Scripta Materialia 144 (2018) 31–35

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

Effects of temperature on the irradiation responses of $Al_{0.1}CoCrFeNi$ high entropy alloy

Tengfei Yang [a,b], Songqin Xia [c], Wei Guo [d], Rong Hu [e], Jonathan D. Poplawsky [d], Gang Sha [e], Yuan Fang [a], Zhanfeng Yan [a], Chenxu Wang [a], Congyi Li [b], Yong Zhang [c], Steven J. Zinkle [b], Yugang Wang [a,*]

[a] State Key Laboratory of Nuclear Physics and Technology, Center for Applied Physics and Technology, Peking University, Beijing 100871, China
[b] Department of Nuclear Engineering, University of Tennessee, Knoxville, TN 37996, USA
[c] State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China
[d] Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA
[e] Herbert Gleiter Institute of Nanoscience, Nanjing University of Science and Technology, Nanjing 210094, China

ARTICLE INFO

Article history:
Received 12 August 2017
Received in revised form 16 September 2017
Accepted 17 September 2017
Available online xxxx

Keywords:
Irradiation effects
Structural damage
Chemical segregation
High-entropy alloy

ABSTRACT

Structural damage and chemical segregation in $Al_{0.1}CoCrFeNi$ high entropy alloy irradiated at elevated temperatures are studied using transmission electron microscopy (TEM) and atom probe tomography (APT). Irradiation-induced defects include dislocation loops, long dislocations and stacking-fault tetrahedra, but no voids can be observed. As irradiation temperature increases, defect density is decreased but defect size is increased, which is induced by increasing defect mobility. APT characterization reveals that ion irradiation at elevated temperatures can induce an enrichment of Ni and Co as well as a depletion of Fe and Cr at defect clusters, mainly including dislocation loops and long dislocations.

Published by Elsevier Ltd on behalf of Acta Materialia Inc.

High-entropy alloys (HEAs) are newly emerging advanced materials, which are defined as a multi-element solid solution composed of four or more principal elements in equimolar or near-equimolar ratios [1,2]. HEAs can exhibit high hardness, fatigue resistance, wear resistance and excellent low temperature fracture-resistance [3–6]. Specially, HEAs have a high softening resistance at elevated temperatures and sluggish diffusion kinetics. For these reasons, HEAs are widely regarded as promising high-temperature materials [7]. Due to their excellent mechanical properties and high temperature stability, HEAs have been proposed as structural materials in advanced nuclear systems [8,9], in which structural materials will be exposed to high temperatures and high irradiation doses.

Irradiation of alloys by energetic particles in nuclear reactors can cause serious structural damage, leading to the degradation of mechanical properties [10]. Therefore, the structural stability and mechanical properties of HEAs under irradiation are crucial for their applications in nuclear energy systems. Generally, irradiation-induced damage accumulation at room temperature is suppressed in HEAs [8], and the volume swelling induced by high temperature ion irradiation can be decreased by controlling the number and the type of alloying elements. As such, the volume swelling of FeCoCrMnNi HEA (<0.2%) is 30 times lower than that of Ni (~6.7%) under irradiation with 5 MeV Ni at 500°C [11]. Although the irradiation response of HEAs has been studied, most of the experimental results are focused on the effects of compositional complexity on the irradiation resistance. The evolution of structural damage and chemical segregation with irradiation temperature in HEAs, which are important for a fundamental understanding of defect behavior and in determining the temperature range for its applications in nuclear system, have not been systematically studied.

In the current work, a single phase HEA $Al_{0.1}CoCrFeNi$ [12,13] is chosen as a model system to study the effects of temperature on the irradiation responses of HEA. The HEA $Al_{0.1}CoCrFeNi$ samples were irradiated by 3 MeV Au ions at 6×10^{15} cm^{-2} (~31 peak dpa); the irradiation temperatures ranged from 250 °C to 650 °C. Irradiation-induced defects and compositional segregation were characterized by transmission electron microscopy (TEM) and atom probe tomography (APT), respectively. Our aims are to investigate defect evolution and chemical segregation of HEA under high temperature irradiations, and provide reliable experimental results for obtaining a deeper understanding of the defect behavior in HEAs and the applications of HEAs in nuclear systems.

$Al_{0.1}CoCrFeNi$ HEA used in this study was synthesized by arc-melting a mixture of pure metals in a Ti-gettered high-purity argon atmosphere [12,14]. The polished samples were irradiated with 3 MeV Au ions at 250, 350, 500 and 650 °C, respectively. The details of ion irradiation can be found in Supplementary material. The corresponding displacements per atom (dpa) was calculated using SRIM 2013 assuming a displacement threshold energy of 40 eV in the Kinchin-Pease option [15].

* Corresponding author.
E-mail address: ygwang@pku.edu.cn (Y. Wang).

https://doi.org/10.1016/j.scriptamat.2017.09.025
1359-6462/Published by Elsevier Ltd on behalf of Acta Materialia Inc.

The irradiation fluences were 6×10^{15} cm^{-2}; the corresponding peak damage is ~31 dpa. The SRIM calculation results of dpa and Au distribution are given in Fig. S1 in Supplementary material. TEM observations were conducted using a 300 keV, Tecnai F30 microscope. Cross-sectional TEM samples were prepared by mechanical polishing, followed by ion milling to form a wedge for electron transparency. Irradiation-induced solute segregation in $Al_{0.1}$CoCrFeNi was characterized using APT (CAMECA LEAP 4000× HR or CAMECA LEAP 4000× SI) with a 50 pJ laser energy, a repetition rate of 200 kHz, and a specimen temperature of 30 K.

Fig. 1 shows the bright field (BF) images and corresponding selected area electron diffraction (SAED) patterns of $Al_{0.1}$CoCrFeNi HEAs irradiated at the four different temperatures. All BF images in Fig. 1 were taken under two-beam BF conditions using a diffraction vector $g = (0\bar{2}2)$ near the [211] zone axis and SAED patterns were taken under the [100] zone axis. No voids were observed at any of the four different sample temperatures. Furthermore, only diffraction spots belonging to a fcc structure can be found in selected area electron diffraction (SAED) patterns, indicating that no phase decomposition or incoherent precipitation occurs. At 250 °C (Fig. 1(a)), most of the visible irradiation-induced defects are located at the peak damage region (450 nm–650 nm). A high density of dislocation loops and small defect clusters, whose nature can't be distinguished, are observed. High-resolution TEM characterization in this depth region (as shown in Fig. 2(a)) revealed two groups of dislocation loops lying on the $(\bar{1}11)$ plane (indicated by red arrows) and $(1\bar{1}1)$ plane (indicated by blue arrows), suggesting that these small dislocation loops are faulted loops on the {111} plane. Fig. 2(b) presents the magnified HRTEM image of a dislocation loop in Fig. 2(a), which demonstrates that the dislocation loops are interstitial-type with $\boldsymbol{b} = 1/3\langle 111\rangle$. The average size of dislocation loops in this region was 7.4 ± 2.0 nm. A few large defect clusters including dislocation loops and dislocation line segments can be observed in the shallower region from 350 to 480 nm, but the densities are much lower.

As irradiation temperature increases to 350 °C (Fig. 1(b)), the depth-dependent microstructure is qualitatively similar to that at 250 °C. The region containing the highest density of small defect clusters and dislocation loops occurs over a somewhat narrower range of depths as compared with that of 250 °C, and the depth range of the region which consists of a low density of large dislocation loops and dislocation line segments is increased. This suggests that a portion of the small defects start to grow or become unstable and dissociate with increasing temperature, but the defect characteristics in both regions are overall similar with that of 250 °C.

Fig. 1(c) shows the BF image of $Al_{0.1}$CoCrFeNi irradiated at 500 °C, and it can be observed that the defect morphology and density are significantly different as compared with that of 250 °C and 350 °C. The defect density in the deeper region (510 nm–620 nm) is significantly decreased but the defect size is increased, with a greater number of easily resolvable isolated dislocation loops. A further microstructure characterization of this region (as shown in Fig. 2(c)) reveals that most of

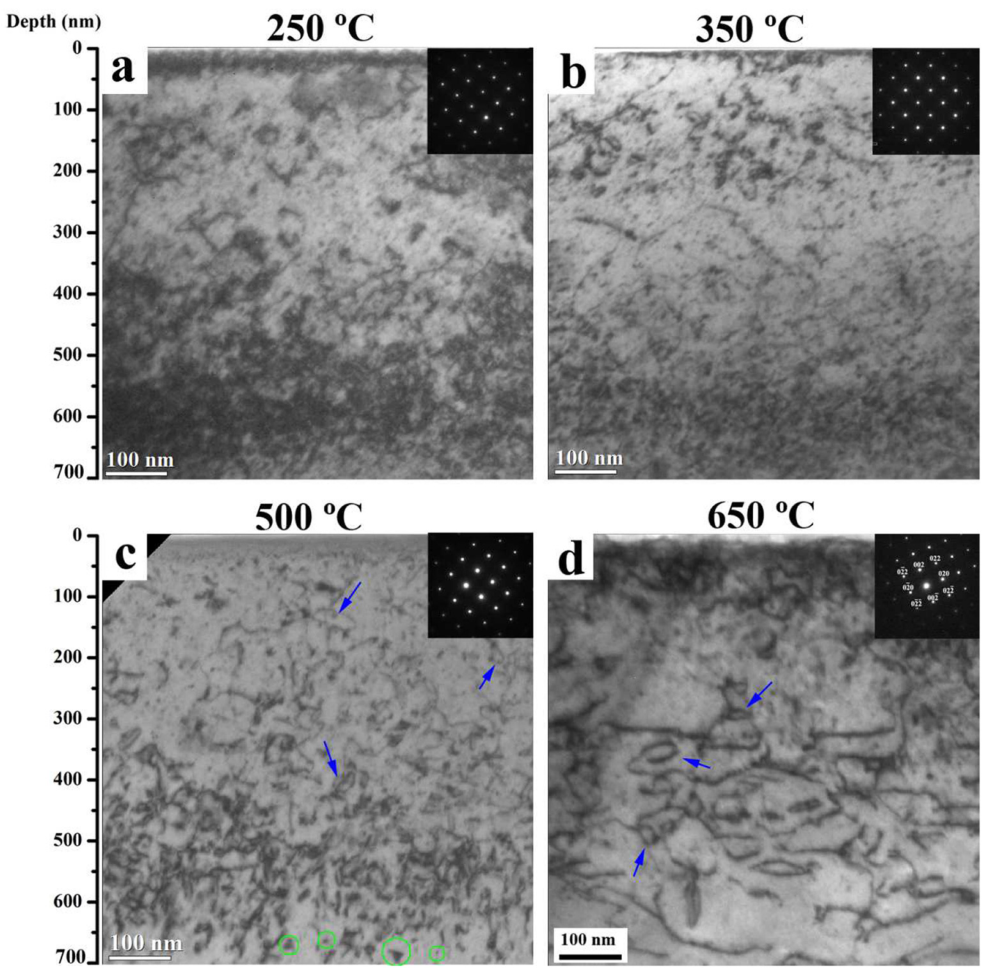

Fig. 1. BF images and corresponding SAED patterns of $Al_{0.1}$CoCrFeNi irradiated by 3 MeV Au ions to 6×10^{15} cm^{-2} at (a) 250 °C; (b) 350 °C; (c) 500 °C and (d) 650 °C, respectively. Perfect loops in (c) and (d) are marked by blue arrows and the green circles in (c) indicate SFTs. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)