Cracks in superalloys

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Cover image: An orientation imaging map illustrating the general crack propagation behaviour of a 2160 s dwell-time tested Inconel 718 bar run at 550 °C, with a 15 % superimposed overload at the beginning of the dwell-time.

During the course of research underlying this thesis, Jonas Saarimäki was enrolled in Agora Materiae, a multidisciplinary doctoral program at Linköping University, Sweden.

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Gas turbines are widely used in industry for power generation and as a power source at hard to reach locations where other possibilities for electrical power supplies are insufficient. New ways of producing greener energy is needed to reduce emission levels. This can be achieved by increasing the combustion temperature of gas turbines. High combustion temperatures can be detrimental and degrade critical components. This raises the demands on the high temperature performance of the superalloys used in gas turbine components. These components are frequently subjected to different cyclic loads combined with for example dwell-times and overloads at elevated temperatures, which can influence the crack growth. Dwell-times have been shown to accelerate crack growth and change cracking behaviour in both Inconel 718, Haynes 282 and Hastelloy X. On the other hand, overloads at the beginning of a dwell-time cycle have been shown to retard the dwell-time effect on crack growth in Inconel 718. More experiments and microstructural investigations are needed to better understand these effects.

The work presented in this thesis was conducted under the umbrella of the research program Turbo Power; “High temperature fatigue crack propagation in nickel-based superalloys”, where I have mainly looked at fatigue crack growth mechanisms in superalloys subjected to dwell-fatigue, which can have a devastating effect on crack propagation behaviour. Mechanical testing was performed under operation-like cycles in order to achieve representative microstructures and material data for the subsequent microstructural work. Microstructures were investigated using light optical microscopy and scanning electron microscopy (SEM) techniques such as electron channeling contrast imaging (ECCI) and electron backscatter diffraction (EBSD).

The outcome of this work has shown that there is a significant increase in crack growth rate when dwell-times are introduced at maximum load (0% overload) in the fatigue cycle. With the introduction of a dwell-time there is also a shift from transgranular to intergranular crack growth for both Inconel 718 and Haynes 282. The crack growth rate decreases with increasing overload levels in Inconel 718 when

an overload is applied prior to the dwell-time. At high temperature, intergranular crack growth was observed in Inconel 718 as a result of oxidation and the creation of nanometric voids. Another observed growth mechanism was crack advance along δ -phase boundaries with subsequent oxidation of the δ phase.

This thesis comprises two parts. Part I gives an introduction to the field of superalloys and the acting microstructural mechanisms related to fatigue and crack propagation. Part II consists of five appended papers, which report the work completed as part of the project.

Populärvetenskaplig sammanfattning

Gasturbiner används över hela världen för el- och kraftproduktion samt i allt från pumpar och skepp till svåråtkomliga områden i berg och djungel där kraftnätet kan vara instabilt och nästintill obefintligt. Idag är efterfrågan på grön el större än någonsin. Det betyder att vi behöver komma på nya sätt att producera renare el t.ex. från sol och vind. Ett annat sätt att producera renare el är att öka förbränningstemperaturen i dagens gasturbiner. Vid en ökning av förbränningstemperaturen ändras kraven på de material som används i de varma delarna av gasturbinerna. Temperaturökningen kan leda till att de mekaniska egenskaperna hos kritiska komponenter försämras. Dessa komponenter utsätts vanligtvis för olika cykliska laster i kombination med hålltider och överlaster som kan påverka sprickinitiering och spricktillväxt och som avsevärt kan förkorta turbinens livslängd.

Inconel 718, Haynes 282 och Hastelloy X är superlegeringar som används till gasturbinkomponenter. Resultaten visar att effekten av hålltider vid maxlasten (0 % överlast) och hög temperatur markant ökar spricktillväxthastigheten under utmattningscykeln. När hålltider introduceras blir spricktillväxten interkristallin i Inconel 718, Haynes 282 och Hastelloy X. När en överlast introducerats i början av hålltidscykeln avtog sprickpropageringshastigheten i samband med att överlasten ökades i Inconel 718. Sprickpropagering skedde längs korngränserna p.g.a. oxidation, bildning av nanometriska kaviteter (krypskada) samt mellan γ och δ -fas. Den ledande sprickpropageringsmekanismen i Haynes 282 var mest sannolikt oxidering av sprickspetsen till skillnad från det additivt tillverkade materialet Hastelloy X där sprickan propagerade i korngränserna till följd av ogynnsamma utskiljningar. För att öka förståelsen för hur de här effekterna påverkar mikrostrukturen och vise versa krävs det mer forskning inom området.

Den här avhandlingen består av två delar. Den första ger läsaren en introduktion till materialområdet superlegeringar och de mekanismer som vanligtvis relateras till utmattning. Den andra delen består av fem artiklar som sammanfattar mitt arbete.

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It is truly hard to write the acknowledgements for any thesis or article, as it is fraught with danger. Mention too few and some might feel left out and vexed because they were not included. Mentioning too many will make you look like one of the artists from the MTV music awards thanking every famous person for inspiration, non-existent assistance, and so forth. So here we go.

Firstly, this research has been carried out at the Division of Engineering Materials, Department of Management and Engineering, Linköping University, Sweden. The project “High temperature fatigue crack propagation in nickel-based superalloys” was financially funded by the Swedish Energy Agency, GKN Aerospace Engine Systems, Siemens Industrial Turbomachinery AB and the Royal Institute of Technology through the Swedish research program Turbo Power, and through faculty grant SFO-MAT-LiU#2009-00971 which are gratefully acknowledged. During the course of the research underlying this thesis, I was enrolled in *Agora Materiae*, a multidisciplinary doctoral program at Linköping University, Sweden. Through this program I have made new friends and learned all kinds of weird things concerning physics and thin films, for this I am truly grateful. So thank you *Per-Olof Holtz* for running *Agora Materiae*.

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A special thanks to *Christopher Tholander* who has made some of my mind-boggling computer and programming problems seem ridiculously simple and *Matias Lundberg*, co-creator of the abstract office, cheers.

To the people outside of work who makes my life the utopia I am always claiming it to be: Världens bästa mamma, my aunt *Mariaana* and uncle *Charlie*. Finally, to supermamman *Jennifer, Plutten (Jasmine)* and Lillplutten (*Joline*) for giving me a wonderful family.

@Plutten and @Lillplutten, I wrote this thesis to the both of you. When I began, I had not yet realised that girls grow (much) quicker than chapters. This thesis will most likely work as an extremely effective bedtime story for quite some time. One day you will be old enough to start thinking about reading this. You can then take it down from some upper shelf or box in the basement, dust it off, and tell me what you think of it. I will probably be too deaf to hear, and too old to understand a word you say, but I will still be your mount papa.

A handwritten signature in blue ink that reads "Jonas Saarimäki". The script is fluid and cursive, with a long, sweeping tail on the final letter.

Jonas Saarimäki
Linköping, Mars 2018

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CHAPTER 1

Introduction

Gas turbines are widely used in industry for power generation and power sources at hard to reach locations where other possibilities for electrical power supplies are limited [1]. In the logistics sector turbines are frequently used in the aviation industry. The effect of global warming has increased the demand on lowered emissions. New ways for producing greener energy is needed. This can be achieved by increasing the combustion temperature of gas turbines [2]. This, in turn, raises the demands on the high temperature performance of the superalloys used in gas turbine components [3]. Different types of turbines have been developed throughout history. The first steam engine was developed by Hero. The “first” gas turbine was developed in 1920, and about twenty years later the first aircraft turbine became available. The key behind successful development of better performing turbines has been improvements in the field of superalloys, coatings and cooling.

Superalloys are not only used in turbine discs and blades but in many applications such as burner nozzles, power plants, aqueous, space and petrochemical applications. Superalloys are a group of nickel-, iron-nickel- and cobalt-based materials which combine mechanical properties and corrosion resistance needed for components subjected to temperatures as high as 1000 °C [4]. At these high temperatures most other material groups such as steel, demonstrate extremely poor properties.

Superalloys are used in some of the worlds most aggressive working conditions inside a gas turbine because they show a positive combination of mechanical properties and corrosion resistance. These harsh working conditions are detrimental to the alloy and cause corrosion, oxidation, erosion, thermal-mechanical fatigue, low cycle fatigue, high cycle fatigue, creep and microstructural degradation. All of these can contribute to catastrophic failure. Components such as turbine discs are subjected to high temperatures and significant tangential forces generated when spinning at speeds up to 9000 rpm. Component life is usually correlated to the

number of stop and start cycles or takeoffs and landings. Land based gas turbines are generally run at a constant load level. This means that the fatigue life is paramount [5] when designing turbine components for frequent starts. It is well known that fatigue cracks commonly initiate at surfaces [6] and that fatigue crack growth rate is highly cycle type dependent. A dwell-time cycle is well suited for simulating a run cycle from start to stop. Dwell-time cycles have been shown to increase crack growth rates [7]. To achieve a better understanding of crack growth rates, microstructure and cycle dependence are of utmost interest and needs further investigation.

Siemens Industrial Turbomachinery AB and GKN Aerospace Engine Systems were the main collaboration partners in this project. The testing methods used have been directed towards the operating cycles of gas turbines such as dwell-times simulating a part of a transatlantic flight or part of a normal run cycle for a land based gas turbine. The materials researched in this thesis are Inconel 718 (bar, grain enlarged bar and cast), Haynes 282 (in the form of a forged ring), and Hastelloy X (additively manufactured (AM) by laser powder bed (SLM) and cast). Inconel 718 is frequently used as a disc material in landbased gas turbines. Haynes 282, a quite newly developed Ni-base superalloy, is a candidate for several high-temperature applications in both aircraft and land based gas turbine engines. AM SLM Hastelloy X is used for burner components. The design of a land based gas turbine and an aircraft engine are quite similar which means that the presented research in this thesis could be of use to both industries. The main goal has been to find a way to describe crack growth and determine which mechanisms are present.

1.1 Aims and research questions

The overall aim of the present work underlying this thesis has been to increase the knowledge regarding which deformation and damage mechanisms affect crack propagation in superalloys depending on cycle type, microstructural and grain morphology. More specifically, the following research questions have been addressed:

- 1. How does the implementation of a dwell-time at maximum load affect crack propagation rates in superalloys? (Papers I–V)**

It is well known that dwell-times can have an accelerating affect on crack propagation rates. So the question to be answered is how big the effect of dwell-times is on crack propagation rates. This is of utmost importance when calculating and modeling turbine life.

- 2. How does the implementation of an overload prior to the start of each dwell-time affect crack propagation rates in Inconel 718? (Paper I)**

Overloads occur repeatedly during gas turbine run cycles. It is therefore important to understand how overloads effect crack propagation rates.

3. How does δ -phase orientation affect the crack propagation path in Inconel 718? (Paper III)

Inconel 718 gas turbine discs are often processed under the δ -solvus temperature giving rise to a forming induced arrangement of the δ -phases. It is important to understand how the forming induced arrangement affects the crack propagation.

4. How does grain size affect crack propagation rates in Inconel 718? (Paper IV)

Choosing the incorrect grain size can lead to catastrophic failure of gas turbine components when fatigue is taken into account. Therefore, research is needed where different grain sizes and differently processed Inconel 718 is investigated to optimize for balance between fatigue and creep.

5. How does microstructural and grain morphology as well as heat treatments affect crack propagation rates in laser powder bed additive manufactured (AM) Hastelloy X? (Paper V)

Today we classify most materials as cast, forged, bar or rolled. How should AM materials be classified? Should they be classified as one of the former ones or should AM materials be considered as a new material group? This is why microstructural and grain morphology as well as heat treatments need to be studied and compared with differently processed materials to fill the knowledge gap between AM materials and other material groups.

1.2 Outline

This thesis builds upon my licentiate thesis work “Effect of dwell-times on crack propagation in superalloys” [8] and comprises two parts. Part I gives an introduction into the field of superalloys and acting microstructural mechanisms needed to give a better understanding of the materials science and experiments discussed in Papers I–V. As well as a summary of all the results obtained from Papers I–V. Part II consists of five appended scientific papers.

CHAPTER 2

Background

Gas turbines are used for various applications. They are used in both civil- and military-aircraft and helicopters. When looking at electrical power generation, gas turbines of all shapes and sizes are used, micro-turbines with a power output from 20 kW up to the largest frame-type turbines with a power output of 480 MW. Gas turbines have a thermal efficiency of 15 – 46 % when used in a single cycle configuration [1]. Gas turbines are preferably run at constant, rather than a fluctuating, load. This makes gas turbines superior for applications like power plants and transcontinental jet aircrafts. Gas turbines like the Siemens SGT-500, 600, 700 and 750 are used to drive generators, cruise ships, destroyers, and to drive oil and gas pipeline pumps all over the world in environments where the weather conditions vary from the arctic colds in Siberia to the hot climate of Thailand. The present work is focused on superalloys used in industrial power generation.

Simple-cycle gas turbines can, according to Boyce [1], be classified into five general groups:

- ***Frame type heavy-duty gas turbines*** are large power generating units that range from 3 MW to 480 MW in a simple cycle configuration, with efficiencies ranging between 30–46 %.
- ***Aircraft-derivative gas turbines*** are power generating units, originating from aerospace industry as the prime mover of aircrafts. These units have been adapted to the electrical generation industry by removing the bypass fans, and adding a power turbine at their exhaust. These units range in power from 2.5 MW to about 50 MW. The efficiency of these units can range between 35–45 %.
- ***Industrial type-gas turbines (IGT)*** vary in range from about 5 MW–40 MW. This type of turbine is used extensively in many

petrochemical plants to drive compressors. The efficiencies of these units are between 15–35 %.

- **Small gas turbines** are generating units that range between about 0.5 MW–2.5 MW. They often have centrifugal compressors and radial inflow turbines. With efficiencies varying between 15–25 %.
- **Micro-turbines** are turbines in the range between 20 kW–350 kW.

2.1 The workings of a gas turbine

Fig. 2.1 shows a model of the SGT-750 gas turbine manufactured by Siemens Industrial Turbomachinery AB in Finspång, Sweden. The SGT-750 is a medium size industrial gas turbine that can be used for mechanical drive and electrical power generation or in a combined power and heat generation cycle. All specific data used in the following description are specific to the SGT-750 and will vary between machines and manufacturers. All gas turbines work according to the same principle and can be explained using the following steps.

Figure 2.1. Siemens Industrial Gas Turbine SGT-750 (Courtesy of Siemens Industrial Turbomachinery AB).

1. An electrical starter motor connected to the shaft is used to start the gas turbine. Which in turn rotates the rotor until a high enough speed has been achieved to make it self-sustained. When the turbine is creating enough power to drive the compressor, the electrical starter engine can be disconnected. Combustion engines can also be used as starter motors. In the early days of gas turbine development pressurised air was blown into the air intake to get it rotating.
2. Approximately 130 m^3 air per second [9] flows through the air intake that acts as a huge funnel to the compressor. The air flow is then directed by variable guide vanes which are used to optimise air flow performance depending on intake air quality and surrounding weather conditions, such as

temperature range and particle size of air-born contaminants. When air is sucked through the 13-stage axial flow compressor it becomes pressurised with a 23.8:1 pressure ratio [9]. Between every compression stage there is a stage of stationary vanes. Vanes are used to redirect air flow yet again to optimise compressor performance.

3. After the compressor stage, air is mixed with fuel in the can-annular combustors using dry low-emission burners. The SGT-750 runs on natural gas even though there are many types of fuel available. The more common fuels in liquid form are oils, ethanol and methanol. In gas form they are natural gas, propane and butane [1]. Natural gas is used because it is the most “ECO” friendly alternative. The fuel mixture is combusted at temperatures exceeding 1400 °C resulting in gas expansion.

Combustor design is divided in two distinct configurations, annular and can-annular. Combustors in heavy industrial gas turbines usually have long combustion chambers which makes them more suitable for burning lower quality fuels which are cheaper and more freely available [10]. The annular combustor is generally placed inside and the can-annular outside the envelope of compressor and turbine. The annular combustor is a single combustor with multiple fuel nozzles with an inner wall that acts as a heat shield to protect the rotor. Can-annular combustors can also be divided into two groups, one with a straight flow-through the combustor and the other with a reverse flow combustor. The reverse flow combustor that is used in heavy industrial gas turbines facilitates the use of a regenerator [11], which improves overall thermal efficiency.

4. In the first hot stage of the turbine, pressure and exhaust temperature begins to decrease. The first hot stage blades are subjected to both high temperatures and pressure which can be detrimental to the alloys used. To protect hot stage components a ceramic thermal barrier coating can be used. In the following and last turbine stages, temperature keeps decreasing. An SGT-750 can rotate at speeds exceeding 6000 rpm [9] which results in strong centripetal forces on blades. Due to the large centripetal forces and high temperatures, all parts must be made from the best possible materials, typically Ni-based superalloys.
5. The exhaust temperature can be around 500 °C [9]. Depending on user needs, exhaust heat can be used as input energy for a combined cycle processes. The exhaust heat can be used to produce steam for steam turbines and/or heat water in regenerative heat-exchangers to supply warm water for district heating.

When turbines are used for combined power and heat generation a total electrical efficiency of > 60 % can be achieved [12] and the overall efficiency can be increased to > 90 % [9].

2.2 Superalloys

Titanium and aluminium was added in 1929 to the by then well-known face centered cubic (FCC) “80/20” Ni-Cr alloy, resulting in the first Ni-based superalloy [13], which exhibited significantly better creep strength compared to its Ni-Cr counterpart. Since then, the development and investigation of superalloys continues. The term superalloy was first used shortly after World War II to describe a group of alloys developed for use in turbosuperchargers and aircraft turbine engines that required high performance at elevated temperatures. Well proven mechanical properties of superalloys in hot sections of jet engines and power generation turbines is paramount since the turbine operating temperature is directly connected to turbine efficiency. Superalloys have been specifically designed to withstand service temperatures exceeding the capability of steels [14] and up to temperatures as high as 1000 °C [4]. These alloys can be Fe-, Co- or Ni-based. The latter has gained the most interest from the gas turbine industry for use in high temperature components due to the good combination of mechanical, oxidation and corrosion resistant properties at high temperatures. Fig. 2.2 shows that the mechanical strength of Ni-based superalloys are far beyond that of the other alloy groups. Therefore, an in-depth knowledge of the acting degradation mechanisms (both mechanical and environmental) in superalloys are of utmost importance enabling not only for alloy development but also modeling used for predicting component life.

Figure 2.2. Maximum service temperature for different groups of creep-resistant alloys, redrawn from [15], where denoted $R_{p0.2}$ is an offset of the yield strength.

At temperatures above ~ 540 °C, ordinary steels and Ti alloys lose their mechanical properties and become inferior in comparison to the Ni-based alloys. Steels are also more prone to corrosion at these elevated temperatures. The only material groups capable of service temperatures in excess of 1000 °C are Ni-based superalloys and refractory metals. Refractory metals such as Tungsten could be

considered. As seen in Fig. 2.2, they lack the desired ductility of superalloys [14]. Co-base superalloys can be used instead of Ni-based alloys, but they are in many cases not as strong and/or corrosion resistant as Ni-based alloys. Fe-Ni-based superalloys like Inconel 718, are used at lower service temperatures (turbine discs), to a larger extent than the Co- or Ni-based superalloys and are less expensive and malleable.

Improvements in both alloy composition and processing has allowed designers to push the limits of gas turbines and improve engine efficiency since the introduction of superalloys. As well as the increased environmental awareness and climate changes [16] and the increased generation of emissions, such as CO₂, by the logistics industry [17] pushes for more efficient gas turbines. Fig. 2.3 shows that the improvement rate of superalloys have stagnated concerning turbine entry temperature. This has instead given rise to the investigation of new alloy systems such as Co-Al-W [18] exhibiting interesting properties, Ti-Al alloys with a lower density compared to Ni-based alloys [19], fibre reinforced ceramic blades [20], and high entropy alloys [21] which have been reported to be less prone to the formation of inimical phases such as topologically closed-packed (TCP) phases observed in Ni-based superalloys. All these new alloy systems are plausible candidates to be used to increase turbine efficiency.

Figure 2.3. Evolution of the turbine entry temperature (TET) capability of Rolls-Royces civil aeroengines, from 1940 till 2010. Redrawn from [22].

The strength of superalloys can be related to chemistry, melting/casting procedures, mechanical work processes and especially to post forming heat treatments. Superalloys contain several elements from minuscule to major amounts. The most common elements are Ni, Cr, Mo, Al, B and C.

Most wrought superalloys have quite high Cr-levels to provide corrosion resistance. However, Cr-content in cast alloys has been reduced over time and replaced by other strengthening elements [14]. Wrought superalloys can be complicated to machine by conventional methods such as turning and milling. The decrease in Cr-content in Ni-based superalloys has been compensated with a higher Al-content

and lowered S-content giving a similar oxidation resistance but decreased resistance to other types of detrimental corrosive attacks.

Generally, superalloys exhibit great oxidation resistance but poorer corrosion resistance. Components in the hotter parts of aircraft turbines ($>760\text{ }^{\circ}\text{C}$) and components subjected to high temperatures ($650\text{ }^{\circ}\text{C}$) for long periods of time, as in land based gas turbines, are coated with a protective thermal barrier coating (TBC). TBCs protect superalloys from heat and harmful elements and extend component life. The need for TBCs is illustrated in Fig. 2.4 where the yield strength of various superalloys start to degrade rapidly at $\sim 700\text{ }^{\circ}\text{C}$.

Figure 2.4. Yield strength of the wrought alloy compositions from Table 2.2 at different temperatures, redrawn from [23–25], where denoted $_{0.2}$ is an offset of the yield strength.

2.2.1 Phases

Ni-based superalloys have a microstructure mainly composed of two coherent phases. Firstly the nonmagnetic disordered FCC γ -matrix, which usually contains high amounts of solid-solution elements like Co, Fe, Cr, Mo and W [26]. Secondly, ordered FCC ($L1_2$) γ' . γ' being the main strengthening precipitate in Haynes 282 is required for high-temperature strength and creep resistance. Al and Ti are required to precipitate γ' coherently with the γ matrix [26]. The FCC γ' structure is illustrated in Fig. 2.5 where the blue corner atoms represent Al or Ti and the grey surface atoms represent Ni. The crystal structures and lattice parameters of γ and γ' are so similar that the two coherent phases share parallel $\{001\}$ planes and $\langle 001 \rangle$ directions. γ' can also be found in grain boundaries as a result of heat treatments and or elevated temperatures during service. Grain-boundary γ' can form a film along grain boundaries which can prevent grain boundary dislocations. However, it can become unstable and instead of preventing dislocations it can act as an undesired low friction grain boundary film [26].

Inconel 718 gains its strength from ordered (DO_{22}) body centered tetragonal (BCT) Ni_3AlNb γ'' . Ni and Nb form γ'' in the presence of Fe, which is coherent

Figure 2.5. γ' crystal structure, generated using Vesta [27].

with the γ matrix. Large mismatch strains are induced by the γ'' phase. This provides high strength at temperatures up to 650 °C, which limits its use to land based gas turbine discs. Over time γ'' is prone to transform into orthorhombic Ni_3Nb δ , another common phase in Inconel 718. In Inconel 718 δ phase is formed in the temperature range $\sim 650\text{--}980$ °C [28]. Hexagonal η , given rise by a high Ti/Al ratio, has been found in 718 Plus, a derivative of Inconel 718. δ and η are quite similar with the main difference being the stacking sequence. Generally, δ and η are observed as a plate like morphology. δ and η are not commonly regarded as strengthening phases but can in small quantities be used to refine grain size, and improve fatigue crack growth in certain directions [29].

Carbides and borides are regularly observed in polycrystalline Ni-based superalloys. They are used to improve creep resistance by impeding grain boundary sliding. Carbides can precipitate when up to 0.2 % carbon is added and combined with carbide reactive elements such as Ti, Ta, Hf and Nb [26]. In Haynes 282 and Hastelloy X carbides exist in the form of grain boundary carbides. Some carbides can precipitate during extended periods of service when subjected to sufficiently high temperatures. As a result carbides decompose and generate other carbides such as M_{23}C_6 and/or M_6C at grain boundaries. The “M” element is Cr, Ni, Co, Fe, Mo, W, Nb, Hf, Th, Zr or Ta [26]. Carbides are considered to have a beneficial effect on rupture strength at high temperature and also to influence ductility [30]. Borides are found in superalloys in the form of tetragonal M_3B_2 . They are formed when boron segregates to grain boundaries. Small additions of boron are essential to improve creep rupture resistance in superalloys. Borides are hard particles, blocky to half moon in appearance and usually observed at grain boundaries [26].

Not all phases are strengthening. Topological close packed (TCP) phases such as tetragonal σ , rhombohedral μ , and hexagonal Laves can be detrimental even in small quantities. σ is most common in Fe- and Co-based superalloys in the shape of irregular globules. σ can precipitate when components are subjected to long service times at temperatures in the range 540–980 °C. μ can be found in high Mo or W content alloys and generally appears as coarse irregular Widmanstätten platelets which are formed at high temperatures. Laves phase precipitate when components are exposed to high temperatures for extended periods of time [31] and is most common in Fe- and Co-based superalloys in the shape of irregular globules or platelets. σ , μ and Laves phase embrittle the microstructure and decrease rupture strength and ductility which results in reduced component life.

2.2.2 Alloying elements

Superalloys have a quite simple microstructure even though they occasionally are comprised of up to 14 elements. They have been developed for their usage and processing technique from the moment they were invented with the purpose to improve mechanical, temperature, oxidation, corrosion, grain boundary strengthening properties and castability. Every element has its specific role in superalloys and can effect mechanical, oxidation and corrosive properties. How a specific element effects a certain property or phase is shown in Table 2.1. Some of the elements can improve mechanical strength through solid solution strengthening and/or precipitation hardening. Precipitates can be in the form of carbides based on various elements. Cr improves corrosion and Al improves oxidation resistance respectively but both are also matrix strengtheners, although Al has a high affinity to create γ' phase. Heavy elements such as Mo, W, Nb and Ta are used for matrix hardening. The downside of using heavy elements is the increased density which is not optimal for aero and space applications. Elements like Cr, W, Nb and Fe can have a positive effect, but when used excessively they can form TCP phases such as σ , μ and Laves phase [26, 32]. The principal elements used for γ' precipitation are Al and Ti, in some cases Nb and Ta. Tantalum can be used to replace Ti in single crystal superalloys. Tantalum also raises the solidus temperature and volume fraction of γ' to 70–80 % in single crystal alloys. More in-depth information on the effect of alloying elements in superalloys can be found in literature [33–35].

Fig. 2.2 shows that Ni-based superalloys are especially suitable for high temperature applications. They often contain multiple alloying elements which make them considered as advanced alloys. They have gained great interest from industry due to their good mechanical and corrosive properties which are often well documented in literature [14, 33–35]. Ni-based superalloys can be classified into three groups: wrought, cast, and powder metallurgy (PM) alloys.

Wrought superalloys are suited for mechanical work such as forging, turning, milling and welding. The compositions of a selection of wrought superalloys are given in Table 2.2. They are often solid solution strengthened as Hastelloy X and/or through the formation of γ' [36] as in Haynes 282 or γ'' in Inconel 718. γ' and γ'' are typically precipitated during heat treatments.

There are three subdivisions of cast superalloys: polycrystalline, directionally solidified (DS), and single crystal, the latter being the most advanced. The compositions of a selection of cast superalloys are given in Table 2.3. Cast superalloys are generally more arduous to machine by turning and milling than wrought superalloys. Cast and wrought superalloys are strengthened through the precipitation of γ' and/or γ'' . The advantage of casting is that not only regular polycrystalline materials can be cast but also directionally solidified and single crystal materials can be cast. The fatigue and creep life of directionally solidified materials can be improved by engineering the amount of grains, grain boundaries and the Young's modulus through anisotropy.

Table 2.1. Effects of major alloying elements in Ni-based superalloys [26].

Effect *	Fe-based	Co-based	Ni-based
Solid-solution strengtheners	Cr, Mo	Nb, Cr, Mo, Ni, W, Ta	Co, Cr, Fe, Mo, W, Ta, Re
FCC matrix stabilizers	C, W, Ni	Ni	–
Carbide formers			
MC	Ti	Ti	W, Ta, Ti, Mo, Nb, Hf
M ₇ C ₃	–	Cr	Cr
M ₂₃ C ₃	Cr	Cr	Cr, Mo, W
M ₆ C	Mo	Mo, W	Mo, W, Nb
Carbionitrides :M(CN)	C, N	C, N	C, N
Promotes general precipitation of carbides	P	–	–
Forms γ' Ni ₃ (Al, Ti)	Al, Ni, Ti	–	Al, Ti
Retards formation of hexagonal η (Ni ₃ Ti)	Al, Zr	–	–
Raises solvus temperature of γ'	–	–	Co
Hardening precipitates and/or intermetallics	Al, Ti, Nb	Al, Mo, Ti**, W, Ta	Al, Ti, Nb
Oxidation resistance	Cr	Al, Cr	Al, Cr, Y, La, Ce
Improve hot corrosion resistance	La, Y	La, Y, Th	La, Th
Sulfidation resistance	Cr	Cr	Cr, Co, Si
Improves creep properties	B	–	B, Ta
Increases rupture strength	B	B, Zr	B***
Grain-boundary refiners	–	–	B, C, Zr, Hf
Facilitates working	–	Ni ₃ Ti	–

* Not all these effects necessarily occur in a given alloy.

** Hardening by precipitation of Ni₃Ti also occurs if sufficient Ni is present.

*** If present in large amounts, borides are formed.

Table 2.2. Wrought superalloy compositions, [wt. %], [37].

Alloy	Ni	Cr	Co	Mo	W	Nb	Al	Ti	Fe	C	B	Zr
Hastelloy X	Bal.	22.0	1.5	9.0	0.6	-	0.25	-	18.5	0.1	-	-
Haynes 282	Bal.	19.6	10.3	8.7	0.01	0.10	1.5	2.2	0.5	0.06	0.005	-
Inconel 718	Bal.	19.0	-	3.0	-	5.1	0.5	0.9	18.5	0.04	-	-

Table 2.3. Cast superalloy compositions, [wt. %], [38].

Alloy	Ni	Cr	Co	Mo	W	Nb	Al	Ti	Fe	C	B	Zr	Other
Hastelloy X	50	21	1	9	1	-	-	-	18	0.1	-	-	-
Inconel 718	53	19	-	3	-	5	0.5	0.9	18	0.04	-	-	0.1 Cu

2.3 Fatigue

Studying fatigue of components is relevant when looking at economy and safety. The manufacturers will try to produce a component to be fatigue free with the highest possible fatigue resistance considering various cyclic stresses, material properties, residual stresses, surface quality, etc. There is a need to consider why the component might still fail as a result of fatigue due to detrimental cycles not considered by the designers such as incidental damage, aggressive atmospheres and improper use of the component. This means that crack initiation points and propagation rates should be of utmost interest. However, crack initiation in a component is component specific since some components are designed to allow for cracking to occur and others not. As a consequence nucleation is not considered as much as propagation rates. For turbine parts, a finite life is to be expected due to the severe and detrimental environments they are subjected to. Therefore, both nucleation and propagation need considering.

Fatigue life can be divided in to three parts: (i) the nucleation part comprising nucleation and microcrack growth, (ii) macrocrack growth, and (iii) final fracture occurring during the final cycle which can be considered quasi-static rather than the effect of fatigue. Fatigue life equals the nucleation part plus the macrocrack growth part. This way we consider nucleation and propagation as two separately occurring phases.

It is well known that fatigue cracks generally initiate at surfaces [6]. This is most likely due to high stress levels K , surface roughness leading to local nonhomogeneous small scale stress distribution, tensile residual stresses, environmental effects, etc. All of these can aid nucleation and propagation of surface cracks. Fatigue crack initiation is commonly explained using cyclic deformation induced surface roughening as proposed by Wood [39]. Surface roughening can be described as microscopic extrusions and intrusions where slip bands appear at the surface. These slip bands, known as persistent slip bands (PSB), form as a result of cyclic slip irreversibilities. Fatigue cracks usually initiate in valleys of the roughened surface and at the PSB-matrix interface due to the fact that strains are highly inhomogeneous in PSB-matrix interface [40, 41]. Grain boundaries, twin boundaries, and inclusions can also work as crack initiation sites [42–44].

In materials containing large amounts of discontinuities like surface and internal inclusions, fatigue cracks initiate at these discontinuities most likely due to the lower toughness of the discontinuities compared with the matrix [45–51]. If a fatigue crack forms at an inclusion and begins to propagate depends on the type of inclusion, size of the inclusion, distribution of inclusion and residual stresses present. Discontinuities such as porosities which are quite common in cast- and powder-metallurgy alloys can also work as possible crack initiation points [52–56]. Crack initiation transition from surface to sub-surface/internal pores and inclusions have been reported to occur during giga-cycle fatigue testing [45, 57]. In addition, texture can affect fatigue crack initiation in Ni-based superalloys and Ti alloys [58–61]. Clustered grains can act more like a single grain exhibiting similar crystallographic orientation and enable slip across low angle grain boundaries, thereby creating the possibility for slip to occur easier over longer distances, resulting in dislocation pile-ups and stress concentrations at grain boundaries.

Figure 2.6. Schematic illustration of the primary fracture mechanisms in steels as a function of the applied stress-intensity range and effects of several major variables on crack growth behaviour, redrawn from [62].

A crack can be considered a macrocrack when the stress intensity factor K has a “real” meaning when describing crack growth. Fatigue crack growth according to the school books is divided into three régimes illustrated in Fig. 2.6.

I *The threshold régime* is characterised by low crack growth rates and is strongly influenced by both microstructure and environment. II *The Paris régime* where crack growth rate is considered linear. The Paris régime is influenced more by testing atmosphere, specimen geometry, and frequency. III is *the final régime* where crack growth is considered unstable and characterised by rapid crack growth leading to final failure. Régime I and II are the ones of the most interest when consid-

ering life assessment and modeling. Researchers have studied fatigue arduously, mainly from two points of view: (i) researchers like myself focusing on microstructure and (ii) with others focusing on modeling crack growth rates. Macroscopic differences such as number of cycles to failure, frequency and cycle type are just some of the parameters influencing crack growth rates. In Papers I–V the effect dwell-times have been investigated focusing on microstructural morphology and crack appearance.

2.4 Dwell-times

It has been well established that time dependent intergranular cracking of Ni-based superalloys, under both sustained and cyclic loads, is dominated by environmental interactions with oxygen at the crack tip [63, 64]. Intergranular cracking does not only occur due to the formation of massive oxidation products along the grain boundaries. The mechanism might be better described as nano scaled dynamic embrittlement (DE) where oxygen diffuses into highly stressed grain boundaries at the crack tip which causes decohesion [65, 66]. Previous studies [67, 68] have shown that complex oxides of Ni, Cr and Fe as well as oxides formed from Nb-carbides, can be formed at the crack tip in Inconel 718. Conforming with the crack tip oxidation observations in Papers I–V crack growth rates increased when a dwell-time was applied at the maximum load illustrated in Fig. 2.7 (a).

Figure 2.7. Inconel 718 crack propagation rates run at 550 °C with $R = 0.05$ presented as da/dN vs. ΔK for (a) fine grained Inconel 718 bar, grain enlarged bar and cast. Data redrawn from [69]. (b) The retarding effect of overloads is exemplified, with data redrawn from [7]. (c) Showing the effect of anisotropy between samples cut in two directions, SC and CS. The dashed circles and filled dots show where the cracks start to grow out of plane more than 10° perpendicular to the loading direction, circles show the theoretical validity of the ΔK calculation according to ASTM E399-97. Data redrawn from [29].

Fig. 2.7 (a) also shows how grain size can affect dwell-fatigue crack growth rates. With the introduction of a dwell-time cracks grew intergranularly compared to samples subjected to pure fatigue which exhibited more transgranular crack growth.

Introducing an overload prior to a dwell-time can significantly influence crack growth rates [7] as exemplified in Fig. 2.7 (b). With an increasing overload the dwell-time effect can be more or less extinguished and crack growth rates converge with that of the purely fatigue loaded. This can partly be explained by the zone of compressive stress formed at the crack tip after partial unloading [70]. It is not obvious whether the decrease in crack growth rate is only due to a reduction of the crack driving force or if the embrittlement effect of the material is also reduced by a reduction of the oxygen diffusion rate at the crack tip.

Morphology or anisotropy such as δ -phase orientation has also been shown to impact dwell-fatigue crack propagation rates as reported in Paper III [29] and shown in Fig. 2.7 (c). When subjected to 2160 s dwell-times cracks grew mainly intergranularly and in the γ/δ interface. As a result the cracks would grow out of plane when the δ -phases were orientated perpendicular to the loading direction. The effect of building direction in additive manufactured (AM) selective laser melted (SLM) Hastelloy X on dwell-fatigue crack propagation at 700 °C was investigated in Paper V showing that it has a unique grain morphology yielding highly anisotropic behavior. The AM material is highly anisotropic as a result of the layer by layer manufacturing technique resulting in elongated grains in the building direction. During dwell-fatigue crack propagation occurred mainly intergranularly. As a result of dwell-fatigue intergranular creep damage was observed in front of crack tips.

Fig. 2.8 redrawn from [71] shows how dwell-time crack growth, creep, tensile and Low cycle fatigue (LCF) properties change with an increasing grain size for Udimet 720. A reduction in both tensile strength and LCF is a result of the larger grain size, but the larger grain size does not only have detrimental effects, it significantly improves the resistance against dwell-time crack growth and creep properties as illustrated in Fig. 2.7 (b).

2.5 Crack propagation mechanisms

Understanding crack propagation mechanisms is important, especially when life modeling comes into play. Vast amounts of research and plenty of books have been written on the subject. By no means does this mean that all problems have been solved. Components used in hazardous environments and other adverse applications commonly exhibit shortened life as a result of fatigue. Many models today can be used to estimate the life of components under very specific conditions. In reality it is very hard to accurately estimate component life, at least with the already preexisting models available. A life prediction model for the overload tests microstructurally investigated in Paper I [7] was developed by Gustafsson *et al.* [70]. The other test series leading up to it are discussed in many articles [70, 72–83] where models for fatigue crack propagation were modified to handle load ratios

Figure 2.8. Normalised life and strength vs. grain size, redrawn from [71].

R , stress-intensity factors K , threshold values and environmental effects, which are vital to accurately enable life prediction. Modeling is of course important, but more so is the understanding and possibility to explain the leading mechanisms behind the crack propagation during fatigue. Dynamic embrittlement (DE) and stress accelerated occasionally termed strain-assisted [67] grain boundary oxidation (SAGBO) are the two most common mechanisms used to explain why crack propagation rates accelerate with dwell-times at elevated temperatures.

Dynamic embrittlement

Wrought fine-grained polycrystalline Ni-based superalloys, such as Inconel 718, are in many situations limited by their susceptibility to expeditious intergranular cracking during prolonged dwell-times at high temperatures and high tensile stresses [73]. It has been well established that time dependent intergranular cracking of Ni-based superalloys, under both sustained and cyclic loads, is dominated by environmental interactions with oxygen at the crack tip [63, 64]. Intergranular cracking is not only due to the formation of massive oxidation products along grain boundaries. The mechanism could be better described as nano scaled DE, where oxygen diffuses into highly stressed grain boundaries at the crack tip and mainspring decohesion [65, 66]. Previous studies [72, 84, 85] have shown that Inconel 718 mainly cracks transgranularly during cyclic testing in the lower temperature range and intergranularly during fatigue and dwell-fatigue at higher temperatures. The same behaviour has been perceived in other superalloys such as Waspalloy [74]. Grain boundary embrittlement has been studied in [64, 74, 75] where it was shown that the crack growth per cycle during unloading-reloading is much higher after a dwell-time period compared to pure cyclic loading. Similar observations during thermomechanical fatigue crack growth tests have been reported in [73].

DE as a mechanism was first mentioned by Liu and White [86] as an explanation to the increase in crack growth when comparing cyclic crack growth

Figure 2.9. Schematic representation of the dynamic embrittlement mechanism where (a) is before cracking and (b) is after cracking.

with sustained and/or dwell-time crack growth. DE is a time-dependent brittle, intergranular fracture mechanism [87] controlled by the diffusion of embrittling elements to grain boundaries, illustrated in Fig. 2.9. Embrittling elements can be pre-existing in the alloy. Sulphur and oxygen supplied by the surrounding atmosphere can induce cracking in alloy steels and Ni-based superalloys [87]. DE might be a generic damage mechanism [65] that occurs if: (i) Embrittling species with low melting temperatures, either pre-existing in the alloy or from the surrounding atmosphere, adsorb at the free surface at the crack tip. (ii) The temperature and elastic stress ahead of the crack tip are high enough to enable grain boundary diffusion of embrittling species. This is of greater concern when looking at high-strength materials since plastic deformation at the crack tip can cause stress relaxation, resulting in a diffusion retardation of the embrittling species. (iii) The embrittling species lowers the grain boundary cohesion enough that crack growth can persevere.

Microstructure can significantly impact the DE process. A microstructure consisting of large grains is more prone to higher grain boundary stress concentrations and usually exhibit a shorter and less tortuous crack appearance. The DE fracture mechanism is not only determined by grain size but could also be due to a crystallographic misorientation relationship [65]. As DE sets in, the build up of diffused species aid the decohesion process. When this build up has reached a high enough level, oscillating macroscopic crack propagation can begin. The oscillation can be explained by a selective intergranular diffusion process depending on the geometry and structure of the grain boundaries [65]. After creep and plastic rupture of uncracked ligaments the crack tip stress intensity increases resulting in intergranular fracture. To improve the resistance against DE of polycrystalline Ni-based alloys small additions of B, C, Hf or Zr can be used [88, 89]. This may improve the grain boundary strength by lowering either the grain boundary energy or the embrittlement diffusivity.

Stress accelerated grain boundary oxidation

SAGBO is an environmentally assisted crack growth mechanism suggested to be either a partial or the leading mechanism for crack growth in alloys tested in air. The SAGBO mechanism was first reported by McMahon *et al.* [90], in which it was called “stress-corrosion cracking”. Carpenter *et al.* [91] wrote one of the first papers discussing SAGBO in connection with crack propagation. Li *et al.* [92] suggested that the sustained load crack growth in RR1000 at 700 °C takes place by a mechanism similar to DE by repeated fracture of an oxide film in the grain boundary ahead of the crack, namely SAGBO. Such films, with penetration depths of around 1–10 μm ahead of the tip, have been observed in dedicated experiments in several Ni-based superalloys [67, 93, 94]. SAGBO as a crack propagation mechanism has been discussed and suggested as a possible crack propagation mechanism in several other papers [64, 65, 86, 95–103]. SAGBO is also supported by observations in Paper IV, Fig. 2.10 (a), where a crack propagated through an oxidized grain boundary in a cast Inconel 718 2160 s dwell-time sample. SAGBO works as a result of grain boundary oxidation which then cracks and exposes new surfaces for further oxidation and subsequent cracking, as illustrated in Fig. 2.10 (b).

Figure 2.10. Stress accelerated grain boundary oxidation illustrated as (a) a crack propagating through an oxidized grain boundary in a cast Inconel 718 2160 s dwell-time sample and in (b) a schematic representation of the stress accelerated grain boundary oxidation (SAGBO) mechanism.

Brittle/cleavage striations

Fatigue striations are commonly found on fracture surfaces of various alloys ranging from Al-alloys to Ni-based superalloys. Generally there are two types of fatigue striations, plastic and brittle striations [104–106]. Brittle striations are also called cleavage striations [107] in various literature. The mechanism behind striation formation can generally be explained as a two-step process. The first step being the result of crack-tip blunting during the loading part of a fatigue cycle, followed by a resharpening of the crack tip during the load reversal. The

lack of striations on fracture surfaces of metals tested in vacuum are due to oxides reducing slip reversal during crack closure. When fatigued in hard vacuum, slip can be almost completely reversed. Both types of striations are transgranular. Ductile striations are called ductile because the material ahead of the crack tip undergoes plastic deformation resulting in the typical curved arrays by which they advance on fracture surfaces [108] as illustrated in Fig. 2.11 (a). Brittle striations, Fig. 2.11 (b), are formed during a two step process, first by cleavage followed by crack-tip blunting and dislocation generation. They appear as concentric circles starting from an initiation point, often from brittle inclusions. This is why brittle striations typically show a flat appearance with no real signs of plastic deformation. Brittle striations are always associated with corrosion assisted fatigue and, in particular, with hydrogen absorption [108]. The striation formation depends on the combination of cleavage as a result of local shear stress [107]. Brittle striations were observed in Haynes 282 in Paper II. These observations are very similar to the striations observed during environmental hydrogen embrittlement of Ti-alloys [109–112]. In Ti-alloys, the brittle striations have been proposed to be due to the repeated stress assisted formation and fracture of hydride films ahead of growing cracks [110]. The crack growth behaviour in gaseous hydrogen exhibited similar behaviour as Haynes 282 in Paper II, including the threshold above which an increased environmental effect could be observed [111, 112].

Figure 2.11. Fracture surfaces of the Haynes 282 90 s dwell-time sample in Paper II [113] (a) Fatigue striations. (b) Brittle striations.

2.6 Crack retarding mechanisms

If we look at a propagating fatigue crack we will see plastic deformation “moving” ahead of the crack tip. The plastic zone increases in size meaning that the plastic zone is proportional to the crack length. The same will be true for the reversed plastic zone. Monotonic plastic deformation (elongation in x- and y-directions)

is left in the wake of the crack as a result of the monotonic plastic zone being larger than the reversed plastic zone. The elongation can induce crack closure or partial crack closure during unloading and result in compressive residual stresses being induced in the wake of the crack, meaning that compressive residual stresses are transmitted through the crack when the fracture surfaces are pressed together causing plastic deformation. If the fracture surfaces are pressed together before the load cycle has been fully unloaded it means that the crack is no longer fully open vis-à-vis crack closure has occurred. Crack closure was first observed by Elber Wolf [114] and is occasionally referred to as the Elber mechanism. The effect of crack closure can be obtained from stiffness measurements using crack opening displacement as well as from more indirect evidence like the effect on fatigue crack propagation.

The plastic zone in front of the crack tip can also result in local hardening leading to crack tip blunting. Crack tip blunting is quite common during dwell-fatigue and occurs when the plastic zone ahead of the crack tip grows large enough [115] so that it starts behaving like a notch [116] with a lower stress concentration than that of the originally sharp crack tip. Blunting of the crack tip will impede the crack growth rate since the energy needed to propagate a blunted crack tip is higher compared to that of a sharp crack tip. The blunting mechanism and its retarding effect on crack growth has been investigated and described in several papers [7, 63, 117–120] which discuss its retarding effect during different loading conditions such as fatigue, dwell-, and creep-fatigue has also been modeled by Ismonov *et al.* in [121].

Other crack growth mechanisms, such as dynamic recrystallization, strain localization in persistent slip bands, deformation bands and vacancy diffusion, have been proposed in several studies [122–126] as possible culprits for aiding crack propagation.

CHAPTER 3

Experimental procedures

Standard heat-treated and grain enlarged Inconel 718 bar was used in Papers I and IV. The standard heat treatment was conducted according to AMS 5663: solution annealing for 1h at 945 °C, followed by ageing for 8 h at 718 °C and 8 h at 621 °C. Inconel 718 with a nominal chemical composition shown in Table 3.1 were machined from a real turbine disc press-forging in Paper III. Forging was performed below the δ -solvus temperature, obtaining a fine grained microstructure. Consequently, the δ -phase orientates along flow lines during the press-forging. After press-forging the disc was sub-solvus heat-treated accordingly: solution annealing for 3.5 h at 970 °C, followed by oil quenching and ageing for 8 h at 720 °C and 8 h at 620 °C.

Haynes 282 in the form of a forged ring was studied in Paper II. The forged ring was heat-treated accordingly: solution heat treated for 2 h at 1100 °C then aged for 2 h at 1010 °C, with a final ageing treatment at 788 °C for 8 h. The material had a chemical composition as shown in Table 3.2 and an average grain size of 120 μm or #3 according to ASTM-E112.

Both cast Hastelloy X and by additive manufacturing (AM) selectively laser melted (SLM) EOS NickelAlloy HX conforming to AMS 5754 / UNS N06002 were utilised in Paper V. The AM SLM material was used in the as-manufactured condition as well as heat treated at 1177 °C being the standard heat treatment for 30 min as well as at 900 °C for 30 min which is a more suitable heat treatment for AM SLM Hastelloy X. No hot isostatic pressing was conducted. AM SLM powder material is gas atomized and sieved to a fraction (10–45 μm) suitable for the SLM process. The nominal composition in wt. % of EOS NickelAlloy HX is shown in Table 3.3. In literature Hastelloy X can also be identified as Alloy X. General mechanical properties for the different alloys are given in Table 3.4

All the material used were supplied by Siemens Industrial Turbomachinery AB and GKN Aerospace Engine Systems. Fatigue crack propagation testing and

microscopy studies were performed at the Division of Engineering Materials at Linköping University.

A Hitachi SU70 FEG analytical scanning electron microscope (SEM), operating at 1.5–20 kV was used together with SEM techniques such as, electron channelling contrast imaging (ECCI) [127, 128] and electron back scatter diffraction imaging was used for microstructural analysis in all included Papers.

Table 3.1. Composition of elements for Inconel 718 [wt.%) [129].

Element	Wt %	Ni	Cr	Fe	Mo	Nb	Co	C	Mn	Si	S	Cu	Al	Ti
	Min.	50	17	bal.	2.8	4.75							0.2	0.7
	Max.	55	21		3.3	5.5	1	0.08	0.35	0.35	0.01	0.3	0.8	1.15

Table 3.2. Nominal chemical composition, Haynes 282 [wt.%) [25].

Element	Ni	Cr	Co	Mo	Ti	Al	Fe	Mn	Si	C	B
	Bal.	20	10	8.5	2.1	1.5	1.5*	0.3*	0.15*	0.06	0.005

*Maximum.

Table 3.3. Nominal chemical composition, EOS NickelAlloy HX [wt.%) [130].

Element	Ni	Cr	Fe	Mo	Co	W	C	Mn	Si	B	Nb	Al	Ti
	Bal.	22	18	9	1.5	0.6	0.1	1*	1*	0.008*	0.5*	0.5*	0.15*

*Maximum.

Table 3.4. Monotonic mechanical properties for Inconel 718 [129], Haynes 282 [25], and Hastelloy X [131].

Alloy	$R_{P0.2}$ [MPa]	R_M [MPa]	Youngs modulus [GPa]
Inconel 718	1034	1240	200
Haynes 282	699	1132	217
Hastelloy X (Wrought)	350	770	192
AM SLM Hastelloy X 0°	676	840	201
AM SLM Hastelloy X 90°	531	720	173

3.1 Kb specimens

Kb-type test specimens illustrated in Fig. 3.1 were used in papers I and IV. They had a rectangular cross-section of 4.3×10.2 mm and an electro-discharge machined starter notch. Testing was conducted in a 160 kN MTS servo hydraulic tensile/compression testing machine, equipped with a three zone high temperature furnace. A fatigue pre-crack was propagated in laboratory air, at room temperature, with a load ratio of $R = 0.05$, using a sinusoidal cyclic frequency of 10 Hz,

resulting in a semi-circular pre-crack. After which high temperature testing was started. All dwell and overload tests were conducted in laboratory air.

Figure 3.1. Drawing of the Kb-specimen with the rectangular cross section, dimensions in mm.

Crack growth was measured according to ASTM-E647-08 using a 12 A channel pulsed direct current potential drop (DCPD) system. Crack length was calculated by dividing the potential drop (PD) over the crack by the PD on the opposite side of the sample as a reference. This ratio was then converted to crack length assuming a semi-circular crack front using an experimentally acquired calibration curve for Inconel 718. The calibration curve showed PD ratio as a function of crack length, based on initial and final crack lengths measured on fracture surfaces as well as by measured induced beach marks [70]. The analytical solution for the stress intensity factor K was obtained using a pre-solved case for a semi-elliptic surface crack according to ASTM E740-03. The test was interrupted when a crack had propagated 2.5 mm according to the PD value.

3.2 Compact tension specimens

Compact tension (CT) samples with side grooves were used in Papers II and III and without side grooves in Paper V. A CT specimen with side grooves and PD instrumentation is illustrated in Fig. 3.2 (a). Sample measurements are shown in Fig. 1 (b) and (c). All CT samples were pre-cracked according to ASTM-E647 using the compliance method for crack length measurements at room temperature. All samples were pre-cracked at room temperature using a sinusoidal cyclic test frequency of 10 Hz. Crack opening displacement was measured using an Instron clip gauge extensometer.

High temperature crack propagation tests were then conducted implementing dwell-times (90 s, 2160 s) as well as sustained loading (creep). Testing was conducted in a 100 kN Zwick servo electric tensile testing machine (Kappa 50DS), equipped with a three zone high temperature furnace. At high temperatures all tests were performed according to ASTM-E647-08 using a 20 A pulsed direct current potential drop (DCPD) system where crack lengths were obtained via the

Johnson formula [132], according to

$$a = \frac{2W}{\pi} \cos^{-1} \frac{\cosh\left(\frac{\pi y}{2W}\right)}{\cosh\left[\frac{U}{U_0} \cosh^{-1}\left(\frac{\cosh\frac{\pi y}{2W}}{\cos\frac{\pi a_0}{2W}}\right)\right]}, \quad (3.1)$$

were U_0 and a_0 are the initial values of the potential and crack length respectively, U and a are the actual values of the potential and crack length, y is one half of the gauge span for U , and W is the sample width. The analytical solution for the stress intensity factor, K , for CT-specimens were obtained from ASTM-E399-97. The tests were stopped at an approximate crack length of 20 mm, after which some specimens were sectioned as-is, perpendicular to the centreline of the crack, so that the crack path could be studied in a cross-section.

Figure 3.2. a) 3D view of an instrumented CT specimen with side grooves. (b), (c) A schematic rear- and side-view drawing respectively.

3.3 Potential drop crack length measurements

There are two main types of PD measuring techniques, DCPD which depends on the specimen resistivity and ACPD which relies on the specimen impedance (skin effect). Pulsed DCPD was used to measure crack propagation at high temperature in all included Papers.

DCPD and ACPD have been in use for decades. DCPD was the first method to be used due to its simplicity. In the early 1980's ACPD started catching up and

gained interest due to better sensitivity when conducting crack measurements. With time the PD techniques were developed and improved. The more sensitive pulsed DCPD and reversing DCPD were introduced resulting in improved sensitivity of the DCPD systems. During the mid 1990's microprocessor control and more advanced electronics were developed reducing the sensitivity differences between DCPD and ACPD. Both DCPD and ACPD entail at least four probe measurements. Two for current and two for PD measurements. Two extra sample connections can be utilized as a reference.

If the specimen resistance is known, the voltage drop can be calculated for any particular current value, which was done for pulsed DCPD measurements using the Johnson formula, Eq. 3.1 in Papers II, III, and V. The main difference in the pulsed DCPD setups between the Kb- and CT-samples where that two extra connections on the back of the Kb-sample were used as a reference in Papers I and IV. Specimen resistance changes when a defect or crack is introduced. Maintaining a constant current enables the possibility to detect both crack initiation as well as crack propagation, by monitoring the potential drop.

When a direct current passes through a specimen, it is usually assumed that the current spreads out instantly and evenly when the current is applied. To achieve a qualitative and measurable signal (good being a signal with a good signal to noise ratio) large currents are usually required. The typical current range used for DCPD is 10-50 A. Continuous direct current can be used but it can also result in self-heating which in turn might alter the specimen resistivity and in the end the measured DCPD. The self-heating problem can be minimised by using pulsed DCPD where the current is pulsed on and held for a short period of time before being turned off again. When the pulsed signal is on the signal is recorded.

Crack length values measured using DCPD change non-linearly due to remaining ligaments/islands which tend to show a shorter crack length than the actual length. This is why DCPD is slightly less sensitive to short cracks and crack initiation compared to ACPD. In theory DCPD response should be linear which would make calibration easier. This is a simplification. Usually calibration is conducted using beach-mark testing and/or interrupted tests. Although DCPD is non-linear, crack length can be approximated using the Johnson formula Eq. (3.1).

3.4 Microscopy

Various microscopy techniques have been utilised in order to investigate and illustrate the microstructural morphology in all included Papers. Light optical microscopy (LOM) was used since it gives a good overview and sense of depth when looking at fracture surfaces. When more microstructural information is wanted regarding morphology such as deformation, orientation, oxidation, slip and twins scanning electron microscopy (SEM) was utilised. A Hitachi SU70 field emission gun SEM operating at 1.5–20 kV was used together with SEM techniques such as electron channelling contrast imaging (ECCI) [127, 128] and electron back scatter diffraction (EBSD) imaging. ECCI was used to achieve high quality, high contrast images of for example crack growth appearance and microstructural morphology,

illustrated in Fig. 3.3 (a). For more information regarding ECCI see references [133–136]. In papers IV and V orientational effects were subject of more interest. To determine for example crystallographic orientation, misorientation angles and coincident site lattice grain boundaries in materials EBSD mappings can be done resulting in orientation imaging maps where different colors correspond to crystallographic orientations presented in Fig. 3.3 (b).

Figure 3.3. (a) Electron channeling contrast image of Haynes 282 subjected to pure cyclic fatigue (sinusoidal wave) at room temperature, 0.05 Hz and $R = 0.1$, showing how channeling can be used to illustrate for example plastically deformed areas [113]. (b) An OIM of a press-forged Inconel 718 turbine disc [29]

CHAPTER 4

Summary

Fatigue crack growth testing has been performed on Inconel 718 at 550 °C in Paper I with the purpose of investigating the effect of overloads on dwell-fatigue crack growth. In Paper II fatigue crack growth testing was performed on Haynes 282 at room temperature as well as at 650 °C and 700 °C under different loading conditions. The purpose of Paper II was to clarify a number of controversial results previously reported for the same material, and to study the effects of dwell-fatigue and sustained loads on the crack growth behaviour. Paper III addresses anisotropy effects during dwell-fatigue caused by δ -phase orientation in forged Inconel 718. Paper IV deals with how grain size effects crack growth during dwell-fatigue in differently processed bar and cast, Inconel 718. Dwell-fatigue of AM SLM Hastelloy X at 700 °C was investigated in Paper V showing that AM materials could be considered as a new separate material group.

The following results and conclusions obtained from my research have been made:

- Both Inconel 718 and Haynes 282 showed an increase in crack growth rate when dwell-times were introduced at the maximum load (0 % overload) in the fatigue cycle. With the introduction of a dwell-time crack growth shifted from transgranular to intergranular. This was also shown in paper II as the result of a transition from cycle-dependent to time-dependent crack growth.
- When an overload was introduced in the load cycle at the beginning of the dwell-time in Inconel 718, the dwell-time effect was retarded. At an overload of 15 %, the dwell-time effect was essentially extinguished and the crack growth rate became the same as during pure fatigue loading without a dwell-time, accompanied by a transition from intergranular to transgranular cracking. High resolution scanning electron microscopy revealed that microscopic

crack growth in Inconel 718 at no or low overloads and high temperature took place as intergranular crack growth along grain boundaries due to oxidation and the creation of nanometric voids. Another growth mechanism was crack advance along δ -phase boundaries with subsequent severe oxidation of the δ -phase. Unbroken ligaments were observed behind the crack front. At such locations, the crack eventually grew along the substructures created by severe local plastic deformation and the high temperature. This substructure consisted of dislocation sub-cells or nano-sized grains created by embryos to dynamic recrystallisation.

- For Haynes 282 some controversial results have been previously published claiming frequency dependence at room temperature which was not observed in the dwell-fatigue tests conducted for paper II. It was shown to be stress ratio dependent at room temperature. At elevated temperatures the crack growth rate was shown to be purely time-dependent at stress intensity factors above $\sim 45 \text{ MPa}\sqrt{\text{m}}$. The leading mechanism controlling the time-dependent crack growth was suggested to be caused by stress-assisted grain boundary oxidation rather than dynamic embrittlement.
- The orientational effect (forming induced arrangement) of the δ -phase in Inconel 718 has been reported to significantly affect crack propagation behaviour. Crack growth rates were higher in samples with δ -phase discs oriented longitudinally perpendicular to the loading direction. The Nb-rich δ -phase discs are prone to oxidation which weakens the γ - δ interface. Delamination of the interface occurs when a tensile load is applied resulting in faster crack growth. Samples cut with the δ -phase discs oriented longitudinally parallel to the loading direction exhibited the slowest crack growth rates because the cracks then propagated in the γ - δ interface parallel to the loading direction. The crack path in the samples with the δ -phase discs oriented longitudinally parallel to the loading direction deviated from planar crack growth when reaching a δ -phase due to the weakening of the γ - δ interface. When the crack deflects, the driving force is retarded, lowering the crack propagation rates even if the crack propagates along the γ - δ interface.
- Grain enlarged bar exhibited slower time dependent crack propagation than the fine grained bar in Paper IV. This could be due to the difference in yield strength at 550°C , where the fine grained bar exhibited the highest yield strength of $\sim 1100 \text{ MPa}$, followed by grain enlarged bar $\sim 900 \text{ MPa}$ and lastly by the cast which should theoretically be in the range of $700\text{--}800 \text{ MPa}$. Depending on the yield strength the size of the plastic zone would change from a relatively small one in the fine grained bar, and become larger with an increasing grain size. The plastic zone is well known to retard crack propagation which can also lead to blunting and in some cases fully hinder crack propagation as was seen in the cast dwell-time sample. The retardation could also be due to the larger grains, resulting in a higher amplitude and longer wave length for the crack to propagate through, giving the same projected crack length. The fine grained bar samples exhibited more orthogonal

crack propagation compared to the loading direction giving a lower amplitude and a shorter wave length. The difference in cyclic crack propagation rates between the pure fatigue and dwell-time samples was approximately a factor ten for the fine grained and grain enlarged bar. Cast samples on the other hand showed none or minute differences between the pure fatigue and dwell-fatigue samples.

- Today we classify most materials as cast, forged, bar, or rolled. How should AM materials be classified. Should they be classified as one of the former ones or should AM materials be considered as a new material group? The effect of building direction in AM SLM Hastelloy X on dwell-fatigue crack propagation at 700 °C was investigated in Paper V showing that AM materials could be considered as a new separate material group, since the microstructural morphology is unique compared to materials manufactured by conventional means. The AM material is highly anisotropic as a result of layer by layer manufacturing technique. Elongated grains can be observed in the building direction. Crack propagation occurs mainly intergranularly during dwell-fatigue resulting in out of plane crack growth depending on building direction versus loading direction. This is why AM materials could be considered as a new material group.

4.1 Scientific contribution

The Swedish higher education act states *“The mandate of higher education institutions shall include third stream activities and the provision of information about their activities, as well as ensuring that benefit is derived from their research findings”*. Thus, it is my responsibility as a researcher to motivate how my research contributes not only the research community but also society in general. Fatigue crack mechanisms in superalloys used in industrial gas turbines have been investigated in this Ph.D. thesis. This is important since it gives a deeper understanding of the fracture behaviour of superalloys. Which is needed to improve the service life of industrial gas turbines and increase their efficiency. Ultimately resulting in a greener environment.

CHAPTER 5

Future work

Dwell-fatigue has been the main focus of the research presented in this thesis which has closed a few knowledge gaps regarding crack propagation. There are still vast amounts of testing and research that can be done in the field of fatigue, dwell-fatigue and creep in superalloys. Aspects requiring more attention are different cycles and loading conditions. Various corrosive atmospheres need to be investigated in order to achieve more real life like component testing conditions. Especially research on AM materials will require significant effort since the anisotropy yields challenges regarding both testing and methodology.

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CHAPTER 6

Results and included papers

The results obtained from my research leading up to this thesis are presented in the form of five appended scientific papers, which report the work completed as part of the project.

6.1 List of publications

- [I] **Mechanical properties of lattice truss structures made of a selective laser melted superalloy**
H. Brodin, J. Saarimäki
13th International Conference Fracture, Beijing, China (2013).

- [II] **Influence of overloads on dwell time fatigue crack growth in Inconel 718**
J. Saarimäki, J. Moverare, R. Eriksson, J. Johansson
Material Science & Engineering A, **612**, 398-405 (2014).

- [III] **Scatter in Dwell Time Cracking for a Ni-Based Superalloy in Combination With Overloads**
E. Storgårds, J. Saarimäki, K. Simonsson, S. Sjöström, D. Gustafsson, T. Månsson, J. Moverare
ASME. Journal of Engineering for Gas Turbines and Power, **138.1**, (2015).

- [IV] **Time and cycle-dependent crack propagation in Haynes 282**
J. Saarimäki, J. Moverare, M. Hörnqvist Colliander
Material Science & Engineering A, **658**, 463-471 (2016).

- [V] **Influence of superimposed vibrational load on dwell time crack growth in a Ni-based superalloy**
E. Storgårds, J. Saarimäki, K. Simonsson, S. Sjöström, T. Månsson, J. Moverare
International Journal of Fatigue, **87**, 301-310 (2016).

- [VI] **Characterization of Hastelloy X Produced by Laser Powder Bed Additive Manufacturing**
J. Saarimäki, M. Lundberg, J. J. Moverare, H. Brodin
World PM2016, Hamburg, Deutschland (2016).

- [VII] **Damage development in thin walled selective laser melted structures**
J. Saarimäki, J. Moverare, H. Brodin
Materials Science and Technology 2016 Proceedings, Salt Lake City, USA, 79-86 (2016).
- [VIII] **Microstructural characterization of as-manufactured and heat treated electron beam melted inconel 718**
D. Deng, J. Saarimäki, H. Söderberg, R. L. Peng, H. Brodin, J. Moverare
Materials Science and Technology 2016 Proceedings, Salt Lake City, USA, 105-112 (2016).
- [IX] **3D residual stresses in AM SLM Hastelloy XTM**
J. Saarimäki, M. Lundberg, J. J. Moverare, H. Brodin
Materials Research Proceedings, **2**, 73-78 (2016).
- [X] **Residual stresses in uniaxial cyclic loaded pearlitic lamellar graphite iron**
M. Lundberg, J. Saarimäki, R. L. Peng, J. J. Moverare
Materials Research Proceedings, **2**, 67-72 (2016).
- [XI] **Surface integrity and fatigue behaviour of electric discharged machined and milled austenitic stainless steel**
M. Lundberg, J. Saarimäki, J. J. Moverare, M. Calmunger
Materials Characterization, **124**, 215-222 (2017).
- [XII] **Anisotropy effects during dwell-fatigue caused by δ -phase orientation in forged Inconel 718**
J. Saarimäki, M. H. Colliander, J. J. Moverare
Material Science & Engineering A, **692**, 174-181 (2017).
- [XIII] **Effective X-ray elastic constant of cast iron**
M. Lundberg, J. Saarimäki, J. J. Moverare, R. L. Peng
Journal of Materials Science, 1-8 (2017).

[XIV] Grain size depending dwell-fatigue crack growth in Inconel 718

J. Saarimäki, M. Lundberg, J. J. Moverare
Advanced Engineering Materials, 1-7 (2018).

6.2 Summary of included papers

Paper I

Influence of overloads on dwell time fatigue crack growth in Inconel 718

Summary

Fatigue crack growth testing has been performed on Inconel 718 at 550 °C with the purpose of investigating the effect of overloads on the dwell-fatigue crack growth. There is a significant increase in crack growth rate when dwell-times are introduced at the maximum load (0 % overload) in the fatigue cycle, and when an overload is applied prior to the dwell-time, the crack growth rate decreases with increasing overload levels.

Author's contribution

I performed parts of the mechanical testing and have been the main contributor regarding the microstructure investigation, evaluation and responsible for the manuscript writing. The crack path analysis was done with the help of my co-author Ph.D. Robert Eriksson.

Paper II

Time- and cycle-dependent crack propagation in Haynes 282

Summary

Crack growth tests were performed on Haynes 282 at room temperature as well as at 650 °C and 700 °C under different loading conditions. The purpose was to clarify a number of controversial results reported previously regarding R -value and frequency effects at room temperature and to study the effects of dwell-fatigue and sustained loads on the crack growth behavior at high temperatures. The crack growth rate was purely time-dependent at elevated temperatures at stress intensity factors above $\sim 45 \text{ MPa}\sqrt{\text{m}}$. The time-dependent crack growth was suggested to be caused by stress-assisted grain boundary oxidation rather than dynamic embrittlement.

Author's contribution

I performed parts of the mechanical testing, instrumentation and have been the main contributor regarding the microstructure investigation, evaluation and responsible for the manuscript writing.

Paper III**Anisotropy effects during dwell-fatigue caused by δ -phase orientation in forged Inconel 718****Summary**

Crack propagation behavior was characterised using compact tension (CT) samples cut in different orientations from a real Inconel 718 turbine disc forging. To investigate the influence of plane strain and plane stress condition on the crack propagation rates. The samples were subjected to dwell-fatigue tests at 550 °C with 90 s or 2160 s dwell-times at maximum load. The forged alloy exhibits strong anisotropic behaviour caused by the non-random δ -phase orientation. When δ -phases were oriented perpendicular compared to parallel to the loading direction, the crack growth rates were approximately ten times faster. Crack growth occurred preferably in the interface between the γ -matrix and the δ -phase.

Author's contribution

I performed parts of the mechanical testing and have been the main contributor regarding instrumentation, microstructure investigation, evaluation and manuscript writing.

Paper IV**Grain size depending dwell-fatigue crack growth in Inconel 718****Summary**

Dwell-fatigue crack propagation behavior in Inconel 718 has been tested at 550 °C using Kb test samples with 2160 s dwell-times at maximum load and pure fatigue tests. The dwell-time effect has been studied for differently processed Inconel 718 namely fine grained bar, grain enlarged bar, and cast material. Time dependent crack propagation rates were strongly affected by grain size. Propagation rates increase with decreasing grain size, whereas crack tip blunting increased with increasing grain size.

Author's contribution

I performed parts of the mechanical testing and have been the main contributor regarding instrumentation, microstructure investigation, evaluation and manuscript writing.

Paper V

Dwell-fatigue crack propagation in additive manufactured Hastelloy X

Summary

Dwell-fatigue crack propagation behavior at 700 °C of as built and heat treated AM SLM Hastelloy X has been investigated. Significant differences in crack propagation rates were observed between the different material conditions. All material condition exhibited intergranular crack growth. Depending on building direction the cracks grew either in plane or out of plane.

Author's contribution

I performed parts of the mechanical testing and have been the main contributor regarding instrumentation, microstructure investigation, evaluation and manuscript writing.

Papers

The papers associated with this thesis have been removed for copyright reasons. For more details about these see:

<http://urn.kb.se/resolve?urn=urn:nbn:se:liu:diva-144397>

Final conclusion:
That's the way the cookie crumbles!