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VHCF life of AM1 Ni-based single crystal superalloy after pre-deformation

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Abstract

VHCF properties of the Ni-based single crystal superalloy AM1 with pre-deformation have been investigated at 1000 °C. Pre-deformed material did not present life debit under fully reversed conditions ($R = -1$). Pre-deformation decreased VHCF lifetime under high mean stress conditions ($R = 0.5$) with a presence mode I surface crack initiation. The pre-deformed material has microstructure coarsened bands lying on the former {111} slip planes, facilitating microstructure shearing during VHCF at $R = 0.5$. Slip traces increased surface roughness, causing surface recrystallization and fatal surface crack. Rejuvenation heat treatment after pre-deformation can restore the microstructure and the fatigue life.

Highlights

- VHCF life of pre-deformed Ni-based single crystal superalloy at 1000 °C.
- Pre-deformation has no effect on the VHCF life at $R = -1$.
- Pre-deformation induces VHCF life debit at $R = 0.5$ and high mean stress.
- Rejuvenation heat treatment restores VHCF life.

1. Introduction

Very high cycle fatigue (VHCF) life of Ni-based single crystal (SX) superalloys in fully reversed stress condition ($R = -1$) is generally controlled by the size and position of the largest casting pores [1–5]. This mainly results from the stress concentration in pores' vicinity that activates γ/γ' microstructure shearing by slip bands [4]. In such conditions, a solidification pore (S-pore) in the interdendritic area becomes the main crack initiation site [6,7]. In a recent article by Cervellon et al., fatigue crack initiation mechanisms have been proposed for the VHCF regime ($N_f \gg 10^7$) under fully reversed conditions at 1000 °C [4]. This study described that stress concentration near a large S-pore induces intense localized shearing of γ' precipitates activating recrystallization and interacting solute element concentration [4,8–10]. The local crack propagation threshold decreases as recrystallization proceeds. The crack propagation at the recrystallized area creates a “rough zone” at the fracture surface of a specimen failed by internal crack initiation [4].

On the other hand, the VHCF life of Ni-based SX superalloys at a positive ratio ($R > 0$) is affected by the γ/γ'

41 microstructure and the intrinsic creep strength [1,2,11]. Overaged and coarsened microstructures have lower creep
42 durability and lower yield stress, especially at high applied stresses or higher strain rates [1,12]. Therefore, since
43 the specimen is in tensile loading during all periods of positive ratio condition with a very high strain rate (in the
44 order of 10^2 /s), creep damages such as microstructure coarsening and cracks initiating from creep voids also take
45 place in this VHCF regime.

46
47 A plastic (pre-)deformation may occur during the processing (i.e. casting, heat treatments ...) of airfoils made
48 from Ni-based SX superalloys [8,13,14]. If such plastic deformation (PD) is introduced in a component after the
49 solution heat treatment, three microstructural events take place during subsequent aging treatments [8,15]: 1)
50 Faster γ/γ' microstructure evolution in the vicinity of the slip band compared to the area far from the slip band; 2)
51 S-pore and homogenization-pore (H-pores [6,16]) growth assisted by enhanced γ/γ' microstructure evolution at the
52 bands; 3) Dislocation climb favoring the nucleation of deformation pores (D-pores [17]) within the bands.
53 Moreover, during creep loading at high temperature (≥ 950 °C), faster microstructure coarsening along the former
54 slip band accelerates the creep damage accumulation at nucleated and enlarged pores, ultimately leading to a
55 shorter creep life [8,15]. When creep testing at a lower temperature with higher applied stresses (e.g., 850 °C / 500
56 MPa), γ/γ' microstructure shearing from creep void was observed locally in the microstructure coarsened band
57 [8,14]. Considering these microstructure evolutions and high-temperature behaviors of pre-deformed AM1
58 Ni-based SX superalloy, coarsened pores lying in the microstructure coarsened band with lower resistance to
59 shearing may decrease the high-temperature fatigue life of the material.

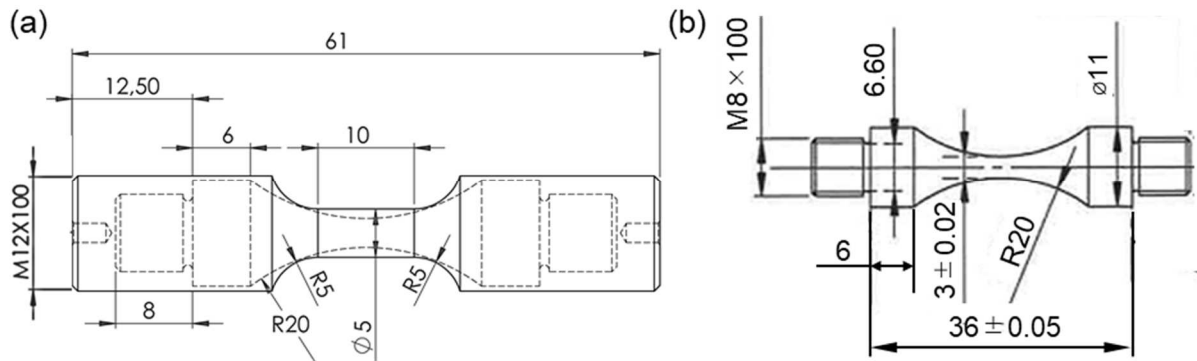
60
61 Within this context, this study aims at investigating the influence of microstructural defects induced by prior
62 plastic deformation on the VHCF lifetime of the AM1 SX superalloy. Another objective is to improve the
63 understanding of fracture mechanisms in the VHCF regime at 1000 °C, both in fully reversed ($R = -1$) and with
64 the application of a mean stress ($R = 0.5$). The experiments are achieved by a specifically-designed two-step
65 machining procedure to obtain pre-deformed VHCF specimens. Because turbine blades may be pre-deformed
66 during production [8,13–15], and since high cycle fatigue properties have been imposed as new turbine airfoil
67 design criteria by the airworthiness authorities (FAA, EASA), evaluating fatigue properties of pre-deformed
68 material is an essential matter. Rejuvenation heat treatment, which can restore microstructure and creep life after
69 pre-deformation [14], is also tested in this study to evaluate its efficiency in restoring VHCF life.

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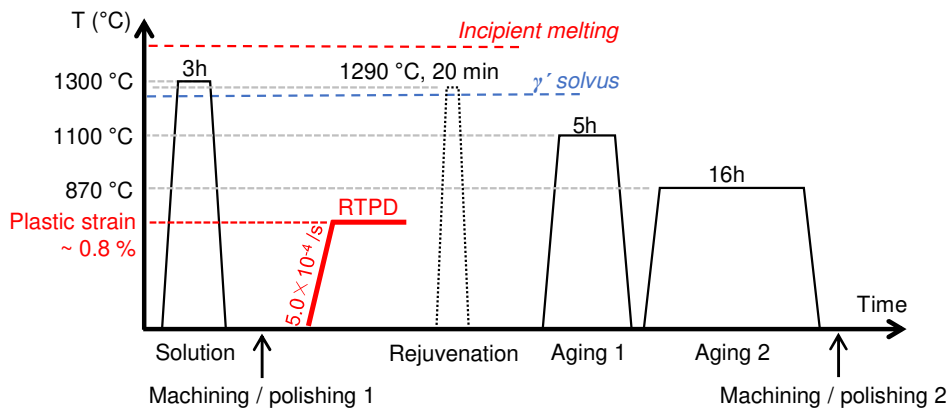
71 **2. Experimental procedures**

72 Solution-treated SX bars (14 mm diameter) of the Ni-based superalloy AM1
73 (Ni-7.6Cr-6.7Co-5.6W-2Mo-8Ta-5.2Al-1.2Ti-0.05Hf, wt.%) used in this study are from the same casting batch as
74 our previous study [15]. SX bars with excellent crystallographic orientation (longitudinal misorientation from the
75 perfect $\langle 001 \rangle$ direction less than 2°) were chosen for this study. VHCF test specimens with pre-deformation were
76 prepared by following steps including two-step machining: 1) Machining SX bars into tensile test specimen
77 (geometry given in Figure 1(a)) with parallel gauge section (i.e. smooth gauge part); 2) Mechanical polishing of a
78 gauge section using P4000 SiC abrasive paper to remove residual stresses inherited from machining; 3) Apply

79 tensile prior plastic deformation at room-temperature at a strain rate of 5.0×10^{-4} /s using an Instron 8562
 80 electromechanical machine. Plastic strain of the pre-deformed specimen was controlled at $\sim 0.8\%$ using an
 81 extensometer; 4) Aging heat treatments consisting of standard first (at 1100 °C for 5 h, air quench) and second (at
 82 870 °C for 16 h, air quench) stages in the air using a resistive furnace; 5) Second machining into hourglass-shaped
 83 VHCF specimen with the geometry given in Figure 1(b) [1]; 6) Surface polishing up to a mirror finish (1 μm
 84 diamond paste) to remove residual stresses and plastically deformed layer coming from machining and to
 85 minimize surface roughness. These steps are presented as a diagram in Figure 2.
 86



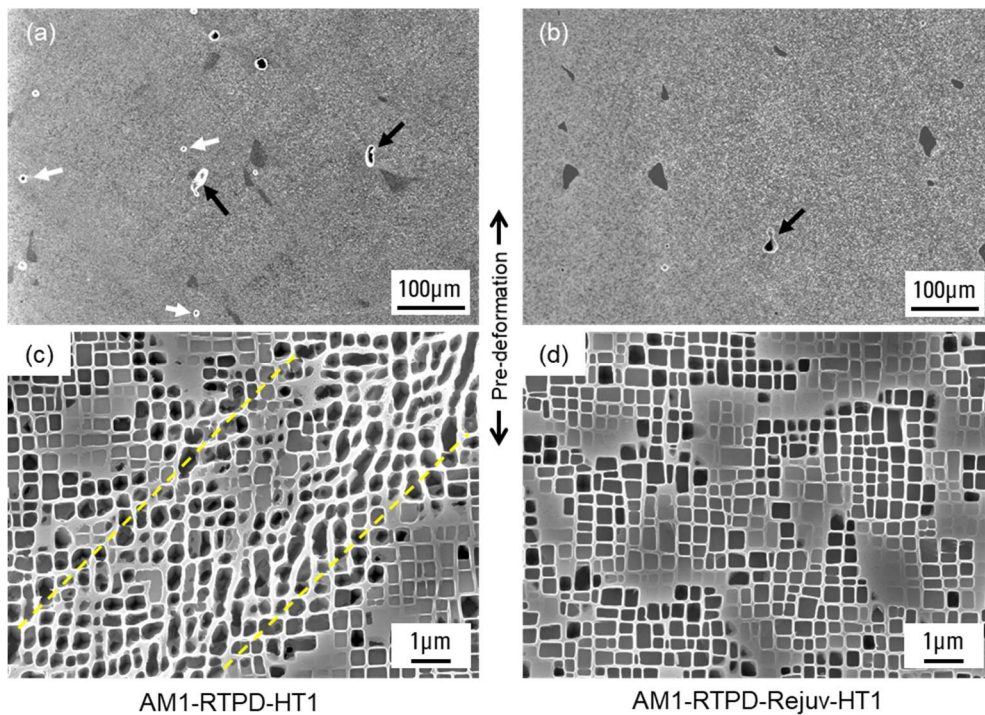
87
 88 Figure 1. Geometries of cylindrical gauge specimens for tensile pre-deformation (a) and hourglass-shaped
 89 specimens for the VHCF test (b).
 90



91
 92 Figure 2. Material history diagram showing an order of machining/polishing, RTPD (room temperature plastic
 93 deformation), rejuvenation, and aging treatments.
 94

95 The pre-deformed specimen is named “AM1-RTPD-HT1”, whereas reference material without PD is named
 96 “AM1-HT1”. The VHCF test using an ultrasonic fatigue machine requires a specimen with precise geometry to
 97 achieve resonance at ~ 20 kHz and 1000 °C. Therefore, VHCF specimens were machined from larger cylindrical
 98 specimens to introduce controlled pre-deformation strain in the central part of the VHCF specimen without
 99 changing the geometry. Rejuvenation treatment at 1290 °C for 20 min (air quench) was inserted between
 100 pre-deformation and aging treatments for microstructure restoration (this specimen is named
 101 “AM1-RTPD-Rejuv-HT1”). The rejuvenation treatment condition for pre-deformed AM1 was investigated and
 102 determined previously [14].
 103

104 Initial microstructures of AM1-RTPD-HT1 and AM1-RTPD-Rejuv-HT1 are shown in Figure 2. Bands of
 105 coarsened γ' precipitates were observed in AM1-RTPD-HT1 and they are called “microstructure coarsened bands”
 106 whereas they were absent in AM1-RTPD-Rejuv-HT1. Diameter of S-pores (black arrows in Figure 2) was
 107 measured using the surrounding diameter method [18] and it was 70 μm at maximum for both AM1-RTPD-HT1
 108 and AM1-RTPD-Rejuv-HT1, a value equivalent to the one measured in AM1 SX superalloy tested in the previous
 109 study [2]. D-pores (white arrows in Figure 2(a)) were nucleated in AM1-RTPD-HT1 during aging treatments.
 110 Details of the microstructure coarsened bands and pores are available in previous studies from the authors [8,15].
 111 In addition, AM1-RTPD-Rejuv-HT1 presented equivalent γ/γ' microstructure to AM1-HT1 [14].
 112



113
 114 **Figure 3.** Initial microstructure of AM1-RTPD-HT1 (a, c) and AM1-RTPD-Rejuv-HT1 (b, d). Black and white
 115 arrows in lower magnification images (a, b) are pointing at S-pores and D-pores, respectively. Dotted
 116 lines in (c) are indicating the microstructure coarsened band.
 117

118 All fatigue tests were performed at 1000 °C, 20 kHz, and $R = -1$ and 0.5 using an ultrasonic fatigue machine
 119 following the procedures described previously [1–4]. The VHCF specimen is designed specifically for the testing
 120 machine and its first tension/compression mode resonant frequency is 20 ± 0.5 kHz. Specimen center was heated
 121 by induction heating method and the temperature was measured and controlled using a dual-wavelength pyrometer,
 122 forming a closed-loop temperature controlling system. PID parameters of the induction heating device were
 123 adjusted so as to achieve an excellent temperature control at 1000 °C (± 2 °C) despite self-heating of the
 124 specimens that may occur at high alternating stresses ($\sigma_a > 200$ MPa) in the beginning of the tests. A piezoelectric
 125 transducer transformed a sinusoidal electrical signal into a longitudinal mechanical wave which was amplified by
 126 an ultrasonic horn. Tests at $R = -1$ were performed under strain-controlled mode. A constant mean load was
 127 applied using a pneumatic device for the tests at $R = 0.5$.
 128

129 Fractographic and microstructural analyses of failed specimens were performed using an optical microscope and a

130 field emission gun scanning electron microscope (JEOL JSM-7000F). Longitudinal cross sections of
 131 AM1-RTPD-HT1 specimens after VHCF tests at 1000 °C, $f = 20$ kHz, $R = -1$, $\sigma_a = 209$ MPa and at 1000 °C, $f =$
 132 20 kHz, $R = 0.5$, $\sigma_{max} = 551$ MPa were prepared for microstructure observations. A combination of mechanical
 133 polishing up to 1- μ m diamond powder and chemical etching by aqua regia was applied for observing γ/γ'
 134 microstructure. The longitudinal cut specimens were finished either by electrochemical polishing using Struers
 135 LectroPol-5 system (electrolyte A3, operated at 45 V for 5 s) or mechanical polishing using colloidal silica
 136 solution (particle size 0.04 μ m) for the electron backscatter diffraction (EBSD) analysis using a JEOL JSM-6100
 137 scanning electron microscope equipped with EDAX forward scatter detector. Crystallographic orientation in
 138 inverse pole figure (IPF) maps refers to the loading direction of the specimen.

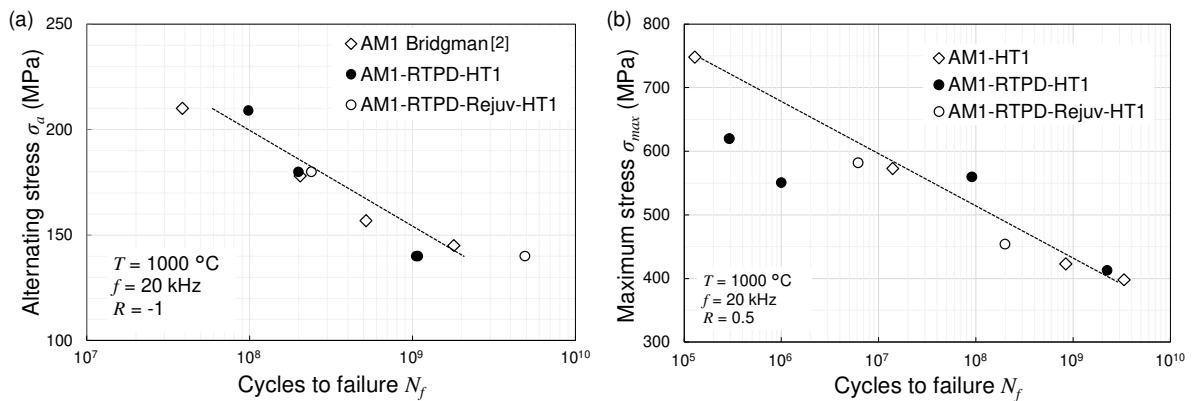
139

140 3. Results

141 A) VHCF tests results

142 VHCF tests were performed on pre-deformed AM1 at 1000 °C with stress ratios of $R = -1$ and 0.5 (Figure 4).
 143 Under fully reversed condition ($R = -1$), both AM1-RTPD-HT1 and AM1-RTPD-Rejuv-HT1 have a similar
 144 lifetime compared to the reference “AM1 Bridgman”, which was processed with standard heat treatments without
 145 pre-deformation [2]. In the tests with a positive ratio of $R = 0.5$, AM1-RTPD-Rejuv-HT1 performed similarly to
 146 the reference AM1-HT1. AM1-RTPD-HT1 failed at a similar life to the reference at $R = 0.5$ and $\sigma_{max} = 413$ MPa
 147 and 560 MPa. However, AM1-RTPD-HT1 exhibited a shorter fatigue lifetime of nearly one decade at higher
 148 maximum stress condition of $\sigma_{max} = 620$ MPa and 551 MPa.

149



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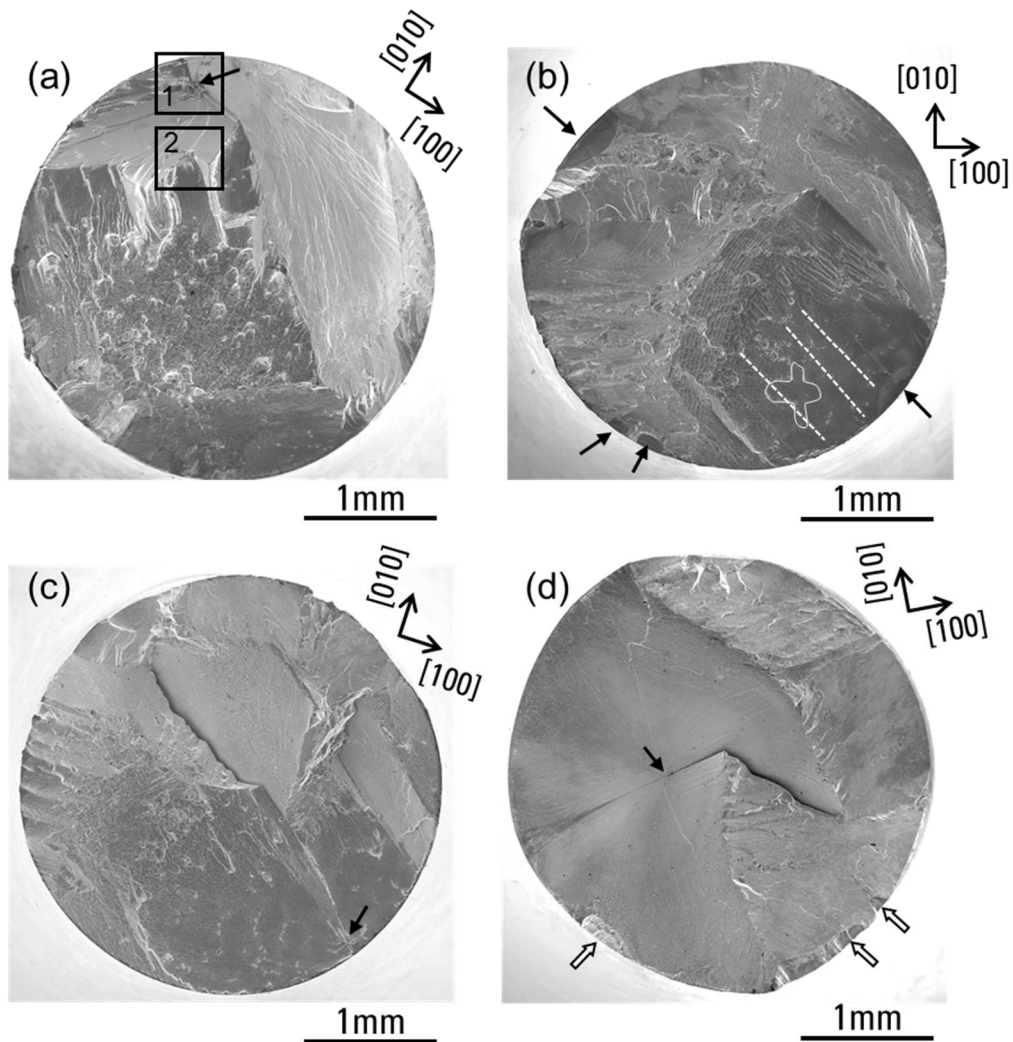
151 Figure 4. Results of VHCF tests at 1000 °C / $f = 20$ kHz in the S-N diagram. The alternating stress is plotted as a
 152 function of the number of cycles to failure for $R = -1$ (a). The maximum stress is plotted as a function
 153 of the number of cycles to failure for $R = 0.5$ (b). Data points of AM1 Bridgman in (a) are taken from
 154 reference [2].
 155

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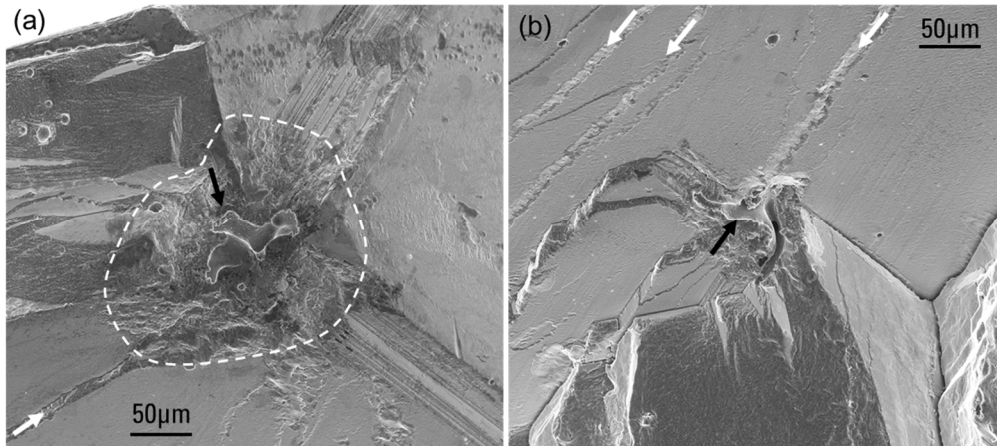
156 B) Fractographic observations

157 Fracture surfaces of specimens after the VHCF test are shown in Figures 5. The fracture plane parallel to the
 158 $\{111\}$ slip system appeared in all specimens. At $R = -1$ conditions, the fatal crack started from S-pore close to the
 159 cylindrical surface. The rough zone was surrounding S-pores on the fracture surface regardless of applied stress

160 and lifetime up to $\sim 10^9$ cycles (Figure 6(a)). The size of critical S-pore and surrounding rough zone are consistent
 161 with previous results from the literature [2]. Also, traces of the microstructure coarsened band were apparent in all
 162 AM1-RTPD-HT1 specimens tested at $R = -1$ (see white arrows in Figures 6). In AM1-RTPD-HT1 tested with $R =$
 163 -1 and $\sigma_a = 209$ MPa, multiple and isolated S-pores on the fracture surface (Figure 6(b)) presented features similar
 164 to the main crack initiating pore (i.e., presence of a rough zone). Multiple S-pores were exposed in the fracture
 165 surface of specimens that failed at lower stresses as well.
 166



167
 168 Figure 5. Fractography of AM1-RTPD-HT1 tested at 1000 °C, $f = 20$ kHz, $R = -1$, $\sigma_a = 209$ MPa (a),
 169 AM1-RTPD-HT1 tested at 1000 °C, $f = 20$ kHz, $R = 0.5$, $\sigma_{max} = 551$ MPa (b), AM1-HT1 tested at
 170 1000 °C, $f = 20$ kHz, $R = 0.5$, $\sigma_{max} = 573$ MPa (c) and AM1-RTPD-HT1 tested at 1000 °C, $f = 20$ kHz, R
 171 $= 0.5$, $\sigma_{max} = 560$ MPa. Black arrows are pointing at the main crack initiating pore (a, c, d) and surface
 172 failure initiating sites (b). White arrows in (d) are pointing at surface cracks. Rectangles 1 and 2 in (a)
 173 are shown as magnified images in Figure 6(a) and 6(b), respectively. Dotted lines in (b) are indicating
 174 the microstructure coarsened bands. White curve in (b) is showing one of dendrite structures.
 175



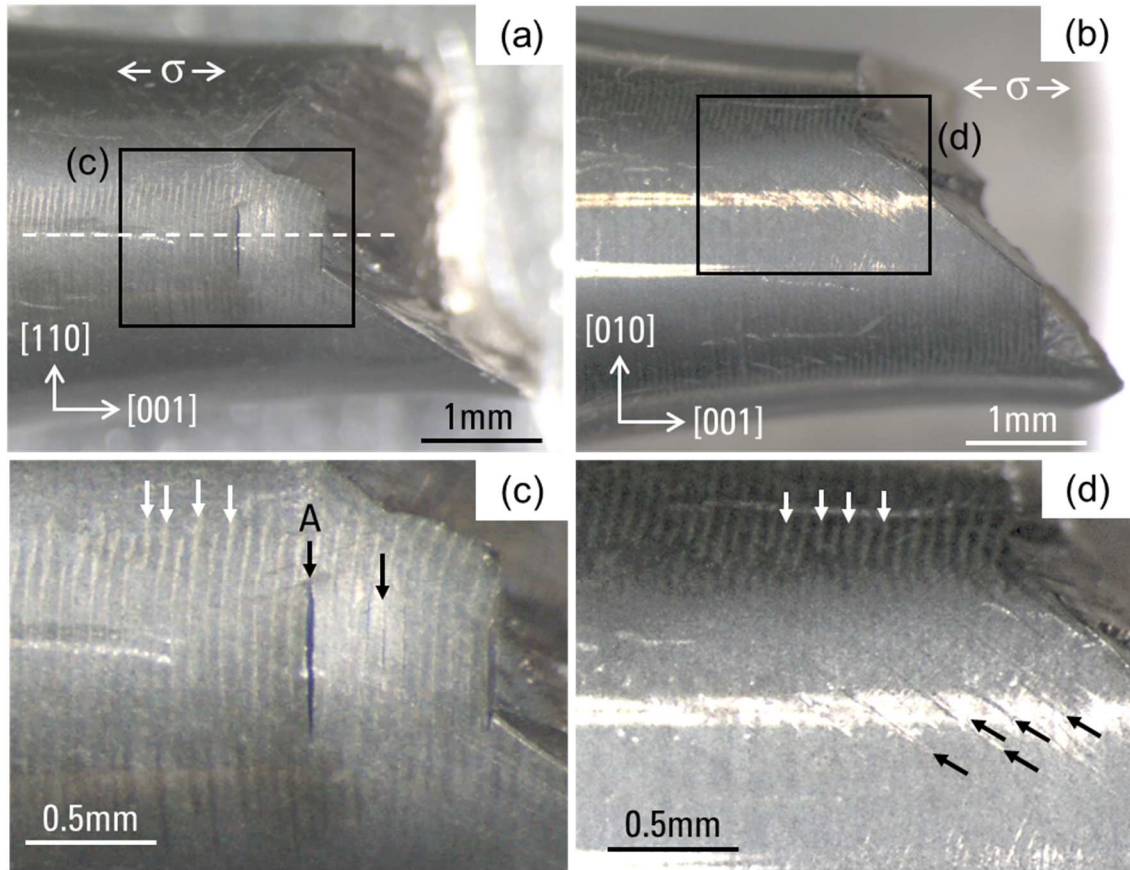
176
 177 Figure 6. Magnified image of VHCF specimen (AM1-RTPD-HT1) tested at 1000 °C, $f = 20$ kHz $R = -1$, $\sigma_a = 209$
 178 MPa shown in Figure 5(a). The main crack initiation site is indicated by a black arrow with the rough
 179 zone surrounded by the dotted line (a). Another solidification pore on the fracture surface is indicated
 180 by a black arrow in (b). White arrows are pointing at microstructure coarsened bands.

181
 182 AM1-HT1 and AM1-RTPD-Rejuv-HT1 at $R = 0.5$ and maximum stress lower than 600 MPa also presented an
 183 internal crack initiation with the major fracture plane lying on $\{111\}$ plane, in good agreement with the previous
 184 studies [1,2]. Figure 5(c) is an example of an internal crack initiation of AM1-HT1 tested at 1000 °C, $f = 20$ kHz,
 185 $R = 0.5$, $\sigma_{max} = 573$ MPa.

186
 187 Exception to these trends with a failure starting from the surface was the test performed with the conditions of
 188 1000 °C, $f = 20$ kHz, $R = 0.5$, and $\sigma_{max} = 551$ MPa (Figure 5(b)). Mode I cracks are initiating from the specimen's
 189 surface at locations with an external normal to the specimen's gauge surface of $\langle 110 \rangle$ type, as indicated by black
 190 arrows in Figures 5(b) and 7(c). At the similar condition of $\sigma_{max} = 560$ MPa that showed no effect of
 191 pre-deformation on the VHCF life, the specimen had both the internal crack initiation from a pore and surface
 192 cracks along $\sim \langle 110 \rangle$ type generatrices, as shown in Figure 5(d) by white arrows. The internal crack was directly
 193 transferring to crystallographic fracture and no mode I crack propagation stage from this internal pore was
 194 observed.

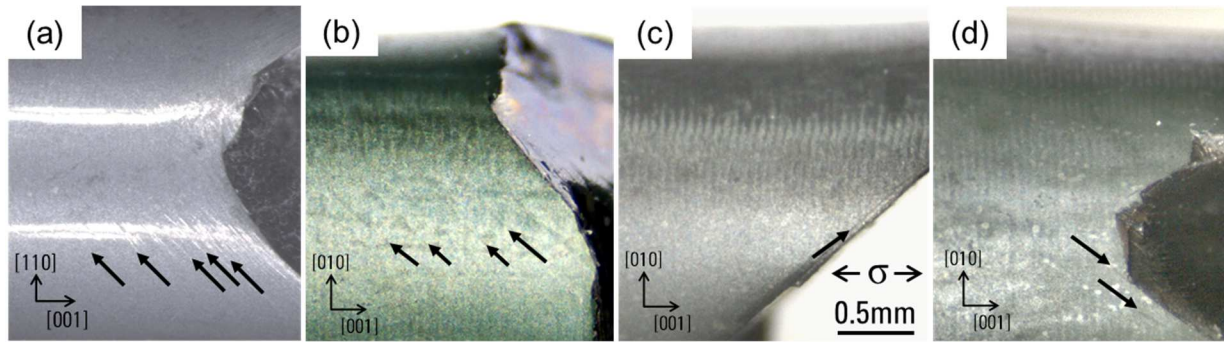
195
 196 Apart from the cracks of AM1-RTPD-HT1 at $R = 0.5$, and $\sigma_{max} = 551$ MPa, line traces transverse to the specimen's
 197 longitudinal direction were also observed (white arrows in Figures 7(c, d)). The same specimen had slip marks
 198 parallel to the $\{111\}$ plane on the surface viewed from a different direction (black arrows in Figure 7(d)). Such
 199 $\{111\}$ slip traces are similar to those previously observed at the specimen's surface just after pre-deformation or
 200 after creep tests at temperatures above 950 °C [8,15]. AM1-RTPD-HT1 at $R = 0.5$ and $\sigma_{max} = 620$ MPa had crack
 201 initiation from the surface as well. This specimen showed a very high density of $\{111\}$ slip traces near fracture
 202 surface (Figure 8(a)) compared to specimen tested at $\sigma_{max} = 551$ MPa.

203



204
 205 Figure 7. Appearances of the VHCF specimen (AM1-RTPD-HT1) tested at 1000 °C, $f = 20$ kHz, $R = 0.5$, $\sigma_{max} =$
 206 551 MPa from two different directions (a, b). Rectangles in (a) and (b) are shown in (c) and (d),
 207 respectively. Black arrows in (c) are pointing at surface cracks. The microstructure of crack A is
 208 presented in Figures 11 and 12. Black arrows in (d) are showing $\{111\}$ slip traces appearing on the
 209 cylindrical surface. White arrows in (c) and (d) are line traces transverse to the loading direction.
 210 Dotted line in (a) is the cutting plane for microstructure observation (see Figure 11).
 211

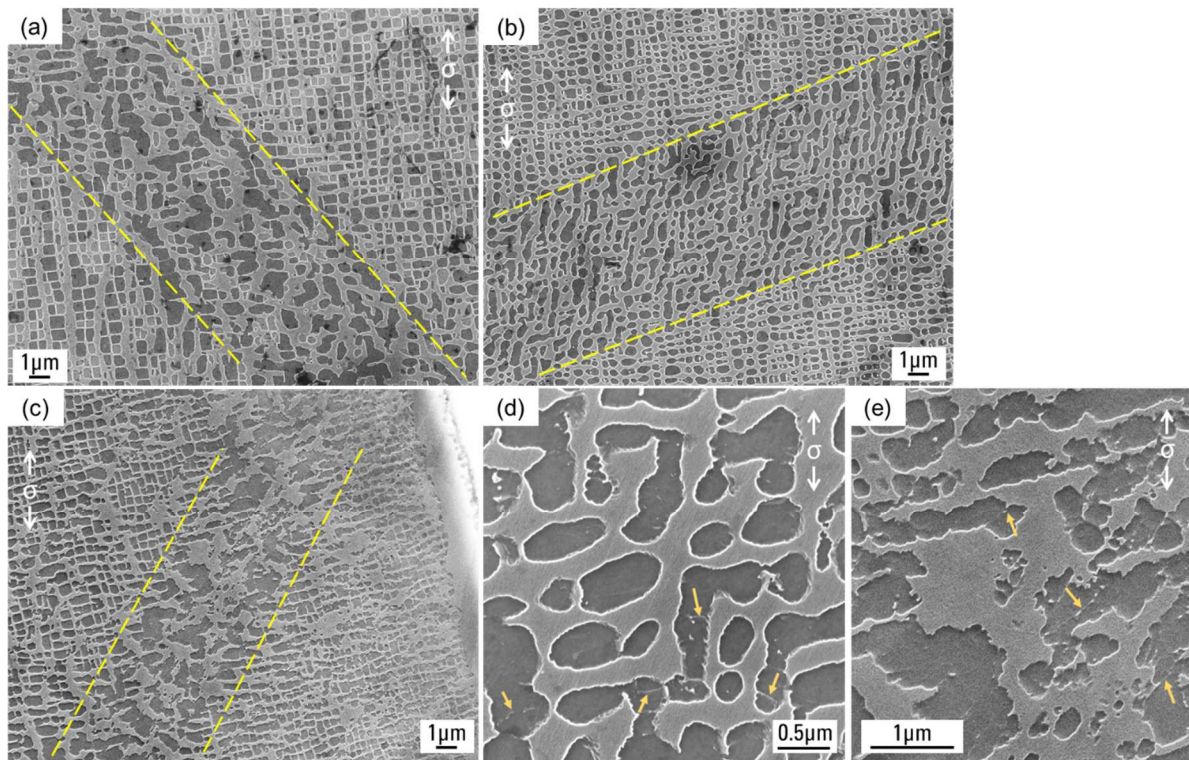
212 The transverse line traces and $\{111\}$ slip bands also appeared on the surface of AM1-HT1 ($R = 0.5 / \sigma_{max} = 582$
 213 MPa), AM1-RTPD-HT1 at lower applied stress ($R = 0.5 / \sigma_{max} = 413$ MPa in Figure 8(b)), and
 214 AM1-RTPD-Rejuv-HT1 ($R = 0.5 / \sigma_{max} = 573$ MPa in Figure 8(c) and $R = 0.5 / \sigma_{max} = 454$ MPa in Figure 8(d)).
 215 AM1-HT1 and AM1-RTPD-Rejuv-HT1 had by far a lower number of $\{111\}$ slip bands compared to
 216 AM1-RTPD-HT1. Surface micro-cracks were also observed in some of those specimens at the intersection of two
 217 slip bands; however, the failure came from an internal crack initiation site (i.e., an S-pore). Non-fatal surface
 218 cracks that do not lead to the failure have been reported in the thesis by Cervellon [19].
 219



220
 221 Figure 8. Appearances of the VHCF specimens tested at 1000 °C, $f = 20$ kHz, $R = 0.5$. AM1-RTPD-HT1 at $\sigma_{max} =$
 222 620 MPa (a), AM1-RTPD-HT1 at $\sigma_{max} = 413$ MPa (b), AM1-RTPD-Rejuv-HT1 at $\sigma_{max} = 582$ MPa (c),
 223 and AM1-RTPD-Rejuv-HT1 at $\sigma_{max} = 454$ MPa (d). Black arrows are pointing at $\{111\}$ slip traces.
 224

225 C) Microstructure characterization of the specimen's longitudinal sections

226 Precipitate scale observations of the microstructure coarsened bands are shown in Figure 9. Figures 9(a, b, d) were
 227 obtained by observing a primary dendrite core inside of the specimen. γ' precipitates in the specimen tested at $R =$
 228 $-1 / \sigma_a = 209$ MPa (Figure 9(a)) had coarsened more than the ones in the specimen tested in $R = 0.5$ (Figure 9(b)),
 229 which is simply because the overall duration of this test was longer. Although microstructure evolution had not
 230 proceeded much, dislocation traces were observed in the microstructure coarsened band of the specimen tested
 231 with $R = 0.5 / \sigma_{max} = 551$ MPa (magnified image in Figure 9(d)), whereas they were not observed in $R = -1 / \sigma_a =$
 232 209 MPa (Figure 9(a)). A notable difference between dendritic cores/interdendritic areas at the bulk has not been
 233 investigated.
 234

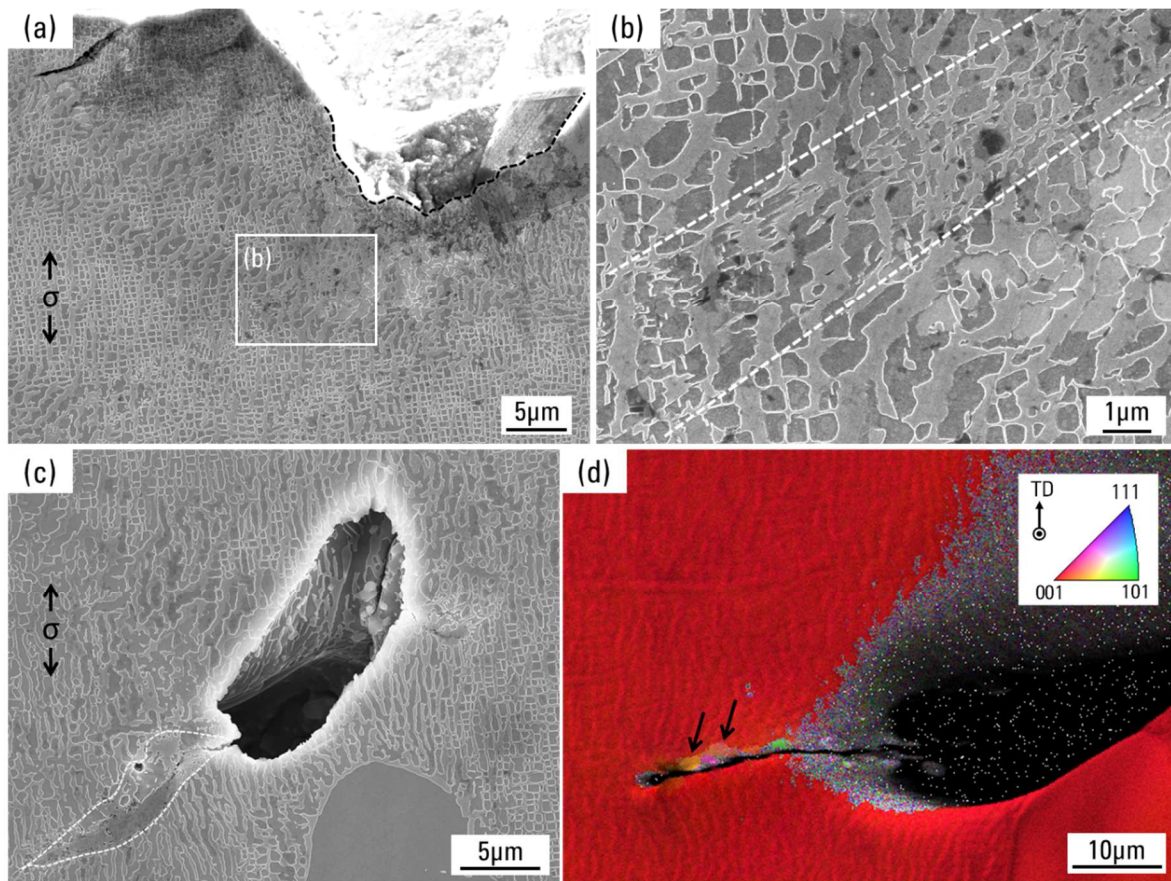


235
 236 Figure 9. Precipitate scale microstructure observed in longitudinal cross section of AM1-RTPD-HT1 specimens
 237 after tests at 1000 °C, $f = 20$ kHz. (a) $R = -1$, $\sigma_a = 209$ MPa, failed at 9.8×10^7 cycles, (b-e) $R = 0.5$,
 238 $\sigma_{max} = 551$ MPa, $N_f = 1.0 \times 10^6$ cycles. Microstructure inside the specimen and that close to the

239 specimen's surface are shown in (b) and (c), respectively. (d) and (e) are magnified images inside the
 240 microstructure coarsened band of (b) and (c), respectively. Arrows are pointing at dislocation traces in
 241 the γ' phase. Dotted lines are indicating the microstructure coarsened bands.
 242

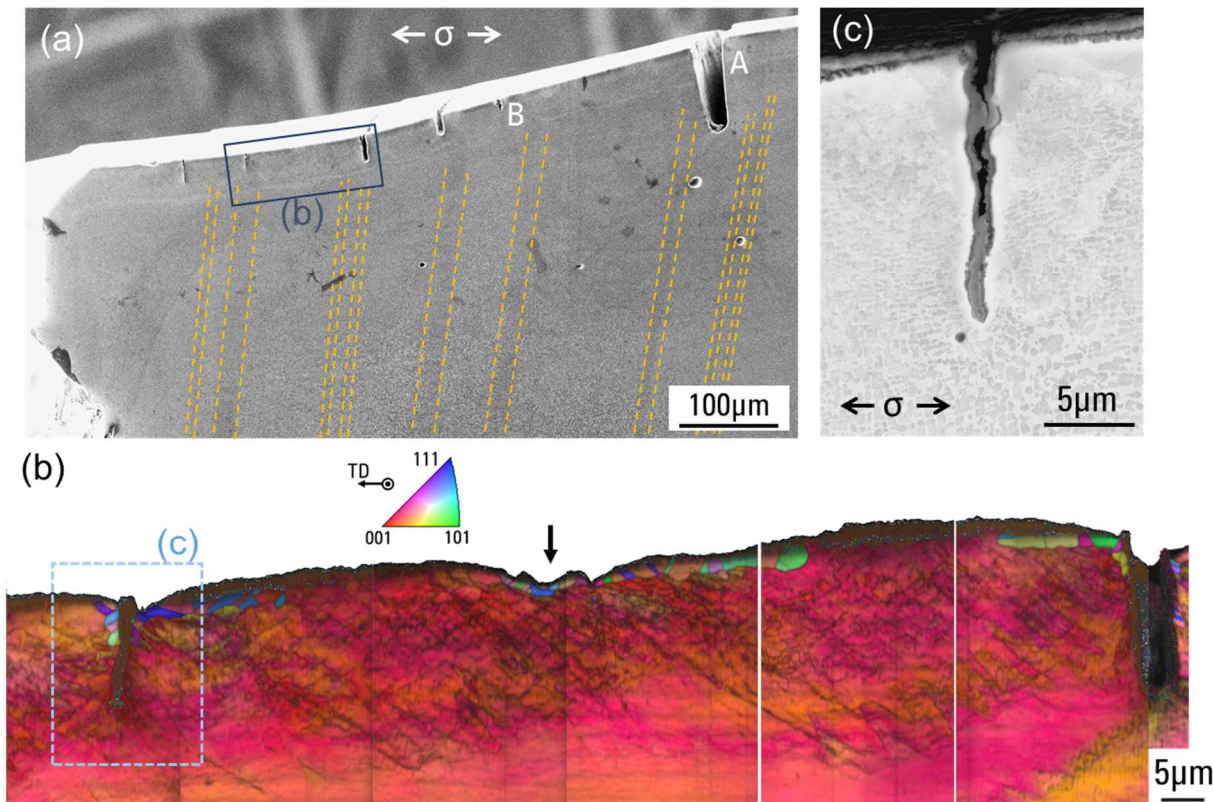
243 Close to the specimen's surface, microstructure coarsening proceeded much faster inside the band (Figure 9(c))
 244 with a very high density of dislocation traces (Figure 9(e)). Because of heavy deformation close to the surface, the
 245 dendritic structure could not be identified. These specimens did not have intermetallic phases other than γ matrix
 246 and γ' precipitates (i.e., TCP phases). AM1 is indeed known to be an alloy with a very stable microstructure
 247 without TCP precipitation after over 400 h of creep test at 1050 °C [15].
 248

249 Figures 10(a, b) are showing γ/γ' microstructure around an S-pore on the fracture surface associated with a rough
 250 zone. Like in previous studies [1,4], a high density of shear traces and disturbed γ/γ' microstructure were observed
 251 below the rough zone (Figure 10(b)). Figure 10(c) is an example of an S-pore where cracks are propagating along
 252 recrystallized grains (see Figure 10(d)), which will become the rough zone and can be considered as an
 253 intermediate stage before failure [4].
 254



255
 256 Figure 10. Microstructure observed along longitudinal cut of AM1-RTPD-HT1 specimen tested at 1000 °C, $f = 20$
 257 kHz, $R = -1$, $\sigma_a = 209$ MPa. (a) The area near the fracture surface with S-pore exhibiting the rough zone
 258 indicated by the dotted curve. (b) Magnification of the rectangle in (a) showing microstructure shearing
 259 in between dotted lines. (c) S-pore inside the specimen. The dotted curve is showing the evolved
 260 microstructure with crack propagation. (d) IPF map of the area around the crack in (c) superimposed on
 261 forward scatter detector signal. Arrows are showing recrystallized grains along the crack path.

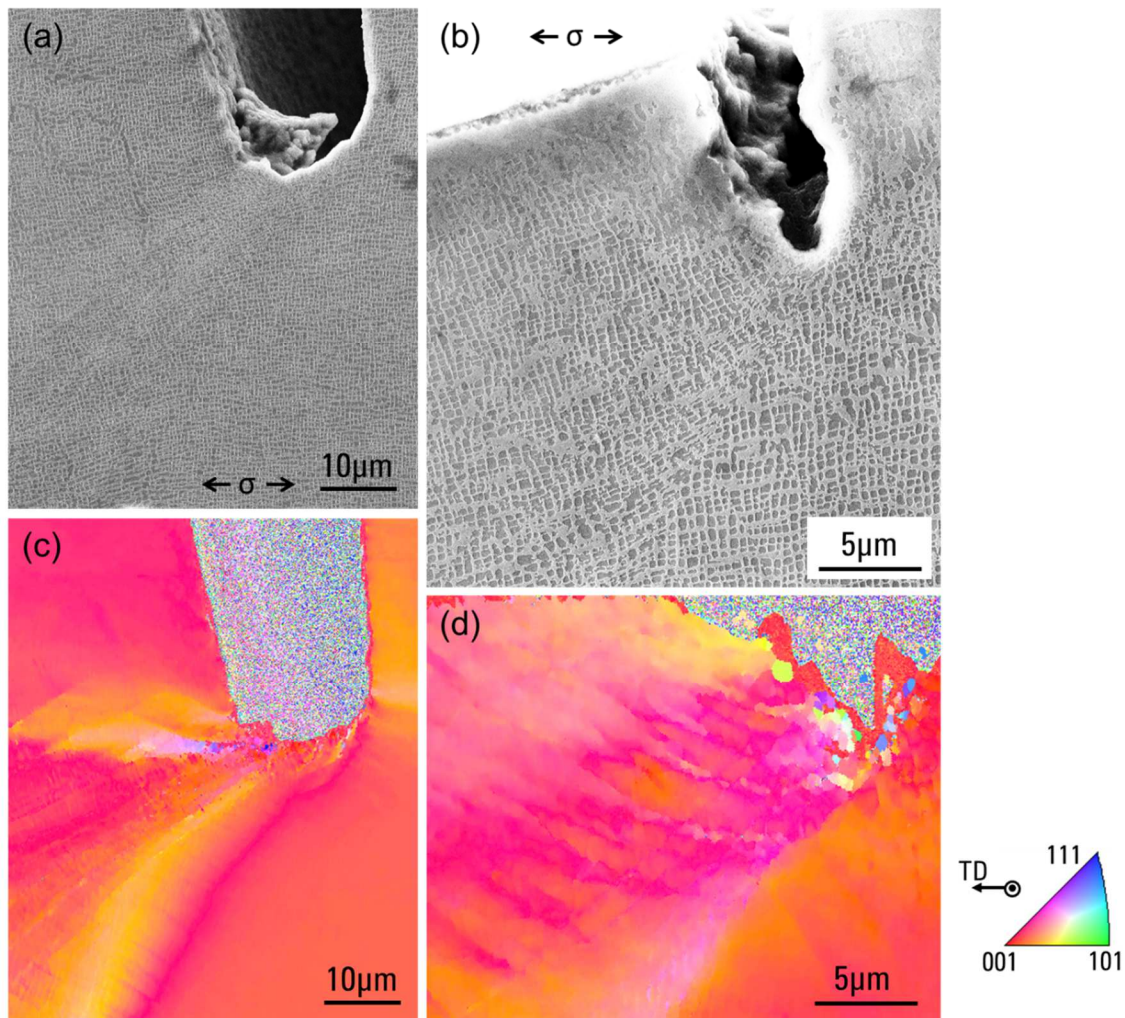
262
 263 Figures 11(a) is showing the low magnification microstructure at the surface of AM1-RTPD-HT1 specimen tested
 264 at 1000 °C, $f = 20$ kHz, $R = 0.5$, $\sigma_{max} = 551$ MPa (cutting plane before polishing is shown in Figure 7(a)). Despite a
 265 very short test duration (~ 50 s), multiple surface cracks were observed, as well as the microstructure coarsened
 266 bands indicated by dotted lines in the same figure to help the reader. Figure 11(b) is a crystallographic orientation
 267 map superimposed to an image quality (IQ) map obtained by EBSD analyses. Three surface recrystallization areas
 268 are in this IPF + IQ map. A darker area in an IQ map corresponds to an area with a higher dislocation density that
 269 decreases diffraction quality. Two areas on the right and left are associated with initiated cracks. The recrystallized
 270 area located in the middle is a potential third crack initiation site (see arrow in Figure 11(b)). The specimen's
 271 surface layer of ~ 30 μm in thickness has dark areas and lines, as observed in Figure 11(b). The bands with
 272 disturbed microstructure, like the one shown in Figure 9(c), are observed in this layer. Figure 11(c) shows the
 273 crack filled with oxide and surrounded by the γ' -depleted zone all along the crack path up to the tip.
 274



275
 276 Figure 11. Microstructure observed along longitudinal cross section ($\sim (110)$ plane) of AM1-RTPD-HT1 specimen
 277 tested at 1000 °C, $f = 20$ kHz, $R = 0.5$, $\sigma_{max} = 551$ MPa. (a) Low magnification image taken from the
 278 area with surface cracks. Dotted lines are indicating the microstructure coarsened bands. (b) Merged
 279 IPF + IQ maps at the surface layer indicated by the rectangle in (a). Arrow is pointing at the
 280 recrystallized area without crack. (c) Backscatter electron image of the area indicated by the rectangle
 281 in (b).
 282

283 Other cracks marked as A and B in Figure 11(a) are presented in Figure 12 with higher magnification and IPF
 284 maps. These cracks have a larger opening (> 3 μm) with crack tips not filled with oxides, whereas the oxides are
 285 filling the crack with a smaller opening (Figure 11(c), ~ 1 μm). The crack on the right in Figure 11(b) has the same

286 features as cracks A and B (i.e., large opening and no oxide filling). Recrystallized grain was not observed around
 287 crack A with the largest opening (Figure 12(c)). Lattice rotation at the tip of this crack A indicates the existence of
 288 extreme stress concentration. The IPF map in Figure 12(d) shows recrystallized grains surrounding crack B. At the
 289 tips of both cracks A and B, slip bands can be observed in secondary electron images and IPF maps. When these
 290 IPF maps are superimposed to an IQ map, slip bands appear as dark lines due to a high dislocation density. It
 291 should be noted that slip bands in the sub-surface layer and at the crack tips are different from the slip bands
 292 introduced during pre-deformation. All of these slip bands are different from the microstructure coarsened bands
 293 that formed during aging treatments.
 294



295
 296 Figure 12. Microstructure observed along longitudinal cross section ($\sim (110)$ plane) of AM1-RTPD-HT1 specimen
 297 tested at $1000\text{ }^{\circ}\text{C}$, $f = 20\text{ kHz}$, $R = 0.5$, $\sigma_{max} = 551\text{ MPa}$, failed at 1.0×10^6 cycles. (a, b) Secondary
 298 electron images showing γ/γ' microstructure. (c, d) IPF maps in the same area as (a, b). (a, c) and (b, d)
 299 are corresponding to the cracks indicated as A and B in Figure 11(a), respectively.
 300

301 4. Discussion

302 A) Effect of pre-deformation in fully reversed condition $R = -1$

303 In the previous study, stereological analyses were performed on void/pore size and counts in AM1-RTPD-HT1

304 [15]. Increase in void size and density induced by pre-deformation before aging treatments has been evidenced
305 [15]. Since void/pore size is the critical factor controlling the VHCF durability in the Ni-based SX superalloys
306 under fully reversed condition [2,5], the pre-deformed material having a lifetime similar to non-pre-deformed
307 material seems contradictory.

308
309 Recalling that both reference and AM1-RTPD-HT1 are produced by the Bridgman SX casting method with similar
310 casting parameters, primary dendrite arm spacings, S-pores, and eutectics are assumed to be comparable in these
311 materials in the as-solutioned state. Thus, the probability of having a critical S-pore within the gauge center
312 section of the VHCF specimens are equivalent. In the previous study, larger voids' counts and size distribution are
313 not significantly different among the two materials, AM1-HT1 and AM1-RTPD-HT1 [15]. Diameter of S-pores in
314 AM1-RTPD-HT1 was within the range of reference data taken from the previous study [2], as described in the
315 experimental procedures. Moreover, it has already been shown that increased void size by over-aging did not
316 affect the VHCF life of CMSX-4 tested under similar testing conditions [1]. An increased number of smaller
317 H/D-pores by pre-deformation (compare Figures 3(a, b)) is not an essential factor controlling the VHCF life of
318 Ni-based SX superalloys under fully reversed conditions, in good agreement with a previous analysis by
319 Cervellon et al. [1].

320
321 The fracture surface of all AM1-RTPD-HT1 specimens after VHCF tests exhibited either a {111} plane or pattern
322 of the microstructure coarsened band. Moreover, in the fully reversed VHCF conditions, multiple S-pores were
323 exposed on the fracture surface with the surrounding rough zone (Figure 6) and were connected to microstructure
324 coarsened bands. These imply that, although pre-deformation may not affect the initiation of the fatal crack and
325 overall durability in these conditions, the microstructure coarsened bands and pores lying on the bands remained
326 as preferable damaging sites that assist crack propagation up to the final fracture. By increasing the duration of
327 VHCF tests at $R = -1$ (tests in excess of 10^{10} cycles), an effect of pre-deformation on VHCF life might be
328 observed, resulting from a prolonged time to allow sufficient microstructure evolution within the coarsened bands.

329
330 In the previous studies performed at Institut Pprime, it was almost impossible to identify secondary cracks outside
331 of the main crack initiation site of specimen tested at $R = -1$ [1–5,11]. Once again, slip bands intersecting with
332 S-pores can help pore growth during subsequent aging treatment, but it did not change pore size drastically. Slight
333 difference in S-pore size was confirmed to be negligible because AM1-RTPD-HT1 showed similar VHCF
334 performance to the non-pre-deformed AM1. Contrarily to the previous studies, several pores were triggering
335 recrystallization in AM1-RTPD-HT1 at $\sigma_a = 209$ MPa, like in Figure 10(c). Those pores were mostly connected to
336 the microstructure coarsened band. The γ/γ' microstructure around this pore and the recrystallized area is coarser
337 compared to the other areas. The micro shearing had probably started from the pore, and it created a local intense
338 strain field. Once the local plastic deformation reaches a certain amount, it accelerates the microstructure
339 evolution and activates the recrystallization. Cervellon et al. have already presented this mechanism in the same
340 VHCF conditions [4]. In the non-pre-deformed specimens, S-pore with surrounding recrystallized area was only
341 present at the fracture surface [2].

342

343 From the present study, it is suggested that coarsened and degraded γ/γ' microstructure surrounding S-pore favors
344 the primary process (microstructure shearing) of a crack initiation. Some S-pores in the non-pre-deformed
345 material have coarse microstructure nearby due to sub-grain boundaries and eutectics. Indeed, S-pore connected to
346 the microstructure coarsened band is a favorable site for this crack initiation process in the pre-deformed material.
347 These degraded γ/γ' microstructures may have locally higher dislocation density before applying load that assisted
348 the shearing process [8], which is different from pre-rafted microstructure that does not affect VHCF life at the
349 same conditions [1]. Therefore, the number of cycles required to initiate a fatal crack is the same regardless of
350 pre-deformation; however, the number of potential primary crack initiating pore is increased by the
351 pre-deformation.

352

353 B) Enhanced shearing and recrystallization at positive stress ratio condition, $R = 0.5$

354 In previous studies, Cervellon et al. have shown that the γ/γ' microstructure (morphology and size) controls the
355 VHCF life at 1000 °C of Ni-based SX superalloys when a high mean stress is superimposed to the alternating
356 stress [1,11]. VHCF damages include a creep contribution when the material is exposed to high-temperature and a
357 tensile stress for all testing periods [11,20]. At a very high strain rate in the tensile deformation, a degraded γ/γ'
358 microstructure presents lower yield stress than the optimal cuboidal microstructure [1,21–24]. In the present study,
359 the microstructure coarsened bands have a higher dislocation density both in bulk (Figure 9(d)) and especially at
360 the surface layer (Figure 9(e)). In addition, during creep at 850 °C / 500 MPa or 1050 °C / 200 MPa,
361 microstructure shearing occurred much more frequently inside the microstructure coarsened bands [14]. Therefore,
362 fatigue damage mechanisms can proceed faster inside the microstructure coarsened bands when the creep damage
363 is active, and the VHCF life of pre-deformed AM1 may decrease at high mean stress conditions. Furthermore,
364 initial dislocations introduced by a combination of pre-deformation and subsequent aging treatments can enhance
365 dislocation penetration from the matrix into the precipitates that accelerate the damaging process.

366

367 The shorter VHCF lifetime of AM1-RTPD-HT1 compared to AM1-HT1 at the conditions $R = 0.5 / \sigma_{max} = 551$
368 MPa and $\sigma_{max} = 620$ MPa is clearly the consequence of pre-deformation. However, unlike other specimens that
369 exhibited internal crack initiation, AM1-RTPD-HT1 presented multiple cracks from the surface and did not have
370 an internal crack from the creep void. The reason for the absence of creep damage is because fatigue loading time
371 was so short (less than 1 min) that the γ' -rafting and creep void growth did not have sufficient time to occur
372 (Figure 9(b)). A unique feature of AM1-RTPD-HT1 is a greater number of the {111} slip band traces shown in
373 Figures 7(d) and 8 compared to that in either AM1-HT1 or AM1-RTPD-Rejuv-HT1, although the test duration of
374 AM1-RTPD-HT1 was by far shorter at high mean stress condition. With a very high density of dislocations at the
375 surface layer shown in Figure 11(b) and a high density of the slip band traces on the surface, the sub-surface
376 microstructure can be considered as the key in this specimen's fatigue failure.

377

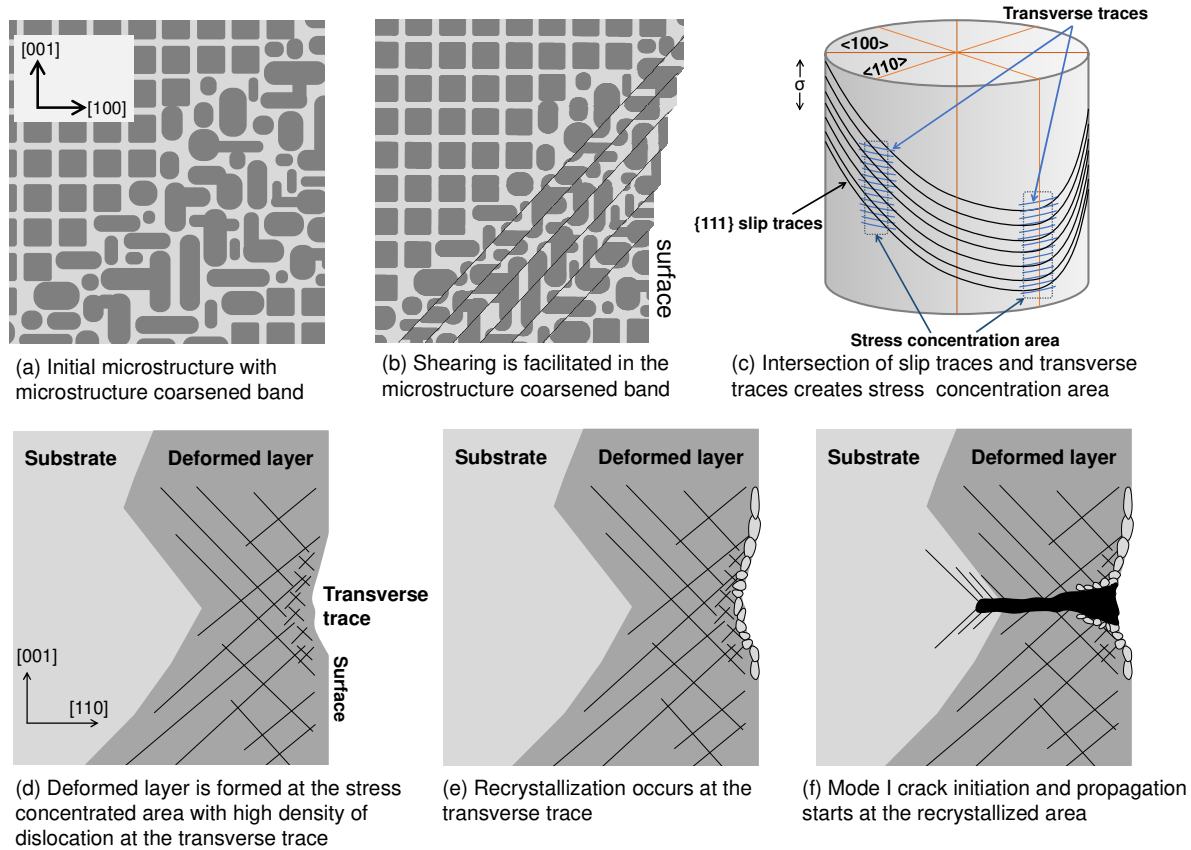
378 The average spacing between transverse traces in Figure 7(a) is 66 μm , and that of cracks observed in Figure 11(a)
379 is 71 μm (recrystallized area pointed by the arrow in Figure 11(b) is counted as a “close-to-initiated-crack”).
380 Recrystallized layers in Figure 11(b) are observed as the transverse traces at lower magnification. These facts

381 confirm that the surface recrystallizations in the pre-deformed specimen are linked to the transverse traces. The
382 transverse band traces along $\sim \langle 110 \rangle$ type generatrices formed in all specimens tested at $R = 0.5$ as a result of
383 preferential slip accumulation and stress concentration created by the alternating stress, and possibly because of
384 elastic anisotropy of SX specimen [25,26]. Strongly marked $\{111\}$ slip traces and their intersection with the
385 transverse traces increase the specimen's surface roughness. Hence, intersection of two slip traces on the surface
386 of the pre-deformed material suffers intense stress concentration and triggering surface recrystallization that leads
387 to very rapid failure.

388
389 The recrystallized layer in Figure 11(b) is limited to only one-grain thickness. Two deeper cracks in Figure 11(b)
390 and the largest one in Figure 12(c) do not have recrystallized grains up to their crack tips. In the same two cracks
391 observed in Figure 11(b), the recrystallized layer below the surface along the crack path has a depth of $\sim 10 \mu\text{m}$.
392 This recrystallization depth is the same as the crack depth shown in Figure 12(d), which has recrystallized grains
393 at its tip. This recrystallized depth suggests that recrystallization causes crack initiation and mode I crack opening
394 at the surface, but it is not necessary for cracks to propagate further. Crack tip plasticity shown as slip bands in
395 Figure 12(a, c) is a typical one observed in mode I crack transitioning into mode II crack [27,28] and is enough to
396 ensure further crack propagation. Once the crack penetrates the deformed layer, crystallographic cracking take
397 place until failure. Fracture surface in Figures 5(b) and 7(a, b) is showing fracture planes similar to what has been
398 shown in the literature [29–32].

399
400 Figure 13 schematically represents the crack initiation mechanism that occurred in AM1-RTPD-HT1 at $1000 \text{ }^\circ\text{C}$, f
401 $= 20 \text{ kHz}$, $R = 0.5$, $\sigma_{max} = 551 \text{ MPa}$ and $\sigma_{min} = 620 \text{ MPa}$. The microstructure coarsened bands lying on $\{111\}$ plane
402 facilitates shearing (see microstructure in Figure 9(c-e) and schematic illustration in Figure 13(b)). This leads to
403 $\{111\}$ slip traces at the specimens' surface, as observed in Figure 7(d). Slip trace intersection with the specimen's
404 $\langle 110 \rangle$ generatrix leads to a greater stress concentration due to the slip accumulation and the creation of a locally
405 greater roughness (Figures 13(b, c)). The deformed layer is created underneath the stress concentration area, and a
406 high density of dislocations is accumulated at the transverse trace (Figure 13(d)). Recrystallization is subsequently
407 triggered at the surface layer (Figure 13(e)), which can be assisted by oxidation (there is also 45 min of soak at
408 $1000 \text{ }^\circ\text{C}$ before the application of the alternating/mean stress used for specimens' emissivity stabilization, ensuring
409 a reliable temperature control during the tests) that leads to an Al depletion and very local γ' dissolution [33,34].
410 Finally, mode I crack opening starts at the recrystallized area and propagates like other typical fatigue mode I
411 cracks (Figure 13(f)) [27,28]. Once these mode I micro-cracks become long enough, they bifurcate into a mode II
412 crack, and specimen ultimately fails with crystallographic cracking (see fracture surface in Figure 5(b)) [29–32].

413



414

415 **Figure 13. Schematic illustration of the crack initiation mechanisms in pre-deformed Ni-based SX superalloys at**
 416 **high temperature and high applied mean stress VHCF test.**

417

418 During isothermal creep tests, local microstructure shearing in the band was more apparent in the creep conditions
 419 with higher stress [14]. This is consistent with the fact that shearing inside the band was facilitated in the
 420 pre-deformed material during the VHCF test at a positive ratio condition with high maximum stress. According to
 421 a previous study from the authors, time dependent creep damage should be accumulated faster inside the
 422 microstructure coarsened band and causes earlier failure [15]. However, at the lower maximum stress condition
 423 (~400 MPa), the VHCF lifetime was not affected by the pre-deformation in the S-N diagram (Figure 2(b)). Fatigue
 424 tests with a lower applied mean stress can be an option to clarify the possibility of localized creep damage
 425 accumulation within the band. Decreasing the testing frequency can be another option to check this possibility;
 426 however, a lower strain rate may change the active deformation and damage mechanisms [11]. Overall, the
 427 difference in VHCF life between pre-deformed and non-pre-deformed specimens at this condition ($\sigma_{max} \sim 400$
 428 MPa) remains within the standard variability observed among all the data generated at Institut Pprime using
 429 different Ni-based SX superalloys and this machine (over 300 VHCF test results at 1000 °C, including different R
 430 ratios).

431

432 Another remaining question is whether the pre-deformation effect on the VHCF life gradually increases with an
 433 increase of σ_{max} from ~400 MPa to ~550 MPa or if there is a critical mean stress that activates a transition in
 434 failure mechanism from an internal to a surface site according to the mechanism shown in Figure 13. Earlier in
 435 this section, we explained that facilitated shearing in the microstructure coarsened band is an important factor of

436 decreased VHCF life. Several studies on Ni-based SX superalloys have shown that the yield strength becomes
437 more sensitive to the alloy's precipitate size as the strain rate increases [1,21–24]. Because coarsened
438 microstructure locally has lower shear resistance, pre-deformed material should undergo higher plastic strain
439 magnitude within the microstructure coarsened bands, creating rough surface that critically affects fatigue life.
440 This is also a reason why only few slip traces were observed very close to the fracture surface of AM1-HT1 and
441 AM1-RTPD-Rejuv-HT1 (see Figure 8). Meanwhile, AM1-RTPD-HT1 at lower mean stress presented more {111}
442 slip traces (Figure 8(b)), which means slip activation is still favorable but without any impact on the fatigue life.
443 Surprisingly, AM1-RTPD-HT1 at $\sigma_{max} = 560$ MPa performed better than at $\sigma_{max} = 551$ MPa. The specimen tested at
444 $\sigma_{max} = 560$ MPa presented both internal and surface cracks (Figure 5(d)). This means that the two failure modes
445 (internal and surface) are competing, but the surface crack did not grow enough to further control the ultimate
446 failure. It is therefore suggested that $\sigma_{max} \sim 550$ MPa could be border condition above which the failure mechanism
447 explained in Figure 13 become active. AM1-RTPD-HT1 at $\sigma_{max} = 620$ MPa had high density of slip traces near the
448 fracture surface (Figure 8(a)), meaning that γ/γ' microstructure shearing is strongly favorable at a higher maximum
449 stress condition.

450
451 The border condition for drastic fatigue life reduction is possibly determined by the sensitivity to the
452 microstructure shearing and resulting surface roughness that activates surface recrystallization. In another words,
453 at a higher maximum stress condition, microstructure shearing becomes locally more important in coarsened
454 microstructure bands and the local level of microstructure degradation within a coarsened band can be a
455 determining factor of the overall VHCF lifetime at $R = 0.5$, and more generally, at high R ratios at this temperature.
456 A reliable quantification would require a larger number of VHCF tests at around suspected border conditions and
457 it remains a field for further investigation.

458

459 C) VHCF life restoration by rejuvenation heat treatment

460 Like creep properties presented in one of our previous studies [14], rejuvenation treatment after pre-deformation
461 successfully restored VHCF properties in all conditions tested in this study. It was especially effective in the
462 positive stress ratio condition where pre-deformation leads to a debit in VHCF lifetime at high maximum stresses
463 ($\sigma_{max} > 550$ MPa). This result supports the fact that a coarsened microstructure affects fatigue life when a high
464 mean stress is applied at high temperatures [1]. On the other hand, further S-pore growth by additional
465 high-temperature re-resolution treatment and stress relaxation, which is one of the risks using a rejuvenation
466 treatment, had not occurred and rejuvenation showed no effect on the VHCF life of the material. The effectiveness
467 of rejuvenation treatment is demonstrated in VHCF behavior in addition to the creep properties [14].

468

469 5. Conclusions

470 Fatigue properties of AM1 Ni-based single crystal superalloy with prior plastic deformation applied between
471 solution and aging treatment has been tested in the very high cycle domain at 1000 °C using an ultrasonic fatigue
472 machine ($f = 20$ kHz). The main conclusions established in this study are:

- 473 • VHCF lifetime and main crack initiation mechanisms in fully reversed ($R = -1$) conditions were not affected

474 by either the microstructure coarsened band or the increased density of smaller pores in pre-deformed
475 material. Fractographic and microstructural observations on pre-deformed material showed multiple pores
476 presenting the primary process of crack initiation associated with local recrystallization around them.

- 477 • Pre-deformation decreased the VHCF lifetime by more than a decade at a positive stress ratio ($R = 0.5$) and
478 high mean stress condition. The microstructure coarsened band in the pre-deformed material facilitated γ/γ'
479 microstructure shearing at a very high strain rate in the ultrasonic fatigue test, resulting in $\{111\}$ slip traces
480 appearing on the specimens' surface. The accumulated slip along $\langle 110 \rangle$ -type generatrices of the specimens
481 led to a locally higher density of dislocations and stress concentration, hence triggering surface
482 recrystallization. Mode I surface cracks were initiated from the recrystallized area and then transitioned to
483 crystallographic cracking, leading to the earlier failure by nearly one decade compared to the specimen
484 without pre-deformation.
- 485 • Rejuvenation treatment can restore VHCF lifetime at a positive stress ratio condition and does not affect
486 fatigue life in all the other conditions tested in this study.

487

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500

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