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A mechanism leading to γ' precipitates with $\{111\}$ facets and unusual orientation relationships to the matrix in γ/γ' nickel-based superalloys

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ABSTRACT

Cast-and-wrought heavily-alloyed γ - γ' nickel-based superalloys may exhibit large recovered grains inherited from the ingot conversion and characterized by a high density of close-to-coherent micrometric γ' precipitates. In the AD730TM nickel-based superalloy, a previous work ^[18] highlighted a new interaction between such precipitates and a recrystallization front passing through. This interaction resulted in γ' precipitates with a close-to-twin orientation relationship to their recrystallized host grain. Called T-type precipitates, they were revealed to be $\{111\}$ bounded plate-like particles. The present paper aims to clarify the mechanism whereby such precipitates form. The formation of T-type precipitates actually is part of a more global mechanism which also produces γ' precipitates slightly misoriented from their surrounding matrix (C-type precipitates) and of same size and morphology as T-type precipitates. Both T and C type precipitates display $\{111\}$ facets and are evidenced in the AD730TM, René65 and PER72 alloys, supporting the idea that the mechanism can more generally occur in all low-lattice-mismatch γ - γ' nickel-based superalloys. Finally, a scenario is proposed: T/C type precipitates form at the recrystallization front of grains sharing a $\langle 111 \rangle$ axis with the recovered grain they consume, and develop $\{111\}$ facets and specific orientations which minimize the interfacial energy on both recrystallized and recovered sides.

I. INTRODUCTION

Wrought polycrystalline γ - γ' nickel-based superalloys are commonly used to manufacture aero-engine turbine disks, thanks to their outstanding mechanical properties at high temperature^[1,2]. Those alloys display γ grains and γ' precipitates of various sizes. The γ phase is a Face Centered Cubic (FCC) non-ordered solid solution, while the γ' -Ni₃(Al,Ti) phase has a L1₂ ordered cubic structure. At a few micrometers in size, primary precipitates are the largest precipitates of the microstructure^[3]. Derived from the as-cast microstructure and high temperature billet forging sequences, they are useful for the grain size control since they pin grain boundaries during subsolvus forging operations. Most of the time, primary precipitates lie on grain boundaries and are incoherent with the surrounding γ matrix, which means that there is no matching at all between the γ' and the γ crystal lattices. Yet, the interaction between a recrystallization front and an incoherent precipitate is quite easy to predict. When a moving grain boundary encounters a precipitate, its curvature is locally modified due to capillarity effects, which is interpreted as a pinning force applied on the boundary and referred to as the Smith-Zener pinning force^[4]. Then, if the grain boundary is subject to sufficient driving force to counteract the pinning force, it will bypass the precipitate; otherwise it will halt at the precipitate. However, when a moving boundary meets a coherent precipitate - which means that the γ and γ' crystal lattices perfectly coincide at the γ/γ' interface^[5] - the interaction can be much more complex. Indeed, because coherent interfaces have low energy values^[6,7], the maximum pinning force a coherent precipitate can enforce is twice that of an incoherent precipitate of the same size^[8,9]. Then, other phenomena which also allow the grain boundary to carry on moving and which are more thermodynamically and/or kinetically interesting than the coherent precipitate bypass can occur. It was reported in the literature that a coherent precipitate may be cut-through - so that the orientation of the precipitate is changed to that of the growing grain^[7,8] - or dissolved^[10,11]. However, if the grain boundary driving force is not sufficient to bypass, dissolve or cut-through the coherent precipitate, the grain boundary will halt at the coherent precipitate which will then coarsen as a result of the interfacial energy equilibrium^[8]. At a few hundred nanometers to few micrometers in size, secondary and tertiary precipitates are always coherent with their surrounding matrix. But much larger precipitates are also found coherent or close-to-coherent. First, Charpagne et al. evidenced large primary precipitates with the same orientation as their surrounding matrix in the case of heteroepitaxial recrystallization^[12-14]. Second, cast-and-wrought heavily-alloyed γ - γ' nickel-based superalloys may exhibit unrecrystallized grains inherited from the ingot conversion process^[15-17] where a high density of micrometric close-to-coherent precipitates is found. These unrecrystallized grains are characterized by intragranular orientation gradients with more or less developed recovery cell structures; thus they will be called recovered grains in the following text. Yet, in the AD730TM γ - γ' nickel-based superalloy a previous work^[18] pointed out a new interaction between the close-to-coherent precipitates of the recovered grain and the recrystallization front passing through. This interaction sometimes led to γ' precipitates with a close-to-twin orientation relationship to their recrystallized host grain. "Close to twin orientation relationship" means that the γ' and γ cubic lattice cells conform to a $60^\circ \langle 111 \rangle$ rotation within the 8.66° tolerance given by Brandon's criterion: both the misorientation angle and misorientation axis do not deviate more than 8.66° to the 60° angle and $\langle 111 \rangle$ axis of the perfect twin boundary^[18,19]. Such precipitates close to the twin orientation relationship have been called T-type precipitates and revealed to be $\{111\}$ bounded plate-like particles. Their size, morphology and narrow orientation spread compared to the precipitates of the recovered grain have proved that they do not result from a simple bypass by the recrystallization front but from a specific mechanism. The aim of

41 the present paper is to clarify this mechanism using EBSD data analyses. It is shown that the formation of T-type
42 precipitates is part of a more global mechanism which also produces precipitates with an orientation very close
43 to that of their surrounding matrix and of the same size and morphology as the T-type precipitates. These
44 precipitates are called C-type precipitates and rotated by few degrees around a $\langle 111 \rangle$ axis they have in common
45 with their surrounding matrix. T and C type precipitates are evidenced in the AD730TM, René65 and PER72
46 alloys, supporting the idea that the studied mechanism can more generally occur in all γ - γ' nickel-based
47 superalloys with a low-lattice mismatch. Finally, a scenario is proposed for this specific mechanism which
48 allows a recrystallization front to get through a high density of close-to-coherent micrometric precipitates and
49 leads to γ' precipitates with unusual morphologies and orientations.

50 II. EXPERIMENTAL

51 In the present paper, five samples are analyzed. They are derived from different thermo-mechanical conditions
52 and from three different alloys: AD730TM, René65 and PER72. The PER72 alloy is an equivalent of the
53 Udimet720 alloy manufactured by the Aubert&Duval Company. All these polycrystalline γ - γ' nickel-based
54 alloys are all heavily-alloyed, derived from the traditional cast-and-wrought route and characterized by a low
55 lattice mismatch^[12,20]. Their chemical compositions are given in Table 1.

56 Three samples were cut out from AD730TM billets. Two of them were observed as-received. The third one,
57 referred to as the “AD730TM Lab-Forged sample”, was deformed at high temperature (compression test) then
58 solution-treated for 4 hours at 1080°C in order to complete recrystallization. Hot-deformation and solution-
59 treatment temperatures were both below the γ' solvus temperature which is about 1110°C. The fourth and fifth
60 samples were cut out from René65 and PER72 billets and heat-treated for two hours at about 40° below their
61 respective γ' solvus temperature. All the samples were ground with Si papers and polished with diamond
62 suspensions down to 1 μ m. To obtain a suitable surface quality for EBSD (Electron BackScatter Diffraction),
63 they were finally polished either by electropolishing (electrolyte: CH₃OH-10% HClO₄) or with a 0.02 μ m
64 colloidal silica solution on a vibratory machine. Observations were carried out with a Zeiss Supra40 Field
65 Emission Gun Scanning Electron Microscope operated at 15kV. The microscope is equipped with the
66 QUANTAX EDS/EBSD system from the Bruker Company which is composed of an XFlash 5030 Energy
67 Dispersive X-ray Spectrometer (EDS) and an e-FlashHR EBSD detector, both controlled by the ESPRIT®
68 software package. Some of the EBSD data post-treatments were performed using MTEX, a freely available
69 MATLAB toolbox^[21].

70

71 III. RESULTS AND DISCUSSION

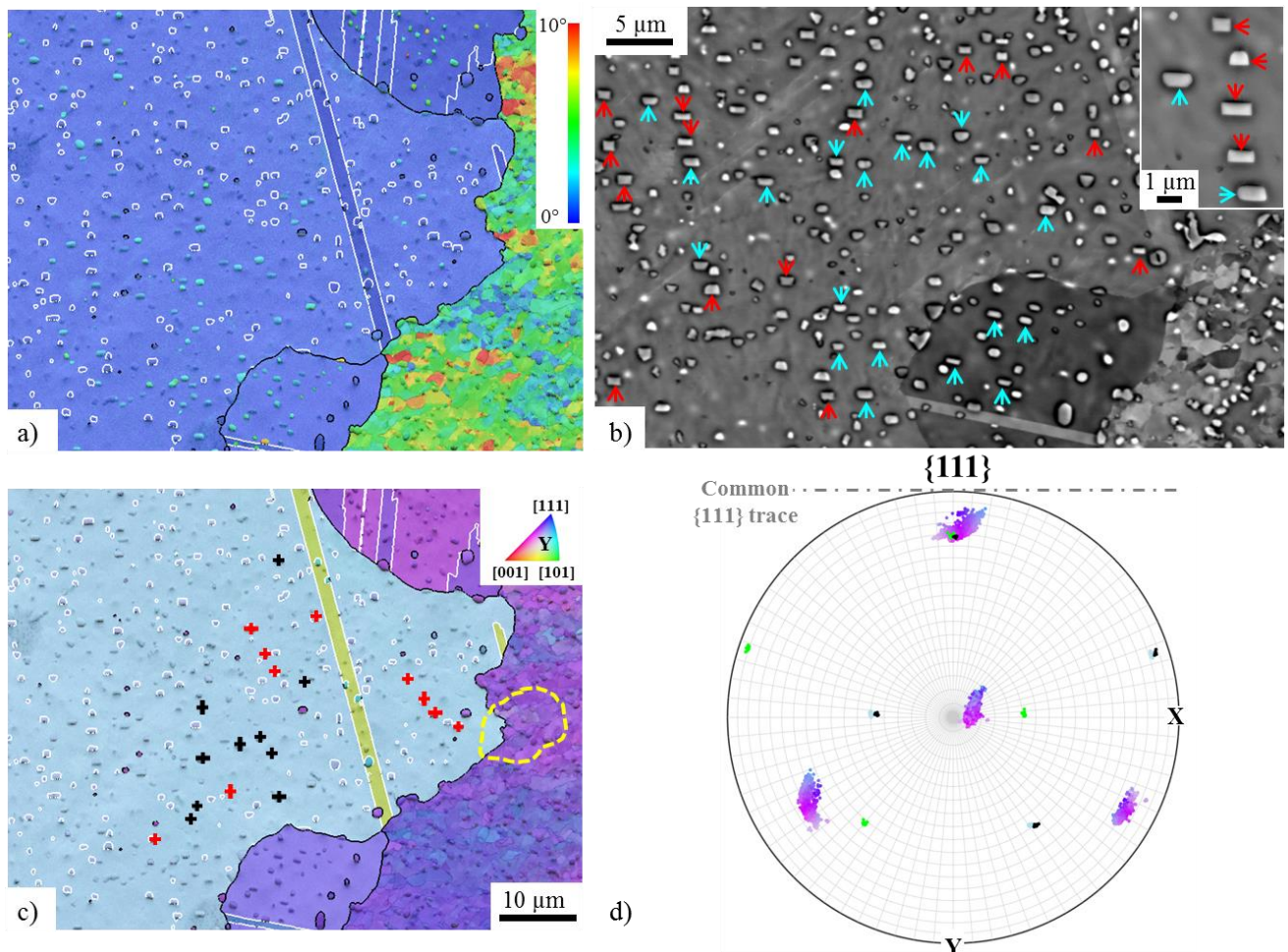
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73 A. T and C type precipitates in the AD730TM alloy : the results of a single mechanism

74

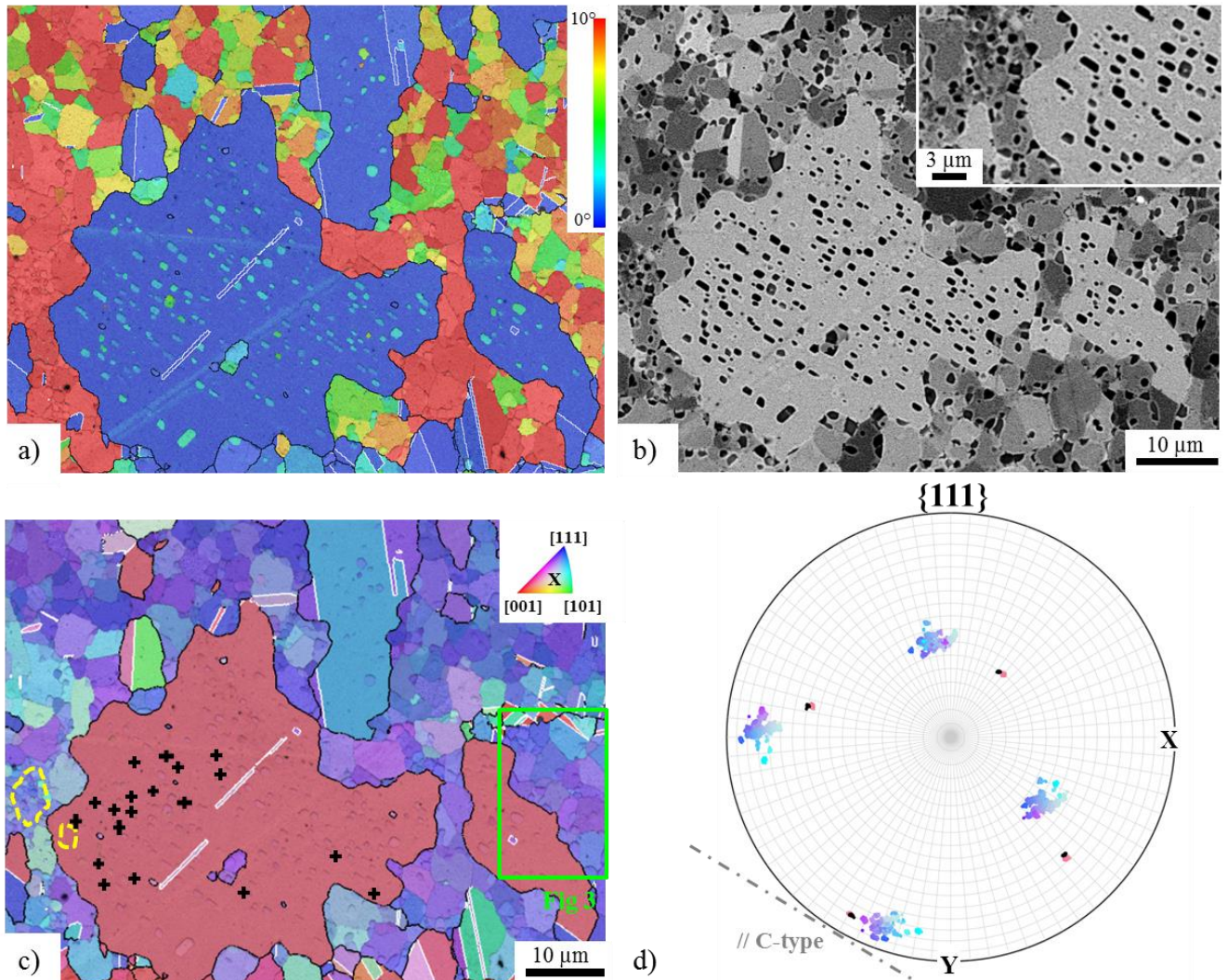
75 T-type precipitates have been reported in the alloy AD730TM, in some recrystallized grains which had
76 extensively grown into a recovered grain and which shared a $\langle 111 \rangle$ axis with it^[18]. Nevertheless, Fig.1 shows
77 that T-type precipitates are not the only type of precipitate which can be found in such recrystallized grains.
78 Indeed, some precipitates which are 2-3° misoriented from their host grain appear among the T-type precipitates
79 (Fig.1.a). These precipitates have a nearly rectangular shape in the sample section like the T-type precipitates
80 (Fig.1.b). Close to the orientation of their surrounding matrix, they will be called C-type precipitates in the

81 following text. The rectangular shapes of the C-type precipitates are parallel and also parallel to those of the T-
 82 type precipitates (Fig.1.b). On the $\{111\}$ pole figure (Fig.1.d), the orientations of T and C type precipitates
 83 (randomly selected) are compared to those of the host recrystallized grain and the recovered grain. First, the
 84 orientations of the two precipitate types appear very homogeneous compared to those of the recovered grain
 85 selected over a much smaller area. Second, the $\langle 111 \rangle$ axis shared by the recrystallized and recovered grains
 86 perfectly matches with a $\langle 111 \rangle$ axis of the C and T type precipitates. Indeed, the three other $\langle 111 \rangle$ axes of the C
 87 type precipitates differ slightly from those of their recrystallized host grain, showing that the misorientation of
 88 the C-type precipitates regarding their host grain originates from a rotation of few degrees around the $\langle 111 \rangle$ axis
 89 they exactly have in common. Moreover, the longest sides of the rectangular shapes of the T and C type
 90 precipitates are parallel to the $\{111\}$ plane trace common to the recrystallized and recovered grains. Yet, it has
 91 been evidenced in ^[18] that T-type precipitates are plate-like particles bounded by the $\{111\}$ plane common to the
 92 T-type precipitates, the recrystallized grain and the recovered grain. Here, the similar morphologies and
 93 alignments of the C and T type precipitates strongly suggests that both types of precipitates are faceted by the
 94 same $\{111\}$ plane, common to the recrystallized and recovered grains.
 95



96
 97 *Fig.1 – C and T type precipitates coexisting in a recrystallized grain (AD730TM Lab-Forged sample). a) EBSD*
 98 *map: misorientation angle to the mean orientation of the grain overlaid on the band contrast map, grain*
 99 *boundaries (misorientation angle threshold: 10°) plotted black and twin boundaries (60° $\langle 111 \rangle$ with 8.66°*
 100 *tolerance, following Brandon's criterion ^[19]) plotted white. b) Backscattered electron image, where the γ'*
 101 *precipitates appear bright despite their lower atomic number because of topographic effects induced by*
 102 *electropolishing. Some C and T type precipitates are highlighted by blue and red arrows respectively. c) EBSD*

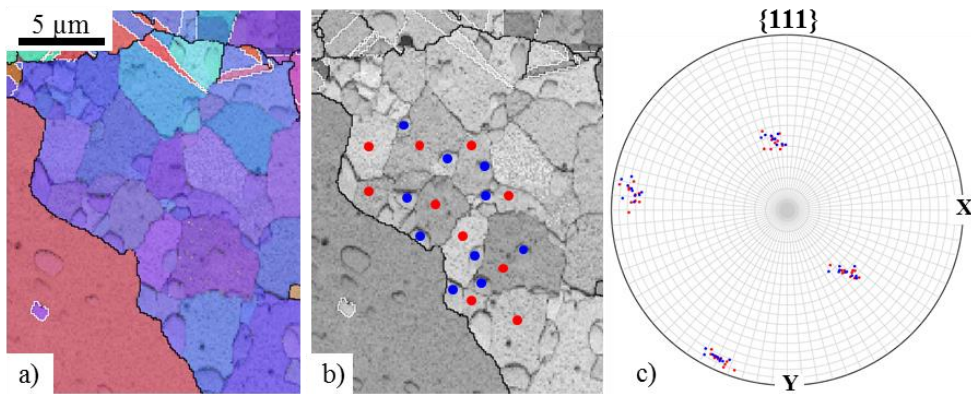
103 map: orientation (IPF color code) overlaid on the band contrast map, grain boundaries plotted black and twin
 104 boundaries plotted white. d) $\{111\}$ pole figure on a spherical grid (spacing: 5°), orientations of ten C-type
 105 precipitates (black crosses in c) plotted black, orientations of ten T-type precipitates (red crosses in c) plotted
 106 green and orientations of the yellow selection in c) plotted with the same color as displayed in c). The $\{111\}$
 107 plane trace common to the C/T type precipitates and the recrystallized matrix is displayed by a dashed line.
 108



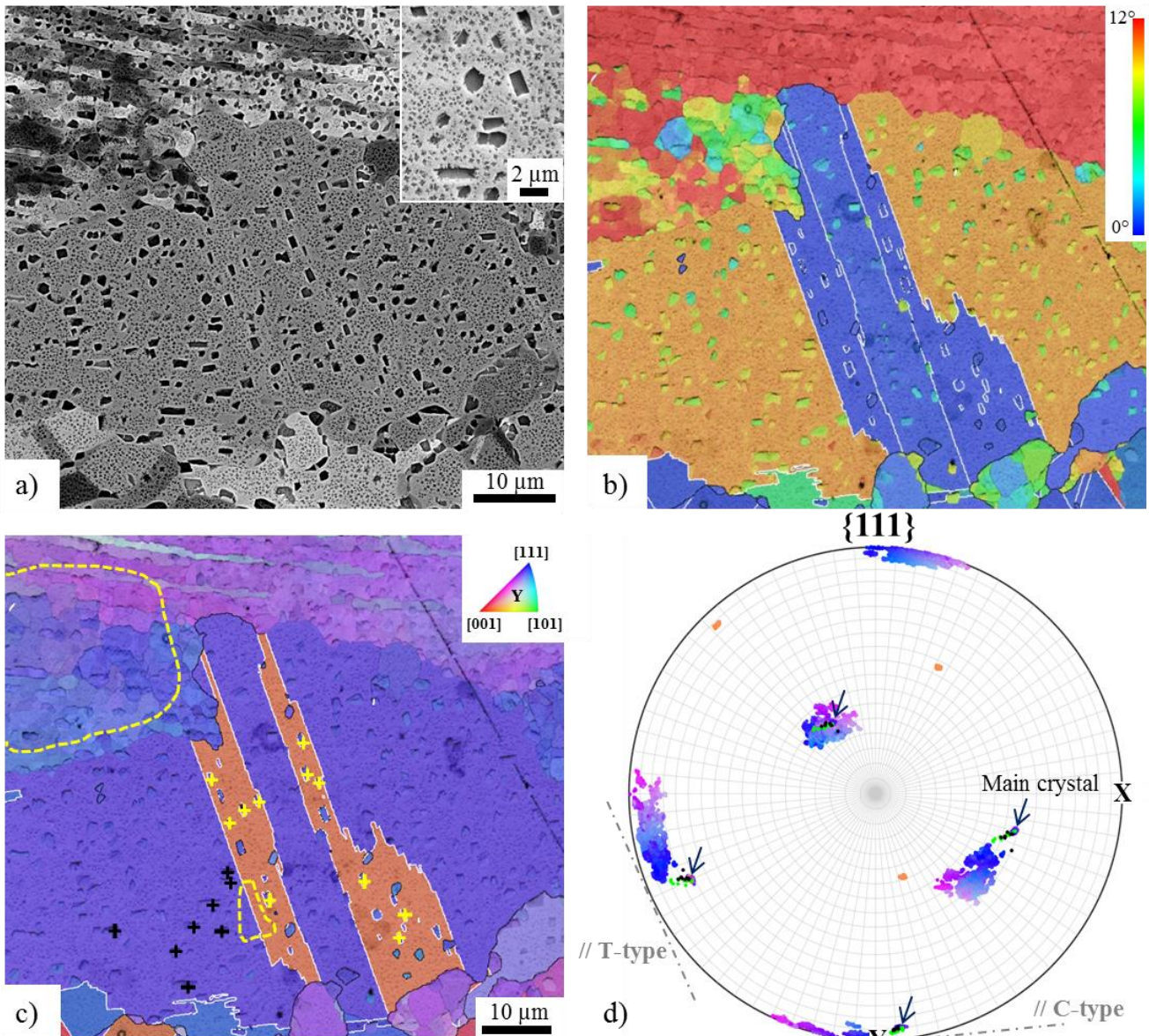
109
 110 Fig.2 – Grain full of C-type precipitates (AD730™ billet sample). a) EBSD map: misorientation angle to the
 111 mean orientation of the grain overlaid on the band contrast map, grain boundaries (misorientation angle
 112 threshold: 10°) plotted black and twin boundaries ($60^\circ < 111 >$ with 8.66° tolerance) plotted white. b)
 113 Backscattered electron image. c) EBSD map: orientation (IPF color code) overlaid on the band contrast map,
 114 grain boundaries plotted black and twin boundaries plotted white. d) $\{111\}$ pole figure on a spherical grid
 115 (spacing: 5°), orientation of the selected C type precipitates (black crosses in c) plotted black, orientations of
 116 the yellow selections in c) plotted with the same color as displayed in c). The $\{111\}$ plane trace common to the C
 117 type precipitates and the recrystallized matrix is displayed by a dashed line.
 118
 119

120 Fig. 2 presents an example of a large recrystallized grain with a single T-type precipitate amongst many C-type
 121 precipitates in an AD730™ billet. The recrystallized grain has grown by consuming a so-called substructured
 122 grain, which is a former grain mainly composed of well-defined low-misoriented crystallites with micrometric
 123 close-to-coherent precipitates located at their boundaries. It is worth mentioning here that the close-to-coherent
 124 precipitates of the substructured grain are not C-type precipitates since they do not have the aligned rectangular
 125 shapes and narrow orientation spread of the neighboring C-type precipitates (Fig.2.b and Fig.3). The

126 substructured and recrystallized grains have a $\langle 111 \rangle$ axis close to each other (Fig.2.d), so that the recrystallized
 127 grain is rotated around this $\langle 111 \rangle$ axis with respect to the substructured grain, but less rotated than what has
 128 been observed in Fig.1. This $\langle 111 \rangle$ axis also coincides with the $\langle 111 \rangle$ axis around which the orientations of the
 129 C-type precipitates are rotated relatively to that of their host grain. Moreover, the corresponding $\{111\}$ plane
 130 trace is parallel to the longest sides of the C-type precipitates, which is again consistent with C-type precipitates
 131 bounded by $\{111\}$ planes.
 132



133
 134 *Fig.3 – Orientations of the precipitates in the substructured grain with respect to their surrounding matrix*
 135 *(AD730™ billet sample – Area highlighted in Fig.2.c). a) EBSD map: orientation (IPF color code) overlaid on*
 136 *the band contrast map, grain boundaries (misorientation angle threshold: 10°) plotted black and twin*
 137 *boundaries ($60^\circ \langle 111 \rangle$ with 8.66° tolerance) plotted white. b) EBSD map: band contrast map, grain boundaries*
 138 *plotted black and twin boundaries plotted white. c) $\{111\}$ pole figure on a spherical grid (spacing: 5°).*
 139 *Orientation of the precipitates (selected by blue points in b)) plotted blue, orientations of the matrix (selected by*
 140 *red points in b)) plotted red.*
 141



142
 143 *Fig.4 – Change from C type to T type precipitates by twinning (AD730™ billet sample). a) Backscattered*
 144 *electron image (overall view) and secondary electron image (insert at high magnification). b) EBSD map:*
 145 *misorientation angle to the mean orientation of the grain overlaid on the band contrast map, grain boundaries*
 146 *(misorientation angle threshold: 10°) plotted black and twin boundaries (60° <111> with 8.66° tolerance)*
 147 *plotted white. c) EBSD map: orientation (IPF color code) overlaid on the band contrast map, grain boundaries*
 148 *plotted black and twin boundaries plotted white. d) {111} pole figure on a spherical grid (spacing: 5°),*
 149 *orientations of the C-type precipitates (black crosses in c) plotted black, orientations of the T-type precipitates*
 150 *(yellow crosses in c) plotted green and orientations of the yellow selections in c) plotted with the same color as*
 151 *displayed in c). The {111} plane traces common to the C/T type precipitates and the recrystallized matrix are*
 152 *displayed by dashed lines.*

153
 154
 155 A last example of C and T type precipitate coexistence in the AD730™ alloy is presented in Fig.4. This time, a
 156 large recrystallized grain consuming a recovered grain displays C-type precipitates in its main crystal and T-type
 157 precipitates in its twin crystals (Fig.4.b). The two precipitate types are again rectangular in the sample section
 158 and can reach few micrometers in length. However, the rectangular shapes of the T and C type precipitates
 159 follow two different directions in space. The {111} pole figure (Fig.4.d) reveals that the main crystal has an
 160 orientation very close to that of the recovered grain and that the longest sides of the C-type precipitates are not so

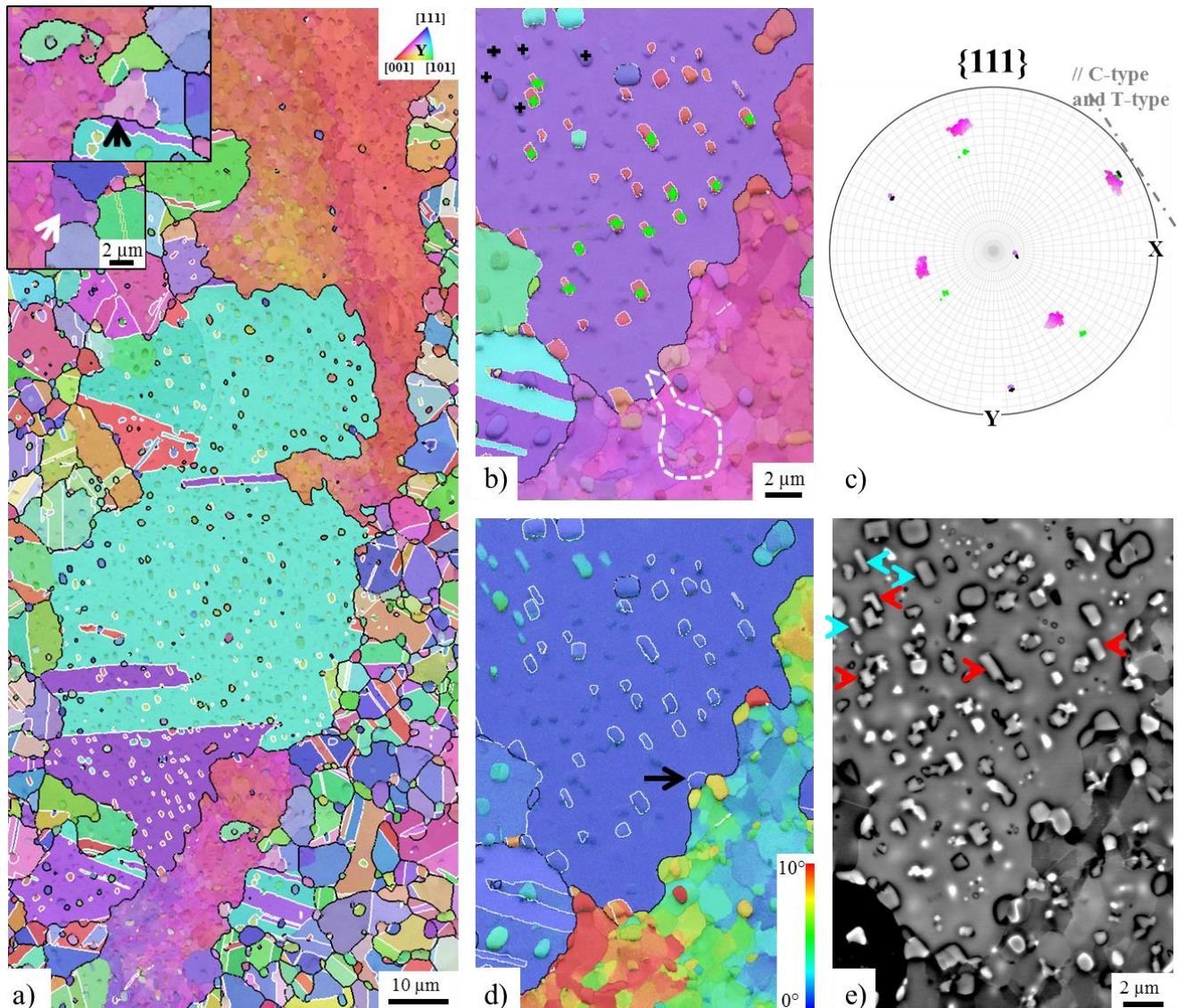
161 far from a $\{111\}$ plane trace. The twin crystals have a single $\langle 111 \rangle$ axis in common with the recovered grain
162 and the longest sides of the T-type precipitates coincide with the corresponding $\{111\}$ plane trace (which differs
163 from that of the C-type precipitate). In this third example, the orientations of the C-type precipitates are very
164 homogeneous but those of the selected T-type precipitates are somewhat more spread than observed in Fig.1.
165 This underlines that the mechanism leading to T-type precipitates does not produce perfect twin boundaries.
166 For the three cases presented above, as well as for the cases which will be presented in the following sections,
167 the misorientation between the recrystallized grain and the recovered (or substructured) grain has been quantified
168 by calculating the average misorientation angle ($\bar{\theta}$) at the recrystallized/recovered boundary (average on
169 boundary segments of at least few tens of micrometers long). The values are summarized in Table 2. From Table
170 2, it can be noticed that recrystallized grains whose misorientation to the recovered grain is lower than $\sim 37^\circ$
171 show C-type precipitates only. On the other hand, recrystallized grains which are misoriented by more than $\sim 50^\circ$
172 show T-type precipitates only. Between these two misorientation angles, a transition zone is observed where
173 recrystallized grains exhibit both C and T type precipitates. So, although a more statistical analysis would be
174 required to obtain more precise transition values, there does exist a link between the misorientation angle of the
175 recrystallized grain relatively to the recovered grain and the precipitate type(s) which form(s).

176
177 To summarize, the C and T type precipitates are located in recrystallized grains which have grown into
178 recovered/substructured grains. Both can display rectangular shapes in sample sections. If so, the longest sides of
179 their rectangular shapes are up to a few micrometers in length and parallel to the trace of the $\{111\}$ plane they
180 share with their host grain. The corresponding shared $\langle 111 \rangle$ axis is always very close to a $\langle 111 \rangle$ axis of the
181 consumed recovered/substructured grain. Thus, C and T type precipitates are both $\{111\}$ bounded plate-like
182 particles which derive from the same mechanism. The misorientation angle between the recrystallized and the
183 recovered grains determines what precipitate type forms: while the less misoriented recrystallized grains only
184 display C-type precipitates, the highest misoriented recrystallized grains display only T-type precipitates, and
185 intermediate misorientations both types. Recrystallized grains with a $\langle 111 \rangle$ axis very close to one of the $\langle 111 \rangle$
186 axes of the recovered grain they consume originate from the Continuous Recrystallization (CRX) and the
187 Heteroepitaxial Recrystallization (HERX) which are the two recrystallization mechanisms identified for the
188 recovered grains of the AD730TM alloy [18]. Both mechanisms produce recrystallized grains whose initial
189 orientations are close to that of the recovered grain. As the recrystallized grain grows into the recovered grain, its
190 misorientation with respect to the recovered grain increases because of the orientation gradients present inside
191 the recovered grain. By growing, the recrystallized grain may also undergo annealing twinning, so that the
192 misorientation of the recrystallization front changes from a rather low misorientation to a high one (or
193 conversely) due to the additional 60° $\langle 111 \rangle$ rotation. This explains why a wide range of misorientation angles is
194 observed at the recrystallized/recovered boundaries (Table 2).

195 196 **B. Observations in other γ/γ' superalloys: René65 and PER72 alloys**

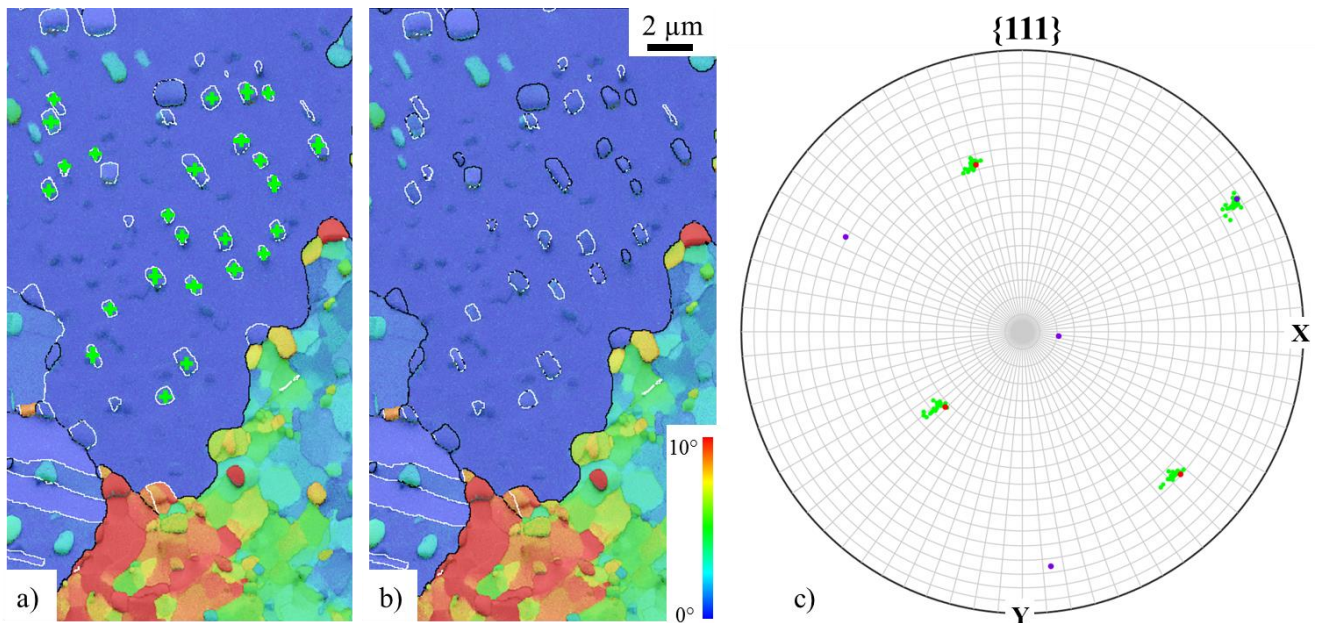
197 In René65 and PER72 billets, recovered grains with their characteristic high density of close-to-coherent
198 precipitates can also be observed (Fig.5.a and Fig.7.a). Besides, the configuration presented in Fig.1 for the
199 AD730TM alloy is found in the René65 billet sample too (Fig.5): a few large recrystallized grains have grown to a
200 large extent inside a recovered grain. These large recrystallized grains are full of micrometric precipitates with

201 some of them well rectangular in the sample section (Fig.5.d-e). Some of the rectangular precipitates are in twin
 202 orientation relationship to their host grain but the twin orientation relationship is imperfect because only realized
 203 within the 8.66° tolerance given by Brandon's criterion (Fig.6). Thus, such precipitates do correspond to the T-
 204 type precipitates as defined in the alloy AD730TM. In the same crystal, rectangular precipitates slightly-
 205 misoriented to their surrounding matrix and parallel to the T-type precipitates are also found (Fig.5.d-e): these
 206 precipitates are C-type precipitates. On the $\{111\}$ pole figure (Fig.5.c), one can check that the crystal hosting the
 207 T and C type precipitates has a $\langle 111 \rangle$ axis very close to a $\langle 111 \rangle$ axis of the recovered grain. Then, T and C type
 208 precipitates are rotated around this common $\langle 111 \rangle$ axis with respect to their host grain and the corresponding
 209 $\{111\}$ plane trace coincides with the longest sides of their rectangular shapes. As observed in the AD730TM
 210 alloy, the orientations of the two precipitate types are more homogeneous than that of the recovered grain.
 211 Finally, CRX cells and HERX grains can be noticed at the borders of the recovered grain (black and white
 212 arrows in Fig.5.a).
 213



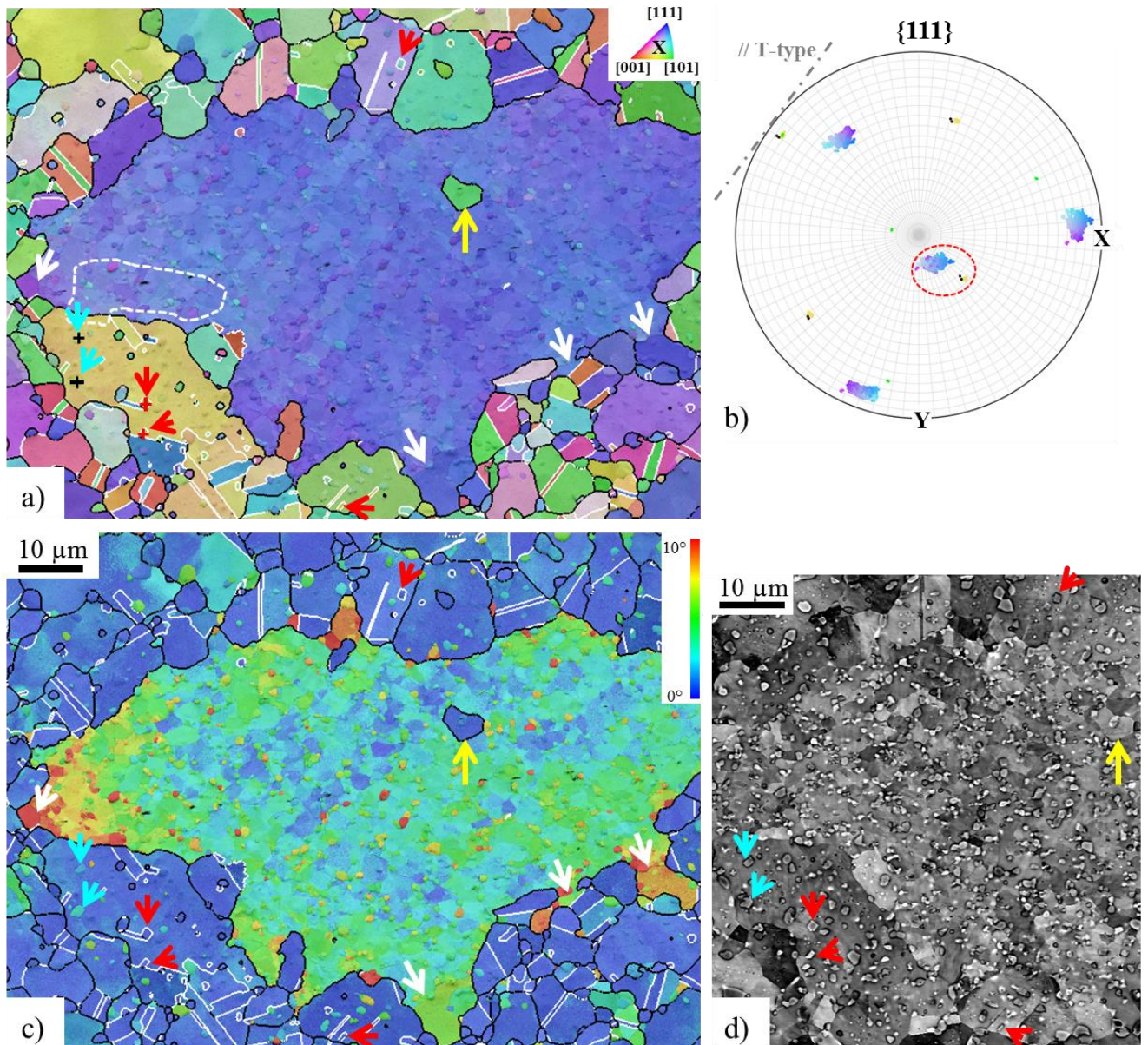
214
 215 *Fig.5 – C and T type precipitates resulting from the recrystallization of a recovered grain in the René65 alloy*
 216 *(René65 billet sample). a) and b) EBSD map: orientation (IPF color code) overlaid on the band contrast map,*
 217 *grain boundaries (misorientation angle threshold: 10°) plotted black and twin boundaries ($60^\circ \langle 111 \rangle$ with 8.66°*
 218 *tolerance) plotted white. In the inserts, the black and white arrows underline a CRX cell and a HERX grain*

219 respectively. c) $\{111\}$ pole figure on a spherical grid (spacing: 5°), orientations of the C-type precipitates (black
 220 crosses in b)) plotted black, orientations of the T-type precipitates (green crosses in b)) plotted green and
 221 orientations of the white selection in b) plotted with the same color as displayed in b). The $\{111\}$ plane trace
 222 common to the C/T type precipitates and the recrystallized matrix is displayed by a dashed line. d) EBSD map:
 223 misorientation angle to the mean orientation of the grain overlaid on the band contrast map, grain boundaries
 224 plotted black and twin boundaries plotted white. The black arrow underlines a T-type precipitate at the
 225 recrystallization front. e) Backscattered electron image, where the γ' precipitates appear bright despite their
 226 lower atomic number because of topographic effects induced by electropolishing. C and T type precipitates are
 227 highlighted by blue and red arrows respectively.
 228
 229



230 Fig.6 – The imperfect twin orientation relationship of the T-type precipitates (René65 billet sample). a) and b)
 231 EBSD maps - misorientation angle to the mean orientation of the grain overlaid on the band contrast map, grain
 232 boundaries (misorientation angle threshold: 10°) plotted black and twin boundaries ($60^\circ \langle 111 \rangle$ with $\Delta\theta$
 233 tolerance) plotted white. In a) twin boundaries are plotted with 8.66° tolerance following Brandon's criterion
 234 ^[19]. In b) twin boundaries are plotted with 2° tolerance. c) $\{111\}$ pole figure on a spherical grid (spacing: 5°).
 235 The orientations of the T-type precipitates selected in a) are plotted green, the orientations of the surrounding
 236 matrix and the corresponding perfect twin crystal ($60^\circ \langle 111 \rangle$ 0° tolerance) are plotted purple and red
 237 respectively.
 238
 239

240 No case of striking heterogeneous recrystallization with large recrystallized grains full of T/C type precipitates
 241 has been observed in the studied PER72 billet sample. It is also worth mentioning here that, in the AD730TM and
 242 René65 alloys, all the recovered grains do not always lead to several huge statically recrystallized grains as
 243 shown throughout the paper. The reason why this phenomenon is not systematic is not understood yet but would
 244 be interesting to investigate in future research. Nevertheless, in the PER72 alloy, isolated T and C type
 245 precipitates are seen in recrystallized grains of “normal” size next to recovered grains (red and blue arrows in
 246 Fig.7). For example, the yellow grain on Fig.7.a contains several C and T type precipitates. This grain has a
 247 $\langle 111 \rangle$ axis not so far from a $\langle 111 \rangle$ axis of the nearby recovered grain (dashed circle in Fig.7.b), although it is
 248 not the twin axis of the T-type precipitates. Some CRX cells and HERX grains are also seen in the recovered
 249 grains of the PER72 alloy (white and yellow arrows in Fig.7). This means that the two recrystallization
 250 mechanisms suspected in the AD730TM alloy as taking part to the formation of the T/C type precipitates also
 251 occur in the PER72 alloy.



252
 253 Fig.7 – A recovered grain in the PER72 alloy (PER72 billet sample). a) EBSD map: orientation (IPF color
 254 code) overlaid on the band contrast map, grain boundaries (misorientation angle threshold: 10°) plotted black
 255 and twin boundaries (60° <111> with 8.66° tolerance) plotted white. b) {111} pole figure on a spherical grid
 256 (spacing: 5°), orientations of the C-type precipitates (black crosses in a) plotted black, orientations of the T-
 257 type precipitates (red crosses in a) plotted green and orientations of the white selection in a) plotted with the
 258 same color as displayed in a). A {111} plane trace is displayed by a dashed line. c) EBSD map: misorientation
 259 angle to the mean orientation of the grain overlaid on the band contrast map, grain boundaries plotted black
 260 and twin boundaries plotted white. d) Backscattered electron image, where the γ' precipitates appear bright
 261 despite their lower atomic number because of topographic effects induced by electropolishing. Red, blue, yellow
 262 and white arrows highlight T-type precipitates, C-type precipitates, HERX grain and CRX cells respectively.
 263

264 Thus, T and C type precipitates, with their characteristic {111} facets and orientation relationships, can be
 265 identified in recrystallized grains near the recovered grains of the René65 and PER72 alloys. In the same way as
 266 in the AD730™ alloy, the recrystallized host grain has a <111> axis very close to a <111> axis of the nearby
 267 recovered grain. Such recrystallized grains are very likely to derive from the CRX and HERX which occur in the
 268 recovered grains of the three alloys studied.

269
 270

271 C. Clarification of the mechanism leading to T and C type precipitates

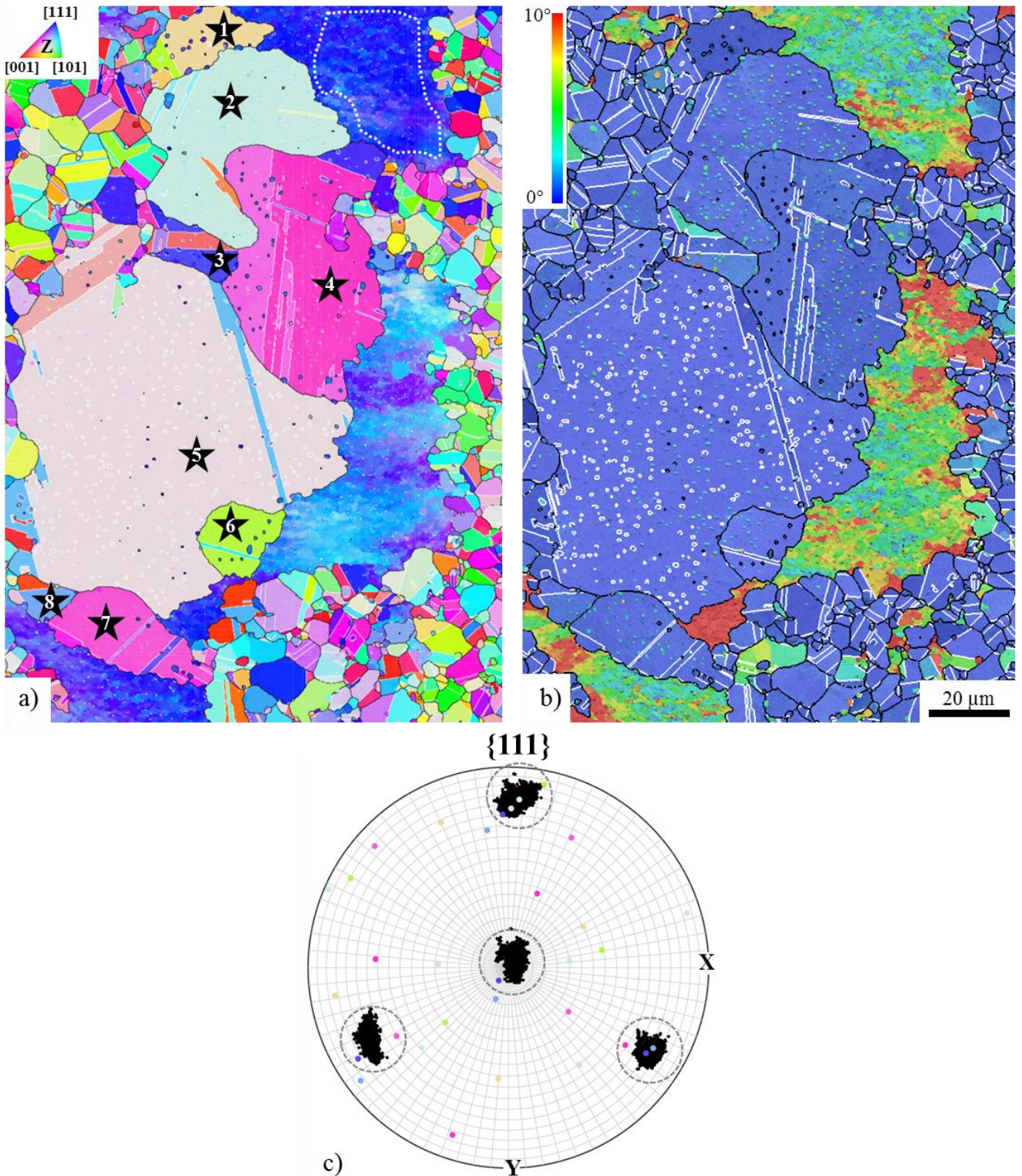
272 1. An advantageous mechanism to overcome a high density of close-to-coherent precipitates

273 Fig.8 displays several large recrystallized grains which have extensively grown by consuming the nearby
274 recovered grain. If the crystallographic orientations of these recrystallized grains (labelled by black stars in
275 Fig.8.a) are compared to those of the recovered grain (Fig.8.c), it appears that all the recrystallized grains –
276 except the grain N°1 in Fig.8.a – have a $\langle 111 \rangle$ axis close to a $\langle 111 \rangle$ axis of the recovered grain they consume
277 (less than 10° deviation). Moreover, all these large recrystallized grains show a high density of intragranular
278 precipitates which are C and/or T type precipitates (Fig.8.b).

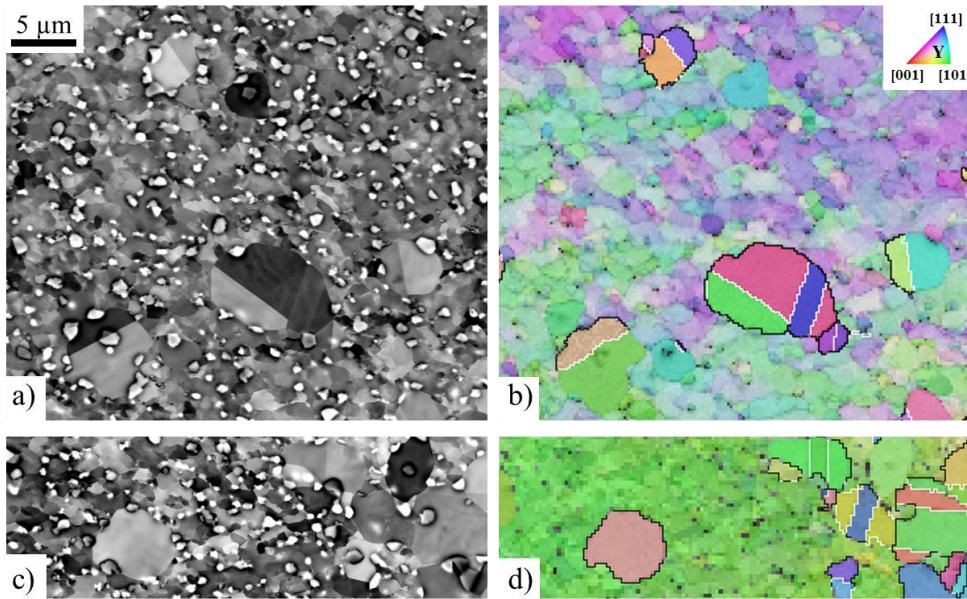
279 On the other hand, many recrystallized grains attempting to consume a recovered grain remain very small (Fig.
280 9). If some of the small recrystallized grains can result from the CRX and HERX occurring in recovered grains,
281 many of them derive from Discontinuous Recrystallization (DRX) [22] and so have random orientations,
282 especially those at the periphery of the recovered grain. Nevertheless, whatever the origin of the small
283 recrystallized grains, each time the same pattern is noticed: i) recrystallized grains are small but larger than the
284 recovery cells of the recovered grain (which are delimited by close-to-coherent precipitates); ii) small
285 recrystallized grains are free from precipitates (except the precipitate which originated HERX); iii) some coarse
286 close-to-coherent precipitates can be pointed out on the boundaries of the small recrystallized grains. From this
287 pattern, a scenario explaining why these recrystallized grains remain so small may be developed. Given that
288 small recrystallized grains are always larger than recovery cells, they must start growing by dissolving the
289 nearby close-to-coherent precipitates. But, since no re-precipitation is observed behind the recrystallization front,
290 at first time all the γ' -forming elements remain stored in the moving boundary. Then, the high solute
291 concentration in the moving grain boundary decreases its mobility [22,23] and the boundary ends up stopping at
292 close-to-coherent precipitates. Finally, these close-to-coherent precipitates are fed by the γ' -forming elements
293 stored in the boundary and their coarsening leads to a high pinning force, which definitely prevents the boundary
294 from moving anymore.

295 The above observations suggest that the recrystallized grains having a $\langle 111 \rangle$ axis close to a $\langle 111 \rangle$ axis of a
296 recovered grain have an advantage over a randomly-oriented recrystallized grain in consuming it. Yet,
297 consuming a recovered grain amounts to overcoming the high Smith-Zener drag pressure exerted by its close-to-
298 coherent precipitates. For spherical particles, the Smith-Zener drag pressure is usually described by the formula
299 $P_{SZ} = \Gamma \frac{3f}{2r}$, where Γ , f and r are respectively the grain boundary energy, the precipitate volume fraction and the
300 precipitate mean radius [4]. In the present case, the drag pressure has not been precisely evaluated but, judging
301 from the precipitate density and size, it is very likely to be quite high. In addition, the intragranular precipitates
302 of the recovered grains are close-to-coherent and the drag pressure enforced by coherent precipitates is twice that
303 enforced by the equivalent incoherent population [8,9]. In literature several possible mechanisms to overcome
304 coherent precipitates have been pointed out: precipitates can be bypassed, dissolved (with a coherent re-
305 precipitation behind the recrystallization front) [10,11] or crossed by the recrystallization front so that their
306 orientation is changed to that of the growing grain [7,8]. However here, none of these mechanisms is observed:
307 while many recrystallized grains stop growing very early inside the recovered grain, some recrystallized grains
308 sharing a $\langle 111 \rangle$ axis with the recovered grain can grow to a large extent producing T/C type precipitates. Thus,
309 the mechanism leading to T/C type precipitates appears as the more thermodynamically/kinetically interesting
310 mechanism to overcome the precipitates of the recovered grain. Yet, considering that T/C type precipitates are
311

312 bounded by $\{111\}$ planes, this is consistent with a mechanism which minimize the energy of the system since
 313 $\{111\}$ planes are known to be low energy interfaces [24].



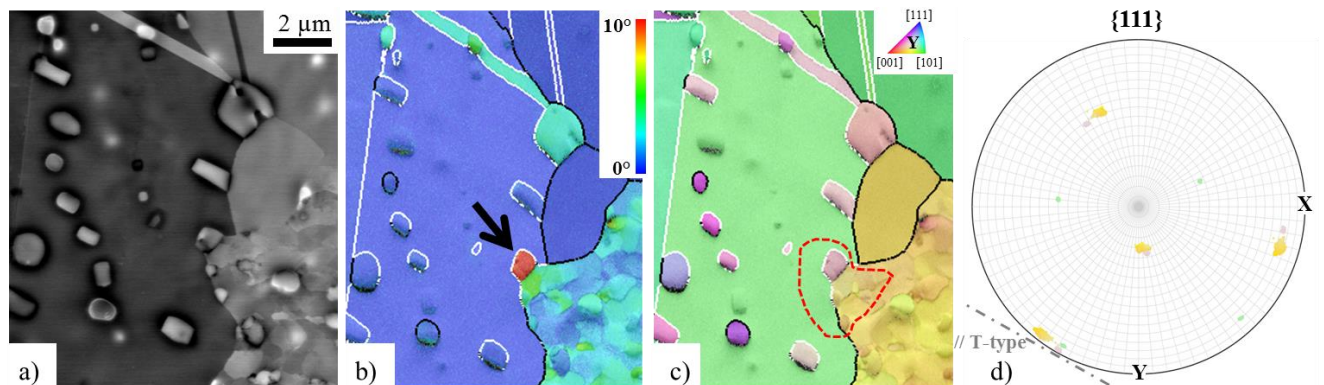
314
 315 *Fig.8 – Analysis of the heterogeneous recrystallization nearby a recovered grain (AD730™ Lab-Forged*
 316 *sample). a) EBSD map: orientation map (IPF color code), grain boundaries (misorientation angle threshold:*
 317 *10°) plotted black and twin boundaries (60° $\langle 111 \rangle$ with 8.66° tolerance) plotted white. b) EBSD map:*
 318 *misorientation angle to the mean grain orientation overlaid on the band contrast map, grain boundaries plotted*
 319 *black and twin boundaries plotted white. c) $\{111\}$ pole figure on a spherical grid (spacing: 5°), orientations of*
 320 *the recovered grain (white selection in a) plotted black, orientations of the grains selected by black stars in a)*
 321 *plotted with the same color they appear in a).*
 322



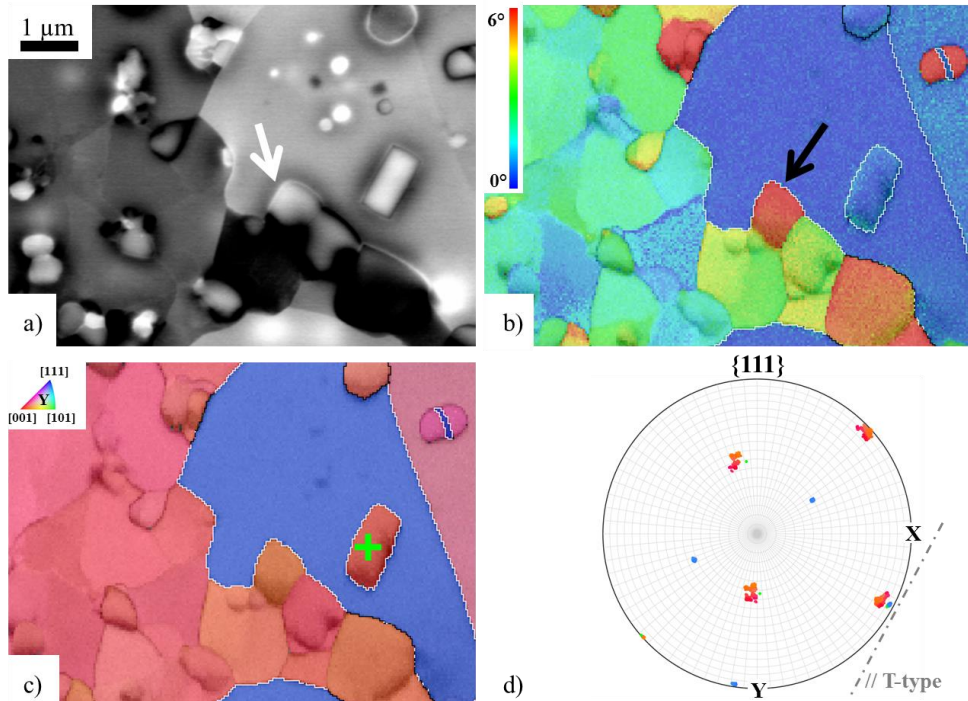
323 Fig.9 – Recrystallized grains unsuccessful in consuming the surrounding recovered grain (AD730™ Lab-Forged
 324 sample). a) and c) Backscattered electron images, where the γ' precipitates appear bright despite their lower
 325 atomic number because of topographic effects induced by electropolishing. b) and d) EBSD map: orientation
 326 (IPF color code) overlaid on the band contrast maps, grain boundaries (misorientation angle threshold: 10°)
 327 plotted black and twin boundaries ($60^\circ \langle 111 \rangle$ with 8.66° tolerance) plotted white.
 328
 329
 330

331 2. Involving grain boundary faceting

332 As highlighted in [18], the few T-type precipitates evidenced at the recrystallization front (black arrows in Fig.5,
 333 Fig.10 and Fig.11) prove that T-type precipitates form at the recrystallization front with their characteristic
 334 orientation and faceted shape. Similarly, a C-type precipitate is seen at the recrystallization front in Fig.12.k
 335 (black cross).
 336
 337

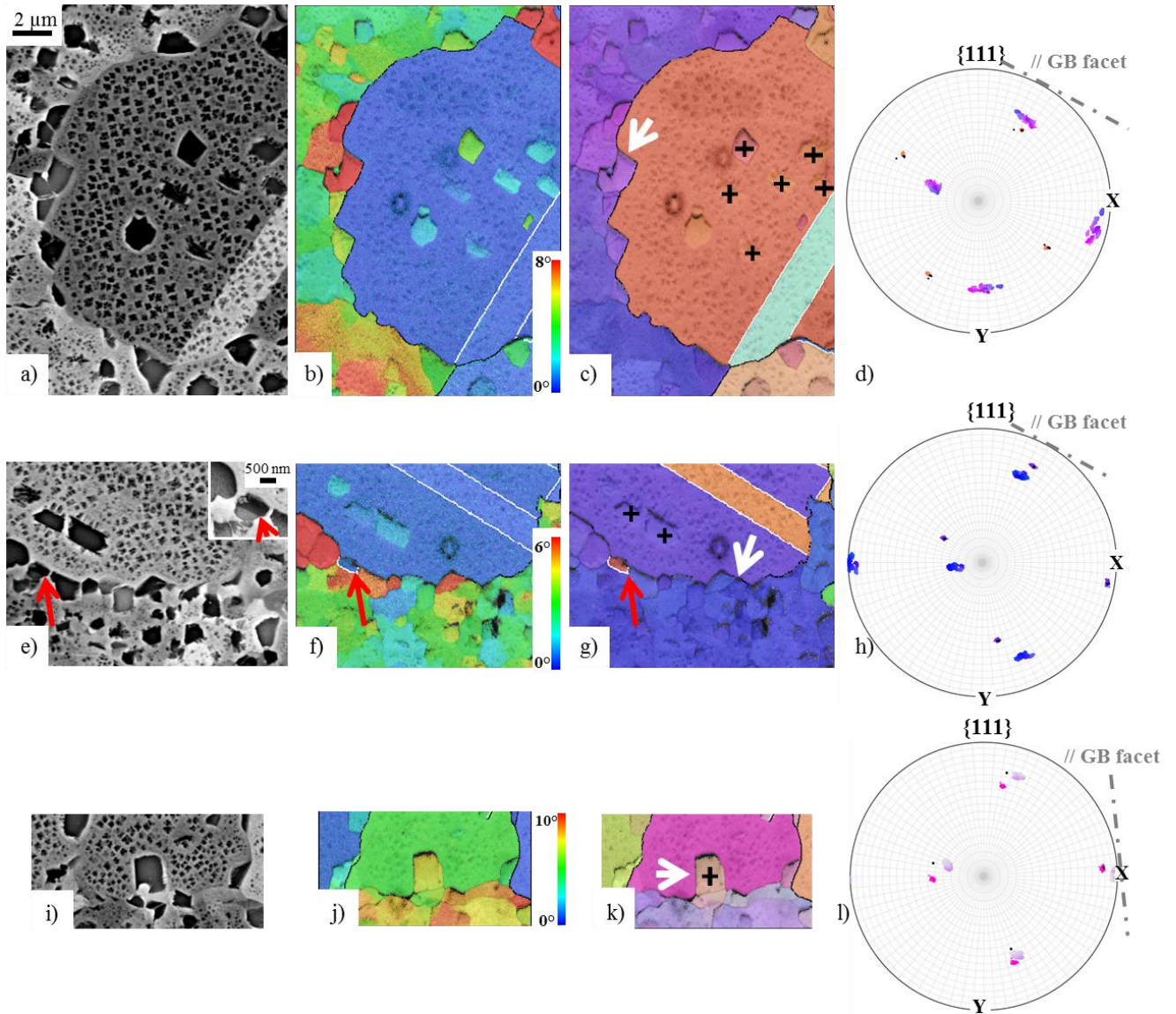


338 Fig.10 – A T-type precipitate at the recrystallization front in the AD730™ alloy (AD730™ Lab-Forged sample).
 339 a) Backscattered electron image, where the γ' precipitates appear bright despite their lower atomic number
 340 because of topographic effects induced by electropolishing. b) EBSD map: misorientation angle to the mean
 341 orientation of the grain overlaid on the band contrast map, grain boundaries (misorientation angle threshold:
 342 10°) plotted black and twin boundaries ($60^\circ \langle 111 \rangle$ with 8.66° tolerance) plotted white. The black arrow
 343 underlines a T-type precipitate at the recrystallization front. c) EBSD map: orientation (IPF color code)
 344 overlaid on the band contrast map, grain boundaries plotted black and twin boundaries plotted white. d) $\{111\}$
 345 pole figure on a spherical grid (spacing: 5°), orientations of the red selection in c) plotted with the same color as
 346 displayed in c). The $\{111\}$ plane trace common to the T-type precipitate and the selected matrix is displayed by a
 347 dashed line.
 348
 349



351
 352 *Fig.11 – A T-type precipitate at the recrystallization front in the René65 alloy (René65 billet sample). a)*
 353 *Backscattered electron image, where the γ' precipitates appear bright despite their lower atomic number*
 354 *because of topographic effects induced by electropolishing. The white arrow highlights a grain boundary facet.*
 355 *b) EBSD map: misorientation angle to the mean orientation of the grain overlaid on the band contrast map,*
 356 *grain boundaries (misorientation angle threshold: 10°) plotted black and twin boundaries ($60^\circ \langle 111 \rangle$ with 8.66°*
 357 *tolerance) plotted white. The black arrow underlines a T-type precipitate at the recrystallization front. c) EBSD*
 358 *map: orientation overlaid on the band contrast map, grain boundaries plotted black and twin boundaries plotted*
 359 *white. d) $\{111\}$ pole figure on a spherical grid (spacing: 5°), orientations of the red selection in c) plotted with*
 360 *the same color as displayed in c), orientation of the selected T-type precipitate (green cross in c)) plotted green.*
 361 *The $\{111\}$ plane trace common to the recrystallized and recovered grains is displayed by a dashed line.*

362
 363



364
 365 *Fig.12 – {111} grain boundary facets at the recrystallization front (AD730™ billet sample). 1st column (a, e and*
 366 *i): Backscattered electron images. Secondary electron image inserted in e), the red arrows point out a twin*
 367 *boundary inside a precipitate. 2nd column (b, f and j): EBSD maps - misorientation angle to the mean*
 368 *orientation of the grain overlaid on the band contrast maps, grain boundaries (misorientation angle threshold:*
 369 *10°) plotted black and twin boundaries (60° <111> with 8.66° tolerance) plotted white. 3rd column (c, g and k):*
 370 *EBSD maps - orientation (IPF color code) overlaid on the band contrast maps, grain boundaries plotted black*
 371 *and twin boundaries plotted white. The white arrows highlight grain boundary facets. 4th column (d, h and l):*
 372 *{111} pole figures on a spherical grid (spacing: 5°), orientations of the recrystallized/recovered interface plotted*
 373 *with the same color as displayed in the orientation maps, orientations of the selected C-type precipitates (black*
 374 *crosses) plotted black. Some {111} plane traces, parallel to grain boundary facets, are displayed by dashed*
 375 *lines.*
 376

377 In the AD730™ billet sample, small facets in the range of 0.5 to 2 μm wide can be observed on the
 378 recrystallized/recovered front (Fig.12). Most of the facets coincide with a {111} plane trace the recrystallized
 379 and recovered grains have in common (white arrows on the orientation maps Fig.12). On these facets,
 380 precipitates large, faceted and slightly misoriented compared to the neighboring recovered matrix are implanted
 381 (misorientation angle to the mean grain orientation maps Fig.12). The insert in Fig.12.e even shows, inside a
 382 precipitate implanted on a recrystallization front facet, a twin boundary being itself parallel to the
 383 recrystallization front facet. Similarly, in the René65 alloy, the T-type precipitate visible at the recrystallization

384 front is implanted on a facet which lies on a {111} plane the recrystallized and recovered grains have in common
385 (white arrow in Fig.11.a).

386

387 The {111} facets of the T/C type precipitates are very unusual for γ' precipitates since coherent {100} cuboidal
388 γ' precipitates are classically observed in nickel-based superalloys [20,25,26]. Yet, γ' precipitates always evolve so
389 as to minimize the system free energy. For instance, the classical {100} cuboidal shape allows the minimization
390 of the elastic strain energy occurring at the coherent interfaces due to the γ - γ' lattice mismatch. For C/T
391 precipitates, the elastic strain energy is small since their imperfect cube-cube/twin orientation relationships
392 require interfacial dislocations to accommodate the elastic strain. Thus, the morphology and orientations of the
393 C/T type precipitates are mainly controlled by their interfacial energy which has to be minimized considering the
394 recrystallized and recovered sides since C/T type precipitates form at the recrystallization front.

395 The recrystallized and recovered grains share a $\langle 111 \rangle$ axis. Yet, $\langle 111 \rangle$ twist boundaries have a much lower
396 boundary energies than any other grain boundary type, with minima at $0^\circ \langle 111 \rangle$ and $60^\circ \langle 111 \rangle$ corresponding to
397 the cube-cube and twin orientation relationships respectively (from [24] the energy of a $\langle 111 \rangle$ twist boundary does
398 not exceed 0.5 J/m^2 while a random boundary can reach 1.5 J/m^2). Thus, by developing $\langle 111 \rangle$ twist boundaries
399 (approximately) lying on the {111} plane common to the recovered and recrystallized grains, the precipitates
400 decrease their interfacial energy on both recovered and recrystallized sides. Finally, the γ/γ' $\langle 111 \rangle$ twist rotation
401 angle which is adopted is the best compromise to minimize the total interfacial energy of the precipitate. This
402 rotation angle is logically close to 0° or 60° , depending on the mutual misorientation of the recrystallized and
403 recovered grains.

404 Regarding the formation of the {111} facets which have been noticed at the recrystallization front, two
405 hypotheses can be considered; they are schematically represented in Fig.13. The first one (H1) is that the
406 recrystallization front naturally tends to develop {111} facets, and thus $\langle 111 \rangle$ twist portions, because of the
407 $\langle 111 \rangle$ axis its shares with the recovered grain. Then, due to the supersaturation of the recrystallization front, γ' -
408 forming elements re-precipitate on these {111} facets in order to minimize the total interfacial energy of the
409 obtained precipitates as just explained above. Coincidence Site Lattice (CSL) boundaries naturally tend to facet
410 and the obtained facets are preferentially the crystallographic planes densely packed with coincidence sites [27,28].
411 Many CSL boundaries in the FCC structure are equivalent to a $\langle 111 \rangle$ rotation and so present the {111} planes as
412 densely packed CSL planes. The names of these CSL boundaries as well as their corresponding rotation angle
413 are given in Table 3. One can notice that the $\langle 111 \rangle$ rotation angles of these CSL boundaries cover the whole
414 recrystallized/recovered misorientation angle range presented in the present paper (Table 2).

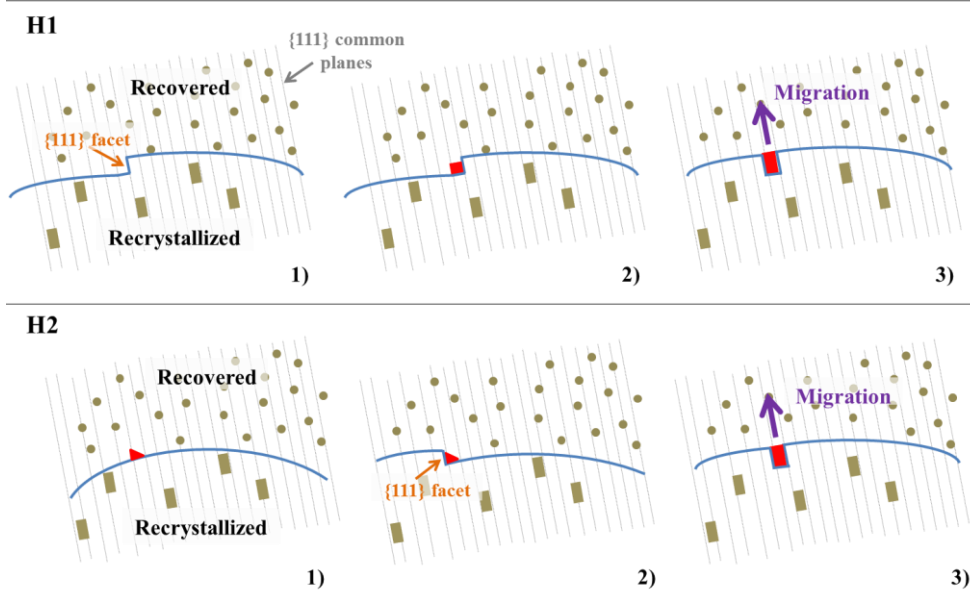
415 In the second hypothesis (H2), the T/C type precipitates first nucleate at an initially rough recrystallization front
416 due to solute supersaturation. This nucleus is $\langle 111 \rangle$ rotated with respect to the recrystallized and recovered
417 grains so as to minimize its interfacial energy. Then, while growing, the precipitate develops its characteristic
418 {111} facets to again minimize its interfacial energy. The γ/γ' {111} facets of the precipitate oblige the
419 recrystallization front in turn to facet so as to bypass it.

420 At the moment, given the available information, deciding which hypothesis between H1 and H2 is the most
421 likely is not possible. But, whatever the hypothesis considered, as the T/C type precipitates form at the
422 recrystallization front but inside the recovered grain, the recrystallization front still has to bypass them to carry
423 on growing. Because T and C type precipitates are {111} bounded plate-like particles, the classical Smith-Zener

424 model which is used for spherical particles and, above all, which ignores the interfacial energies between the
 425 grains and the particle is not the most appropriate here. However, the work of Ringer et al. [29] on grain boundary
 426 pinning by stable cubic-shaped particles is interesting to consider. Indeed, they highlighted that the pinning
 427 enforced by a cubic or spherical particle strongly depends the “degree of particle coherency” defined by the
 428 ratio:

$$D_{PC} = \frac{\Gamma_{1p} - \Gamma_{2p}}{\Gamma_{12}}$$

430 Where the grain 1 is non-recrystallized, the grain 2 is recrystallized, Γ_{ip} is the energy of the grain i/particle
 431 interface and Γ_{12} is the grain boundary energy. Ringer et al. evidenced that for a cubic (or spherical) particle the
 432 pinning force is minimal for low D_{PC} values. On the contrary, in case of a coherent particle, the D_{PC} is close to 1
 433 and the pinning force is much higher. As said above, $\langle 111 \rangle$ twist interfaces have low energy values. Thus,
 434 considering the T/C type precipitates delimited by $\langle 111 \rangle$ twist boundaries whose corresponding $\{111\}$ plane is
 435 common to the recrystallized and recovered grains, Γ_{1p} and Γ_{2p} are very low compared to Γ_{12} , so the D_{PC} is small
 436 and the pinning enforced is minimized.



437
 438 *Fig.13 – Schematic diagrams describing the two hypotheses (H1 and H2) for the formation of {111} facets at the*
 439 *recrystallized/recovered front.*

440 3. Scenario proposed for the formation of C and T type precipitates

441
 442
 443 At this point, a scenario for the formation of T and C type precipitates can be proposed. Under hot-deformation
 444 and subsequent heat-treatments, recovered grains partially recrystallize by forming CRX and HERX grains
 445 which are low-misoriented to the surrounding recovered matrix. Recrystallized grains are also produced by DRX
 446 occurring at the periphery of the recovered grain as everywhere in the equiaxed grain areas. During an annealing
 447 stage, all these recrystallized grains start growing inside the recovered grain due to its relatively high stored
 448 energy. This first growth step is supported by the dissolution of the precipitates encountered in the recovered
 449 grain, so that the recrystallization front gets more and more saturated with γ' -forming elements. Then, there are
 450 two cases. In the first case, the recrystallization front gets so saturated that the high solute concentration
 451 decreases significantly its mobility. Thus, the recrystallization front stops at some precipitates of the recovered

452 grain and the γ' -forming elements stored in the front feed those precipitates. Those precipitates coarsen, pin and
453 prevent the boundary from further motion. The second case concerns the recrystallized grains sharing a $\langle 111 \rangle$
454 axis with the nearby recovered grain. These recrystallized grains mainly derive from the CRX and HERX
455 occurring in the recovered grain. The common $\langle 111 \rangle$ axis can allow: H1) the formation on the recrystallization
456 front of facets lying on the $\{111\}$ common planes, then the implantation of γ' -forming elements on the $\{111\}$
457 facets as a consequence of supersaturation; H2) the formation of precipitates faceted by the $\{111\}$ common
458 planes on a rough recrystallization front due to supersaturation, this then obliges the recrystallization front to
459 form $\{111\}$ facet in order to bypass them. Whatever the hypothesis (H1 or H2), what is sure is that the T/C type
460 precipitates form inside the recovered grain at the recrystallization front, and develop these $\{111\}$ facets
461 combined with specific orientations to minimize their interfacial energy including both the recrystallized and
462 recovered sides. Indeed, $\langle 111 \rangle$ twist boundaries have low energies with minima at $60^\circ \langle 111 \rangle$ and $0^\circ \langle 111 \rangle$. This
463 implies that the lowest misoriented recrystallized grains ($< \sim 37^\circ$) lead to precipitates implanted approximately at
464 $0^\circ \langle 111 \rangle$ on the front, the highest misoriented ones ($> \sim 50^\circ$) lead to precipitates implanted approximately at
465 $60^\circ \langle 111 \rangle$ on the front, and that for the intermediate misorientations both implantations are possible. The low
466 interfacial energies on both recrystallized and recovered sides make the faceted precipitates little costly to
467 bypass. The obtained precipitates are few degrees rotated to their host recrystallized grain ($\sim 0^\circ \langle 111 \rangle$ - C-type)
468 or in close-to-twin orientation relationship ($\sim 60^\circ \langle 111 \rangle$ - T-type). Released from the γ' elements it contained, the
469 recrystallization front can carry on moving inside the recovered grain and repeat the same mechanism.
470 Nevertheless, the scenario which is proposed here has some weaknesses. First, the reason why all the grains
471 derived from the CRX and HERX in the recovered grain do not succeed in growing to a large extent inside the
472 recovered grain is not understood yet. Thus, the mechanism leading to T/C type precipitates must require
473 additional conditions to a common $\langle 111 \rangle$ axis to happen. Second, the reason why the γ' precipitates re-arrange
474 inside the recovered grain and not coherently behind the recrystallization front as it is expected from the
475 literature is unclear too. One can imagine that the γ' -forming elements precipitate around a precipitate pre-
476 existing in the recovered grain with a slight change in orientation to minimize the interfacial energy. However,
477 no such sub-boundary has been observed into the T/C type precipitates yet. Another reason which could account
478 for this is the enhanced γ' -forming element diffusion which occurs in the recovered grain due its highly
479 developed network of dislocations [30]. All these points need to be clarified in future research.

480

481 IV. CONCLUSION

482 A previous work [18] has pointed out a close-to-twin orientation relationship between micrometric γ' precipitates
483 and their host recrystallized grain in the AD730TM γ - γ' nickel-based superalloy. The aim of the present paper is
484 to clarify the mechanism leading to such precipitates called T-type precipitates.

485 This paper shows that:

- 486 1. The mechanism leading to T-type precipitates is also able to form C-type precipitates, which are
487 micrometric γ' precipitates slightly misoriented to their surrounding matrix by a rotation of few degrees
488 around a $\langle 111 \rangle$ axis. Both C and T type precipitates are found in recrystallized grains which have
489 grown into a recovered grain and which (approximately) share a $\langle 111 \rangle$ axis with this recovered grain.

- 490 The recrystallized grains sharing a $\langle 111 \rangle$ axis with a recovered grain derive from the CRX and HERX
491 occurring in the recovered grain.
- 492 2. The misorientation angle between the recrystallized and recovered grains determines if the precipitates
493 are mainly C-type ($< \sim 37^\circ$), mainly T-type ($> \sim 50^\circ$) or both (between ~ 37 and $\sim 50^\circ$).
 - 494 3. Both C and T type precipitates are faceted by $\{111\}$ planes whose corresponding $\langle 111 \rangle$ axes are
495 common to the T/C type precipitates, the surrounding recrystallized matrix and the consumed recovered
496 grain.
 - 497 4. Like the AD730TM alloy, the René65 and PER72 alloys can exhibit large recovered grains. In the
498 René65 and PER72, T and C type precipitates with their characteristic shape and orientation
499 relationships are found in recrystallized grains sharing a $\langle 111 \rangle$ axis with the nearby recovered grain.
500 Those recrystallized grains derive from the CRX and HERX mechanisms which have been also pointed
501 out in the recovered grains of the René65 and PER72 alloys. So, the mechanism leading to T/C type
502 precipitates may possibly occur in all the low-lattice-mismatch γ - γ' nickel-based superalloys.
 - 503 5. The mechanism leading to T/C type precipitates is an advantageous mechanism to overcome the high
504 density of close-to-coherent precipitates in a recovered grain. Thus, regarding a random recrystallized
505 grain, recrystallized grains derived from the CRX and HERX occurring in the recovered grain are
506 privileged for consuming it.
 - 507 6. T/C type precipitates appear in the recovered grain at the recrystallization front. They grow onto $\{111\}$
508 facets that the recrystallization front developed due to its crystallographic structure (H1), or grow
509 directly with $\{111\}$ facets on a rough recrystallized front which is then obliged to form $\{111\}$ facets in
510 order to bypass them (H2). Whatever the hypothesis, T/C type precipitates develop $\langle 111 \rangle$ twist
511 boundaries with specific rotation angles ($\sim 60^\circ \langle 111 \rangle$ and $\sim 0^\circ \langle 111 \rangle$) in order to minimize their
512 interfacial energy including both the recovered and recrystallized sides. Then, the low interfacial energy
513 on both the recrystallized and recovered sides of the T/C precipitates makes them little costly to bypass.
 - 514 7. However, some points of the suggested scenario remain unclear. First, as all the CRX and HERX grains
515 formed from the recovered grain do not display T/C type precipitates, additional conditions must
516 certainly be required for the mechanism to happen. Second, the re-precipitation in the recovered grain
517 and not in the recrystallized grain as it is expected from the literature may be perhaps due to the
518 enhanced diffusion occurring in the recovered grain because of its network of dislocations.

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520

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522

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598

599 TABLES

600

601 *Table 1 – Chemical compositions of the alloys used in the present work (wt. %)*

Element	Ni	Fe	Co	Cr	Mo	W	Al	Ti	Nb	B	C	Zr
AD730 TM [31]	Bal.	4.00	8.50	15.70	3.10	2.70	2.25	3.40	1.10	0.01	0.015	0.03
René65 [15]	Bal.	1	13	16	4	4	2.1	3.7	0.7	0.016	-	0.05
PER72	Bal.	0.138	14.5	16.06	2.88	1.21	2.57	5.07	-	0.016	0.017	-

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623 *Table 2 – Average misorientation angle $\bar{\theta}$ calculated at recrystallized/recovered grain boundaries*

$\bar{\theta}$ at grain boundary	Precipitate Type	REX Grain reference
23.2°	C	Purple crystal in Fig.4.c
24.2°	C	Grain N°2 in Fig.8.a
25.2°	C + 1 single T	Red grain in Fig.2.c
29.7°	C	Grain N°4 in Fig.8.a
31.8°	C	Grain N°6 in Fig.8.a
32.4°	C	Grain N°7 in Fig.8.a
36.3°	C	Orange grain in Fig.12.c
37.7°	C + T	Grain N°5 in Fig.8.a
47.9°	C + T	Purple grain in Fig.5.b
53.2°	T	Orange twin crystals in Fig.4.c
53.8°	T	Green grain in Fig.10.c

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626 *Table 3 – CSL boundaries equivalent to a $\langle 111 \rangle$ rotation in a FCC structure ^[32]*

CSL boundary	Rotation axis	Rotation angle
$\Sigma 3$	$\langle 111 \rangle$	60°
$\Sigma 7$	$\langle 111 \rangle$	38.21°
$\Sigma 13b$	$\langle 111 \rangle$	27.79°
$\Sigma 19b$	$\langle 111 \rangle$	46.82°
$\Sigma 21a$	$\langle 111 \rangle$	21.78°
$\Sigma 31a$	$\langle 111 \rangle$	17.9°
$\Sigma 37c$	$\langle 111 \rangle$	50.57°
$\Sigma 39a$	$\langle 111 \rangle$	32.2°
$\Sigma 43a$	$\langle 111 \rangle$	15.18°
$\Sigma 49a$	$\langle 111 \rangle$	43.57°

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