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Fundamental mechanisms of surface damage associated to the localization of the plastic deformation in fatigue

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Abstract

Nowadays fatigue damage is becoming a very important issue for it may lead to a service failure at any moment. In low cycle fatigue, the crack initiations influence strongly the fatigue life of a material. The study done here is carried out on WaspaloyTM at its under-aged state, a superalloy based Nickel characterized by the hardening precipitates, γ' in matrix Nickel, γ . Fatigue resistance depends on the material microstructure. Damage mechanisms due to the localization of plastic deformation by fatigue are studied on the precipitate and grain from nano to meso scale. This work aims to have a better understanding on the crack initiation mechanisms related to the metallurgical parameters: the influence of the grain and γ' size. Complete investigation is performed by using multiple analysis instruments: SEM and AFM.

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Keywords: low cycle fatigue, superalloy based Nickel, extrusion height, crack nucleation, microstructure, heat treatment

1. Introduction

Fatigue damage in polycrystal such as a face-centered cubic (fcc) metal especially in superalloy based-nickel, WaspaloyTM is related to a surface phenomenon. Under cyclic loading during fatigue, slip bands may emerge in form of extrusions and intrusions on the material surface, as a result of a localization of the plastic deformation induced by the irreversible motion of dislocations [1-5]. These irreversible surface markings arisen from the surface step called extrusion heights can be analysed by atomic force microscopy (AFM) [3-6]. Slip bands may be the origin of fatigue crack initiation in which its presence is used to define fatigue crack nucleation [1-3].

In previous work, *Risbet et al.* [2-5, 7] has shown that attaining a critical extrusion height of 50 nm is not a sufficient condition to induce the apparition of a transgranular crack. Crystallographic Schmid factor does not have any influence on the crack initiation. Further investigations are to be made to clarify the crack nucleation condition.

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Microstructure of a material in term of the grain and precipitate sizes may influence strongly on the fatigue resistance particularly in cracks initiation [6-8].

This paper work is an extension study done by *Risbet et al.* [2-5, 7]. The purpose of this paper work is to have a better understanding on the surface damage mechanisms leading to crack nucleation related to the localization of the plastic deformation of an under aged polycrystalline Ni-base superalloys (Waspaloy™) in term of metallurgical parameters : grain and precipitate sizes. Heat treatments are performed in order to obtain controlled grain and precipitate sizes for this study [9-11]. Interrupted fatigue tests are carried out until the crack initiation state in order to track the evolution of the extrusions heights in which certain extrusion may transform to cracks. In this work, crack is stated as in the initiation phase when its length is equivalent to a dimension of a grain size.

Present study focuses on the influence of grain and precipitate sizes on the distribution of extrusion height in both damaged and undamaged grains characterized by the variance (σ^2), average and maximal extrusion heights values. Analyses were performed with scanning electron microscopy (SEM) in order to detect the crack nucleation and with AFM in order to investigate the extrusion height for both interrupted fatigue state and crack nucleation phase. Chemicals compositions in damaged grains are as well analysed to see if there is any influence of chemical elements on the crack nucleation.

Nomenclature

ϵ_a	plastic strain amplitude
d_{ave}	average grain size
$d_{\gamma_{ave}}$	average precipitate size
h_{ave}	average extrusion height
h_{max}	maximal extrusion height
σ^2	variance
$\Delta\sigma$	difference in strength

2. Materials and experimental procedure

2.1. Materials

Experiments were performed in a face-centered cubic (fcc) polycrystal, Waspaloy™, a superalloy based nickel having the following chemical compositions in wt %: 1.80 Al; 3.33 Ti; 12.64 Co; 19.45 Cr; 7.31 Mo; balance Ni. This material is strengthened by spherical precipitates, γ' (Ni_3Al). In the as-received state the average grain size is 67 μm and the average precipitate diameter is 45 nm. No crystallography texture was observed. Only under aged specimens were investigated in these studies. Under cyclic loading during fatigue test, precipitates are sheared due to plastic deformation causing localization of plastic strain within bands.

2.2. Description of experimental procedure

2.2.1. Heat treatment

Before proceeding with fatigue tests, heat treatment experiments have been performed in vacuum furnace to select an appropriate range of grain and precipitate size. It is proposed in this work that precipitate size should not cross the threshold value of 50 nm in order to remain in the dislocation particle shearing deformation mechanism. Precipitates achieving larger size of 50 nm will become bypassed and looped by the dislocation rather than cut or

sheared by dislocation. As a consequence slip planarity and strain localization mechanisms will be reduced and this is not the purpose of our study.

Heat treating process parameters are temperature and aging time which play an important role in determining grain and precipitate size. Increased temperature and aging time lead to precipitate coarsening and result in Orowan looping due to the transition from dislocation particle shearing to precipitate bypassing with increasing precipitate size. A set of heat treatments under different conditions providing a range of controlled grain size with a same precipitate size inversely was chosen for further investigation. In this paper, only one heat treatment condition is selected. The specimen underwent 2 heat treatments steps in a Thermolyne 4800 non vacuum furnace. The initial heat treatment step was a solutionizing treatment at 1100 °C for 4 h followed by oil quenching. This stage consists in dissolving precipitates. Subsequently, treatment was conducted at 550 °C for 4h followed by air quenching. This is a re-precipitation and precipitation growing step. This treatment condition resulted in an average grain size of 100 μm and an average precipitate dimension of 7 nm.

As mentioned above, the mean grain size is respectively 67 μm in an as received state, called C3 and 100 μm in the heat treated state, called C2. The difference in strength, $\Delta\sigma$ calculated for these 2 grain sizes using Hall-Petch relationship is equal to 28 MPa which is negligible. From the metallurgical point of view, grain sizes of these 2 metallurgical states can be considered to be the same [11].

2.2.2. Surface preparation and SEM observation

Besides facilitating surface investigation by both SEM and AFM, surface preparation is important as the surface roughness may have an impact on the life duration. Specimens were mechanical polished down to 4000 grit SiC paper. In order to visualize microstructures like grains, specimens were chemical etched in a mixed solution containing 2/3 hydrochloric acid and 1/3 nitric acid for a duration of 1min 30.

Surface observation has been performed on a (FEG) Zeiss Sigma scanning electron microscope (SEM) especially in crack nucleation detection. Chemical composition analysis has been performed on energy dispersive X-ray spectrometer (EDX) where EDX and SEM were coupled together. The purpose of this analysis is to see the influence of chemical elements and compositions on crack initiation mechanism.

2.2.3. Fatigue experiments

Low cycle fatigue tests were conducted using Instron servohydraulic machine at ambient temperature on hourglass cylindrical specimens with a gauge length of 15 mm and a gauge diameter of 6 mm. Tests were carried out on a controlled plastic strain amplitude of $\Delta\varepsilon_p/2 = 0.3\%$ with a fully reversed triangular wave form and were interrupted after 100 cycles in order to track the evolution of the extrusions heights with AFM and also to detect crack nucleation by SEM investigation. Experiment was stopped when cracks fulfill this defined initiation criterion: crack is stated as in the initiation phase when its length is equivalent to a dimension of a grain size. Crack initiation life for the as received specimen, C3 is in order of 1000 cycles. At 1000 cycles, early stage of crack propagation in about 10 grains is already observed on the specimen surface.

2.2.4. Procedure to perform AFM measurements

The surface relief was examined using Dimension 3100 Nanoscope microscopy at ambient temperature in Tapping Mode to obtain images of slip bands. Due to the hourglass cylindrical form, the observed zone is limited to 300 μm x 300 μm . The microscope is equipped with an optical camera which facilitate probe tip positioning on a pre-selected area. The topographic images on selected area were monitored until cracks nucleation phase.

In average for every test, 10 grains was investigated. Each image represents a section of 10 μm x 10 μm showing slip bands of each investigated grains. By using the implemented software, 2 types of measurements were made for each micrograph: (1) a transversal extrusion direction measurement is used to measure the mean level of grain considered as the origin of extrusion ($h = 0$) (2) a lengthways extrusion direction along extrusion line measurement is used to measure the evolution of extrusion height for each extrusion. Each profile showing the extrusion height was then discretized and interpolated into a length step of 40 nm in order to obtain statistic distribution of extrusion height [6].

Statistic distribution of extrusion height in each grain was compared and analyzed using normalization procedure. Probability of the density function, P normalized with the mean height $\langle h \rangle$, $\langle h \rangle P$ was plotted against $h/\langle h \rangle$. $P = P(h, h+\Delta h)$ defined probability that any emergence height has a size between h and $h+\Delta h$. The bin width $\Delta h = \Delta h/3 =$

20 nm. The metallurgical situation is analyzed by using gamma distribution with 2 parameter for each grain, average extrusion height, $\langle h \rangle$ and variance, σ^2 .

3. Results and Discussions

Table 1 : Investigated specimens with different metallurgical states

Name	d_{ave} (μm)	$d\gamma'_{ave}$ (nm)
C1	30	7
C2	100	7
C3	65	45

Investigated specimens having different metallurgical states are shown in) 000–000 7. C1 is a specimen investigated previously by *Risbet et al.* C2 is a heat treated specimen while C3 is a specimen in the as received state. These specimens are different in term of metallurgical states. C1 and C2 have different grain size with a constant precipitation size while C2 and C3 have different precipitation size with a constant grain size.

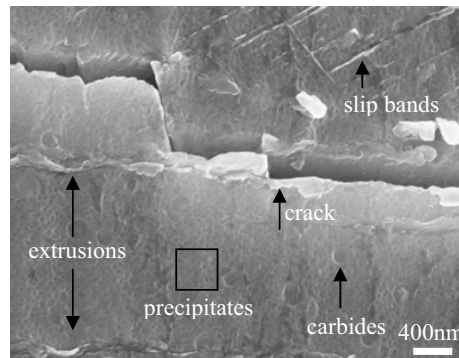


Fig. 1 : Microstructures of Waspaloy™, C3 cycled at $\epsilon_a = 0.3\%$ after attaining crack initiation life in order of 1000 cycles

Microstructural features of one metallurgical state of Waspaloy™, C3 cycled at $\epsilon_a = 0.3\%$ after attaining crack initiation life in order of 1000 cycles are shown in Fig. 1. In this SEM image, extrusions, precipitates, carbides and transgranular cracking can be clearly observed. Fatigue loading on this specimen after 1000 cycles results in most of transgranular damages initiations at extrusion. A few crack initiations are observed to be intergranular.

3.1. Chemical composition investigation

Chemical composition analysis has been performed on energy dispersive X-ray spectrometer (EDX). EDX and SEM were coupled together. The purpose of this analysis is to see if there is any influence of chemical elements and compositions on crack initiation mechanism.

Chemical compositions of a few elements such as aluminum, titanium, cobalt, chromium and molybdenum have been analyzed on a few damaged grains of specimen C3 after cracks nucleation. Tests have been conducted on 2 different zones within a grain: one nearby crack and the other far from crack. The distance of the second zone away from crack varies from 11 to 23 μm depending on the grain size. The width of these zones is not more than 1 μm .

No convincing result concerning the variation of chemical elements compositions were seen between both zones except for molybdenum. It is shown that there is a variation of molybdenum content in both studied zones of a few

analyzed damaged grains. The weight percentage of this element is higher in cracking zone as shown in Table 2, except for G1.

To validate this observation, a further analysis of high resolution chemical element mapping done by using SEM-EDX will be done in order to identify the presence of molybdenum in cracking zone. Besides chemical composition analyses on C2 are also in progress.

Table 2 : Variation of molybdenum quantities in weight percentage in 2 different zones within a same grain of a few grains.

Grains	(Wt% of Mo)	
	Near crack	Away from crack
G1	7.96	8.47
G2	8.38	7.43
G3	8.83	7.91
G4	11.81	8.81

3.2. AFM investigation on interrupted tests

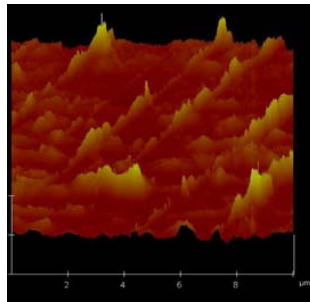


Fig. 2 : Extrusion of an undamaged grain of specimen C3 cycled at $\epsilon_a = 0.3\%$ after attaining crack initiation life in order of 1000 cycles

Surface relief of specimens was examined by AFM in order to investigate the evolution of extrusion height for both interrupted fatigue state and crack nucleation phase. Fig. 2 shows an example of extrusions of an undamaged grain on the cylindrical surface of C3 after cracks nucleation. h_{ave} for this topographic view is 47 nm while h_{max} is 187 nm. In this figure, extrusions are parallel one to another and are inclined about 45° to the loading axis.

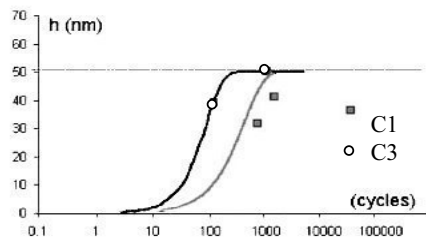


Fig. 3 : Average height evolution with the number of cycles N for different specimens undergo plastic strain amplitude of 0.3%.

Evolution of h_{ave} with number of cycles N of 2 specimens, C1 (specimen investigated previously by *Risbet et al.* [6]) and C3 are shown in a continuum curve in Fig. 3. Both specimens undergo same experimental condition. SEM observation is performed to detect extrusion appearance and cracks nucleation.

Analyses for specimen C3 show that cracks nucleate earlier than 1000 cycles. SEM observation shows that at 1000 cycles, early crack propagation is observed in few damaged grains. Global evolutions of extrusion height for both specimens seem to be a like. The behavior of C3 differs from C1 as the extrusion height reaches a saturation of 50 nm with an increasing N earlier in C3.

Table 3 : Quantitative results of AFM investigation on (a) interrupted state and (b) on nucleation phase.

$\epsilon_a = 0.3\%$ (interruption)						$\epsilon_a = 0.3\%$ (crack nucleation)					
Name	d_{ave} (μm)	$d\gamma_{ave}$ (nm)	h_{ave} (nm)	h_{max} (nm)	Cycles	Name	d_{ave} (μm)	$d\gamma_{ave}$ (nm)	h_{ave} (nm)	h_{max} (nm)	Cycles
C1	30	7	32	157	750	C1	30	7	41	250	1500
C2	100	7	80	192	100	C3	65	45	51	178	1000
C3	65	45	40	129	100						

The investigation results by AFM on 3 specimens are shown in Table 3. C1, C2 and C3 are used in order to compare the influence of different metallurgical states on the average and maximal extrusion heights. C1 and C2 do have same precipitate size with different grain size whereas C2 and C3 do have same grain size with different precipitate size. As mentioned in §2.2.1, the difference in strength, $\Delta\sigma$ is negligible, so that from the metallurgical point of view, these 2 metallurgical states have almost the same grain size [11].

As observed in Table 3a), C2 has the highest average and maximal height among all investigated specimens during interrupted state. Table 3b) shows results of C1 and C3 at cracks nucleation phase. C1 has a lower average extrusion height contrary to C3. Inversely C1 has a higher maximal extrusion height compared to C3.

These studies show that cracks might only nucleate in damaged grains with extrusion height attaining 50 nm, as reported by *Risbet et al.* [6]. It seems in these investigations that grain size does have an effect on the evolution of the extrusion height for both specimens having same precipitate size. Smaller precipitate size with constant grain size may probably induce higher extrusion height. Meanwhile further investigations on other metallurgical states are necessary in order to validate these remarks.

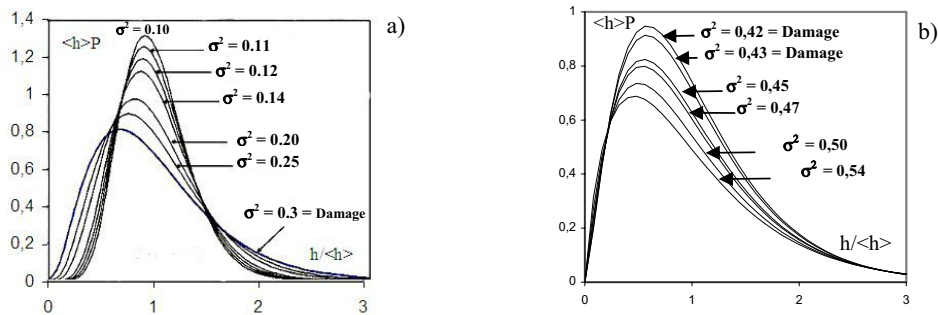


Fig. 4 : Distribution functions of extrusion height for damaged and undamaged grains in (a) specimen C1 and (b) specimen C3

Fig. 4 shows distribution of extrusion height of both damaged and undamaged grains for specimen C1 and C3. It is reported in the previous work as shown in Fig. 4a) that the variances, σ^2 of specimen C1 are equal to 0.3 for damaged grains and are lower than 0.3 for undamaged grains. The variances found for specimen C3, as shown in Fig. 4b) are not in accordance with this previous work. It is shown that the σ^2 is equal to 0.42 ± 0.01 for damaged grains where as σ^2 of undamaged grains range from 0.43 to 0.54. It is interesting to note that all damaged grains in C1 or C3 do have same variance value although this value is different in C1 and C3.

Both specimens do not result in same variance value in damaged grains. To explain this fact, the effect that precipitate size distribution may have on the dislocation free movement in slip bands is being examined. As the

precipitate size distributions are different in both specimens, the dislocation free movement in slip bands is probably not the same from one specimen to another. As the precipitate inter-distance is related to precipitate size distribution, transmission electron microscopy (TEM) investigations are in progress in order to go into detail.

4. Conclusion

Present study focuses on the influence of grain and precipitate sizes on the average extrusion height defining crack nucleation criterion. Heat treatments were performed in order to obtain controlled grain and precipitate size for this work. A greater Molybdenum content is shown nearby crack within damaged grain by chemical composition analysis. Based on the statistical analysis of the extrusion height, all damaged grains have the same variance. Besides the variance calculated for damaged grains is not in accordance with previous work. As a consequence, the value of variance may vary from one metallurgical state to another. Microstructures like grain and precipitate size may influence the average and maximal height of extrusions as well as crack initiation life. Larger grain size results in a higher value of average extrusion height. Further and additional investigations by TEM are in progress in order to validate remarks reported in this work

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