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Luciana Maria Bortoluci Ormastroni[1, *], Inmaculada Lopez-Galilea[2], Julian Pistor[3], Benjamin Ruttert[2], Carolin Körner[3], Werner Theisen[2], Patrick Villechaise[1], Fernando Pedraza[4], and Jonathan Cormier[1, *]

[1] Institut Pprime, UPR CNRS 3346, Physics and Mechanics of Materials Department, ISAE-ENSMA, 1 avenue Clément Ader, BP 40109, 86961 Futuroscope-Chasseneuil Cedex, France.
[2]Ruhr-Universitat Bochum, Institut fur Werkstoffe, lehrstuhl Werkstofftechnik, Universitasstr. 150, D-44801 Bochum, Germany.
[3]Friedrich-Alexander-Universität Erlangen-Nürnberg, Department Werkstoffwissenschaften, Lehrstuhl für Werkstoffwissenschaften, Martensstr. 5, D-91058 Erlangen, Germany.
[4]Université de La Rochelle, Laboratoire des Sciences de l'Ingénieur pour l'Environnement (LaSIE UMR-7356 CNRS), Avenue Michel Crépeau, 17042 La Rochelle, cedex 01 – France

*Corresponding authors: luciana-maria.bortoluci-ormastroni@ensma.fr, jonathan.cormier@ensma.fr

Highlights

- Additively manufactured and Bridgman solidified CMSX-4 superalloy have comparable VHCF life under high temperature.
- The superalloy chemistry controls the fatigue life of defect-free specimens.
- Defect-free specimens fails by surface crack initiation.
- The fatigue limit is controlled by the superalloy oxidation resistance at temperature of 1,000 °C.

Abstract

A single crystalline (SX) nickel-based superalloy additively manufactured (AM) by electron beam-based powder bed fusion (PBF-E) was investigated under very high cycle fatigue (VHCF) at 1,000 °C in fully reversed conditions (R_ε = -1). Specimens processed using a classical Bridgman solidification route and the impact of a hot isostatic pressing (HIP) treatment were also considered. It is shown that the fatigue lifetime of the AM specimens is higher or in the same range of the Bridgman processed ones with the same chemical composition. All defect-free AM samples fail by surface initiation with very long VHCF lives. In the absence of metallurgical defects such as grain boundaries or pores, the superalloy chemical stability against oxidation governs VHCF failure.

Keywords: Nickel-based superalloys, additive manufacturing, electron beam-based powder bed fusion, very high cycle fatigue.

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1 Introduction

Nickel-based superalloys in single-crystalline (SX) form are widely used in the hot-sections of aero-engines for the manufacturing of blades and vanes thanks to their excellent mechanical properties at temperatures up to ≈ 90 % of the material's melting temperature [1,2]. During service operations at (very) high temperatures, these components undergo a variety of damages including creep, corrosion, and oxidation [2–4]. The entire replacement of damaged components results in material and time costs. The use of additive manufacturing (AM) techniques allows for the design of turbine blades with complex geometrical designs (e.g., intricate cooling channels) combined with the possibility of refurbishment. Electron beam based powder bed fusion (PBF-E) is one of the methods that can be adopted by the industry in the near future [5]. One of the most promising interests in employing PBF-E to produce Ni-based SX superalloys is to control different processing parameters to obtain a superalloy with specific microstructural characteristics [6].

CMSX-4 is one of the "legacy" Ni-based SX superalloys used in the hottest-sections of civil and military aero-engines as blade material [1,7,8]. It is a γ/γ' superalloy which presents a quite high degree of chemical segregation after casting compared to first generation Ni-based SX superalloys. A specific full heat treatment (HT) with subsequent aging(s) (AG) is required to obtain a desired γ' size and volume fraction so as to optimize mechanical properties, especially monotonic ones [6,9–11]. A collaboration between Cannon-Muskegon, the producer of CMSX-4 alloy, and the research center SFB/TR 103 [12] allows SFB/TR 103 to reproduce this superalloy chemistry for the subsequent preparation of SX PBF-E bars.

Considering that fatigue is responsible for most of the crack initiation events/failure cases of internally cooled blades of aero-engines [13], it is of utmost importance to investigate fatigue properties of a PBF-E processed SX superalloy, especially in the very high cycle fatigue (VHCF) regime in view of using such a processing route for the manufacturing of Ni-based SX airfoils in future aero-engines. Fatigue tests under high and very high temperatures with a lifetime higher than 10^8 cycles are very time consuming using a conventional fatigue machine (≈ 100 Hz). Ultrasonic machines (in the kHz regime) became a solution in order to study the damage mechanisms at very high vibratory frequencies [14]. It is also recalled that HCF/VHCF durability is a certifying criteria of airfoils according to airworthiness authorities (e.g. EASA and FAA) [15–17], as the loading by vibratory stresses turns out to be a matter of safety requirement of aero-engines.

From the authors' very best knowledge, there is only one published study focused on the fatigue properties of a PBF-E SX superalloy in the low cycle fatigue (LCF) regime [9]. In fact, Meid et al. investigated the PBF-E SX performance of CMSX-4 alloy in stress-controlled LCF at 950 °C/0.25 Hz and $R_\sigma = 0.6$ [9]. It was shown in this pioneering study that the PBF-E SX alloy can perform similar to the conventionally processed SX alloy, provided that an adequate heat treatment sequence is performed after the AM process. This result is not surprising considering the fact that in such LCF conditions (high temperature, high mean stress, low frequency), a pronounced cyclic ratcheting is observed, indicating that creep damage is also contributing to the overall LCF durability [9].

In contrast, the present paper focuses on the durability of the same PBF-E SX alloy in the VHCF domain at high temperature/ $R_\varepsilon = -1$. In such conditions, the VHCF life of conventionally processed Ni-based SX superalloys is known to be highly dependent on the solidification parameters [14–20]. In fact, VHCF crack initiation occurs at the largest casting pores in nearly 95 % of the cases at $R_\varepsilon = -1/1000$ °C [15,17,19,20] and only occasionally at the

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surface due to oxidation [15,19,21,22] or due to a prior plastic deformation, inducing surface recrystallization [23]. VHCF life in fully reversed conditions at high temperature is hence really sensitive to the presence of defects such as pores and/or grain boundaries. In the absence of large solidification pores in the PBF-E material due to the AM process itself [24,25], a better fatigue performance is expected and/or different crack initiation mechanisms, but this needs to be investigated and therefore, it constitutes the main objective of this manuscript.

To achieve this aim, the PBF-E CMSX-4 SX samples have been tested with and without hot isostatic pressing (HIP) treatment [15,25,26] in the present investigation. The results were compared with the database available from the literature [15] using a Bridgman-processed CMSX-4 alloy after full HT, established using the same VHCF set-up.

2 Materials and methods

2.1. Chemical composition and heat treatments

The nominal chemical compositions of CMSX-4/Bridgman, ERBO/1 and CMSX-4 atomized powder used to build PBF-E specimens is listed in **Table 1**.

Table 1

CMSX-4/Bridgman, ERBO/1, and CMSX-4 atomized powder nominal chemical compositions (in wt. pct.).

Alloy	**Ni**	**Cr**	**Mo**	**Co**	**W**	**Re**	**Al**	**Ti**	**Ta**	**Hf**
CMSX-4/Bridgman [15]	Bal.	6.5	0.6	9.0	6.0	3.0	5.6	1.0	6.35	0.1
ERBO/1 [27]	Bal.	6.2-6.6	0.5-0.7	9.3-10.0	6.2-6.6	2.8-3.1	5.4-5.75	0.9-1.1	6.3-6.75	0.07-0.12
CMSX-4 Atomized powder [31]	Bal.	6.6	0.6	9.9	6.4	2.9	6.2	1.1	6.6	0.08

The CMSX-4/Bridgman fatigue results are extracted from the literature [15]. For this reason, the HT was described in the previous article [15]. The ERBO/1 plate used to machine the fatigue specimens was provided by Doncasters Bochum company in partnership with the Ruhr-Universitat Bochum. This plate had a 20 mm thickness. The HT (ERBO/1C according to [27]) was presented in the literature [27].

Square bars with 12 mm edges were built (**Fig.1 a**) by the PBF-E method. The PBF-E CMSX-4 SX samples were divided in two different groups depending on whether they have or not a HIP treatment. First, the AM/HT/REC (AM: Additive manufacturing, HT: Heat treated, REC: Recycled atomized powder) specimens were built with recycled powder, HT by standard method in air at 1,315 °C/35 min and finally aged at 1,140 °C/2 h/AQ (air quench) and at 870 °C/20 h/AQ. The AM/HIP (HIP: HIP treatment) specimens were homogenized in the QIH-9 HIP (hot isostatic pressing) at 1,315 °C/35 min under 100 MPa Argon pressure. The material was directly quenched after holding by applying the fast-cooling capability of the HIP set-up to achieve ultrafine γ′ precipitates. Then, the

material was subjected to the same AG treatment as defined for the non-HIPed material. The AM/HIP samples were received in two different batches. The first batch containing eight bars had been built using recycled powder (AM/HIP/REC). The second batch contained four bars fabricated with fresh powder (AM/HIP/FRS) (FRS: Fresh atomized powder). The nomenclature is detailed in **Table 2**.

Table 2

Sample's nomenclature. HT is the denomination for full heat treatment. PBF-E is the denomination for electron beam based powder bed fusion. SHT is the denomination for solution heat treatment. AG is the denomination for aging treatment. AQ is the denomination for air quench. HIP is the denomination for hot isostatic pressing.

Nomenclature	Superalloy/Solidification process/Heat treatment
CMSX-4/Bridgman	CMSX-4 solidified by classical Bridgman method → HT described in the literature [15]
ERBO/1	ERBO/1 solidified by classical Bridgman method → HT described in the literature as ERBO/1C [27]
AM/HT/REC	Additive manufacturing (PBF-E) samples built w/ recycled CMSX-4 atomized powder → SHT in air at 1,315 °C/35 min and AG steps at 1,140 °C/2 h/AQ and 870 °C/20 h/AQ
AM/HIP/REC	Additive manufacturing (PBF-E) samples built w/ recycled CMSX-4 atomized powder → HIP homogenization at 1,315 °C/35 min under 100 MPa Argon pressure
AM/HIP/FRS	Additive manufacturing (PBF-E) samples built w/ fresh CMSX-4 atomized powder → HIP homogenization at 1,315 °C/35 min under 100 MPa Argon pressure

2.2. Stereological analyzes

A γ/γ' stereological analysis was performed using a JEOL JSM-7000F scanning electron microscope, by applying the secondary electron imaging (SEI) mode operating at 25 kV. The microstructures are shown in **Fig. 1 b-g**. The observations have been made in both primary dendrite arms (D) and interdendritic spacing (ID) for Bridgman processed CMSX-4/Bridgman and ERBO/1 alloys. As the PBF-E processed alloy has very small primary dendrite arms spacings [6,31], almost no remaining difference in precipitate size and morphology can be observed in a fully heat treated/HIPed state across this very fine dendritic structure (**Fig. 1 f-g**). The software ImageJ was employed to analyze the γ' size. All measurements were done in the dendrite core for Bridgman solidified alloys. The γ' average edge length of CMSX-4/Bridgman alloy is 590 ± 120 nm, and it is 560 ± 80 nm for the ERBO/1 one. The AM/HT/REC presents 420 ± 80 nm, and AM/HIP/REC 470 ± 100 nm sizes. The average edge length of γ' precipitates in the PBF-E SX is hence smaller compared to the one in CMSX-4/Bridgman and ERBO/1 materials. This difference in γ' size between all the investigated specimens has been neglected in the following of this article since it is known from the prior literature that the VHCF properties of Ni-based SX superalloys are not dependent on the precipitation size and morphology at R_ε = -1/1000 °C [17]. Moreover, a possible small variation in γ' volume fraction around 70 % does not seem neither to have any impact on the VHCF properties in these conditions [15].

The CMSX-4 PBF-E SX stereological characterization detailed in [25,29,30] has shown that the SX part developed pores having a ≈ 1.7 µm average diameter size [24,25], which contrasts with the Bridgman method that results in solidification pores of up to 200 µm after the complete solution heat treatment (SHT) for this alloy [28].

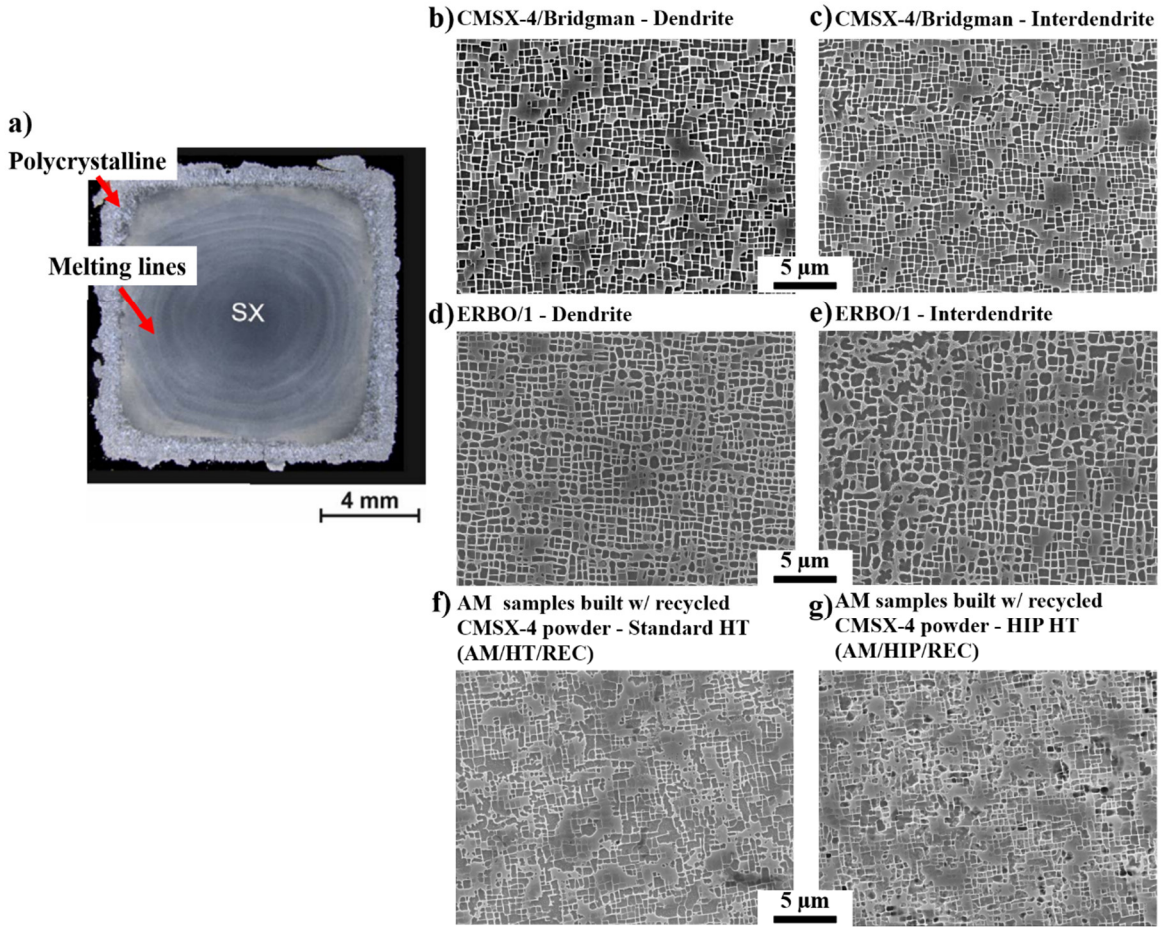

Fig. 1. (a) PBF-E SX 12 mm square bars microstructure details (aqua regia etched). (b) γ/γ' microstructure of CMSX-4/Bridgman observed in primary dendrite arms and (c) in interdendritic spacing. (d) ERBO/1 alloy solidified by a Bridgman method in primary dendrite arms and (e) in interdendritic spacing. (f) CMSX-4 alloy processed by PBF-E after standard heat treatment and (g) after HIP treatment.

2.3. Specimens' machining and very high cycle fatigue tests details

VHCF tests were performed using hourglass-shaped samples (see **Fig. 2**) [32]. The samples were extracted by electric discharge machining and subsequent turning to reach the final shape. Two samples for AM/HT/REC, twelve samples for AM/HIP of the two batches (8 specimens AM/HIP/REC and 4 specimens AM/HIP/FRS) and eight samples for ERBO/1 were machined at Pprime Institute. A thickness of 0.2 mm from the original diameter was first removed by SiC grinding to remove residual stresses inherited from the machining steps. This polishing step was essential to avoid surface recrystallization during the fatigue tests at high temperature. Thereafter, the

surfaces were gently ground and polished to a final mirror polish (1 µm diamond suspension) with the final steps parallel to the loading axes.

Strain-controlled VHCF tests were performed at 1,000 °C (± 2 °C), f = 20 ± 0.5 kHz, R_ε = -1 with an ultrasonic fatigue machine used in previous studies [16–19,21,23,32]. The fatigue tests were performed with a sinusoidal waveform. These tests were performed using alternating stresses allowing i) a direct comparison with conventionally processed Ni-based single crystalline specimens previously tested at Institut Pprime [16–19,21,23,32], ii) a good control of specimen's temperature and iii) failure of specimens in a reasonable time. An induction coil was used as a heating source, the power of which was controlled by a closed loop with a dual-wavelength pyrometer measuring the specimen's central temperature. A pre-heating of 45 min at 1,000 °C was applied before starting the tests to stabilize the surface oxides and resulting surface emissivity. Due to the high density of dislocations stored in the AM processed specimens, especially in the HIPped ones, only low alternating stresses were used to limit the impact of specimens' self-heating on their temperature control. Hence, AM specimens were all tested with an alternating stress σ_a below 180 MPa. With this high temperature VHCF set-up used in these previously described conditions, fluctuations in alternating stress of +/- 3 MPa at maximum have been observed throughout tests.

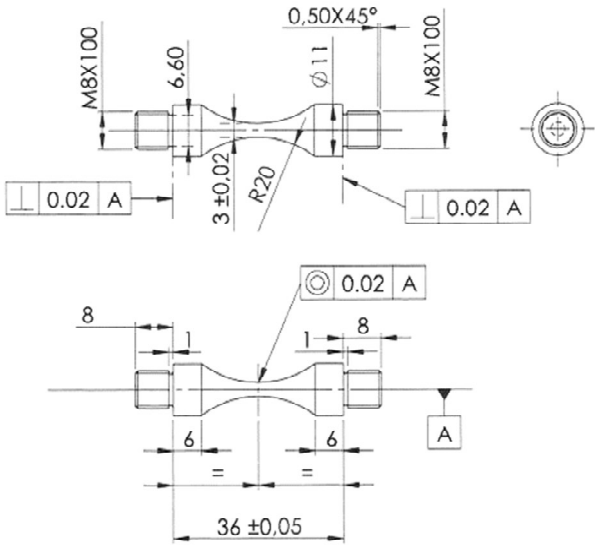

Fig. 2. Ultrasonic fatigue sample dimensions (in mm), used for VHCF tests at 1,000 °C.

2.4. Post-mortem analyses

Optical and scanning electron microscopy (OM and SEM, respectively) were employed to perform post-mortem observations. Fractographic analyses were carried out using a field emission gun (FEG) microscope JEOL JSM-7000F operating at 25 kV. The longitudinal sections were mirror polished down to 1 µm to observe the microstructure under secondary (SEI) and backscattered electrons (BSE) imaging modes in the same equipment operating at ≈10 mm work distance. The chemical analyses were conducted with an EDAX energy dispersive spectrometer (EDS). The analyses were made on un-etched surfaces to avoid any potential dissolution of oxides, carbides and other metallurgical phases that could fake the interpretation. In addition, electron backscatter

diffraction (EBSD) analyses were performed to characterize the crystallographic orientation around the internal crack initiation. It was performed using a second JEOL JSM-6100 SEM, operating at 25 kV, and the orientation imaging microscopy (OIM) software EDAX, version 6.1. A 0.2 µm scanning step was chosen. The specimens were polished using colloidal silica solution (particle size 0.04 µm).

3 Results

3.1. VHCF performances and fractographic analyses

The alternating stress (σ_a) as a function of the number of cycles to failure (Nf) diagram gathering all VHCF results obtained in the present study is presented in **Fig. 3**. It can also be observed that fatigue lives ranging from $\approx 10^6$ cycles up to $\approx 3.0 \times 10^{10}$ cycles have been obtained at σ_a = 145 MPa. The latter is probably the longest fatigue test ever performed on this class of alloy at high temperature. Significant scattering in ERBO/1 can result from different crack initiation mechanisms that will be detailed later in this article.

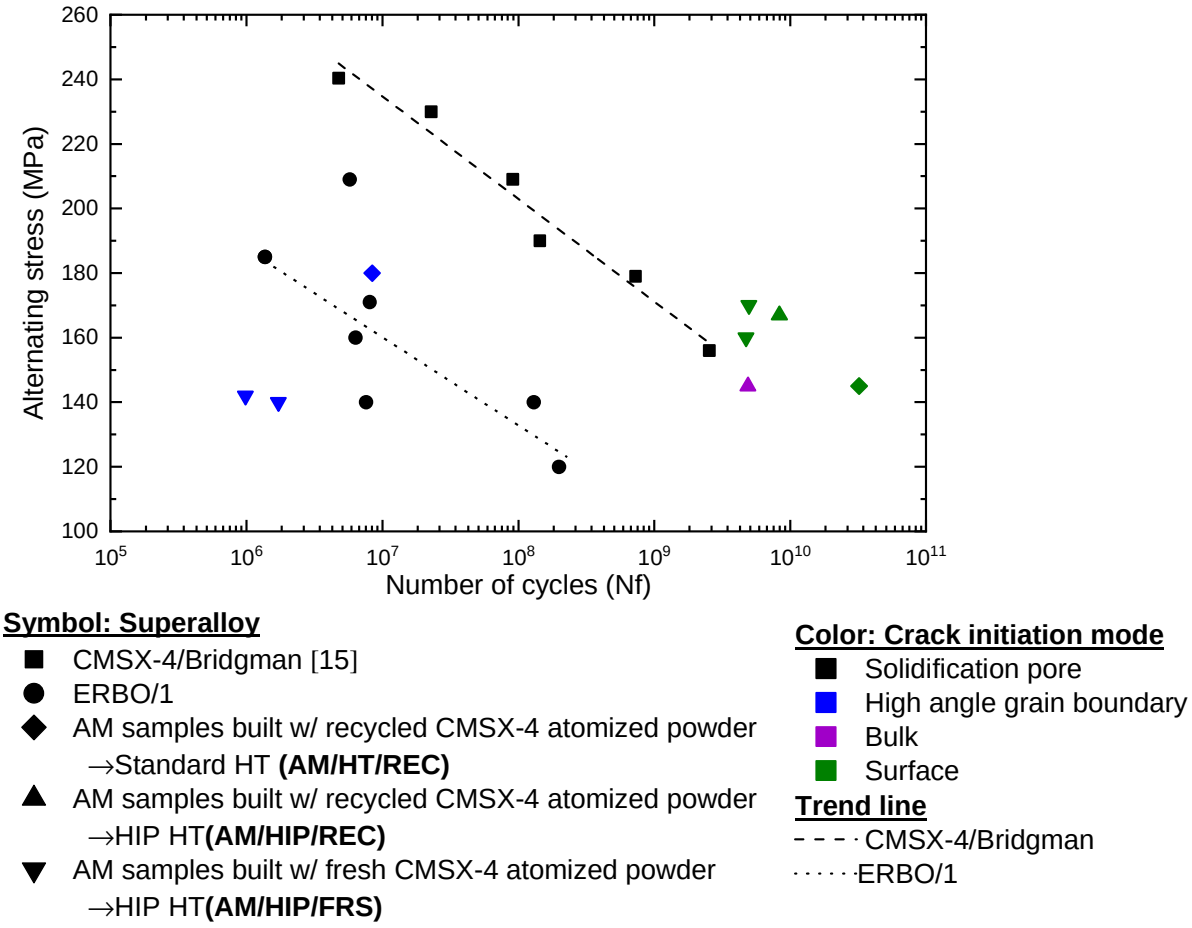

Fig. 3. S-N diagram at 1,000 °C, R_ε = -1 / f = 20 kHz. The alternating stress σ_a is plotted as a function of the number of cycles to failure. The CMSX-4/Bridgman data, represented with black squares, were extracted from the literature [15]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The AM processed specimens present very different VHCF lives, either very long ones in excess of 5.0×10^9 cycles, or short ones below 10^7 cycles. In fact, six AM/HIP/REC samples failed from stray grains, which presumably formed during the PBF-E and/or HIPing processes. The presence of such grains within the gauge part of VHCF specimens, especially close to the surface, results in very low VHCF lives. Failure during the initial ramping-up in alternating stress up to reaching the desired value in σ_a even occurred in six different samples containing stray grains (**Fig. 4** and **Table 3**). These tests were considered as "unsuccessful" since no stabilized alternating stress was reach.

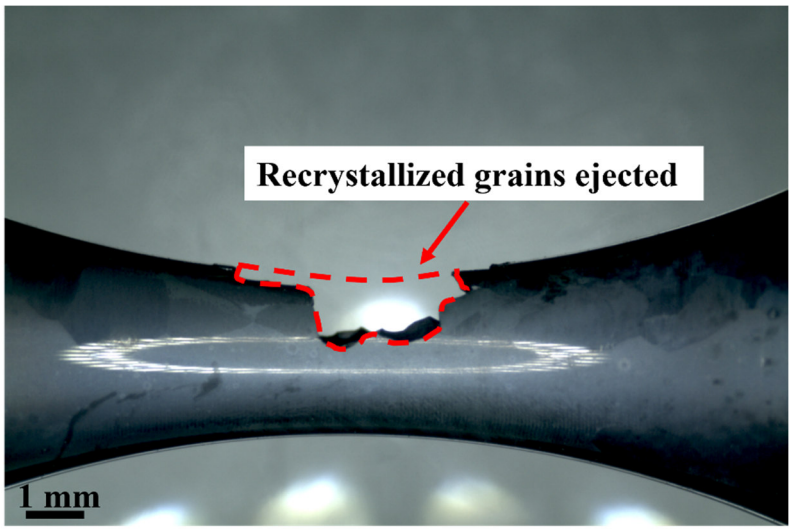

Fig. 4. Optical microscope observations of two AM/HIP/REC samples that presented immediate failure. The red dotted region illustrates the location of a recrystallized grains ejected from the specimen gauge part during the application of the alternating stress.

All other successful tests using PBF-E processed specimens (i.e., AM/HT/REC, AM/HIP/REC and AM/HIP/FRS samples) with a few cycles close to the CMSX-4/Bridgman trend line failed from a surface crack initiation or from an internal site (see **Fig. 3**). The presence of grain boundaries within the gauge part of specimens appears to be detrimental to the VHCF life of AM processed specimens and may lead to fatigue lives well below the ones obtained for Bridgman processed alloys (see **Fig. 3** and **Table 3**).

After mechanical testing, fractographic observations were performed. The fracture analysis of CMSX-4/Bridgman specimens was already presented in detail by Cervellon et al. [15]. Crack initiation from internal solidification pores was systematically obtained, these critical pores having a maximum diameter ranging from 50 to 150 µm [15]. In good agreement with this reference database, ERBO/1 specimens systematically presented a crack initiation from solidification pores with a size ranging from 80 to 200 µm (see **Table 3** and **Fig. 5**). Such a crack initiation mode has already been well explained in the literature [15,20,32]. The quite pronounced scatter in VHCF life of ERBO/1 specimens displayed in **Fig. 3** results from the quite large variation in casting pore size serving as the main crack initiation site (**Fig. 5)**, since specimens were machined out from a thick SX plate. In fact, using the same casting parameters, large SX castings are known to introduce larger solidification pores, overall reducing the VHCF life [15]. Hence, the variability in VHCF life of ERBO/1 does not deserve further analysis/discussion.

Focusing on the successful results shown in **Fig. 3** for PBF-E-processed specimens (i.e., no consideration of specimens containing high angle grain boundaries), most of AM/HT/REC, AM/HIP/REC and AM/HIP/FRS samples presented crack initiation at the surface, as shown in **Fig. 6**. According to **Fig. 6**, surface cracks assisted by oxidation as deep as 0.8 mm have been observed after failure. The fracture surfaces of these specimens are also characterized by a subsequent crack propagation zone in mode I (highlighted by yellow dotted lines), which seems to be less oxidized according to the lighter contrast, and then by a final failure area in shear mode. Specimens with a high VHCF life ($Nf > 4 \times 10^9$ cycles) presented several surface cracks (secondary cracks) that were perpendicular and parallel to the main loading axis, as shown in **Fig. 7** for two different specimens tested at $\sigma_a = 145$ MPa. Both specimens present a non-negligible density of surface cracks. However, the main crack initiation site was not necessarily at the specimen's surface (see **Table 3** and **Fig. 8a**). In fact, only one specimen had crack initiation from an internal site (AM/HIP/REC-2, see **Fig. 8b**) with the characteristic rough zone commonly observed around the main crack initiation site of samples tested under VHCF conditions [15,20], see yellow insert in **Fig. 8b**. However, it was not possible to unambiguously conclude on the nature this crack initiation site from fractographic observations and further observations along longitudinal cuts were thus performed.

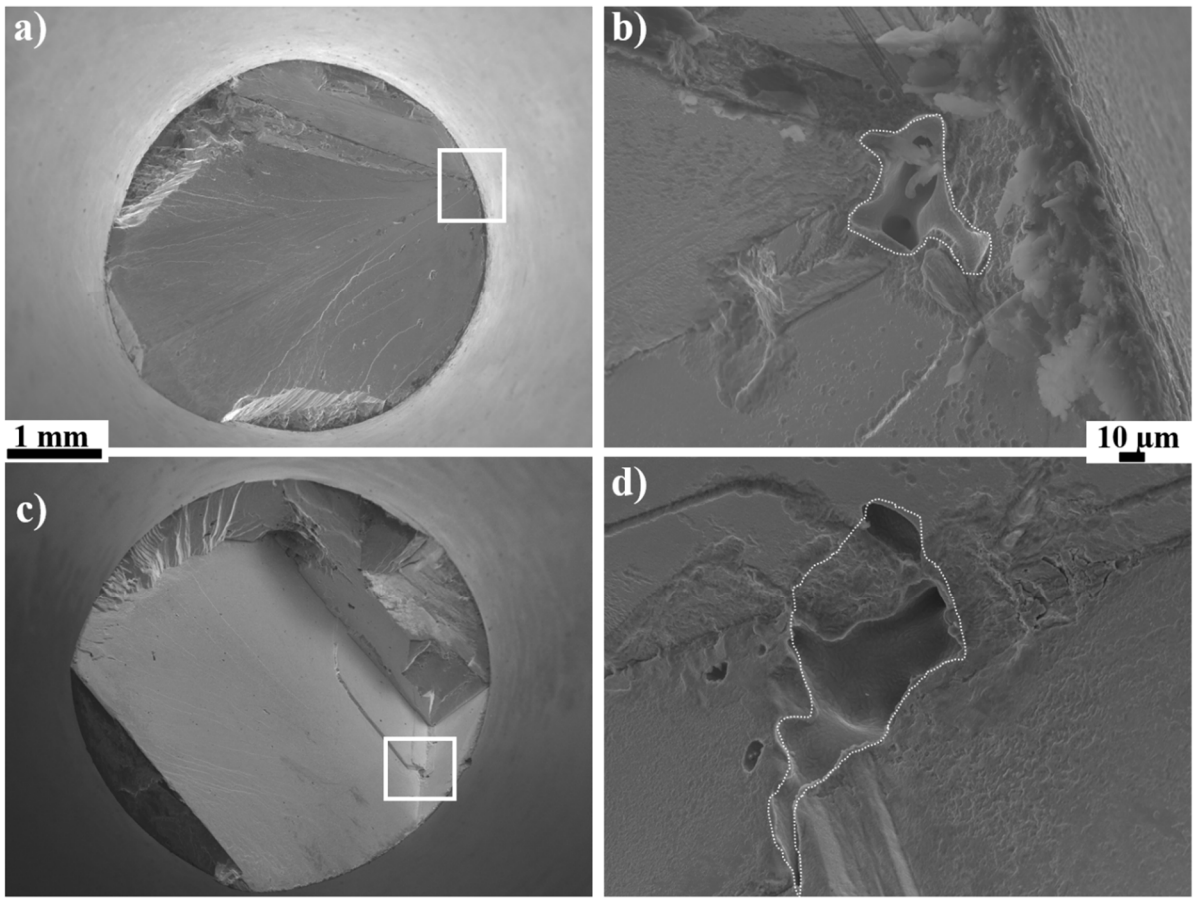

Fig. 5. Fractographic observations of ERBO/1 samples with main crack initiation from solidification pores. (a) ERBO/1-6 ($\sigma_a = 209$ MPa, with $Nf = 5.7 \times 10^6$ cycles) and (b) crack zone magnification, detail to the presence of a casting pore close to the surface with diameter of ≈ 40 µm. (c) ERBO/1-1 ($\sigma_a = 160$ MPa, with $Nf = 6.6 \times 10^6$ cycles) and (d) crack zone magnification, detail to the presence of a casting pore with diameter of ≈ 170 µm.

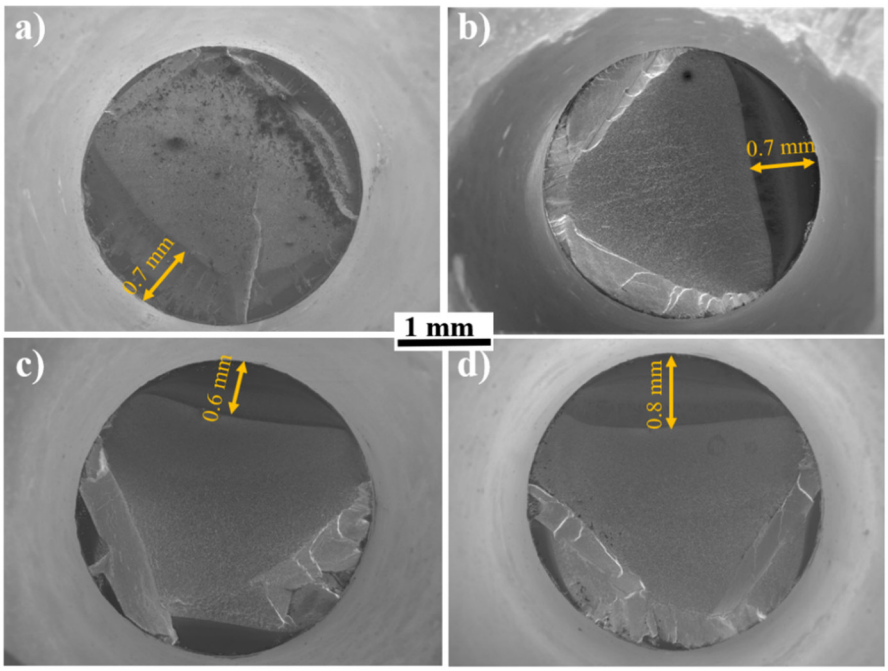

Fig. 6. Fractographic observations of PBF-E processed specimens with main crack initiation from the surface: (a) AM/HT/REC-1 (σ_a = 145 MPa, Nf = 3.2 × 10^{10} cycles), (b) AM/HIP/REC-1 (σ_a = 167 MPa, Nf = 8.3 × 10^9 cycles), (c) AM/HIP/FRS-2 (σ_a = 170 MPa, Nf = 4.9 × 10^9 cycles), (d) AM/HIP/FRS-3 (σ_a = 160 MPa, Nf = 4.7 × 10^9 cycles). The maximum depth of the main fatal crack initiated at the surface is added for each specimen.

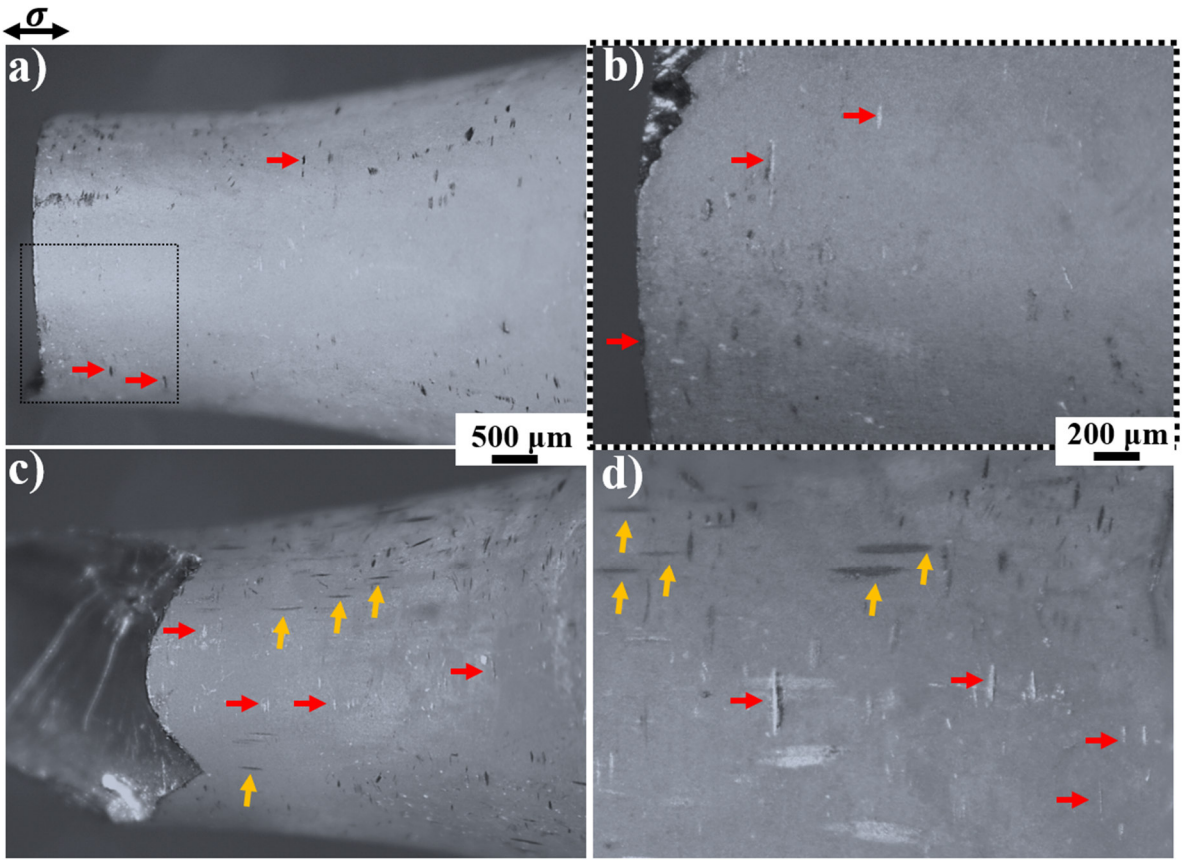

Fig. 7. Two examples of PBF-E processed specimens with surface cracks. (a and b) AM/HT/REC-1 and (c and d) AM/HIP/REC-2. Both tests were performed at σ_a = 145 MPa, with Nf = 3.2×10^{10} cycles for AM/HT/REC-1 and Nf = 4.9×10^{9} cycles for AM/HIP/REC-2. Red and yellow arrows indicate, respectively, cracks perpendicular and parallel to the loading axis. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 3

VHCF results at 1,000°C/R_ε = -1/f = 20 kHz of Bridgman and PBF-E processed ERBO/1 SX specimens.

	Specimen	σ_a (MPa)	Nf (cycles)	Crack initiation
PBF-E	**AM/HT/REC-1**	145	3.2×10^{10}	Surface initiation
	AM/HT/REC-2	180	8.4×10^{6}	High angle grain boundary
	AM/HIP/REC-1	167	8.3×10^{9}	Surface initiation
	AM/HIP/REC-2	145	4.9×10^{9}	Bulk initiation
	AM/HIP/REC-3			Immediate failure during alternating stress ramp-up
	AM/HIP/REC-4			Immediate failure during alternating stress ramp-up
	AM/HIP/REC-5			Immediate failure during alternating stress ramp-up
	AM/HIP/REC-6			Immediate failure during alternating stress ramp-up
	AM/HIP/REC-7			Immediate failure during alternating stress ramp-up
	AM/HIP/REC-8			Immediate failure during alternating stress ramp-up
	AM/HIP/FRS-1	142	9.8×10^{5}	High angle grain boundary
	AM/HIP/FRS-2	170	4.9×10^{9}	Surface initiation
	AM/HIP/FRS-3	160	4.7×10^{9}	Surface initiation
	AM/HIP/FRS-4	140	1.7×10^{6}	High angle grain boundary
Bridgman	**ERBO-BR-1**	160	6.3×10^{6}	Solidification pore
	ERBO-BR-2	140	7.6×10^{6}	Solidification pore
	ERBO-BR-3	120	1.9×10^{8}	Solidification pore
	ERBO-BR-4	171	8.0×10^{6}	Solidification pore
	ERBO-BR-5	140	1.3×10^{8}	Solidification pore
	ERBO-BR-6	209	5.7×10^{6}	Solidification pore
	ERBO-BR-7	185	1.3×10^{6}	Solidification pore
	ERBO-BR-8	185	2.6×10^{7}	Solidification pore

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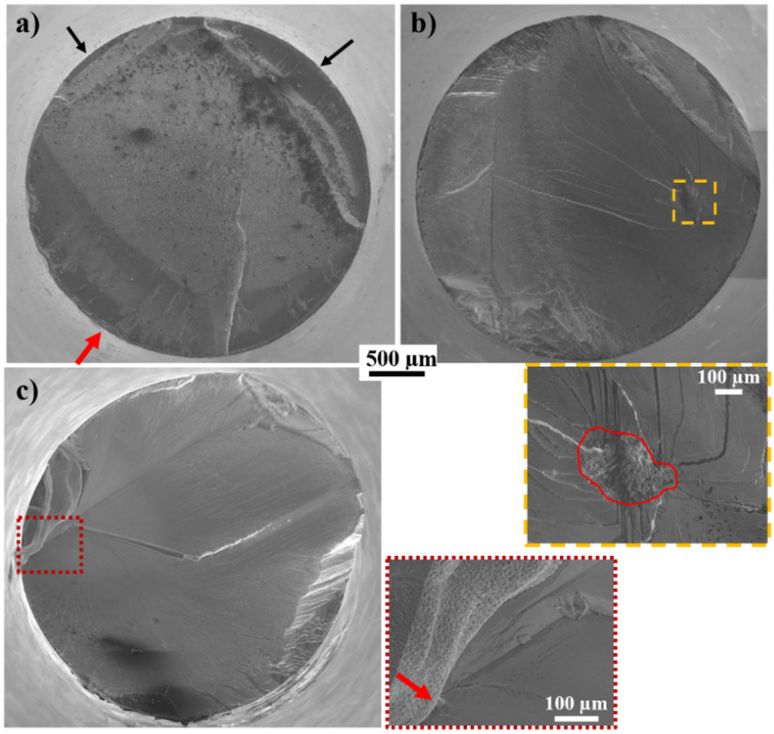

Fig. 8. Fractographic observations of three PBF-E processed specimens: (a) AM/HT/REC-1 (σ_a = 145 MPa, Nf = 3.2 × 10^{10} cycles) with surface crack initiation pointed by the arrows, the main initiation is highlighted by the red arrow; (b) AM/HIP/REC-2 (σ_a = 145 MPa, Nf = 4.9 × 10^9 cycles) with a main internal crack site, presenting the characteristic rough zone pointed by the dotted yellow square (see insert); and (c) AM/HIP/FRS-1 (σ_a = 142 MPa, Nf = 9.8 × 10^5 cycles) with a main initiation from a surface grain boundary pointed by the red dotted square (see insert). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

3.2. Microstructure investigation along longitudinal sections

The longitudinal cross-sections were prepared in AM/HT/REC-1 and AM/HIP/REC-2 specimens to better understand crack initiation mechanisms for both PBF-E processed specimens.

3.2.1. Crack initiation from an internal site: AM/HIP/REC-2 specimen

The crack initiation site observed in **Fig. 8b** has been investigated at a high magnification and along a longitudinal section intercepting the initiation site as shown in **Fig. 9**. Different imaging modes have been used to try to determine its exact nature. From this figure, it was possible to identify needle-shaped precipitates (red arrows in **Fig. 9 c**). The rough zone presented discontinuities that look very similar to vacancies as previously observed by Cervellon et al. [20] within the rough zone due to locally very high deformation magnitude (**Fig. 9 c** and **d**). These small pores whose size is below 1 µm may have also been inherited from the AM process.

Fig. 10 shows the EBSD analysis performed just below the crack initiation site according to the localization provide in **Fig. 9a** by the green rectangle. The IQ+IPF images show that most of this region below the crack

initiation site presents angular variations lower than 1° indicating the absence of recrystallization. However, a small "recrystallization" zone of ≈ 60 µm in extension (see **Fig. 10d**) was observed close to the crack initiation zone. It is composed a several low angle boundaries of local misorientation typically lower than 10°. The internal stresses remaining from the melting lines combined with the HIP treatment can be the explanation of the recrystallized zone indicated in **Fig. 10**.

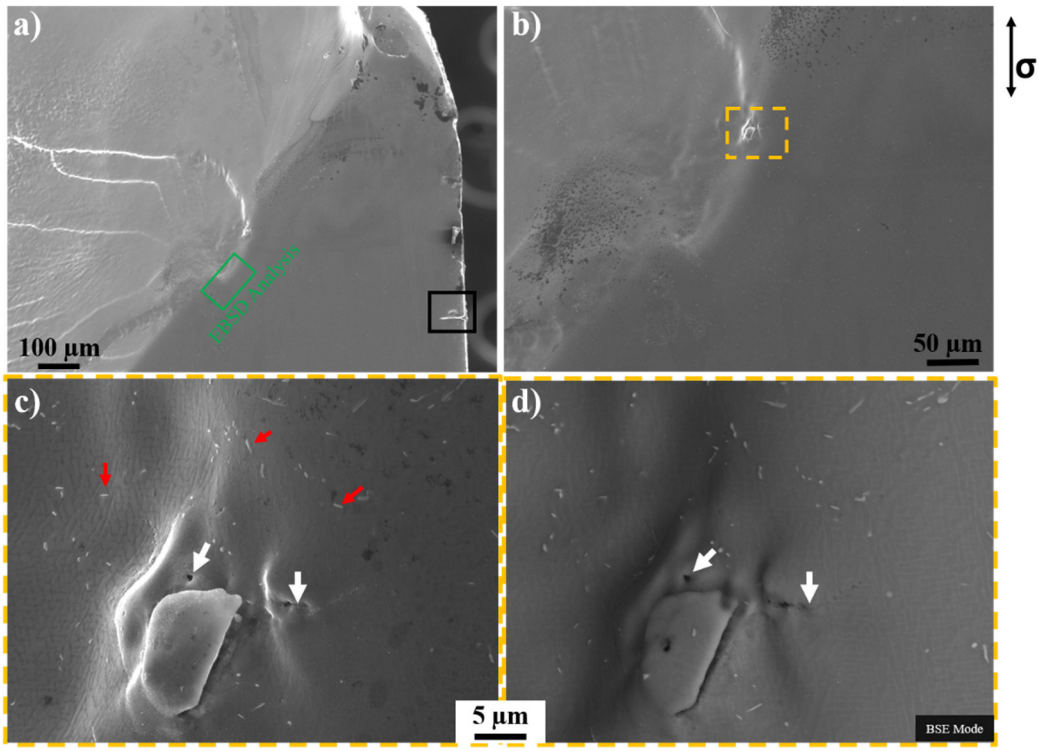

Fig. 9. (a) Crack initiation site of AM/HIP/REC-2 (σ_a = 145 MPa, Nf = 4.9 × 10^9 cycles) observed along a longitudinal section. Few secondary cracks are observed at the surface (see black square). (b) Crack initiation site identified by the rough zone, (c) magnification images using the SEI and (d) BSE imaging mode. White arrow is pointing out a remaining pore and red arrows highlight needle-shaped precipitates. The green square in (a) shows the location where the EBSD analyses have been performed. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

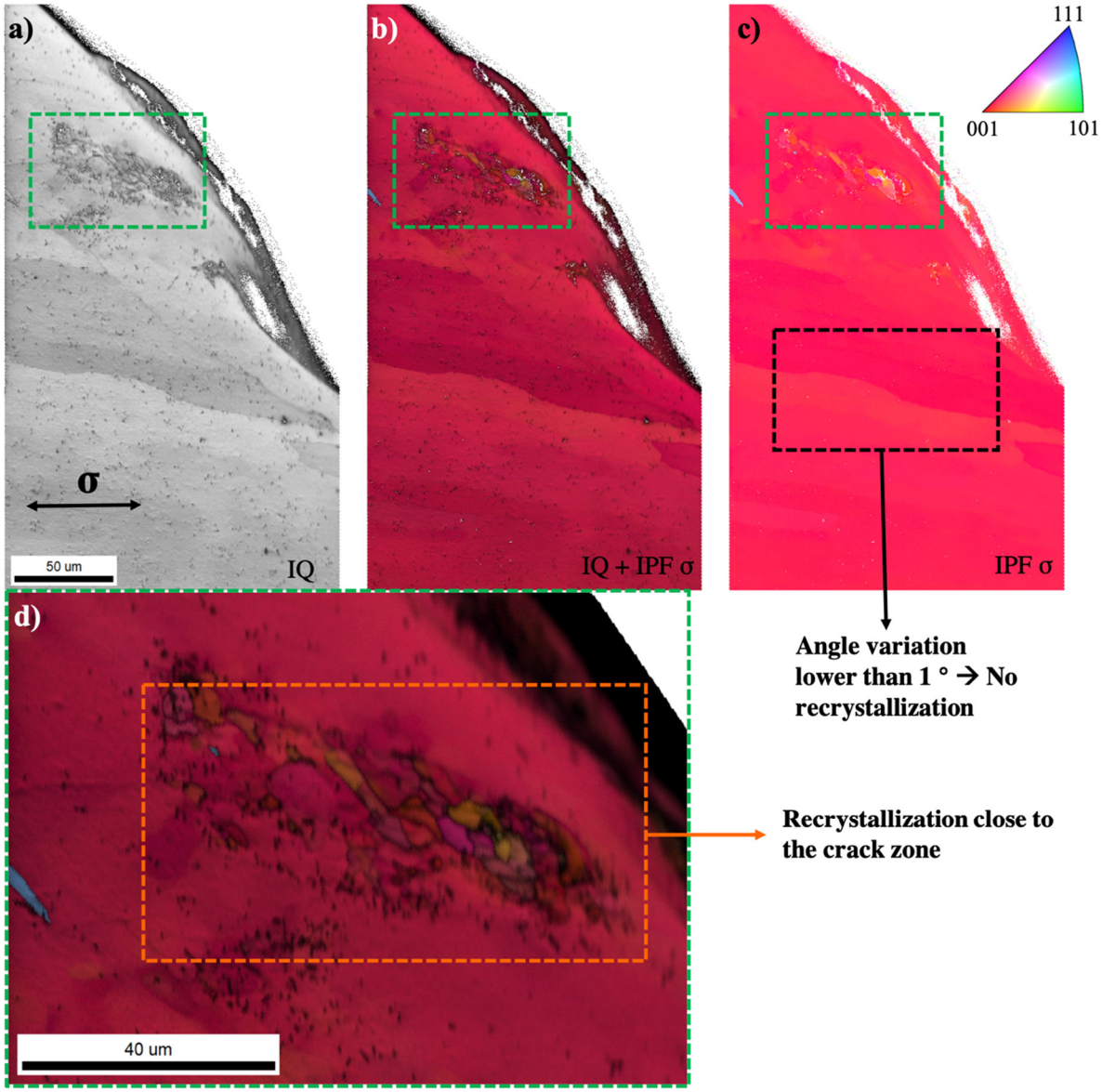

Fig. 10. EBSD characterization of AM/HIP/REC-2 (σ_a = 145 MPa, Nf = 4.9 × 10^9 cycles) crack initiation zone (green square in **Fig. 9a**) including (a) image quality (IQ). (b) IQ and inverse pole figure (IPF), (c) and IPF relative to the loading direction. Angle variation lower than 1° is pointed by the black dotted square. A region (dotted green rectangles in (a-c)) containing several recrystallized cells with low angle grain boundaries is shown in (d). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

3.2.2. Crack initiation from the surface: AM/HT/REC-1 specimen

Fig. 11 shows the features found along a longitudinal section of the AM/HT/REC-1 specimen. Failure from surface initiation is only shown for the HT sample since the results obtained for AM/HIP/REC-1, AM/HIP/FRS-2 and AM/HIP/FRS-3 (**Fig.6** and **Table 3**) are equivalent**. Fig. 11a** illustrates the presence of a secondary crack whose depth is ≈ 70 µm. This crack seems to be filled by oxides up to its tip. **Fig. 11b** and **c** are observations in BSE and SEI modes, respectively, allowing to highlight significant features like the square-like precipitates around the crack initiation zone and the oxidation inside the crack. A ≈ 30 µm-thick layer with Kirkendall porosity and precipitates

of different shapes appear underneath the surface (**Fig.11 d** and **e)**. **Fig. 11e** illustrates the oxides growing outwardly and leaving behind Kirkendall voids. None of these particular features were previously observed either in CMSX-4/Bridgman in VHCF at 1,000 °C by Cervellon et al. [15] or in the CMSX-4 PBF-E samples after LCF tests at 950 °C by Meid et al. [6], probably due to shorter duration of the tests.

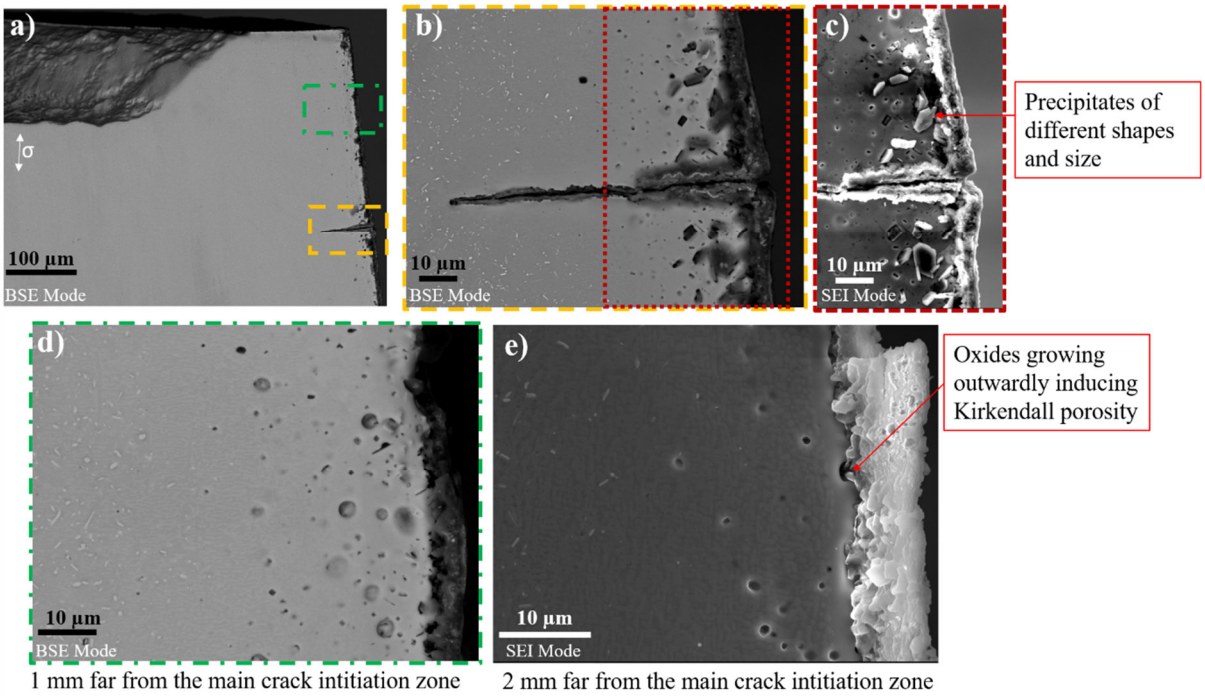

Fig. 11. Microstructure characterizations of AM/HT/REC-1 (σ_a = 145 MPa, Nf = 3.2 × 10^{10} cycles) along a longitudinal section: (a) high magnification overview of one side of the sample in BSE mode. (b) Secondary crack detail (orange dotted rectangle in (a)) in BSE mode and (c) SEI mode. (d) Surface detail (green dotted rectangle in (a)) 1 mm far from the main crack initiation site in BSE mode, and (e) 2 mm far from the main crack initiation zone in SEI mode. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fig. 12a highlights the chemical elements that constitute the oxides and the precipitates. The external oxide scale outside the crack is mainly composed of O, Ni and Cr (light yellow and green). The internal crack oxides comprise Ti, Ta and Al (purple and dark pink). The needle-like precipitates are rich in Re (yellow) ≈ 20 wt. % (see also Re map in **Fig. 12b**), and several big precipitates are composed of Ta, W and Hf (red). The large square-shaped precipitates close to the surface are composed of N and Al (orange), and the small ones are composed of N and Ti (blue). Re-rich needle-shaped precipitates are probably TCP ones, presumably µ or P phases, based on their morphology and on their very high Re content [33].

The EDS composition profile of the oxide and the substrate is displayed in **Fig. 12b**. The oxide is divided in three main layers: an external NiO, an intermediate Cr_2O_3 and internal Al_2O_3 layers. Depletion of Al, Ta and Ti between the bulk and the surface is also observed (**Fig. 12c)**, which results in the countercurrent increase in Re and W.

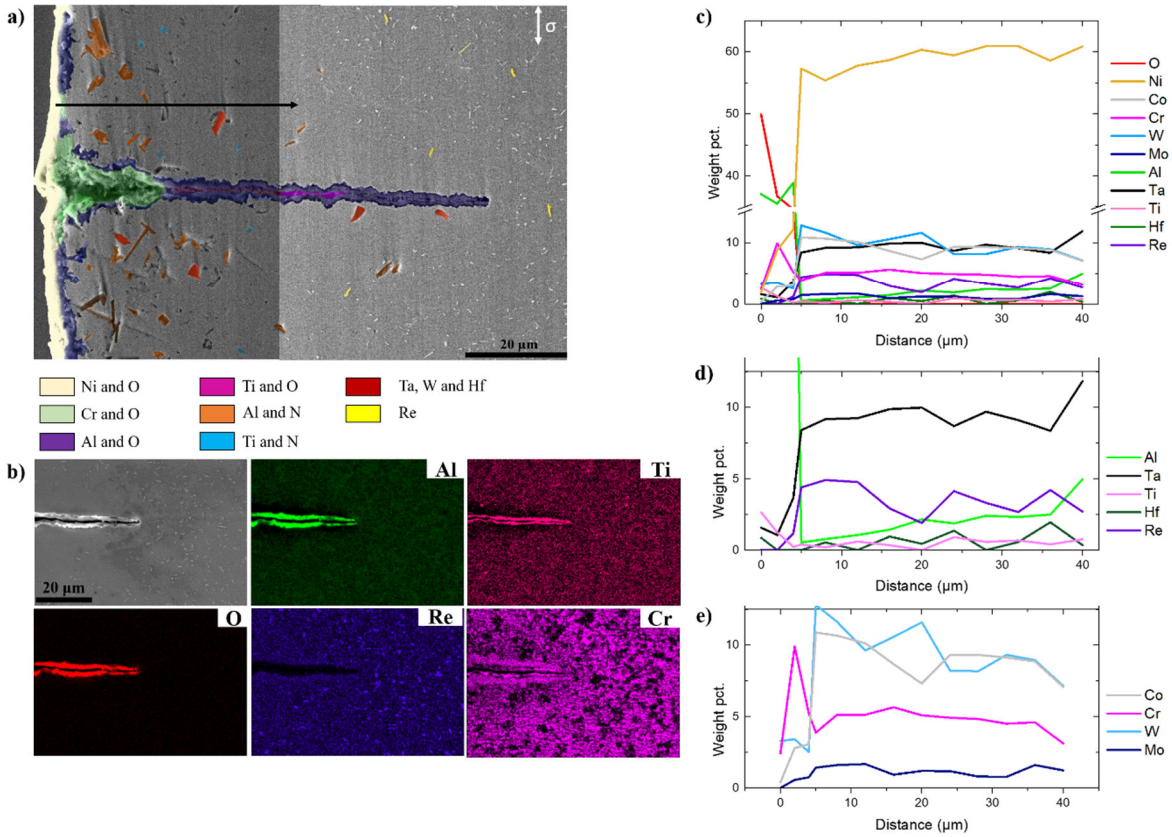

Fig. 12. AM/HT/REC-1 (σ_a = 145 MPa, Nf = 3.2 × 10^{10} cycles) EDS analysis. (a) SEI image artificially colored with the Photoshop software in agreement with the chemical elements X-ray maps analysis in (b). (b) X-ray maps of the crack tip highlighting the elements O, Al, Re, Ti, and Cr. (c) Chemical composition through the profile illustrated by the black arrow in **Fig. 12a** including all elements, (d) with a focus on Al, Ta, Ti, Hf, and Re elements and (e) with a focus on Co, Cr, W, Mo elements. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

4. Discussion

From the present experiments, it has been observed that PBF-E processed specimens performed extremely well in VHCF at 1,000 °C/R_ε = -1/f = 20 kHz provided that they are defect-free, i.e., they are fully single crystalline. Moreover, in comparison to conventionally processed (Bridgman solidification) Ni-based SX superalloys, which usually present crack initiation from the largest casting pores in these conditions irrespective of the alternating stress (see **Figs. 3** and **5** and refs. [14–20,23]), nearly all defect-free PBF-E processed specimens failed from the surface, with a crack initiation process assisted by oxidation (see **Figs. 3, 6, 7a, 8a, 11** and **12**) and equivalent results for fresh and recycled atomized powder. Since only AM specimens failed from an internal site, one can hypothesize on a very high dislocation density at local scale inherited from the building/HIP processes (**Fig. 9** and **10**). Yet, further analyses and additional results with a similar crack initiation mechanism would be required to unambiguously assess this potential mechanism of crack initiation. In the previous literature investigating fatigue properties of PBF-E-processed Ni-based SX superalloys, Meid et al. [6] compared the LCF life at 950 °C at a

stress ratio 0.6 of the AM SX with its parent cast material fully heat treated with and without HIP. They showed that the former performed better than the latter. In contrast, they did not report any failure related to the presence of grain boundaries probably because these stray grains were avoided by machining the samples from the center of bars. According to Meid's results [6] the crack initiation for PBF-E SX has three potential sources: (i) the interface of melting layers, (ii) the micro-porosity generated during solidification or, (iii) for the HIPed specimens, the precipitates formed at the location of collapsed pores. However, none of their specimens initiated or failed from the surface and we have to recall that the fatigue conditions used by Meid et al. introduced a pronounced cyclic ratcheting, i.e. creep strongly contributed to the damage mechanisms [6].

The following discussion will hence focus on the VHCF damage mechanisms of PBF-E-processed specimens, with a special focus on the crack initiation mechanism from the surface assisted by oxidation.

4.1. Crack initiation at grain boundaries

According to **Fig. 3** and **Table 3**, the presence of stray grains within the gauge part of VHCF specimens induces a spectacular debit in fatigue life. Six specimens even failed during the initial ramping-up in alternating stress (see **Fig. 4**). These grains are either inherited from the PBF-E building process (i.e., the polycrystalline shell as shown in **Fig. 1a** could have been deeper in some AM processed specimens leading to the presence of grains within the gauge part of specimens after machining/polishing) and/or the HIPing process. While (surface) recrystallization is known to decrease the LCF life of Ni-based single crystalline superalloys [34,35], no prior study reported on the consequence of stray grain on the VHCF life at high temperature of Ni-based SX superalloys, according to the authors' best knowledge. We may only mention that a directionally solidified alloy (DS200+Hf), presenting several ≈ <001> columnar grains within the gauge part of VHCF specimens (i.e. low angle grain boundaries with respect to the loading direction [36]), performs slightly worse in VHCF at 1,000 °C/R_ε = -1/f = 20 kHz compared to SX specimens solidified with the same casting parameters [15]. Hence, highly misoriented grains from the perfect [001] crystallographic orientation is critical to the VHCF life. In fact, as VHCF specimens used in the present study are designed so as to obtain a frequency of resonance in tension/compression of 20 kHz +/- 500 Hz [32], such recrystallized grains have a Young's modulus far higher than the one along the <001> orientation (i.e. 93 GPa for CMSX-4/Bridgman). Hence, the local frequency of resonance is far different from 20 kHz leading to a local stress concentration at grain boundaries. Failure in shear mode at the grain boundary is obtained in such a condition. The analysis of the role of recrystallized grains on the VHCF life of Ni-based SX superalloy specimens will be analyzed in greater details in a forthcoming article, by using finite element simulations and bicrystalline specimens tested in VHCF in the same condition.

4.2. Microstructure evolution

PBF-E specimens that failed through cracks whose nucleation/micropropagation is assisted by oxidation presented a very high VHCF life (between 70 and 447 h) at 1,000 °C. According to **Figs. 11** and **12**, these specimens presented very different intermetallic precipitates in addition to the usual γ′ precipitates. AM/HT/REC and AM/HIP/REC coupons were then overaged in isothermal conditions for 480 h at 1,000 °C to compare the microstructure evolutions with and without ultrasonic fatigue loading. Such a comparison is shown in **Fig.13**.

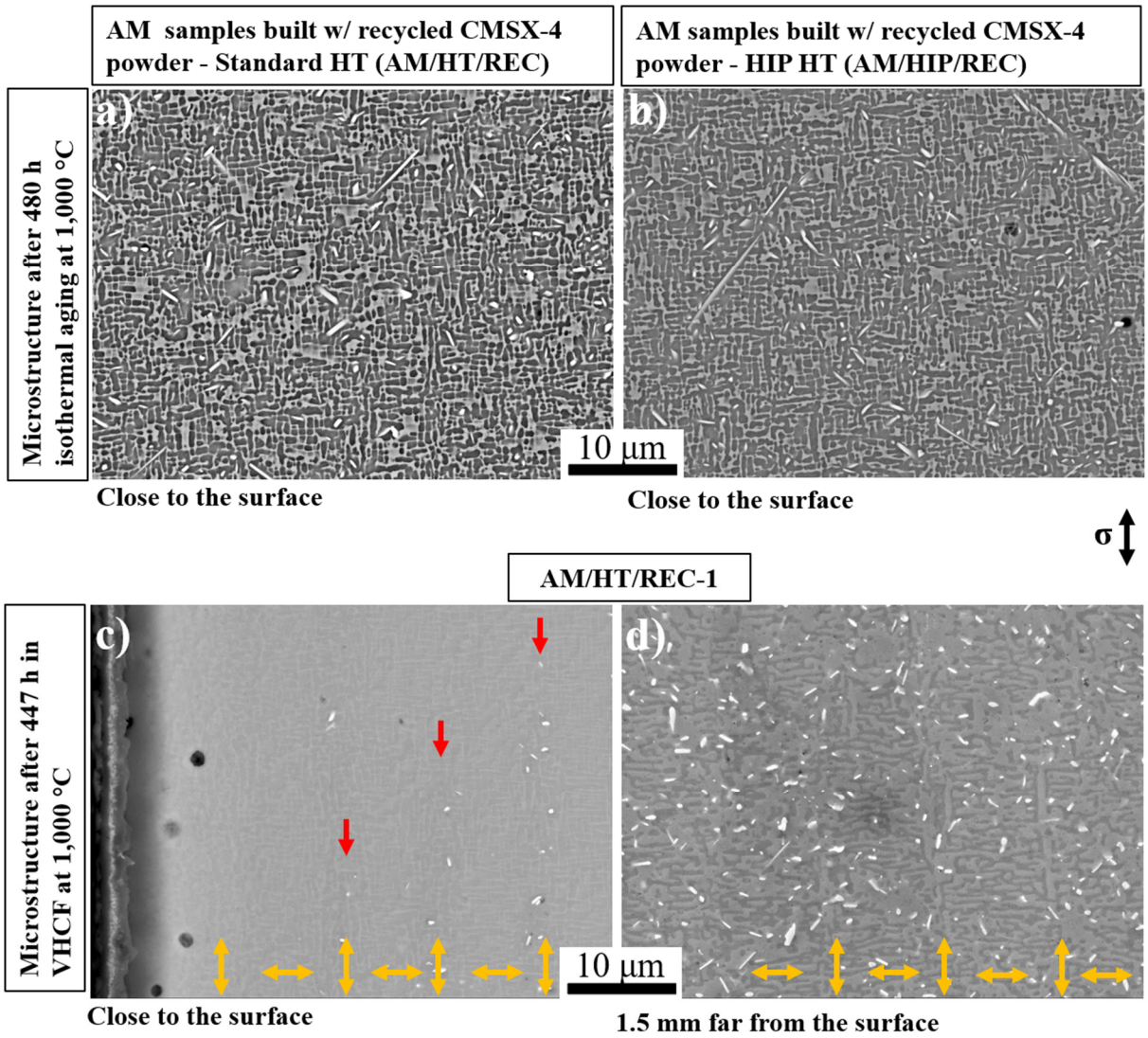

Fig. 13. (a) Microstructure evolution after 480 h at 1,000 °C for AM/HT/REC and (b) AM/HIP/REC. Both microstructures were taken 0.5 mm below the fracture surface. (c) Microstructure evolution after ≈ 447 h in VHCF at 1,000 °C for AM/HT/REC-1 (σ_a = 145 MPa, Nf = 3.2 × 10^{10} cycles) close to the surface and (d) 1.5 mm below the surface. Red arrows are showing TCP precipitates along the dendritic zones. Yellow arrows are showing the directions of local γ' coarsening. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

At 1,000 °C, the superalloy CMSX-4/Bridgman is known to be prone to topologically close-packed (TCP) precipitates [14,37–40] that are well known to lower the mechanical properties [41,42]. This TCP precipitation is also known to be enhanced at high temperature by the introduction of pre-deformation [43,44] or by the very high local plastic deformation in the rough zone close to stress raisers in VHCF [20]. As already investigated by Pistor et al. [30], TCP phases precipitate in PBF-E CMSX-4 alloy at high temperatures (around 1,050°C) and for longtime exposures. In our study, based on the morphology and EDS analysis showing a very high Re content shown in **Fig. 12**, we believe that needle-type precipitates are also TCP phases [39]. Moreover, AM/HT/REC-1 test lasted ≈ 447

h, i.e. long enough to be in the precipitation domain of TCP phases at high temperatures according to previous literature [33,38,39], as opposed to the tests performed by Meid et al. [9] that only lasted 48 h in LCF.

Such TCP phases precipitate homogeneously (≈ 1 % in area fraction) in the material without loading (**Figs. 13a** and **b**). Comparing **Fig. 13d** (2.5 % in area fraction) with **Figs. 13a** and **b**, imposing the VHCF loading does not seem to deeply affect the overall amount of TCP phases, but their location varies close to the surface, where they seem to mainly precipitate in former primary dendrite arms (red arrows at **Fig. 13c**). Further, their density increases from the surface towards the bulk. This can be explained by the local change in chemical composition due to the depletion of the other alloying elements to form the oxides, especially Cr and W known to favor TCP precipitation in CMSX-4/Bridgman [33], in addition to Re. This result is also in good agreement with the Re distribution map of **Fig. 12b** showing almost no TCP precipitates close to the crack tip.

The square-type precipitates rich in N are the nitrides developed close to the surface due to the low oxygen pressure. As the oxygen is being consumed by the Ni, Al, and Cr elements to form the oxide layer, the oxygen pressure locally drops, and the 78 % of N_2 present in the air diffuses in the substrate reacting with Al and Ti. Nitrides are known to be brittle structures, decreasing the mechanical properties of superalloys, especially in fatigue at low temperatures [45–47]. The Ta/W/Hf-rich carbides previously observed in MAR-M247 [48] and in Mar-M200+Hf/DS200+Hf [49] are also known to favor crack initiation in LCF [50]

The voids appearing just beneath the oxide layer (**Figs. 11 b-e)** have been previously observed after the high temperature oxidation of various Ni-based superalloys [51–54] and are related to the vacancy supersaturation according to Evans et al. [55]. Such vacancies originate by the major outward cation diffusion and the countercurrent injection of vacancies into the bulk resulting in the well-known Kirkendall porosity [56]. One shall notice that the Kirkendall porosity often driven by the gradient of chemical potential [57] (**Fig. 12c**) was not observed in the absence of mechanical loading. This finding suggests that greater outward diffusion was most likely enhanced through the dislocations introduced upon the VHCF [58] allowing to reach voids of ≈ 5 μm diameter. One may also speculate on the high dislocations' density inherited from the very PBF-E additive manufacturing process that can also foster oxidation. Indeed, it is known that a very high dislocation density is present within the material after the PBF-E process [59], and this was also confirmed by the fact it has been impossible to conduct VHCF tests in the present conditions at alternating stresses greater than 180 MPa (see Fig. 2), due to a too pronounced self-heating. Regarding oxidation, Juillet et al. demonstrated that the instantaneous parabolic rate (k_p) constants could not be calculated in an AM IN-718 superalloy due to enhanced growth of the oxides in comparison with the forged counterparts and related this to the significantly higher density of internal defects assessed by transmission electron microscopy (TEM) [60]. As a result, the selective outward diffusion of the most oxidizable elements to form the oxide scale brings about the precipitation of the strong carbide and nitride formers underneath [61].

4.3. Crack initiation and propagation mechanism assisted by oxidation

This is the first time that a VHCF life controlled by oxidation is presented in the open literature for a Ni-based SX superalloy at high temperature in fully reversed conditions, and in the absence of any surface recrystallization mechanisms. In fact, several EBSD maps have been performed in this work for specimens showing surface crack

initiation and no evidence of recrystallization close to secondary cracks in PBF-E processed specimens has been observed (see, e.g., Fig. 14). Cervellon already observed that surface cracks may develop in fully reversed conditions at the same temperature for long VHCF tests [15,17], but the main crack initiation (almost) always occurred at an internal site (a casting pore). Our present results are somehow different from previous examples of fatal crack initiation from the surface in VHCF in this class of alloys showing the presence of recrystallized grains (associated or not to a coating deposition process) [21,22] or from a stress concentration at the surface of specimens, due to a prior plastic deformation before testing specimens in VHCF at 1,000°C/R=0.5/f = 20 kHz [23].

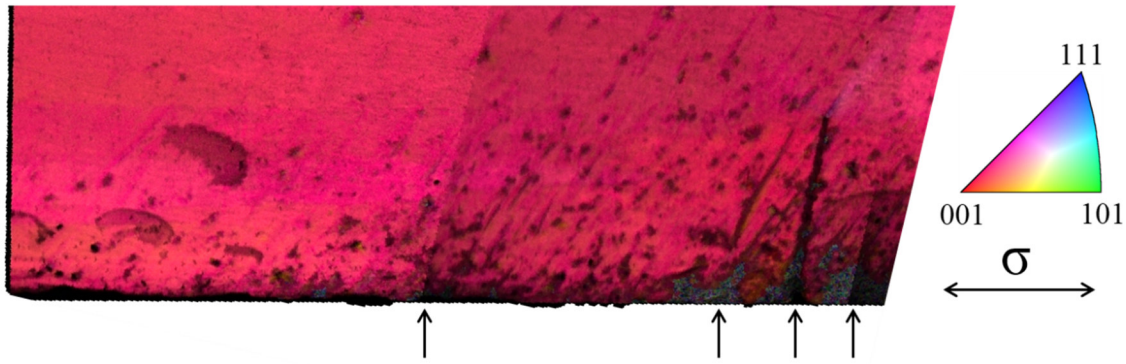

Fig. 14. EBSD characterization of AM/HT/REC-1 (σ_a = 145 MPa, Nf = 3.2 × 10^{10} cycles) close to surface secondary cracks. The Index Quality map is superimposed to the inverse pole figure (IPF) relative to the loading direction. Black arrows indicate secondary cracks initiated from the surface.

Fig. 15 presents the typical crack initiation mechanism as understood from the microstructures and chemical analyses shown before. Starting from the initial optimized γ/γ' microstructure (**Fig. 15a**) that likely contains intrinsic processing defects (incl. a high dislocation density), oxidation rapidly occurs at the specimens' surface, leading to a rougher surface and to the depletion of (mostly) Al and Cr and the subsequent precipitation of nitrides and carbides below the surface (**Fig. 15b**). Dislocation climbing may be then fostered with the VHCF loading, results in enhanced outward cation diffusion and the derived formation of Kirkendall voids just below the free surface (**Fig. 15c**). Increasing the duration of VHCF exposure at high temperature leads to TCP precipitation and to non-directional coarsening of γ' precipitates (see **Fig. 15c**), resulting from a small remaining dendritic segregation, as already proposed by Hazotte et al. [62]. Overall, the subsurface layer is composed of voids, a γ matrix depleted from its main refractory elements filled with quite brittle particles (nitrides, carbides). The increased surface roughness due to oxidation combined with the presence these voids/non-metallic inclusions lead to a surface crack initiation at a void or a non-metallic particle. This micro-crack then propagates in mode I through the microstructure, assisted by oxidation, up to failure (**Figs. 15d** and **e**).

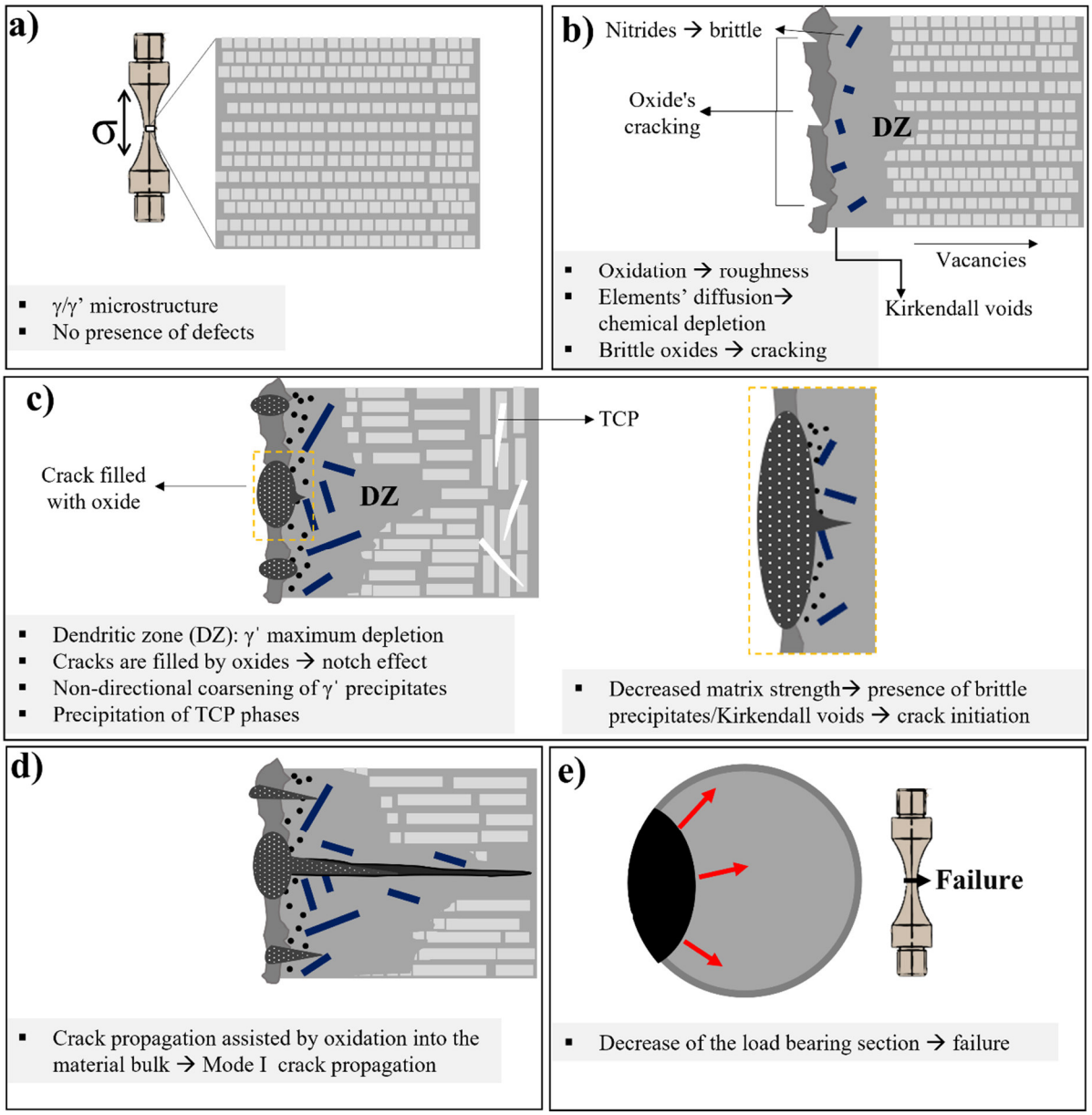

Fig. 15. Schematic illustration of the oxidation-controlled crack initiation mechanism of PBF-E CMSX-4 SX Ni-based superalloys in VHCF conditions at 1,000 °C/R_ε = -1/f = 20 kHz. (a) The initial optimized γ/γ' microstructure is oxidized leading to the precipitation of nitrides (b) and carbides below the surface and simultaneously developing a rougher surface. (c) Subsequently the oxidation leads to the formation of Kirkendall voids just below the free surface, followed by the precipitation of TCP phases as well as non-directional coarsening of γ' precipitates. The increased surface roughness due to oxidation combined with the presence these voids/non-metallic inclusions lead to a surface crack initiation at a void or a non-metallic particle. (d) The micro-crack then propagates in mode I through the microstructure, assisted by oxidation, up to failure (e). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

From the present investigation, it has been shown that provided PBF-E processed CMSX-4 specimens are defect-free (i.e., no stray grain within the gauge part of specimens), a very high VHCF life is obtained, comparable or

even longer than the one obtained for conventionally processed Ni-based SX superalloys. A S-N diagram at 1,000 °C/R_ε = -1/f = 20 kHz including defect-free PBF-E processed CMSX-4 specimens and several other results obtained for conventionally processed Ni-based SX superalloys with an internal crack initiation at a solidification pore is shown in the **Fig. 16**. It is important to point out that the results from the internal database in Institut Pprime/ISAE-ENSMA (France) have been obtained using "legacy" Ni-based SX superalloys such as Rene N5 and AM1 [63] and but also newer generations such as CMSX-4 Plus, DD33, TMS-238, MC-NG, and TROPEA [18,19,63]. Their chemical composition is given in **Table 4**. Further, they were solidified using a Bridgman or an LMC process. According to **Fig. 16**, an environmental VHCF limit under such conditions can clearly be identified, towards the highest VHCF lives. Up to now, most of the authors focused their investigations on VHCF damage mechanisms from either casting pores or from non-metallic inclusions (TCP phases, eutectics, carbides…), i.e. from process related "defects" [15,19,20,57,64]. When nearly all such defects are removed from the material, and provided that a good surface preparation of specimens is performed to avoid any surface recrystallization due to too large residual stresses known to be detrimental the fatigue life [34,35], the intrinsic VHCF life is controlled by the alloy's chemistry. In such conditions, the VHCF life of single crystalline Ni-based superalloys is hence controlled by the environmental resistance of the alloy, i.e., by its own chemistry and the overall microstructure of the alloys. The present study also suggests that no fatigue limit exists in these fatigue conditions at high temperature, but such a question should also be investigated at higher and lower temperatures to get a better picture of oxidation controlled VHCF crack initiation mechanisms.

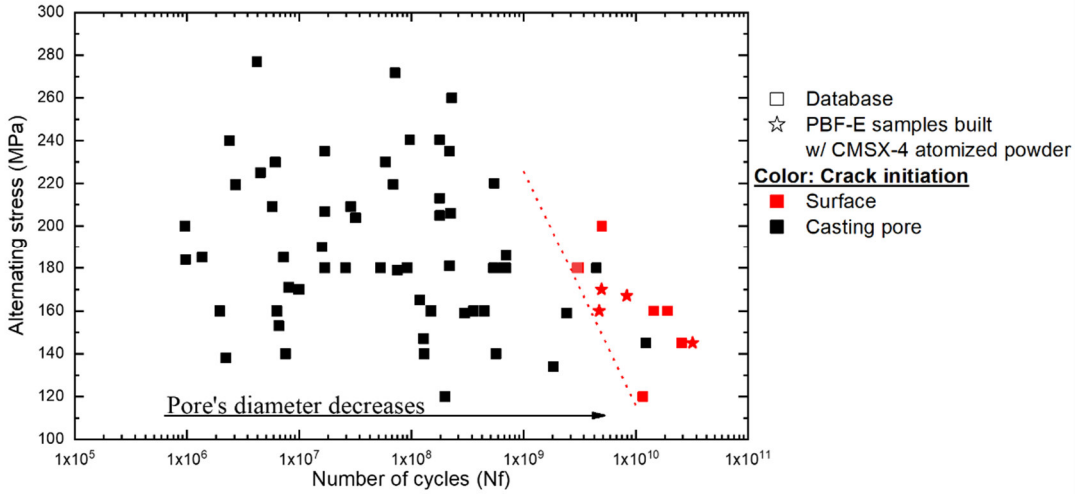

Fig. 16. S-N diagram at 1,000 °C, R_ε = -1 / f = 20 kHz including defect-free PBF-E processed CMSX-4 SX specimens and other "conventionally" processed SX specimens made of Rene N5, AM1, CMSX-4 Plus, DD33, TMS-238, MC-NG and TROPEA alloys with different solidification parameters [18,19,63]. The alternating stress σ_a is plotted as a function of the number of cycles to failure. A transition from an internal crack initiation site at a large casting pore to the surface is illustrated. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 4

Chemical composition (in wt. pct) of AM1, Rene N5, CMSX-4 Plus, DD33, TMS-238, MC-NG and TROPEA alloys

Alloy	Ni	Cr	Mo	Co	W	Al	Ti	Ta	Hf	Re	Others
AM1	Bal.	7.5	2.0	6.6	5.5	5.2	1.2	7.9	0.1		
Rene N5	Bal.	7.0	2.0	8.0	5.0	6.2		7.0	0.2	3.0	
CMSX-4 Plus	Bal.	3.5	0.6	10.0	6.0	5.7	0.9	8.0	0.1	4.8	
DD33 [65]	Bal.	3.0	1.0	12.0	6.0	6.0		8.0	0.1	4.0	
TMS-238 [66]	Bal.	4.6	1.1	6.5	4.0	5.9		7.6	0.1	6.4	5.0 Ru
MCNG [67]	Bal.	4.0	1.0	<0.2	5.0	6.0	0.5	5.0	0.1	4.0	4.0 Ru
TROPEA [68]	Bal.	6.4	0.6	8.9	6.1	5.4	1.0	9.1	0.08	1.0	1.95 Pt

5. Conclusions

Very high cycle fatigue performances and crack initiation mechanisms at high temperature of PBF-E CMSX-4 SX Ni-based superalloy have been investigated under fully reversed conditions (R_ε = -1), f = 20 kHz and 1,000 °C. Comparisons have been made with conventionally processed Ni-based SX superalloys of ERBO/1 and CMSX-4/Bridgman type. The following main conclusions have been obtained:

- All Bridgman processed specimens failed from an internal casting pore site.
- The VHCF lifetime of PBF-E processed specimens in these conditions is comparable or higher than the one of Bridgman solidified Ni-based SX superalloys provided that no defect (e.g., stray grain) is present within the gauge part of the specimens.
- The presence of high angle grain boundaries/stray grains leads to a spectacular (more than 3 decades) decrease in VHCF lifetime compared to defect-free specimens.
- Due to the absence of large casting pores/non-metallic inclusions, nearly all defect-free PBF-E processed specimens failed by an oxidation assisted surface crack initiation process. Hence, the superalloy chemistry and microstructure stability below the surface controls the crack initiation and first stages of propagation and an environmentally controlled VHCF life limit has been identified in these conditions.

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