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Achieving Ultrahigh Fatigue Resistance in AlSi10Mg Alloy by Additive

Manufacturing

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18 Abstract: Since the first discovery of fatigue phenomenon in the late 1830s, the efforts to fight against fatigue failure have never been stopped by materials scientists and mechanical 19 engineers. Here we report a fatigue immunisation phenomenon in nano-TiB2 decorated 20 AlSi10Mg (NTD-Al) enabled by additive manufacturing. This fatigue immunisation 21 22 mechanism benefits from the 3D dual-phase cellular nanostructure, which acts as a strong 23 volumetric 3D dislocation nanocage to prevent the localised damage accumulation, thus 24 inhibiting fatigue crack initiation. The intrinsic fatigue strength limit of NTD-Al was proven to be close to its tensile strength through *in situ* fatigue test of a defect-free micro-sample, 25 26 where sign of neither crack initiation nor accumulated fatigue damages was observed. To demonstrate the practical applicability of this mechanism, the printed bulk NTD-Al achieved 27

record high fatigue resistance tested by ASTM standard, which was more than double those of other additive manufacturing Al alloys and surpassed those of high-strength wrought Al alloys. A paradigm of achieving high fatigue resistance through additive manufacturing phase engineering has been established from nanoscale structural design to the fabrication of geometrically intricate industrial components, which will be extended to the design and development of other fatigue-resistant metals.

Fatigue resistance is a critical design criterion of structural components, as it is estimated that 37 over 80% of engineering failures are due to fatigue¹. Insufficient fatigue strength of metals has 38 remained an unsolved problem for almost two centuries, where conventional metals exhibit 39 fatigue strength generally lower than half of their tensile strength. In particular for Al alloys, 40 41 which are the second widely used metals in industry, the typical fatigue strength ratio (fatigue 42 strength limit/tensile strength) is only about $1/3^2$. This leads to a very important fact, but seldom 43 mentioned in industry that for all the moving Al components suffering from cyclic loads, the load efficiency of materials utilisation is roughly estimated to be less than 20%, considering 44 the safety factors and the inhomogeneous stress distribution in engineering components. 45 Despite the metallurgical defects introduced during manufacturing, the intrinsic mechanism for 46 47 the lower fatigue resistance is the irreversible dislocation slips taking place in particular grains 48 due to the severe heterogeneous strain distribution in polycrystalline metals, therefore enabling 49 localised damage accumulation that initiates fatigue cracks. Recent efforts have shown that 50 through microstructure design, fatigue resistance of metals can be dramatically enhanced, e.g. 51 bone-like nano-laminated substructure arresting fatigue cracks in steel³, fatigue-induced precipitation strengthening weak grain boundaries in Al alloy² and highly oriented nanoscale 52 twins constraining reversible cycling damages in copper⁴. 53

Laser powder bed fusion (LPBF), as one of the most widely used metal additive manufacturing (AM) techniques, is known for its capability of directly fabricating geometrically complex components with free topology optimisation design. It is worth more attention that the rapid cooling rate of LPBF also provides unrivaled material microstructure

58	designability down to nanoscale. Here we report a fatigue immunisation mechanism in
59	AlSi10Mg alloy fabricated by LPBF AM. We demonstrate that continuous 3D dual-phase
60	cellular nanostructure (3D-DPCN) of the LPBF AM AlSi10Mg alloy is immune to fatigue by
61	inhibiting localised damage accumulation and fatigue crack initiation. This fatigue
62	immunisation mechanism is distinct from the fatigue mechanisms of conventional metals that
63	have been studied for over a century. Through an <i>in situ</i> micro-fatigue experiment, it is found
64	that the intrinsic fatigue strength limit (> 10^7 cycles) of a defect-free sample is close to its
65	tensile strength, and the post-fatigue sample is observed to be crack-free and absent of localised
66	fatigue damage accumulation, where a fatigue ratio of ~ 0.9 is achieved. As a proof of the
67	fatigue immunisation mechanism in bulk sample, we fabricated a record high fatigue-resistant
68	nano-TiB2-decorated AlSi10Mg (NTD-Al) alloy through optimizing printing defects. The
69	fatigue strength limit of the NTD-Al alloy, following ASTM standards, was the best among all
70	Al alloys including the conventional high-strength wrought Al alloys with limited metallurgical
71	defects, and more than double those of other reported AM Al alloys ^{2,5-22} (Fig. 1A and B). It is
72	worth mentioning that the NTD-Al alloy has been applied to fabricate large thin-walled
73	structures, including fan blades of aircraft engine designed by virtue of its high fatigue strength,
74	and successfully passed the qualifying fatigue test (Fig. 1C). These findings not only provide
75	a novel path to designing fatigue resistant alloys in metal research community, but also raise a
76	new opportunity to weight reduction by increasing the materials load efficiency of moving
77	components. Combined with the various advantages of 3D printing, such as topological
78	optimisation (Fig. 1D), this work would have everlasting impacts on modern industry in terms

of component weight reduction, and influence future society in terms of low-carbon emission
from aircrafts and vehicles with the development and mass application of never-fatigue metals.





Fig. 1 Achieving high fatigue strength of the AM NTD-Al alloy. (A) Number of cycles to failure plotted against the maximum stress of AM NTD-Al (stress ratio (R) = 0.1 and -1), compared with other reported AM AlSi10Mg and AlMgScZr alloys^{5-9,14-22}. (B) Fatigue limit to tensile strength ratio vs. fatigue strength of AM NTD-Al, compared with Al alloys, $R = -1^{2,10-13}$. (C) Multi-scale specimen validations for unprecedented fatigue performances. (D) Multiscale optimisation strategies for performance improvement.

89	We applied LPBF-AM to fabricate the samples using AlSi10Mg powders decorated with
90	TiB ₂ nanoparticles ²³ , as demonstrated in Extended Data Fig. 1. Samples for standard fatigue
91	tests were printed by ProX DMP 200 made by 3D Systems (Extended Data Fig. 2). The
92	optimised powder chemical composition and printing parameters are given in Extended Data
93	Table 1. Micro-computed tomography (μ -CT) analysis was used to investigate the printing
94	defects in the NTD-Al, where uniformly distributed nearly spherical pores were found
95	throughout the sample (Fig. 2A). The widely reported detrimental defects in common AM
96	AlSi10Mg, the irregular shaped lack of fusions (LOFs, often in size of hundreds of
97	micrometers, Extended Data Fig. 3A and B), resulted from the improper fusion between
98	successive tracks or layers due to insufficient energy density ²⁴ were absent in our NTD-Al
99	sample. Electron backscatter diffraction (EBSD) showed a quasi-equiaxed grain morphology
100	with an average size of approximately 2–3 μ m (Fig. 2B). Further characterisation revealed a
101	typical DPCN that consisted of a network of solidification cellular structure with an average
102	diameter of ~ 500 nm and α -Al inside the cellular structure (Fig. 2C). Continuous 3D-DPCN
103	were revealed by the tomographic reconstruction of backscattered electron (BSE) images from
104	sections sliced by focused ion beam (FIB) (Fig. 2D). Close-up transmission electron
105	microscopy (TEM) images showed that the cellular structure was composed of polycrystalline
106	Si grains of several to tens of nanometers in size and random crystallographic orientations (Fig.
107	2E and F). The main component of the 3D-DPCN was Si phases containing a few TiB_2
108	nanoparticles that could be observed in the energy dispersive spectroscopy (EDS) maps (Fig.
109	2F and G). EOS M280 was also used to print NTD-Al samples, showing no notable difference

- 110 compared to ProX DMP 200 (Extended Data Fig. 3 and 4), validating the applicability of our
- 111 design strategy in different printing scenarios.





Fig. 2 Microstructure of the AM NTD-Al alloy. (A) µ-CT analysis showing the spatial and 113 size distributions of printing defects, where the largest defect is measured to be 73 μ m, only 114 about 1/3 of the LOF defects in AM AlSi10Mg alloy without TiB₂ decoration (Extended Data 115 Fig. 3A and B). (B) Grain morphology of the printed sample. (C) A network of solidification 116 cellular structure with an average diameter of \sim 500 nm. (D) Continuous 3D Si cellular structure 117 revealed by BSE/FIB tomography. (E) Cellular structure consisting of nanosized Si phases 118 119 revealed by TEM. (F-G) TEM image and Al, Si, Mg and Ti EDS maps illustrating that the 120 distribution of Si coinciding with the cellular structure. Inset showing the selected area electron diffraction (SAED) pattern illustrating Si existing as nanosized phases with random 121 orientations. 122



131	element solutions, precipitations, grain boundaries et.al, and thus determines the intrinsic
132	fatigue behaviour. The size of the micro-samples fabricated using FIB was chosen to be gauge
133	length of ~ 14 μm and cross section of ~ 4 \times 4 $\mu m^2,$ which contains around a thousand cellular
134	nanostructures (Fig. 3A). Micro-tensile tests were first performed to find out the intrinsic
135	mechanical response without the printing defects interference, where yield strength of ~ 470
136	MPa and ultimate tensile strength of ~ 550 MPa were measured (Extended data Fig. 5D, the
137	in-situ movie available in Supplementary Video 1). The micro-tensile samples exhibited ductile
138	behaviour with significant necking accompanied by gradual reduction of the cross-sectional
139	area, as shown in Supplementary Video 1. The stress-strain curve was smooth without any
140	strain burst or sudden load drop. It should be noticed that the dislocation slip bands $\frac{29}{29}$
141	conventionally appeared during the <i>in situ</i> micro-test of metallic materials were not observed
142	(Extended Data Fig. 5D), indicating that localised dislocation slips were suppressed. According
143	to the tensile test results, two series of fatigue tests were conducted, one with maximum stress
144	at the yielding stress, the other near the ultimate tensile stress, with sinusoidal cyclic loading
145	tests at 100 Hz (Fig 3A, inset) and stress ratio of $R = 0.1$. Surprisingly, neither sign of damage
146	accumulation (such as the persistent slip bands, PSBs) nor crack initiation could be observed
147	on the sample surface after 10 ⁷ cycles loading at a maximum stress of 470 MPa (Fig. 3B, the
148	in situ movie available in Supplementary Video 2). By contrast, when micro-sized Ni crystal
149	was examined by a similar in situ fatigue test, slip band markings characteristic of PSB
150	formation were observed ³⁰ . The transmission Kikuchi diffraction (TKD) and local
151	misorientation map of thin-section sample prepared from the fatigued area in Fig. 3C-E
152	demonstrated that very limited residual strain was generated, indicating strain localisation was

153	totally restrained by the cell walls after long-term cyclic loading. These results suggest that the
154	3D-DPCN possesses an ultrahigh intrinsic fatigue strength limit above 470 MPa, which is more
155	than doubled the highest fatigue strength limit of reported Al Alloys. The corresponding fatigue
156	strength/tensile strength ratio is above 0.85, which has never been reported in any other metals
157	to the best of our knowledge (Fig. 1B). When the maximum stress was raised to 520 MPa,
158	detectable strain accumulation damage gradually developed in the form of necking during the
159	course 10^5 loading cycles (after which the test was stopped), while the intactness of the sample
160	was still preserved (Fig. 3F and G, and see Supplementary Video 3). This result further proves
161	that strain damage accumulation only generates in a reluctant manner at stress close to the
162	tensile strength, which deserves future investigation. A same test was also conducted on LPBF-
163	AM AlSi10Mg without TiB ₂ decoration, where a fatigue strength limit ratio exceeding 0.85
164	was verified (Fig. S1 and see Supplementary Video 4), confirming that the intrinsic fatigue
165	performance arises from the 3D-DPCN. These findings reveal a new route to the design of
166	fatigue immunisation metals through additive manufacturing phase engineering.



168Fig. 3 In situ micro-fatigue testing results. SEM images of the micro-sample (A) before the169fatigue test and (B) after 10^7 loading cycles at the maximum stress of 470 MPa. Inset: loading170curve showing 50 cycles in 0.5s (part of the total loading curve as an example) of stress from171~ 50 MPa to ~ 470 MPa. (C) Band contrast image of the sample after the fatigue test, with (D)172TKD map and (E) local misorientation map. (F) SEM images of the micro-sample before the173fatigue test and (G) after 10^5 loading cycles at the maximum stress of 520 MPa. Inset: loading

174 curve showing 50 cycles in 0.5s of stress from \sim 50 MPa to \sim 520 MPa.

175	To validate the functioning of the fatigue immunisation mechanism in bulk NTD-Al,
176	fatigue tests with ASTM/ISO standard specimens (Extended Data Fig. 2) were performed on
177	the as-printed NTD-Al alloy. Despite the relatively low ductility of approximately 6%-8%
178	(Extended Data Fig. 6) and the abundance of printing defects (Fig. 2A), the fatigue strength
179	limits of the NTD-Al alloy reached 260 MPa (pull-pull fatigue testing, stress ratio $R = 0.1$;
180	printed by ProX DMP 200) and 250 MPa (rotary bending fatigue testing, stress ratio $R = -1$;
181	printed by M280), which were more than double those of reported AM AlSi10Mg and high-
182	strength AM AlMgScZr alloys (Fig. 1A) ⁵⁻⁹ ,14-22. The rotary bending fatigue strength limit of
183	250 MPa (R = -1) even exceeded those of conventional 2XXX, 6XXX and 7XXX series
184	wrought Al alloys (Fig. 1B) ^{2,10-13} . These findings indicate the vast potential applicability of our
185	alloy for the lightweight structures necessary in industries, where the fatigue property is the
186	key design criterion. Furthermore, the intrinsic fatigue strength limit of defect-free samples
187	almost doubled the macro-fatigue strength, denoting that there is plenty space for further
188	enhancement of fatigue resistance as shown in Fig. 1B.

The fatigue testing results illustrate that the fatigue strength limit of the ASTM standard 189 NTD-Al samples almost doubled those of traditional AM AlSi10Mg alloys but was still half 190 lower than that of the defect-free micro-size NTD-Al sample. Previous literature has shown 191 192 that the fatigue strength limit of macro-size 3D printing samples is strongly associated with the largest printing defects $\frac{24,31}{2}$. μ -CT results showed that obvious LOF defects with irregular 193 morphology were distributed throughout the AM AlSi10Mg alloys (Extended Data Fig. 3), 194 195 while nearly spherical pores instead of LOF defects were uniformly distributed throughout the 196 NTD-Al samples (Fig. 2A). Meanwhile, the size of the largest defect in AM NTD-Al alloys

197 was reduced to no more than one-third of that of the AM AlSi10Mg alloys (Extended Data Fig. 198 3). As the stress concentration from the defects is a crucial factor related to the fatigue 199 resistance, the stress concentration factors of the largest defects drawn from the µ-CT results were estimated by the finite element method (FEM) in the AM NTD-Al and AM AlSi10Mg 200 samples, respectively (Extended Data Fig. 7). The stress concentration factors of AM NTD-Al 201 202 were generally between 2.2 and 3.0, while those of the irregular LOF defects in AM AlSi10Mg 203 were 4.3 to 7.9. The greatly decreased pore sizes and the optimised porosity morphology are 204 primarily responsible for the superior fatigue-resistance of AM NTD-Al compared with conventional AM AlSi10Mg. 205

We investigated the macro-specimens after 10^7 fatigue cycles at a maximum stress of 260 206 MPa (R = 0.1) to assess the microstructural features after high cycle fatigue test. The fatigued 207 area of the sample ($2 \text{ mm} \times 2 \text{ mm} \times 6 \text{ mm}$ in the middle of the specimen) was first characterised 208 209 by μ -CT (Fig. 4A). Compared with the initial sample (Fig. 2A), no detectable changes in the 210 defect characteristics were found, including the size, morphology and distribution of pores. 211 Notably, no obvious cracks were observed in the characterised region after fatigue testing (Fig. 212 4A). However, as shown by the fracture surface of samples that failed at higher stress 213 (Extended Data Fig. 8), the fatigue cracks usually initiate from the site of the largest defect 214 near the surface of the sample, the size of which is in agreement with the μ -CT results. 215 Therefore, two of the largest pores (P1 and P2) near the sample surface and two of the largest 216 pores in the middle of the sample (P3 and P4) were selected for high-resolution µ-CT scanning of 0.5 µm voxel size. No cracks emerged from these pores as shown in the insets of Fig. 4A, 217 218 confirming that the crack initiation was entirely suppressed even around the largest pores. In 219 order to reveal the fatigue immunisation mechanism of our macro samples, two TEM thin foils were lifted out from the fatigued areas using FIB to characterise the fatigued microstructure. 220 221 One was from a randomly chosen area and the other was from the stress concentration area 222 next to P1, which was located and exposed by μ -CT-guided laser polishing (Fig. 4B, details of the process can be found in Supplementary Information Fig. S2). The latter was lifted out at 223 224 the edge of P1 on the plane parallel to the loading axis (Fig. 4C). Fig. 4D to F and Extended 225 Data Fig. 9 showed that the Si cellular structure was not destroyed after fatigue loading. Even 226 through the material at the edge of P1 was exposed to much larger cyclic loading due to the stress concentration effect, no notable changes were observed in the Si cellular structure (Fig. 227 4D). Under the applied stress of 260 MPa (fatigue limit of macro-sized sample), stress 228 229 concentration of over 500 MPa estimated by FEM (Extended Data Fig. 7D) would be 230 developed around the spherical pores, indicating that the critical stress of fatigue immunisation 231 without damage accumulation is around 500 MPa, in agreement with our micro-fatigue test 232 results (sample passed fatigue test at maximum stress of 470 MPa and damage accumulation 233 manifested at maximum stress of 520 MPa, Fig. 3B and G).



235

Fatigue immunisation mechanism

Fig. 4 Fatigue mechanism investigation on the specimen after 10⁷ fatigue cycles at a 236 237 **maximum stress of 260 MPa (\mathbf{R} = 0.1).** (A) No obvious cracks were observed in the specimen 238 after fatigue testing; no cracks emerged from the largest pores (P1-P4); P1 and P2 were located 239 close to the sample surface while P3 and P4 were in the middle of the sample. (B) μ -CT scanning cross-section of P1. (C) Locating and exposing the cross-section of P1 by µ-CT-240 guided laser polishing. (D) TEM characterisation of intact Si cellular structure in the region 241 242 next to P1 (TEM foil lifted out from the rectangular region in Fig. 4C). (E-F) EDS maps illustrating the distribution Al and Si elements. (G-H) Individual dislocations distributed inside 243 one Si nano-cellular structure. Schematics showing (I) the conventional fatigue damage 244 originating from PSB and (J) the fatigue immunisation mechanism of 3D-DPCN structure. 245

246	The <i>in situ</i> fatigue test and the particular post-fatigue microstructure of the bulk sample
247	prove a cyclic response totally immune to fatigue damage accumulation in our printed alloy.
248	Based on the above analysis, we rationalised that the defects optimisation and 3D-DPCN were
249	responsible for the high fatigue resistance of the LPBF-AM NTD-Al alloy. The 3D-DPCN
250	hampers the conventional strain localisation associated with irreversible damage during fatigue
251	loadings (Fig. 4I), such as persistent slip bands, veins, and dislocation cells, which were widely
252	observed in various coarse-grained metals ^{32,33} . Instead, individual dislocations were observed
253	inside the Si nano-cellular structure (Fig. 4G and H). This coincides with the results observed
254	in copper film that as the grain reduced to below 1 μ m, the generation of dislocation
255	substructures was inhibited and only individual dislocations were formed after fatigue test ³⁴ .
256	Similar microstructural fatigue features have also been observed in other ultrafine-grain
257	alloys ³⁵ , where the limited space for dislocation movement inhibits the formation of fatigue
258	dislocation patterns, resulting in increased resistance to crack initiation and high fatigue
259	resistance. For instance, an ultrafine-grained high-entropy alloy has been found to exhibit a
260	high fatigue ratio of approximately 0.62 times its ultimate tensile strength ³⁶ . Besides, A recent
261	in situ TEM compression test on AM AlSi10Mg showed that during deformation, the
262	dislocations generated were constrained inside the cells, where no obvious penetration through

the cell boundaries was observed²⁸. These results provide microstructural evidence confirming that the 3D nano-cellular Si phase of approximately 500 nm in size acts as a strong volumetric 3D dislocation barrier nanocage that traps dislocation movement inside during fatigue load. By this manner, long-range dislocation slips and dislocation reorganisation were strongly inhibited during fatigue loading, thereby preventing the formation of strain localisation fatigue damage (Fig. 4J). Thus, a fatigue strength limit close to its tensile strength can be guaranteed without fatigue damage accumulation and crack initiation.

The 3D-DPCN shows a characteristic fatigue response with no shearing or damage 270 accumulation below the critical stress, which has significant advantages in industry compared 271 with conventional alloys where the unpredictability of fatigue damage accumulation raises 272 great uncertainty. Based on our work a paradigm of achieving high fatigue resistance through 273 274 additive manufacturing phase engineering has been established from nanoscale microstructural 275 design to the fabrication of geometrically intricate industrial components. Utilizing in situ 276 micro-scale tests, the intrinsic fatigue strength limits of the 3D-DPCN can be measured, the 277 results of which could be incorporated with defects distribution from the CT analysis and stress 278 distribution from the simulation of industrial components, so that the fatigue response of an 279 AM component can be predicted to assist performance-oriented processing optimisation. One 280 such example of meter-scale engine blade design is given in Fig. 1C and D. In this work, we reported a defect-optimised bulk AM NTD-Al alloy that has a record 281 high fatigue strength that greatly surpassed that of previously reported AM Al alloys and 282 conventional high-strength wrought Al alloys obtained by different thermal-mechanical 283

284 processes. A novel fatigue immunisation mechanism was revealed in Al-alloys prepared by

LPBF-AM, where the inherent 3D-DPCN network resulted from the rapid solidification is able 285 to confine fatigue damage via acting as a strong volumetric 3D dislocation barrier cage to 286 prevent the localised irreversible damage accumulation during fatigue cyclic loading. Further 287 enhancement of the fatigue resistance of NTD-Al is expected by decreasing the printing defect 288 size to the sub-micron range or by eliminating defects through future technological innovations. 289 290 Our anti-fatigue paradigm has also been validated in industry scale components, including 291 aircraft door hinges and engine blades. Furthermore, this fatigue damage immunisation mechanism enabled by additive manufacturing phase engineering approach could also be 292 293 extended to the design of other fatigue-resistant dual/multi-phase alloy systems (another example is given in supplemental materials) and to other additive manufacturing techniques 294 295 including electron beam melting and directed energy deposition.

297 Methods

298 **Powder fabrication**

The nano-TiB₂-decorated AlSi10Mg powder was fabricated by an *in situ* salt-metal reaction method and gas atomisation¹⁴. Extended Data Fig. 1 shows the spherical morphology of our *in situ* synthesised powders, which ensures a much better flowability. The flowability of our powders was measured to be ~ 20 s/50 g through Hall flowmeter measurement. It can be observed from the magnified image of the cross section of powder that the *in situ* synthesised TiB₂ nanoparticles are homogeneously distributed in the powder (Extended Data Fig. 1B and C).

Previous works shows that TiB₂ particles can ameliorate the laser printing process through 306 (a) increasing the average laser energy density¹⁴; (b) modifying the solidification path by 307 promote grain nucleation and mitigate solidification defects^{23,37-39}; and (c) stabilizing melt pool 308 309 fluctuations and preventing liquid droplet coalesce, which result in the elimination of large spatters and the reduction of printing defects^{23,40}. As shown in Extended Data Fig. 1E and F. 310 311 the laser reflectivity of Al-Si-Mg alloy can be reduced by $\sim 40\%$ with 5% addition of TiB₂, while the K-M absorption factor was raised by ~ 220%. With the addition of TiB₂ particles, the 312 313 detrimental LOF defects have been eliminated (Fig. 2A and Extended Data Fig.3). The 314 chemical composition is shown in Extended Data Table 1.

315

Laser powder bed fusion additive manufacturing

LPBF additive manufacturing of NTD-Al samples for standard fatigue test was performed using 3D Systems Prox DMP 200 and EOS M280 selective laser melting machines. The fan blades of aircraft engine were manufactured by a customised BLT-S500 selective laser melting machine. An inert, high-purity (\geq 99.99%) argon gas atmosphere flow with an oxygen content of < 600 ppm was used throughout the process to minimise oxidation. The AM processing was completed at room temperature without applying heat to the build plate. The printing parameters used in our work were listed in Extended Data Table 1. The samples consisted of 3 mm × 21 mm × 105 mm and 12 mm × 28 mm × 130 mm blocks for the fatigue tests; 5 mm × 5 mm blocks for microstructure characterisation; and 2 mm × 10 mm × 60 mm blocks for the tensile tests. Images of the as-printed samples are shown in Extended Data Fig. 2.

326 Sectioning and sample preparation

As-printed samples were removed from the build plates by wire electrical discharge machining. The fatigue samples were polished using 400, 800, 1200 and 2500 grit sandpaper consecutively and then with 5 µm diamond suspensions, which ensured a deformation-free surface. The scanning electron microscopy (SEM) and EBSD samples were polished with 400, 800, 1200 and 2500 grit sandpaper. Final polishing of these samples was conducted with the diamond suspensions, followed by ion polishing in a Leica EM TIC 3X. Some of the polished samples were etched using Keller's etchant to reveal the 3D cellular Si structure (Fig. 2C).

The TEM and TKD lamellas were prepared in a Tescan Lyra3 FIB/SEM microscope. A protective platinum layer was deposited at 30 kV and a current of 200 pA. FIB trenches were roughly milled under 30 kV and 10 nA. Cross-sections were finely polished using a 3 nA current. After lifting out the sample and welding it to a Cu TEM grid, the lamellas were thinned under 30 kV, with the current reduced in the sequence of 1 nA, 500 pA, 150 pA and 50 pA. Final polishing was conducted at 5 kV and a 50 pA current.

340 Microstructure and defect characterisation

The microstructures of the as-fabricated alloys were characterised with a SEM (Tescan Lyra3) equipped with EBSD (Oxford Nordlys Max3). The EBSD maps were recorded with step size of 0.1 μm. The orientation evolution of the fatigued samples was characterised using TKD with step size of 30 nm (Oxford Nordlys Max3). TEM (FEI Tecnai G2 operating at 200 kV) was used to examine the Si cellular structure.

Reconstruction of printing defects

347 X-ray μ -CT was performed using Xradia 510 Versa (Zeiss) to analyse the printing defects 348 inside the SLM-printed samples. A voxel size of 3.0 μ m was selected to allow the large 349 specimen volumes (up to 2 mm × 2 mm × 6 mm) to be imaged. High-resolution μ -CT scanning 350 (0.5 μ m voxel size) was further performed on localised regions. Reconstruction of the scanned 351 volumes was conducted using Dragonfly software. The defect population was resolved with 352 respect to the position and volume.

353 **Reconstruction of 3D dual-phase cellular nanostructure**

354 To obtain 3D volumes of the studied materials, SEM was coupled with the FIB milling 355 technique (Lyra3 Tescan). The angle between the FIB and SEM columns was 55°. Before 356 running the acquisition, a Pt layer $(10 \times 10 \times 1 \ \mu m)$ was deposited on an area of interest by ion beam deposition (IBD) to smooth out the surface roughness and protect the target surface. A 357 micropillar ($10 \times 10 \times 20 \ \mu m$) was prepared for the 3D acquisition. After acquiring an BSE 358 359 image at the side surface of the micropillar, a 15 nm step of material was removed using FIB. 360 Serial FIB sectioning was done with a current of 1 nA at 5 keV. The following BSE 2D image 361 was acquired and this process was repeated several hundred times to obtain an 3D volume with a resolution of 15 nm. The collected 2D images of the elemental-Si contrast were reconstructed
 by Dragonfly software.

Tensile and Fatigue test

Dog-bone tensile testing specimens were machined according to the ASTM-E8M standard. The tests were carried out on a Zwick/Roell Z100 testing machine at a constant strain rate of 10^{-4} s⁻¹.

368 Fatigue testing specimens were machined according to the ASTM E466-21 standard for 369 pull-pull fatigue test, R = 0.1 (Extended Data Fig. 2C) and the ISO 1143-2008 for rotating bending fatigue test, R = -1 (Extended Data Fig. 2D), respectively. High-frequency fatigue 370 testing machines were used for the rotating bending fatigue test (R = -1) (Giga Quad YRB200; 371 372 Yamamoto Corporation, Japan) and the pull-pull fatigue test (R = 0.1) (Changchun Qianbang 373 Corporation, China). The cyclic loading followed a sinusoidal wave. A frequency of 80 Hz was used for both fatigue tests. The tests were conducted in air at room temperature (approximately 374 375 21 °C and 30% relative humidity). The high cycle fatigue tests were terminated after 10^7 cycles, 376 with the surviving samples being identified as run-outs.

377 Micro-sample fabrication

For the *in situ* micro-tests, "T"-shaped micro-specimen geometry was fabricated as shown in Extended Data Fig. 5. The gauge regions were rectangular in cross-section with approximate dimensions of $4 \times 4 \ \mu\text{m}^2$, while the gauge lengths were approximately 14 μ m. The "T" head region was approximately 15 μ m in width and the same thickness as the gauge section. During the test, the "T" head would be lifted from the underneath at two sides to induce tensile load at 383 the gauge region. Chamfering was introduced at the two ends of the gauge region to reduce 384 local stress concentration.

The micro-specimens were fabricated at the top edge of the printed sample after mechanical surface polishing, so that the top and front views of micro-specimen could be exposed to laser and ion milling. The first step of fabrication was rough-milling the geometry using a Gemini2 (Zeiss) femtosecond laser operated at 4.5 W with a repetition rate of 1.0 kHz. After laser milling, FIB in Tescan Lyra3 SEM-FIB was used to obtained the final gauge geometry. Surface imperfections and curtaining, as well as tapering of the specimen, were minimised by performing a low-current final polishing step at 30 kV, 150 pA.

392 Micro-testing methodology

The micro-tensile and fatigue tests were performed utilizing an Alemnis Standard Assemble platform inside of a Tescan Lyra3 SEM. A customised diamond tensile grip was used to induce tensile load to the specimen (Extended Data Fig. 5A and B). During the FIB polishing of "T"shape geometry, several adjustments was made to ensure full contact between the "T"-head and the tensile grip at the two sides, so that the tensile load will be applied along the gauge axis.

For the tensile test, a nominal strain rate of 10^{-3} s⁻¹ was applied using the displacementcontrolled mode. For the fatigue test, the cyclic loading was introduced through applying sinusoidal displacement signal at 100 Hz. The stress level was controlled by adjusting the input displacement value and amplitude to perform the R=0.1 fatigue test. The load and displacement data were tracked and recorded at 1kHz frequency. The displacement signal was recalibrated after each 10^4 loading cycles (100 s) to minimise the effect of mechanical drifting.

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528	Methodology: YW, CD, HW, HL, MW, QS, SM, LW
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540 to Zhe Chen.



- 560 powders have been significantly increased by introducing nano-TiB₂ particles.



562 Extended Data Fig. 2 As-printed NTD-Al alloy fatigue testing specimens. (A) Samples
563 constructed by machine EOS M280. (B) Samples constructed by machine 3D Systems ProX
564 MP 200. Schematics of fatigue testing specimen dimensions. (C) Pull-pull fatigue test

565 sample geometry in mm, R = 0.1 (ASTM E466-21). (D) Rotating bending fatigue test sample geometry in mm, R = -1 (ISO 1143-2008). 566



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Extended Data Fig. 3 µ-CT analysis showing the spatial and size distributions of defects. 569

570 (A) Defects with irregular morphology in AM AlSi10Mg alloy. (B) The maximum defect in 571 the AlSi10Mg sample with the length about 266 µm. (C) Defects with near spherical 572 morphology in AM NTD-Al alloy manufactured by EOS M280. (D) The maximum defect in the NTD-Al sample with the diameter about 77 µm. (E-F) Comparison of number and volume 573 574 fractions of defects in different size ranges.



575

576 Extended Data Fig. 4 Microstructure characterisation of the NTD-Al sample 577 manufactured by EOS M280. (A) Grain morphology of the plane perpendicular to the 578 printing direction. (B) Grain morphology of the plane parallel to the printing direction. (C) 579 Network of solidification cellular structure with an average diameter of ~ 500 nm. (D) Selected-580 area electron diffraction pattern indicating that Si existed as nanosized phases with random 581 orientations.



583 **Extended Data Fig. 5** *In situ* **micro-mechanical testing.** (A) Geometry of the tensile grip 584 and micro-specimen. SEM images of (B) the customised diamond tensile grip and (C) the 585 micro-specimen. (D) Stress-strain curve measured from the *in situ* micro-tensile test, inset: 586 SEM images of the necked region at different strain levels.



587

588 Extended Data Fig. 6 Tensile Properties of as-printed NTD-Al. Engineering stress-strain









Extended Data Fig. 8 Fractographic observations of fatigue failures in AM NTD-Al alloy. (A-C) Fracture surface of AM NTD-Al alloy at different fatigue loading strength, R=0.1; (D-F) three typical regions pointed out by white arrows in Extended Fig. 8A. The fatigue crack usually initiates at the largest pore next to the sample surface. Many smaller defects were also found on the fracture surface. The interspacing between neighboring defects ranges from tens to hundreds of microns. Obvious Si cellular boundaries were also found on the fracture surface.



Extended Data Fig. 9 Si cellular structure of the AM NTD-Al alloys after 10^7 fatigue testing cycles at a maximum stress of 260 MPa (R = 0.1). (A-B) HAADF STEM images showing the intact Si cellular structure. TEM samples were prepared from randomly selected area from the gauge section of the sample after fatigue testing. (C-F) EDS maps illustrating the distribution Al, Si, Mg and Ti elements.

614 Extended Data Table 1 Chemical composition (wt.%) and additive manufacturing processing parameters

615 of NTD-Al and AlSi10Mg powders (The corresponding weight and volume fractions of the TiB₂ nanoparticles

Element	Si	Mg	Cu	Mn	Ti	В	Al
NTD-AI 9.8		0.32	<0.03	<0.01	3.84	1.76	Balance
AlSi10Mg	AlSi10Mg 10.1		<0.03	<0.01	/	/	Balance
		Laser	Laser	Scanning	Layer	Rotation	Hatching
Parameter		power	diameter	speed	thickness	angle	space
		(W)	(µm)	(mm/s)	(µm)	(°)	(µm)
AlSi10Ma	ProX DMP	280	75	1000	30	66 7°	100
AIGHTUNIg	200	200	75	1000	50	00.7	100
NTD-AI	ProX DMP	280	75	1200	30	66.7°	100
	200						
NTD-AI	EOS M280	320	75	1000	30	66.7°	100

616 were \sim 5 wt.% and \sim 3.4 vol.%, respectively)











Fatigue damage originating from PSB

Fatigue immunisation mechanism





























с.										
•	Defects in AlSi10Mg alloy	6th		*	and the second sec				S	G.
	Stress concentration factor	7.9	6.1	7.3	6.2	5.2	7.8	7.7	7.0	4.3
	Pores in NTD-Al alloy	٢							Ŷ	
	Stress concentration factor	2.8	2.4	2.3	2.3	2.2	2.3	2.5	3.0	2.8

Loading direction







