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Abstract

Powder of a nickel-based superalloy, RR1000, has been hot isostatically pressed (HIPped) at a supersolvus temperature and post-HIP heat treated to produce different microstructures. Microstructures were investigated using a scanning electron microscope (SEM) together with an energy dispersive X-ray spectrometer (EDX) and a wave-length dispersive X-ray spectrometer (WDX). High cycle four-point bending fatigue and tension-tension fatigue tests have been performed on the fabricated samples. It was found that HIPped and aged samples showed the best four-point bending fatigue limit while HIPped and solution treated and aged samples had the lowest fatigue limit. The four-point bending fatigue crack initiations all occurred from the sample surfaces either at the sites of inclusion clusters or by cleavage through large grains on the surfaces. The tension-tension fatigue crack initiation occurred mainly due to large hafnia inclusion clusters, with lower fatigue lives for samples where inclusions were closer to the surface. Crack initiation at the compact Al_2O_3 inclusion cluster led to a much higher fatigue life than found when cracks were initiated by large hafnia inclusion clusters. The tension-tension fatigue limits were shown to decrease with increased testing temperature (from room temperature to 700°C).

Key words: Hot isostatic pressing; Powder Metallurgy; Nickel-based superalloys; High cycle fatigue; Fracture behaviour

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1. Introduction

Net-shape hot isostatic pressing of alloy powder enables production of complex components with reduced cost due to the elimination of forging or extrusion and of massive machining operations and thus has been considered as one of the advanced manufacturing routes for production of high performance components and structures for aerospace application. Several components have been made from Ti alloys and nickel-based superalloys [1-4]. However, because no thermo-mechanical processing such as forging and extrusion is involved, defects such as inclusions, porosity and prior particle boundaries (PPBs) which may be present in the powder may also be present in as-HIPped or HIPped and heat treated materials. These could downgrade the mechanical properties of the HIPped components. In the case of as-HIPped Ti-based alloys, PPBs and porosity are generally not present whereas inclusions are observed occasionally which could act as crack initiators and cause scatter in ductility [5]. As far as as-HIPped nickel-based superalloys are concerned, PPBs due to carbide or oxide decoration are a very common issue and thermally-induced pores could be formed if high temperature post-HIP heat treatments are conducted [6-14]. PPBs are vulnerable sites for crack initiation and propagation and thus very harmful for high temperature mechanical properties of Ni-based superalloys, especially for hot ductility, low cycle fatigue (LCF) properties and stress rupture properties [15-16]. Inclusions and pores could also act as crack initiation sites and were found to affect adversely fatigue properties. Several reports suggested that LCF lifetime is closely related to the size, number and location of inclusions [16]. Therefore, it is necessary to study the microstructure and defects and their influence on mechanical properties when it comes to net-shape HIPping of nickel-based superalloy powder.

In our previous work [17], RR1000 was selected for net-shape HIPping and an optimum HIPping condition that minimized PPBs has been developed. A post-HIP heat treatment which avoided the formation of thermally-induced porosity and gave rise to optimum tensile

properties has also been identified [18]. In this paper, we try to investigate the high cycle fatigue (HCF) properties and fracture of the as-HIPped samples and the HIPped and heat treated samples and to find out the key factors that will affect the fatigue properties of net-shape HIPped nickel-based superalloy samples.

2. Experimental

The powder used in this study is an argon-atomised nickel-based superalloy powder RR1000 which has a size range below 53μm in diameter. The chemical composition of this alloy is listed in Table 1 and the γ′ solvus (S) of this alloy is around 1160°C. The as-received powder was HIPped at a super-solvus temperature (S+20ºC) and at a pressure of 100MPa for 4 hours, followed by cooling at 5°C/min to room temperature. Some samples were aged at 760°C for 16h and air cooled (the cooling rate was measured to be around 105°C/min) directly after HIPping without a prior solution treatment (HIPped+aged) while other samples were solution-treated at (S-40)°C for 2h followed by air cooling (at around 105°C/min) and ageing at 760°C for 16h (HIPped+solution treated+aged).

Metallographic specimens were prepared using conventional method and then electrolytically etched in 10% H_3PO_4 in H_2O at 25V for 2-5 seconds. This preferentially dissolves the γ phase leaving the γ′ in relief. The samples were examined using SEM in a JEOL 7000 FEG-SEM microscope fitted with EDX and WDX. For EDX analysis, a 20KV accelerating voltage and a 10mm working distance were used while for WDX analysis, a 10KV accelerating voltage together with a beam current of more than 10nA was used. Electron back scattered diffraction (EBSD) was also conducted in the SEM to study the grain structure. The samples were tilted 70° for EBSD mapping. The accelerating voltage used was 20 kV and the working distance was around 18 mm. A step size of 10nm and a pattern resolution of 128x128 were used to

develop the EBSD maps which were then analyzed using OXFORD INCA software supplied by OXFORD INSTRUMENTS.

Both four-point bending fatigue tests and tension-tension fatigue tests were performed to evaluate the HCF properties of the HIPped and heat treated materials. To be mentioned, the purposes for these two types of tests are different. The 4-point bending test is designed to assess the surface-sensitiveness of materials under cyclic stress condition while tension-tension tests are used to examine the fatigue behaviour of a volume of material as a whole. The fatigue limit in HCF was defined as the fatigue strength corresponding to a fatigue life of 1.0×10^7 cycles.

Samples with a dimension of $60\times10\times10mm^3$ were cut out using EDM for four-point bending fatigue testing. Prior to testing, the sharp edges of samples were ground to be round and the surface was polished using 800 grit silicon carbide papers to remove the surface deposits formed during EDM machining. The four-point bending fatigue tests were performed on an Amsler Vibrophore electro-magnetic resonance machine at a stress ratio R ($R=\sigma_{min}/\sigma_{max}$) of 0.1 and at frequencies in the range of 100–120Hz.

Tension-tension fatigue tests were performed on cylindrical samples at both room temperature and 700℃ in air on an Amsler Vibrophore machine at frequencies in the range of 70–85Hz with a stress ratio R of 0.1. For high temperature testing, a heating furnace was set up around the testpieces and prior to each test, samples were held at 700℃ for 0.5 hour.

Fracture surfaces after fatigue failure were ultrasonically cleaned in acetone for at least three minutes to remove any dirt and then examined using SEM.

3. Results

3.1 Room Temperature Four-point Bending Fatigue Behaviour

Figure 1 shows the microstructure of as-HIPped sample and HIPped and heat treated samples. A typical trimodal γ′ distribution, composed of large primary γ′ at grain boundaries, intragranular secondary γ′ and very fine tertiary γ′, could be observed in all the samples investigated. The development of tertiary γ′ in as-HIPped sample is believed to be due to the slow cooling (only 5°C/min) from HIPping temperature which would give more time in the middle or late cooling stage for the remnant γ′ formers to diffuse and gather to form fine γ′ particles. The as-HIPped sample and HIPped and aged sample both show irregular-shaped secondary γ′ within grains and coarse primary γ′ at grain boundaries. Ageing led to the formation of a very high population of fine tertiary γ′; see Figure 1(d). Solution treatment and ageing significantly decreased the volume fraction of secondary γ′ and changed the irregular-shaped γ′ in the as-HIPped samples into cuboidal or near-triangular morphology (see Figure 1(e-f)) and also led to the formation of a number of much finer tertiary γ′. The refined microstructure has been shown to lead to considerable improvement of tensile strengths in HIPped and solution treated and aged samples [18]; see Table 2. Apart from γ-γ′ microstructure, the samples also contain some large inclusions, as shown in Figure 2. These inclusion particles are usually rich of Hf, Zr and O (also see Table 3), which could be attributed to (Hf, Zr)O_2 or hafnia.

Figure 3 shows the room temperature four-point bending fatigue properties of as-HIPped samples and of HIPped and heat treated samples. It can be seen that at lower stress levels (σ_{max}＜850MPa), those samples that were HIPped and then aged show the best fatigue life. In this stress range, the HIPped and aged samples all run out (greater than 10^7 cycles) whereas

most of the samples from other processing conditions fail at about 10^6 cycles. However, it is noted that at higher stress levels ($\sigma_{max} \geq 850$MPa), the fatigue lifetimes for samples prepared under different processing conditions are generally comparable when they were tested at the same stress levels.

The fracture surfaces of all failed samples were examined in order to understand the factors which led to initiation of fatigue failure. The fatigue crack initiators together with fatigue lifetimes of the tested samples are summarised in Table 4. All the fatigue crack initiations occur from the sample surface where high tension-tension stress is expected. At lower stresses ($\sigma_{max} < 850$MPa), the HIPped and aged samples all run out whereas those samples that were solution treated and aged fail either at the site of inclusion clusters or in a brittle manner characterized by the large cleavage facets at the initiation sites; also see Figure 4. This may account for the reduction of fatigue limit for the samples that were solution treated. EBSD image of a cross section through the crack initiation site (as shown in Figure 5) shows that the large cleavage facets are actually due to the presence of large grains on the sample surface. These grains are obviously subject to stress concentration and it is possible that some of the favourable slip system may have been activated so that slip and fracture could proceed through them. The observation is consistent with previous report which suggested coarse grains are favourable for fatigue crack initiation [19]. At higher stresses ($\sigma_{max} \geq 850$MPa), samples failed either due to the presence of inclusion clusters or due to cleavage fracture; see Table 4. Also, it is noted that the presence of large inclusion clusters does not downgrade the four-point bending fatigue properties significantly. Thus for samples that were tested at the same stress level only slightly smaller fatigue lives are associated with failures which occur at inclusion clusters than are found when no inclusions are seen and large cleavage facets are seen at the initiation sites. Moreover, despite the fact that the fatigue lifetime is impaired to some extent by the presence of inclusion clusters, the samples prepared all show a very high

fatigue limit; even the samples (that were solution treated) with lowest fatigue limit among the samples investigated, still show a fatigue limit around 700MPa.

In terms of fatigue crack propagation, all the samples show an identical fatigue crack propagation sequence, e.g., the fatigue crack initiation from sample surface is followed by a cleavage fracture as evidenced by sharp and angular facets in the near-threshold area or lower Paris regime and then a long-range striated fracture in higher Paris regime and finally a monotonic but dimple-ductile fracture in the final stage; see Figures 6 and 7. Moreover, some opened-up pores could be observed occasionally in the monotonic fracture stage which may be due to the crack propagation along remnant prior particle boundaries (PPBs); see Figure 7(g). The bottom of these pores usually shows massive fine dimples (see Figure 7(h)), indicating a ductile fracture.

3.2 Room Temperature Tension-Tension Fatigue Behaviour

Figure 8 shows the room temperature tension-tension HCF properties for HIPped and heat treated samples. Samples, irrespective of processing conditions, all run out when the testing stress level is below 850MPa. Solution treatment seems to show no obvious influence on tension-tension HCF limit which is 850MPa in this case. However, compared with four-point bending fatigue tests, tension-tension fatigue tests lead to much better fatigue limit, especially for the samples that were solution treated.

Figure 9 shows some typical fracture surfaces of samples after tension-tension fatigue testing at room temperature. A summary of fatigue lifetimes together with crack initiation origins of the samples tested at room temperature are shown in Table 5. In contrast to the observations made on samples tested by four-point bending fatigue as summarised in Table 4, all the tension-tension fatigue samples tested show fatigue crack initiation always associated with inclusion clusters. These inclusion clusters are found to be rich in Hf, Zr, O and C through

EDX analysis, as shown in Figure 10. EDX quantitative analysis (see Table 6) suggests that they could be mainly (Hf, Zr)O_2, consistent with the above WDX analysis. For most of the samples, fatigue cracks initiate from the internal inclusion clusters but in some cases, fatigue crack initiation tends to occur at clusters of inclusions present in the surface region (see Figure 9(e-f). Also, it seems that the fatigue lifetime is associated with the location of the crack initiation site. Those internal crack initiators usually lead to much longer fatigue lifetime than surface crack initiators when the samples were processed and tested under exactly the same conditions; see Figures 9(a-b) against Figures 9(e-f). Moreover, in another group's comparison under the same processing and testing conditions, when the crack initiator is further from the sample surface, the fatigue life is better; see Figures 9(g) and (h). These results are consistent with previous reports on other powder-processed nickel-based superalloys [16, 20].

Around the crack initiators, a radiating fracture surface could be observed. Depending on the distance from crack initiation site, several distinct crack propagation regions with different fracture modes could be seen. In the near-threshold and lower portion of Paris regimes, crystallographic cleavage mode dominates; see Figure 11(a). This is followed by a striated fracture with continued crack propagation (Figure 11(b)). In the high ΔK (ΔK, crack-tip intensity factor range) regime, a ductile-dimpled fracture mode is observed. Moreover, in this regime, whenever inclusion clusters are encountered, cracks would be developed and pores formed due to material separation at these sites; see Figures 11(e-f). This is further evidenced by the observation on the sections of the tested samples where secondary cracks are developed at the inclusions/matrix interfaces; see Figure 12. The results further suggest that inclusion clusters are very harmful for fatigue initiation and propagation of directly HIPped and heat treated samples.

Based on the above observation, it is obvious that the samples for tension-tension fatigue tests show generally similar crack propagation micromechanism to those for four-point bending fatigue tests, irrespective of the difference in sample geometry.

3.3 High Temperature Tension-Tension Fatigue Behaviour

Figure 13 shows the tension-tension fatigue properties at 700℃ for HIPped and heat treated samples. Solution treatment seems to show no appreciable influence on the high temperature tension-tension fatigue properties. Samples with or without solution treatment both show a fatigue limit at 750MPa, which however, is 100MPa lower than the room temperature tension-tension fatigue limit (850MPa, see Figure 8). Moreover, those samples that failed at 700℃ usually show a relatively short lifetime as compared with those failing at room temperature; see Table 5.

The high temperature tension-tension fatigue properties together with fatigue crack initiators are summarized in Table 5. Similar to the case at room temperature, the fatigue crack initiation of these samples tested at 700ºC is also due to large inclusion clusters and the fatigue lifetime of the samples (irrespective of processing condition) is also highly dependent on the location of large inclusion clusters, i.e., the distance from the site of inclusion cluster to the sample surface (*d*). When a sample fails from the sample surface due to inclusion clusters, it suffers the lowest fatigue life; see Figure 14(a-b). Also the fatigue lifetime increases with the increased distance between the fatigue crack initiator and the sample surface; see Figure 14(e-h). For the same type of inclusion clusters, the size of an inclusion cluster seems to be insignificant with the depth of inclusion cluster from the surface having a far larger effect. This is evidenced by both Figure 14(a-d) and Figure 14(e-h) where the samples fail at larger inclusion clusters but show even higher fatigue lifetime, because of the larger distance of the inclusion clusters from the sample surface. In addition, the type of

inclusion cluster may influence the fatigue properties. Figure 15 shows that samples that were processed under exactly the same condition fail at different types of inclusion clusters and consequently show a big difference in fatigue lifetime. The compact Al_2O_3-type inclusion cluster (Figure 15(c-d)), the composition of which is listed in Figure 15(e) and Table 6, leads to a higher fatigue lifetime than those dispersed and large hafnia-type inclusion clusters, even though the hafnia cluster is further from the surface than the Al_2O_3 cluster.

Samples tested at 700℃ show generally similar fatigue crack propagation behaviour to those tested at room temperature, that is, crack initiation followed by a cleavage fracture and then a striation stage and finally a transgranular fracture stage; see Figure 16. However, the final monotonic fracture stage seems to be more faceted and crystallographic at 700℃ than at room temperature (see Figure 16(e-f)), which is consistent with the observations on tensile deformation [18] and obviously is due to the easier planar slip at elevated temperature. Besides, it is noted that samples tested at room temperature all show a very flat fracture surface while samples tested at 700℃ show part flat fracture surface and part slant fracture surface; see Figure 17. Given that the slant fracture surface area actually corresponds to the final monotonic fracture, this part of fracture is obviously dominated by the plain stress condition, which is also consistent with the previous study on tensile behaviour [18].

4. Discussion

It was demonstrated that the HIPped and aged samples appear to show the best four-point bending fatigue limit whereas samples which were solution treated show the lowest fatigue limit. The latter tend to suffer from an early failure at the lower stress levels (below 800MPa) due to the presence of either large inclusion cluster or large grain clusters in surface area. The clusters of large grains on the sample surface seem to be favourable for local stress/strain concentration which then led to early crack initiation by slipping through them. This is

consistent with the previous report which suggested that large grains are favourable for fatigue crack initiation although they are beneficial for crack propagation [19].

In terms of tension-tension fatigue, solution treatment shows no obvious influence on fatigue limit and fatigue lifetime; see Figure 8 and Figure 13. Instead, the location and type of primary fatigue crack initiators (i.e., large inclusion clusters) are found to show pronounced influence on fatigue lifetime at both room temperature and 700°C. Thus, the internal crack initiation generally show higher fatigue lifetime as compared with surface initiation and with the crack initiator being further away from the sample surface, the fatigue life is better. This is consistent with previous reports which also suggest that internal crack initiation usually leads to a better fatigue lifetime than surface initiation [16, 20]. It is generally believed that the less constrained surface allows relatively easy slip [13, 17-18]. This, when interacting with surface inclusions that are usually subjected to stress concentration, would lead to accelerated localized strain concentration and consequently to earlier crack initiation. Internal inclusions, however, are subject to plastic constraint and thus more difficult to lead to crack initiation. Moreover, it is noted that in the current study the fatigue lifetime does not necessarily degrade with increased inclusion cluster size, which is evidenced by both Figure 14(a-d) and Figure 14(e-h) where the samples fail at larger inclusion clusters but show even higher fatigue lifetime due to the longer distance between these inclusion clusters to the sample surface. This is not consistent with previous reports which indicated that inclusion size shows great influence on fatigue properties [16] but this further suggests that the location of crack initiator tends to dictate the fatigue lifetime of the investigated samples. In addition, it is demonstrated that even the type of inclusions could show great influence on fatigue property. Actually, crack initiation at the compact Al_2O_3 inclusion cluster was found to give rise to much higher fatigue lifetime in contrast to the initiation due to the dispersed and large

hafnia inclusion clusters under the same stress condition even though the hafnia clusters were closer to sample surface; see Figure 15. Also, unlike previous studies [16] where PPBs were found to cause fatigue crack initiation, the current study does not witness fatigue crack initiations at PPB defects although during the later stage of fatigue crack propagation, cracks could propagate along some of the remained PPBs and lead to formation of pores on the fracture surfaces. This may be due to the fact that PPBs have been minimized in the current samples thanks to the use of a super-solvus HIPping temperature [17].

As compared with room temperature four-point bending fatigue tests, the room temperature tension-tension fatigue tests show improved fatigue limit, which is particularly obvious for those samples which have been solution treated. Also, for four-point bending fatigue, the crack initiations all occurred from the sample surfaces whereas the crack initiation for tension-tension fatigue could happen at the sample surface or internally. This is associated with the different stress conditions that the samples have experienced. For four-point bending fatigue tests, one sample surface suffers not only high tension-tension stress but also large normal shear stress and thus is the most vulnerable area for stress-concentration and crack initiation whereas in tension-tension fatigue tests the whole volume of material experiences a relatively more homogeneous stress distribution. The stress concentration could only exist at inclusion clusters that are present either internally or at surface region. The latter is thus less surface-sensitive and demonstrates better fatigue life under the same stress conditions.

High temperature tension-tension fatigue was shown to have lower fatigue limit (100MPa lower) and shorter fatigue lifetimes than room temperature tension-tension fatigue although all the samples at room temperature and 700°C failed due to inclusion clusters. The reduction may be mainly due to the cyclic fatigue complicated by creep at elevated temperature. As seen in Table 2, the yield strengths of relevant samples have actually dropped by around

100MPa with elevated testing temperature, suggesting that the plastic deformation would be easier at higher temperature. Actually, previous study [18] has already demonstrated that deformation of HIPped and heat treated RR1000 became much easier and more planar at 700°C. The easier deformation around the stress concentrated large inclusion clusters would be more likely to promote local plastic strain buildup and thus accelerated the separation of inclusions/matrix interface to induce earlier crack initiation. At elevated temperatures, oxidation is believed to be another common factor that could affect fatigue properties depending on loading frequency. In particular, during a LCF test where low loading frequencies (such as 0.25Hz) are generally used, oxygen could penetrate through the crack tip leading to the formation of an oxide layer which causes embrittlement and accelerated fatigue crack propagation to nickel-based superalloys. One of the typical features for oxidation-induced fatigue fracture is the formation of intergranular fracture due to the preferential oxygen diffusion at grain boundaries [21-22]. Increasing loading frequency (like from 0.25Hz to 5.0Hz) was reported to significantly reduce the oxidation effect and could change the failure mode from intergranular to transgranular fracture in LCF testing [23-26]. Here for the HCF testing in the current study, the loading frequencies are much higher (85-110 Hzs) and thus the effect of oxidation on HCF properties could be considered insignificant. This is further supported by the observation that the fracture modes of the samples remain almost unchanged with increased testing temperature. Fatigue crack initiations all occurred at inclusion clusters while fatigue crack propagation proceeded in a highly crystallographic and faceted mode first followed by a long-range striation stage (the widespread striation implies that cyclic fatigue deformation had dominated the crack propagation instead of oxidation) and a final transgranular fracture mode. Transition of failure mode from transgranular fracture at room temperature to intergranular fracture at elevated temperature which is typical to LCF tests does not happen in the current study. Instead, fracture in the final stage of crack

propagation actually becomes even more crystallographic and faceted at 700℃; see Figure 15(e-f). All of these suggest that oxidation atmosphere may have a negligible effect on fatigue crack initiation (most of the crack initiation happened internally) and propagation and on fatigue lifetime during HCF testing. The use of high loading frequencies may have effectively suppressed the diffusion and penetration of oxygen to grain boundaries to cause intergranular fracture [27-29].

Given the fact that most of the fatigue crack initiations were due to large hafnia clusters and that the PPB decoration in as-HIPped RR1000 was mainly due to the formation of Hf-rich oxides or carbides [17], it is suggested that Hf should be removed from this alloy. The alloy was designed for P/M use where PPBs and inclusion clusters are usually smashed and scattered by extrusion or forging. In the case of net-shape HIPping, no thermomechanical processing is employed, it is necessary to optimize compositional design that will enable better mechanical performance.

5. Conclusions

- HIPped and aged samples showed the best four-point bending fatigue limit while HIPped and solution treated samples had the lowest fatigue limit due to failure at lower stress levels. The room temperature four-point bending fatigue crack initiations all occurred from the sample surfaces either at the sites of inclusion clusters or by cleavage through coarse grains on the surfaces.
- The tension-tension fatigue crack initiations occurred mainly due to large hafnia inclusion clusters. Internal crack initiation generally led to improved fatigue life as compared with surface crack initiation and the fatigue life increased with the distance between crack initiator and the sample surface under the same stress condition.

- Tension-tension fatigue crack initiation at the compact Al_2O_3 inclusion cluster led to a much higher fatigue life than the initiation due to the dispersed and large hafnia inclusion clusters under the same stress condition.
- Tension-tension fatigue limit and fatigue life were generally decreased with increased testing temperature probably due to easier deformation and increasingly weakened interface between matrix and inclusion clusters at elevated temperature.
- Inclusion clusters are more harmful for fatigue properties than the remained PPBs.

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Table 1 Nominal compositions of RR1000 powder in wt.%

Alloy	Cr	Co	Mo	Al	Ti	Ta	Hf	C	B	Zr	Ni
RR1000	15.0	18.5	5.0	3.0	3.6	2.0	0.5	0.027	0.015	0.06	Bal.

Table 2 Comparison in tensile properties between HIPped and heat treated RR1000 samples

Processing Conditions	Tested at Room Temperature			Tested at 700°C		
	$\sigma_{0.2}$ (MPa)	UTS (MPa)	Elongation (%)	$\sigma_{0.2}$ (MPa)	UTS (MPa)	Elongation (%)
HIP 1180℃+Ageing	925 922	1474 1470	24 25	827 835	1304 1308	35 30
HIP 1180°C + Heat treated+Ageing	1070 1072	1580 1589	25 24	975 982	1473 1476	25 24

Table 3 The compositions in at% of the large inclusion particles in the as-HIPped RR1000 sample analysed by WDX

Inclusions / Elements	Particle 1	Particle 2	Particle 3	Particle 4	Particle 5	Average
Zr	2.192	3.150	3.047	2.553	2.270	2.642
Hf	24.530	27.464	27.018	25.602	24.391	25.801
O	57.504	61.892	64.568	60.441	56.432	60.167
C	15.774	7.494	5.367	11.405	16.906	11.389

Table 4 Summary of four-point bending fatigue lifetime and crack initiation sites of the as-HIPped samples or HIPped and heat treated samples

Conditions / σ_{max} (MPa)	HIPped	HIPped+Aged	HIPped+Solution treated+ Aged
700		Run out	Run out or 2.8×10^6, hafnia clusters
750	Run out for 2 times	Run out	2.2×10^6 Large cleavage facets
800	1.35×10^6,or 1.67×10^6 hafnia clusters	Run out	1.85×10^6, Large cleavage facets
825			1.25×10^6, Large cleavage facets
850	0.74×10^6, hafnia clusters	Run out or 1.29×10^6, Large cleavage facets	0.61×10^6, hafnia clusters
900	0.99×10^6, Large cleavage facets	0.68×10^6, hafnia clusters	0.81×10^6, hafnia clusters
950	0.91×10^6, Large cleavage facets	0.61×10^6, hafnia clusters	
1000	0.62×10^6, Large cleavage facets		

Table 5 Summary of tension-tension fatigue properties and crack initiators of the HIPped and heat treated samples

Testing conditions	Processing conditions	750 (MPa)	800 (MPa)	850 (MPa)	900 (MPa)	925 (MPa)	950 (MPa)
Room Temperature	HIP+Age	Run out	Run out	Run out	Run out twice	Run out	0.2648×10^6, 9.2189×10^6, hafnia clusters
	HIP+ST+Age	Run out	Run out	Run out	Run out, or 4.9240×10^6, hafnia clusters	-	2.7886×10^6, 6.9034×10^6, hafnia cluster
700℃	HIP+Age	Run out twice	0.7898×10^6, 0.6634×10^6, 0.8499×10^6, hafnia cluster; 9.3996×10^6, Al_2O_3 cluster	0.0243×10^6, 0.9667×10^6 hafnia clusters	-	-	-
	HIP+ST+Age	Run out	Run out, 1.1947×10^6, 0.9933×10^6, hafnia clusters	1.5393×10^6, 1.7901×10^6, hafnia clusters	-	-	-

Table 6 Compositions in at.% of particles in the inclusion clusters analysed using EDX

Element	1	2	3	4	5	6	7	8	Average
C	18.90	16.86	18.68	18.99	22.92	17.47	19.28	16.83	18.74
O	51.66	57.17	49.45	47.90	38.85	38.28	45.49	49.57	47.30
Ti	1.23	0.87	0.94	1.03	1.90	2.14	0.83	0.39	1.17
Cr	1.25	1.37	1.60	1.60	1.98	2.57	1.72	1.66	1.72
Co	1.74	1.72	2.03	1.98	2.30	2.97	1.98	2.24	2.12
Ni	4.79	5.27	5.51	5.75	7.58	8.37	5.94	5.39	6.07
Zr	4.23	4.05	3.89	4.33	3.06	5.03	3.73	1.87	3.77
Hf	16.20	12.68	17.90	18.41	21.41	23.18	21.00	22.06	19.10

Table 7 Chemical composition in at.% of the inclusion shown in Figure 15(d-e) analysed by EDX

Element	O	Al	Ti	Cr	Co	Ni
Concentration, at.%	59.83	37.64	0.19	0.70	0.35	1.29

Figure Captions

Figure 1 Secondary electron SEM micrographs showing γ′ size, distribution and morphology in samples that were HIPped at (S+20)°C and then subject to different heat treatments, (a-b) no heat treatment; (c-d) ageing; (e-f) solution treatment and ageing [18]. GB stands for grain boundary.

Figure 2 (a-b) Back scattered electron SEM micrographs showing the presence of a large inclusion particle in as-HIPped samples and (c) WDX spectrum showing the typical composition of an inclusion particle.

Figure 3 Room temperature four-point bending fatigue properties of samples that were prepared under different conditions. The trend lines are drawn based on the general trend of fatigue lifetime with stress level and the arrows indicate that some of the samples run out during testing at relevant stresses.

Figure 4 Secondary electron SEM micrographs showing the four-point bending fatigue crack initiatiors for the samples that were HIPped, solution treated and then aged, (a) tested at σ_{max}=700MPa, failed at 2.8397×10^6 cycles; (b-c) tested at σ_{max}=750MPa, failed at 2.2087×10^6 cycles; (d) tested at σ_{max}=800MPa, failed at 1.8467×10^6 cycles.

Figure 5 (a) Secondary electron SEM micrograph showing the crack initiation via cleavage during four-point bending fatigue testing. The sample was HIPped and aged, tested at σ_{max}=850MPa and failed at 1.29×10^6 cycles; (b) EBSD image showing grain structure of a cross section right through the fatigue crack initiation facets; (c)Inverse pole figure for the EBSD micrograph. The arrow shows the grain which has been sheared to form the large facets at the crack initiation site.

Figure 6 Secondary electron SEM micrographs showing the typical four-point bending fatigue fracture sequence: (a) fatigue crack initiation followed by striated propagation; (b) transition of striated propagation to monotonic fracture (rougher surface); (c) catastrophic monotonic fracture. The arrow shows the crack growth direction.

Figure 7 Secondary electron SEM micrographs showing the typical four-point bending fatigue crack propagation sequence of as-HIPped or HIPped and heat treated samples at each stage, (a-b) highly crystallographic and faceted crack propagation right after crack initiation, in the lower Paris regime; (c-d) striated crack propagation in higher Paris regime; (e-f) dimple-ductile crack propagation in the final monotonic/static fracture stage; (g-h)crack propagation along some remenant PPBs

Figure 8 Room temperature tension-tension fatigue properties of the samples that were prepared under different conditions. The trend lines are drawn based on the general trend of fatigue lifetime with stress level and the arrows indicate that samples run out during testing at relevant stresses.

Figure 9 Secondary electron SEM micrographs showing the fatigue crack initiation of samples after room temperature tension-tension fatigue testing. (a-f)HIPped and aged samples, tested at σ_{max}=950MPa; (a-d) internal crack initiation, failed at 9.2189×10^6 cycles; (e-f) surface crack initiation, failed at 0.2648×10^6 cycles. (g-h) HIPped+solution treated and aged samples, tested at σ_{max}=950MPa; (g) failed at 2.7886×10^6 cycles, the distance between crack initiator and sample surface *d*=0.57mm; (h) failed at 6.9034×10^6 cycles, *d*=1.32mm.

Figure 10 (a) secondary electron SEM micrograph showing an inclusion cluster (also shown in Figure 9(d)); (b) EDX analysis showing the composition of inclusion particles.

Figure 11 Secondary electron SEM micrographs showing the room temperature tension-tension fatigue fracture surfaces of a HIPped and heat treated sample, (a) cleavage facets at the lower Paris regime; (b) fatigue striations at the upper Paris regime; (c-d) dimple-fracture at the high ΔK regime of the fatigue crack growth, and (e-f) crack propagation at the sites of inclusion clusters. The sample shown here was HIPped, solution treated and then aged, tested at σ_{max}=950MPa and failed at 6.9034×10^6 cycles

Figure 12 Back scattered electron SEM micrographs showing secondary cracks at inclusion clusters on a section of a HIPped, solution treated and aged sample after room temperature tension-tension fatigue test. The sample was tested at σ_{max}=950MPa and failed at 2.7886×10^6 cycles

Figure 13 Tension-tension fatigue properties of samples that were prepared with different conditions and tested at 700°C. The trend lines are drawn based on the general trend of fatigue lifetime with stress level and the arrows indicate that samples run out during testing at relevant stresses.

Figure 14 Secondary electron SEM micrographs showing the crack initiators of samples after tension-tension fatigue testing at 700℃. (a-d) the samples investigated were all HIPped+Aged and tested at σ_{max}=850MPa; (a-b) failed at 0.0246×10^6 cycles; (c-d) failed at 0.9667×10^6 cycles. (e-h)The samples investigated were HIPped+Solution treated+Aged and tested at σ_{max}=800MPa; (e-f) failed at 0.9933×10^6 cycles; (g-h) failed at 1.1947×10^6 cycles.

Figure 15 Secondary electron SEM micrographs showing the crack initiators of samples after tension-tension fatigue test at 700℃. The samples investigated were HIPped+Aged and tested at σ_{max}=800MPa. (a-b) failed at 0.7898×10^6 cycles; (c-d) failed at 9.3996×10^6 cycles; (e)EDX analysis showing the composition of the inclusion in Figure 14 (d), suggesting that this inclusion should be Al_2O_3.

Figure 16 Secondary Electron SEM micrographs showing the fracture surfaces of samples after tension-tension fatigue test at 700℃, (a-b)crystallographic and faceted fracture after crack initiation; (c-d)fracture in a ductile striation mode; (e-f)transgranular fracture in the final crack propagation stage. The sample shown here was HIPped+Solution treated+Aged and tested at σ_{max}=800MPa, failed at 1.1947×10^6 cycles. The arrow shows the detailed structure of a facet showing γ′ have been cut through.

Figure 17 (a-b) Photographs showing the samples after tension-tension fatigue testing and (c-d) secondary electron SEM micrographs showing the typical general fracture surfaces of samples after tension-tension fatigue testing; (a,c) tested at room temperature; (b,d) tested at 700℃. Type I samples, HIPped+Aged; type II samples, HIPped+Solution treated+Aged.