



Thermal cycle induced solid-state phase evolution in IN718 during additive manufacturing: A physical simulation study

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ABSTRACT

Additive manufacturing (AM) is well-known for its capability to produce and repair intricate parts e.g., Ni-based superalloy components used in aero-engine applications. However, a comprehensive understanding of the microstructure evolution and its link to properties remains elusive, with gaps in the through-process evolution of γ grains, γ' , γ'' , δ , and other phases in response to thermal cycling. The outstanding challenge here is to observe these transformations dynamically during the 3D printing process, as this usually requires complex in-situ measurements. Here we apply a different approach to systematically reveal the impact of thermal cycling on the solid-state microstructure evolution of IN718, via a combination of physical simulations and thermo-kinetic modelling.

We replicate the thermal cycles IN718 experiences during laser directed energy deposition. Solid-state thermal cycles are shown to have minor effects on the grain morphology due to short holding times at critical temperatures, however, they do induce plastic strain accumulation. Significantly, we show how thermal cycling influences the evolution of γ' , γ'' , and δ phases. While high initial peak temperatures inhibit the precipitation of γ' , γ'' , and δ phases, prolonged thermal cycling to gradually decreasing peak temperatures promotes their precipitation. The δ phase forms along grain and twin boundaries, while both γ' and γ'' predominantly precipitate in Nb-enriched regions, causing heterogeneities in hardness. Our results underpin the suitability of physical simulations for replicating superalloy AM, highlighting its potential as a tool to advance the current limited understanding of the microstructure and property evolution.

1. Introduction

Additive manufacturing (AM) is an increasingly popular manufacturing route for making 3D components in a layer-by-layer manner, using powder, wire, or other types of feedstocks [1–3]. It enables the fabrication of complex geometries with short lead times, high efficiency, and minimal waste. Laser directed energy deposition (L-DED) in particular is a metal AM technique that has seen rapid growth over the past decade, with widespread applications across various industries due to its ability to fabricate larger components than powder bed fusion techniques [4–9]. Additionally, ability of L-DED to add to existing components and its suitability for repair make it an economically preferable choice [10,11]. IN718 is a Ni-based superalloy commonly used for manufacturing engineering parts such as gas turbines for aircrafts

and power generation [12,13]. Consequently, DED repairs using IN718 for components such as turbine blisks are of significant industrial interest [14,15]. The popularity of IN718 can be attributed to its excellent weldability and manufacturability, combined with its exceptional properties, including high strength, corrosion resistance, and remarkable microstructural stability at high temperatures [16]. The as-printed IN718 microstructure consists of fine dendrites due to micro-segregation of solute elements, and solidification phases such as Laves and carbides [17,18]. In addition, it may comprise nano-scale, ordered intermetallic phases of γ' - (L_{12} Ni₃(Al, Ti)) and γ'' - (DO_{22} Ni₃(Nb)) as well as the μ m-scale orthorhombic δ Ni₃(Nb,Ti) (DO_a) phase [19,20]. These phases form during solid-state phase transformations, and their presence and morphology determine the mechanical properties of the component.

Despite the increasing enthusiasm by the aerospace industry in L-

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DED for making engineering parts from IN718 and other high-performance alloys, many challenges remain. This slows down the more widespread applications of metal AM across new industries. This is related to the perhaps greatest challenge in AM, i.e., the complex as-built microstructure of AM components, which is vastly different to their traditionally processed (cast and wrought) counterparts. For instance, in casting, the continuous slow cooling causes the precipitation of desirable γ' , γ'' , and δ phases [21]. However, in L-DED, it is difficult to achieve γ' , γ'' , and δ precipitation during printing, due to its inherent complex thermal profile [21]. L-DED, like other metal AM processes, involves multiple thermal cycles with high heating and cooling rates, starting with the melting and solidification of the first layer on a substrate. The additional heat generated by the laser during the deposition of the subsequent layers may cause partial re-melting and re-heating of already deposited layers. The previous layers experience additional short heat treatments, and this may lead to solid-state phase transformations and recrystallisation phenomena to occur. The same sequence of events is repeated for multiple cycles until the final layer has been deposited. Hence, individual layers experience multiple additional thermal cycles after solidification, and this has been shown to determine the local solid-state microstructure evolution of each layer or material voxel [22].

Several studies report heterogeneities in the distribution and morphology of solid-state phase transformation products throughout builds. Li et al. [23] and Tian et al. [24] both reported a lower volume fractions of γ' , γ'' , and δ at the top compared to the bottom of their builds. This affected the resulting mechanical properties such as hardness and tensile strength and was attributed to local variations in thermal conditions across the build. Conversely, other studies report no precipitation due to less intense thermal cycling experienced [25,26]. Based on these findings and as recently reviewed [27], it can be concluded that solid-state phase transformations are determined by thermal cycling. However, the current understanding of the through-process microstructure evolution in AM builds as a function of process parameters is lagging. Advancements in this area are critical to enable AM of high-performance alloys with optimized and reproducible properties.

To better understand how thermal cycling is linked to the microstructure evolution in AM builds, complex in-situ measurements are usually required. While post-AM materials characterization such as transmission electron microscopy of various layers offer valuable insights [28], they are not sufficient to completely elucidate various stages of the microstructural evolution. Generally, in-situ studies enable the observation of microstructural changes as they occur during fabrication. However, performing such experiments requires significant resources in terms of time, effort, and cost [29]. For instance, monitoring the printing process in real-time requires the use of sophisticated and expensive tools such as high-resolution infrared cameras, high-speed X-ray imaging, synchrotron X-ray, or neutron diffraction techniques [30–34]. More straightforward approaches to monitor the thermal history during AM have their own challenges. For instance, the thermocouples typically used for monitoring the temperature have their own limitations and often fail to provide accurate measurements. This is because they are typically attached to the substrate and not the build, and because they provide accurate readings only across certain temperature ranges [24, 35].

An alternative approach to tackle this challenge is to simulate AM processes in physical simulators instead of actual 3D printers. This enables systematic studies of the effect of AM thermal cycling, independently of other variables. An important example is the work by Mantri et al. [36], where laser surface engineering was utilized to mimic the microstructural evolution during L-DED of IN718. Physical simulation techniques such as Gleeble thermo-mechanical simulations and quench dilatometry are promising alternatives to reproduce the thermal history of the complex AM process [37]. These techniques have been demonstrated to be able to replicate processing conditions that voxels of AM builds are subjected to during printing on somewhat larger volumes, which is beneficial for subsequent materials characterization and

testing, and in a more controllable manner [31,38]. In particular, the Gleeble's fast resistivity heating and water cooling capabilities coupled with precise thermal history control have been recently harnessed to replicate thermo-mechanical cycles experienced by the Ti-6Al-4 V alloy by Kumar et al. [31]. Furthermore, unlike the actual manufacturing process, this enables not only systematic parameter studies but also quenching of selected conditions throughout the thermal profile for ex-situ characterization and testing.

The opportunities and limitations of AM thermal cycling using a Gleeble have so far been explored and validated in the Ti-6Al-4 V alloy only. An example is the work of Xiao et al. [38], where a Gleeble 3500 was utilized to simulate four different AM thermal cycles to investigate the phase evolution. Kumar et al. [31] utilized the Gleeble to explore the impact of thermomechanical gyrations on the phase transformation response and flow behavior. However, there does not seem to be any research available on the use of Gleeble to replicate AM thermal cycles for Ni-based superalloys. The only available relevant literature focuses on utilizing the Gleeble to reproduce welding thermal cycles [39]. Through these tests, significant variations were observed after the Ni-17Mo-7Cr superalloy was subjected to typical welding thermal cycles, providing insights into the microstructure evolution in the heat affected zone.

Therefore, the current study aims to extend the Gleeble physical simulation strategy to replicate the thermal history of select layers of IN718 during the L-DED process. This enables us to achieve an advanced understanding of the relationship between AM thermal cycles and the microstructure evolution of superalloys, as will be revealed by various microscopy techniques in the following. Physical Gleeble simulations are supported by MatCalc through-process thermokinetic modeling to comprehensively uncover how the thermal cycles affect the solid-state phase evolution. Given the dynamic nature of these transformations, thermokinetic modeling offers another alternative to in-situ observations. Our results demonstrate that the combined Gleeble and MatCalc simulation strategy is indeed suitable for replicating superalloy AM to systematically advance the current understanding of the microstructure evolution.

2. Materials and methods

2.1. Material and sample fabrication

The material used for this investigation was wrought IN718 with a chemical composition of Ni-18.5Fe-18.0Cr-5.3 Nb-3.0Mo-0.95Ti-0.5Al-0.02 C-0.003B (wt%). We opted for a homogeneous, wrought starting material to limit the parameter space, by avoiding the additional complexity related to the inherent heterogeneity of 3D printed starting materials. The ingot was produced by the typical triple melting route for aerospace grade IN718 (vacuum induction melting, electroslag remelting, and vacuum arc re-melting), followed by homogenization, ingot breakdown, and radial forging. The resulting billet was cut into slices with a radius of 50 mm and a height of 200 mm. Dog-bone shaped samples for the Gleeble (Fig. 1a) specifically designed to ensure fast and uniform heating and cooling in the region of interest (smaller diameter gauge length) were machined from this billet slice. [Supplementary Fig. S1](#) shows the starting microstructure containing metal carbides and nitrides, with a volume fraction and size range (diameter) of about 0.7 % and 1–4 μm , respectively. There are also δ phase precipitates decorating the grain boundaries, and γ' and γ'' precipitates inside of the grains.

2.2. Gleeble thermal cycle simulation setup

The cyclic heating and cooling experiments were performed in a Gleeble 3500-GTC thermo-mechanical simulator (Dynamic Systems Inc.), as depicted in Fig. 1b. Copper grips were used to hold the samples in place, and resistance heating was employed to provide the necessary heating. The samples were cooled via a water-cooling system that

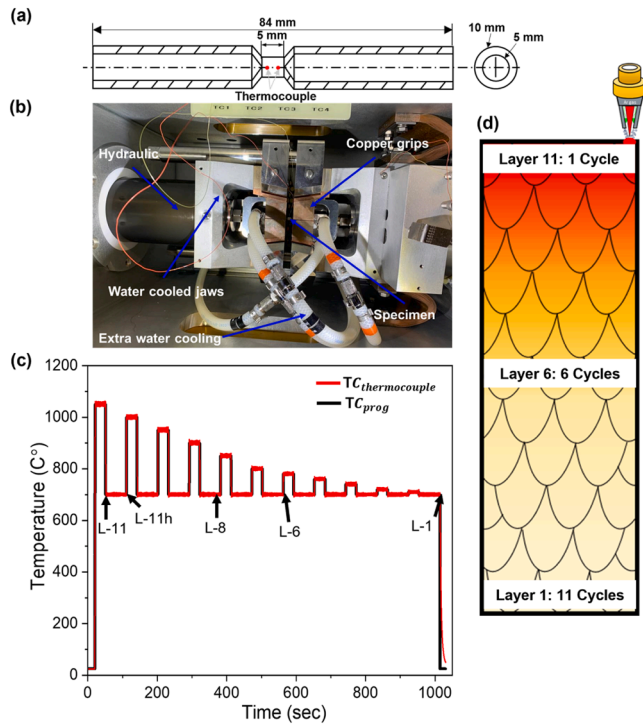


Fig. 1. (a) Schematic and dimensions of the sample used for the Gleeble tests. Two red dots indicate the regions where thermocouples were attached. (b) Test chamber of the Gleeble 3500 used for this research. (c) Targeted (black) versus actual (red) Gleeble thermal cycles. (d) Schematic of a L-DED build as simulated in c.

pumped water through the copper jaws. Additional cooling was provided simultaneously through high-speed water flow in the hollow cylinder design. Throughout the cyclic heating and cooling process, the chamber was flooded with Ar gas to prevent oxidation. To monitor the temperature readings, in-situ, K-type thermocouples were spot-welded to the center of each sample. Heating and cooling rates of 5000 °C/sec and 300 °C/sec were applied, respectively. These rates are close to the limits achievable with this set-up, and are within the range of heating and cooling rates used in actual L-DED processes, which is 100–6000 °C/sec [40,41].

To simulate the thermal cycle of a selected layer or voxel, repeated quenching and heating was conducted at progressively decreasing temperatures. The targeted (TC_{prog}) and actual simulated thermal cycles ($TC_{thermocouple}$) are presented in Fig. 1c. The strong correlation between TC_{prog} and $TC_{thermocouple}$ confirms the Gleeble's ability to effectively replicate the thermal cycles of AM. Fig. 1d shows a representation of the simulated layers on a L-DED build. The samples were held for 30 s after heating to the peak temperatures and 60 s after quenching to the lower temperature of 700 °C. The simulated thermal cycles represent the first 11 layers of a L-DED process. Five samples were quenched at different stages of the process (as shown in Fig. 1c) to systematically observe the microstructural changes during thermal cycling. Table 1 provides

Table 1
Sample designation and the characterization techniques used.

Sample name	Layer number	Number of thermal cycles	SEM/EBSD/ Hardness	APM
As-received	–	–	✓	✓
L-11	11	1	✓	✓
L-11 h	11 (hold)	1	✓	–
L-8	8	4	✓	–
L-6	6	6	✓	–
L-1	1	11	✓	✓

information on the sample designation and the various characterization techniques applied to each sample. L-11 h was included to investigate the microstructure difference after holding L-11 at 700 °C for 60 s. The thermal cycles were chosen based on a literature survey for L-DED with short interlayer dwell time [21]. The quenching temperature of 700 °C reflects the average temperature of the deposited material. This temperature results from heat accumulation from previous layers that have insufficient time to cool down. Tian et al. [24] observed comparable thermal cycles in the first ten layers of their L-DED IN718 build.

2.3. Microstructure characterization

The regions of interest of the Gleeble samples (5 mm in length) were sectioned, ground, and polished using standard metallography preparation methods. Backscattered electron (BSE) imaging and energy dispersive spectroscopy (EDS) were conducted using a JEOL 7001 F scanning electron microscope (SEM) with an accelerating voltage of 15 kV and a probe current of 13 nA. Further SEM microstructure analyses were performed to reveal nano-scale phases after electropolishing at 25 V for 30 s using the Struers A2 electrolyte. For electron backscatter diffraction (EBSD), an EDAX 'Hikari Super' system installed on the JEOL 7001 F SEM was utilized. EBSD data were acquired with an accelerating voltage of 20 kV, a probe current of 13 nA, a 6×6 binning, and a step size of 1 μ m. The acquired data were analyzed using the TSL OIM 8.0 software. A grain dilation clean-up was performed with a minimum grain size of 5 points and an angle of tolerance of 5° for plotting inverse pole figure (IPF) maps. For analyses of the grain size, twin boundaries were excluded based on the guide provided in [42].

For additional nano-scale characterization of selected samples, atom probe microscopy (APM) was used. Due to the limited size of the gauge length, APM tips were prepared using plasma focused ion beam (PFIB) without lift-out, following the procedures described in [43]. A ThermoFisher Helios G4 Xe PFIB was used for FIB milling. A CAMECA local electrode atom probe (LEAP) 4000X Si was utilized in the laser-assisted mode, with a detector efficiency of 57 %. Data was collected using the following settings: pulse rate of 200 kHz, laser energy of 50 pJ, and detection rate of 1.0 %. The experiments were conducted under ultra-high vacuum conditions of $<3.7 \times 10^{-11}$ Torr in the analysis chamber, with the temperature in channel B kept at 50 K. Data analyses were performed using the CAMECA AP suite version 6, and the reconstructions were calibrated using the tip morphology from SEM images and the tip radius evolution. The enhanced iso-surface method, as proposed by Theska et al. [44], was employed to extract iso-surface threshold values. This method leverages differential concentration profiles at the interfaces to identify the largest concentration gradient. Cluster count analyses were conducted in tip volumes of 50,000 nm³ in the γ matrix, avoiding other μ m-scale phases. To identify a solute cluster, the algorithm was searching for cluster of at least 5 ions, with a step size of 0.01 nm [45].

2.4. Hardness testing

Vickers hardness testing was performed on the gauge lengths of the Gleeble samples using a Struers DuraScan hardness tester with a maximum load of 100 mN and a dwell time of 10 s. Ten micro-indentations were made, and the average values reported. The ASTM standard E92–82 [46] was followed to ensure that the indentations were sufficiently distant from each other to prevent any interference from the strain-hardened areas.

2.5. Thermo-kinetic modeling

Thermo-kinetic modeling was performed using the MatCalc software (version 6.04 rel. 0.011) [42], along with the thermodynamic ('mc.ni.tdb', version 2.034) and diffusion ('mc.ni.ddb', version 2.007) databases. MatCalc has previously been used to simulate solid-state phase

transformations in Ni-based superalloys [47,48], steels [49], and Al alloys [50] during complex traditional thermo-mechanical processes. The precipitation kinetic simulation in MatCalc is based on classical nucleation theory and the Svoboda-Fischer-Fratzl-Kozeschnik (SFFK) growth model [51]. The basic principles of MatCalc have been elaborated in several publications [47,48]. The parameters used for the MatCalc simulation are defined in Section 3.2.

3. Results

3.1. Hardness testing

Fig. 2a presents a bar plot depicting the hardness variation with the number of thermal cycles. The as-received sample exhibited a typical hardness of 385 HV, which is comparable to that of most IN718 Ni-based superalloys in the as-forged (air-cooled) condition [52]. Upon fast heating to 1050 °C followed by quenching, the hardness experienced a sharp drop to 176 HV. This drop indicates a significant change in the microstructure, specifically in terms of grain and precipitate number density [53,54]. Subsequent holding at 700 °C for 60 s did not result in any significant hardness change. However, after 4, 6, and 11 cycles, the hardness slightly increased to 184, 202, and 239 HV, respectively. This

gradual change in hardness is consistent with the frequently reported heterogeneities in the hardness of DED Ni-based superalloys, which are attributed to the change in morphology and number density of γ' , γ'' , and δ phases within the build [23,24].

3.2. Microstructure characterization of the as-received material and Gleeble samples

Fig. 2b displays inverse pole figure (IPF) maps of the as-received and five Gleeble samples investigated in this study. The grains in the as-received material exhibit random orientations, and there is no significant change in orientation during thermal cycling. This grain structure is similar to that of the majority of as-forged (air-cooled) IN718 Ni-based superalloys [52]. The grain size slightly increases from 104 μm in the as-received condition to around 120 μm in all Gleeble samples. It should be noted that all Gleeble samples show grain sizes with differences within the error bars, implying that no significant change in grain size occurs during subsequent thermal cycling.

Fig. 3 shows the kernel average misorientation (KAM) maps of the as-received and Gleeble samples, with misorientation values ranging from 0 to 5°. KAM is used to qualitatively determine the amount and distribution of deformation (plastic strain) throughout individual grains after

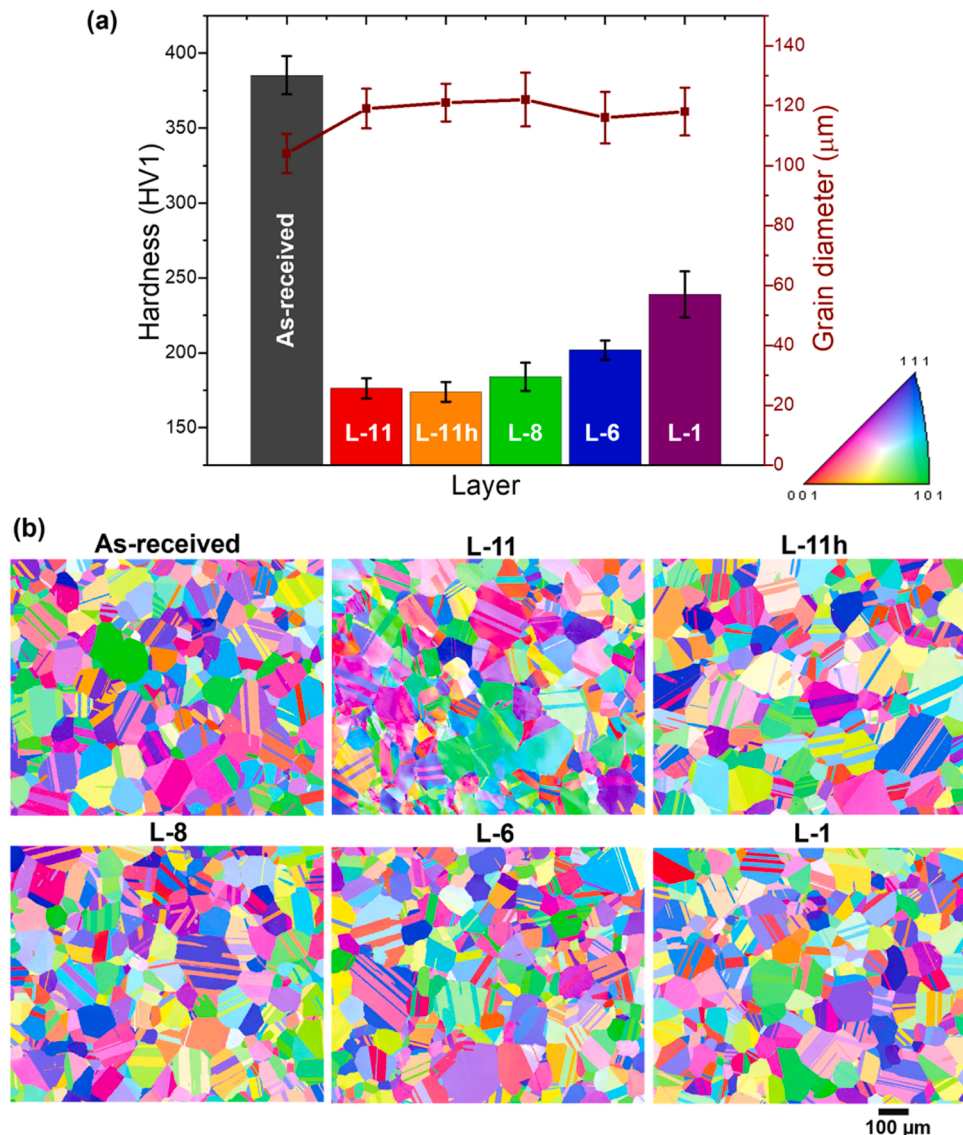


Fig. 2. (a) Average hardness and grain size of the as-received and Gleeble samples. (b) EBSD inverse pole figure maps of as-received, L-11, L-11 h, L-8, L-6, and L-1.

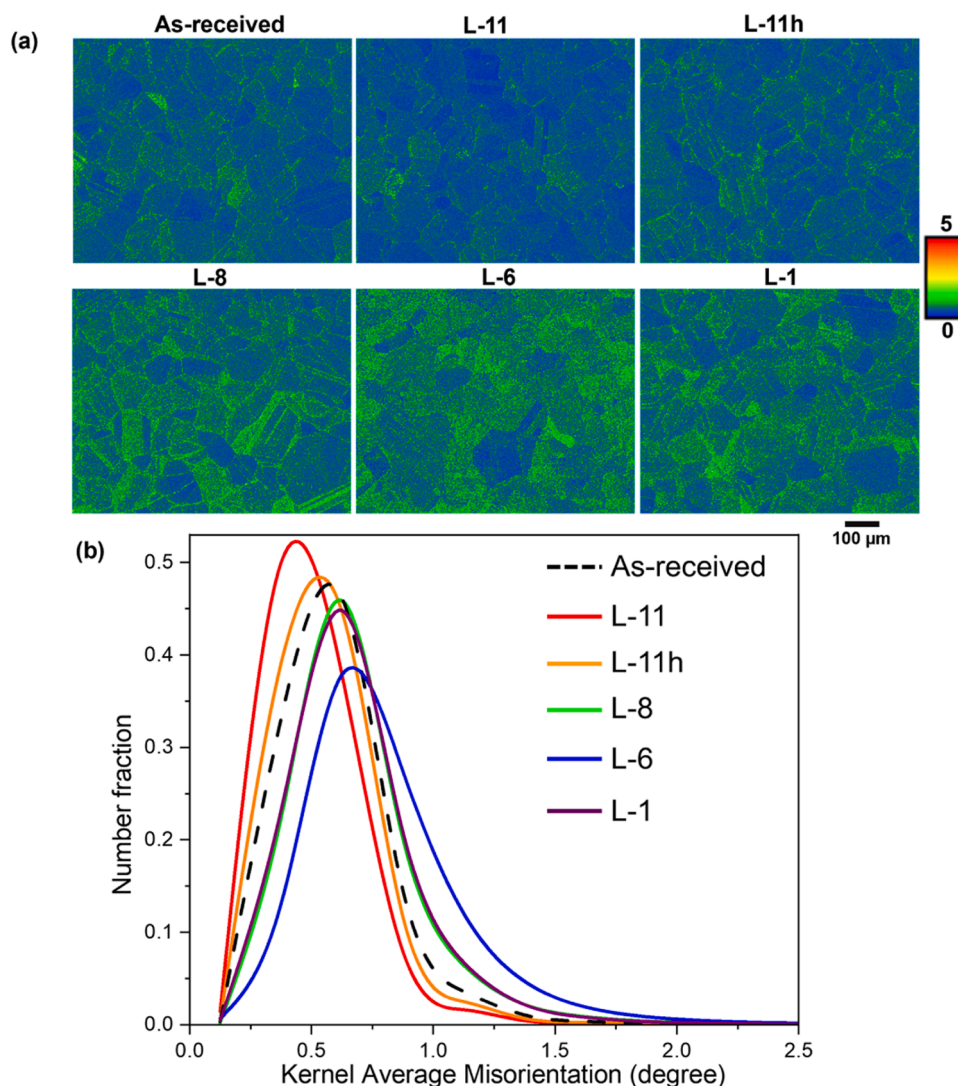


Fig. 3. (a) EBSD KAM maps of as-received, L-11, L-11 h, L-8, L-6, and L-1. (b) Number fractions of KAM in various conditions.

thermal cycling [55]. As shown in Fig. 3a and b, low local misorientations are observed in the as-received sample, which decrease slightly after the first heating and cooling cycle (L-11). Local misorientations exhibit a slight increase during subsequent heating and cooling cycles.

Supplementary Fig. S2 displays BSE images of the as-received and Gleeble samples. The carbides and nitrides present in the as-received microstructure are identified as (Nb,Ti)C (white) and TiN (dark) as confirmed by EDS point analyses shown in Supplementary Fig. S3. These phases are well-known to form in IN718 [56,57]. During thermal cycling, there is no significant change in the size and volume fraction of these phases.

Fig. 4 shows higher magnification images of selected samples. Similar to the trend in hardness, there is a variation in the microstructure as the number of thermal cycles increases. After the first heating and cooling cycle (L-11), the microstructure appears to be free of γ' , γ'' , and δ precipitates, indicating their dissolution (Fig. 4b). A similar microstructure is also observed in L-11 h, despite holding at 700 °C for 60 s. For L-8, the microstructure shows comparably smaller δ phase precipitates (ranging from ~17–50 nm in length) distributed along grain and twin boundaries, as seen in Fig. 4d and Supplementary Fig. S4(a, b). No γ' and γ'' precipitates, however, seem to be present. The size and number of precipitated δ phases increases after additional cycles to ~90 and 300 nm, as observed in L-6 (Supplementary Fig. S4c) and L-1

(Supplementary Fig. S4d), respectively.

The distribution of γ' and γ'' phases is non-uniform, as shown in L-6 (Fig. 4e) and L-1 (Fig. 4f). These phases seem to mainly precipitate around eutectic products (e.g., NbC) at grain boundary edges, as indicated in Fig. 4e and f. The short thermal cycles and high cooling rates prevent their further precipitation, making it difficult to distinguish γ' from γ'' based on SEM only. However, since these regions are richer in Nb as seen in the EDS line scan in Fig. 5, the majority of those precipitates are assumed to be γ'' [58,59].

3.3. Atom probe microscopy

To further investigate the nm-scale precipitate evolution of γ' and γ'' during thermal cycling, APM was used. Fig. 6a, b, and c depict the atom maps for tips extracted from L-11, L-1, and as-received samples, respectively. Based on the atom maps, it appears that all atoms are distributed homogeneously in both L-11 and L-1 samples, indicating the absence of γ' and γ'' precipitates in the γ matrix. The atom maps of Ti and Nb in the as-received state, however, show heterogeneities, indicating the presence of γ' and γ'' precipitates within the γ matrix. There, γ' and γ'' precipitates are encapsulated by iso-surface concentration values of 10.65 at% of Al + Ti) and 12.95 at% of Nb, respectively. These observations are consistent with the SEM images in Fig. 4.

Fig. 7a and b display the cluster count analyses of Al and Nb atoms

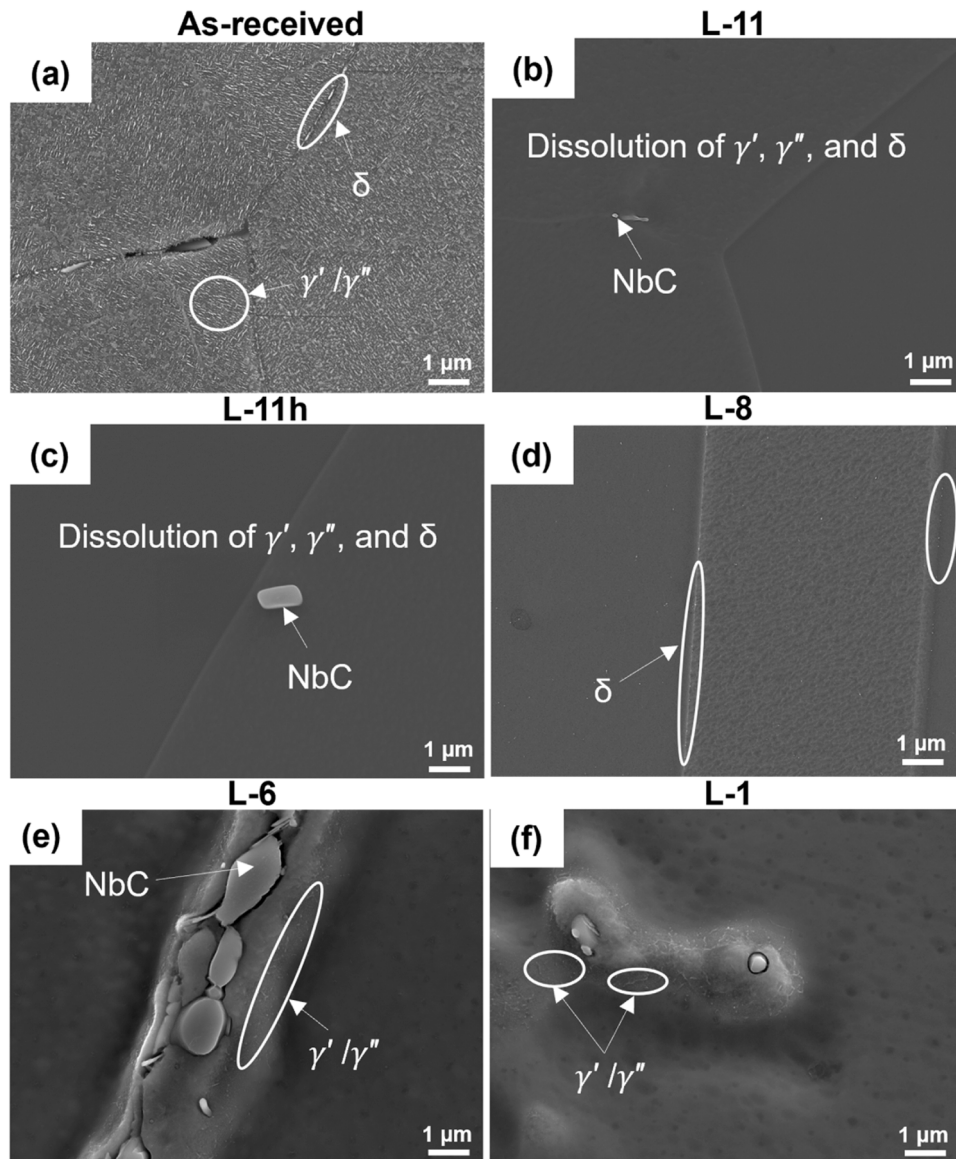


Fig. 4. The microstructure evolution of the as-received and Gleeble samples showing various phases such as γ' , γ'' , δ , and carbides: (a) as-received, (b) L-11, (c) L-11 h, (d) L-8, (e) L-6, and (f) L-1.

for L-11 and L-1, respectively. The purpose of this analysis is to further investigate the presence or absence of early stage solute clustering and/or precipitation as is commonly done using APT [45,59,60]. The experimental distribution curve of L-11 for Nb atoms indicates no deviation from a randomized count, suggesting no solute clustering in this sample. Similarly, for Al atoms, no significant deviation from a randomized count is observed. In the L-1 dataset, the experimental distribution and the randomized curve for Nb atoms are almost identical. However, a slight deviation is present for Al, indicating that the onset of Al clustering must have occurred in regions further away from eutectic products or grain boundaries during thermal cycling.

3.4. Thermokinetic modelling

MatCalc modelling of the thermal profile during the Gleeble experiments were performed with γ defined as the matrix phase and γ' , γ'' , MC carbide, and δ -phases included as possible precipitates. In the kinetic parameter setup for Ni-based superalloys, the MatCalc user needs to define nucleation sites for various precipitates, e.g., bulk, dislocations, and/or grain boundaries. This will result in different nucleation rates,

phase fractions, and number densities [61]. Although γ' and γ'' typically form inside grains via homogeneous nucleation [62,63], experimental observations show that there are no γ' or γ'' precipitates in the bulk of the simulated IN718 microstructure in this work (see Fig. 4e and f). Instead, these precipitates tend to form in regions that are richer in Al, Ti, and Nb. Hence, based on the experimentally observed kinetics, the nucleation sites for γ' and γ'' were assumed to be dislocations and grain boundary edges, and the nucleation sites for the δ -phase grain boundaries. This assumption introduces a form of compositional heterogeneity in the model, creating conditions similar to the experimental setup for in-situ observation of the phase evolution. It should be noted that restricting the nucleation site of γ' and γ'' to the bulk (homogenous) would be suitable for modeling other AM processes like E-PBF and ageing treatments where the precipitation of γ' and γ'' occurs more homogeneously [17,64]. MC carbides were restricted to grain boundaries and the bulk. The regular solution critical temperature for γ' and γ'' was adjusted to 2227 °C [65]. The regular solution critical temperature is the highest temperature at which two phases are present in the system, regardless of the composition. All other parameters were kept at their default values, following the recommendations from MatCalc version

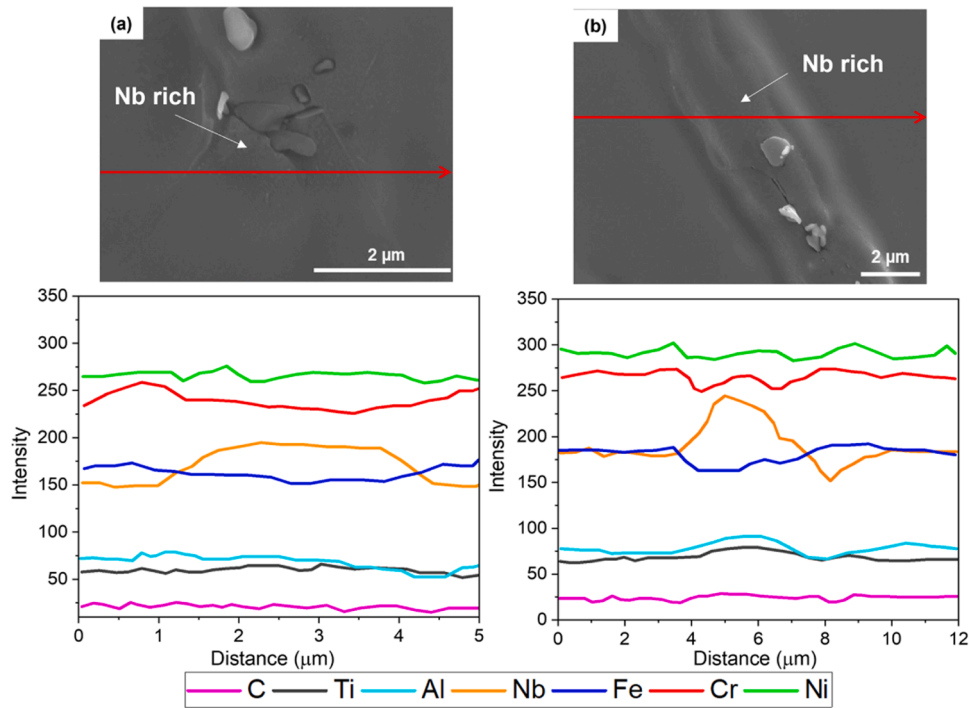


Fig. 5. SEM micrographs and EDS line scan results across (a) a eutectic region and (b) a grain boundary of sample L-6. The red lines indicate the line scan trace.

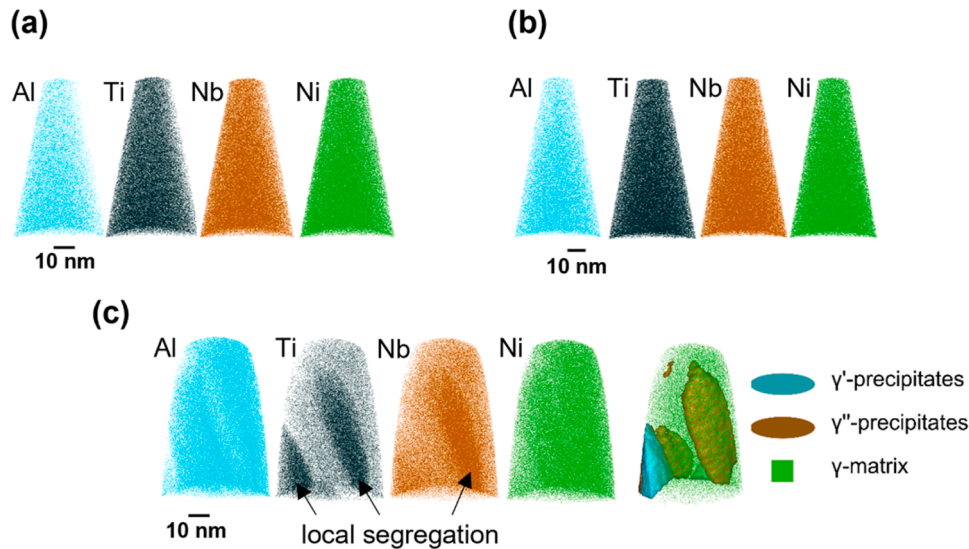


Fig. 6. Atom maps of γ matrix in (a) L-11, (b) L-1 without signs of clustering and precipitation, and (c) as-received with segregation and precipitation of γ' and γ'' .

6.04.0011. Fig. 8a displays the thermal cycles used in the Gleeble experiment as input for the MatCalc simulation.

Fig. 8b shows the simulated phase fractions of γ' , γ'' , MC carbide, and δ -phases during thermal cycling. The inset in Fig. 8b is a magnified view of the simulated phase fractions in the first 400 s. The top layer (L-11) represents the microstructure without undergoing additional thermal cycling. As no melting occurred in the current study, this can be compared to the last layer in an actual L-DED build, representing the microstructure after solidification and final cooling to room temperature. The microstructure of layers below the top layer is determined by the number of thermal cycles, which increases with distance to the top, as seen in Fig. 8a. The phase fraction plot shows small phase fractions ($<1\%$) for all precipitates considered. In the top layer, the precipitation of γ' , γ'' , and δ occurs during cooling. δ nucleates first, followed by γ' and

γ'' , as seen in the nucleation rate (inset in Fig. 8c) and phase fraction plots (inset in Fig. 8b). The phase fractions of γ' , γ'' , and δ increase to 0.00005 %, 0.00015 %, and 0.0005 %, respectively, upon cooling to and holding at 700 °C. The increase in phase fraction is accompanied by the growth of precipitates, with γ' , γ'' , and δ sizes (diameter) reaching 29, 20, and 40 nm, respectively (Fig. 8d). As subsequent layers are deposited, prior layers are subjected to additional thermal cycles with gradually lower peak temperatures, leading to phase transformations of γ' , γ'' , and δ -phases.

During the deposition of any subsequent layer, γ' and γ'' in the previous layer (layer 10) begin to dissolve as the temperature crosses the solvus temperature. This is witnessed by the reduction of the γ' and γ'' phase fractions, number densities, and sizes approaching 0 nm. On the other hand, δ does increase in both size and phase fraction. Upon

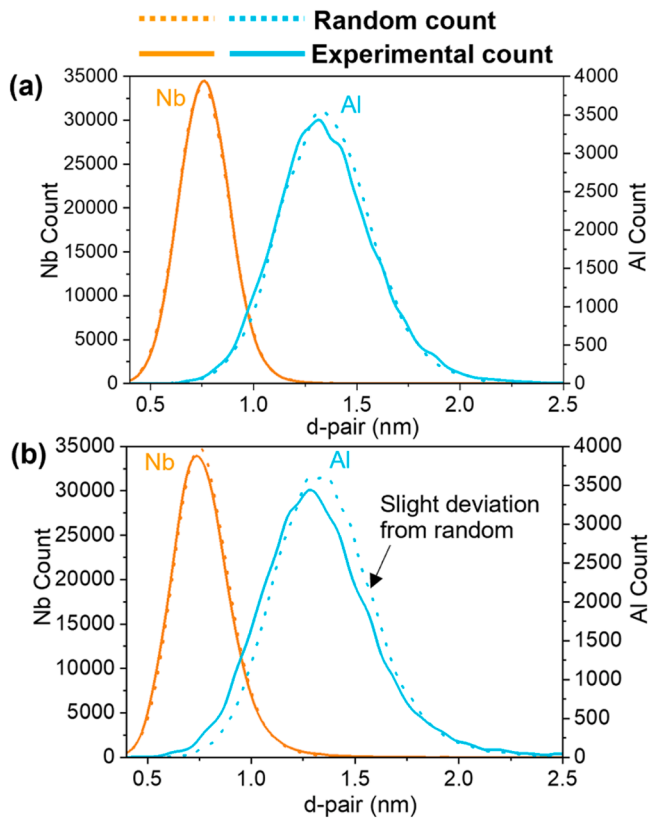


Fig. 7. Cluster count analyses of (a) L-11 and (b) L-1 for Al and Nb atoms.

cooling, γ' and γ'' precipitates that were dissolved during heating reprecipitate with a surge in their phase fractions until the next layer is deposited. The complete dissolution and reprecipitation of γ' and γ'' continue for the next layer deposition while the phase fraction and size of the δ phase gradually increases. In layer 7, the phase fraction of γ' decreases, while the size undergoes slight but noticeable fluctuations. In contrast, there is an increase in both the phase fraction and size of γ'' . Moreover, δ shows further increments in phase fractions and growth. The coarsening of the δ phase is further underpinned by the constant number density after nucleation. The fluctuation in size of γ' indicates partial dissolution. Upon cooling to and holding at 700 °C, the dissolved γ' reprecipitates (4th nucleation burst). From then on, deposition of subsequent layers causes the phase fraction and size of all of precipitates to increase. It should be noted that the increase in precipitate sizes and phase fractions is more pronounced during deposition (heating) than holding at 700 °C. For MC carbide, upon precipitation, the phase fraction and size remain constant without any significant fluctuations.

4. Discussion

Our results have successfully demonstrated the capability of using the Gleeble technology to simulate the L-DED process on IN718. The changes in hardness observed during thermal cycling provide evidence for the correlation between the microstructure evolution during L-DED thermal cycles and properties. In the following sections, we will discuss the effects of thermal cycling on various microstructural features such as the grain morphology, carbides, δ -phase, γ' , γ'' precipitates, and the influence of the starting microstructure. Our experimental data is underpinned by MatCalc thermokinetic simulations, which provide additional valuable insights into the dynamic behavior of these phases during thermal cycling.

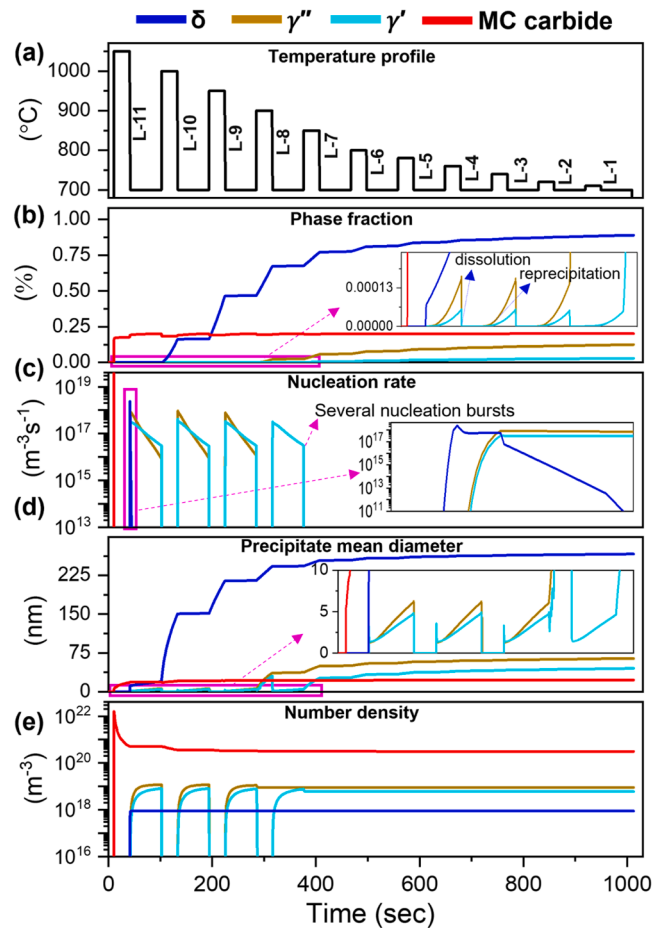


Fig. 8. MatCalc prediction of the phase evolution of γ' , γ'' , MC carbide, and δ -phases during thermal cycling: (a) thermal profile as applied in the Gleeble simulator, (b) phase fractions, (c) nucleation rates, (d) precipitate mean diameters, and (e) number densities.

4.1. γ Matrix evolution

The slight increase in grain size observed in all Gleeble samples compared to the as-received material can be attributed to the high peak temperature of the first cycle (1050 °C). This temperature is reported to be sufficient to achieve the desired grain growth in most forged IN718 alloys, usually for a dwell time of >2 h [66]. However, due to the short dwell time in the current study (30 s), only a slight increase in grain size was observed. The lack of significant change in grain morphology and size during subsequent thermal cycling can be attributed to the much lower peak temperatures and/or short annealing times. This suggests that solid-state thermal cycles with high peak temperatures have a negligible influence on the as-deposited grain morphology and size in AM materials. This also means that the thermal cycles experienced by the as-built part may promote grain growth only if there is sufficient holding time at critical temperatures for grain coarsening [22].

It should be noted that variations in solidification mode affecting the cooling rate and thermal gradient during AM are responsible for changes in the grain morphology and size [67,68]. For example, in most DED builds, there is a transition of dendrite and grain morphology from columnar at the bottom to equiaxed at the top of a build, due to a higher cooling rate and thermal gradient at the bottom layers [69,70]. Although current research shows that thermal cycles do not directly influence the grain morphology and size in the solid-state, they play a major role in changes of the solidification mode. For instance, heat accumulation from previous layers causes upper layers to experience a lower cooling rate compared to the bottom ones, due to the difficulty of

heat transfer from the molten pool into the substrate [27]. It should be noted that most IN718 builds with heterogeneities in grain size exhibit heterogeneities in their hardness as a function of build height [71–73]. However, in the current study, the negligible variation in grain size suggests that this did not play a major role in the hardness variation. This finding aligns with microstructure analysis of DED IN718 builds in literature, where variations in hardness were attributed to factors such as solidification and precipitation rather than grain size [24,74,75].

The low local misorientations observed in the as-received sample could be attributed to recrystallization, grain growth and dislocation annihilation, a characteristic of limited stored residual stress in most as-forged IN718 alloys [76]. The slight reduction in local misorientation after one cycle (L-11) in the current study indicates a slight relief of internal plastic strain, which may be attributed to the high peak temperature that is close to the homogenization temperature. This elevated temperature activates recovery and dislocation annihilation, influencing the strain distribution. However, the short dwell time was not sufficient to cause a significant annihilation of plastic strain. Longer holding times (i.e. >1 h) at temperatures above 1000 °C have been reported to be required to significantly reduce the induced plastic strain in AM IN718 [77]. Although subsequent thermal cycles could anneal out residual stresses or plastic strains caused by previous ones, it should be noted that the restrictive mechanical constraint in AM and our Gleeble setup make plastic strain accumulation significant [78–81]. This restrictive mechanical constraint influences the thermal expansions and contractions during fast heating and cooling cycles, leading to strain accumulation. This is because the constrained material is unable to expand or contract freely, resulting in the buildup of internal stresses, which explains the observed increase in local misorientations during subsequent heating and cooling cycles as seen in L-6 and L-1. It should be noted that DED builds are generally prone to high plastic strain due to the low heat accumulation from previous layers [27,82,83]. This differs from electron beam powder bed fusion, where the build is subjected to elevated pre-heat temperatures that significantly reduce thermal gradients and relieve stresses throughout the builds [84–86].

4.2. Microscale precipitates

The presence of TiN in the as-received sample can be attributed to its formation during solidification [87]. In AM, TiN can also be introduced via the feedstock material and may remain unmelted due to its high melting point (~2930 °C) [88]. This explains its presence in all Gleeble specimens. In IN718, precipitation of (Nb,Ti)C is known to occur during solidification and usually forms in the interdendritic regions via eutectic reactions [21,89]. In the current study, the size and volume fraction of these carbides are much higher compared to actual L-DED process due to the slower solidification and cooling rate of the starting microstructure. The constant and stable phase fraction of the carbides during thermal cycling can be attributed to their high melting point (~1050–1360 °C) and the short holding time at the peak temperatures [90–92]. This is underpinned by the MatCalc prediction, where we do not observe any fluctuations in the carbide morphology during thermal cycling. The solvus temperature for most MC carbides exceeds all peak temperatures observed in our Gleeble study, and carbide dissolution kinetics at temperatures above the solvus are known to be comparably slow [93]. The stable phase fraction and random distribution of MC carbides indicate that they have a negligible influence on the gradient in hardness.

4.3. Nanoscale precipitates

4.3.1. Evolution during thermal cycling

The γ' , γ'' , and δ phases are the prevailing precipitates that typically form during solid-state phase transformations in IN718. [94–96]. Their presence in the as-received sample in the current study is a consequence of slow cooling after forging, indicating there was sufficient time for their nucleation and growth. The as-forged sample spends a relatively

longer time in the temperature ranges of the γ' , γ'' , and δ precipitation windows, that are in the range of 600 to 1000 °C [21]. In IN718, the δ phase is formed at interfaces (e.g., grain or twin boundaries) and/or inside grains through the $\gamma'' \rightarrow \delta$ transformation [21,97]. In this study, the precipitation of the δ phase is seen to occur at the grain boundaries only (see Fig. 4d, and Supplementary Fig. S4).

The dissolution of all precipitates after the first heating cycle is due to the high peak temperature, which is above the solvus temperatures of γ' (~850 °C), γ'' (~900 °C), and δ (~1000 °C), respectively [98]. The MatCalc simulation (Fig. 8) supports this observation, i.e., no precipitates are seen at peak temperatures above the solvus. The complete dissolution of the various precipitates despite the short dwell time at the peak temperature can be attributed to the exponential increase in solute diffusion rates at high temperatures [37]. Since dissolution occurred during heating and holding at the peak temperature, the γ' , γ'' , and δ phases should have precipitated upon cooling as the temperature profile passes through their precipitation window. However, no precipitation is observed, which can be attributed to the rapid cooling rate achieved in the Gleeble. This rapid cooling rate does not allow for sufficient time for phase nucleation and growth to occur in sample L-11 [23]. This explains why in DED IN718 the precipitation of γ' , γ'' , and δ phases and their growth rates are less pronounced compared to traditional manufacturing. Our MatCalc model shows a minor precipitation of δ in L-11 during cooling (Fig. 8). This can be attributed to the high sensitivity of precipitation and nucleation related physical parameters in the software [47].

The presence of a small fraction of δ phase precipitates (17–50 nm) in L-8 after three cycles indicates the onset of δ precipitation, as the peak temperatures of the previous layers were above the δ solvus (~1000 °C). The peak temperature experienced by layer 8 is within the precipitation window of δ and thus enables its precipitation. This is in line with the work of Jang et al. [99], where the precipitation of δ in wire-arc DED IN718 was attributed to the sufficient time the layer spent in the δ precipitation window. The same authors drew this conclusion by utilizing the thermal history of the deposit and the precipitation kinetics of the δ phase, using Continuous Cooling Transformation / Time-Temperature-Transformation (TTT) diagrams. As the peak temperature of L-8 in our study is still above the solvus temperature of γ' and γ'' , these phases do not precipitate. MatCalc predictions confirm this via the cyclic dissolution and reprecipitation of γ' and γ'' , while the δ phase remains stable. The increase in phase fraction and growth of δ during subsequent thermal cycling (from L-6 to L-1) indicate prolonged exposure of these layers within the precipitation temperature window. This is also supported by MatCalc predictions, which point out that the growth of δ and increase in phase fractions is more pronounced during heating to and holding at peak temperatures. Similarly, the presence of γ' and γ'' in L-6 (after 6 cycles) indicates the onset of γ' and γ'' precipitation, as the peak temperature is within the precipitation window in this case. This means the layer remained in this window for a long enough period. The various reported TTT diagrams of γ' and γ'' in IN718 indicate that typically a longer incubation time (>200 s) is needed for the onset of precipitation, especially at low temperatures [21,23]. This explains why even holding at 700 °C for 60 s in previous layers (e.g., L-11 h) did not result in the formation of γ' and γ'' .

The presence of δ in layers L-1 to L-8 and its absence in layer L-11 align with the observed heterogeneity in the distributions of δ phases in L-DED IN718 materials [24,100,101]. Similarly, the presence of γ' and γ'' in L-1 to L-6, and their absence in L-4 to L-11, support the observed heterogeneity in the distributions of γ' and γ'' phases. Li et al. [23] reported the precipitation of γ' , γ'' , and δ in the middle and bottom of a L-DED IN718 build in contrast to the top, due to the intense thermal cycle in the middle and bottom regions that satisfy the precipitation conditions of these phases. In the current work, the heterogeneity in precipitation with increasing numbers of cycles explains the potential slight increase in average hardness, within error bars. In addition to γ'' strengthening, small amounts of δ phase in IN718 are known to improve

the mechanical properties by providing grain boundary pinning [102].

4.3.2. Influence of the starting microstructure

In the current study, the starting microstructure lacks typical AM dendrites due to its wrought processing after casting. This contrasts with real L-DED printed materials, which typically exhibit distinct dendritic structures. As a result, the current study exhibits less compositional heterogeneity compared to real L-DED printed parts. In AM, after the non-equilibrium solidification of IN718, there is a composition gradient from the dendrite core to the interdendritic region or eutectics in the matrix due to segregation. This leads to a shift in the local equilibrium conditions and driving forces for solid-state phase transformation from the dendrite core to the interdendritic or eutectics regions [103,104]. Despite compositional gradients, γ' and γ'' precipitation is expected to be homogeneous if the alloy spends sufficient time in the γ' and γ'' precipitation window. This is evident in most aged IN718 and E-PBF samples, where uniformly distributed γ' and γ'' are reported [52,53,105]. For instance, the elevated preheat temperature and slow cooling during E-PBF cause the material to spend sufficient time in the γ' and γ'' precipitation window. However, in L-DED, the time is insufficient due to low global heat accumulation and fast cooling rate [21,27]. Consequently, regions richer in γ' and γ'' forming elements become favourable for nucleating γ' and γ'' , as these regions provide a lower-energy surface for nucleation [23,99,102,106]. A typical example can be found in the work of Tian et al. [24], where they reported the precipitation of γ' and γ'' primarily around eutectic products (e.g., NbC and Laves) in interdendritic regions. This indicates that the non-uniform distribution of γ' and γ'' is influenced by compositional gradients determined by the initial solidification conditions and the lower heat accumulation.

In real L-DED processing, the high concentration of solute elements near eutectic products along grain or dendrite boundaries can also be attributed to the partial remelting of those eutectic products and diffusion during additional thermal cycles. L-DED thermal cycles exhibit higher peak temperatures, potentially approaching the solidus of various eutectic products such as Laves and carbides [24]. This is less likely to occur in the current study due to the less complex thermal profile and absence of dendritic structures. However, it is worth noting that the compositional heterogeneity observed (Figs. 5a and 5b) can be attributed to the potential excessive overshoot of the target temperature during the initial thermal cycles. Temperature overshooting in Gleeble testing typically occurs when the heating rate exceeds 500 °C/s [107]. Considering the high heating rate employed in the current study (5000 °C/s), this overshoot could have led to the temperature temporarily exceeding the solidus temperature of selected eutectic products, leading to micron-scale solute diffusion. The localized enrichment of Nb near eutectic products increases the driving force for the nucleation of precipitates. Furthermore, the areas that have undergone remelting are exposed to a slower cooling process during subsequent melting cycles, which ultimately makes them favorable sites for precipitation [75,108].

In regions further away from grain boundaries or eutectic products, APM confirms that the first heating cycle in this study dissolved all precipitates previously present, and that all solute elements were distributed homogeneously thereafter (Fig. 6). Although the APM samples studied were taken from random regions, the lack of local segregation allows us to conclude that the tips in Fig. 6 are from regions further away from eutectic products or grain boundaries. The slight deviation of the experimental distributions of Al from random ones indicates that Al responds more strongly to clustering than Nb does during extended thermal cycling [59]. This phenomenon could be attributed to the slow cooling rate during the initial material manufacturing, which enabled Al to diffuse to lattice sites that could become nucleation sites for Al-rich γ' precipitates during prolonged thermal cycling. A similar phenomenon was reported in L-PBF IN718, where Rielli et. al [45] observed Al clustering in the dendrite core due to slow cooling from the chessboard laser scanning strategy. These clusters impacted precipitation during direct ageing, by acting as potential nucleation sites for

Al-rich γ' .

4.4. Outlook

The current study demonstrates that physical Gleeble simulations are indeed suitable to replicate AM thermo-mechanical cycling, and this is underpinned by MatCalc simulations. Immediate future research using these tools should extend our approach to AM starting materials fabricated by wire arc additive manufacturing (WAAM). This will provide valuable insights into the role of AM solidification microstructures including dendrites and solidification cells on solid-state precipitation during thermal cycling. As mentioned earlier, these features were absent in the initial microstructure studied here. To conclude this section, it is worth noting that the capabilities of Gleeble for AM research extend beyond studying the role of thermal cycling on the solid-state microstructure evolution. For example, the Gleeble simulation strategy may provide valuable insights into how the solidification microstructure evolves in response to multiple melting cycles and shed light on the mechanisms behind micro crack formation during AM [85]. These future applications of Gleeble in AM research will help to advance the current understanding AM processing – materials properties relationships.

5. Conclusions

We successfully apply a combined physical and computational modelling approach to systematically reveal the impact of typical thermal cycling experienced during additive manufacturing on the solid-state microstructure evolution of IN718. We replicate thermal cycles IN718 experiences during laser directed energy deposition (L-DED). We systematically study the effect of thermal cycling on the microstructure and hardness evolution. Our main findings are summarized below:

1. The microstructure evolution of IN718 thermally cycled in a physical simulator shows excellent correlation with the actual L-DED process, highlighting the capability of using physical simulations for future AM research on Ni-based superalloys. The microstructure evolution is in good agreement with predictions made by thermo-kinetic simulations.
2. Typical L-DED thermal cycles in the solid-state are demonstrated to have a negligible influence on the grain morphology of IN718 due to the short holding time at critical peak temperatures.
3. L-DED thermal cycles, coupled with the restrictive mechanical constraint of the sample or build, induce plastic strain accumulation, similar to those experienced during AM processing due to the presence of additional layers.
4. L-DED thermal cycling influences the microstructure evolution and phase transformation pathways during AM, leading to significant effects on the precipitation and distribution of γ' , γ'' , and δ phases. A negligible influence is observed for carbides present in the starting material, owing to their high solvus temperature.
5. During initial thermal cycles, no γ' , γ'' , and δ phases are observed due to the high peak temperatures above the solvus. The short holding time at peak temperatures is shown to be sufficient to dissolve all phases from the starting material.
6. Prolonged thermal cycling, which is representative of the middle and bottom regions of the actual L-DED process, leads to the precipitation of γ' , γ'' , and δ phases. The δ phase is shown to precipitate along grain and twin boundaries, while γ' and γ'' forms in regions richer in Nb. Prolonged thermal cycling also causes clustering of Al atoms.
7. The heterogeneity in the distribution of nanoscale precipitates leads to a variation in hardness.

The knowledge gained from this study is crucial for controlling solid-state phase evolution in IN718 during AM and will be applicable to other AM processes and alloys. The ability to systematically explore the solid-

state phase evolution through process parameter optimization using such combined modelling approaches will facilitate the fabrication of Ni-based superalloy parts with superior properties for critical applications such as aerospace.

CRediT authorship contribution statement

X.Z. Liao: Funding acquisition, Project administration, Writing – review & editing. **N. Haghdadi:** Conceptualization, Methodology, Funding acquisition, Supervision, Writing – review & editing. **N. Adomako:** Conceptualization, Formal analysis, Investigation, Methodology, Writing – original draft. **S. Primig:** Conceptualization, Funding acquisition, Methodology, Project administration, Software, Supervision, Writing – review & editing. **S.P. Ringer:** Funding acquisition, Project administration, Writing – review & editing.

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.

Data Availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jallcom.2023.173181](https://doi.org/10.1016/j.jallcom.2023.173181).

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