

Article

Comparative Study on Helium Ion Irradiation Resistance in Additively Manufactured 316L Stainless Steel and CoCrFeNi High-Entropy Alloy

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Abstract

The 316L stainless steel (316L SS) and high-entropy alloys (HEAs) are leading candidates for radiation-tolerant structural materials in nuclear environments. Additive manufacturing (AM) enables tailored microstructures through unique thermal histories, producing high dislocation densities and sub-grain features that act as effective sinks for irradiation-induced defects. In this work, a direct quantitative comparison of helium (He) irradiation response, particularly bubble formation, is conducted between 316L SS fabricated using laser powder bed fusion (LPBF) and CoCrFeNi HEAs fabricated by laser-directed energy deposition (LDED), both possessing a face-centered cubic (FCC) crystal structure and comparable principal elemental constituents. The samples were subjected to ex situ He ion irradiation using 200 keV He⁺ ions to a peak damage dose of 10 dpa at 25 °C, 400 °C, and 600 °C at the CINT User Facility at Los Alamos National Laboratory. Post-irradiation microstructural characterization was performed using transmission electron microscopy at the IVEM-Tandem Facility at Argonne National Laboratory. For LPBF 316L SS, the areal bubble density decreases from approximately $5.1 \times 10^4 \mu\text{m}^{-2}$ at 25 °C to $2.1 \times 10^3 \mu\text{m}^{-2}$ at 600 °C, while the mean bubble diameter increases from 2.9 nm to 37.4 nm. The CoCrFeNi HEA exhibits a similar trend but retains a higher areal bubble density at elevated temperatures, with values of $2.1 \times 10^4 \mu\text{m}^{-2}$ at 400 °C and $3.7 \times 10^3 \mu\text{m}^{-2}$ at 600 °C, along with a larger mean bubble size at 400 °C compared to 316L SS. These results highlight the combined roles of AM-induced microstructures, alloy compositions, and irradiation temperatures in governing He damage evolution in FCC alloys, providing guidance for the development of radiation-tolerant materials for advanced nuclear energy applications.

Keywords: additive manufacturing; helium ion irradiation resistance; 316L stainless steel; high-entropy alloys



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1. Introduction

Additive manufacturing (AM) has emerged as an attractive processing route for structural materials in nuclear energy systems due to its capability to fabricate complex geometries and, more importantly, to generate non-equilibrium microstructures through rapid solidification and cyclic thermal histories [1–3]. Laser-based AM processes such as laser powder bed fusion (LPBF) and laser-directed energy deposition (LDED) produce refined cellular substructures, high dislocation densities, and heterogeneous defect distributions

that are fundamentally different from those in conventionally manufactured alloys [4–6]. These AM-induced microstructural features are increasingly recognized as critical factors governing the evolution of irradiation damage, making AM materials scientifically relevant candidates for nuclear applications beyond their geometric advantages.

In nuclear environments, structural materials are exposed to energetic neutron irradiation, which produces displacement damage and generates helium (He) through transmutation reactions [7,8]. Among irradiation-induced species, He is particularly detrimental because of its extremely low solubility in metals. He readily binds with vacancies to form vacancy–He clusters, which subsequently evolve into nanoscale He bubbles [9,10]. The accumulation and growth of these bubbles can lead to swelling, embrittlement, and degradation of mechanical properties, thereby limiting component lifetime [11]. Consequently, understanding He bubble evolution is central to assessing the irradiation resistance of candidate nuclear materials.

Austenitic stainless steel, particularly 316L stainless steel (316L SS), is widely used in nuclear systems and has therefore served as a model material for studying irradiation effects [12–14]. Recent studies have shown that AM 316L SS exhibits greater resistance to He-induced degradation compared to conventionally manufactured counterparts. For example, Sun et al. [11] investigated the hardening behavior of He-ion-irradiated LPBF 316L SS at 450 °C and reported reduced irradiation hardening in AM samples relative to conventionally manufactured materials. Fu et al. [15] evaluated He bubble evolution at 700 °C and found that the bubble density in AM 316L SS was approximately two times lower than that in conventionally manufactured material. In a subsequent study, the same authors examined He bubble evolution over a range of irradiation temperatures, demonstrating that bubble size increases with temperature while bubble number density decreases [16]. Their work further revealed that different AM-induced microstructural features, such as dislocation structures and oxide particles, can exert varying influences on He bubble evolution. More recently, Zhao et al. [17] investigated irradiation damage behavior in multi-laser powder bed fusion-fabricated 316L SS. Their results indicated that multi-laser overlap regions exhibit lower irradiation hardening than single-laser regions, an effect primarily associated with differences in He bubble size and number density. While these findings highlight the potential of advanced AM processing strategies to tailor irradiation response, the inherent complexity and process-control challenges associated with multi-laser systems limit the reliable translation of these benefits to large-scale nuclear component fabrication.

Although these studies demonstrate that AM-induced microstructural features can improve the He irradiation resistance of 316L SS, these improvements primarily rely on defect-sink engineering through processing. This raises an important question as to whether microstructural control alone is sufficient to mitigate He-induced damage at higher temperatures, or whether additional mitigation strategies based on alloy design are required. Beyond microstructural engineering through processing routes, alloy design offers an additional strategy for mitigating He-induced damage. High-entropy alloys (HEAs), composed of multiple principal elements in near-equiatomic ratios, exhibit pronounced chemical complexity and lattice distortion, which can hinder defect migration and reduce the irradiation-induced defect formation [18–20]. Among these materials, CoCrFeNi has emerged as one of the few HEAs exhibiting a stable single-phase face-centered cubic (FCC) structure [21,22]. Several studies were conducted based on the CoCrFeNi HEA family, with particular emphasis on swelling behavior, He bubble evolution, and the effects of compositional modifications on irradiation-induced hardening [23–25]. For instance, Chen et al. [26] investigated He bubble evolution over a temperature range from 250 °C to 400 °C and reported an inverse relationship between He bubble density and temperature, while

bubble size showed limited coarsening. Their study further indicated that HEAs exhibit superior irradiation resistance compared to steels, which was attributed to severe lattice distortion and chemical disorder. Despite these promising findings, most irradiation studies on HEAs have been conducted using conventionally manufactured materials. The He irradiation behavior of AM HEAs remains largely unexplored.

The present work is not intended as a single-variable decoupling study; rather, it is designed as a comparative study of two representative AM FCC materials (LPBF 316L SS and LDED CoCrFeNi HEA). Under identical He irradiation conditions and a consistent TEM-based quantification protocol, we investigate the temperature dependence of He bubble size and area density in these two AM alloys, with the goal of comparing He bubble evolution in these representative AM states, establishing a comparable microstructural baseline, and identifying trend-level differences that motivate more rigorously controlled follow-up studies. In this work, we investigate He bubble evolution in LPBF-fabricated 316L SS and LDED-fabricated CoCrFeNi high-entropy alloys irradiated with 200 keV He⁺ ions to a peak damage level of 10 displacement per atom (dpa) at temperatures of 25 °C, 400 °C, and 600 °C. High-resolution electron microscopy was used to characterize He-bubble morphology across the irradiated depth, and quantitative measurements of bubble size and area density were performed within the peak He concentration region identified by SRIM simulations. This study provides a systematic comparative assessment of He bubble evolution in AM FCC alloys, elucidating the combined roles of irradiation temperature, AM-induced microstructure, and compositional complexity in governing irradiation resistance for nuclear energy applications.

2. Materials and Methods

In this work, bulk 316L SS samples were fabricated via LPBF on a Renishaw AM 400 system (Renishaw plc, Wotton-under-Edge, UK), using spherical 316L SS powder (Renishaw plc, Wotton-under-Edge, UK) with an average size of 27 µm. The manufacturer's standard processing parameters were used to produce nearly fully dense specimens, thereby providing a representative high-density LPBF 316L SS condition for comparison with the LDED CoCrFeNi HEA under identical irradiation conditions. However, optimization of LPBF process parameters for irradiation resistance remains an important direction for future work. The hatching laser power was 195 W, the layer thickness was 50 µm, the scan speed was 750 mm/s, the point distance was 60 µm, and a laser rotation angle of 67° was used in pulsed laser mode.

For comparison, a CoCrFeNi HEA was fabricated via LDED on an Optomec LENS MR-7 system (Optomec Inc., Albuquerque, NM, USA). A pre-alloyed HEA powder feed-stock (ATT Advanced Elemental Materials Co., Ltd., Albany, NY, USA) was sieved to the 45–150 µm range before use in the printing operation. The LDED process parameters were optimized through multiple trial builds, and the final parameter set was selected based on print quality, surface morphology, and minimal porosity. The optimized parameters were a laser power of 480 W, a scan speed of 10 mm/s, a powder feed rate of 3 g/min, a hatch spacing of 305 µm, and a layer height of 295 µm. It is noted that LPBF and LDED processes exhibit distinct thermal histories and solidification conditions, resulting in differences in grain morphology and defect structures. In the present study, these differences are treated as characteristics of the selected AM material states arising from their respective processing routes, rather than as independently controlled variables. Also, the bulk samples were fabricated in an inert atmosphere, with the O₂ level in the chamber below 20 ppm.

Following bulk fabrication, thin rectangular specimens with approximate dimensions of 3 × 3 × 1 mm³ were sectioned from both LPBF 316L SS and LDED HEA builds. The samples were mechanically polished to a mirror finish using progressively finer polishing

steps, achieving a final surface roughness of approximately 20 nm. Care was taken to ensure that the samples were free of surface cracks and exhibited uniform flatness prior to irradiation.

The prepared specimens were mounted on a thin molybdenum holder and irradiated with He ions at the Ion Beam Materials Laboratory at Los Alamos National Laboratory. Irradiation was performed using a single-beam He ion source at 200 keV. The total He ion fluence was 3.85×10^{17} ions/cm², corresponding to a peak damage level of approximately 10 dpa. Irradiations were conducted at three temperatures: 25 °C, 400 °C, and 600 °C. Each irradiation experiment was carried out for a total duration of approximately 8 h. The depth-dependent He concentration and displacement-damage profiles were estimated using SRIM simulations. The SRIM profile (Figure 1) indicates that the peak He concentration and associated bubble formation region occur at depths between approximately 510 and 610 nm from the irradiated surface. This peak damage band was used as the primary region of interest for subsequent microstructural characterization and quantitative analysis.

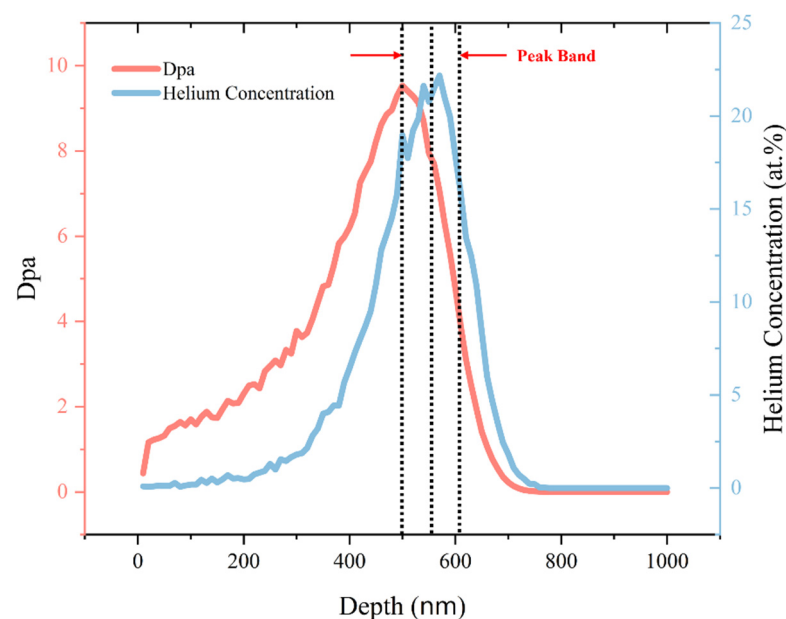


Figure 1. SRIM-simulated depth profiles of displacement damage (dpa) and He concentration for He ion irradiation. The peak damage band, highlighted by dashed lines, corresponds to the region that is used to guide TEM analysis of irradiation-induced microstructural features.

He-induced microstructural features in both 316L SS and HEA samples were characterized using transmission electron microscopy (TEM). Cross-sectional TEM lamellae corresponding to each irradiation temperature were prepared using focused ion beam (FIB) milling in a Hitachi NB5000 dual-beam scanning electron microscope (SEM) (Hitachi High-Technologies Corp., Tokyo, Japan). A tungsten protective surface layer was deposited prior to milling to preserve irradiation-induced features. TEM observations were conducted using a Hitachi-9000 TEM (Hitachi High-Technologies Corp., Tokyo, Japan) operated at an accelerating voltage of 300 keV at the IVEM-Tandem Facility, Argonne National Laboratory. Bright-field TEM images were acquired at multiple magnifications to capture the spatial evolution of He bubbles across the irradiated depth, with particular emphasis on the region of peak He concentration.

3. Results and Discussion

3.1. As-Printed Microstructure of LPBF 316L SS

Figure 2a shows the SEM microstructure of the LPBF-fabricated 316L-SS. The microstructure is characterized by large columnar grains aligned along the build direction, within which a mixture of equiaxed and columnar cellular substructures is observed. These fine substructures are a direct consequence of the rapid solidification conditions inherent to the LPBF process, where cooling rates can exceed $\sim 10^5$ K/s. Such high cooling rates promote cellular solidification and result in a refined sub-grain morphology within the larger columnar grains [27,28].

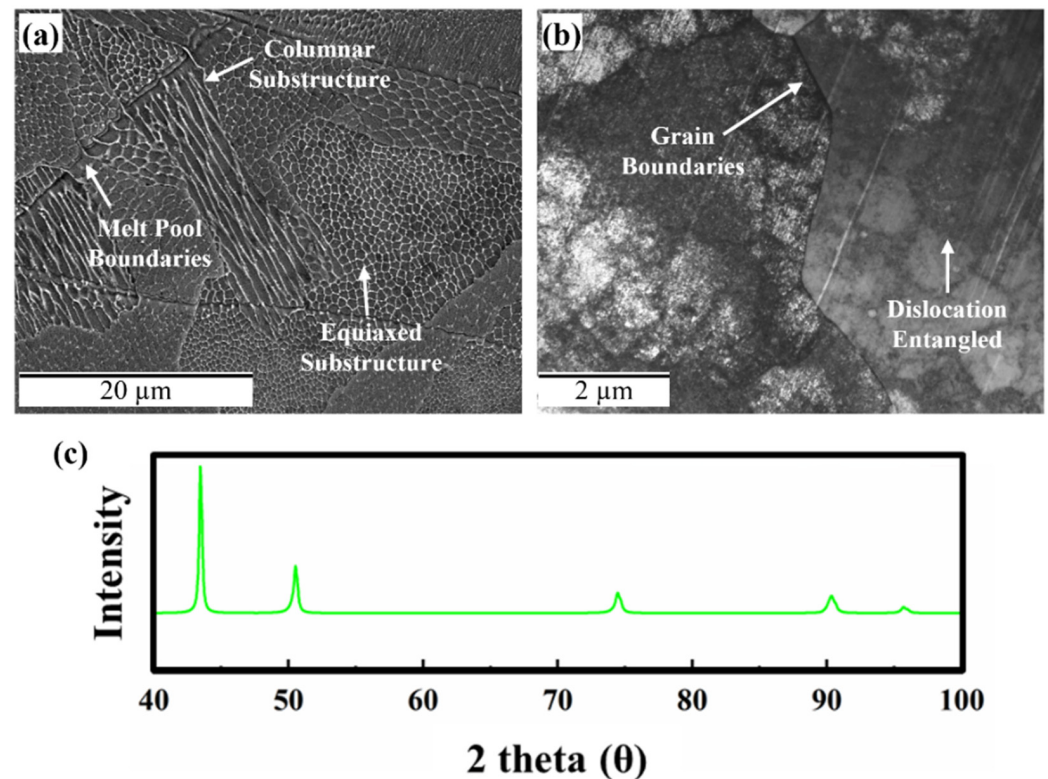


Figure 2. As-printed microstructure of LPBF-fabricated 316L SS: (a) SEM image showing melt pool boundaries and the coexistence of columnar and equiaxed cellular substructures formed during rapid solidification; (b) bright-field TEM image revealing grain boundaries and a high density of entangled dislocations within the austenitic matrix; and (c) XRD pattern confirming a single-phase austenitic FCC structure with no detectable secondary phases.

The TEM image shown in Figure 2b reveals a high density of entangled dislocations distributed primarily along the cellular boundaries. This dislocation-rich network is commonly reported in LPBF-fabricated 316L SS and is attributed to steep thermal gradients, repeated thermal cycling, and constrained solidification during layer-by-layer processing [29]. Quantitative analysis of the as-printed microstructure was performed to determine the characteristic cell size, sub-grain wall thickness, and dislocation density. The average cellular substructure size was measured to be approximately 807 ± 110 nm, while the average sub-grain wall thickness was found to be 102.7 ± 20.3 nm. The dislocation density was estimated to be $1.3 \times 10^{15} \text{ m}^{-2}$ using the Williamson–Hall method, following the procedure described in our previous work [27]. Despite the presence of these deformation-induced defects, phase analysis (Figure 2c) confirms that the as-printed 316L SS retains a single-phase austenitic structure, with no evidence of secondary phase formation. The combination of fine cellular substructures and high dislocation density is

expected to influence irradiation-induced defect evolution by providing a high density of potential defect sinks, which is discussed in subsequent sections.

3.2. Irradiation Microstructure of LPBF 316L SS

3.2.1. At 25 °C

Figure 3 presents TEM images of the LPBF 316L SS irradiated with He ions at room temperature, obtained from different depths within the irradiated layer. A clear depth-dependent variation in defect morphology and density is observed across the near-surface (top), peak-damage band, and bottom regions. In the top region (Figure 3b), the microstructure exhibits relatively uniform, diffuse contrast, without clearly resolved large cavities or voids, which is consistent with limited point-defect mobility at room temperature.

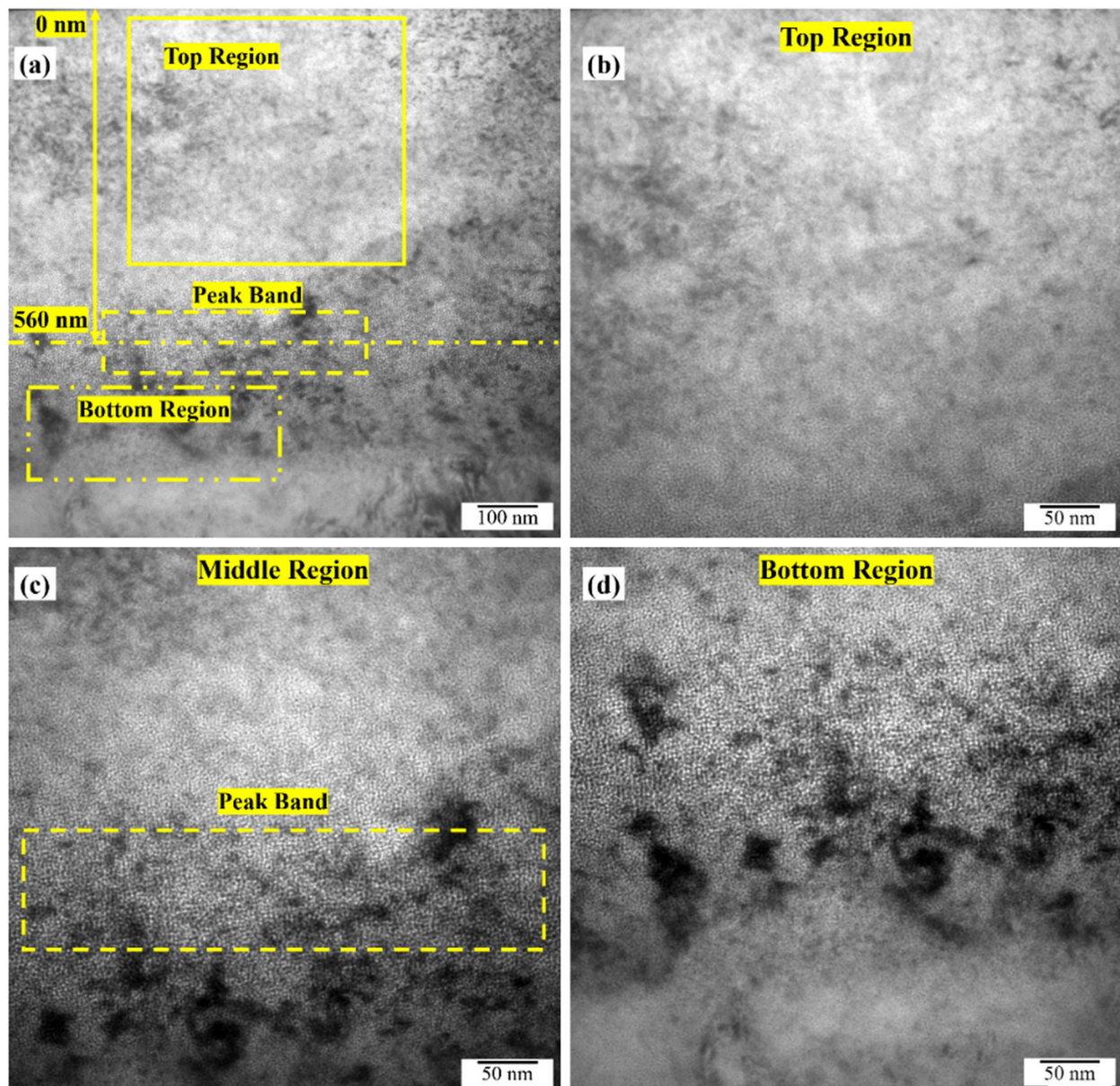


Figure 3. TEM images of LPBF-fabricated 316L SS irradiated with He ions at 25 °C: (a) cross-sectional overview showing the top region, peak damage band, and bottom region; (b) top region with diffuse contrast from fine irradiation-induced defect clusters; (c) peak damage band exhibiting enhanced defect contrast; and (d) bottom region showing localized defect clustering. Dashed lines indicate the peak damage region identified from simulated damage profiles in Figure 1.

Moving toward the middle of the irradiated layer (Figure 3c), a distinct damage band is observed at a depth corresponding to the peak of the displacement damage and He concentration profile. This peak band shows enhanced dark contrast relative to the surrounding regions, indicating a higher density of irradiation-induced defects. The defects in this region remain finely distributed and form a continuous band rather than discrete, well-defined bubbles or voids. Under overfocus imaging conditions, the sizes of these fine defects were statistically measured, with an average size of approximately 2.9 nm. Such localization of damage is commonly observed in ion-irradiated austenitic steels and reflects the strong coupling between the He implantation profile and the displacement-damage peak, where vacancy–He complexes are most likely to accumulate.

In the bottom region (Figure 3d), below the peak damage band, the defect contrast becomes more heterogeneous, with the appearance of darker clustered features and localized agglomeration. Despite the increased contrast, no large cavities or well-developed He bubbles are observed, suggesting that defect coarsening remains strongly suppressed at room temperature. The gradual reduction in defect density away from the peak band further confirms that the observed microstructural features are governed by the implanted He and damage depth profile rather than uniform bulk damage.

The observed defect morphology at 25 °C can be rationalized by the limited mobility of vacancies and He atoms at low temperature. Under these conditions, irradiation primarily leads to the formation and retention of small defect clusters, including vacancy–He complexes rather than the growth of resolvable bubbles or voids. In LPBF-built 316L SS, the high density of pre-existing dislocations and cellular sub-grain structures introduced during rapid solidification provides an enhanced sink strength for irradiation-generated point defects. These microstructural sinks promote defect recombination and limit long-range defect migration, thereby suppressing bubble growth and void formation. Similar behavior has been reported in previous studies on additively manufactured austenitic stainless steels, where the cellular dislocation network plays a critical role in mitigating defect coarsening under irradiation at low temperatures.

Overall, the room-temperature irradiation response of LPBF 316L SS is characterized by a high density of fine defect clusters localized near the peak-damage region, with no evidence of significant He-bubble growth or void swelling. This microstructural state is expected to contribute primarily to irradiation hardening via dispersed barrier mechanisms rather than volumetric swelling, providing a baseline for understanding the temperature-dependent evolution of irradiation damage.

3.2.2. At 400 °C

Figure 4 presents TEM images of LPBF-built 316L SS irradiated with He ions at 400 °C, revealing a pronounced evolution in defect morphology compared to room-temperature irradiation. A clear depth-dependent variation in defect size, density, and spatial distribution is observed across the top region, peak damage band, and bottom region of the irradiated layer. Unlike the fine, diffuse defect contrast observed at room temperature, the microstructure at 400 °C is dominated by well-resolved nanoscale He bubbles and defect clusters, indicating thermally activated defect migration and clustering.

In the top region (Figure 4b), a high density of small, spherical nanoscale bubbles is uniformly distributed throughout the microstructure. The bubbles are finely dispersed and relatively homogeneous in size, suggesting that He atoms and vacancies possess sufficient mobility at 400 °C to form stable vacancy–He complexes and small bubbles, but not enough mobility to promote extensive coarsening or bubble agglomeration. This region reflects a balance between defect generation and limited bubble growth, resulting in a dense population of small He bubbles.

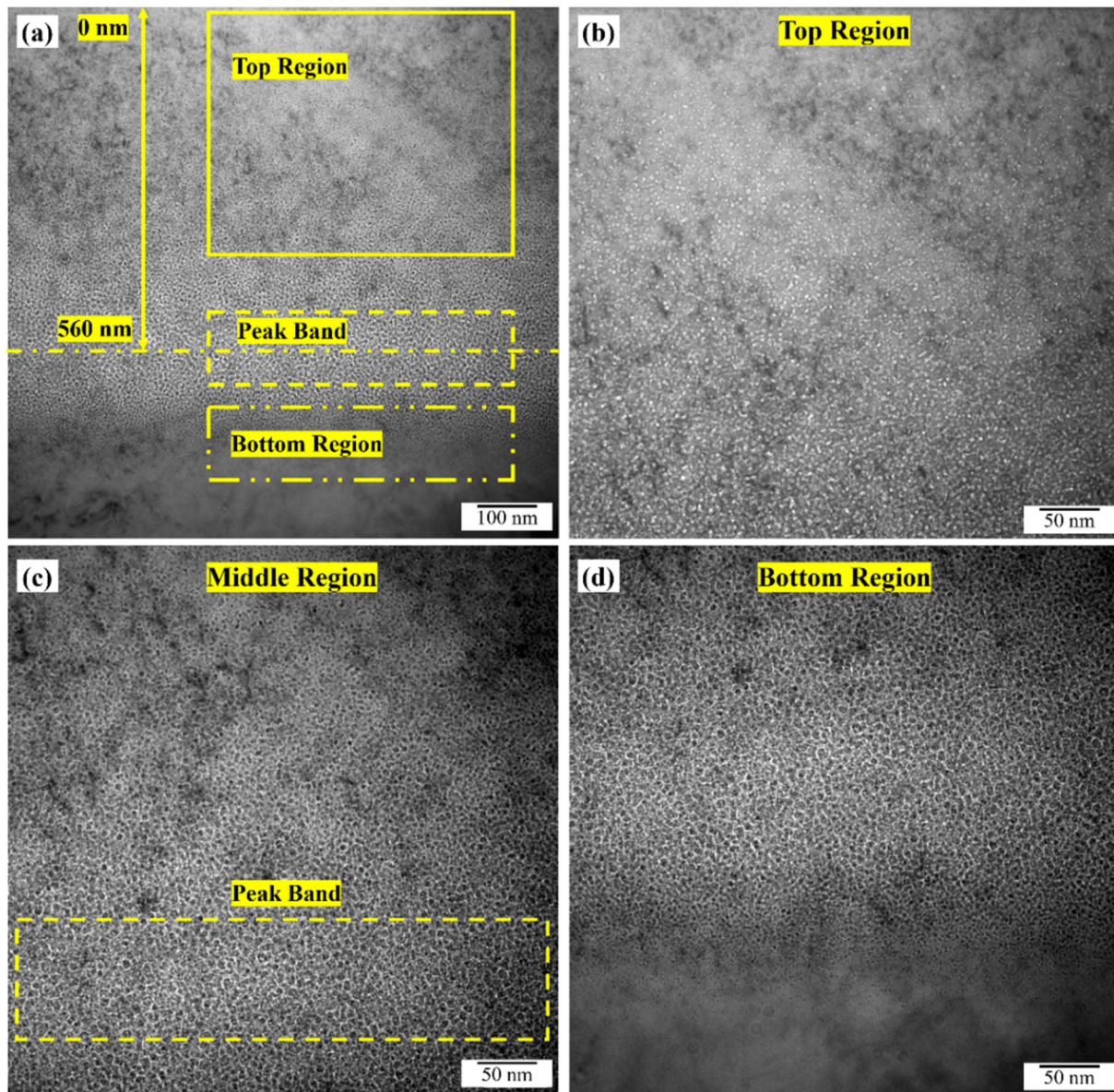


Figure 4. TEM images of LPBF-fabricated 316L SS irradiated with He ions at 400 °C: (a) cross-sectional overview showing the top region, peak damage band, and bottom region relative to the irradiated surface; (b) top region exhibiting a high density of nanoscale He bubbles; (c) middle region corresponding to the peak damage band with enhanced bubble density and size; and (d) bottom region showing uniformly distributed nanoscale bubbles below the peak damage band. Dashed lines indicate the peak damage region identified from simulated damage profiles.

The middle region corresponding to the peak damage band (Figure 4c) exhibits a distinct increase in defect density and bubble population. This region shows the highest concentration of nanoscale He bubbles, forming a continuous damage band aligned with the projected range of implanted He and the peak displacement-damage profile. In the bottom region (Figure 4d), the microstructure continues to exhibit a high density of nanoscale bubbles; however, the spatial distribution becomes more uniform and less localized compared to the peak band. Although bubble populations remain significant, there is no evidence of large cavities or void formation, indicating that while defect mobility is sufficient for bubble nucleation and growth at 400 °C, large-scale coarsening and void swelling mechanisms remain limited. The absence of large voids suggests that bubble growth is still kinetically constrained by defect trapping and recombination processes.

The defect morphology observed at 400 °C reflects a transition from a defect-retention-dominated regime at room temperature to a mobility-assisted clustering regime. At this intermediate temperature, vacancies and He atoms acquire sufficient thermal energy to migrate and interact, enabling the formation of stable He bubbles. However, the dense dislocation networks and cellular sub-grain structures inherent to the LPBF microstructure continue to act as effective defect sinks, limiting long-range defect migration and suppressing extensive bubble coarsening.

3.2.3. At 600 °C

Figure 5 shows the TEM images of LPBF-fabricated 316L SS irradiated with He ions at 600 °C, revealing a pronounced transformation in defect morphology and spatial distribution compared to irradiation at lower temperatures. At this elevated temperature, irradiation damage is dominated by the formation, growth, and coarsening of He bubbles, accompanied by strong depth-dependent variations across the top region, peak damage band, and bottom region. The defects are significantly larger and more clearly resolved than those observed at 25 °C and 400 °C, indicating that thermally activated defect mobility plays a dominant role in microstructural evolution at 600 °C.

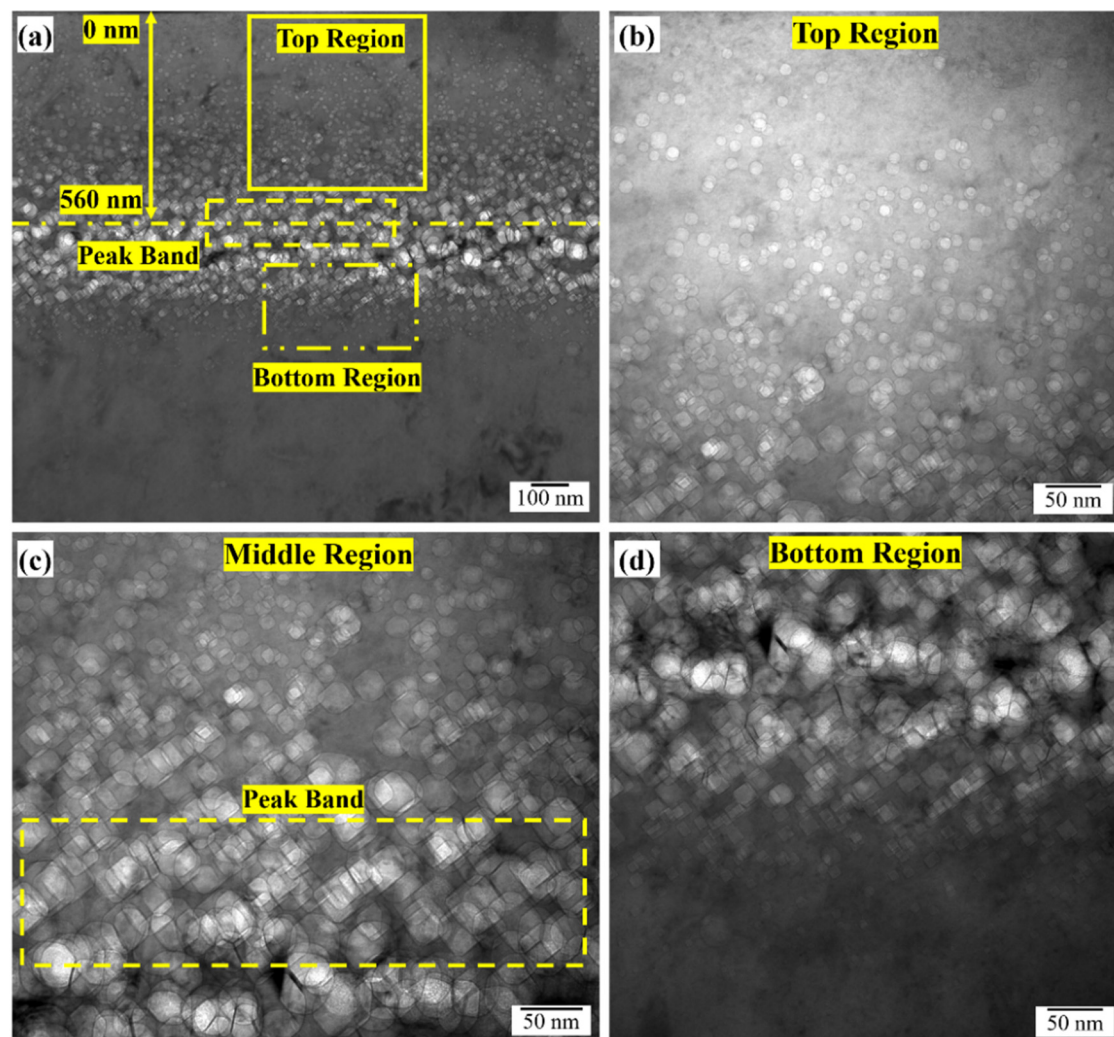


Figure 5. TEM images of LPBF-fabricated 316L SS irradiated with He ions at 600 °C: (a) cross-sectional overview showing the top region, peak damage band, and bottom region relative to the irradiated surface; (b) top region displaying coarsened He bubbles; (c) middle region corresponding to the peak

damage band with the highest bubble density and pronounced bubble coarsening; and (d) bottom region showing further bubble growth and coalescence below the peak damage band. Dashed lines indicate the peak damage region identified from simulated damage profiles.

In the top region (Figure 5b), a high population of He bubbles with relatively larger diameters is observed, distributed throughout the matrix. Compared to the finer, more uniform bubble populations observed at 400 °C, those at 600 °C exhibit larger sizes and greater contrast, reflecting enhanced diffusion of both He atoms and vacancies. This increased mobility enables vacancy–He complexes to grow by absorbing additional vacancies and He atoms, resulting in noticeable bubble coarsening even near the surface, where displacement damage is relatively lower.

The middle region corresponding to the peak damage band (Figure 5c) shows the most pronounced microstructural evolution. This region exhibits the highest bubble density and the largest bubble sizes, forming a well-defined band aligned with the projected He range and peak displacement damage. The simultaneous presence of high He concentration and elevated vacancy production, combined with enhanced thermal diffusion at 600 °C, promotes rapid bubble growth and coalescence. The increased bubble size indicates that bubble growth kinetics dominate over defect recombination, leading to localized accumulation of large He-filled cavities. In the bottom region (Figure 5d), the microstructure shows extensive bubble coarsening and clustering, with some bubbles coalescing into elongated or irregular features.

The defect morphology observed at 600 °C reflects a fundamental shift from sink-controlled defect retention at lower temperatures to diffusion-dominated defect evolution. At this temperature, the mobility of vacancies and He atoms is sufficiently high to enable long-range defect migration, promoting He bubble growth, coalescence, and spatial redistribution. Although dislocations and cellular sub-grain boundaries inherent to the LPBF microstructure continue to interact with irradiation-generated defects, their effectiveness in suppressing bubble coarsening is significantly reduced under these conditions. This reduction may be further influenced by the combined effects of elevated temperature and prolonged irradiation at 600 °C. Previous studies have shown that under purely thermal annealing, the cellular dislocation network characteristic of LPBF-fabricated 316L SS remains moderately stable up to approximately 600 °C [30], exhibiting slight dislocation rearrangement and modest cell coarsening, whereas more pronounced cellular dissolution is typically reported at higher temperatures (≥ 800 °C) [31]. However, the present experiments involve simultaneous He irradiation and thermal exposure. Under irradiation conditions, continuous displacement cascades generate nonequilibrium point defects and can enhance atomic mobility through irradiation-enhanced diffusion, thereby accelerating dislocation climb, defect annihilation, and solute redistribution beyond what would occur during thermal annealing alone. Similar irradiation-assisted recovery behavior has been reported in the literature, demonstrating that proton irradiation at intermediate temperatures (300 °C) promoted recovery of the cellular structure in LPBF 304L SS, attributed to irradiation-enhanced diffusion [32]. Therefore, although direct evidence of cellular degradation was not obtained in the present study, the observed bubble coarsening at 600 °C is consistent with diffusion-dominated defect evolution, combined with thermally and irradiation-assisted reductions in effective sink density.

3.3. As-Printed Microstructure of CoCrFeNi HEA

Figure 6a shows the SEM microstructure of the LDED-fabricated CoCrFeNi HEA, which is characterized by well-defined cellular substructures with an average sub-grain size of approximately 2.5 μm . In this, no evidence of elemental segregation is observed, as a uniform elemental distribution is maintained across the cellular substructure regions. This

was confirmed from the SEM-EDS maps shown in Supplementary Figure S1. Figure 6b presents a TEM image of the as-printed CoCrFeNi HEA, revealing a high density of dislocations distributed throughout the FCC matrix. The dislocations appear randomly distributed, and no distinct dislocation cell structures are observed within the grains. This suggests that, unlike LPBF-fabricated 316L SS, the as-printed CoCrFeNi HEA does not exhibit a pronounced cellular dislocation network, but instead accommodates thermal stresses primarily through a more homogeneous dislocation distribution. The phase constitution of the as-printed CoCrFeNi HEA is confirmed by the XRD pattern shown in Figure 6c, which indicates a single-phase FCC structure with no detectable secondary phases or intermetallic compounds.

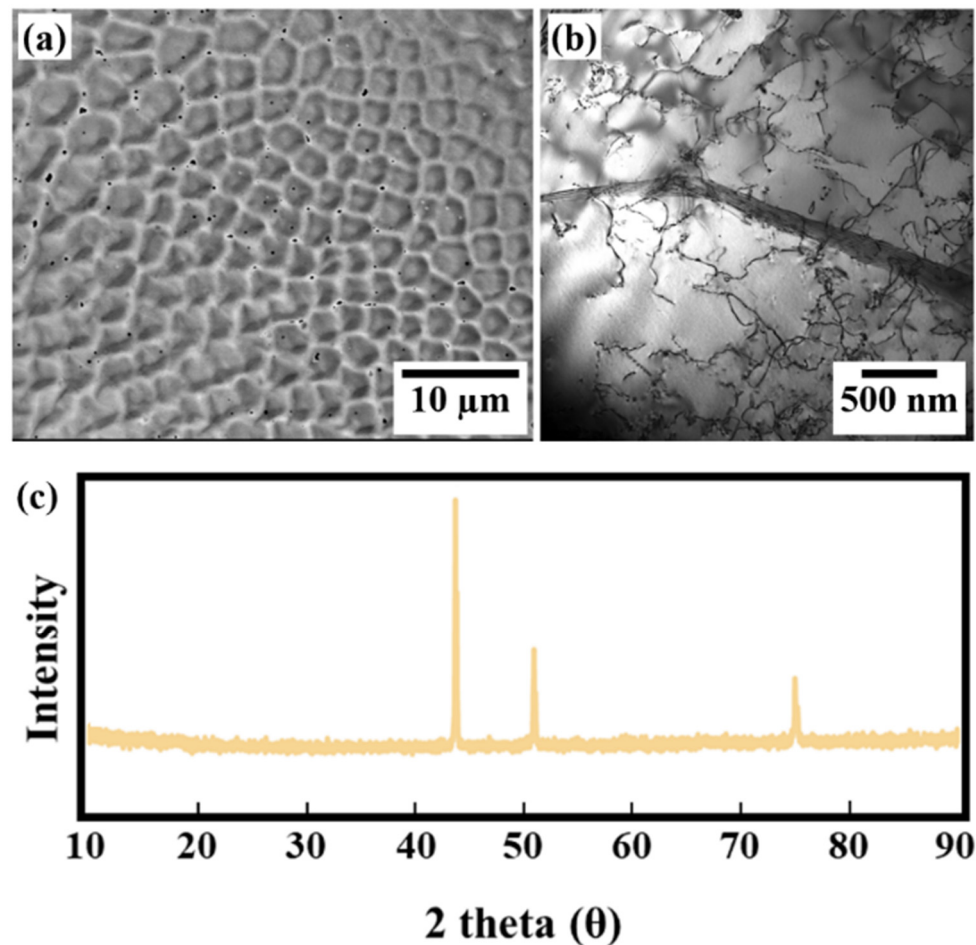


Figure 6. As-printed microstructure of LDED-fabricated CoCrFeNi HEA: (a) SEM image showing cellular sub-grain structures formed during solidification; (b) TEM image revealing a dislocation distributed within the FCC matrix; and (c) XRD pattern confirming a single-phase FCC structure with no detectable secondary phases.

Although both alloys exhibit a single-phase FCC structure, their as-printed microstructures differ significantly due to the different AM processes employed. The LPBF-fabricated 316L SS contains a refined cellular substructure with an average cell size of ~ 807 nm and a high dislocation density of $\sim 1.3 \times 10^{-15} \text{ m}^{-2}$, forming a dense cellular dislocation network that acts as an effective sink for irradiation-induced point defects. In contrast, the LDED-fabricated CoCrFeNi HEA exhibits larger sub-grain structures with an average size of $\sim 2.5 \mu\text{m}$ and a more homogeneous dislocation distribution without a pronounced cellular dislocation network. These quantitative differences in microstructural length scale and dislocation organization may influence the effective sink density available for defect

trapping, which can affect He bubble nucleation and growth during irradiation. Therefore, while the present study compares two representative AM FCC alloys under identical irradiation conditions, the observed irradiation response reflects the combined effects of alloy chemistry and process-induced microstructure.

3.4. Irradiation Microstructure of CoCrFeNi HEA at 25 °C, 400 °C, 600 °C

Figure 7 presents the irradiation-induced microstructural evolution of the LDED-fabricated CoCrFeNi HEA subjected to He irradiation at 25 °C, 400 °C, and 600 °C. For direct comparison with LPBF-fabricated 316L SS, the analysis focuses on the peak-damage band region at all irradiation temperatures, corresponding to the projected He range and maximum displacement damage. Both materials were irradiated under identical conditions.

At 25 °C (Figure 7(a1,b1)), the peak damage region in the CoCrFeNi HEA exhibits a high density of fine defect contrast without clearly resolved He bubbles, indicating that irradiation damage is primarily retained in the form of small defect clusters, with an average defect size of approximately 3.4 nm. A similar defect-retention-dominated regime is observed in LPBF-fabricated 316L SS at 25 °C. In both materials, the limited mobility of vacancies and He atoms at low temperature plays the primary role in suppressing bubble nucleation and growth, leading to the retention of irradiation damage as nanoscale defect clusters rather than resolvable bubbles. While this low-temperature defect retention is common to both alloys, the microstructural features governing defect trapping differ. In LPBF 316L-SS, defect retention is further influenced by the presence of a dense cellular dislocation network formed during rapid solidification, which provides additional trapping sites for irradiation-generated defects. In contrast, the CoCrFeNi HEA lacks a pronounced cellular dislocation structure, and defect retention is instead likely associated with restricted defect mobility and lattice distortion arising from its chemical complexity. Despite these differences in microstructural characteristics, both materials effectively suppress the formation of resolvable He bubbles at 25 °C.

At 400 °C (Figure 7(a2,b2)), the CoCrFeNi HEA forms nanoscale He bubbles within the peak damage band, indicating enhanced defect mobility compared to 25 °C. Unlike LPBF-fabricated 316L SS, where bubble spatial distribution is influenced by the underlying cellular sub-grain structure, the HEA does not show obvious bubble localization associated with specific microstructural features within the observed region. This behavior is consistent with the absence of a dense cellular dislocation network in the HEA and suggests that defect evolution at this temperature is less constrained by localized microstructural sinks. Bubble growth remains limited at this intermediate temperature, with no evidence of extensive coarsening or void formation.

At 600 °C (Figure 7(a3,b3)), pronounced He bubble growth and coarsening are observed in the CoCrFeNi HEA, indicating a transition toward diffusion-dominated defect evolution at elevated temperature. While similar bubble coarsening is observed in LPBF-fabricated 316L SS under identical irradiation conditions, the mechanisms governing defect evolution differ due to their distinct as-printed microstructures. In the HEA, bubble growth at 600 °C primarily reflects enhanced defect mobility within a chemically complex FCC lattice, rather than changes in microstructural sink stability.

At this stage, the comparison between LPBF-fabricated 316L SS and LDED-fabricated CoCrFeNi HEA is based on qualitative microstructural observations. Quantitative analyses of the He bubble size distributions and area bubble densities will be presented in the subsequent section to further elucidate differences in the evolution of irradiation damage between the two materials.

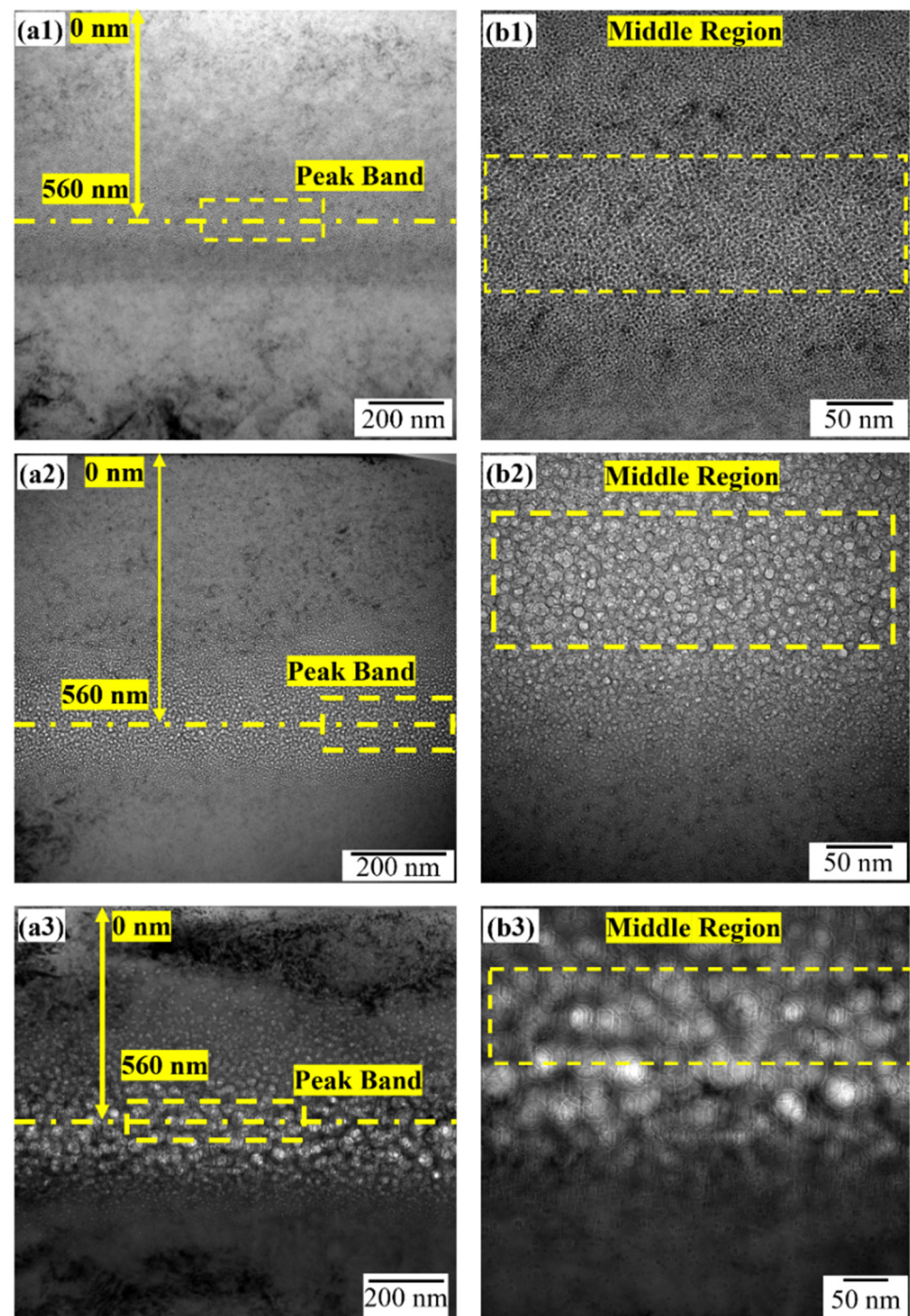


Figure 7. TEM images of the irradiation microstructure of LDED-fabricated CoCrFeNi HEA at different temperatures, focusing on the peak-damage band region: (a1,b1) 25 °C, showing fine defect contrast without clearly resolved He bubbles; (a2,b2) 400 °C, showing the formation of nanoscale He bubbles within the peak damage band; and (a3,b3) 600 °C, showing pronounced He bubble growth and coarsening. Dashed boxes indicate the peak damage band corresponding to the projected He range.

3.5. Quantitative Comparison of Bubble Size and Areal Bubble Density for 316L SS and HEA

Quantitative analyses of the He bubble size and areal bubble density were quantified from bright-field TEM images acquired within the SRIM-predicted peak-damage band (Figure 1). For each material and irradiation temperature, a single cross-sectional lamella was examined. Within the peak-damage band of each lamella, multiple independent fields of view were analyzed to ensure statistical representativeness. For bubble size

measurements, three independent fields of view were selected along the horizontal direction of the peak band. For areal bubble density calculations, four independent fields of view within the peak-damage region were analyzed for each condition. The analyzed peak-band region corresponded to an approximate height of ~ 100 nm across an image width of ~ 1 μm , yielding an analyzed areal density of ~ 0.1 μm^2 per condition. Bubble diameters were measured using image analysis software, and areal bubble densities were calculated by counting the number of bubbles within the defined peak-band region and normalizing by the analyzed area. The reported mean values represent the average over all analyzed fields of view. Error bars correspond to the standard deviation derived from field-to-field measurements within the peak band.

The mean He bubble diameter as a function of irradiation temperature for both materials is shown in Figure 8a. At 25 °C, both 316L SS and HEA exhibit small bubble sizes, with mean diameters of only a few nanometers, indicating limited defect mobility and suppressed bubble growth. With increasing irradiation temperature to 400 °C, a noticeable increase in bubble size is observed in both materials, reflecting enhanced vacancy–He diffusion and clustering. At 600 °C, a significant increase in bubble diameter is observed for both materials, accompanied by larger standard deviations, suggesting extensive bubble coarsening and partial coalescence.

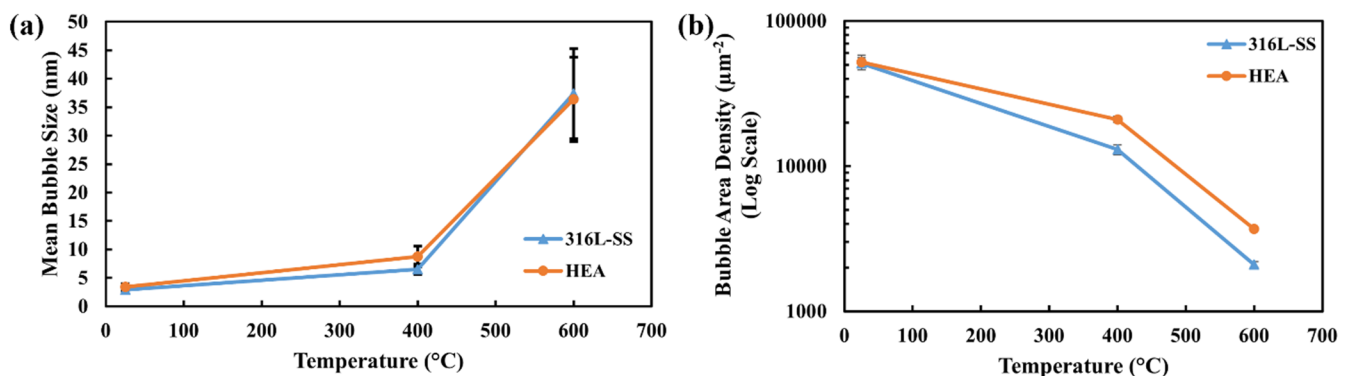


Figure 8. Quantitative comparison of He bubble characteristics in LPBF-fabricated 316L SS and LDED-fabricated CoCrFeNi HEA as a function of irradiation temperature: (a) mean He bubble size showing a pronounced increase with temperature for both materials and (b) area bubble density plotted on a logarithmic scale, showing a systematic decrease with increasing temperature.

A direct comparison reveals that the HEA exhibits a larger average bubble diameter than 316L SS at 400 °C, while also retaining a higher areal bubble density, as discussed below. This simultaneous increase in both bubble size and density at the intermediate temperature suggests that He bubble nucleation and growth are influenced by the complex chemical environment of the HEA, which may promote He retention while still allowing bubble growth. At 600 °C, the mean bubble sizes in both materials become comparable within experimental uncertainty, indicating that thermally activated coarsening processes dominate bubble evolution at higher temperatures.

The corresponding variation in He areal bubble density with irradiation temperature is shown in Figure 8b. The areal bubble density, N_A , was calculated using the relation $N_A = \frac{N}{A}$, where N is the total number of resolvable bubbles within a defined region of interest, and A is the analyzed area of that region. The areal density values are plotted on a logarithmic scale in Figure 8b to clearly capture the large variation in areal bubble density across irradiation temperatures.

For both materials, the areal bubble density decreases monotonically with increasing irradiation temperature. At 25 °C, a high density of small bubbles is observed, consistent with nucleation-dominated behavior and limited bubble growth. As the temperature

increases to 400 °C and 600 °C, the areal bubble density decreases substantially as bubbles grow, coalesce, and consume smaller bubbles, leading to a reduced number of discrete bubble features. This inverse relationship between bubble size and areal bubble density reflects the transition from nucleation-controlled to coarsening-dominated regimes as temperature increases.

Comparatively, the HEA consistently exhibits a higher areal bubble density than 316L SS at both 400 °C and 600 °C, despite also showing larger bubble sizes at 400 °C. This behavior suggests that the compositional complexity and associated lattice distortion in the HEA may reduce defect mobility and suppress bubble coalescence, allowing a higher number of bubbles to be retained even as individual bubbles grow. At the highest irradiation temperature, the difference in areal bubble density between the two materials diminishes, indicating that thermal effects increasingly govern He bubble evolution in both systems.

For clarity, the average bubble diameters and corresponding area densities for all irradiation temperatures are summarized in Table 1. The tabulated data complements the graphical trends shown in Figure 8 and provides a concise quantitative comparison between LPBF 316L SS and LDED HEA.

Table 1. Bubble size and areal bubble density for LPBF 316L SS and LDED CoCrFeNi HEA at different temperatures.

Temperature (°C)	316L SS		CoCrFeNi HEA	
	Bubble Size (nm)	Areal Bubble Density (Number/ μm^2)	Bubble Size (nm)	Areal Bubble Density (Number/ μm^2)
25	2.9 ± 0.6	$(5.1 \pm 0.5) \times 10^4$	3.4 ± 0.6	$(5.2 \pm 0.6) \times 10^4$
400	6.5 ± 0.9	$(1.3 \pm 0.1) \times 10^4$	8.7 ± 1.8	$(2.1 \pm 0.1) \times 10^4$
600	37.4 ± 7.9	$(2.1 \pm 0.1) \times 10^3$	36.4 ± 7.4	$(3.7 \pm 0.1) \times 10^3$

The variation in the bubble size and areal bubble density also depends upon the difference in composition between the 316L SS and CoCrFeNi HEA. Compared with 316L SS, equiatomic CoCrFeNi is a representative FCC multi-principal-element alloy. Its higher chemical complexity and configurational mixing entropy lead to more pronounced local chemical fluctuations, thereby altering the interplay between point defects and gas atoms in the lattice and modifying defect evolution pathways [33,34]. In CoCrFeNi, variations in the local atomic environment from site to site lead to a markedly broad distribution of vacancy formation energies [35], and this broader defect-energy landscape may alter the evolution pathway of He–defect complexes and the coarsening behavior of the bubble population, thereby contributing to the retention of a higher areal bubble density under the same irradiation conditions [36]. In addition, bubble growth depends not only on a continuous supply of He but also strongly on the availability of vacancies, which provide the free volume required for bubble expansion [36,37]. First-principles calculations for CoCrFeNi-based multi-principal-element alloys have shown that vacancy formation energies span a wide range and include relatively low-energy segments at certain sites, which favor the formation and stabilization of local vacancies and vacancy clusters within a specific temperature window [35]. Therefore, at 25 °C and 400 °C, CoCrFeNi not only more readily generates and retains a larger number of vacancy–He nucleation sites, but may also access a more sufficient vacancy supply at an earlier stage, promoting faster growth of some bubbles and leading to a larger average bubble size than in 316L. As the temperature increases to 600 °C, bubble growth is significantly enhanced and becomes increasingly dominated by thermally activated kinetics, causing the average bubble size in the two materials to converge. However, because nucleation and population evolution remain influenced by composition-dependent distributions of defect energetics and dif-

ferences in vacancy–He stability, CoCrFeNi still maintains a higher bubble density. The present experimental observations are qualitatively consistent with this interpretation. In particular, the CoCrFeNi HEA exhibits both a larger bubble size and a higher areal bubble density than 316L SS at 400 °C, suggesting that bubble nucleation and growth processes may be influenced by differences in vacancy availability and defect energetics associated with the chemically complex lattice. Nevertheless, the current TEM observations do not directly resolve the early-stage nucleation behavior of vacancy–He complexes. In future work, in situ ion irradiation TEM experiments will be performed to directly monitor the nucleation and growth of He bubbles, which would provide further experimental insight into the proposed mechanism.

4. Conclusions

In this work, the He irradiation-induced bubble evolution in LPBF 316L SS and LDED CoCrFeNi HEA was quantitatively investigated using 200 keV He⁺ irradiation to a peak damage level of 10 dpa at 25 °C, 400 °C, and 600 °C. TEM combined with image-based analysis was employed to evaluate the He bubble size and areal bubble density within the peak He concentration region identified by SRIM simulations. The study provides a direct, side-by-side comparison of He damage behavior in two AM-fabricated FCC alloy systems:

1. In both LPBF 316L SS and LDED CoCrFeNi HEA, increasing irradiation temperature leads to significant bubble coarsening accompanied by a monotonic decrease in areal bubble density, indicating a transition from nucleation-dominated behavior at 25 °C to coarsening-dominated behavior at 600 °C.
2. The areal bubble density in LPBF 316L SS decreases from $(5.1 \pm 0.5) \times 10^4 \mu\text{m}^{-2}$ at 25 °C to $(1.3 \pm 0.1) \times 10^4 \mu\text{m}^{-2}$ at 400 °C and $(2.1 \pm 0.1) \times 10^3 \mu\text{m}^{-2}$ at 600 °C, while the mean bubble diameter increases from $2.9 \pm 0.6 \text{ nm}$ to $37.4 \pm 7.9 \text{ nm}$, reflecting strong temperature-activated bubble growth and coalescence.
3. The HEA follows a similar temperature-dependent trend but retains a higher areal bubble density than 316L SS at elevated temperatures, decreasing from $(5.2 \pm 0.6) \times 10^4 \mu\text{m}^{-2}$ at 25 °C to $(2.1 \pm 0.1) \times 10^4 \mu\text{m}^{-2}$ at 400 °C and $(3.7 \pm 0.1) \times 10^3 \mu\text{m}^{-2}$ at 600 °C.
4. At 400 °C, the HEA simultaneously exhibits a larger mean bubble size ($8.7 \pm 1.8 \text{ nm}$) and a higher areal bubble density compared to LPBF 316L SS ($6.5 \pm 0.9 \text{ nm}$), suggesting that compositional complexity and lattice distortion reduce defect mobility and suppress bubble coalescence. At 600 °C, bubble sizes in both materials become comparable within experimental uncertainty.

Overall, these results demonstrate that while AM-induced microstructures influence He bubble evolution, alloy chemistry plays a critical role in controlling He bubble retention at intermediate irradiation temperatures, whereas temperature-driven diffusion and coarsening dominate at higher temperatures. The combined consideration of processing route, microstructure, and compositional complexity is therefore essential for designing irradiation-tolerant materials for advanced nuclear energy applications.

The differences observed in this study arise from the combined effects of alloy chemical complexity and AM process-induced microstructure. The current experimental design has not yet fully decoupled the contributions of these two factors. In future work, we will design more controlled experiments to separate these variables and enable a more rigorous comparison of the irradiation tolerance of the two materials.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app16062750/s1>, Figure S1: (a) SEM-BSE image of LDED fabricated CoCrFeNi HEA, and the corresponding EDS elemental maps showing the distribution of (b) Cr,

(c) Fe, (d) Ni, and (e) Co. All elemental maps indicate homogeneous elemental distribution at the micron scale.

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