

Site-specific Cu clustering and precipitation in laser powder-bed fusion 17–4 PH stainless steel

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ABSTRACT

Thermal gyrations inherent to metal additive manufacturing (AM) result in complex microstructural heterogeneities that are linked to the process parameters, geometry, and alloy composition. AM 17–4 precipitation hardened (PH) stainless steel displays heterogeneities due to its multi-phase microstructure where different as-built phases have been reported. These phases include austenite, martensite, and ferrite, as well as varying degrees of AM induced Cu clustering and/or precipitation. To enable a systematic study of the evolution of these strengthening phases, we utilize a non-standard concentric scan strategy to achieve variations in thermal conditions across a build. Using atom probe microscopy and thermal modeling, we show that the response of Cu varies across the build from no clustering at the outer regions to an increased tendency for Cu precipitation towards the center. Our results will enable the tuning of processing parameters to achieve homogenous Cu precipitation, and thus improve mechanical properties of AM 17–4 PH.

A major hallmark of powder bed fusion (PBF) additive manufacturing (AM) is the unprecedented microstructural variation possible. Due to the impacts related to processing parameters and geometry, variations in grain structure, phase fractions, and precipitate morphology can be seen within a single build geometry [1–4]. Progress has been made in advancing the understanding of the evolution of grain structure and texture control. However, due to the complexity of phase transformations during AM, there is still a paucity in understanding the site-specific control of microstructural features such as μm -scale secondary phases and nm-scale precipitation phenomena [5]. Explanations for the differences in complexity between grain control versus phase control can be given based on solidification conditions and the inherent thermal gyrations exhibited in AM. While site-specific grain structure control is primarily reliant on thermal gradients (G) and solidification velocities (R), the phase and precipitation evolution is dependent not only on the G and R, effecting liquid-solid transformations, but also the preceding thermal gyrations effecting solid-solid transformations [6, 7].

As a multi-phase alloy, 17–4 precipitation hardened (PH) stainless steel has shown to be particularly sensitive to changes in the processing parameters, geometry, and composition during laser-PBF (L-PBF) [4, 8–11]. The matrix microstructure in 17–4 PH has been characterized within the literature as being either mostly martensitic with small

amounts of austenite (FCC) or a mixture of austenite and ferrite (BCC) with diverse reported phase fractions [12–14]. Greater complexity is added when considering Cu precipitates. Only a few studies have reported the formation of Cu precipitates in as-built conditions [15,16]. Numerous other studies, in contrast, report the need for an aging step to form Cu precipitates [12,17–19]. Naturally, Cu clustering and precipitation are strongly dependent on the thermal conditions. Slower cooling rates will provide more time for Cu to diffuse, form clusters, and precipitate. As Cu precipitates are the main strengthening phase in 17–4 PH, it is important to understand the thermal histories that lead to their formation [20]. To build a better understanding, the current work utilizes a non-standard concentric scan strategy that elicits a variety of thermal signatures. This enables a systematic study of the precipitation response of Cu in L-PBF 17–4 PH. The experimental results are reasoned through thermal simulations eliciting an understanding between how the local thermal history influences the behavior of Cu. A framework is presented for using the concentric scan strategy as a method for studying precipitate evolution in other alloys due to its wide variation in thermal history.

17–4 PH samples were produced using powder provided by 3D Systems with the composition given in Table 1. Builds were printed using a 3D System ProX 300 L-PBF machine. An inward travelling concentric scan strategy was utilized as outlined by Moyle et al. [4]. The

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processing parameters are as follows: laser power of 127.5 W, scanning speed of 1200 mm/s, layer height of 40 μm , laser diameter of 100 μm , and hatch spacing of 50 μm . Builds were printed in an N_2 atmosphere. Samples had a geometry of $10 \times 10 \times 15 \text{ mm}^3$. Simulation of the process was performed using OpenFOAM to obtain information on the local thermal history in the concentric scan strategy. More details on the theory behind the simulation are given in [21]. Parameters used in the current simulation are given in the supplementary file. Calibration of the simulation was done through comparison to melt pool geometries revealed through light optical microscopy (LOM) of etched samples.

For characterization, samples were sectioned and ground using SiC papers up to 4000 grit before being polished with 3 μm and 1 μm diamond suspensions. Following mechanical preparation, samples were electro-polished using Struers A2 electrolyte for 20 s at 25 V. Electron back scattered diffraction (EBSD) was performed on a JEOL 7001f scanning electron microscope equipped with a Hikari-EDAX camera. EBSD data was captured using 20 kV acceleration voltage, 13 nA probe current, and 8×8 binning. TSL OIM 8.0 was used for EBSD data analyses.

Atom probe microscopy (APM) tips were extracted from a comb-shaped cross-sectional sample taken from a cut made at the center region, perpendicular to the build direction. Five tips were pre-prepared with their locations shown through LOM in Fig. 1a. The tips were sharpened using a focused ion beam technique as described by Rielli et al. [22] in a ThermoFisher Helios G4 Xe PFIB. A straight flight CAMECA LEAP 4000 atom probe in laser-assisted mode with 57 % detector efficiency was used with 50 pJ laser energy, 200 kHz pulse rate, and 0.5 % detection rate at a temperature of 50 K. Data analysis was performed in the CAMECA AP Suite 6.1 software. Reconstructions were performed via tip profile from scanning electron microscopy images of the tips.

The results from EBSD analyses are overlayed on the LOM image in Fig. 1 and show a multiphase response with the sample experiencing a mostly FCC microstructure at the center and a mostly BCC structure at the outer region. Greater details on the evolution of these phases has been discussed elsewhere [4,11,23]. In general, the different phases can be linked to the changing thermal conditions that occur due to the laser's motion. Higher cooling rates suppress formation of FCC in the outer regions. The laser's faster concentric motion towards the center of the build results in greater reheating and decreased cooling rates causing transformation of BCC to FCC.

Atom maps for tips 1 and 3 are shown in Fig. 2a and b respectively for Cu, Fe, Ni, Cr, Nb, Mn, NbN, and C and reveal the general trends in their distributions. Further, one dimensional (1D) concentration plots are provided in Fig. 2c for Tip 3. Tip 2, 4, and 5 atom maps are given in the supplementary file (Figure S1). In these maps, NbN instead of N is shown due to the overlapping of N peaks with Si. As the Nb atom maps do not correlate to the NbN maps, and the NbN shows the expected segregation behavior of N, the NbN maps can be used for qualitative analysis of the segregation of N. Overall, Tip 1 contains no identifiable segregation in its atom map. Tip 3 contains an interface based on the larger amount of C and NbN towards the top of the tip. The upper region is identified as FCC due to its higher solubility for C and NbN [14,24]. The lower region is depleted in C and NbN and enriched in Nb, and hence, is identified as the BCC phase. Further, as will be shown later, the lack of Cu precipitates within the upper region of the tip further confirms the formation of FCC based on previous literature [15,17,19]. No obvious difference in the atomic distributions of Fe, Ni, and Cr can be seen in the atom maps. However, the concentration maps do reveal slight variations in Ni and Fe

around the interface of the FCC and BCC regions. Further, a few striated lines of NbN can be seen in the BCC phase that are likely enrichments along dislocations. The other tips, not shown here, show mostly non-partitioned distributions of elements in their atom maps similar to tip 1.

Bulk compositional analyses for Tips 1 and 3 are provided in Table 2. When comparing Tip 1 and Tip 3, Tip 3 shows a significantly higher content of Ni and qualitatively less NbN. Considering the differences between the FCC and BCC regions of Tip 3, the FCC region shows higher amounts of NbN and C, and less Nb. This is in line with expectations that partitioning of interstitials primarily occurs under high cooling rates while other elemental partitioning is limited [25]. Overall Tips 2, 4, and 5 show similar trends to Tip 1, and are provided in the supplementary file (Table S3).

The calculated cooling rates for each atom probe tip location are presented in Fig. 3 as violin plots showing the kernel density estimated distribution of the underlying data. Wider regions in the vertical axis represent a greater occurrence of any given cooling rate. Fig. 3 demonstrates that the concentric scan strategy results in different thermal histories between the outer and inner regions of the build. The outer region has maximum cooling rates greater than 10^6 K/s . The center only sees cooling rates slightly above 10^5 K/s . The inner region also sees a broader distribution of cooling rates between 10^1 and 10^3 K/s , while the cooling rates at the outer regions are more likely to be at the extremes. An explanation is that, due to faster initial cooling rates at the outer regions, these regions reach lower temperatures faster. These lower temperatures will be associated with a higher number of lower cooling rates as the heat flux within the build decreases. Overall, the simulations demonstrate that a variety of thermal histories exist within the concentrically scanned build. This is expected to affect the Cu precipitation response.

1st nearest neighbor (1st-NN) distributions in Fig. 4 reveal the presence of Cu clustering in each tip. For comparison, a fully randomized distribution of Cu atoms is given in gray. Only tips 3 and 4 show any significant amount of clustering with a noticeable shift in their d-pair distributions to lower d-pair values when compared to the randomized distribution. These shifts in the distributions are an indication that the average distance between Cu atoms has decreased. Comparing tips 3 and 4 to work by Moyle et al. [15], who printed samples using a more traditional raster scan strategy, the peak count for the tips presented in this work show lower d-spacing at around 0.3 nm compared to 0.4 nm respectively. In tip 1, 2, and 5, the Cu 1st-NN mostly aligns with the random distribution, indicating a lack of any major Cu clustering. The lack of Cu clustering can be attributed to the higher cooling rates present towards the outer regions of the build. Cooling was fast enough such that there was minimal Cu diffusion. Analysis of tip 3 in the FCC region, given in the supplementary file (Figure S2), shows no clustering despite similar Cu concentrations to that of the BCC region. The lack of Cu clustering within FCC is related to the lower diffusion rate in FCC compared to BCC [26] and Cu's higher solubility in the FCC matrix.

The formation of Cu precipitates is desirable as it will increase the strength of 17-4 PH through Orowan mechanisms [14,27,28]. Research has shown that peak strength (without the consideration of Cr-rich precipitates) is achieved at a radius around 0.8 to 1.5 nm and number densities of 1.2×10^{24} to $8.0 \times 10^{24} \text{ m}^{-3}$, respectively [29-31]. Information on the radius and number density of Cu precipitates were calculated from 10 at. % Cu isoconcentration maps obtained from the APM data [30] and are presented in Fig. 5a. Only tips 3 and 4 showed the formation of Cu precipitates in the isoconcentration maps. Tips 3 and 4

Table 1

Composition of 17-4 PH stainless steel powder measured from inductively coupled plasma mass spectrometry (ICP-MS).

Element	Fe	Cr	Ni	Cu	Mn	Si	Nb	Mo	N	C
Concentration (wt. %)	Bal.	16.10	4.34	3.81	0.87	0.71	0.32	0.13	0.10	0.03
Concentration (at. %)	Bal.	17.04	4.07	3.30	0.87	1.39	0.19	0.07	0.39	0.14

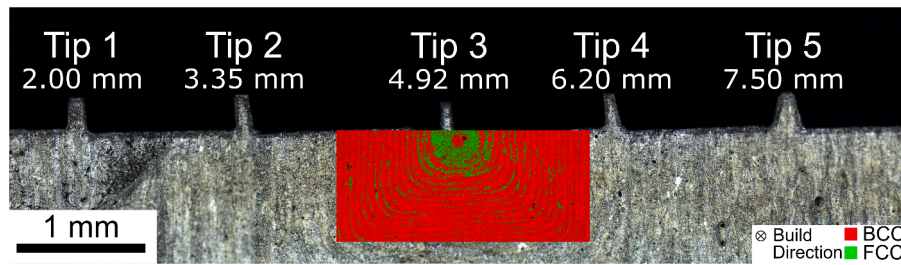


Fig. 1. LOM of the sample and tips to be extracted for APM. Distances calculated from the left edge of the build. Overlaid is an EBSD of the center of the sample showing a mostly austenite (FCC) center and a ferrite (BCC) outer region. The LOM and EBSD image are of the same scale.

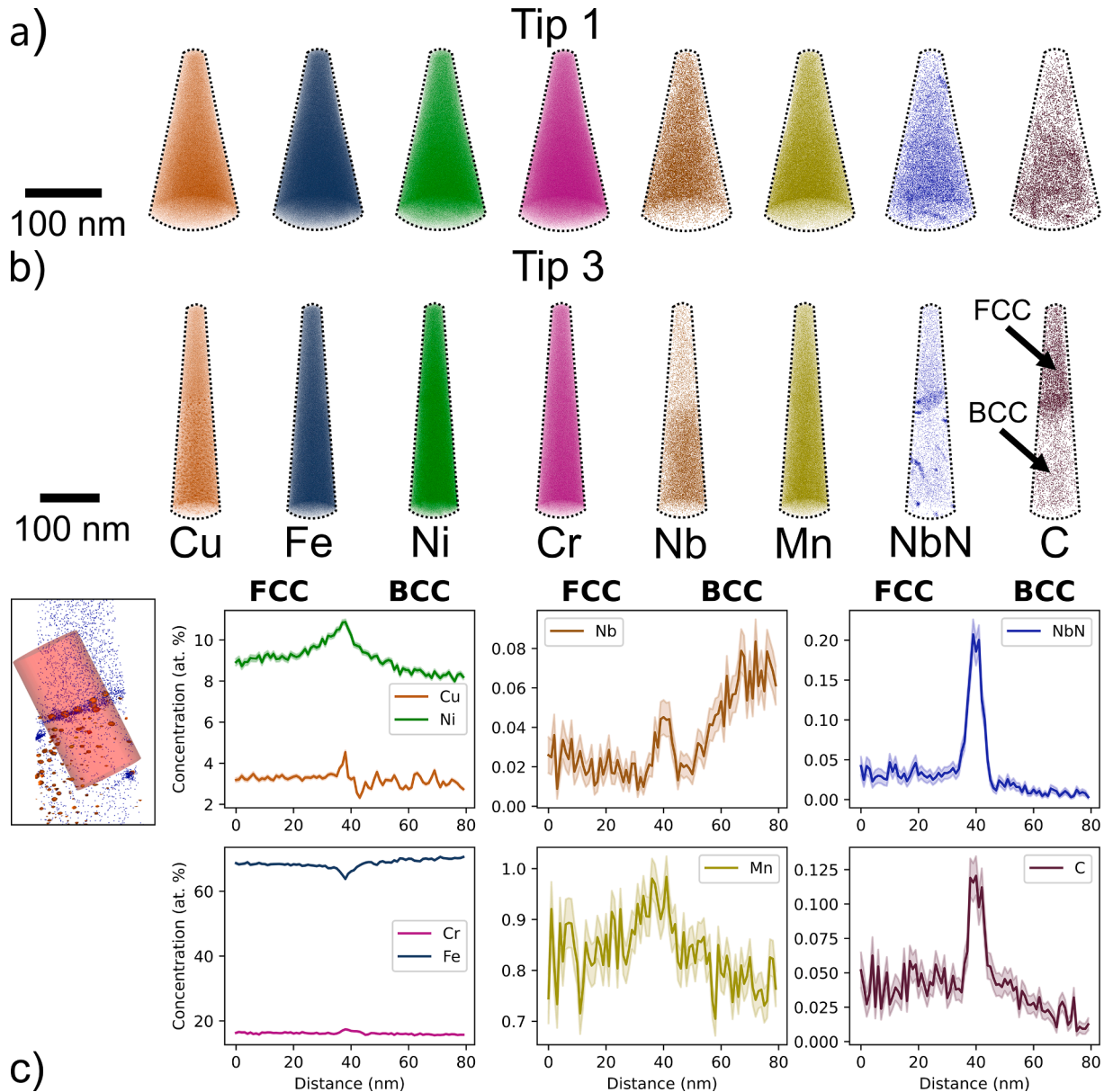


Fig. 2. a) Atom maps from tip 1. b) Atom maps from tip 3. Black arrows point towards characteristic phase regions within tip 3. c) 1d concentration plots from tip 3 intersecting the interface between the FCC and BCC phase.

have an average Cu precipitate radii of 4.29, and 1.71 nm and number densities of 4.67×10^{23} and $1.65 \times 10^{24} \text{ m}^{-3}$ respectively. For peak strengthening in 17-4 PH, the size and number density of the Cu-precipitates for tip 3 are larger and lower respectively when compared to the ranges quoted from literature [29–31] while tip 4 sees

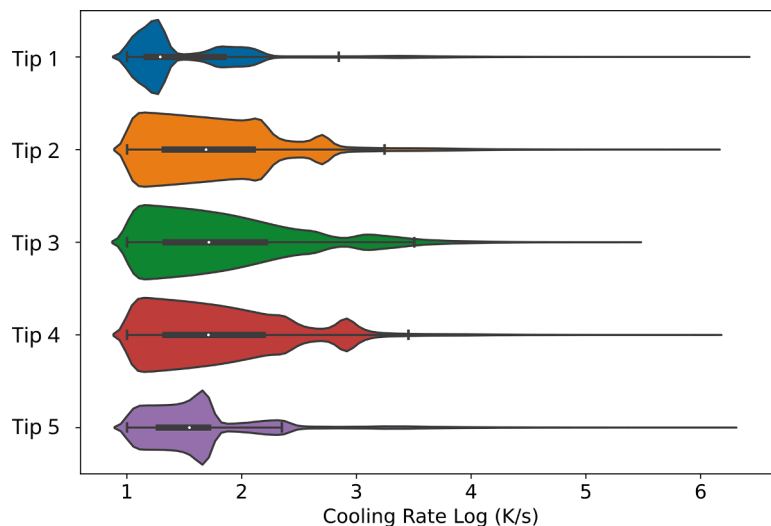
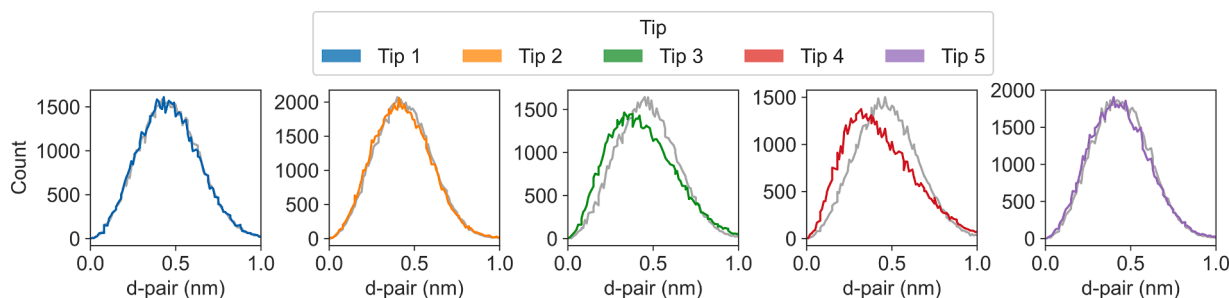
larger sizes but a number density within the quoted ranges.

Overall, the formation of larger Cu precipitates in tip 3 is in line with the lower cooling rates, thus, indicating greater aging based on cooling rates presented in Fig. 3. Interesting, however, is the difference in Cu precipitate formation between tip 2 and 4. These tips are similar

Table 2

Bulk Composition of APM tips 1 and 3 in at. %.

	Cu	Fe	Ni	Cr	Nb	Mn	NbN	C
Tip 1	3.24	71.90±0.01	4.90	17.10	0.08	0.73	0.13	0.02
Tip 3 – FCC (Top)	3.25 ±0.01	68.85±0.04	8.72 ±0.02	16.26±0.03	0.03	0.80	0.06	0.05
Tip 3 – BCC (Bottom)	3.28 ±0.01	69.49±0.07	8.35 ±0.02	15.96±0.03	0.07	0.79	0.03	0.01

**Fig. 3.** Violin plots showing the distribution of cooling rates experienced within the 17-4 PH build at the positions where the APM tips were extracted.**Fig. 4.** 1st nearest neighbor distributions for the individual APM datasets. The gray lines show the distribution for the randomized datasets.

distances from the center of the sample with tip 2 taken ~1.2 mm and tip 4 taken ~1.6 mm from the center. They also have similar bulk compositions (Table S3). Their differences in Cu precipitation response can either be linked to thermal conditions or broader compositional variations not captured by APM. While they have similar maximum cooling rates, there are minute differences in their thermal signatures, with tip 4 seeing a broader range of cooling rates that may be encouraging Cu precipitate formation. However, the formation of Cu precipitates in L-PBF 17-4 PH may also be sensitive to broader compositional differences. Studies have shown in L-PBF that due to solidification conditions, variations in elemental distribution occur between the interdendritic and dendritic regions [13,32]. As the volume probed by APM results is small (largest volume presented here is $0.724 \mu\text{m}^3$ from tip 3), heterogeneities in microstructure may not be present in APM. Lastly, it was ruled out that local FCC may be inhibiting the Cu precipitation due to its higher Cu solubility based on Cu precipitation behavior seen in tip 3 (Fig. 5a). Here FCC has a significantly higher volume phase fraction, yet Cu precipitates still form in the neighboring BCC.

Local thermal conditions may influence the composition of precipitates [33]. Therefore, analyses of the Cu precipitates in tips 3 and 4

were done through proxigrams (Fig. 5b) using the Cu isoconcentration surfaces shown in Fig. 5a. The proxigrams show the concentration of elements within these Cu precipitates for tips 3 and 4. Both tips show similar trends in major elements such as Fe, Cr, Cu, and Ni. Fe is noted to decrease to 20 at. % while Cu increases up to 60 at. %. Some minor elements, however, show significant differences between the tips. The concentrations of NbN and Mn are notably higher in tip 4 compared to tip 3. It may be convenient to identify the FCC in tip 3 as a major driver for the difference in composition in the Cu precipitates due to localized depletion of elements between the FCC and BCC phase. However, it was noted in the APM maps (Fig. 2) that while NbN does show strong partitioning to FCC, Mn does not favor one phase over the other. Hence, another factor may be contributing to the depletion of Mn in the Cu precipitates in tip 3 such as the thermal history. When comparing the proxigrams here to proxigrams for conventionally processed 17-4 PH after a heat treatment at 450 °C for 8 hr, results presented here for tips 3 and 4 show Cu precipitates that are richer in Cu, and, for tip 3, leaner in Mn [31].

Overall, this study shows the sensitivity of Cu precipitation to thermal conditions within L-PBF. This work demonstrated these changes

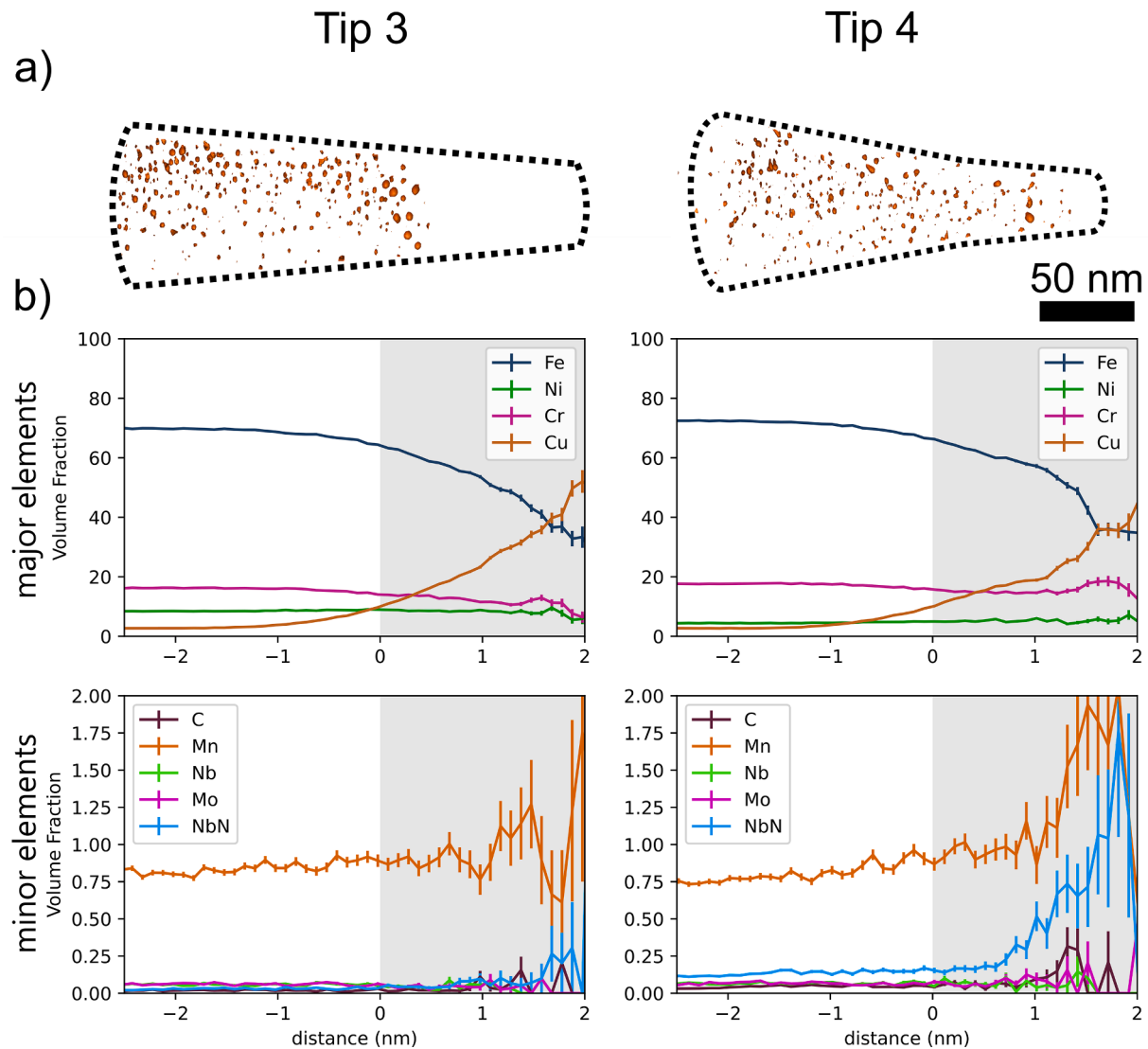


Fig. 5. a) 10 at. % Cu isoconcentration surfac es in tips 3 and 4 showing Cu precipitates. b) Proxigrams showing the concentration of elements along the radius of the Cu precipitates shown in a).

through the use of a concentric scan strategy offering a unique way to study the impact of a wide range of thermal histories on the microstructure and precipitation response. Arguably, a similar concentric scan strategy could be applied to other alloys to reveal the interplay between L-PBF thermal conditions on the microstructure evolution. For 17–4 PH, as Cu precipitation is the main strengthening mechanism, the capability to tune the L-PBF processing parameters to obtain optimal Cu precipitation size and number density in the as-built condition is of high interest. From the results presented here, this would involve a scan strategy that decreases the cooling rate. Such candidates, based on previous literature, include examples such as shorter raster scan lengths to reheat local areas quickly and repeatedly [34] or the utilization of more unique computationally calculated scan strategies designed at maintaining consistent lower cooling rates [35,36].

In summary, we applied a special concentric scan strategy and revealed increasing Cu clustering and precipitation from the outside towards the center of a L-PBF 17–4 PH build. This was attributed to a decrease in cooling rate towards the center of the build. Ultimately, the laser's faster concentric motion towards the center of the build reheats this region more rapidly and slows down cooling compared to the outer regions. Further, we showed site-specific evolution of these Cu precipitates. Cu precipitates at the center of the build (tip 3) had larger radii

and were depleted in N and Mn compared to those Cu precipitates away from the center of the build (tip 4). Overall, this work paves the way towards optimization of processing parameters to encourage the formation of Cu precipitates in the as-built state of 17–4 PH to achieve superior strength without post-AM thermal treatments.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.scriptamat.2023.115891](https://doi.org/10.1016/j.scriptamat.2023.115891).

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