



## **An Investigation on the Plane-Strain Fracture Toughness of a Water Atomized 4130 Low-Alloy Steel Processed by Laser Powder Bed Fusion**

M. Abdelwahed<sup>a, b, \*</sup>, S. Bengtsson<sup>c</sup>, M. Boniardi<sup>a</sup>, A. Casaroli<sup>a</sup>, R. Casati<sup>a</sup>, M. Vedania<sup>a</sup>

This is a post-peer-review, pre-copyedit version of an article published in journal title. The final authenticated version is available online at: <https://doi.org/10.1016/j.msea.2022.143941>

This content is provided under [CC BY-NC-ND 4.0](https://creativecommons.org/licenses/by-nc-nd/4.0/) license



1  
2  
3  
4  
5  
6  
7  
8  
9  
10  
11  
12  
13  
14  
15  
16  
17  
18  
19  
20  
21  
22  
23  
24  
25  
26  
27  
28  
29  
30  
31  
32  
33  
34  
35  
36  
37  
38  
39

## **An Investigation on the Plane-Strain Fracture Toughness of a Water Atomized 4130 Low-Alloy Steel Processed by Laser Powder Bed Fusion**

M. Abdelwahed<sup>a, b, \*</sup>, S. Bengtsson<sup>c</sup>, M. Boniardi<sup>a</sup>, A. Casaroli<sup>a</sup>, R. Casati<sup>a</sup>, M. Vedania<sup>a</sup>

<sup>a</sup>Department of Mechanical Engineering, Politecnico di Milano, Milan, Italy.

<sup>b</sup>Department of Design and Production Engineering, Faculty of Engineering, Ain Shams University, Cairo, Egypt.

<sup>c</sup>Höganäs AB, Höganäs, Sweden.

### **Abstract**

A water atomized 4130 steel powder was processed by Laser powder bed fusion and investigated both in the as built condition and after the quench and tempering heat treatment. Analyses were focused on the different microstructures developed and on steel fracture behaviour in terms of tensile fracture elongation, Charpy impact properties and linear elastic fracture toughness. Comparisons were also drawn by testing a reference 4130 steel produced using gas atomized powders. The slightly higher oxygen content found in the water atomized powders led to the formation of finely dispersed nano-size oxide particles in the steel matrix. It was found that these nano-sized inclusions have a minor effect on the tensile properties, but a significant influence on the impact toughness response. The fracture toughness tests showed that the orientation leading to propagation of cracks along the inter-layer planes represented the most critical situation, and the steel toughness could be significantly improved after the quench and tempering treatment owing to the achievement of a fully martensitic and more homogeneous microstructure.

The results suggest that the investigated water atomized low-alloy steel powder feedstock can be considered as a suitable and cheaper alternative for structural parts produced by additive manufacturing, which could replace the more popular gas atomized steel grades.

### **Keywords**

Laser Powder Bed Fusion; Water Atomized Powder; Low-Alloy Steel; Non-metallic Inclusions; Hardenability; Fracture Toughness.

### **1. Introduction**

40

41 The laser powder bed fusion (L-PBF) technology has been well recognized among the  
42 different manufacturing processes due to the remarkable ability of facilitating the  
43 production of complex-shaped metallic components with outstanding mechanical  
44 properties for light-weight applications. Though, the commercially available steels for L-  
45 PBF are still limited to stainless steels, maraging grades, and few other tool steels [1].  
46 Recently, the interest on low-alloy steels for L-PBF gained an increasing interest owing  
47 to their low-cost and favorable properties for structural applications [2–11].

48

49 The expected low cost of these steels not only derives from the lower amount of alloy  
50 elements, but it could stem from the adoption of cheaper powder production routes such  
51 as water atomization (WA). Indeed, WA is a popular process which provides cost-  
52 effective feedstocks for several powder metallurgy applications, especially in large  
53 batches for mass-production of components [20,21]. It is to consider that the nature of  
54 the atomization process, which uses water jets to atomize the steel melt, contributes to  
55 increase the oxygen level in the powder, and to the formation of micrometer- and sub-  
56 micrometer oxide-based inclusion in the steel matrix after L-PBF [9,22]. According to a  
57 previous study, the L-PBF of WA 4130 alloy could deliver outstanding tensile properties,  
58 even after various powder recycling events, though a slight reduction in strength and  
59 ductility could be measured when compared to a counterpart steel fabricated by GA  
60 powder with similar chemical composition [23].

61

62 Several efforts have been made to identify the role of alloying elements, the as-built (AB)  
63 microstructure and effects of thermal treatments on the properties of low-alloy steel  
64 grades, even if obtained from gas atomized (GA) powders, which represent the reference  
65 processing route for LPBF powders. A first investigation was carried out on a 24CrNiMo  
66 steel highlighting the heterogeneous and spatial-dependent microstructure generated by  
67 the different local thermal cycles experienced along the build height [12]. Directional-  
68 dependent tensile properties were also recorded in most of the L-PBF processed steels,  
69 which could be reduced by producing a more homogenous microstructure through a post-  
70 process thermal treatment of quench and tempering (Q&T) [3,4,10,13]. The investigated  
71 low-alloy steels showed high tensile properties, over those of the corresponding wrought  
72 alloys. However, the knowledge on other mechanical properties such as the impact and  
73 fracture toughness still needs to be improved in order to gain a greater confidence level  
74 on low-alloy steels processed by L-PBF for structural applications. In this respect, Wang  
75 and co-authors [5] tested the impact toughness of a 30CrMnSi alloy after L-PBF, and also  
76 proposed few thermal treatments to enhance their performance. The AB steel was able  
77 to absorb 11.4 J that is equivalent to almost 25% of the corresponding forged and  
78 annealed counterpart, due to the dissimilarity in microstructures. It was confirmed that the  
79 tempering temperature of a quenched microstructure had a significant effect on the

80 toughness values, since the tempering treatments performed at 550 °C and 650 °C led to  
81 impact energies of 12.4 J and 29.1 J, respectively. On the contrary, in an investigation  
82 carried out on a 30CrNiMo8 grade [10], the authors reported impact toughness values of  
83 around 100 J in both AB and Q&T conditions. It is noteworthy to mention that these energy  
84 values are significantly higher than those generally achieved by the wrought alloy. Hence,  
85 it becomes clear that a general trend on the fracture behavior of L-PBF processed steels  
86 cannot be estimated yet, and further investigations on the toughness of low-alloy steels  
87 produced by L-PBF are necessary for their adoption in structural applications.

88  
89 Fracture toughness is a significant material property that describes the structural integrity  
90 and reliability of materials, considering the combined effect of an applied load in presence  
91 of defects [14]. In L-PBF of ferrous alloys, the main factors affecting fracture toughness  
92 are directly associated to microstructure, process-related defects, residual stresses, and  
93 steel cleanliness. Suryawanshi et. al. [15] reported that the  $K_{IC}$  value of a L-PBF  
94 processed 316L steel ranges between 63 and 87  $\text{MPa}\sqrt{\text{m}}$ , which is much lower compared  
95 to the same alloy in wrought form (112 to 278  $\text{MPa}\sqrt{\text{m}}$ ). It was claimed that this drop is  
96 mainly due to reduced ductility, presence of defects, lack of transformation-induced  
97 plasticity effect in the L-PBF alloy. The authors also investigated the anisotropy in  
98 properties and showed that the fracture toughness is slightly higher when the cracks grew  
99 orthogonally to the building direction. Conversely, the  $K_{IC}$  value of a L-PBF 18Ni300  
100 maraging steel grade after aging was found to be 70 – 75  $\text{MPa}\sqrt{\text{m}}$ , which is comparable  
101 to the wrought alloy despite the dissimilarity in the microstructures [16].

102  
103 It is well known that also the presence of non-metallic inclusions in steels affects ductility  
104 and toughness. Several investigations have been carried out to understand the effect of  
105 sulfide-based inclusions on the fracture toughness of steels [17,18]. It was found that the  
106 sulfides act as crack initiators and reduce the  $K_{IC}$  value by 25 – 50%. Oxide-based  
107 inclusions are more likely to be found in L-PBF processed steels, since the feedstock  
108 powder already contains limited amounts of oxygen from the atomization process itself  
109 [9], as a result of contamination during powder handling and storage, or due to the oxygen  
110 pickup from the chamber environment during the L-PBF process. Lou and co-workers  
111 [19] reported that oxide-based inclusions stimulate an early initiation of micro-voids that  
112 dramatically affects the toughness of L-PBF 316L stainless steel compared to the wrought  
113 alloy.

114  
115  
116 The scope of this investigation is therefore primarily oriented towards a better  
117 understanding of the properties of a WA 4130-steel processed by L-PBF. The  
118 microstructures developed after L-PBF and Q&T treatments, hardness and tensile  
119 properties as well as plane strain fracture toughness of the WA steel was especially

120 studied considering the different orientations with respect to the building direction.  
121 Moreover, the contribution of oxide inclusions on the impact toughness response was  
122 evaluated by comparing the WA powder with a standard GA powder of the same alloy.  
123

124

125

126

## 2. Material and Experimental Procedures

127

128 A water atomized 4130 low-alloy steel powder was mainly used for the present  
129 investigation, whereas a reference 4130 steel obtained by GA powders was also  
130 considered for selected comparative tests. Accordingly, the steels were labelled as W-  
131 4130 and G-4130, respectively. The G-powder had a spherical morphology with particle  
132 size distribution characterized by ....., while the W-powder consisted of irregular-shaped  
133 particles featuring a size of ....., As expected, the W-type powder showed a relatively  
134 lower flowability compared to the gas atomized powder, Further powder characteristics  
135 have been provided in a previous paper [13].  
136

137

138 An open L-PBF system featuring flexible control over the laser system and optical chain  
139 (LLA150R, 3DNT) was adopted to process the steel powders under an Argon-shielding  
140 atmosphere, in which the oxygen level was kept below 2500 ppm. A single mode fiber-  
141 laser of 190 W was applied on a powder layer of 40  $\mu\text{m}$  in thickness to process the  
142 specimens. Based on a preliminary optimization of parameters, the hatching distance was  
143 set to 90  $\mu\text{m}$ , while the laser scanning velocity was defined to 650 mm/s and 500 mm/s  
144 for the G-4130 and the W-4130 powders, respectively. Moreover, a stripe scanning  
145 strategy was used with an incremental rotation angle of  $67^\circ$  for each deposited layer.

146

147 Chemical analyses were performed by a Bruker Q4 TASMAN equipment to measure the  
148 composition of the steel specimens after L-PBF. Microstructural investigations were  
149 performed on the processed specimens directly after polishing down to 50 nm colloidal  
150 silica and etching. Nital reagent was generally used as etchant to reveal the  
151 microstructure whereas Picral was preferred to highlight the melt pool boundaries. The  
152 microstructure was characterized by a Nikon Eclipse LV150NL optical microscope, and a  
153 Zeiss Sigma 500 VP field-emission scanning electron microscope equipped with energy-  
154 dispersive X-ray spectroscopy. Further characterization was carried out by electron  
155 backscattered diffraction (EBSD) operating with an accelerating voltage of 20 KV and a  
156 step size resolution of 0.35  $\mu\text{m}$ .

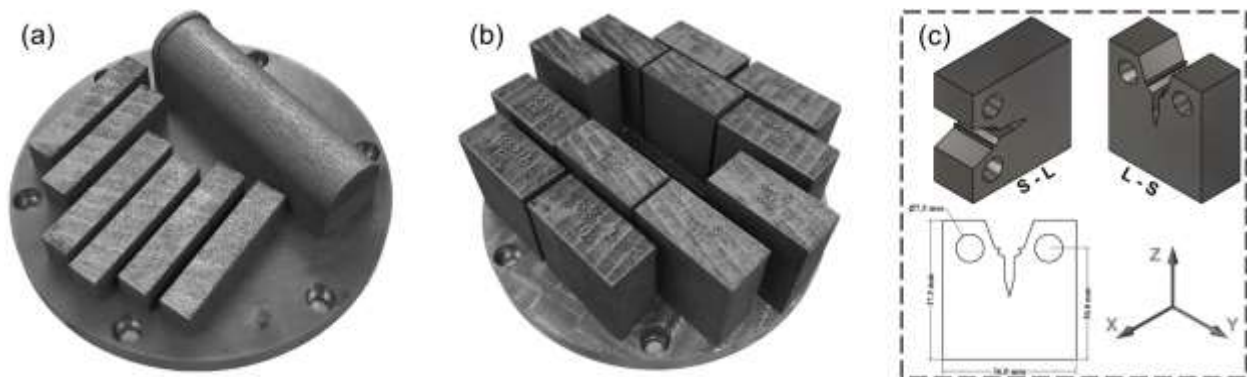
157 Thermodynamic simulations to support phase identification were conducted with  
158 ThermoCalc AB software relying on the TCFE9 Steels/Fe-alloys database.

159

159 Jominy specimens of 27 mm in diameter and 103 mm in length were fabricated by L-PBF

160 and machined to a final standard size in order to measure the hardenability of both steels.  
161 The mechanical properties of the steels were evaluated by tensile testing, impact  
162 toughness, and plane-strain fracture toughness tests. Sub-sized cylindrical dog-bone  
163 specimens were extracted from bars fabricated orthogonally to the building direction and  
164 tested according to ASTM E8M standard. Charpy V-notch specimens were machined  
165 from 2 mm-oversized bars oriented in the same direction. EN ISO 148-1 standard  
166 procedures were followed to determine the impact toughness at room temperature.

167  
168 Oversized coupons (41 mm x 39 mm x 18 mm) for the fracture toughness tests were  
169 fabricated from the WA feedstock considering two orientations, where the crack  
170 propagation plane was parallel (L-S) or orthogonal (S-L) to the building direction, as  
171 displayed in Fig. 1. The specimens were tested either in AB state or after water quenching  
172 from 840 °C and tempering at 550 °C for 1 hour (Q&T). Afterwards, the coupons were  
173 machined to obtain compact tension (CT) fracture toughness specimens, according to  
174 ASTM E1820 standard with step notches to measure the crack opening displacement  
175 (COD) by a clip-on extensometer. Side grooves were also machined to ensure a crack-  
176 front flatness and to provide reliable measurements of the crack length. Fatigue pre-  
177 cracking and fracture testing campaign were conducted by using a servo-hydraulic  
178 machine (type MTS 810). After performing the tests, specimens were oxidized in a muffle  
179 furnace, immersed in liquid nitrogen and broken in a brittle manner. The standard was  
180 followed to assess the initial and the final crack length, using nine equally-spaced  
181 positions to determine the average initial crack size. All tests were carried out at room  
182 temperature, using at least three specimens for each condition. The fracture surfaces of  
183 specimens were analysed by a Zeiss SteREO Discovery v12 stereoscope as well as a  
184 Zeiss EVO-50XVP SEM to define the failure mechanisms.



185  
186 Fig. 1: views of the fabricated (a) Jominy and Charpy specimens, (b) fracture toughness  
187 coupon blocks, and (c) a schematic representation of the orientation of the fracture  
188 toughness specimens. The z-axis corresponds to building direction, normal to baseplate  
189 plane (x-y)

190

191 **3. Results and Discussion**

192

193 3.1. Chemical composition after L-PBF

194

195 The chemical composition of the investigated steels after L-PBF is given in Table 1. A  
196 lower carbon content is measured for the L-PBF W-4130 steel compared to the gas  
197 atomized counterpart. This could be due to variability of elemental composition during  
198 feedstock preparation and partially to loss of carbon during L-PBF processing. It has been  
199 reported that the laser processing of GA low-alloy steel powders is generally associated  
200 with a reduction in carbon content between 5% and 12% in the AB components,  
201 regardless the differences in composition and processing conditions [11,12,24,25]. A  
202 similar amount of 6% loss in carbon was also measured for a L-PBF steel processed from  
203 a WA powder with 0.33 wt.% C [26].

204

205

Table 1. Chemical analysis of the L-PBF processed steels.

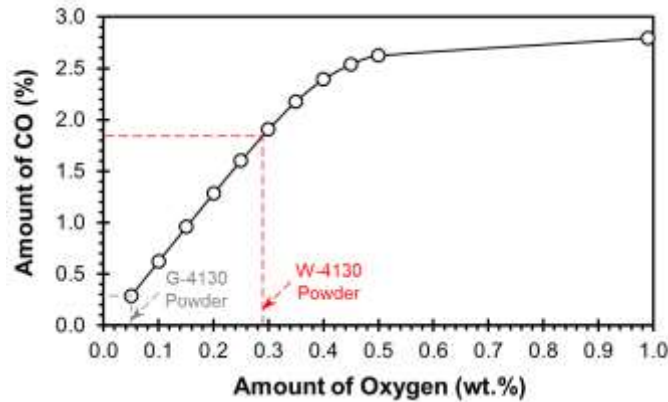
Alloy	Chemical Composition (wt. %)						
	C	Cr	Mo	Si	Mn	Ni	O*
G-4130	0.317	1.182	0.152	0.271	0.694	0.144	0.05
	± 0.005	± 0.006	± 0.005	± 0.008	± 0.003	± 0.009	
W-4130	0.234	1.330	0.285	0.629	0.050	0.090	0.29
	± 0.004	± 0.005	± 0.007	± 0.017	± 0.000	± 0.004	

206

\*Amount of oxygen was measured on the starting feedstock powders.

207

208 The reduced amount of carbon in the as-built W-4130 specimens could be partially related  
209 to the higher oxygen level in the starting powder, which stimulate the formation of CO gas  
210 during the melting stages. Thermodynamic simulations were performed on a simplified  
211 Fe-0.3C system with various oxygen contents to better quantify this effect. The results  
212 are displayed in Fig. 2 and indicate that higher contents of oxygen contribute to a  
213 pronounced increase in the fraction of CO above the liquidus temperature. Therefore, it  
214 could be assumed that the oxygen content in the starting powder plays the significant role  
215 on the carbon loss during L-PBF of steels.



216

217 Fig. 2: Mole fraction of CO gas above 1600 °C as a function of oxygen content in a Fe-  
 218 0.3C system (computed under equilibrium hypothesis).

219

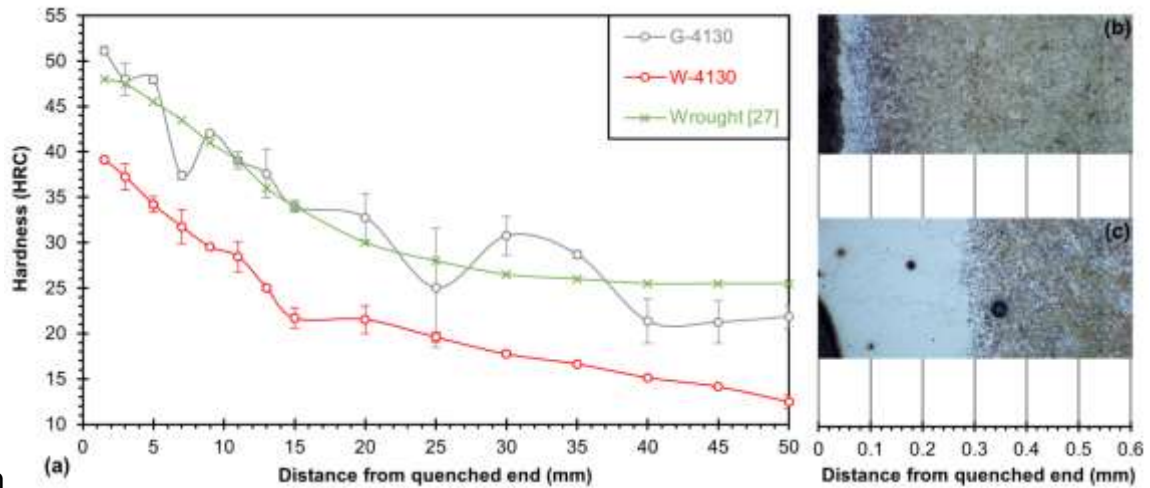
### 220 3.2. Hardenability of the L-PBF 4130 Steels

221

222 Given the differences in composition (especially for C and Mn) of the two investigated  
 223 steels, their hardenability was experimentally measured in order to improve the  
 224 understanding of the achievable properties. The results of the Jomini tests are  
 225 summarized in Fig. 3. It is revealed that the hardness profile of the G-4130 steel follows  
 226 the mean values also expected by a similar wrought alloy [27]. However, a significant  
 227 reduction in the hardness values was measured for the W-4130 steel, indicating a lower  
 228 hardenability. This effect is supposed to be related to the modified composition of the W-  
 229 4130 powder, featuring lower C and lack of Mn. In addition, oxide-based inclusions, more  
 230 frequent in the W-4130 version, could stimulate the nucleation of other constituents rather  
 231 than martensite, hence play an important role on hardenability. Finally, the micrographs  
 232 of Fig. 3(b) and 3(c) show the decarburized layer formed on surface of Jomini samples  
 233 during the austenitizing step, prior to quenching. Based on microstructure observation, a  
 234 ferrite layer of about 300  $\mu\text{m}$  was formed on the surface of the W-4130 specimen,  
 235 corresponding to almost 3 times that of the G-4130 steel. It is assumed that the lower  
 236 starting value of C would ease the achievement of the ferritic field when the steels are  
 237 held at high temperature in a furnace with oxidizing air environment, hence widening the  
 238 ferritic layer in the W-4130 specimens.

239 Beyond the decarburized surface, a fully martensitic matrix up to 6 mm could be observed  
 240 for the G-4130, while a mixture of martensite and other ferrite constituents could be  
 241 detected for the WA alloy confirming the relatively lower hardenability after quenching.





242

m

243 Fig. 3: (a) Hardenability curves of the investigated steels. The hardness values of a  
 244 wrought 4130 steel [27] is also reported. Optical micrographs of sections portions of the  
 245 Jominy specimens close to surface at the end quench location for (b) G-4130 and (c) W-  
 246 4130 steel.

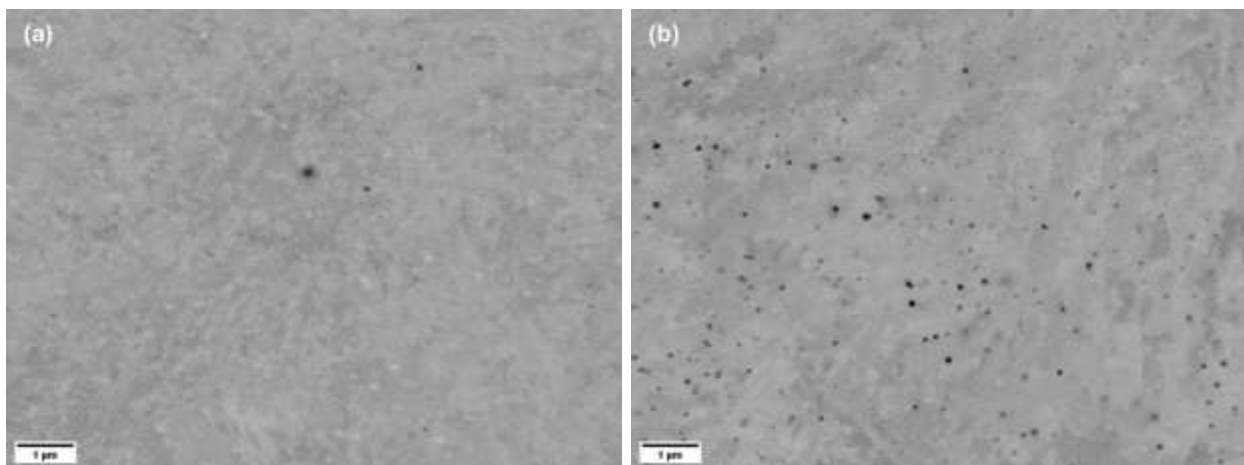
247

### 248 3.3. Microstructure Evolution

249

250 Fig. 4 depicts high magnification FE-SEM micrographs, highlighting the finely dispersed  
 251 sub-micrometric oxide inclusions in the matrix of the investigated steels. The  
 252 thermodynamic simulations estimated the formation of dual-phase oxides of type MnO-  
 253 SiO<sub>2</sub> in the G-4130 steel, whereas mono-phase SiO<sub>2</sub> inclusions in the W-4130 alloy are  
 254 expected. The elemental compositions measured by EDS point analyses confirmed the  
 255 hypotheses by which the oxides found in the G-4130 steel were rich in Mn, Si, and O  
 256 while those embedded in the matrix of the W-4130 steel were mainly formed by Si and  
 257 O. Moreover, it could be observed that the fraction of oxides occupying the W-4130 matrix  
 258 was about 0.5% while that observed in the G-4130 steel was lower than 0.1%.

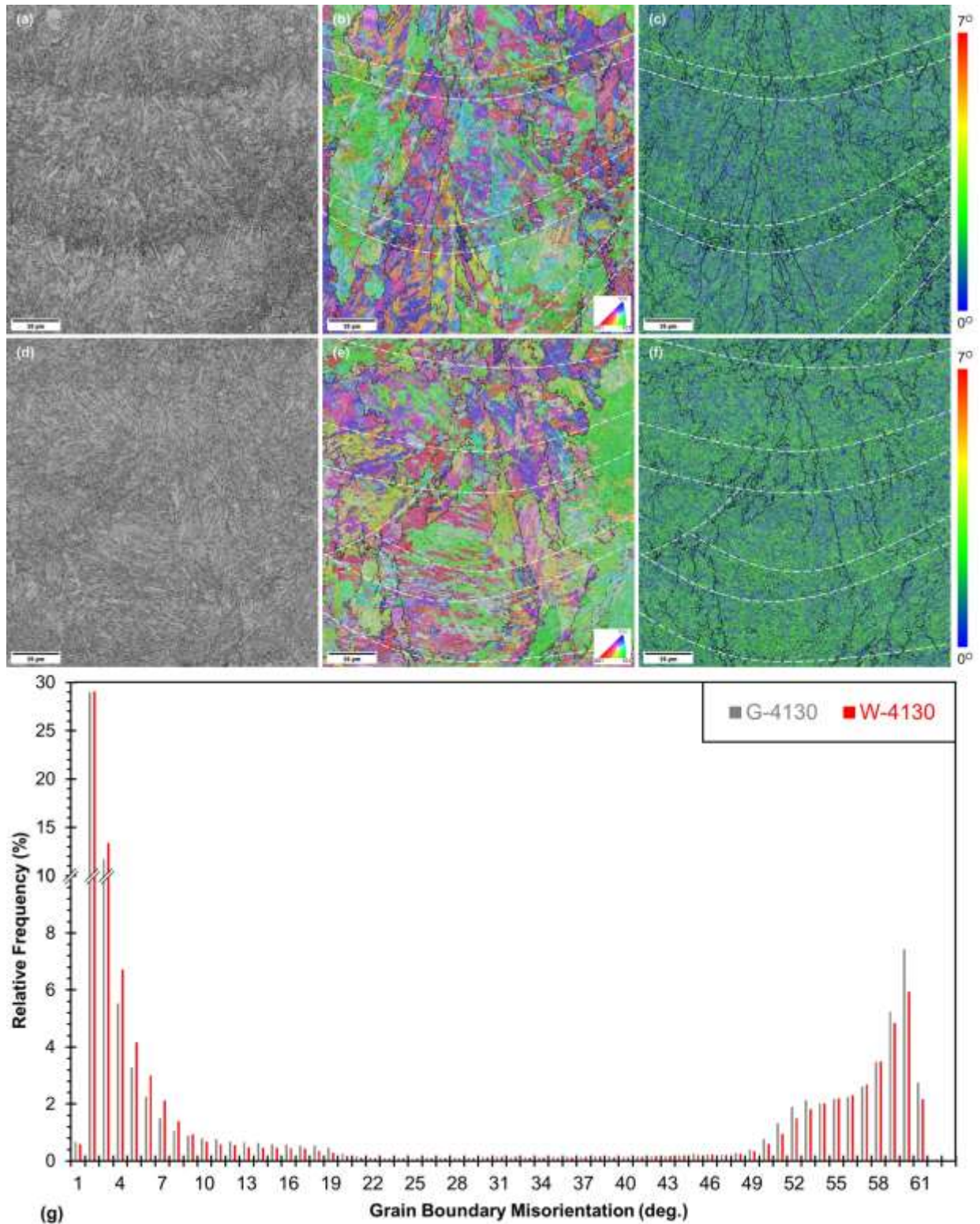
259



260 Fig. 4: FE-SEM micrographs showing the non-metallic inclusions in the (a) G-4130 and  
261 (b) W-4130 steels.  
262

263 EBSD analyses results carried out on the investigated steels after L-PBF are collected in  
264 Fig. 5. In Fig. 5 (a) and 5(d) band contrast (BC) images are provided. The BC method  
265 has been widely used as an approach to differentiate between phases having similar  
266 crystal structure such as martensite, bainite, and ferrite [28,29]. This method is based on  
267 the diffraction pattern intensity during data acquisition in which the phase with higher  
268 lattice imperfections provides a lower pattern quality, corresponding to darker regions.  
269 From obtained results, it could be clearly observed that the melt pool boundaries in the  
270 steel are highlighted by relatively lower pattern intensities, indicating more imperfections  
271 located in those regions. In addition, finer microconstituents are found at the melt pool  
272 boundaries compared to other zones. This is presumably due to the relatively higher  
273 cooling rates that are experienced close the melt pool boundaries during solidification  
274 [30]. The inverse pole figures (IPFs) provided in Fig. 5(b) and 5(e) show highly  
275 misoriented microstructures and an epitaxial growth of the grains across several melt  
276 pools. Moreover, the Kernel average misorientation (KAM) analyses depicted in Fig. 5(c)  
277 and 5(f) indicate a nearly homogenous local strain distribution within the microstructures  
278 after L-PBF.

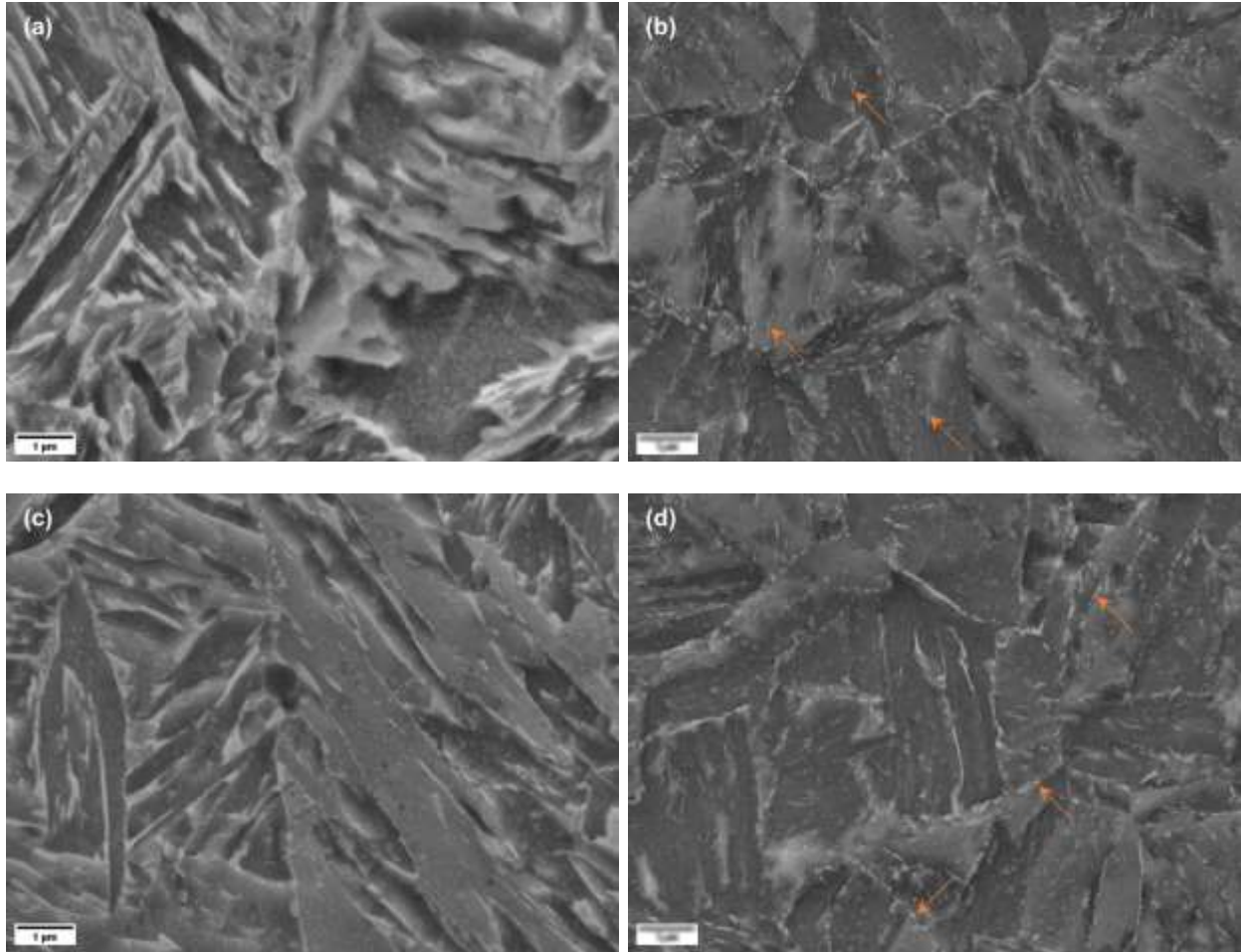
279 Finally, the analysis about grain boundary misorientation profiles provided in Fig 5(g)  
280 reveals a substantially comparable trend between the two investigated steels. The as-  
281 built microstructures are primarily separated by a high fraction of low angle grain  
282 boundaries denoting the dominant presence of sub-structures within the martensitic  
283 blocks. Furthermore, a second population of high-angle grain boundaries centered at  
284 about  $60^\circ$  was detected. It has been demonstrated that a strong peak at this particular  
285 angle is usually detected for martensitic structures [31,32], which corresponds to the  
286 block boundaries [33].



287

288 Fig. 5: EBSD measurements showing band contrast micrographs, IPF, and KAM maps,  
 289 respectively, for (a-c) G-4130 and (d-f) W-4130 steels. (g) grain boundary misorientation  
 290 profile for the investigated steels.

291 Fig. 6 displays high magnification FE-SEM micrographs of the investigated steels in both  
292 AB and Q&T conditions. During L-PBF, the progressive overlapping of layers and related  
293 heat dissipation generates a partial tempering effect on the rapidly cooled microstructure,  
294 which in turn leads to the precipitation of carbides [8,12]. Conversely, the conventional  
295 quenching and isothermal tempering resulted in fine dispersed secondary phase particles  
296 decorating the block and lath boundaries, as shown in Fig 6(b) and 6(d). According to the  
297 literature, such phases are assumed to be carbides mainly of the  $M_{23}C_6$  type [23].  
298



299

300

301 Fig 6: FE-SEM micrographs of G-4130 in (a) AB and (b) Q&T conditions, W-4130 in (c)  
302 AB and (d) Q&T conditions. (arrows refers to the precipitated carbides).  
303

### 304 3.4. Mechanical Properties

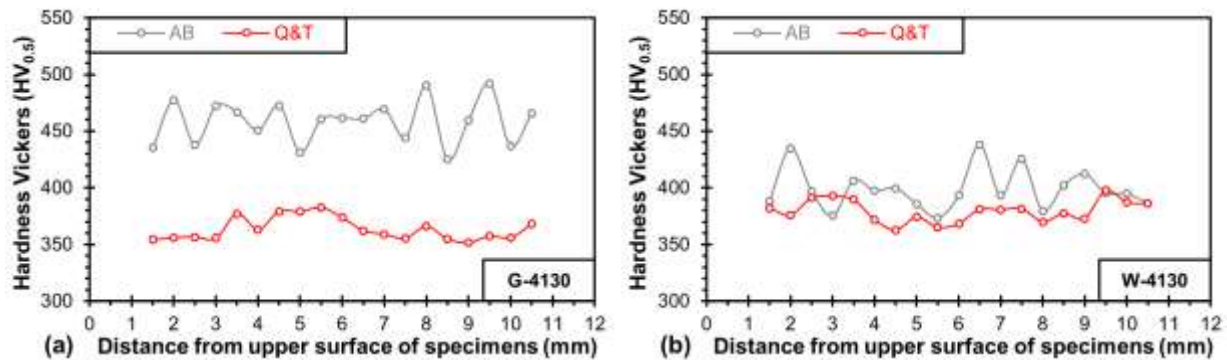
305

#### 306 3.4.1. Microhardness

307

308 Hardness profiles were measured across the thickness of the Charpy specimens to  
309 collect further information about the actual hardening achieved by the two steels either

310 after rapid quenching from the L-PBF process (AB condition) or after the conventional  
 311 Q&T treatment. The results are displayed in Fig. 7. Both steels in AB condition showed  
 312 fluctuations in the microhardness values owing to the heterogeneous microstructure  
 313 encountered when crossing the partially overlapped melt pools (see description in section  
 314 3.3). According to the lower C content and the reduced hardenability measured for the  
 315 W-4130 steel, the average hardness level achieved ( $398 \pm 18 \text{ HV}_{0.5}$ ) was lower than that  
 316 of the G-4130 version ( $459 \pm 23 \text{ HV}_{0.5}$ ) in AB condition. On the contrary, after Q&T  
 317 treatment, the investigated steels provided a more homogeneous microstructure and  
 318 reached similar hardness values, of  $379 \pm 9 \text{ HV}_{0.5}$  and  $363 \pm 9 \text{ HV}_{0.5}$ , for the W-4130 and  
 319 G-4130 steels, respectively. It is to remark that both alloys did not show any drop in  
 320 hardness towards the center of specimen thickness, suggesting an acceptable  
 321 hardenability within the 12- thickness sample volume.

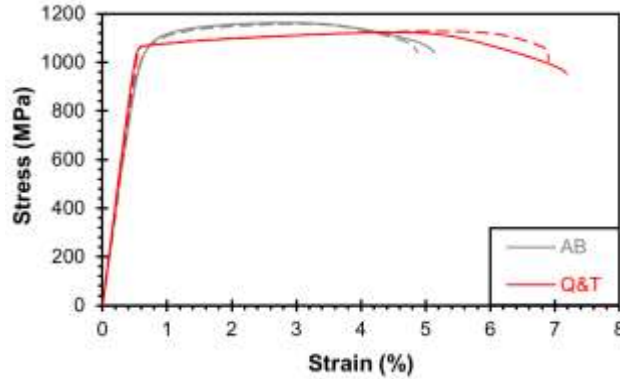


322 (a) Distance from upper surface of specimens (mm) (b) Distance from upper surface of specimens (mm)

323 Fig. 7: Microhardness profiles of the investigated (a) G-4130 and (b) W-4130 steels.

324  
 325 3.4.2. Tensile Behavior

326  
 327 The tensile curves recorded from the W-4130 steel in AB and Q&T conditions are  
 328 displayed in Fig. 8. The curves show an almost flat plastic plateau for the heat-treated  
 329 steel after the linear elastic stage, while a more evident strain hardening response was  
 330 detected in AB condition. Moreover, the acquired results showed that the steel in AB  
 331 condition was able to deliver the highest offset yield and ultimate tensile strength, of  $1068$   
 332  $\pm 6 \text{ MPa}$  and  $1160 \pm 2 \text{ MPa}$ , respectively. Comparably, the Q&T steel provided a yield  
 333 strength of  $1070 \pm 0 \text{ MPa}$  and tensile strength of  $1126 \pm 2 \text{ MPa}$  with a significant  
 334 improvement in the fracture elongation from  $5.00 \pm 0.10 \%$  to  $7.05 \pm 0.15 \%$ , which is  
 335 supposed to be promoted by the more homogeneously tempered microstructure generated  
 336 after the post-process thermal treatment.



337

338 Fig. 8: Tensile curves of the investigated W-4130 steel (dashed-lines correspond to the  
 339 replicated results).

340

### 341 3.4.3. Impact Toughness

342

343 The energy absorbed during the impact tests of both steels are summarized in Table 2.  
 344 It can be observed that the Q&T treatment enhances the impact toughness of the G-type  
 345 steels with respect to the AB condition, though the measured values are still lower than  
 346 those expected by the standard 4130 wrought steel [27]. Conversely, in the WA steel, the  
 347 Q&T treatment did not provide any substantial improvement in toughness with respect to  
 348 AB condition. From a direct comparison between the two steels variants, it is readily seen  
 349 that the W-4130 steel shows lower toughness properties in both investigated conditions.  
 350 This behaviour is supposed to be related to the larger presence of oxide-based inclusions,  
 351 that could assist crack initiation and growth. A similar behaviour was also reported by  
 352 Salandre et. al. [34] who investigated an L40 tool steel which provided an impact  
 353 toughness of 50 J when using gas atomized powders, that significantly dropped to 13 J  
 354 using water atomized powders of the same steel grade.

355

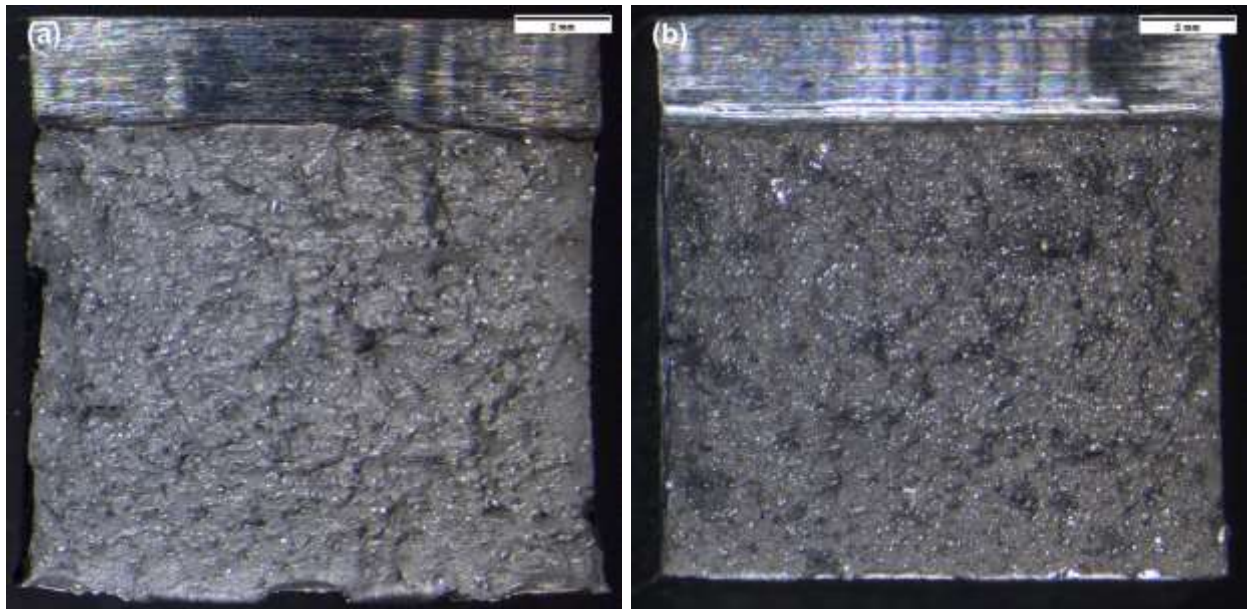
356 Table 2. Impact energy of the investigated low-alloy steels along with available data  
 357 from the literature for similar grades.

Steel Grade	Condition	Impact Energy (J)
<b>G-4130</b>	AB	18.9 ± 3.1
	Q&T-550 °C	25.3 ± 2.4
<b>W-4130</b>	AB	12.3 ± 0.5
	Q&T-550 °C	13.3 ± 0.5
<b>30CrMnSi</b> [5]	AB	11.4 ± 0.5
	Q&T-550 °C	12.4 ± 0.6
	Q&T-650 °C	29.1 ± 1.0
<b>30CrNiMo8</b> [10]	AB	87.8 ± 11.9
	Q&T-560 °C	108.8 ± 2.3

358

359 Fig. 9(a) and 9(b) display representative stereo-microscope images of Charpy fracture  
360 surfaces of the G-4130 and the W-4130 alloys, respectively, in Q&T condition. A slightly  
361 higher area fraction occupied by lateral shear lips could be observed for the surfaces of  
362 the G-4130 steel, in agreement with its more ductile behaviour. A similar trend was also  
363 observed for the same steels in AB condition. The higher magnification SEM micrographs  
364 given in Fig 10, generally reveal the presence of fine dimples covering most of the fracture  
365 surfaces of both steels in both investigated tempers, indicating a microscopically ductile  
366 fracture mechanism taking place during the impact test. A few quasi-cleavage flat  
367 surfaces were additionally observed (more frequent in the Q&T alloys), as highlighted by  
368 the yellow arrows in Fig. 10(b) and 10(d). The relatively large craters observed in Fig.  
369 10(d) refer to micro-porosities, presumably generated during the laser process by  
370 entrapped or released gases. It is to remark that few typical L-PBF lack of fusion defects  
371 were also occasionally detected on the fracture surfaces.

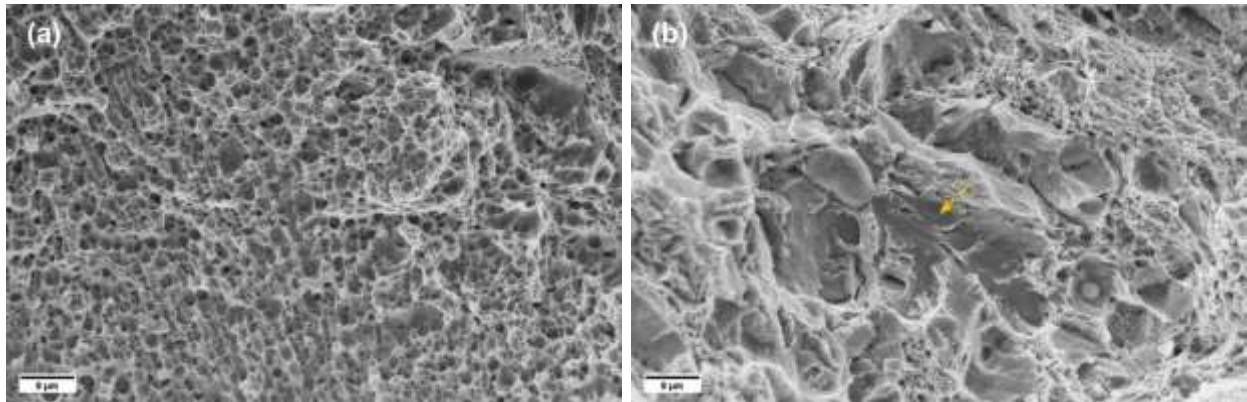
372 The most significant difference between the surfaces of the two investigated steels mainly  
373 consists of a wider population of spherical oxide-based inclusions found in micro- and  
374 submicrometer dimples (compare Fig 10(a) and Fig. 10(c)) of the W-4130 steel samples,  
375 that are believed to rule the different impact toughness response of the investigated  
376 steels.



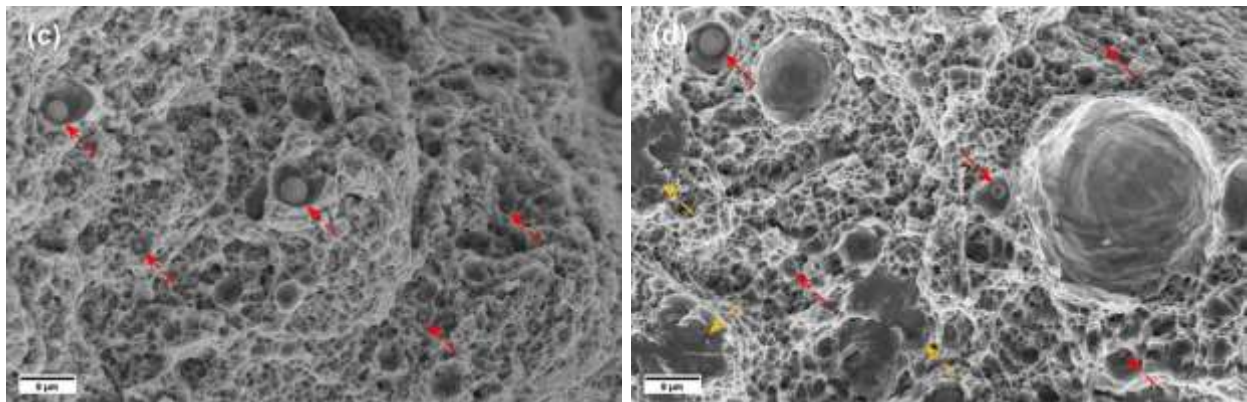
377

378 Fig. 9: Stereographic images of the fracture surface after Charpy impact tests of the (a)  
379 G-4130 and (b) W-4130 steels, both in Q&T condition.

380



381



382 Fig. 10: SEM fractographs of the G-4130 steel in (a) AB and (b) Q&T condition, W-4130  
383 steel in (c) AB and (d) Q&T condition. (yellow and red arrows refer to quasi-cleavage  
384 planes and oxide-based inclusions, respectively).

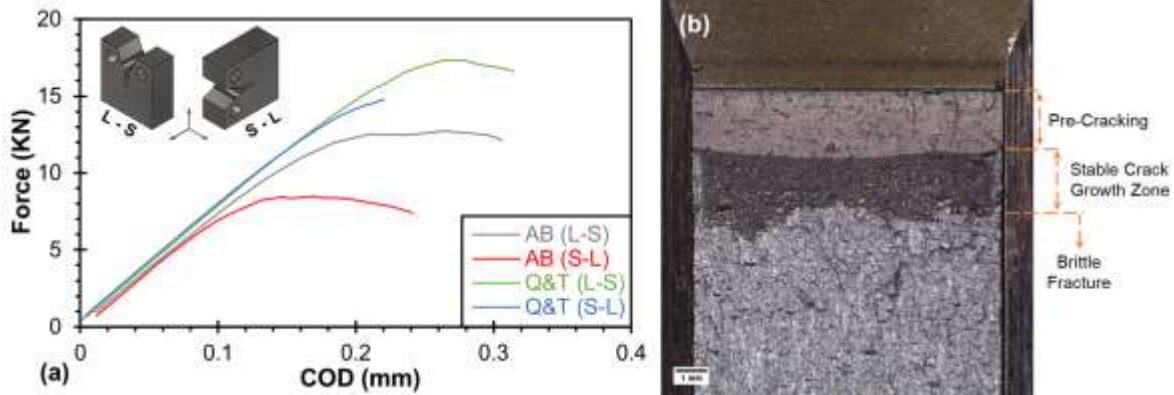
385

#### 386 3.4.4. Fracture Toughness

387

388 Typical curves of the load vs. COD of the W-4130 steel in different conditions are provided  
389 in Fig.11(a), while a stereomicrograph of a fractured CT specimen showing the different  
390 zones of the crack development is given in Fig. 11(b). An elastic-plastic deformation  
391 behavior was observed for the WA steel in both conditions investigated and an anisotropic  
392 response was measured along the different loading orientations. The obtained  $J_{IC}$  values  
393 and the calculated  $K_{JIC}$  data are summarized in Table 3. It is observed that the crack  
394 propagation in a plane orthogonal to the building direction is the most critical situation in  
395 the L-PBF W-4130 steel. It is also found that the Q&T treatment is able to reduce the  
396 anisotropy and to improve the material toughness. Additionally, it is worth mentioning that  
397 the performance of the Q&T W-4130 alloy was found to be comparable with that of similar  
398 low-alloy steel grade processed from a GA powder, as reported in Table 3.





399

400 Fig. 11: Load vs. COD curves (a) of the W-4130 steel tested in different conditions and  
 401 (b) typical CT fracture toughness specimens after fracture.

402

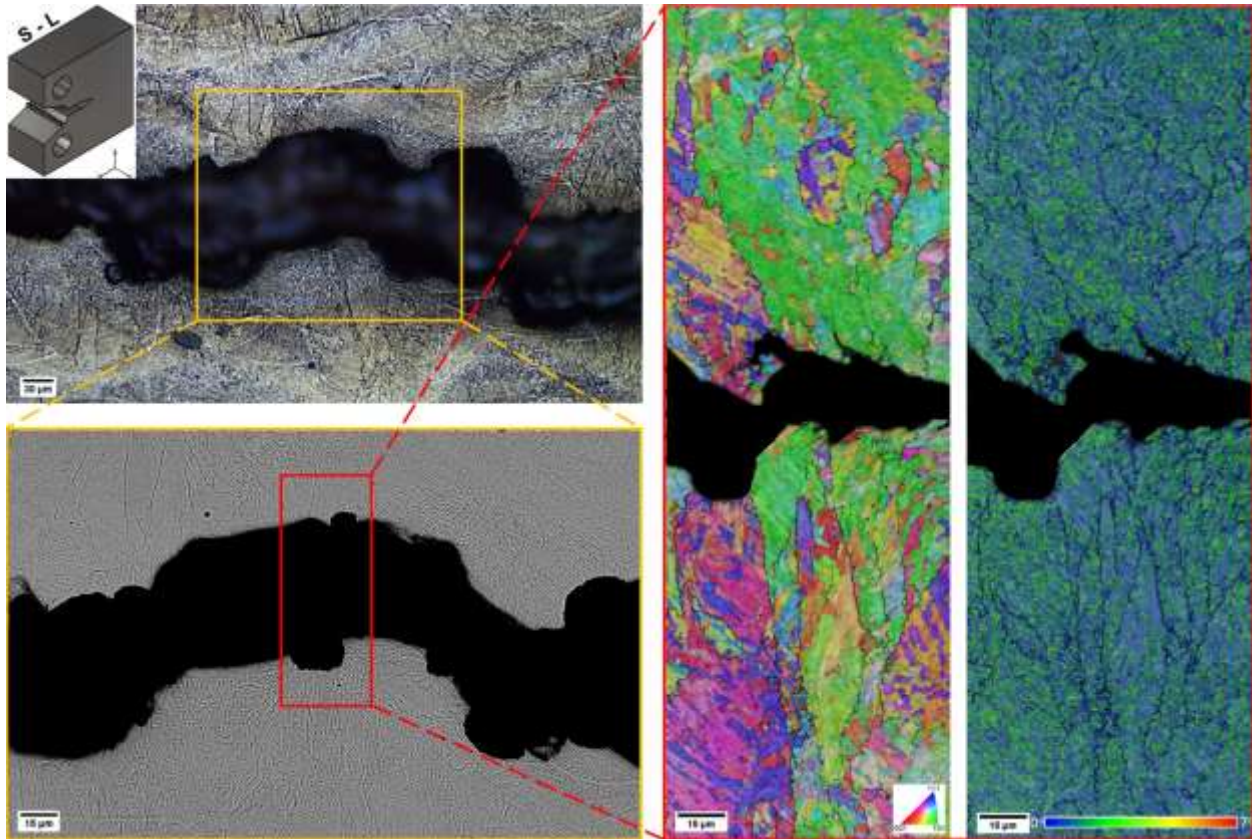
403 Table 3. Plane-strain fracture toughness values of the L-PBF processed W-4130 steel  
 404 in different conditions.

Alloy	Condition	Orientation	$J_{IC-effective}$ (KJ/mm <sup>2</sup> )	$K_{JIC}$ (MPa√m)
W-4130	AB	L-S	14.3 ± 1.9	56.0 ± 3.8
		S-L	5.70 ± 0.6	35.5 ± 1.8
	Q&T	L-S	25.1 ± 0.2	74.3 ± 0.3
		S-L	15.6 ± 3.8	58.0 ± 7.5
4340 [4]	Q&T	L-S	-	54.3
		S-L	-	52.9

405

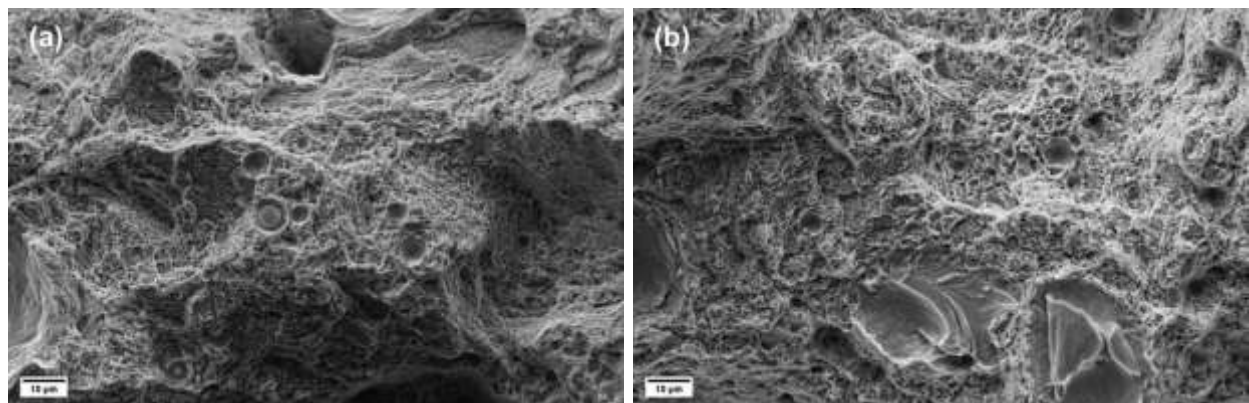
406 Fig. 12 displays the analyses performed on a cross section of a S-L W-4130 specimen  
 407 tested in as built condition, when considering a region corresponding to the stable crack  
 408 growth. Fractographs collected from the region corresponding to stable crack growth of  
 409 the tested CT specimens of the W-4130 steel are depicted in Fig. 13. SiO<sub>2</sub> inclusion were  
 410 again observed on the fracture surfaces along with few microporosities. A dimple rupture  
 411 was detected for both investigated conditions and specimen orientation, highlighting the  
 412 plastic deformation which took place during testing and the intrinsically ductile failure  
 413 mechanism in the stable crack growth zone.

414



415

416 Fig. 12: Microstructural analyses on the vertical cross section of a fractured W-4130 CT  
 417 specimen in AB condition and tested along S-L orientation.  
 418



419

420 Fig. 13: Fracture surfaces of (a) L-S and (b) S-L W-4130 specimens in as-built condition.  
 421

#### 422 4. Conclusions

423

424 The present study focused on the microstructural and fracture behavior of a water  
 425 atomized 4130 low-alloy steel processed by Laser powder bed fusion starting. The main  
 426 outcomes are summarized as follows:

427  
428  
429  
430  
431  
432  
433  
434  
435  
436  
437  
438  
439  
440  
441  
442  
443  
444  
445  
446  
447

- The water atomized feedstock contains a higher level of oxygen than a corresponding gas atomized powder, which leads to a different population of non-metallic inclusions after Laser processing. A reduced hardenability of the water atomized steel was also measured in comparison to the gas atomized counterpart, due to variations in chemical compositions and as a result of the larger presence of SiO<sub>2</sub>-type inclusions that could stimulate on cooling the nucleation of other phases rather than martensite.
- Consistently the Charpy-V impact toughness of the Water atomized 4130 steel showed a slight drop over the gas atomized counterpart. Despite the effects induced by the dispersed inclusions, the obtained impact toughness values were comparable to those provided by other L-PBF low-alloy steels published in the literature.
- The fracture toughness behavior suggested an easier condition for crack propagation in a direction orthogonal to the building direction, when considering both as-built and quench and tempered conditions.
- The quench and tempering treatment generated a more homogenous microstructure and significantly enhanced both the tensile fracture elongation and the fracture toughness behavior with respect to as built condition. However, no pronounced improvement was measured in the case of impact toughness property.

448

## 449 **Acknowledgements**

450  
451  
452  
453  
454  
455  
456  
457  
458

The investigation was carried out within the frame of the SPAcEMAN project funded by EIT-Raw Materials in 2018-2021. The Italian Ministry of Education, University and Research is also acknowledged for the support provided through the project "Department of Excellence LIS4.0 - Lightweight and Smart Structures for Industry 4.0". Marawan Abdelwahed was funded by a full Ph.D. scholarship from the Ministry of Higher Education of the Arab Republic of Egypt. The authors would like to express their gratitude to Luca Signorelli, Lorenzo Giudici, Ludovica Rovatti, and Michael Ishola for their collaboration and technical support at Politecnico di Milano.

459

## 460 **Data availability**

461  
462  
463

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

464

## 464 **References**

465  
466

[1] P. Bajaj, A. Hariharan, A. Kini, P. Kürnsteiner, D. Raabe, E.A. Jäggle, Steels in

- 467 additive manufacturing: A review of their microstructure and properties, *Mater. Sci.*  
468 *Eng. A.* 772 (2020) 138633. <https://doi.org/10.1016/j.msea.2019.138633>.
- 469 [2] W. Wang, S. Kelly, A Metallurgical Evaluation of the Powder-Bed Laser Additive  
470 Manufactured 4140 Steel Material, *JOM.* 68 (2016) 869–875.  
471 <https://doi.org/10.1007/s11837-015-1804-y>.
- 472 [3] J.J.S. Dilip, G.D.J. Ram, T.L. Starr, B. Stucker, Selective laser melting of HY100  
473 steel: Process parameters, microstructure and mechanical properties, *Addit.*  
474 *Manuf.* 13 (2017) 49–60. <https://doi.org/10.1016/j.addma.2016.11.003>.
- 475 [4] E. Jelis, M.R. Hespos, N.M. Ravindra, Process Evaluation of AISI 4340 Steel  
476 Manufactured by Laser Powder Bed Fusion, *J. Mater. Eng. Perform.* (2018).  
477 <https://doi.org/10.1007/s11665-017-2989-8>.
- 478 [5] L. zhi Wang, J. jiao Wu, X. fei Huang, X. fu Hong, SLM-manufactured 30CrMnSiA  
479 alloy: Mechanical properties and microstructural effects of designed heat treatment,  
480 *Opt. Laser Technol.* 107 (2018) 89–98.  
481 <https://doi.org/10.1016/J.OPTLASTEC.2018.05.020>.
- 482 [6] M. Wei, S. Chen, L. Xi, J. Liang, C. Liu, Selective laser melting of 24CrNiMo steel  
483 for brake disc: Fabrication efficiency, microstructure evolution, and properties, *Opt.*  
484 *Laser Technol.* 107 (2018) 99–109.  
485 <https://doi.org/10.1016/J.OPTLASTEC.2018.05.033>.
- 486 [7] X. Li, Y.H. Tan, H.J. Willy, P. Wang, W. Lu, M. Cagirici, C.Y.A. Ong, T.S. Herng, J.  
487 Wei, J. Ding, Heterogeneously tempered martensitic high strength steel by  
488 selective laser melting and its micro-lattice: Processing, microstructure, superior  
489 performance and mechanisms, *Mater. Des.* 178 (2019) 107881.  
490 <https://doi.org/10.1016/J.MATDES.2019.107881>.
- 491 [8] J. Damon, R. Koch, D. Kaiser, G. Graf, S. Dietrich, V. Schulze, Process  
492 development and impact of intrinsic heat treatment on the mechanical performance  
493 of selective laser melted AISI 4140, *Addit. Manuf.* 28 (2019) 275–284.  
494 <https://doi.org/10.1016/j.addma.2019.05.012>.
- 495 [9] M. Abdelwahed, R. Casati, S. Bengtsson, A. Larsson, M. Riccio, M. Vedani, Effects  
496 of Powder Atomisation on Microstructural and Mechanical Behaviour of L-PBF  
497 Processed Steels, *Metals (Basel)*. 10 (2020) 1474.  
498 <https://doi.org/10.3390/met10111474>.
- 499 [10] L. Zumofen, A. Kirchheim, H.-J. Dennig, Laser powder bed fusion of 30CrNiMo8  
500 steel for quenching and tempering: examination of the processability and  
501 mechanical properties, *Prog. Addit. Manuf.* 5 (2020) 75–81.  
502 <https://doi.org/10.1007/s40964-020-00121-x>.
- 503 [11] R. Seede, D. Shoukr, B. Zhang, A. Whitt, S. Gibbons, P. Flater, A. Elwany, R.  
504 Arroyave, I. Karaman, An ultra-high strength martensitic steel fabricated using  
505 selective laser melting additive manufacturing: Densification, microstructure, and  
506 mechanical properties, *Acta Mater.* 186 (2020) 199–214.  
507 <https://doi.org/10.1016/J.ACTAMAT.2019.12.037>.
- 508 [12] Y.R. Han, C.H. Zhang, X. Cui, S. Zhang, J.B. Zhang, Y. Liu, The formability and  
509 microstructure evolution of 24CrNiMo alloy steel fabricated by selective laser  
510 melting, *Vacuum.* 175 (2020) 109297.  
511 <https://doi.org/10.1016/j.vacuum.2020.109297>.
- 512 [13] M. Abdelwahed, S. Bengtsson, R. Casati, A. Larsson, S. Petrella, M. Vedani, Effect

513 of water atomization on properties of type 4130 steel processed by L-PBF, Mater.  
514 Des. 210 (2021) 110085. <https://doi.org/10.1016/j.matdes.2021.110085>.

515 [14] T.H. Becker, P. Kumar, U. Ramamurty, Fracture and fatigue in additively  
516 manufactured metals, Acta Mater. 219 (2021) 117240.  
517 <https://doi.org/10.1016/j.actamat.2021.117240>.

518 [15] J. Suryawanshi, K.G. Prashanth, U. Ramamurty, Mechanical behavior of selective  
519 laser melted 316L stainless steel, Mater. Sci. Eng. A. 696 (2017) 113–121.  
520 <https://doi.org/10.1016/J.MSEA.2017.04.058>.

521 [16] J. Suryawanshi, K.G. Prashanth, U. Ramamurty, Tensile, fracture, and fatigue  
522 crack growth properties of a 3D printed maraging steel through selective laser  
523 melting, J. Alloys Compd. 725 (2017) 355–364.  
524 <https://doi.org/10.1016/j.jallcom.2017.07.177>.

525 [17] J. Maciejewski, The Effects of Sulfide Inclusions on Mechanical Properties and  
526 Failures of Steel Components, J. Fail. Anal. Prev. 15 (2015).  
527 <https://doi.org/10.1007/s11668-015-9940-9>.

528 [18] R. Vaz Penna, L.N. Bartlett, T. Constance, Understanding the Role of Inclusions on  
529 the Dynamic Fracture Toughness of High Strength Lightweight FeMnAl Steels, Int.  
530 J. Met. 13 (2019) 286–299. <https://doi.org/10.1007/s40962-018-0273-9>.

531 [19] X. Lou, P.L. Andresen, R.B. Rebak, Oxide inclusions in laser additive manufactured  
532 stainless steel and their effects on impact toughness and stress corrosion cracking  
533 behavior, J. Nucl. Mater. (2018). <https://doi.org/10.1016/j.jnucmat.2017.11.036>.

534 [20] D.F. Heaney, Powders for metal injection molding (MIM), in: Handb. Met. Inject.  
535 Molding, Elsevier, 2012: pp. 50–63. <https://doi.org/10.1533/9780857096234.1.50>.

536 [21] S. Cacace, Q. Semeraro, Influence of the atomization medium on the properties of  
537 stainless steel SLM parts, Addit. Manuf. 36 (2020) 101509.  
538 <https://doi.org/10.1016/j.addma.2020.101509>.

539 [22] M. Abdelwahed, S. Bengtsson, R. Casati, A. Larsson, M. Vedani, L-PBF Processing  
540 of Steel Powders Produced by Gas and Water Atomization, BHM Berg- Und  
541 Hüttenmännische Monatshefte. 166 (2021) 40–45. <https://doi.org/10.1007/s00501-020-01071-1>.

542 [23] M. Abdelwahed, R. Casati, A. Larsson, S. Petrella, S. Bengtsson, M. Vedani, On  
543 the Recycling of Water Atomized Powder and the Effects on Properties of L-PBF  
544 Processed 4130 Low-Alloy Steel, Materials (Basel). 15 (2022) 336.  
545 <https://doi.org/10.3390/ma15010336>.

546 [24] R. Seede, B. Zhang, A. Whitt, S. Picak, S. Gibbons, P. Flater, A. Elwany, R.  
547 Arroyave, I. Karaman, Effect of heat treatments on the microstructure and  
548 mechanical properties of an ultra-high strength martensitic steel fabricated via laser  
549 powder bed fusion additive manufacturing, Addit. Manuf. 47 (2021) 102255.  
550 <https://doi.org/10.1016/J.ADDMA.2021.102255>.

551 [25] E. Jelis, M. Clemente, S. Kerwien, N.M. Ravindra, M.R. Hespos, Metallurgical and  
552 Mechanical Evaluation of 4340 Steel Produced by Direct Metal Laser Sintering,  
553 JOM. 67 (2015) 582–589. <https://doi.org/10.1007/s11837-014-1273-8>.

554 [26] T. Nakamoto, N. Shirakawa, Y. Miyata, H. Inui, Selective laser sintering of high  
555 carbon steel powders studied as a function of carbon content, J. Mater. Process.  
556 Technol. 209 (2009) 5653–5660.  
557 <https://doi.org/10.1016/J.JMATPROTEC.2009.05.022>.

558

- 559 [27] E.N. DIN, 10083-3 (2007) Steels for quenching and tempering-Part 3: Technical  
560 delivery conditions for alloy steels, English version, of DIN EN 10083-3: 2007-01,  
561 Deutsches Institut für Normung e. V., Berlin. (n.d.).  
562 <http://www.skl.by/files/information/flat/EN10083-3.pdf>
- 563 [28] Y. Wang, J. Hua, M. Kong, Y. Zeng, J. Liu, Z. Liu, Quantitative analysis of  
564 martensite and bainite microstructures using electron backscatter diffraction,  
565 *Microsc. Res. Tech.* 79 (2016) 814–819. <https://doi.org/10.1002/jemt.22703>.
- 566 [29] M.-S. Baek, K.-S. Kim, T.-W. Park, J. Ham, K.-A. Lee, Quantitative phase analysis  
567 of martensite-bainite steel using EBSD and its microstructure, tensile and high-  
568 cycle fatigue behaviors, *Mater. Sci. Eng. A.* 785 (2020) 139375.  
569 <https://doi.org/10.1016/j.msea.2020.139375>.
- 570 [30] H. Chen, D. Gu, D. Dai, C. Ma, M. Xia, Microstructure and composition  
571 homogeneity, tensile property, and underlying thermal physical mechanism of  
572 selective laser melting tool steel parts, *Mater. Sci. Eng. A.* 682 (2017) 279–289.  
573 <https://doi.org/10.1016/j.msea.2016.11.047>.
- 574 [31] P.P. Suikkanen, C. Cayron, A.J. DeArdo, L.P. Karjalainen, Crystallographic  
575 Analysis of Martensite in 0.2C-2.0Mn-1.5Si-0.6Cr Steel using EBSD, *J. Mater. Sci.*  
576 *Technol.* 27 (2011) 920–930. [https://doi.org/10.1016/S1005-0302\(11\)60165-5](https://doi.org/10.1016/S1005-0302(11)60165-5).
- 577 [32] J.C.F. Jorge, L.F.G.D. Souza, M.C. Mendes, I.S. Bott, L.S. Araújo, V.R.D. Santos,  
578 J.M.A. Rebello, G.M. Evans, Microstructure characterization and its relationship  
579 with impact toughness of C–Mn and high strength low alloy steel weld metals – a  
580 review, *J. Mater. Res. Technol.* 10 (2021) 471–501.  
581 <https://doi.org/10.1016/J.JMRT.2020.12.006>.
- 582 [33] T. Tsuchiyama, M. Natori, N. Nakada, S. Takaki, Conditions for grain boundary  
583 bulging during tempering of lath martensite in ultra-low carbon steel, *ISIJ Int.* 50  
584 (2010). <https://doi.org/10.2355/isijinternational.50.771>.
- 585 [34] M. Salandre, S. Garabedian, G. Gaillard, T. Williamson, T. Joffre, Development of  
586 a New Tooling Steel (L40) for Laser Powder Bed Fusion: Influence of Particle Size  
587 Distribution and Powder Atomization on Mechanical Performance, *Adv. Eng. Mater.*  
588 23 (2021). <https://doi.org/10.1002/adem.202100350>.
- 589