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# On the fatigue small crack behaviors of directionally solidified superalloy DZ4 by in situ SEM observations

Xianfeng Ma, Hui-Ji Shi\*

AML, School of Aerospace, Tsinghua University, Beijing 100084, PR China

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#### ABSTRACT

Small fatigue crack behaviors in a nickel-based directionally solidified superalloy DZ4 were studied by in situ Scanning Electron Microscopy. The crack initiation and propagation manners were identified under different temperatures, i.e. 25 °C, 350 °C, 700 °C. Fatigue crack growth occurred preferentially along slip bands at 25 °C and 350 °C but by Mode-I type at 700 °C. The crack growth rate generally increased with temperature, especially between room temperature and 350 °C. The anomalous small crack growth was analyzed by in situ examining the effect of microstructure. The small cracks were found to be primarily microstructurally small and secondly mechanically small.

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#### 1. Introduction

Ever since the pioneer work of Pearson [1] on the "small" crack behavior in aluminum alloys, it has been gradually recognized that the cracks on the order of, or less than, the microstructural size tend to grow faster than the large cracks driven by the same nominal stress intensities. Morris's following research on Al alloys [2] showed that the interactions between fatigue crack and grain boundary played an important role in the crack opening behavior. Nowadays, with the progress of the directionally solidification processing technique, the work temperature of turbine has been greatly increased in the past decades due to the application of directionally solidified (DS) superalloys with excellent fatigue and creep resistance [3]. For aerospace blades and discs, it is found that the critical crack size for final fracture can be close to the small crack regime [4]. It has been widely accepted that [5,6] the small crack propagation takes up most of the fatigue life time, especially for the cleaner materials of high surface quality in aerospace structures. Moreover, regarding the so-called small crack phenomenon, i.e. much faster crack growth rate and lower crack threshold, nonconservative fatigue life prediction could occur if small crack behavior is not well incorporated in fatigue life calculation [6]. Insofar as the research goes, the anomalous small fatigue crack growth behaviors have been widely studied in polycrystalline metals [4-7] and more recently in single crystal superalloys [8,9]. It is The small-crack effects in polycrystals have been usually attributed to the blocking contribution of grain boundary (GB) [6]. Hence, studying the small crack growth in DS or single crystal alloys would have the advantage of eliminating the pronounced GB effects, focusing on the interaction with other factors (e.g. metallurgical microstructure and crystal orientation [9]) and other mechanisms responsible for small crack [8]. Furthermore, it has been recognized that the effect of temperature on the small crack behavior can be significant, as reported in aluminum alloys, etc. [10]. Stephens's study [6] on Waspaloy alloys showed that the temperature can evidently altered the fatigue crack formation, the slip character and thus, the crack growth rate. However, due to lack of solid experimental studies, this influence has not been well understood in DS superalloys, especially in the realistic engine temperature regime.

DZ4 is a directionally solidified nickel-based superalloy developed for fabricating high-performance gas-turbine blades, components, etc. in China [3]. In this study, the fatigue small crack behaviors of DZ4 superalloy at different temperatures, i.e. 25 °C, 350 °C, 700 °C, were investigated by in situ SEM fatigue testing. The effects of temperature on the fatigue fracture mechanisms and the small crack behaviors were addressed.

#### 2. Material and experimental procedure

The material used in this investigation is a directionally solidified nickel-based superalloy DZ4 with a nominal composition (in

E-mail addresses: maxianfeng@gmail.com (X. Ma), shihj@mail.tsinghua.edu.cn (H.-J. Shi).

amazing to notice that this phenomenon has been rarely reported in DS superalloys.

<sup>\*</sup> Corresponding author.

wt.%): 0.14C, 9.5Cr, 6.0Co, 5.3W, 3.8Mo, 6.0Al, 1.8Ti, 0.02B and Ni balance [11]. Specimens with detailed dimensions shown in Fig. 1a were cut with gauge length parallel to the longitudinal orientation. The single edge notched specimen had a notch at the center of the specimen with a radius of about 30  $\mu m$  and a depth of about 50–80  $\mu m$ . The notch was produced by the edge of a knife to create local stress concentration and favor crack initiation consequently.

The polished faces of the samples were etched in a aqueous solution of 4 g CuSO<sub>4</sub> + 20 mL HCl + 20 mL H<sub>2</sub>O at room temperature prior to fatigue tests in order to reveal the prevailing microstructure for in situ observations under SEM. The directionally solidified structure is composed of paralleled columnar grains with their  $\langle 0\,0\,1\rangle$  axis along the growth direction (Fig. 1b). No effect of the etching on the crack path was found.

The fatigue crack propagation tests were performed in the vacuum chamber of the Scanning Electron Microscopy (SEM) using a specially designed servo-hydraulic testing system. This machine provided pulsating loads at 10 Hz of ±1 kN maximum capacity and a displacement range of  $\pm 25$  mm. This servo-hydraulic testing system provides excellent control of cyclic loading. A maximal stress of 850 MPa was adopted through the test to study the fatigue crack propagation in low-cycle fatigue. The waveform utilized was sinusoidal. All fatigue tests were load controlled at a stress ratio of 0.1 with a loading frequency of 2 Hz. The specimens were heated by resistance coils. Uniform heating of the specimen gage length was obtained by carefully monitoring and controlling the specimen temperature using adjustable thermocouples attached to the gage length. The temperature profile remained within about ±1 °C of the nominal temperature throughout each test. The SEM was operated at an accelerating voltage of 15 kV. The fatigue tests were paused at different cycles of loading to capture the images of the samples with fatigue cracks in the secondary-electron mode of SEM. After the in situ fatigue tests, the fracture surfaces were examined and analyzed using a Scanning Electron Microscopy of Hitachi S-4500 for all specimens.

#### 3. Results

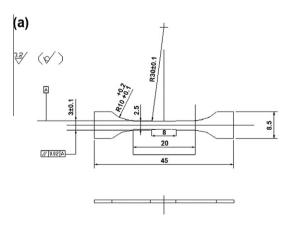
#### 3.1. In situ observations of crack growth

Fig. 2a shows the propagation of a small crack initiated from the notch root due to local stress concentration after 2569 cycles at 25 °C. The loading axis is along the vertical direction in Fig. 2a–f. The crack propagated in zigzag manner in its early period as in Fig. 2a. Notch depth is excluded in the crack length in the caption of Fig. 2, also adopted in Figs. 3 and 4. A remarkable

decrease in the fatigue crack growth rate (FCGR) was noticed when the crack approached the carbide in Fig. 2b. The crack subsequently passed around the carbide, partly because the carbide was located along the crack propagation direction (Fig. 2c). Afterwards, the crack penetrated the carbides perpendicular to the crack path (Fig. 2d). Here, it is distinct that fatigue small crack propagation is greatly associated with the direction of carbides and local inhomogeneities relative to the crack. With the extension of the crack, duplex slip traces on both sides of the crack became evident (Fig. 2e and f).

Two DZ4 specimens were fatigue tested under in situ SEM at 350 °C. Fig. 3a-f shows the typical process of crack evolution in one specimen, under the vertical cyclic loading. With the advance of a small crack from the notch root (Fig. 3a), a secondary crack emerged at the interdendritic carbide ahead of the primary crack. partly due to the incoherent property of the carbide. This secondary crack grew in shearing mode along the preferential slip plane. indicated by Fig. 3b. Coalescence of the secondary crack and the primary crack coalesce occurred obviously in Fig. 3c. This fatigue cracking mode was distinct from the continuous crack propagation in the other specimen at 350 °C. Many secondary small cracks were observed near the primary crack in Fig. 3d. Of interest is that the original crack tip in Fig. 3c was replaced by a new one accompanied with slip traces, as the old crack was impeded by certain barriers. It is noticed in Fig. 3e that the crack leaned to the small crack preinitiated from fractured carbide ahead of crack tip (Fig. 3d), indicating the effect of carbide on crack coalescence. Further, remarkable shearing deformation was observed in between the primary crack tip and the carbide-initiated small crack (Fig. 3e). After 16,627 cycles in total, crack coalescence occurred and a large crack tip open displacement (CTOD) was noticed, which led to sharp increase in crack length and crack growth rate correspondingly (Fig. 3f). The specimen was fractured 2 cycles later. It is evident at 350 °C that the cracking mode of coalescence can greatly aid the crack propagation with the help of cracked carbides.

For the crack propagation at higher temperatures, e.g. 700 °C, degradation of the photograph quality under SEM is evident. After 2480 cycles, a small crack perpendicular to the vertical loading direction was observed at the notch root (see Fig. 4a). The subsequent crack propagation was not completely straight but appeared to advance by avoiding the local microstructural inhomogeneities ahead of the crack tip (Fig. 4b). The crack was impeded by carbide in Fig. 4c, similar to that in Fig. 2d. In Fig. 4d, the crack overcame the carbide and bifurcating crack was noticed, with the new primary crack tip indicated by 'A'. Another small crack away from the crack tip was marked by 'B'. By comparing Fig. 4e with Fig. 4d, it is indicated that the bifurcated



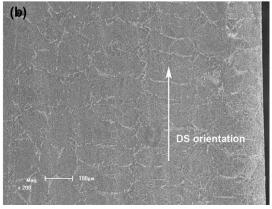


Fig. 1. (a) LCF testing specimen geometry (in mm), (b) typical microstructure of DZ4 superalloy.

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