Low Cycle Fatigue and Thermo-Mechanical Fatigue of Uncoated and Coated Nickel-Base Superalloys

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Low Cycle Fatigue and Thermo-Mechanical Fatigue of Uncoated and Coated Nickel-Base Superalloys

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Abstract

High strength nickel-base superalloys have been used in turbine blades for many years because of their superior performance at high temperatures. In such environments superalloys have limited oxidation and corrosion resistance and to solve this problem, protective coatings are deposited on the surface. The positive effect of coatings is based on protecting the surface zone in contact with hot gas atmosphere with a thermodynamically stable oxide layer that acts as a diffusion barrier. During service life, mechanical properties of metallic coatings can be changed due to the significant interdiffusion between substrate and coating. There are also other degradation mechanisms that affect nickel-base superalloys such as low cycle fatigue, thermo-mechanical fatigue and creep.

The focus of this work is on a study of the low cycle fatigue and thermo-mechanical fatigue behaviour of a polycrystalline, IN792, and two single crystal nickel-base superalloys, CMSX-4 and SCB, coated with four different coatings, an overlay coating AMDRY997, a platinum aluminide modified diffusion coating RT22 and two innovative coatings with a NiW interdiffusion barrier called IC1 and IC3. An LCF and TMF device was designed and set-up to simulate the service loading of turbine blades and vanes. The LCF tests were run at 500 °C and 900 °C while the TMF tests were run between 250 °C and 900 °C. To simulate service life, some coated specimens were long-term aged at 1050 °C for 2000 h before the tests.

The main conclusions are that the presence of the coatings is, in most cases, detrimental to low cycle fatigue lives of the superalloys at 500 °C while the coatings do improve the low cycle fatigue lives of the superalloys at 900 °C. Under thermo-mechanical fatigue loading conditions, the coatings have negative effect on the lifetime of IN792. On single crystals, they are found to improve thermo-mechanical fatigue life of the superalloys, especially at lower strains. The tests also indicate that long-term aging influences the fatigue life of the coated superalloys by oxidation and diffusion mechanisms when compared to the unaged specimens. The long-term aged specimens exhibit longer life in some cases and shorter life during other test conditions. Fatigue cracks were in most cases initiated at the surface of the coatings, growing both intergranularly and transgranularly perpendicular to the load axis.

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Contents

1 Introduction

According to a rapport from Elforsk [1], "gas turbines are rapidly moving towards higher output" leading to an increase of turbine inflow temperatures and stresses. This harsh environment are further aggravated by oxidation and corrosion factors that limit the life of the hot gas turbine components such as combustors, turbine blades, vanes and disks. Today, turbine parts are manufactured from nickel-base superalloys which provide a sufficiently high level of mechanical properties at elevated temperatures [2]. Above this temperature, superalloys must be protected by coatings in order to prevent oxidation and corrosion attack. A coating used for deposition on superalloys at high temperatures is defined as a surface layer of material, either ceramic, metallic or a combination of those. Forming a thermodynamically stable oxide layer with elements like aluminium and chromium on the surface of the superalloy, coatings are acting as a diffusion barrier to slow down the reaction between the substrate material and the aggressive environment [3]. There are other sources of loads imposed on turbine blades and vanes; centrifugal forces due to rotation, creep, low cycle fatigue and time varying thermo-mechanical fatigue due to sequential engine start-ups and shutdowns.

Ideally coatings should not have any effect on mechanical properties of the superalloy but in practise the coatings can modify those properties in positive or negative way [4]. Except oxidation and corrosion attack, the life of the coatings can be reduced by cracking caused by thermal and mechanical cycling due to poor mechanical behaviour of the coatings. For example, platinum modified coatings have low ductility while other coatings suffer from brittle phase formation during engine operation, which causes failure of the coatings. In such a case the coatings loose their protective function and crack propagation leads to failure of the substrate.

Low cycle fatigue (LCF) is isothermal fatigue where the strain amplitude during fatigue cycling exceeds the yield strength and causes inelastic deformations so that the material suffers from damage in a short number of cycles [5]. Most turbine blades have a variety of features like holes, interior passages, curves and notches that raise the local stress level to the point where plastic strains occur. Isothermal LCF test has been used to determine the performance of materials used in components as turbine blades and disks [6] and data from such tests can be an important consideration in the design of materials for turbine components. Results from an LCF test may be used in the formulation of empirical relationships between cyclic variables like stress, total strain, plastic strain and fatigue life (number of cycles to failure). Thermo-mechanical fatigue (TMF) is cyclic damage induced under thermal and mechanical loading. Like the LCF test, the purpose of TMF tests is to simulate behaviour of material in a critical location. Strain and temperature variations during TMF are classified according to the phase relation between mechanical strain and temperature, for example, in-phase TMF means that peak strain coincides with maximum temperature while out-of-phase TMF means maximum strain at minimum temperature [7] that is investigated in this study.

This research deals with the study of LCF and TMF behaviour of three coated and plus coated long-term aged nickel-base superalloys. As a comparison, uncoated specimens made of the same batch as the superalloys are also tested and presented. Four coatings were deposited on the superalloys; an overlay NiCoCrAlYTa coating (AMDRY997), a platinum modified diffusion aluminide RT22 coating and two innovative coatings with a NiW diffusion barrier in the interface called IC1 and IC3 that were developed during this project.

2 Aims

This research has been a part of a European project called Advanced Long Life Blade Coating Systems-ALLBATROS. The main objectives of the Allbatros project were to:

- increase efficiency, reliability and maintainability of industrial gas turbine blades and vanes by developing coatings with a life of 50 000 h even when using fuels with high pollutant levels,
- characterize advanced existing coatings to assess lifetime and performance of coatings and coated materials,
- provide material coating data, and
- increase understanding of the degradation and fracture behaviour of coated superalloys under low cycle fatigue and thermo-mechanical fatigue test conditions.

One of the limiting life factors in a coating is cracking caused by fatigue due to poor mechanical behaviour of the coating. Important factors are damage due to incompatibility with the substrate, low ductility, high ductile to brittle transition temperature and brittle phase transformation during engine operation. Fatigue failure often can come earlier than damage due to oxidation and the coating looses the protection function and subsequent crack propagation leads to accelerated corrosion of the substrate. The aims of this project have been to:

- develop and set-up low cycle fatigue and thermo-mechanical fatigue test equipment,
- perform low cycle fatigue and thermo-mechanical fatigue tests on three uncoated and coated nickel-base superalloys with test conditions as closely as possible service conditions,
- generate and provide low cycle fatigue and thermo-mechanical fatigue data of uncoated and coated polycrystalline and single crystal nickel-base superalloys,
- emphasize influence of temperature on low cycle fatigue life,
- study the effect of long-term aging on microstructure, low cycle fatigue and thermo-mechanical fatigue of the coated superalloys,
- characterize and define microstructural changes caused by low cycle fatigue and thermo-mechanical fatigue test conditions, and
- characterize cyclic stress behaviour, cyclic hardening, cyclic softening and fatigue life under low cycle fatigue and thermo-mechanical fatigue loading conditions.

Low cycle fatigue and thermo-mechanical fatigue tests have been performed on 148 and 73 specimens, respectively, including uncoated, coated and long-term aged specimens. Investigation of fracture properties and microstructural characterization of fatigue damage are also presented. The scope of the next chapter called **Background** is to give more information about superalloys and coatings and to review low cycle fatigue, thermo-mechanical fatigue behaviour and main failure mechanisms of coated superalloys published in the literature.

3 Background

"Superalloys as a class constitute the currently reigning aristocrats of the metallurgical world. They are the alloys which have made jet flight possible, and they show what can be achieved by drawing together and exploiting all the resources of modern physical and process metallurgy in the pursuit of a very challenging objective", from [8].

In the following chapters the nickel-base superalloys and coatings used for deposition on the superalloys are presented from microstructural point of view. Then, low cycle fatigue (LCF) and thermo-mechanical fatigue behaviour (TMF) of some uncoated and coated superalloys found in the literature is given.

3.1 Gas turbines

A gas turbine extracts energy from a flow of hot gas produced by combustion of gas or fuel in a stream of compressed air. It has an upstream air compressor mechanically coupled to a downstream turbine and a combustion chamber in between, Figure 3.1. Gas turbine may also refer to just the turbine element. Energy is released when compressed air is mixed with fuel and ignited in the combustor. The resulting gases are directed over the turbine's blades, spinning the turbine, and, mechanically, powering the compressor. Finally, the gases are passed through a nozzle, generating additional thrust by accelerating the hot exhaust gases by expansion back to atmospheric pressure [9]. Energy is extracted in the form of shaft power, compressed air and thrust, in any combination, and used to power aircraft, trains, ships, electrical generators, and even tanks. When

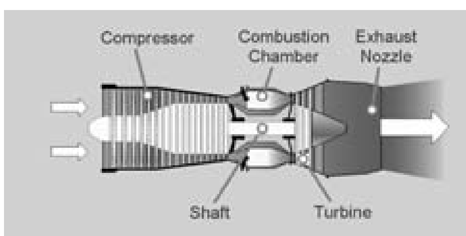

Figure 3.1: Illustration of the basic components of a simple gas turbine engine [10].

designing a gas turbine engine, special materials providing strength, environmental resistance and temperature stability are used.

3.2 Nickel-base superalloys

Nickel base superalloys have been in use since 1940 [2] primarily in aero and land turbine blades, disks and vanes because of their good mechanical properties such as long-time strength and toughness at high temperatures. The operating temperature of the turbine components ranges from 150 °C to almost 1500 °C [3]. These performances depend on the microstructure of the alloys, additional alloying elements, type of heat treatment applied and production methods. The superalloys can have an iron, cobalt or nickel base and a combination of alloying elements such as chromium, titanium, aluminium, tantalum, etc. added to improve certain properties. Nickel-base superalloys are the most widely used superalloys and are studied here. They have as main element nickel (Ni) and have a significant amount of chromium (Cr) that gives them high corrosion resistance.

Nickel-base superalloys have a γ matrix that has a face-centred cubic (FCC) crystal structure [11] containing a dispersion of ordered intermetallic precipitates of the type Ni_3Al called γ'. Other elements are added to, for example, strengthen the matrix, increase oxidation resistance and increase fraction of γ' [12]. Strength can be provided by molybdenum, tantalum, tungsten or rhenium, oxidation resistance by chromium, aluminium or nickel, phase stability by nickel, increased volume fraction of γ' by cobalt and reinforcement of grain boundaries by zirconium, boron or hafnium that form carbide precipitations. As matrix hardeners not associated with γ', heavy metals such as molybdenum and tungsten, together with niobium and tantalum are added to form topologically close packed (TCP) precipitates as σ, μ, δ, Laves or R-phases.

To summarize, the microstructure of nickel-base superalloys consists mainly of the following phases:

- γ-FCC matrix
- Coherent intermetallic precipitates, γ'-Ni_3Al
- Topologically close-packed (TCP) phases
- Carbides
- Borides

The superalloys are produced by three processing routes, casting, powder metallurgy and wrought processing, for more information about the processing of superalloys see [13].

For this study a conventional cast polycrystalline nickel-base superalloy IN792 and two single crystal nickel-base superalloys, CMSX-4 and SCB were chosen and examined.

Table 3.1: Nominal chemical composition of IN792 in wt.%

Alloy	Co	Mo	W	Ta	Re	Al	Ti	Hf
IN792	9.0	1.9	3.93	4.175	–	3.375	3.975	–
Alloy	Cr	C	Si	Mn	Fe	B	Zr	Ni
IN792	12.5	0.08	0.2	0.15	0.5	0.015	0.03	Bal.

Table 3.2: Mechanical properties of IN792 at different temperatures

Temperature	σ_{uts} [MPa]	$\sigma_{0.2}$ [MPa]	A [%]	RA [%]
21 °C	1170	1060	4	4
650 °C	861	827	4	4
790 °C	545	–	5	–
980 °C	180	–	5	–

3.2.1 Polycrystalline IN792

IN792 is a polycrystalline nickel-base superalloy with γ-FCC Ni matrix containing hard precipitates of γ' and several alloying elements such as tungsten, chromium, cobalt, tantalum, titanium and aluminium and grain boundary strengtheners boron and zirconium. The nominal composition of IN792 is presented in Table 3.1. The volume of the soft matrix contains up to 50 % of γ'. The resistance of the material to plastic deformation is due to interaction between dislocations and γ' phase [13] and [14].

IN792 is fabricated by conventional casting. During the solidification, the material is subjected to crack formation and it often suffers of poor castability. The blanks made of IN792 were received from Nuovo Pignone, Florence, Italy with the mechanical properties at different temperatures presented in Table 3.2. The data at the room temperature comes from [13] while the other properties are from Nuovo Pignone's material specification. The blanks of IN792 were not hot isostatically pressed. The as-received billets were subject to a solution treatment at 1120 °C for 2 hours, followed by fast cooling with argon down to 500 °C and then cooling to room temperature. After the heat treatment, the alloy was first aged at (845 ± 10) °C for 24 hours, followed by argon fast cooling to room temperature, then second aging treatment was performed at (760 ± 10) °C for 16 hours, followed by forced argon fast cooling to room temperature.

Table 3.3: Nominal chemical composition of CMSX-4 in wt.%

Alloy	Co	Mo	W	Ta	Re	Al	T	Hf	Cr	Ni
CMSX-4	9.0	0.6	6.0	6.5	3.0	5.6	1.0	0.1	6.5	Bal.

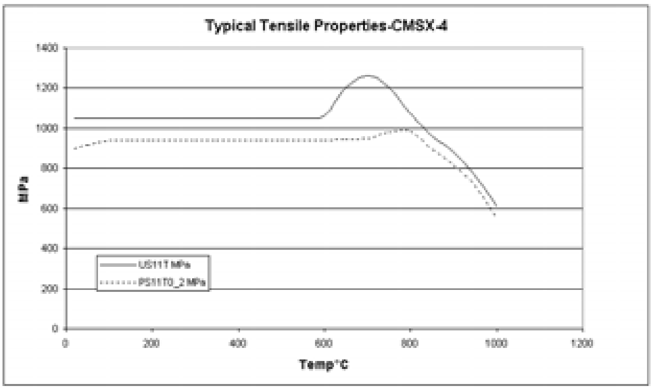

Figure 3.2: Tensile properties of CMSX-4 at different temperatures (by courtesy of Mick Whitehurst, Siemens, Lincoln, UK). US11T is ultimate strength in MPa, PS11T0_2 is yield strength in MPa.

3.2.2 Single crystal CMSX-4

The single crystal CMSX-4 is the second-generation rhenium-containing superalloy developed by Cannon-Muskegon Corporation [13]. The alloy is widely used because of its good high temperature creep resistance. The bars were received from Siemens, Lincoln, UK. For chemical composition of CMSX-4 see Table 3.3. The crystallographic orientation of the bars was chosen so that one of the $\langle 001 \rangle$ directions were aligned with a maximum deviation of 13° to the specimen and load axis. The addition of rhenium improves the strength of the alloy by acting as a powerful obstacle against dislocation movement in the γ matrix. Its stress-rupture temperature endurance is better than the first generation CMSX alloys [15] such as CMSX-2 and CMSX-3. The level of aluminium is increased in CMSX-4 while the chromium level is decreased, which together with rhenium gives the alloy better oxidation resistance and decreased coarsening of γ'. The tensile properties of CMSX-4 are presented in Figure 3.2. For the changing of tensile elongation with temperature see Figure 3.3 and for the effect of temperature on dynamic modulus of the alloy see Figure 3.4.

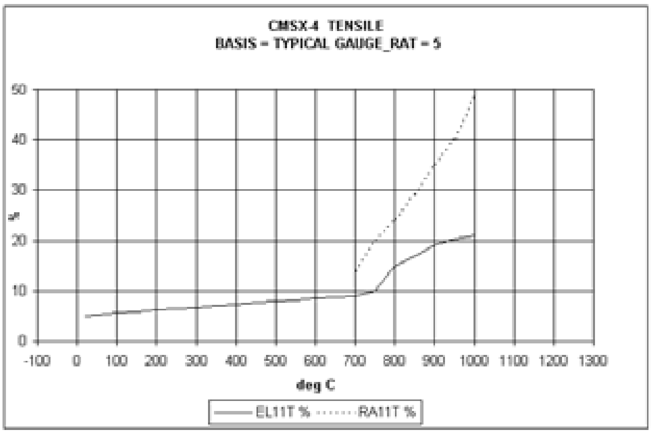

Figure 3.3: Tensile elongation of CMSX-4 at different temperatures for CMSX-4 (by courtesy of Mick Whitehurst, Siemens, Lincoln, UK). EL11T is elongation in % and RA11T is reduction of area in %.

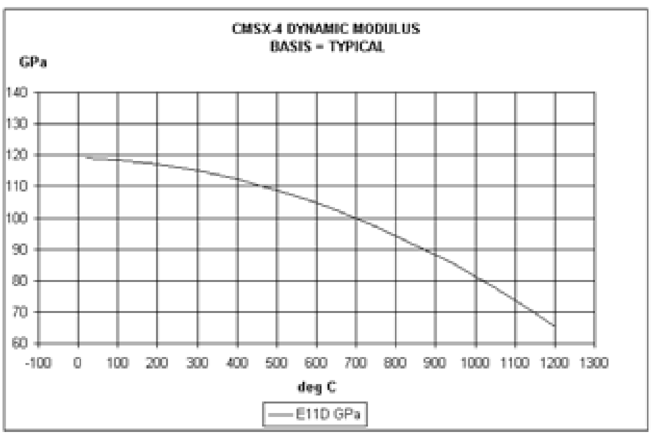

Figure 3.4: Dynamic modulus of elasticity for CMSX-4 at different temperatures (by courtesy of Mick Whitehurst, Siemens, Lincoln, UK). E11D is Young's modulus in GPa.

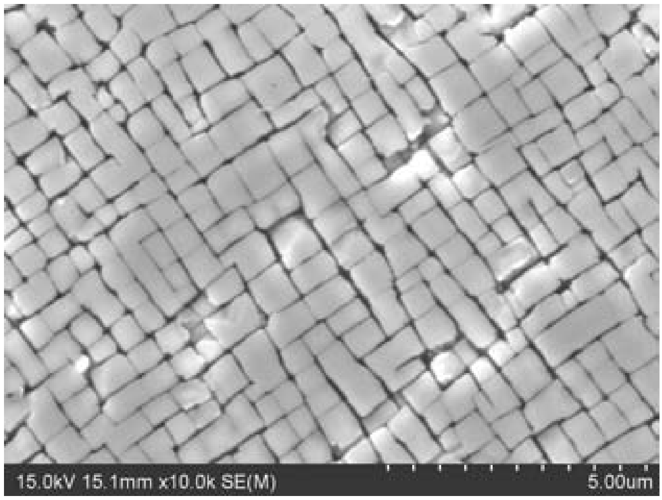

Figure 3.5: SEM micrograph showing microstructure of CMSX-4 after aging treatment at 1140 °C/4 h with uniformly distributed cuboidal γ' precipitates in γ matrix

The following heat treatments were applied to the bars, heating up to (1037 ± 15) °C and held for 20 minutes, ramped to (1256 ± 6) °C at 4 °C/min, ramped to (1308 ± 2) °C at 0.5 °C/min and held for 60 minutes, then gas fan quenched at 60 °C/min minimum to below 1150 °C in argon and cooled to room temperature with gas fan; temperature was again raised under vacuum or high purity argon to 1140 °C and the bars were held two hours and then cooled by air. The third heating cycle was obtained under vacuum at 870 °C, the bars were held for 20 hours and then air cooled to room temperature. A typical microstructure of a CMSX-4 alloy after the heat treatment is presented in Figure 3.5. The fully heat treated CMSX-4 contains about 70 % of γ' phase.

3.2.3 Single crystal SCB

SCB single crystal with higher chromium level than CMSX-4 has been developed by Onera, France, in the frame work of an European research program for turbine blade applications [16] with a hot corrosion resistance comparable to IN792. The nominal chemical composition of the material is given in Table 3.4. The bars for the mechanical tests were received by Onera with their axis parallel to $\langle 001 \rangle$ direction and a maximum deviation of 13°. Heat treatment of SCB involves solution heat treatment in the temperature range of 1250-1260°C for good homogenization,

Table 3.4: Nominal chemical composition of SCB in wt.%

Alloy	Co	Mo	W	Ta	Al	Ti	Cr	C	Ni
SCB	4.95	0.99	3.93	2.03	4.02	4.58	11.8	0.019	Bal.

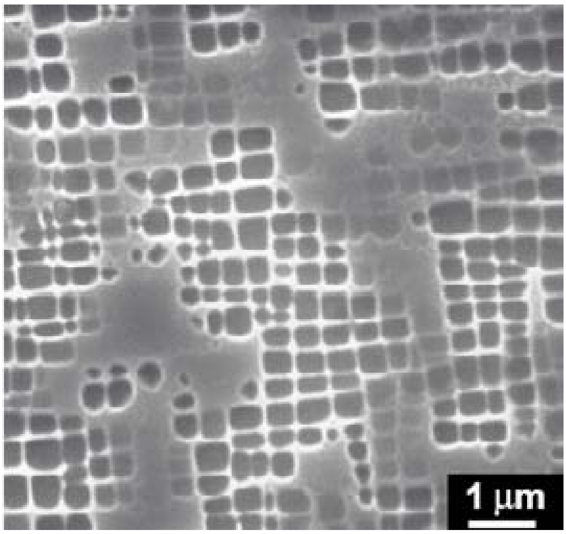

Figure 3.6: Cubical γ' precipitates in SCB after fully heat treatments at 1270 °C/4 h and 1100 °C/4 h, [16]

then first aging under 1100°C/4h/AC and second aging under 850°C/24h/AC. The fully heat treated SCB alloy contains about 57 % of γ' phase dispersed as cubical precipitates with sizes within the range 200 to 500 nm, see Figure 3.6. Tensile tests on SCB have been carried out at Onera. For mechanical properties of the alloy see Figure 3.7, 3.8 and 3.9 where the test results are compared with two other single crystals. The creep strength has shown to be better than for IN792 alloy.

3.3 Coatings

The superalloys alone have limited oxidation and corrosion resistance at high temperatures. To solve this problem nickel-base superalloys are protected by oxidation and corrosion resistant coatings. Oxidation is the primary reaction between the coating or base alloy, if no coatings are deposited on the alloy, with

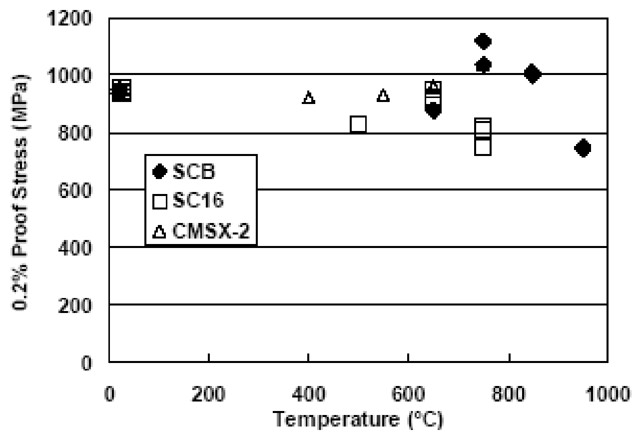

Figure 3.7: Yield strength of SCB at different temperatures, [16].

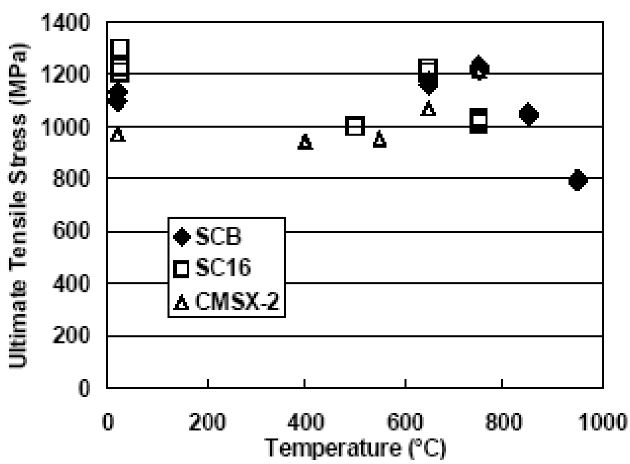

Figure 3.8: Ultimate tensile strength of SCB at different temperatures, [16].

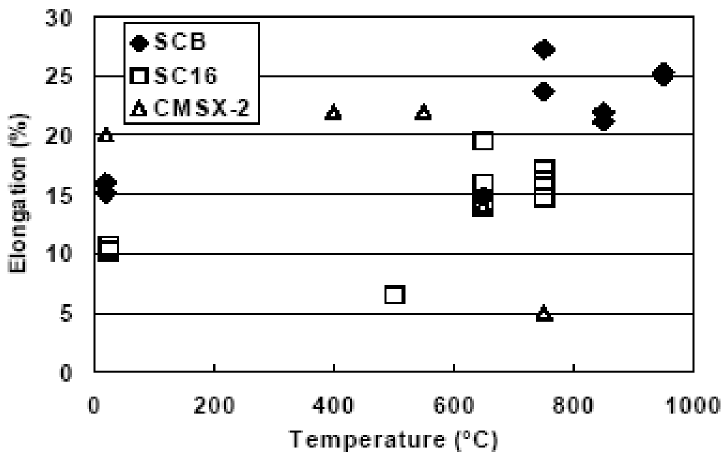

Figure 3.9: Elongation of SCB at different temperatures, [16].

the oxidants present in the hot gases. Hot corrosion occurs from surface reactions with salts deposited from the vapour phase [17]. The positive effect of coatings is based on protecting the surface zone in contact with hot gas atmosphere with elements like aluminium, chromium, which form a thermodynamically stable oxide layer. The coatings act as diffusion barriers to slow down the reaction between the substrate material and the aggressive environment [3]. In addition to oxidation and hot corrosion, coatings will change with time by interdiffusion with the substrate alloy, as they are not in thermodynamic equilibrium with the substrate alloy. This is of concern, not only because it may modify the mechanical properties of the substrate, but also because loss of Al to the substrate will reduce the oxidation life of the coating [18]. When the Al content of the coating is too low, other oxides than Al_2O_3 may form the nature of which depends on the composition of the coating, often brittle spinel type, which can result in failure and therefore is to be avoided.

Typical coatings for high-temperature applications are overlay coatings, aluminide and platinum aluminide diffusion coatings. The most widely used types of coatings are aluminides (NiAl or Ni_2Al_3) and MCrAlY where M is Ni and/or Co element. The diffusion coatings are obtained through surface enrichment through diffusion reactions, that includes pack cementation, slurry cementation and metallizing. The overlay coatings are applied by plasma spray and physical vapour

Table 3.5: Chemical composition of the overlay coatings in wt.%

Coating	Ni	Co	Cr	Al	Y	Ta	Pt
AMDRY997	44.0	23.0	20.0	8.0	0.6	4.0	–
AMDRY995	32.0	38.5	21.0	8.0	0.5	–	–

deposition (PVD) techniques [19].

3.3.1 Overlay coatings

The composition of overlay coatings are independent from the substrate alloy as opposite to diffusion coatings. A typical MCrAlY overlay coating is NiCoCrAlYTa called AMDRY997 used in this study as a reference coating. The M of MCrAlY stands here for a combination of both nickel or cobalt. The presence of a significant amount of chromium give these coatings excellent corrosion resistance combined with good oxidation resistance. Chromium also provides hot corrosion resistance, but the amount that can be added is limited by the formation of Cr-rich phases in the coating. Aluminium content for an MCrAlY is typically around 10 to 12 %, in AMDRY997 coating it is only 8 %. Since oxidation life is essentially controlled by the availability of aluminium, it would be tempting to increases the aluminium content but this can lead to a significant reduction of ductility. Yttrium enhances adherence of the oxide layer [20] while additions of tantalum enhances the hot corrosion resistance. For the composition of the coatings see Table 3.5. Other MCrAlY coatings can have an addition of hafnium that plays a similar role as yttrium. Adding silicon significantly improves cyclic oxidation resistance by forming SiO_2 but decreases the melting point of the coating. Additions of rhenium have been shown to improve isothermal or cyclic oxidation resistance and thermal cycle fatigue [21]. The coating can be deposited by different methods such as air plasma spray (APS), vacuum plasma spray (VPS) and low pressure plasma spray (LPPS). Deposition is followed by a high-temperature heat treatments in vacuum or argon gas to allow interdiffusion and therefore improve the adhesion of the coating [22].

MCrAlY coatings typically exhibit a two-phase microstructure consisting of B2 type β-NiAl intermetallic and γ phases. AMDRY997 has also some γ' precipitated in the γ phase. The presence of γ phase increases the ductility of the coating thereby improving thermal fatigue resistance. As for β-NiAl coatings, high temperature exposure results in depletion of the Al both by formation of aluminium oxide on the top surface of the coating and by diffusion into the substrate. As the amount of Al decreases, the β phase tends to transform to other phases, γ' and γ.

3.3.2 Diffusion coatings

Diffusion aluminide coatings are based on the intermetallic compound β-NiAl. Pack cementation is the most widely used process to form NiAl as it is inexpensive and well adapted to coating of small parts. Pack cementation falls in the category of chemical vapour deposition processes where the components to be coated are immersed in a powder mixture containing Al_2O_3 and aluminium particles. About 1 to 2 wt% of ammonium halide activators are added to this pack. The whole batch is then heated to temperatures around 800 to 1000 °C in argon. At these temperatures, aluminium halides are formed, which then diffuse through the pack and react with the substrate to deposit Al metal. The activity of Al at the surface of the substrate defines two categories of deposition methods: low and high activity, referred to as outward and inward diffusion, respectively [23].

In cements with low aluminium contents (low activity/outward diffusion), the formation of the coating occurs mainly by Ni diffusion and results in the direct formation of a nickel rich NiAl layer. The process requires high temperature (1000 to 1200 °C). In cements with high aluminium contents (high activity/inward diffusion), the coating forms mainly by inward diffusion of aluminium and results in formation of Ni_2Al_3 and possibly β-NiAl. Aluminizing temperatures can in this case be lower (700 to 950 °C). There can, in this way, be a high Al concentration gradient in the coating, and also significant interdiffusion with the substrate during service. For these reasons, a diffusion heat-treatment is generally given at 1050 to 1100 °C to obtain a fully β-NiAl layer. The structure and composition of the coating depends on the substrate but aluminide coatings lack ductility below 750 °C [24]. One of the major problems with aluminide coatings is revealed during thermo-mechanical fatigue, as cyclic strains induced by temperature gradients in the blades can lead to thermal fatigue cracks.

In low activity/outward diffusion coatings, the alloying elements present in the substrate will also tend to diffuse into the coating layer. In high activity/inward diffusion coatings, they enter into solution in the compound layer to form precipitates during the treatment that can change the mechanical properties of the coated alloy. A typical microstructure of low activity aluminide coating is illustrated in Figure 3.10. The external zone is typically Al rich β-NiAl phase, while the internal zone is Ni rich. Platinum was introduced as a diffusion barrier into aluminide coatings by electroplating the base alloy with Pt. The layer of Pt deposited is typically of 5 to 10 µm thick for platinum modified RT22 [26], which was chosen as the reference coating in this study. It was found that Pt additions enhanced Al diffusion [27] when deposited on CMSX-4 and also formed TCP (topologically close-packed) phases with some elements of the substrate such as Re, W, Mo and Cr. The microstructure of RT22 can be characterized as being two phase, based on an outer zone of $PtAl_2$ embedded in NiAl or single phase, based on an outer

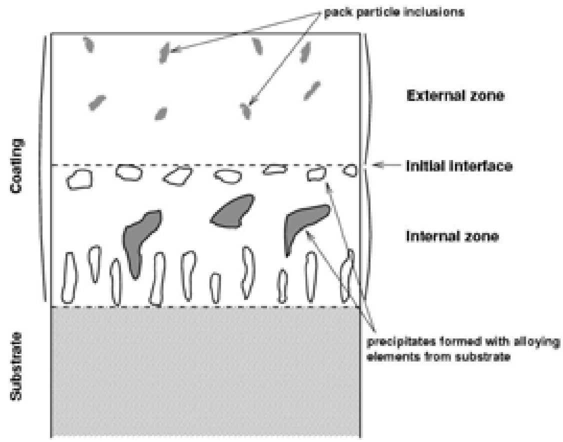

Figure 3.10: Typical microstructure of an aluminide coating, [25].

Table 3.6: Chemical composition of the diffusion coating RT22 in wt%

Coating	Ni	Co	Cr	Al	Y	Ta	Pt
RT22	35.0	4.8	1.8	42.8	–	–	15.7

zone of platinum enriched NiAl. For composition of RT22 see Table 3.6.

3.3.3 Innovative coatings

The innovative coatings, IC1 and IC3, with a diffusion barrier between the coating and substrate were developed at Onera, France, to keep a high aluminium concentration and activity in the coating. Aluminium forms a scale on the surface that protects the base material against the aggressive environment. During long term exposure of the coating under service, loss of aluminium occurs by interdiffusion into the substrate causing spallation of the protective oxide layer [28]. Addition of chromium improves hot corrosion resistance of the coatings but this requires a high chromium concentration. The composition of the coating is confidential and is not presented in this paper.

The coating IC1 consists of a NiW electrolytic layer that acts as a diffusion barrier and prevents diffusion of aluminium into the substrate. A VPS AMDRY997 (NiCoCrAlYTa) is an intermediate layer that acts as a chromium reservoir. Platinum modified nickel aluminide is applied as a top layer by electrolytic deposition. The total thickness of the coating varies from 200 to 250 µm. The NiW deposit is about 11 µm thick. AMDRY997 is a mixture of γ and β phases with some Ta precipitates. The outer layer is a β-(Ni,Pt)Al phase. The amount of chromium in the outer layer is about 8 at.% and the underlying layer is a chromium reservoir with 24 at.% of chromium [28]. IC3 is another three layer innovative coating with an NiW diffusion barrier between the coating and substrate as a bottom layer. The top layer is a CN91 diffusion coating consisting of a single Pt/Al phase. The middle layer is an LCO22-CoNiCrAlY or AMDRY995 coating with the chemical composition presented in Table 3.5. The typical thickness of the coating was from 120 to 150 µm.

3.4 Low cycle fatigue

The principles behind low cycle fatigue tests and mechanisms will be presented in the following chapters. Finally an overview of the LCF lives of coated nickel-base superalloys found in the literature will be given at the end of the chapter.

3.4.1 Principles behind LCF testing

There are commonly three recognized forms of fatigue, high cycle fatigue (HCF), low cycle fatigue (LCF) and thermo-mechanical fatigue (TMF) [29]. HCF is usually associated with low stress levels and low amplitude high frequency elastic strains. LCF is isothermal fatigue where the strain range during fatigue cycling exceeds elastic strain range and causes inelastic deformations so that the material exhibits a short number of cycles to failure [5]. TMF describes fatigue under simultaneous changes in temperature and mechanical strain [30]. Large temperature changes during TMF result in significant thermal expansion and contraction that are also reinforced by changes in mechanical strains associated with centrifugal loads as engine speed changes.

Isothermal LCF test has been used to determine the performance of materials applied in components as turbine blades and disks [6]. Results of a LCF test may be used in the formulation of empirical relationships between cyclic variables of stress, total strain, plastic strain and fatigue life. They are normally presented as curves of cyclic stress or strain versus life or cyclic stress versus plastic strain and examination of such curves and comparison with monotonic stress stress-strain curves gives useful information regarding cyclic stability of a material.

LCF life lies usually in the range between 10^2 to 10^5 cycles [31]. The response of a material subjected to cyclic loading is presented in the form of hysteresis loops, an example is presented in Figure 3.11. The total width of the loop is the total strain range $\Delta\varepsilon_{\mathrm{t}}$ while the total height of the loop is the total stress range $\Delta\sigma_{\mathrm{t}}$. The total strain is the sum of the elastic strain and the plastic strain, see

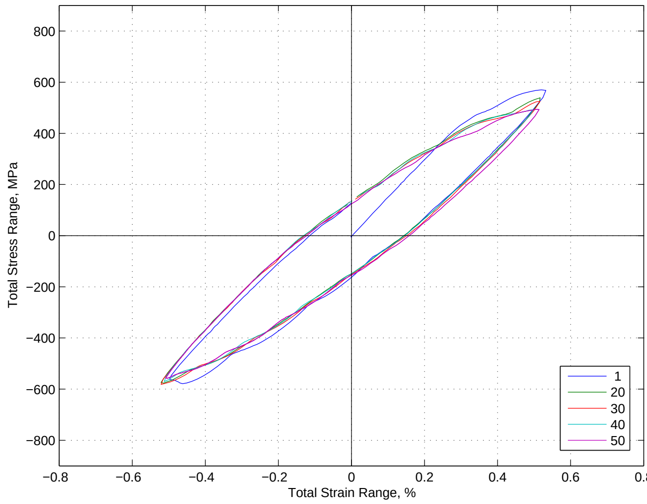

Figure 3.11: An example of a hysteresis loop obtained for IN792 tested at 900 °C and a strain range of 1 %.

eq. (3.1), [32]:

$$\Delta\varepsilon_{\mathrm{t}} = \Delta\varepsilon_{\mathrm{e}} + \Delta\varepsilon_{\mathrm{p}} \tag{3.1}$$

Based on the Basquin and Coffin-Manson relationship, the total strain range can be expressed as, [33]:

$$\frac{\Delta\varepsilon_{\mathrm{t}}}{2} = \frac{\sigma'_{\mathrm{f}}}{E}(2N_{\mathrm{f}})^{b} + \varepsilon'_{\mathrm{f}}(2N_{\mathrm{f}})^{c} \tag{3.2}$$

where σ'_{f} is the cyclic strength coefficient, b is the cyclic strength exponent, E is Young's modulus, $2N_{\mathrm{f}}$ is the number of reversals to failure, $\varepsilon'_{\mathrm{f}}$ is fatigue ductility coefficient and c is fatigue ductility exponent. The strains in eq. (3.2) are expressed as strain amplitudes that are a half of the strain ranges. The relation between strain and numbers of reversals to failure can be presented in a log-log plot as shown in Figure 3.12, [34].

3.4.2 Mechanisms of fatigue crack initiation and growth

The fatigue life of a material can be divided in three stages:

- crack initiation,
- crack propagation, and
- failure. This stage happens very quickly.

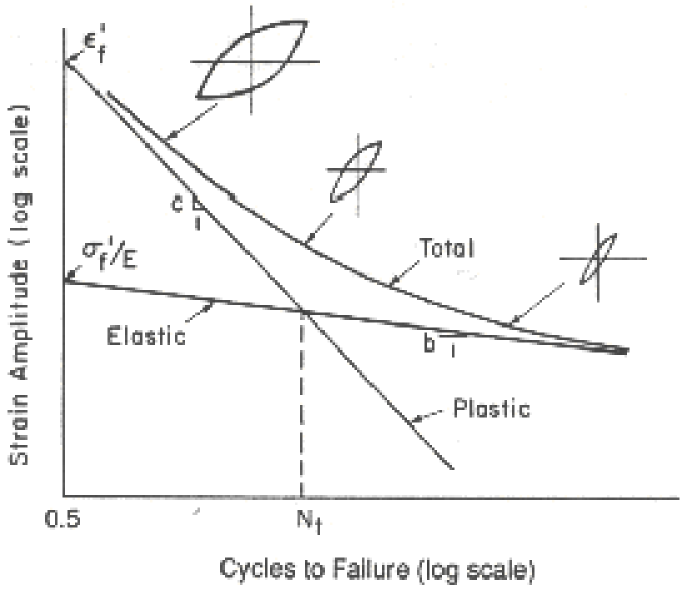

Figure 3.12: Strain-life curves showing total, elastic and plastic strain versus reversals failure.

Dislocations play a major role in the fatigue crack initiation phase. It has been observed in laboratory testing by clean FCC materials that after a large number of loading cycles, dislocations pile up and form structures called persistent slip bands (PSB). Persistent slip bands are areas that rise above (extrusion) or fall below (intrusion) the surface of the component due to movement of material along slip planes. This leaves tiny steps in the surface that serve as stress risers where fatigue cracks can initiate. An example of a crack initiated at the edge of a PSB is presented in Figure 3.13.

3.4.3 Low cycle fatigue

The deformation microstructure of a single crystal superalloy AM1 under LCF test conditions was investigated at 950 °C [35]. Two types of behaviour were found depending on number of cycles and total strain amplitude. The first one presents

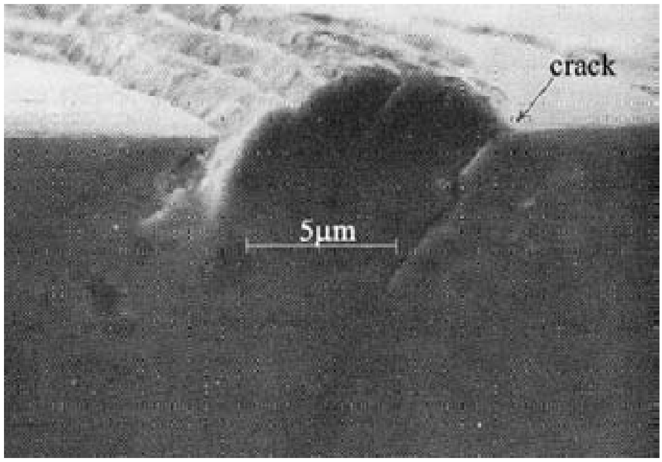

Figure 3.13: Fatigue crack initiation in a copper crystal,[33].

anisotropic microstructure behaviour due to the plasticity partition throughout the γ channels and oriented coarsening of the γ' precipitates. The second one presents homogeneous deformation behaviour. The same single crystal coated with a coating called C_1A was tested under LCF at 600 ℃, 950 ℃ and 1100 ℃ with various frequencies [36]. It was shown that the coating had a favourable effect on the fatigue life at 950 ℃ and 1100 ℃ but not at 600 ℃ because of its brittle properties at low temperatures. At higher frequencies many cracks were initiated at the surface propagating perpendicularly to the load axis into the substrate. At lower frequencies the surface damage was accelerated by oxidation.

A directionally solidified nickel-base superalloy DSCM247 coated with a plasma sprayed NiCrAlY coating was investigated under LCF test conditions by [30]. The LCF results showed that the fatigue life of the coated superalloy was controlled by the fatigue behaviour of the bulk material and that the cyclic deformation behaviour could depend on the strain rate. The area fraction of the coating in the load-bearing cross-section was about 11 % of the coated superalloy. The fatigue life of the coated superalloy was lower at 400 ℃ and slightly higher at 1000 ℃ when compared to the uncoated superalloy. The cracks started at the surface of the coating and at the pores in the coating when tested at temperatures below the ductile to brittle transition temperature (DBTT) of the coating while above the DBTT the cracks initiated from the brittle oxide layer build above the ductile coating.

According to a study of [37], the LCF life of aluminized nickel-base superalloy was lowered by 60 % compared to that of the substrate at 600 °C. At 800 °C, the presence of the coating was beneficial for total strain ranges less than 1.2 %. No effect of the coating was observed at 1000 °C. At cyclic strain ranges less than 1 %, the diffusion aluminide coating RT22 was protective when deposited on Udimet 720 alloy and tested at 732 °C in salt [38]. At higher strain ranges the coating produced a sharp notch that extended into the substrate leaving a free way for salt attack on the base material.

The effect of a plasma sprayed NiCoCrAlY coating (PWA 1365-2) and a diffusion aluminide coating (ELCOAT 101) on the fatigue life of the superalloy IN738LC was examined under LCF and TMF loading conditions at 850 °C, [39]. The LCF life of the plasma sprayed superalloy was longer than that of the aluminized coating because of the higher ductility of the plasma sprayed coating. All cracks were initiated at the coating surfaces. The LCF life of the aluminized coated IN783LC was compared with the data for the uncoated IN738LC found in the literature [40] and small differences were found in fatigue life.

Low cycle fatigue behaviour of a free-standing NiCoCrAlY (a specimen made only of the coating) and NiCoCrAlY coated single crystal PWA 1480, $\langle 001 \rangle$, was tested at 650 °C and 1050 °C [41]. At 650 °C, the fatigue life of the coating was less than that of the coated superalloy while at 1050 °C the fatigue life of the coating was five time greater than that of coated PWA. The strain in the coating was largely inelastic as the coating was ductile with a tensile elongation of 250 %. The increase in life was attributed primary to lower crack growth rates at 1050 °C where multiple branching transgranular crack growth was observed. This was unlike the situation at 650 °C when singular cracks were found.

Uncoated and MCrAlY coated single crystal SX60A was tested at room temperature and 800 °C [42] under LCF test conditions. At room temperature the presence of the coating reduced the fatigue life of the substrate while at 800 °C the coating has little or no effect on the fatigue life of the substrate. The decrease in the life of the coated superalloy was linked to an increase in the inelastic strain range.

3.5 Thermo-mechanical fatigue

Thermo-mechanical fatigue was developed in the early 1970s to simulate loading conditions experienced by turbine blades and vanes in laboratory scale [43]. Superalloys are subjected to thermal and thermo-mechanical loads. Under thermal loading, stresses are developed under thermal cycling without external loading while under thermo-mechanical fatigue, stresses are developed under simultaneous changes in temperature and mechanical strain [13]. Mechanical strain $\varepsilon_{\mathrm{mech}}$ is defined by subtracting thermal strain $\varepsilon_{\mathrm{th}}$ from net strain $\varepsilon_{\mathrm{net}}$ according to eq. (3.3):

$$\varepsilon_{\mathrm{net}} = \varepsilon_{\mathrm{th}} + \varepsilon_{\mathrm{mech}} = \alpha * (T - T_0) + \varepsilon_{\mathrm{mech}} \tag{3.3}$$

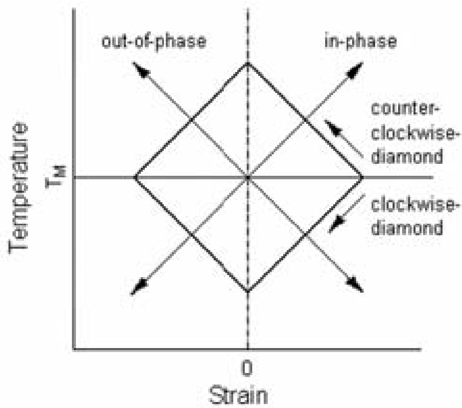

Figure 3.14: Strain and temperature variations for different TMF cycles [44].

where α is thermal expansion coefficient and $(T - T_0)$ is temperature range with T as current temperature.

Usually two kind of TMF tests are conducted in the laboratory with proportional phasing: in-phase (IP), where the maximum strain is applied at the maximum temperature, and out-of-phase (OP), where the maximum strain is applied at the minimum temperature. The variation of thermal and total strains with time in IP and OP cases is illustrated in Figure 3.14 together with other TMF cycle types, a clockwise-diamond (CD) cycle and counter-clockwise-diamond (CCD) cycle, with the intermediate temperature applied at largest tensile and compressive strains, respectively, and the highest and lowest temperatures at zero strain. In the OP-TMF case, the material undergoes compression at high temperatures and tension at low temperatures. The inverse behaviour is observed for the IP-TMF case. The mean stress of the cycle is tensile in the OP-TMF case, while it is compressive in the IP-TMF case. Under TMF conditions, damage mechanisms existing in metals are: fatigue, environmental and creep damage. These damage mechanisms may act independently or in combination with other operating conditions, such as strain rate or dwell time. Traditionally, fatigue damage is the cyclic plasticity-driven, time and temperature independent damage existing when cyclic loading occurs. Creep is a viscous deformation at a constant stress level that leads to intergranular creep cavity growth and rupture. Under TMF loading, creep deformation can contribute to the formation and propagation of microcracks. Metals exposed to environments at high temperatures are also subjected to corrosion by oxidation that can be accelerated by tensile stresses. Frequency is observed to influence both low cycle fatigue and thermo-mechanical fatigue life of superalloys [13]. The effect of different frequencies on fatigue is not presented here because all tests were done at the same frequency.

3.5.1 Polycrystalline superalloys

In gas turbine components, where coatings are deposited to protect against corrosion and oxidation, it is found that coatings can effect thermo-mechanical fatigue life of substrates. The study of [43] showed that a NiCrAlY coating had no effect on the OP-TMF life of IN738 in the temperature range of 450 ℃ and 850 ℃ tested at a strain rate of $10^{-5}\mathrm{s}^{-1}$ and with a strain ratio of $R = \varepsilon_{\min}/\varepsilon_{\max} = -1$. They concluded that oxidation did not play significant role in this temperature range. Chen et al. [45] performed LCF and two different TMF tests on IN738LC at 750 ℃ and 950 ℃ to study fatigue life and damage. The results showed that OP-TMF gave much shorter life than IP-TMF and LCF tests. High tensile stresses developed during OP-TMF tests were possible reason for the observed behaviour. In [46], LCF life was observed to lie between IP and OP-TMF life data where shortest life was found for OP-TMF tested specimens. They concluded that the differences in the life were caused by stress-temperature relations. In work of [47] , Beck et al. run LCF, IP-TMF and OP-TMF tests on uncoated IN792CC and found that the LCF life was two times higher than for all TMF tests. It was also explained that the difference in the lives was probably due to higher stress amplitudes developed during TMF tests causing more damage during each TMF cycle than during LCF. LCF life of uncoated IN738LC was found to be similar to the IP-TMF life in the study of [48] in the temperature range of 750 ℃ to 950 ℃. Zamrik and Renauld [49] tested NiCoCrAlY coated IN738LC specimens under OP-TMF test conditions between 482 ℃ to 816 ℃ and observed lower life for the coated specimens. Extensive research of [50] on three different nickel-base superalloys, IN738LC, CMSX-4 and CM247LC, coated with aluminized CoNiCrAlY and tested under LCF and OP-TMF test conditions revealed that all fatigue life was substrate dependent and that OP-TMF life was "remarkably" shorter compared to the uncoated specimens. They related the detrimental effect of the coatings on the OP-TMF life to the ductility of the coating. They also found that the LCF life was improved by presence of the coatings at 900 ℃ while the diamond TMF life was comparable to those of the uncoated specimens. At higher applied strain, IP-TMF life between 400 ℃ and 850 ℃ was longer for IN738LC specimens coated with a diffusion ELCOAT 101 coating when compared to the uncoated IN738LC tested under same test conditions [40]. A cross-over in life was found between IP-TMF and LCF tests but with small differences. Plasma sprayed IN738LC specimens with a NiCoCrAlY coating gave longer life during LCF than during IP-TMF tests due to slow propagation of cracks through the coating at constant temperature [51]. Hardening occurred during both IP and OP-TMF tests with higher hardening tendency than during

LCF in a cast superalloy K417 [52]. In a study of [46], cyclic hardening is observed for an FNiCrCoMo alloy exposed to OP-TMF tests between 600 °C and 1050 °C.

3.5.2 Single crystal superalloys

Reduced asymmetric CCD-TMF life was reported by [53] for single phase Pt-modified single crystal PWA 1484 in the temperature range of 400 °C and 1100 °C. The negative effect of the coating was attributed to brittle cracking in an early stage of the test. Single crystal CMSX-4 was coated with low-pressure plasma sprayed NiCoCrAlY and tested under OP-TMF at two maximum (800 °C and 1000 °C) and two minimum temperatures (100 °C and 400 °C) [54]. They observed lower life for the coated specimens, differences in crack initiation at the surface of the coatings and differences in the stress strain behaviour between the uncoated and coated specimens. During cooling cycles in TMF tests, coatings become strong and take over thermal mismatch stresses leading to high tensile stresses in coatings. Coatings are found to have a positive effect on TMF life of a single crystal AM1 between 650 °C and 1100 °C by [55] when the specimens were tested into ductile region of the coating. Shorter fatigue was also found during OP-TMF test conditions than during IP-TMF for uncoated single crystal SRR99 in the study by [56]. Chataigner, Fleury and Rémy [57] studied the influence of C1A coating on a single crystal AM1 during a specific TMF cycle between 600 °C and 1100 °C with a peak strain at 950 °C. The life of the coated specimens was found to be the same as for the uncoated specimens and cracks were found initiating from the surface of the C1A coating. The same results are found in [58]. Bain [59] examined the effect of two coatings, a CoCrAlY overlay and an aluminide coating, on LCF and TMF life of a single crystal. The application of the coatings degrades the TMF life which was lower than the LCF life. Aluminide coatings exhibited longer life at lower strains while the inverse occurred at higher strains. Sakaguchi and Okazaki [60] summarized observations of the influence of test type on the fatigue lifetime of CMSX-4 saying that OP-TMF and IP-TMF cannot be comparable to the LCF lives. In a report by [61], it is observed that the uncoated CMSX-4 specimens have significantly longer life than the coated specimens due to “greater number of crack initiation sites provided by the coating” when tested during OP-TMF loading conditions at two temperature ranges, 400 °C and 1050 °C and 650 °C and 1050 °C. No “principal difference” in mechanical stress response was found between aluminide coated and uncoated single crystal CMSX-6 tested under an asymmetric TMF cycle in a temperature range of 400 °C and 1100 °C due to the coating thickness which was about 50 µm [62].

3.6 Summary of LCF and TMF review

This brief survey of the low cycle fatigue and thermo-mechanical fatigue lives of a number of uncoated and coated superalloys found in the literature has pointed to different conclusions. It highlights significant differences in damage mechanisms and material behaviour observed during both low cycle fatigue and thermo-mechanical fatigue. A few general conclusions can be made about the behaviour of both uncoated and coated nickel-base superalloys during low cycle fatigue and thermo-mechanical fatigue test conditions:

- Fatigue life differs with type of load applied, i.e. isothermal low cycle fatigue or thermo-mechanical fatigue.
- Coatings are detrimental during low cycle fatigue load at test temperatures under their ductile to brittle transition temperature. Cracks were found to initiate both from the surface of coatings and pores in the coatings.
- Above the ductile to brittle transition temperature, coatings can have a favourable effect on low cycle fatigue life of superalloys. Cracks were found to initiate from brittle oxide layers built on the top of coatings during tests. It was also found that at temperatures of about 1000 °C, coatings did not have any effect.
- Life of overlay coatings were generally longer than that of aluminide coatings due to their higher ductility.
- During thermo-mechanical fatigue, some researchers state that coatings do not have positive effect of fatigue life while other state the opposite (for single crystals).
- Hardening is found both during LCF at lower temperatures and TMF test conditions between certain temperatures.
- Isothermal tests cannot be used to describe a material's behaviour under thermo-mechanical fatigue because of different deformation mechanisms.

Considerable work has been done in the past on low cycle fatigue and thermo-mechanical fatigue of uncoated and coated nickel-base superalloys. However, many questions still remain, other factors which affect fatigue life of coated superalloys have not been discussed here, for example, the effects of corrosion, interdiffusion, different coating techniques, heat treatment and aging. These factors give additional effects that further complicate an already complex issue. Structural stability of coatings is an important factor if coatings have to maintain their protective qualities over extended periods of time at high temperatures. Coatings degrade

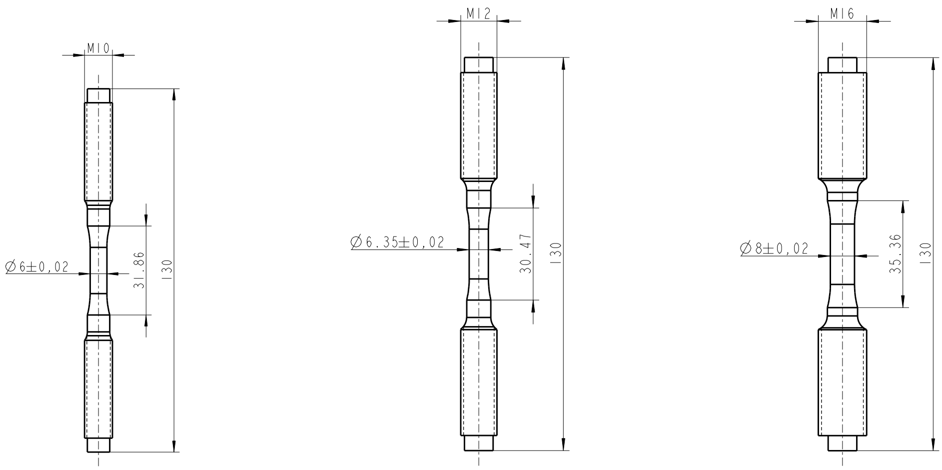

Figure 3.15: Low cycle fatigue, a and b, and thermo-mechanical fatigue, b and c, solid specimens used in the experiments.

not only by loss of scale forming elements to the surface, but also by interdiffusion with the substrate. This can result in additional problems such as the formation of topologically-close-packed phases, for example σ, below the coatings which cause embrittlement of the substrate. There is a great need of better understanding of the complex behaviour of coatings as well as coated superalloys.

3.7 Experimental procedure

Cylindrical solid specimens were machined from polycrystalline IN792 and from single crystals superalloys in such a manner that the crystallographic $\langle 001 \rangle$ directions of the single crystals were aligned with a maximum deviation of 13° from the specimen axis. The test specimens were designed according to ASTM Standard E606-80, see Figure 3.15. All mechanical LCF and TMF tests were performed at Linköping University on a closed loop servo hydraulic testing machine INSTRON Model 8801 equipped with a 100 kN load cell. The equipment used for both LCF and TMF tests is presented in Figure 3.16.

Cyclic fatigue tests were conducted in axial total strain amplitude control mode under fully reversed tension-compression loading with $R = \varepsilon_{\min}/\varepsilon_{\max} = -1$. All tests were initiated in tension load and triangular waveform was used as a test command signal. An axial 10-mm gauge length EPSILON high-temperature

Figure 3.16: Equipment used for both low cycle fatigue and thermo-mechanical fatigue tests.

extensometer, model 3448, was attached on the all specimens to monitor and control total strain amplitude during the test. The specimens were heated by induction heating furnace Hüttinger, Model TIG5/300, with different rates. Eurotherm Controller System model 2408 was used to control and regulate the heating of the specimens. The temperatures were controlled and monitored using a pyrometer PZ20AF, Keller, and N-type thermocouples wound around the circumference of the specimen near the gauge length extremity at both sides. Maximum temperature variation during any given test was well within ±1 ℃.

The LCF tests were conducted at two temperatures, 500 ℃ and 900 ℃, in laboratory air without any dwell time and applied strain ranges of $\Delta\varepsilon_{\mathrm{t}} = 0.8, 1.0, 1.2, 1.4$ and 1.6%. The applied strain rate was $10^{-3}\mathrm{s}^{-1}$ (6 %/min). The experiments were stopped after a 50 % load range drop from the saturation load range or after the total fracture of the specimens. A triangular waveform was used at all tests. To simulate as close as possible long term exposure of gas turbine engines during service life, aging were performed at 1050 ℃ for 2000 h on some coated specimens.

The out-of-phase TMF tests ($\mathrm{T}_{\mathrm{min}}$ at $\varepsilon_{\mathrm{max}}$) were run between 250 ℃ and 900 ℃ with $\mathrm{R} = \varepsilon_{\mathrm{min}}/\varepsilon_{\mathrm{max}} = -1$ in laboratory air at applied total mechanical strain ranges of $\Delta\varepsilon_{\mathrm{t}} = 0.4, 0.6, 0.8$ and 1.0% until a load drop of 20 % or the catastrophic failure of the specimen. The period of the TMF cycle was 100 sec without any dwell time. Prior to each TMF test, the thermal strain, $\varepsilon_{\mathrm{therm}}$, was determined for each sample as a function of temperature under zero mechanical load. A forced air cooling was applied by placing three nozzles with compressed air around the middle of the specimen, Figure 3.17.

The test matrix for isothermal LCF and TMF tests on all uncoated and coated superalloys is presented in Table 3.7. Number behind plus sign in the test matrix means the number of long-term aged specimens that have been tested under both LCF and TMF test conditions.

3.7.1 Experimental analysis

Specimens were examined using light optical microscopy (OP) and scanning electron microscopy (SEM) equipped with an energy-dispersive X-ray spectroscopy (EDS) and a wavelength dispersive spectrometer (WDS) to determine microstructural features, fracture modes, damage and microscopic mechanisms governing the final fracture. Specimens for the observations were first sectioned using a diamond saw taking both the radial and axial sections for a thorough overview of cracks in the specimens. After sectioning, specimens were mounted in an epoxy resin and ground using emery paper of various grades from 120 to 1200 grit and mechanically polished using 3 µm diamond paste. For final polishing an acid alumina suspension OP-A was used. To reveal phases and grains, a solution of 80 % hydrochloric and

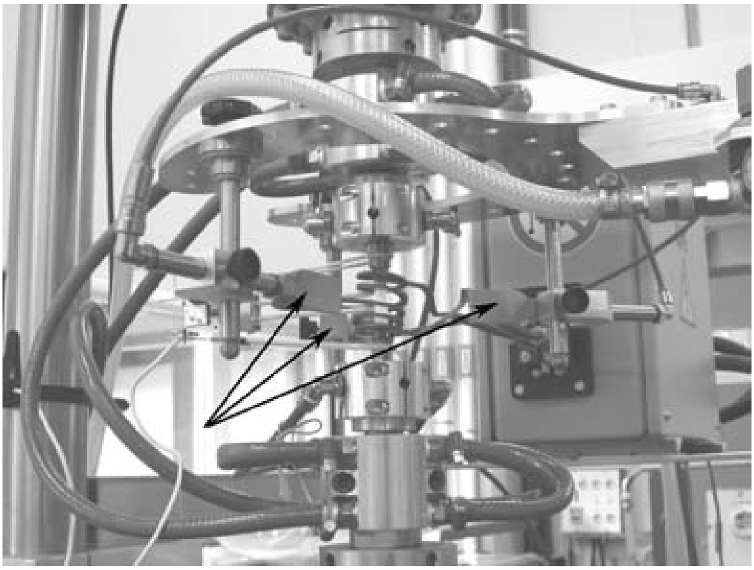

Figure 3.17: Set-up of a thermo-mechanical fatigue tests. The arrows show the nozzles used during cooling cycles.

20 % nitric acid was used for 5 to 6 s. Afterwards, the specimens were immediately rinsed in water and ethanol and dried with compressed air. Microstructural and chemical analysis were performed using a Leo 435 SEM with a PGT IMIX-PC EDS analyzer and a FE-SEM Hitachi SU-70 with INCA EDS Energy system. The optimal accelerating voltage to obtain images with backscattered electrons for best contrast between phases was 20 kV.

3.8 Conclusion

The research in this effort has been directed towards clarification and better understanding of the influence of load, temperature and coating on fatigue behaviour of superalloys. The main achievements of this work can be summarized as follows:

- A low cycle fatigue and thermo-mechanical fatigue test rig has been set-up on a servo-hydraulic machine to simulate complex cycles experienced in gas turbines and aircraft engines.
- A lot of low cycle fatigue and thermo-mechanical fatigue data has been generated, which can be used for numerical simulation, life prediction and

Table 3.7: Test matrix

Superalloy Coating	CMSX-4 RT22	AMDRY997	IC1	IC3	Uncoated	Totally
LCF-500 °C	4+3	4+2	3+2	2	3	23
LCF-900 °C	3+3	4+2	3+2	2	3	22
OP-TMF	5	3	5	3	4	20
Superalloy Coating	SCB RT22	AMDRY997	IC1	IC3	Uncoated	
LCF-500 °C	4+3	4+3	3+3	2	3	25
LCF-900 °C	4+3	4+3	3+3	2	3	25
OP-TMF	5	2	6+3	5	6	26
Superalloy Coating	IN792 RT22	AMDRY997	IC1	IC3	Uncoated	
LCF-500 °C	4+3	4+3	4+2	3	4	27
LCF-900 °C	4+3	4+2	4+2	3	4	26
OP-TMF	4	4	5+2	6	6	27
Totally						221

modelling of material behaviour under fatigue.

- It was shown that the coatings have detrimental effect on low cycle fatigue at lower test temperature, i.e. under ductile to brittle transition temperature of the coatings.
- The effect of presence of the coatings on thermo-mechanical fatigue life is different for the three superalloys, enhancing the TMF life of the single crystals and degrading the TMF life of the polycrystalline superalloy.
- The dominant damage mechanisms were due to fatigue damage (surface crack initiation and in some cases from pores) during LCF tests at lower temperature and coupled oxidation-fatigue damage during LCF tests at higher temperature and OP-TMF. Long-term aging degrades the coatings by forming a brittle aluminium oxide layer on the surface which serves as initation site for cracks.
- The fatigue life of the long-term aged specimens was generally less than that of the unaged specimens. No cracks were initiated from TCP phases formed under the interdiffusion zone in CMSX-4 and IC1 coated IN792.

- Cyclic hardening occurs during both LCF at 500 °C and OP-TMF but not during LCF at 900 °C. The hardening effect is higher in the uncoated specimens compared to the coated specimens.

3.9 Future work

Surface engineering and coatings technology play a crucial role in the operation of all high temperature engines. The desire for higher operating temperatures, improved performance, extended component lives and cleaner fuel-efficient power generation places severe demands on the structural materials. Many components operating at high temperature are coated to enable cost-effective component lifetimes to be achieved. Coatings are generally applied to provide oxidation, corrosion or thermal protection depending on the nature of the operating environment and thermal loads. Any coating should possess the requisite mechanical properties, adhesion and metallurgical stability in contact with the substrate to withstand thermal and mechanical cyclic loadings.

Development of computational based modelling methods is needed to limit the costs associated with testing and to provide increased flexibility. The development of computer models that can account, for example, for defects and residual stress distributions in coated systems, corrosion and oxidation attack and for the effects of complex fatigue cycle with and without dwell time on lifetime for gas turbine components is needed. In this thesis a lot of data, that can be used for modelling, has been generated but the results show that the small number of tested specimens contributes to scatter in the fatigue data. A larger number of specimens would likely decrease the overall scatter of the data and provide a higher level of confidence in the differences found in LCF and TMF and in modelling. Developing an improved understanding of the mechanical properties and behaviour of coatings and substrates under service like loading conditions and environments is very still important.

Improved coatings that are capable of providing increased thermal protection and more reliable integrity with substrates for longer periods, i.e. strain tolerance, reduced thermal mismatch and increased phase stability, are needed. Emphasis should be placed on the importance of microstructure, in particular during long service lives. Microstructure and composition of coatings can change during operation at higher temperatures as a result of Al consumption by oxidation and diffusion of main alloying elements. The initial microstructures can transform to other phases such as γ solid solution of Ni and can be accompanied by grain growth in overlay coatings or martensitic transformations in β-NiAl coatings. To study such microstructural transformation of coatings longer LCF and TMF tests are needed.

Furthermore, research topics in thermo-mechanical fatigue should include mapping of failure and damage accumulation process, mechanisms of microcracking, strength of coated systems, crack nucleation, initiation and crack growth, micromechanical and constitutive modelling and life prediction methods which account for thermal and cyclic loadings as well as for corrosion and oxidation.

Under the Allbatros programme, extensive experimental fatigue tests on many coated systems have been done and some of the data has not yet been published. For future work it is planned to publish all data.

4 Summary of the appended papers

Paper I - Low Cycle Fatigue and Fracture of a Coated Superalloy CMSX-4
A coated single crystal nickel-base superalloy CMSX-4 has been tested under low cycle fatigue test conditions to study effects of coatings on the fatigue life. For this purpose three different coatings have been chosen, an overlay coating AMDRY997, a platinum modified aluminide diffusion coating RT22 and an innovative coating with a NiW diffusion barrier in the interdiffusion zone called IC1. For comparison, tests also were done on the uncoated specimens taken from the same batch. Cylindrical solid specimens were cyclically deformed with fully reversed tension-compression loading with total strain amplitude control at two temperatures, 500 ℃ and 900 ℃. The applied total strains were 1.0 , 1.2 and 1.4 %. The waveform of the fatigue cycle was triangular at a strain rate of $10^{-3}\mathrm{s}^{-1}$ or 6 %/min. The stress and strain response was calculated during the test. The empirical relationship between strain amplitudes and number of reversals to fatigue failure was determined by Basquin and Coffin-Manson equations and presented with a plot. Fracture behaviour of the coated specimens was examined by scanning electron microscope to determine fracture modes and mechanisms. The investigation shows that the coatings have detrimental effect on the fatigue life of CMSX-4 at 500 ℃ while at 900 ℃ an improvement of the LCF life is observed. The reduction of the fatigue life at 500 ℃ can be related to early cracking of the coatings under their ductile to brittle transition temperature (DBTT), where their surface roughness can serve as notches to fatigue crack initiation.

Paper II - Low-Cycle Fatigue and Damage of an Uncoated and Coated Single Crystal Nickel-Base Superalloy SCB
This paper presents low cycle fatigue behaviour and damage mechanisms of uncoated and coated single crystal nickel-base superalloy SCB tested at 500 ℃ and 900 ℃. Four coatings were deposited on the base material, an overlay coating AMDRY997, a platinum-modified aluminide diffusion coating RT22 and two innovative coatings called IC1 and IC3 with a NiW diffusion barrier in the interface with the substrate. The low cycle fatigue tests were performed at three strain amplitudes, 1.0, 1.2 and 1.4 %, with $R = -1$, in laboratory air and without any dwell time. The low cycle fatigue life of the specimens is determined by crack initiation and propagation. Crack data are presented for different classes of crack size in the form of crack density, that is, the number of cracks normalized to the investigated interface length. Micrographs of damage of the coatings are also

shown.

The effect of the coatings on the LCF life of the superalloy was dependent on the test temperature and deposited coating. At 500 °C all coatings had a detrimental effect on the fatigue life of SCB. At 900 °C both AMDRY997 and IC1 prolonged the fatigue life of the superalloy by factors ranging between 1.5 and 4 while RT22 and IC3 had a negative effect on the fatigue life of SCB. Specimens coated with RT22 exhibited generally more damage than other tested coatings at 900 °C. Most of the observed cracks initiated at the coating surface and a majority were arrested in the interdiffusion zone between the base material and the coating. No topologically close-packed phases were found. Delamination was only found in AMDRY997 at higher strains. Surface roughness or rumpling was found in the overlay coating AMDRY997 with some cracks initiating from the rumples. The failure morphology at 900 °C reflected the role of oxidation in the fatigue life, the crack initiation and propagation of the coated specimens. The wake of the cracks grown into the substrate was severely oxidized leading to the loss of Al and Ti to the oxide and resulting in the formation of a γ' depleted zone. The cracks grew more or less perpendicular to the load axis in a Stage II manner.

Paper III - Comparison of Low-Cycle Fatigue Properties of Two Coated Single Crystal Nickel-Base Superalloys, CMSX-4 and SCB

Damage mechanisms during low cycle fatigue of the two uncoated and coated single crystal nickel-base superalloys CMSX-4 and SCB were investigated at three strain levels and two temperatures. At 500 °C, the superalloys have similar fatigue lives while at 900 °C, SCB gives slightly longer life than CMSX-4. Both AMDRY997 and RT22 when coated on CMSX-4 give longer life than when coated on SCB at all temperatures. IC1 performs better when coated on SCB. The coatings have contributed to a change in the fatigue life of the substrates. The main conclusion is that at 500 °C all coatings have a detrimental effect on the fatigue life of both superalloys. At 900 °C fatigue lives of the coated specimens are longer than those of uncoated ones except for RT22 coated on SCB. The initiation mechanism at 500 °C was brittle fatigue fracture of the coatings. This mechanism provided an initial defect at the coatings from which a crack grew into the substrate. Since RT22 is a brittle coating due to the brittleness of NiAl-phase it is impossible to comply with more ductile substrate leading to early fatigue crack initiation in the substrate under the DBTT. The beneficial effect of the coatings at 900 °C can be coupled to the good ductility over the DBTT. The NiW diffusion barrier can slow down crack propagation by acting as an obstacle to the crack growing into the substrate. Primarily, observed cracks were initiated at the coating surface growing more or less perpendicular to the load axis. Crack growth path is mainly intergranular through RT22. In the other coatings the cracks grew both intergranularly and transgranularly.

Paper IV - Low Cycle Fatigue, Thermo-Mechanical Fatigue and Failure Mechanisms of an Uncoated and Coated Polycrystalline Nickel-Base Superalloy IN792

Isothermal low cycle fatigue tests under fully reversed cyclic conditions were performed at 500 °C and 900 °C on uncoated and coated polycrystalline nickel-base superalloy IN792. Thermo-mechanical fatigue behaviour was studied under out-of-phase (T_{min} at ε_{max}) loading in the temperature range from 250 °C to 900 °C of same coated systems. The coatings investigated were an overlay AMDRY997-NiCoCrAlYTa coating, a diffusion platinum modified aluminide coating RT22 and two innovative coatings called IC1 and IC3. This report presents also the effects of long-term aging on low cycle fatigue and thermo-mechanical fatigue behaviour. The stress-strain response and the cyclic life of the material were measured during the tests. The results showed that the tendency to cyclic hardening during thermo-mechanical fatigue was higher for RT22 at 0.8 % and IC1 coated systems than that during low cycle fatigue tests at 500 °C while isothermal fatigue at 900 °C was observed to cause softening and lower cyclic stress than thermo-mechanical fatigue.

At corresponding strain amplitude, the thermo-mechanical life was lower than that of the isothermal fatigue at 500 °C and higher when compared to the isothermal fatigue tests done at 900 °C. Additional damage is produced by the reaction between mechanical stress cycles and temperature cycles in thermo-mechanical fatigue, which could lead to the decrease in the thermo-mechanical fatigue life when compared to the low cycle fatigue life. The uncoated specimens exhibited longer thermo-mechanical fatigue life than low cycle fatigue life. The specimens deposited with the overlay coating AMDRY997 exhibited slightly longer thermo-mechanical fatigue life than other studied coatings. The cracks were initiated at the surface of the coatings in most cases but they were also found to initiate in the substrate. Microscopic observations of the fracture surfaces and the longitudinal sections revealed mixed initiation mode and transgranular fracture mode under thermo-mechanical fatigue.

Paper V - Influence of Long Term Aging on Microstructure and Low Cycle Fatigue Behaviour of Two Coated Nickel-Base Single Crystal Superalloys

Long-term aging of metallic coatings results in changes of mechanical properties due to the significant interdiffusion of the main alloying elements between substrate and coatings. The objective of this study is to examine and describe the influence of long-term thermal aging at 1050 °C for 2000 h on microstructure evaluation, low cycle fatigue life and fracture behaviour of two coated single crystal nickel-base superalloys, CMSX-4 and SCB. The coatings used in the study were AMDRY997 and RT22 as reference coatings and two innovative coatings with a NiW interdiffusion barrier, IC1 and IC3. A number of cylindrical solid specimens were first aged at to simulate long-term exposure of aircraft engine and gas turbine service environment and then cyclically deformed with fully reversed tension-compression loading under total strain amplitude control at two elevated temperatures of 500 ℃ and 900 ℃ and a constant strain rate of $10^{-3}s^{-1}$ (6 %/min) in air atmosphere without any dwell time.The tests indicate that long-term aging influences fracture and fatigue behaviour of coated superalloys exposed to oxidation and diffusion. Aged specimens exhibit longer fatigue life in some cases depending on coating system and shorter life during other test conditions. Fatigue cracks in most cases were initiated at the surface of the coating, growing intergranularly perpendicular to the load axis. Major degradation mechanism in AMDRY997 coating deposited on CMSX-4 tested at 900 ℃ is surface oxidation and interdiffusion with the substrate. Cracks in this aged coated system in the aged condition propagated transgranularly through the coating changing the path behaviour when passing the interdiffusion zone.

My contribution to each paper is indicated by the position of my name in the list of the authors. In all papers I set-up all LCF and TMF tests, run 148 LCF and 73 TMF tests, prepared the tested specimens to analysis, analyzed the results, researched the literature and wrote the papers.

5 Summary of the papers not included in the thesis

Paper I - Low Cycle Fatigue of Single Crystal Nickel-Base Superalloy CMSX-4 with a New Coating IC1
S. Stekovic

Published in ASME International Mechanical Engineering Congress and Exposition, IMECE 2005, Orlando, USA, p. 235-241, 2005

Paper II - Damage Occurring During Low Cycle Fatigue of a Coated Single Crystal Nickel-Base Superalloy SCB
S. Stekovic

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Paper III - ALLBATROS advanced long life blade turbine coating systems
M. P. Bacos and P. Josso and N. Vialas and D. Poquillon and B. Peraggi and D. Monceau and J. R. Nicholls and N. Simms and A. Encinas-Oropesa and T. Ericsson and S. Stekovic

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My contribution to each paper is indicated by the position of my name in the list of the authors. In all papers, except the last one on the list, I set-up and run all LCF and TMF tests, prepared the tested specimens to analysis, analysed the results, researched the literature and wrote the papers. In the last article, that was published in my licentiate thesis, my supervisor Torsten Ericsson wrote the main part of the last page in the paper, which was about our research in the European project called Allbatros. Madam Dr. Marie-Pierre Bacos put contribution from all project partners together into an article.

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Low Cycle Fatigue and Thermo-Mechanical Fatigue of Uncoated and Coated Nickel-Base Superalloys

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Sammanfattning
Abstract

High strength nickel-base superalloys have been used in turbine blades for many years because of their superior performance at high temperatures. In such environments superalloys have limited oxidation and corrosion resistance and to solve this problem, protective coatings are deposited on the surface. The positive effect of coatings is based on protecting the surface zone in contact with hot gas atmosphere with a thermodynamically stable oxide layer that acts as a diffusion barrier. During service life, mechanical properties of metallic coatings can be changed due to the significant interdiffusion between substrate and coating. There are also other degradation mechanisms that affect nickel-base superalloys such as low cycle fatigue, thermo-mechanical fatigue and creep.

The focus of this work is on a study of the low cycle fatigue and thermo-mechanical fatigue behaviour of a polycrystalline, IN792, and two single crystal nickel-base superalloys, CMSX-4 and SCB, coated with four different coatings, an overlay coating AMDRY997, a platinum aluminide modified diffusion coating RT22 and two innovative coatings with a NiW interdiffusion barrier called IC1 and IC3. An LCF and TMF device was designed and set-up to simulate the service loading of turbine blades and vanes. The LCF tests were run at 500^{o}C and 900^{o}C while the TMF tests were run between 250^{o}C and 900^{o}C. To simulate service life, some coated specimens were exposed at 1050^{o}C for 2000h before the tests.

The main conclusions are that the presence of the coatings is, in most cases, detrimental to low cycle fatigue lives of the superalloys at 500^{o}C while the coatings do improve the low cycle fatigue lives of the superalloys at 900^{o}C. Under thermomechanical fatigue loading conditions, the coatings have negative effect on the lifetime of IN792. On single crystals, they are found to improve thermo-mechanical fatigue life of the superalloys, especially at lower strains. The tests also indicate that long-term aging influences the fatigue life of the coated superalloys by oxidation and diffusion mechanisms when compared to unaged specimens. The long-term aged specimens exhibit longer life in some cases and shorter life during other test conditions. Fatigue cracks were in most cases initiated at the surface of the coatings, growing both intergranularly and transgranularly perpendicular to the load axis.

Nyckelord
Keyword
Aero engine, AMDRY995, AMDRY997, CN91, CMSX-4, Coatings, crack initiation, diffusion barrier, gas turbine, IC1, IC3, IN792, innovative coatings, LCO22, long-term aging, microstructure, nickel-base superalloy, polycrystalline superalloy, RT22, SCB, single crystal