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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 $\beta$ -metastable titanium alloy

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## Abstract

Surface Mechanical Attrition Treatment (SMAT) has been carried out at cryogenic and room temperatures on a 5553  $\beta$ -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

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## Highlights

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

## 1. Introduction

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

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

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

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

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



49 anism such as the formation of kink bands [42–47]. Although common to materials with  
50 high anisotropy [48, 49], kink bands rarely appear in body-centered cubic structures. Zheng  
51 et al. described in detail the formation of those deformation bands [43]. They are due to the  
52 accumulation of edge dislocations resulting in bands with random misorientations ranging  
53 mostly between  $10^\circ$  and  $25^\circ$  compared to the initial orientation. Three kink slip systems  
54 may be activated:  $(112)[11\bar{1}]$ ,  $(123)[11\bar{1}]$ ,  $(101)[1\bar{1}\bar{1}]$ . They induce a rotation of the crystal  
55 around three rotation axes, also called Taylor axes:  $[1\bar{1}0]$ ,  $[5\bar{4}1]$ ,  $[12\bar{1}]$ , respectively. As the  
56 kink bands are relatively thin, this mechanism effectively refines the microstructure by the  
57 introduction of a high density of high-angle boundaries. It has been confirmed that this  
58 deformation mechanism depends on the alloy composition ; as Sadeghpour et al. used the  
59 same deformation process on four different  $\beta$ -metastable titanium alloys and obtained kink  
60 bands in only one of these alloys [50]. Surprisingly, for Sadeghpour et al., one of the alloys  
61 that did not produce any kink band was the 5553 while Qin et al. showed the possibility to  
62 form kink bands in this 5553 alloy [45].

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

## 71 **2. Experimental Procedures**

### 72 *2.1. Material and surface treatments*

73 The 5553  $\beta$ -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  $\mu\text{m}$ .

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

89 Three treatment conditions were studied and provided the following samples:

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

96 Table 1 gives the different treatment conditions with their corresponding temperature  
97 variations during SMAT as well as the resulting surface roughness. The temperature was  
98 probed with thermocouples placed in holes drilled from one sample head to the central  
99 section along the longitudinal axis of some samples that were used only for that matter. For  
100 SMAT at room temperature, the sample core temperature rose from 20°C to about 80°C,  
101 due to the heat generated by the shot impacts, before reaching a steady state after about  
102 2 min. The samples treated at cryogenic temperature were first cooled down to -170°C

103 with liquid nitrogen and were subsequently SMATed. Their core temperature increased by  
104 roughly the same amount to about  $-100^{\circ}\text{C}$  before stabilizing after less than 4 min.

## 105 *2.2. Microstructure and surface analyses*

106 The linear surface roughness was measured along the longitudinal axis on the central sec-  
107 tion with an average of 6 measurements per sample made with a stylus surface profilometer  
108 which had a standard radius of  $5\ \mu\text{m}$ .

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

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

131 profiles. To emerge from the background noise, the volume fraction of a given phase needs  
132 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  $\mu\text{m}$  below the surface and continued every  
135 30  $\mu\text{m}$  toward the core. The profiles were obtained from an average of 10 measurements.

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

### 144 *2.3. Fatigue and failure analyses*

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

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

### 159 **3. Results**

160 This section is divided into four different subsections dealing with the deformed mi-  
161 crostructure, the surface properties, the fatigue properties and, lastly, analyses of the frac-  
162 ture surfaces.

#### 163 *3.1. Deformed Structure Analyses*

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

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

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

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

### 224 *3.2. Surface and Subsurface Properties*

225 Since the surface integrity is of major importance in high-cycle fatigue due to the pro-  
226 motion of surface cracks for low integrity, determining the surface roughness is mandatory.  
227 Table 1 displays the Ra and Rz parameters of the surface roughness for polished and SMATed  
228 samples. The SMAT-RT surface samples present a roughness about 4 times higher compared  
229 to the SMAT-CT surface condition due to the increased mechanical strength of materials  
230 at cryogenic temperature leading to a lower deformation of the surface for a given imparted  
231 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  $\mu\text{m}$  below the surface. The affected depth dropped from 200  $\mu\text{m}$  to 150  
237  $\mu\text{m}$  with the use of the cryogenic temperature.

238 The residual stresses significantly limit the crack propagation in the regions in compres-  
239 sion and as such they significantly impact the fatigue behavior. Fig. 5b displays the residual  
240 stress profiles for both SMAT-RT and SMAT-CT sample conditions. Both profiles appear  
241 extremely similar in shape and intensities. The compressive residual stresses are about -400  
242 MPa at the surface and the residual stress profiles reach a compression peak at a depth

243 slightly over  $100\ \mu\text{m}$  with a value of about  $-650\ \text{MPa}$  for the SMAT-RT samples and  $-600$   
244  $\text{MPa}$  for the SMAT-CT samples. The residual stresses then increase until they reach  $0\ \text{MPa}$   
245 at a depth of about  $350\ \mu\text{m}$ .

### 246 *3.3. Fatigue Properties*

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

### 255 *3.4. Fracture Surfaces*

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



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

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

296 The tomography technique was used to eventually detect in-depth features without hav-  
297 ing to cut or alter the samples. Due to the resolution limitation, only large cracks can be  
298 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  $\mu\text{m}$  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

307 The results, previously reported for the  $\beta$ -metastable titanium alloy, have shown the  
308 effects of the SMAT processing conditions and, in particular, the effect of the SMAT tem-  
309 perature. The effects of SMAT on (i) the microstructure and on (ii) the fatigue properties  
310 will be discussed successively in the following sections.

### 311 4.1. Deformed microstructure

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

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

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

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

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

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

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

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

#### 400 *4.2. Impact on fatigue*

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

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

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

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

## 438 5. Conclusions

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

- 443 • Kink bands were formed after SMAT in the 5553  $\beta$ -metastable titanium alloy at its  
444 full  $\beta$  state due to the local high strain rates induced by SMAT. The random impact  
445 directions, inherent in the SMAT process, allowed the activation of different kink band  
446 systems. The kink band density was the highest in the subsurface for both SMAT at  
447 room (RT) and at cryogenic temperature (CT).
- 448 • The formation of martensite was observed within the kink bands for all SMAT condi-  
449 tions due to the concentration of the deformation in these regions.
- 450 • SMAT at cryogenic temperature allowed the formation of martensite deeper in the  
451 subsurface compared to SMAT done at room temperature. Contrary to what was  
452 observed in some austenitic stainless steels [21, 22], this higher amount of martensite  
453 was not associated with an increase in subsurface hardness. On the contrary, the  
454 SMAT-CT samples were found softer than the SMAT-RT ones when subsequently  
455 tested by micro-hardness at room temperature.
- 456 • The roughness after SMAT was reduced by a factor of 4 when the treatment was  
457 done at cryogenic temperature compared to the treatment done at room temperature  
458 due to the increased mechanical strength of the alloy at lowest temperature of SMAT  
459 processing.
- 460 • SMAT-CT resulted in an improvement of the fatigue resistance by 8% compared to  
461 the SMAT-RT. Two hypotheses may explain this difference: (i) the deeper martensite  
462 and the largest kink bands formed by SMAT-CT may have delayed the final fracture;  
463 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.

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

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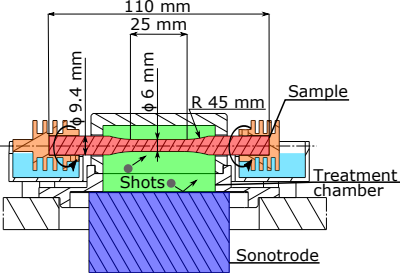
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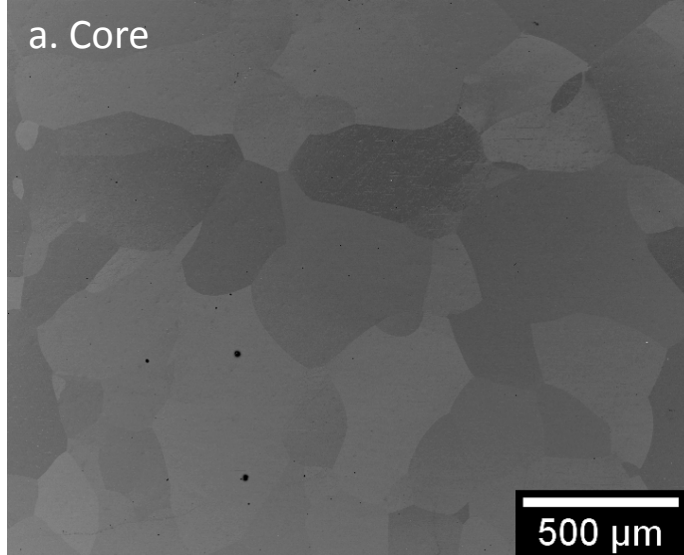
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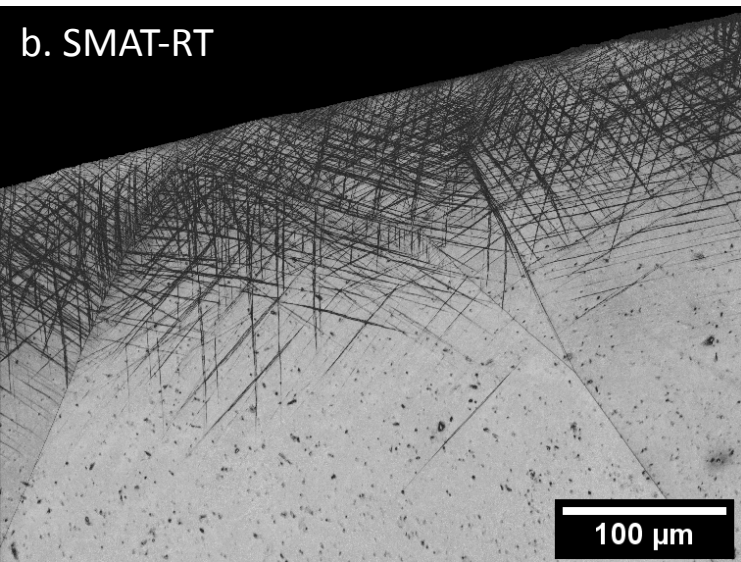
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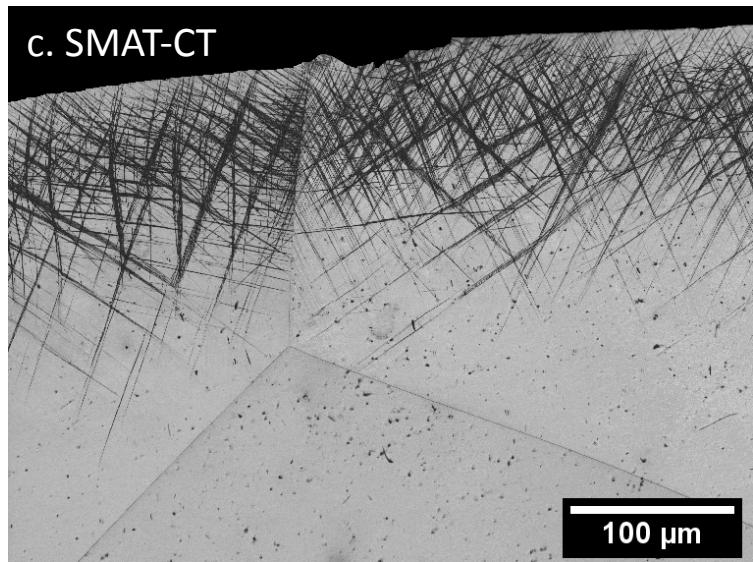
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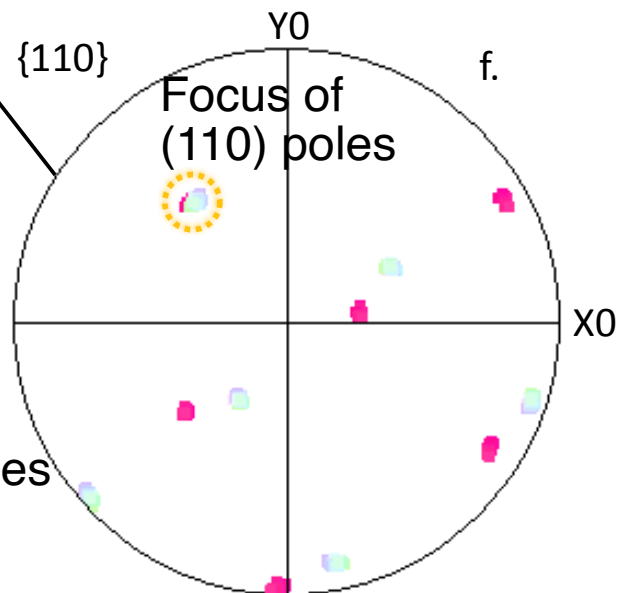
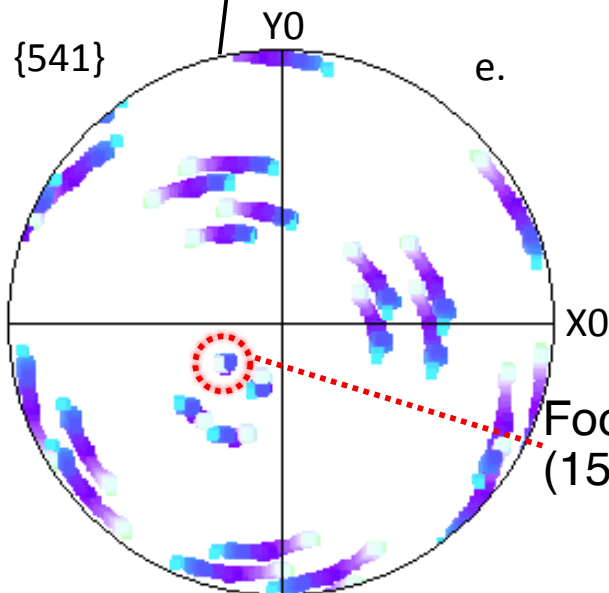
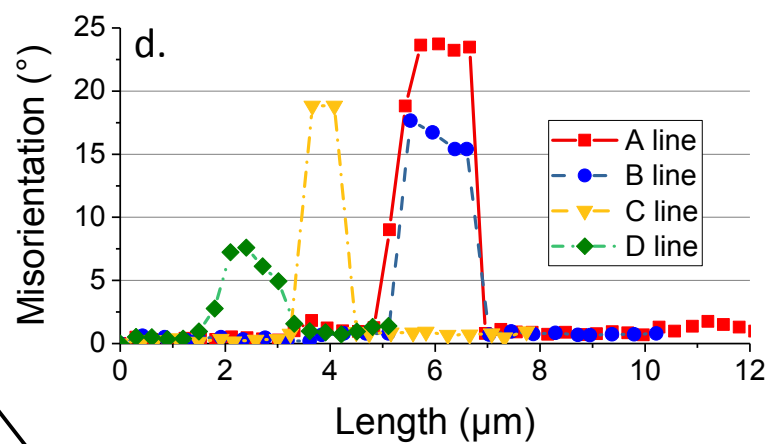
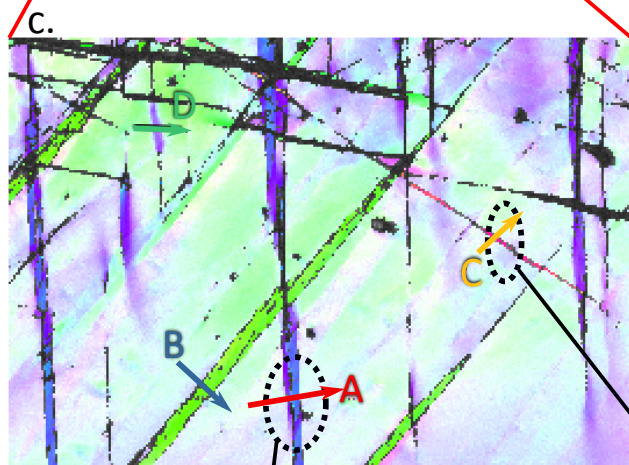
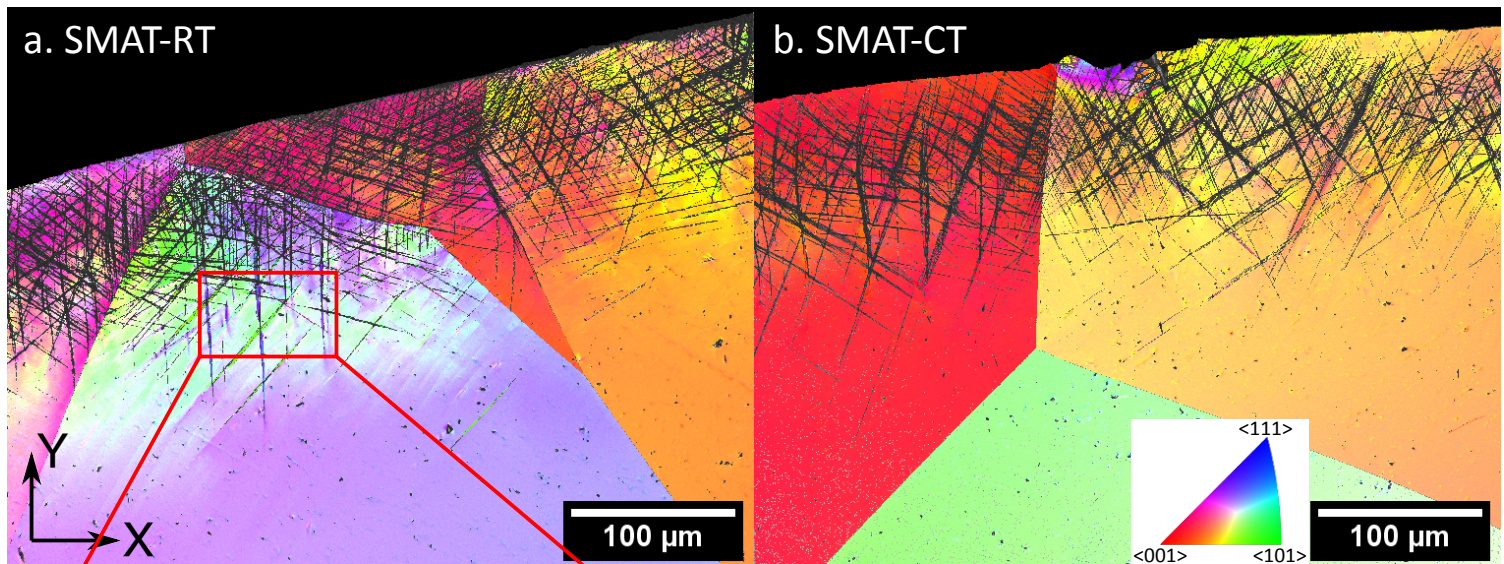


b. SMAT-RT

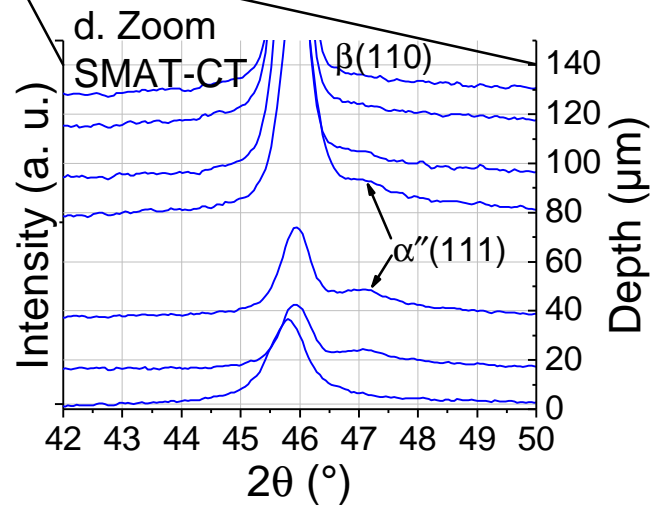
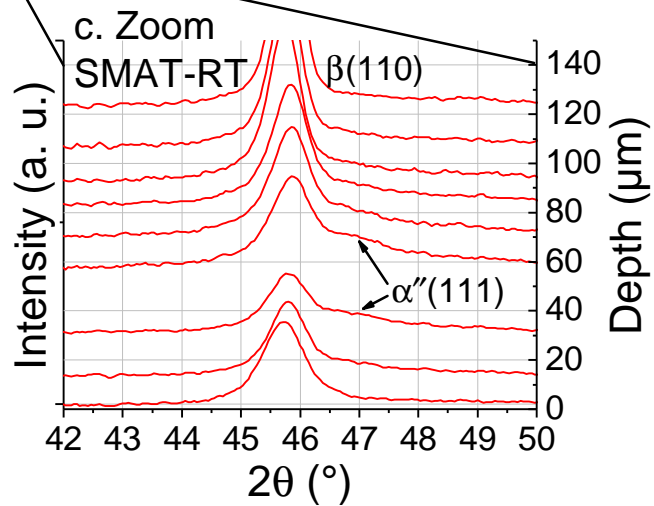
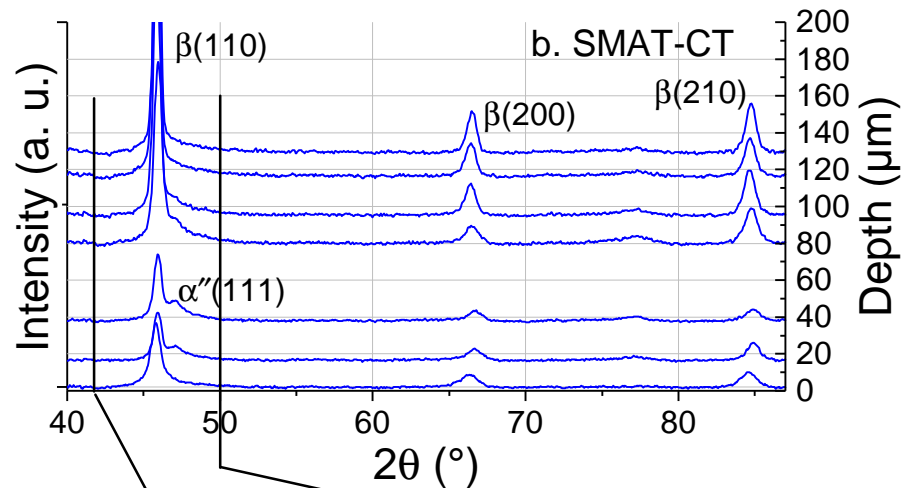
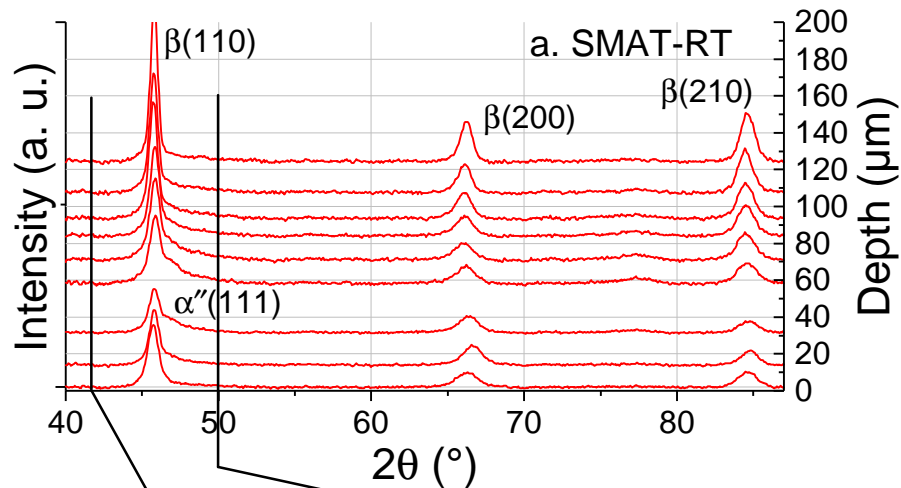


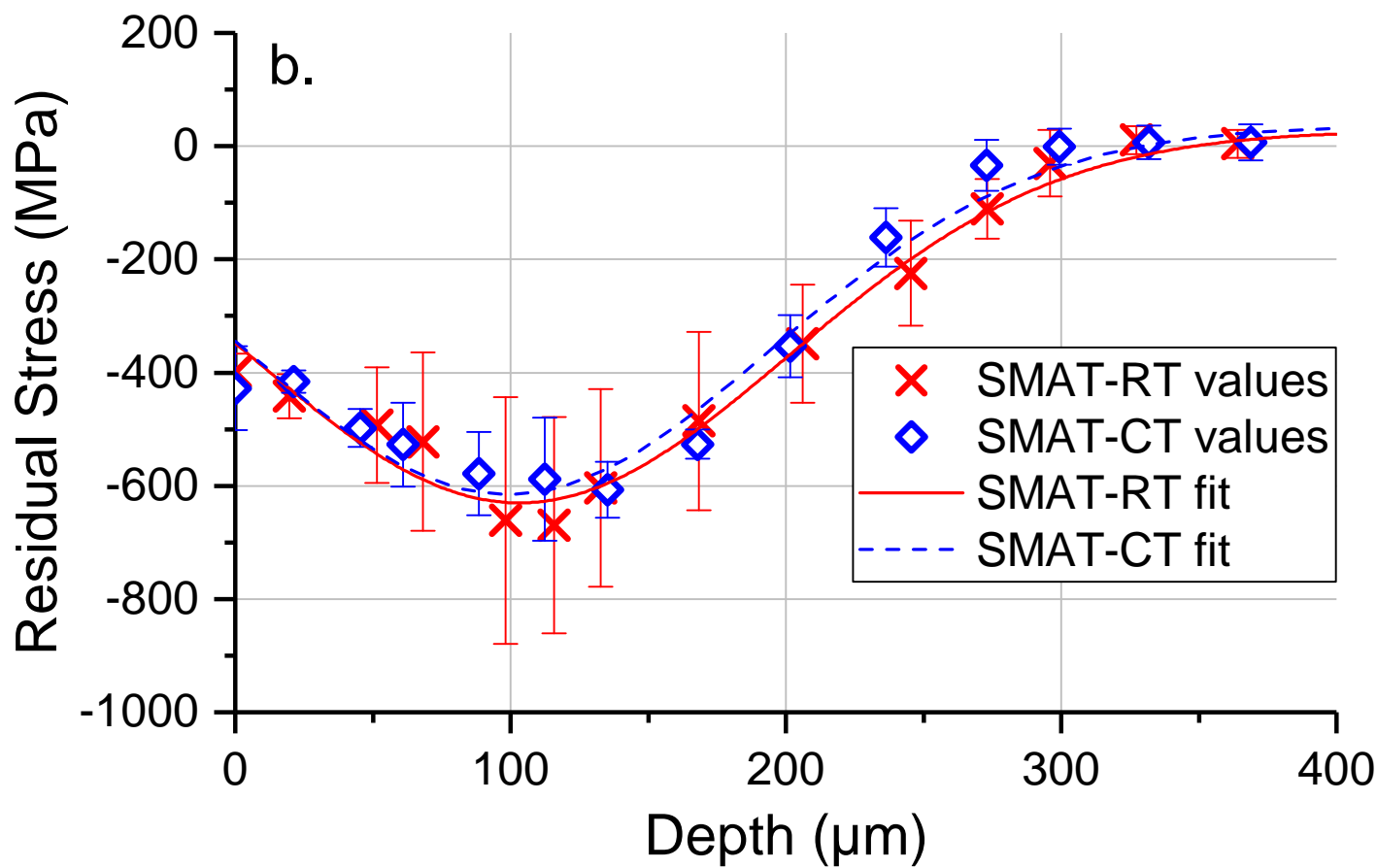
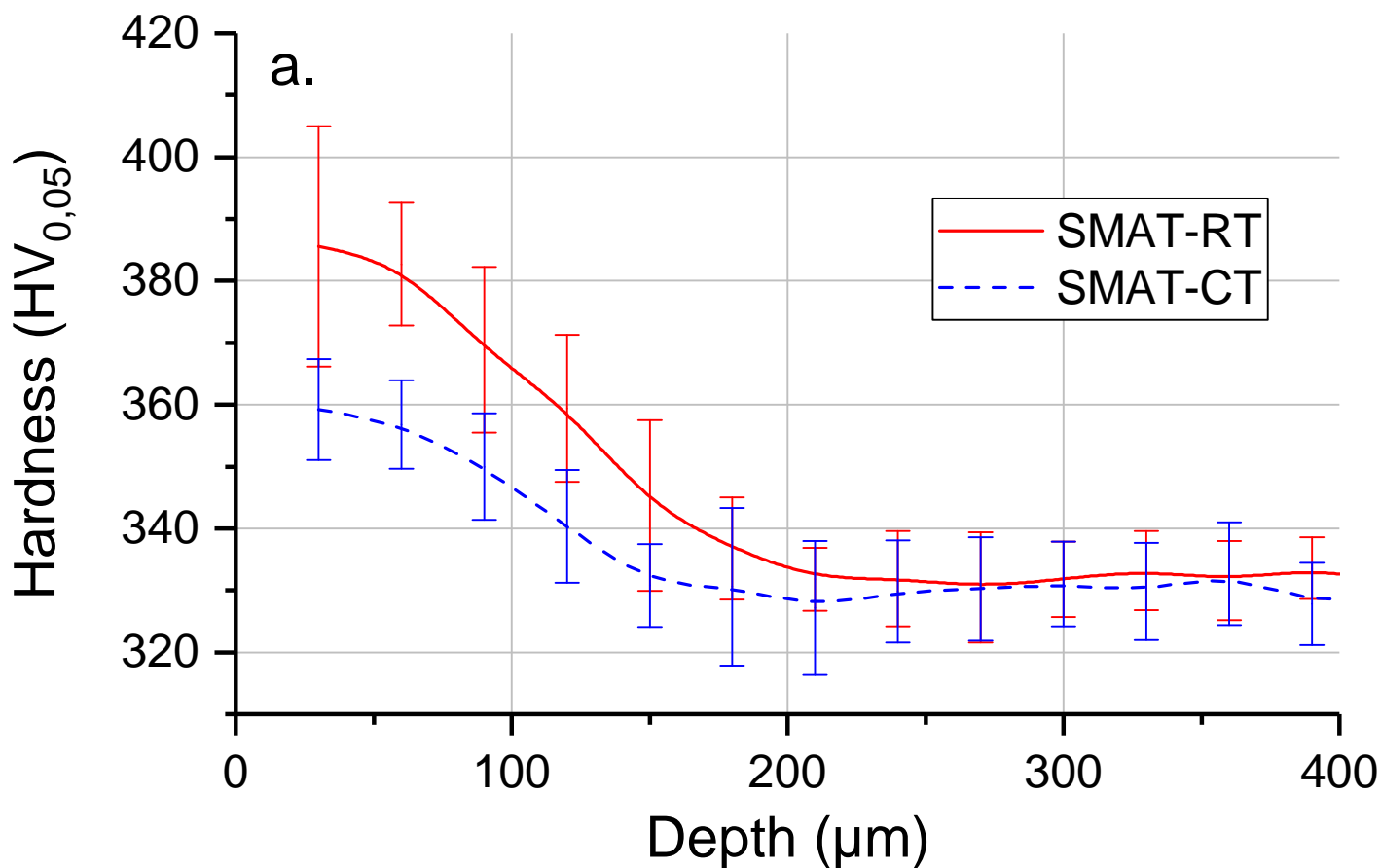
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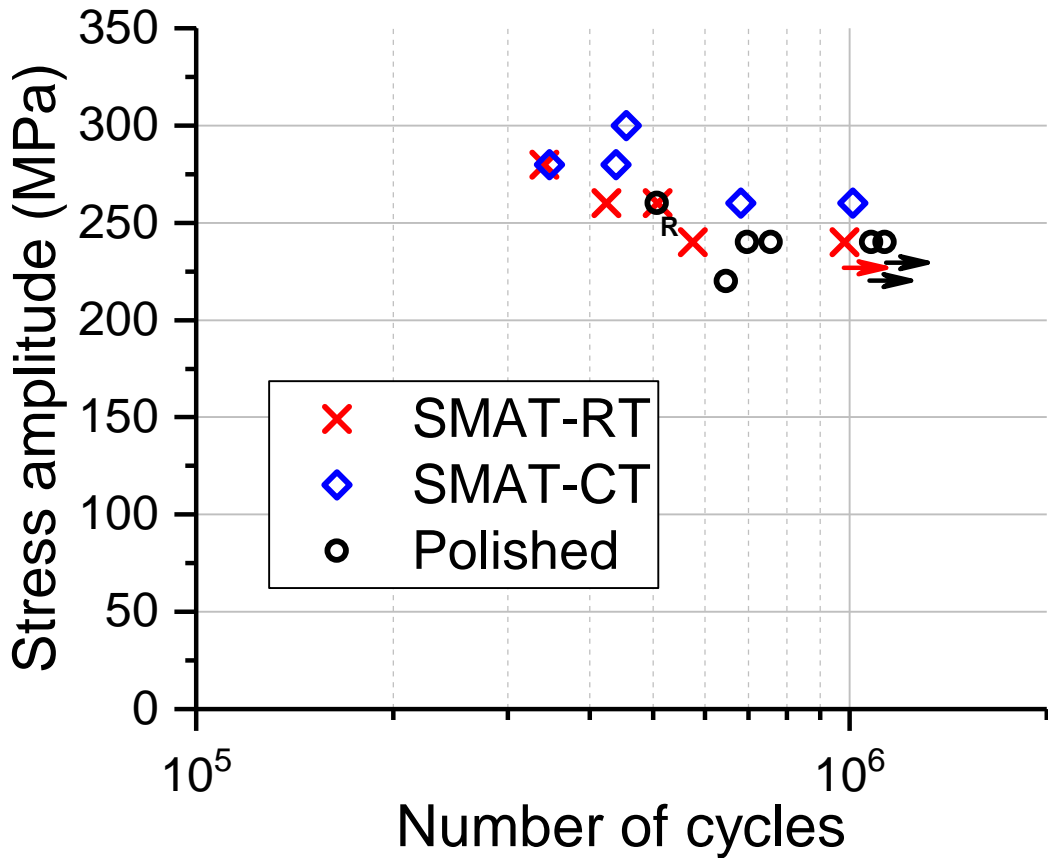


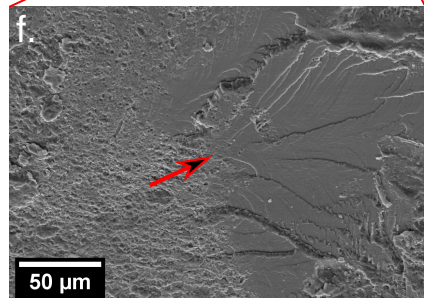
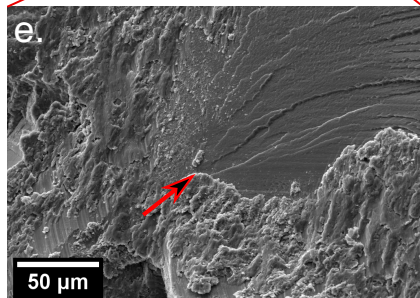
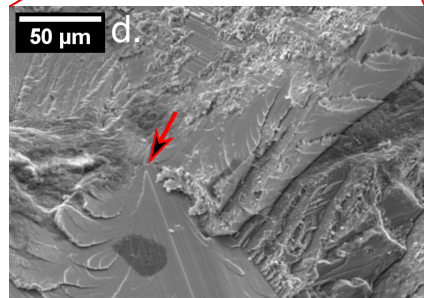
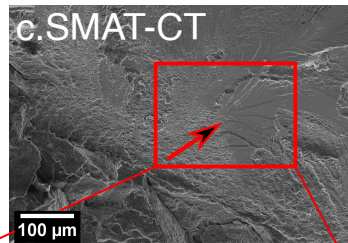
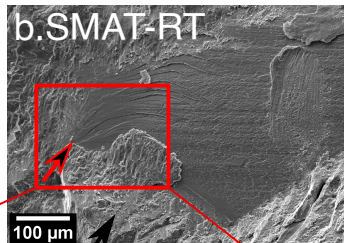
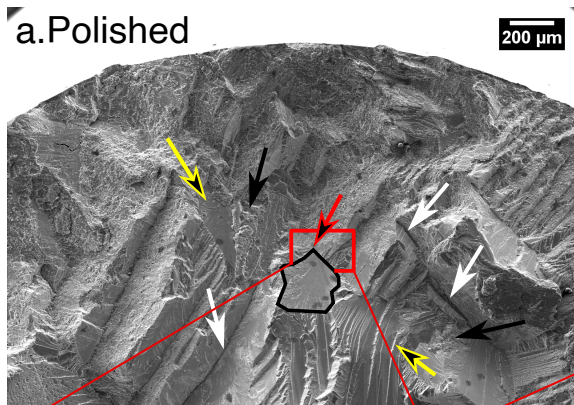




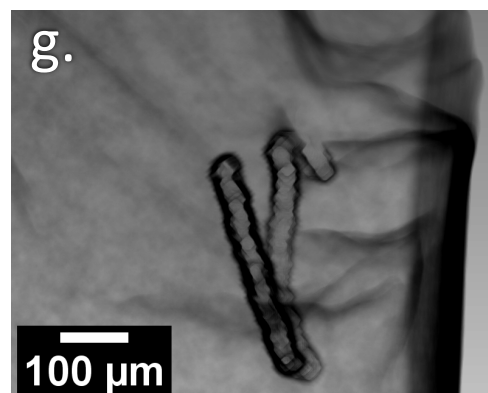
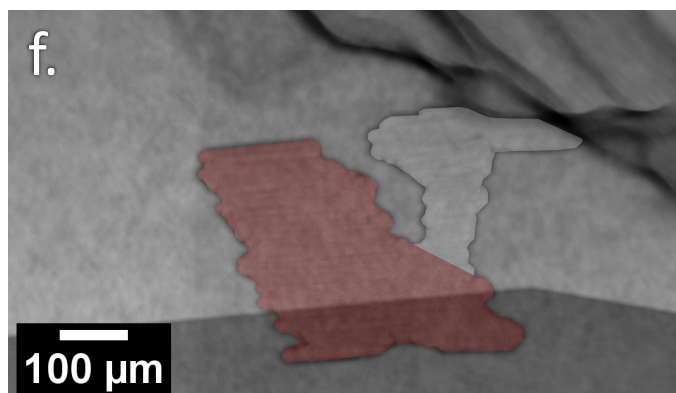
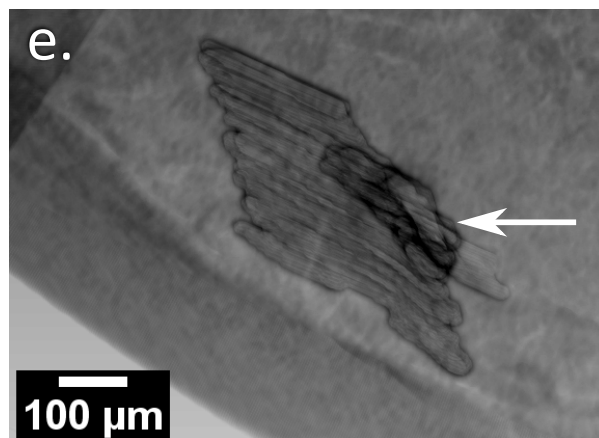
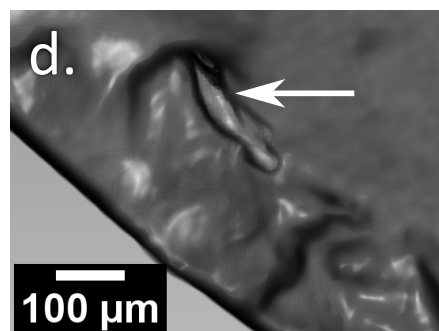
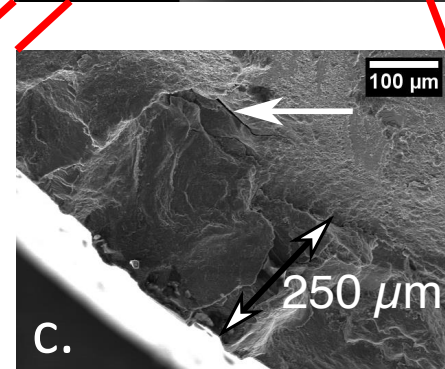
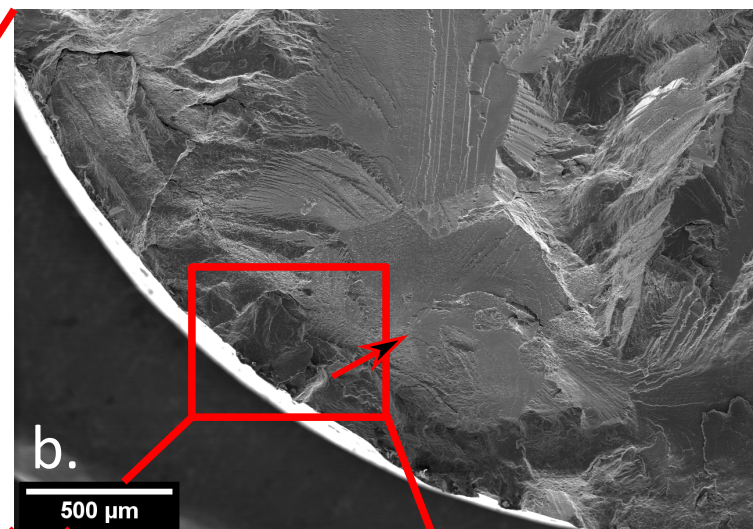
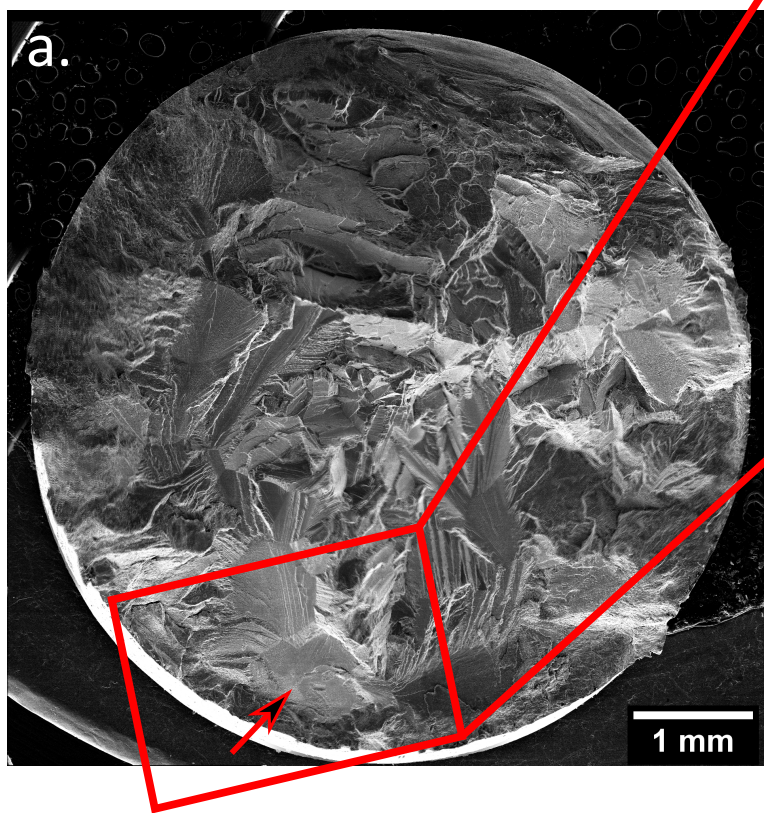












Sample	Treatment temperature	Ra ( $\mu\text{m}$ )	Rz ( $\mu\text{m}$ )
Polished (P)	<i>None</i>	$0.05 \pm 0.01$	$0.34 \pm 0.07$
SMAT-RT	20 to 80°C	$3.13 \pm 0.39$	$14.89 \pm 1.75$
SMAT-CT	-170 to -100°C	$0.81 \pm 0.13$	$3.88 \pm 0.64$

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

# SMATed microstructures of a $\beta$ -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

Temperature

- ↗ Surface hardening + 7%
- ↗ Hardened depth + 30%

