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Research Paper

Multi-scale microstructural investigation of a laser 3D printed Ni-based superalloy



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ABSTRACT

The heterogeneous microstructure of a laser 3D printed Ni-based superalloy was examined at multiple length scales. The sub-millimeter-sized columnar crystal grains are composed of micron-sized cellular colonies. The crystal grains grow in epitaxy with the substrate under the large temperature gradient and high cooling rate. The cell boundaries, decorated with γ/γ' eutectics, μ -phase precipitates and high density of dislocations, show enrichment of γ' forming elements and low-angle misorientations. Dislocations trapped in the intra-cellular regions are characterized as statistically stored dislocations with no detectable contribution to lattice curvature, and are the results of the interaction between dislocations and γ' precipitates.

1. Introduction

Laser additive manufacturing, which is commonly referred to as laser 3D printing (L3DP), has great potential for near net shape manufacturing as well as repair of gas turbine engine components composed of single crystal or directionally solidified Ni-based superalloys with high γ' content (> 60 %) [1]. Based on the powder feeding strategy, L3DP can be categorized into either direct energy deposition (DED) or powder bed fusion (PBF). Due to the focused heat source and reduced heat input, a steep temperature gradient, parallel to the building direction, is present during both the DED and PBF processes, facilitating epitaxial crystal growth with the base plate metal orientation. Simultaneously, heterogeneous microstructures with length scales ranging from nanometer to sub-millimeter are generated from the rapid solidification of the DED and PBF process [2-5]. These are otherwise not feasible through conventional methods of manufacturing. The high cooling rates inherent to L3DP severely suppress the growth of secondary dendrite arms, making it challenging to distinguish between cells and dendrites [6] in the absence of crystal orientation knowledge. Hence, the term "cell" is generally used to denote the cellular/dendritic structures in 3D printed alloys. The fine cellular structures,

accompanied by both elemental and dislocation partitioning, have been reported in a variety of 3D printed face-centered cubic alloys, including Al alloys [7], austenite stainless steels [2,8,9], high-entropy alloys [10] and Ni-based superalloys [11–14].

The simultaneous enhancements in strength and ductility of 3D printed alloys, compared to their conventionally manufactured counterparts, are attributed to such cellular structures [2,8]. There is however no consensus on the exact mechanism leading to the observed strengthening. In the absence of detailed characterization of the misorientations across them, the cell boundaries are treated as conventional high-angle grain boundaries (HAGBs), leading to a Hall-Petch relation based description of strength [2]. Some authors however argue that cell boundaries cannot be regarded as HAGBs, and attribute the observed strengthening to dislocation interactions (work hardening) together with elemental segregation (solid solution strengthening) and crystal grain refinement (if any) [10,15]. In this context, it is worth noting that most of such observations and discussions are based on austenite stainless steels, high entropy alloys and Ni-based superalloys with low γ' content (e.g. IN718 and IN625) [13,16]. However, only a few studies have been reported in laser 3D printed directionally solidified and single crystalline Ni-based superalloys with high Ti/Al

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Table 1Nominal compositions of the substrate and powders.

Composition (wt%)	С	Cr	Co	Мо	W	Al	Ti	Та	Ni
DZ125L substrate	0.12	8.74	9.54	2.21	6.46	5.03	3.18	3.96	Balance
DD4 powders	0.004	8.97	7.26	1.94	6.91	3.94	4.22	3.73	Balance

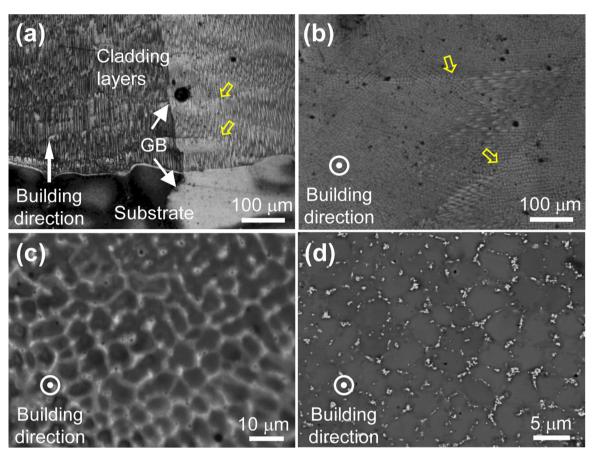


Fig. 1. The cellular microstructures in the DEDed superalloy. OM images collected from the (a) longitudinal section and (b) cross-sectional specimens. The yellow arrows in (a) and (b) denote the fusion lines. (c) An enlarged view of (b) showing the typical cell morphology. (d) BSE SEM image showing the dispersed precipitates along the cell boundaries from the cross-sectional view.

contents and thus high γ' volume fraction, especially without post processing heat treatment [11,17]. In particular, detailed knowledge of the dislocation type and distribution, and how the chemical inhomogeneity associated with the cellular structure influences the size, morphology, and phase of the strengthening precipitates is essential to understand the mechanical performance of 3D printed Ni-based superalloys. Keeping this in view, we report here the results of a comprehensive microstructural study on a Ni-based superalloy manufactured by DED technique.

2. Materials and methods

Gas atomized DD4 superalloy powders with a diameter range of $40\text{--}120\,\mu\text{m}$ were used as the feedstock for the DED process. An in-house developed co-axial laser cladding apparatus was employed and a 7 mm diameter 15 mm long cylinder was deposited on the (001) surface of a directionally solidified Ni-based superalloy DZ125L substrate using a scan protocol where the scanning direction of each layer was perpendicular to the previous one. The nominal compositions of DD4 powders and DZ125L substrate are listed in Table 1. The DED process was carried out with a fixed laser power of 230 W and a scan rate of 10 mm/s under an Ar protective atmosphere. Powders carried by Ar gas were

injected into the melt pool at a rate of $\sim 9 \text{ mm}^3/\text{s}$. More detailed information about the manufacturing process can be found elsewhere [18].

After the DED process, the specimens were cut from the cylinders. After mechanical polishing and electrical etching in an aqueous solution consisting of 30 vol% H₃PO₄, microstructural investigation was carried out under an optical microscope (OM, Carl Zeiss Axio Scope A1) and a scanning electron microscope (SEM, Hitachi SU6600) equipped with energy dispersive spectroscopy (EDS). Electron backscattered diffraction (EBSD) equipped in a focused ion beam system (FIB, FEI Helios 600 NanoLab DualBeam) was conducted for the study of grain structures. An EBSD measurement from the cross-sectional area up to $1350 \times 1150 \,\mu\text{m}^2$ was acquired with 5 μ m step size and post-processed by MTEX toolbox [19]. Synchrotron X-ray microdiffraction (μXRD) was conducted on beamline 12.3.2 at the Advanced Light Source of the Lawrence Berkeley National Laboratory. The cross-sectional specimen was mounted on the sample stage at a 45° incline angle relative to the polychromatic X-ray beam (5-24 keV). 3600 Laue Patterns (LP) were collected using a two-dimensional X-ray detector (DECTRIS Pilatus 1M) over an area of 60 \times 60 μm^2 with 1 μm step size and 0.5 s exposure time at each scan position, and subsequently indexed by using the custom-developed software package XMAS for mapping the orientation

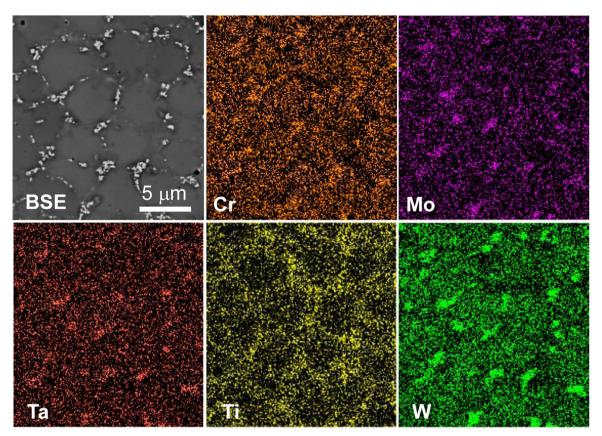


Fig. 2. EDS element distributions collected from the cross-sectional specimen.

deviations. Full details of the μXRD setup and LP indexing process can be found in Ref. [20,21]. The EBSD and μXRD specimens were fabricated by electrical polishing in a solution of 92 % alcohol and 8% perchloric acid at 25 V. In addition, the cross-sectional specimen was thinned with a twin-jet electro-polishing machine, and investigated under transmission electron microscope (TEM, 200 kV JOEL 2100F) to examine the microstructures at the nano-scale.

3. Results

Low magnification optical micrograph (Fig. 1a) of the longitudinal section of the specimen, shows the substrate/cladding layer interface and the layer-by-layer deposition patterns outlined by the fusion lines, which are also visible from the cross-sectional view in Fig. 1b. The thickness of each deposited layer was measured to be about 100 µm. The observation of grain boundaries (GBs), one of which is marked with white arrows in Fig. 1a, running across multiple deposited layers from substrate, implies epitaxial growth of the columnar grains. In each grain, columnar cells orient preferentially parallel to the building direction. From the cross-sectional view in Fig. 1c, these cell colonies appear equiaxed in the direction transverse to the building direction. The cells can therefore be viewed as long cylindrical rods with an average diameter of ~6 µm. Under the backscattered electron (BSE) mode of SEM, the cells are outlined by the high density of nano-sized particles with bright contrast at the boundaries (Fig. 1d). These regions are enriched in Cr, Mo, Ta, Ti and W, as revealed in the energy dispersive spectroscopy (EDS) maps (Fig. 2).

From the SEM image (Fig. 3a) and dark field TEM image (Fig. 3b) illuminated by the 100 reflection of γ' precipitates (Fig. 3c), two different morphologies of γ' precipitates are observed. In the intra-cellular regions, small, close-packed and neatly ordered near-cuboidal γ' precipitates with an average size of \sim 30 nm can be seen. Along the cell boundaries, however, rectangular γ/γ' eutectic phases, with a typical γ'

size of ~ 80 nm (width) $\times 200$ nm (length), aggregate in a comb-like morphology. Moreover, a topologically closed-packed (TCP) particle near the γ/γ' eutectic was identified as the μ -phase with rhombohedral crystal structure, as evidenced from the corresponding selected area electron diffraction (SAED) study in Fig. 3d.

From the out-of-plane inverse pole figure (IPF-Z) map obtained using EBSD in Fig. 4a, a broad grain size distribution is observed and the largest grain is close to 1 mm in diameter. Most of the grains in red reveal that their [001] directions are almost parallel to the building direction. Since the aim of the present study is to surrogate the repair of a turbine blade tip, the substrate surface normal is also along the [001] direction. Thus the deposited layers are epitaxial with the substrate. The uneven colors in the grain interior indicate the presence of intragranular orientation gradients, and the low angle grain boundaries (LAGBs) visible in the kernel average misorientation (KAM) map (Fig. 4b) confirm the existence of a sub-grain structure. It should be noted that the value of the extent of orientation variance between cellular colonies is barely accessible by conventional EBSD method [2] due to its poor angular resolution ($\sim 0.5^{\circ}$). Therefore, synchrotron μXRD technique with superior orientation resolution (< 0.01°) was employed [22].

As seen in the IPF-Z map of the synchrotron μ XRD study (Fig. 4c), the whole scanned area has a similar orientation, yet an isolated subgrain domain, in orange, deviates $\sim 12^\circ$ relatively to its surroundings. Since the sub-grain orientation variation is hardly discernable from the IPF-Z map, the misorientation angle of each scan spot relative to the average orientation of the whole scan is calculated and shown in Fig. 4d. The map shows the whole scan area is composed of a multitude of cellular colonies in distinctive colors. This suggests that the growth direction of each cell is slightly misaligned, while the orientation within each single cell is nearly identical. Consequently, LAGBs observed from the KAM map (Fig. 4e) delineate the cell boundaries and hence outline the size/shape of cell colonies, in agreement with the optical

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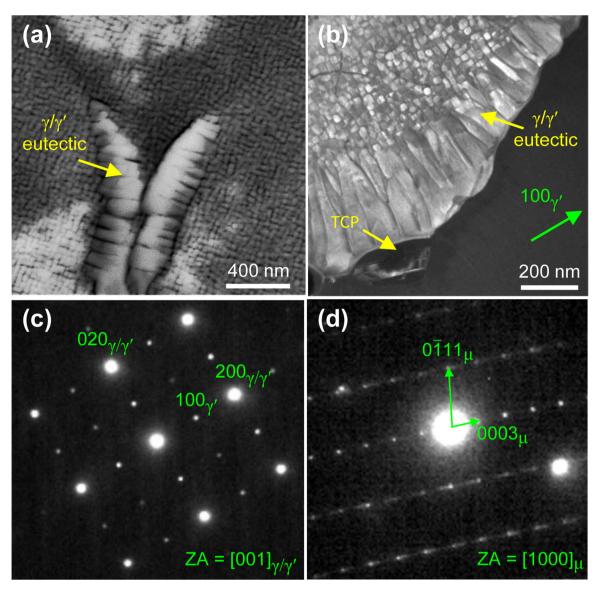


Fig. 3. The phase distribution in the DEDed superalloy. The combination of (a) BSE SEM and (b) TEM dark field images reveals two types of γ' morphologies. SEAD patterns evidence (c) the coherent γ/γ' microstructure and (d) the μ -phase precipitates.

microscopy and SEM images displayed in Fig. 1c and d. As seen in Fig. 4f, ten cells can be clearly distinguished from the nine peaks along the misorientation profile obtained through the blue horizontal line in Fig. 4e. The misorientation angle between each pair of adjacent cells ranges from 1° to 4°, while the low amplitude orientation variations (< 0.1°) in the intra-cellular scale are negligible.

In general, LAGBs are formed as dislocations arrange into energetically favorable configurations and are necessary for the accommodation of lattice curvatures [23]. To verify this, we examined the dislocation structure in the cross-sectional specimen using TEM in scanning transmission electron microscopy (STEM) mode. As seen in Fig. 5a, the cell boundaries, in dark contrast and denoted by hollow arrows, are decorated with γ/γ' eutectics and irregular shaped μ particles with average size of about 200 nm. An enlarged view displayed in Fig. 5b suggests large populations of dislocations tangling along the cell boundary. Since these dislocations are not homogeneously distributed, possibly because their mobility is impeded by μ particles, LAGBs, and coarse γ' precipitates of γ/γ' eutectics, it is difficult to estimate their density quantitatively. In the interior of the cells, however, the dislocation lines are distributed more homogeneously (Fig. 5c). Using the line-intercept method adopted by Norfleet et al. [24], the dislocation

density in the cell interior is estimated to be $\sim 1.4 \times 10^{14} \, \mathrm{m}^{-2}$.

4. Discussion

From the multi-scale characterization approach described above, the heterogeneous microstructure of the 3D printed superalloy, schematically displayed in Fig. 6, can be constructed. Although the crystal grains obtained in 3D printed superalloys are usually coarse (grain size of up to hundreds of microns or even at a centimeter scale for a single crystal [25]), they are composed of submicron to micron sized cellular structures, exhibiting elemental segregation, inhomogeneous distributions of dislocations and precipitates, and small misorientation between adjacent cells. The cell boundaries are enriched in γ' forming elements, resulting in larger γ' precipitates in the inter-cellular regions than in the cell interiors [26]. In some of the superalloys like the one studied here, particles with TCP phases are detected [27]. High and non-uniform density of tangled dislocations are observed along the cell boundaries. Dislocations also exist in the cell interior; although their density is lower than those along the cell boundaries, it is higher than those in the cell interiors of 3D printed stainless steels [2,8] and high entropy alloys [10]. Typically, the rapid cooling rate and the repeated heating/cooling

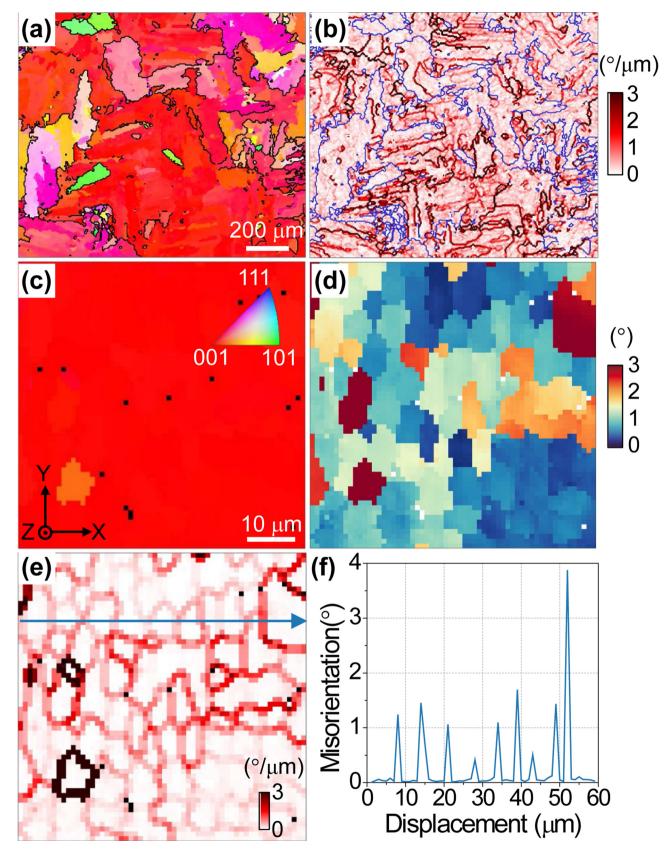


Fig. 4. Crystal orientation and misorientation analysis of cross-sectional specimen. (a) IPF-Z and (b) KAM maps of an EBSD measurement. The black and blue lines in (a) and (b) delineate the GBs with misorientation angles larger than 15° , respectively. (c) IPF-Z, (d) misorientation angle and (e) KAM maps of the μ XRD scan. (f) A linear profile of point-to-point misorientation along the horizontal arrow in (e).

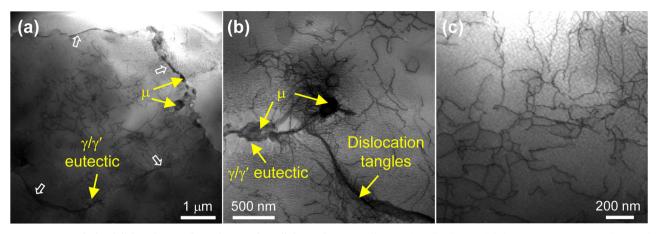


Fig. 5. STEM TEM analysis of dislocation configurations at the cellular scale. (a) A full view of a cell colony and dislocation configurations, (b) near the cell boundary, and (c) in the cell interior.

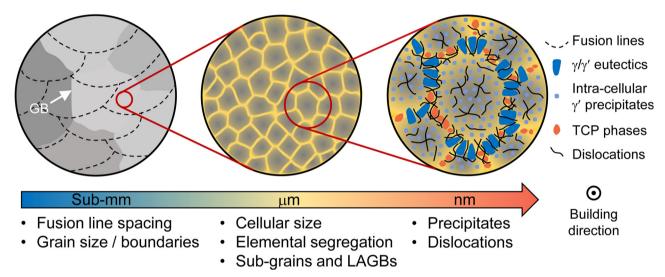


Fig. 6. Schematic illustration of the microstructures from the sub-millimeter to nanometer scales in the cross-sectional view of DEDed Ni-based superalloy.

cycles generated by the motion of the laser beam induce intense thermal stresses that can exceed the yield strength of the cladding materials [18]. Additionally, for γ' -strengthened Ni-based superalloy, the rapid precipitation of γ' will also introduce pronounced phase transition stresses due to the γ/γ' misfit. The superimposition of thermal stresses and phase transition stresses induce a large number of dislocations both in the cell center and in the intercellular region. A possible reason for the distinguished intra-cellular dislocation distribution of superalloys studied here from the 3D printed stainless steels and high entropy alloys is the strong hindrance to dislocation mobility caused by the γ' particles.

The cellular microstructure is due to the rapid solidification process during 3D printing. According to the undercooling criterion [28], solidification microstructure depends on temperature gradient (G), solidification rate (R), and undercooling temperature (ΔT) of the melt pool. Cellular solidification prevails if $G/R > \Delta T/D$, where D is the diffusion coefficient. In L3DP, G usually reaches $10^5 \sim 10^7$ K/m [29], which is at least 3 orders of magnitude higher than the temperature gradient in traditional casting conditions. As the cooling rate, i.e. the multiplication of G and R, is inversely proportional to the cell spacing (or primary dendrite arm spacing), the observed cell spacing ($\sim 6~\mu$ m) is larger than most of other reported superalloys fabricated by PBF ($< 1~\mu$ m) [11,12], suggesting that R is relatively low in the present study, although still 2 orders of magnitude higher than that for conventional casting conditions [29]. The resulting large G/R ratio facilitates the formation of

cellular structures. It is also suggested that a high *G* gives rise to strong thermo-capillary convection due to the surface-tension driven instability, or Benard-Marangoni surface instability [30], which promotes the ejected γ' forming elements to accumulate in the inter-cellular liquid, and, in turn, triggers the eutectic reaction $L \rightarrow \gamma + \gamma'$ in the final stage of solidification [26,31]. Since the γ' solvus rises with increasing content of γ' forming elements, inter-cellular γ' particles precipitate at a higher temperature and experience longer period over which they can coarsen [26]. Since superalloys with high W or/and Mo content show a strong tendency for µ-phase formation during conventional solidification, the μ-phase observed in this study is believed to nucleate directly from the inter-cellular liquid, i.e. primary µ-phase [32]. Moreover, as shown in Fig. S1 in the Supplementary Material, the lamellar features observed within the TCP particle imply the presence of growth faults, which may have been introduced by the thermal stresses that arise during the 3D printing process [32].

As for the layer-by-layer deposition process, partial remelting of the underlying layers is necessary for a strong metallographic bond between two adjacent deposit layers. Simultaneously, the unmolten layers also serve as the substrate for the epitaxial growth of the newly formed cells. However, affected by the flowing field, cell growth deflects toward upper stream and the deflection angles varies at different locations in the melt pool [33], leading to a slight orientation variation between different cells. On the other hand, although the solidification cell growth front aligns along the favored [001] crystallographic

direction, which is anti-parallel to the heat flux, the individual cells, regarded as cantilever beams, are anchored at the position of the solidus isotherm once the inter-cellular liquid starts to freeze and is subjected to bending moments and torques caused by thermal stresses [34]. Therefore, the accumulated misorientation is an outcome of the layer-by-layer building process. Because of the complex solidification process and the presence of plastic stresses, dislocations are also formed in the deformable γ channels and bow out between hard γ' precipitates. In addition, the complex solidification paths, including melting, partial re-melting, cyclic annealing, γ' dissolution and re-precipitation, lead to the inhomogeneous distribution of γ' precipitates, resulting in complicated interactions between the dislocations and the γ' precipitates. Thus the dislocations are distributed in the cells with random signs [35]. In other words, these dislocations are statistically stored dislocations (SSDs). This is why the lattice curvature is not observed in the intracellular regions using the µXRD technique.

5. Conclusions

In summary, heterogeneous microstructures, including fusion lines, columnar grains, LAGBs as well as the elemental distribution, phase constituents and dislocation configurations at the cellular scale are investigated in a DEDed superalloy. The epitaxial growth of cells and columnar grains are attributed to the partial remelting of underlying deposit layers and the maximum heat flow antiparallel to the building direction. Cell boundaries, identified as LAGBs, are decorated with γ/γ' eutectics, μ -phases and non-uniform dislocation arrangements, which are linked to the inter-cellular segregation of γ' forming elements during the DED process. In contrast to the cell structures in other 3D printed alloys with small to negligible volume fraction of strengthening precipitates, intra-cellular dislocation density in the γ' -strengthened superalloys is significantly higher but do not contribute to lattice curvature, and is attributed to the interactions between dislocations and γ' precipitates leading to the formation of SSDs.

Declaration of Competing Interests

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.

CRediT authorship contribution statement

Yao Li: Investigation, Formal analysis, Writing - original draft. Kai Chen: Conceptualization, Writing - original draft, Supervision. R. Lakshmi Narayan: Writing - review & editing. Upadrasta Ramamurty: Writing - review & editing. Yudong Wang: Visualization. Juncheng Long: Visualization. Nobumichi Tamura: Formal analysis, Data curation, Writing - review & editing. Xin Zhou: Resources.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the

online version, at doi:https://doi.org/10.1016/j.addma.2020.101220.

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