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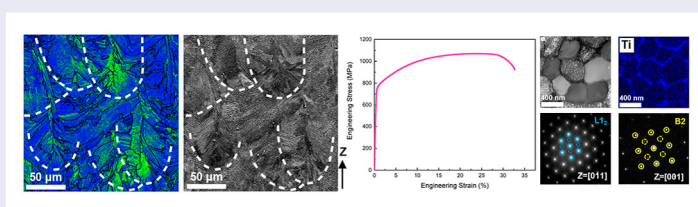
Dual heterogeneous structure facilitating an excellent strength-ductility combination in an additively manufactured multi-principal-element alloy

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ABSTRACT

The (FeCoNi)₈₆Ti₇Al₇ multi-principal-element alloy with a dual heterogeneous microstructure was successfully fabricated by selective laser melting, exhibiting an excellent combination of strength (ultimate tensile strength, 1085.2 ± 23.2 MPa) and ductility (30.5 ± 2.6%). It is evidenced that the joint effects of the hetero-deformation induced hardening from grains with heterogeneous geometrically necessary dislocations densities, in-situ formed B2 phase, and the coherent precipitation hardening from in-situ formed nano L1₂ phase were responsible for the strength. This work sheds light on the feasibility of simplifying the production of multi-mechanism strengthened alloys within one step and paves a new avenue to produce high-performance complex-shaped components.



IMPACT STATEMENT

(FeCoNi)₈₆Ti₇Al₇ multi-principal-element alloy exhibiting heterogeneity on the grains structure and in-situ precipitation was successfully fabricated by selective laser melting. It shows both good tensile strength and ductility.

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KEYWORDS

Additive manufacturing; multi-principal-element alloy; dual heterogeneous microstructure; mechanical properties; hetero-deformation induced hardening

1. Introduction

The idea of multi-principal-element alloy (MPEA) has been considered to be a breakthrough to the traditional single-principal-based alloy design strategy, and hence potentially to further extend the up-limit of strength-ductility combination [1–3]. In particular, the face-centered-cubic (fcc) MPEAs often show high work-hardening capability due to the intrinsically activated multiple dislocation slip system during straining, leading to good resistance to crack propagation and the consequent descent tensile ductility [2]. However, most fcc-MPEAs generally present low strength [2], making strengthening fcc-MPEAs a quite hot topic.

Inducing precipitation of the L1₂ phase and introducing heterogeneous grain structure are both effective

and widely employed strengthening methods for fcc-MPEAs. The coherent interface between the L1₂ phase and the fcc matrix relieves the interfacial stress concentration, significantly improving the strength without sacrificing too much ductility [4–10]. While the heterogeneous grain structure can induce a gradient distribution of strain around interfaces, triggering an extra work-hardening effect [11–15]. For further improvement on the mechanical performance, the precipitation hardening and heterogeneous grain structure have been simultaneously employed in MPEAs with complex processing methods that involve multi-pass rolling and annealing [16–19]. During the annealing, the significant difference in the driving force provided by the heterogeneous matrix usually causes heterogeneity in the phase, morphology,

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and distribution of precipitates, which enables the formation of a dual heterogeneous microstructure (heterogeneous grain structure and heterogeneous precipitates) and achieves an even better strength-ductility combination [16,17]. However, tuning the dual heterogeneous microstructure by thermomechanical treatments is still too inconvenient for a wide industrial application. Fortunately, additive manufacturing, a recently developed fabrication route, seems to provide a possible solution to this dilemma. Additive manufacturing (AM) is a one-step near-net manufacturing method that can produce parts with complex geometry, while the complex thermal cycles during processing give rise to the tendency to form in-situ precipitation and microstructure in different degrees of heterogeneity [20–23], making it a promising method to combine the precipitation and heterogeneous grain structure within one step.

In this work, by alloying a high amount of Ti and Al simultaneously, we designed an MPEA with a composition of $(\text{FeCoNi})_{86}\text{Ti}_7\text{Al}_7$ (at.%) that refers from the literature [9] with good mechanical performance yet only after complex processing. The SLMed alloy exhibits a heterogeneous matrix, which presents grains with different geometrically necessary dislocations (GNDs) densities, and the pronounced heterogeneity on the precipitation, which relates to the B2 phase and L_{12} phase. This dual-heterogeneity endows the alloy with a decent combination of strength and ductility, without any in-following thermomechanical process. The strengthening mechanism was discussed in accompany with microstructure analyses.

2. Materials and methods

The pre-alloyed $(\text{FeCoNi})_{86}\text{Ti}_7\text{Al}_7$ (in at. %) powders were produced by gas atomization, and the particle size ranges from 8 to $45\text{ }\mu\text{m}$ with a mean value of $29.8\text{ }\mu\text{m}$ (Figure S1, Supporting information). The SLM process was conducted on a commercial machine (FS271M, Farsoon, China), with a laser power of $400\text{ }\mu\text{m}^2/\text{s}$ and a scanning speed of $1200\text{ }\mu\text{m}/\text{s}$. The beam size is $90\text{ }\mu\text{m}$, the hatching space is $110\text{ }\mu\text{m}$, and the layer thickness is 0.04 mm . Argon atmosphere was used to prevent oxidation during SLM. The laser was rotated 67° in-between each scanning layer. The geometry of the as-built material block was designed in dog-bone shape, with a gauge length of 9.525 mm , a width of 2 mm , and a thickness of 10 mm . The specimens for tensile tests were prepared via slicing the as-built blocks by wire-electrode. Each tensile specimen has a thickness of $\sim 2\text{ mm}$ after polishing. The density of the as-built block was measured three times by the Archimedes method.

The phase constitution of the as-built $(\text{FeCoNi})_{86}\text{Ti}_7\text{Al}_7$ HEA was identified by an X-ray diffractometer (XRD, Rigaku D/MAX-2250, Japan) with a $\text{Cu K}\alpha$ radiation. Electron backscatter diffraction (EBSD) characterization was conducted using an SEM (Zeiss Supra 55) equipped with an EBSD Detector. Electron channeling contrast imaging (ECCI) was conducted on a scanning electron microscope (SEM, Carl Zeiss SMT AG, Germany) with a Gemini-type field emission gun electron column. Conventional TEM characterizations were performed by transmission electron microscope (TEM, JEOL-2100F, Japan) equipped with an energy dispersive spectrometer (EDS). Elemental distribution was further characterized by another TEM (Thermo Fisher Talos 200X) equipped with a super EDS.

All tensile tests were conducted at room temperature by using an Autograph AGS-X 50 kN tester with an extensometer. The test was repeated three times to ensure the reproducibility. The strain rate of conventional tensile tests is $1 \times 10^{-3}\text{ s}^{-1}$. The loading-unloading-reloading (LUR) tensile tests were performed at room temperature with a strain rate of $1 \times 10^{-3}\text{ s}^{-1}$. In each LUR cycle, the sample was first stretched to a predefined strain at a constant strain rate of $1 \times 10^{-3}\text{ s}^{-1}$ and then unloaded in a load-control mode to 50 N. Subsequently, the sample was stretched again at a strain rate of $1 \times 10^{-3}\text{ s}^{-1}$ before the next unloading.

3. Results and discussion

The bulk density is measured to be $7.718 \pm 0.001\text{ g/cm}^3$. The low magnification SEM characterization and EDS mapping of the undeformed sample are presented in Figure S2, showing no macroscopic voids and homogeneous element distribution. Figure 1(a, b, d, e) present the hierarchical microstructure and heterogeneous grains morphology resulted from the layer-by-layer fabrication mode and the accompanied remelting [24,25]. Overlapped melt tracks are observed in Figure 1(f). The scanning strategy is shown in Figure 1(j). Slanted columnar grains (white arrows) grow perpendicular to the melt pool boundaries due to the sizeable and directional thermal gradient, while relatively finer grains (black arrow in Figure 1(b)) may form at regions near multiple melt pool boundaries (marked with dashed lines in Figure 1(c)) due to the multiple remelting and reheating brought by overlapping of melt pools [26].

Figure 1(g and h) show the local misorientations on XZ plane and XY plane. Previous investigations have confirmed that the density of geometrically necessary dislocations (GND) is positively proportional to the KAM value [27]: $\rho_{\text{GNDs}} = 2\theta_{\text{KAM}}/ub$. θ_{KAM} is the average local misorientation, b is the Burger's vector and u is the

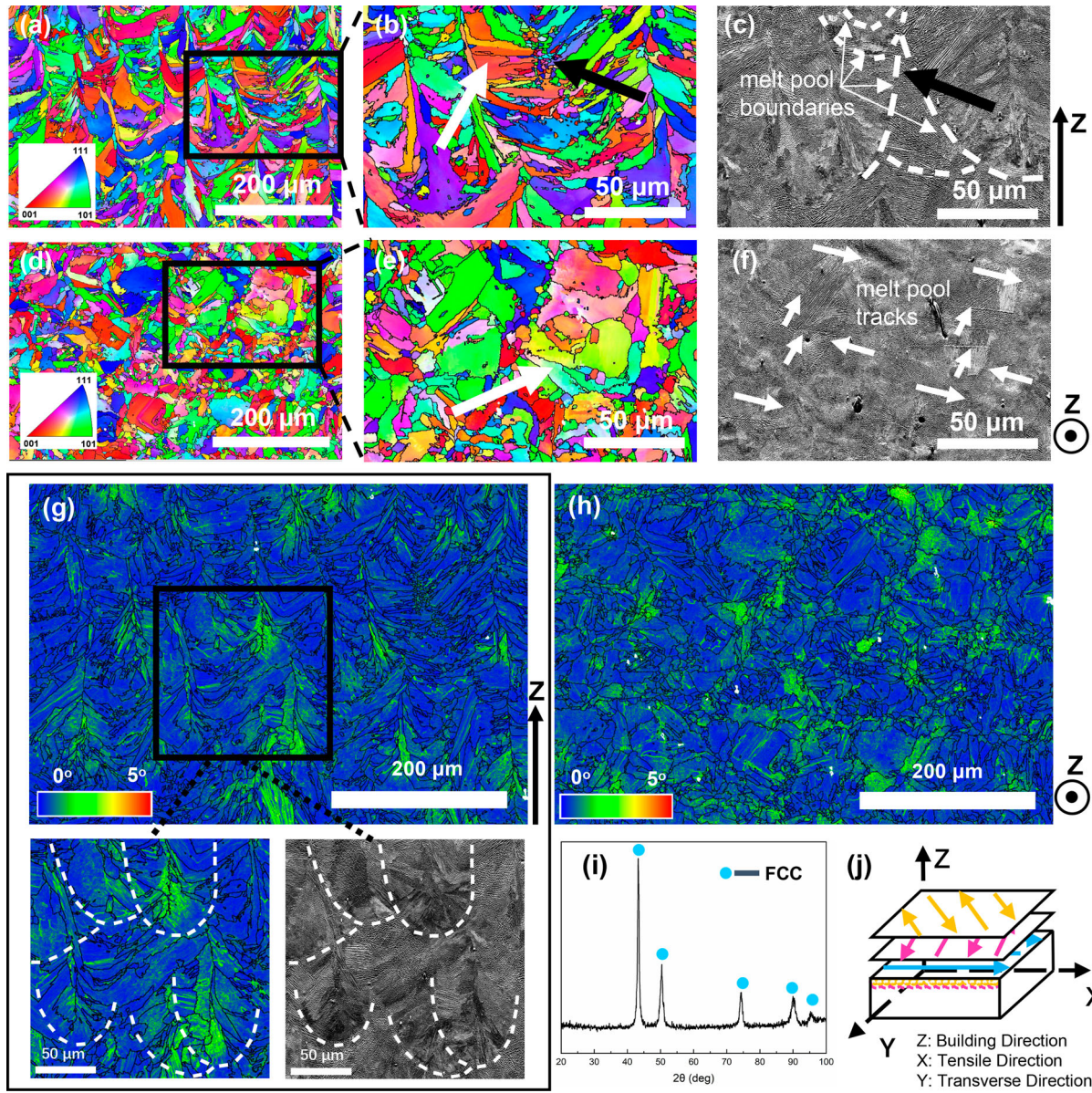


Figure 1. EBSD characterization of the (FeCoNi)₈₆Ti₇Al₇ alloy. (a) EBSD inverse pole figure (IPF) map on XZ plane; (b) Enlarged view in (a); (c) Integrated forward scatter detector (FSD) map of (b); (d) EBSD IPF map on XY plane; (e) Enlarged view in (d); (f) Integrated forward scatter detector (FSD) map of (e); (g) KAM map corresponding to (a) (step size = 0.5 μm); (h) KAM map corresponding to (d) (step size = 0.5 μm); (i) XRD pattern of the investigated alloy; (j) Schematic diagram of the scanning strategy in SLM process.

unit length (step size used in EBSD characterization). Figure 1(g) exhibits an inherent heterogeneity in the local misorientations, reflecting more severe strains and higher densities of GNDs from the centre to the bottom of melt pools. The initial strain and the high densities of GNDs in materials produced by AM are ascribed to the expansion/contraction heterogeneities produced by the localized heating/cooling [28]. The unstable solidification process enables uneven initial stress and strain among grains, thus GNDs are generated to coordinate the microscopic deformation and maintain the continuity [29]. Furthermore, the shrinkage of the whole melt pool

area will be restricted by the heat affected area beneath the melt pool, leading to even intenser strain localization, which is consistent with the phenomenon that there is higher GNDs density around grains at the bottom and centre area of melt pools [28,30]. It is worth noting that except for GNDs, there may also be statistically stored dislocations (SSD) in the alloy, but the distribution of SSDs cannot be displayed via KAM map since they have no cumulative effect on the local misorientation [31,32]. Most of SSDs may be annihilated under cyclic heating, and GNDs may be dominant in the remaining dislocations, so more attention is paid to the GNDs. The XRD

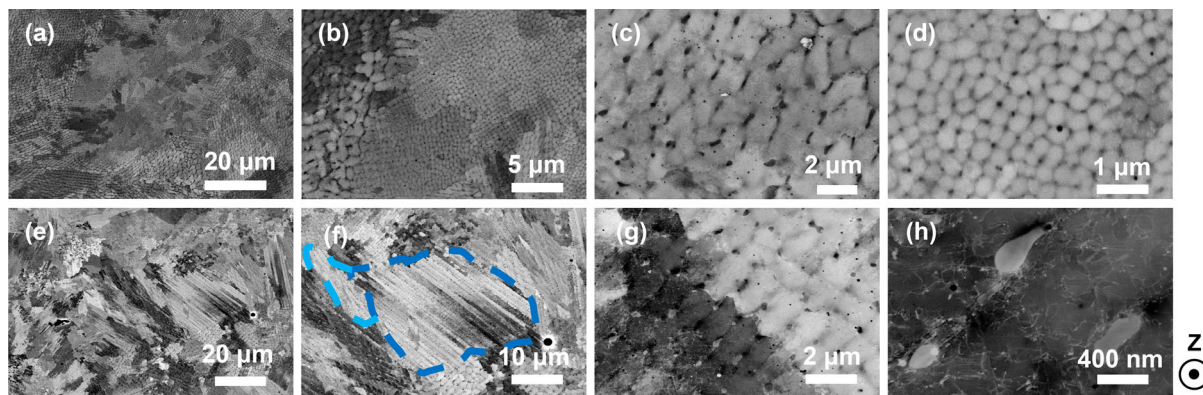


Figure 2. SEM (a-d) and ECC images (e-h) of the XY plane of the investigated alloy: (a), (e) Grains composed of cellular structures; (b), (c), and (d) cellular substructures in different sizes; (f) Cellular structures exhibited in columnar morphology; (g) Precipitates distributed along cellular structures boundaries; (h) A close-up on precipitates with dislocations around.

pattern in Figure 1(i) shows a single fcc crystalline structure, yet no additional peaks can be identified for other phases.

Figure 2(a, b, e, f) present that all grains are composed of fully-developed sub-grain cellular structures or columnar structures (Figure 2(f)), respectively. According to the references [33–36], the cellular structures are columnar structures that are displayed cross-sectionally. To facilitate discussion, both of them are denoted as ‘cellular structures’ hereafter. Figure 2(b–d) present the obvious discrepancy in the size of cellular structures and inter-cellular precipitates from region to region. Moreover, the size of inter-cellular precipitates shows a positive dependent relationship with the size of cellular structures. Figure 2(g–h) exhibit more details about precipitates. High-density dislocations are observed around the inter-cellular precipitates as well as to lie along the cellular boundaries as shown in Figure 2(h).

To further uncover the constituent phase structure, TEM characterizations were carried out. The failure to characterize precipitates in XRD pattern may be probably due to the small amount (volume fraction of 3.6%) of the inter-cellular precipitates, slightly below the detectable limit of the XRD technique. Figure 3(d), the selected area electron diffraction (SAED) pattern of the circled area in Figure 3(a), reveals that the inter-cellular precipitate is a B2 phase, which is frequently observed in the other Al/Ti containing MPEAs [37–39]. Figure 3(c) shows the columnar structures, which is the cellular structure exhibited in another view. The inter-cellular B2 precipitate corresponding to Figure 2(c–d, g–h) are observed along the boundaries as circles indicate, and there are tangled high-density dislocations around the cellular structure boundaries. Figure 3(e), the SAED pattern of the circled area in Figure 3(b) also shows that the matrix is an fcc structure. Interestingly, the appearance of the ordered

superlattice that marked by the dash lined circles indicates the formation of the $L1_2$ precipitates. Figure 3(f) shows that the $L1_2$ precipitates are pretty small, mostly below 5 nm, consistent with the weak superlattice spots. Figure 3(g) reveals that there are Ti segregation and Fe depletion along the cellular structure boundaries.

In summary, the SLMed (FeCoNi)86Ti7Al7 alloy displays a dual heterogeneous microstructure both in the matrix and the precipitates. The matrix containing grains in different morphologies exhibits an inherent GNDs variation from region to region. The precipitates include the B2 phase in size ranging from tens to hundreds of nanometers distributing along cellular structure boundaries, and $L1_2$ in size below 5 nm inside cellular structures.

The remelting and reheating fabrication mode brings about the variation of the solidification rate and cooling rate, plays a similar effect of aging, subsequently inducing the heterogeneous precipitation of the B2 phase and $L1_2$ phase. As Figure S3 and Table S1 and Table S2 show, the matrix where the size of cellular structures are finer contains more Ti and Al, and the inter-cellular precipitates also exhibit finer size and lower content of Ti and Al. This local composition difference may also result from the remelting and varied cooling rate, which also enhances the heterogeneity of microstructure. As the substitutional solute, Ti atoms tend to move to the loosely packed region like the cellular structure boundaries due to its relatively larger size [40]. The high Ti/Al ratio along cellular structure boundaries promotes the formation of the B2 phase, while the $L1_2$ phase precipitates inside the cellular where there is a lower Ti/Al ratio [41]. Since the interface between the B2 phase and the fcc matrix is incoherent [42], the inter-cellular boundaries, which are kind of defects, can act as an excellent position for precipitation to lower the lattice distortion.

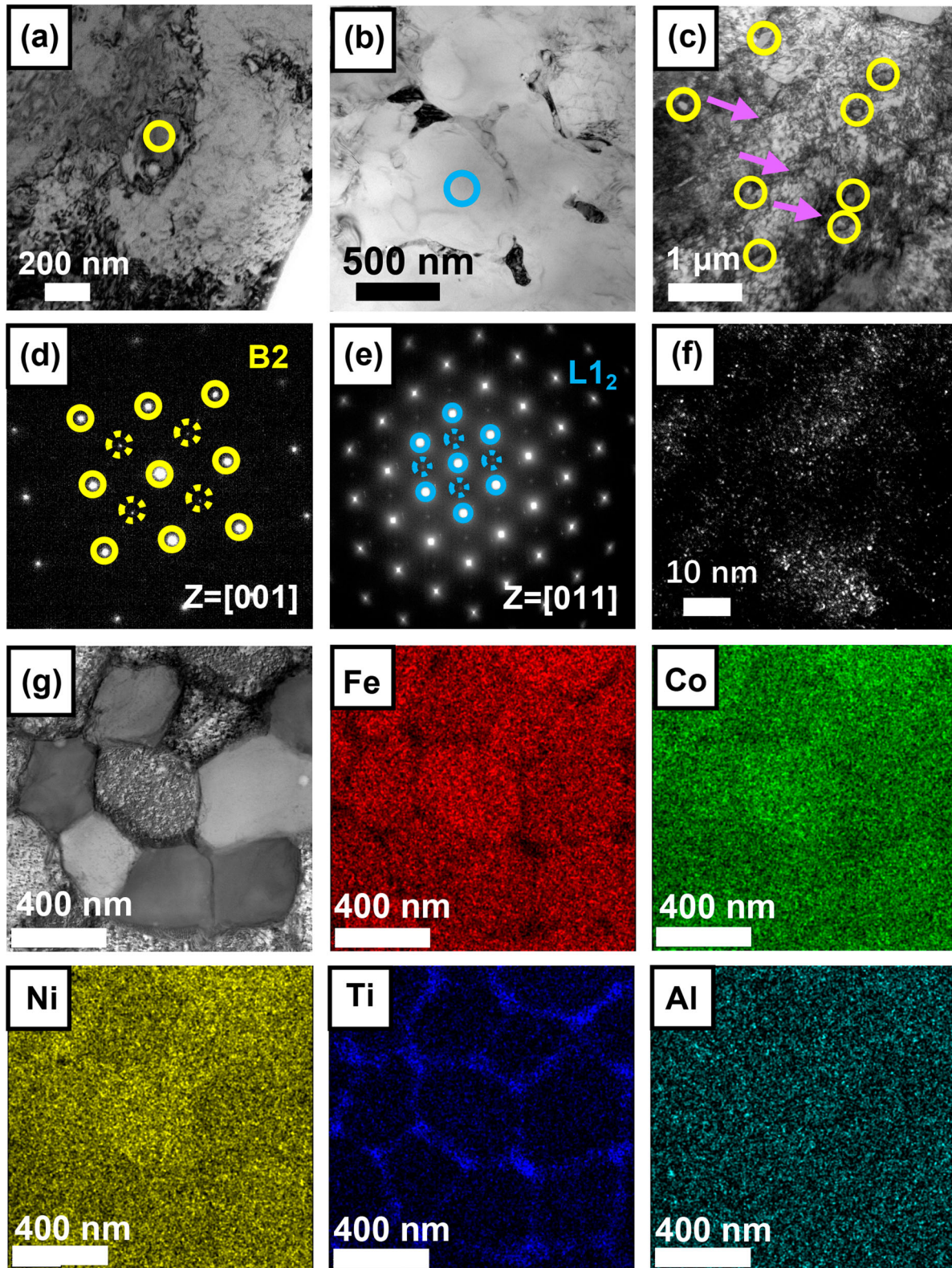


Figure 3. (a, b) Bright-field image of the investigated alloy; (c) Bright-field image of cell substructures decorated with dislocations and B2 precipitates along boundaries; (d, e) SAED patterns corresponding to the circled areas in (a) and (b), respectively; (f) Dark-field image of the L₁₂ precipitate inside; (g) Bright-field image and TEM-EDS mapping of cellular structures.

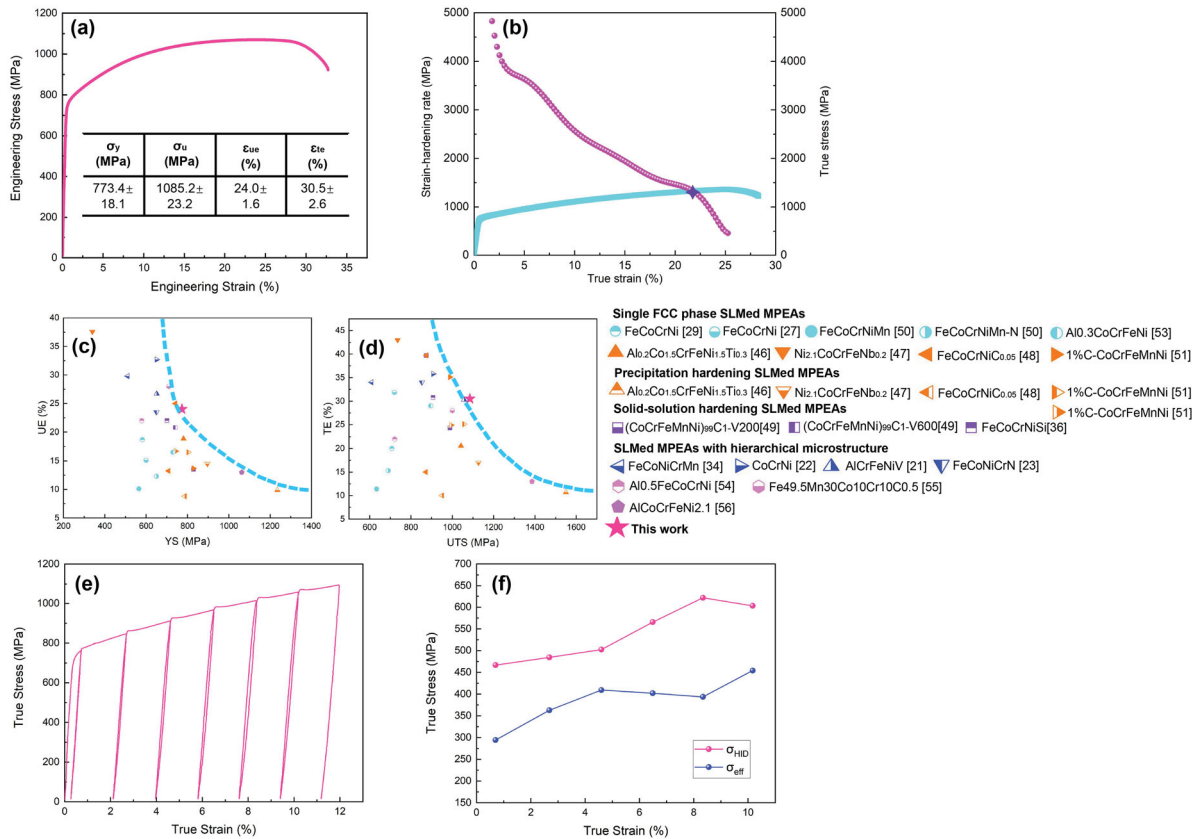


Figure 4. (a) Representative tensile curve of SLMed (FeCoNi)₈₆Ti₇Al₇ alloy; (b) The true stress-true strain curve and strain-hardening rate curve corresponding to (a); (c and d) Comparisons of the tensile properties of our present alloy with other excellent SLMed MPEAs; (e) LUR true stress-true strain curve; (f) Evolution of σ_{HDI} and σ_{eff} with increasing true strain.

As for the cellular structures, the formation mechanism is still incompletely revealed. Previous studies have ascribed it to the cellular solidification accompanied by solutes redistribution or segregation induced by constitutional supercooling and vertex inside melt pool [43]. The local composition variations among cellular structures in different sizes in the SLMed (FeCoNi)₈₆Ti₇Al₇ alloy indicates that redistribution of solutes does happen while the formation of cellular structures. The addition of Ti and Al may facilitate the constitutional supercooling and further promote cellular solidification. But how solute segregation interacts with the inherent dislocations, and whether it is one of the origins of dislocations are still questions. It has been verified that there still is a high density of dislocations and cellular structures inside additive manufactured pure Cu [28]. So the microscopic compression-tension brought by the periodic heating and cooling is regarded as one of the origins of dislocations [28,44,45], which also agrees with the GNDs distribution here. Moreover, Ti-6Al-4V, which exhibits inherent weak elemental segregation and fast phase transition, shows no cellular structures even with a large density of dislocations inside when fabricated via additive manufacturing [44]. This phenomenon indicates that the elemental

composition and crystal structure have an effect on the formation of cellular structure since they influence the solidification microstructure and the intrinsic moving behavior of dislocations.

Figure 4(a) shows the representative tensile curves of the as-built (FeCoNi)₈₆Ti₇Al₇ alloy tested at room temperature. The alloy shows a yield strength (YS) of 773.4 ± 18.1 MPa, an ultimate tensile strength (UTS) of 1085.2 ± 23.2 MPa, a uniform elongation (UE) of $24.0 \pm 1.6\%$, and a total elongation to fracture (TE) of $30.5 \pm 2.6\%$. The corresponding true stress-true strain curve and strain-hardening rate curve reveal that our present alloy can keep enough strain-hardening rate to avoid the plastic instability even when the true strain exceeds 20%, as shown in Figure 4(b). Figure 4(c-d) is a comparison of our present alloy with other excellent SLMed MPEAs [21–23,27,29,34,36,46–56], showing the superiority in the combination of strength and ductility. It is worth noting that the size of tensile test samples may affect the mechanical properties [57].

LUR test was also carried out, as shown in Figure 4(e). The distinct occurrence of hysteresis loops indicates a strong Bauschinger effect [11]. The flow stress of the alloy at a specific strain can be divided into the HDI stress

(σ_{HDI}) and the effective stress (σ_{eff}) [58], both showing a growing trend with the increasing strain, as Figure 4(f) exhibited.

Since the higher KAM value indicates a higher density of geometrically necessary dislocations (GNDs) and a severer strain, the regions with higher GNDs density may have experienced stronger work hardening compared with the regions with lower GNDs density. The region

with a higher KAM value shows higher Vickers hardness as the nanoindentation results presented in Figure S5 and Table 3. Hence, the region with higher GNDs density are hard domains, regions with lower GNDs density are soft domains. The strain incapability between soft and hard domains can bring out the HDI hardening.

Figure 5 presents the EBSD IPF maps, KAM maps, and FSD maps of microstructures on the XZ plane before and

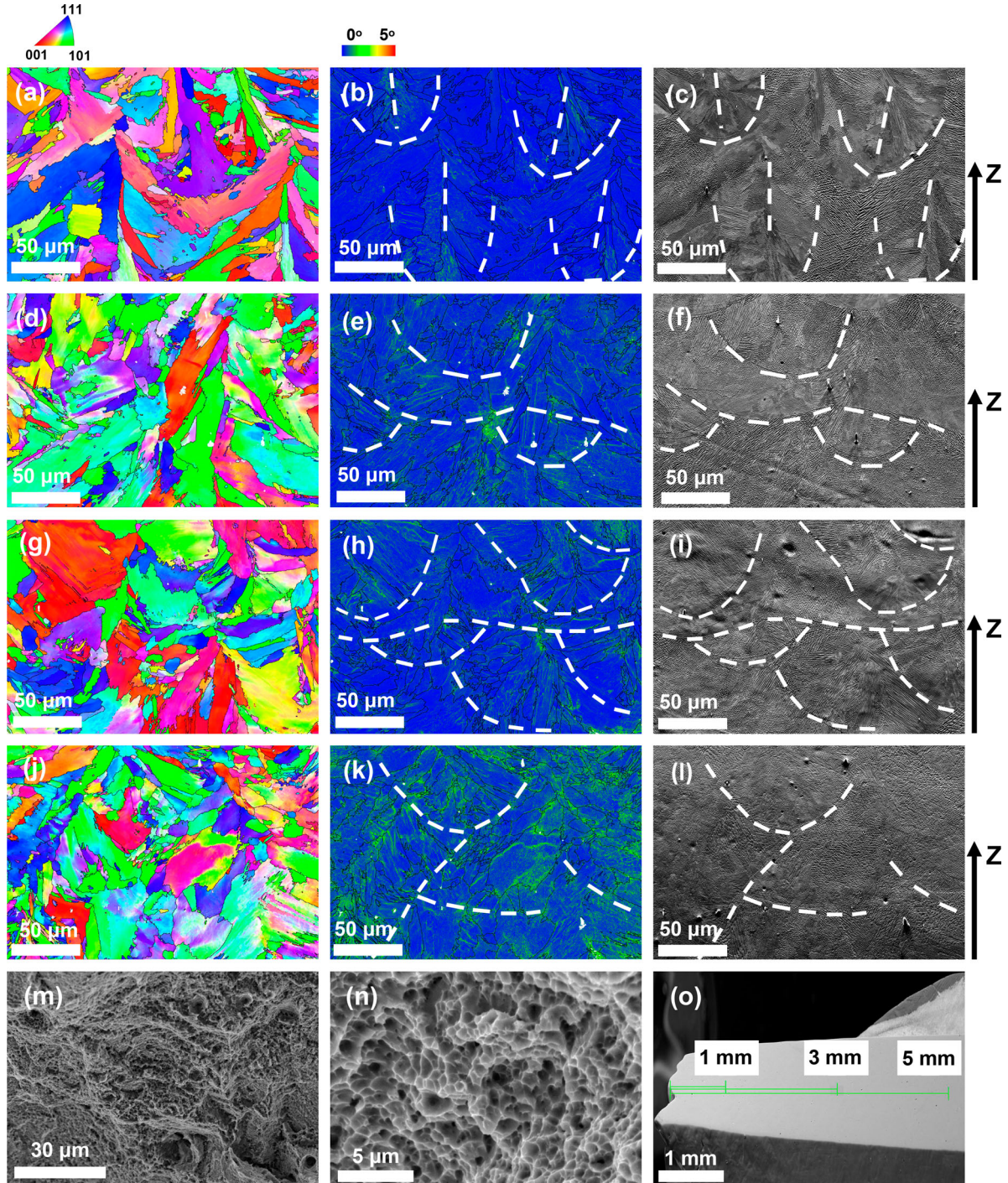


Figure 5. EBSD IPF map, KAM map, and FSD map of microstructure on XZ plane (step size $\mu = 0.1 \mu\text{m}$): (a–c) undeformed area; (d–f) 5 mm from the fracture; (g–i) 3 mm from the fracture; (j–l) 1 mm from the fracture; (m–n) SEM images showing the fractographic features of the investigated HEAs; (o) illustration of the regions picked for the characterization.

after the tensile test. Microstructures after deformation are picked at regions 5, 3, and 1 mm away from the fracture to represent microstructure with different degrees of strain, which is illustrated in Figure 5(o). As Figure 5(a-c) show, in undeformed microstructure, the grains at the bottom area or centre area of melt pools show evident higher misorientations, which is consistent with Figure 1(g). In the region 5 mm away from the fracture (Figure 5(d-f)), the KAM concentration phenomenon becomes less obvious, indicating that soft regions (regions with less initial GNDs) start to deform first at the initial stage of deformation. In the region 3 mm away from the fracture (Figure 5(g-i)), the KAM concentration phenomenon was enhanced. The grains near the centre and bottom of melt pools show even stronger local misorientations, indicating that when the strain increases, the hard domain is also activated. Finally, the region 1 mm away from the fracture (Figure 5(j-l)) shows much severer orientations changes compared to the initial state, suggesting the severe deformation near the fracture.

At the initial deforming stage, soft domain is firstly activated. To maintain the strain continuity, GNDs would be generated and pile up near the interface between soft and hard domains. The accumulated GNDs produce the long-range internal stress that reduces the applied effective resolved shear stress for dislocation slip [58], leading to an increase of the yield strength. As the deformation proceeding, GNDs keep accumulating, thus provide a persistent long-range internal stress and the subsequent high strain hardening, stabilizing the plastic deformation and leading to good ductility [11,59,60]. Pronounced dimples can be observed at the fracture surface as Figure 5(m and n) show, exhibiting a ductile fracture.

The σ_{HDI} is the directional long-range internal stress provided by the pile-up and accumulation of GNDs against barriers including dislocation walls, dislocation cells, grain boundaries, and incoherent interfaces. The σ_{eff} is usually attributed to the interaction between the moving dislocations, pre-existed dislocation forest, and coherent precipitates [16]. As the interface between B2 precipitates and the matrix is incoherent [42], dislocations pile up at the interface, or bow out and leave dislocation loops when passing [61,62]. B2 precipitates, and the following piled dislocations, and dislocation loops actually also provide long-range internal stress [18,63], playing the same reducing effect on the applied effective resolved shear stress for dislocation slip as which the GNDs accumulation induced by heterogeneous deformation provides. Thus, the strengthening contribution from B2 precipitates is also part of the HDI hardening [64]. In contrast, due to the small size of L_{12} particles and low misfit between the L_{12} particles and the matrix, dislocations can shear the L_{12} particles during

the deformation [5]. Therefore, the interaction between moving dislocations and the L_{12} phase contributes to effective stress.

The strengthening contribution from HDI hardening, solid-solution hardening, and precipitation hardening are calculated to be around 466.7, 33.4, and 170.1 MPa, respectively (detailed calculations are presented in Supplementary information). The calculation values indicate the HDI hardening makes the most contribution, suggesting the superiority of the dual heterogeneous structure.

4. Conclusion

In this study, the $(\text{FeCoNi})_{86}\text{Ti}_7\text{Al}_7$ alloy accompanied by a dual-heterogeneous microstructure was successfully fabricated through a selective laser melting process. It exhibits a good combination of both the strength and ductility. The excellent mechanical properties result from the HDI hardening and precipitation hardening. Introducing the dual-heterogeneous microstructure via selective laser melting sheds light on the feasibility of simplifying the production of dual heterogeneous microstructure, and the capability to produce high-performance complex-shaped components within one step.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of other ongoing studies.

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