Effects of In-Situ Formed Oxide Inclusions on High-Temperature Mechanical Properties of 304L Stainless Steel Processed by Laser Powder Bed Fusion

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Abstract

Laser Powder Bed Fusion (LPBF) enables the formation of in-situ oxide inclusions, offering a cost-effective alternative to conventional Mechanical Alloying (MA) for fabricating Oxide Dispersion Strengthened (ODS) alloys. This method enhances the dispersion of oxide particles and their compatibility with the matrix. The present study systematically investigates the microstructure and high-temperature mechanical properties of LPBF-processed 304L stainless steel (SS). By precisely controlling chamber oxygen content at 50 ppm, 500 ppm, and 1500 ppm and optimizing process parameters, in-situ 304L ODS alloys were successfully fabricated. High-temperature tensile and creep rupture testing demonstrates that the 500 ppm sample exhibits exceptional high-temperature strength and creep performance, attributable to stable oxide particles that effectively inhibit grain coarsening and pin dislocations. This sample achieves a yield strength (YS) of approximately 294 MPa, ultimate tensile strength (UTS) of approximately 351 MPa, elongation (EL) of 26%, and a rupture life of 194 hours at 600°C. The LPBF process realizes uniform distribution of oxide particles, significantly improving high-temperature mechanical properties. This progress provides a new, more stable, and effective approach for manufacturing core structural materials for nuclear power applications.

Full Text

Preamble

A Study on High-Temperature Mechanical Properties of 304L Stainless Steel Processed by Laser Powder Bed Fusion Under the Effect of In-Situ Formed Oxide Inclusions

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Abstract

In-situ oxide inclusions formed during laser powder bed fusion (LPBF) provide a cost-effective alternative to traditional mechanical alloying (MA) for fabricating oxide dispersion strengthened (ODS) alloys, enhancing both oxide particle dispersion and matrix compatibility. This study investigates the microstructure and high-temperature mechanical properties of 304L stainless steel (SS) fabricated via LPBF. By controlling the oxygen content in the chamber at 50 ppm, 500 ppm, and 1500 ppm while optimizing processing parameters, in-situ 304L ODS alloys were successfully prepared. High-temperature tensile and durability tests reveal that the 500-ppm sample exhibits excellent high-temperature strength and durability, attributed to stable oxide particles that prevent significant grain coarsening and pin dislocations. This sample achieved a yield strength (YS) of ~294 MPa, ultimate tensile strength (UTS) of ~351 MPa, elongation (EL) of 26%, and a durability of 194 hours at 600 °C. The LPBF process improves the uniform distribution of oxide particles and significantly enhances high-temperature mechanical performance. This advancement provides a new, more stable and effective approach for manufacturing core structural materials in nuclear power applications.

Keywords: In-situ ODS, LPBF, oxygen content, high temperature tensile, mechanical properties

1 Introduction

Over the past decades, oxide dispersion strengthened (ODS) alloys have been widely used in high-temperature applications, including heat exchangers, gas turbines, and fusion reactor divertors [1, 2]. Nano-sized oxides uniformly dispersed within the matrix enhance mechanical properties by inhibiting dislocation movement through Hall-Petch or Orowan mechanisms [3-6]. Traditionally, ODS steels were produced via mechanical alloying (MA), which involves high-energy milling of steel mixed with nano-oxide powders, followed by hot extrusion or hot isostatic pressing (HIP). While this method effectively disperses

nano-oxides [6] and has been commercially adopted, it has limitations: the addition of oxides like yttria can cause carbide agglomeration, and the ball milling process often reduces the sphericity of 304 stainless steel matrix powders, adversely affecting material formability. Additionally, applications of ODS steels are limited mainly to tubes and bars due to inadequate joining techniques [7-9]. Oxygen-controlled laser powder bed fusion (LPBF) offers the potential to overcome these limitations by enabling the production of ODS steel components with complex geometries [6, 10-14].

LPBF, an advanced additive manufacturing (AM) technique, can achieve metastable solidified microstructures such as submicron-scale cellular structures and nano-oxide inclusions [15, 16]. These microstructures provide enhanced strength and ductility at high temperatures compared to conventional counterparts [17-19]. Ghayoor et al. [17] demonstrated that LPBF-processed 304L alloys mixed with 0.5 wt% Y_2O_3 nanoparticles possessed a yield strength (YS) of 290 MPa even after tensile testing at 600°C, significantly superior to hot-rolled 304L SS (YS: 150 MPa) [18]. Additionally, aging at 1200°C indicated good retention of ultra-fine cellular structures due to nano-oxide pinning. The presence of nano-oxide particles impedes recrystallization and further grain growth, which are the main reasons for the improvement in high-temperature mechanical properties. Phaniraj et al. incorporated powder (Fe-20%Ni-14%Cr-2.5%Mo-2.5%Al-2%Mn) and produced austenitic ODS SS via the MA method to study the effect of oxide particles on thermal stability. After aging at 1100°C for 24 hours, the oxide particles remained thermodynamically stable and obstructed grain growth (limiting grain size to ~8 nm). This stabilization enables the material to maintain high strength at temperatures approaching its melting point, thereby potentially enhancing its creep resistance.

A deeper understanding of oxygen uptake, oxide nucleation, and growth during AM processes is crucial for optimizing high-temperature performance and maintaining the integrity of AM-fabricated steels. Most studies have examined the high-temperature performance of ODS alloys produced by MA, indicating that the high-temperature performance of ODS steels may be hindered by oxide loss, oxygen depletion, and oxide coarsening [9, 22-25]. However, the current understanding of the high-temperature mechanical properties of ODS alloys prepared by LPBF remains insufficient. The coarsening of oxides and subsequent slag formation likely result from the depletion of supersaturation in nano-oxide particles under high-temperature conditions. Gong et al. [25] observed that the formation of larger Y-Al-O particles in 14Cr-Al-ODS steel adversely affects the material' s strength after high-temperature tension at 700 °C, as particle coarsening leads to performance decline. Moreover, annealing at high temperatures can induce grain growth and further coarsening of oxide particles in ODS steels. This microstructure evolution, along with recrystallization, particle coarsening, or dissolution, potentially diminishes the material' s creep resistance at high temperatures. Ha et al. [24] reported that the properties of ODS steel deteriorate when exposed to high temperatures, specifically noting that recrystallization temperatures nearing 0.9Tm (0.9 times the melting temperature) result in

coarser grains and oxides. From the above research, it is evident that ODS alloys prepared by MA tend to have problems such as excessive oxide particle size, non-uniform distribution, and instability at high temperatures. Consequently, this method cannot meet the requirements for the stable use of ODS alloys at high temperatures.

Very limited research has shown that adjusting the oxygen content in the LPBF chamber during processing can obtain in-situ ODS alloys with improved mechanical properties. Hsu et al. [20] prepared 17-4PH SS with nano-oxide particles by regulating oxygen content to 500 ppm, achieving a 154.5 MPa increase in YS, primarily due to nano-oxides and finer martensite. Dryepondt et al. [21] fabricated LPBF HK30Nb steel (Fe-25Cr-20Ni-Nb-C), where residual oxygen during the printing process led to the formation of carbon oxides. These carbon oxides, along with the cellular structure, contributed to maintaining a stable YS of 480 MPa during high-temperature tensile tests conducted between 400-600 °C. At 600 °C, ultimate tensile strength (UTS) also reached 600 MPa. However, the impact of oxygen content on the high-temperature mechanical performance and stability of oxide particles in LPBF ODS alloys remains underexplored.

In this work, we explored the effects of varying oxygen content on the high-temperature tensile properties of LPBF 304L SS by regulating the printing chamber atmosphere. Techniques such as optical microscopy (OM), x-ray diffraction (XRD), scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM) were used for microstructure characterization. Simulations using ANSYS Mechanical APDL 2022R1 software estimated theoretical mechanical properties under different oxygen levels. The findings provide insights into how oxygen concentration impacts microstructural evolution and mechanical properties after high-temperature stretching, offering pathways to optimize LPBF-manufactured materials for high-temperature applications.

2.1 Sample Preparation and LPBF Process

This study utilized gas-atomized 304L SS powder, with its elemental composition detailed in Table 1 and particle sizes ranging from 10 m to 53 m. The LPBF process was conducted using a Hanbon HBD-150 machine in an argon gas-protected environment. The processing parameters used in the LPBF system are presented in Table 2. A gradient range of oxygen content (50 ppm, 500 ppm, and 1500 ppm) was modulated during the printing process to obtain three kinds of ODS material. Raw rectangular bars were fabricated at three oxygen levels for tensile and rupture tests. To distinguish different samples in the LPBF process, a coordinate system was established as shown in Fig. 1 [Figure 1: see original paper]. Specifically, specimens aligned with the Z-axis (building direction) are termed vertical samples, while those perpendicular to the Z-axis (in the XY plane) are designated horizontal samples.

2.2 Mechanical Performance Testing

Tensile tests were conducted at 600 °C with a strain rate of 1$×10^{-3}$ s^{-1} using an ETM 205D testing machine. The specimens, aligned parallel to the building direction, were prepared following the Chinese GB/T 228.2-2015 standard with a 4 mm gauge diameter, 25 mm gauge length, and 55 mm total length. Two specimens per oxygen content were tested to minimize processing errors. High-temperature durability tests were also conducted at 600 °C under 225 MPa load using an RJ-30 creep durability testing machine. The specimens, designed per the HB 5150-1996 standard, had a 5 mm gauge diameter, 25 mm gauge length, and 60 mm total length. One sample was tested for each condition.

2.3 Microstructural Characterization

The microstructural morphology of samples before and after high-temperature tensile tests was characterized using an FEI Quanta 450 scanning electron microscope (SEM). Fracture surfaces were also examined after rupture. For SEM observation, cross-section samples with 10 mm distance from the fracture site were prepared by grinding with SiC sandpaper up to 3000 grit, polishing with 40 nm colloidal silicon slurry, and electrochemically etching in 30% NaOH at 25 V. EBSD analysis was utilized to characterize the texture, grain morphology, and size distribution. These scans covered an area of approximately 500 m$×$400 m with a 0.5 m step at 25 kV. Data acquisition and analysis were performed using Aztec Crystal 2.1 software (Oxford Instrument). For TEM analysis, thin foils were ground to 100 m and electropolished using 5% perchloric acid in 95% ethanol at -30 °C and 30 V. The microstructure and elemental composition were analyzed with an FEI Tecnai F30 Field-Emission TEM at 200 kV. Image J software (version 1.53) was used for quantitative analysis of nano-oxide particles following ASTM E1245 standards, focusing on differences in contrast, size, and distribution at varying oxygen levels. Phase identification was carried out using D8 XRD with a 30-90° scanning angle and 0.01 °/s speed, following JCPDS cards for LPBF 304L SS.

2.4 Theoretical Calculation of Material Strength

The finite element model was developed using the Mechanical APDL 2022R1 software of ANSYS to simulate the interaction between nano-oxides and matrix deformation. A 1 m$×$1 m square region was generated using standard code with dispersed nano-oxide particles. The oxygen content and particle size were considered for better understanding the deformation mechanism in this study. These specifications were based on the number density and average size of small and large nano-oxide particles as presented in Table 2 . Meshing and constraints were applied using the ANSYS APDL program. The left side of the square region was fixed, while the right side was constrained, as illustrated in Fig. 11 [Figure 11: see original paper] (a1-c1), to accurately simulate the actual tensile experiments. In the APDL program, nano-oxide particles with different

sizes were randomly incorporated to closely replicate the random distribution of these nano-oxide particles within the material under high-temperature conditions. This simulation was conducted under uniform conditions of introduction area, constraints, and loading. Theoretical YS and material deformation values for three distinct oxygen content scenarios were subsequently analyzed. This method clearly illustrates that modification in particle size, resulting from variations in oxygen content at 600 °C, substantially affects the material' s strength and deformation.

3.1 Microstructural Analysis

Fig. 2 Figure 2: see original paper shows the ultrafine cellular structures within the austenite grains, exhibiting a consistent average cell size of approximately 330 nm across all samples. These samples exhibited a random distribution of numerous small nano-oxide particles, both within and at the boundaries of the cellular structure. At higher magnification, as shown in Fig. 2(a2-c2), the base material reveals a widespread distribution of nano-oxide particles throughout the matrix. The density of these nano-oxide particles increases with higher oxygen content, indicating a strong correlation between oxygen levels and particle concentration, which significantly influences the material' s mechanical properties. Notably, under high oxygen conditions, the density of nano-oxide particles exhibits a pronounced improvement.

Fig. 3 [Figure 3: see original paper] presents the XRD patterns of LPBF 304L SS samples after high-temperature stretching at 600 °C. In these patterns, the austenite phase with FCC structure was the dominant phase across all three conditions, marked by black triangles for clarity. Additionally, a subtle peak observed in the XRD spectra between 50° and 60° corresponds to SiO_2, as identified by PDF card 47-1144, and is marked with a black dot. Besides these, the extensive variation in oxygen content and temperature did not produce obvious new phases.

Fig. 4 Figure 4: see original paper shows the detailed microstructural morphologies of these samples in as-deposited conditions after high-temperature tensile testing at 600 °C, where the presence of austenite grains is evident across all oxygen content levels. In Fig. 4(a2-c2), the cellular structures within the austenitic grain boundaries reveal uniformly distributed submicron-sized cellular structures (~400 nm) across all specimens. Oxide particles are distributed randomly in the interior and at the grain boundaries. Compared to the cellular structures of about 500 nm observed in LPBF 316L SS materials [15, 27], the LPBF 304L ODS SS specimens in this study exhibit notably finer cellular structures. This refinement is attributed to the rapid cooling rates (10^3-10^7 °C/s) during LPBF, which generate thermal shrinkage stress and create a balance between the thermal gradient and the solidification rate, thereby constraining the cellular structure size to ~400 nm. Additionally, Fig. 4(a3-c3) uncovers a multitude of fine, dispersed nano-oxide particles (~20 nm) within the matrix, with their quantity increasing with oxygen content. The variation in the quantity

and size of these nano-oxide particles is expected to significantly influence the material' s mechanical properties, which is thoroughly investigated in section 4.2 of this study.

Fig. 5 Figure 5: see original paper displays the distribution of nano-oxide particles within the non-cellular structure regions of the LPBF 304L SS matrix, identifying two distinct types of nano-oxide particles with differing contrasts (white particles and black particles). Fig. 5(a2-c2) illustrates the distribution of these contrasting nano-oxide particles, which are located not only along the boundaries but also within the interiors of the cellular structures.

The grain misorientations of LPBF 304L SS samples subjected to high-temperature tensile tests are shown in Fig. 6 Figure 6: see original paper. The inverse pole figure (IPF) reveals that the misorientation of the austenite grains is primarily concentrated on <111>. Fig. 6(a2-c2) presents the kernel average misorientation (KAM) for the samples, revealing that variations in process parameters across different oxygen contents minimally affect the stress distribution in the as-deposited samples. Notably, stress levels remained low across the samples, except for localized stress increases at melt pool edges attributable to enhanced energy density and thermal cycling during fabrication, aligning with prior research findings [28]. Furthermore, EBSD effectively analyzes the phase composition within the alloy (Fig. 6(a3-c3)), identifying ferrite against an austenite background. Such results revealed that all samples contained approximately 40% Σ3 twin boundaries and around 2% Σ9 twin boundaries, indicating that deformation twins formed during the 600 °C tensile process significantly contribute to mechanical property enhancement, as supported by references [29-31]. Xiong et al. [29] highlighted that the synergy between nano-sized twins and nanograins restricted dislocation slip, thus reinforcing material strength. Additionally, twins mitigate the plastic behavior of crack propagation, elevating crack toughness. Jazaghi et al. [31] conducted uniaxial tensile tests on 316 austenitic stainless steel, acquired in the form of hot-forged billets from 20-500 °C and compared the results with fully annealed samples. They observed an increase in the high-temperature YS of austenitic stainless steel, which is attributed to nano-sized twins. These twins can restrict dislocation movement, enhance dislocation storage, and consequently amplify material strength. This phenomenon can reduce the dislocation-free path, increase dislocation storage, and ultimately boost material strength. Moreover, the average grain sizes of the samples after 600 °C high-temperature tensile testing are 9.05 m, 8.97 m, and 10.22 m for 50 ppm, 500 ppm, and 1500 ppm oxygen contents, respectively (Fig. 7 [Figure 7: see original paper]). These values are comparable to those reported by Yang et al. [16] for SLM 304L SS after 600 °C heat treatment (10.41 m), indicating consistency in grain size across various studies.

Fig. 8 Figure 8: see original paper further examines the nano-oxide particle distribution in non-cellular regions of the LPBF 304L SS matrix after high-temperature testing, again revealing white and black contrast particles. Fig.

8(a2-c2) illustrates the distribution of these contrasting nano-oxide particles, which are located not only along the boundaries but also within the interiors of the cellular structures. Additionally, Fig. 8 reveals that particle size increases with rising oxygen content, indicating the occurrence of coarsening in nano-oxides. Table 3 shows a significant increase in the density of large-sized oxide particles within the matrix, from 0.58 m^{-2} (average size 61 nm) at 50 ppm to 1.89 m^{-2} (average size 70 nm) at 1500 ppm. Similarly, the density of small-sized oxide particles rose markedly from 8.02 m^{-2} (average size 22 nm) at 50 ppm to 12.82 m^{-2} (average size 16 nm) at 1500 ppm. These findings suggest that elevated oxygen content during high-temperature stretching at 600 °C not only increases the size of nano-oxide particles but also promotes the formation of new, smaller particles.

To further explore the impact of oxygen content changes on the compositional variations of nano-oxide particles after high-temperature stretching, element distribution mappings were obtained on selected areas within the cellular structures of specimens (50 ppm, 500 ppm, and 1500 ppm), respectively. Fig. 9 Figure 9: see original paper shows that the majority of nano-oxide particles across the three oxygen levels are composed of Si-O-Cr. These particles are randomly distributed throughout both the boundaries and interiors of the cellular structures. This observation is consistent with the findings of Deng et al. [32], who noted that silicon predominantly determines the chemical composition of oxides, while other elements such as Mn and Cr are present in varying concentrations. Additionally, a coarsening behavior was observed for both the black and white particles with the increase in oxygen content, which was triggered by the sufficient oxygen content. (The particles appearing brighter are referred to as white particles, while those that appear darker are termed black particles.)

Fig. 10 Figure 10: see original paper presents TEM images of nano-scale deformation twins and acicular ferrite formed during high-temperature deformation. These features, identified as typical twin spots through selected-area electron diffraction, indicate that deformation was activated during the deformation process. The formation of deformation twins at high temperatures interacts with dislocations, thereby restricting dislocation movement and enhancing the YS of the material [31]. Additionally, the high-energy boundaries generated at high temperatures facilitate high-temperature deformation and microstructural refinement [32, 33]. Notably, the oxygen content in the material also plays a crucial role. Regarding the relationship between oxide particles and oxygen content, during high-temperature tension, oxygen content differences significantly impact nano-twin crystals. In high-oxygen regions, oxygen atoms at grain boundaries change atomic arrangements, boosting nano-twin nucleation. Thus, the number of nano-twin crystals increases, with smaller size, denser distribution, and more complex morphology. In low-oxygen regions, the weak nucleation driving force leads to fewer nano-twin crystals with larger size and more orderly growth.

For the relationship between ferrite and oxygen content, high-oxygen samples promote ferrite phase transformation, increasing nucleation sites and ferrite

number. Low-oxygen samples have reduced acicular ferrite nucleation due to insufficient phase-transformation driving force. In high-oxygen samples, abundant nucleation restricts acicular ferrite growth, resulting in smaller size, while in low-oxygen samples, fewer nucleations allow acicular ferrite to grow larger. After deformation, all three samples demonstrated the formation of acicular ferrite, which is significantly influenced by the cooling rate; faster cooling rates promote acicular ferrite formation [34]. Acicular ferrite has been shown to increase the tensile strength of the material while reducing EL [15].

3.2 Mechanical Properties

The influence of oxygen content on the mechanical properties of LPBF 304L SS samples was assessed through high-temperature tensile tests conducted at 600 °C. The engineering stress-strain curves are depicted in Fig. 11 and the values of UTS and YS are presented in Table 4 . An improvement in material strength was obtained both in YS and UTS at 600 °C by modulating the oxygen content from 50 to 500 ppm. Specifically, the YS for samples at 500 ppm surpassed that of 50 ppm by ~20 MPa and the UTS increased by ~11 MPa. The elongation, however, remained relatively stable. As the oxygen content increased to 1500 ppm, materials were not further strengthened, which may be attributed to tiny defects caused by high oxygen levels. Further analysis of Fig. 11 shows serrated yielding in the stress-strain curves of all samples, indicative of dynamic strain aging (DSA), aligning with prior research on high-temperature tensile behavior of austenitic SS [35, 36]. DSA can lead to a decrease in plasticity of the material and contribute to an increase in the short-term strength of the material at high temperatures. The yield strength-to-tensile strength ratio of the samples was calculated and shown in Table 4, which shows a trend of rising first and then declining with the increase of oxygen content. A higher YS/UTS ratio indicates that the material is not easy to undergo plastic deformation under stress and has better tensile properties and higher strength.

SEM observations of high-temperature tensile fractures across different oxygen levels, as shown in Fig. 12 [Figure 12: see original paper], facilitate analysis of the specimens' high-temperature tensile fracture mechanisms. The macroscopic fracture (Fig. 12(a1-c1)) exhibits characteristics of ductile fracture, including shear lips and cup-and-cone morphology. A detailed examination of the magnified areas (Fig. 12(a2-c2)) reveals that the fracture surface is interspersed with micrometer-sized dimple features, signaling substantial plastic deformation before rupture. This fracture surface pattern correlates well with tensile test results. Inside the fine toughness nests, small oxide particles were detected ranging from a minimum diameter of ~15 nm to a maximum of ~100 nm (Fig. 12(a3-c3)). These findings suggest that some fine particles form during high-temperature tensile testing at 600 °C, with the size of some larger particles increasing with oxygen content. These larger particles may serve as potential sites for crack initiation, influencing the part' s failure.

Table 5 indicates that the 500-ppm sample exhibits an endurance life of 194 h,

which significantly surpasses the 50-ppm sample (131 h) by ~63 h. Conversely, the endurance life of the 1500 ppm sample decreased by ~94 h to 99 h compared to the 500-ppm sample. Fracture surface analysis of the endurance specimens (Fig. 13 Figure 13: see original paper) reveals that fractures predominantly exhibit dissociative characteristics at 600 °C and 225 MPa, characterized by a brittle fracture mode along crystal boundaries. Tearing prongs are minimal, the number and size of toughness sockets decrease, and the fracture surface is relatively flat, dominated by dissociative surfaces of various sizes. A closer examination of the magnified areas within the white box revealed the presence of fine nano-oxide particles (Fig. 13(a3-c3)).

3.3 Theoretical Yield Strength Simulation

The high-temperature tensile tests conducted at 600 °C revealed a trend where the YS of the material initially increases and then decreases with rising oxygen content. Using Mechanical APDL 2022 R1 software, we calculated the theoretical YS values at oxygen contents of 50 ppm, 500 ppm, and 1500 ppm. This process involved simulating the random distribution of nano-oxide particles and analyzing the microscopic strengthening mechanism across the three oxygen levels.

The APDL model employs the von Mises criterion, based on material plasticity theory and the fourth strength theory (deformation energy theory), to characterize material yielding and failure under three-dimensional stress. This criterion suggests that material yields when its elastic deformation energy per unit volume reaches a certain threshold. Specifically, the von Mises criterion is expressed as [37]:

$$\sigma_{vonMises} = \sqrt{\frac{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2}{2}} = K(\sigma_{yield})$$

where K is the yield point of the material and σ_{yield} is the shear yield strength of the material. Based on TEM experimental data, we obtained information on the number density and type of nano-oxide particles. Three oxygen content scenarios were set in the software to simulate the high-temperature tensile process at 600 °C, calculating the theoretical mechanical properties based on particle density per unit area (m^2).

According to Table 3, the number of oxide particles per square micrometer varies from 50 to 1500 ppm. In our idealized model, nano-oxide particles were defined as SiO_2 within a 304L SS matrix. The simulation of the high-temperature stretching process at 600 °C requires the setting of both the random distribution of nano-oxide particles and appropriate temperature and material constraints. Table 3 provides detailed information on the particle counts and sizes for different oxygen content levels, along with the simulation' s predefined constraints and applied loads. Specifically, at 600 °C, for 50 ppm, the model induces 8 small particles with an average diameter of 22 nm and 1 large particle with a

diameter of 61 nm per unit area (m^2). For 500 ppm, there are 11 small particles with an average diameter of 18 nm and 1 large particle with a diameter of 69 nm per unit area (m^2). At 1500 ppm, the configuration consists of 13 small particles with an average diameter of 16 nm and 2 large particles with an average diameter of 70 nm per unit area (m^2), as shown in Fig. 14 Figure 14: see original paper.

Constraints are applied with fixed conditions on the left side and loads on the right side of the specimens, as illustrated in Fig. 14(a2-c2). Theoretical calculations require the setting of several nonlinear solution options, including options for larger deformation, prediction correction, and others to accurately simulate material behavior. The simulation results, illustrated in Fig. 14(a3-c3), show that the YS at 500 ppm is 320 MPa, surpassing the YS at 50 ppm (282 MPa) by 38 MPa and at 1500 ppm (291 MPa) by 29 MPa. The varied distribution of nano-oxide particles enhances the material' s YS, but differences in particle size have distinct impacts on YS. Large oxide particles cause significant stress concentrations and deformation within the material, while smaller particles induce weaker stress concentrations and less work hardening, as shown in Fig. 14(a4).

4.1 Strengthening Mechanism: Micro-Mechanisms and Kinetics of Oxide Coarsening

The coarsening of in-situ oxides formed during the LPBF process in 304L SS, as observed in Fig. 8 and Fig. 9, occurred and was predominantly controlled by the Ostwald ripening mechanism. This observation aligns with findings from Deng et al. [32], who discovered similar coarsening behaviors in AM 316L SS treated at temperatures ranging from 650 to 1150 °C. This process adheres to the Lifshitz-Slyozov-Wagner (LSW) theory, which is based on the Gibbs-Thomson equation (1) [38, 39]. It is posited that the solute concentration inversely correlates with particle radius. The smaller particle size signifies higher solute concentration around them, leading to element diffusion from smaller to larger particles. The small particles thus dissolve and large particles grow into larger sizes to maintain local chemical equilibria.

$$C_r = C_\infty \left(1 + \frac{2\sigma V_m}{rRT}\right)$$

where C_r is the solute concentration, V_m is the molar volume, r is the radius of the nano-oxide particles, C_∞ is the equilibrium solubility of the precipitated phase, R is the molar gas constant, σ is the specific interfacial energy, and T is the absolute temperature.

Further analysis, incorporating diffusion dynamics and the quasi-steady-state approximation diffusion equation, yields the particle coarsening rate as Eq. (2) [40]:

$$\frac{dr}{dt} = \frac{2D\sigma V_m C_\infty}{rRT}\left(\frac{1}{r} - \frac{1}{r_c}\right)$$

where t is the coarsening duration and D is the diffusion coefficient. Equation (2) clarifies the dynamics of particle coarsening: when $r < r_c$, smaller particles begin dissolving. With the radius in the range of $r_c < r < 2r_c$, the growth rate of particles accelerates with increasing r. At size of $r = 2r_c$, the growth rate reaches maximum, marking the peak growth rate of particles. Beyond $r > 2r_c$, further particle growth is halted and the growth rate declines until solute concentration equilibrium is achieved. Consequently, smaller nano-oxide particles dissolved, whereas larger particles underwent coarsening during the 600 °C high-temperature stretching process, as demonstrated in Fig. 8 and Fig. 9.

The size disparity of nano-oxide particles formed during high-temperature stretching also significantly influences their interaction with dislocations [41-44]. Naoko et al. [42] discovered that the coherence degree of nano-oxide particles is size-dependent, with coherent particles being smaller than 5 nm, semi-coherent particles ranging from 5-20 nm, and incoherent particles exceeding 20 nm in size. Fu et al. [43] observed that dislocations can slice through particles when they are small and either semi-coherent or coherent, leading to a predominance of the cut-through mechanism. Conversely, for larger particles, the bypass mechanism becomes dominant. Ukai et al. [41] found that coherent nanoparticles are advantageous as they reduce the driving force required for bypass mechanisms facilitated by dislocation climbing. Taking into account the number density and size of oxide particles detailed in Table 3, the Orowan mechanism' s impact during high-temperature stretching across three oxygen content levels is illustrated in Fig. 15 [Figure 15: see original paper].

Fu et al. [43] also developed a model to describe the contribution of oxide particles to yield strength through both strengthening mechanisms. These models collectively demonstrate that the enhancement effect of oxide particles is predicated on a critical size of 1.56 nm, with diffuse strengthening increasing as nano-oxide particle size remains below 1.56 nm and diminishing as nano-oxide particle size exceeds 1.56 nm. This observation aligns with the average particle size in this work, where the intensity at 500 ppm (22.1142857 nm) is higher than that at 50 ppm (24.630232 nm) and 1500 ppm (22.942857 nm). Sakasaegawa et al. [44] found that the presence of larger particles (100 nm) scarcely contributes to oxide dispersion strengthening and may even diminish the creep properties of the material. The occurrence of ~100 nm oxide particles in high-temperature tensile samples with high oxygen content (as shown in Fig. 7c) may account for the observed reduction in tensile properties of the samples at 600 °C with high oxygen levels.

4.2 Strength Theory Contributions

The modulation of oxygen content in LPBF 304L SS demonstrated the possibility of enhancing high-temperature strength through in-situ ODS technology. The specimens under three different oxygen contents exhibit superior strength compared to reported results in traditionally manufactured ODS stainless steels, which is attributed to the in-situ-generated nano-scale oxide particles, facilitated by rapid cooling and thermal cycling [14, 45]. Previous studies have reported the YS of forged austenitic SS under high-temperature stretching at 600 °C to be ~150 MPa (Nikulin et al.) [46] and 185 MPa at 650 °C in super 304H SS (Peng et al.) [47]. The enhanced strength at high temperatures can be attributed to fine, diffusely distributed nano-oxide particles, which hinder dislocation motion through a pinning effect during tensile deformation [12]. Additionally, the presence of numerous cellular structures within the grains further amplifies mechanical properties. Therefore, the combined reinforcement effect of cellular structures and nano-oxide particles within the matrix significantly boosts both yield and tensile strengths in LPBF ODS 304L stainless steel. The mechanical properties are also influenced by the size, distribution, and deformation capacity of the oxide particles [12, 48].

In this study, the observed discrepancy in mechanical properties could be attributed to the size variation of the nano-oxide particles formed under different oxygen content. Specifically, the smaller newly formed nano-oxide particles might be too minuscule to effectively anchor dislocations following high-temperature stretching. Conversely, some oxide particles could be excessively large, detrimentally impacting the mechanical properties. As Darling et al. [49] have pointed out, nano-oxide particles around 5 nm in size are more sensitive to thermal softening compared to larger nano-oxide particles, and these smaller particles are more easily bypassed at higher temperatures. Therefore, at 600 °C, the contribution of small particles to enhancing and maintaining strength is less significant than at room temperature. The enhancement mechanisms of metal matrix composites mainly include interfacial enhancement, particle enhancement, and dislocation enhancement [32]. Within the scope of this study, both the cellular structure and the nano-oxide particles, as depicted in Fig. 4 and Fig. 8, play critical roles in materials strengthening at high temperature. The principal strengthening mechanisms identified for LPBF 304L SS include the uniform distribution of nano-oxide particles, the promotion of high-density dislocation networks, and grain boundary strengthening. Consequently, the theoretical YS increment ($\Delta\sigma_{YS}$) of the material was estimated using formula (1) [45]:

$$\Delta\sigma_{YS} = \Delta\sigma_{GB} + \Delta\sigma_{Dis} + \Delta\sigma_{OX}$$

where $\Delta\sigma_{GB}$ represents the increment in grain boundary strength, $\Delta\sigma_{Dis}$ is the contribution of dislocation strengthening, and $\Delta\sigma_{OX}$ is the contribution of nano-oxide particle strengthening.

The EBSD micrograph of the LPBF 304L ODS alloy showed the average grain size of the samples after high-temperature tensile testing under three different contents (Fig. 7). Therefore, the Hall-Petch relationship could be utilized for estimating the grain boundary strengthening contribution as:

$$\Delta\sigma_{Hall-Petch} = K(d_{oxygen1}^{-1/2} - d_{oxygen2}^{-1/2})$$

where the constant K is defined as 0.274 $MNm^{-3}/^2$ [45] and d represents the grain size. As illustrated in Fig. 7, the inverse square root of the grain size $d^{-1/2}$ varies with oxygen content, being 9.05 m for 50 ppm, 8.97 m for 500 ppm, and 10.22 m for 1500 ppm. Thus, an increase in oxygen content to 500 ppm results in $\Delta\sigma_{GB}$ of ~0.405 MPa. However, this value declines by about 5.777 MPa as the oxygen content escalates from 500 ppm to 1500 ppm.

TEM analyses, showcased in Fig. 8 and Fig. 9, reveal that the LPBF 304L SS ODS alloy maintains a high dislocation density after undergoing high-temperature stretching at 600 °C. This is attributed to the rapid melting and cooling processes that induce significant strain in the matrix, thereby enhancing its dislocation density. The dislocation density, measured via AZtecCrystal software from EBSD images post-stretching at 600 °C, recorded 19.422$×10^{14}$ m^{-2} for 50 ppm, 20.955$×10^{14}$ m^{-2} for 500 ppm, and 19.828$×10^{14}$ m^{-2} for 1500 ppm, indicating an initial increase followed by a decrease with rising oxygen content. This trend in strength enhancement via dislocation density aligns with the Bailey-Hirsch equation [45]:

$$\Delta\sigma_{Dis} = \alpha MGb\sqrt{\rho}$$

where α is 0.25, M is the Taylor factor (3 for FCC polycrystals), G is the shear modulus of 80 GPa [14], b is the Burgers vector of 0.25 nm, and ρ is the dislocation density (m^{-2}). The dislocation strengthening contribution between 500 ppm and 50 ppm oxygen content is calculated to be 25.546 MPa.

Furthermore, studies have indicated that nano-oxide particles within the matrix can hinder dislocation movement [6]. Fig. 8 and Fig. 9 depict these particles distributed randomly along the boundaries and within the cellular structure, potentially interacting with dislocations to improve material properties. Post-stretching at 600 °C, variations in the size and volume fraction of nano-oxide particles are documented, highlighting the impact of oxygen content on their distribution. The results in Table 3 indicate that the size and volume fraction of the nano-oxide particles inside the material change after the three oxygen content samples are stretched at high temperatures. Further calculations on the high-temperature stretched samples show that the average size of the nano-oxide particles in the 50 ppm sample is 24.630 nm with a volume fraction of 0.273%; the average size in the 500 ppm sample is 22.114 nm with a volume fraction of 0.305%; and the average size in the 1500 ppm sample is 22.943 nm with a volume fraction of 0.405%. The nano-oxide particles were mainly identified as

silica precipitates, and their size and distribution density were closely related to the oxygen content. The theoretical reinforcement value contributed by the nano-oxide particles can be estimated by the Orowan-Ashby model [45]:

$$\Delta\sigma_{OX} = \frac{0.13Gb}{\lambda}\ln\left(\frac{D}{2b}\right)$$

where $\lambda = D\left(\sqrt{\frac{\pi}{4f}} - 1\right)$ represents the interparticle spacing, f is the volume fraction of precipitation (vol%), and D is the average particle size (m).

Calculations suggest that the enhancement attributed to nano-oxide particles at 500 ppm is ~96.978 MPa, showing an increase of 10.763 MPa over 50 ppm and an 11.507 MPa rise relative to 1500 ppm. This distribution positively influences the theoretical mechanical properties of the material. Assessing the theoretical YS after high-temperature stretching reveals that the theoretical YS at 500 ppm is ~36.714 MPa higher than at 50 ppm and ~12.874 MPa greater than at 1500 ppm. These theoretical values corroborate the observed experimental trends in mechanical property enhancements.

4.3 Prospects for Irradiation Performance

ODS alloys produced through traditional technology are embedded with uniformly dispersed nano-oxide particles, thereby enhancing the material' s mechanical properties. These particles serve as strong trapping centers for point defects such as helium and vacancies, significantly reducing the formation of large helium bubbles and boosting irradiation resistance [50, 51]. Research by Odette et al. [52] confirmed the effectiveness of oxide particles in absorbing helium atoms and limiting helium bubble growth through He ion injection tests on MA957 steel. Similarly, K. Yutani [50] observed superior swelling resistance in high-chromium ODS ferrite steel, attributed to the dense distribution of fine oxide particles that trap helium atoms and vacancies. Xie et al. [51] carried out simultaneous irradiation of Fe and He ions at 350-550 °C on ODS ferrite/martensite (ODS-F/M) steel and found that ODS-F/M steel displayed a tendency to gather helium bubbles around oxide particles, suggesting these interfaces are more effective at trapping helium compared to grain boundaries and internal grains. Furthermore, irradiation at 450 °C resulted in fewer and smaller irradiation-induced dislocation loops in ODS-F/M steel compared to Chinese Low Activation Martensite (CLAM) steel under identical conditions, suggesting an inhibitory influence of ODS-F/M steel on the proliferation of such dislocation loops.

Compared to traditionally manufactured (TM) ODS materials, those produced via LPBF are distinguished by ultrafine cellular sub-grains [15], precipitated phases [53], and oxide nano-inclusions [11], serving as defect sinks that fortify irradiation resistance. It has been discovered that grain boundaries [11], particle-matrix interfaces [54, 55], and precipitation phases [56] can effectively absorb

point defects arising during irradiation [57], thus bolstering the material' s resilience to such exposure [15, 58, 59]. Fu et al. [58] irradiated SLM 316L SS and TM 316L SS with 700 keV He ions at 500 °C and found that SLM 316L SS exhibits a reduced number density of helium bubbles due to its intricate microstructures, including entangled dislocation network walls and sub-grain boundaries, thereby showing greater resistance to irradiation hardening than its traditionally manufactured counterpart. Similarly, Li et al. [59] found that unique microstructures in SLM 304L SS—such as sub-grain boundaries, nano-inclusions, and fine grain structures—can inhibit formation and growth of dislocation loops induced by irradiation when both SLM 304L SS and TM 304L SS were irradiated with 304 MeV Xe ions at room temperature. This makes SLM 304L SS more resistant to irradiation hardening compared to TM 304L SS. Hou et al. [15] subjected LPBF 304L SS, solution-annealed LPBF 304L SS, and conventionally rolled 304L SS to irradiation with 24 keV He ions at 350 °C. Upon comparing and analyzing the microstructural changes of the three samples after irradiation with theoretical calculations, they determined that LPBF 304L SS exhibits stronger helium resistance than the other two samples. This enhanced resistance is attributed to the cellular sub-grains and uniformly distributed nano-inclusions in LPBF 304L SS, which can inhibit the nucleation of helium gas bubbles.

In this work, finer cellular structure and high-density in-situ oxides were obtained simultaneously in the LPBF 304L stainless steel through atmosphere adjustment. It is speculated that the material should possess better irradiation resistance due to the simultaneous acquisition of finer cellular structure and oxide particles. Besides, the mechanical properties at both room temperature and high temperature have been significantly improved. Therefore, this approach should be a potential technology to meet the higher material requirements of future nuclear power.

5 Conclusion

This study provides a comprehensive analysis of the microstructural and mechanical properties of LPBF 304L SS subjected to high-temperature stretching and durability tests at 600 °C under three oxygen contents, offering insights into how oxygen modulation affects these properties:

1. Through oxygen content regulation, uniform and fine in-situ oxide particles, mainly SiO_2, were obtained. The presence of these particles refined the size of cellular substructure.

2. Larger particles tend to cause more stress concentrations compared to smaller particles, while very small particles have a negligible impact on high-temperature mechanical properties. The LPBF 304L SS with an oxygen content of 500 ppm exhibited superior mechanical properties under high-temperature tensile testing and durability evaluation at 600 °C and 225 MPa, achieving a YS of 294 MPa, UTS of 351 MPa, EL of 26%, and

an endurance life of 194 h.

3. This material shows potential in enhancing irradiation resistance. Moreover, it significantly improves the mechanical properties at room and high temperatures, making it a potential solution for future nuclear power applications with higher material demands.

CRediT Authorship Contribution Statement

Yanlin Gu: Methodology, Investigation, Formal analysis, Writing –Original draft.
Zhen Yan: Investigation.
Yuyu Guo: Investigation.
Aijun Huang: Writing –Review & Editing.
Juan Hou: Funding acquisition, Project administration, Writing –Review & Editing.

Conflicts of Interest

The authors declare no conflict of interest.

Data Availability

Data will be made available on request.

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Note: Figure translations are in progress. See original paper for figures.