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## Effect of building directions on the surface roughness, microstructure, and tribological properties of selective laser melted Inconel 625

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# 1           **Effect of building directions on the surface roughness,** 2           **microstructure, and tribological properties of selective laser** 3           **melted Inconel 625**

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## 15       **Abstract**

16           To study the effect of the building directions on surface macro- / micro- structure and  
17           tribological properties of Inconel 625 samples manufactured by selective laser melting (SLM)  
18           technology, parts were fabricated in three different building directions (0°, 45°, 90°). The  
19           microstructure of SLM samples was characterized using optical microscope (OM) and  
20           scanning electron microscopy (SEM). Only the  $\gamma$ -Ni phase was detected in the X-ray  
21           diffraction (XRD) patterns of the SLM samples. The lattice constant of the  $\gamma$ -Ni phase in the  
22           SLM 45° sample was the lowest among these SLM samples. The value of the surface  
23           roughness of the SLM 45° specimen was the highest than other parts, while, the value of the  
24           surface roughness of the SLM 0° sample was the lowest. The surface microstructure of the  
25           SLM samples was primarily composed of columnar dendrites and equiaxed grains. Among  
26           these samples, the finest equiaxed grains (about 0.38-0.63  $\mu\text{m}$ ) was evenly distributed in the  
27           SLM 0° sample, while, the roughest equiaxed grains (approximately 0.73-1.26  $\mu\text{m}$ ) was  
28           formed in the SLM 45° sample. Furthermore, the SLM 0° specimen showed the highest  
29           microhardness value (332 HV<sub>0.2</sub>) and the best tribological performances, such as the lowest  
30           average coefficient of friction (COF=0.31) and the wear rate (57.2 $\times 10^{-6}$  mm<sup>3</sup> / (N•m)).

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31 **Keywords:** Selective laser melting, building directions, Surface roughness, Microstructure  
32 evolution, Tribological performances.

## 33 **1 Introduction**

34 When it comes to the metal additive manufacturing (AM) technology, selective laser  
35 melting (SLM), the represent of sustainable metal production, is one of the most attracting  
36 technologies employed in recent years, as stated by Chen et al. (2019) and Martin et al. (2017).  
37 In the process of the SLM, metal powder is spread in thin layers and rapidly fused using  
38 high-density laser beam in the layer-by-layer method in order to create object components  
39 with complex geometric shapes. This technique opens a wide field of possible applications for  
40 many industrial sectors thanks to its eye-catching advantages, such as unlimited geometric  
41 variability, design freedom and material flexibility, as revealed by Yan et al. (2019b) and Yan  
42 et al. (2019a). Also, apart from the commonly used commercial pre-alloyed powder e.g.,  
43 nickel-based, cobalt-based, and iron-based alloy powder, this technique, due to the high  
44 energy density of the input laser beam, can also manufacture high melting point refractory  
45 metals like molybdenum, tantalum, tungsten and their alloys. SLM technology can, therefore,  
46 be regarded as an emerging technology to produce metal components directly from the raw  
47 material.

48 Inconel 625 is a nickel-based superalloy promoted by solid-solution hardening of various  
49 elements like C, Cr, Mo, and Nb, which has been used in widespread applications such as  
50 aerospace, marine, chemical and nuclear industries due to its outstanding yield, tensile and  
51 creep strength at elevated temperature, as investigated by Paul et al. (2007). Inconel 625  
52 components are generally manufactured by conventional manufacturing processes e.g.,  
53 casting, powder metallurgy and stir friction welding (Song and Nakata, 2010). With the  
54 bombarding development of AM technology, manufacturing Inconel 625 components by AM  
55 for industrial applications has steadily emerged as a promising approach. Currently, in the  
56 review paper by Mumtaz and Hopkinson (2009), the SLM technology was reported to  
57 successfully fabricate Inconel 625 parts. Compared to other AM techniques such as direct  
58 metal deposition (DMD) technology (Dinda et al., 2009), SLM, being able to efficiently  
59 utilize material and achieve cost-effective processing, has obtained a great attention. The  
60 investigations conducted by Frazier (2014), Yadollahi and Shamsaei (2017) and Yap et al.  
61 (2015) showed that since the laser spot of SLM is focused in a tiny area of less than 100  $\mu\text{m}$   
62 in diameter, extremely high temperature is generated in the center of the laser molten pool.  
63 Also, owing to energy distribution occurring in the laser spot, a great thermal gradient in the  
64 Inconel 625 material is produced, which directly affects the microstructure and mechanical  
65 performances of the processed parts.

66 To research the microstructure characteristics of the SLM Inconel 625 parts, recently,  
67 some studies focusing on the microstructure features of this material have been published. Li

68 et al. (2017) found that fine columnar  $\gamma$  dendrite microstructure with strong texture was a  
69 significant feature of the SLM samples, and the heat treatment could recrystallize  
70 microstructures, increase grain size, decrease dislocation density and diminish grain boundary  
71 misorientation. Anam et al. (2014) investigated that cellular substructure grains with enriched  
72 zones of Nb and Mo in the inter-arm spacing were detected using SEM and EDS, and the  
73 grains were also proved to grow preferentially in the build direction within the microstructure  
74 of SLM Inconel 625 parts. Li et al. (2015) studied the microstructure evolution of SLM  
75 Inconel 625 components and concluded that the microstructure was composed of elongated  
76 columnar crystals. In addition, they reported that the effect of the applied heat treatment on  
77 the microstructures of the SLM parts was not significant. Though many scholars have done  
78 various experiments for describing the microstructure features of SLM Inconel 625 parts in  
79 the different heat-treated states, the effect of the building directions on the microstructure of  
80 the surface and its evolution are still rarely analyzed.

81 As well known, manufacturing methods have a great impact on the microstructure and  
82 mechanical performances of the SLM Inconel 625 parts. Therefore, some other researchers  
83 have also studied the impact of applying different SLM manufacturing strategies on the  
84 microstructure and mechanical properties of the SLM Inconel 625 parts. Yadroitsev et al.  
85 (2009) investigated the effect of the different building directions on the mechanical properties  
86 of the SLM Inconel 625 parts, and the results displayed that the Young's modulus value for  
87 the "horizontal" samples was by 1.5 times higher than that for the "vertical" specimens and  
88 was close to that of wrought Inconel 625 component (about 200 GPa). However, the yield  
89 strength and ultimate strength of the samples were not significantly affected by the change in  
90 the building directions. Yadroitsev and Smurov (2010) also found that elongation of the SLM  
91 Inconel 625 parts was only 8-10 % compared to 40 % for conventionally cast and wrought  
92 components. Moreover, Gonzalez et al. (2019) compared the microstructures and mechanical  
93 properties of Inconel 625 parts produced by different AM techniques. The results illustrated  
94 that SLM specimens had potential to display outstanding mechanical properties as compared  
95 to ones fabricated using other technologies. As described above, the majority of the current  
96 studies were primarily concentrated on the effect of laser processing parameters and building  
97 directions on the microstructure and mechanical properties of SLM parts.

98 In summary, building directions can be adjusted to modify the microstructure of the  
99 SLM components, as demonstrated by Rashid et al. (2018). However, to date, the impact of  
100 the building directions upon the surface microstructure of the SLM Inconel 625 components,  
101 particularly the diverse microstructure caused by the complex thermal history and  
102 micro-reaction occurring in the SLM process, has not been explored in depth. Besides,  
103 relationships existing between the printing orientations and the tribological behavior of the  
104 SLM Inconel 625 parts have not been systematically studied, either. Obviously, there is still a  
105 research gap about the effect of SLM building directions on the wear performance of the SLM  
106 Inconel 625 samples.

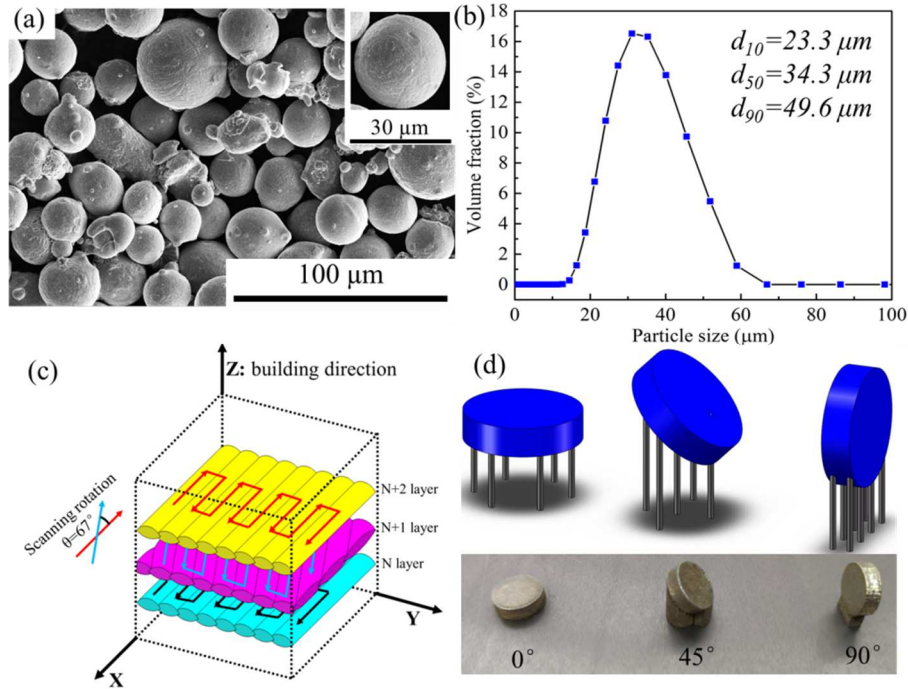


107 Hence, this problem should be of great interest to researchers as it not only helps to dig  
108 in the depth of the wear mechanism of SLM parts but also is deemed to be useful for the  
109 design and fabricating strategy of SLM components for generating high wear-resistant  
110 surfaces. Thus, the influence of the building directions on the macrostructure characteristics  
111 such as surface roughness, the surface microstructure evolution, and the wear mechanism of  
112 the SLM Inconel 625 parts are discussed and analyzed in detail within this work.

## 113 **2 Experimental details**

### 114 **2.1. Manufacturing procedure**

115 In this work, Inconel 625 powder (EOS GmbH, Germany) with a spherical shape was  
116 used as the raw material. The chemical composition of this used powder is shown in Table 1.  
117 The overview and magnified surface morphology of the used Inconel 625 powder are  
118 displayed in Fig. 1a. The particle size distribution ( $d_{10}=23.3\ \mu\text{m}$ ,  $d_{50}=34.3\ \mu\text{m}$ , and  $d_{90}=49.6$   
119  $\mu\text{m}$ ) of the powder, measured by the laser diffraction powder sizer (Mastersizer 2000,  
120 Malvern Instruments Ltd., UK), is given in Fig. 1b. As described in Fig. 1c, the laser scanning  
121 strategy was chosen as zigzag pattern with  $67^\circ$  rotation between the neighboring layers. These  
122 specimens were built up in three different orientations by SLM using an EOS M290 (EOS  
123 GmbH, Germany) system. As shown in Fig. 1d, the different category of the processed  
124 samples is marked as  $0^\circ$ ,  $45^\circ$ ,  $90^\circ$ . After conducting a systematic investigation, laser spot of  
125  $100\ \mu\text{m}$ , laser power of 280 W, layer thickness of  $30\ \mu\text{m}$ , hatch distance of  $110\ \mu\text{m}$  and  
126 scanning speed of 950 mm/s were used to process these samples. In order to avoid oxidation,  
127 the chamber of the SLM machine was filled with high purity nitrogen (99.99 %) to maintain  
128 the residual oxygen content below 0.13 %.



129  
 130 Figure 1. (a) Macroscopic morphology of the raw material; (b) particle size distribution; (c)  
 131 SLM scanning strategy used to process the samples; (d) SLM Inconel 625 samples produced  
 132 by applying different building directions.

133 Table 1: Chemical composition of the Inconel 625 raw powder

| Element | Ni      | Cr          | Mo         | Nb        | Fe    | Co    | Al    | C     |
|---------|---------|-------------|------------|-----------|-------|-------|-------|-------|
| wt.%    | Balance | 20.00-23.00 | 8.40-10.00 | 3.15-4.15 | ≤5.00 | ≤1.00 | ≤0.40 | ≤0.10 |

## 134 2.2. Material characterization

135 The surface roughnesses of these SLM parts were evaluated by a DektakXT profilometer  
 136 (Bruker, American). The surface area of 1000 μm×1000 μm of the each SLM sample at  
 137 different building directions was selected as the surface roughness test plane. The surface  
 138 microstructure characteristics of the SLM Inconel 625 samples were observed using an  
 139 optical microscope (OM, Leica Dmi5000m) and a scanning electron microscopy (FEI Nova  
 140 Nano SEM 450). Before conducting the metallographic analyses, all the samples were  
 141 polished by SiC grinding papers with different grits and then etched for 20 s in aqua regia  
 142 solution containing 100 ml HNO<sub>3</sub> and 300 ml HCl. Phase composition of the SLM parts was  
 143 utilized by X-ray diffraction (XRD, Siemens, Germany) at 40 kV and 40 mA in a 2θ range of  
 144 30-100°.

## 145 2.3. Mechanical performances

146 The Vickers microhardness was measured using a microhardness tester (Leitz-Wetzlar,  
 147 Germany) with a load of 200 g and an indentation time of 25 s. Before testing the

148 microhardness, surface roughness of the testing plane of the each SLM sample were polished  
 149 to below 0.15  $\mu\text{m}$ . The microhardness values were recorded after measuring different  
 150 locations on each surface of the SLM specimens, and then the average value of 10 times  
 151 measurements was calculated.

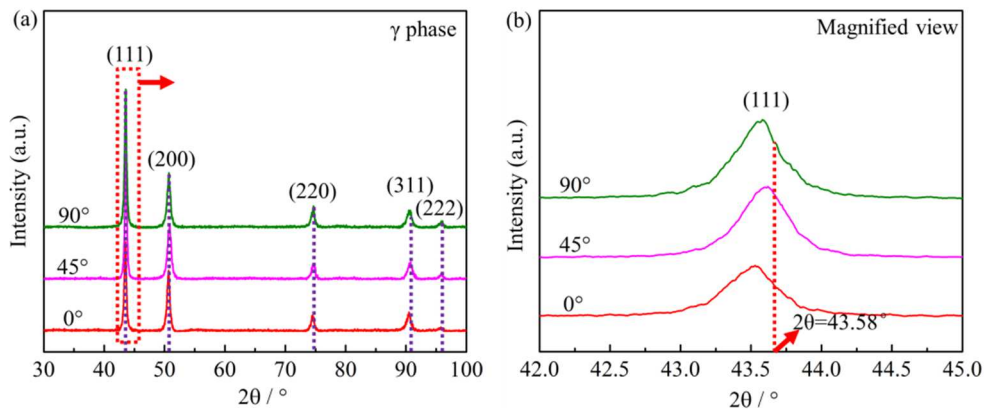
152 Dry sliding ball-on-disk wear tests were evaluated at room temperature with a CSEM  
 153 tribometer equipment. Before testing the friction and wear performance, the surface  
 154 roughness (Ra) of all SLM specimens were polished to below 0.15  $\mu\text{m}$ .  $\text{Si}_3\text{N}_4$  balls with a 4  
 155 mm diameter, cleaned with ethanol before performing the tests, were chosen as the  
 156 counterpart material sliding on the produced samples. The wear test conditions included a  
 157 testing load of 500 g, a rotational speed of 100 mm/s, a sliding distance of 200 m, and a  
 158 rotation radius of 3 mm. The coefficient of friction (COF) was automatically recorded by the  
 159 machine during the sliding tests. The worn surface of each sample was observed and analyzed  
 160 by SEM after finishing the wear tests. The macroscopic morphology of the worn tracks was  
 161 measured by the 3D surface profilometer. The wear rate ( $\omega$ ) of the samples was calculated via  
 162 the formula as follows and counted the mean value after three measurements:

$$163 \quad \omega = \frac{2\pi r S}{W \times L}$$

164 where r is the radius of the wear track, mm; S is the cross-sectional area of the worn tracks,  
 165  $\text{mm}^2$ ; W is the testing load, N; and L is the sliding distance, m.

### 166 3 Results and discussion

#### 167 3.1 X-ray diffraction analysis



168  
 169 Figure 2. (a) XRD diffractograms of the SLM specimens fabricated in different building  
 170 directions; (b) the magnified view of the (111) crystal plane

171 According to the X-ray diffraction patterns of the SLM samples printed in different  
 172 building directions, as shown in Fig. 2a, only peaks related to  $\gamma$ -Ni (fcc) phase was found.  
 173 From the phase diagram of the Fe-Cr-Ni ternary alloy, it can be further concluded that the  
 174 matrix is  $\gamma$  phase with the rather high solubility of Fe in  $\gamma$ -(Cr, Ni) based on the study of Yen

175 et al. (2008).

176 The diffraction peaks of some intermetallic phases like  $\gamma''$  phase ( $\text{Ni}_3\text{Nb}$ ),  $\text{Ni}_2(\text{Cr}, \text{Mo})$ ,  
177 Laves phase ( $\text{NbCr}_2$ ), etc., were not detected, although these intermetallic compounds may  
178 have been formed at elevated temperature. The reasons of that may be explained below: as the  
179 laser scanning speed (950 mm/s) and the cooling rate of the molten pool ( $10^5\text{K/s}$ - $10^6\text{K/s}$  (Yan  
180 et al., 2020b, 2020a)) were extremely high, these features would cause the velocity of the  
181 atomic reconstruction at the front of the solid-liquid interface is quite higher than the diffusion  
182 velocity. Hence, the solution atoms were trapped in the solid-liquid interface leading to the  
183 occurrence of the so called “solution trapping” phenomenon. Because of this, metallic solute  
184 atoms such as Cr, Nb, Mo, and etc., which were enriched at the front of the solid-liquid  
185 interface, did not have much chance to form intermetallic compounds and these solute atoms,  
186 therefore, were captured in the  $\gamma$ -Ni matrix. As a result, other phases were hard to observe in  
187 the XRD patterns of the SLM parts printed in various building directions. Note that a small  
188 number of intermetallic compounds might have been formed in the SLM parts, however,  
189 probably because of the low content and the small sizes of these precipitates, they were still  
190 not presented in the XRD diffractograms (Dinda et al., 2009).

191 Table 2: Lattice constants of the  $\gamma$ -Ni phase formed in the SLM Inconel 625 parts.

| Sample   | Lattice constant ( $\text{\AA}$ ) | Fitting error |
|--|-----------------------------------|---------------|
| Standard $\gamma$ -Ni phase measured by<br>Graulis et al. (2009) | 3.5911                            | /             |
| SLM $0^\circ$ part   | 3.59474                           | $\leq 10.33$  |
| SLM $45^\circ$ part  | 3.59204                           | $\leq 10.07$  |
| SLM $90^\circ$ part  | 3.59353                           | $\leq 10.11$  |

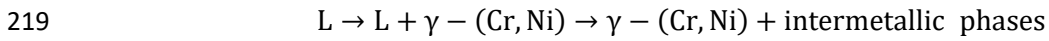
192 The lattice constants of SLM Inconel 625 parts fabricated in the various building  
193 directions were calculated using the Rietveld Refinement method proposed by Alexander and  
194 Klue (1989) and Materials Information Company (1991) and are listed in Table 2.

195 As shown in Fig. 2b, the diffraction peaks of the SLM samples were shifted to a  
196 low-angle direction as compared to the (111) crystal plane of the standard  $\gamma$ -Ni phase.  
197 Amongst all the printed samples, the offset value of the SLM  $45^\circ$  sample was the lowest.  
198 According to Table 2, the measured lattice constants of  $\gamma$ -Ni phase in the SLM parts were  
199 larger than the standard lattice constant of  $\gamma$ -Ni phase ( $a=3.5911\text{ \AA}$ ), which further led to the  
200 occurrence of lattice distortion of the  $\gamma$ -Ni phase. Additionally, the lattice constant measured  
201 in the SLM  $0^\circ$  sample was larger than that of the SLM  $45^\circ$  and SLM  $90^\circ$  sample, while, the  
202 lattice constant was the lowest in the SLM  $45^\circ$  sample.

203 Numerous factors such as element solid solution, surface stress, and etc. will lead to the  
204 diffraction peak shift and the lattice distortion. As the laser scanning strategy was the same for  
205 fabricating these SLM samples, it is estimated that the surface stress was not significantly

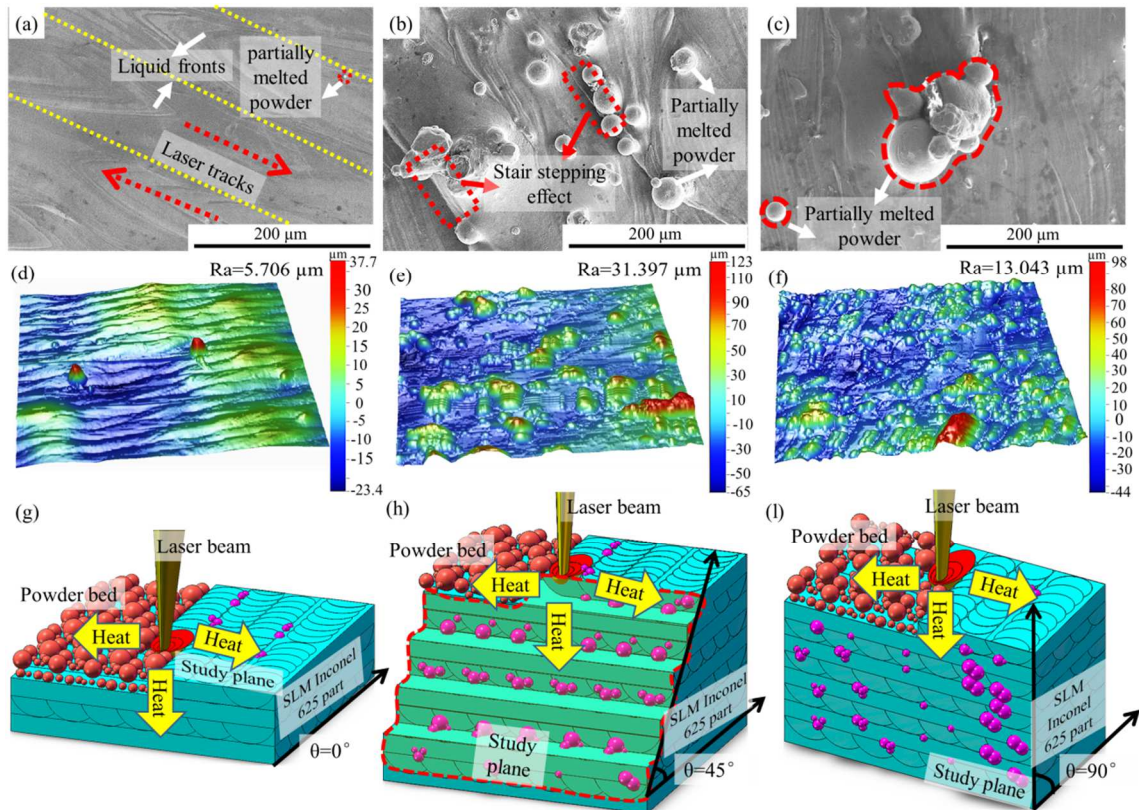
206 different among these SLM specimens. Thus, the difference of the above phenomenon that  
 207 occurred in the SLM parts could be primarily attributed to the difference existing in the  
 208 amount of the multi-element solid-solution  $\gamma$ -Ni phase of the printed parts. This situation  
 209 might be explained by the fact that the solid solubility of the alloying elements was high  
 210 within the fine microstructure of the SLM 0° sample due to the extremely rapid solidification  
 211 rate. In contrast, as the cooling rate **might be** relatively slower during the solidification  
 212 process of the SLM 45° and the SLM 90° samples, there might have been enough time for the  
 213 diffusion process of the alloying elements in these samples. As such, it is thought that a  
 214 supersaturated solid-solution  $\gamma$ -Ni phase with slightly lower solid solubility was produced in  
 215 the mentioned samples as compared to that of the SLM 0° sample.

216 After considering the above results and researching the analysis of Aliofkhazraei (2015)  
 217 and Cieslak et al. (1988), the formation sequence of the phases after solidification of the  
 218 molten pool can be proposed as follows:



220 **3.2 Macro- / Micro-structure characteristics**

221 **3.3.1 Surface roughness**



222  
 223 Figure 3. SEM macroscopic morphology of the SLM samples: (a) 0°; (b) 45°; (c) 90°;  
 224 three-dimensional surface topographies of the SLM specimens: (d) 0°; (e) 45°; (f) 90°;  
 225 surface roughness formation mechanism of the SLM parts: (g) 0°; (h) 45°; (i) 90°.

226 To investigate the impact of applying different building directions on the surface  
227 roughness, the surface morphology of the SLM Inconel 625 samples processed in different  
228 printing directions is shown in Fig. 3. The SLM 0° sample showed a relatively smooth surface  
229 having only a small number of partially melted powder particles within the borders between  
230 the neighboring tracks (Fig. 3a). Whereas, in the SLM 45° sample, plenty of partially melted  
231 particles were noticeable on the interfaces between the adjacent layers, which are thought to  
232 be essentially caused by the prominent stair steps (Fig. 3b). Similarly, multiple partially  
233 melted powder particles were found in the SLM 90° sample (Fig. 3c). However, it is noted  
234 that these small stair steps were no longer present in the SLM 90° sample compared to that in  
235 the SLM 45° sample. Moreover, the above macrostructure characteristics (Fig. 3a-c) were  
236 quantitatively analyzed by the Ra measurement of these samples (Fig. 3d-f). The SLM 0°  
237 sample had the smoothest surface ( $R_a=5.706\ \mu\text{m}$ ), while the SLM 45° sample exhibited the  
238 roughest surface ( $R_a=31.397\ \mu\text{m}$ ).

239 To further understand the influence of the building orientations on the surface roughness  
240 of the printed parts, the forming mechanism of the observed macroscopic morphology is  
241 presented in Fig. 3g-l. It is known that the balling phenomenon (Gu and Shen, 2009) and the  
242 stair stepping effect (Strano et al., 2013), also promoting the occurrence of the balling  
243 phenomenon, are the essential factors affecting the surface roughness of the SLM parts  
244 produced under the different building orientations. With regard to the balling phenomenon,  
245 the micro-sized balls and large-sized balls can be assumed as the two major types of balling  
246 phenomenon. When the preset powder bed is irradiated by the laser beam, the molten pool  
247 having heterogeneous surface tension is produced, owing to the different volumes of different  
248 elements existing in the molten pool based on the study of Steen and Mazumder (2010). The  
249 partial kinetic energy of the laser beam is converted into the surface energy of the molten  
250 metal, due to a high scanning velocity producing some smaller metal droplets of alloy  
251 material. To ensure that these droplets have a minimum surface energy, a variety of  
252 micro-sized balls will be formed under the joint influence of the surface tension and gravity.  
253 Besides, a small quantity of metal droplets also tend to splash from the surface of the molten  
254 pool and then solidify into the micro-sized balls on the surface of the SLM parts.

255 In general, the phenomenon of forming the micro-sized balls is uncommon due to the  
256 limited kinetic energy created by the high scanning velocity. The relatively smooth surface  
257 observed on the macro-surface appearance of the SLM 0° sample (Fig. 3a and Fig. 3g) is an  
258 evidence for this claim. Additionally, the laser remelting process existing in these overlapping  
259 regions is beneficial for inhibiting the balling phenomenon and improving the surface finish  
260 of SLM parts (Yasa and Kruth, 2011). Consequently, the smoothest macroscopic surface  
261 could be obtained on the SLM 0° sample.

262 Moreover, the formation of the large-sized balls on the macroscopic surface is rather  
263 typical, which can be marked as a wetting problem between the molten metal and the surface  
264 of the solidified SLM parts. The equilibrium of interfacial free energies existing among the

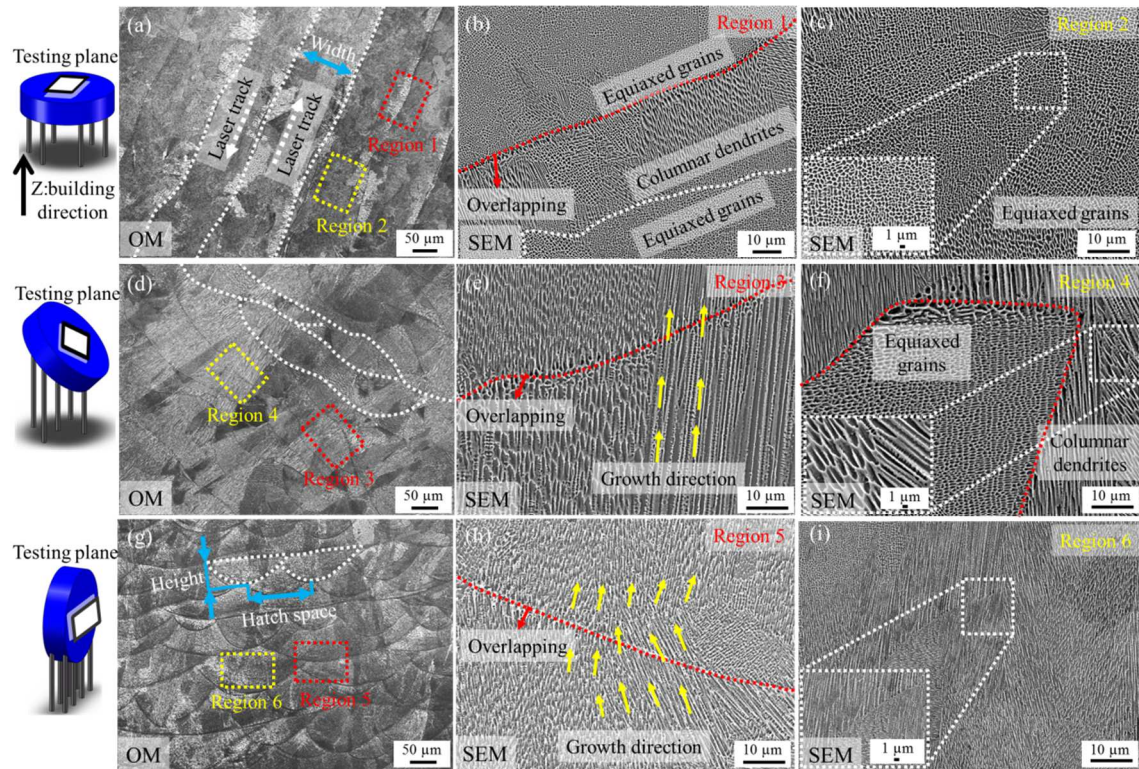


265 molten metal, the solidified printed samples and the ambient air can be governed by (Das,  
266 2003):

$$267 \quad S = \gamma_{sg} - \gamma_{sl} - \gamma_{lg}$$

268 where S is the spreading coefficient and  $\gamma_{sg}$ ,  $\gamma_{sl}$  and  $\gamma_{lg}$  are the interfacial energies of the  
269 solid-gas, the solid-liquid and the liquid-gas, respectively. According to the above equation,  
270 the value of the S reflects the degree of spontaneous liquid spreading. The molten pool of the  
271 SLM Inconel 625 parts is inevitably susceptible to oxidation during the rapid solidification  
272 process, due to the existence of some active elements (e.g., Ni, Cr, etc.) prone to oxidation in  
273 the composition of these samples (Das, 2003). However, the  $\gamma_{sg}$  of these metal oxides are  
274 quite lower than the corresponding  $\gamma_{lg}$  (Bunnell et al., 1995) causing a negative S. Hence,  
275 under this condition, the molten metal tends to diminish surface energy by forming dozens of  
276 large-sized balls, instead of wetting the metal oxide films. Furthermore, owing to the  
277 extremely short dwelling time of the laser beam (normally less than 4 ms, as demonstrated by  
278 Simchi (2006) and Simchi and Pohl (2004)) and the exceedingly high solidification rate of the  
279 molten pool (up to 30 m/s, as reported by Gholipour et al. (2011) and Rack and Kalish,  
280 (1974)), the molten metal cannot perfectly wet the surface. This phenomenon, therefore,  
281 facilitates the formation of the large-sized balls on the surface of the printed samples.

282 As a result, the large-sized balls were detected on the macroscopic surfaces of the SLM  
283 45° sample (Fig. 3b) and the SLM 90° sample (Fig. 3c). Moreover, the macro-surface  
284 appearance of the SLM 45° sample (i.e., the study plane shown in Fig. 3h) was composed of  
285 multiple micro-sized stair steps. Since there doesn't exist the remelting process in the regions  
286 of the stair steps, therefore, the balling effect produced on the surface of the previous cladding  
287 layer cannot be eliminated. Consequently, under the combined effect of the balling effect and  
288 stair stepping effect, the value of the surface roughness of the SLM 45° sample is the largest.  
289 As for the surface of the SLM 90° sample, it is noted that, thanks to the remelting  
290 phenomenon occurred between the neighboring layers, a relatively fine surface finish is  
291 obtained. However, the balling phenomenon especially the large-sized balls was also evident  
292 (Fig. 3c and Fig. 3l) in the sample prepared under this condition.



294

295 **Figure 4. Microstructures of the SLM parts: (a) the SLM 0° sample; (b) enlarged view of**  
 296 **region 1 in a; (c) enlarged view of region 2 in a; (d) the SLM 45° specimen; (e) enlarged view**  
 297 **of region 3 in d; (f) enlarged view of region 4 in d; (g) the SLM 90° part; (h) enlarged view of**  
 298 **region 5 in g; (i) enlarged view of region 2 in g.**

299 Fig. 4 presents the microstructures of the SLM parts, manufactured in different building  
 300 directions (0°, 45°, 90°) using OM and SEM. As for the microstructure of the SLM 0° sample,  
 301 the width of the laser track shown in Fig. 4a was approximately 86-108 μm, which was  
 302 basically equal to the laser spot size (100 μm). The microstructure close to the overlapping  
 303 line (region 1) was mainly composed of columnar dendrites (near the overlapping line) and  
 304 equiaxed grains (near the middle of the laser tracks), as depicted in Fig. 4b. In contrast,  
 305 basically only fine equiaxed grains with a size of about 0.38-0.63 μm (region 2) existed in the  
 306 middle of the laser tracks as displayed in Fig. 4c. Fig. 4d shows the large fish-scale shape  
 307 molten pool distributed within the microstructure of the SLM 45° sample. The coarse  
 308 columnar structures and equiaxed grains were still observed next to the overlapping line  
 309 (region 3) between these two adjacent layers (Fig. 4e). Fig. 4f illustrates that the columnar  
 310 structures were grown on the surfaces of the equiaxed grains with a size of approximately  
 311 0.73-1.26 μm (in the region 4). The fish-scale morphology with the size of 21-45 μm in  
 312 height, formed in the SLM 90° specimen, was roughly equal to the powder layer thickness (30  
 313 μm). In addition, the distance between the neighboring tracks (about 105-129 μm), measured  
 314 from the Fig. 4g, was substantially equivalent to the hatch space. As displayed in Fig. 4h and



315 Fig. 4i, the columnar dendrites and equiaxed grains (about 0.45-0.91  $\mu\text{m}$ ). Interestingly, both  
316 existed in the overlapping regions between the adjacent laser layers (region 5) and in the  
317 molten pool (region 6).

318 In short, after observing and analyzing the microstructures of the SLM parts in different  
319 building directions, it is found that the microstructures, regardless of the building orientations,  
320 are primarily made up of columnar dendrites and equiaxed grains.

321 It should be noted that in these specimens the finest microstructure and the largest  
322 number of equiaxed grains were distributed in the SLM  $0^\circ$  sample, while, the SLM  $45^\circ$   
323 sample possessed the coarsest microstructure and the least quantity of equiaxed grains. In  
324 order to explain the above phenomenon, the influence of the building directions on the surface  
325 microstructure evolution process is analyzed. The microstructure features in Fig. 4 can be  
326 characterized and analyzed by considering the surface microstructure evolution process of  
327 multi-tracks (region A) and multi-layers (region B) of the SLM Inconel 625 parts, as depicted  
328 in Fig. 5. The heat input ( $Q_s$ ) distribution in the molten pool can be estimated by the Gaussian  
329 heat source and thus the corresponding  $Q_s$  is expressed below (Luo et al., 2018):

$$330 \quad Q_s = Q_m \exp\left(-\frac{2r^2}{R^2}\right)$$

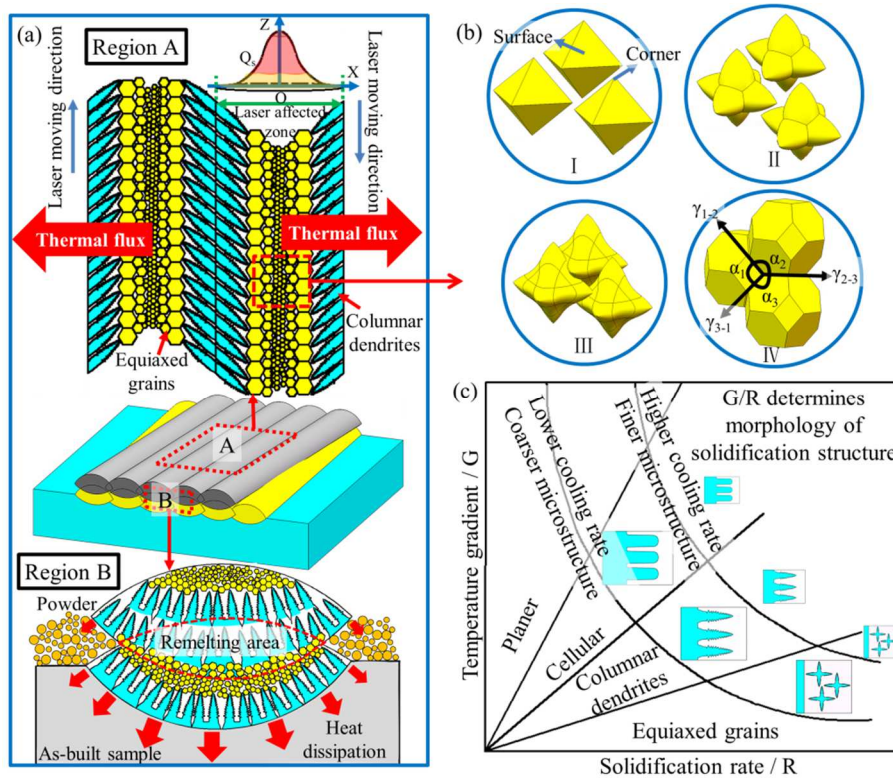
331 where  $Q_m$  is the maximum heat flux density;  $r$  is the radial distance between the observing  
332 point and the laser spot center and  $R$  is the laser spot radius. Therefore, in the same plane  
333 irradiated by the laser beam, as  $r$  gradually increases, the heat input amount will gradually  
334 decrease. This trend will result in the temperature in the middle of the molten pool to be much  
335 higher than that in the boundary region of the molten pool. In the central area of the molten  
336 pool, the convective heat transfer process occurs mainly in two directions, i.e., from the center  
337 to the periphery of the molten pool, and from the surface of the molten pool to the ambient  
338 environment. This phenomenon will make the actual temperature ( $T_q$ ) gradient in the liquid  
339 metal at the central region of the molten pool much lower than the liquidus temperature ( $T_l$ )  
340 gradient. Consequently, the resulted large degree of constitutional undercooling ( $\Delta T = T_l -$   
341  $T_q$ ) satisfies the nucleation conditions of the equiaxed grains. Hence, a heterogeneous crystal  
342 nucleation process in the central region of the molten pool will occur, and they will gradually  
343 grow into polyhedral grains, i.e., stage 1 (as shown in Fig. 5b).

344 Based upon the requirement for the minimum Gibbs free energy, crystal interfaces are  
345 often composed of crystal planes with lower interfacial energy. Therefore, the polyhedral  
346 surfaces formed are the interfaces with low interfacial energies, while, the “corner” regions  
347 are crystal planes with high interfacial energies. According to the XRD spectrums (as  
348 displayed in Fig. 2), it can be interpreted that the crystal plane of the minimum interfacial  
349 energy of the  $\gamma$  phase grains in the SLM Inconel 625 component is  $\{111\}$  crystal plane. In a  
350 non-equilibrium solidification process of the molten pool, a solute concentration gradient in  
351 the liquid metal at the front of the polyhedron is high and thus the diffusion speed is quite  
352 large. It is owing to this reason that the growth morphology of the polyhedral grains is shown

353 in stage 2. When the equiaxed grains rapidly grow larger and approach each other, the grain  
 354 boundaries tend to have equal angles and the intersecting interface of three grains will be  
 355 preferentially formed (remarked in stage 3). To meet a requirement of the minimum grain  
 356 boundary energy, as displayed by Callister and Rethwisch. (2015) among these grains, the  
 357 following equation can be presented:

$$358 \quad \frac{\gamma_{1-2}}{\sin\alpha_3} = \frac{\gamma_{2-3}}{\sin\alpha_1} = \frac{\gamma_{3-1}}{\sin\alpha_2}$$

359 where  $\gamma_{1-2}$ ,  $\gamma_{2-3}$ , and  $\gamma_{3-1}$  are interfacial tensions;  $\alpha_1$ ,  $\alpha_2$ ,  $\alpha_3$  are interface angle between adjacent  
 360 grains. Thus, in the final stage, the interface angles between adjacent grains will be prone to  
 361 be  $120^\circ$  and also be substantially equal. Finally, the equiaxed grains at the center of the  
 362 molten pool exhibit hexagonal structures (as shown in stage 4). Besides, since the marginal  
 363 area of the molten pool is surrounded by the powder bed, the temperature gradient at the edge  
 364 of the molten pool will be much greater than that at the center of the molten pool.  
 365 Additionally, there is little difference in the solidification rate between the center area and the  
 366 boundary area of the molten pool. Under such conditions, the columnar dendrites will form  
 367 and distribute along the margin of the molten pool.



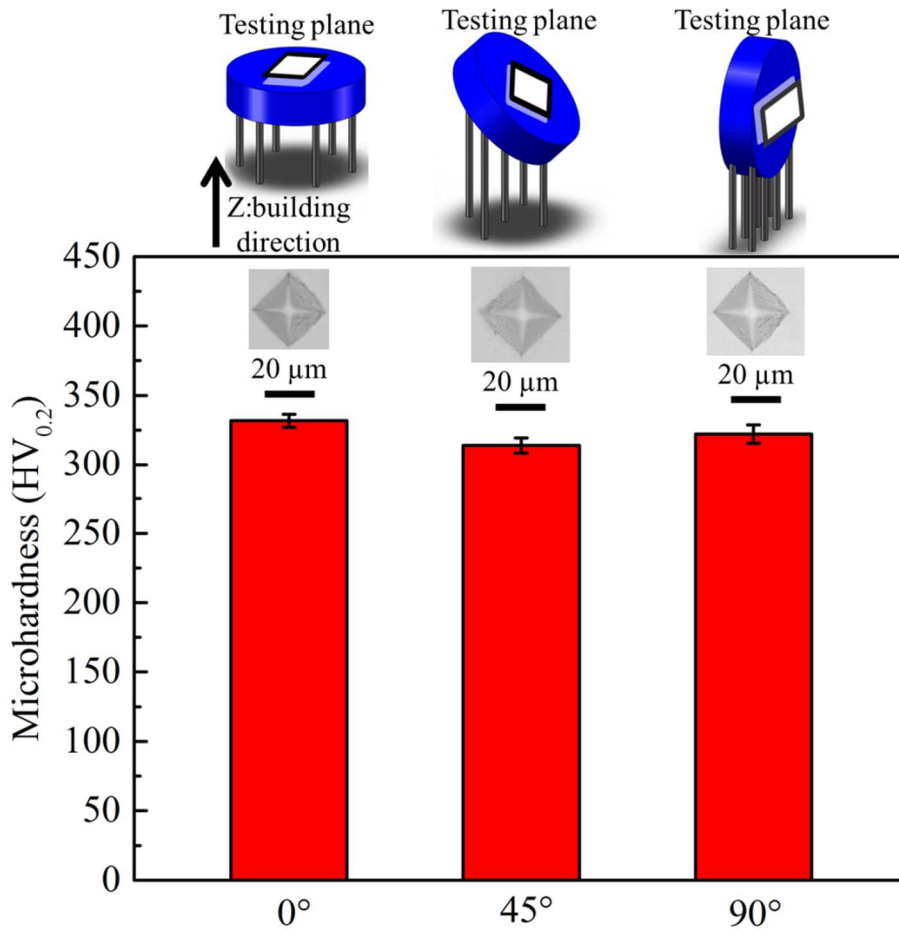
368  
 369 Figure 5. (a) Surface microstructure characteristics of the multi-tracks and the multi-layers  
 370 regions of the SLM parts; (b) equiaxed grains formation mechanism diagram; (c) schematic  
 371 profile illustrating the effects of temperature gradient G and growth rate R on the morphology  
 372 of solidification microstructure described by Liu et al. (2016).

373 Combining the above analysis and Fig. 5c, the microstructure of the SLM  $0^\circ$  sample is  
 374 the one with the multi-tracks overlapping region (as depicted in region A of Fig. 5a). The

375 microstructure evolution from the boundary of the laser track to the middle of that consist of  
376 separately coarse columnar dendrites, fine columnar dendrites, coarse equiaxed grains and  
377 fine equiaxed grains. This microstructure features denote that the columnar dendrites  
378 primarily grew along the laser tracks (as shown in Fig. 4b), while, the equiaxed grains were  
379 distributed in the center of that (as displayed in Fig. 4c).

380 Regarding the SLM 45° specimen, its microstructure is substantially equivalent to the  
381 microstructure of the SLM 90° sample being intercepted at a 45° angle. The microstructure of  
382 the SLM 90° sample is the one with the multi-layers region (as illustrated in region B of Fig.  
383 5a). Hence, the microstructure formation mechanism of both samples can be depicted by  
384 region B of Fig. 5a and the microstructure evolution of these two specimens can be  
385 principally determined using the heat dissipation direction, the preferred orientation of the  
386 dendrites, and the ratio of the temperature gradient ( $G$ ) to the solidification rate ( $R$ ) (Fig. 5c).  
387 At the bottom of the molten pool, since  $G$ , close to the substrate, is rather small and  $R$  is quite  
388 large, therefore, a typical characteristic of epitaxial columnar growth perpendicular to the  
389 interface of the molten pool will occur. In the middle of the molten pool, due to the further  
390 reduction of  $G/R$ , fine dendritic structures are distributed on the surface of the coarse  
391 columnar dendrites. Moreover, since the heat dissipation direction of the solidification  
392 microstructure is mainly through the multi-layers region and powder bed, the growth  
393 orientation of the dendrites is only in the opposite direction of the heat dissipation direction.  
394 This results in the directional growth of the columnar structures (as shown in Fig. 4e and Fig.  
395 4h). Ascending from the middle of the molten pool, the  $G$  is the lowest and the  $R$  is the largest,  
396 so the value of  $G/R$  is the smallest. Consequently, in addition to the formation of the fine  
397 dendrites, the fine equiaxed grains can be also formed at the top of the molten pool (as  
398 demonstrated in Fig. 4f and Fig. 4i.). As for the remelting areas, the microstructure will  
399 maintain the growth direction of the microstructure in the previous layers and continue to  
400 grow because of the phenomenon of structural inheritance. This feature not only ensures the  
401 bonding strength between the adjacent layers but also makes the epitaxial structure have  
402 continuity in the growth direction (Fig. 4e). Another interesting phenomenon in the remelting  
403 zone is that the melt flow will produce a scouring effect on the solute-rich layer at the front of  
404 the columnar dendrites. This makes the columnar dendrites grow obliquely just like Fig. 4h.

405 **3.3 Microhardness**



406

407

Figure 6. Microhardness of the SLM parts built in different building directions

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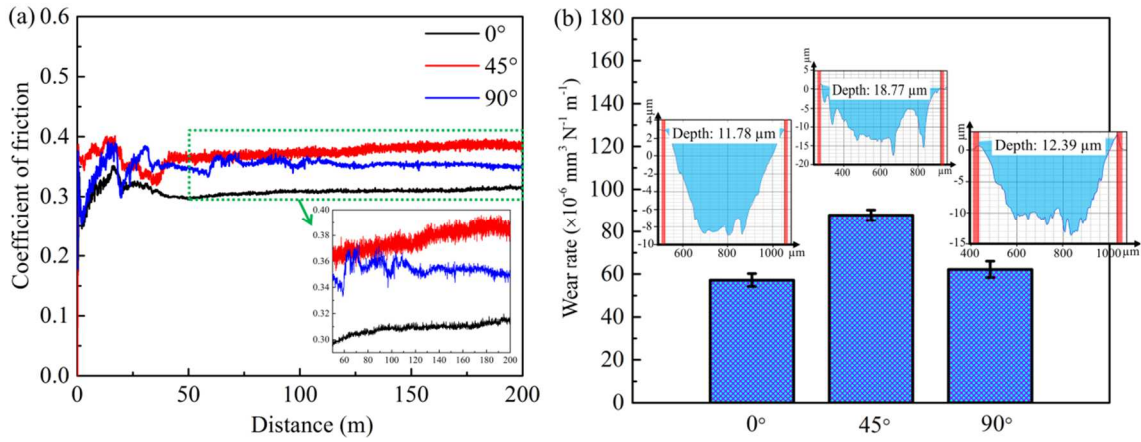
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418

As presented in Fig. 6, the average microhardness of the SLM 0° sample, the SLM 45° sample and the SLM 90° sample is 332 HV<sub>0.2</sub>, 314 HV<sub>0.2</sub> and 322 HV<sub>0.2</sub>, respectively. The mechanical performances of the SLM samples have a strong correlation to their microstructure characteristics. Comparing these three types of the SLM samples built in different orientations, the massive fine equiaxed grains distributed within the surface microstructure of the SLM 0° sample have a significant effect on the improvement of its hardness. In contrast, the comparably coarse columnar dendrites and equiaxed grains formed in the surface of the SLM 45° and the SLM 90° samples will induce lower hardness values in these samples as compared to the SLM 0° one. Additionally, the average microhardness of the SLM 45° part is the lowest among these specimens by the cause of having the coarsest microstructure.

419 **3.4 Anisotropy in tribological properties**

420 **3.5.1 Coefficient of friction and wear rate**



421

422 Figure 7. (a) COF - distance profiles and (b) wear rate of the SLM samples fabricated in the  
423 different building directions.

424

425 Fig. 7 displays the effect of the building directions on the tribological properties  
426 including the COF and wear rate of the SLM specimens. The corresponding statistical results  
427 are listed in Table 3. As for the SLM 0° sample, the average COF of the stable stage of  
428 friction (i.e., the wear distance from 50 m to 200 m) is 0.31 and the average wear rate is only  
429 57.2×10<sup>-6</sup> mm<sup>3</sup>/(N•m). In contrast, the average COF and the wear rate of the SLM 45° sample  
430 and the SLM 90° sample are relatively higher, as compared to the SLM 0° one. These can be  
431 attributed to the coarser columnar dendrites and equiaxed grains dispersed within the surface  
432 of these specimens as compared to the SLM 0° one. As a result, amongst these parts, the SLM  
433 45° sample exhibits the worst tribological performance, as it contains the coarsest surface  
434 microstructure.

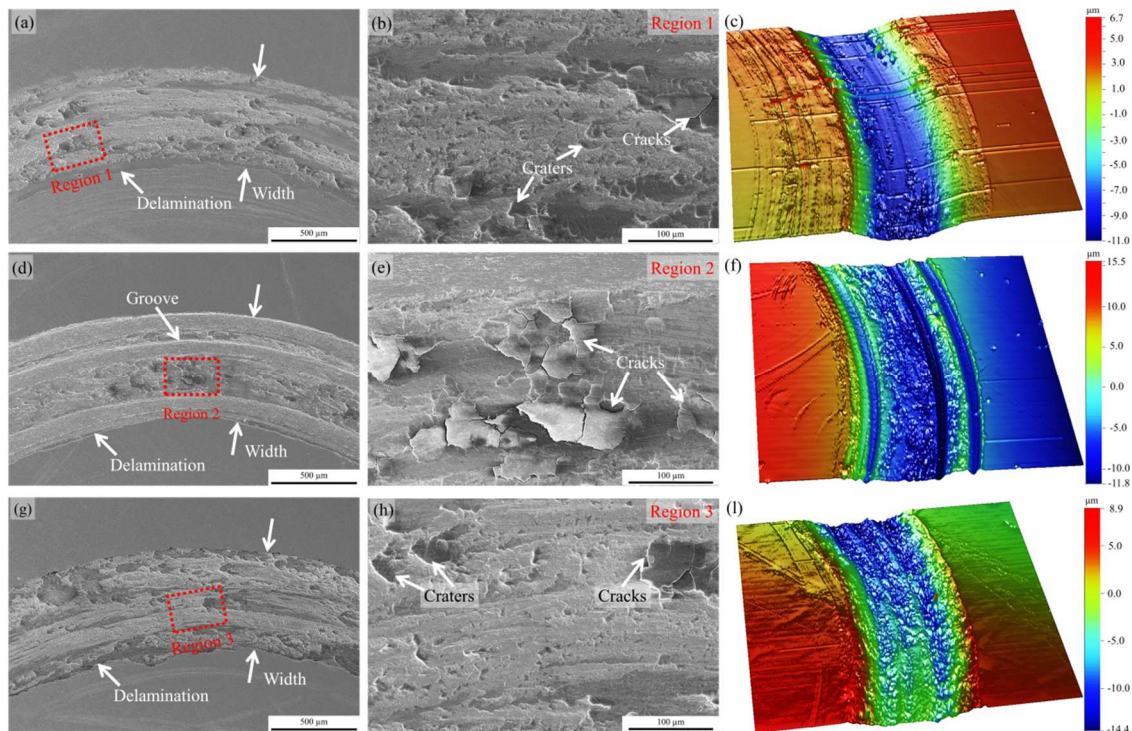
434

435 Table 3: Tribological properties of SLM Inconel 625 specimens produced in different  
436 building orientations

| Sample       | Average COF | Average wear rate (×10 <sup>-6</sup> mm <sup>3</sup> /(N•m)) |
|--------------|-------------|--|
| SLM 0° part  | 0.31        | 57.2±3   |
| SLM 45° part | 0.38        | 87.8±2.5   |
| SLM 90° part | 0.35        | 62.2±3.8   |



436 **3.5.2 Worn track morphology and mechanism**

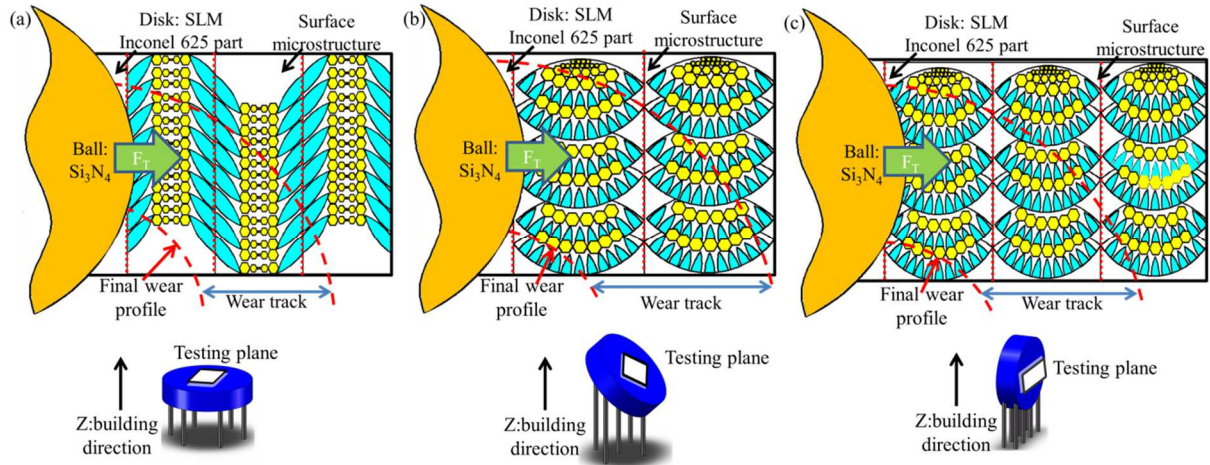


437  
 438 Figure 8. Worn surface morphology of (a) SLM 0° sample; (b) magnified view of region 1 in  
 439 a; (c) 3D surface topography of a; (d) SLM 45° sample; (e) magnified view of region 2 in d; (f)  
 440 3D surface topography of d; (g) SLM 90° sample; (h) magnified view of region 3 in g; (i) 3D  
 441 surface topography of g.

442 Details about the worn surface morphology of the SLM Inconel 625 parts are revealed in  
 443 Fig. 8. For the SLM 0° specimen, the average width of the worn track was about 531.6 μm.  
 444 Numerous grooves and furrows were scattered on the surface of the worn track, illustrating  
 445 that the main wear mechanism causing damage during the test was abrasive. A small quantity  
 446 of micro craters and cracks could also be presented in region 1 (Fig. 8b), which demonstrate  
 447 the occurrence of delamination in the worn surface. The 3D surface topography of the SLM 0°  
 448 sample after experiencing the friction stage was relatively smooth (Fig. 8c). While, as for the  
 449 SLM 45° part, the width of the worn track was 646.5 μm (Fig. 8d). As depicted in Fig. 8d-e, a  
 450 great number of cracks and parallel deep grooves were presented on the worn surface of this  
 451 sample which is indicative of delamination and deformation phenomenon occurring on the  
 452 worn surface. Thus, the 3D surface topography of the SLM 45° part was comparably rougher  
 453 than that of the SLM 0° part (Fig. 8f). As seen in Fig. 8g, the width of the wear track for the  
 454 SLM 90° sample was 602.6 μm. In addition, a small number of cracks and craters were  
 455 produced due to the plowing effect. This indicated that the ploughing were also another  
 456 primary wear mechanism damaging this sample. It is noted that the 3D surface of the wear  
 457 trace of the SLM 90° sample was rather smoother than that of the SLM 45° part.

458 In short, the abrasive and micro-ploughing wear mechanisms are the main wear

459 mechanism damaging these SLM Inconel 625 parts. Moreover, it is concluded that the effect  
 460 of the building directions on the tribological performances of these samples (such as average  
 461 COF, wear rate and worn track morphology) cannot be neglected. Hence, the influence of the  
 462 printing orientations upon the wear performance and the friction behavior of the samples are  
 463 discussed below.



464

465 **Figure 9. Schematic diagrams of sliding wear mechanism on the surface of the SLM Inconel**  
 466 **625 parts manufactured at different printing directions: (a) 0°; (b) 45°; (c) 90°.**

467 Fig. 9 illuminates the relationship between the surface microstructure and the wear  
 468 mechanism of the SLM Inconel 625 samples manufactured in different building orientations.  
 469 Compared with the columnar dendrites, the fine equiaxed grains are easier to accommodate  
 470 large strains announced by Martin et al. (2017). During the sliding wear process, thus, the  
 471 columnar dendrites are liable to break up under the tangential force ( $F_T$ ) resulting in a poor  
 472 wear resistance performance.

473 Among these specimens, the finest microstructure (e.g., columnar dendrites and  
 474 equiaxed grains) were primarily formed in the surface of the SLM 0° specimen (Fig. 4a-c).  
 475 Therefore, under the illustration of Fig. 9a, as the columnar structures are peeled off during  
 476 the wear process, the equiaxed grains will be also gradually peeled off. Then, upon the  
 477 influence of this process, some inconspicuous plows (Fig. 8a), a small quantity of micro  
 478 craters, and some micro cracks (Fig. 8b) were progressively formed on the worn track surface.  
 479 As mentioned, this phenomenon produced the smoothest worn surface with the narrowest  
 480 average width of the worn track (Fig. 8c) in SLM 0° sample. On the contrary, according to the  
 481 surface microstructure analysis of the SLM 45° specimen (Fig. 4d-f), the roughest columnar  
 482 dendrites and the smallest number of equiaxed grains were dispersed within the  
 483 microstructure of the SLM 45° component. Consequently, according to Fig. 9b, after a large  
 484 quantity of rough columnar dendrites are peeled off in the process of the sliding wear, a huge  
 485 number of craters and cracks are produced in the damaged areas. As the wear process is  
 486 further performed, these numerous micro craters and cracks will connect to each other, and  
 487 then produce the massive larger cracks. This phenomenon could be observed in Fig. 8e.

488 Hence, the roughest worn trace surface with the largest average width of the worn track was  
489 obtained in this sample, as displayed in Fig. 8f. As for the SLM 90° component, under the  
490 description of Fig. 4g-l, the surface microstructure of the SLM 90° sample was finer than that  
491 of the SLM 45° part and the proportion of the equiaxed grains in the microstructure was more  
492 than that of the SLM 45° specimen. Therefore, under the illumination of Fig. 9c, only a small  
493 quantity of columnar structures within the surface of the SLM 90° specimen will begin to  
494 break up and generate some tiny craters and cracks during the dry sliding wear process. As  
495 compared to the SLM 45° part, these craters and cracks were not joined together but only  
496 scattered on the worn surface of the SLM 90° specimen (Fig. 8g-h). Finally, as seen in Fig. 8l,  
497 a relatively rough worn surface was obtained.

## 498 **4 Conclusion**

499 In summary, a variety of SLM Inconel 625 **samples** were successfully fabricated by SLM  
500 technology to study the effect of the building directions upon the performances of the SLM  
501 parts. In this work, experiments were conducted to investigate the impact of the building  
502 orientations on the surface roughness, microstructure evolutions, and tribological properties  
503 of the selective laser melted Inconel 625 parts. Primary conclusions drawn from this work  
504 were listed as follows:

505 1. The phase structure of the SLM Inconel 625 **specimens** was predominately  
506 composed of the  $\gamma$ -Ni (fcc) phase. The lattice constant of the  $\gamma$ -Ni phase in the SLM 45°  
507 **sample** (3.59204 Å) was lowest than other SLM samples. The microstructure of the SLM  
508 parts was composed of the columnar structures and the equiaxed grains. Among these samples,  
509 the finest microstructure and the largest number of equiaxed grains (0.38-0.63  $\mu\text{m}$ ) were  
510 distributed in the SLM 0° sample, while, the SLM 45° sample possessed the coarsest  
511 microstructure and the least quantity of equiaxed grains (0.73-1.26  $\mu\text{m}$ ).

512 2. The value of the surface roughness of the SLM 45° **sample** (31.397  $\mu\text{m}$ ) was the  
513 roughest owing to the balling phenomenon and the stair stepping effect. On the contrary, the  
514 value of the surface finish of the SLM 0° specimen (5.706  $\mu\text{m}$ ) was the smoothest due to the  
515 remelting phenomenon.

516 3. For the SLM 0° specimen, the average COF and the wear rate was 0.31 and  
517  $57.2 \times 10^{-6} \text{ mm}^3 / (\text{N} \cdot \text{m})$ , respectively. In contrast, for the SLM 45° sample, the average COF  
518 and the wear rate was respectively 0.38 and  $87.8 \times 10^{-6} \text{ mm}^3 / (\text{N} \cdot \text{m})$ .

519 The present work demonstrates that the SLM **building direction** has an obvious influence  
520 on the surface macro- / micro- structure and tribological properties of the Inconel 625 parts.  
521 This paper will attract the attention of the engineering community for designing a proper  
522 building direction to obtain a desirable surface with better tribological performances.



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535

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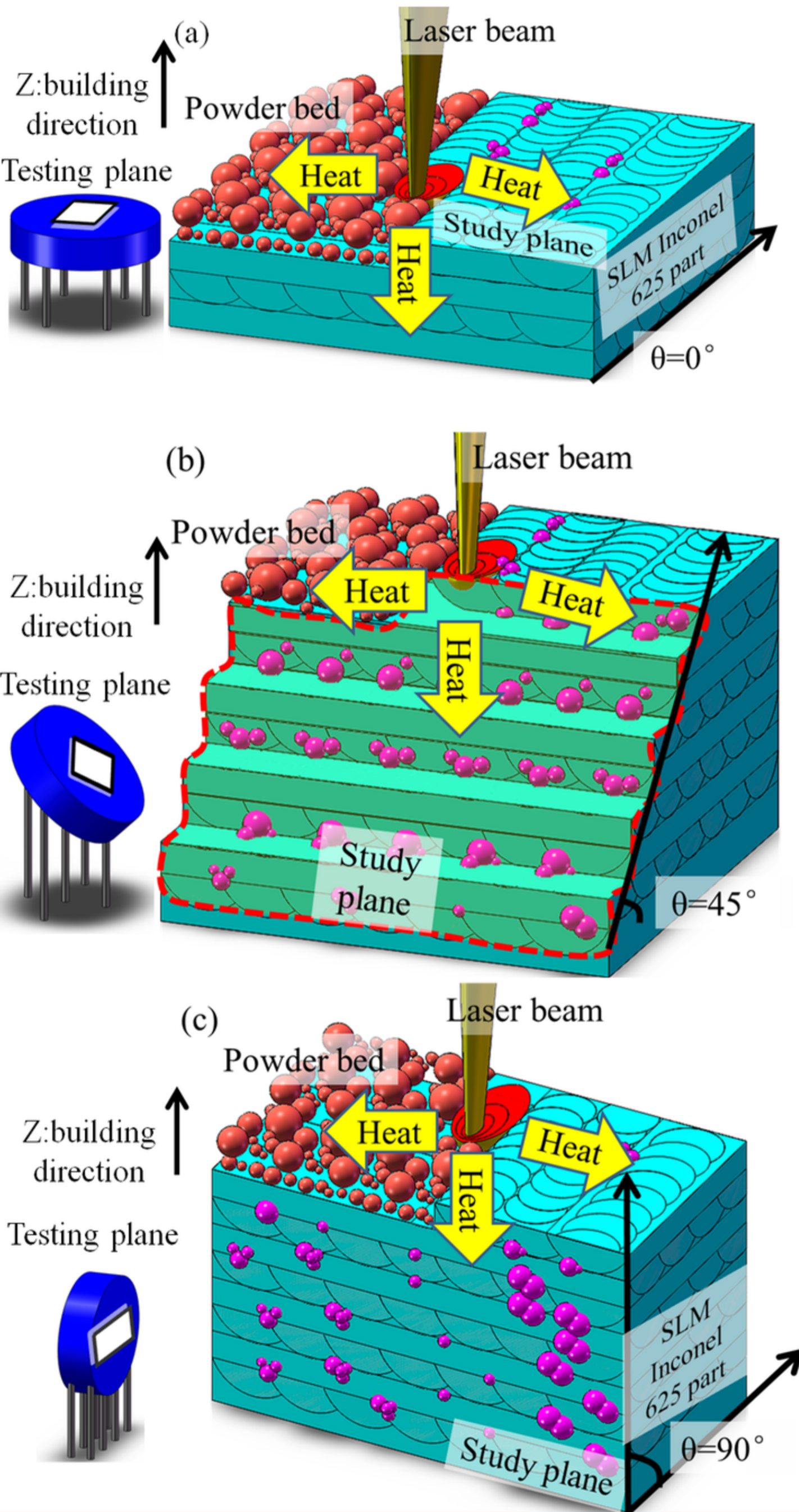
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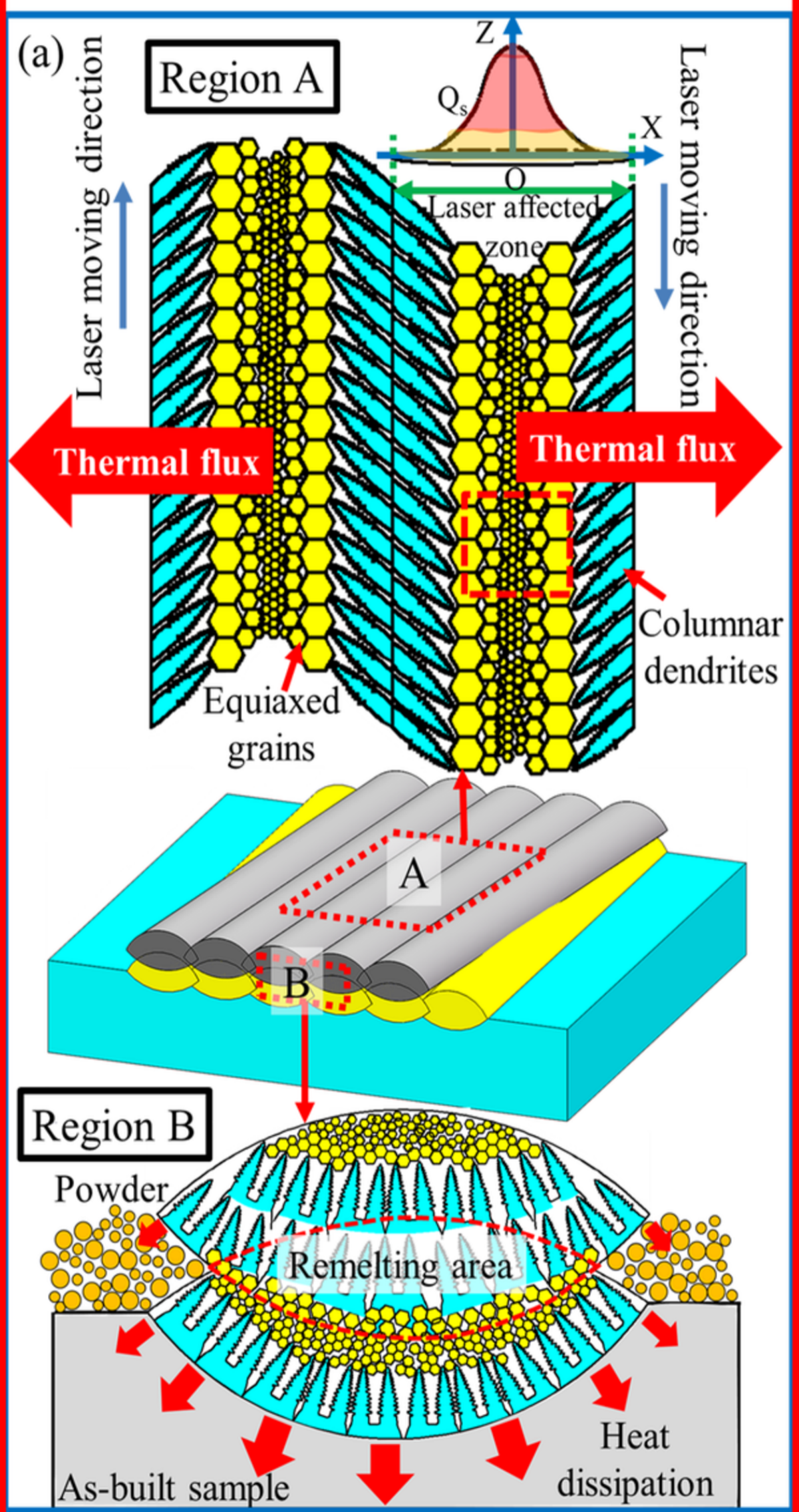
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### Surface roughness formation mechanism



### Microstructure characteristics



### Schematic diagrams of sliding wear mechanism

