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Strong and ductile reduced activation ferritic/martensitic steel additively manufactured by selective laser melting

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ABSTRACT

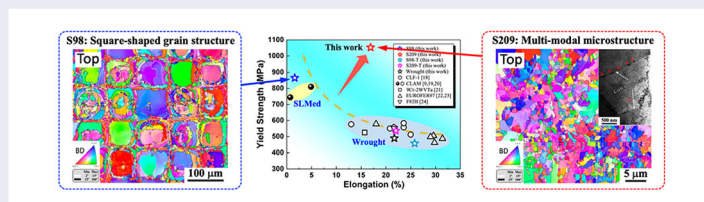
Selective laser melting (SLM) was successfully applied to fabricate strong and ductile reduced activation ferritic/martensitic (RAFM) steel by introducing heterogeneous multi-/bimodal microstructure. The SLM-built RAFM steel exhibited an excellent combination of strength and ductility (i.e. yield strength of 1053 ± 4.7 MPa and elongation of $16.9 \pm 0.4\%$), well surpassing the previously reported counterparts. The superior strength was expected from the refined grains and lath martensites, and the high ductility was associated with the improved dislocation-controlled strain hardening capability. This work provides a basis for developing high-performance RAFM steel by microstructural design via SLM.

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KEYWORDS

Selective laser melting; reduced activation steel; heterogeneous microstructure; strength; ductility



IMPACT STATEMENT

A new strategy, refers to heterogeneous multi-/bimodal microstructure, is proposed to develop strong and ductile RAFM steel via SLM and the achieved superior mechanical properties overcome the classic strength-ductility trade-off.

Introduction

Reduced activation ferritic/martensitic (RAFM) steels have been recognized as the primary candidate structural materials for Test Blanket Module (TBM) of fusion reactors because of their good mechanical and thermo-physical properties as well as mature industrial bases [1]. Due to the geometry complexity of TBM component, the manufacturing process requires several critical steps including hot isostatic pressing, diffusion welding and post machining [2,3], which leads to low manufacturing efficiency and thus high costs.

Selective laser melting (SLM) emerges as a promising additive manufacturing technique to break the bottleneck

of conventional manufacturing process with a number of distinct advantages, such as high geometrical freedom, efficient material usage and high production flexibility [4,5]. Previously, Ordás et al. [6] fabricated the TBM relevant mock-ups with 9Cr-1Mo-V (wt.%) steel by SLM. Furthermore, SLM was successfully applied to produce geometrically complex TBM First Wall Panel part made of 316L stainless steel with superior mechanical properties [7,8]. These studies fully confirm the feasibility of manufacturing the complex nuclear fusion related components based on SLM technique, but the above-mentioned metallic materials do not satisfy the reduced activation requirement for the fusion reactor. To date, few

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studies concerned the additively manufactured RAFM steels. Recently, Huang et al. [9] have attempted to utilize SLM to fabricate RAFM steel component for the first time, but the SLM-built RAFM steels exhibited an undesirable combination of strength and ductility, especially with low elongation less than 5% [9], which can not meet the fundamental requirement for mechanical properties in the fusion reactor and thus limits their potential applications in this field.

The current study, therefore, aims to fabricate strong and ductile RAFM steel by SLM via optimizing processing parameters and better understand the corresponding microstructure and tensile deformation behavior, further unveiling the strengthening and toughening mechanisms. This finding is expected to provide a new pathway for developing the high-strength and ductile RAFM steels by tailoring the microstructure via SLM.

Experimental procedures

The CLF-1 steel (one of the RAFM steels) powder was fabricated from CLF-1 slabs by gas atomization and the chemical composition is Fe-0.08C-8.59Cr-1.46W-0.58Mn-0.28V-0.17Ta (wt.%). The obtained powder is spherical with an average size (d_{50}) of 31.3 μm (Supplementary Figure S1(a,b)). SLM was conducted in a Concept Laser M2 machine equipped with a 400 W fiber laser at a constant layer thickness (30 μm) and hatch spacing (85 μm) while varying the laser power (160 ~ 320 W) and scanning speed (400 ~ 1200 mm/s). The scanning strategy was zigzag scan pattern with 90° rotation between adjacent layers (Figure S1(c)). Specimens with two geometries were fabricated: rectangular cuboid specimens (9 × 10 × 5 mm) for parameter optimization and microstructural observation, and tensile specimens with a gauge dimension of 20 × 5 × 3 mm. Table S1 provides the optimized SLM parameters with good surface quality ($R_a = \sim 10 \mu\text{m}$) and high relative density ($\geq 99.6\%$). The specimens manufactured under the volumetric energy density of 98.04 J/mm³ (laser power of 200 W and scanning speed of 800 mm/s) and 209 J/mm³ (laser power of 320 W and scanning speed of 600 mm/s) (Figure S2) were selected to comparatively study the microstructure and mechanical properties, which are denoted as S98 and S209, respectively. The specimens tempered at 740 °C for 1.5 h followed by air cooling are referred to as S98-T and S209-T, respectively. The relative density was determined using the Archimedes method and the surface roughness (R_a) was measured by a laser microscope (Keyence VK-X250 K). Phase analysis was performed by X-ray diffraction (XRD) using a Rigaku MiniFlex 600 with Cu K α radiation. The microstructures were characterized by

optical microscope (Keyence VHX-5000), field-emission scanning electron microscope (FE-SEM, FEI Quanta 450 FEG), electron back-scatter diffraction (EBSD) and transmission electron microscope (TEM, JEOL JEM-2100F). EBSD was performed on a FEI Quanta450 FEG FE-SEM equipped with an EDAX-TSL system. The tensile test was conducted using a Zwick/Roell Z050 testing machine under a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at room temperature. Three specimens were tested for each condition to ensure the data reproducibility.

Results and discussion

The XRD patterns (Figure S3) indicate that ferrites/martensites formed in the SLM-built CLF-1 steel without residual austenite. Figure 1 shows the heterogeneous microstructures of the SLM-built CLF-1 steel with different morphologies. From the top views (Figure 1(b,k)), the S98 showed a square-shaped grain structure with checkerboard pattern due to the alternating 90° rotation of scanning strategy between each layer [10], while the S209 exhibited an entirely modified multimodal microstructure with a grain size distribution ranging from 0.17 μm to 5.65 μm (Figure 2). The front views (Figure 1(f,o)) reveal that the S98 and S209 both exhibited a bimodal microstructure consisting of domains with predominantly two different grain sizes (Figure 2), in which the columnar-shaped coarse grains were enveloped by the equiaxed fine grains with size of 1 ~ 3 μm . Compared to the S98, the S209 contained a higher fraction of fine grains and finer coarse grains with less evident epitaxial grain growth along the building direction (BD) (Figure 1(f,o) and Figure 2), as well as many lath martensites with lath width of 50 ~ 1000 nm as presented in the TEM bright field (BF) image (Figure 1(m)). As confirmed from the grain size distributions (Figure 2), the S209 experienced a significant grain refinement with average grain size of 2.5 μm and 16.5 μm in the top and front views, respectively. This can be mainly ascribed to the increased energy density, which is known to decrease the temperature gradient (G), and thus both $G \times$ growth rate (R) and G/R , facilitating the formation of fine equiaxed grains during the solidification [11,12].

The SEM images (Figure 1(c,g,l,p)) show many second phase particles in the top and front views. The TEM BF images (Figure 1(d,e,m,n)) further reveal that these fine spherical precipitates distributed at the grain/lath boundaries and within the grain/lath interiors as indicated by red arrows, and the particle size varied from 10 to 100 nm, which were expected to be the M_{23}C_6 or MX phase [13,14]. In addition, dislocations were observed to form in some fine and coarse grains as well as lath

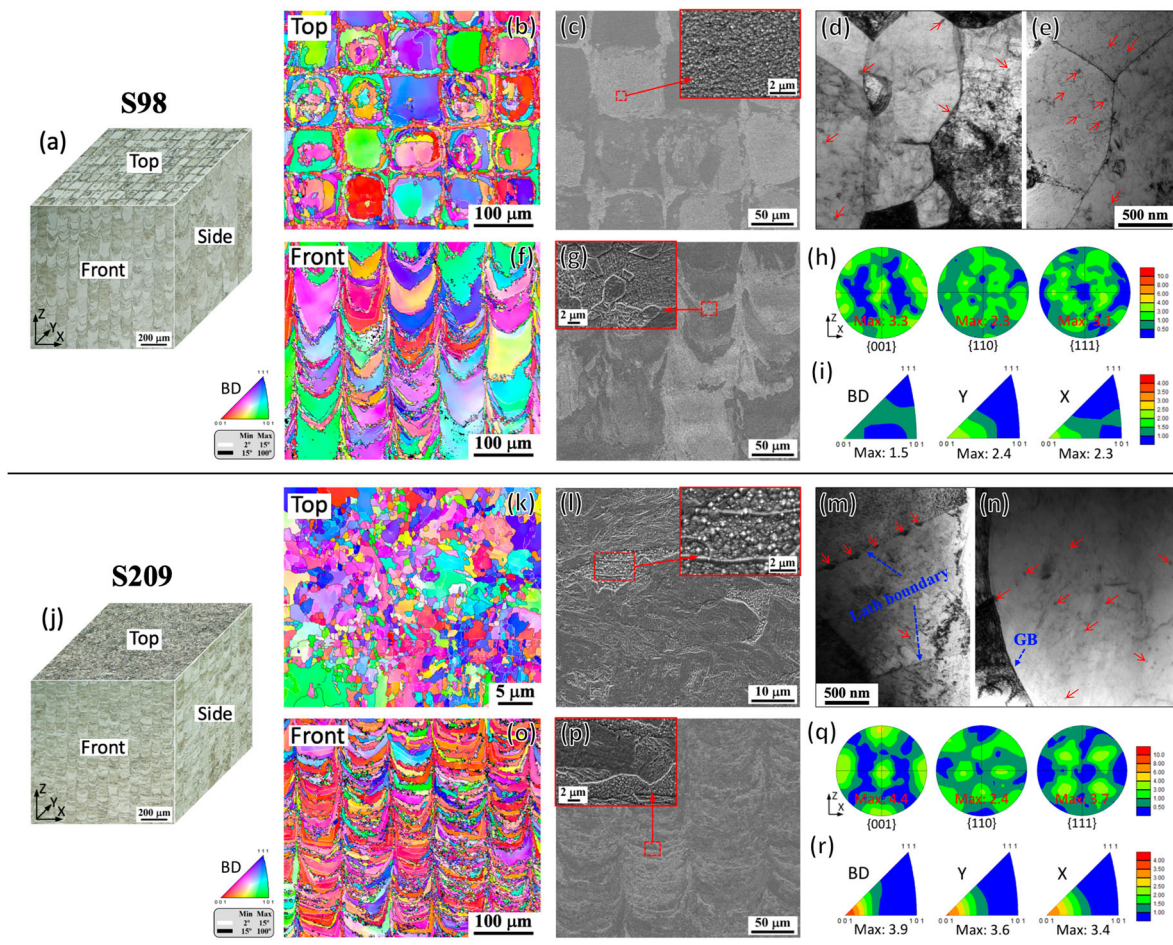


Figure 1. Microstructures and textures of the SLM-built CLF-1 steel. (a,j) Three-dimensional view of optical microstructures. (b,f,k,o) EBSD inverse pole figure maps and (c,g,l,p) corresponding SEM images. TEM BF images examined in the domains of (d) fine grains and (e,n) coarse grains as well as (m) lath martensite. (h,q) Pole figures and (i,r) corresponding inverse pole figures measured from the front view.

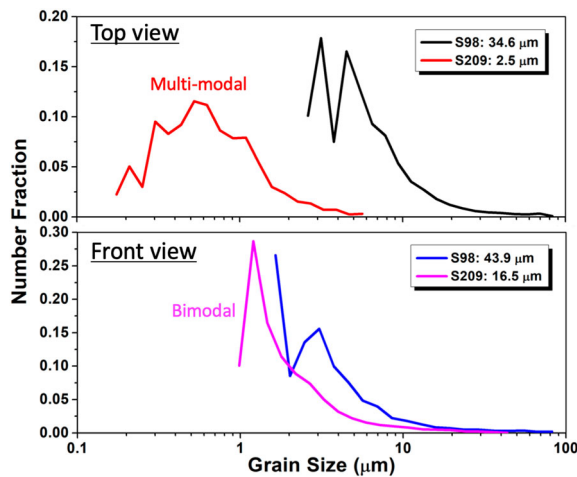


Figure 2. Grain size distributions of the SLM-built CLF-1 steel analyzed based on the EBSD results.

martensites (Figure 1(d,e,m,n)). In terms of the textures, the S98 showed a random texture (Figure 1(h,i)), while the S209 exhibited a typical cube texture, i.e. $\langle 001 \rangle$

direction parallel to the BD, with weak texture intensity of 4.4 (Figure 1(q,r)). The $\langle 001 \rangle$ is an easy growth direction for a cubic material and thus grows preferentially when the crystallographic axis of the $\langle 001 \rangle$ family is close to the thermal gradient direction [15,16]. With increasing energy density, the random texture altered into the cube texture, most likely due to a change in the melt-pool shape from shallow to protuberant [17].

Figure 3(a) shows the typical engineering stress-strain curves of the SLM-built CLF-1 steel and the relevant data are listed in Table S2. The S209 exhibited high strength with tensile yield strength (TYS) of 1053 ± 4.7 MPa and high ductility with elongation of $16.9 \pm 0.4\%$, which is much superior to the S98 (TYS of 865 ± 8.5 MPa and elongation of $1.6 \pm 0.6\%$). After subsequent tempering, the S98 and S209 showed a drastic increase in elongation with the sacrifice of TYS, but still comparable to the wrought CLF-1 steel. The data for various RAFeM steels fabricated by SLM [9] and conventional methods [18–24], as well as the present tensile properties are

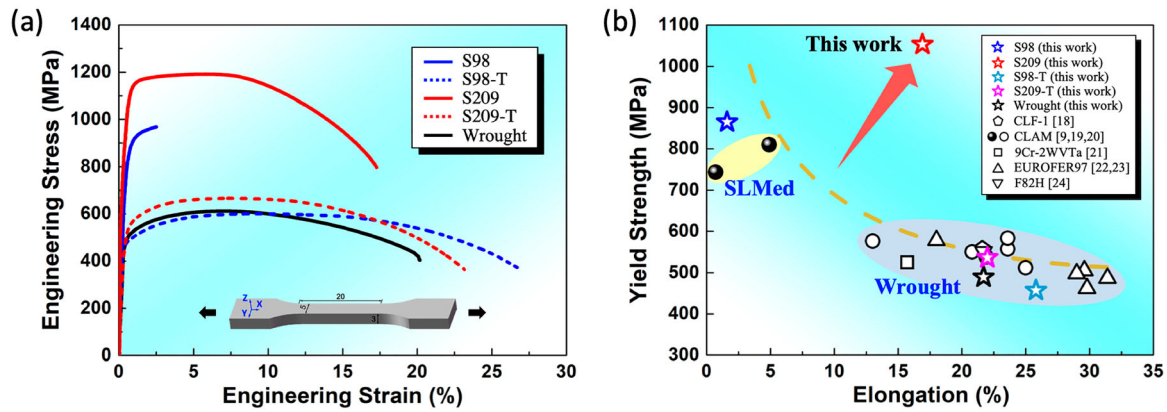


Figure 3. Tensile properties of the SLM-built CLF-1 steel. (a) Room temperature engineering stress-strain curves. (b) Relationship between TYS and elongation of various RAFA steels, including SLM-built [9] and conventionally processed [18–24] steels, as well as the present tensile properties.

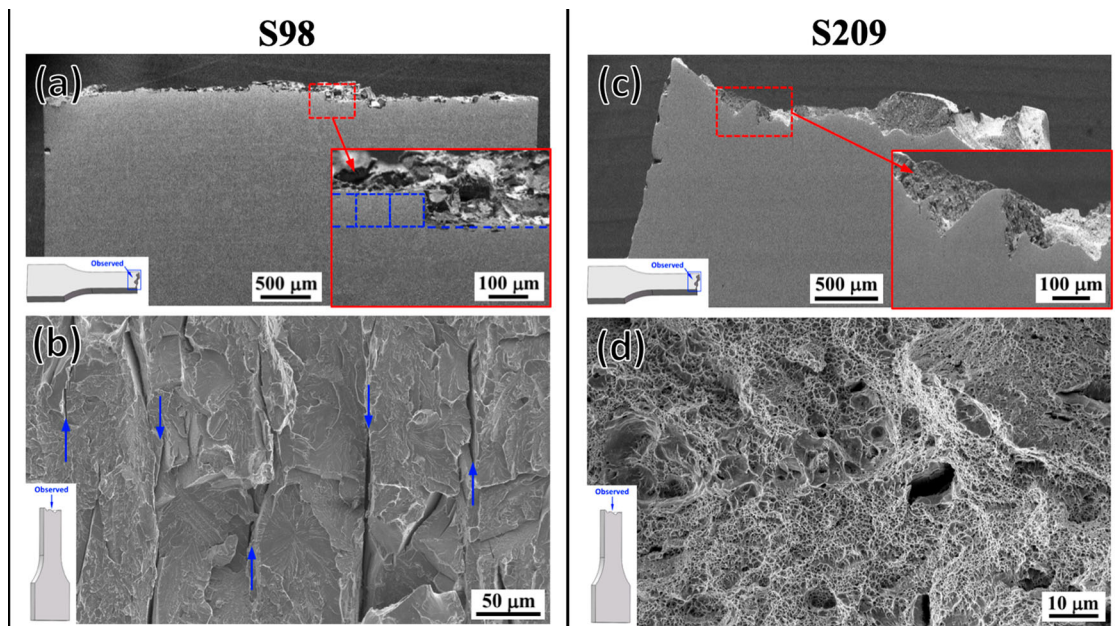


Figure 4. SEM images of the tensile fractured SLM-built CLF-1 steel on the (a,c) cross-sectional and (b,d) fracture surfaces.

included for comparison in Figure 3(b). Apparently, the S209 exhibited an excellent combination of strength and ductility, which sets the present CLF-1 steel apart from all the previously reported RAFA steels. Such comparison highlights the unprecedented properties obtained in this work and suggests that SLM process is a promising approach to synchronously enhance the strength and ductility.

Figure 4 illustrates the SEM images of the tensile fractured SLM-built CLF-1 steel with two distinct fracture features. As for the S98, fracture occurred mainly along the periphery of square-shaped grains as highlighted by blue lines, showing a flat cross-sectional profile (Figure 4(a)). The corresponding fracture surface (Figure 4(b)) displays typical river-patterned cleavage planes in accompany with deep and long cracks parallel

to the BD as indicated by blue arrows. Interestingly, such parallel-distributed cracks corresponded to the periphery of square-shaped grains observed in Figure 4(a). These observations reveal adequate evidences of intergranular brittle fracture, which results from the stress concentration at the periphery of square-shaped grains and its further conversion into micro-cracks that would propagate along such periphery upon straining. It is noted that the S157 in Table S1 exhibited a similar microstructure with the S98 and thus ended in a brittle fracture (Figure S4). Comparatively, the S209 experienced evident necking during tension (Figure 4(c)), exhibiting a typical ductile-type failure with the presence of numerous fine dimples (Figure 4(d)). Hence, the modified microstructure for the S209 alleviates the propensity for intergranular fracture and thus gives rise to a tensile fracture mode

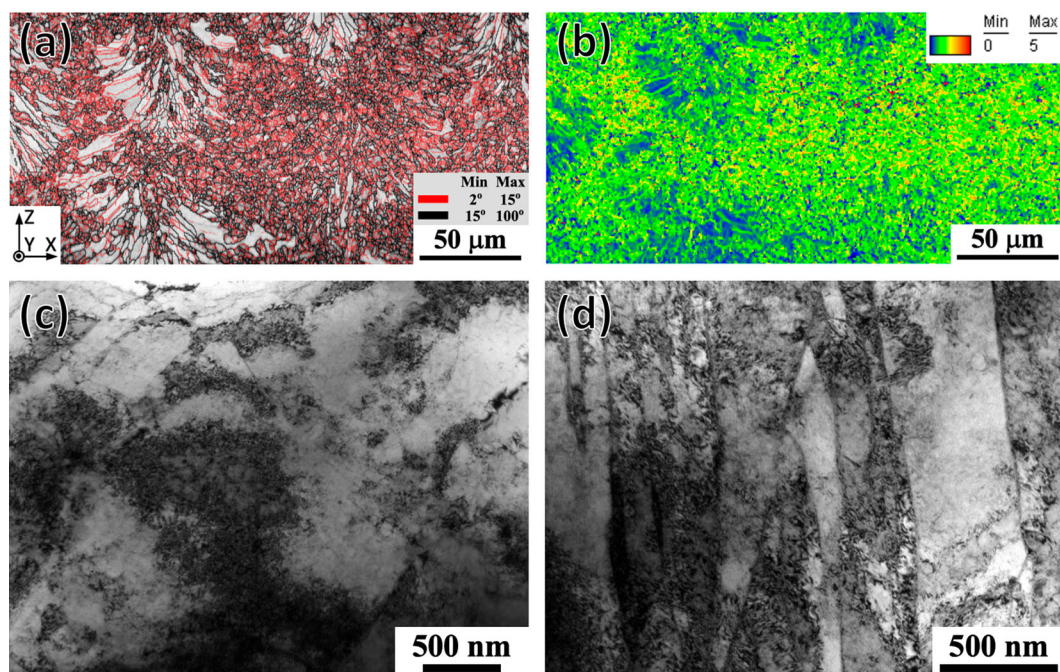


Figure 5. Deformation structures of the S209 tensile strained to (a–c) 2.5% and (d) fracture. (a) EBSD image quality map with superimposed LAGBs (red line) and HAGBs (black line) and (b) corresponding KAM map. TEM BF images at the location of (c) coarse grains and (d) lath martensites.

transition from cleavage to ductile, contributing to the ductility improvement.

In order to clarify the underlying deformation mechanisms of the SLM-built CLF-1 steel during tension, the microstructures of the S209 strained to 2.5% and fracture were further examined using EBSD and TEM, as presented in Figure 5. In the EBSD image quality map with superimposed low angle grain boundaries (LAGBs, 2–15°) and high angle grain boundaries (HAGBs, > 15°) (Figure 5a), the S209 contained a high fraction (~0.31) of LAGBs at a relatively low strain of 2.5%. Usually, the LAGBs come from the accumulated dislocations in deformed structure [25]. The corresponding kernel average misorientation (KAM) map (Figure 5(b)), which signifies the local misorientation and strain energy within the grain [26,27], reveals that the spots with high KAM value were accompanied with lots of LAGBs, suggesting the predominance of dislocation-accommodated deformation during tension. The TEM BF image (Figure 5(c)) further confirms the high activity of dislocations observed in the domain of coarse grains. Compared to the initial state (Figure 1(n)), the S209 strained to 2.5% showed an apparent increase of dislocation density in the form of dislocation walls (Figure 5(c)). As expected, the dislocation density further increased drastically and the well-aligned lath martensites were decorated with high density of dislocations when deformed to fracture (Figure 5(d)). Thus,

in general, the dislocation slip dominated the tensile deformation for the SLM-built CLF-1 steel.

Considering the dense and crack-free SLM-built CLF-1 steel, the high strength and high ductility of the S209 arise from the modified heterogeneous multi-/bimodal microstructure with negligible effect of metallurgical defects such as porosity. Specifically, the high strength can be mainly attributed to the refined grains from Hall-Petch relation and introduced lath martensites through its effect on the dislocation motion. Besides, fine precipitates and high density of dislocations are other two important strengthening factors for the SLM-built CLF-1 steel, while the texture makes limited contribution due to its weak texture intensity. The tempering treatment generally reduces the dislocation density, and it is observed in Figure S5 that after tempering the second phase particles grew obviously with nearly stable multi-/bimodal microstructures and textures, leading to the much lower TYS. This in turn supports the contribution of the dislocation strengthening and second-phase strengthening and also confirms the effectiveness of solid solution strengthening in the SLM-built CLF-1 steel.

On the other hand, it is expected that the heterogeneous multi-/bimodal microstructure contributes to the steady strain hardening capability that stabilizes the tensile deformation, which postpones the initiation of necking and thus avoids premature fracture [26,28]. In fact, the bimodal structure was firstly proposed for

high-strength and ductile nanostructured metals with hard domain of fine grains embedded inside a matrix of soft domain of coarse grains [29]. For the case of the S209, hard fine grains improve the flow stress, and soft coarse grains serve to offer sufficient space to further store dislocations (Figure 5(c)) and typically accommodate more strains during deformation. Additionally, relative to a uniform structure long-range back stress may exist during deformation in such heterogeneous microstructure due to the plasticity mismatch at the interface between the domains of fine and coarse grains [26–28,30], which provides additional strain hardening and consequent high ductility. Also, the S209 contained many fine lath martensites, which act as strong obstacles against dislocation movement and force the dislocations to tangle and accumulate around the lath boundaries (Figure 5(d)), increasing the strain hardening. Therefore, architecting the heterogeneous multi-/bimodal microstructure is highly desirable for additively manufactured CLF-1 steel to achieve an excellent combination of strength and ductility. Given that the size and volume fraction of different domains in the multi-/bimodal microstructure can be further modified feasibly through optimizing the SLM parameters and scanning strategy, the strength and ductility are expected to be improved even beyond the levels presented in this work.

Conclusions

In summary, strong and ductile CLF-1 steel was additively manufactured by SLM successfully with an excellent combination of strength and ductility (i.e. TYS of 1053 ± 4.7 MPa and elongation of $16.9 \pm 0.4\%$), which is much superior to the previously reported RAFM steels. The simultaneous improvement in strength and ductility has been attributed to the unique structure formed during SLM, i.e. heterogeneous multi-/bimodal microstructure consisting of domains of fine and coarse grains, as well as lath martensites. The refined grains and lath martensites mainly contributed to the superior strength, and the multi-/bimodal microstructure facilitated the steady strain hardening controlled by dislocation activities, which postponed the necking and thus improved the ductility. This work proposes a new strategy for developing high-strength and ductile RAFM steel by designing the multi-/bimodal microstructure via SLM, potentially enhancing the steel's suitability for the application in the fusion reactor.

Disclosure statement

No potential conflict of interest was reported by the authors.

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