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ORIGINAL REPORT



Response of solidification cellular structures in additively manufactured 316 stainless steel to heavy ion irradiation: an *in situ* study

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ABSTRACT

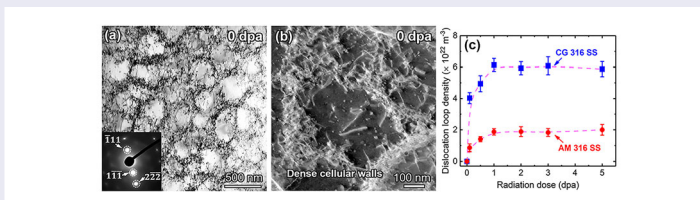
In-core or cladding structural materials exposed to heavy ion irradiation often suffer serious irradiation-induced damages. Introducing defect sinks can effectively mitigate irradiation-induced degradation in materials. Here, we investigated the radiation response of additively manufactured 316 austenitic stainless steel with high-density solidification cellular structures via *in situ* Kr⁺⁺ irradiation at 400°C to 5 dpa. The study shows that the cellular walls with trapped dislocations can serve as effective defect sinks, thus reduce dislocation loop density compared with the conventional coarse-grained counterparts. This study provides a positive step for the potential applications of radiation-resistant, additively manufactured steels in advanced nuclear reactors.

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Additive manufacturing; 316 austenitic stainless steels; cellular structures; *in situ* radiation; radiation damages



IMPACT STATEMENT

The solidification cellular walls with trapped dislocations in additively manufactured 316 SS can serve as effective defect sinks that prominently reduce irradiation-induced defect density compared with the conventional coarse-grained counterparts.

1. Introduction

Additive manufacturing of metallic materials has drawn increasing attentions because of its advantages, such as relatively short lead times and design freedom to process complex parts and geometry compared with other conventional manufacturing techniques [1,2]. Among various additive manufacturing methods, laser powder-bed-fusion (L-PBF) technique is one of the popular selective laser melting (SLM) methods for layer-by-layer construction of bulk metallic materials [2,3]. Previous studies on 316 austenitic stainless steel (SS) processed by L-PBF showed better overall mechanical properties in terms of high strength and high ductility compared with their wrought counterparts [4–6]. These excellent properties were ascribed to the hierarchically heterogeneous microstructure consisting of solidification cellular structures, low angle grain boundaries (LAGBs), and

precipitates [4]. Of particular interest is the solidification cellular structures, which have been frequently reported in additively manufactured (AM) Fe-, Co- and Al-based alloys [7]. These cellular structures, consisting of cellular walls with different composition from the matrix and trapped dislocations, can strongly influence the mechanical properties, and may impact radiation resistance of materials as well. Note that these solidification cellular structures are not the same as conventional dislocation cell walls despite the morphology similarities [4].

Recently, there are increasing studies that explore the feasibility of applying AM 316 stainless steel (SS) as components for nuclear industry by judging their microstructure stability, mechanical properties, and corrosion sensitivity under high-temperature irradiation [8–11]. One major challenge is that the microstructure of metallic materials subjected to high-energy particle irradiation

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undergoes serious degradation, forming various types of defects, such as Frenkel pairs, dislocation loops, etc., which further undermine the mechanical properties of irradiated materials [12–14]. An effective way to mitigate irradiation-induced degradation in materials is to introduce high-density defect sinks [15], such as free surfaces [16], high angle grain boundaries [17,18], twin boundaries [19], and phase interfaces [20]. Recent *ex situ* proton and helium ion irradiation studies on AM 316 SS found that dense dislocations trapped inside solidification cellular structures tended to undergo recovery and recrystallization during radiation, and may serve as defect sinks, thus help alleviate void swelling and helium bubble formation [9,10]. However, studies on heavy ion irradiation of AM 316 SS with cellular structures remain scarce, and the underlying mechanisms of the microstructural evolution of cellular structures subjected to high-temperature irradiation are largely unknown.

In this work, we investigated the radiation response of cellular structures in AM 316 SS up to 5 dpa at 400°C using *in situ* Kr^{++} irradiation technique in comparison with conventional coarse-grained (CG) 316 SS. The microstructure evolutions, including the variation of cellular structures and their interactions with irradiation-induced defects were analyzed in detail. Possible defect-sink interaction mechanisms are discussed. This study provides a forward step for the potential applications of radiation-tolerant AM austenitic SS for nuclear industry.

2. Materials and methods

The 316L SS was fabricated by a Concept Laser M2 PBF machine, with a Gaussian beam size of 54 μm in diameter. Transmission electron microscopy (TEM) samples for *in situ* irradiation experiment were prepared by first mechanically grinding 3 mm disks down to 70–80 μm , and then twin-jet polished in the solution of 5vol% perchloric acid and 95vol% ethanol with a voltage of 65 V at -25°C . *In situ* irradiation experiment on CG and AM 316 SS was performed in the Intermediate Voltage Electron Microscopy (IVEM) facility at Argonne National Laboratory. Before irradiation, the TEM samples were annealed at 400°C for 30 min to evaluate the thermal stability of the cellular structures. Then the samples were irradiated to a maximum fluence of 3.2×10^{15} ions/ cm^2 (~ 5.5 dpa) at 400°C with 1 MeV Kr^{++} beam at a dose rate of 0.0011 dpa/s.

3. Results

Bright field (BF) and corresponding dark field (DF) TEM micrographs of the solidification cellular structures before irradiation in Figure 1(a,b) show that high-density

dislocations are decorated along cellular walls, while the cell interior is relatively clean (dislocation-free). The irradiated region has an average cell size of $\sim 480 \pm 130$ nm (Figure 1(c)). EDS chemical maps in Figure S1(a1–a6) illustrate obvious Fe depletion, Cr and Mo segregation to the cellular walls, and some Si-rich particles with a dimension of ~ 20 –50 nm along the cellular walls. Figure 1(d) depicts the depth dependent irradiation dose and Kr^{++} concentration profiles calculated by SRIM software.

DF TEM micrographs in Figure 2 compare the microstructural evolution of CG 316 with AM 316 during *in situ* irradiation up to 2 dpa at 400°C. The cellular structures in AM 316 SS are very stable at 400°C, $\sim 0.4 T_m$ (T_m : melting temperature), as confirmed in Figure S2 and Figure S3. After irradiation to 0.1 dpa, CG 316 SS sample was swarmed with high-density small defect clusters or loops (small white dots), while the AM 316 SS was nearly intact with fewer white dots in the cell interiors (Figure 2(b,g)). The magnified DF images show obvious defect density difference in the two samples irradiated to 0.1 dpa. As the dose increased to 0.5 dpa in Figure 2(c,h), the loop density in both samples increased, but appeared to be greater in the CG specimen than in the AM 316 SS. After irradiation to higher doses (1 and 2 dpa) in Figure 2(d,e) and (i–j), the loop density in both samples remained unchanged. The corresponding BF TEM images (Figure S4) show similar phenomenon.

To further understand the different loop densities in irradiated CG and AM 316 SS, post-irradiation studies and detailed *in situ* video analyses were performed. Scanning transmission electron microscopy (STEM) images of cellular structures in Figure 3(a,b) and Figure S5 show that, after irradiation to 5 dpa, the dense cellular walls became relatively diffused and more dislocation lines marked by arrows were observed in the irradiated cell interior (Figure 3b). The image intensity profiles across the cellular wall in Figure 3(c) (measured at the same tilting/imaging condition) illustrate that the cellular wall thickness increased from ~ 100 nm (before radiation) to 200 nm after irradiation to 5 dpa. Comparison of dose-dependent variation of loop density in Figure 3(d) shows that the loop density in CG 316 SS is nearly four times as much as that in the irradiated AM 316 SS across all dose level (0–5 dpa). In addition, post-irradiation TEM analysis in Figure S6 confirms a prominently higher loop density in irradiated CG 316 than that in AM sample.

Figure 4 shows the detailed *in situ* video snapshots captured from 0.11 to 5.5 dpa. The observation is mainly focused on the interactions between loops, dislocation lines and cellular walls, and the migration of trapped dislocation lines. At least four representative scenarios,

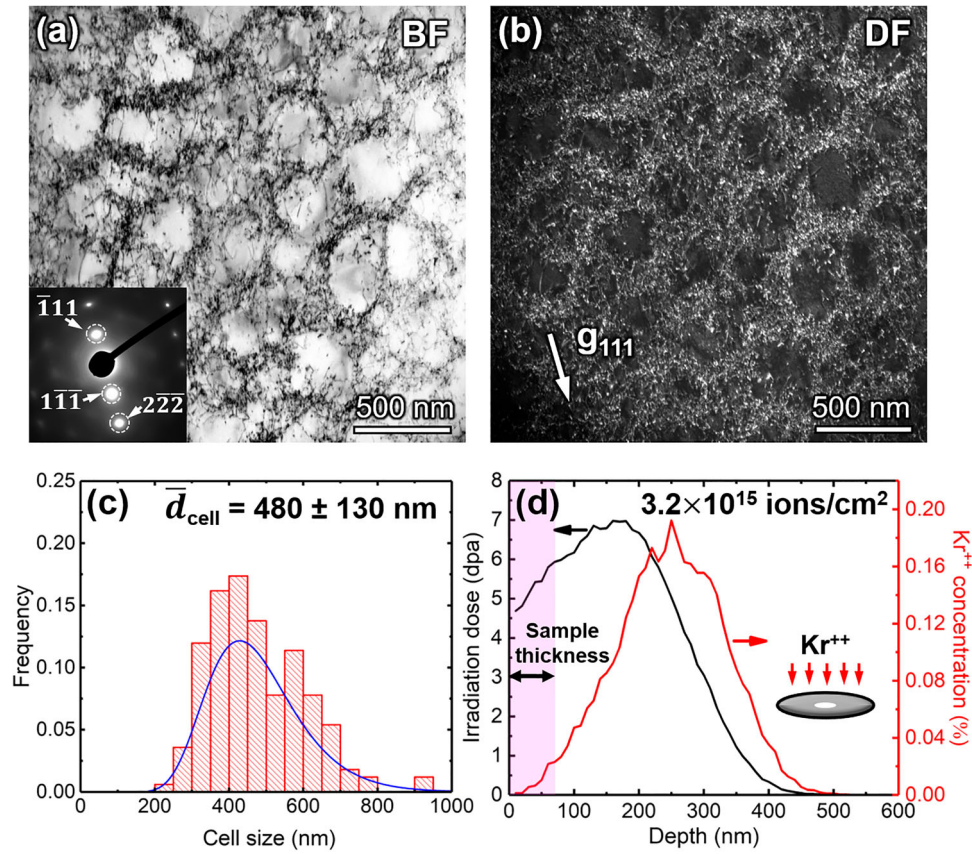


Figure 1. (a–b) BF and corresponding DF TEM images of the solidification cellular structures in AM 316 SS before irradiation. (c) The size distribution of cells indicates an average cell diameter of ~ 480 nm in the irradiated area. (d) Depth dependent irradiation dose and Kr^{++} concentration profiles show the radiation conditions.

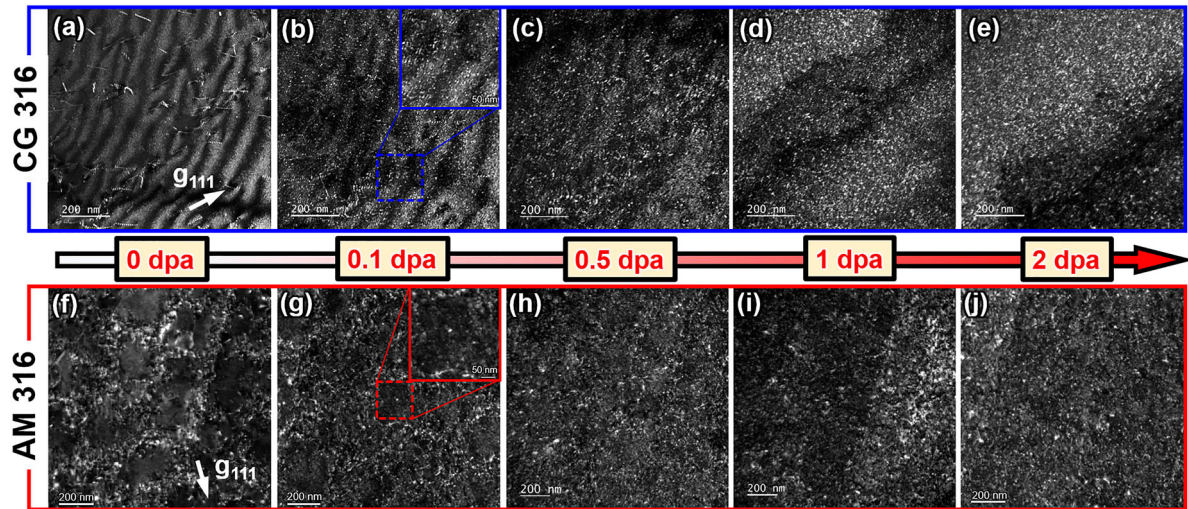


Figure 2. *In situ* TEM DF images show the microstructure evolution with irradiation dose in (a–e) CG and (f–j) AM 316 SS up to 2 dpa at 400°C . At the same dose level, AM 316 SS exhibited a much lower loop density compared with the CG counterpart. Inserted in (b) and (g) are the magnified DF images. All the DF images were captured using g_{111} close to $\langle 110 \rangle$ zone axis.

including absorption, climbing, annihilation, and detangling, were observed. First at 1.3 dpa in Figure 4(a1), a newly formed dislocation loop L1 induced by irradiation is ~ 19 nm away from the cellular wall with a thickness

of ~ 50 nm. By 1.4 dpa, L1 migrated towards the wall by ~ 7 nm. After another 0.01 dpa, L1 was absorbed by the wall. The wall thickness increased by ~ 6 nm during the irradiation. Second, at 3.7 dpa in Figure 4(b1), dislocation

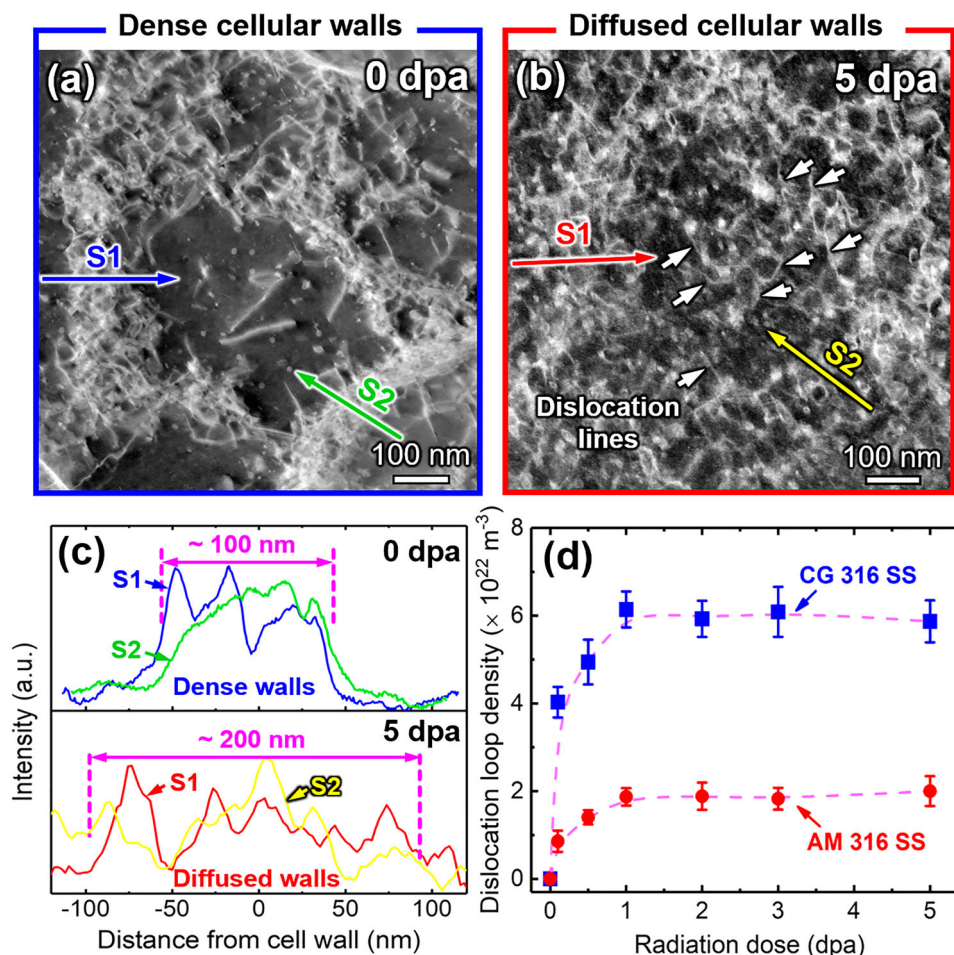


Figure 3. Representative DF STEM images of cellular structures (a) before and (b) after irradiation to 5 dpa. (c) DF intensity profiles measured from the line scans (S1 and S2) in (a) and (b) showing the average cellular wall thickness increased from ~ 100 to 200 nm after irradiation. (d) Dose-dependent variation of loop density shows a much lower saturated loop density in AM 316 sample.

line D1 and loop L2 were in contact with each other. After 0.7 dpa, D1 moved away from L2 by ~ 12 nm, and as the irradiation dose increased to 5.5 dpa, the distance between D1 and L2 reached 18 nm (Figure 4(b2,b3)). In the third case at 3.7 dpa in Figure 4(c1), two dislocation lines D2 and D3 were separated with a distance of ~ 35 nm, and as the dose increased to 5.2 dpa, D3 moved towards D2 by ~ 22 nm. Finally, they partially annihilated with each other at 5.5 dpa (Figure 4(c3)). In the last case, at 0.11 dpa two dislocation lines D4 and D5 were entangled with each other by the pinning effect from D4 in Figure 4(d1). After 0.05 dpa, D5 suddenly detangled from D4 and migrated ~ 15 nm away from D4. Then D5 departed slowly from D4, and finally was ~ 23 nm away from D4 at 0.22 dpa in Figure 4(d3). Schematic diagrams in Figure 4(a4–d4) illustrate the absorption of a dislocation loop by cellular wall, dislocation climb, annihilation and detangling of trapped dislocation lines. More *in situ* video analyses on dislocation migration and defect cluster absorption by cellular walls are shown in Figure S7

and Figure S8 (See supplementary videos S1–S6 for more details).

4. Discussion

Among various defect sinks, dislocations are known as biased sinks for interstitials [21,22]. However, cellular walls with high-density dislocations are more likely to be neutral defect sinks in this study. Prior theoretical studies suggest that different from an isolated dislocation, the sink strength of a group of dislocations for interstitials may approach that for vacancies, when capture radii of these dislocations overlap, resulting in a partial cancellation of long-range stress fields of individual dislocations [23,24]. In addition, an experimental study proposed that high-density dislocations may become relatively neutral defect sinks, and decrease the swelling rate of certain ferritic/martensitic steels [25].

Nevertheless, conventional deformation-induced dislocation cellular walls are often considered thermally

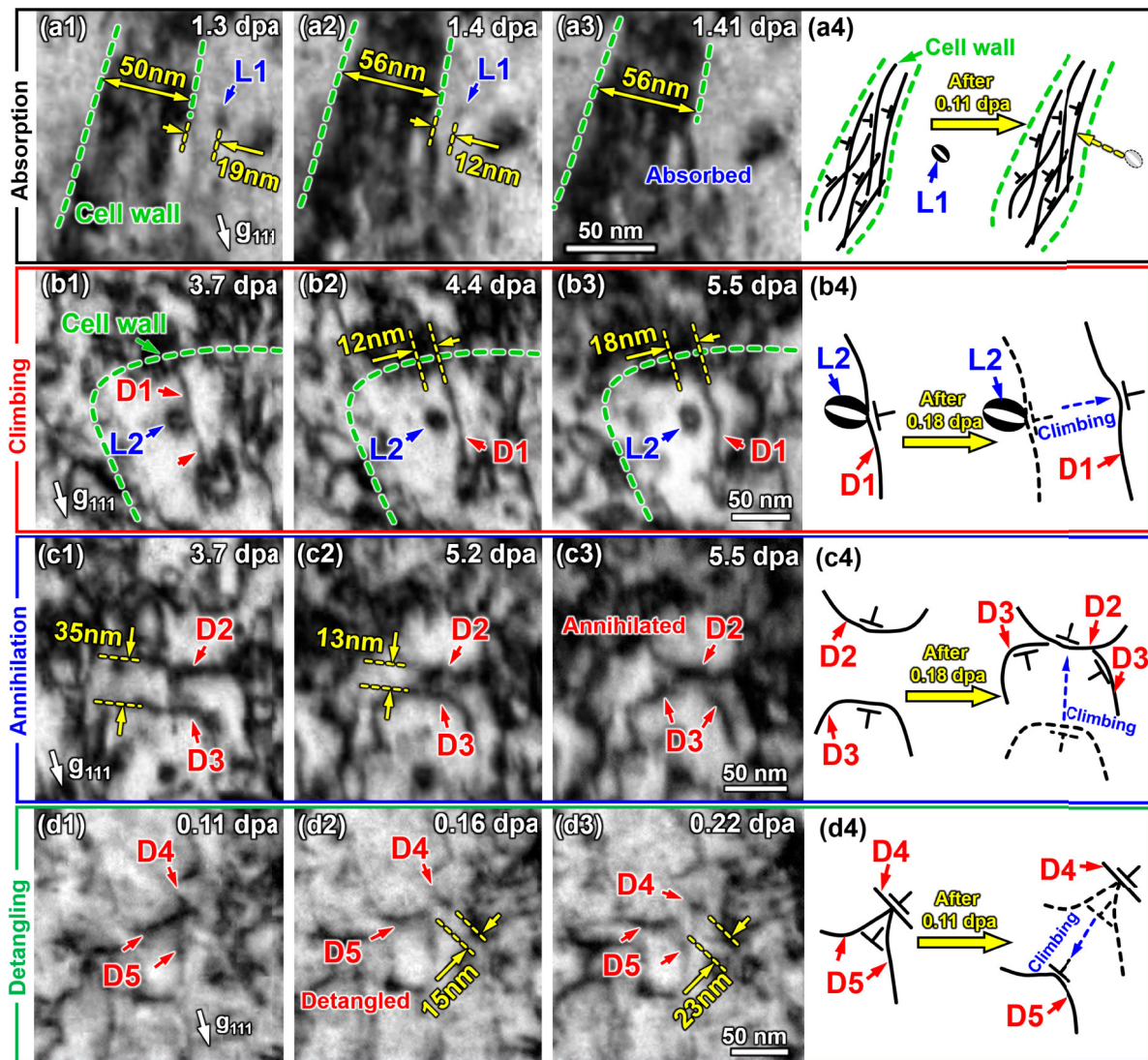


Figure 4. *In situ* video snapshots show the interaction between dislocation lines, loops and cellular walls. (a1–a3) A dislocation loop L1 was absorbed by cellular wall, which became thicker during 1.3–1.41 dpa. (b1–b3) A dislocation line D1 moved away from an irradiation-induced loop L2 during 3.7–5.5 dpa by climbing. (c1–c3) Dislocation line D3 moved towards D2 and partially annihilated each other while in contact during 3.7–5.5 dpa. (d1–d3) Two dislocation lines D4 and D5 detangled from each other as D5 retracted and departed from D4. (a4–d4) The corresponding schematic diagrams illustrate the four scenarios (See supplementary video S1–S4 for more details).

unstable, thus suffer from obvious recovery and recrystallization due to the climb and annihilation of dislocations at elevated temperatures ($\geq \sim 0.4 T_m$), and irradiation can further accelerate these annealing processes by introducing abundant point defects [23,24]. In the present study, however, dislocations trapped at the cellular walls exhibit excellent thermal stability at 400°C ($\sim 0.4 T_m$) and a low average climb rate of ~ 0.01 nm/s, which was measured from Figure 4(b1–b3). *In situ* studies in Figure 4 show that L1, D1, D3 and D5 were relatively mobile, while L2, D2 and D4 remained stationary. The absorption of dislocation loops by the cellular walls indicates the strong sink strength of the wall. The migration of dislocations (such as D1) near cellular walls

may arise from the irradiation-assisted dislocation climb at elevated temperatures, but the slow migration speed may be related to the pinning effect from the cellular walls. The *in situ* studies indicate that cellular walls play an important role in stabilizing the trapped dislocations and their tangles, and, in turn, make the cellular structures relatively neutral defect sinks for vacancies and interstitials.

Compared with high angle grain boundaries (HAGBs), solidification cellular walls are equivalent defect sinks, but have inherently low energy, and thus are much more stable at high temperatures than HAGBs. The crystallographic orientation analysis on the cellular structures in the AM 316 SS in Figure S9 shows small

misorientations ($\leq 2.5^\circ$) between adjacent cells. Therefore, the boundary energy of cellular walls is similar to LAGBs, whose energy is determined by an array of dislocations and is much lower than that of conventional HAGBs [26,27]. Hence these cellular walls have sink strength comparable to HAGBs, but have low energy comparable to LAGBs, making them appealing stable defect sinks.

During the irradiation process, the dense cellular walls containing high-density dislocations gradually evolve to wide and diffuse walls due to irradiation-assisted dislocation climb. However, the EDS chemical maps in Figure S1(b1–b6) confirm that the cellular structures with high density dislocations are still stable even after irradiation to 5 dpa at 400°C in spite of a relatively diffuse appearance, meaning that cellular walls are still effective defect sinks in capturing defects. Dislocation climb can be classified into two categories during high-temperature irradiation: thermally activated climb by forming supersaturated vacancy concentration and irradiation-induced climb by introducing high-density point defects including self-interstitials and vacancies [14,23]. In this study, dislocation climb is more likely to be dominated by irradiation as the dislocations and the cellular walls are very stable during annealing at 400°C (without radiation). Irradiation-induced climb rate ($v_{c,irr}$) is obtained from the local net flux of point defects (either vacancies or interstitials) from and into the dislocation core, and can be expressed by [14,24]:

$$v_{c,irr} = \frac{\Omega_v}{b} Z_v D_v (C_v - C_{0,v}) - \frac{\Omega_i}{b} Z_i D_i C_i \quad (1)$$

where Ω_v and Ω_i are the atomic volume for a vacancy and an interstitial, respectively. $C_{0,v}$ is the equilibrium vacancy concentration. b is the Burgers vector. Factors affecting the radiation-dominated climb rate are: Z_v and Z_i , dislocation sink strength for vacancy and interstitial; C_v and C_i , vacancy and interstitial concentration induced by irradiation; D_v and D_i , diffusivities of vacancy and interstitial.

The local net flux of point defects necessary for dislocation climb can be achieved by point defect diffusion driven by concentration gradient and the different diffusivities of self-interstitial and vacancy, and can be described by locally valid chemical rate equations [14]:

$$\frac{\partial C_v}{\partial t} = K_0 - K_{iv} C_i C_v - K_{vs} C_v C_s + \nabla \cdot D_v \nabla C_v, \quad (2)$$

$$\frac{\partial C_i}{\partial t} = K_0 - K_{iv} C_i C_v - K_{is} C_i C_s + \nabla \cdot D_i \nabla C_i, \quad (3)$$

where K_0 is the defect production rate; K_{iv} is vacancy-interstitial recombination rate coefficient; K_{vs}/K_{is} is the

vacancy/interstitial-sink reaction rate coefficient. In this work, due to the existence of cellular walls as defect sinks, the point defect concentration would be lower in the region close to walls than that in the cell center. During irradiation, defect concentration gradient (∇C_i or ∇C_v) may drive the diffusion of point defects from the cell center to the cellular walls.

However, the respective local fluxes of self-interstitial ($D_i \nabla C_i$) and vacancy ($D_v \nabla C_v$) from cell center to the wall are expected to be different as a consequence of different migration energies (E_m) of an interstitial and a vacancy at the same temperature [28,29]. The E_m of a vacancy in γ -Fe has been calculated to be ~ 1.32 eV, which is nearly eight times greater than that of a single interstitial, ~ 0.15 eV [30]. Thus, the Arrhenius' equation ($D = D_0 \exp(-E_m/kT)$) would predict that $D_i \gg D_v$, if we assume D_0 and T to be the similar for self-interstitials and vacancies. Moreover, the diffusivity of an interstitial in γ -Fe was calculated to $\sim 2 \times 10^{-5}$ cm²/s at 700°C, which is also nearly three times greater than that of a vacancy, $\sim 6.5 \times 10^{-6}$ cm²/s [31]. Therefore, a local net flux of vacancies or interstitials towards the cellular walls was introduced by different diffusivities of interstitials and vacancies, and facilitated dislocation climb. The *in situ* irradiation studies also suggest that the climb of dislocations may gradually change the nature of cellular walls at very high doses, thus the point defect absorption efficiency (sink strength) of the cellular walls may be modified gradually. Stabilization of the cellular walls to high-temperature and high-dose irradiations may be a critical task for the design of radiation-resistant SS in the future. In addition, foregoing discussions focus primarily on the role of cellular walls in stabilizing the trapped dislocations, while the sink strength of the wall itself to radiation-induced defects remains a subject that deserves future investigations.

5. Conclusions

In situ Kr⁺⁺ irradiation experiment was performed to study the irradiation response of solidification cellular structures in an AM 316 SS up to 5 dpa at 400°C. Major findings are summarized as follows: (1) AM 316 SS exhibits a much lower dislocation loop density compared with its CG counterpart subjected to the same irradiation dose, 5 dpa. (2) The trapped dislocations are relatively stable at 400°C during the *in situ* irradiation due to the existence of solidification cellular walls. (3) Cellular walls with high-density dislocations tend to absorb irradiation-induced dislocation loops and serve as effective defect sinks. (4) High-dose radiation leads to diffused cellular structures.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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