Intergranular fracture events at high temperature in a high strength Ni-based superalloy

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Introduction and Motivation

Polycrystalline high strength Ni-based superalloys produced by powder metallurgy (PM) are predominately used for high pressure disc rotors in jet engines. There is significant interest in quantifying the mechanical performance of these superalloys at temperatures over 700°C. In part, this is because the outer rim of the disc is likely to experience such temperatures during portions of future mission cycles [1]. The disc must operate in harsh environments where damage due to oxidation and time dependent deformation can lead to intergranular crack growth.

The effect of oxygen on a crack tip is still not understood to the fullest extend. Currently two mechanisms are proposed to explain the damage to the grain boundary (GB) in front of the crack tip: stress accelerated grain boundary oxidation (SAGBO) [2, 3] and dynamic embrittlement (DE) [4]. Studies on the fundamental damage mechanism are traditionally performed using conventional large-scale test pieces. The distribution of oxygen at the crack tips in these specimens has been recently studied using high resolution characterisation techniques mainly transmission electron microscopy combined with energy dispersive X-ray spectroscopy (TEM-EDX) [5], nanoscale secondary ion mass spectrometry (NanoSIMS) [6], and atom probe tomography (APT) [7]. The effect of oxygen on the local mechanical properties ahead of the crack tip has not been considered. Novel micro-mechanical testing techniques allow site-specific investigations into the effect of oxygen and therefore allow the study of the failure mechanisms at the right length scale.

In this work, a multi-scale approach is used to study oxygen-assisted cracking. Firstly, slow strain-rate tests building upon the previous work of [8] were performed at temperatures above 700°C. Rather then using conventional tensile specimens miniaturised test pieces were used. These miniaturised tests pieces were found to be of sufficient size to represent large scale material behaviour. Crack tips have been characterised using high resolution scanning electron microscopy (SEM) with analytical capabilities. Site specific samples for micro-bend tests at crack tips have been manufactured using focused ion beam (FIB) milling. The full paper will discuss the effect of oxygen at high temperatures on mechanical properties ahead of the crack tip in a Ni-based superalloy.

Experimental Procedures

Slow strain-rate tests are a low cost screening tool to compare the susceptibility of Ni-based superalloys to oxygen-assisted cracking [8]. The strength of being a low cost testing technique was extended further by using miniaturised specimen with a cross-section of 1x1 mm and a gauge length of 16 mm. These miniaturised specimen were tested at 800°C with a strain-rate of 0.0001 $/s$ using a 5 kN electrical thermal mechanical testing (ETMT) system in oxygen and argon atmosphere. The specimens are heated using direct resistance heating. In contrast to the experiments Gabb et al. [8] performed, instead of fast strain-rate tests slow strain-rate tests were used in argon to isolate the effect of oxygen on the failure mechanism.

A part of the aim was to measure the ductility. Subsequent fractography was carried out using three dimensional (3D) topographical analysis and scanning electron microscopy (SEM). In addition, cross-sections perpendicular to the fracture surfaces were metallurgically prepared in order to identify the crack path. The oxygen profile ahead of crack tips was studied using high resolution energy-dispersive X-ray spectroscopy (EDX). Electron backscatter diffraction (EBSD) was performed to further characterise the failed GBs. A single beam FIB instrument was used to manufacture 14 μm long and 3 μm wide micro-cantilever with a triangular cross-section. Micro-cantilever were manufactured at the fracture surface with the previously analysed GB in front of the crack tip close to the fixed-end. In addition micro-cantilever were prepared in the shoulder of the ETMT specimen as a reference. Micro-bend tests were performed at room temperature (RT) with a constant displacement rate of 5 nm/s in a nanoindentation system. After testing the deformed cantilever were imaged using a dual beam FIB-SEM.

A typical cast and wrought γ/γ' Ni-based alloy for high pressure and turbine discs has been chosen: Alloy 720Li (Table 1). Samples were machined out of a isothermal forging ring with the dimensions of 18x14 mm. This ring was then solution annealed and age heat treated.

Table 1: Composition of the alloy studied in wt-% (Ni-base)

Name	Cr	Co	W	Al	Ti	Mo	C	B	Zr
Alloy 720Li	16.3	14.6	1.3	2.5	5	3	0.025	0.02	0.03

Results and Discussion

Examples of slow strain-rate stress-strain curves for alloy 720Li obtained from tests in an oxygen environment are shown in Figure 1a. Tests performed in laboratory air had all a relatively small inelastic fracture strain of ∼5 %. Fractography performed at lower magnifications showed a fracture surface normal to the applied load with clear signs of oxidation. Higher magnifications proved the purely intergranular nature of the fracture. Tests performed in an argon atmosphere showed an inelastic fracture strain of >10 % in all cases (Figure 1a). Their fracture surface did not show signs of oxidation and was transgranular. Before failure ductile deformation was observed. It can be concluded that tests performed in an oxygen atmosphere at 800°C all failed in a less ductile, intergranular manner while tests performed in an argon atmosphere showed more ductility before

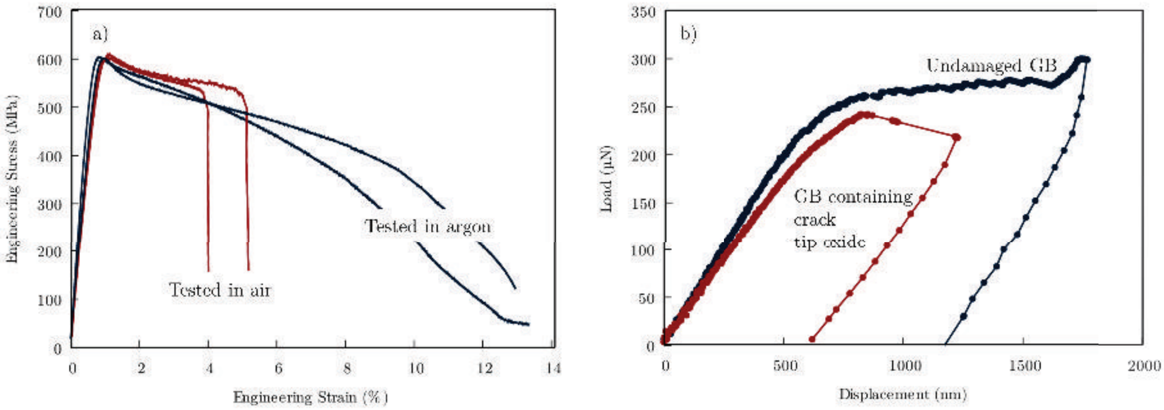

Figure 1: a): Slow strain-rate tensile stress-strain curves for alloy 720Li tested in oxygen and argon atmosphere. b): Load-displacement curves for a micro-cantilever containing a crack tip oxide and a reference micro-cantilever from bulk alloy 720Li.

failure deformed ductile before failure. No effect of the environment on the yield stress was observed. The presence of oxygen clearly reduced the ductility and led to a thermally activated and time-dependent embrittlement which is in accordance with literature [9, 10]. These results therefore demonstrate that miniaturised slow strain-rate tests are capable to study oxygen-assisted cracking.

The crack path within miniature specimens which suffered oxygen induced failure was studied using SEM and EDX. Increased levels of oxygen were observed alongside the purely intergranular crack (Figure 2a). The investigation showed that a substantial amount of the cracking took place between γ-grains and primary γ'-precipitates. Signs of plastic deformation was observed in the γ-grains (Figure 2b). High resolution EDX revealed a Cr- and Mo-rich oxide ahead of the crack between a primary and secondary γ' (Figure 2c). The same area showed a Ni-depletion. This result is evidence that oxygen affects the GB locally. This finding is in accordance to APT results of an oxidized intergranular crack tip in Allvac 718Plus® [7]. The presents of a crack tip oxide could be interpreted as an argument supporting the SAGBO theory. However, it is not yet clearly evident whether the oxide formed before or after the crack propagated.

In order to investigate the impact of the observed oxide on the local mechanical properties a site specific micro-bend test was performed (Figure 2d). The oxide was close to the fixed-end of the cantilever. The load displacement curve shows a fracture event just after the yield point. A second micro-cantilever which was milled into the shoulder of the same specimen did not fail but rather deform ductile (Figure 1b). Examination of the brittle cantilever showed no obvious crack in the oxide and it can be assumed that the fracture event occurred internally. However, this preliminary result confirmed that intergranular fracture is directly connected to the presents of oxygen at the crack tip. Reconstructing the crack path inside the cantilever is subject to ongoing research and will be achieved by using SEM and FIB methods. The results of this work will be presented in the paper.

These results demonstrates that micro-bend tests are suited to study the mechanical properties of isolated features at a crack tip and motivate the further study comprising of more detailed examinations of multiple crack tips and a number of micro-bend tests.

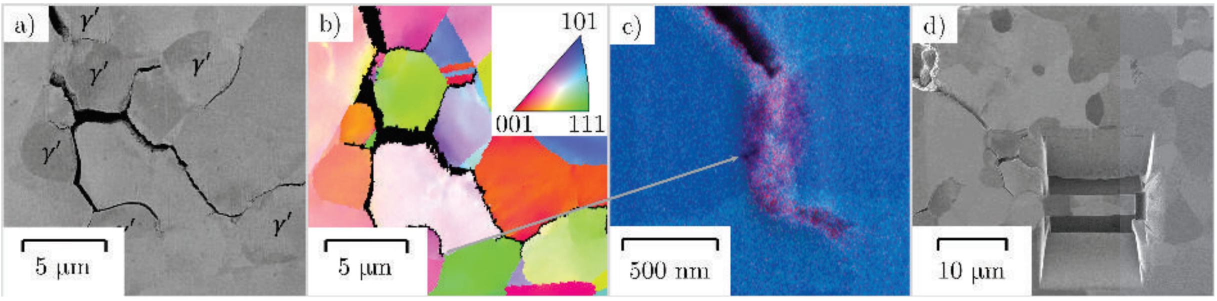

Figure 2: a): SEM image showing a crack connected with the fracture surface. b): EBSD image of the crack path. c): High resolution EDX of a isolated crack tip. Ni, Cr, Co, W and Mo are highlighted in light blue. Ti and Al are highlighted in dark blue and O is highlighted in red. d): FIB-SE image showing an untested micro-cantilever containing a crack tip oxide.

Summary

The full paper will summarise the following points:

- Slow strain-rate tests have been miniaturised building upon the work of Gabb et al. [8] in order to study the effect of oxygen on the mode of failure.
- Crack tips in cross-sectioned intergranular fracture surfaces have been studied using combination of SEM, EDX and EBSD.
- It has been demonstrated that small-scale bend tests using micro-cantilevers can be applied to test isolated features in front of a crack-tip giving insight into the failure mechanism of oxygen-assisted cracking.

Acknowledgements

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