLA Corporation, Santa Clara, CA, USA) nanoindenter with a Berkovich tip. A 5 × 5 indent matrix (each indent was 30 μ m apart) was utilized for each sample to reduce statistical error.

3. Results and Discussions

3.1. Structural, Microstructural, and Chemical Analysis

Figure 1a shows the room-temperature XRD patterns of the pristine studied materials. It can be seen that all the prepared materials have a single bcc solid-solution phase. We can see that the (110) diffraction peaks of CrFeV are particularly high, which depicts that texture exists in the CrFeV sample. The diffraction peaks shift towards a lower diffraction angle, which means the lattice is expanded. From the results of the XRD, the lattice constants of materials increased in the order α -Fe < CrFeV < AlCrFeV. After Au ions irradiation, grazing incidence XRD with grazing incident angle 3° (the penetration depth was about 600 nm) was performed with the irradiated samples, and the results are illustrated in

Figure 1b. Although the (200) diffraction peak of irradiated CrFeV is not obvious (because of coarse grain and irradiation induced partial atomic rearrangement), the irradiated studied materials still have bcc structures and no other phases formed. Here, the (211) diffraction peak is obviously higher than others, showing texture of another orientation in the irradiated CrFeV sample.



Figure 1. XRD results of (a) pristine α-Fe, CrFeV, and AlCrFeV and (b) irradiated samples.

The microstructure of the alloys after polishing and prior to irradiation are shown in Figure 2. It can be seen from the SEM back-scattered images that all alloys exhibit a coarse equiaxed grain structure with sizes ~200 μ m and not any other microstructural characters were found. The XRD and SEM results verify that CrFeV and AlCrFeV both have single bcc structures. Additionally, the inserted EDS-mapping results show that the distribution of these elements in the materials is extremely homogeneous. It should be noted that the alloys are nominal equiatomic alloys, and the actual contents were characterized by SEM-EDS. The measured atomic percent of elements are displayed in Table 1. Considering the element evaporation in the preparation process, the atomic percentages of elements are nearly equal.

For the two above CrFeV and AlCrFeV multi-component alloys, we used several geometric and thermodynamic parameters to assess the stability of the bcc phase. These parameters are applicable to all kinds of alloys, including binary alloys. Here, the atomic size difference δ ; Pauling electronegativity difference $\Delta \chi$; mixing enthalpy ΔH_{mix} ; ratio of entropy to enthalpy value Ω ; and valence electron concentration VEC were calculated as follows:

$$\delta = \sqrt{\sum_{i=1}^{n} c_i (1 - r_i / \bar{r})^2} (\bar{r} = \sum_{i=1}^{n} c_i r_i)$$
(1)

$$\Delta \chi = \sqrt{\sum_{i=1}^{n} c_i (\chi_i - \overline{\chi})^2} \ (\overline{\chi} = \sum_{i=1}^{n} c_i \chi_i)$$
(2)

$$\Delta H_{mix} = \sum_{i=1, i \neq j}^{n} \Omega_{ij} c_i c_j = \sum_{i=1, i \neq j}^{n} 4\Delta_{mix}^{AB} c_i c_j$$
(3)

$$\Omega = \frac{T_{\rm m} \Delta S_{\rm mix}}{|\Delta H_{\rm mix}|} \tag{4}$$

$$VEC = \sum_{i=1}^{n} c_i (VEC)_i$$
(5)

where \bar{r} and $\bar{\chi}$ are the average atomic radius and average Pauling electronegativity, and c_i , r_i , χ_i , and (VEC)_i are the molar ratio, atomic radius, Pauling electronegativity, and valence electron concentration of i-th element, respectively. The Δ_{mix}^{AB} is the mixing enthalpy of binary liquid AB alloys [22]. The atomic radius and Pauling electronegativity were reported in previous work [23–25]. Table 2 displays the calculated values of δ , $\Delta\chi$, ΔH_{mix} , ΔS_{mix} , T_m , VEC, and Ω in CrFeV and AlCrFeV alloys. It has been reported in previous studies that multi-component alloys possessing small values of δ (<6.6%), $\Delta\chi$ (~≤17.5%), near zero absolute values of ΔH_{mix} (–22 kJ/mol ~5 kJ/mol), and large values of Ω (~≥1.1) tend to form solid solutions, instead of intermetallic compounds. In addition, Guo et al. [24] concluded that the VEC could effectively predict the alloy's phase and found that the bcc phase was stable when the VEC was lower than 6.87. As we can see from Table 2, the VEC values of these two alloys both satisfy the bcc phase formation criteria, which are consistent with actual results.



Figure 2. SEM back-scattered images and EDS-mapping of (a) CrFeV, and (b) AlCrFeV.

Table 1. The measured atomic percent of elements in the CrFeV and AlCrFeV alloys (at %).

Alloy	Al	Fe	Cr	V
CrFeV AlCrFeV	$\stackrel{\backslash}{ 24.22\pm0.98}$	$\begin{array}{c} 32.48 \pm 1.23 \\ 24.40 \pm 0.75 \end{array}$	$\begin{array}{c} 33.07 \pm 1.01 \\ 24.67 \pm 0.99 \end{array}$	$\begin{array}{c} 34.45 \pm 0.97 \\ 26.71 \pm 0.88 \end{array}$

Alloys	δ	Δχ(%)	$\Delta H_{mix}(kJ/mol)$	$\Delta S_{mix}(J/mol \cdot K)$	T _m (K)	VEC	Ω
CrFeV	2.65	8.81	-4.44 -11.75	9.13	2037.67	6.33	4.19
AlCrFeV	5.84	8.70		11.53	1761.56	5.50	1.73

Table 2. Certain geometric and thermodynamic parameters of CrFeV and AlCrFeV multicomponent alloys.

3.2. Voids TEM Observation and Swelling

In the present study, Au ions irradiation firstly leads to many point defects in the cascade region induced by nuclear collisions, and then the point defects evolve to form other microstructural damage, such as black dots, dislocations/dislocation loops, faults, voids, and irradiation segregation. This microstructural damage has an obvious impact on the materials' mechanical properties. As mentioned in Section 2, the irradiated voids were our primary objects of observation at the fluence of 1.5×10^{16} cm⁻² at 500 °C.

Figure 3 displays the bright field (BF) TEM images of three FIB specimens and the void statistic results for α -Fe and CrFeV. The curve graph shown in Figure 3a displays the damage and implanted ion profiles in α -Fe, which were calculated with SRIM 2008 [26] based on the quick Kinchin–Pease option. The peak dose in α -Fe is about 108 dpa, and the dose rate was about 4.5×10^{-3} dpa/s. In our statistics, at least 300 clearly visible voids in the whole FIB specimen spanning across ~10 μ m were measured in the α -Fe FIB specimen, and all visible voids were counted in the CrFeV FIB specimen spanning across $\sim 6 \mu m$. No voids were observed in the AlCrFeV FIB specimen, indicating the alloy's strongest irradiation swelling resistance among the three samples. Figure 3d-f show the statistical results of void average diameter, void density, and void swelling at various depths in α -Fe, respectively, and Figure 3g–i show the same for CrFeV. As we can see, voids were distributed in a wide range (100–1300 nm) in α -Fe, and some voids were located far beyond the Au ions range, which means some vacancies move outside the range and then agglomerate to form voids. Although the average size of voids does not significantly change at different depths, the large voids mainly occurred at 200–600 nm, resulting in large void swelling in this range. In CrFeV, voids were distributed in a narrow range (100–400 nm) which implies the vacancy migration, and agglomeration was more local in CrFeV compared to α -Fe. Considering the maximum void swelling region, the swelling in α -Fe (200–600 nm) is ~1.70%, which is more than 100 times the swelling of ~0.013% in CrFeV (100-400 nm). For these two samples, there were no voids in the region near the surface (0–100 nm) because the surface is a huge sink for point defects. As for AlCrFeV, no voids were observed in the specimen. Here, clear experimental results show that the multi-component alloys have significantly improved void swelling resistance compared to pure reference metal, and the Al element in AlCrFeV has a significant effect on void swelling control.

We confirm that the Al element in AlCrFeV considerably affects the alloy's irradiated swelling resistance. Al has a larger atomic radius compared to the three 3d TM (transition metal) elements. The δ values are shown in Table 2, indicating that the lattice of AlCrFeV is much more distorted than that of CrFeV. Yang et al. [27] found that NiCoFeCrPd alloy with larger Pd atoms showed a stronger suppression effect on void growth than NiCoFeCrMn at elevated temperatures. Based on the above facts, we anticipate that vacancy migration in AlCrFeV is obstructed more by a heavily distorted lattice compared to CrFeV, which remains to be explored by simulated calculations.

In addition to the increased lattice distortion of AlCrFeV originating from the Al element, other effects of Al element in multi-component alloys are discussed here. Al inevitably enriches element species of an alloy. Jin et al. [7] reported that the irradiation swelling in Ni-based equiatomic alloys is in the order Ni > NiCo > NiCoCr > NiCoCrFe \approx NiFe > NiCoFeCrMn \approx NiCoFe. Although alloying with Fe can more effectively hinder defect evolution than alloying with Co, the results mainly showed that the more complex the composition, the lower the irradiation swelling. In addition, in Ni-based equiatomic

alloys, Zhang et al. [6] reported that materials' electrical and thermal conductivities decrease with more complex compositions. The large reduction of electrical and thermal conductivities in NiCoFeCr compared to pure Ni, NiFe, and NiCo shows a shrinkage of the electron mean free path, which means slow energy dissipation and increased defect recombination. Therefore, the least accumulated irradiation damage occurs in NiCoFeCr. In the present study, the irradiation swelling is in the order α -Fe > CrFeV > AlCrFeV, which is consistent with the phenomena discussed above.



Figure 3. BF TEM images of: (**a**) α -Fe (the yellow arrows show some voids beyond Au ions range); (**b**) CrFeV (the yellow arrows indicate voids for clarity); and (**c**) AlCrFeV (the yellow circles show some defect clusters in the irradiated region) irradiated by 6 MeV Au ions at a fluence of 1.5×10^{16} cm⁻² at 500 °C. (**d**–**f**) Void average diameter, void density, and void swelling at various depths in α -Fe and (**g**–**i**) in CrFeV.

Al brings about change of electron system to TMs. As a 3p metal element, Al holds three electrons on its outermost shell and has a high Fermi level, meaning a small work function and a high ionization tendency [28]. Combination of Al and TMs signifies that Al transfers electrons to TMs, so the Al-TM bonds become shorter than the sum of corresponding metallic radii. The phenomenon of bond shortening has been reported in some Al-TM compounds and glasses [29–32]. Because the electrical and thermal conductivities are closely related to the state of free electrons, the strong electron transfer among elements in AlCrFeV induces low electrical and thermal conductivities. We infer that the electrical and thermal conductivities of AlCrFeV are at a relatively low level compared to pure α -Fe and CrFeV. We therefore infer that the energy dissipation process in AlCrFeV is sustained for a longer period, more defects are eliminated, and less damage accumulates.

Al has a much lower melting point (T_m) than the three other TM elements, so the T_m of AlCrFeV is obviously lower than the value of CrFeV (as shown in Table 2). As is revealed in Reference [21], the vacancy formation energy E_v^f is approximately proportional to the T_m of the material ($E_v^f \approx T_m/1067$). Thus, AlCrFeV possesses lower E_v^f compared with CrFeV and a larger equilibrium thermal vacancy concentration C_v^0 , which is expressed as [33]:

$$C_{\rm v}^0 = \frac{1}{\Omega} \exp\left(\frac{S_{\rm v}^{\rm f}}{k}\right) \exp\left(-\frac{E_{\rm v}^{\rm f}}{kT}\right) \tag{6}$$

according to the common form of the void growth equation:

$$\frac{\mathrm{d}R}{\mathrm{d}t} = \dot{R} = \frac{\Omega}{R} \left[D_{\mathrm{v}} \left(C_{\mathrm{v}} - C_{\mathrm{v}}^{\mathrm{V}} \right) - D_{\mathrm{i}} C_{\mathrm{i}} \right] \tag{7}$$

where *R* is the radius of void, Ω is the atomic volume, *D* and *C* are diffusion coefficient and concentration of point defects (subscript v denotes vacancy, i denotes interstitial). C_v^V is the vacancy concentration at the void surface, which is expressed as:

$$C_{\rm v}^{\rm V} = \frac{1}{\Omega} \exp\left(\frac{S_{\rm v}^{\rm f}}{k}\right) \exp\left(-\frac{E_{\rm v}^{\rm f}}{kT}\right) \exp\left(-\frac{p\Omega}{kT}\right) = C_{\rm v}^{0} \exp\left(\frac{2\gamma\Omega}{RkT}\right) \tag{8}$$

where S_v^t is the vacancy formation entropy, k is the Boltzmann constant, T is the absolute temperature, and p is the surface tension of a void ($p = -\frac{2\gamma}{R}$, γ is the surface energy). Assuming other variables are nearly identical, we can deduce that a larger value of C_v^0 results in a larger value of C_v^V , and we then obtain a smaller $\frac{dR}{dt}$ value. The lower E_v^f therefore greatly slows void swelling. The similar mechanism of irradiation swelling resistance has been observed for electron-irradiated Fe-Cr-Ni solid solutions [34].

Due to the large atomic size of Al, it was combined with three other 3d TM elements to increase the alloy's lattice distortion, and the AlCrFeV alloy still maintained a single bcc phase. Al is a relatively cheap metal, and its combination with 3d TM elements is an economical choice for lattice distortion research. Here, we discuss the origin of void swelling resistance and the Al effect in multi-component alloys, which is helpful for the future design of irradiation resistant materials. Based on the above results, we can conclude that large lattice distortion, chemical complexity, change of electron system, and lower E_v^f of AlCrFeV play important roles in void swelling control.

3.3. Nanoindentation

For ion-irradiated samples, the nanoindentation test is one of few applicable characterization methods for mechanical properties because of the shallow irradiation region (~ μ m level). Load control was performed for all irradiated samples. The load was set as 5gf, and for comparison, the pristine samples were also tested. Figure 4a–c illustrate the representative load–unload curves for α -Fe, CrFeV, and AlCrFeV, respectively. Under the same load force, the irradiated samples of α -Fe and CrFeV are indented to a shallower depth, exhibiting larger hardness values compared with pristine samples. As for AlCrFeV, there is little difference between the load–unload curves of irradiated and pristine samples. Figure 4d illustrates a comparison of hardness before and after irradiation. The hardening rate was calculated by $\Delta H(\%) = 100(H_f - H_i)/H_i$, where H_f and H_i are the hardness of final and initial state. The hardening rate of α -Fe is close to 31%, and the value of CrFeV is about 12.69%, while the change of hardness in AlCrFeV is inverse. Not only is hardening not observed, but also slight softening (~1.15%) occurs in AlCrFeV.



Figure 4. Representative load–unload curves of: (**a**) irradiated and pristine α -Fe; (**b**) CrFeV; and (**c**) AlCrFeV. (**d**) Comparison of hardness.

Obvious hardening effects are observed in α -Fe and CrFeV, while a slight softening effect is found in AlCrFeV. Considering the void size and number density of α -Fe is much larger than that of CrFeV, and there are no voids observed in AlCrFeV, we conclude that void hardening plays an important role in hardening of materials. Although the change of hardness in AlCrFeV is contrary to our expectation, we speculate that the as-casted AlCrFeV with low T_m went through a "striking" annealing process after ion irradiation at 500 °C sustained for several hours, which resulted in a softening effect. A previous work [35] attributed irradiation softening in neutron irradiated molybdenum to small defect clusters with strain field. AlCrFeV possesses a large distorted lattice, so the interfaces between defect clusters (as shown in Figure 3c) and lattice have a large mismatch, which is the origin of strain field. There are no voids in AlCrFeV, so void hardening does not exist. This, of course, does not imply that no irradiation condition overpowers the hardening effect, so the softening effect in the current irradiation condition overpowers the hardening effect, so the softening effect, so the softening effect.

4. Conclusions

In the present study, we prepared CrFeV and AlCrFeV multi-component alloys, and the nominal equiatomic alloys both had near-equal molar ratios. The results of XRD and TEM verified that CrFeV and AlCrFeV had single bcc structures. Then, the alloys were irradiated with 6 MeV Au ions to over 100 dpa at 500 °C, and α -Fe was used as the reference sample.

Under the current irradiation condition, the voids in CrFeV were much smaller and sparse compared with α -Fe, while no voids were observed in AlCrFeV. Al in AlCrFeV brings about many changes, such as large lattice distortion, chemical complexity, change of electron system, and lower E_v^f , so AlCrFeV has excellent irradiation swelling resistance. In general, we discovered that multi-component alloys exhibit good irradiation resistance compared to pure reference metal, and the AlCrFeV alloy with larger lattice distortion performs better.

Based on the nanoindentation tests, the irradiated samples of α -Fe and CrFeV become harder compared to pristine samples. The hardening rate for α -Fe (~30.98%) is much larger than that of CrFeV (~12.69%). As for AlCrFeV, scarce irradiation softening is found, which is thought to be associated with sustained high temperature irradiation and large lattice strain field.

Author Contributions: S.X. and Y.W. conceived the idea of exploring the bcc structured novel multicomponent alloys. Y.S. carried out the Au ions irradiation and subsequent TEM observation and nanoindentation tests of the samples. J.H., Q.L., H.L. and C.W. made contributions to writing, reviewing, and editing. All of the authors discussed the results and reviewed the manuscript. All authors have read and agreed to the published version of the manuscript.

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