



Irradiation tolerance of Co-free high entropy alloy: a quantitative microstructural evaluation of the effect of irradiation damage dose

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ABSTRACT

High entropy alloys (HEAs) are potential structural materials for next-generation nuclear systems due to their excellent high-temperature strength and corrosion resistance. However, their viability in extreme environments remains uncertain, necessitating a rigorous understanding of microstructural stability under irradiation. This work provides a comprehensive quantitative evaluation of microstructure as a function of irradiation fluence at 300 °C. Transmission electron microscopy revealed a dynamic disorder–order phase transformation (FCC-L1₂), resulting from the competition between irradiation-enhanced diffusion and ballistic dissolution. At low doses, pre-existing dislocations led to a heterogeneous distribution of dislocation loops. This effect became less pronounced at higher doses as the strength of competing defect sinks increased. Further irradiation facilitated the unfaulting of 1/3 <111> Faulted/Frank loops into 1/2 <110> Perfect loops, eventually evolving into a complex dislocation network. While void nucleation was suppressed at lower doses, discrete voids appeared at 10 dpa, due to vacancy supersaturation following enhanced interstitial absorption by the established dislocation network. Furthermore, Ni segregation at grain boundaries (initially driven by an interstitial drag mechanism) was less prominent at higher damage dose. This study offers mechanistic insight into irradiation induced microstructural evolution and the critical role of sink strength evolution in governing the long-term microstructural integrity of HEAs under irradiation.

1. Introduction

The realization of Generation IV fission reactors and future fusion energy systems hinges on the development of structural materials capable of enduring extreme environments. These materials must maintain dimensional stability and mechanical integrity under high displacement damage (exceeding 100 dpa in some designs), high operating temperatures (300–800 °C), and exposure to chemically aggressive coolants such as molten salts or liquid metals [1,2]. Conventional austenitic stainless steels, while well-understood and easily fabricated, exhibit a vulnerability to void swelling and radiation-induced segregation (RIS) [3]. At intermediate temperatures (0.3–0.5 T_{melting}), the accumulation of vacancies leads to the nucleation of voids, resulting in macroscopic volume expansion and a catastrophic loss of fracture toughness [4]. While ferritic-martensitic (FM) steels and oxide dispersion strengthened (ODS) alloys offer improved swelling resistance, they are limited by low-temperature embrittlement/poor high temperature

properties (above 550 °C) and high processing costs, respectively [5,6]. This creates a critical demand for a new class of radiation-tolerant alloys that bridge the gap between high-temperature strength and irradiation stability.

High entropy alloys (HEAs) and complex concentrated alloys (CCAs) are novel candidates for extreme environments, exhibiting exceptional high-temperature strength [7,8], wear resistance [9], and superior resistance against corrosion and oxidation [10–12]. Unlike traditional dilute alloys based on a single solvent element, HEAs leverage high configurational entropy to lower the Gibbs free energy, thereby stabilizing simple solid solutions particularly at higher temperatures [13]. However, intermetallic phases can still form in HEAs because of enthalpy driven effects [14]. Beyond initial thermodynamics, the enhanced radiation tolerance of HEAs is increasingly attributed to their unique core effects specifically severe lattice distortion and sluggish diffusion [15,16]. The extreme chemical disorder in these systems significantly reduces the electron and phonon mean free paths having a

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significant effect on defect formation energies and migration barriers. This resulting orders of magnitude decrease in thermal and electrical conductivity is hypothesized to extend the lifetime of the thermal spike immediately following a displacement cascade. A prolonged thermal spike enhances the probability of athermal recombination of Frenkel pairs, effectively reducing the number of surviving primary defects before they can aggregate into larger clusters [17,18]. Furthermore, the variable energy landscape created by local chemical fluctuations leads to sluggish diffusion, which acts as a kinetic barrier for the long-range migration of point defects. This complexity fundamentally alters defect trajectories, often forcing interstitial clusters into localized 3D motion rather than the long-range 1D glides characteristic of pure metals. This localization suppresses the growth of dislocation loops and delays the onset of void swelling by preventing the rapid supersaturation of vacancies [16,19,20].

While early research focused on equi-atomic/multi-principal systems like CoCrFeNi [16,17,21], CoCrFeMnNi [16,19,20,22] etc. practical nuclear application necessitates a transition to reduced activation compositions. The presence of Cobalt is a significant drawback due to the transmutation of ^{59}Co to ^{60}Co under neutron flux, a reaction that produces a long-lived radioactive isotope emitting high-energy gamma radiation, thereby complicating maintenance and increasing decommissioning costs [23]. To replace the role typically played by Co while enhancing the alloy's corrosion resistance, Aluminum (Al) could become a strategic addition. In Generation IV environments, the ability of a material to form a self-healing, protective oxide scale (such as Al_2O_3) is vital for resisting the corrosive nature of advanced coolants [24–26]. Furthermore, Aluminum acts as a potent solute for the formation of ordered intermetallic phases (e.g., L_{12}) which can provide excellent high temperature strength [27,28]. Under irradiation, the excess flux of vacancies and interstitials facilitates radiation-enhanced diffusion (RED), which can overcome the kinetic barriers that inhibit the nucleation of nanoprecipitates during standard thermal processing leading to radiation enhanced precipitation (REP) [29,30]. Additionally, irradiation can also result in the ballistic dissolution (BD) of these precipitates [31]. The dynamic competition between REP and BD plays a crucial role in long term stability of these precipitates under irradiation.

The macroscopic radiation tolerance of an alloy is fundamentally governed by its aggregate sink strength, which is defined by the density and efficiency of internal microstructural features such as pre-existing dislocations, grain boundaries, and phase interfaces in absorbing and annihilating point defects [31–34]. However, the evolution of these sinks is a highly dynamic process. During the initial stages of irradiation, point defects aggregate into isolated sessile $1/3\langle 111 \rangle$ Faulted/Frank dislocation loops which can unfault into glissile $1/2\langle 110 \rangle$ Perfect dislocation loops at higher doses or higher temperatures [29,35–37]. In conventional alloys, these loops rapidly grow and coalesce into complex dislocation networks. In contrast, in HEAs, this transition is significantly retarded by sluggish diffusion and the localized 3D motion of defect clusters [16,19]. The transition from a sink-limited regime to a saturated network regime represents a critical microstructural threshold that governs the onset of steady-state void growth during low temperature irradiation [33]. In systems exhibiting concurrent REP/BD, the microstructural interplay becomes increasingly sophisticated. Emerging precipitates act as additional, highly efficient sinks for recombination but also serve as potent obstacles that restrict dislocation mobility [29,35]. In addition to internal sinks like dislocations and phase interfaces, grain boundaries serve as highly effective sinks for irradiation-induced defects, establishing a defect flux that is strongly sensitive to the homologous irradiation temperature. The enrichment of Ni and Co at grain boundaries has been extensively documented in various HEAs, most notably in the equiatomic CoCrFeMnNi (Cantor alloy) and its derivatives [38]. This phenomenon is proposed to be governed by the inverse Kirkendall mechanism, a non-equilibrium process where radiation-induced fluxes of vacancies and interstitials toward defect sinks drive a redistribution of alloying elements. Because Ni and Co typically possess the

lowest self-diffusion coefficients in these complex systems they preferentially enrich at sinks such as grain boundaries while faster-diffusing species like Mn and Cr are depleted. This elemental redistribution is highly sensitive to the homologous irradiation temperature. It necessitates sufficient vacancy mobility to establish and sustain the required defect concentration gradients, a condition that is typically inhibited at room temperature but becomes prominent at intermediate temperatures (e.g., $0.3\text{--}0.5T_{\text{melting}}$) [3,39–41]. While the sluggish diffusion kinetics inherent to HEAs can possibly mitigate the overall magnitude of this segregation compared to conventional stainless steels, the persistent enrichment of Ni and Co raises a critical question into its susceptibility to irradiation-induced degradation.

Despite the documented potential of HEAs for extreme environments, a comprehensive mechanistic understanding of the void incubation period remains elusive, particularly regarding the transition from loop aggregation to late-stage nucleation in HEAs undergoing dynamic REP/BD. Also, while irradiation induced grain boundary segregation is well-documented, the collective evolution of competitive sinks, including dislocation networks and REP requires further exploration to determine their impact on aggregate sink strength.

This study tries to address these knowledge gaps using a novel Cobalt-free, Al-bearing HEA strategically designed to mitigate the radiological hazards of ^{60}Co . By subjecting this alloy to 5 MeV Fe^{2+} ion irradiation at 300 °C, we probe quantitatively the microstructural evolution across a range of fluences, providing insights into the unfaulting mechanism. Where sessile $1/3\langle 111 \rangle$ Faulted/Frank loops transform into glissile $1/2\langle 110 \rangle$ Perfect loops. Another focus of the study is to evaluate the role of irradiation damage dose on elemental segregation at the grain boundary, void incubation period and the role of REP/BD in defect microstructure evolution. This investigation seeks to elucidate how chemical complexity and dislocation microstructure can be leveraged to engineer advance materials for next-generation nuclear applications.

2. Experimental methods

2.1. Material processing

A novel Co-free HEA was designed as a part of the European project “INNUMAT” [42] and was synthesis using induction casting at the Tecnalia research centre in form of 7 Kg ingot [26]. The ingot was cut into smaller slices of thickness 2–2.6 cm followed by homogenization at 1100 °C for 4 h, subsequently at 1200 °C for 4 h and water quenched. The slices were further cold rolled to achieve a 50% reduction in the thickness by multiple passes. To mitigate the cold working induced hardening, recrystallization was carried out at 950 °C for 15 min. The composition of the alloy measured by scanning electron microscopy – energy dispersive x-ray spectroscopy (SEM-EDS) along with the nominal composition is summarized in Table 1.

2.2. Ion irradiation

The alloy was irradiated with 5 MeV Fe^{2+} ions at the Ion Beam Center (Helmholtz-Zentrum Dresden-Rossendorf). To ensure homogenous irradiation, a focused beam was raster-scanned at ~ 1 kHz in both the x and y directions over the $10 \times 5 \times 0.8$ mm samples. Irradiations were performed at 300 °C to fluences of 2.7×10^{15} , 8.1×10^{15} , and 2.7×10^{16}

Table 1

Chemical composition of the alloy measured by energy dispersive x-ray spectroscopy measurements.

Composition (at. %)	Cr	Fe	Mn	Ni	Al
Nominal	11.0	33.5	6.0	43.5	6.0
Measured	10.46 ± 0.82	34.35 ± 2.49	5.96 ± 0.5	43.84 ± 3.16	5.43 ± 0.43

ions/cm², corresponding to a damage level of 1, 3, and 10 dpa, respectively at a depth of 400–700 nm under an average pressure of 12.5×10^{-7} , 3.5×10^{-7} , 3.3×10^{-7} mbar for 1, 3, 10 dpa samples respectively. Damage profiles and ion ranges were simulated using the SRIM-2008 code [43] via the Quick Kinchin-Pease (K-P) method. Following the recommendations of Stoller [44], threshold displacement energies (E_d) were set at 25 eV for Al and 40 eV for all other constituent elements. The resulting displacement damage and ion implantation profiles are illustrated in Fig. 1. For quantitative microstructural and chemical characterization, an analysis zone between 400–700 nm was selected (highlighted in yellow, Fig. 1) to mitigate the influence of surface sinks and the injected interstitial effect from implanted ions.

2.3. Microstructural characterization

Microstructural characterization was performed using scanning electron microscope (SEM), electron backscattered diffraction (EBSD), and transmission electron microscope (TEM). EBSD mapping was conducted on a Zeiss Evo 10 SEM equipped with a Bruker e-flash^{HR} detector, operating at 20 kV and 1.5 nA. Cross-sectional TEM lamellae were prepared using an FEI Scios 2 Gallium ion Focused Ion Beam (FIB) system. To achieve electron transparency while minimizing surface damage, the accelerating voltage was systematically stepped down from 30 kV to 5 kV. Final surface cleaning was performed at 2 kV and 8 pA with a $\pm 5^\circ$ coincidence angle to eliminate ion beam induced artifacts (refer to Fig. S1 in supplementary materials for surface quality validation).

TEM was performed using an FEI Tecnai G2 G20 Twin microscope. Dislocation loop characterization included the determination of size distributions, Burgers vector *b* identification via *g.b* analysis, and the quantification of the relative fractions of $1/2 \langle 110 \rangle$ Perfect versus $1/3 \langle 111 \rangle$ Faulted/Frank loops using statistical method (refer to supplementary data). To avoid the misinterpretation of the FIB damages as irradiation induced defects, loops/black dots smaller than 2 nm were excluded from quantification. The loop nature (interstitial or vacancy) was established using the inside-outside contrast method [45,46]. These analyses were conducted under conventional two-beam Kinematic Bright-Field (KBF) and Weak-Beam Dark-Field (WBDF) conditions

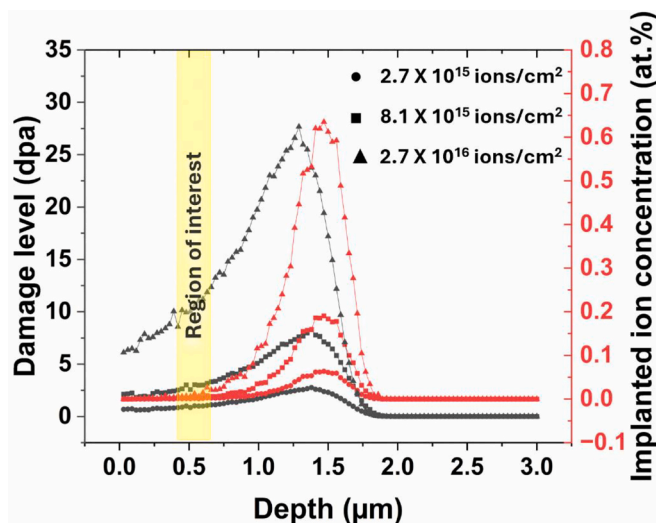


Fig. 1. SRIM simulations showing the depth profile of displacement per atom (dpa) and implanted ion concentration (at. %) irradiated with 5 MeV Fe²⁺ ions at 300 °C to 2.7×10^{15} , 8.1×10^{15} and 2.7×10^{16} ions/cm². The region 400–700 nm is chosen for in depth quantitative evaluation to avoid effect of free surface and implanted Fe²⁺ ions. The region of interest corresponds to ~ 1, 3, 10 dpa damage corresponding to 2.7×10^{15} , 8.1×10^{15} and 2.7×10^{16} ions/cm² fluences, respectively.

(g.3.1 g). For the identification of voids, a defocus series technique utilizing Fresnel fringes was implemented [47]. To facilitate accurate defect density calculations, the local thickness of the TEM lamellae was measured using electron energy loss spectroscopy (EELS). Chemical characterization and scanning transmission electron microscopy- energy dispersive x-ray spectroscopy (STEM-EDS) mapping were performed using a Thermo Scientific Talos F200X TEM equipped with a four-detector Super-X EDS system and subsequently processed using Velox software.

3. Results

3.1. Microstructure of pristine specimen

Metallographic characterization of the pristine specimen confirms a single-phase face-centered cubic (FCC) crystal structure. The SEM image (Fig. 2a) and inverse pole figure (IPF) (Fig. 2b) map reveals the absence of any pronounced crystallographic texture following recrystallization, indicating a nearly random grain orientation distribution. The area-weighted grain size distribution (inset of Fig. 2b) shows a limited number of coarse grains exceeding 200 μm, while the average grain size is 74.2 μm. Approximately 50.8% of the grain boundaries were identified as $\Sigma 3$ ($60^\circ \langle 111 \rangle$) twin boundaries (Fig. 2c). Based on their morphology, these boundaries are characteristic of annealing twins, which is indicative of low stacking fault energy (SFE) [48,49]. Kernel average misorientation (KAM) maps (Fig. 2d) indicate the presence of residual deformation induced strain within a few larger grains, suggesting that recrystallization is not fully completed.

Fig. 3a presents an inverted high-angle annular dark-field (HAADF) STEM image acquired along the [011] zone axis, revealing a non-uniform distribution of dislocations confined to specific slip planes. This indicates pronounced localized planar slip, a behaviour typically attributed to glide plane softening [50]. In complex alloys, this phenomenon is frequently correlated with the presence of short-range order (SRO) [51]. The dislocation density was estimated to be $2.17 \pm 0.89 \times 10^{13} \text{ m}^{-2}$ using the line intercept method [52]. As shown in Fig. 3b, the microstructure contains few stacking faults alongside dislocations exhibiting “curvy” slip trajectories. This morphology suggests dislocation pinning, likely resulting from local chemical fluctuations associated with SRO [50]. The inset in Fig. 3b displays a selected area electron diffraction (SAED) pattern consistent with a single-phase FCC crystal structure ($a_0 = 0.369 \text{ nm}$). Notably, despite the mechanical evidence of SRO, no diffuse scattering is observed in either the SAED pattern or the Fast Fourier Transform (FFT) derived from the high resolution (HR) TEM imaging (Fig. 3c) suggesting the SRO is below nm scale. Furthermore, STEM-EDS elemental mapping (Fig. 3d), the line scan taken across the grain boundary (supplementary Fig. S2) and high-resolution elemental mapping (supplementary Fig. S3) confirms nanoscale chemical homogeneity, with no detectable elemental segregation at grain boundaries or within the grain interiors.

3.2. Microstructure of irradiated specimens

The irradiation-induced defect depth distribution across the three fluences is illustrated in Fig. 4a–c. In all cases, the affected region extends to a depth of approximately 1.5 μm, which is in close agreement with the peak damage depth predicted by SRIM simulations (Fig. 1). While the 1 dpa sample exhibits a distinct, non-homogeneous distribution of dislocation loops (Fig. 4a), the defect structure in the 3 and 10 dpa samples (Fig. 4b, c) appears significantly more homogeneous (1, 3, 10 dpa samples correspond to the 3 fluences). To investigate phase stability, SAED was performed on the irradiated regions (red rectangles in Fig. 4), the presence of additional $\{100\}$ and $\{110\}$ superlattice reflections along with the fundamental FCC reflections confirms an irradiation-induced transformation to an ordered L1₂ phase. Since SAED patterns from the unirradiated zones (green rectangles in Fig. 4) reveal

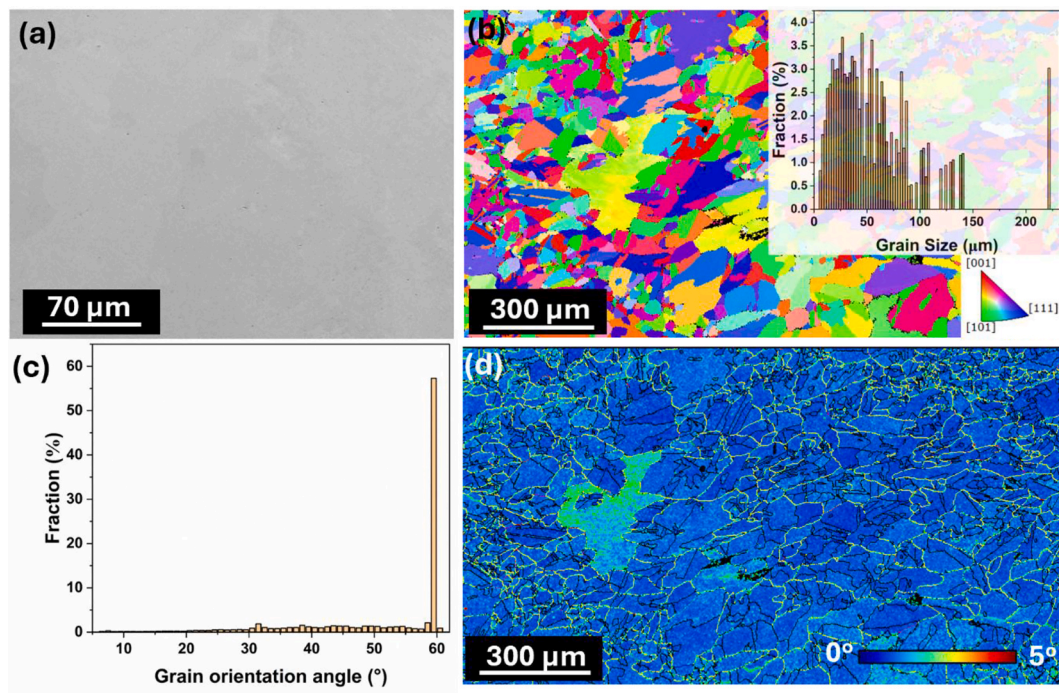


Fig. 2. Microstructural characterization of pristine HEA. (a) Secondary electron (SE) image of the pristine HEA. (b) Inverse pole figure (IPF) map of the recrystallized HEA, indicating a random crystallographic orientation with no strong texture; the inset shows the area-weighted average grain size distribution. (c–d) Grain boundary orientation angle distribution highlighting a high fraction of $\Sigma 3$ (60° @ $\langle 111 \rangle$) twin boundaries. In the kernel average misorientation (KAM) maps, black lines denote $\Sigma 3$ twin boundaries, while yellow lines represent general grain boundaries with misorientations $> 5^\circ$.

only fundamental FCC reflections despite 300°C isothermal exposure, it is evident that the $L1_2$ ordering transformation is driven by radiation-enhanced kinetics rather than purely thermal activation. While sub-nanometer SRO below the spatial or contrast detection limit of SAED may still exist within these control regions, extensive diffraction characterizations across multiple specimen depths and grain orientations (Supplementary Fig. S4) confirm that no long-range $L1_2$ ordering or thermal phase transformation occurs at 300°C .

The evolution of irradiation-induced defects as a function of depth is illustrated in Fig. 5, where magnified regions facilitate a comparative analysis between the near-surface (0–300 nm), the bulk-representative (400–700 nm), and the peak damage (1.2–1.3 μm) zones. In the near-surface region (Fig. 5a'–c'), the proximity to the free surface, which acts as a potent unbiased sink for point defects, results in the formation of relatively larger dislocation loops despite the lower local displacement damage dose. This surface-driven modification of the defect population renders the near-surface quantification non-representative of the bulk irradiation response. At the peak damage region at a depth of 1.2–1.5 μm (Fig. 5a'''–c'''), the microstructure transitions from a discrete loop population in 1 dpa sample to a high-density, interconnected dislocation network in 3 and 10 dpa samples. However, interpretation of this peak region is complicated by the significant accumulation of implanted ions, which according to SRIM simulations (Fig. 1), increases from ~ 0.06 at. % to ~ 0.6 at. % across the tested fluences. This substantial shift in local stoichiometry likely alters the defect mobility and SFE of the alloy, further complicating the direct correlation between displacement damage (dpa) and the observed microstructural evolution at these depths. Consequently, the 400–700 nm depth (Fig. 5a''–c'') was utilized for primary defect quantification to avoid surface-sink artifacts and the implanted ions effect. At the maximum damage dose of 10 dpa, the peak concentration of implanted Fe ions is approximately 0.2 at%. Because this is a self-ion irradiation, this negligible concentration does not significantly alter the local chemical composition or artificially influence the observed microstructural evolution.

Microstructural analysis of the regions of interest corresponding to a damage of 1, 3, and 10 dpa (Fig. 5a''–c'') reveals a clear evolution in defect morphology. The loops size distribution at the depth of interest is presented in Fig. 6. At 1 dpa, the dislocation loop distribution is notably non-homogeneous. In regions lacking pre-existing dislocation lines, loops are smaller (~ 5 nm) but more densely packed. Conversely, regions containing pre-existing lines exhibit larger loops (~ 7 nm) at lower densities (Table 2). As the irradiation dose increases to 3 dpa, the average loop size increases significantly to ~ 18 nm before decreasing to ~ 12 nm at 10 dpa. While loop and loop line density remains within the same order of magnitude across these doses, it follows a similar non-monotonic trend as the loop size (Fig. 7). In contrast, the dislocation line density exhibits a continuous, monotonic increase with cumulative irradiation dose.

The representative KBF micrographs for all irradiation conditions are presented in Fig. 8, featuring pairs of images captured from the same region using two distinct vectors near the same zone. To unambiguously identify the Burgers vectors of the highlighted loops (labeled A–D), the standard $g \cdot b$ invisibility criterion was applied in conjunction with simulated loop projections along the corresponding zone axes [53]. As an illustrative case, Fig. 8a and a' display KBF images obtained under $g = [020]$ and $g = [220]$ conditions along the $[001]$ zone axis, respectively. Loop A is clearly resolved under $g = [020]$ but exhibits invisibility (or vanishingly weak contrast) under $g = [220]$. Based strictly on the $g \cdot b$ criterion, the possible b vectors for loop A include $\pm 1/3 [1\bar{1}1]$, $\pm 1/3 [-8111]$, or $\pm 1/2 [-110]$. However, by comparing the experimental morphology with the simulated projection of the loop along the $[001]$ zone axis [53], the loop was definitively indexed as either $\pm 1/3 [1\bar{1}1]$ or $\pm 1/3 [-111]$ (Fig. 8a" and a1).

Following the systematic statistical analysis of dislocation loops (for more details refer to supplementary data), a distinct trend in loop character emerges across the dose range. At the lower dose of 1 dpa, there is a high population of faulted loops (Table 2). This fraction undergoes a slight reduction as the dose increases to 3 dpa, suggesting a potential transition or un-faulting process as the defect density evolves.

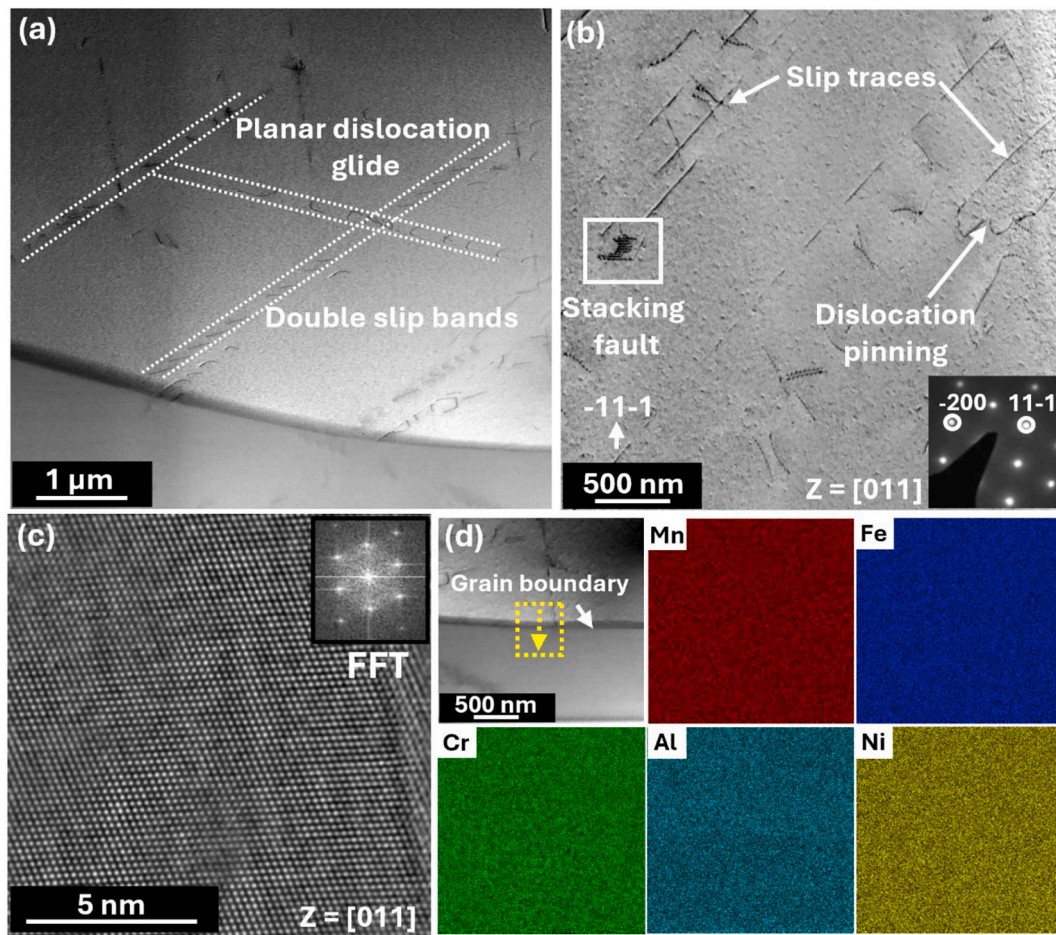


Fig. 3. TEM characterization of the pristine HEA. (a) Inverted HAADF micrograph highlighting planar dislocation glide. (b) Representative KBF TEM image, showing the presence of stacking faults and dislocation pinning. The inset displays the SAED pattern recorded along the [011] zone axis. (c) HR-TEM image captured along the [011] zone axis; the inset displays the corresponding FFT. (d) STEM-EDS mapping and line scan (yellow box) across a grain boundary, confirming the absence of any elemental segregation.

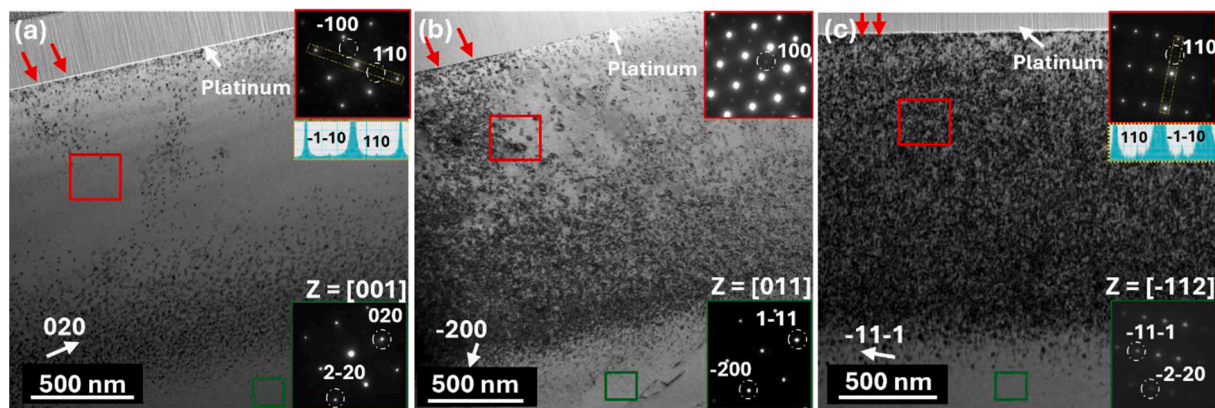


Fig. 4. Overview TEM micrographs of regions irradiated at three different fluences. Red arrows indicate the direction of irradiation. Panels (a–c) show two-beam KBF micrographs taken along the [001], [011], and $[-112]$ zone axes for the 1, 3, and 10 dpa samples, respectively. Insets display SAED patterns from the irradiated (red box) and unirradiated (green box) regions along with the line intensity profile marked with yellow box showing presence of additional reflections.

Interestingly, as the damage reaches 10 dpa, the fraction of faulted loops increases once more, likely because of the evolution of the dislocation network. The quantitative distribution and statistical breakdown of perfect vs. faulted loops for all irradiation conditions are summarized in Table 2.

To understand the microstructural evolution under irradiation, it is

critical to determine the nature (vacancy or interstitial) of the observed Frank loops. In this study, the inside-outside contrast technique [46] was employed on the 3 dpa sample. Fig. 9(a, a') and (b, b') illustrate three Frank loops (marked with red circles) and a perfect loop (marked with a blue circle). The direction of the Burgers vector was determined using simulated projections of the loops imaged near the [111] zone axis [53],

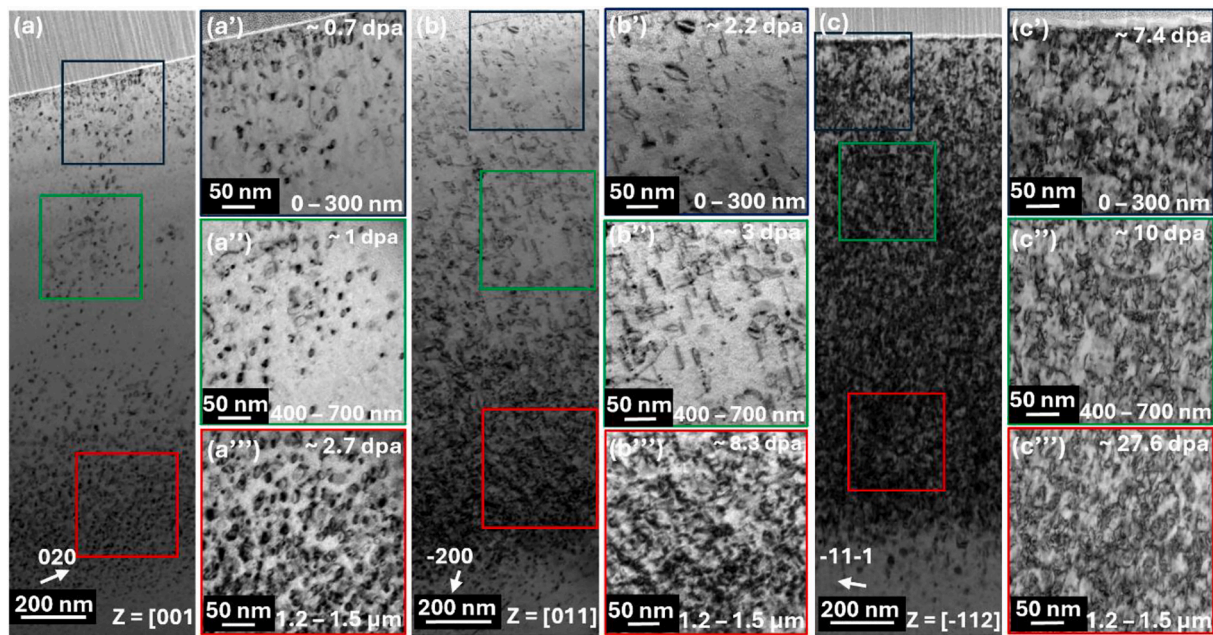


Fig. 5. (a–c) Two-beam KBF micrographs illustrate the microstructure at three depths: the near surface (0–300 nm, blue box), the region of interest (400–700 nm, green box), and the peak damage zone (1.2–1.5 μm , red box). SRIM calculated doses for each region are indicated in the top-right corner of the respective images.

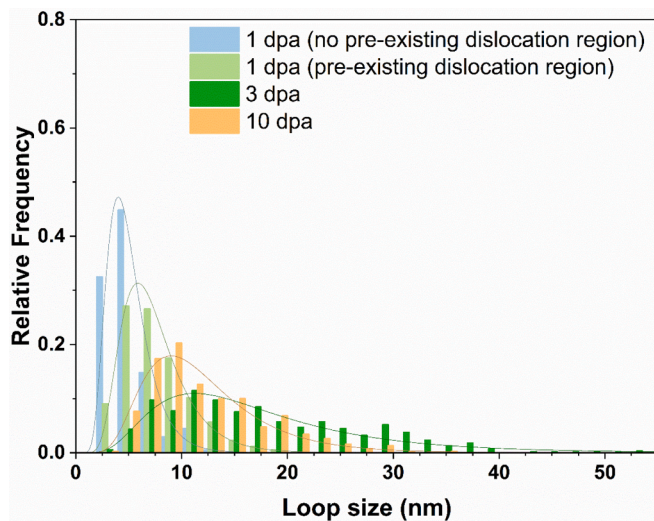


Fig. 6. Dislocation loop size distributions for 1, 3, and 10 dpa samples. Due to the non-homogeneous spatial distribution of loops in the 1 dpa sample, two separate regions were analyzed to ensure statistical accuracy. Each histogram is fitted with a log-normal distribution to determine the mean loop size and corresponding standard deviation.

as presented in Fig. 9c.

The identification of the Burgers vector $\mathbf{b}$ relies on the relationship between the diffraction vector $\mathbf{g}$ and the deviation parameter (s_g). For $s_g > 0$: ($\mathbf{g} \cdot \mathbf{b}$). $s_g < 0$ results in inside contrast (image smaller than the actual defect). And ($\mathbf{g} \cdot \mathbf{b}$). $s_g > 0$ results in outside contrast (image larger than the actual defect). By adopting the Finish-to-Start/Right-Hand (FS/RH) convention and defining the line direction $\mathbf{u}$ as clockwise, a Burgers vector pointing “upward” (parallel to the loop plane normal $\mathbf{n}$) defines an interstitial-type loop. In the present analysis, the condition $\mathbf{b} \cdot \mathbf{z} < 0$ was satisfied, indicating that $\mathbf{b}$ points upward in the same direction as the loop plane normal. Consequently, the Frank loops are confirmed to be interstitial in nature (Fig. 9d). For Loop C, a Perfect loop with $\mathbf{b} = \pm 1/2[110]$, the angle between $\mathbf{b}$ and $\mathbf{z}$ is 90° which is $>55^\circ$ the

Table 2

Summary of dislocation loop quantitative analysis, including number density and the respective fractions of Perfect and Faulted loops. The mean loop sizes, derived from the distributions in Fig. 6, are also provided for each sample.

Irradiation condition	1/3(111) loop density (m^{-3})	1/2(110) loop density (m^{-3})	Total loop density (m^{-3})	Fraction of 1/3(111) loops (%)	Mean loop size (nm)
1 dpa (no prior dislocation region)	$8.97 \pm 2.53 \times 10^{21}$	$2.54 \pm 2.52 \times 10^{21}$	$11.52 \pm 0.14 \times 10^{21}$	77.87 ± 21.48	5.04 ± 2.04
1 dpa (prior dislocation region)	$4.87 \pm 0.63 \times 10^{21}$	$2.62 \pm 0.62 \times 10^{21}$	$7.49 \pm 0.31 \times 10^{21}$	65.01 ± 7.16	7.46 ± 3.11
3 dpa	$6.48 \pm 0.91 \times 10^{21}$	$3.68 \pm 1.11 \times 10^{21}$	$10.15 \pm 0.54 \times 10^{21}$	63.8 ± 10	17.85 ± 10.74
10 dpa	$6.77 \pm 0.61 \times 10^{21}$	$1 \pm 0.6 \times 10^{21}$	$7.77 \pm 0.35 \times 10^{21}$	87.11 ± 7.72	12.16 ± 5.73

conditions were found to be “unsafe” or ambiguous for definitive characterization.

As previously noted, the appearance of superlattice reflections from $\{100\}$ and $\{110\}$ planes in the SAED patterns confirms an irradiation-induced phase transformation from a disordered FCC matrix to a chemically ordered L_{12} phase (Fig. 4). To visualize the spatial distribution of these ordered domains, Dark field (DF) micrographs were captured using these superlattice reflections from the depth of interest (400–700 nm). As shown in Fig. 10, these images reveal the morphology of the L_{12} precipitates across all irradiation conditions. The quantitative evolution of these features is summarized in Fig. 10d and Table 3. It is important to note that precipitates smaller than 1 nm were excluded from the analysis due to the inherent challenges of accurately quantifying sub-nanoscale defects in a 100 nm thick TEM lamella. At this thickness, the interaction of the diffracted beam along the sample depth and limited contrast for features nearing the resolution limit, complicates the detection of small defects. Nevertheless, the remaining data provides a clear qualitative trend. Table 3 reveals a non-monotonic

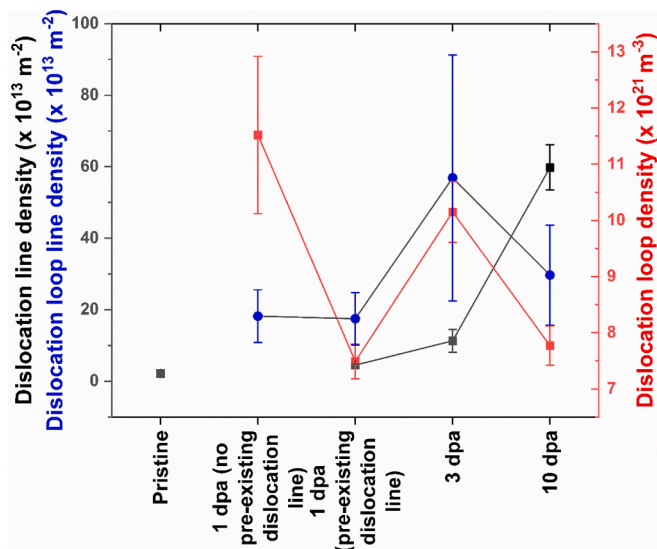


Fig. 7. The graph illustrates the quantitative evolution of dislocation line, loop and loop line densities across all experimental conditions, highlighting the correlation between irradiation dose and defect accumulation.

response of precipitate size and number density to increasing dose. Specifically, from 1 to 3 dpa, the average precipitate size decreases from ~ 3 nm to ~ 2.5 nm while the number density increases, suggesting a regime dominated by irradiation-induced fragmentation or enhanced nucleation. However, at 10 dpa, this trend reverses, the average size recovers to ~ 3 nm and the number density declines to levels comparable to the 1 dpa state. This behavior underscores a dynamic competition between ballistic dissolution, where high-energy displacement cascades physically dissolves precipitates and radiation-enhanced diffusion, which promotes subsequent reprecipitation and coarsening. This cyclic evolution provides critical insight into the phase stability of the alloy under high irradiation damage doses. The chemical composition of the L_{12} precipitates was further investigated using STEM-EDS mapping (supplementary data Fig. S5). The simultaneous enrichment of Ni and Al, accompanied by the localized depletion of the matrix elements, confirms that these ordered features are most likely Ni_3Al type precipitates formed during the irradiation process.

The chemical stability of grain boundaries under irradiation was evaluated using STEM-EDS to quantify the extent of radiation-induced segregation (RIS) (Fig. 11a–c). Consistent with the RIS mechanisms commonly observed in nickel-based FCC alloys, the elemental maps reveal a localized enrichment of Ni and a corresponding slight depletion of Mn, Fe and Cr in the immediate vicinity of the grain boundaries. This chemical partitioning was further quantified via EDS line profiles at the depth of interest (supplementary Fig. S6), which confirms the inverse correlation between Ni and Mn, Fe concentrations across the boundary interface (Table 4).

The magnitude of radiation-induced segregation exhibits a non-linear dependence on dose. Initially, the Ni enrichment amplitude increases from ~ 2.5 at. % at 1 dpa to ~ 4.0 at. % at 3 dpa. However, at 10 dpa, the measured peak amplitude decreases to ~ 1.5 at. %. Rather than a true reduction in total segregant, this apparent decrease is attributed to significant broadening of the segregation profile. It should be noted that the measured amplitude and profile width are highly sensitive to electron beam-specimen geometry, minor deviations from a perfectly edge-on grain boundary alignment within the TEM lamella can artificially exacerbate this apparent peak broadening. Nevertheless, a similar dose-dependent evolution is observed for the segregation profiles of Mn, Fe, and Cr, whereas Al shows no preferred segregation/depletion. Ultimately, this profile broadening and subsequent saturation suggests that the local grain boundary chemistry has reached a steady-state condition,

where the defect-driven solute flux is effectively balanced by thermal back-diffusion and the competing influence of a high defect sink density within the matrix. To eliminate thermally driven segregation/depletion because of exposure to 300°C during irradiation, STEM-EDS mapping and a line scan across the grain boundary in the unirradiated region of the 10 dpa sample was performed (Fig. S7 & S8). Which shows no elemental segregation/depletion due to thermal exposure alone.

In addition to dislocation loops and elemental segregation, the evolution of voids (vacancy clusters) was investigated as a function of irradiation dose. To accurately resolve these features, off-axis focus series TEM imaging was conducted (Fig. 12a–c). This approach effectively suppresses the strain-contrast originating from the dense dislocation network and loops, allowing for a clearer observation of the underlying void morphology.

The presence of voids was confirmed using the Fresnel contrast technique, where the features exhibit characteristic light or dark fringes depending on the applied over-focus or under-focus values. While no detectable voids were observed in the 1 dpa and 3 dpa samples, a density of small, randomly distributed voids, ranging from 1 to 10 nm in diameter are observed in the 10 dpa material. A high-magnification view of a representative void, located at the depth of interest corresponding to 10 dpa dose, is presented in insets of Fig. 12c. Since the voids are sparsely distributed across the depth and the number is extremely low, providing a quantitative assessment would be statistically inaccurate.

The absence of significant void formation at lower doses, followed by their appearance at 10 dpa, suggests that a threshold dose is required for the nucleation and stable growth of vacancy clusters within this specific alloy matrix. However, it is important to consider that a population of sub-nanometer vacancy clusters may exist below the resolution limit of the TEM. These invisible clusters could act as precursors for the larger voids observed at higher doses and likely contribute to the overall radiation-induced hardening of the material despite their lack of visible contrast.

4. Discussion

4.1. Dose-dependent evolution of the dislocation microstructure

4.1.1. Role of pre-existing dislocations and non-homogeneous loop distribution (1 dpa)

At the early stage of irradiation (1 dpa), the microstructure exhibits a pronounced non-homogeneous distribution of dislocation loops. This spatial variation is primarily dictated by the pre-existing dislocations present in the pristine material, which are non-homogeneously distributed within the grain due to dislocation channeling (as shown in Fig. 3a, b). According to dislocation bias theory [54,55], these pre-existing lines act as potent primary sinks for irradiation-induced point defects. As interstitials possess a significantly larger elastic strain field than vacancies, they experience a stronger long-range elastic attraction to the edge components of mixed dislocation lines [54]. In contrast, Chang et al. [56] found that the core of screw dislocation segments often exerts a relatively repulsive force on incoming interstitials due to the strong compressive forces and acts as a preferential sink for vacancies [57]. Consequently, vacancies are more prone to be absorbed by these screw components core of mixed dislocations where the interstitial bias is minimized leading to a dislocation climb, resulting in the evolution of these lines into the helical dislocation structures [58,59]. Similar helical dislocation structure is observed at 1 dpa as shown on Fig. 13a. This differential absorption of vacancies leads to a localized supersaturation of interstitials around the dislocation leading to the formation of interstitial loops. These interstitial loops can act as a competitive sink once they reach a critical size for other interstitials and grow which leads to the observation of larger loops and lower density in the vicinity of these dislocation lines as compared to the matrix (as shown in table 2). In contrast, the matrix (Fig. 13b) which lacks these high-strength primary sinks maintains a higher, uniform supersaturation of both species. The

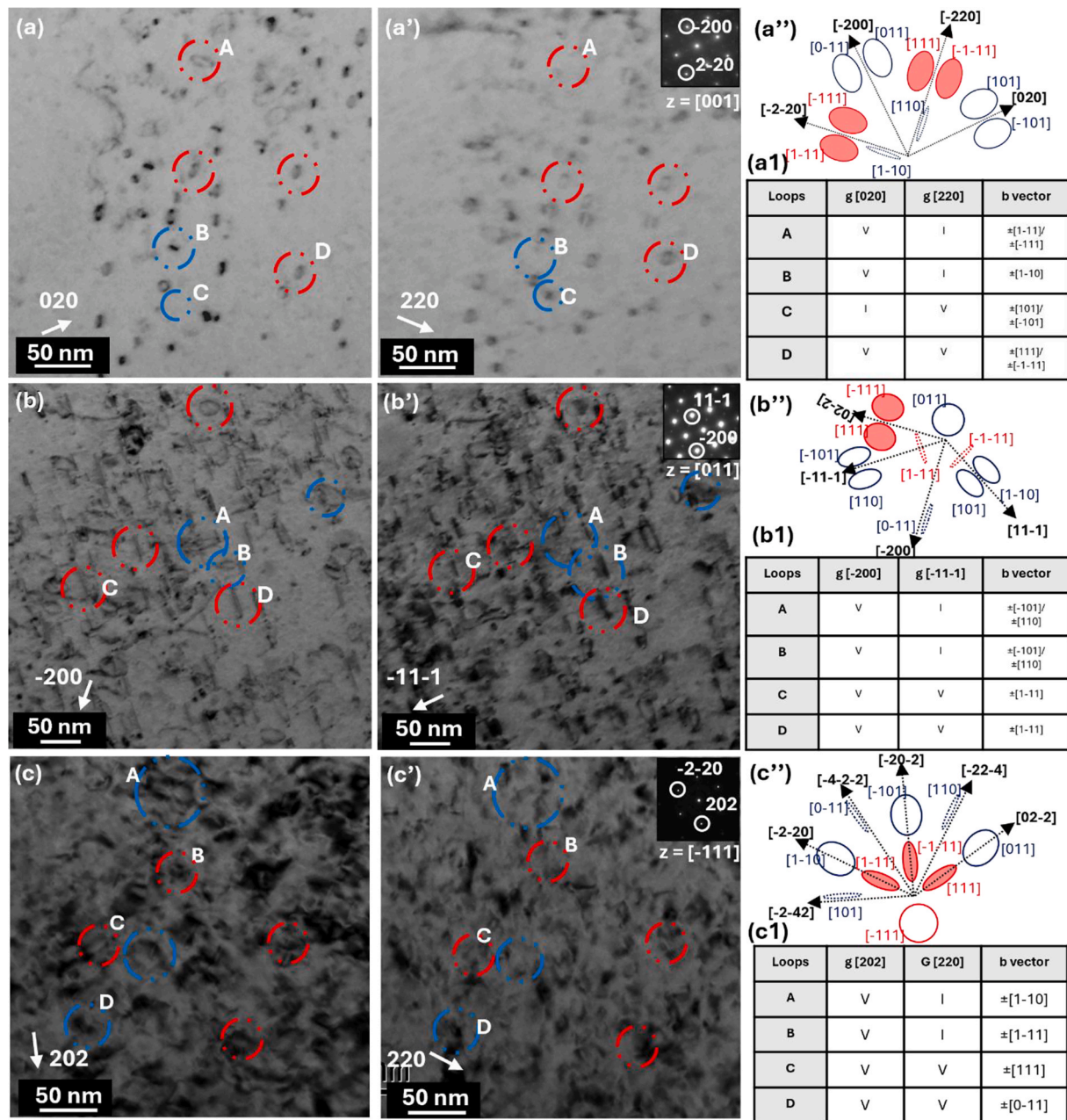


Fig. 8. Determination of dislocation loop Burgers vectors: (a, a'), (b, b'), and (c, c') show two-beam KBF micrographs captured using distinct g vectors to identify the Burgers vectors of the observed loops. Each pair corresponds to the same region under different diffraction conditions. The SAED patterns in the upper-right corners of (a'), (b'), and (c') indicate the respective zone axes. Panels (a'')–(c'') provide the simulated loop projections for these zone axis orientations adopted from [53]. (a1)–(c1) highlight the analysis of specific loops based on the $g \cdot b$ invisibility criterion and geometric projection; red circles denote faulted loops, while blue circles indicate perfect loops.

smaller size of these matrix loops is attributed to the irradiation temperature of 300 °C where vacancies have limited mobility and the suggested sluggish diffusion characteristics resulting from high local lattice distortion in HEAs [60,61]. In these sink-free regions, the restricted long-range mobility of interstitials, coupled with the trapped vacancy population, significantly increases the probability of mutual recombination [62]. The high recombination rate effectively limits the net number of interstitials available for sustained loop growth, resulting in the observed higher density of smaller defects. Additionally, the higher deviations in the density of the perfect loops could be associated with the local chemical variations within the matrix.

4.1.2. Loop morphology and the unfaulting mechanisms (3 dpa)

At a cumulative damage level of 3 dpa, the microstructural evolution shifts from being initially governed by pre-existing sinks to being dominated by the nucleation and growth of a dense population of irradiation-induced defects. While the defect density remains within the same order of magnitude as the 1 dpa state, the average loop diameter undergoes a significant increase, increase from ~ 5 –7 nm to ~ 18 nm. This suggests that the microstructure has transitioned into a growth-dominant regime where the high density of nascent loops establishes a competitive and globally uniform sink strength. Which is well reflected by the increased dislocation loop sink strength from 1 dpa to 3 dpa (Table 5). Consequently, the localized influence of the initial dislocation is eclipsed, leading to the observed homogenization of the defect

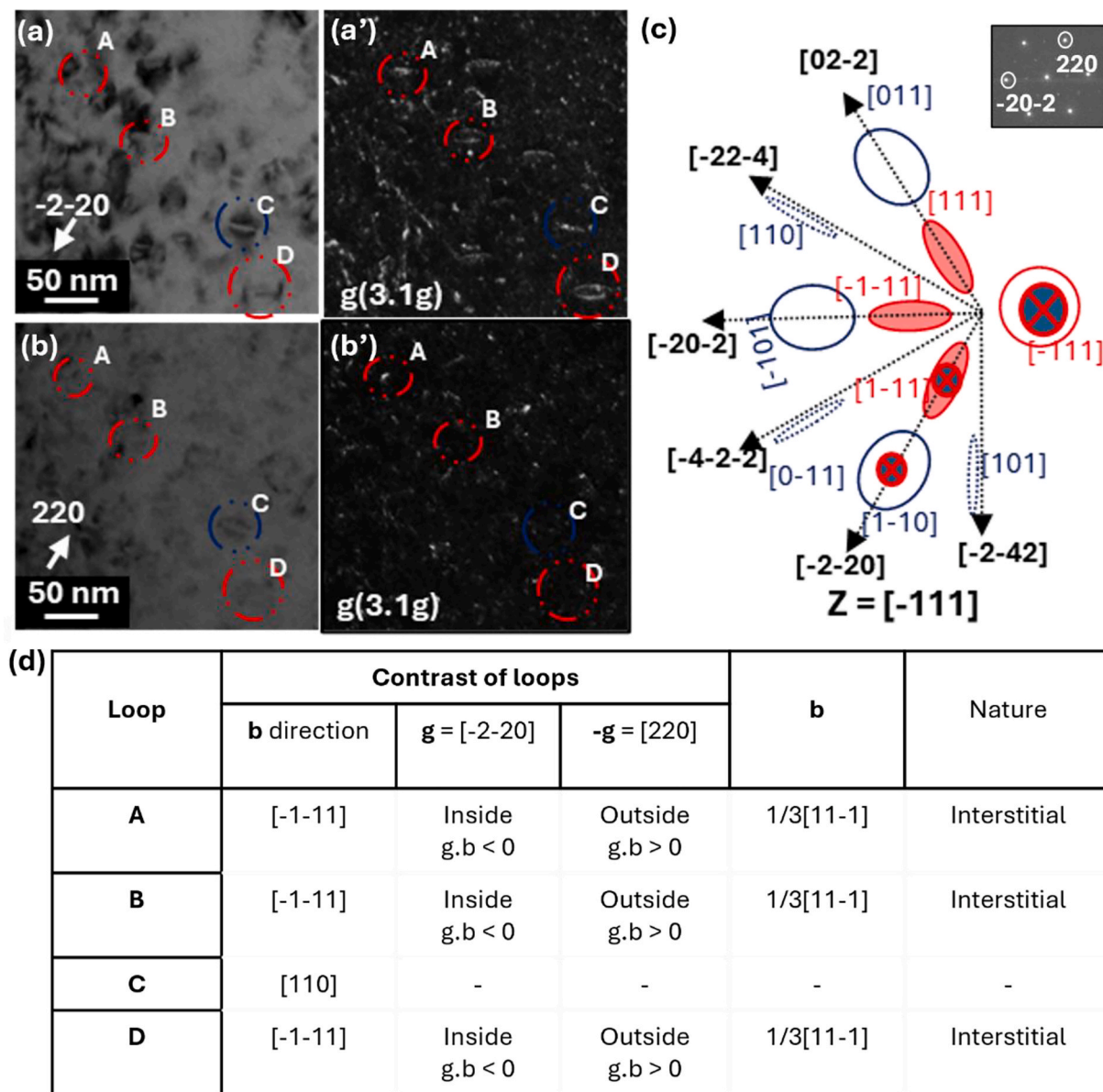


Fig. 9. Determination of loop nature using the inside-outside contrast method. (a, a') and (b, b') present KBF and WBDF micrographs captured with $g = [-2-20]$ and $g = [220]$ diffraction vectors near the $[-111]$ zone axis. Frank loops analyzed for contrast shifts are marked with red circles. (c) Simulated projection of loops along the $[-111]$ zone axis [53], highlighting invisibility conditions for certain orientations (marked with 'X'). (d) Analysis of Frank loop nature (interstitial vs. vacancy) based on the image contrast changes between the $\pm g$ conditions shown in (a-b').

distribution (Fig. 13c). A critical observation at this dose is the presence of few Frank/faulted loops with segmented (faceted) morphologies. In many compositionally complex FCC alloys, including equi-atomic NiCoFeCr [63], $\text{Ni}_{40}\text{Fe}_{40}\text{Cr}_{20}$ [64], and Austenitic stainless steels [65,66], interstitial Frank/faulted loops maintain a circular geometry prior to unfauling, as this geometry is energetically favorable [67]. Conversely, in pure FCC metals with lower SFE such as Cu [68,69], Ag [68], and Au [70] both interstitial and vacancy-type loops frequently exhibit faceted or polygonal morphologies.

In FCC materials, a Frank loop is sessile due to its associated stacking fault and is bounded by a dislocation line at the periphery. To minimize overall energy, Frank loops with Burgers vector, $1/3 \langle 111 \rangle$ dissociate into a $1/6 \langle 112 \rangle$ Shockley partial and a $1/6 \langle 110 \rangle$ stair-rod dislocation [67]. This dissociation leads to a polygonal (often hexagonal) shape, with faceted edges aligned along the $\langle 110 \rangle$ close-packed directions. Similar polygon morphology of the dissociated faulted loops can be seen in Fig. 14a, which are schematically represented in Fig. 14c to illustrate

the hexagonal loop segments and their corresponding projections along the $[011]$ zone axis. These results align with the findings of Ma et al. [67], who established that all segments of such hexagonal loops are parallel to the close-packed $\langle 110 \rangle$ directions. From an energetic standpoint, this transformation is highly favorable for vacancy-type loops in FCC systems like Al, Ni, and Cu, as the dissociation releases the significant elastic strain concentrated at the dislocation core, thereby lowering the total formation energy [67]. In contrast, for interstitial-type loops, the stress is typically distributed across the fault plane, whereas in vacancy loops, the stress is localized at the loop periphery, which more readily triggers the dissociation reaction. Theoretically, circular interstitial loops should exhibit the lowest formation energy and maximum stability due to this distributed stress. However, in low-SFE materials and chemically complex HEAs, the energy barrier for dissociation is notably reduced. Consequently, minimal external stimuli, such as the localized stress fields generated by the high density of radiation-induced defects or thermal stimuli are sufficient to promote

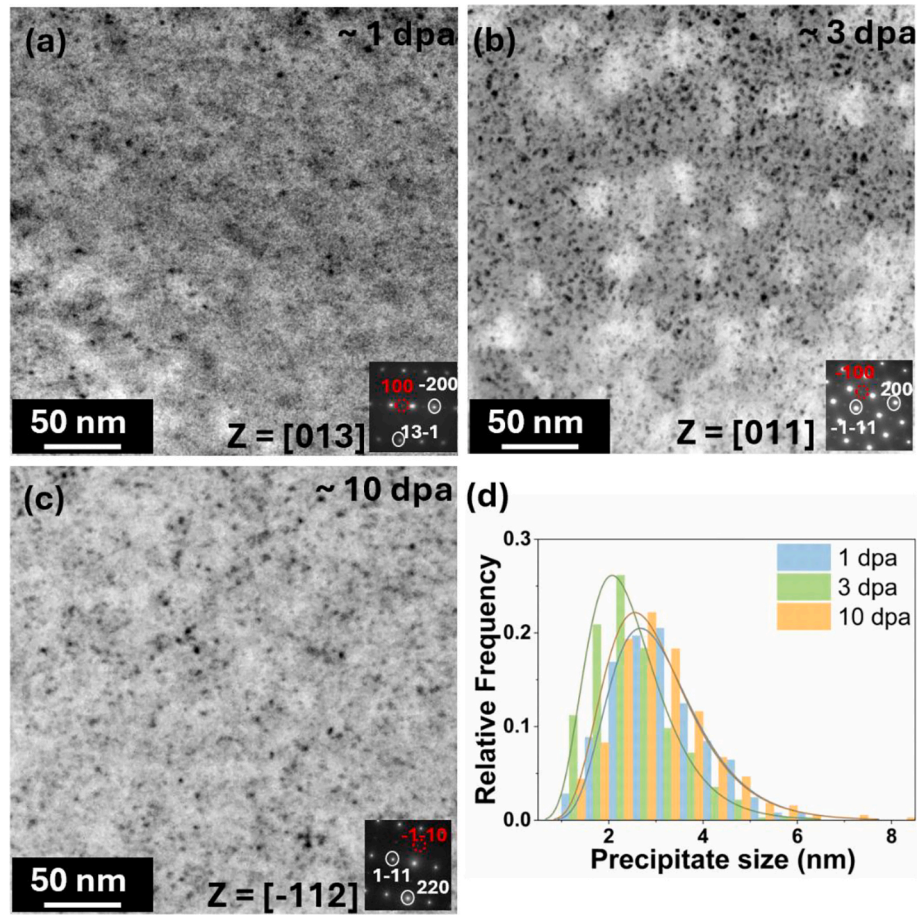


Fig. 10. Evolution of L₁₂ precipitates under irradiation: (a–c) Inverted dark-field (DF) micrographs for the 1, 3, and 10 dpa regions. The images were captured using the (100), (−100), and (−1-10) diffraction spots, respectively (indicated by red circles in the SAED insets). The corresponding zone axes are [013], [011], and [−112]. (d) Size distribution histograms for the L₁₂ precipitates across the three irradiation conditions, each fitted with a log-normal distribution to determine the mean diameter and standard deviation.

Table 3

Quantitative analysis of L₁₂ precipitates, summarizing the mean particle size and number density across the studied irradiation conditions.

Irradiation condition	Precipitation size (nm)	Precipitate density (m ^{−3})
1 dpa	3.14 ± 1.06	4.92 ± 1.21 × 10 ²²
3 dpa	2.49 ± 0.89	10.47 ± 0.82 × 10 ²²
10 dpa	3.02 ± 1.02	3.56 ± 0.31 × 10 ²²

segmentation even in interstitial-type loops [67]. The fact that majority of the loops retain a circular or slightly elliptical morphology (Fig. 14d) while few appear sharply faceted (Fig. 14b) suggests a heterogeneous SFE landscape within the HEA matrix. HEAs have shown to have low intrinsic SFE (10–40 mJ m^{−2}) [67,71,72] but also possess the highest unstable SFE (>400 mJ m^{−2}) [67] which can act as a strong barrier for dissociation. This variation is a direct consequence of local chemical fluctuations and short-range order (SRO) [73] and in this case could also be impacted by the ordered phase transformation observed in our material, which creates a region of varying stacking fault stability. In regions where the SFE is locally elevated, the dissociation of Frank loops is difficult, maintaining a circular geometry. Conversely, in regions with low SFE, dissociation into Shockley and stair-rod components is promoted, resulting in the observed faceted polygonal morphology.

The quantitative evaluation of the dislocation loop populations presented in Table 2 reveals an increase in the fraction of perfect loops as the damage level increases from 1 to 3 dpa. While a minority of Perfect loops may nucleate directly within high-energy displacement cascades,

most of the ½ <110> Perfect loop population is attributed to the unfaulting of pre-existing 1/3 <111> Frank loops. This transition is governed by three primary mechanisms documented in the literature: the energy reduction mechanism, the loop-loop interaction reaction, and the nucleation and growth of Shockley partial dislocations. According to isotropic elasticity theory, a Perfect dislocation loop becomes energetically more stable than a Faulted loop only when the defect reaches a critical radius (r_c). The unfaulting reaction becomes thermodynamically favorable when the energy saved by eliminating the stacking fault associated with the faulted loop exceeds the energy required to create the new Burgers vector (perfect loops). This is expressed by the following equation [74,75]:

$$\gamma_{SFE} > \frac{\mu b^2}{3\pi r_c} \left| \frac{2-\nu}{2(\nu-1)} \right| \ln \left(\frac{4r_c}{r_0} - 2 \right) \quad (1)$$

Where γ_{SFE} (45 mJ m^{−2}) [26] is the stacking fault energy, μ (82 GPa in case of austenitic steel [75]) is the shear modulus, b (0.2 nm) is the modulus of the Burgers vector of the Frank loops, ν (0.3) is the Poisson's ratio, and r_0 (0.5 nm) is the dislocation core radius. Solving for r_c , the critical radius for unfaulting to be energetically favorable is ~57 nm (loop size of 114 nm) which is very large when compared to the average loop sizes observed in this study in all the 3 conditions. This suggests unfaulting occurs below the theoretical critical size. Supporting this, Chen et al. [76] found no evidence of a definitive critical size in ion irradiated stainless steel at 400 °C, they observed small perfect loops (8–16 nm) coexisting with a higher density of much larger Frank loops (40–44 nm). Similar conclusions were made in in-situ TEM observation

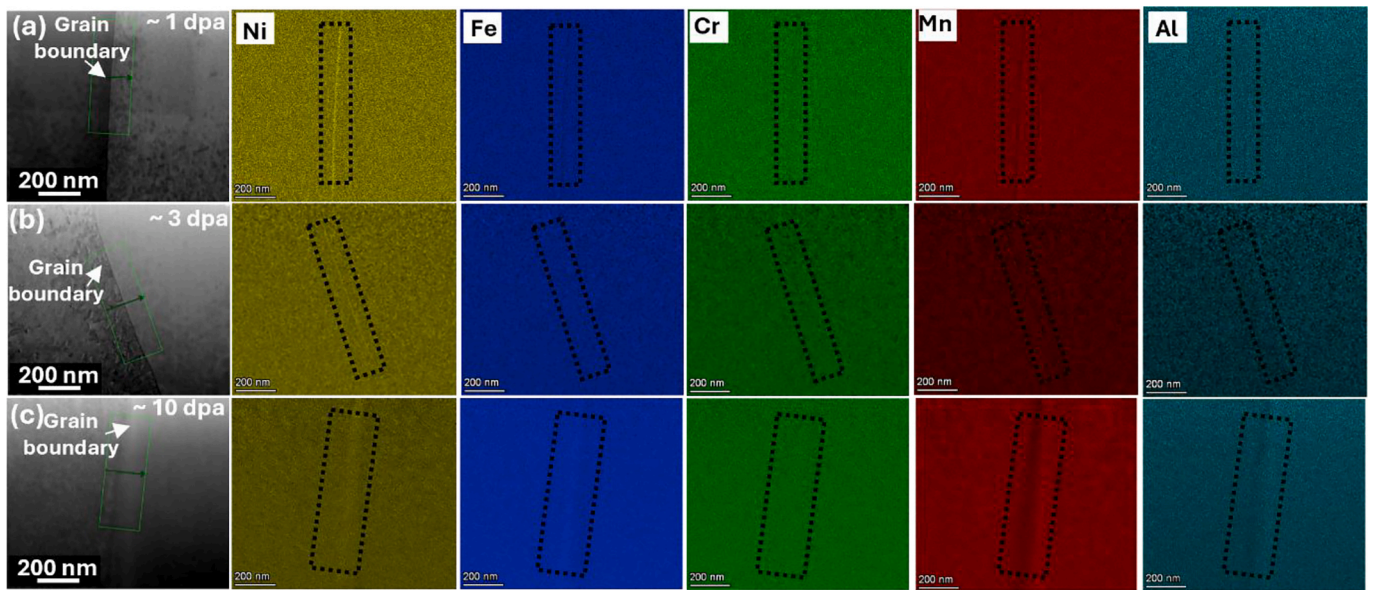


Fig. 11. STEM-EDS elemental mapping across grain boundaries. (a–c) Chemical maps are shown for the 1, 3, and 10 dpa irradiation conditions. The specific depth of interest analyzed for each sample is denoted by a green rectangle on the corresponding overview image and the grain boundary has been highlighted using black dashed rectangle.

Table 4

Enrichment amplitude calculated across the grain boundary at the depth of interest.

Irradiation condition	Ni (at. %)	Fe (at. %)	Cr (at. %)	Mn (at. %)
1 dpa	2.58 ± 0.42	−1.29 ± 0.18	−0.45 ± 0.17	−1.03 ± 0.14
	3.93 ± 1.17	−1.47 ± 0.47	−0.6 ± 0.36	−1.97 ± 0.56
3 dpa	1.51 ± 0.16	−0.16 ± 1.02	−0.16 ± 0.32	−1.38 ± 0.08

of H_2^+ & He^+ irradiated Pd where the theoretical critical radius was calculated as ≈ 16 nm and still much larger faulted loops (80–120 nm) were observed [77]. In HEAs, the high local chemical fluctuations leading to variable SFE [67] may significantly alter this r_c compared to pure FCC metals. Previous work has shown that the reduction in the loop energy is a necessary condition but by itself is insufficient and requires additional local stresses such as from interaction between high density loops or the nucleation of the Shockley partials within the Faulted loops

or because of cascade collision [76–78].

The loop-loop/line interaction and nucleation and growth mechanisms have been considered the driving mechanisms for unfaulting. Consistent with the in-situ observations by Li et al. [77], our 3 dpa sample (Fig. 14a) displays similar possible unfaulting pathway triggered by the interaction between proximate Faulted-Faulted and Faulted-Perfect loops shown in Fig. 14a. Experimental and computational studies has shown the unfaulting process driven by these loop-loop or loop-line interactions [79–81]. MD simulations by Ustrzycka et al. on a Cr-rich FeNiCr alloy showed that interactions between edge dislocations and Frank loops lead to loop splitting, transformation, or absorption by the dislocation line. A primary mechanism proposed for this configuration change is the interaction-driven unfaulting of the interstitial Frank loop into a perfect loop [82]. We can also observe the presence of the secondary loops within the interior of Frank loops (Fig. 14b, d). These internal features might be $1/6 \langle 112 \rangle$ Shockley partials which could eventually sweep across the stacking fault removing it. For an extrinsic fault to be removed two Shockley partials must sweep across the faulted plane as shown by the following dislocation reaction [66]:

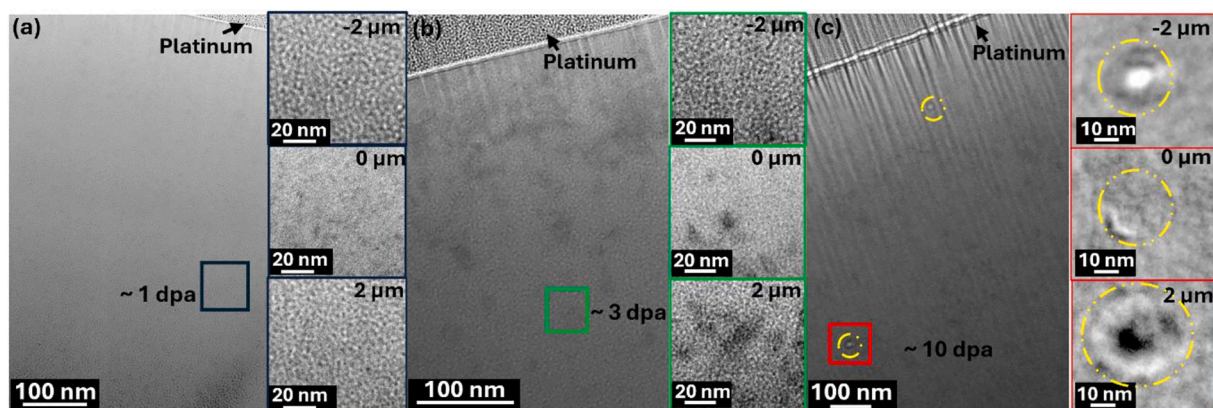


Fig. 12. Fresnel contrast analysis of voids. (a–c) Off-axis under-focused (−2 μm) overview micrographs for the three irradiation fluences. Corresponding high-magnification images of the depth of interest are shown under three −focus conditions: under-focus (−2 μm), exact focus (0 μm), and over-focus (+2 μm). Voids are highlighted with yellow circles to illustrate the characteristic contrast reversal.

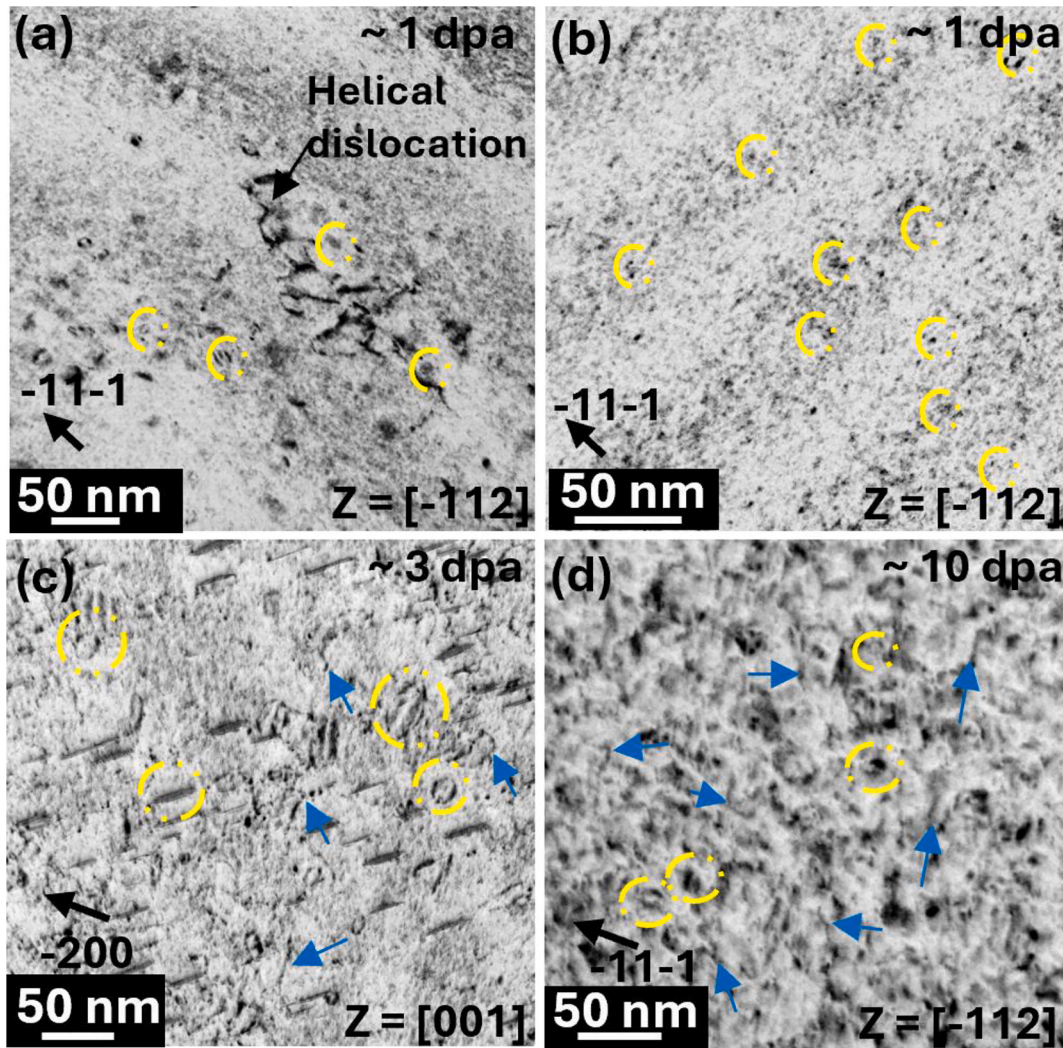


Fig. 13. Irradiation induced microstructure evolution (a) Inverted Weak-Beam Dark-Field (WBDF) micrograph at 1 dpa, showing preferential clustering of large interstitial loops (yellow circles) in the vicinity of pre-existing dislocations (b) Inverted WBDF image of the 1 dpa matrix in a region devoid of prior dislocations, exhibiting a high density of fine, isolated loops. (c–d) Inverted WBDF micrographs at 3 dpa and 10 dpa, respectively. Illustrating the progressive coarsening of the loop population and the transition toward a saturated dislocation network. Yellow circles highlight representative loop morphologies, while blue arrows indicate primary dislocation lines.

Table 5

Calculated average sink strength of microstructural features.

Irradiation condition	Pristine	1 dpa	3 dpa	10 dpa
Sink strength of GBs (m^{-2})	1.09×10^{10}	1.09×10^{10}	1.09×10^{10}	1.09×10^{10}
Sink strength of dislocation network (m^{-2})	2.6×10^{13}	2.7×10^{13}	1.36×10^{14}	7.18×10^{14}
Sink strength of dislocation loops (m^{-2})		2.15×10^{14}	6.82×10^{14}	3.56×10^{14}
Total sink strength – matrix (m^{-2})	2.6×10^{13}	2.42×10^{14}	8.18×10^{14}	1.07×10^{15}

$$\frac{1}{3}[111] + \frac{1}{6}[2\bar{1}\bar{1}] + \frac{1}{6}[\bar{1}2\bar{1}] \rightarrow \frac{1}{2}[110] \quad (2)$$

The observation of nested loop configurations within larger Frank loops provides strong empirical support for the hypothesized unfaulting pathway via Shockley partial nucleation. This interpretation aligns well with previous in-situ TEM irradiation studies on Pd at 300 °C, which reported a similar unfaulting mechanisms where Shockley partial nucleation initiated at doses as low as 0.1 dpa [77]. Bilyk et al. reported similar observations of nested loops inside a Frank loop in ion irradiated

CrFeMnNi and proposed possible division of the faulted loop onto smaller perfect loops through increasing the fraction of perfect loops [83]. While the unfaulting of Perfect loops is traditionally characterized as a thermally activated process requiring a critical energy to nucleate the Shockley partial, the increasing radiation dose in this HEA introduces a potent mechanical driver. As the defect density rises with fluence, the overlapping elastic strain fields of neighboring loops generate significant local internal stresses. These localized stress concentrations effectively lower the activation barrier for Shockley partial nucleation, allowing the partial to sweep across the fault plane and eliminate the stacking fault. This dose-driven, stress-assisted unfaulting mechanism accounts for the transition from sessile Faulted/Frank loops to glissile Perfect loops observed in our quantitative data at higher dpa.

4.1.3. Dislocation network formation and void nucleation (10 dpa)

By 10 dpa, the microstructural evolution is characterized by the extensive interaction and coalescence of glissile Perfect loops, marking a fundamental transition from discrete defect clusters to a complex, interconnected dislocation network (Fig. 5c'', 13d). Quantitatively, this is reflected in a sharp decrease in the Perfect loop fraction from ~36% to ~13%, as Perfect loops are incorporated into the complex

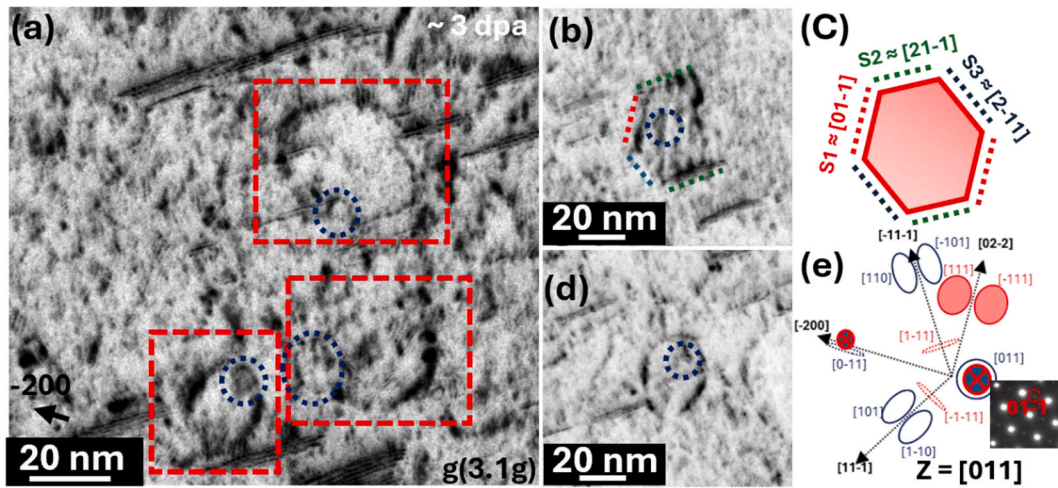


Fig. 14. Morphology and transformation of Frank loops (these micrographs correspond to 3 dpa region). (a) Inverted WBDF micrograph highlighting three large Frank loops (red dotted rectangles), blue circles indicate the nucleation/interaction of perfect loops along the periphery of the larger Frank loops. (b,d) Representative inverted WBDF micrographs illustrating typical Frank loop morphologies (b) represent a segmented loop and (d) represent a circular loop which looks ellipsoidal due to the projection on the imaging plane. (c) schematic projection of the segmented loop. (e) simulated loop projections for [011] zone axis orientation, including the invisible loop configurations for the $g = -200$ diffraction condition marked by the cross, adopted from [53].

network. This shift is further corroborated by the inverse relationship between loop density and dislocation line density observed at higher fluences (Fig. 7). This microstructural evolution is significantly affected by the presence of L_{12} nanoprecipitates, which serve as strong obstacles to dislocation migration [84]. For dislocations to bypass these precipitates, they must either shear the particles or undergo bowing. This requires either higher stress or higher temperatures. This dislocation pinning elevates the dislocation density and stabilizes a high sink strength within the matrix (Table 5), thereby enhancing the annihilation of radiation induced defects.

The emergence of voids at 10 dpa is a direct consequence of this saturated network microstructure. In HEAs, long-range defect mobility is drastically impeded because of high lattice distortion, variable energy landscape and 3-D motion of interstitials which tend to enhance defect recombination thus suppressing void growth [16,19,85]. Recent work by Gui et al. shows a broad activation energy spectrum for monovacancy migration (1.55 to 1.9 eV) in the same alloy which arises from chemical diversity and lattice distortion leading to thermodynamic instability of defect clusters [86]. At lower fluences (1–3 dpa), the limited thermal mobility of vacancies at $0.3T_{\text{melting}}$ promotes a recombination-dominated regime, preventing the formation of resolvable voids. While sub-resolution vacancy clusters likely exist below the TEM detection limit (Fig. 12a, b), the high recombination rate with relatively short-range interstitial mobility effectively suppresses void nucleation. However, as the dislocation line density at 10 dpa increases six-fold compared to 3 dpa (Fig. 7), the microstructure transitions into a sink-dominated regime. Table 5 shows the evolution of the sink strength of various microstructural features with damage dose (see supplementary data for details). At 10 dpa we see considerable increase in the sink strength of the dislocation network (~ 7 times as compared to 3 dpa). In this state, the dense dislocation network provides a potent, biased sink for interstitials, which due to their larger strain fields are preferentially absorbed without the need of long-range diffusion. This localized absorption of interstitials from the matrix drastically reduces the probability of mutual recombination, finally allowing the vacancy concentration to reach the critical supersaturation threshold. Only when the network is sufficiently dense to capture the competing interstitials can the sluggish vacancies survive and coalesce into the observed 1–10 nm voids. This mechanism aligns with observations in NiCoFeCrPd HEAs, where indentation-induced dislocations accelerated swelling [33], and in cold-worked copper, where increased dislocation density

promoted void nucleation [87]. Consequently, our results demonstrate a prolonged incubation period for swelling, governed by the kinetic evolution from sessile Faulted/Frank loops to a saturated dislocation network capable of facilitating vacancy survival at 300°C. This transition is schematically illustrated in Fig. 15. Additionally, 10 dpa damage dose at 300°C represents only the early-stage irradiation regime and further high temperature, high-dose irradiation studies are required to fully comment on the irradiation tolerance behavior in advanced nuclear systems.

4.2. Irradiation enhanced phase transformation

The irradiation enhanced formation of the ordered L_{12} phase (Ni_3Al type) is governed by a homogeneous nucleation process, facilitated by the low lattice misfit ($\sim 0.15\%$) with the FCC matrix. This coherency minimizes both elastic strain energy and interfacial energy (γ), significantly reducing the thermodynamic barrier for nucleation $\left(\Delta G^* \propto \frac{\gamma^2}{\Delta G_v}\right)$

where G^* is the Gibbs free energy and G_v is the volumetric free energy, and suppressing capillary coarsening during irradiation [31]. In contrast to semi-coherent nanoparticles (nano-oxides) or complex superlattices (e.g., L_{21} in BCC matrix [88]) which often dissolve under heavy-ion bombardment due to high interfacial energies, the coherent L_{12} phase in this HEA remains in the matrix even after a damage dose of 10 dpa. The formation of L_{12} precipitates is chemically rooted in the Ni-Al binary pair, which possesses the most negative enthalpy of mixing ($\Delta H_{\text{mix}} \approx -22 \text{ kJ/mol}$) [91] among the constituent elements, providing a strong chemical driving force for local ordering that competes with the high configurational entropy of the matrix. The phase evolution observed between 1 and 10 dpa reflects a dynamic competition between RED and BD. The negligible change in precipitate size and number density between 1 dpa and 10 dpa suggests that the microstructure has reached a steady-state condition. In this regime, the radiation-enhanced diffusion (RED) driven solute flux toward the precipitates is balanced by the ballistic dissolution (BD) of solute atoms away from them. Interestingly, the number density increases by nearly a factor of two at 3 dpa relative to 1 dpa (Table 3). This transient rise likely arises from a dual process, the ballistic fragmentation/dissolution of larger existing particles (evidenced by the decrease in average size from 3.14 nm to 2.49 nm) alongside the RED-driven growth of sub-resolution clusters

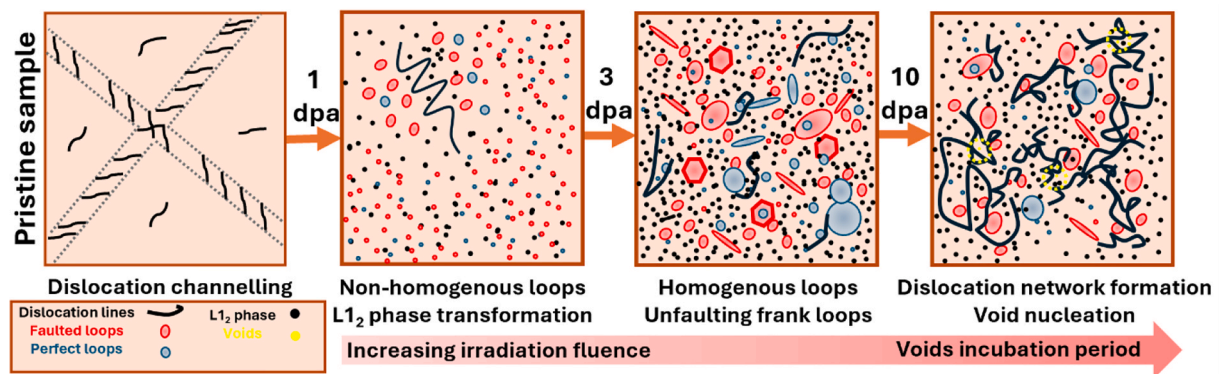


Fig. 15. Schematic of the dose-dependent microstructural evolution in the HEA. 1 dpa: Heterogeneous nucleation of loops near pre-existing sinks. 3 dpa: Transition to faceted morphologies and onset of unfaulting to perfect loops. 10 dpa: Formation of a dense, precipitate-pinned dislocation network, triggering vacancy supersaturation and void incubation.

into the detectable TEM size range. Because no precipitation occurs in the unirradiated control regions, thermal diffusion is negligible at this irradiation temperature. Consequently, the observed phase stability is driven directly by RED via irradiation-induced supersaturated vacancy concentrations. Beyond 3 dpa, as the damage accumulates to 10 dpa, both average precipitate size and number density revert toward values similar to the 1 dpa state, further confirming a dynamic equilibrium between RED and BD. Nevertheless, higher-resolution spatial techniques, such as Atom Probe Tomography (APT), would be beneficial to quantitatively analyses the evolution of the ordered phase as a function of damage dose.

Du et al. [31] proposed an irradiation induced disordering-ordering transition in a BCC-B₂ and FCC-L₁₂ system. According to which during ion irradiation, energetic displacement cascades effectively shuffle atoms, dissolving precipitate embryos in tens of picoseconds [89]. Al being the lightest atom tends to be displaced larger distances from the precipitates leading to the disordering of the ordered phase forming a Ni rich region along with high density of point defects. And before the cascade cools down the Al atoms bound back to the Ni rich regions due to higher affinity toward Ni [90], forming L₁₂ nuclei. This process is a rapid process and could happen under 1 s. and due to the supersaturation of Al around these nuclei there is a growth of these low energy highly coherent precipitates due to short-range reshuffling. A pivotal factor in this kinetics is the low thermal conductivity inherent to concentrated solid solutions [17]. High chemical disorder and severe lattice distortion shorten the phonon mean free path, slowing the dissipation of energy from the displacement cascade [91]. This extends the thermal spike duration and increases localized peak temperatures [18,92]. This prolonged “liquid-like” state within the cascade core provides a critical temporal window for atomic reordering, even at a bulk temperature of $\sim 0.3T_{\text{melting}}$.

Finally, while the L₁₂ precipitates are highly stable due to ordering-disordering behavior, their role as recombination centers is limited by their coherent interface. Unlike incoherent/semi-coherent interfaces found in oxide-dispersed (ODS) steels [93], which possess large misfit strain fields that act as potent sinks for both interstitials and vacancies, the minimal strain field surrounding these L₁₂ precipitates results in a lower capture efficiency for point defects [94]. Consequently, while these precipitates are excellent for high temperature mechanical strengthening, their contribution here has rather been on stabilizing the dislocation network structure through dislocation pinning. It would be interesting to see how these order-disorder phase transformations would impact the defect evolution at the temperatures where there is significant thermal vacancy mobility.

4.3. Irradiation induced grain boundary segregation

Radiation-induced segregation (RIS) in materials is driven by the persistent flux of vacancies and interstitials toward defect sinks or low energy sites, such as free surface, grain boundaries and dislocations [95–97]. The STEM-EDS mapping (Fig. 11) reveals a slight enrichment of Ni at the grain boundaries, a trend that peaks at 3 dpa before becoming less prominent at 10 dpa (Table 4). Theoretically, RIS is governed by two primary mechanisms: (i) Inverse Kirkendall (IK) effect [96] where an oversized elements exchange with the vacancy flux. Since the vacancy flux moves towards the sink these elements typically deplete at the sink. (ii) Interstitial Drag Mechanism [97] where an undersized atoms or those with a high binding affinity for interstitials are dragged toward the sink by the interstitial flux, leading to enrichment. In both cases there is an enrichment of smaller size atoms at the interfaces. The decreasing order of the atomic size is Al > Mn > Fe > Cr > Ni [90] in the studied HEA. Accordingly, the enrichment of Ni at the grain boundaries or loops can be explained with either of the mechanisms. At an irradiation temperature of $\sim 0.3T_{\text{melting}}$, vacancy mobility is significantly restricted in comparison to interstitials making the IK mechanism unlikely driver for the observed Ni enrichment. Instead, the segregation is more likely coupled to the relatively higher mobility of interstitials and Perfect dislocation loops in the vicinity of grain boundaries. While Faulted/Frank loops are sessile and only grow via climb, Perfect loops are glissile [4,98]. Ni and Co segregations and Fe, Cr and Mn depletion near irradiation induced loops have been previously observed in several studies [35,41]. The enrichment of Ni at the boundaries likely occurs because Ni atoms preferentially associate with these mobile defects, which then migrate toward and are absorbed by the grain boundary.

The evolution of the dislocation enriched zone (DEZ) (Fig. 16) provides a clear explanation for the observed segregation trends. Which is fundamentally driven by the high defect flux toward grain boundaries at elevated temperatures [99,100], the differing mobilities of various defects e.g. perfect loops are mobile in comparison to faulted loops, and a low intrinsic stacking fault energy that favors sessile faulted loop formation [101]. At 1 and 3 dpa, a high density of loops accumulates near the grain boundary; unlike traditional dislocation denuded zones (DDZs) observed at room temperatures [35], this DEZ forms because sessile Frank loops remain stationary in the matrix while mobile perfect loop nuclei are captured by the grain boundary's strain field [35]. However, the DEZ width contracts from ~ 180 nm at 1 dpa to ~ 140 nm at 3 dpa, eventually vanishing by 10 dpa. The decrease of the DEZ at 3 dpa maybe attributed to enhanced absorption of the perfect loops and the growth of the faulted loops near the grain boundary which also correlates to the increase in the Ni segregation at the grain boundary. The disappearance of DEZ and the subsequent reduction in Ni segregation at 10 dpa are

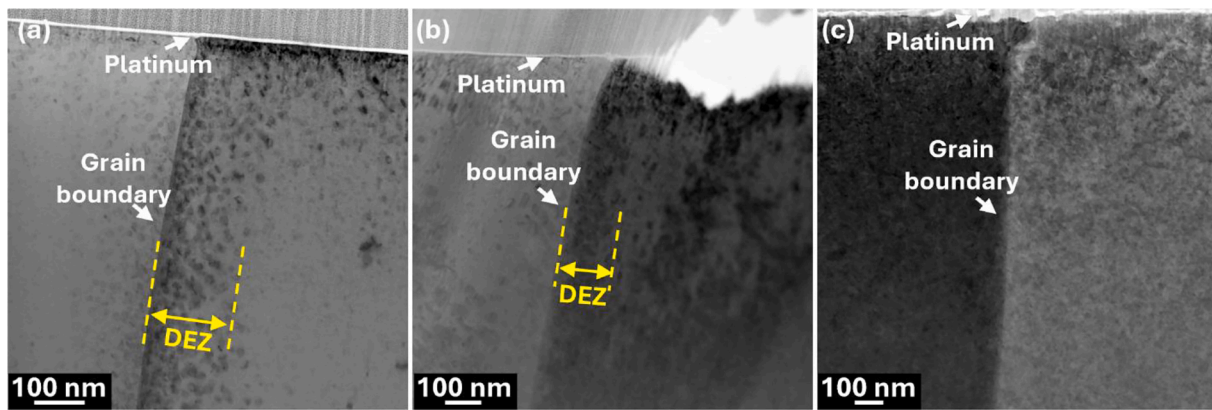


Fig. 16. High-density dislocation loops near grain boundaries. (a–c) Inverted on-axis Dark-Field STEM (DF-STEM) micrographs recorded along the $[-112]$, $[013]$, and $[001]$ zone axes for 1, 3, and 10 dpa conditions, respectively. The images reveal a significant accumulation of dislocation loops localized near the grain boundary in 1 and 3 dpa regions. The dislocation enriched zone (DEZ) is highlighted at the target depth.

attributed to the emergence of competitive sinks. At 10 dpa, the matrix develops a dense, saturated dislocation network sink (matrix sink strength increases by an order higher compared to 1 dpa) which absorbs the defect flux originally destined for the grain boundaries. As internal sink strength intensifies, the net flux of defects and associated Ni solutes toward the interface is drastically attenuated. Interstitials are sequestered by the local dislocation network before they can reach the boundary, leading to decreased Ni segregation observed at high doses and highlighting that RIS in HEAs is dynamically regulated by the evolving topology of the dislocation substructure.

5. Conclusion

This study systematically investigates the irradiation induced microstructural evolution of a novel Co-free high-entropy alloy (HEA) using 5 MeV Fe^{2+} ion irradiation across various fluence corresponding to damage range of 1 to 10 dpa at 300 °C at a depth of 400–700 nm. By correlating quantitative defect density measurements with analytical TEM analysis, It was demonstrated that the alloy's radiation tolerance is governed by a complex interplay between dislocation topology, sluggish diffusion kinetics, and the stability of coherent nanoprecipitates. The main highlights from the study are presented below:

- **Dose-dependent microstructural evolution:** The alloy transitions from a sink-limited regime dominated by sessile Faulted/Frank loops at 1 dpa because of the pre-existing dislocation lines to a saturated dislocation network at 10 dpa.
- **Loop segmentation and unfauling:** At 3 dpa, low Stacking Fault Energy (SFE) and local chemical order trigger the faceting of Faulted/Frank loops into polygonal shapes. The unfauling of Faulted/Frank loops into glissile Perfect loops is most likely through the nucleation of Shockley partials within the Faulted/Frank Loop driven by high density of defects in the vicinity and loop-loop/line interaction mechanisms.
- **Irradiation induced L_{12} phase stability:** Irradiation induced coherent ($\sim 0.15\%$ lattice mismatch) L_{12} nanoprecipitates exhibits a competition between ballistical dissolution and irradiation enhanced diffusion. Their low lattice misfit and strong Ni-Al bonding acts as an obstacle for dislocation mobility that stabilizes the dislocation network at higher dpa and enhances radiation hardening.
- **Void incubation mechanism:** Voids (1–3 nm) emerge only at 10 dpa. This indicates a significant incubation period where the dense dislocation network must reach a critical density to absorb interstitials, allowing sluggish vacancies to reach the supersaturation required for nucleation.

- **Radiation-induced segregation (RIS) and sink competition:** Ni enrichment at grain boundaries (dislocation enriched zone formation) peaks at 3 dpa due to interstitials and mobile loop migration. By 10 dpa, segregation diminishes as the high-density internal dislocation network acts as a competitive sink, absorbing solute-defect fluxes before they reach the boundaries.
- **Radiation tolerance:** The alloy's shows delayed void nucleation period which is intrinsically linked to sluggish vacancy diffusion and the delayed topological transition of dislocations, which together suppress the early-stage accumulation of vacancy clusters. However, the formation of L_{12} phases along with high density of loops and dislocation network would drastically increase the hardness of the alloy.

While the current findings demonstrate delayed void nucleation at 300 °C, this temperature and damage dose remains below the actual projected application environment for next-generation nuclear alloys. Consequently, further irradiation induced defects investigation is required at elevated temperatures and at higher damage doses, where increased thermal vacancy mobility will play a more decisive role in the evolution of the defect microstructure and long-term phase stability.

CRediT authorship contribution statement

Lakshay Chauhan: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Conceptualization. **Neelam Meena:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Ermile Gaganidze:** Writing – review & editing, Supervision, Resources, Funding acquisition, Conceptualization. **Dimitri Litvinov:** Writing – review & editing, Investigation. **Cornelia Kaden:** Writing – review & editing, Resources. **Jarir Aktaa:** Writing – review & editing, Supervision, Resources, Funding acquisition, Conceptualization.

Declaration of competing 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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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.matdes.2026.116704>.

Data availability

Data will be made available on request.

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