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High Temperature Fatigue Behaviour of Austenitic Stainless Steel

Microstructural Evolution during Dwell-Fatigue
and Thermomechanical Fatigue

Hugo Wärner

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Hugo Wärner

Division of Engineering Materials
Department of Management and Engineering
Linköping University, SE-581 83, Linköping, Sweden
<http://www.liu.se>

Linköping, June 2021

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Crack path in a thermomechanical fatigue tested Sanicro 25 specimen.

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Abstract

The global energy consumption is increasing and together with global warming from greenhouse gas emission, a need for more environmentally friendly energy production processes is created. Higher efficiency of biomass power plants can be achieved by increasing temperature and pressure in the boiler section, this would increase the generation of electricity along with the reduction in emission of greenhouse gases e.g. CO₂. The generation of power must also be flexible to be able to follow the demands of the energy market and this results in a need for cyclic operating conditions with alternating output and multiple start-ups and shut-downs.

Because of the need for flexibility, higher temperature and higher pressure of future biomass power plants, the demands of improved mechanical properties of the materials used for the components are also increased. Properties like creep strength, maintained structural integrity, thermomechanical fatigue resistance and high temperature corrosion resistance are critical for materials used in the next generation biomass power plants. Highly alloyed austenitic stainless steels are known to possess such good high temperature properties and are relatively cheap compared to the nickel-base alloys, which are already used in high temperature cyclic conditions for other applications. The behaviour of austenitic stainless steels subjected to future biomass power plants operating conditions are not yet fully investigated.

This thesis presents research that includes investigations of the mechanical and microstructural behaviour during high temperature cyclic conditions of austenitic stainless steels. This is done using thermomechanical fatigue testing, dwell-fatigue testing and impact toughness testing at elevated temperatures. Material service degradation as an effect of microstructural evolution is investigated by ageing of some test specimens before testing. Microscopy is used to investigate the connection between the mechanical behaviour and the microstructural deformation- and damage mechanisms of the austenitic stainless steels after testing.

The results show that creep-fatigue interaction damage, creep damage and oxidation assisted cracking are present during high temperature cyclic conditions. In addition, ageing results in a less favourable microstructural configuration which negatively affects the resistance to high temperature damage mechanisms. An example of this is the lowering of impact toughness due to precipitation and coarsening of detrimental phases of some aged austenitic stainless steels. Moreover, TMF testing of aged austenitic stainless steels induce oxidation assisted cracking and an embrittling effect that cause significant cyclic life decrease. The creep-fatigue interaction behaviour during dwell-fatigue testing of two austenitic stainless steels generates various crack propagation characteristics. The higher alloyed material shows interchanging intra- and intergranular propagation with dynamic recrystallization, while the lower alloyed material shows propagation exclusively along the grain boundaries by the assistance of fatigue induced slip bands interaction with grain boundary precipitates.

The research of this thesis provides a deeper understanding of the structural integrity, deformation mechanisms, damage mechanisms and fracture mechanisms during high temperature cyclic conditions of austenitic stainless steels. Long term, this is believed to contribute to development of suitable materials used as components of future biomass-fired power plants to achieve sustainable power generation.

Populärvetenskaplig sammanfattning på svenska

På grund av den ökande globala energikonsumtionen tillsammans med global uppvärmning från växthusgasutsläpp, finns det ett behov av miljövänlig hållbar energiproduktion. Genom att öka tryck och temperatur hos biomassaeldade kraftverkens ångpannor kan energiproduktionen effektiviseras, vilket skulle bidra till både minskning av biogasutsläpp och ökad energiproduktion. Energiproduktionens flexibilitet har även blivit allt viktigare till följd av en mer osäker energimarknad innehållande ökande miljövänliga alternativ, som till exempel vind- och solenergi, med varierande utgående effekt beroende på väderomställningar. Detta innebär ett ändrat drifttillstånd i form av flera uppstarter och nedstängningar för biomassaeldade kraftverken som delvis kommer användas som uppbackningsalternativ.

De ökande kraven på biomassaeldade kraftverkens flexibilitet, temperatur och tryck ger ökande krav på de mekaniska egenskaperna hos materialen som används för kritiska komponenter. Detta innefattar motstånd till termomekanisk utmattning, högttemperaturkorrosion, kryp och bibehållen strukturell integritet. Höglegerade austenitiska rostfria stål har visat potential att uppnå dessa krav och är dessutom billigare än andra alternativ som till exempel nickelbaserade superlegeringar som vanligtvis används för andra högttemperatursapplikationer med cyklisk drift. Dock har det inte helt utretts hur austenitiska rostfria stål beter sig vid cyklisk drift med förhöjd tryck och temperatur.

Forskningen i denna doktorsavhandling behandlar austenitiska rostfria ståls mekaniska och mikrostrukturella beteende under cykliska driftförhållanden vid hög temperatur. Detta undersöks med hjälp av termomekanisk utmattningsprovning, slagseghetsprovning och utmattningsprovning med hålltid. En del av provstavarna åldras innan mekanisk provning för att undersöka påverkan av mikrostrukturell utveckling över tid. Mikroskopi används för att

undersöka kopplingen mellan de mekaniska egenskaperna och de mikrostrukturella deformations- och skademekanismerna.

De mekaniska och mikroskopiska undersökningarna tyder på att krypskador, interaktion mellan utmattning och kryp och oxidationsassisterad sprickbildning negativt påverkar livslängden för austenitiska rostfria stål under cyklisk drift vid hög temperatur. Åldring innan provning förändrar mikrostrukturens ursprungliga konfiguration vilket resulterar i minskat skydd mot livsreducerande deformations- och skademekanismer. Höglegerade austenitiska rostfria stål visar sig ha bättre högtemperatursbeteende jämfört med andra austenitiska legeringar på grund av dess förstärkta mikrostruktur som ökar motståndet för oxidation, kryp och plastisk deformation.

Sammanfattningsvis har denna forskning resulterat i ökad förståelse för det mekaniska och mikrostrukturella beteendet hos austenitiska rostfria stål under cyklisk drift vid hög temperatur. Långsiktigt kommer detta bidra till förbättrad utveckling av högpresterande material för biomassaeldade kraftverk, vilket gagnar övergången till en mer hållbar framtida energiproduktion.

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Finally, I would like to thank my parents, Claes and Ann, for always supporting and believing in me throughout my endeavours.

Hugo Wärner
Linköping, April 2021

List of Papers

In this thesis, the following papers have been included:

- I. H. Wärner, M. Calmunger, G. Chai, S. Johansson and J. Moverare, **Creep-fatigue interaction in heat resistant austenitic alloys**, Published online May 25, MATEC Web of Conferences, vol. 165, 2018.
I performed the mechanical testing and was the main contributor of the manuscript writing.
- II. H. Wärner, M. Calmunger, G. Chai, S. Johansson and J. Moverare, **Thermomechanical fatigue behaviour of aged heat resistant austenitic alloys**, International Journal of Fatigue, vol. 127, pp. 509-521, 2019.
I performed the mechanical testing of the aged TMF specimens, the hardness testing, the microstructural investigation and was the main contributor of the manuscript writing. The IP-TMF tests was performed by Johan Moverare.
- III. H. Wärner, M. Calmunger, G. Chai, J. Polák, R. Petráš, M. Heczko, T. Kruml, S. Johansson and J. Moverare, **Fracture and Damage Behavior in an Advanced Heat Resistant Austenitic Stainless Steel During LCF, TMF and CF**, Presented at ECF22, Belgrade (Serbia), 2018, Procedia Structural Integrity, ECF22 Proceedings, vol. 13, pp. 843-848, 2018.
I performed the aged IP-TMF tests, the unaged- and aged CF tests. For these test specimens I also performed the microstructural imaging and was the main contributor to the manuscript writing. The LCF tests was performed by Jaroslav Polák, IP-TMF tests at $\Delta T=300\text{ }^{\circ}\text{C}$ - $750\text{ }^{\circ}\text{C}$ was performed by Roman Petráš and the IP-TMF tests at $\Delta T=100\text{ }^{\circ}\text{C}$ - $800\text{ }^{\circ}\text{C}$ was performed by Johan Moverare.

- IV. H. Wärner, M. Calmunger, G. Chai, S. Johansson and J. Moverare, **Structural Integrity and Impact Toughness of Austenitic Stainless Steels**, Presented at ICM13, Melbourne (Australia) and published online in ICM13 proceedings, 2019.

I performed the microstructural investigation and was the main contributor of the manuscript writing. The ageing and the impact toughness testing were performed at AB Sandvik Materials Technology in Sandviken, Sweden. The Thermo-Calc simulations was performed by Johan Moverare.

- V. H. Wärner, J. Xu, G. Chai, J. Moverare and M. Calmunger, **Microstructural evolution during high temperature dwell-fatigue of austenitic stainless steels**, International Journal of Fatigue, vol. 143, 105990, 2021.

I performed the dwell-fatigue testing and was the main contributor of the manuscript writing. I performed the SEM investigation and the TEM investigation was performed by me and Jinghao Xu.

- VI. H. Wärner, Robert Eriksson, G. Chai, S. Johansson, J. Moverare and M. Calmunger, **Influence of ageing on thermomechanical fatigue of austenitic stainless steels**, Presented at MSMF9, Brno (Czech Republic) and published online in Procedia Structural Integrity, vol. 23, pp. 354-359, 2019.

I performed the TMF testing and was the main contributor of the manuscript writing. The microstructural investigation was performed by me and Mattias Calmunger. The modelling part was performed and written by Robert Eriksson. The ageing was performed at AB Sandvik Materials Technology in Sandviken, Sweden.

- VII. H. Wärner, G. Chai, J. Moverare and M. Calmunger, **High temperature fatigue of aged heavy section austenitic stainless steels**, In manuscript, submitted to International Journal of Fatigue.

I performed the TMF testing, the hardness testing, the microstructural investigation and was the main contributor of the manuscript writing.

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Abbreviation

BCC	body-centred cubic
BCT	body-centred tetragonal
BF	bright field
BSE	backscattered electron
CF	creep-fatigue
COM	crystal orientation map
DF	dark field
DRX	dynamic recrystallization
EBSD	electron backscatter diffraction
ECCI	electron channelling contrast imaging
EDS	energy-dispersive spectroscopy
FCC	face-centred cubic
FEG	field emission gun
GB	grain boundary
GBS	grain boundary sliding
IP	in-phase
IPF	inverse pole figure
LAGB	low angle grain boundary
LCF	low cycle fatigue
MAD	mean angular deviation
OP	out-of-phase
RT	room temperature
SE	secondary electron
SEM	scanning electron microscopy
SFE	stacking fault energy
SRC	stress relaxation cracking
STEM	scanning transmission electron microscopy
TEM	transmission electron microscopy
TMF	thermomechanical fatigue
WDS	wavelength-dispersive spectroscopy
Z	atomic number

Part I

Background and Theory

1

Introduction

This chapter introduces the background and goals of the research that has been conducted within the work of this PhD thesis. It also addresses the research project, of which the conducted research is a smaller part, which puts the thesis in a wider context.

1.1 Introduction to the research project

This doctoral thesis is a part of KME-701, *Influence of high-temperature environments on the mechanical behaviours of high-temperature austenitic stainless steels* [1], and KME-801, *Heavy section austenitic stainless steel for the future header and piping material in high-efficient biomass-fired power plants* [2], which are research projects in collaboration between Linköping University, AB Sandvik Materials Technology and Sandvik Heating Technology AB. The research project is partly financed by the Swedish Energy Agency and by industries, through the research consortium of materials technology for thermal energy processes (KME) [3].

1.2 Background

With rising energy consumption the need for renewable energy is great in order to avoid excessive emission of greenhouse gases (e.g. CO_2) [4, 5]. Biomass fuel is one alternative that is renewable in the sense that the amount CO_2 consumed during growth is released during combustion [6, 7]. In the future a need for flexible generation of power is critical if only renewable power generation is to be achieved. The power plants must therefore be able to alter their output, have access to back up power plants or even shutdown during certain periods to be able to follow the demands of the energy market [8–12]. In addition, higher efficiency in power plants is desirable and this can be achieved by increasing pressure and temperature in the boiler sections [13–15]. This results in not fully investigated operating conditions which may negatively affect the materials used for critical components in power plants. These conditions include alternating- and long term high-temperature, cyclic loading and corrosive environments and they are of great interest if the goals for future power generation are to be achieved. During start-up and shut-down, the thermal transient and mechanical strain cycles induce strain-temperature phasing, which is known as thermomechanical fatigue (TMF) [16, 17]. Especially for a heavy section component, temperature gradients are problematic since it causes problems related to thermal expansion and where the expansion close to a stress concentrator is often constrained by the cooler material in its surroundings. The thermal strain is then converted into mechanical strain which induce material damage. TMF testing in an out-of-phase (OP) condition show similar temperature and mechanical profile that simulates the real component condition for a hot-spot surrounded by cooler material or the hot-side of a thicker wall with a temperature gradient [18, 19]. OP-TMF corresponds to a material that undergoes compression at maximum

temperature, T_{max} , and tension at minimum temperature, T_{min} . In-phase (IP) TMF has the opposite configuration, with T_{max} during tension and T_{min} in compression. Furthermore, the cyclic operating conditions contribute to a combined accumulation of cyclic fatigue damage and time-dependent creep damage, that reduces the ductility at high temperature, thus accelerating the fatigue damage [20]. The interaction behaviour of creep and fatigue of a boiler material is complex and can be an individual, a competing or an additive process [21, 22]. This combined damage effect is known as creep-fatigue (CF) interaction and together with TMF, they are key damaging factors during these new power plant operating conditions [15, 20, 23, 24].

Traditionally, ferritic stainless steels have been used for critical power plant components, but with highly corrosive environments and the demands of increased efficiency, other material groups must be considered for components of future biomass-fired power plants [15, 23]. There are power plants in which medium alloyed stainless steel are used for components such as boilers, digesters, pumps and valves [15, 25]. Another possible candidate could be the nickel-base alloys, which are already operating at these new conditions in other applications. However, they are expensive compared to austenitic stainless steels, which is illustrated by Fig. 1. The chart presents the price against the maximum service temperature for different candidate groups of power plant material. However, it should be noted that during cyclic conditions and in combination with fatigue the behaviour and suggested service temperature is not yet fully defined.

Figure 1: Difference in price [SEK/kg] vs. Maximum service temperature [°C], for power plant materials (From GRANTA EduPack 2020).

The alloy definitions and the degree of alloying for the materials in Fig. 1 compared to the investigated materials of this thesis are shown in Fig. 2. Highly alloyed austenitic stainless steels have comparable oxidation- and creep resistance in aggressive high temperature environments to the nickel-base alloys and superior resistance compared to the traditional ferritic stainless steels [26, 27]. These properties make them potentially a suitable candidate for the next generation biomass power plants. However their behaviour, especially for thick wall components (given its higher thermal expansion), at the new cyclic operating conditions have not been fully investigated and understood [13, 25, 27].

Definition	Alloy	Approximate concentration of main alloying elements [wt%]
Nickel-base superalloy	Hastelloy X	Cr=22, Ni=47, Mo=9, Co=1.5, W=0.6, Mn=0.5, C=0.7, Si=0.5, Fe=17.5
Highly alloyed austenitic stainless steels	Sanicro 31HT(*) (alloy 800HT)	Cr=20.5, Ni=30.5, C=0.7, Mn=0.6, Si=0.6, Ti=0.5, Al=0.5 Nb=0.5, Fe=bal
	Sanicro 25 (*)	Cr=22.5, Ni=25, W=3.6, Cu=3.0, Co=1.5, Mn=0.5, Nb=0.5, N=0.23, Si=0.2, C=0.1, Fe=bal
	Alloy 904L	Cr=20.5, Ni=25.5, Mo=4.5, Cu=1.5, Mn=1, Si=0.5, C=0.1, N=0.05, Fe=bal
	AISI 310M (*)	Cr=25.4, Ni=19.2, Mn=0.84, Si=0.55, Mo=0.11, Cu=0.08, N=0.04, C=0.015, Ti=0.001, Fe=bal
Medium alloyed austenitic stainless steels	Esshete 1250 (*)	Cr=15, Ni=9.5, Mn=6.3, Nb=1, Mo=1, Si=0.5, V=0.3, C=0.1, Fe=bal
	AISI 304 (*)	Cr=18.3, Ni=10.3, Mn=1.4, Si=0.3, Cu=0.3, W=0.05, N=0.07, C=0.015, Fe=bal
	AISI 316L	Cr=17, Ni=12, Mn=1, Si=0.5, C=0.015, Fe=bal
Ferritic stainless steels	ASTM CB-30	Cr=19.5, Ni=1, Si=0.75, Mn=0.5, C=0.015, Fe=bal
	T/P122 [15]	Cr=12, W=2.0, Cu=1, Mn=0.6, Mo=0.4, V=0.2, Si=0.2, C=0.11, Nb=0.05, N=0.06, Fe=bal

Figure 2: The definitions and the degree of alloying of the alloys in Fig. 1 compared to the investigated materials of this thesis (marked with *).

1.3 Purpose of the research

The KME-701 and KME-801 projects specifically addresses the following aims of the KME-program: "To evaluate the mechanical properties and service life of various materials in relation to new material requirements for more efficient electricity production (elevated pressures and temperatures)" and

"To develop methods for quantifying processability for new materials, as well as creating an understanding of microstructure development and mechanical properties for more efficient energy plants" [1, 3]. These aims comprise the main purpose of the research in this doctoral thesis, namely "to evaluate the mechanical- and microstructural behaviour of materials that are candidates for future power plant applications, which will suffer from tougher environment due to the increasing efficiency demands and cyclic operations". The results are believed to be used for material design and improvement to enhance the development of future biomass power plant boilers.

1.4 Research questions

The research in this doctoral thesis aims to investigate the mechanical- and microstructural behaviour during high temperature cyclic conditions of austenitic alloys, which could be suitable candidates for critical components in the next generation biomass-fired power plants. For safety reasons, the effect of prolonged microstructural degradation is also investigated to evaluate the durability of the materials. More specifically, the following research questions (RQ) have been addressed:

1. During TMF and CF conditions, what are the mechanisms governing the fatigue response and behaviour of the investigated materials?
2. How will prolonged microstructural degradation by pre-ageing influence the high-temperature behaviour?
3. For the investigated austenitic alloys, how will the different operating conditions influence their performance? Given the selected research methods what important properties should future biomass power plant boiler materials possess?

1.5 Overview of the thesis

This thesis is divided into two parts. Part I provides background and theory to the research area and consists of six chapters. This part is based on the licentiate thesis *High Temperature Fatigue Behaviour of Austenitic Stainless Steel - Influence of Ageing on Thermomechanical Fatigue and Creep-Fatigue Interaction* [28] which was presented in December 2018. However, the content has been significantly modified and extended since then. After an introduction to the research project in Chapter 1, a general presentation of austenitic

stainless steels and the effect of alloying elements with precipitation characteristics are given in Chapter 2. Chapter 3 consists of a description of microstructural mechanisms, TMF and CF interaction together with their influence on austenitic stainless steels. The research methods, mechanical test procedures and the investigated materials are then described in Chapter 4. In Chapter 5, a short summary and discussion of the included papers are provided. This is followed by the overall conclusions of the thesis, in Chapter 6, which also includes suggestions for future work. In Part II, the main research of the thesis is described by the seven included research papers.

2

Austenitic stainless steel

This chapter provides a theoretical background to the material group austenitic stainless steels, which includes definition, alloying elements and commonly formed precipitates.

2.1 Austenitic stainless steel

Iron (Fe)-based alloys with at least 10.5 wt.% chromium (and maximum 1.2 wt.% carbon) are considered stainless steels [29, 30]. The high chromium content leads to formation of a thin dense chromium oxide (Cr_2O_3) film at the surface, providing the corrosion resistance. If the protective layer is damaged or removed, regeneration can be achieved if the surface is exposed to an environment that can deliver enough oxygen. This process is referred to as self-passivation [29, 31]. Commonly stainless steel is divided into four groups (see the list below) depending on their main phases: austenite, ferrite and martensite. The last group in the list is defined by the heat treatment used [29, 32, 33].

- Ferritic
- Austenitic
- Martensitic
- Duplex (austenitic-ferritic)
- Precipitation-hardening

The crystal structure of these main phases is face centred cubic (FCC) for austenite, body centred cubic (BCC) for ferrite and body centred tetragonal (BCT) for martensite. However, the focus of this project is the behaviour of austenitic stainless steels. In general, the austenitic stainless steels includes 15-26 wt.% chromium, 8-25 wt.% nickel, 0-6 wt.% molybdenum and lower amount of other alloying elements [30, 31, 34]. More about these and their general purposes are covered in the next section. Because of the high chromium content, austenitic stainless steels exhibit great corrosion resistance which includes high steam oxidation resistance at elevated temperatures. Other properties such as good weldability and creep resistance, excellent formability and ductility are commonly associated with austenitic stainless steels [33–35]. But compared to other stainless steel groups, they also have high coefficient of thermal expansion which can be problematic when used in dissimilar welds or in thick section components (stress relaxation cracking (SRC) and OP-TMF issues). Due to the fact that austenitic stainless steels are not susceptible to hydrogen embrittlement, in most cases there is no need for pre- and post-weld heating [35]. Increase of strength can be achieved by cold working but normally not by heat treatments [30, 34].

2.1.1 Alloying elements

Different main alloying elements of austenitic stainless steels promote different types of microstructures. In order to keep the appropriate microstructure and gain the best properties for a specific application, a balance of the stabilizing elements must be achieved. The alloying additions below are considered in an iron-base material.

Chromium (Cr) is the main alloying element in stainless steel, and it provides the alloy with corrosion resistance at high temperatures, as described in the last section. Promotion of the ferritic microstructure is also increased by chromium [34, 36].

Nickel (Ni) promotes the austenitic microstructure and generally increases ductility and toughness and together with chromium it provides the high temperature resistance in austenitic stainless steels [30, 34].

Manganese (Mn) generally improves hot ductility, but it also increases the solubility of nitrogen (N) and promotes the austenitic microstructure. Due to the high cost of nickel, manganese is often used to replace some of the nickel content in certain alloys [36, 37].

Copper (Cu) promotes the austenitic microstructure and when finely homogeneously dispersed, it enhances strength and hardening during high-temperature fatigue [38]. It also enhances the formability and machinability [37].

Silicon (Si) promotes the ferritic microstructure and increases the oxidation resistance [34, 37].

Both carbon (C) and nitrogen (N), strongly promote the austenitic microstructure and increase the mechanical strength by solid solution. Carbon reduces resistance of intergranular corrosion due to carbide formation (most often chromium carbides). Titanium (Ti) and niobium (Nb) are added if the carbon content is relatively high and these two elements react more easily with carbon than chromium does, thus lowering the amount of carbide formation in the grain boundaries. In nitrogen containing austenitic stainless steels, niobium also forms nitrides which provides strength when finely dispersed [38]. Both niobium and titanium promote the ferritic microstructure [30, 34, 39].

Tungsten (W) generally increase pitting corrosion resistance by stabilization of the oxide layer and also provides solid solution strengthening when fine precipitates are homogeneous dispersed throughout the grains [37, 38].

Molybdenum (Mo) is a ferrite stabiliser and improves both the creep properties and the corrosion resistance [30, 39].

2.1.2 Precipitation

At high-temperature conditions, austenitic stainless steels commonly form secondary phases, carbides, nitrides and intermetallic phases [36, 40]. Precipitates like these are often undesirable, because of their detrimental effect on the mechanical properties and corrosion resistance. However, when stable precipitates act as obstacles for dislocation movements they can strengthen alloys. Below, common precipitates in austenitic stainless steels are presented.

$M_{23}C_6$ has a FCC crystal structure and in austenitic stainless steels, the main element (M) is usually chromium. The carbide generally nucleates relatively easy and fast at grain boundaries, twin boundaries and as intragranular precipitates. It commonly depletes chromium in the grain boundaries, leading to intergranular corrosion by sensitisation [36, 41]. For some cases the creep strength is improved, but the fatigue properties are reduced due to an embrittling effect [41].

M_6C has a variable combination of elements for M, these includes chromium, iron, molybdenum, niobium and silicon [36]. It has a FCC structure and generally forms in austenitic stainless steel with enough molybdenum or niobium, after long-term ageing [40]. It can be hard to distinguish M_6C from $M_{23}C_6$ because of resembling formation sites and morphologies [42].

Cr_2N has a hexagonal close packed (HCP) crystal structure. At 900 °C, it is found in non-stabilized high niobium containing austenitic stainless steels. In both low nitrogen non-stabilised creep resistant austenitic stainless steels and in stabilised nitrogen grades, Cr_2N is not commonly found. In the latter case it is because Z-phase forms instead [36]

MX (MC) precipitates forms in austenitic stainless steels containing strong carbide/nitride formers, such as titanium, niobium, vanadium, zirconium, tantalum (as M) and nitrogen or carbon or both (as X) [36, 43]. The MX precipitates usually form within dislocations or stacking faults and on grain- and twin boundaries. Dispersed MX precipitates increase the creep rupture strength and intergranular corrosion resistance, but they can lower the creep ductility if not the optimum stoichiometry balance is achieved [36, 41].

Z-phase has a BCT crystal structure and is a carbonitride that forms in niobium containing, high nitrogen, austenitic stainless steels. The Z-phase is usually stable at high-temperature and similar to MX precipitates, it generally forms at grain boundaries, on twin boundaries and within the matrix where dislocations exists [42]. Finely dispersed Z-phases, are usually beneficial for the creep resistance, but in some materials the precipitates coarsen with increasing temperature and time and this has the opposite effect on the creep properties [36, 41]. In niobium containing austenitic stainless steels,

which have a relatively high level of nitrogen compared to carbon, this effect occurs when Z-phase nucleates instead of Nb(C,N) precipitates. However, both Nb(C,N) and the Z-phase can be present at the same time in regions of high niobium content [36, 38, 41].

Sigma phase (σ -phase) is an intermetallic phase that consists of iron, chromium, nickel, molybdenum, silicon and it has a tetragonal crystal structure [36]. It often precipitates between 600 °C and 1000 °C and due to its brittle and hard matrix, it increases the hardness of the material. However, it also decreases the toughness, elongation, thermal fatigue and corrosion resistance (by depletion of chromium and molybdenum). If σ -phase forms as large precipitates or as a film at grain boundaries, it is detrimental to the creep resistance. However, if it forms as small intragranular precipitates, it is found to increase the creep resistance [44]. Generally, σ -phase forms at grain and twin boundaries [36, 44]. Nickel and nitrogen usually suppress formation of σ -phase in austenitic stainless steels [41, 44].

Laves phase is also an intermetallic phase with hexagonal crystal structure and it usually consists of iron, molybdenum, titanium, niobium, silicon, chromium and nickel [45, 46]. In niobium containing steels, Laves phase is detrimental for the creep properties because it promotes formation of M_6C [36]. But in high nickel austenitic stainless steels with tungsten, it is found that the formation of Laves phase at the grain boundaries may improve the creep strength [47]. Commonly, Laves phase is found dispersed inside the grains, but sometimes also at grain boundaries after long time high-temperature exposure between 600 °C and 1000 °C. In niobium and tungsten containing austenitic stainless steels it can form as Fe_2Nb and/or Fe_2W after ageing around 600 °C and 800 °C [40, 45, 46].

3

Microstructural mechanisms, thermomechanical fatigue, creep-fatigue interaction and thier influence on austenitic stainless steels

TMF- and CF like processes appear to limit the fatigue life of critical components in power plants and are considered key factors, along with oxidation, in the understanding of the material degradation behaviour during future operating conditions. This chapter will start with a general description of several microstructural mechanisms and then focus on the behaviour of austenitic stainless steels during TMF- and CF like conditions.

3.1 Deformation mechanisms

Elastic deformation occurs when shear stress causes stretching of the chemical bonds, but after stress removal the deformation disappears. In contrast, inelastic deformation involves permanent rearranging of the atoms after the deformation is completed. Instantaneous inelastic deformation is defined as plastic deformation and deformation which is inflicted by stress only after passage of time, is termed creep [48–50].

3.1.1 Plastic deformation by dislocation motion

All crystalline materials contain imperfections such as point (vacancies, interstitials and impurities), volume (voids), surface (grain boundaries, twins and microcracks) or line defects (dislocations). These are disturbances to the local arrangement of the atoms that greatly influence the properties of the material [48, 51]. This section focuses on plastic deformation by dislocation motion, however the behaviour and effects of the various imperfections are interconnected [48, 49]. There are three types of dislocations: screw dislocation, edge dislocation and mixed dislocation. An edge dislocation has an extra half-plane of atoms midway through the crystal, distorting nearby planes of atoms. The dislocation has two properties, a line direction, which is the direction along the bottom of the extra half plane, and the Burgers vector which describes the distortion in relation to the lattice. In an edge dislocation, the Burgers vector is normal to the line direction and for a screw dislocation the Burgers vector is parallel to the line direction [48, 49, 51].

For a certain critical stress, plastic deformation is created by dislocation motion and if the movement is along the plane of the dislocation line, it is called slip or glide. To allow these movements, all the bonds between the atoms must be broken. Dislocation movement out of the dislocation plane by the means of vacancy-atom exchange is known as dislocation climb. This occurs when the dislocation movement is impeded by an obstacle (a point defect) and when vacancy diffusion is present [52, 53]. If multiple dislocations move in the same direction on the same plane, a slip plane is created. Regions where numerous dislocations move on adjacent slip planes are called slip bands and these are associated with regions of intense plastic shear deformation separated by regions of almost no shear [49, 52, 53]. Evidence of slip planes and slip bands are commonly found when investigating the microstructural features of high temperature fatigued austenitic stainless steels [54–56]. Wavy slip originates from multiple screw dislocations changing from one plane to another, called cross-slip. Cross-slip is influenced by stacking faults, which are errors in the stacking sequence of close-packed planes, and

these interruptions carry a certain stacking fault energy (SFE), which is the associated energy per unit area of fault. The cross-slip mechanism is limited for common low-to-medium alloyed austenitic stainless steels which contains wider stacking faults and therefore lower SFE [48, 49, 53]. For highly alloyed austenitic stainless steels the SFE is found to be higher compared to low- and medium alloyed austenitic stainless steels [57].

In FCC metals such as austenitic stainless steels, slip commonly occurs on the close packed $\{111\}$ planes in $\langle 110 \rangle$ directions, which gives a total of 12 potential slip systems. An illustration of this can be seen in Fig. 3. A slip system is activated when the critical shear stress is reached. Thermal vibrations of atoms may lower the required stress for the dislocations to overcome obstacles and assist the slip process. Thus, an increase in temperature will reduce the flow stress [49].

Figure 3: Slip systems and directions of a FCC crystal, (a) a slip system of $\{111\}\langle 110 \rangle$ within the FCC unit cell, (b) the three $\langle 110 \rangle$ slip directions within the slip plane from (a).

3.1.2 Twin boundaries

Deformation twinning is a process which produces a mirror image misorientation of the crystal structure. Twins can be produced when a shear force, acting along the twin boundary, causes the atoms to shift out of position. Twinning occurs either during deformation or heat treatment of certain metals and experiments have shown that twins often form at very low plastic strain level in BCC and HCP alloys, whilst FCC metals do not generally twin until considerable plastic deformation by slip has occurred [49, 58, 59].

The twin boundaries interfere with the slip process and increase the strength of the material. For austenitic stainless steels, the misorientation angle is commonly 60° and the formation is associated with low SFE due to increased critical twinning shear stress needed with increasing SFE. Temperature and strain rate also affects the twinning stress similarly to SFE but they are not as prominent [59–61].

3.1.3 Creep

A time-dependent plastic deformation under constant stress, where the strain increases with time, is generally defined as creep [48, 49, 52, 62]. The general mechanisms that contribute to creep are commonly classified as dislocation slip, dislocation climb, grain-boundary sliding and diffusion flow caused by vacancies [52, 62]. Both dislocation slip and dislocation climb are often referred to as dislocation creep, but they have different movement characteristics as explained in the 3.1.1 section. Creep by dislocation slip is active in the whole temperature range and at high temperatures creep can occur below the yield stress, but the contribution is usually insignificant at low temperatures [62, 63]. For creep by dislocation climb, the dependence on vacancy diffusion makes the process more active during elevated temperatures [63, 64].

The process in which shear stresses cause grains to slide past each other along the boundaries is called grain-boundary sliding (GBS). Small-grained materials contain large numbers of grain boundaries that acts as barriers against sliding, thus providing creep strength. However, materials that have large grains (a low number of grain boundaries) does not contain as many vacancies and thus dislocation climb is reduced compared to small-grained materials. Grain size has therefore a dual effect on the creep properties of a material [62–64]. Voids and cavities are thought to be preceded by GBS, but it is unclear if the void formation is associated with or exists as a consequence of GBS [50, 65]. As presented in the previous chapter, GBS can be prevented by precipitation of the grain boundaries [63].

Another creep mechanism is diffusion flow, and it is caused by migration of vacancies through the lattice structure. This process requires the chemical bonds to be broken and reformed, thus a certain amount of activation energy is needed. At higher temperatures creep by diffusion flow is therefore more easy [62, 64, 65].

3.1.4 Dynamic recrystallization

For low or medium SFE alloys at elevated temperatures, a microstructural process called dynamic recrystallization (DRX) can occur if a certain critical

strain has been reached [66–68]. In addition to the temperature and deformation rate, the chemical composition and initial grain size also influence the critical strain for initiation of DRX [66, 67, 69, 70]. From the review study of high temperature deformation of metals by Huang and Logé [68] the two mechanisms discontinuous- and continuous dynamic recrystallization (DDRX and CDRX) are usually most active in low/medium SFE metals (DDRX) and in medium/high SFE metals (CDRX), respectively. The mechanism for DDRX is commonly attributed to accumulation of dislocations to a certain critical level, or critical strain, that cause DDRX nuclei groups to form by shearing/sliding of partial pre-existing grain boundaries that induce inhomogeneous local strain and bulging of serrated grain boundaries together with dislocation sub-boundaries or twins. DDRX nuclei groups are commonly observed to develop into so called necklace structures on grain boundaries. Mechanisms attributed to CDRX are homogeneous misorientation increase under high temperature straining so that accumulation of dislocations progressively increase their misorientation and low angle grain boundaries (LAGBs) are created. With further straining, high angle grain boundaries (HAGBs) are formed when misorientation angles reach about 10° [68, 71]. However, interaction with pre-existing grain boundaries have also been described by [68] to nucleate CDRX and microshear bands is known to assist CDRX during severe plastic deformation (SPD).

3.2 Thermomechanical fatigue

Critical power plant components, such as piping for boilers, suffers from stresses and strains originating both from non-uniform temperature fields and internal pressure, which together are found to lower the predicted life of the component [18, 72–75]. Depending of the nature of future cyclic conditions in power plants, different type of thermomechanical phasing may occur and the most common ones during testing are the in-phase (IP) and out-of-phase (OP) [16, 18, 19, 76]. Their respective characteristics are shown in Fig. 4. The main damaging contributions are considered to be creep, fatigue and environmental effects (commonly oxidation) [16]. In the work by Nitta and Kuwabara [77], TMF behaviour of both IP- and OP-TMF are described. Type-I scenario is when IP loading conditions cause earlier failure than OP loading and, in this case, creep is the predominant damaging mechanism. This is because of high temperature and tensile stresses, which is more detrimental during IP cycling. In the type-O scenario, OP loading conditions lead to shorter fatigue life due to environmental effects such as embrittlement by oxidation of the surface which causes early crack initiation. This process

Figure 4: The most common strain controlled TMF test cycles, in-phase (IP) and out-of-phase (OP).

affects OP cycling more than IP cycling. If neither environmental effects nor creep damage dominate, IP and OP loading conditions show nearly the same fatigue life. This is the Type-E scenario. In the last scenario, Type-E', IP loading conditions lead to short fatigue life at high strain amplitudes, which means that creep only occurs at high loads [77].

The study of H.J. Christ [16], investigates environmental effects during TMF conditions on the austenitic stainless steel AISI 304L. It was found that the alloy had moderate influence from environmental effects and compared to vacuum conditions, interaction with air caused grain boundary oxidation which consequently caused premature crack initiation. This effect was active during OP conditions when creep damage was negligible. Bae et al. [78] also performed investigations focusing on the TMF behaviour of AISI 304L, but at a higher temperature range (450 °C to 700 °C) and it was found that creep was the dominant damaging mechanism for IP loading conditions.

TMF testing at a lower temperature range (100 °C to 340 °C) and at a higher temperature range (300 °C to 650 °C) of the austenitic stainless steels 316L(N) were performed by Mageshwaran et al. [79] and Nagesha et al. [80]. At the lower temperature range the TMF life of the IP condition was longer than both OP cycling and isothermal cycling (at $T_{\max}$). However, with increasing TMF peak temperature this difference was found to diminish. At the higher temperature range, the environmental effect by oxidation limited the OP-TMF life.

Petráš et al. [81] performed investigations on the highly alloyed austenitic stainless steel Sanicro 25, subjected to both IP- and OP loading conditions

in the temperature range of 250 °C to 700 °C. Sanicro 25 showed prominent cyclic hardening during these conditions and the most detrimental condition was IP-TMF. This was due to the difference in oxidation assisted crack initiation and growth between IP and OP. For IP tests, oxide cracking occurred in the grain boundaries while the OP tests showed delayed local oxide cracking in the perpendicular direction. In this case, creep should be the most damaging mechanism according to [77].

3.3 Creep-fatigue interaction

It is well known that the combination of creep and fatigue exert detrimental effects on components in the boiler sections of power plants and conservative design development is suggested by Code Cases of ASME, for these types of conditions [20, 22, 82, 83]. The effect of creep damage on the fatigue endurance at elevated temperature is referred to as creep-fatigue (CF) interaction. The acceleration of fatigue damage due to the reduction of ductility caused by creep, is thought to be the main creep-fatigue damage mechanism [20, 84]. However, a more pessimistic analysis should also account for the effects of fatigue upon creep [22]. ASME Code [82] uses CF interaction damage diagrams for proposed service cycles, where the creep and fatigue damage are evaluated separately. This is done by linear summation, using Miner's rule for the fatigue damage (independent of strain rate) and time fraction summation to account for creep damage. The combined formulation can be seen in eq. 1.

$$\phi_f + \phi_c = \sum(n/N_d)_j + \sum(\Delta t/T_d)_k \leq D \quad (1)$$

In the above expression D is the allowable creep-fatigue damage factor, ϕ_f is the separated fatigue damage, ϕ_c is the separated creep damage, N_d is the number of allowable cycles of type j , n is the actual number of cycles of type j , T_d is the allowable time at stress level k and Δt is the actual time at stress level k [22, 82]. D is consequently the factor that account for the combined effect of creep damage (due to the time subjected to an applied stress) and generated fatigue cyclic damage. Values of D for steels from different groups; one ferritic-martensitic (T91), three austenitic (304, 316, 800HT) and one bainitic (2.25Cr1Mo), at elevated temperatures (≈ 425 °C) are shown in the creep-fatigue interaction diagram in Fig. 5. As an example: for 9CrMoV, the figure indicates high CF interaction effect and that the allowable damage fraction for creep is very low in order to be in the

safe design region. However, Skeleton et al. [22] argues that this method of damage assessment can be somewhat pessimistic. Presented below are the types of CF interaction damages in austenitic alloys assessed by Plumbridge [21] and Hales [85].

- Transgranular crack initiation and growth
- Transgranular initiation followed by intergranular growth
- Intergranular initiation followed by transgranular growth
- Intergranular initiation and growth
- Internal triple point wedge crack
- Grain boundary voids
- Initiation from matrix pore or inclusion

Figure 5: Example of a creep-fatigue damage envelope (interaction diagram) re-plotted from ASME Code [82] with axes interchanged (same as in [22]).

In addition to these different possible interaction damages, the crack propagation can be "competing", "interacting" and "additive" according to [22, 85]. In "competing" propagation, each mechanism is independent of the other and follows its own failure criterion. In "additive" propagation, the mechanism acts independently at the beginning but failure occurs when the combined damage reach unity. In "interacting" propagation, creep damage formation

influences transgranular cracks so that the crack propagation is changed to intergranular characteristics. It is found that in such cases the total damage D becomes less than unity [22].

High-temperature CF behaviour of the highly alloyed austenitic stainless steel, Alloy 709, with various tensile hold times and T_{max} of 650 °C and 750 °C, was investigated by Alsmadi and Murty [84]. The study results revealed that the CF life at 650 °C fluctuates due to dynamic strain ageing (DSA) during the dwell times. A Linear damage summation (LDS) was employed to construct the CF interaction diagram. The calculated creep and fatigue damage fractions resulted in data points that mostly lay below the ideal curve and some data points lying close to the ideal curve, which indicates a life limiting effect originating from CF interaction. From the microstructural investigation, an increased density of cracks and creep cavities were found at 750 °C with less dislocation density compared to testing at 650 °C.

4

Experimental- and analytical methods

In this chapter the experimental- and analytical procedures are described. The investigated materials of the research project, the ageing specifics and the phase equilibrium- and precipitation simulations are presented first. Then the mechanical test procedures are explained, followed by presentation of the different microstructural investigation methods used.

4.1 Material

This thesis consists of investigations of five commercial austenitic alloys: Sanicro 25, Sanicro 31HT, Esshete 1250, AISI 310M and AISI 304. These five alloys were chosen to represent different types of austenitic alloy candidates for critical power plant components. Sanicro 25 and AISI 310M are defined as highly alloyed austenitic stainless steels. Sanicro 31HT has high nickel content and is closer to the nickel-base alloys but is in some studies also referred to as an austenitic stainless steel. Esshete 1250 and AISI 304 are medium alloyed and the less expensive candidates. The nominal chemical composition in wt.% of these materials (marked with *) are given in Fig. 2. The materials were provided and heat-treated by Sandvik Materials Technology AB. The heat treatment specifics are given in Table. 1.

Table 1: Solution heat treatments of the investigated materials.

Alloy	Temperature [°C]	Time [min]
AISI 304	1060	15
AISI 310M	1050	10
Sanicro 25	1220	10
Sanicro 31HT	1200	15
Esshete 1250	1100	15

4.2 Ageing

The ageing of some TMF specimens used in *Paper II, III and VI*, was performed at 800 °C for 2000 hours in laboratory air before machining of the specimens. The ageing of *Paper VII*, was performed at 600 °C, 650 °C and 700 °C for 3000 hours, also in laboratory air before machining of the specimens. The ageing of the dwell-fatigue specimens in *Paper III*, was performed at 650 °C and 700 °C for 2000 hours, in laboratory air on already machined specimens. The long-term ageing of *Paper IV*, was performed at 650 °C and 700 °C for 1000, 3000, 10 000, 20 000 and 30 000 hours in laboratory air on already machined specimens.

4.3 Phase equilibrium- and precipitation simulations

The software Thermo-Calc together with the TCFE9 (Steels and Fe-Alloys) database and TC-Prisma together with the MOBFEE3 mobility database [86,

87], were used for equilibrium simulations of the precipitation process for the investigated materials of *Paper II and IV*.

4.4 Mechanical testing

In the following sections the mechanical testing is presented. The TMF testing (*Paper II, III, VI and VII*), hardness testing (*Paper II and VII*), dwell-fatigue testing (*Paper I and III*), some creep tests (*Paper I*) and some ageing (*Paper III*) were conducted at Linköping University in Linköping. Long-term ageing (*Paper II, III, IV, VI and VII*), long-term creep (*Paper I*) and impact toughness tests (*Paper IV*) were carried out at AB Sandvik Materials Technology in Sandviken, Sweden. The Vickers hardness testing was performed using Struers DuraScan G5, following ISO 6507 and ASTM E384 standards [88, 89] using a HV5 indenter configuration.

4.4.1 Thermomechanical fatigue testing

The used test procedure was strain controlled thermomechanical fatigue (TMF) and for the IP-TMF tests (*Paper II and III*) there were a 5 minute dwell time at maximum mechanical strain ($\varepsilon_{\text{mech, max}}$) and maximum temperature. The used test machine was a servo-hydraulic TMF machine from Instron with induction heating and forced air-cooling. The configuration of the test machine is displayed in Fig. 6. Before the TMF tests the machine

Figure 6: The configuration of the TMF test machine.

was carefully aligned according to "the validated code of practice" [17], to prevent buckling and other instability effects. The research of this thesis includes both IP- and OP-TMF tests with $R_\epsilon = 0$ and $R_\epsilon = -\infty$, respectively. In Fig. 7, the TMF specimen used for the testing of *Paper II, III and VI* is given. For the tests done in *Paper VII* the specimen geometry was changed (see Fig. 8) to avoid potential barrelling problems, discovered by the investigation of *Paper VI*. The temperature range used was 100 °C to 800 °C (*Paper II, III and VI*) and 100 °C to 650 °C (*Paper VII*). The heating and cooling rate was 5 °C/s. For all the TMF papers the test set-up was done according to [17], with spot welded thermocouples in the middle of the gauge length at a distance of 1 mm between each other. The strain was measured with a high-temperature extensometer with a gauge length of 12.5 mm. The thermal strain was subtracted from the measured total strain, so that the mechanical strain could be controlled as suggested by [17]. The number of cycles to failure (N_f) was defined as the point at which the amplitude stress (σ_a) decrease 10 % below the tangent line constructed at the last point of zero curvature. However, the tests were not stopped until a load drop of 60 % occurred.

Figure 7: Schematics of the original TMF specimen.

Figure 8: Schematics of the altered TMF specimen.

4.4.2 Dwell-fatigue testing

The testing (*Paper I*) was performed using an MTS servo hydraulic testing machine equipped with an Instron 8800 control system. The strain was measured by an Instron 2632-055 extensometer and the isothermal condition, at 700 °C, was controlled by an MTS 652.01 furnace. The $R_\epsilon = 0$ -type test cycle involved a strain-controlled part, dashed line in Fig. 9, and a load controlled part, solid line in Fig. 9. Strain ranges from 0.5 % to 1.0 % and dwell times, t_d , from 0 to 1800 seconds were tested. Set-up and employment of the test as well as definition of plastic strain range, $\Delta\epsilon_p$, stress range, $\Delta\sigma$, and total strain amplitude, $\Delta\epsilon/2$, were conducted according to ASTM standard E2714-3 for creep-fatigue testing [90]. The number of cycles until failure, N_f , was defined as the point at which the amplitude stress, $\Delta\sigma/2$, decrease 10% below the tangent line constructed at the last point of zero curvature, as suggested by [90].

Figure 9: Sketch of the dwell-fatigue test cycle, strain vs. time.

4.5 Microscopy

This section presents the microscopical techniques used in the research of this thesis. However, firstly the steps of the sample preparation are described which are important for both investigation- and image quality.

4.5.1 Specimen preparation

The surface of the investigated samples must be carefully prepared in order to reduce the surface roughness. This is important to be able to perform

qualitative scanning electron microscopy (SEM) techniques like backscatter electron (BSE) and electron backscatter diffraction (EBSD). Mechanical polishing according to the steps in Table. 2 were performed on all the samples before the SEM investigations.

For the scanning transmission electron microscopy (STEM), thin foils prepared from the gauge section of the tested specimens were ground to 60 μm and then punched into 3 mm discs. Then twin-jet electro-polishing was conducted with an electrolyte solution containing 10% perchloric acid and 90% ethanol, using a polishing temperature of -25 $^{\circ}\text{C}$, to receive a disc thin enough to perform transmission of electron without too much energy loss.

Table 2: Steps of the sample preparation.

Grinding paper/ polishing cloth	Grain size [μ]	Time [min]	No. papers
SiC paper, 500	30	4	2
SiC paper, 1200	15	4	1
SiC paper, 4000	5	4	3
Silk cloth	Diamond suspension, 3	8	
Woven wool cloth	Diamond suspension, 1	10	
Rayon-viscose fibres cloth	Diamond suspension, 0.25	15	
Neoprene foam cloth	Colloidal silica suspension, 0.04	5	
Neoprene foam cloth	water	1	

4.5.2 Scanning electron microscopy

The SEM techniques secondary electron (SE), backscattered electron (BSE), electron backscatter diffraction (EBSD), energy dispersive spectroscopy (EDS) and wavelength-dispersive spectroscopy (WDS) were used in the microstructural investigations of the appended papers.

In investigations of the topographical information of cracks, the SE-technique was used. Electrons emitted from the sample with very low energies (below 50eV) are considered secondary electrons (SE) and they originate from inelastic scattering on loosely bound outer shell electrons in the surface or the near surface regions of the samples (emission depth of $\approx 1\text{nm}$). This gives a shallow escape depth and the surface contrast comes from the difference of number of electrons leaving the sample at different beam locations (number component) and contrast effects from the electron travel paths after leaving the sample (trajectory component) [91–93].

In the investigations that required compositional contrast imaging, the BSE-technique was used. The contrast comes from high energy (50 eV)

backscattered electrons (BSEs), that elastically returns from the sample and are collected by the BSE detector. Larger atoms, with greater atomic number (Z) have higher probability of producing an elastic collision because of their greater cross-sectional area. The number of BSEs reaching the BSE detector is proportional to the mean atomic number of the sample. Thus, "brighter" BSE intensity correlates with greater average Z in the sample, and "dark" areas have lower average Z [91, 94]. In the investigations of this thesis, the BSE-technique was used to distinguish between different phases and precipitates, but the technique also provided topographic and crystallographic information.

The EDS technique was used for qualitative (element present) and quantitative (number of elements) chemical analysis. This was done by measuring of the energy and intensity distribution of the x-ray photon signals that are generated from the focused electron beam and specimen interaction volume beneath the specimen surface. X-ray photons that emerge from the specimen have specific energies that relate to the elements in the sample. The relative weights of the characteristic X-ray lines in each family (different energies for different transitions between the subshells e.g. K, L, M of the specific atom) are consistent and this, along with their energies, allows related peaks in the X-ray spectrum to be recognized [91, 94]. The relative intensities of the elemental peaks were evaluated using a commercial software from Oxford. Information about the composition of phases and precipitates were collected with this technique. The WDS technique was mainly used for small spot analysis when the EDS spatial resolution was too poor for accurate qualitative investigation e.g., in the case of separating tungsten and silicon peaks. In contrast to EDS, the WDS technique recognizes and separates elements by their wavelength using Bragg diffraction, instead of sorting based on energy. Bragg diffraction occurs when x-rays, with a specific wavelength (λ) similar to the spacing of the crystal lattice planes (d) undergoes constructive interference. This occurs only when the differences in the X-rays travel path are equal to an even number multiple (n) of the wavelength. When constructive interference occurs, a diffracted beam of X-rays will leave the crystal at an angle equal to that of the incident beam angle (θ) [94, 95]. In Fig. 10, Bragg diffraction is illustrated and in eq. 2 the Bragg's law is given, which defines the relation between the wavelength of the incident beam and the condition on θ for constructive diffraction to occur.

$$n\lambda = 2d\sin(\theta) \quad (2)$$

Although the data collection in WDS is slower than for EDS, as only one

Figure 10: Bragg diffraction. The lower beam is a length of $2d\sin(\theta)$ longer than the upper beam and Bragg's law is fulfilled when that distance is equal to $n\lambda$.

element can be monitored at a time in WDS while EDS can gather a spectrum of all the elements in the sample simultaneously, the spectral resolution of WDS (about 5 eV) is much higher than for EDS (above 100 eV) [91, 94].

Close to the Bragg condition, the intensity of backscattered electrons (BSEs) is strongly dependent on the crystal orientation and the effect can be used to image local misorientations, defects and strain fields near the surface of bulk materials. This technique is termed electron channelling contrast imaging (ECCI) [96, 97]. When applying ECCI for defect observation in a polycrystalline material it is not known which lattice planes leads to the observed defect contrasting and if perfect channelling conditions are achieved. However, this can be corrected for by measuring of the crystal orientation combining ECCI with EBSD [98–100].

In the research performed within this thesis, the EBSD technique was used for obtaining information on the individual orientation of the microscopic grains. The EBSD pattern is gathered by illuminating a tilted specimen with the stationary electron beam. The diffraction/channelling of the electrons causes the electrons to form very flat cones. These cones have an angle of $(90^\circ - \theta_b)$, where θ_b is the angle for Bragg reflection to occur. Because of diffraction from both back and front of the atomic planes, two pairs of cones intercept the imaging plane. These are viewed as two nearly straight lines separated by an angle of θ_b . The intersections of the different lines ("Kikuchi lines") represent zone axes and are related to a specific crystallographic direction within the crystal [91, 95, 101, 102]. EBSD software uses the Kikuchi pattern to identify the phase and the orientation of the crystal lattice in the point which generated the pattern, this process is referred to as indexing. During this process the actual Kikuchi pattern is compared to theoretical pattern and the mean angular deviation (MAD value) between these two is considered

a quality factor for the indexing process [91, 95, 103]. Position of grain boundaries are defined with a minimum crystallographic orientation between neighbouring grid points (typically 10°).

The orientation, shape and size of grains can be displayed using inverse pole figures (IPFs), which gives a two-dimensional illustration of a three-dimensional orientation relation. The IPF uses the stereographic triangle to express which crystal direction is parallel to the sample direction (surface normal, Z0). In Fig. 11a, the construction of an IPF is demonstrated. The coordinate system (cube axes in the figure) lay inside a unit sphere. In the figure the projection plane is defined by the 100 and the 010 axis (the (100) plane) which is located in the middle of the sphere. Normal vectors of the unit cells three planes of orientation (the green of $\{110\}$, red of $\{100\}$, and blue of $\{111\}$) are then drawn and the intersection points between the vectors and the unit sphere is then projected to the bottom of the sphere in a straight line. The point of intersection with the projection plane is defined as the pole of the normal of the projected plane. The same procedure is done for the other plane normals in the unit cell and then the projection plane with its projected poles is the actual IPF. In an IPF, each orientation is displayed as a single point, rather than a number of points as is the case with a pole figure (PF). IPFs is commonly used as a colour key for crystal orientation maps (COMs) of cubic materials. By assigning red, blue and green to the corners of the figure, with smooth gradation of colours in between (Fig. 11b), any given measurement (due to cubic crystal symmetry) can be related to a distinct colour in the COM [95, 104–106].

Figure 11: Illustration of stereographic projections, (a) construction of an IPF, (b) the 24 equivalent triangles for an FCC unit cell and the IPF colour key reference.

It follows that:

- If red is displayed in the sample normal COM, the face of the crystal $\{100\}$ lies parallel to the sample surface
- If green is displayed in the sample normal COM, the edge of the crystal $\{110\}$ lies parallel to the surface
- If blue is displayed in the sample normal COM, the corner of the crystal $\{111\}$ lies parallel to the surface

The microscopic investigations using EBSD, EDS, SE, WDS and BSE were performed with a HITACHI SU-70 FEG-SEM equipped with OXFORD EBSD and BSE detector. The EBSD system used the HKL software CHANNEL 5. The EBSD maps were measured with acceleration voltage of 20 kV using a working distance of 22 mm to 27 mm and step sizes of 0.5 μm to 1.5 μm . The SE and BSE images were captured using 10kV to 20 kV acceleration voltage and a working distance from 7.4 mm to 15 mm. The EDS and WDS process used 20 kV acceleration voltage and a working distance of 15 mm.

4.5.3 Transmission electron microscopy

When performing transmission electron microscopy (TEM) a beam of electrons is emitted from an electron gun and accelerated to an energy of typically 80-300 keV and it allows for imaging and chemical analysis with resolution below 1 $\text{\AA}$ [107]. As mentioned in section 4.5.1 the samples must be well prepared and thin enough (<150 nm) for transmission of sufficient electrons to form an image with minimum energy loss. Usually, the microscope can be operated in different modes. In conventional TEM, the various magnetic lenses focus the electrons into a small probe that is transmitted through the sample. Another set of lenses then forms images either by bright field (BF), dark field (DF), or lattice phase contrast. BF images are formed by the direct beam (non-diffracted beam), preventing all other beams to pass the recording system by use of the objective aperture. If the signal from a diffracted beam is allowed, a DF image is received. By adjusting the magnetic lenses in a way that the back focal plane of the lens rather than the imaging plane is focused, a diffraction pattern can be produced. In the scanning transmission electron microscopy (STEM) mode, the electron beam is focused as a fine probe which is scanned over the specimen (by scanning coils) and the transmitted intensity is recorded. The STEM mode BSE, SE as well as emitted X-rays can be collected from the sample and the images are similar to those obtained by SEM, although the resolution is much higher (around 0.1 nm) [107–109].

A FEI Tecnai G2 STEM microscope operating at 200 kV were used to investigate the deformation substructures and nano sized precipitates of *Paper V*. Images were captured using different camera lengths: high angle annular dark field images were obtained with a camera length of 140 mm, dark field with 350 mm and bright field with 2100 mm.

5

Summary and discussion of appended papers

This chapter provides a short summary of the included papers and a discussion of the results in relation to the research questions.

Paper I

Creep-fatigue interaction in heat resistant austenitic alloys

In this study the combined effect of creep and fatigue was investigated for the two austenitic stainless steels, Sanicro 25 and Esshete 1250. To simulate this effect, strain controlled low-cycle fatigue tests with load-controlled dwell times (dwell-fatigue tests) were used. The tests had variable strain amplitudes and dwell times, and the test temperature was 700 °C. Hardening/softening curves (stress vs. cycles/time), hysteresis curves (stress vs. strain) and a creep-fatigue interaction diagram, were constructed and analysed. With the mechanical response analysis and findings by previous investigations, estimated damage initiation mechanisms were discussed. It was found that both the investigated materials suffered from interaction damage by creep and fatigue for most of the test configurations. Different types of estimated initiation- and propagation damage were active depending on the duration of the dwell time and the amplitude of the strain. Of the two investigated materials, the higher alloyed Sanicro 25 suffered higher stresses, but showed longer dwell-fatigue life.

Paper II

Thermomechanical Fatigue Behaviour of Aged Heat Resistant Austenitic Alloys

This study focuses on the influence of ageing on the TMF behaviour for the three austenitic stainless steels Sanicro 25, Esshete 1250 and Sanicro 31HT. The aim was to investigate the effect of prolonged microstructural evolution, simulated by pre-ageing at 800 °C for 2000 hours. The TMF tests were performed with IP loading, temperature ranges of 100 °C to 800 °C and dwell times of 5 minutes at maximum temperature. The role of precipitation and damaging mechanisms were investigated using the SEM techniques BSE, EDS and EBSD. The investigation showed that all the tested materials experienced detrimental effects from pre-ageing because of the deterioration of the microstructure by precipitation, which foremost enabled oxidation assisted cracking and reduced the creep resistance. Fast fracture was then induced along the embrittled grain boundaries leading to fast failure.

Paper III

Fracture and Damage Behaviour in an Advanced Heat Resistant Austenitic Stainless Steel During LCF, TMF and CF

This work summarises and discusses fracture and damaging behaviours of Sanicro 25 exposed to various test conditions. These involved dwell-fatigue tests, LCF tests and two types of IP-TMF tests. The cyclic behaviour for the different test conditions was analysed and compared. The results revealed that Sanicro 25 subjected to TMF- and CF conditions suffered shorter cyclic life compared to the LCF condition. The mechanical test data indicated that the cyclic life of the TMF- and CF condition were influenced differently by pre-ageing. However, the ageing temperatures were lower in the CF case and there were too few test data points to be able to establish a definite trend. During the TMF test condition, Sanicro 25 showed prominent cyclic hardening and this effect was further promoted by higher strain ranges, lower dwell times, lower temperature ranges and pre-ageing.

The microscopic investigations of the fracture and damage behaviour showed evidence of oxidation assisted cracking in TMF test specimens and damage by void formation in CF test specimens. For TMF and CF conditions, EBSD mapping showed crack initiation in multiple grain boundaries close to the specimen surface.

Paper IV

Structural Integrity and Impact Toughness of Austenitic Stainless Steels

This study investigates the effect of long-term ageing on the toughness of the three austenitic alloys AISI 304, AISI 310M and Sanicro 31HT. The samples were aged at 650 °C and 700 °C in air-environment for 1000, 3000, 10 000, 20 000 and 30 000 hours before the impact toughness tests. All the investigated materials showed a severe impact toughness decrease after ageing compared to their unaged reference. For AISI 304 and AISI 310M, evidence of the precipitates $M_{23}C_6$ and σ -phase was found during the microscopic investigation, which was supported by Thermo-Calc simulations with the TCFe9 (Steels and Fe-Alloys) database. TC-Prisma with the MOBFE3 mobility database predicted increased volume fraction and coarsening of these precipitates with increasing ageing time. This was considered to be the reason for the decreased impact toughness. The decrease was not as prominent in

the case of Sanicro 31HT and this was linked to the low number and origin of detrimental precipitates predicted and found in the microstructure.

Paper V

Microstructural evolution during high temperature dwell-fatigue of austenitic stainless steels

This paper provides an in-depth microstructural investigation of the two austenitic stainless steels whose mechanical CF response were evaluated in *Paper I*. The microscopic techniques EBSD, EDS, WDS and STEM were used to investigate the crack propagation characteristics, deformation mechanisms and precipitation processes. The materials displayed different crack propagation and CF interaction behaviour: Sanicro 25 exhibited interchanging intra- and intergranular crack characteristics which appeared to involve dynamic recrystallization (DRX) of the crack tip plastic zone. Esshete 1250 solely experienced fracture along the grain boundaries, while the interior of the grains showed high plasticity in the form of pronounced fatigue induced slip bands. The interaction of the slip bands and the grain boundary precipitates was considered to enhance the intergranular damage process.

Paper VI

Influence of ageing on thermomechanical fatigue of austenitic stainless steels

The OP-TMF behaviour of three pre-aged austenitic stainless steels was investigated in this paper, where the IP-TMF tests were used as references. The TMF tests were strain controlled with a temperature range of 100 °C to 800 °C and only lower mechanical strain ranges were considered ($\varepsilon_{\text{mech}} \leq 0.4\%$). All the OP-TMF specimens suffered inhomogeneous deformation (barrelling effect) and most of them failed by thermocouple detachment instead of the load drop criteria and this phenomenon became the main focus of the study. To investigate if the test specimen geometry was contributing to the change of the diameter with cyclic load i.e., the barrelling, a simplified finite element analysis (FEA) was performed. The same characteristic inhomogeneous deformation was simulated by the linear-elastic-ideal-plastic modelling of the test specimen, subjected to an OP-TMF cycle but with a temperature range of 0 °C to 700 °C (since no data at 800 °C were available) and $\Delta\varepsilon_{\text{mech}} = 0.6\%$

(since no plastic deformation occurred for lower strain ranges at 700 °C). The FEA showed that the plastic strain was concentrated in the "waist" rather than the middle of the deformed specimen, where the plastic strain of the "waist" was almost double that of the plastic strain in the middle region. LAGB density calculations were then applied on a Sanicro 31HT (that had failed by the load drop criteria) to check the accuracy of the FEA and these two agreed reasonably well. In addition, no barrelling effect could be distinguished before 500 OP-cycles when the LAGB density calculations were performed on the fixed OP-TMF test specimens.

Paper VII

High temperature fatigue of aged heavy section austenitic stainless steels

This study is dedicated to the behaviour of Sanicro 25 during OP-TMF testing to evaluate if the material is a suitable candidate for heavy section components in biomass-fired power plants. The test procedure was strain-controlled with a temperature range of 100 °C to 650 °C. Both unaged and aged specimens were tested to simulate service degradation resulting from long-term usage. The aged Sanicro 25 specimens suffered increased plastic straining and shorter TMF life compared to the unaged specimens. The SEM techniques EBSD and EDS showed that the difference in TMF life of the two test conditions was attributed to an accelerated microstructural evolution that provided decreased effectiveness for impeding dislocation motion. Ageing did not affect the OP-TMF life of the reference material, Esshete 1250. However, the structural stability and the resistance for cyclic deformation were greatly reduced due to coarsening and cracking of the strengthening niobium carbide precipitates. Comparing the two tested materials, Sanicro 25 showed higher structural stability and longer cyclic life during OP-TMF testing.

Discussion

This thesis has the overall aim to give a better understanding of the influence of detrimental high-temperature cyclic conditions on the material candidates for critical components in boiler sections of next generation biomass power plants. In section 1.4 the overall aim was expressed as three research questions, RQ1, RQ2, and RQ3. RQ1 and RQ2 are partly answered by the summary of the appended papers above. Where the response and behaviour of the investigated materials subjected to pre-ageing (*Paper II, III, IV, VI and VII*), CF conditions (*Paper I, III and V*) and TMF conditions (*Paper II, III, VI and VII*) are presented. To be able to address RQ3, an overall assessment of the research performed within this thesis must be considered. Below, the papers in Part II are related to the research questions to discuss the success of fulfilling the goals of this thesis more easily.

- *RQ1: During TMF and CF conditions, what are the high-temperature fatigue response and behaviour of the investigated materials?*

During dwell-fatigue conditions, both the medium alloyed Esshete 1250 and the highly alloyed Sanicro 25 experienced CF interaction damage, which is displayed by the CF interaction diagram in *Paper I*. However, the interaction damage characteristics were different. Sanicro 25 exhibits high stress levels foremost because of the homogeneously spread coherent copper precipitates and incoherent niobium carbides/nitrides. In addition, the occurrence of Z-phase, chromium carbides and solid solution strengthening elements e.g., tungsten, contribute to effective impeding of dislocation motion and induce a prominent hardening effect. The most favourable crack path seems to be locally determined as described in *Paper V*, which involved a DRX process of the crack tip plastic zone that caused the interchanging intra- and intergranular crack propagation. In contrast to what was predicted in *Paper I*, the microscopic investigation of *Paper V* showed that Esshete 1250 solely experienced fracture along the grain boundaries. This CF behaviour was owing to the interaction of the plastically induced slip bands and the grain boundary precipitates that was considered to enhance the intergranular damage process.

During TMF, the previously discussed hardening effect of Sanicro 25 appears to apply in the both the OP- and IP condition. However, in conformity with the theory in section 3.2 there is a notable difference of the high temperature behaviour for these two conditions. During OP-TMF, Sanicro 25 suffers higher stresses and increased hardening because of tension at lower temperature and as a result, fatigue is the dominant damaging mechanism in the

bulk and oxidation assisted initiation cracking at the surface. Cracks initiate and propagate transgranularly, assisted by slip bands, oxidation and cracking or decohesion of brittle precipitates. The IP condition instead induces intergranular crack propagation and initiation, because of creep damage formed in the grain boundaries and together with the oxidation assisted propagation process described in *Paper II* it makes for a more favourable failure mode. The IP-TMF damage and failure mechanisms of Sanicro 25 are similar for the other two investigated materials, but with an accelerated failure process due to less effective strengthening mechanisms against plastic deformation and lower resistance to grain boundary creep damage. Considering the highly alloyed Sanicro 31HT, only the IP-TMF behaviour was investigated, and the high temperature strengthening mechanisms involved solid solution and precipitation of titanium, aluminium and carbon. As discussed in *Paper II*, evidence of oxidation assisted cracking was not found after IP-TMF testing and this was most likely due to the high amount of chromium and nickel which suppress the oxidation process. However, for Esshete 1250 this is a life limiting mechanism which is attested by the findings of *Paper II*. This is an effect of using manganese additions and lower nickel content for achieving the stable austenitic phase to reduce alloy cost, but at the loss of oxidation resistance. High temperature strength and hardening attains from fine homogeneously spread niobium carbides, but with extended IP- and OP-TMF cycling they seem to coarsen, thus allowing for increased plastic straining and eventually crack or lose its cohesion, promoting crack propagation.

- *RQ2: How will prolonged microstructural degradation by pre-ageing influence the high-temperature behaviour?*

As given in the review of papers above, the influence of ageing on the impact toughness of two highly alloyed- and one medium alloyed austenitic alloy was investigated in *Paper IV*. For all the materials, the impact energy quite severely decreased even after short time ageing, comparing the unaged samples to aged samples after 1000 hours. An increase in ageing time and higher ageing temperature (650 °C to 700 °C) lowered the impact energy and for long term ageing (up to 30 000 hours) only Sanicro 31HT still exhibited a reasonable level of impact toughness (~ 100 J) and showed evidence of maintained ductility in the form of fracture surface dimples. The highly alloyed AISI 310M and the medium alloyed AISI 304 owed their loss in impact toughness and structural integrity to excessive grain boundary precipitation of detrimental phases like σ -phase and $M_{23}C_6$. Similarly, the high temperature cyclic properties of austenitic alloys during TMF are negatively affected

by pre-ageing which is demonstrated in *Paper II, III, VI and VII*. For the IP-TMF condition (*Paper II*) pre-ageing produces an embrittling effect for all the investigated materials. For Sanicro 31HT, this effect was due to excessive grain boundary precipitation of titanium and chromium carbides. The effect was most detrimental for Esshete 1250, where it considerably lowered the structural integrity so that tests with equivalent $\Delta\varepsilon_{\text{mech}}$ to the unaged tests, result in immediate fracture. The microscopic imaging of the performed pre-aged tests with lower $\Delta\varepsilon_{\text{mech}}$ show coarsening of niobium carbides, which also precipitate on the grain boundaries and make them less ductile and susceptible for creep damage. With less homogeneously spread niobium carbides the resistance for plastic deformation inside the grains decreases. With further straining, the niobium carbides crack or lose their cohesion and with creep- and oxidation assisted grain boundary cracking due to sensitisation (depletion of Cr and Mo), the IP-TMF life is significantly lowered. For OP-TMF, pre-ageing of Esshete 1250 specimens do not lower the cyclic life much, but the structural integrity is plainly decreased as evident by the increased plastic strain range, decreased hardness and lowered hardening effect. The pre-ageing of the OP-TMF Sanicro 25 specimens resulted in negative microstructural evolution in the form of intra- and intergranular precipitation of needle shaped Laves phase, chromium carbides and coarsening of Z-phase and niobium carbides. For the IP-TMF condition, the embrittlement of the grain boundaries together with the enhanced oxidation process, lowered the TMF life significantly compared to the unaged specimens. The life was also reduced for the OP-TMF condition (*Paper VII*), but this was due to the decreased intragranular ability for impeding dislocation motion, which enhanced the fatigue govern crack propagation. Furthermore, from the investigations of *Paper VI* the reduction in structural integrity of pre-aged austenitic stainless steel specimens can cause inhomogeneous deformation e.g., barrelling, if no prior analysis of specimen geometry is conducted. This knowledge was later used in *Paper VII* to exclude the influence of barrelling due to unfavourable dimensions of the specimens.

- *RQ3: For the investigated austenitic alloys, how will the different operating condition influence their performance? Given the selected research methods what important properties should future biomass power plant boiler materials possess?*

Paper III investigates Sanicro 25 subjected to the different high temperature loading conditions LCF, CF and TMF. It was found that both the CF- and TMF conditions are shifted to the lower fatigue life compared to the LCF

condition in the Coffin-Manson fatigue life curve. That the conditions with the addition of dwell time and thermal cycling are more detrimental is of course easy to anticipate. The two TMF experiments show similar cyclic life but different plastic strain for the same number of cycles, where the TMF tests with greater temperature range and added relaxation at maximum temperature show lower plastic strain for equal number of cycles. One reason for this behaviour is that the relaxation reduces the hardening effect but induce creep damage and studying their cyclic stress-strain curves in *Paper III*, the stress is higher to inflict the same plastic strain for the test without the dwell-time and lower temperature range compared to the other TMF tests. The difference in cyclic life (at equal plastic strain) is then understandable, given that the test set-up is strain controlled which yield higher stresses with higher resistance to straining, and this accelerate other damaging mechanisms like oxidation assisted cracking and creep. The OP-TMF condition was not included in *Paper III* which makes it hard to directly analyse its influence compared to the other loading conditions, even though the material behaviour was discussed for RQ1. The OP-tests of *Paper VII* were performed with lower maximum temperature to avoid barrelling (see *Paper VI*), no dwell time, but higher $\Delta\varepsilon_{\text{mech}}$ to avoid unnecessary long test times. The lower maximum temperature of the OP condition increases the cyclic life compared to the IP condition, however it is believed that the much longer cyclic life of the OP test should still surpass the positive influence that lowering of the maximum temperature would have on the IP tests, making the IP condition the more detrimental condition. Other TMF studies [79–81] comparing the OP- and IP-TMF supports that the IP condition is more detrimental for austenitic stainless steels at high temperatures.

Given that TMF is the most detrimental investigated loading condition and that the behaviour of the investigated austenitic stainless steels during such conditions show foremost life limiting damage originating from high temperature plastic straining, creep and oxidation. Then the straightforward recommendation of important properties for boiler material to be used in future power plants should be resistance to these mechanisms. However, the reality of tailoring an alloy to enhance resistance of one mechanism will influence another and there is also often an economical goal to be met, which makes the process quite complex. All the investigated materials are tailored for use at high temperature cyclic conditions but with various success to endure the enhanced future power plant conditions with increased temperature and pressure, that have been investigated in this thesis. The higher alloyed austenitic stainless steels show the best overall performance, however as discussed previous, the increased alloying for preventing oxidation (higher levels of Cr and Ni) and creep (addition of N, Nb, Mo, W and C) influence

the high temperature long-term microstructural evolution which enhance the fatigue driven plasticity. However, according to the investigations of the appended papers the drawbacks of other creep- and oxidation resistance tailoring were shown to be greater. An example of this is Esshete 1250, where reducing the nickel content (economical reason) and obtain the austenitic phase by addition of manganese, instead reduces the oxidation resistance at the investigated high temperature conditions. Furthermore, the addition of molybdenum to enhance the creep and oxidation properties by solid solution strengthening can lead to precipitation of the detrimental σ -phase. This was seen for some of the alloys in *Paper IV*, which greatly reduced their structural integrity and impact toughness after long term ageing. Another important consideration is the role of the stabilising elements such as niobium, titanium and vanadium and their role of tying up carbon to e.g., avoid sensitisation.

6

Conclusions and outlook

In this chapter, the conclusions from the research performed within this thesis are presented. In addition, suggestions of further research that could be performed based on this PhD thesis are given.

The presented research within this doctoral thesis investigates the high-temperature behaviour of austenitic stainless steels focusing on the damaging mechanisms during thermomechanical fatigue (TMF) and creep-fatigue (CF) interaction. In addition, the influence of prolonged microstructural degradation was evaluated for the investigated materials by pre-ageing.

During dwell-fatigue testing, it was found that the investigated austenitic stainless steels suffered from creep-fatigue interaction damage and it was found that depending on the material, different damage behaviour and crack propagation characteristics were active. A highly alloyed austenitic stainless steel showed interchanging intra- and intergranular crack characteristics which appeared to involve dynamic recrystallization (DRX) of the crack tip plastic zone. A medium alloyed austenitic stainless steel solely experienced fracture along the grain boundaries, while the interior of the grains showed high plasticity in the form of pronounced fatigue induced slip bands. The interaction of the slip bands and the grain boundary precipitates was considered to enhance the intergranular damage process. The thermomechanical fatigue (TMF) investigations showed that austenitic stainless steels experience detrimental effects from pre-ageing because of the deterioration of the microstructure, which foremost enhanced oxidation assisted cracking, induced grain boundary embrittlement and reduced the creep resistance. Cracking was induced along the grain boundaries leading to fast fracture for the in-phase (IP) condition and transgranular propagating cracks for the out-of-phase (OP) condition. The OP-TMF propagation was assisted by slip band formation, cracking or decohesion of brittle precipitates and by oxidation assisted cracking, while the IP-TMF propagation was enhanced by the embrittlement of the grain boundaries due to excessive precipitation. Important properties that future power plant material should possess are resistance to life limiting damage originating from high temperature plastic straining, creep, creep-fatigue interaction and oxidation. For the investigated austenitic stainless steels, thermomechanical fatigue was the most detrimental high temperature condition which involved most of these mechanisms.

The work of present thesis involved analysis of the material behaviour at the identified most detrimental and not fully understood, future biomass-fired power plant operating conditions. Real life component testing would be the next step to further investigate the long-term reliability of highly alloyed austenitic stainless steels used for critical components. Comparisons to even more various reference materials that are commonly used in present power plants, such as different new highly alloyed ferritic stainless steels and nickel-base superalloys would be of great interest. To receive a broader picture of the damaging behaviours of the materials during these conditions, investi-

gations including other important high temperature properties like fatigue crack propagation resistance are valuable. In this context the investigations could also include the behaviour of welded components, which are common in power plants. Future investigations would also benefit from including interrupted or more advanced in-situ testing to assess and confirm the progression of the discovered detrimental mechanisms by present work.

Finally, the research of this thesis has contributed to deeper understanding of the mechanical- and microstructural behaviour of austenitic stainless steels during high temperature cyclic conditions, which will provide useful knowledge for the development of the next generation materials used for components of future biomass-fired power plants.

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Part II

Papers Included

Papers

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Linköping Studies in Science and Technology, Dissertation No. 2140, 2021
Division of Engineering Materials, Department of Management and Engineering

Linköping University
SE-581 83 Linköping, Sweden

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