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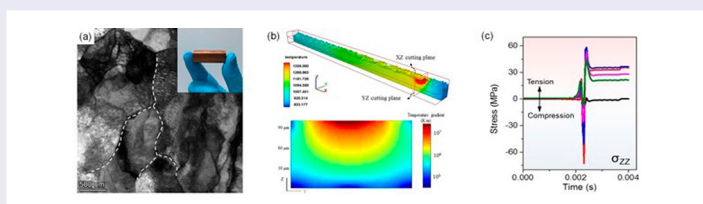
The origin of high-density dislocations in additively manufactured metals

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ABSTRACT

The origin of dense dislocations in many additively manufactured metals remains a mystery. We here employed pure Cu as a prototype and fabricated the very challenging high-purity (> 99.9%) bulk Cu by laser powder-bed-fusion (L-PBF) technique. We found that high-density dislocations were present in the as-built samples and these high-density dislocations were introduced on the fly during the L-PBF process. A newly developed multi-physics modeling was further employed to interpret the origin of these pre-existing dislocations, demonstrating that the compression-tension cycles rendered by the localized heating/cooling heterogeneity upon laser scanning are responsible for the residual high-density dislocations.



IMPACT STATEMENT

The origin of dense dislocation structures in many as-built additively manufactured metals, which incurs a long-standing dispute, was found to be inherent to the additive manufacturing process.

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KEYWORDS

Additive manufacturing; Cu; microstructure; dislocation; multi-physics modeling

1. Introduction

The deformation of metals is normally associated with dislocations. The primary dislocation density and distributions are critical factors which control dislocation nucleation, storage and annihilation during straining, therefore dominate the strengthening and hardening responses of a metal [1,2]. High-density dislocations were considered to be less possible in additively manufactured (AM) metals, owing to the high-temperature and melting-solidification nature of the additive manufacturing processes [3,4]. However, dislocations were found to prevail in many AM metals, particularly in metallic materials with face-centered cubic (FCC) crystalline structure, mediating (or governing) their deformation mechanisms and the resultant mechanical properties. For instance, low-energy dislocation cells were commonly observed in AM 316L stainless steels (SS)

[2,5,6], and this kind of dislocation arrangement shows the ability to regulate dislocation glide/storage and facilitate abundant interactions with deformation twins, thus producing an extended hardening stage and an outstanding strength–ductility combination. Dislocation hardening was also evidenced to play a major strengthening contribution to the yield strength of AM CoCrFeNiMn high entropy alloy [7], and the sophisticated dislocation activities, including dislocation-twin and dislocation cell interactions, promote remarkable dislocation trapping and retention, rendering a steady strain hardening behavior in this alloy. In the context of the significance of primary dislocation on the deformation mechanisms, and further, the great potential of controlling these pre-existing dislocations in turn to render better properties of AM metallic materials, a basic understanding of the physical origin of these dislocations is therefore highly demanding.

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However, to date, a precise depiction of the underlying mechanism remains incompatible. Taking as an example, the dislocation network (dislocation cell) formation mechanism in AM 316L SS was interpreted from the constraint of baseplate, which promotes the plastic deformation of the building samples [8]. On the other hand, there were evidences suggesting that the dislocation patterns are associated with solute atom microsegregation, which provides a spatial framework for dislocation pinning as they propagate upon cooling [6,8,9]. In particular, it follows that the spatially periodic compositional scaffolding was also widely adopted in other AM FCC metals that revealing cell structures, despite a lack of convincing experimental evidences [10,11]. In consideration that almost all of the current AM metallic materials contain multiple compositions, it is therefore extremely difficult to separate the dislocation formation mechanism from cell solidification or any other impurity-correlated solidification processes. To this end, we here employed pure Cu as a prototype of AM FCC metals to avoid the solute redistribution effect. An interpretation of the following questions could then be more straightforwardly provided: (1) Are high-density dislocations or dislocation cell structures still present in the as-built high-purity samples? Or in other words, are dense dislocations an intrinsic feature associated with AM processing? And if yes, (2) What is the physics behind this phenomenon? A convincing answer to these two questions may help to solve the long-standing debating of dislocation origin in the AM field, and benefit a deep understanding and new thinking of the layer-wise melting advantage of AM technology.

We here fabricated nearly full-dense bulk pure Cu by laser powder-bed-fusion (L-PBF) technique. After examining its microstructures, we surprisingly found that high-density dislocations (in the order of 10^{16} m^{-2}) were present. This result suggests that high-density dislocations may be an intrinsic feature of L-PBF metals and are not associated with the previously proposed mechanisms, such as oxide or solute atom redistribution. We further developed a novel multi-physics model to provide insight into the physical origin, demonstrating that the repeated mechanical cycles rendered by local thermal heterogeneities are responsible for the residual high-density dislocations. We believe that this study can provide future directions for a rational foundation concerning the sophisticated dislocation cells and other cellular structures that widely observed in AM metals/alloys.

2. Experimental

We conducted plenty of experiments to probe the processing window for porosity-free AM Cu, using the

L-PBF printing system (S210, BLT). It was well known that the high laser-reflectivity and high thermal conductivity of Cu make it very challenging to be prepared by L-PBF [12]. Our key optimized printing parameters include a spot size of 60 μm , laser power of 230 W, scan speed of 0.5 m/s, hatch spacing of 60 μm and build layer thickness of 20 μm . A continuous scanning strategy with a rotation of 67 degrees for alternate layers was adopted in order to randomize crystal orientation and alleviate residual stress in the as-built samples [13]. The density of the as-printed Cu with dimensions of 25 mm (length) $\times$ 8 mm (width) $\times$ 8 mm (height) (Figure 1(a)) was measured to be $99.3 \pm 0.2\%$ using the Archimedes method. To reveal the fusion boundaries and voids in the samples, cross-sections of the pieces were cut, polished and etched by the ethanol solution of ferric trichloride/hydrochloric acid to enhance the image contrast for scanning electron microscopic (SEM, FEI Scios) observations. Electron backscatter diffraction (EBSD, Bruker QUANTAX), transmission Kikuchi diffraction (TKD, Bruker OPTIMUS) equipped in the SEM, and transmission electron microscopy (TEM, Thermo Fisher TALOS F200X) operated at the voltage of 200 kV were employed for microstructural analyses. Dislocation density was measured by the combination of linear-interception method from weak-beam dark-field scanning transmission electron microscopy (STEM) images and high-resolution TEM (HRTEM) images (please refer to Supplementary Materials for more details) [14], and compositional mapping was done using energy-dispersive X-ray spectroscopy (EDS) attached to the TEM. TEM/TKD samples with a final thickness of $\sim 100 \text{ nm}$ were prepared by the lift-out method using a focused ion beam (FIB, FEI Scios) system. For TKD experiments, the orientation data during scanning were collected with a pattern resolution of 800×600 pixels (2×2 binning) and an exposure time of 20 ms. The step size was set to be 30 nm and 6 nm for normal and high-resolution scan purposes, respectively. The evolution of individual powder particles during the L-PBF process was simulated using a meso-scale thermal-fluid flow model. More details of our modeling framework can be found elsewhere [15] and were described in Supplementary Materials.

3. Results and discussion

3.1. Microstructures

Figure 1(b) displays the cross section of our as-built Cu, where the melt-pools (MPs) were found to be partially overlapped without macroscopic voids at the MP boundaries. The depth and width of the typical MPs, as illustrated by the yellow region in Figure 1(b), were

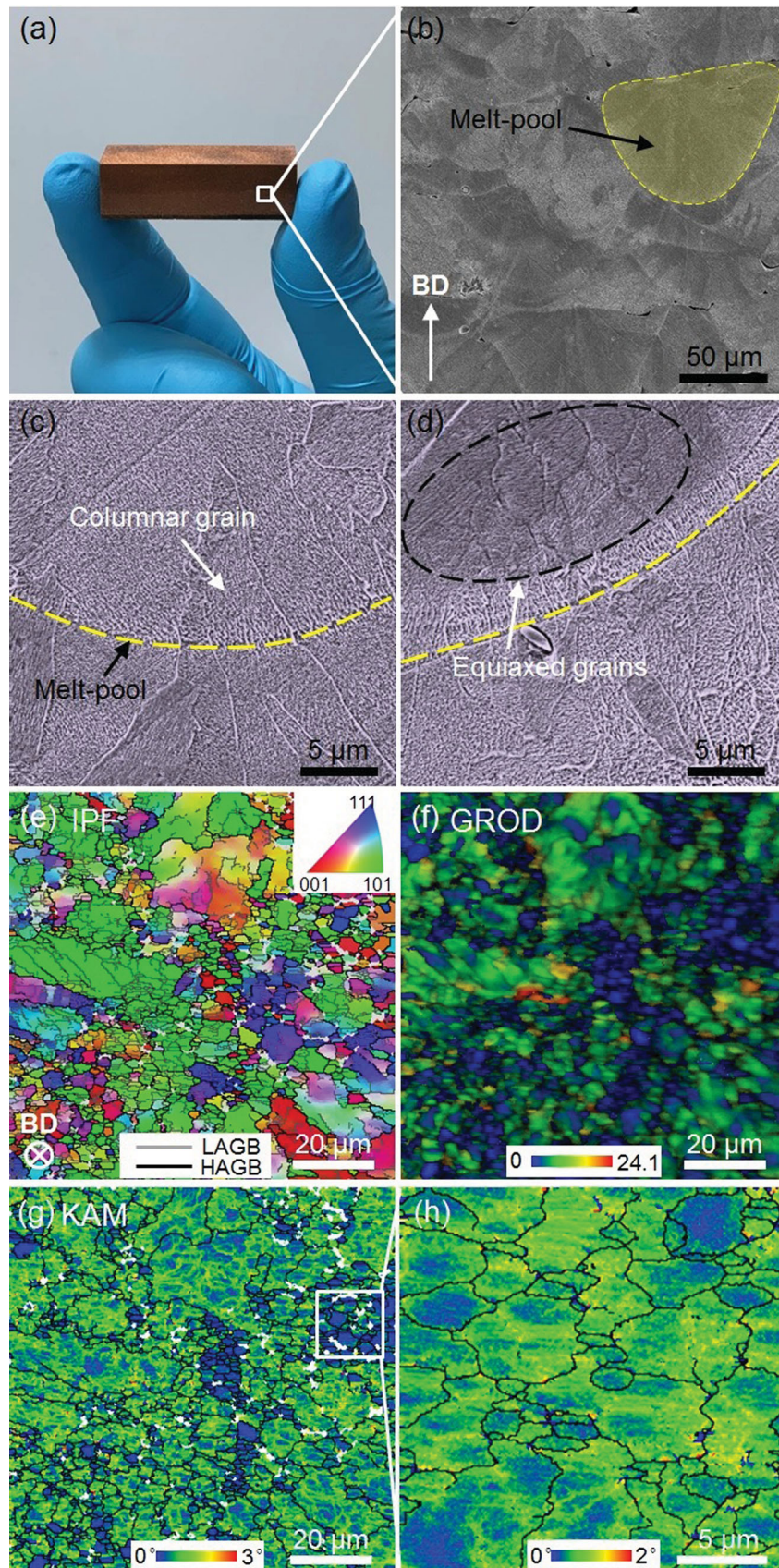


Figure 1. Microstructures of L-PBF Cu. (a) A photograph of the as-built sample. (b) A cross-sectional SEM image, showing fusion boundaries. Build direction (BD) was shown by a white arrow. A representative melt-pool was also indicated. (c,d) are higher-magnification SEM images of the regions containing melt-pools. (e–h) are in-plane EBSD maps. (e) IPF map with HAGBs (grain misorientation larger than 10°) and LAGBs (grain misorientation between 2° and 10°) superimposed. (f,g) are GROD and KAM maps, respectively. (h) KAM map of the selected fine-grained region in (g).

estimated to be 75 and 100 μm , respectively. By a closer examination of the grain structures on the etched surface, we found coarse grains grew epitaxially from the MP boundaries (Figure 1(c)), which phenomenon had been commonly observed and well elucidated in many L-PBF metals [6,7,11]. Nevertheless, equiaxed grains with much smaller grain sizes ($\sim 5\ \mu\text{m}$) were also frequently exhibited along the MP boundaries (Figure 1(d)), suggesting that the far-from-equilibrium nature of L-PBF process may impact the solidification kinetics of pure Cu. This bimodal grain size distribution was also demonstrated by the EBSD results, where small equiaxed grains with random orientations were embedded within the coarse grains (Figure 1(e)). Notably, a large fraction of low-angle grain boundaries (LAGBs, 63% of the total GBs) was observable, especially in the coarse grains, revealing a

deformation microstructure-like feature [16]. This phenomenon can be further evidenced by an in-depth EBSD analysis. Grain reference orientation deviation (GROD), a grain-based misorientation parameter based on the orientation difference between one specific point and the average orientation of the grain, is particularly useful for highlighting localized microstructural differences [17]. We employ the average orientation for a grain to be the reference point for GROD evaluation of this grain. As displayed in the GROD map of L-PBF Cu, Figure 1(f), the color variations as well as the presence of ‘hot spots’ were attributed to the high local misorientations. Moreover, the kernel average misorientation (KAM) maps were also shown to contain orientation gradient or local misorientations (as large as 1.5°) in both large and small grain regions (Figure 1(g,h)), indicative of the existence of

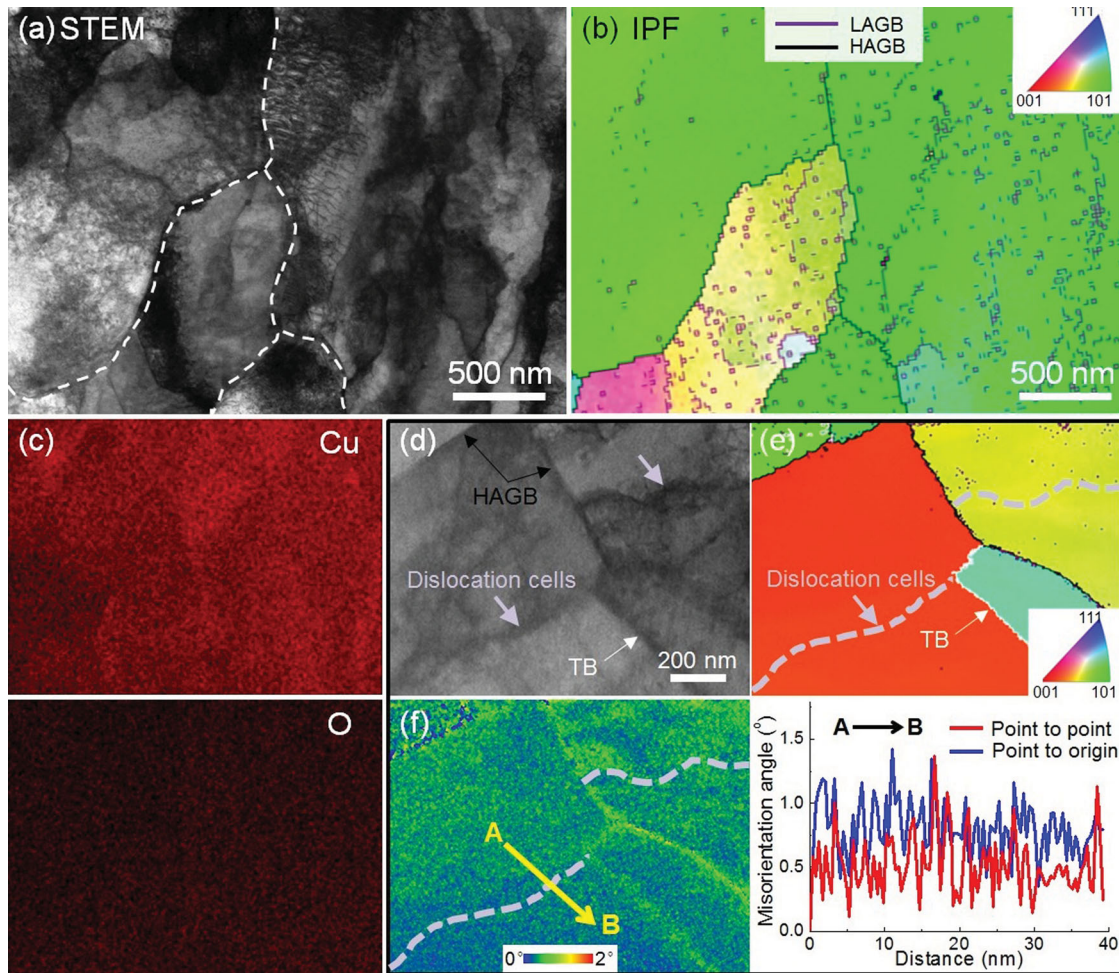


Figure 2. TEM and TKD characterizations. (a) A cross-sectional bright-field STEM (BF-STEM) image, in which grain boundaries were indicated by white dashed lines. (b) The corresponding IPF map, where HAGBs and LAGBs were superimposed. (c) Cu and O EDS maps of the region shown in (a), and the intensity fluctuation as revealed in Cu EDS map may be associated with the local variations in sample thickness. (d–f) are BF-STEM image, its corresponding IPF and KAM maps, respectively. Dislocation cell boundaries and twin boundary (TB, $\Sigma 3$) were indicated in (d,e). The right panel of (f) shows the plots of misorientation angle variations across the representative dislocation cell boundary. Misorientations were found to be concentrated at grain boundaries and twin boundaries in (f), implying the existence of GNDs to accommodate deformation incompatibility between grains/twins.

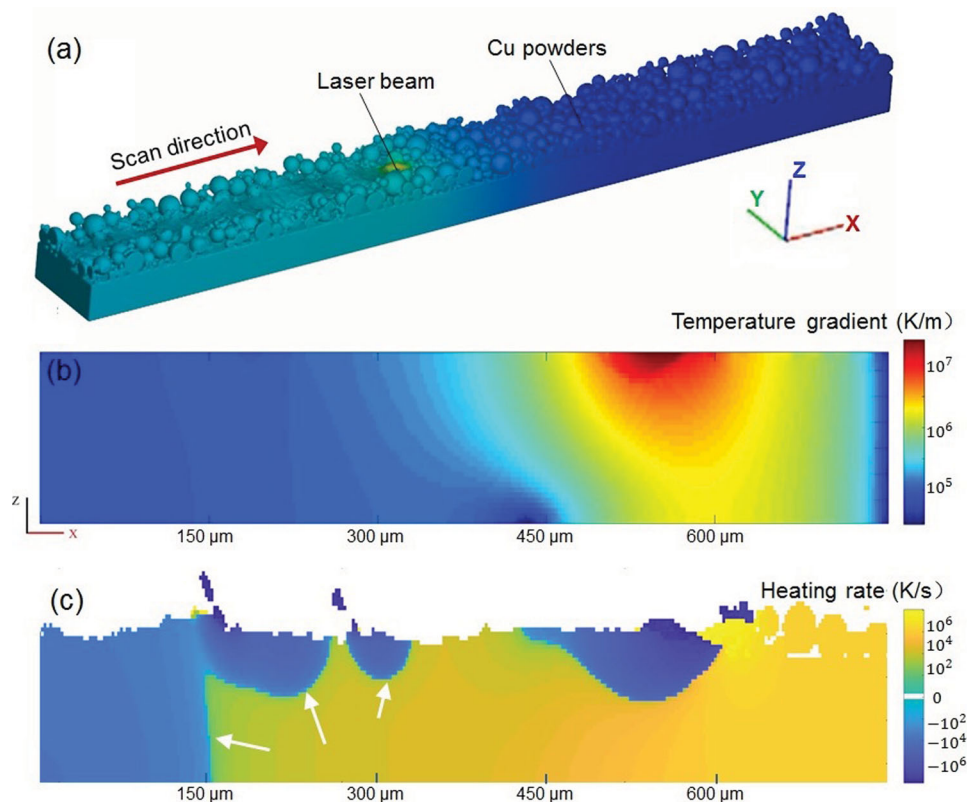


Figure 3. Multi-physics modeling of L-PBF Cu. (a) The meso-scale modeling process. (b,c) are temperature gradient and heating rate maps during laser scanning, respectively. The sharp transitions of heating/cooling regions were indicated by white arrows in (c).

geometrically necessary dislocations (GNDs) in the as-built samples [6,17]. In a word, large fractions of LAGBs and local misorientations are the generic microstructural features of our L-PBF Cu samples.

We demonstrated that these microstructural features were attributed to the presence of high-density dislocations. As exhibited by a representative STEM image (Figure 2(a)) and its corresponding inverse pole figure (IPF) map (Figure 2(b)) of as-built Cu, dislocations prevailed and were self-organized into sub-grain cells with the length scale between 200 and 500 nm. We further estimated the average dislocation density to be $\sim 10^{16} \text{ m}^{-2}$ (Text S1 in Supplementary Materials), which value is also in line with that of severely deformed metals [18]. Unexpectedly, the LAGBs were distributed in a fragmentary manner without obvious overlap with the dislocation cell boundaries (Figure 2(b)), suggestive of a specific arrangement of dislocations at these boundaries.

3.2. The origin of dense dislocations

In order to determine the governing mechanisms for dislocation formation, three possible mechanisms, i.e. cellular solidification, GNDs rendered by thermal gradient and repeated deformation of as-built parts due to thermal heterogeneities, were posited and critically examined.

We first considered the widely adopted proposal for the pre-existing dislocation cells in AM Cu, i.e. cell solidifications of impurities (e.g. oxygen) or oxide [12,19]. However, as revealed by the EDS maps (Figure 2(c)), there was no discernible oxygen segregated along the cell boundaries, implying that the cells as exhibited in Figure 2(a) are decorated solely by dislocations. Our elemental composition analysis (Table S1) also shows that few metallic impurities and oxygen were incorporated during the L-PBF process, resulting in a Cu content higher than 99.9 wt.% in the as-built samples. As there are no solute atoms or particles available for redistribution, which are critical factors for dislocation pinning and accumulations [19], the cellular solidification mechanism is unlikely to account for the observed high-density dislocations. We next focused on the nature of the dislocations and considered whether these dislocations were completely comprised of GNDs. This hypothesis is reasonable, because a remarkable attribute of the L-PBF process relies on the inhomogeneity in heating/cooling upon laser scanning, resulting in local misorientations as revealed in Figure 1(e–h). GNDs are then ready to form to accommodate the local misorientations [8]. While in contrast to this line of reasoning, our nanometer-resolution orientation mapping (Figure 2(d,e)) and line profile analysis (Figure 2(f)) showed no obvious (or

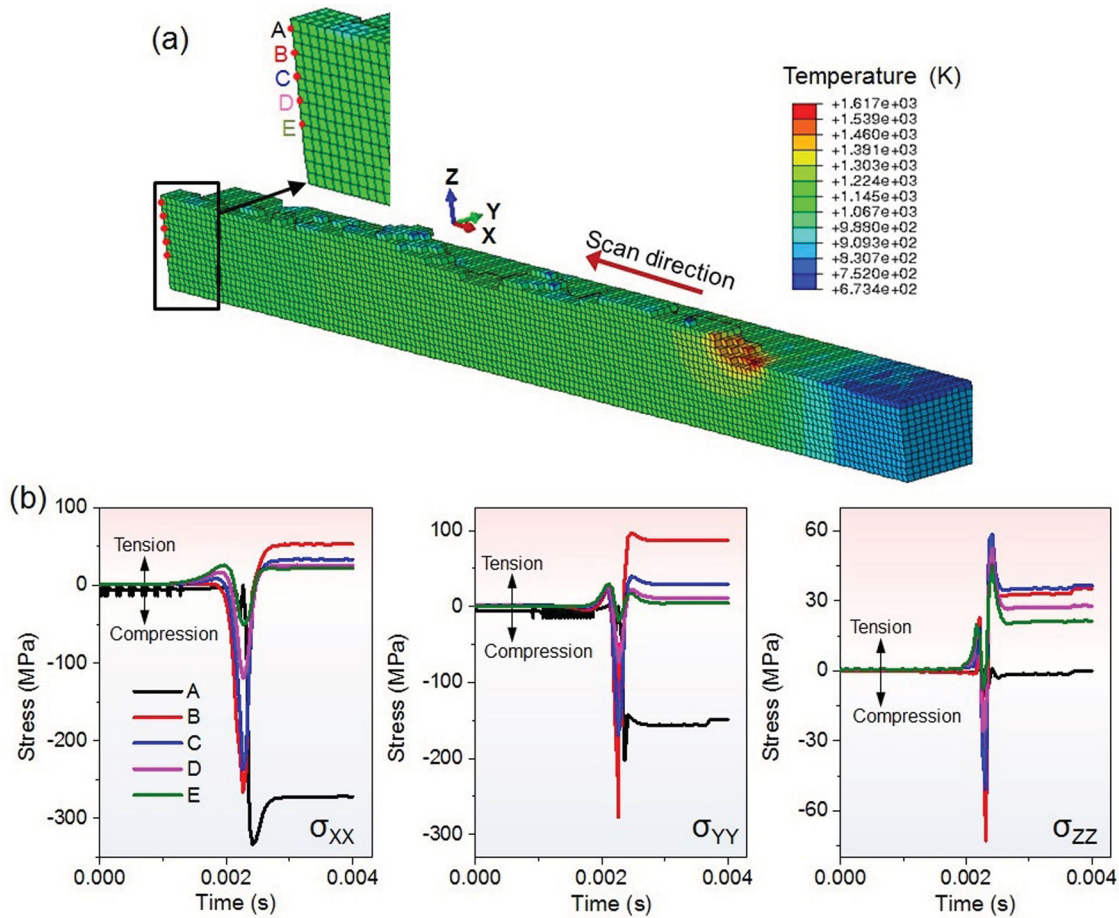


Figure 4. Detailed analyses of stress evolution during a single track. (a) Multi-physics simulation of the P-LBF process, where five points in the as-built part, with an average spacing of 10 μm , were deliberately selected for local stress analysis. (b) Stress components (σ_{XX} , σ_{YY} and σ_{ZZ}) distributions of these selected points as a function of time, showing the presence of compression-tension cycles. The other stress components revealed similar trends and were provided in Supplementary Materials (Figure S3).

undetectable) grain misorientations across the dislocation cell boundaries in spite of some concentrations at high-angle grain boundaries (HAGBs) or twin boundaries (TBs). Similarly, our previous investigations on as-built AM 316L SS also confirmed the absence of any local misorientation across the cell boundaries [6]. Due to the fact that dislocations in cell-walls take over the majority of the measured dislocations (Figure S1), these observations thus indicate that the pre-existing dislocations are not, or at least not entirely, originated from the deformation heterogeneity induced GNDs. What's more, GNDs here represent dislocations that are required to accommodate the non-uniform thermal expansion/contraction and are therefore difficult to move during sample building attributed to their 'geometrically necessary' feature [20]. In this context, it is less likely for these dislocations to glide and be self-assembled into the low-energy dislocation cell patterns as observed experimentally. We finally posited that it is the numerous thermomechanical cycles that result in the high-density dislocations. The localized heating/cooling

during the L-PBF process naturally produces expansion/contraction heterogeneities [4], leading to microscopic compression-tension actions on the as-built components. Unfortunately, significant challenges are associated with the experimental observation of this dynamic thermomechanical process, and we herein developed a novel multi-physics modeling to demonstrate the robustness of this hypothesis.

3.3. Multi-physics modeling

We employed an integrated modeling framework to model the L-PBF process of Cu (Figure 3(a)). Our simulation result on the temperature gradient in the as-built part was depicted in Figure 3(b). Temperature gradient values as high as 10^7 K/m were exhibited inside the melt-pool and in the region beneath the laser interaction domain. Such a strong temperature gradient creates local perturbations (Marangoni convections) [21], which break up the plane front in the growth of columnar grains and benefit the formation of equiaxed grain

structure as shown in Figure 1(d,e). In addition, as indicated by the white arrows in Figure 3(c), sharp variations in the heating/cooling rate were present during and after laser interaction. These intrinsic heating/cooling heterogeneities facilitate the introduction of residual stress to accommodate the localized expansion or contraction misfit, and if the residual stress is high enough to render plastic deformation, dislocations accumulate and self-assemble into the patterns as we observed experimentally (Figure 2). We further made a detailed analysis of the evolution of localized residual stress, by tracking the stress components of different positions upon laser scanning (Figure 4(a)). The results reveal that these positions suffer from dynamic loadings, that is, first compression followed by tension, as the laser scans over the reference points (Figure 4(b)). These deformation cycles arise from localized heating and cooling heterogeneities and are expected to play crucial roles in dislocation nucleation and accumulation. Note that peak stresses of both compression and tension states at different positions are high, at least tens of MPa for various stress components (Figure 4(b)). Although the peak stresses gradually attenuate for reference point that away from the sample surface, it is rational for these high residual stresses to activate plastic deformation in the as-printed, yet re-heated regions.

We, therefore, put forth to claim that the multiple compression-tension cycles that inherent to the L-PBF process are the governing mechanisms responsible for the pre-existing high-density dislocations. During the thermomechanical cycles, dislocation patterns are gradually self-organized into low-energy configurations via dislocation cross-slip/climb to minimize local strain energies. These processes are greatly facilitated by the high-temperature environment upon fabrication, rendering no or marginal (if existed) local misorientations across the dislocation cell boundaries (Figure 2(d-f)). This low-energy dislocation configuration shows similar microstructural feature with those of metals after fatigue tests [22,23], which is understandable due to their similarities in mechanical history. The heterogeneous dislocation assemblies facilitate the development of a high long-range internal stress (i.e. Type III residual stress) in the as-built Cu (Figure S2), agreeing with the observation that microscale residual stress is widely existed in the AM metals [24]. We finally note here that the observed dislocation structures should be a generic feature of L-PBF metals, unless that other deformation mechanism (e.g. phase transformation or twinning) is preferentially activated to release the residual stress. These dislocation cell boundaries may act as sinks for solute diffusion, accounting for, as least in part, the previously observed chemical segregation at dislocation cells in AM 316L SS

[5,6]. Moreover, this theory also hints at the formation mechanism of sub-grain cells in many other AM FCC metallic materials, such as Al-Si alloy and Inconel 718 alloy [25,26], suggesting that these cellular substructures might, in fact, inherit from the dislocation cell structures.

4. Conclusions

To conclude, we here employed pure Cu as a prototype to uncover the origin of dislocations that existed in many as-built L-PBF metals. High-density dislocations were unexpectedly observed in the as-built Cu. Three possible mechanisms were then posited and critically examined to determine the underlying mechanisms, and we found that these high-density dislocations were not introduced through previously proposed mechanisms, such as cell solidification or nanoparticle blockage. New multi-physics modeling was further developed to elucidate their physical origin, unraveling that the repeated compression-tension cycles rendered by the local thermal inhomogeneity are a major contribution to the residual high-density dislocations. Thus, the origin of dislocations is specific to AM processing, and may not rely on metal systems. The layer-wise melting nature coupled with the local thermomechanical control may also facilitate AM technology an indispensable new tool in tailoring microstructures of the as-built samples, thereby creating materials with properties far-exceeding their conventional counterparts.

Disclosure statement

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

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