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Multi-Scale Investigation of Oxygen-Induced Damage in Nickel-Based Superalloy Alloy 718

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Abstract. Understanding damage mechanisms across scales is crucial to ensure the structural integrity of nickel-based superalloy components under demanding conditions. This study highlights key aspects of a multi-scale experimental approach for analyzing oxygen-induced cracking in Alloy 718. Microcantilever bending tests on specific grain boundaries were combined with corrosion tests and detailed analyses using high-resolution scanning electron microscopy, electron backscatter diffraction, and energy-dispersive X-ray spectroscopy. The results suggest that susceptibility to oxidative attack is significantly impacted by the type of grain boundary, emphasising the importance of local crystallography in oxygen diffusion and elemental redistribution. By bridging local microstructural features with global mechanical response, the presented multi-scale approach allows the parameterization of physically based material models and identifies grain boundary engineering as a promising strategy for improving damage tolerance.

Introduction

The nickel-based superalloy Alloy 718 is widely used in high-temperature applications due to its exceptional mechanical strength, oxidation resistance, and corrosion resistance. A notable example is its application in forged aircraft turbine blades and discs, where components must withstand both high thermal and mechanical loads. Its outstanding performance is primarily attributed to precipitation hardening: γ'' (Ni_3Nb) and, to a lesser extent, γ' ($Ni_3(Al,Ti)$) precipitates, playing a central role in strengthening the microstructure [1-4]. Despite this, Alloy 718 is susceptible to environmental degradation in oxidizing atmospheres. The combination of mechanical stress, high temperatures, and oxygen weakens the grain boundaries, which can ultimately result in brittle intergranular fracture (Figure 1, [5]).

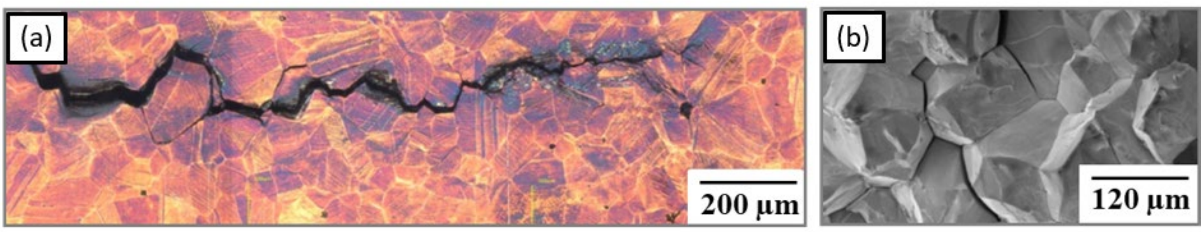

Fig. 1. Brittle intergranular cracking in Alloy 718. a) crack propagation path on the surface of a polished fatigue specimen. b) intergranular crack surface after fatigue testing [5].

Two mechanisms are commonly discussed: (i) dynamic embrittlement (DE), where oxygen diffuses into microcracks and embrittles grain boundaries in a manner similar to hydrogen embrittlement in steels [6-8], and (ii) stress-aided grain boundary oxidation (SAGBO), where oxides form ahead of the crack tip and promote crack propagation through repeated fracture of brittle oxides [9, 10]. While the dominant mechanism remains under debate, oxygen ingress is widely recognized as weakening grain boundaries in the vicinity of surface and microcracks.

In the present paper, a methodological workflow for experimental investigation and physical-based simulation of damage mechanisms in Ni-based superalloys under complex service-like conditions is presented. Due to the complex interplay of different degradation factors and the limited reproducibility due to technical constraints, a single experimental method is not sufficient to fully characterize material behavior across all relevant length scales. Therefore, this work emphasizes the necessity of combining multiple complementary experimental techniques, ranging from microscale investigations targeting individual microstructural features, such as specific grain boundaries, to oxidation tests at elevated temperatures. The overall study (see Figure 2) aims not only to identify and understand the underlying damage mechanisms and key influencing factors, but also to systematically generate comprehensive material data for simulation-based predictions of material failure using a multi-scale approach. Experimental data serves as the foundation for a coupled finite element (FEM) model that accounts for both mechanical and diffusion-driven processes, and ab initio density functional theory (DFT) simulations focusing on grain boundaries [11]. While key aspects of the experimental program are presented in this publication, future studies will explore both the results and interplay of several other experiments (e.g., dwell-time fatigue, four-point bending tests) and advanced evaluation methods (e.g., atom probe tomography, 3D-EBSD), as well as the integration of the experimental data into FEM and atomistic simulations in more detail. Ultimately, the developed experimental strategies aim to bridge the gap between localized damage initiation at the microstructural level and the global material response, thereby enabling a deeper mechanistic understanding and more reliable service life prediction of components made from Ni-based superalloys.

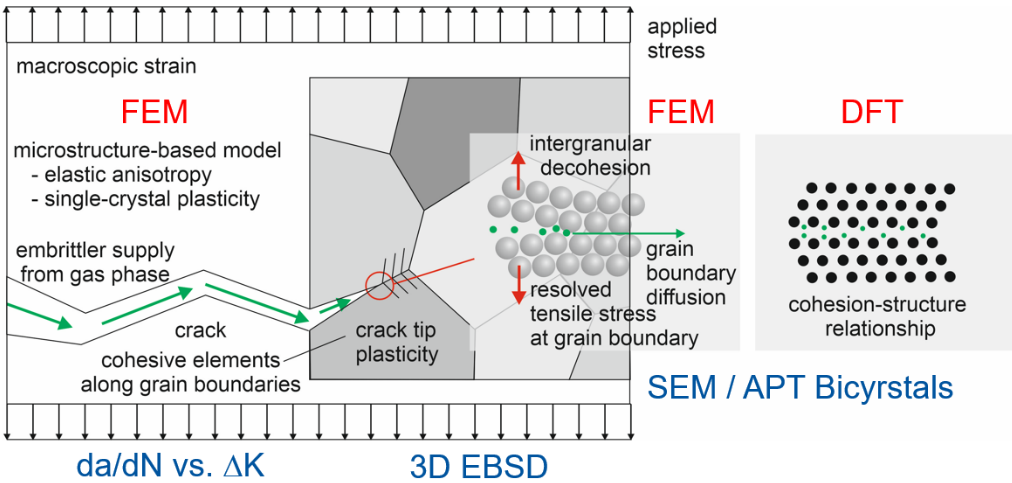

Fig. 2. Schematic overview of overall approach: stress assisted grain boundary diffusion of elemental oxygen lowering the interface cohesive forces and eventually leading to crack growth: multi-scale methodology, integrating experimental aspects, finite element modelling, and ab initio DFT simulations.

Material

The nickel-based superalloy Alloy 718 (material number 2.4668) used in this study was produced by VDM-Metals via multiple vacuum melting steps. The measured chemical composition is listed in Table 1. Forged round ingots were solution annealed to dissolve alloying elements and subsequently enable controlled precipitation of γ' and γ''. Solution annealing was conducted at 1050 °C for 1 h, followed by water quenching, according to industrially established process parameters to obtain a coarse-grained microstructure. Subsequently, the typical two-stage aging treatment process for the high-strength material variant was applied, comprising 8 h at 718 °C, subsequent furnace cooling with 50 K/h to 620 °C, holding for 8 h, and air cooling. The resulting microstructure is texture-free, with an average grain size of 20.5 µm (equivalent diameter), as shown in the inverse pole figure (IPF) and grain size distribution maps in Figure 3, as obtained by EBSD.

Table 1. Measured chemical composition of 2.4668 Alloy 718 (wt.-%).

Ni	Cr	Fe	Nb	Mo	Ti	Al	Others
54.1	18.0	Bal.	5.4	3.0	1.0	0.5	< 0.5

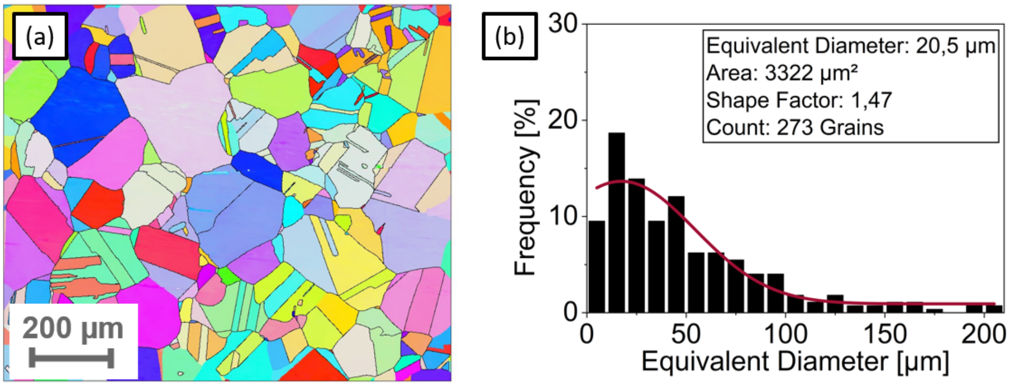

Fig. 3. a) IPF map and b) grain size distribution map of Alloy 718 after solution annealing and high-strength heat treatment.

Experimental Methods

Various experimental methods across multiple length scales were combined to investigate damage behavior, from microstructural processes to macroscopic diffusion phenomena. It is generally known that oxygen-induced damage is controlled by local phenomena, particularly the local microstructure, short-range stress fields and the local diffusion properties of oxygen. The properties of individual grain boundaries, especially regarding crystallographic misorientation, boundary strength and oxygen diffusivity, are therefore of great importance. To characterize these influences, room temperature micro-bending tests were carried out at various characteristic locations in the microstructure, both within the grain and on selected grain boundaries. Therefore, compact specimens measuring 15×7×3 mm³ were wet-ground using SiC abrasive papers up to P4000 grit, followed by sequential polishing with 3 µm and 1 µm diamond suspensions, and finishing with a colloidal silica suspension (0.04 µm). Based on grain orientation maps from electron backscatter diffraction (EBSD) analysis, pentagonal micro-bending cantilevers (undercut angle: 52°) with a length of 15 µm, a width of 6 µm and a thickness of 3 µm at the sides (correspondingly more in the middle due to the tetragonal geometry) were prepared on selected grain boundaries, for example CSL3 grain boundaries and high-angle grain boundaries, using plasma focused ion beam (PFIB) milling (Helios 5, Thermo Fisher Scientific). For high dimensional accuracy and low influence of the ion beam on the surface material layer, the last step was carried out with a low ion beam current of 0.3 nA. Particular focus was placed on the orientation of the grain boundary, located in the front part of the cantilever (2 µm from bulk material) in the area of maximum stress, and oriented parallel to the axis of applied force. Furthermore, a sharp notch is required to ensure a controlled failure of the grain boundary. This notch had a depth of 0.3 µm and was milled using the lowest possible current of 10 pA. Mechanical loading up to fracture was carried out using a pico-indenter (Bruker Hysitron Pi 89) installed in a scanning electron microscope (SEM). A conical diamond tip (Synton-MDP) with a tip radius of 2 µm and a tip angle of 60° was used as the indenter. Force and indentation depth were recorded throughout the test, followed by detailed post-mortem analysis using high-resolution SEM to map dislocation slip traces and the fracture surface.

Oxidation tests were conducted to determine the oxygen diffusion rate along different grain boundary types. For this purpose, samples with dimensions of 20×12×4 mm³ were ground and polished as described above. The Oxidation experiments were performed at 800 °C and 900 °C in air (21% oxygen, ambient pressure) for 100 h each. The samples were coated with conductive silver and subsequently galvanized with zinc. They were longitudinally cut in the middle, and the cross-sectional surfaces were metallographically prepared as described above. The depth of the oxidation attack was quantified in SEM using the concentric backscattered electron detector (CBS) (10 kV, 0.8 nA). Additionally, energy dispersive X-ray spectroscopy (EDS) was used to determine local elemental distribution (15 kV, 0.8 nA) and EBSD (20 kV, 13 nA) to analyse the grain orientation and grain boundary type, enabling correlation of local microstructure with oxidation attack.

Results and Discussion

The results of the micro-bending tests for a cantilever positioned at a CSL3 grain boundary are shown in Figure 4 as an example. EBSD analysis prior to loading (IPF map in Figure 4a) provides grain orientation information and characterizes the grain boundary type. Mechanical loading of the cantilever with a picoindenter is shown Figure 4b. the corresponding force–displacement curve (Figure 4c) exhibits a typical response, which is used for calibration of the crystal plasticity FE model. The cantilever initially undergoes elastic and then plastic deformation, followed by grain boundary fracture, indicated by a sudden drop in stress (red arrow). Post-mortem SEM images clearly show pronounced slip traces from localized dislocation motion, as well as a crack along the CSL3 grain boundary.

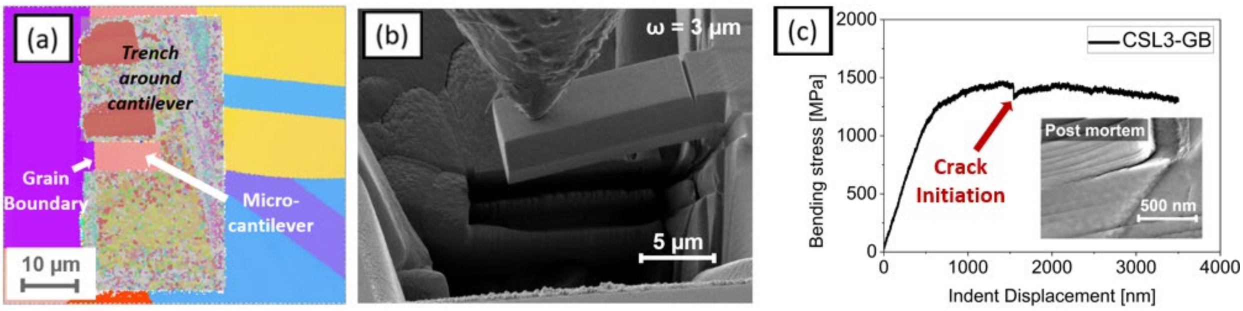

Fig. 4. Microcantilevers bending tests on specific GBs: a) Position of a selected microcantilever in the IPF map; b) In-situ snapshot of the microcantilever during bending at 3 µm displacement; c) Bending stress vs. displacement curve of a representative microcantilever placed on a CSL3 grain boundary, with a high-resolution SEM image showing a crack along the boundary after testing.

The corrosion tests reveal significant surface degradation at both 800 °C and 900 °C. Consistent with literature [12, 13], the EDS images in Figure 5 show a chromium oxide layer at the surface. Below this oxide layer, there is a noticeable enrichment of niobium, chromium, and molybdenum, accompanied by elevated oxygen levels. Further into the material, internal oxidation of aluminum (Al_2O_3) is observed.

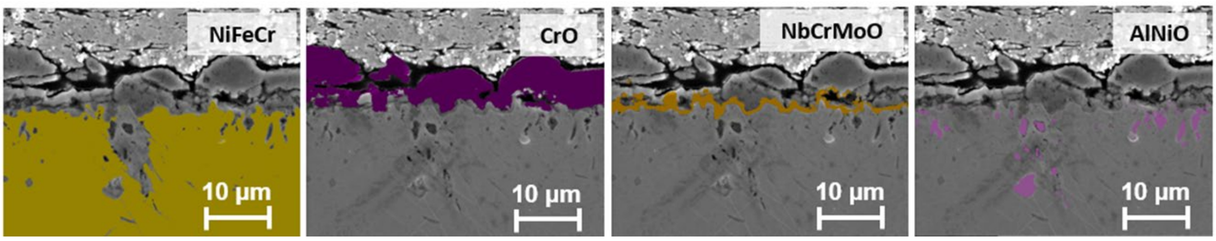

Fig. 5. Representative oxidation profile after oxidation experiment (900 °C for 100 h).

As expected from other studies [14, 15], oxidation attack is more severe at grain boundaries than in the bulk material. Figure 6 shows an example of the evaluation procedure, presenting an SEM image of different grain boundaries and enabling measurement of oxidation at various grain boundary types. Twin grain boundaries (CSL3) thus show no discernible oxidation attack, while the attack on the other grain boundary types varies. The analysis focuses in particular on the points at which the oxidation attack abruptly ceased. This is often assumed to be due to an interaction between the microstructural composition (e.g., location and morphology of the precipitates) and the specific grain boundary orientation. However, further analyses are required to fully characterize this process.

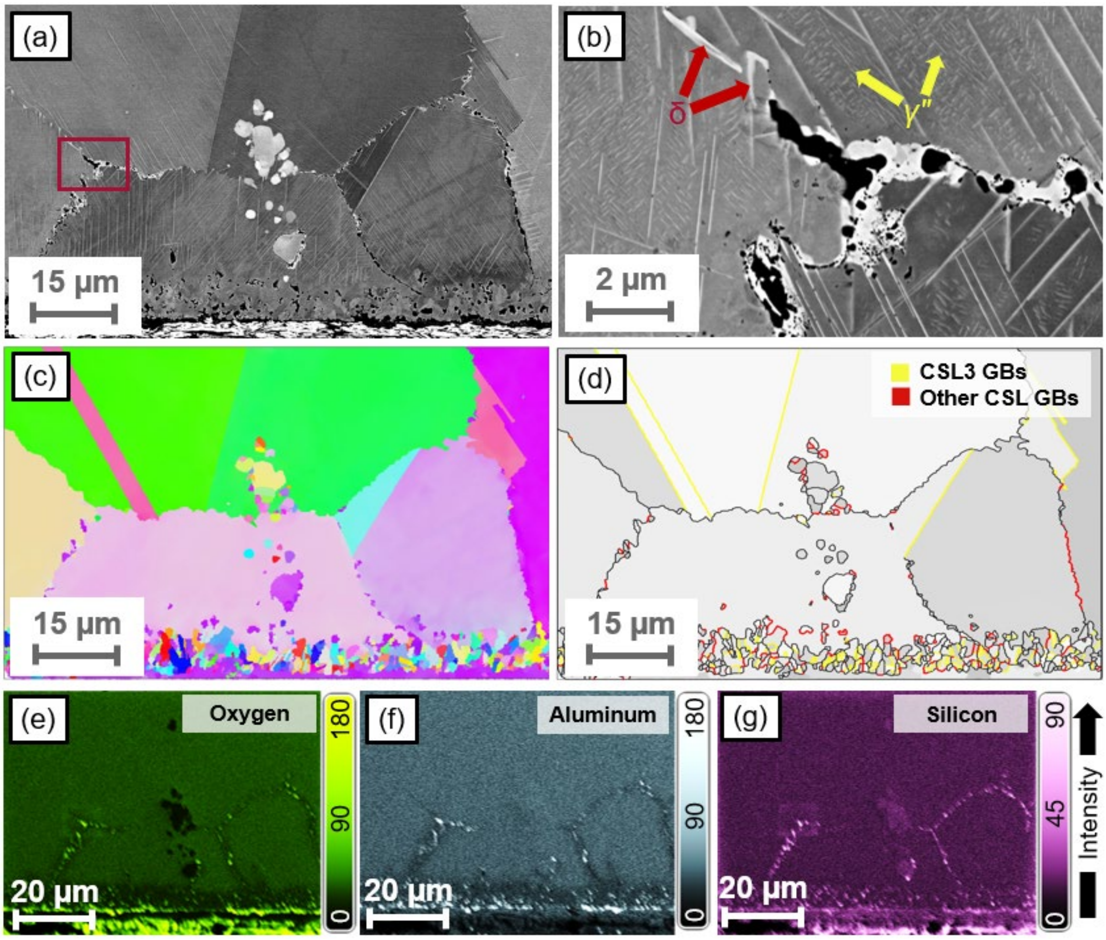

Fig. 6. Exemplary analysis of an oxidized area (900 °C, 100 h). a) Overview image of the region, recorded using the CBS detector; b) High-resolution view of the area marked in red, showing oxidation damage and the presence of δ- and γ″-precipitates; c) IPF map; d) Grain boundary type map; e–g) EDS maps of O, Al, and Si, respectively, for the same area.

Summary and Outlook

Key aspects of a comprehensive experimental approach for investigating damage mechanisms in the nickel-based superalloy Alloy 718 under service-relevant conditions are presented in the present paper. Oxidation tests (800 °C and 900 °C; 100 h) were combined with SEM, EBSD, and EDS analyses. Variations in oxidation depth indicate the importance of the local environment, specifically grain boundary character and precipitate morphology. Oxidation preferentially occurs at high-angle grain boundaries and is influenced by precipitate distribution, while CSL3 twin boundaries remain largely unaffected. Additionally, differences in grain boundary strength, which were quantitatively assessed through micro-bending experiments conducted in the SEM, are assumed to play a significant role. While these experiments provide valuable insights into the mechanical behavior of individual grain boundaries, the current setup, operating under vacuum conditions in the SEM, limits the ability to replicate service-like degradation mechanisms due to interfacial oxygen diffusion. Future work will therefore focus on testing micro-cantilevers prepared from oxygen-damaged samples at elevated temperatures (e.g., 650 °C) in a high-temperature-capable SEM environment. The goal is to gain a deep understanding of grain boundary-dependent failure mechanisms. Such findings could contribute to significantly improved high-temperature fatigue and oxidation resistance of nickel-based superalloys, thereby extending component service life.

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