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ORIGINAL REPORT



Revealing in-situ phase transition during laser additive manufacturing via high-speed synchrotron X-ray diffraction and imaging

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ABSTRACT

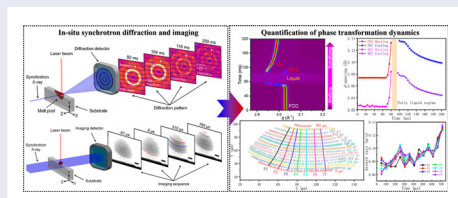
Phase transition at the initial solidification stage notably affects subsequent phase structure of peritectic alloys, however, detailed dynamics between entangled three-phase region remains ambiguous in the far-from-equilibrium condition during laser additive manufacturing. Here, in-situ peritectic phase transition during single-layer laser printing of an exemplary 304L stainless steel is directly investigated and quantified at high temporospatial resolution by high-speed synchrotron X-ray diffraction and imaging. These quantitative observations clarify the mechanism of coupled peritectic growth and local remelting at three-phase region. The results revealed provide in-depth understanding of phase transformation dynamics and delicate design of phase structure for metal additive manufacturing.

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laser additive manufacturing;
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Introduction

Laser additive manufacturing (AM), being a novel manufacturing route for three-dimensionally printing customized and geometrically complex metallic components directly from a digital file [1,2], creates diverse solidification modes from equilibrium to nonequilibrium, depending on the solidification rate from ~ 1 mm/s to ~ 1 m/s range and cooling rate up to 10^6 K/s. As for highly transient laser printing process, the resultant phase structure can become complex and unpredictable [3,4]. Austenitic 304L stainless steel (SS), for instance, varies from single-phase to multiphase structures when

widely printed in aerospace and automotive and biomedical fields due to exceptional printability [5,6]. At the initial solidification stage, there are three phases entangled due to a peritectic phase transformation for this alloy: parent liquid (L) phase, body-centered cubic (BCC) ferrite (δ) phase and face-centered cubic (FCC) austenite (γ) phase. When cooling down high-temperature liquid, primarily-solidified BCC phase appears first from melt. Primary BCC phase contacts with liquid and reacts to form peritectic FCC phase at the triple point, which corresponds to three-phase region in the phase diagram. The connectivity of Liquid, BCC and FCC phases and

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equilibrium peritectic reaction at isothermal temperature usually requires a slow cooling rate [7]. Because fast cooling rate results in the combined effects of temporal changes in the interface composition and compositional gradient during solidification, BCC δ phase grows with difficulty and liquid is inclined to directly transform to peritectic phase. FCC γ phase can primarily grow and original δ phase is suppressed at higher cooling rate of 1.5×10^6 K/s [4]. But the presence of δ phase found in the final FCC matrix suggests incomplete peritectic reaction during initial solidification and the hypothesis whether three-phase ($L + \delta + \gamma$) reaction exists in the far-from-equilibrium conditions during laser AM process demands direct experimental evidence [8–11].

The initial solidification of peritectic alloys undergoes two stages of peritectic reaction and transformation, greatly controlling the solidified phase structure and desired properties afterwards [12]. Peritectic reaction terminates once primary phase is enclosed by peritectic phase and cut off contact with liquid and subsequently peritectic transformation occurs [13]. For the peritectic reaction, mechanisms of thermal diffusion of solute atoms in the liquid phase, remelting of properitectic phase by accompanying the growth of peritectic phase, and effective dissipation of latent heat and solute mixing ahead of peritectic tip are proposed. For the subsequent peritectic transformation, mechanisms of solute diffusion from liquid to properitectic phase through peritectic layer and massive transformation of crystal structure change at atomic scale without compositional partitioning or solute diffusion are proposed. Visualizing the growth of properitectic and peritectic phases from liquid directly helps better understand the mechanisms of peritectic reaction and transformation and well tune associated phase structure and defects upon initial solidification.

However, directly observing the three-phase reaction in nonequilibrium conditions is really challenging and lacks of in-situ experiments. Because there are many obstacles that block real-time investigation of initial solidification behavior during laser printing process: (1) it needs subsurface detection due to solidification front located at back rim of melt pool but conventional techniques only capture surface profiles; (2) thick feedstock and substrate obstruct the transmission observation because of intrinsic opaqueness of metals; (3) localized melting and solidifying of melt pool demand high temporospatial detecting resolution in terms of microsecond- and micrometer-scale laser-metal interaction; (4) mechanisms of real-time phase transformation dynamics stay illusion in destructive and time-consuming postmortem characterizations. Recently, high-speed synchrotron X-ray imaging and

diffraction techniques unveil melt pool-scale phenomena and mechanisms of defect formation and evolution [14–18], which provide promising solutions to in situ investigate the phase transformation dynamics in nonequilibrium condition.

Here we deploy the state-of-the-art high-speed synchrotron X-ray diffraction and imaging capabilities to investigate the initial liquid-to-crystal solidification process during single-layer laser printing of 304L SS. An intriguing peritectic phase transition is delicately quantified, specifically for the high solidification and cooling rates and phase structure evolution as function of time and location. The quantification of three-phase (Liquid+BCC+FCC) reaction at millisecond and micrometer scales, to the best of our knowledge, is the first in-situ observation of peritectic reaction and transformation during the laser AM process. These corroborate the mechanism of peritectic FCC growth along the liquid/BCC interface with intense heat transfer and solute mixing during peritectic reaction and effective thermal diffusion at millisecond scale during peritectic transformation.

Results

Phase transformation by in-situ synchrotron X-ray diffraction

Aiming to find out the existence of peritectic reaction in far-from-equilibrium condition, in-situ high-speed high-energy synchrotron X-ray diffraction of single-layer laser printing of exemplary 304L SS has been conducted (Figure 1). Focused X-ray beam is aimed at melt pool to real-time monitor solidification sequence and connectivity of Liquid, BCC and FCC phases. The laser printing parameters are selected based on our previous experience [3]. As sketched in Figure 1(a), upon cooling, phase transformation dynamics is recorded by high-quality diffraction patterns (see Supplementary Material). Original diffraction patterns with high frame rate (250 Hz) have high q -space range and low d -spacing crystallographic planes, which are good enough for phase identification and quantification. The halo rings of diffraction pattern at the time of 92 ms indicate that the specimen is melted after turning on the laser. At the transmission path of synchrotron X-ray beam, the whole melt pool is fully liquid under the exposure of scanning laser. At 104 ms, intensive crystalline spots of BCC phase (δ -ferrite) come up among the halo rings: the brightest spots from (110) plane is immersed in the first halo ring; the spots from (200) plane locate between the two main halo rings. Weak FCC (111) and (200) crystalline spots (γ -austenite) show up at one diffraction frame later. It should be noted that

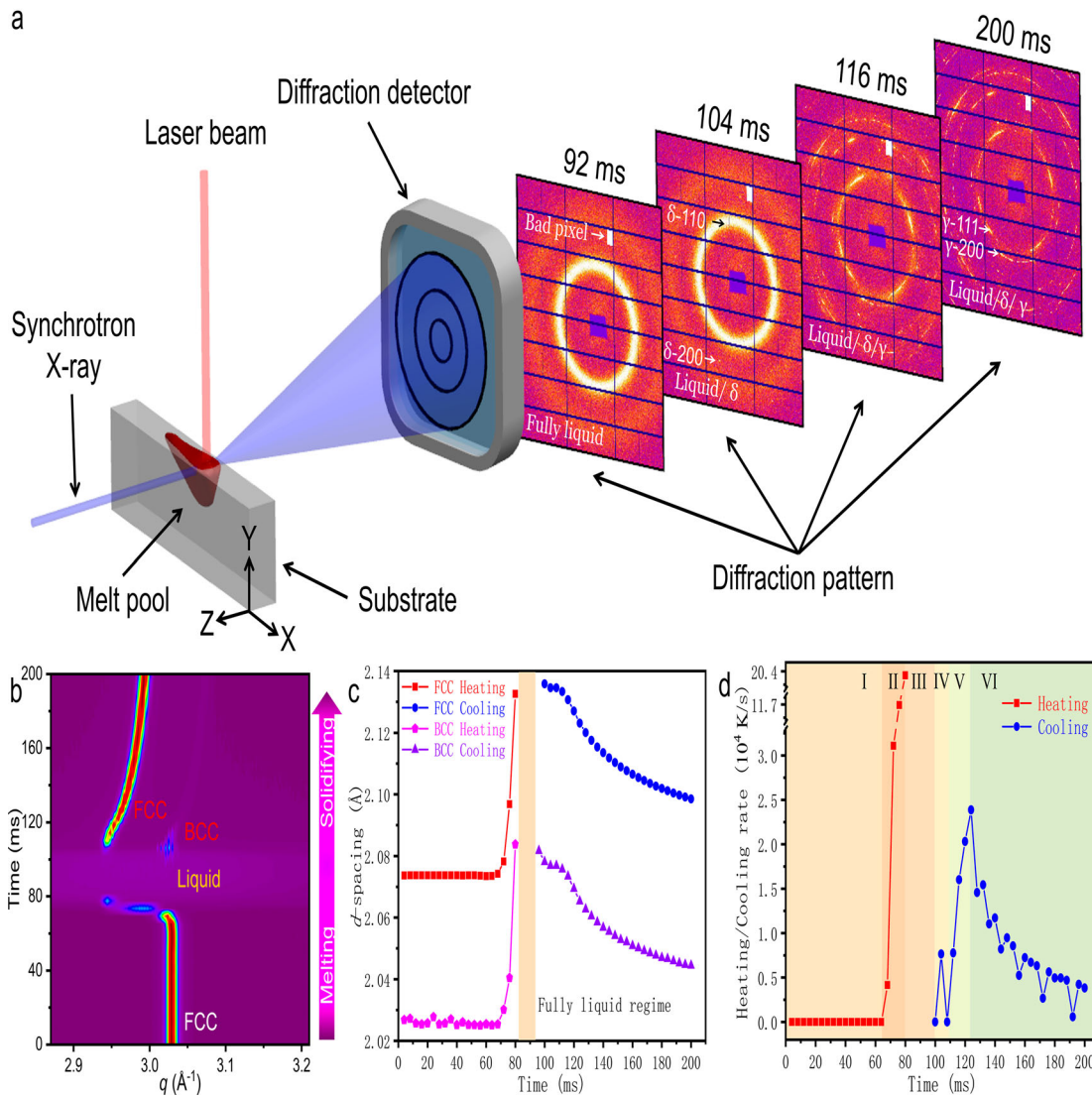


Figure 1. Time-resolved synchrotron X-ray diffraction observation of phase transformation dynamics during single-layer laser printing of exemplary 304L SS. (a) Schematic illustration of in-situ high-speed synchrotron X-ray diffraction experiments during laser printing process. The diffraction system is synchrotronized with laser printing system and high-quality sequential 2D patterns are recorded when laser scans through X-ray beam location. (b) 2D phase evolution plot (time vs. the scattering vector q , with diffraction peak intensity as color map) during in-situ XRD measurements upon melting and solidifying. Clearly, there is a three-phase (Liquid, BCC and FCC) region upon solidifying. (c) Evolution of principal FCC (111) and BCC (110) peak positions when laser scans through X-ray beam location, demonstrating a plateau of d-spacing curves and isothermal transformation stage upon rapid solidification. (d) Heating and cooling rates of 304L SS during single-layer laser printing calculated from the lattice d-spacing difference over given time gap. Six marked stages of heating and cooling rates represent six distinct locations with different thermal history of powder bed during laser AM process.

the spots of BCC phase also become brighter and the halo rings of liquid are still observable during liquid-to-crystal transformation. At 200 ms, peaks from BCC phase and halo rings of liquid die down and peaks from FCC phase intensify.

Figure 1(b) displays high-resolution diffraction patterns zoomed in at the q -space range of 2.85–3.25 $\text{\AA}^{-1}$ and presents the time-resolved phase transformations upon melting and solidifying during the single-layer laser printing process. BCC phase in the substrate can be

detected but negligible due to limited volume fraction as compared to FCC phase. Peak positions of FCC phase expand towards low q -space direction quickly before 80 ms when the laser is turned on. Fully-liquid state is achieved after 16 ms and lasts for another 12 ms. BCC phase emerges 4 ms before the appearance of FCC phase, then intensifies for 16 ms and becomes faint afterwards. The intensity of liquid phase decreases quickly, but it still remains noticeable after 200 ms because of observable halo rings. FCC phase appears and become dominant

after the die-down of BCC phase. Clearly, there is an overlap among Liquid, BCC and FCC phases and the three-phase region is observed upon cooling.

Principal FCC (111) and BCC (110) peaks are representatively selected to trace the evolution of peak positions (Figure 1c). Peak positions of both phases consistently maintain a decreasing trend overall under laser beam processing and dramatically drop after 200 ms. Interestingly, a plateau shows up both at the d -spacing curves of BCC and FCC phases in the cooling stages, suggesting an isothermal transformation stage for the rapid solidification inside melt pool. It is in agreement with the hypothesis that primarily-solidified BCC phase contacts with liquid and reacts to form peritectic FCC phase resulting in the isothermal three-phase region.

Further, after turning off the laser and cooling down melt pool, the peak shifts of both BCC and FCC phases within 200 ms are mainly ascribed to thermal effect and reflect local temperature change. The real-time cooling rate is calculated by using time-dependent lattice d -spacing shifts with the consideration of in-situ diffraction patterns and consistent with temperature-dependent thermal expansion coefficient of FCC phase (Figure 1d). The calculated heating and cooling rates exhibit six remarkable stages: at stage I (0–64 ms), it is flat zero as the laser is far away to the X-ray beam location; at stage II (68–80 ms), it soars up to 2.04×10^5 K/s when the laser melts the substrate; at stage III (84–96 ms), it has no data points because melt pool stays fully liquid; at stage IV (100–108 ms), it fluctuates near zero at the emergence of FCC phase corresponding to the plateau of lattice d -spacing curve; at stage V (112–124 ms), it slowly climbs and peak at 2.39×10^4 K/s; at stage VI (128–200 ms), it exponentially decreases.

Solidification dynamics by high-speed synchrotron X-ray imaging

A fully-liquid spherical droplet quickly solidifies onto the substrate due to the combination of thermal conduction and radiation effects when generated from powder bed fusion of 304L SS and two successive solid–liquid interfaces are observed, which provides supportive evidence for peritectic phase transition upon rapid solidification (Figure 2). The two solid–liquid interfaces during cooling are entirely filmed by high-speed synchrotron X-ray imaging (Figure 2a and supplementary Video 1). The primary solid–liquid interface corresponds to the growth of primary BCC phase into liquid region whilst the second solid–liquid interface results from the growth of peritectic FCC phase at the expense of primary BCC and liquid phases. A primary solidification front highlighted in red grows upward into top

liquid region of spherical droplet at 0 μ s. Meanwhile, another moving solid–liquid interface follows the primary solidification front, which is highlighted in blue and distinctly depicted at 510 μ s. These two solid–liquid interfaces propagate deeply into top liquid region and successively disappear due to fully transformation after 765 μ s.

To quantify the growth rates of primary BCC and subsequently peritectic FCC phases, two solid–liquid interfaces observed in the X-ray images are extracted and plotted in Figure 2(b,c). The primary solid–liquid interface of BCC phase in Figure 2(b) overlays over time (open circles) and incurves toward top side of the spherical droplet, similar to the reported results [19,20]. Seven spots evenly distributed at the reference line of primary solid–liquid interface are selected to track the evolving trajectories as function of time. Each spot moves with the growth direction normal to the current interface line at the reference time point and intersects with successive interface lines at following time steps. Thus trajectories of all spots can be determined sequentially, highlighted as solid lines in Figure 2(b). Knowing the moving distance at specific time difference, the growth rate of seven tracked spots is calculated by dividing its displacement by its traveling time and presented in Figure 2(d). The growth rate of primary solid–liquid interface fluctuates and gradually climbs from 0.08 to 0.10 m/s before 612 μ s, and speedily rises to 0.14 m/s from 612 to 765 μ s. Before 612 μ s, the intensive fluctuation of growth rate might result from the competition between thermal conduction of substrate and generation of latent heat from Liquid-to-BCC transformation. After 612 μ s, the combination of thermal removal from conduction substrate and top surface radiation mainly contributes to the effective cooling and makes the growth rate escalating and surging afterwards. Figure 2(c) displays the real-time evolution of second moving boundary following the primary solid–liquid interface and trajectories of five tracked spots. The line curvature of second moving boundary is similar to that of primary solid–liquid interface with aspect of different time scale due to the subsequent Liquid-BCC-to-FCC phase transformation. The growth rate of tracked five spots in Figure 2(e) is in line with the results observed in Figure 2(d) and it keeps increasing from 0.08 to 0.14 m/s when cooling to the top of spherical droplet. The evolving consistency from the seven spots of primary solid–liquid interface and the five spots of second moving boundary indicates the progressive propagation of liquid-to-crystal interfaces and suggests potential epitaxial growth as a result of effective dissipation of latent heat inside liquid regime ahead of first solid–liquid interface and subsequent second moving boundary.

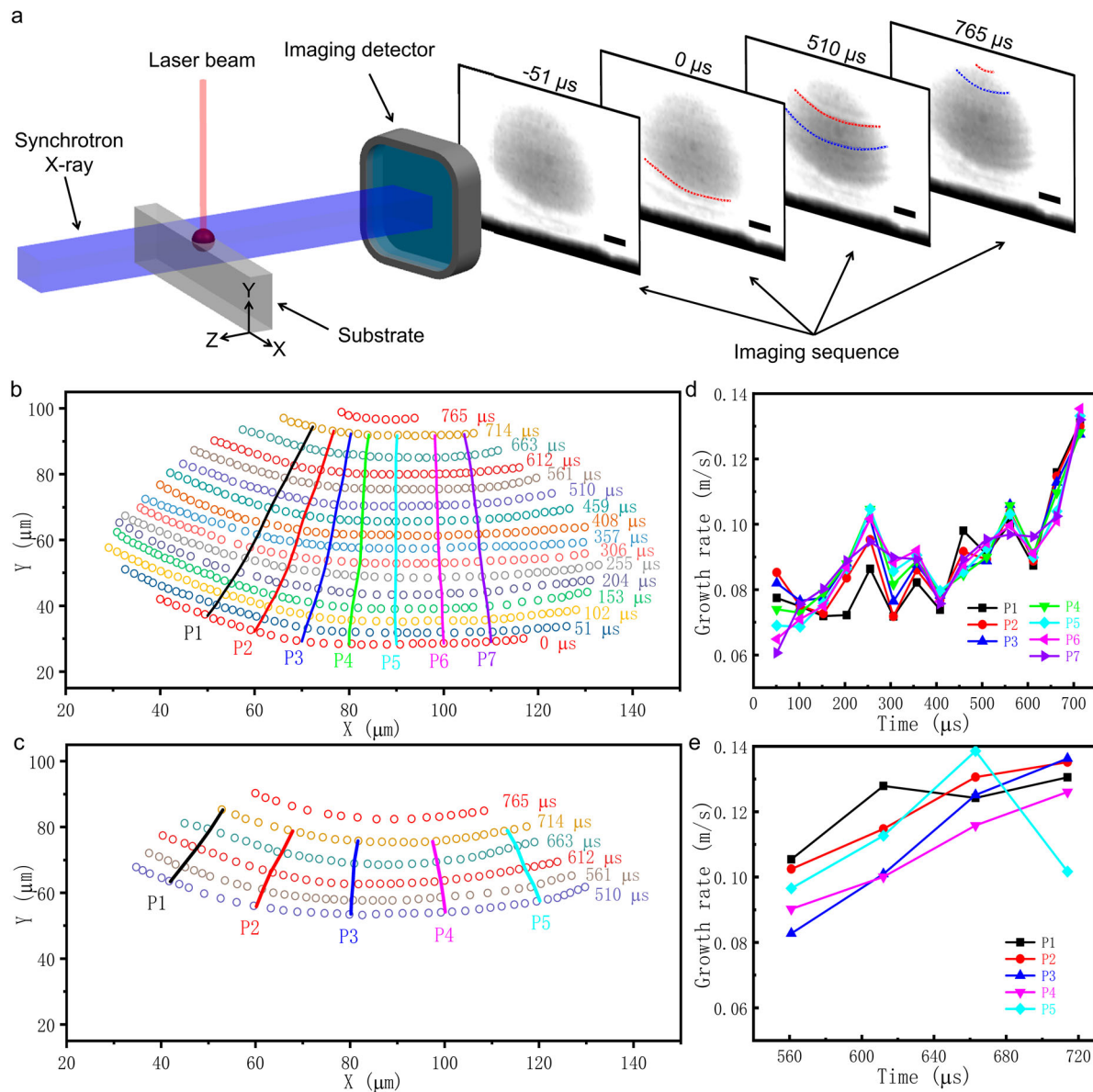


Figure 2. High-speed synchrotron X-ray imaging quantification of double solid-liquid interfaces during rapid solidification of 304L SS droplet. (a) Schematic of high-speed synchrotron X-ray imaging during rapid solidification of a fully-liquid spherical droplet. Representative X-ray images show the evolution of double solid-liquid interfaces of a laser melted droplet. Scale bars are 20 μm . (b) Evolution of primary solid-liquid interface upon cooling of the spherical droplet. The appearance of detectable solidification front is set as reference point. Seven spots are tracked for the entire solidification lifetime by calculating the moving trajectories according to normal direction of growth at the solidification front. (c) Evolution of second moving boundary following the first solidification front. Five spots are tracked to quantify the moving trajectories. (d) Growth rate of seven tracked locations as function of time at primary solid-liquid interface. (e) Growth rate of five tracked locations as function of time at second moving solid-liquid interface.

Microstructure evolution of rapid solidification

To confirm the in-situ observation of peritectic phase transformation during rapid solidification, grain-scale microstructure from ex-situ laser spotwelding of 304L SS with comparable melting conditions of fully-liquid spherical droplet is characterized by electron backscatter diffraction (EBSD) measurements (Figure 3). Epitaxial grains inside melt pool highlighted by dashed

lines directly grow on top of equiaxed grains of the substrate base (Figure 3a). The symmetric grain structure inside melt pool suggests the effective dissipation of latent heat inside melt pool upon solidifying during laser spotwelding. Interestingly, the existence of retarded BCC phase distributed between grain boundaries (Figure 3b) proves that peritectic transformation from liquid and BCC to FCC phases undergoes incompletely after primary BCC phase is enclosed by peritectic FCC phase

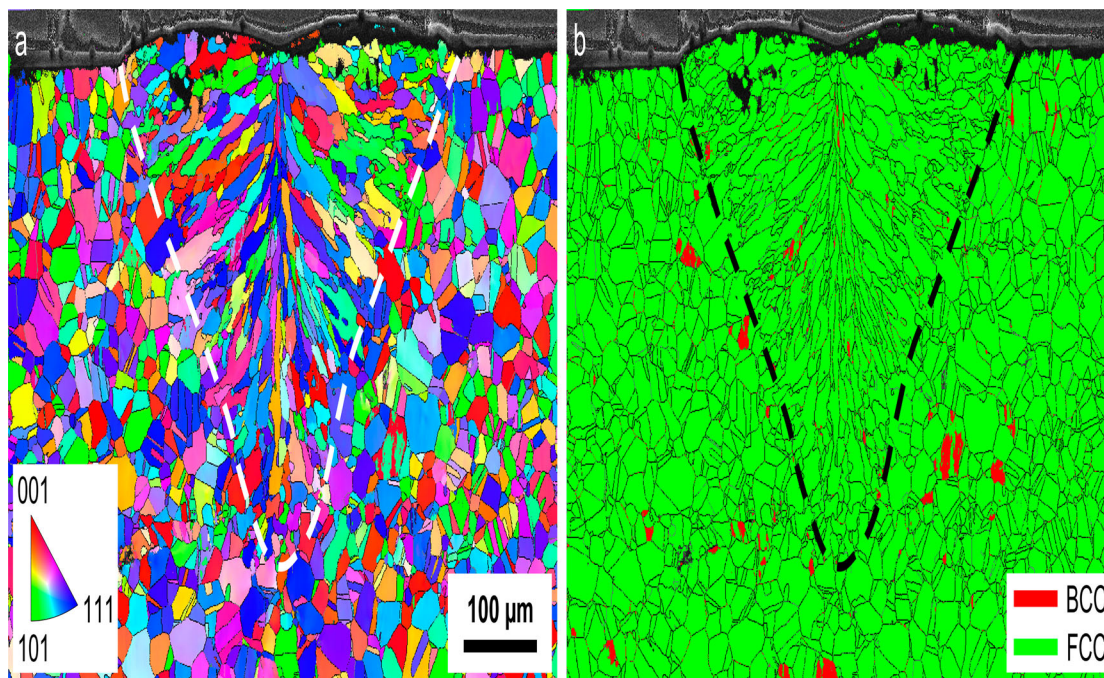


Figure 3. Microstructural characterization of fusion zone and heat-affected zone after laser spotwelding. (a) Cross-sectional EBSD image showing the grain-scale morphology after localized laser melting treatment. (b) Phase mapping of cross-sectional morphology at the same location of **a**, illustrating the distribution of BCC and FCC phases after laser melting treatment. BCC phase is found between grain boundaries both inside fusion zone and heat-affected zone.

in the nonequilibrium condition of rapid solidification. This is also consistent with the observable BCC peaks in the printed AM sample from in-situ XRD measurements and fast advancement of second moving boundary that follows the primary solid–liquid interface from high-speed X-ray imaging observation. As both BCC and FCC phases grow rapidly, solute diffusion between solid BCC and FCC phases declines quickly and slows down the transformation, resulting in the scattering of BCC phase at final FCC matrix. BCC phase is also observed in the heat-affected zone (HAZ). This corresponds to fast thermal accumulation and dissipation from melt pool to HAZ and results in partial melting of HAZ, which is evidenced by in-situ diffraction measurements by aiming focused synchrotron X-ray beam at HAZ (see Supplementary Material).

Discussion

For the peritectic solidification sequence, BCC phase precipitates first from liquid upon cooling and subsequently FCC phase forms at the expense of primary BCC and remaining Liquid phases as the three-phase region is observed for peritectic reaction due to thorough contact between phases (Figure 1). Amorphous liquid inclines to transform to primary BCC phase at the initial stage of solidification and FCC phase appears 4 ms later than BCC phase. The short peritectic incubation time suggests extensive three-phase contact and thermal undercooling

[21]. Quick transformation to FCC phase occurs at the sacrifice of Liquid and BCC phases as the halo rings and BCC crystalline spots become weak afterwards. The halo rings of Liquid phase and both BCC and FCC crystalline peaks stay observable beyond 200 ms, suggesting that three-phase region lasts much longer than what expect before [22]. The plateaus at lattice d -spacing curves of BCC and FCC phases and flat cooling rate curve after the emerging of FCC phase indicate the occurrence of isothermal peritectic phase transition in nonequilibrium condition upon rapid solidification. These provide direct and solid evidence for the peritectic reaction and transformation during laser AM of 304L SS and reveal the solidification sequence and phase structure of twisted FCC-BCC mix in the nonequilibrium condition of rapid solidification.

The real-time evolving of two solid–liquid interfaces observed in high-speed X-ray imaging coincides with the two stages of Liquid-to-BCC and Liquid-BCC-to-FCC phase transformation and provides quantifications of peritectic transition dynamics under far-from-equilibrium condition. First solid–liquid interface advancing into top region of fully-liquid spherical droplet is in line with the primary BCC phase that grows into Liquid phase (Figure 2b). Upon further cooling, second moving boundary comes up and grows at the same pace of ~ 0.14 m/s (Figure 2c). As the growth rate affects either γ -austenite or δ -ferrite phases advancing into Liquid favorably [12], the comparable growth velocities

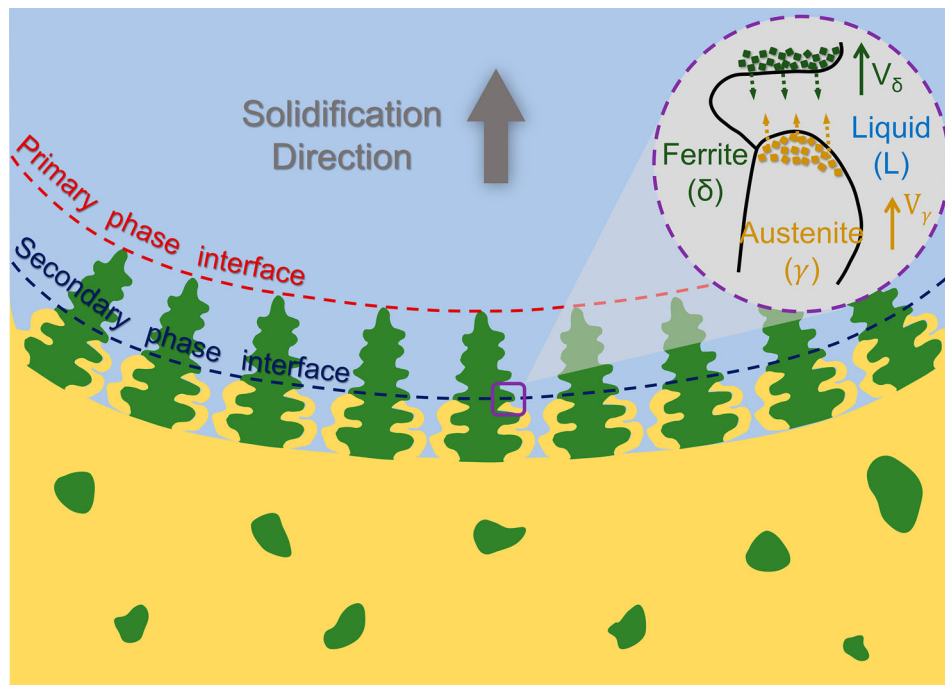


Figure 4. Schematic of local growth mode during peritectic phase transition. Local growth of BCC and FCC phases with effective latent heat dissipation and solute mixing during rapid solidification occurs during rapid solidification. Inset shows the local remelting of δ phase and vertical growth of γ phase at the triple point.

of FCC and BCC phases seem to suggest coupled peritectic growth mode during peritectic reaction. The rapid growth velocities are two to three orders of magnitude faster than those of confocal laser scanning microscopy observations [23,24], but close to those of containerless solidification processes such as electrostatic and electromagnetic levitations and drop tube [21]. The main difference is the lack of large enough undercooling as evidenced by the epitaxial growth of grain structure in the rapid solidification during laser treatment (Figure 3). Thus the likelihood of diffusionlessly massive transformation that requires large undercooling may be not possible in nonequilibrium solidification here [25].

A local growth mode of peritectic phase transition is proposed based on in-situ observation (Figure 4). The epitaxial growth of primary δ phase from support substrate and subsequent growth of γ phase removes the peritectic nucleation barrier and avoids solute pile-up at $L/\delta/\gamma$ three-phase point, suggesting that solute mixing can be effectively achieved under rapid cooling state [26]. As primary dendrite of δ phase grows into deep Liquid region and peritectic γ phase advances behind, local remelting of δ phase occurs due to microscale heat transfer and solute mixing (inset of Figure 4). Because the release of latent heat as a result of fast growth of γ phase changes local temperature gradient and greatly affects the thermal diffusion [24]. The local remelting reduces the solute diffusion to a large distance and cooperates with the fast dendritic growth, which is consistent

with temperature gradient dependent solidification rate [27]. Then γ phase rapidly thickens laterally because of effective solute and thermal diffusion. The existing of δ phase in the solidified matrix also illustrates the peritectic transformation is controlled by diffusion of solute from the Liquid phase to δ phase through the γ phase layer (Figure 3).

Conclusion

In summary, we directly observe the Liquid-to-BCC and Liquid-BCC-to-FCC peritectic transition in-situ and carefully quantify the real-time peritectic solidification with a combination of high-speed high-resolution high-energy synchrotron X-ray diffraction and imaging; isothermal plateaus in cooling paths of primarily-solidified BCC and subsequently-transformed FCC phases indicate the peritectic phase transition at the triple point; growth velocities of BCC and FCC phases both maintain an identical rising trend up to 0.14 m/s; Λ -shape cooling rate calculated by time-resolved FCC lattice d -spacing evolution peaks at 2.39×10^4 K/s. These in-situ observations suggest coupled BCC/FCC growth along the Liquid/BCC/FCC at three-phase region under nonequilibrium conditions with intense heat transfer and solute mixing during peritectic reaction and effective thermal diffusion at millisecond scale during peritectic transformation. These new findings contribute to improving understanding of peritectic solidification and

allow more accurate predictions on phase transformation dynamics and delicate control of solidified phase structure for scientific and engineering applications of metal additive manufacturing.

Disclosure statement

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

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