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# 1                    **Plasticity and brittleness of the ordered $\beta_0$ phase in a TNM-TiAl alloy**

2

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12

## 13    **Abstract**

14    In order to identify the reasons for the brittleness of a TNM-TiAl alloy  
15    ( $\text{Ti}_{51.05}\text{Al}_{43.9}\text{Nb}_4\text{Mo}_{0.95}\text{B}_{0.1}$  in at.%), the plastic deformation behaviour of the ordered  $\beta_0$  phase  
16    is investigated. The corresponding powder was densified by Spark Plasma Sintering to produce  
17    a near lamellar microstructure made of  $\gamma/\alpha_2$  lamellar colonies surrounded by  $\gamma$  and  $\beta_0$  grains.  
18    Then, the room temperature tensile behaviour is studied by carrying out tensile tests and  
19    additional in-situ straining experiments in a transmission electron microscope (TEM). The  
20    TNM alloy exhibits a limited ductility, with the fracture surface of test specimens showing  
21    cleavage facets along lamellar interfaces. In addition, the  $\beta_0$  grains contain nano-precipitates of  
22    the  $\omega_0$  phase and deform plastically by  $\langle 111 \rangle$  superdislocations dissociated into two  
23    superpartial dislocations separated by an antiphase boundary. These dislocations glide in  $\{011\}$   
24    planes and are observed to be localized into pile-ups, which is related to the  $\omega_0$  strengthening

25 precipitation within the  $\beta_0$  grains. This localised hardening behaviour leads to stress  
26 concentration in grain boundaries which is assumed to be responsible for delamination along  
27 lamellar interfaces in neighbouring lamellar colonies.

28

29 **Keywords:** A. titanium aluminide alloys; B. brittleness and ductility, dislocation structure,  
30 plastic deformation mechanisms; C. powder metallurgy; F. transmission electron microscopy;

31

## 32 **1. Introduction**

33 The new generation of intermetallic  $\gamma$ -TiAl-based alloys suitable for high-temperature  
34 structural applications usually contains refractory elements in order to improve mechanical  
35 properties at elevated temperatures via the reduction of dislocation mobility. These elements,  
36 e.g. V, Nb, Cr, Mo, Re or W, also promote the formation of the body-centred cubic (bcc) phase  
37 as disordered  $\beta$  phase (A2 structure) at high temperatures and ordered  $\beta_0$  phase (B2 structure)  
38 at lower temperatures. As mentioned in the exhaustive review of Appel et al. [1] on TiAl alloys,  
39 the role of this phase on mechanical properties is still unclear: “*A possible concern for the  $\beta$ -*  
40 *solidifying alloys is the reported reduction in mechanical properties such as room temperature*  
41 *ductility, high-temperature strength and creep resistance associated with the presence of the*  
42  *$\beta$ /B2 phases””. A review of the current literature does confirm that the ductility of these  $\beta$ / $\beta_0$   
43 phases remains an open question.*

44 Indeed, on one hand, the  $\beta_0$  phase is considered to present very limited plastic  
45 deformation. In a Nb and Mo containing TiAl alloy, Hoppe et al. [2] showed a dislocation-free  
46  $\beta_0$  grain located between two deformed lamellar colonies and assumed that an elastically  
47 transmitted deformation occurs between colonies promoted by a high internal stress. In-situ  
48 high-energy X-ray diffraction tensile experiments were performed at room temperature (RT)

49 on sheets of the so-called TNM-TiAl alloy with a nominal composition of Ti-43.5Al-4Nb-1Mo-  
50 0.5B (in at.%) [3]. In this study, a linear variation of the lattice strain for the  $\beta_0$ -reflections was  
51 found as a function of the applied stress, which was interpreted as indicative of no plastic  
52 deformation in this phase. Sun et al. [4] measured the tensile behaviour of TiAl alloys with  
53 different amounts of Fe, Cr, V and Nb and found a decrease in plastic elongation with the  
54 increase in the content of alloying elements, which is associated with an increase in the  
55 proportion of  $\beta_0$  phase. Based on the observation of cleavage fracture facets in  $\beta_0$  grains and  
56 despite the presence of dislocations, these authors concluded to the brittleness of this phase.

57 On the other hand, Morris et al. [5, 6] investigated the RT mechanical properties of a  
58 Ti-44Al-2Mo (at%) alloy which shows that plastic elongation (0.5%) was higher in the as-  
59 HIPed condition with a higher  $\beta_0$  phase volume fraction (15%), compared to the other process  
60 conditions. Besides, these authors observed two types of dislocations in the  $\beta_0$  grains:  $\langle 100 \rangle$   
61 dislocations and dissociated  $\langle 111 \rangle$  superlattice dislocations, which formed pile-ups nucleated  
62 at grain boundaries under the effect of deformation in adjacent grains. The limited ductility of  
63 this alloy was attributed to both a low mobility of dislocations in the  $\beta_0$  phase and the lack of  
64 slip activity in the  $\alpha_2$  phase. Finally, Leitner et al. [7], who listed all the microstructural features  
65 playing a role on the fracture toughness of  $\beta_0$  containing TNM-TiAl alloys, did propose a  
66 negative effect of this phase as it forms a continuous seam at lamellar colony boundaries.

67 Based on these discrepancies in the literature about the role of the  $\beta_0$  phase on plasticity  
68 and fracture properties of  $\gamma$ -TiAl based alloys, the present work aims at clarifying this open  
69 question by combining an experimental approach of post-mortem and in-situ Transmission  
70 Electron Microscope (TEM) observations.

71 For this purpose, a pre-alloyed powder with the chemical composition of the TNM-TiAl  
72 alloy,  $\text{Ti}_{51.05}\text{Al}_{43.9}\text{Nb}_4\text{Mo}_{0.95}\text{B}_{0.1}$ , in at.%, was densified by Spark Plasma Sintering (SPS). The

73 metallurgical features and mechanical properties of this alloy family are summarised elsewhere  
74 [8, 9]. Consistently with the Nb and Mo contents, the  $\beta/\beta_0$  phase is a constituent of this alloy  
75 associated with the  $\gamma$  and  $\alpha_2$  phases.

76 In TiAl alloys, the  $\beta$ -stabilizing element Nb limits the formation of the  $\gamma$ -TiAl phase  
77 ( $L1_0$  structure) and shifts the  $\beta$  and  $\beta_0$  phase field region to higher Al concentrations. The  $\beta_0$   
78 phase is known to transform into  $\omega$ -type phases at temperatures ranging between 500 °C and  
79 1100 °C, depending on alloy composition and thermal treatments [10, 11]. This transformation  
80 of  $\omega$ -type hexagonal phases in bcc phases is similar to that commonly observed in commercial  
81  $\beta$  or near- $\beta$  Ti base alloys [12-17], or more generally in alloys of group IV elements such as Ti,  
82 Zr and Hf [17-19]. The transformation occurs through local chemical rearrangements associated  
83 with the collapse of  $\{111\}$  bcc planes, leading to the formation of double layers, the latter  
84 alternating with single layers. Because there are four  $\{111\}_{\beta_0}$  planes in a single grain, four  
85 rotational orientation variants can be generated.

86 In the case of intermetallic TiAl alloys, extensive research has been done to determine the  
87 completeness of the bcc/hexagonal transformation, as well as the related chemical  
88 rearrangements [20-23]. In particular, this transformation was investigated in a cast TNM alloy  
89 with a chemical composition similar to that of the alloy studied here [21,23]. In-situ high-energy  
90 X-ray diffraction heating experiments allowed Schloffer et al. [21] to measure the solvus  
91 temperature of  $\omega_0$  phases to be 825 °C. Klein et al. [23] proposed that the growth sequence was  
92 dominated by the redistribution of Mo into the  $\beta_0$  matrix, i.e. by the Mo diffusivity.

93

94

95

## 96 2. Materials and methods

97 The pre-alloyed TNM powder produced by argon gas atomization with the Electrode  
98 Induction Melting Gas Atomization (EIGA) technique [24] was densified by SPS using the  
99 thermal and pressure cycle described elsewhere [25]. To achieve a lamellar microstructure  
100 containing  $\beta_0$  grains, the dwell temperature ranged between 1300 °C and 1360 °C [26].  
101 Throughout the paper, the indicated temperatures correspond to the actual temperatures of the  
102 powder and material (for detailed information regarding the SPS process the reader is referred  
103 to Ref. [25]). Cylindrical billets with a 36 mm diameter and an 8 mm thickness were sintered  
104 under four different conditions: three different dwell temperatures, one of them (1360 °C)  
105 repeated with a 60 min instead of the usual 2 min (see [Table 1](#)). No heat treatment was applied  
106 to the material after sintering. Three specimens suitable for RT tensile tests ( $10^{-4} \text{ s}^{-1}$  strain rate)  
107 were machined from each billet.

108 A JEOL 6490 Scanning Electron Microscope (SEM) was used to characterize the  
109 microstructures in back-scattered electron (BSE) mode, and to analyse the fracture surfaces in  
110 secondary electron (SE) mode. Electron dispersive spectroscopy (EDS) analyses were  
111 performed with a FEI Helios 600i SEM equipped with an Oxford detector. The volume fractions  
112 of the  $\beta_0$  phase in the different materials were measured by X-ray diffraction (XRD) analysis  
113 on an AXS D8 Advanced diffractometer from Bruker. A JEOL 2020 TEM operating at 200 kV  
114 was used to analyse both the dislocations microstructures generated by the tensile tests (post-  
115 mortem microscopy) and the controlling dislocations mechanisms (in-situ RT straining  
116 experiments) with a Gatan straining holder). Every TEM samples for both post-mortem and in-  
117 situ experiments were taken from the AM504 billet ([Table 1](#)).

118

119

### 120 3. Microstructure of the as-SPSed TNM alloy

121           Regarding the four billets under examination (Table 1), it appears that billet AK639 with  
122 the lowest dwell temperature exhibits a lower volume fraction of the  $\beta_o$  phase compared to the  
123 three other billets. Billet AM504 with a specific long duration at dwell temperature shows larger  
124 lamellar colonies. The common microstructural feature for the four materials corresponds to  
125 large lamellar colonies with  $\beta_o$  grains along boundaries (see Fig. 1). As described in section 4,  
126 these similar microstructures result in similar RT mechanical behaviour (Fig. 3).

127           Regarding the lamellar microstructure, electron backscatter diffraction (EBSD) fails to  
128 delimit the colony size since this technique analyses each lamellae of a given colony as a  
129 different grain (indeed, in a colony the lamellae can take six different orientations, which  
130 manifest in ordered domains, twins or pseudo-twin relationships [27]). Therefore, the colony  
131 size has been measured using a manual procedure applied on SEM micrographs. For each  
132 measurement, one hundred adjacent colonies were isolated and their size measured using the  
133 free ImageJ software. The grain size is defined as the diameter of a circle with the same area as  
134 the respective colony. The average values as well as the corresponding standard deviations ( $\sigma$ )  
135 are summarized in Table 1 for the four specimens. The relatively high value of  $\sigma$  (in the order  
136 of half the average colony size) reflects a large scattering of the colony sizes that typically range  
137 from a few microns to nearly one hundred  $\mu\text{m.}$ , The volume fraction of the  $\beta_o$  phase as measured  
138 by XRD is also reported in Table 1, does increase with dwell temperature up to 1325 °C and  
139 then remains more or less constant.

140

141

142

143

144 **Table 1**

145 Microstructural characterization of the obtained as a function of the SPS dwell temperature.

Dwell temperature (°C)	1300	1325	1360	1360 - 60 min -
Specimen designation	AK639	AN849	AL824	AM504
Other SPS billets used in the study		AK746 AM797		
Average colony size (µm)	17.27	17.38	17.11	28.04
Standard deviation (µm)	11.27	9.07	8.95	13.15
Vol.% of the $\beta_o$ phase	5.6	8.2	8.4	9

146

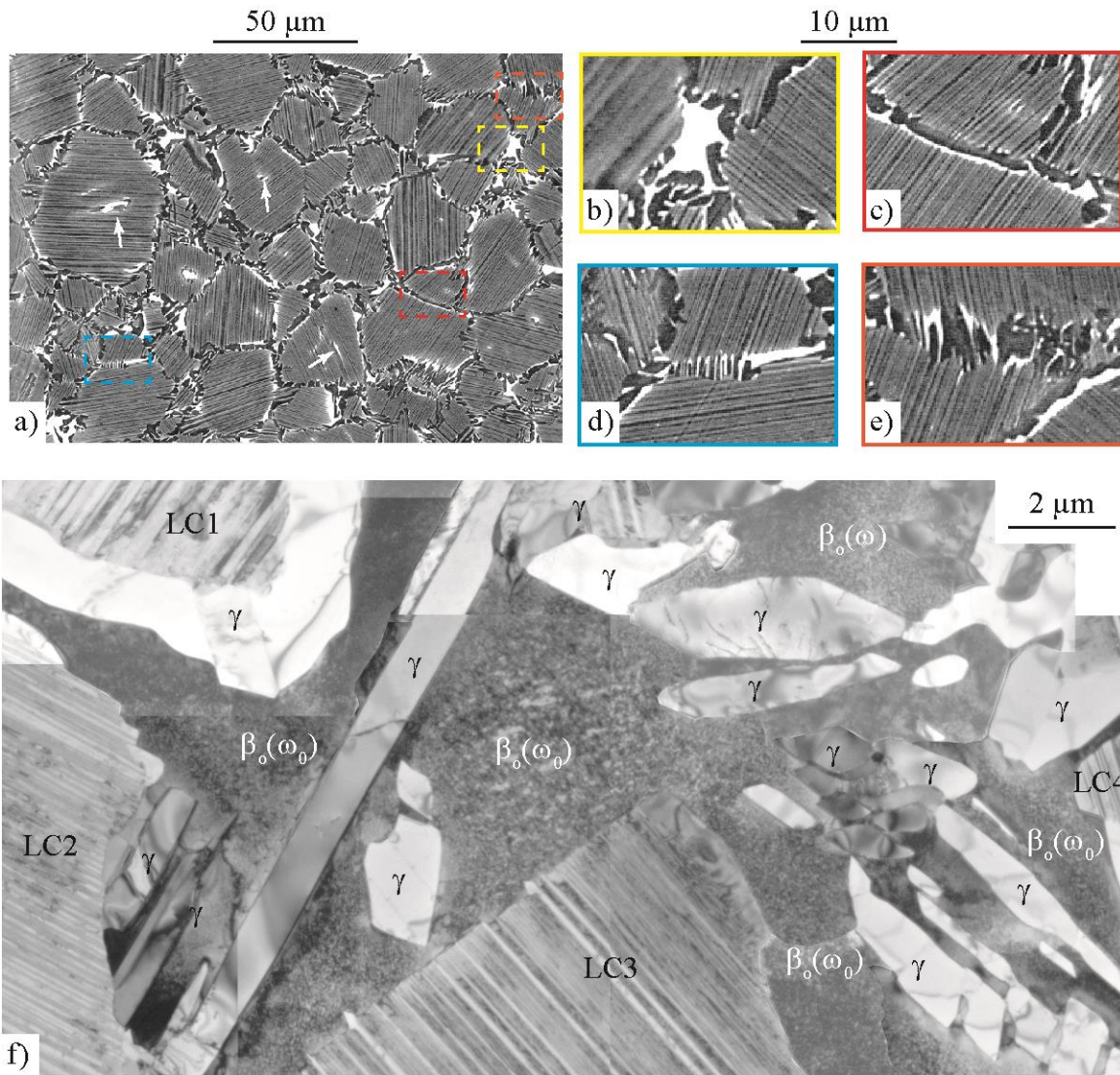
147 Therefore, the microstructure presented in Fig. 1 (billet AK746 for SEM analysis, Figs.  
 148 1a-e, and billet AM 504 for TEM image of Fig. 1f) is representative for the different sintered  
 149 materials. The SEM micrograph of Fig. 1a shows a near lamellar microstructure made of  $\gamma/\alpha_2$   
 150 lamellar colonies surrounded by borders with  $\gamma$  and  $\beta_o$  grains exhibiting black and white colours,  
 151 respectively. The  $\beta_o$  phase is distributed in different ways as evidenced by the corresponding  
 152 enlargements (Figs. 1b-e) of Fig. 1a:

- 153 - Extended shapes, situated at junctions between colony boundaries (enlargement framed in  
 154 yellow, Fig. 1b),
- 155 - Elongated features, generally located along a boundary between a border and a lamellar colony  
 156 (enlargement framed in red, Fig. 1c),
- 157 - Series of parallel, elongated and thin noodle-shaped phase immersed in a  $\gamma$  grain, generally  
 158 oriented perpendicularly to the boundaries (enlargement framed in blue, Fig. 1d),
- 159 - Small precipitates, lath- or noodle-shaped (enlargement framed in orange, Fig. 1e) and,
- 160 - Bright zones located inside lamellar colonies (bright arrows in Fig. 1a).

161 Figure 1f shows a TEM micrograph of an area with the  $\beta_o$  phase distributed at the  
 162 junction between several colony boundaries according to the situation illustrated in Fig. 1b.



163 Here, lamellar colonies (LC) and  $\gamma$  grains (bright contrast) are easily distinguishable. The  
 164 darkest parts marked  $\beta_o(\omega_o)$  exhibit the so-called tweed contrast typical for the  $\beta_o$ -phase  
 165 containing  $\omega$ -type precipitates.



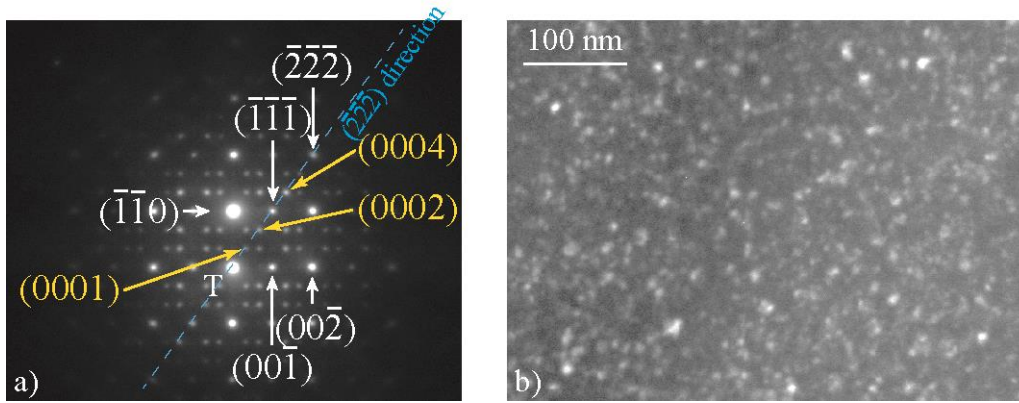
166 f)

167 **Fig. 1.** Microstructure of TNM-TiAl: (a) SEM micrograph of specimen AK746 taken in BSE  
 168 mode with lamellar colonies surrounded by borders with  $\gamma$  and  $\beta_o$  grains in black and white  
 169 colours, respectively. The four colour-framed micrographs correspond to four different  
 170 enlargements displayed in (b), (c), (d) and (e) showing different distributions of the  $\beta_o$  phase  
 171 (see text for details); (f) TEM micrograph of specimen AM504 with  $\beta_o$  phase distributed  
 172 according to the situation imaged in Fig. 1b.

173

174 [Figure 2a](#) depicts a diffraction pattern comparable to that previously reported for the bcc phases  
175 of conventional disordered Ti alloys [13,15,17,19] or intermetallic TiAl alloys. [10,11]. The  $\beta_o$   
176 phase is oriented with a  $\langle 110 \rangle_{\beta_o}$  direction of the zone axis parallel to the electron beam in this  
177 diagram, and the additional spots are due to the presence of two  $\omega_o$  variants. Looking at the  
178  $(\overline{222})_{\beta_o}$  direction in particular, several spots are believed to originate from the variant for which  
179 the c axis is parallel to the  $(\overline{222})_{\beta_o}$  direction: one fundamental reflexion and two superstructure  
180 spots, resulting from the long range ordered structure. However, it may be argued that similar  
181 patterns have been seen in Ti and Zr alloys exhibiting a disordered  $\beta$  structure [13,15,17,19].  
182 On the basis of image simulations, Sabeena [28] proposed that double diffraction explains the  
183 presence of these spots in a Ti-15Mo alloy.

184 [Figure 2b](#) depicts a dark field micrograph of a  $\beta_o$  area taken with  $g=(0002)_{\omega_o}$  superlattice  
185 diffraction vector, where the  $\omega_o$  precipitates seem to have circular morphology. By counting the  
186 number of precipitates and determining the precipitates volume fraction, the  $\omega_o$  precipitates are  
187 found to occupy a few volume percent (0.4 to 3.6 %) of the material and the order of magnitude  
188 of the distance between precipitates is about 27 nm (390 precipitates counted for one variant in  
189 a  $182 \times 394 \times 434 \text{ nm}^3$  volume). Using the methods described in Ref. [29], a precise determination  
190 of the volume fraction, the precipitates size and spacing will be carried out in a forthcoming  
191 investigation. In addition, work is underway to determine the precise crystallographic structure  
192 of these  $\omega_o$  precipitates, with a particular attention to their ordering state. Former studies have  
193 shown that the precipitation of  $\omega_o$  phase requires either an ageing at intermediate temperatures,  
194 e.g.  $700^\circ\text{C}$  [10] or  $850^\circ\text{C}$  [21], or a slow cooling rate of 10 K/min [20]. This is consistent with  
195 our SPS experiments that exhibit cooling down from the dwell temperature ( $1300^\circ\text{C}$  or above,  
196 see [Table 1](#)) at a cooling rate of 100K/min inside the graphite mold maintained in the SPS set-  
197 up [25].



198

199 **Fig. 2.** Characterization of the  $\omega_0$  precipitates in  $\beta_0$  grains. a) Diffraction pattern with  
 200  $\langle 011 \rangle_{\beta_0}$  zone axis (the spots labelled in white correspond to the  $\beta_0$  phase; those labelled in  
 201 yellow to the  $\omega_0$  phase; the blue line indicates the  $(\bar{2}\bar{2}\bar{2})_{\beta_0}$  direction); b) Dark field micrograph  
 202 of the  $\omega_0$  precipitates taken with  $g=(0001)_{\omega_0}$  superlattice diffraction vector.

203

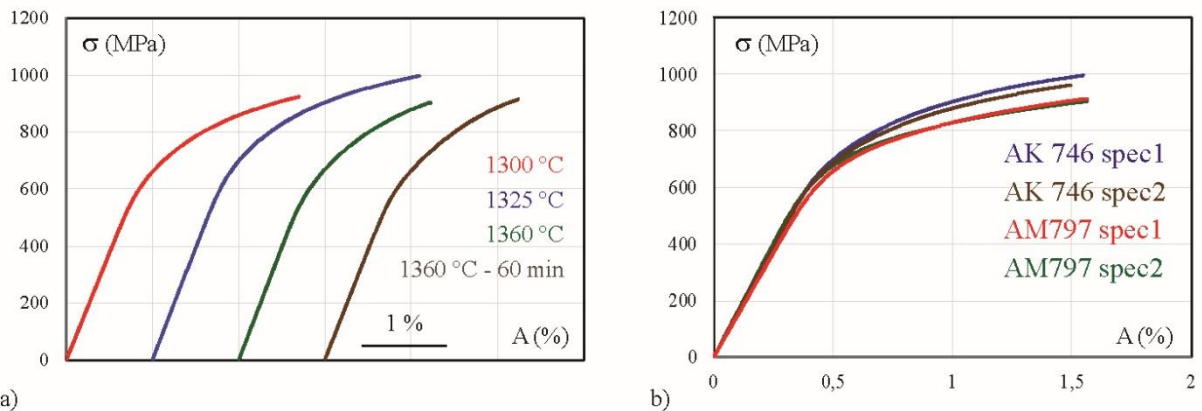
#### 204 4. Mechanical tests and fracture surfaces

205 The RT true stress vs. true strain curves ( $10^{-4} \text{ s}^{-1}$  strain rate) are shown in [Figure 3a](#) for the four  
 206 SPS billets.

207 Yield stress (YS) is the stress at which plastic deformation starts and is defined as the stress at  
 208  $10^{-3} \%$  plastic elongation,  $R_{0.2}$  denotes the tensile stress at 0.2 % plastic elongation, UTS the  
 209 ultimate tensile stress, and the term A represents the plastic elongation at rupture. As can be  
 210 seen in Table 2, the four billets exhibit similar mechanical properties. Indeed, as already  
 211 emphasized in as-SPSed B-containing TiAl alloys with the so-called GE composition (Ti-48Al-  
 212 2Cr-2Nb, in at.%) [30], as soon as the dwell temperature is situated in the single  $\alpha$  or  $\alpha+\beta$  phase  
 213 field, microstructures and mechanical properties are highly reproducible. The alloy densified at  
 214  $1325 \text{ }^\circ\text{C}$  appears to have slightly improved properties resulting from a higher plastic elongation,  
 215 which leads to a higher ultimate tensile strength (UTS). This trend is not easily explained from  
 216 the measurements of the average grain size and of the volume fraction of  $\beta_0$  phase. [Figure 3b](#)

217 shows the curves obtained for four specimens extracted from two billets both densified at  
 218 1325°C (billets AK746 and AM797). These four samples exhibit very similar properties with  
 219 reduced deviations on the different measured stresses (YS, R0.2, UTS) and on the plastic  
 220 elongation at failure.

221 The plastic elongation at rupture for all hot-worked billets tested is between 0.5 and 1.0 %. As  
 222 noted by Appel et al. [1], this limited RT ductility is typical for  $\beta$ -solidifying alloys, which  
 223 possess a fairly high RT strength. However, using SPS as processing route appears to result in  
 224 a slight increase in RT plastic elongation by generating uniform and non-textured  
 225 microstructures.



226 a) b)  
 227 **Fig. 3.** True RT stress-strain curves for the four investigated SPS treatments. (a) one curve for  
 228 each condition; (b) four curves for the AK746 and AM797 specimens - 1325 °C dwell  
 229 temperature (see [Table 2](#) for details).

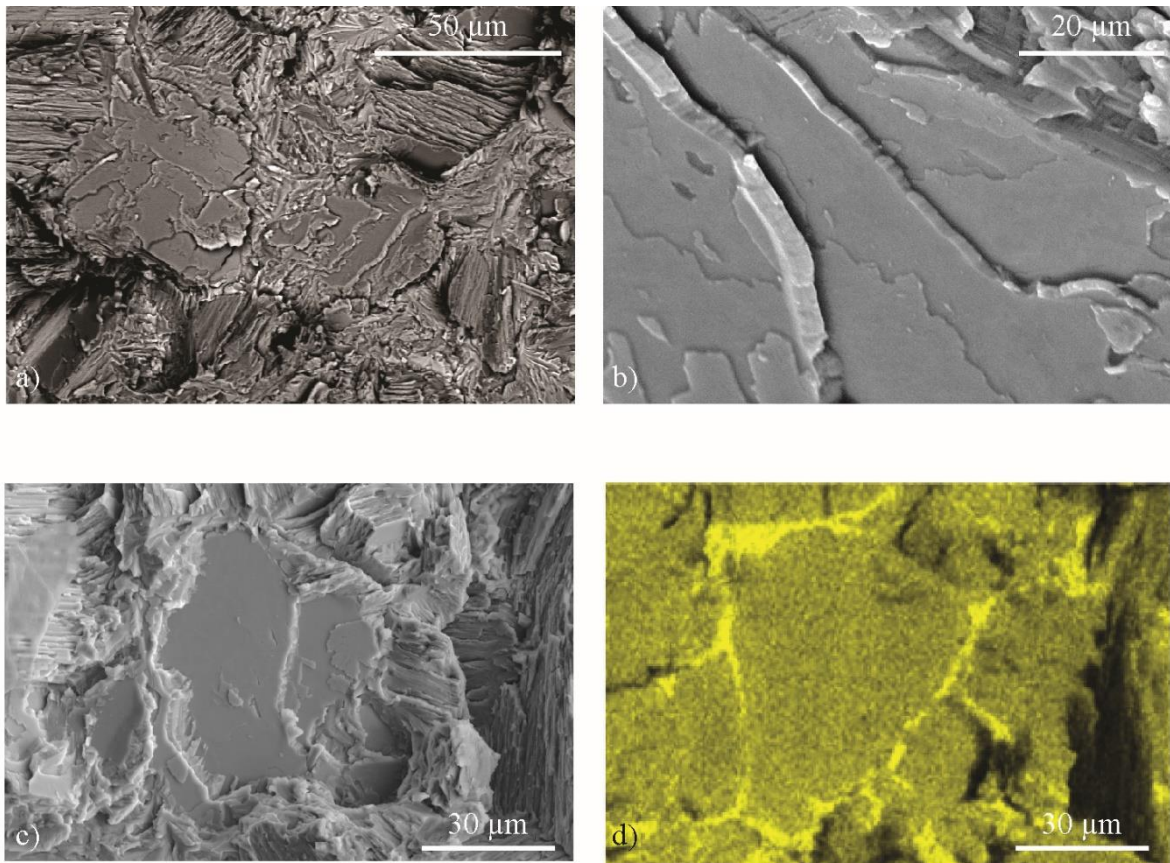
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238 **Table 2**239 RT tensile tests results ( $10^{-4} \text{ s}^{-1}$  strain rate).

Specimen name	Dwell temperature		YS (MPa)	R <sub>0.2</sub> (MPa)	UTS (MPa)	A (%)
AK 639	1300 °C	Spec 1	559	771	923	0.73
		Spec 2	554	759	910	0.75
AK 746	1325 °C	Spec 1	591	814	995	0.92
		Spec 2	583	792	961	0.81
AM797		Spec 1	559	755	914	0.93
		Spec 2	545	748	905	1
AL 824	1360 °C	Spec 1	538	770	901	0.54
AM 504	1360 °C - 60 min -	Spec 1	554	779	914	0.52
		Spec 2	528	763	880	0.44

240

241 A SEM study of the fracture surfaces is presented in [Figure 4](#) for two RT deformed samples  
242 (specimens AK 639 – [Figs. 4a-b](#) and AM504 - [Figs. 45c-d](#), [Table 2](#)). [Figure 4a](#) shows  
243 representative cleavage facets, the size of which matches that of lamellar colonies. The cleavage  
244 is likely to occur along lamellar interfaces. Between these cleavage facets, transgranular crack  
245 propagation is observed, as emphasized in [Figure 4b](#) showing at higher magnification some  
246 steps between the cleaved planes, which might be in the  $\gamma$  lamellae. The micrographs in [Fig. 4c](#)  
247 and [Fig. 4d](#) depict an area both in backscattered mode and by a Mo EDX SEM map,  
248 respectively. The  $\beta_0$  phase is indicated by the bright yellow regions. This demonstrates that the  
249  $\beta_0$  areas do not show any particular brittle behaviour.



250

251 **Fig. 4.** SEM characterization of the fracture surface of RT deformed samples until rupture (AK  
 252 639 – Figs. 4a-b and AM504 - Figs. 4c-d, Table 2). (a) and (b) fracture surfaces; (c) and (d)  
 253 same area in BSE mode and as a Mo EDX SEM map, respectively.

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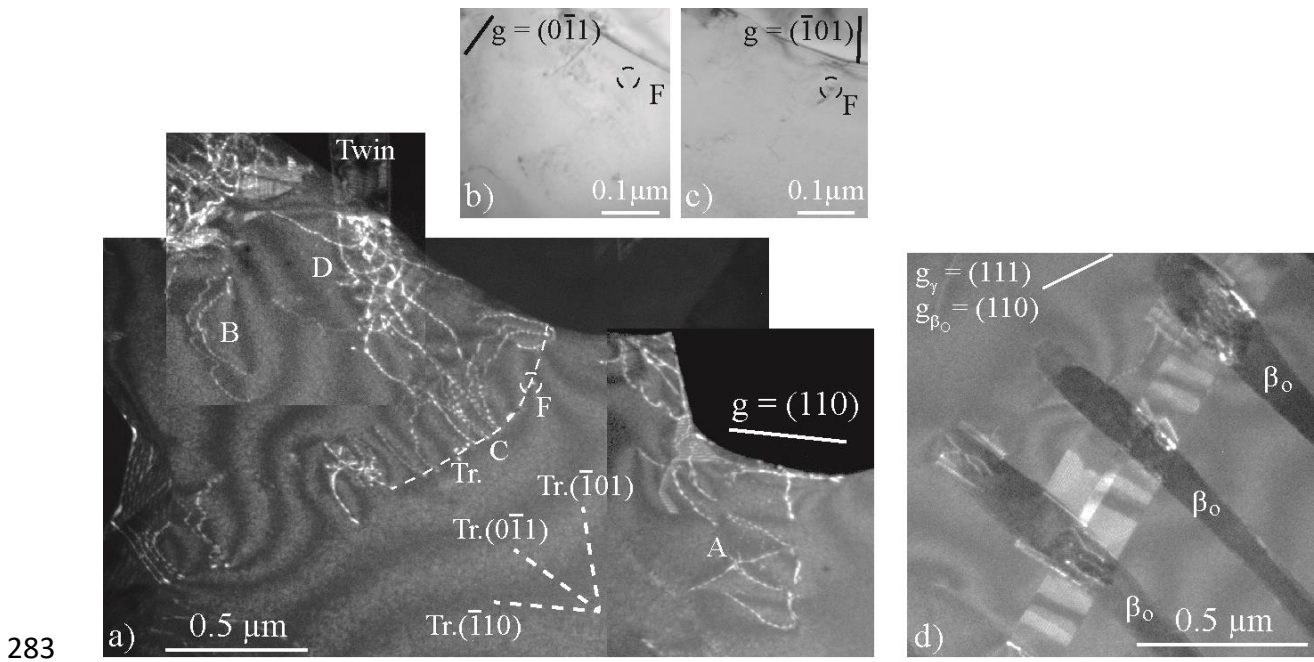
## 256 5. Study of the deformation processes for $\beta_0$ grains

257 Characterization of the deformation processes operating in  $\beta_0$  grains was carried out by both  
 258 TEM post-mortem analyses of thin foils extracted from RT deformed samples until rupture  
 259 (specimen AM 504, Table 2) and TEM in-situ straining experiments in samples cut from the  
 260 same AM 504 billet.

261

### 262 *5.1. Dislocation microstructure in deformed samples*

263 About fifteen grains were examined by TEM and the presence of dislocations was observed in  
264 most of them (Figures 5 and 6). On the general view (Fig. 5a), several groups of dislocations  
265 are observed. As shown in the two inserts (Figs. 5b and c), dislocations are out of contrast with  
266 the  $(0\bar{1}1)$  and  $(\bar{1}01)$  reflexions demonstrating that their Burgers vector is parallel to the  $[111]$   
267 direction, as this is the case for every dislocations in this area (circled detail labelled F serves  
268 as a mark in Figs. 5a-c). Dislocations are generally organized in pile-ups, where traces on the  
269 thin foil surfaces permit to determine their glide plane. For the pile-up of three dislocations  
270 (marked A, Fig. 5a), the trace is parallel to the  $(\bar{1}01)$  plane. A tilt experiment (not reproduced  
271 here), for which this pile-up was observed at different inclinations tilted around this trace has  
272 clearly confirmed that it is located in the mentioned  $(\bar{1}01)$  plane, which is a glide plane for these  
273  $[111]$  dislocations. This is also the case for the pair of dislocations marked B. The trace of the  
274 main pile-up marked C is not straight but curved (see dashed line) indicating changes in the  
275 glide plane. The analysis of the surrounding  $\gamma$  grains showed that these pile-ups can be  
276 associated with the locking of a twin on the grain boundary, as in case D, or not, as in the case  
277 of pile-up C. Hence, Fig. 5d confirms the strong influence of the deformation occurring in the  
278  $\gamma$  phase on that proceeding in the  $\beta_o$  phase. In this area, three parallel noodle-shaped  $\beta_o$  grains  
279 are situated within one  $\gamma$  grain deformed by twins. Clearly, some dislocations are seen in the  
280 noodle-shaped constituents in planes following those of the twin habit planes, thus indicating  
281 that the emission of dislocations in the  $\beta_o$  phase results from the transmission of deformation  
282 from surrounding  $\gamma$  phase.

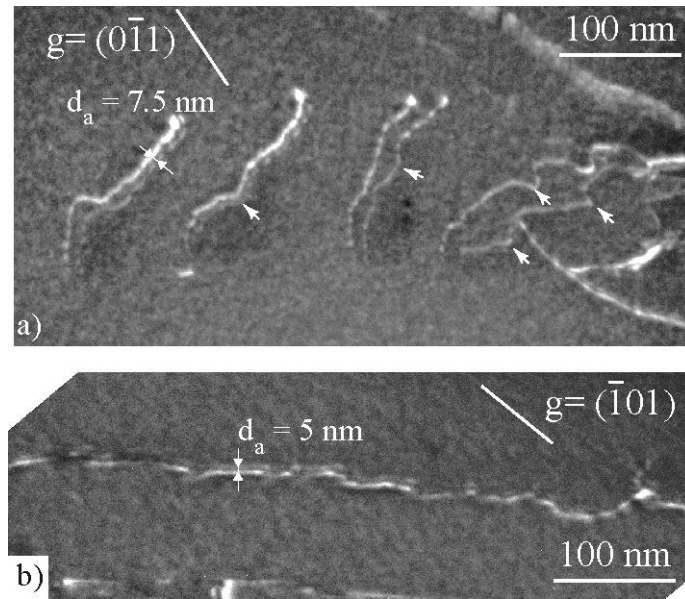


284 **Fig. 5.** Characteristic occurrence of dislocations observed by TEM (weak-beam conditions) in  
 285 a sample macroscopically strained until rupture (AM504, Table 2); (a) general view; (b) and  
 286 (c) same dislocations out of contrast under two imaging conditions; (d) three parallel noodle-  
 287 shaped  $\beta_o$  phase containing some dislocations inside a  $\gamma$  grain deformed by mechanical twins.

289 **Figure 6**, which is taken under higher magnification, gives two examples of the structure of  
 290 these  $\langle 111 \rangle$  dislocations. In **Fig. 6a**, dislocations are localized in a pile-up, whereas **Fig. 6b**  
 291 shows an isolated long dislocation, the rectilinear segments of which are parallel to the screw  
 292 direction of the dislocation. Dislocations are often pinned at small obstacles, as indicated by  
 293 small white arrows in **Fig. 6a**. Every dislocations clearly appear as paired dislocations in every  
 294 cases but with notable differences in spacing. In particular in the pile-up, various spacing is  
 295 likely to occur from the presence of pinning points (arrowed). Without pinning points, the  
 296 spacing of paired dislocations is approximately constant all along the line with an apparent  
 297 value ( $d_a$ ) ranging from 5 to 10 nm. The long dislocation also exhibits macrokinks, which are  
 298 reminiscent of the dislocation structures in  $L1_2$  intermetallic compounds such as  $Ni_3Al$  and



299 Ni<sub>3</sub>Ga [31, 32]. It is even tempting to underline an alignment of the leading and trailing partials  
 300 bordering a macrokink as evidenced in Ni<sub>3</sub>Al and Ni<sub>3</sub>Ga alloys deformed at RT [31, 33] as the  
 301 result of jump amplitudes equal to the width of the antiphase boundary separating the two  
 302 superpartial dislocations of the a<011> dislocations involved in these face-centered cubic type  
 303 intermetallic alloys.

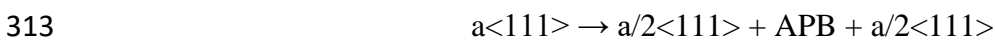


304  
 305 **Fig. 6.** Two examples of the structure of a<111> superdislocations observed by post-mortem  
 306 TEM under weak-beam conditions (AM504). (a) dislocations localized in a pile-up; (b) isolated  
 307 dislocation.  $d_a$  corresponds to the apparent average distance between superpartial  
 308 dislocations.

309

310 The obtained experimental results can be interpreted as follows:

- 311 • a<111> dislocations are dissociated into two identical dislocations bordering an  
 312 antiphase boundary (APB), consistently with the following dissociation scheme:



314 (as the lengths of the Burgers vectors of these  $a/2\langle 111 \rangle$  partial dislocations and  $a\langle 111 \rangle$   
315 dislocations result from the long range ordered structure, they are generally called  
316 superpartial dislocations and superdislocations).

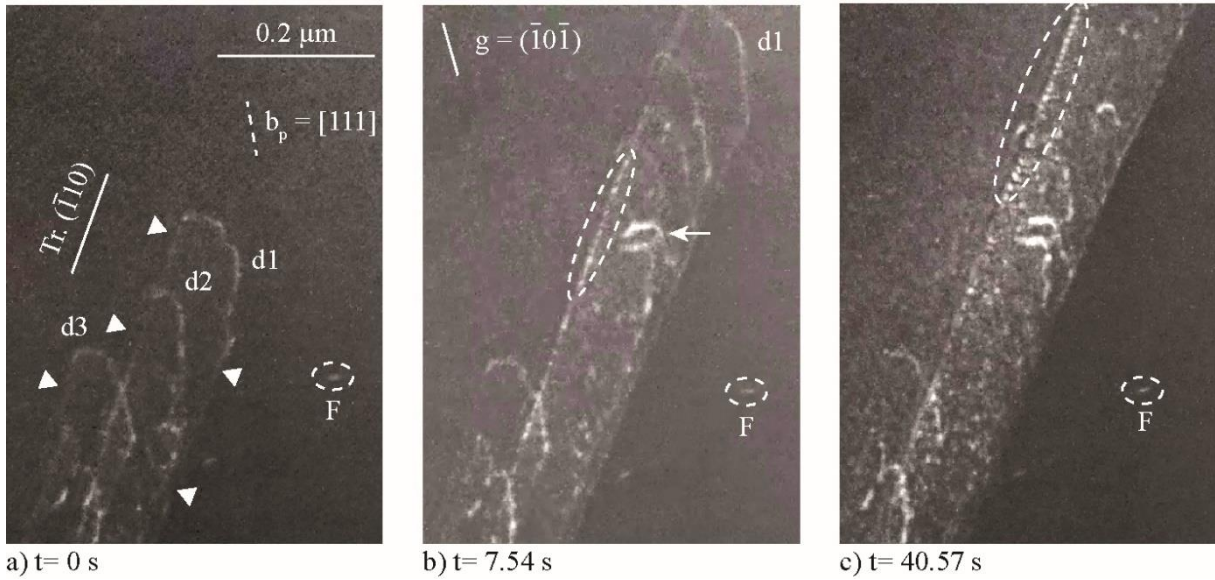
- 317 • Dislocations are often confined in pile-ups, gliding in  $\{110\}$  type planes.
- 318 • Assuming that the APB ribbon lays in the gliding plane, a dissociation width ranging  
319 from 10 to 20 nm can be deduced from the apparent dissociation distances measured on  
320 the rectilinear dissociated dislocation segments.
- 321 • Extrinsic obstacles pin superpartial dislocations, leading to a wide range of dissociation  
322 distances of the superdislocations.

323

## 324 ***5.2. In-situ observation of the movement of $a\langle 111 \rangle$ dislocations in $\beta_0$ grains***

325 The movement under stress of these  $a\langle 111 \rangle$  dislocations was studied by performing in-situ RT  
326 straining experiments in a TEM (note that [Figs. 7-10](#) were made from the video sequences  
327 available for each figure; it is worth noting that these sequences are at real speed). The main  
328 tendency of these dislocations is to move in pile-ups as illustrated by [Fig. 7](#) (video [here](#)), where  
329 a small defect, labelled F, is used as a reference point. In [Fig. 7a](#), three dislocations (labelled  
330 d1, d2 and d3) with a  $a[111]$  Burgers vector are visible. They have left slip traces behind them  
331 (marked by white triangles) in the form of weak contrasted lines on the two thin foil surfaces.  
332 The determination of the grain orientation through a stereographic analysis has shown that these  
333 traces are parallel to the  $(\bar{1}10)$  plane containing the  $a[111]$  Burgers vector. With elapsing time  
334 ([Fig. 7b](#)), d1 and d2 have glided on upward in this plane and a lot of dislocations are now present  
335 in a pile-up created in the wake of d1 (each bright dot on the upper trace of the pile-up indicates  
336 the presence of one gliding dislocation; the dashed oval surrounded some of these dots). In this  
337 figure, the pile-up tip is visible in the observed area and a dislocation loop (arrowed) was formed  
338 due to the pinning of one dislocation. Between [Fig. 7b](#) and [Fig. 7c](#), d1 has moved on, followed

339 by the pile-up. In Fig 7c, the pile-up tip is out of the frame and the dashed oval shows some  
 340 twenty dots/piled-up dislocations. The dynamic sequence allows claiming that several hundreds  
 341 of dislocations glided in the  $\beta_0$  grain in a few seconds following the path of dislocation d1.



342 a)  $t = 0$  s                      b)  $t = 7.54$  s                      c)  $t = 40.57$  s

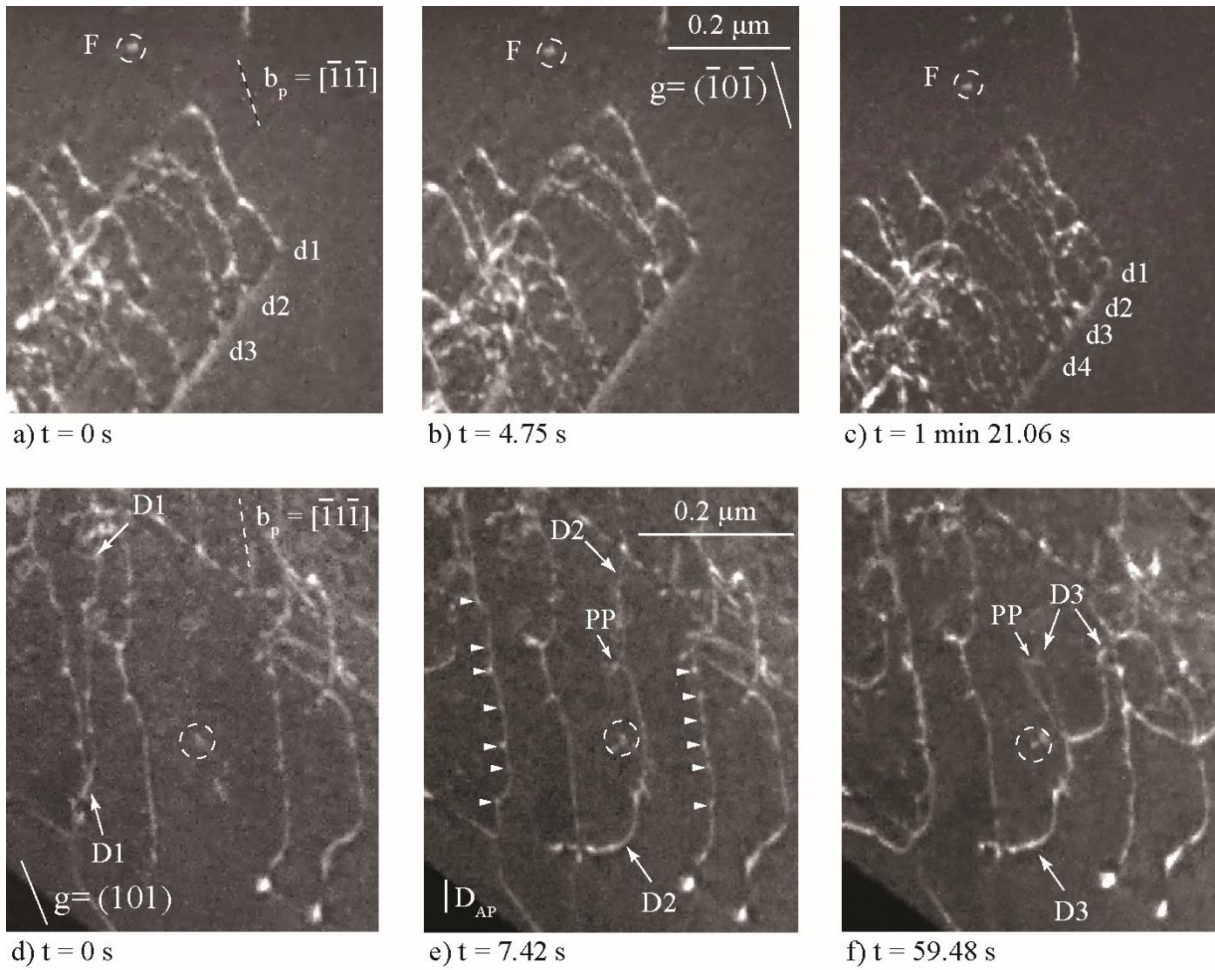
343 **Fig. 7.** Formation of a  $(\bar{1}10)$  pile-up of  $a[111]$  dislocations during an in-situ RT straining  
 344 experiment in a TEM (see corresponding video sequence and text for more details). (a) initial  
 345 situation with dislocations d1, d2 and d3 ( $b_p$  direction corresponds to the projected direction  
 346 of the  $[111]$  Burgers vector on the image plane); (b) creation of the pile-up in the wake of  
 347 dislocation d1; (c) progress of the pile-up made of hundreds of dislocations.

348

349 Figure 8 details the elementary mechanisms of propagation of these dislocations. Figures 8a-c  
 350 (video [here](#)) depict another pile-up generated in the same grain as shown in Fig. 7, whereas  
 351 Figs. 8d-f show the situation in another sample. Figs. 8a-c show a pile-up tip, where dislocations  
 352 appear dissociated according to the dissociation mode analysed in section 5.1 (F is a reference  
 353 point). In this area, the pile-up tip is made of three superdislocations (Figs. 8a-b) joined later  
 354 on by a fourth one (Fig. 8c). It can be seen that the spacing between the two superpartials of a  
 355 given superdislocation is increased with the distance of the considered superdislocation from

356 the pile-up tip (in Fig. 8a, dislocation d4 is more widely dissociated than dislocation d2, for  
357 example), as that was observed in the bulk (Fig. 6a). The spacing between the two superpartial  
358 dislocations of leading dislocation d1 is even difficult to observe due to the resolution limit of  
359 this micrograph. Between Fig. 8a and Fig. 8b, however, all three dislocations have moved on.  
360 Examination of the video shows that these dissociated superdislocations move in a jerky  
361 manner, mainly by independent jumps of the superpartials. Figs. 8d-f (video [here](#)) follow the  
362 movement of another  $a\langle 111 \rangle$  superdislocation in a different grain, which is not strictly  
363 speaking part of a pile-up even though several other dislocations are simultaneously active in  
364 the same area. In this case, imaging conditions did not allow the right resolution to be achieved  
365 for visible dislocation dissociations. Dislocations are elongated along their screw orientation  
366 and anchored at many pinning points (some of them are indicated by small triangles on two  
367 dislocations visible in the frame corresponding to Fig. 8e). A dislocation marked D is followed  
368 between frame 1 (D1 in Fig. 8d) and frame 3 (D3 in Fig. 8f, 1 minute later). The whole  
369 dislocation jumps between positions D1 and D2 between frames Fig. 8d and Fig. 8e (dashed  
370 circled small defect is a fixed spatial reference). In position D2, the dislocation is divided into  
371 two segments separated by a pinning point (marked PP). Subsequently, probably due to the  
372 higher strength of this pinning point, dislocation D splits into two segments that move  
373 independently by the same jerky motion (Fig. 8e), leading to the formation and expansion of a  
374 loop.

375 From these images, it is possible to estimate the distance between the pinning points anchoring  
376 these moving  $a\langle 111 \rangle$  screw dislocations: the apparent distance  $D_{AP}$  is about 30 nm (distance  
377  $D_{AP}$  plotted on frame Fig. 8e), which corresponds to a true distance of 35 nm considering the  
378 inclination of the screw dislocation line. This order of magnitude is compatible with the  
379 apparent distance between  $\omega_6$  precipitates (see Fig. 2b), thereby confirming that dislocation  
380 anchoring occurs on these  $\omega_6$  precipitates.



381

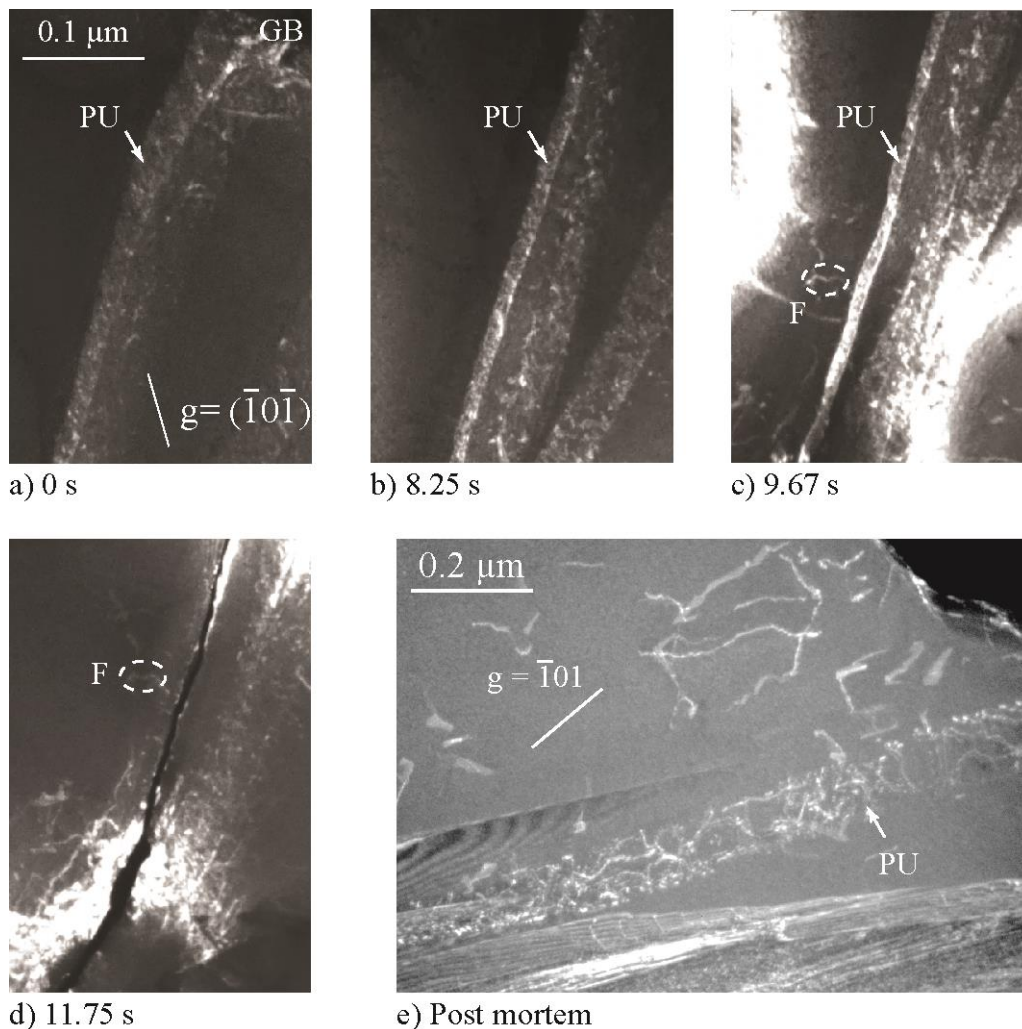
382 **Fig. 8.** Propagation of  $a\langle 111 \rangle$  superdislocations in  $\beta_0$  grains during in-situ RT straining  
 383 experiments in a TEM (see corresponding video sequence and text for more details). Figs. 8a-  
 384 c depict dislocations of a pile-up generated in the same grain shown in Fig. 8; Figs. 8d-f are  
 385 imaging isolated dislocations in another grain of another thin TEM foil.

386

## 387 6. Investigation of the cracking process by in-situ straining experiment

388 In the  $\beta_0$  grain, where the sequence shown in Fig. 7 was taken, four pile-ups were in fact  
 389 observed in almost parallel planes. Figs. 9a-d (video [here](#)) present the evolution of one of them  
 390 labelled PU in Fig. 9a. The dislocations moved upwards in the observed area and Fig. 9a is a  
 391 first micrograph of the area when PU already reached the grain boundary labelled GB located  
 392 at the upper part of the micrograph. The rest of the dynamic sequence was recorded inside the

393  $\beta_0$  grain ('below' GB). Between Figs. 9b and c, the apparent width of PU decreases with the  
 394 accumulation of new dislocations in the pile-up. This effect is due to the relative slip of the  
 395 respective part of the grain located on both sides of pile-up PU. Suddenly, the grain cracked at  
 396 the level of the pile-up, see Fig. 9d. Regarding Fig. 9e, it shows a  $\beta_0$  grain characterized by  
 397 post-mortem TEM in a thin foil extracted from a RT tensile specimen. The pile-up labelled PU,  
 398 evidenced in Fig. 9e, demonstrates that localization of deformation in the form of large pile-  
 399 ups (in addition to the small pile-ups already mentioned in Figs. 5 and 6) is also possible in the  
 400 bulk and not limited to in-situ tests in thin specimens.



401  
 402 **Fig. 9.** Pile-up growing and crack formation in a  $\beta_0$  grain; (a-d) dynamics of a growing pile-  
 403 up leading to the nucleation of a crack (in-situ TEM; see corresponding video sequence and

404 *text for more details); (e) pile-up of the same nature observed by post-mortem TEM conducted*  
405 *in a foil taken from tensile test specimen.*

406

## 407 **7. Discussion**

408 The present TEM studies have shown that the  $\beta_0$  phase can be plastically deformed at RT by  
409 dislocations, consistently with the work of Morris et al. [5, 6]. It is recalled that these authors  
410 investigated a Ti-44Al-2Mo (at%) alloy and observed  $a\langle 111 \rangle$  and  $a\langle 001 \rangle$  dislocations  
411 presumably nucleated in  $\beta_0$  grains by the local stress generated on the grain boundaries by  
412 mechanical twins emitted in neighbouring  $\gamma$  grains. In the present work, deformation is shown  
413 to occur by  $a\langle 111 \rangle$  superlattice dislocations, which are often situated in pile-ups. Some  
414 evidences of deformation in  $\beta_0$  areas originated by twins in  $\gamma$  grains have also been observed  
415 (Fig. 5). Not shown here, other families of dislocations have also been observed and are  
416 currently under investigation. Many of the features of these  $a\langle 111 \rangle$  superlattice dislocations  
417 are reminiscent of observations previously obtained for deformation systems active in other  
418 intermetallic compounds (ordinary dislocations in the  $\gamma$  phase of TiAl, superlattice dislocations  
419 in  $L1_2$  ( $Ni_3Al$  and  $Ni_3Ga$ ) and  $D0_{19}$  structures ( $Ti_3Al$ )):

- 420 • The dissociation scheme determined in section 5.1 is the one expected from the literature  
421 concerning intermetallic alloys with B2 structure [34], even though in NiAl B2-type  
422 intermetallic alloys the core of these dislocations has been found to be very compact by  
423 TEM [35, 36]. These superlattice dislocations are dissociated into two identical  
424 superpartials bordering an antiphase boundary ribbon as in  $Ni_3Al$  [31, 32] and  $Ni_3Ga$   
425 [33], both with the  $L1_2$  fcc-type ordered structure, and also in  $\alpha_2$ - $Ti_3Al$  [37].  
426 Superpartials exhibit superkinks, each of them resulting of a dislocation elementary  
427 jump.

- 428 • The dislocations are elongated along their screw directions, with sometimes some  
429 rectilinear parts for the superpartials, as observed in Ni<sub>3</sub>Al and Ni<sub>3</sub>Ga, as well as during  
430 the prismatic glide in  $\alpha_2$ -Ti<sub>3</sub>Al. This is generally explained by a superpartial core  
431 spreading out of the glide plane, which leads to a lattice friction stress (Peierls stress),  
432 thus hindering dislocation glide.
- 433 • Dislocations glide is made of quick jumps (in less than the time between two successive  
434 video frames i.e. 1/50 s) between two elongated screw configurations. This jerky motion  
435 is also the result of the lattice friction stress [38]. Indeed, dislocations transit from a  
436 stable sessile configuration due to the core spreading, to a non-stable glissile  
437 configuration allowed by the thermally activated recombination of the core under the  
438 effect of the local stress, before again falling in the stable configuration.
- 439 • Dislocations are anchored at pinning points, which are  $\omega_0$  precipitates. This situation is  
440 analogous to that of ordinary dislocations in the  $\gamma$  TiAl phase [39-41], in which the  
441 pinning was attributed to small chemical clusters [42], but also to the case of  
442 precipitation hardened Al base alloys [43], in which dislocations anchor on precipitates  
443 made of crystallographic phases different from that of the matrix.
- 444 • Loops formation leading to dislocation multiplication processes are often observed.  
445 These mechanisms are frequently evidenced when extrinsically pinned dislocations are  
446 subjected to friction forces: their screw character induced by the Peierls stress allows  
447 intense cross-slip events, in a similar manner as ordinary dislocations in TiAl [39-41].

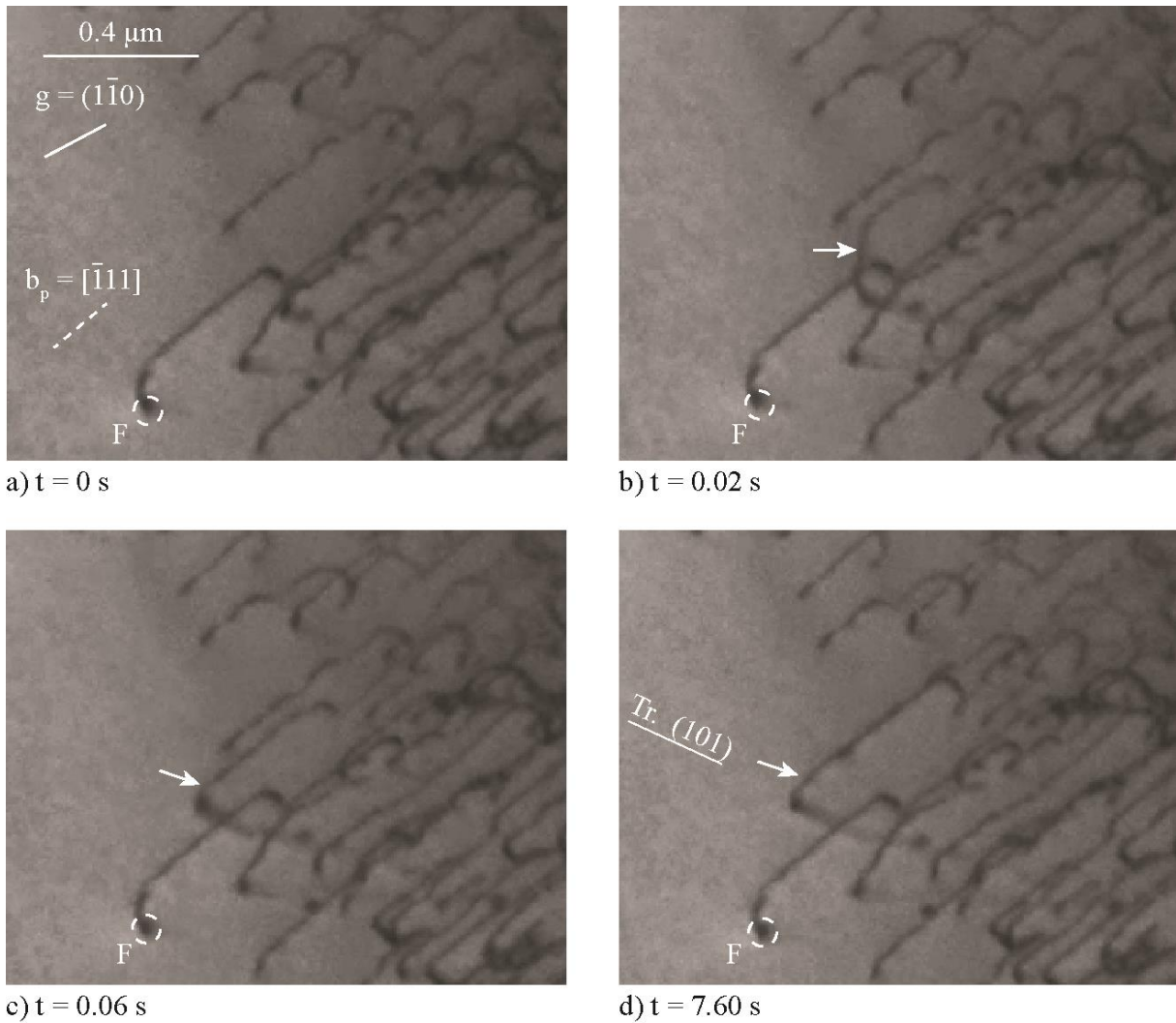
448 Regarding the  $\beta_0$  phase present in the investigated TNM-TiAl alloy,  $a\langle 111 \rangle$  superdislocations  
449 experience a friction force and must overcome the  $\omega_0$  precipitates in order to continue gliding,  
450 precisely in the  $\{110\}$  planes, the densest planes of the B2 structure. The tendency for these  
451 dislocations to align along their screw direction is less marked than in other intermetallic alloys,  
452 especially by considering whole dislocations (Figs. 8 d-f). Sometimes, this alignment seems to



453 be more pronounced for partial dislocations (Figs. 8 a-c). On the other hand, the anchoring  
454 points are very close with a separation distance of the order of 35 nm smaller than that measured  
455 for ordinary dislocations in the TiAl  $\gamma$ -phase (150 nm [41, 42]). On the reverse, this distance is  
456 close to that separating the precipitates of Al alloys (30 nm) [43], in which the structural  
457 hardening due to these precipitates is known to control deformation. This suggests that  $\omega_0$   
458 precipitates have a dominant role here on the dislocations mobility.

459 Furthermore, the trend of having dislocations moving in pile-ups with a tendency for  
460 decorrelation of the superpartials, provides a strong evidence ev that superdislocations are far  
461 from the pile-up tip, as been observed during in-situ straining experiments. It is tempting to  
462 conclude that this pile-up effect results from the structure of the  $\omega_0$  precipitates which are  
463 crossed by dislocations. An analogous situation is found in the short-range order effect detected  
464 in the  $\gamma$ -phase of Ni base superalloys [44], where the formation of pile-ups results from the  
465 greater facility of dislocations to move in the wake of the head dislocations once the ordered  
466 structure has been sheared. To check the key role of  $\omega_0$  precipitates on the formation of  
467 dislocations pile-ups , a specific TEM in-situ RT experiment was carried out in  $\beta_0$  grains of a  
468 Ti-44Al-3Mo alloy (at. %) characterized by the absence of  $\omega_0$  precipitates due to the absence  
469 of Nb. As shown in Fig. 10 (video [here](#)), dislocations observed in the starting situation (Fig.  
470 10a) have been progressively emitted in the  $\beta_0$  grain from a grain boundary situated out of the  
471 frame on the right side of the picture. Dislocations are spread all over the area. The extremity  
472 of a dislocation marked by the letter F which will not move anymore is used as a reference point  
473 in the next three images. An indexation of these dislocations allowed us to determine that their  
474 Burgers vector is parallel to the  $\langle 111 \rangle$  crystallographic direction and to deduce that these  
475 dislocations have a marked screw character. A TEM analysis under weak-beam conditions,  
476 which is not reproduced here, showed that these dislocations are dissociated into two partials  
477 separated by an APB ribbon of the order of 7 nm on screw segments. Regarding the dynamics

478 of these dislocations, Figs. 10b-d allow to follow the path of a dislocation indicated by an arrow:  
479 this dislocation occurs in the observed area (Fig. 10b) and moves individually to position  
480 indicated in Fig. 10.c, until reaching the position indicated in Fig. 10.d. The trace left on the  
481 surface corresponds to a (101) gliding plane. Dislocations activated in the  $\beta_0$  phase of this  
482 material with no  $\omega_0$  precipitates are therefore similar to the dislocations investigated in the  $\beta_0$   
483 phase of the TNM alloys with  $\omega_0$  precipitates, but keeping in mind that they move individually  
484 without forming pile-ups. It is worth noting that even if it would be interesting to carry out  
485 mechanical tests to determine the mechanical impact of the absence of  $\omega_0$  precipitates, we did  
486 not do so: a fair comparison with the TNM alloys would indeed require the manufacturing of a  
487 material with a similar microstructure. This is really a long and difficult task including  
488 atomization of the right pre-alloyed powder, SPS densification etc, that is out of the scope of  
489 this work.



490

491 **Fig. 10.** Propagation of  $a\langle 111 \rangle$  superdislocations in a  $\beta_0$  grain with no  $\omega_0$  precipitates during  
 492 an in-situ RT straining experiment in a TEM; (a) starting situation with the extremity of  
 493 dislocation used as a reference point; (b-d) arriving and propagation of a dislocation  
 494 (arrowed).

495 In summary, the movement of  $a\langle 111 \rangle$  dislocations and the formation of corresponding pile-  
 496 ups appear to result from the presence of  $\omega_0$  nanoprecipitates that seem to play a key role in the  
 497 dynamics of the dislocations involved. It should be noted that further information is needed to  
 498 fully understand the glide mechanism of these dislocations. Work is currently in progress to  
 499 obtain quantitative results on that particular topic.

500 This experimental study did not reveal any particularly brittle behaviour of the  $\beta_0$  phase since  
501 we did observe deformation dislocations and no brittle rupture surface. On the other hand, we  
502 evidenced a strong tendency to form pile-ups of large numbers of dislocations. Even though  
503 pile-ups are enhanced by in-situ experiments, however, it is worthwhile pointing out that a pile-  
504 up of the same type has been found in a post-mortem sample and pile-ups made of few  
505 dislocations, probably embryos of large pile-ups, were also evidenced. As failure is linked to a  
506 localised event that does not necessarily need to occur many times in the material, it can be  
507 assumed that the occurrence of only a few pile-ups in the bulk material (or even just one if it is  
508 well oriented with respect to the neighbouring lamellar colony) is sufficient to induce failure of  
509 the TiAl alloy. These pile-ups contain many dislocations, involving high stress concentrations  
510 that can lead to the cleavage along a lamellar interface if the lamellar colony is well oriented.  
511 Consistently with the limited slip activity of the  $\alpha_2$  phase [5, 6] and with the observation of  
512 intensive cleavage in lamellar interfaces, the low ductility of the present alloy is thus attributed  
513 to a stress mediated cleavage process along lamellar interfaces. As [Tables 1 and 2](#) show, beyond  
514 a few microns (around 20  $\mu\text{m}$ ) the size of the lamellar colonies has no additional detrimental  
515 effect on fracture ( $A= 0.5 \%$ ) because the surface of the cleaved interface certainly exceeds the  
516 critical size for an irreversible failure. Similarly, the proportion of  $\beta_0$  phase has little influence  
517 since the fracture results from a localised process in the material and not from the average  
518 deformation of the  $\beta_0$  phase. However, only a certain volume fraction of  $\beta_0$  phase is required to  
519 make the stress concentration possible in some sites.

520

## 521 **8. Conclusion**

522 The deformation microstructure and the fracture surface have been studied in a TNM-TiAl alloy  
523 ( $\text{Ti}_{51.05}\text{Al}_{43.9}\text{Nb}_4\text{Mo}_{0.95}\text{B}_{0.1}$ ) densified by Spark Plasma Sintering. RT tensile tests were coupled

524 with TEM in-situ RT straining experiments. A cleavage mechanism was observed in the  
525 lamellar interfaces of the  $\gamma/\alpha_2$  colonies but not in the  $\beta_0$  grains. The latter were found to be  
526 plastically deformable by  $a\langle 111 \rangle$  superlattice dislocations, which are dissociated into two  
527 superpartials separated by an antiphase boundary. These dislocations mainly glide in pile-ups  
528 lying in  $\{110\}$  planes and involving many dislocations. In addition, the  $\beta_0$  phase contains  $\omega_0$   
529 nanoprecipitates, which anchor the dislocations, thus limiting their propagation and enhancing  
530 pile-ups configurations.

531 In summary, this study shows that fracture of  $\beta_0$ -containing TiAl alloys occurs by cleavage  
532 along the interface plane of the lamellar  $\gamma/\alpha_2$  colonies, in a similar manner as in lamellar TiAl  
533 alloys with no  $\beta_0$  phase. However, this cleavage mechanism is premature since  $\omega_0$  precipitates  
534 that are present in the  $\beta_0$  phase, as a result of Nb addition in the TNM alloy, promote stress  
535 concentrations by the formation of dislocation pile-ups in this phase. Therefore, such  
536 dislocation pile-ups are believed to trigger cleavage in the neighbouring lamellar colonies.

537

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544

545

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