EFFECT OF NON-METALLIC CERAMIC INCLUSIONS ON STRAIN LOCALIZATION DURING LOW CYCLE FATIGUE OF A POLYCRYSTALLINE SUPERALLOY

Jean-Charles Stinville¹, Victoria M. Miller¹, Tresa M. Pollock¹

¹Materials Department; University of California, Santa Barbara; Santa Barbara, CA 93106

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Abstract

The presence of inclusions in powder metallurgical processed disk materials often leads to competing failure modes and a wide variability in fatigue durability in low cycle fatigue. For robust models for fatigue and prediction of the variability in this property, the impact of such inclusions on the fatigue crack initiation process has been studied. Fatigue experiments in the low cycle fatigue regime on an intentionally seeded René 88DT polycrystalline nickel-base superalloy were performed at 400 °C and 650 °C to evaluate the effect of non-metallic ceramic inclusions on the strain distribution and fatigue initiation process. Advanced high resolution digital image correlation measurements were performed to related strain fields at the grain scale to microstructure surrounding non-metallic inclusions.

Introduction

Nickel-base polycrystalline superalloys exhibit superior high temperature mechanical properties required for turbine disk applications [1]. To minimize segregation induced by high alloy content of these advanced nickel-base disk alloys, powder metallurgy processing is preferred over conventional cast and wrought processing [2, 3]. One concern is the introduction in the alloy of ceramic inclusions inherent in the powder metallurgy process. The presence of inclusions often leads to competing failure modes in low cycle fatigue [3] and to a wide variability in fatigue durability [4].

For models to predict fatigue endurance and characterise the variation in fatigue life, the impact of such inclusions must be assessed. In the superalloy René 88DT, it has been shown [3] that inclusions can serve as the initiation sites for fatigue, with lifetime strongly correlated to inclusion size. However, the significant scatter observed in fatigue lifetimes indicated that other parameters such as inclusion shape and microstructure in the vicinity of the inclusion affect the fatigue initiation and propagation processes. Of particular interest, recent studies [5, 6] have shown that the heterogeneous distributions of plastic strain and stress that develop in the vicinity of inclusion drives the initiation and early propagation processes. In addition, it appears that the cohesion of the inclusion-nickel matrix interface and inclusion fracture are factors that affect significantly the local strain distribution around inclusions [5, 6], and therefore the crack initiation process.

The objective of the study is to characterize experimentally and numerically the crack initiation and propagation process related to the presence of inclusions in low cycle fatigue at intermediate temperature. Critical information on microstructure, strain distribution, fatigue initiation and propagation processes has been gathered through advanced experimental techniques including

Electron BackScatter Diffraction (EBSD), high resolution digital image correlation under scanning electron microscopy (HR-DIC) at the grain scale and transmission electron microscopy (TEM) to provide critical data for development and validation of fatigue models.

Experimental

Because inclusions are relatively rare in conventionally processed materials, a seeding approach was employed. The seeded René 88DT was prepared by powder metallurgy processing where alumina (Al_2O_3) seeds were added to the powders. Fine powders of René 88DT alloy and Al_2O_3 particles were mixed and hot isostatic pressed into compacts. The seeded and pressed compacts were then forged and heat treated to produce the final microstructure. The polycrystalline powder metallurgy processed nickel-base superalloy, René 88DT, has a nominal composition of 13 %Co, 16 %Cr, 4 %Mo, 4 %W, 2.1 %Al, 3.7 %Ti, 0.7 %Nb, 0.03 %C, 0.04 %Zr, 0.015 %B (wt%) [7]. The standard heat treatment consists of a super solvus solution of 1 hour at 1150 °C, followed by a delayed oil quench, and aging for 8 hours at 760 °C. The microstructure of the alloy consists of a γ matrix and two populations of gamma prime (γ') precipitates: larger secondary and nm-scale tertiary γ' within the γ grains [7]. The size of the secondary γ' phase is about 100-200 nm, while tertiary γ' precipitates are typically less than 30 nm in diameter. Crystallographic features have been previously studied using EBSD measurements [8] showing that the material possesses very weak crystallographic texture, a large population of $\Sigma 3$ boundaries (58 % of the total boundary fraction), an average grain size of 26 μm , and a low fraction of large grains on the order of two to five times of the average grain size. Figure 1(a and c) present two electron microscopy images of inclusions embedded in the superalloy matrix. The associated neighboring microstructure is presented in Figure 1(b and d) using EBSD measurements. Monotonic testing revealed a 0.2 % offset yield strength of 985 MPa (≈ 0.7 % total strain) at 593 °C [9].

Cyclic fatigue testing was performed in air at 400 °C and 650 °C in the low cycle fatigue regime at a high and low level of plasticity on seeded specimens. Low cycle fatigue tests were performed in a symmetric, uniaxial, push-pull mode on an electromechanical machine in stress control mode at maximum stresses of 758 MPa and 965 MPa, with a R-ratio of -1 or -0.5, respectively, and a frequency of 1 Hz. Stress-strain curves from the first cycle showed elastic behavior for the low stress condition and limited plasticity in the hysteresis loop from the high stress condition. Cylindrical dogbone-shaped specimens with a gage diameter of 5 mm and gage length of 16 mm were used in this study. The samples were heated using an induction coil and the testing temperature, measured with an infrared pyrometer, was controlled within ± 10 °C along the gage section of fatigue specimens.

Figure 1. (a), (c) Secondary electron images of non-metallic inclusions of Al_2O_3 type prior to fatigue loading. (b), (d) Associated microstructure from EBSD measurements showing embedded inclusions.

Figure 4. (a) Crack initiation at the inclusion-matrix interface during an interrupted test (80 % lifetime) in the low cycle fatigue regime for a seeded specimen tested at 400 °C and at maximum stress of 965 MPa. (b) High magnification image of the interface inclusion-matrix. The loading direction is horizontal.

Figure 5. Crack size in the superalloy matrix during interrupted test at different cycle number (1 cycle, 5 %, 23 % and 60 % lifetime) in the low cycle fatigue regime for a seeded specimen tested at 400 °C and at maximum stress of 965 MPa for two inclusions. The associated microstructures surrounding the inclusions are presented in Figure 1(a and c)

the previously described advanced high-resolution digital image correlation technique combined with imaging within a scanning electron microscope. This technique permits characterizing the plastic deformation in relation to the microstructure of the superalloy that develops during cycling at 400 °C and 650 °C.

Significant plastic deformation in the form of bands of concentrated strain along {111} face centered cubic slip systems occurred after the first cycle around the inclusion in the pole zones (top and bottom region surrounding the inclusion in reference to the horizontal loading direction). The equatorial zones (left and right region) around the inclusion exhibit low levels of residual plasticity. Strain localization is observed along octahedral slip systems and near twin boundaries. Such observation is consistent with strain localization classically observed in the unseeded René 88DT superalloy [8]. Cracks displayed in Figure 5 initiated and propagated in the superalloy matrix in the pole zones around the inclusions where strain localization was significant. It must be noted in Figure 6(d) that residual strain localization is observed in tension in the pole zones around the inclusion and in compression in the equatorial zone. Strain localization in compression indicated by bands of negative concentrated strain (purple bands indicated by black arrows) was observed near twin boundaries, in Figure 6(d). This indicates that during the compressive phase of the cycling, the equatorial zone experienced compressive yielding. Residual strain localization in the pole zones are the highest at the inclusion-matrix interface indicating that these interfaces are more likely the location of crack initiation in the superalloy matrix. Defects or local damage at the interface in the pole zone prior to cycling are the origin of crack initiation (Figure 4(b)).

Low Stress Cycling

For the low level of plasticity (maximum stress of 758 MPa, significantly below the yield stress of the superalloy), plastic strain fields develop during cycling (Figure 7). At lower stresses, strain localizes in the form of bands at specific locations within the microstructure. Enhanced straining is observed near twin boundaries during cycling and favor crack initiation and propagation around the inclusion as displayed in Figure 8. Contrary to strain localization at high level of plasticity, most of the bands of concentrated strain are disconnected from the inclusion-matrix interface. Figure 8(a) shows the surface of the matrix surrounding the inclusion in Figure 7(d) after 70 % of the lifetime. The exact location of the secondary electron image in Figure 8(a) is displayed by the arrow in Figure 7(d). Figure 8(b) presents a transmission electron microscopy image at the location indicated by the dashed line in Figure 8(a). Plasticity developed during cycling at high temperature in the form of multiple slip bands parallel to twin boundaries at specific locations in the matrix, dependent on the microstructure and the strain field induced by the presence of the inclusion. Such observation is consistent with the crack initiation process involved in the unseeded René 88DT superalloy [8]. At low levels of plasticity, microstructural features (specific twin boundaries) around the inclusion favor crack initiation. Inclusions act as a stress raisers and favor crack initiation in the microstructure surrounding the inclusion. It must be noted that defects or local damage at the interface have been also observed to initiate strain localization and cracks at the low level of plasticity as displayed in the bottom pole zone in Figure 7(d).

Interface Cohesion

Experimental strain fields are compared to simple elastic finite element calculations within the commercial ABACUSTM FE code considering both cohesion (Figure 9(a and b)) and no cohesion (Figure 9(c and d)) at the inclusion-matrix interface for the inclusions displayed in Figure 1. For simplicity the matrix is considered as a homogenous material with the average elastic modulus of the superalloy ($E=176$ GPa). Al_2O_3 ceramic inclusions display higher modulus ($E=300$ GPa). In addition, the effect of thermal expansion is not considered. Comparing the experimental strain field (Figure 6) and the equivalent elastic calculation (Figure 9), it is apparent that the deformation during the tensile phase of the cycle in the matrix is consistent with weak cohesion at the inclusion-matrix interface. This observation explains that crack initiation in the matrix will occur with crack oriented normal to the applied tensile stress in the pole zone of the inclusion and highlight the importance of the shape and size of the inclusion in the fracture processes. Fast Fourier transformation calculations using the full 3D description of the local microstructural environment are in progress to better assess the role of local grain orientations in influencing the strain distributions around the inclusions. Experimental strain fields in Figure 6(d) display compressive strain localization in the equatorial zones surrounding the inclusion that develop during compressive phase of cycling. This suggests cohesion or strong inclusion-matrix contact may exist during the compressive phase. It was found that elastic deformation of the inclusion during compression maintained good cohesion with the alloy at the interface, which appeared to affect the strain distribution in the superalloy matrix.

Conclusions

The experiments performed using HR-DIC provide evidence of strain localization around inclusions in relation to the geometrical characteristics of the inclusions and to the microstructure features around them. These observations collectively indicate that fatigue initiation is governed primarily by geometrical characteristics of the inclusion and the strain localization adjacent to the inclusion during cycling at high levels of plasticity. It has been observed that the damage prior to the fatigue test induced during the powder metallurgy process in the matrix (micro cracks) and at the inclusion-matrix interface is also a major factor influencing crack initiation in the seeded specimens especially at high levels of plasticity. During cycling at low level of plasticity, strain localization is observed near twin boundaries that can be disconnected to the interface inclusion-matrix. In addition to initiation at the interface, initiation at microstructural features was observed. Experimental strain fields indicate that ceramic inclusions are weakly adhered to the matrix and therefore do not sustain significant stresses during tensile loading.

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Figure 6. Sub-grain plastic strain field using advanced HR-DIC by electron microscopy at the grain scale during fatigue cycling at intermediate temperature at high level of plasticity around the inclusion displayed in Figure 1(a and c) after the first cycle (a-b) and 5 % lifetime (c-d). The arrows in (d) indicate areas of compressive strain.

Figure 7. (b-d) Sub-grain plastic strain field using HR-DIC by electron microscopy at the grain scale during fatigue cycling at intermediate temperature at low level of plasticity around the inclusion displayed in (a) after the first cycle (b), 10 % lifetime (c) and 70 % lifetime (d). (a) EBSD measurement of the microstructure around the inclusion.

Figure 8. (a) Secondary electron image of the surface of a seeded specimen after cycling at 400 °C and at maximum stress of 758 MPa. (b) Transmission electron microscopy image at the location indicated by the dashed line in (a).

References

1. R.C. Reed, *The Superalloys* (Cambridge, UK: Cambridge University Press, 2006).
2. D.R. Chang, D.D. Krueger, and R.A. Sprague, "Superalloy Powder Superalloy Powder Processing, Properties, and Turbine Disk Applications," *Superalloys 1984*, ed. by M. Gell et al. (Warrendale, PA: TMS-AIME, 1984), 251-257.
3. E.S. Huron and P.G. Roth, "The Influence of Inclusions on Low Cycle Fatigue Life in a P/M Nickel-Base Disk the Influence of Inclusions on Low Cycle Fatigue Life in a P/M Nickel-Base Disk the Influence of Inclusions on Low Cycle Fatigue Life in a P/M Nickel-Base Disk Superalloy," *Superalloys 1996*, ed. by R.D. Kissinger et al. (Warrendale, PA: The Minerals, Metals Materials Society, 1996), 359-368.
4. D. Texier et al., "Microstructural Features Controlling the Variability in Low-Cycle Fatigue Properties of Alloy Inconel 718 at Intermediate Temperature," *Metallurgical and Materials Transactions A*, 2016, 1-14.
5. T. Zhang et al., "Slip Localization and Fatigue Crack Nucleation Near a Non-Metallic Inclusion in Polycrystalline Nickel-Based Superalloy," *Materials Science and Engineering: A*, 641 (2015), 328 - 339.
6. J. Jiang et al., "On the Mechanistic Basis of Fatigue Crack Nucleation in Ni Superalloy Containing Inclusions Using High Resolution Electron Backscatter Diffraction," *Acta Materialia*, 97 (2015), 367 - 379.

Figure 9. Elastic finite element calculations of Von Mises stress from uniaxial tension at 965 MPa considering (a-b) perfect cohesion at the inclusion-matrix interface and (c-d) no cohesion at the inclusion-matrix interface for the inclusions displayed in Figure 1. The loading direction is horizontal.

7. S.T. Wlodek, M. Kelly, and D.A. Alden, "The Structure of René88DT," *Superalloys 1996*, ed. by R.D. Kissinger et al. (Warrendale, PA: The Minerals, Metals Materials Society, 1996), 129-136.
8. J. Miao, T.M. Pollock, and J.W. Jones, "Crystallographic Fatigue Crack Initiation in Nickel-Based Superalloy René 88DT at Elevated Temperature," *Acta Materialia*, 57 (20) (2009), 5964-5974.
9. M.J. Caton et al., "Divergence of Mechanisms and the Effect on the Fatigue Life Variability of the René 88DT," *Superalloy 2004*, ed. by K.A. Green et al. (Warrendale, PA: The Minerals, Metals Materials Society, 2004), 305-312.
10. J.C. Stinville et al., "A Combined Grain Scale Elastic-Plastic Criterion for Identification of Fatigue Crack Initiation Sites in a Twin Containing Polycrystalline Nickel-Base Superalloy," 103 (2015), 461–473.
11. Vic-2D, "[software](correlated Solution Inc., Colombia, Sc)," 2009.
12. M.A. Sutton et al., "Scanning Electron Microscopy for Quantitative Small and Large Deformation Measurements Part I: SEM Imaging at Magnifications From 200 to 10,000," *Experimental Mechanics*, 47 (6) (2007), 775–787.
13. J.C. Stinville et al., "Sub-Grain Scale Digital Image Correlation by Electron Microscopy for Polycrystalline Materials During Elastic and Plastic Deformation," *Experimental Mechanics*, 2015, 1–20.
14. A.D. Kammers and S. Daly, "Small-Scale Patterning Methods for Digital Image Correlation Under Scanning Electron Microscopy," *Measurement Science and Technology*, 22 (12) (2011), 125501.
15. A.D. Kammers and S. Daly, "Self-Assembled Nanoparticle Surface Patterning for Improved Digital Image Correlation in a Scanning Electron Microscope," *Experimental Mechanics*, 53 (8) (2013), 1333–1341.