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Effects of SMAT at cryogenic and room temperatures on the kink band and martensite formations with associated fatigue resistance in a β -metastable titanium alloy

Pierre Maurel^{a,b,c,*}, Laurent Weiss^a, Philippe Bocher^{b,**}, Thierry Grosdidier^{a,c,**}

^aUniversité de Lorraine, Laboratoire LEM 3, UMR CNRS 7239, 7 Rue Félix Savart, 57073 Metz, France ^bLaboratoire d'Optimisation des Procédés de Fabrication Avancés (LOPFA), École de Technologie Supérieure, 1100 rue Notre-Dame Ouest, Montréal, Canada

Abstract

Surface Mechanical Attrition Treatment (SMAT) has been carried out at cryogenic and room temperatures on a 5553 β -metastable Ti alloy to describe its effects on the modification of the microstructure and fatigue properties. The cryogenic temperature promoted the martensitic transformation and the width of the kink bands produced by SMAT. The kink bands formation was promoted by the high strain rates of the SMAT process. The samples SMATed at cryogenic temperature showed an increase in fatigue resistance, by about 8%, compared to samples processed by SMAT at room temperature. This improvement was possibly due to a combination of several factors: compressive residual stresses, lower roughness, deeper formation of martensite and larger kink bands. On the contrary, the SMAT at room temperature did not provide any improvement in terms of fatigue compared to the polished condition. The large grain size provoked the appearance of subsurface crystallographic defects on which primary cracks initiated. This greatly limited the effectiveness of the reinforced surface layer produced by SMAT.

Keywords: Severe Plastic Deformation (SPD), Beta Titanium alloy, High Cycle Fatigue, Surface Mechanical Attrition Treatment (SMAT), Kink band, Cryogenic treatment

^cLABoratoire d'EXcellence "Design of Alloy Metals for low-mAss Structures" (LABEX-DAMAS), 7 rue Félix Savart, 57073 Metz, France

^{*}Corresponding author.

^{**}Secondary corresponding authors. Email addresses: pierre.maurel@univ-lorraine.fr (Pierre Maurel), philippe.bocher@etsmtl.ca
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Highlights

- Kink bands generated by the high strain rates induced by SMAT
- A lower kink band density in the first μ m of SMATed surfaces
- Deeper martensitic formation and lower roughness for cryogenic SMAT
- Increased fatigue resistance for SMAT at cryogenic temperature

1 1. Introduction

The development of Severe Plastic Deformation (SPD) allowed the processing of mate-2 rials with highly enhanced properties. Several reviews of the different processes highlighted 3 the links between the microstructure refinement, high dislocation densities and significant 4 increases of the strength and hardness [1–3]. Unfortunately, the resulting ductility drop is 5 the main drawback of those property improvements. Most SPD techniques need immense 6 loads on small sized samples with basic geometries, such as the High-Pressure Torsion [4] or 7 the Equal Channel Angular Pressure [5]. This remains a significant limitation for up-scaling 8 those processes to industrial applications. 9

As the external surface is usually the most demanding location in industrial parts due 10 to high loads, corrosion, or friction, surface SPD processes were developed to promote the 11 surface resistances in some applications. To this end, examples such as Burnishing [6], Laser 12 Shock Peening [7], Ultrasonic Shot Peening (USP or USSP) [8] or Surface Mechanical At-13 trition Treatment (SMAT) [9–13] were developed. SMAT and USSP processes deform the 14 surface by propelling a media on it (mainly metallic balls) and were developed from pre-15 strain shot-peening [10, 12, 14]. A long enough treatment also produces a significant grain 16 refinement and surface property gradients. These surface SPD processes are well known 17 for modifying wear resistance [15, 16], corrosion resistance [17, 18] and for introducing im-18 portant compressive residual stresses. These high compressive residual stresses and finer 19

⁽Philippe Bocher), thierry.grosdidier@univ-lorraine.fr (Thierry Grosdidier)

microstructures at the surface are known to retard fatigue crack propagation and thus increase the fatigue resistance in aluminium alloys [14]. However, the roughness induced by surface nanostructuring may negatively impact the fatigue resistance, as shown on superalloys, stainless steels and aluminium [13, 19, 20].

In metastable austenitic stainless steels, in which the stress-assisted or strain-induced 24 martensitic transformations could be triggered, performing SMAT at cryogenic temperature 25 could be used to increase the fraction of hardening martensite towards higher depth as 26 well as to modify the interplay between slip activity and martensite / twining formation 27 [21, 22]. The deformation gradient formed by the surface modification generally produces 28 gradients of different natures such as grain size, hardness, martensitic [22] and/or twinning 29 [23] fractions. If the material is harder to deform at cryogenic temperatures, a significant 30 decrease of the apparent surface roughness can be obtained [22]. Thus, an appropriate 31 selection of processing parameters allowed to tailor the surface towards a broad range of 32 hardness and roughness values [22]. 33

The β -metastable Ti alloys are prone to the formation of stress assisted or strain induced 34 martensites as well as mechanical twinning which can both compete with conventional plas-35 ticity mediated by dislocations. The occurrence of these deformation mechanisms depends 36 on the stability of the β phase which, itself, is affected by its chemical composition [24, 25]. 37 the size of the β grains [26, 27], as well as the possible presence of a secondary phase [28, 29]. 38 These mechanisms can be activated in various Ti alloys thereby exhibiting shape memory 39 effect [30, 31], superelasticity [31–36] or strain-hardening behavior [3, 4, 37, 38] and affecting 40 their tensile properties [28, 39, 40]. Superelasticity, due to the formation of stress-assisted 41 martensite, significantly improves the low-cycle fatigue behavior in these alloys due to the 42 reversible phase transformation which suppressed microplastic deformations [35]. The high-43 cycle fatigue domain would be less sensitive to this mechanism as the lower stress involved 44 may not trigger the phase transformation. Also, the martensitic transformation may en-45 hance the grain refinement obtained by severe plastic deformation [41]; this in turns may 46 improve the high-cycle fatigue behavior. In β -metastable titanium alloys with a complete β 47 microstructure, few studies revealed the possibility to activate a different deformation mech-48

anism such as the formation of kink bands [42–47]. Although common to materials with 49 high anisotropy [48, 49], kink bands rarely appear in body-centered cubic structures. Zheng 50 et al. described in detail the formation of those deformation bands [43]. They are due to the 51 accumulation of edge dislocations resulting in bands with random misorientations ranging 52 mostly between 10° and 25° compared to the initial orientation. Three kink slip systems 53 may be activated: $(112)[11\overline{1}], (123)[11\overline{1}], (101)[1\overline{11}]$. They induce a rotation of the crystal 54 around three rotation axes, also called Taylor axes: $[1\overline{10}], [541], [12\overline{1}],$ respectively. As the 55 kink bands are relatively thin, this mechanism effectively refines the microstructure by the 56 introduction of a high density of high-angle boundaries. It has been confirmed that this 57 deformation mechanism depends on the alloy composition; as Sadeghpour et al. used the 58 same deformation process on four different β -metastable titanium alloys and obtained kink 59 bands in only one of these alloys [50]. Surprisingly, for Sadeghpour et al., one of the alloys 60 that did not produce any kink band was the 5553 while Qin et al. showed the possibility to 61 form kink bands in this 5553 alloy [45]. 62

According to the previously described information, three main goals were set for this 63 paper. First, exploring the possibility to trigger or not kink band formation using the 64 SMAT process in the β -metastable 5553 titanium alloy. Indeed, kink bands were triggered 65 in this same alloy by Split-Hopkinson bars [45] but not by shear punching [51]. Second, 66 comparing the surface effects of SMAT at room and at cryogenic temperatures in terms of 67 material properties and especially crystallographic transformations (martensitic and/or kink 68 formations). Finally, discriminating any possible impact on fatigue resistance of the SMAT 69 process. 70

71 2. Experimental Procedures

72 2.1. Material and surface treatments

⁷³ The 5553 β -metastable titanium alloy studied in this paper was provided by Timet Savoie ⁷⁴ (France) as a slice of a forged billet. Its chemical composition was [wt%]: Mo 4.84, V 4.93, Al ⁷⁵ 5.29, Cr 3.04, Fe 0.3, O 0.14, C 0.008, N 0.004, Ti (balance). The alloy was solution-treated ⁷⁶ at 925°C for 1 h in a vacuum furnace and water-quenched. This treatment allowed the alloy ⁷⁷ to retain a complete beta microstructure with an average grain size of about 260 μ m.

Fig. 1 gives a sketch of the Surface Mechanical Attrition Treatment (SMAT) device used 78 to treat the R. R. Moore fatigue samples (red) [52]. Two millimeters in diameter AISI 79 52100 steel shots were confined within the treatment chamber (green) and moved using an 80 ultrasonic device (blue) vibrating at a frequency of 20 kHz with a 60 μ m amplitude. When 81 the vibrating device (sonotrode developed by SONATS [53]) was unused, the shots covered 82 roughly 20% of the vibrating surface. The central section surface of the fatigue samples was 83 set at a distance of 20 mm from the sonotrode. The SMAT duration for the treatment of the 84 cylindrical samples was set to 40 min while the samples were rotating at a constant speed 85 of 20 rpm. Copper heat sinks (in orange in Fig. 1) partly immersed in liquid nitrogen were 86 fixed on both sample heads to maintain the samples at cryogenic temperature during the 87 cryogenic treatments. 88

89

Three treatment conditions were studied and provided the following samples:

- Polished samples (P): these samples were machined to the dimensions, then heattreated and finally polished with SiC papers from 500 to 4000 grit.
- SMATed samples at Room Temperature (SMAT-RT): these samples were prepared in the same way as P samples and were SMATed at room temperature.
- SMATed samples at Cryogenic Temperature (SMAT-CT): these samples were prepared
 like the P samples and were SMATed at cryogenic temperature.

Table 1 gives the different treatment conditions with their corresponding temperature variations during SMAT as well as the resulting surface roughness. The temperature was probed with thermocouples placed in holes drilled from one sample head to the central section along the longitudinal axis of some samples that were used only for that matter. For SMAT at room temperature, the sample core temperature rose from 20°C to about 80°C, due to the heat generated by the shot impacts, before reaching a steady state after about 2 min. The samples treated at cryogenic temperature were first cooled down to -170°C with liquid nitrogen and were subsequently SMATed. Their core temperature increased by
roughly the same amount to about -100°C before stabilizing after less than 4 min.

¹⁰⁵ 2.2. Microstructure and surface analyses

The linear surface roughness was measured along the longitudinal axis on the central section with an average of 6 measurements per sample made with a stylus surface profilometer which had a standard radius of 5 μ m.

Microstructural observations were made on mirror-polished cross-section surfaces cut 109 perpendicularly to the longitudinal axis of the samples. Scanning Electron Microscopy 110 (SEM) images were acquired with an acceleration voltage of 20 kV and an aperture size of 90 111 μm with a Supra 40 apparatus. The working distance was either 18 mm on fracture surfaces 112 or 10 mm for other SEM observations. A Symmetry detector and the Aztec software from 113 Oxford Instruments were used for the Electron BackScattered Diffraction (EBSD) mappings. 114 EBSD mappings were acquired with an acceleration voltage of 10 kV for a working distance 115 of 15 mm, a scanning step size of 300 nm and an aperture size of 60 μ m. 116

The gradient of the phases present in the surface and subsurface microstructures was 117 measured using $\theta - 2\theta$ X-Ray Diffraction profiles acquired during 10 min with a 2D detector 118 on a Bruker D8 apparatus. A Co K_{α} source was used with a 40 kV tension and a 30 mA 119 current. It should be noted that the gradients obtained from these measurements are only 120 qualitative since the present Co K_{α} radiation had a penetration depth of about 30 μm 121 in titanium [54]. As such, the XRD profiles measured are significantly affected by the 122 phases within the first 30 μ m below the measured surface. The profiles were obtained 123 after successive electro-polishing using an electrolytic solution composed of: 6% acid, 35%124 butoxyethanol and 59% methanol, and a tension of 24 V. A silicone mask was used to select 125 and limit the electropolished surface of the sample. A numerical micrometer was used to 126 measure the removed depth after each polishing step. The cylindrical samples were rotated 127 at 20 rpm along the longitudinal direction to increase the number of diffracting grains 128 during the XRD profile measurement. The background noise of the diffraction spectra was 129 approximated by a 6^{th} degree polynomial expression and subtracted to the measured XRD 130

profiles. To emerge from the background noise, the volume fraction of a given phase needs
to be higher than 5%.

¹³³ Vickers microhardness profiles were acquired with a 25 g load on the cross-sections ob-¹³⁴ served by SEM. The measurements started at 30 μ m below the surface and continued every ¹³⁵ 30 μ m toward the core. The profiles were obtained from an average of 10 measurements.

The residual stress profiles were acquired with a μ -X360s Pulstec apparatus using a 136 vanadium source and the $\cos \alpha$ method [55]. The residual stresses profiles were made after 137 successive electro-polishing on fatigue samples - similarly to the $\theta - 2\theta$ XRD profiles - along 138 the longitudinal axis. In order to significantly increase the number of diffracting grains and 139 improve the reliability of the measurements, the samples were rotated at a speed of 20 rpm 140 for the residual stresses measurements which lasted 1 min. The effects of surface material 141 removal on the residual stresses were corrected according to Moore et al. as the geometry of 142 the pocket allows the proposed simplifications [56]. 143

144 2.3. Fatigue and failure analyses

Four points rotative bending fatigue tests were conducted with an R. R. Moore test 145 apparatus at a rotating frequency of 30 Hz (R = -1). The samples were loaded to given 146 stress levels for an intended fatigue life below 10^6 cycles. Due to the limited number of 147 samples for each condition, a staircase method was used. Run-out samples were tested 148 again with an increased load by increments of 20 MPa. The result of the new test was 149 considered valid only if failure occurred after 100 000 cycles. The run-outs with the highest 150 value are also plotted on the fatigue graph and retested samples are identified by an R letter 151 at the bottom right of the corresponding symbol using the same color. 152

After fracture, tomography measurements were carried out on the regions located below the fracture surfaces with a XT H 225 apparatus from Nikon Metrology in order to detect large subsurface fractures in these regions and to determine their shapes. The acquisition was made with a tension of 210 kV, an intensity of 43 μ A and an exposure time of 1 s per projection for a total of 2634 projections. The apparatus had a resolution of 3.96 μ m in this configuration. The acquired data were processed with the Dragonfly software.

159 3. Results

This section is divided into four different subsections dealing with the deformed microstructure, the surface properties, the fatigue properties and, lastly, analyses of the fracture surfaces.

¹⁶³ 3.1. Deformed Structure Analyses

SEM observations allow to show the initial microstructure of the material and the subse-164 quent effects of the two types of SMAT processing. Fig. 2a is a SEM micrograph of the core 165 microstructure at a low magnification and reveals large β grains of about 260 μ m. Figs. 2b 166 and c present band contrast images obtained from EBSD mappings along the cross-section 167 at the surface of the SMAT-RT and SMAT-CT samples, respectively. The band contrast 168 displays the quality of the indexation of EBSD patterns and it is represented by shades of 169 grey, with white being the best and black showing points where the indexation failed. The 170 indexation can be improved by better regularity of the crystallographic cell (less structural 171 defects like dislocations, for example). Deformation bands of different widths and oriented 172 along different directions were formed. For both SMATed samples, these deformation bands 173 are present from the surface down to a depth of about 200 μ m but they are mainly present 174 within the first 100 μ m. The SMAT-RT samples (Fig. 2b) show a narrow range of band 175 widths and a homogeneous distribution around an average width. The SMAT-CT samples 176 (Fig. 2c) display a larger range of deformation band widths. Some are extremely thick while 177 others are significantly thinner, and the band widths are not homogeneously distributed over 178 this range. Instead, both extremes of this range are considerably more common than the 179 rest. Surprisingly, both SMAT-RT and SMAT-CT samples band contrast mappings show 180 a microstructure at the top surface with a better indexation rate and a lower deformation 181 band density compared to the microstructure deeper in the material (Fig. 2b and c). This 182 effect is particularly visible for SMAT-CT samples as the deformation bands are less dense 183 at the top surface. The deformation at the extreme surface was not significant enough to 184 generate a nanostructure which allowed such a high indexation rate. 185

In order to understand the nature of these deformation bands, more observations are 186 needed. Figs. 3a and b represent the inverse pole figures (IPF) maps extracted from the 187 same EBSD mappings shown in Fig. 2 for the SMAT-RT (3a) and SMAT-CT (3b) samples, 188 respectively. The indexation rate drops significantly in the bands due to the significant 189 deformation. The IPF only provides information concerning the changes in crystallographic 190 orientation for the large deformation bands as the smallest ones visible in Figs. 2b and c 191 were thinner than the 300 nm stepsize. A zoom on the region marked by a red rectangle 192 in Fig. 3a is shown in Fig. 3c. The high indexation rate due to the lower deformation at 193 this depth allows the identification of the deformation bands characteristics. The cumulative 194 misorientation variation across the deformation bands was determined for four of these bands 195 along the lines labeled A to D and was represented in Fig. 3d. The misorientations between 196 the bands and their surroundings vary between 5° and 24°. Interestingly, the A and D 197 lines cross two bands aligned in the same direction but they present different misorientation 198 variations. The D-line presents a progressive misorientation variation through the entire 199 band whereas the A-line variation is far steeper at the edges of the band while it is constant 200 at its center. The pole figures of the regions around the A and C lines are given in Fig. 3e 201 and f, respectively. Fig. 3e displays the $\{541\}$ poles while the Fig. 3f shows the $\{110\}$ poles. 202 On both figures, a rotation around one pole, indicated by the circles in dashed lines, can be 203 observed. All these characteristics indicate that these deformation bands are actually kink 204 bands as depicted in detail by Zheng et al. [43]. 205

The effects of SMAT on the martensitic transformation in the 5553 β -metastable titanium 206 alloy can be determined thanks to XRD techniques. Figs. 4a and b give XRD profiles, 207 respectively on the SMAT-RT and SMAT-CT samples, that were obtained at different depths 208 after successive electro-polishing and they permit to determine the evolution of the phases 209 presence depending on the depth. The depth of each measurement can be seen on the right 210 y-axis at the baseline level of each profile. Notable β and α " peaks are indicated. Zooms 211 on the $\beta(110)$ and $\alpha''(111)$ peak positions are displayed in Figs. 4c and d for SMAT-RT 212 and SMAT-CT, respectively. On both SMATed samples, no martensite is detected at the 213 surface. The α "(111) peak begins to appear after the first electro-polishing at a depth of 214

15 μ m for the SMAT-RT and SMAT-CT samples. Between 30 and 60 μ m, the martensite 215 peak is clearly visible for both sample conditions. For the SMAT-RT sample, the peak is 216 barely visible at 80 μ m and disappears at further depths. For the SMAT-CT sample, the 217 martensite can be detected down to 120 μ m. The appearance of only a single well-defined 218 peak prevents an accurate quantification of the martensitic phase. The other peaks are 219 covered by the background noise. However, the fact that the martensite $\alpha^{"}(111)$ peak is 220 visible means that the volume fraction of martensite should be at least about 5%. Thus, 221 the formation of martensite was promoted when the treatment was carried out at cryogenic 222 temperature and the transformation was lower at the surface compared to the subsurface. 223

224 3.2. Surface and Subsurface Properties

Since the surface integrity is of major importance in high-cycle fatigue due to the promotion of surface cracks for low integrity, determining the surface roughness is mandatory. Table 1 displays the Ra and Rz parameters of the surface roughness for polished and SMATed samples. The SMAT-RT surface samples present a roughness about 4 times higher compared to the SMAT-CT surface condition due to the increased mechanical strength of materials at cryogenic temperature leading to a lower deformation of the surface for a given imparted energy of the shots.

²³² Hardness measurements allow to determine the effectiveness of SMAT in reinforcing the ²³³ considered surfaces by severe plastic deformation. Fig. 5a gives the hardness evolutions as a ²³⁴ function of the depth from the surface in the SMAT-RT and SMAT-CT samples. The SMAT-²³⁵ RT sample shows an increase in hardness of about 15% whereas the SMAT-CT presents only ²³⁶ a 9% increase, at 30 μ m below the surface. The affected depth dropped from 200 μ m to 150 ²³⁷ μ m with the use of the cryogenic temperature.

The residual stresses significantly limit the crack propagation in the regions in compression and as such they significantly impact the fatigue behavior. Fig. 5b displays the residual stress profiles for both SMAT-RT and SMAT-CT sample conditions. Both profiles appear extremely similar in shape and intensities. The compressive residual stresses are about -400 MPa at the surface and the residual stress profiles reach a compression peak at a depth slightly over 100 μ m with a value of about -650 MPa for the SMAT-RT samples and -600 MPa for the SMAT-CT samples. The residual stresses then increase until they reach 0 MPa at a depth of about 350 μ m.

246 3.3. Fatigue Properties

The samples were submitted to a stress amplitude for an intended fatigue life between 10^5 247 and 10^6 cycles. Fig. 6 gives the results of the fatigue tests in the form of the stress amplitude 248 level as a function of the number of cycles to failure for SMAT-RT treated samples (red 249 crosses), SMAT-CT treated samples (blue diamonds) and Polished samples (black circles). 250 Arrows below the marks represent the run-out tests for which the experiments were stopped 251 at the indicated number of cycles. From these results, the Polished and SMAT-RT conditions 252 display similar fatigue resistances. The SMAT-CT condition presents an improvement in 253 the fatigue resistance by about 20 MPa (or about 8%) compared to the other conditions. 254

255 3.4. Fracture Surfaces

The observation of fracture surfaces allows to determine how cracks initiated and prop-256 agated, thus providing insights into understanding the fatigue behaviors of the studied con-257 ditions. Based on the observations made on the fracture surfaces, all samples present sub-258 surface nucleation sites and only the polished samples presented some initiation sites at the 259 surface (not documented below). Figs. 7a, b and c show SEM images of the fracture sur-260 faces, respectively for the Polished, SMAT-RT and SMAT-CT conditions. The analyses of 261 the fracture surfaces are not straightforward, as several primary cracks are found and may 262 propagate over similar distances before merging. The term "primary crack" is used in this 263 context to designates the cracks leading to failure while the term "secondary crack" refers to 26 those found either on the fracture surface or elsewhere in the sample without affecting the 265 failure mechanism. Fig. 7a displays clear pseudo-cleavage features in the region within the 266 black outline. In all conditions, such pseudo-cleavage features are recognizable on the frac-267 ture surface and help backtracking to the initial nucleation points on which they converge 268 (identified with red arrows). Higher magnification images of the identified initiation sites 269

are given in Figs. 7d, e and f, for Polished, SMAT-RT and SMAT-CT samples, respectively. 270 The primary cracks were found to nucleate on subsurface defects and they propagated with 271 a pseudo-cleavage mechanism along preferential planes in their original grain before contin-272 uing on to the next grains along other preferential planes with similar orientations. Some 273 ductile fracture features typical of overload can be observed between the pseudo-cleavage 274 regions, indicated by the full black arrows. No fatigue striations were observed. Due to 275 the interaction of different cracks, all initiation sites may not necessarily be present on the 276 fracture surfaces. Some cracks initiated below the fracture surface, indicated by the white 277 arrows. Their importance is unclear and some of them may have been the initiation location 278 of primary cracks. In Fig. 7a, the features on two large pseudo-cleavage regions, pointed 279 by yellow arrows, could not be used to backtrack to their initiation sites, but their origins 280 were clearly disconnected from the initiation site identified with the red arrow. The fact 281 that these large pseudo-cleavages have different origins shows that these primary cracks may 282 independently nucleate and propagate to an important size before merging. 283

Further observations of the complete fracture surfaces showed a change in fracture mech-284 anisms at the surface of SMATed samples. Fig. 8a displays the complete fracture surface 285 of the SMAT-CT sample presented partially in Fig. 7c. The primary crack initiation site 286 indicated in Fig. 7c is marked with red arrows in Figs. 8a and b. This global image confirms 287 that primary cracks have propagated along preferential planes through successive grains, as 288 described in Fig. 7. A higher magnification image of the primary crack initiation site in 289 Fig. 8b displays a 250 μ m large band along the surface that present rough aspects typical 290 of regions formed during the final rupture. The main crack did propagate preferentially to-291 ward the sample center, and only later propagated through this band. Similar observations 292 were made on SMAT-RT samples. A close-up observation of this band (Fig. 8c) shows an 293 emerging crack at the boundary between the band and the main primary crack, indicated 294 by the white arrow. 295

The tomography technique was used to eventually detect in-depth features without having to cut or alter the samples. Due to the resolution limitation, only large cracks can be identified with this method, but Figs. 8d to g show that tomography analyses can reveal ²⁹⁹ the presence of the emerging crack between the band and the primary crack (Fig. 8c). The ³⁰⁰ opening of the crack was magnified for the sake of visibility. Figs. 8e to g show the same ³⁰¹ region in transparency and display a crack network where an enclosed large secondary crack ³⁰² can be found. This crack initiated at a depth of about 250 μ m and propagated roughly ³⁰³ parallel to the surface main fracture as highlighted in red in Fig. 8f. A bridging that may ³⁰⁴ have appeared during the final fracture (highlighted in white in Fig. 8f) joins this secondary ³⁰⁵ crack to the fracture surface.

306 4. Discussion

The results, previously reported for the β -metastable titanium alloy, have shown the effects of the SMAT processing conditions and, in particular, the effect of the SMAT temperature. The effects of SMAT on (i) the microstructure and on (ii) the fatigue properties will be discussed successively in the following sections.

311 4.1. Deformed microstructure

The present results point out that the formation of kink bands is the main deformation 312 mechanism for the SMATed 5553 titanium alloy under the present SMAT loading conditions. 313 The martensitic transformation was concomitant with the kink band formation, as this phase 314 appears within these bands. The presence of kink bands is consistent with observations 315 done in few studies conducted on titanium alloys deformed by a split-Hopkinson pressure 316 bar system (strain rate of $5 \times 10^3 \ s^{-1}$). Zheng et al. focused on the Ti-35V-15Cr-0.3Si-0.1C 317 titanium alloy [43] and Qin et al. worked on the 5553 alloy [45]. The appearance of kink 318 bands in the 5553 alloy after SMAT in the present study, but not after shear punching at 319 strain rate up to 200 s^{-1} in [51] nor in compression tests at strain rates of $10^{-3} s^{-1}$ in [36] 320 and $0.7 \times 10^{-3} s^{-1}$ in [50], tends to support the hypothesis that their formation is promoted 321 at very high strain rates. A strain rate above 200 s^{-1} seems necessary for their formation 322 in the 5553 titanium alloy. According to the calculation of Chan et al. [57] on steel, typical 323 SMAT strain rate can be above $10^3 s^{-1}$ within the first 300 μ m from the surface. However, 324 the local deformation conditions under SMAT did not induce the appearance of adiabatic 325

shear bands; as noted in the case of split-Hopkinson pressure bar system for similar strain 326 rates [43, 45]. The progressive misorientation seen in the pole figure in Fig. 3e by a gradual 327 rotation of the {541} poles around the circle in dashed red lines illustrates what Zheng 328 et al. have proposed as a mechanism for kink bands formation in β titanium alloys [43]. 329 The kink bands are formed by the migration of edge dislocations in dislocation walls along 330 the slip modes: $\{112\}\langle 111\rangle$, $\{123\}\langle 111\rangle$, $\{101\}\langle 111\rangle$. They show a rotation of the crystal 331 respectively around the three rotation axes, also called Taylor axes: $\langle 110 \rangle, \langle 541 \rangle, \langle 121 \rangle$. The 332 difference in kink band misorientations measured along the A line (A kink band) and D 333 line (D kink band) in Fig. 3d also highlights the hypothesis that kink bands are formed 334 progressively by an accumulation of dislocations in dislocation walls. The D kink band 335 had just begun its formation with a misorientation of 7° while the A kink band is already 336 well formed with a misorientation of 24°. Interestingly, the kink band that is still forming 337 (D) presents a gradual variation of misorientation, compared to the sharp variation of the 338 well-formed kink bands (A). 330

The fact that the density of kink bands is higher in the subsurface compared to the surface 340 (Figs. 2b and c) can be explained by complementary mechanisms. Firstly, an important 341 part of the dislocations formed close to the surface have the possibility to be annihilated 342 by migrating and reaching the surface; comparatively, the dislocations formed deeper in 343 the subsurface block and interact with each other in order to minimize the stored energy. 344 Secondly, a higher dislocation density is formed in the subsurface due to the location of 345 the higher amplitude of the Hertz stress during impact tests [22, 58]. For a given impact 346 energy of the SMAT, the SMAT-CT condition appears to result in a lower density of large-347 sized kink bands compared to the SMAT-RT condition. The increase in yield strength 348 at cryogenic temperature, resulting from a decreased mobility of dislocations, reduces the 349 number of dislocations formed after each impact. The distance between the dislocations 350 should allow them to move without immediately interacting and pinning each other down in 351 dense dislocation walls or dislocation entanglements, as would be the case for the SMAT at 352 room temperature. Thus, the mean free path of dislocations should be higher for the SMAT 353 at cryogenic temperature. The dislocations more easily migrate to form fewer but larger 354

kink bands, albeit at a slow velocity due to the low temperature, increasing their size and
resulting in the appearance of the largest kink bands (Fig. 2c). The multitude of thinner
kink bands formed for SMAT-RT is a testimony of the low mean free path of dislocations
at this temperature (Fig. 2b).

SMAT allowed the formation of martensite only within the kink bands in the subsurface. 359 While no martensite was detected at the surface for both SMAT-RT and SMAT-CT, it was 360 visible on the XRD profiles deeper in the subsurface suggesting that the volume fraction of 361 martensite was higher than 5% in the subsurface. The cryogenic temperature improved the 362 ability to trigger the martensitic transformation as lower temperature result in the presence 363 of martensite deeper in the subsurface. This easier martensitic transformation within the 364 kink bands may interact with the dislocation walls at the interface between the kink bands 365 and their surroundings, forcing them apart and expanding them beyond the size they would 366 reach otherwise. This hypothesis could potentially explain why the thickest kink bands were 367 obtained for SMAT-CT samples. However, the ability of the martensitic transformation to 368 assist the formation of kink bands remains to be understood. 369

Under the present processing parameters, no ultrafine grains were observed at the top 370 surface after SMAT at both RT and CT showing that the processing condition was mild. 371 Instead, the microstructure at the top surface was fragmented only by the kink bands. 372 We have found that the fragmentation process continues and results in the formation of a 373 nanostructure when the SMAT duration increased to several hours, the SEM images used for 374 this observation are not given here as the relevance to add them as a new figure is minimal. 375 However, the SMAT duration that was already 40 min long in the present case should be 376 increased to several hours to generate similar nanostructures, which may not be practical for 377 industrial applications as it would add high manufacturing costs (low production rate and 378 rapid wear of SMAT equipment). Besides, in the present study, the goal was to investigate 379 the effects of the SMAT temperatures on the fatigue resistance, and not the effects of the 380 SMAT duration. Indeed, it has been demonstrated that an increased SMAT duration leads to 381 a loss of surface integrity, due to the alteration generated by the intensity of the deformation 382 treatment; and negative impact on the fatigue resistance was observed for long treatments 383

in superalloys, stainless steels and aluminium alloys [13, 19, 20].

It is relevant to compare the effects of SMAT at cryogenic temperature on the present 385 β -metastable Ti alloy to those reported in the case of austenitic stainless steels [21, 22]. 386 In the work done by Novelli et al. as well as in the present work, the SMAT-CT seems 387 to significantly promote the martensitic transformation as less stress and deformation are 388 necessary to trigger the transformation at the low temperature. Both works also show that 389 SMAT-CT allows to significantly reduce the roughness due to the increase in mechanical 390 strength of materials at cryogenic temperature which induces a lower surface deformation 391 for the same energy provided by the shots. However, the present SMAT-CT condition on the 392 5553 alloy displays less surface hardening compared to the SMAT-RT condition, providing 393 quite different results from these reported by Novelli et al. [21, 22]. Indeed, Novelli et 394 al. showed that using SMAT at CT could significantly increase the subsurface hardness 395 over a larger depth range compared to SMAT at RT thanks to the promoted formation 396 of hard martensite with a significant volume change during the transformation. The low 397 volume change during the α " martensitic transformation in titanium limit the martensitic 398 hardening in these alloys. 399

400 4.2. Impact on fatigue

The fatigue behavior of the present 5553 alloy is extremely dependent on crystallographic features, both in the initiation of fatigue cracks and in their propagation.

The fracture surfaces are rather difficult to read and interpret (Fig. 7), but according to 403 the observations gathered on all broken samples, a primary crack appeared in a subsurface 404 grain and started to propagate through the grain along a preferential plane. As the primary 405 crack reached the grain boundary, it spread through the next grain along another preferential 406 plane with similar orientation. Additionally, the crack reduced the local load-carrying capa-407 bility and thus increased the stress concentration of the neighboring region. Several cracks 408 nucleated and propagated according to the same pattern and once the load-carrying capa-409 bility reached a critical level, secondary cracks started to bridge the gaps between already 410 fractured regions, leading to the failure. 411

The initiations mainly started at subsurface defects, such as triple junctions, for all 412 sample conditions, i.e. polished and SMATed samples. For the polished samples, the low 413 surface roughness and early surface plasticity were apparently less detrimental than some 414 subsurface defects as main initiations are located in the subsurface. For SMATed surfaces, 415 the initiations appeared at a depth over 250 μ m from the surface which corresponds to 416 the depth where the compressive residual stresses contribution is relatively low. For the 417 SMATed samples, cracks only propagated toward the surface during the final stage of the 418 crack propagation as they have to go through the region with high compressive residual 419 stresses. This may result in the increase in the number of cycles to failure. 420

Despite SMAT producing an increased surface hardness and important compressive resid-421 ual stresses, the SMAT-RT samples had similar fatigue resistances to that of Polished ones. 422 This is surprising since the cracks on Polished samples could easily reach the surface and 423 propagate more rapidly to a critical size. The propagation for the SMAT-RT was prevented 424 in the 250 μ m layer from the surface during the initial propagation stage, as such the crack 425 could only grow to the critical size by propagating toward the center of the samples, where 426 the stress amplitude is lower. From these observations, the SMAT-RT should have a fatigue 427 resistance noticeably higher than that of the Polished samples. Maybe the high surface 428 roughness of SMAT-RT samples acted as stress concentrators and helped the crack in prop-429 agating through the hardened layer in compression, thus reducing the critical crack size 430 needed before the final fracture. This hypothesis may also explain why SMAT-CT condition 431 produced an 8% increase in fatigue resistance as the surface roughness was significantly low-432 ered, even if these samples also presented a lower hardness and a lower kink band density 433 compared to SMAT-RT samples. Another hypothesis that may explain the better fatigue 434 resistance of SMAT-CT samples is the presence of martensite deeper in the subsurface and 435 of the largest kink bands as both characteristics may also hinder the crack propagation 436 through the deformed layer. 437

438 5. Conclusions

The main goal of this study was to investigate the effects of Surface Mechanical Attrition Treatment (SMAT), in particular at cryogenic temperature, on a β -metastable 5553 titanium alloy and the impact on its fatigue properties. The main results can be summarized as follows:

- Kink bands were formed after SMAT in the 5553 β -metastable titanium alloy at its full β state due to the local high strain rates induced by SMAT. The random impact directions, inherent in the SMAT process, allowed the activation of different kink band systems. The kink band density was the highest in the subsurface for both SMAT at room (RT) and at cryogenic temperature (CT).
- 448

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• The formation of martensite was observed within the kink bands for all SMAT conditions due to the concentration of the deformation in these regions.

- SMAT at cryogenic temperature allowed the formation of martensite deeper in the subsurface compared to SMAT done at room temperature. Contrary to what was observed in some austenitic stainless steels [21, 22], this higher amount of martensite was not associated with an increase in subsurface hardness. On the contrary, the SMAT-CT samples were found softer than the SMAT-RT ones when subsequently tested by micro-hardness at room temperature.
- The roughness after SMAT was reduced by a factor of 4 when the treatment was
 done at cryogenic temperature compared to the treatment done at room temperature
 due to the increased mechanical strength of the alloy at lowest temperature of SMAT
 processing.
- SMAT-CT resulted in an improvement of the fatigue resistance by 8% compared to
 the SMAT-RT. Two hypotheses may explain this difference: (i) the deeper martensite
 and the largest kink bands formed by SMAT-CT may have delayed the final fracture;
 and (ii) the high roughness produced by SMAT-RT may have introduced stress raisers

464 at the surface. As these two hypotheses are not in contradiction with each other, a 465 combination of both may be possible.

• Surprisingly, SMAT-RT and polished samples both had similar fatigue resistance. The 466 core microstructure in the investigated condition of the 5553 alloy presented significant 467 crystallographic defects due to its large grain size and this resulted in the weakest re-468 gion for fatigue being located in the subsurface. Consequently, the impact on fatigue of 469 the surface features induced by SMAT - such as martensite, kink bands and compres-470 sive residual stresses - was limited. Additionally, if the second hypothesis presented 471 above is true, the significant surface roughness may have reduced the effectiveness of 472 the compressive residual stresses induced by SMAT. 473

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Sample	Treatment temperature	Ra (μ m)	Rz (μ m)
Polished (P)	None	0.05 ± 0.01	0.34 ± 0.07
SMAT-RT	$20 \text{ to } 80^{\circ}\text{C}$	3.13 ± 0.39	14.89 ± 1.75
SMAT-CT	$-170 \text{ to } -100^{\circ}\text{C}$	0.81 ± 0.13	3.88 ± 0.64

Table 1: Treatment temperature ranges for SMAT-RT and SMAT-CT samples, Ra and Rz roughness char-

acteristic values.

SMATed microstructures of a β-metastable titanium alloy

Martensitic transformation

Kink band formation



Mechanical properties of SMAT at Cryogenic Temperature (CT) versus SMAT at Room Temperature (RT)

SMAT-CT



✓ Fatigue resistance + 8% 🛰 Roughness - 75% ✓ Martensite depth + 50%

SMAT-RT

Room

