Effects of NiAl-\(\beta\) precipitates on fatigue crack propagation of INCONEL alloy 783 under time-dependent condition with various load ratios

Longzhou Ma \(^a,^*\), Keh-Minn Chang \(^b\), Sarwan K. Mannan \(^c\), Shailesh J. Patel \(^c\)

\(^a\) Harry Reid Center for Environmental Studies, University of Nevada, Las Vegas, NV 89154, USA
\(^b\) Department of Mechanical and Aerospace Engineering, West Virginia University, Morgantown, WV 26505, USA
\(^c\) Special Metals Corporation, Huntington, WV 25705, USA

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Abstract

Hold time fatigue crack growth with various load ratios and sustained loading crack growth tests were conducted on INCONEL\(\textsuperscript{\textregistered}\) alloy 783 for different microstructures at 650 °C in air. The results showed that the presence of intergranular NiAl-\(\beta\) precipitates significantly improves the resistance to fatigue crack propagation under time-dependent conditions. Crack growth retardation was observed as the cyclic loading approached the sustained loading status.

Keywords: NiAl-\(\beta\) precipitate; Fatigue; Fracture; Oxides; Scanning electron microscopy

1. Introduction

Recently developed INCONEL alloy 783 (nominal composition of Ni–34Co–25Fe–5.4Al–3Nb–3Cr) is a low coefficient of thermal expansion (CTE) superalloy with a high strength and good resistance to stress accelerated grain boundary oxidation (SAGBO). The SAGBO resistance of alloy 783 is superior to the conventional low CTE iron–nickel-based superalloys such as INCOLOY\(\textsuperscript{\textregistered}\) alloys 907, 908 and 909. Alloy 783 is unique since it contains very high Al (5.4 wt.%) compared to conventional low CTE alloys. Al results in precipitation of NiAl-type \(\beta\) phase in an austenite matrix in addition to \(\text{Ni}_3\text{Al}\)-type \(\gamma'\) phase. \(\beta\) phase is reported to improve the SAGBO resistance of alloy 783 [1]. It is well documented that, in most conventional superalloys, time-dependent fatigue crack propagation (FCP) under hold time condition is attributed to SAGBO [2,3]. The FCP rate, \(\frac{da}{dn}\), at the full time-dependent stage can be correlated to the sustained loading crack growth rate, \(\frac{da}{dr}\) [2,3]. The full time-dependent FCP is a rate-controlled thermal activation process mainly governed by the maximum stress intensity factor \((K_{\text{max}})\). The cyclic feature such as the minimum-to-maximum load ratio, \(R\), plays a negligible role in affecting \(\frac{da}{dn}\) [2,3]. The purpose of this study is to compare the effects of NiAl-\(\beta\) precipitates on...
FCP of alloy 783 under hold time condition with various values of $R$.

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2. Procedure

2.1. Material and heat treatment

A hot rolled flat of cross-section 120.00 mm x 9.50 mm was supplied by Special Metals Corporation. Analyzed chemical composition with wt.% in parenthesis was as follows: Ni (28.21), Fe (24.88), Co (34.39), Al (5.32), Nb (3.11), Ti (0.32), Cr (3.24), Ti (0.32), C (0.008), and Si (0.06). A set of specimens was subjected to the standard heat treatment. This consisted of the following steps: (i) anneal at 1120°C for 1 h and air cool; (ii) age at 843°C for 4 h and air cool; (iii) age at 718°C for 8 h and furnace cool at 55°C/h to 621°C for 8 h and air cool. Step (ii) will be referred as $\beta$ aging in the text. To produce a microstructure essentially devoid of intergranular $\beta$ phase, the second set of specimens were heat-treated using step (i) and (iii) only. Following heat treatments, single edge notched (SEN) specimens were machined with width of 19.05 mm, length of 69.85 mm, thickness of 3.18 mm, and notch depth of 3.81 mm. All SEN specimens were along longitudinal orientation.

2.2. Crack growth testing

The specimens were fatigue precracked up to 2.5 mm at room temperature with a low constant cyclic-stress intensity factor $\Delta K$ of 22.5 MPa$\cdot$\sqrt{m}. The precracked specimens were then heated to 650°C in air by a quartz light heater mounted on a MTS servohydraulic system. After reaching the test temperature, a 3-s cycle with 100 s hold at the maximum load and a sinusoidal 3-s cycle were alternatively applied to the specimens. Earlier work had indicated that FCP of alloy 783 showed the time-dependent FCP mode when a $3 + 100$ s hold cycle was imposed [4]. The application of 3-s cycle permitted the fatigue crack of alloy 783 to grow in a cycle-dependent FCP mode [4]. This was done to remove the previous hold time FCP testing history. To measure FCP rate, $da/dn$, the crack growth was controlled to reach a steady state in all the tests. In all 3-s tests, the $R$ was kept at 0.05. However, in all $3 + 100$ s hold cycle tests, a series of $R$ were used ranging from 0.1 to 0.9. In addition to the standard FCP tests, sustained loading crack growth tests were carried out to obtain sustained loading crack growth rate, $da/dt$, which was used to identify the full time-dependent FCP behavior. The sustained loading is a special cycle with an $R$ ratio of 1.0, i.e., when $R$ is equal to 1.0, the cyclic loading attains the sustained loading status. Constant maximum stress intensity factor, $K_{\text{max}}$, was 38.5 MPa$\cdot$\sqrt{m} in all the testing. This stress intensity factor is greater than the threshold needed to produce sustained crack growth in alloy 783 [5]. The crack growth process was monitored by a computer controlled d.c. potential drop technique. This technique has a measurement resolution of 5–10 μm and an error of <10%. This system is capable of recording the load and potential variation within each individual cycle.

3. Results and discussions

3.1. Microstructure and tensile properties

Alloy 783 is a three phase alloy containing $\gamma'$ and $\beta$ phases in $\gamma$ austenite matrix. Both heat treatments produced an isotropic microstructure with grain size of ASTM 5–7. Fig. 1(a) shows plate-like intergranular $\beta$ phase particles in a scanning electron microscopy (SEM) photograph. This photograph is taken on as-polished sample. These $\beta$ particles are formed primarily on $\beta$ aging. Fig. 1(a) also shows inter and intragranular globular $\beta$ particles. Since the solvus temperature of $\beta$ phase is approximately 1175°C and the annealing temperature of alloy 783 is 1120°C, these globular $\beta$ particles are primarily formed during processing but are not completely dissolved during the annealing. Grain boundaries of the specimen without $\beta$ aging are not revealed due to the lack of intergranular plate-like $\beta$ particles, Fig. 1(b). Isolated globular $\beta$ particles are revealed. Room temperature tensile data of standard heat-treated specimen
were as follow: 0.2% yield strength, 852 MPa; ultimate strength, 1225 MPa; and elongation, 25.9%. Room temperature tensile data of the specimen heat-treated without β aging were as follows: 0.2% yield strength, 884 MPa; ultimate strength, 1233 MPa; and elongation, 18.5%. The hardness values with and without β aging were 36 Rc and 41 Rc respectively. Elimination of β aging would have resulted in an enrichment of Al in an austenite matrix resulting in a higher volume fraction of γ on the subsequent aging. This could explain higher hardness and yield strength of the specimen heat-treated without β aging.

3.2. Crack growth

Constant ΔK controlled FCP curves under 3 + 100 s hold condition with various load ratios at 650 °C are shown in Fig. 2. The increment of crack length has a linear relationship with the number of cycles for the specimens on which the β aging was omitted, Fig. 2(a). This is due to constant ΔK mode for testing. The slope of FCP curve represents the steady fatigue crack growth rate, da/dn, which was observed for each R. However, for the specimen with β aging, the steady FCP was observed only for R of 0.05 and 0.2 as shown in Fig. 2(b). When R was increased to 0.5, FCP showed some fluctuations and crack growth retardation was observed from 150 to 220 cycles. At R of 0.7, a small crack growth occurred, and the portion of crack growth retardation was increased from 50 to 200 cycles. When R was increased to 0.9, the crack growth was completely retarded from 0 to 250 cycles. As R approaches 1.0, the fatigue becomes sustained loading without any cyclic feature. Fig. 3 shows sustained loading crack growth plots of samples with and without β aging. The specimen without β aging shows a linear steady crack growth behavior with an incubation

Fig. 2. Fatigue crack growth of alloy 783 at 3 + 100 s hold time fatigue, $K_{\text{max}} = 38.5$ MPa$\sqrt{\text{m}}$ and 650 °C (a) without β aging; and (b) with β aging.
period of about 550 s for constant $K$, Fig. 3(a). The slope of crack growth portion represents the sustained loading crack growth rate, $da/dt$. Interestingly, the specimen with $\beta$ aging does not show any significant sustained loading crack growth for about 6000 min except a slight crack extension at about 3000 min. The test was interrupted after 110 h due to crack growth retardation. To identify the full time-dependent FCP behavior of alloy 783 with the different microstructures, the regression analysis results of Figs. 2 and 3 for $da/dn$, and $da/dt$ were coupled in Fig. 4. Mathematically at the full time-dependent stage, the sustained loading crack growth rate, $da/dt$, can be correlated to the FCP rate ($da/dn$), i.e., $da/dn = t * (da/dt)$, where $t$ is the period of cycle. Fig. 4 shows the sustained loading crack growth rate, $da/dt$, as a function of load ratio, $R$. Crack growth rate of $1 \times 10^{-10}$ mm/s implies crack retardation. Sustained loading crack growth rate of the specimen without $\beta$ aging is constant within the experimental scatter, Fig. 4. This shows that the FCP rate under $3 + 100$ s condition is not affected by cyclic portion of the waveform. The FCP of alloy 783 without $\beta$ aging for $3 + 100$ s falls in the domain of fully time-dependent FCP.

The heat treatment with $\beta$ aging increased the resistance to FCP by two orders of magnitude compared to the heat treatment without $\beta$ aging under the time-dependent condition, Fig. 4. Further, only partial time-dependent FCP occurs for $R$ from 0.05 to 0.5. When $R$ is 0.7, the crack growth rate starts to decrease. On increasing $R$ to 0.9 and 1.0, the crack growth is fully retarded. This comparison demonstrates that NiAl-type $\beta$ phase significantly improves the resistance to crack growth and consequently alloy 783 displays an abnormal time-dependent FCP behavior. This will be discussed later.

### 3.3. Stress relaxation examination

One of crucial findings in this study is that $\beta$ aging introduces an abnormal time-dependent FCP and crack growth retardation behavior. It has been reported that the crack blunting, which is usually caused by severe creep deformation ahead of crack tip or heavy corrosion products on the fracture surface, could contribute to the decreased crack growth rate or crack growth retardation in conventional alloys [6,7]. Since the heat treatment without $\beta$ aging produced the higher yield strength as illustrated above, the crack growth retardation could be related to creep deformation. To deter-
mine if creep deformation contributes to the crack growth retardation, the tensile stress relaxation behavior of the specimens with and without β aging were examined at 650 °C following the ASTM E328-86-1991 test procedure. Overlapping of two stress relaxation curves implies that heat treatments with and without β aging do not affect the creep resistance of alloy 783 at 650 °C, Fig. 5. Therefore, it can be inferred that the different crack growth behaviors of the materials heat-treated with and without β aging cannot be ascribed to creep deformation.

3.4 Fracture surface characterization

Fig. 6 shows the SEM photographs of the fracture surfaces under different loading conditions. For the specimens without β aging, the fractographs in all the cases were typical intergranular suggesting that there was very little grain boundary cohesion during crack growth process, Fig. 6(a), (c) and (e). Lots of the protruding globular β particles and the same size holes on the fracture surface indicate that the globular β particles were separated from the matrix during crack growth. For the β aged specimens at R of 0.05, the fracture is predominantly intergranular with a relatively high fraction of rough surface showing numerous oxidized globular β particles, Fig. 6(b). Fracture surface has a few clean grain boundaries and a few rough grain boundaries containing oxidized intergranular β film. On increasing R was to 0.9, the crack growth was retarded with a slight crack extension. The fracture surface still shows intergranular features but the grain boundaries are completely covered with oxidized β film, Fig. 6(d). When R approaches 1.0, i.e. sustained loading status, the crack growth was retarded for 6000 min. Fracture surface is much rougher than R of 0.9. Also, the oxide film appears to be thicker, and the presence of oxide products appears to be more abundant, Fig. 6(f). Energy dispersive X-ray (EDX) analysis showed that these oxide products were Al-rich.

Absence of the intergranular NiAl-β precipitates allows the grain boundaries to act as a dislocation pipe without significant reaction-induced absorption to block the oxygen diffusion. This could explain clean grain boundaries of the specimen for which β aging was omitted. In contrast, the presence of β precipitates along grain boundaries could prevent the oxygen diffusion and dilute the intergranular oxygen concentration due to its particular oxidation resistant characteristics [8,9]. Under time-dependent condition, the rough fracture surface suggests that the crack had to repetitively rupture the intergranular oxidation-resistant β films during crack growth. The driving force to rupture β film arises from the cyclic loading, ΔK. Increase in R value lowers ΔK. For small R values (0.05–0.5), ΔK is sufficient to produce the crack growth with some fluctuation. When R is increased to 0.7 or 0.9, the crack growth slows down and starts to retard due to the reduction of ΔK. Under the sustained loading condition (R = 1.0), the cyclic loading disappears, and only the static load exists ahead of the crack tip. Under these conditions, the intergranular β films could interact with oxygen to produce numerous Al-containing oxides such as Al₂O₃. These heavy oxides would prevent further oxidation after long time exposure due to the passivation effect of NiAl-β [8]. Eventually, the growth of Al-containing oxide scales could blunt the crack tip and retard the crack growth.

4. Conclusions

NiAl-β precipitates in alloy 783 play a very significant role in the time-dependent FCP behavior. The oxidation resistant β phase precipitation along the grain boundaries dramatically
increases the SAGBO resistance, and thereby causes an abnormal time-dependent FCP behavior and crack growth retardation.

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