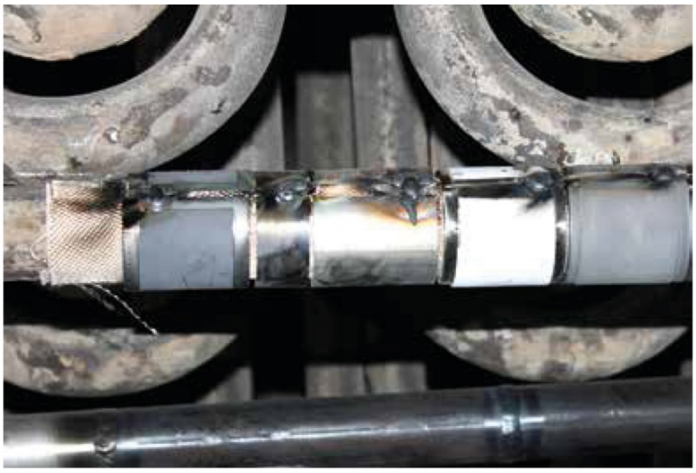
MECHANICAL BEHAVIOURS OF AUSTENITIC STAINLESS STEELS IN HIGH-TEMPERATURE ENVIRONMENTS

KME-701



CONSORTIUM MATERIALS TECHNOLOGY
for thermal energy processes

KME
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Mechanical behaviours of austenitic stainless steels in high-temperature environments

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Preface

The project has been performed within the framework of the materials technology research programme KME, Consortium materials technology for thermal energy processes, period 2014-2018. The consortium is at the forefront of developing material technology to create maximum efficiency for energy conversion of renewable fuels and waste. KME has its sights firmly set on continuing to raise the efficiency of long-term sustainable energy as well as ensuring international industrial competitiveness.

KME was established 1997 and is a multi-cliental group of companies over the entire value chain, including stakeholders from the material producers, manufacturers of systems and components for energy conversion and energy industry (utilities), that are interested in materials technology research. In the current programme stage, eight industrial companies and 14 energy companies participate in the consortium. The consortium is managed by Energiforsk.

The programme shall contribute to increasing knowledge within materials technology and process technology development to forward the development of thermal energy processes for efficient utilisation of renewable fuels and waste in power and heat production. The KME goals are to bring about cost-effective materials solutions for improved fuel flexibility, improved operating flexibility, increased availability and power production with low environmental impact.

KME's activities are characterised by long term industry and demand driven research and constitutes an important part of the effort to promote the development of new energy technology with the aim to create value and an economic, environmentally friendly and long term sustainable energy society.

The industry has participated in the project through own investment (60 %) and the Swedish Energy Agency has financed the academic partners (40 %).

Bertil Wahlund, Energiforsk

Abstract

Increased knowledge about the mechanical performance of virgin and aged austenitic alloys for future application in the area of power generation has been achieved by using several mechanical testing methods, such as thermo-mechanical fatigue, creep/fatigue interaction and toughness tests, of austenitic alloys at elevated temperature and room temperature. The results show that the structural development during testing is affecting properties differently depending on alloy composition. This knowledge can lead to improvements of the performance of the materials.

Sammanfattning

Den globala ökningen i energianvändning och därmed sammanhängande ökning i CO_2-utsläpp vid förbränning har skärpt kraven på energileverantörer och användare att i större utsträckning använda hållbara biobränslen samt att höja verkningsgraden genom att höja tryck och temperatur i energiomvandlingsanläggningarna. Sådana omställningar leder alltid till någon form av problem med koppling till materialegenskaper.

Dessutom ser man i framtiden tydliga tecken på att behovet av reglerkraft för att kompensera för väderbaserade energianläggningar som sol och vindkraft, vilket leder till att anläggningar måste stoppas och startas betydlig oftare än nu, vilket skapar behov av provningsmetoder som tar hänsyn till cykliska mekaniska och temperaturbaserade laster. Detta tillsammans med att framtidens material måste tåla högre temperaturer och ökad miljöpåverkan innebär att befintliga austenitiska material måste förbättras inte bara genom en ökning av andelen nickel och andra verksamma legeringselement utan även genom att generera ny kunskap om hur de mekaniska egenskaperna påverkas av detta.

I detta projekt har inverkan av långtidsåldring, cyklisk och kombinerad cyklisk och statisk belastning vid höga temperaturer på deformation, skadeuppbyggnad och brottegenskaper studerats. Kopplingar till utvecklingen av mikrostrukturer har undersökts med ökad förståelse för hur mekaniska egenskaper och prestanda påverkas. Denna kunskap kan leda till förbättring av materialens prestanda. Så snart inverkan av legering och långtidsvärmepåverkan på de mekaniska egenskaper och prestanda är till fullo klarlagd kan nya och bättre material för användning i olika applikationer utvecklas.

Projektets mål har till en acceptabel nivå kunnat nås och samverkan med industrin får i detta projekt anses vara mycket tillfredsställande och antalet publikationer är åtta samt en doktorsavhandling.

Nyckelord: Biomasseldade kraftvärmeverk, Termomekanisk utmattning, Kryp-utmattnings interaktion, Långtidsåldring, Austenitiska rostfria stål, Deformation och Skada.

Summary

A global increase in use of energy connected to an increase in CO_2 emission during combustion has increased the demand on energy producers and consumers to use sustainable biomass fuels and to increase efficiency by increasing temperature and pressure in energy conversion plants. This often leads to problems connected to materials properties.

There is also a need for the future to introduce plants with the purpose of regulating the systems and compensate for fluctuations caused by weather based sustainable energy production like solar and wind energy with a large number of start and stop cycles. This is increasing the need for new testing methods taking into account variation in temperature and load. This together with the trend of increasing temperature and corrosiveness in the environment means a demand for new austenitic materials improved by higher Ni-content and other minor additions alloying elements.

In this project, the influence of long-term ageing, cyclic and combined cyclic and static loading at high temperatures on deformation, damage development and fracture properties are studied. Couplings to the development of microstructure has been investigated, resulting in an increased understanding of how mechanical properties and performance are affected. This knowledge can lead to improvements of the performance of the materials. As soon as the influence of alloying and treatment on these mechanical properties and performance is wholly understood new materials with better properties can be developed.

The goal fulfilment is acceptable and the cooperation with the industry partner is to be considered extremely good. The number of publications produced within the frame of this project is eight and one PhD thesis.

Key words: Biomass-fired power plants, Thermo-mechanical fatigue, Creep-fatigue interaction, Long-term ageing, Austenitic stainless steels, Deformation and Damage.

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1 Introduction

1.1 BACKGROUND

Biomass is the largest global contributor to renewable energy and has a great potential to expand in the production of heat and electricity [1]. It is a sustainable fuel because it gives no net contribution of CO_2 to the atmosphere and it can be considered endless [1, 2]. However, the global increase in energy consumption and the increase in emissions of greenhouse gases (e.g. CO_2) causing global warming, make needs for both an increase in energy production and a reduction of greenhouse gas emission [3, 4]. One way to accomplish both needs is to increase the efficiency of biomass power plants, which could be reached by increasing temperature and pressure in the boiler sections [5]. Thus, the requirement of more energy production is met and since biomass has no net contribution of CO_2 to the atmosphere less emissions of greenhouse gases is the result.

The materials used for future biomass power plants with higher efficiency are required to display improved properties such as higher yield strength, creep strength and high-temperature corrosion resistance. The performance of austenitic stainless steels at these elevated temperatures is not yet fully understood [6, 7].

For instance, the degradation mechanism acting on austenitic alloys at elevated temperatures called stress relaxation cracking (SRC) is not fully understood. It may result in failures in components after short time of operation despite of the use of approved materials [8, 9]. Standardised tests at ambient- or high temperatures like creep-, tensile-, fracture toughness- or fatigue tests have not been successful in predicting the failures. Examples of SRC have been reported in chemical process industry for example by van Wortel [8], and the alloys treated are also used in boilers. Common denominators for the failures experienced in applications are the presence of residual stresses from either cold forming like for example bending, or from welding. The operating metal temperature is generally between 550 °C and 750 °C, and the alloys used have shown a poor ductility also recognised in low creep ductility at these temperatures [8, 9]. It is reported that SRC can be avoided by post-weld and post-bend heat treatments [10], however these actions may be costly and sometimes even impossible to perform for geometrical reasons. There is a need for an efficient method to evaluate SRC to increase the knowledge about SRC and also supporting material design to prevent SRC. A first step to achieve such a method has been taken in the preceding of the KME 501 project. However, more work has to be made to reach a useful and efficient method [11].

Since the life time of a power plant is expected to be 30 years or more [12], materials with better long term high-temperature performances, as safety and structural integrity [13], are desirable. More knowledge is needed in order to verify that austenitic stainless steels are the long-term solution for power plants with increased steam temperatures and more corrosive environments. Biomass power plants are supposed to withstand many start and stop cycles to maintain efficiency in the global energy and electricity network. This advocates for investigations of how cyclic operation conditions influence the high-temperature performance of austenitic stainless steels.

One way to investigate the cyclic high-temperature properties of the materials used in new more efficient biomass power plants is to study the thermomechanical fatigue (TMF) behaviour [14]. It is very likely that an SRC-type of failure mechanism will be

active during TMF provided that the dwell time in the IP-TMF tests is sufficiently long. However, this is a topic where very little research has been conducted [15].
Other suggested methods to evaluate high-temperature behaviour of advanced heat resistant materials is very slow strain rate testing (SSRT) which can lead to similar analysis as creep tests at high stresses [16]. The advantage of SSRT compared to regular creep testing is that SSRT are much faster, and in the early stage of material development this could be a large benefit if the method gives reliable and appropriate data. Especially if modelling and estimation of life could be based on SSRT data instead of or as a complement to creep test data. In KME 501 damage and fracture mechanisms have been identified for a range of alloys and conditions, as varied strain rates and temperatures [11, 17, 18, 19, 20] and data for modelling have been established [11].

This project is a continuation of KME 501 [11] and KME 521 [15] and the results from this project can be used for improving the design of new and more advanced components used in energy conversion systems based on biofuel. Better knowledge of thermal fatigue and degradation will lead to increased safety and longer component life. The results will also be an important basis for proper material design and selection of alloys for high-temperature environments.

1.2 DESCRIPTION OF THE RESEARCH FIELD

The research project is mainly concerned with two different groups of austenitic alloys, austenitic stainless steels and nickel-based alloys. The materials are normally used in a wide variety of applications ranging from room temperature aqueous applications to high temperatures in corrosive environments. The most advanced application are super heater tubes in power plants. Some of the alloys are used in the biomass power plants of today and some are potential materials for the next generation biomass power plant [6].

Both groups of austenitic alloys show good mechanical and chemical properties at the operation temperatures of today's biomass power plants [6]. However, the materials used for the future biomass power plants with higher efficiency are required to display improved properties as higher yield strength, creep strength and high-temperature corrosion resistance. The performance of austenitic stainless steels at these elevated temperatures is not fully understood, but the nickel-based alloys are already operating under such conditions in other applications and the group of nickel-based alloys is a possible option [6, 7]. The nickel-based alloys are more expensive than the austenitic stainless steels and the austenitic stainless steels are therefore an interesting option to investigate as a material for the future biomass power plants. The austenitic stainless steels are of main concern in this work and the nickel-based alloys are mainly acting as reference materials.

1.3 RESEARCH TASK

One of the main objectives for KME during the program period 2014-2017 is improved operation flexibility, and the option of cyclic operation of thermal energy processes. Investigation of opportunities and obstacles for greater steam data corresponding to the long-term ambition of increasing power plant efficiency which means at minimum an increase of 40 °C to 50 °C in steam temperature for biomass power plant is another goal for this program period. To avoid cracking or failure due to the increased steam data and cyclic operation conditions, studies are necessary to establish the most detrimental mechanism for the mechanical service life of the materials in relation to the new demands. However, the temperature limit for long-term application of the present ferritic steels in steam power plants has been reached and new material groups needs to be explored for this purpose. The need for more corrosion resistant material also increases with the introduction of biofuel and renewable waste fractions and for this purpose austenitic materials can be an attractive alternative. In general, austenitic materials have a higher coefficient of thermal expansion and lower thermal conductivity compared to ferritic steels. As a consequence, higher thermal stresses will develop in austenitic materials compared to ferritic steels for a given thermal cycle. This means that better knowledge of the thermomechanical fatigue behaviour of stainless steels are needed in order to verify that these materials are a long-term solution for power plants with increased steam temperatures and more corrosive environments. The results from this project can be used for improving the design of new and more advanced boilers for biofuel. Better knowledge of thermal fatigue and degradation mechanisms as e.g. stress relaxation cracking, will lead to increased safety and longer component life times. The results will also be an important basis for proper material selection of more corrosion resistant alloys.

1.4 GOAL

Increase of the efficiency of a biomass power plant is obtained mainly by increasing temperature and pressure. The materials used will suffer from a tougher environment resulting in safety and reliability problems. The main purposes of this project are to evaluate the mechanical behaviours for structure safety and integrity analysis, namely:

a) To evaluate the creep and LCF interaction diagram of the boiler materials for material selection and integrity analysis since the boiler materials can undertake both creep and low cycle fatigue during the service.

b) To evaluate the structure stability and the toughness of the materials after long term service at a temperature higher than 650°C and service time longer than 20000 hours for safety analysis.

c) To evaluate thermo-mechanical fatigue properties of the boiler materials for safety and life evaluation since the power plants can start/shutdown quite often during service for energy saving and flexibility purposes in the future.

d) To evaluate the stress relaxation cracking behaviour of the boiler material. It is a critical problem for some boiler materials.

1.5 PROJECT ORGANISATION

The project has been run in close cooperation between Linköping University (LiU) division of Engineering materials and Sandvik Materials Technology (SMT) together with Sandvik Heating Technology (SHT). All activities from project planning, experiments and publishing have been shared activities in most cases. The project group have been consistent of Sten Johansson (LiU), project leader from September 2014 until June 2016, Mattias Calmunger (LiU), PhD student from September 2014 until December 2015 and project leader from July 2016 until April 2018, Guocai Chai (SMT/LiU), Jan Högberg (SMT), Magnus Olaison (SMT), Johan Moverare (LiU) and Hugo Wärner (LiU). Hugo was employed as PhD student within the project in July 2016. The reference group has included local participants and external members, Bo Jönsson (SHT), Edgardo Coda (Foster Wheeler) and Rikard Norling (Swerea KIMAB).

SMT has contributed with 3.968 Mkr and SHT with 0.340 Mkr as industrial in-kind, and the total budget contribution from Swedish Energy Agency (SEA) to LiU is 2.872Mkr.

2 Materials and methods

2.1 MATERIALS

Below the tested materials are presented, not all materials have been used in all investigations. To remove different kinds of pre-deformation, e.g. material manufacturing, all the tested materials were solution heat treated according to table 1. Haynes 282 was also heat treated by using a two-stage stabilizing and ageing treatment at 1010 °C for 2 hours and 788 °C for 8 hours. Seven austenitic stainless steels (Esshete 1250, AISI 304, AISI 310, AISI 316L, AISI 347H, Sandvik Sanicro™ 25 (Sanicro 25) and Sandvik Sanicro™ 28 (Sanicro 28)) and three nickel-based alloys (Alloy 617, Alloy 800HT/Sandvik Sanicro™ 31HT (Sanicro 31HT) and Haynes 282) have been used in the conducted experiments. The nominal chemical composition in wt.% for each alloy is presented in table 2. These commercial heat resistant austenitic alloys represent a range of materials from relatively low alloyed stainless steels (e.g. AISI 304) to nickel-based superalloys (e.g. Haynes 282) and the relatively new group of relatively high alloyed stainless steels (e.g. Sanicro 25).

Table 1: Solution heat treatments.

Alloy	Temperature [°C]	Time [min]
Esshete 1250	1100	15
AISI 304	1060	15
AISI 310	1050	10
AISI 316L	1050	10
AISI 347H	1050	10
Sanicro 25	1250	10
Sanicro 28	1150	15
Alloy 617	1175	20
Sanicro 31HT	1200	15
Haynes 282	1100	120

Table 2: Chemical composition in wt.% of the austenitic alloys, B = balance.

Alloy	C	Mn	Cr	Ni	Mo	Cu	Co	Nb	Fe
Esshete1250	0,1	6,3	15	9,5	1,0	-	-	1,0	B
AISI 304	0,02	1,2	18,3	10,3	-	0,3	-	0,01	B
AISI 310	0,05	0,84	25,43	19,21	0,11	0,08	-	-	B
AISI 316L	0,04	1,7	17,0	12,0	2,6	-	-	-	B
AISI 347H	0,06	1,7	17,5	11	-	-	-	>0,6	B
Sanicro 25	0,07	0,47	22,33	24,91	0,24	2,95	1,44	0,52	B
Sanicro 28	0,02	1,83	27,02	30,76	3,39	0,9	0,09	-	B
Alloy 617	0,06	0,02	22,53	B	9,0	**Ti** 0,46	12,0	**Al** 0,94	1,1
Sanicro 31HT	0,06	0,5	20,32	30,06	-	**Ti** 0,52	0,03	**Al** 0,47	B
Haynes 282	0,06	-	19,6	B	8,7	**Ti** 2,2	10,3	**Al** 1,5	0,5

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2.2 METHODS

This section presents the test methods used within this project.

2.2.1 Long term ageing

The long term ageing before the toughness test was performed at 650 °C and 700 °C for 1 000, 3 000, 10 000, 20 000 and 30 000 hours in laboratory air on already machined specimens.

The long term ageing before the TMF test was performed at 800 °C for 2 000 hours in laboratory air on already machined specimens.

2.2.2 Toughness testing

The impact toughness tests were performed using the Charpy V method according to ISO 14556 standard. Samples with a dimension of 10x10x55mm and V-type notch were used. The impact toughness testing was performed at room temperature (RT). Two to three specimens from each ageing condition and non-aged specimens were tested.

For crack tip opening displacement (CTOD) testing, single edge notched bend (SENB) specimens with a dimension of 90x20x10 mm were used. A sharp crack front was initiated with a K-decreasing process by a pre-cracking fatigue process according to ASTM E 647. The initial stress intensity factor range was 26 MPa√m, and the stress ratio $R=P_{min}/P_{max}$ was 0.1. The test was stopped when a crack length of 10 mm was detected. The CTOD test at RT was performed according to BS 7448:1991 Part 1 using the Instron software K_{IC}/CTOD and a constant strain rate of 0.01 mm/s. The testing was stopped when the maximum load had just passed.

2.2.3 Tensile testing

For the tensile testing performed according to EN 10 002–1 standard, a Roell-Korthaus and an Instron 5982 tensile test machine were used. The machines were equipped with an MTS 653 furnace and a Magtec PMA-12/2/VV7-1 extensometer and an Instron SF16 furnace and an Instron 7361C extensometer respectively, both used in laboratory air. For the tensile tests, round-bar specimens with a diameter of 5 mm and a gauge length of 50 mm were used. Strain rates from $10^{-2}s^{-1}$ down to $10^{-7}s^{-1}$ and temperatures at 23 °C (referred to as RT), 300 °C, 400 °C, 500 °C, 600 °C, 650 °C and 700 °C were used. The slow strain rate testing (SSRT) and the very slow strain rate testing (VSSRT) were performed on the Instron 5982 electromechanical tensile test machine.

2.2.4 Creep–fatigue interaction testing

The creep–fatigue interaction (CF) tests were conducted according to ASTM E2714–13 standard, using an MTS servo hydraulic test machine equipped with an Instron 8800 control system, an Instron 2632-055 extensometer and an MTS 652.01 furnace. The tests were performed in only strain control and both strain controlled fatigue part and load controlled creep part using different strain ranges (from 0,5 to 2 %) and dwell times (from 5 to 30 min). The testing was performed at 700 °C.

2.2.5 Thermal cycling in water vapour environment

Thermal cyclic testing in a corrosive environment was performed at 650 °C in a thermal cyclic rig. The specimens rested on a stationary ceramic table and the thermal cycling was accomplished by lowering and rising a furnace over the specimens. Since the specimens rested on the ceramic table, five sides of the specimen were fully exposed to the corrosive medium. Consequently, all analyses were made on the surface facing upwards. One thermal cycle consisted of a 96 hour dwell time at 650 °C followed by natural cooling until the specimens reached 100 °C which took approximately 18 minutes. The specimens were subjected to 2, 5, 10, 20 and 30 thermal cycles. During the hot part of the cycle, water was introduced as an air-water mist which was sprayed into the furnace (not directly onto the specimens). The water mist immediately evaporated as it was sprayed into the furnace and increased the water vapour content in the furnace chamber. The amount of water vapour was controlled by the water-to-air ratio in the water mist and was adjusted to ~15 mol%. This was achieved by controlling the water inlet flow into the air stream. Since the furnace was not airtight, a new burst of air-water mist was injected every 3rd minute which evaporated and flushed the furnace through with water vapour.

2.2.6 Thermo-mechanical fatigue

The employed testing procedure was strain controlled thermomechanical fatigue (TMF) testing with 5 minutes dwell time at maximum mechanical strain range ($\Delta\varepsilon_{mech,max}$). The test machine was a servo-hydraulic TMF machine from Instron with induction heating and forced air-cooling. Before the TMF-tests the machine was carefully aligned to prevent buckling and other instability effects. The temperature range used was 100-800°C and the heating and cooling rate was 5°C/s. The setup and employment of the tests were done according to the “the validated code of practice” [21], with spot welded thermocouples and 1mm distance between them. The number of cycles until failure (N_f) was defined as a 10 % load-drop.

2.2.7 Stress relaxation – screening method

In the previous KME-project KME-501 [11], it was found that cold deformed specimens, using bending, relax and straighten out if they are not fixed at a constant displacement. This specimen relaxation is not wanted since it affects the load relaxation and subsequently the SRC.

In order to achieve a constant displacement the bent specimens (Fig. 1 (a)) are placed in the rig (Fig. 1 (b)) when heated.

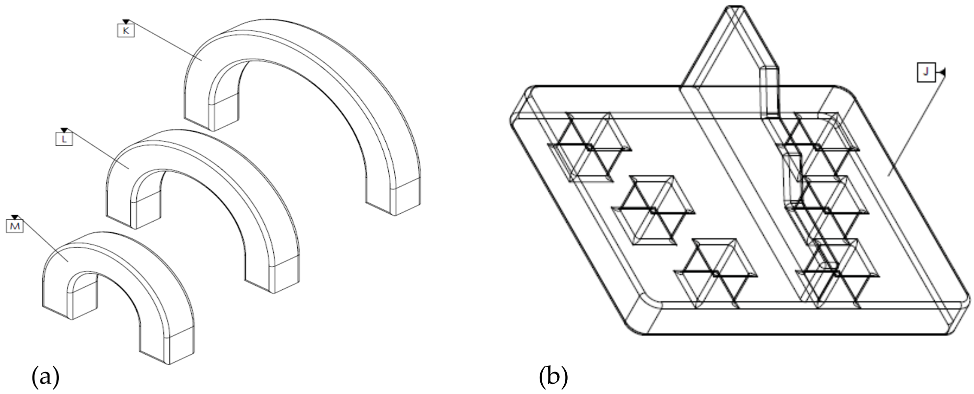

Figure 1: SRC screening method (a) specimen in different deformation degrees and (b) the rig.

The rig with specimens that have different cold deformation degrees achieved by bending the specimens to corresponding radii, the deformation degrees are 16%, 21% and 30% respectively, are placed in a furnace at 650 °C for 500 h and 1000 h. After heating the specimens are studied by LOM and SEM to determine the presence of SRC.

2.2.8 Stress relaxation - Mechanism evaluation method

For the testing method to investigate the mechanisms of SRC, a CT-specimen (Fig. 2 (a)) and tensile test machine with potential drop (PD) (Fig. 2 (b)) were used. A crack was propagated giving a plastic zone. Different plastic zones were used to represent different deformation degrees, plastic zone sizes of 0.11, 0.21 and 0.31 mm have been used. The plastic zones were calculated using the load range variable. The specimens were tensile loaded up to the maximum load from the crack propagation and fixed at that position. The load was monitored by the load cell and the crack propagation was monitored using a PD-method. The tests have been performed at 700°C for at maximum 500 hours. Fracture surface and microstructure will be analysed using SEM.

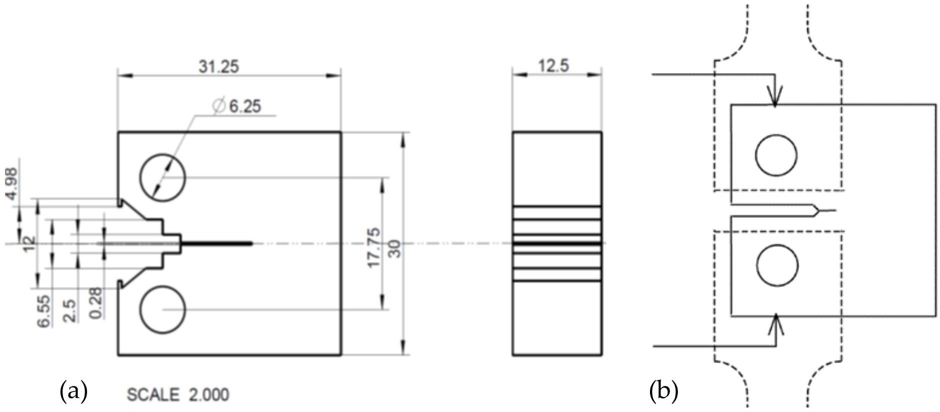

Figure 2: (a) schematic drawing of CT-specimen with PD and (b) drawing of the CT-specimen that have been used.

2.2.9 Scanning electron microscopy

The microstructural investigations were performed using different SEM related techniques like electron channelling contrast imaging (ECCI), electron backscatter diffraction (EBSD), energy–dispersive spectroscopy (EDS) and wave–dispersive spectroscopy (WDS).

ECCI investigations were performed on a HITACHI SU-70 field emission gun (FEG)-SEM and a Zeiss XB 1540 FEG-SEM, both equipped with a solid state 4-quadrant backscattered electron (BSE) detector, using 10 kV acceleration voltage and working distances between 5 mm and 7 mm.

EBSD investigations were performed in a HITACHI SU-70 FEG-SEM equipped with an OXFORD EBSD detector and a 6500 F JEOL FEG-SEM equipped with a TSL OIM EBSD system, both EBSD systems used the HKL software CHANNEL 5. The EBSD-maps were measured at 15 kV and 20 kV acceleration voltage using a working distance of 12 mm up to 25 mm and step sizes of 0,1 µm up to 4 µm.

The EDS and WDS investigations were performed using a HITACHI SU-70 FEG-SEM and some additional EDS measurements were performed using a 6500 F JEOL FEG-SEM. EDS and WDS were performed at 20 kV acceleration voltage and a working distance of 15 mm.

3 Project overview

In order to fulfil the project goals the work has been divided into four parts with tasks and subtasks.

3.1 PART 1: CREEP AND LOW CYCLE FATIGUE INTERACTION AND VERY SLOW STRAIN RATE TESTING AND CREEP

3.1.1 Task 1-1: Creep and LCF interaction behaviour

Creep-fatigue interaction tests have been performed in strain-control and combined strain-controlled fatigue part with load-controlled creep part. The testing is completed, analysis regarding the creep-fatigue interaction behavior have been performed, and the creep-fatigue interaction diagram has been constructed.

3.1.2 Task 1-2: Creep testing

Very slow strain rate tensile testing has been performed using a strain rate down to 10^{-7}/s at elevated temperatures. Information regarding the deformation mechanisms have been revealed.

3.2 PART 2: LONG-TERM AGEING

3.2.1 Task 2-1: Influence of long-term ageing on toughness

Impact and fracture toughness test have been performed after 20 000 hours and 30 000 hours ageing process at 650 °C and 700 °C respectively, using Charpy V and CTOD methods. The ageing has been performed in lab furnaces and not in a real power plant.

3.2.2 Task 2-2: Structure integrity evaluation

The influence of long-term exposure in high-temperature environments on structural integrity of the heat resistant materials has been evaluated using data from task 2-1 and KME-501 along with microstructural investigations such as ECCI, EDS and EBSD. Structure stability has been reported and the influence of precipitates on toughness.

3.3 PART 3: THERMOMECHANICAL FATIGUE TESTING

3.3.1 Task 3-1: Long-term ageing before TMF testing

Long-term ageing has been performed at 800 °C for 2000 hours, in order to take the effect of service degradation into account.

Task 3-2: Thermomechanical fatigue testing

The resistance to TMF failure for both an IP and an OP TMF cycle has been analysed and compared. Scanning electron microscopy (SEM) techniques such as ECCI and EBSD have been used to study the damage and failure mechanisms. TMF life times for different austenitic alloys in IP condition have been reported. TMF life times in OP could not be tested due to too high risk of buckling.

3.4 PART 4: STRESS RELAXATION CRACKING

3.4.1 Task 4-1: Method development

Development of two methods, a screening method and a mechanism evaluation method, were to be performed. The testing of the screening method is completed and has been conducted on cold deformed materials. The test consists of bending to different degrees of deformation and then the specimens are subjected to heat under constant displacement. The mechanism evaluation method uses potential drop (PD) and an electromechanical tensile test machine to monitor both crack propagation and load relaxation. A small compact tension (CT) specimen with notch and pre-crack with a predefined plastic zone has been used.

3.4.2 Task 4-2: Stress relaxation cracking behaviour of heat resistant materials

The methods have been conducted to the alloys for characterisation. For both methods LOM and SEM have been used for the analysis if SRC is present in the microstructure and how it influences the microstructure. Further understanding and development of the methods are needed before the SRC behavior can be characterized using the methods.

4 Results

This section present the main results of the work that have been performed, divided into the four subsections 4.1 to 4.4, related to the project goals. The results presented in section 4.1 to 4.4 can also be found in a PhD thesis [22], two MSc theses [23, 24] and eight scientific articles [25-32] written within this project.

4.1 CHARACTERIZATION OF AUSTENITIC STAINLESS STEELS STATIC AND CYCLIC DEFORMED AT ELEVATED TEMPERATURE

Creep-fatigue interaction behaviour

Within the project creep-fatigue interaction (CF) testing has been performed to evaluate two austenitic stainless steels CF behaviour. Two austenitic stainless steels have been investigated, namely Esshete 1250 and Sanicro 25. The tests have been performed according to section 2.2.4.

The results show that the introduction of dwell time causes a larger plastic strain range, see Fig. 3. The results also show that dwell time gives a shorter CF life at a lower strain range, but has no or small effect on the life at a higher strain range, see Fig. 4. Fracture investigation shows that dwell time results in more intergranular cracking. [25]

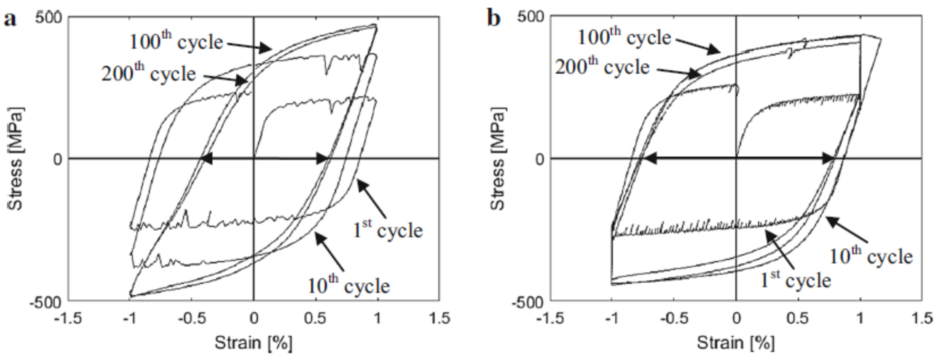

Figure 3: Hysteresis loops at 700 °C, using strain range of 2 % and two dwell times. a No dwell time. b Dwell time 30 min. The inelastic strain ranges corresponding to the 100th cycle are pointed out by the double arrows.

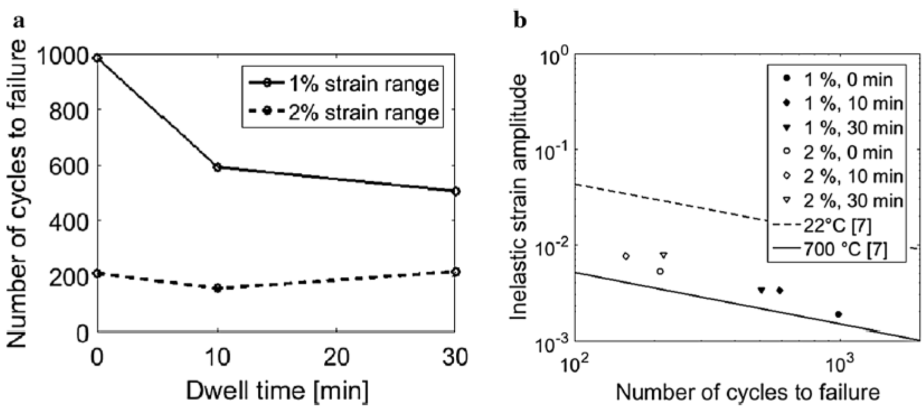

Figure 4: Influence of applied strain range and dwell time on the fatigue life. a Life versus dwell time, b inelastic strain versus life, the lines are taken from Pola´k J et al., Mater Sci Eng A 2014;615:175.

In addition, data of CF behavior for Sanicro 25 and Esshete 1250, with strain controlled fatigue part and load controlled creep part, is presented. Sanicro 25 has a slightly greater CF life but at higher stresses compared to Esshete 1250, see Fig. 5 (a). Fig. 5 (b) show the CF diagram for Esshete 1250 and Sanicro 25, it is seen that both alloys show interaction damage depending on testing conditions (e.i. strain range and dwell time). Esshete 1250 (Fig. 6 (a)) show greater creep strains during the dwell times compared to Sanicro 25 (Fig. 6(b)), even though Sanicro 25 experience higher stresses. Subsequently Esshete 1250 have larger plastic strain ranges compared to Sanicro 25. [26]

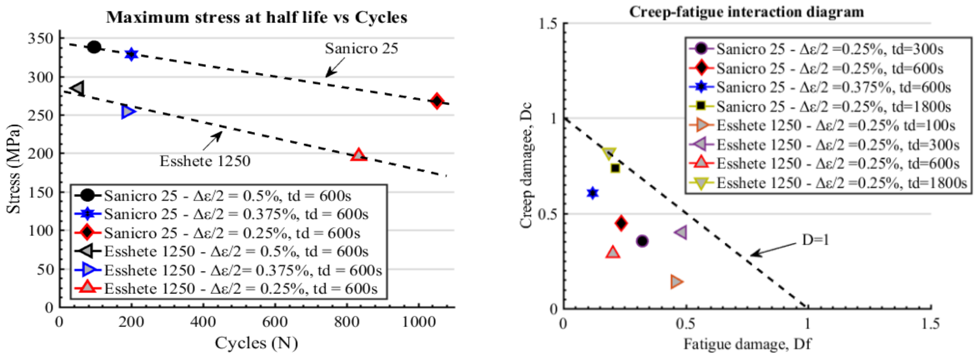

Figure 5: Creep-fatigue interaction tested Sanicro 25 and Esshete 1250 at 700 °C showing max stress at half life vs. number of cycles to failure using a dwell time of 600 s (a) and creep-fatigue interaction diagram.

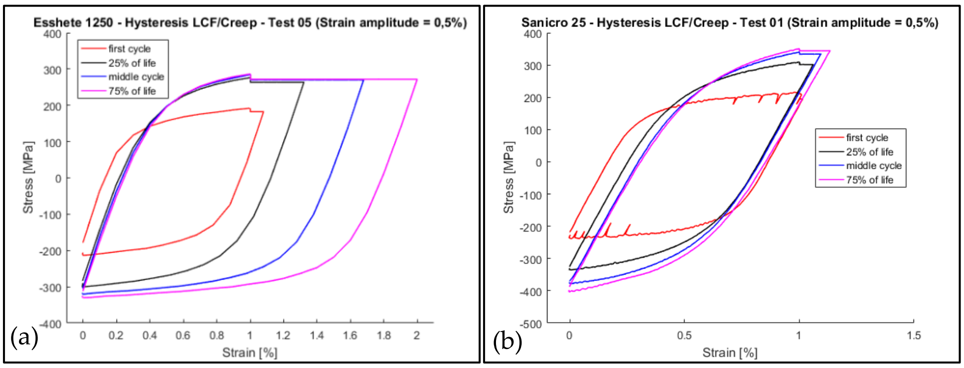

Figure 6: Hysteresis loops of Esshete 1250 (a) and Sanicro 25 (b) from creep-fatigue interaction testing at 700 °C using a strain amplitude of 0.5% and a dwell time of 600 s.

Deformation, damage and fracture mechanisms at elevated temperature

Deformation, damage and fracture mechanisms at elevated temperatures for commercial austenitic stainless steels and nickel-based alloys. The alloys were characterised in terms of microstructure, mechanical properties and stress–strain response. The alloys cover a range from relatively low-alloyed austenitic stainless steel (e.g. AISI 316L) to nickel–based alloys (e.g. Alloy 617) and the interesting area between austenitic stainless steels and nickel-based alloys consisting of the advanced, highly alloyed, heat resistant austenitic stainless steel Sanicro 25. The different austenitic alloys were tested by uniaxial tensile testing at different elevated temperatures from 400 °C up to 700 °C (673-973 K) using different strain rates, according to section 2.2.3.

The different alloys show planar dislocation driven deformation at elevated temperature as dominating deformation mechanism. Deformation twinning is an active deformation mechanism, even at elevated temperatures up to 700 °C (973 K), in austenitic stainless steels, see Fig. 7. Different influence of temperature on ductility was observed, see Fig. 8, where a better high-temperature ductility is governed by a higher amount of nickel and solid solution hardening elements. [27]

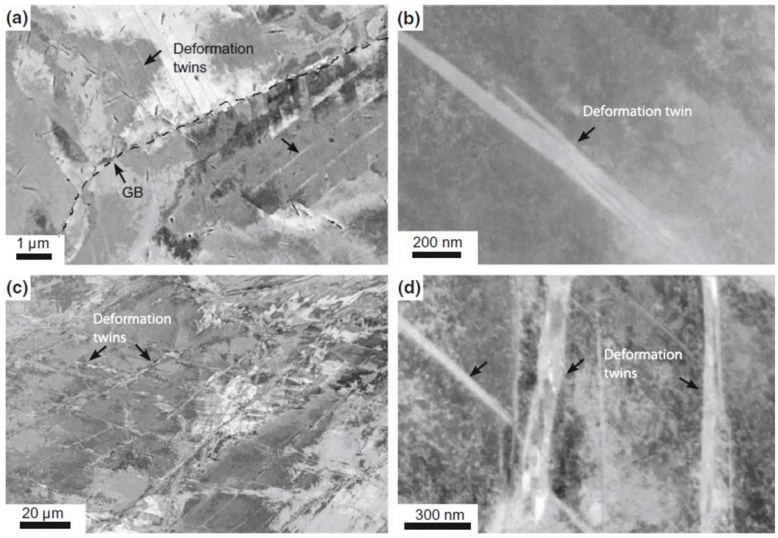

Figure 7: Deformation twins at 650 °C (923 K) in AISI 310 (a), in Sanicro 25 (b), multi-directional twinning in Alloy 800HT (c) and multi-directional twinning in Alloy 617 (d). GB means grain boundary.

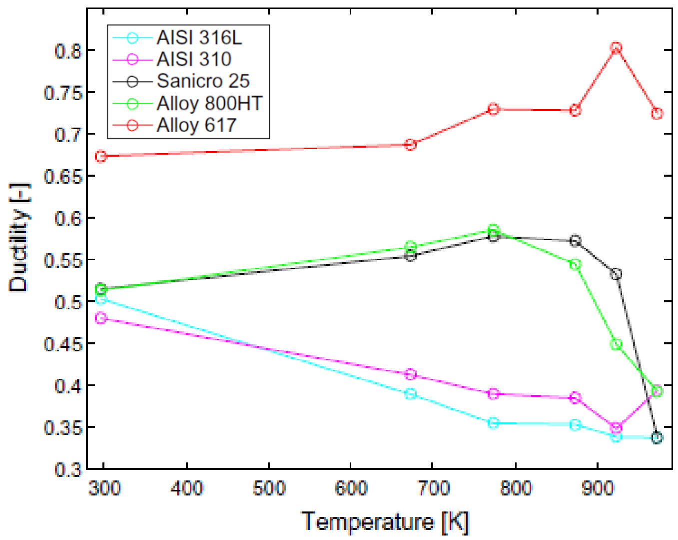

Figure 8: Ductility for all materials at elevated temperatures.

Slow strain rate testing (SSRT) and very slow strain rate testing (VSSRT, using a strain rate of $10^{-7}s^{-1}$) has been performed in order to evaluating creep properties, see Fig. 9 for Sanicro 25 and Fig. 10 for Haynes 282. The mechanical response supports that time dependent deformation is an active mechanism during SSRT at elevated temperature, see Fig. 10 (b). The microstructure development was analysed and evidence of creep-like deformation was found in Haynes 282, see Fig. 11. Therefore, the strain rate dependent deformation during SSRT should be considered as a creep deformation contribution to the total deformation. [22]

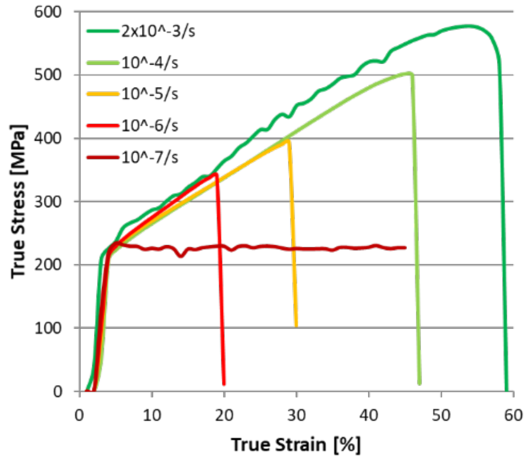

Figure 9: Stress-strain responses of Sanicro 25 tested using different strain rates at 700 °C. The 10^{-7}/s test was stopped before fracture.

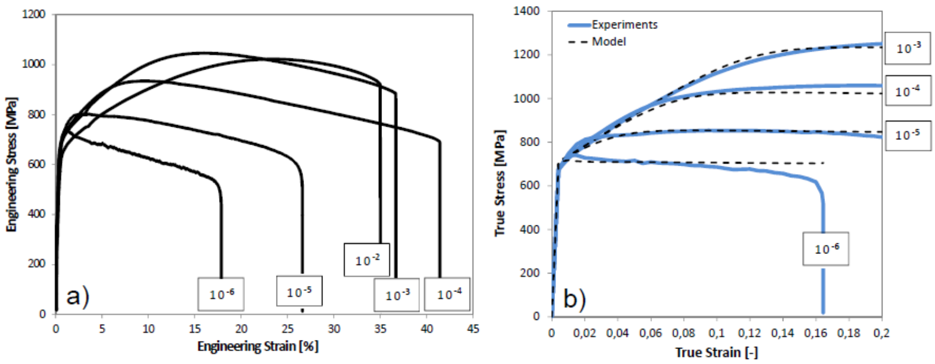

Figure 10: Stress-strain responses of Haynes 282 tested using different strain rates at 700 °C, a) engineering stress-strain curves and b) true stress-strain curves and modelled results.

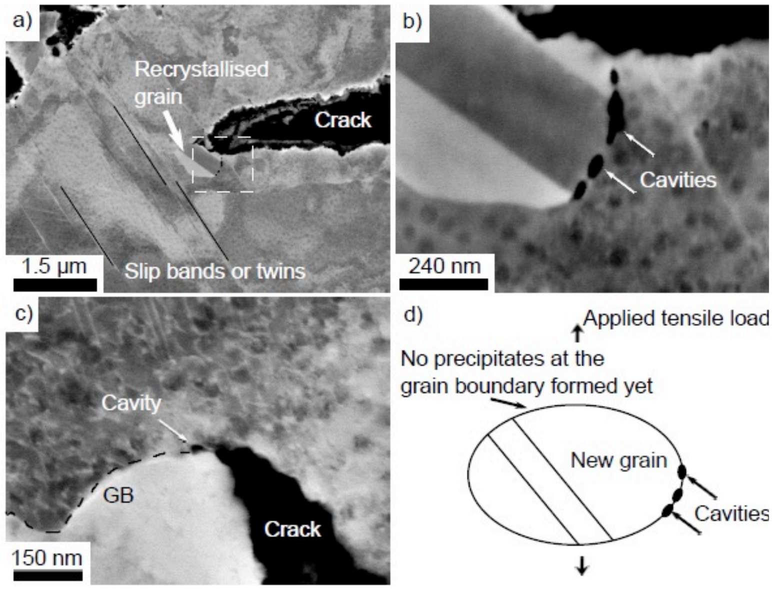

Figure 11: Microstructure development due to a creep-like deformation mechanism, in a material deformed using a strain rate of $10^{6}s^{-1}$, showing in a) recrystallised grain formed during the deformation process at elevated temperature, b) a magnification of the new grain where cavities have been formed at the grain boundary and no sign of precipitates can be seen, c) crack propagation assisted by the formation of cavities within a grain boundary (GB) where no precipitates can be found. In d) a schematically illustration of formation of cavities are shown. The applied tensile load axes are vertical in the micrographs.

Damage and fracture micro-mechanisms have been evaluated of AISI 316L and Alloy 617 from tensile test and SSRT at RT, 650 °C and 700 °C. The dominant damage micro-mechanism in Alloy 617 is interaction between slip bands and grain boundaries, causing strain concentrations. When using strain rates down to $10^{-3}s^{-1}$, the dominant fracture micro-mechanisms are cracked particles or cracks between particle and matrix. When lower strain rate ($10^{-6}s^{-1}$) is used at elevated temperatures, small cracks in the grain boundaries, due to the embrittlement from precipitates, becomes the dominant fracture micro-mechanism for Alloy 617. At RT, near the fracture surface, AISI 316L has a microstructure that is heavily deformed from multi-directional slip bands and planar slip interacting with grain boundaries causing damage such as strain concentrations. At elevated temperatures, when using a low strain rate ($10^{-6}s^{-1}$), AISI 316L also shows the same fracture micro-mechanism as Alloy 617, which means small cracks appear in the grain boundaries due to embrittlement from precipitates, see Fig. 12. [28]

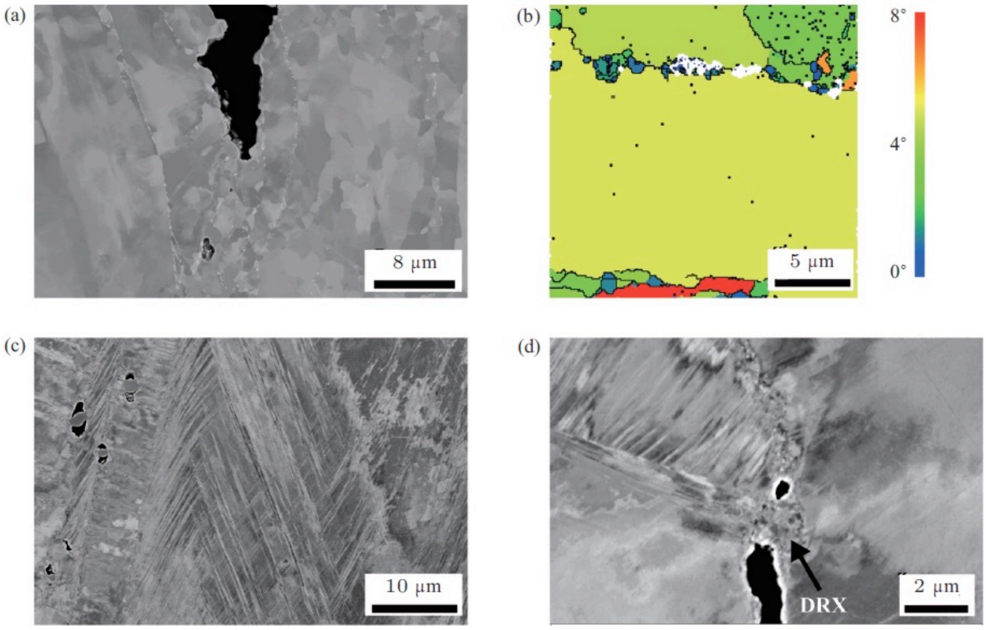

Figure 12: Damage and fracture micromechanisms. AISI 316L (10^{-6} s^{-1}) (a) at 700 °C and (b) DRX at 650 °C (using EBSD grain average misorientation (GAM) map, where GAM below 3° means recrystallized grain, black lines correspond to grain bounderies >10 ° and white color means zero solution which in this case is caused by precipitates). Alloy 617 at 650 °C (c) 10^{-4} s^{-1}, (d) 10^{-6} s^{-1}.

4.2 STRUCTURAL STABILITY AFTER LONG-TERM AGEING AND THERMAL CYCLING

The influence of long-term aging on structural stability and toughness

The austenitic alloys were long-term aged for up to 30 000 hours at 650 and 700 ° C. The influence of ageing on structural development was investigated by impact and toughness testing and followed by microstructural investigations.

The results showed that the austenitic stainless steels suffered from embrittlement due to precipitation, both within the grain and in the grain boundaries. With increased aging time and aging temperature, the amount of precipitates increased. In several of the stainless steels the intermetallic sigma phase was found. Precipitations of Sanicro 25 after up to 30 000 hours ageing at 650 °C and 700 °C are shown in Fig. 13. Precipitates like the Z-phase, Laves phase and $M_{23}C_6$ were most likely found in the aged Sanicro 25.

These precipitates are thought to influence the grain boundary integrity, thus lowering the toughness. [23]

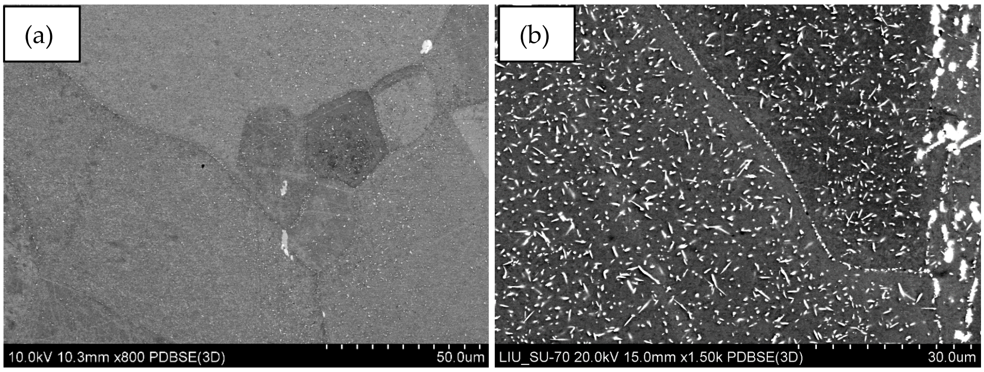

Figure 13: Precipitations in Sancro 25 after 30 000 hours at 650 °C (a) and 20 000 hours at 700 °C (b).

Long-term ageing in Alloy 617 can lead to the precipitation of γ′, Mo-rich and Cr-rich carbides, see Fig. 14. This causes a reduction of toughness of the material, see Fig. 15 (a). However, the material still has a high toughness even after an ageing at 700 °C for 20 000 hours, see Fig. 15 (b). Besides dislocation slip, twinning (Fig. 15 (c)) is another toughening mechanism in Alloy 617 during toughness testing. [29, 30]

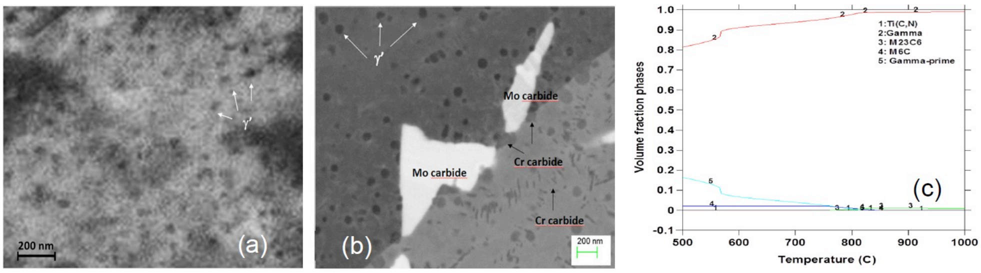

Figure 14: Precipitates in long term aged material, (a). At 700°C for 3000 hours, (b). At 700°C for 20 000 hours, (c). Phase diagram of alloy 617 from 500°C to 1000°C.

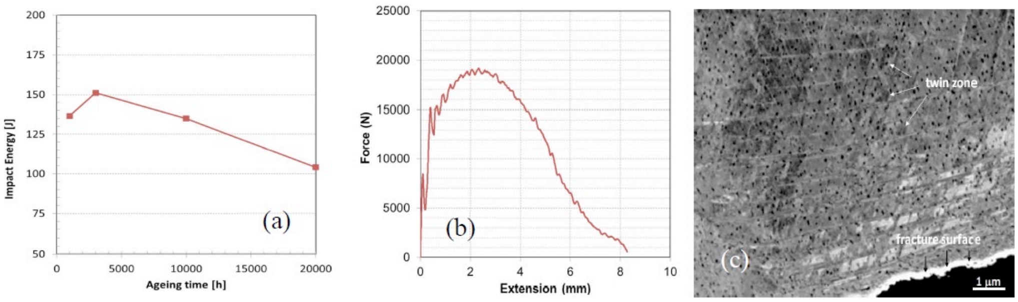

Figure 15: Influence of ageing on impact toughness, deformation and fracture behavior in the specimen aged at 700°C for up to 20 000 hours, (a). Impact toughness, (b). Impact force versus crack extension, (c). deformation, damage and fracture behavior.

The influence of thermal cycling on structural stability

Thermal cycling in humidified air, according to section 2.2.5, has been performed on AISI 304, AISI 316L and Sanicro 28. The results show that the main outward growing oxide is Fe_2O_3 for all three alloys. The main inward growing oxides are probably an Ni-poor (Cr, Fe, Ni)$_3O_4$ and an Ni-rich (Cr, Fe, Ni)$_3O_4$ spinel for AISI 304 and AISI 316L and an $NiCr_2O_4$ spinel oxide for Sanicro 28, see Fig. 16 and table 3. Sanicro 28 has much better oxidation resistance at 650 °C and in water vapour environment than the two other alloys. This is attributed to the higher Cr/Fe-ratio of Sanicro 28. [31]

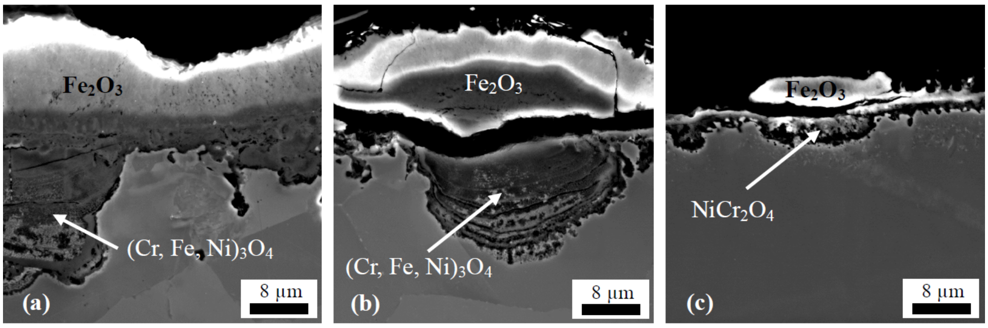

Figure 16: SEM cross-section images of the oxidation after 30 thermal cycles in water vapor environment. (a) AISI 304, (b) AISI 316L and (c) Sanicro 28.

Table 3: EDS typical chemical compositions of OAZ after 30 cycles for the tested alloys [wt.%].

Oxide	O	Si	Cr	Mn	Ni	Fe
Fe_2O_3	52	0,09	1,68	0,34	0,08	45,8
Ni-rich spinel	41,3	0,93	18,61	1,95	25,35	11,86
Ni-poor spinel	55,34	0,92	20,03	0,64	8,37	14,69
$NiCr_2O_4$	50,4	0,8	22,12	0,49	24,18	2,02

It was found that the Cr/Ni-ratio in the oxidation affected zone (OAZ), see Fig. 17, is important for the local surface phase stability. Where a low Cr/Ni-ratio decreases the probability of local surface phase transformation and stabilizes the FCC structure, see table 4. [31]

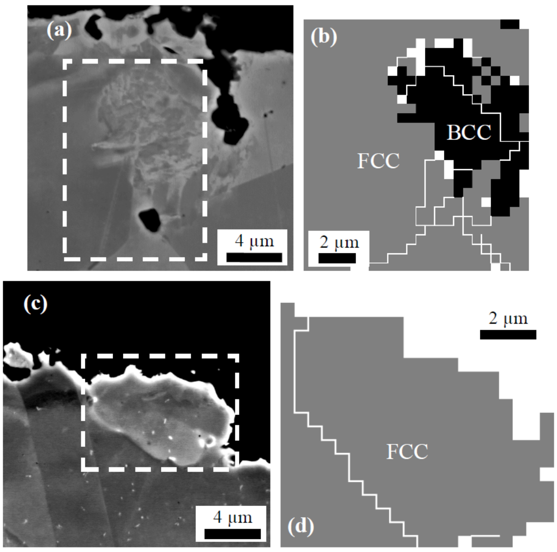

Figure 17: SEM images (a) and (c) and EBSD grain boundary and phase-maps (b) and (d) where the white small lines are grain boundaries >10 °, gray correspond to FCC, black corresponds to BCC and the white larger dots corresponds to zero-solutions. (a) and (b) AISI 304, (c) and (d) AISI 316L.

Table 4: EDS typical chemical compositions of OAZ after 30 cycles for the tested alloys [wt.%].

Alloy	Si	Cr	Mn	Ni	Fe	Cr/Ni-ratio
AISI 304	0,4	11,48	0,38	12,96	74,33	0,89
AISI 316L	0,25	9,65	0,74	20,32	68,75	0,47
Sanicro 28	0,19	17,04	1,05	39,42	41,31	0,43

The FCC structure in AISI 304 corresponds to α' martensite that forms during cooling from 650 °C to 100 °C. This is due to chromium depletion caused by oxidation at the surface, which locally rises the M_s temperature. The α' martensite grows thicker with increasing number of cycles during the thermal cycling. The α' martensite transforms back to FCC at 650 °C during the thermal cycling. The formation of α' martensite will give several smaller grains at the surface compared to the grains in the FCC matrix. Even though the α' martensite transforms back to FCC at 650 °C, the increased number of grain boundaries will improve the chromium diffusion within this region. A more chromium-rich protective scale is formed above the α' martensite layer compared to regions where no α' martensite layer rapidly formed. For this reason, areas that lack the α' martensite are more consumed by inward corrosion and the outer oxide scale is thicker than at the areas with α' martensite, see Fig. 18. Thus, the formation of α' martensite is locally decreasing the in- and outward scale growth. [32]

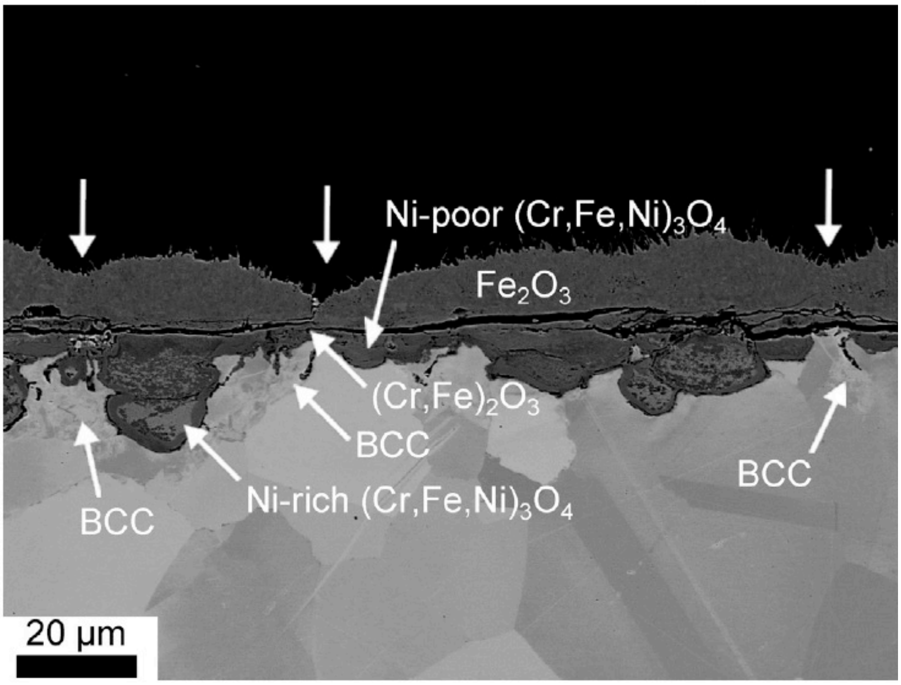

Figure 18: ECCI-image of the microstructure after 10 thermal cycles between 100 °C and 650 °C in ~15 mol% water vapour. The image shows the effect of grain boundary diffusion of Cr, resulting in a thinner outer oxide layer (presumably Fe_2O_3) indicated by the three arrows at the α' martensite (BCC) regions. Above the α' martensite there is a Cr-rich $(Cr, Fe)_2O_3$ scale on the metal surface, compared to the inward growing oxides (presumably a Ni-rich $(Cr, Fe, Ni)_3O_4$ and a Ni-poor $(Cr, Fe, Ni)_3O_4$ spinel oxides) next to it.

4.3 THERMOMECHANICAL FATIGUE BEHAVIOUR

Thermomechanical fatigue testing has been performed on three commercial austenitic alloys: Esshete 1250, Sanicro 25 and Sanicro 31HT. The materials were exposed to IP and OP TMF testing according to section 2.2.6. Pre-aged (2 000 hours at 800 °C) TMF-samples were tested to simulate service degradation from long-term usage. The pre-aged samples showed a shorter TMF-life compared to non-aged samples, see Fig. 19. The shorter TMF life for the pre-aged samples were attributed to an embrittling effect from precipitation, see Fig. 20. [23, 33]

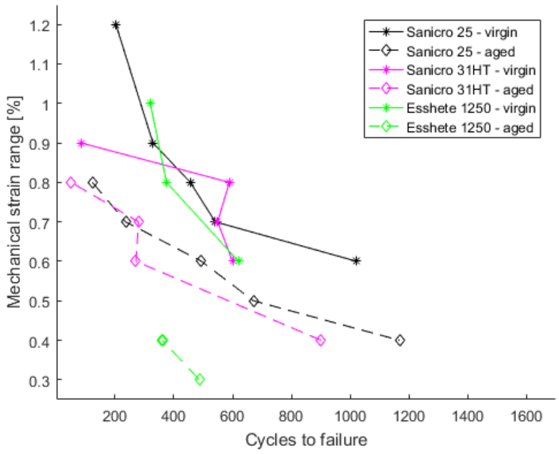

Figure 19: Mechanical strain range over number of cycles to failure for IP TMF-tested virgin and aged austenitic stainless steels.

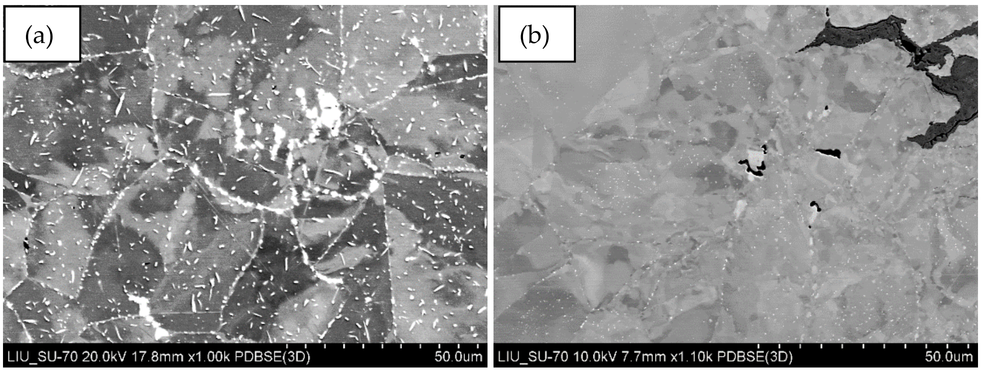

Figure 20: Cross-section of Sanicro 25 in aged (a) and non-aged (b) condition, showing the amount of precipitates both in the grains and in the grain boundaries after TMF testing using a mechanical strain range of 0.6%.

In addition, OP test have been performed on aged specimens of Sanicro 25, Sanicro 31HT and Esshete 1250. These tests have been performed for 500 cycles and then stopped, this was to be able to compare the alloys since the risk of buckling was too high. Esshete 1250 had to be performed using a strain range of 0.3% for this reason. Fig. 21 shows the development of maximum stress during the 500 cycles for each material. The reason for the higher maximum stress of Sanicro 25 compared to Sanicro 31HT is probably a greater high-temperature strength.

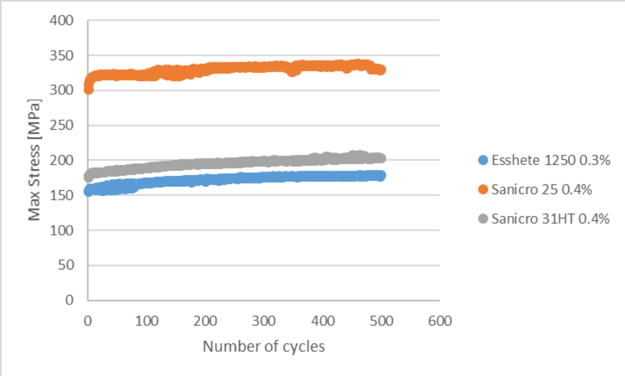

Figure 21: Max stress vs. number of cycles up to 500 cycles.

4.4 STRESS RELAXATION CRACKING

Screening method

AISI 347H and Sanicro 31HT have been tested using the screening method, according to section 2.2.7. The results show no signs of cracks in the cross section after testing at 700 °C for 500 hours, but small precipitates in the grain boundaries have been observed in Sanicro 31HT, see Fig.22. However, further analysis is needed to evaluate the method. [24]

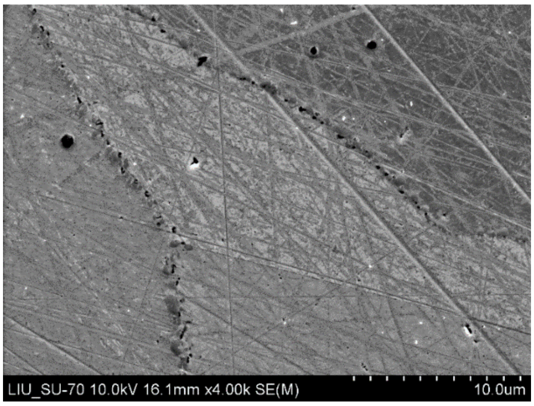

Figure 22: SEM image showing different small precipitates in the grain boundaries in Sanicro 31HT after testing at 700 °C for 500 hours.

Mechanism evaluation method

AISI 347H and Sanicro 31HT have been tested using the mechanism evaluation method, according to section 2.2.8. The only sign of SRC have been found in Sanicro 31HT with a plastic zone of 0.11 mm. The SRC sign means that the PD value increases, corresponding to a crack propagation, when the stress decreases as in Fig. 23 (a), the initial drop in the PD curve is due to the PD-wires that have been used. None of the other plastic zones or AISI 347H showed this behavior and instead the PD curve was constant or decreased as in Fig. 23 (b). However, further analysis is needed to evaluate the method and the mechanism of SRC. [24]

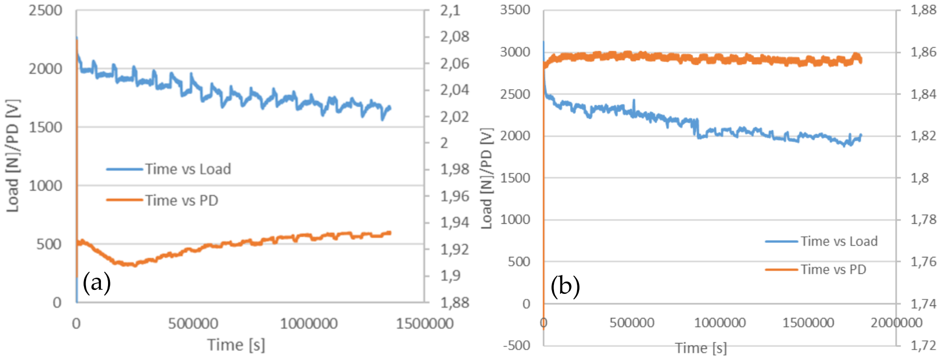

Figure 23: Load and PD development over time for Sanicro 35HT with 0.11 mm (a) and 0.21 mm (b) plastic zone size.

5 Analysis of the results

This project is a study of austenitic materials and their behaviour at high temperature. In all seven austenitic stainless steel and three nickel-based alloys have been investigated. Deformation, damage and fracture behaviour during thermo-mechanical fatigue, creep-fatigue interaction, slow strain rate deformation together with long term ageing have been focused on.

During CF testing on Sanicro 25 in strain control, stress relaxation due to the dwell time is observed. As a consequence of the relaxation a larger inelastic strain range occurs. A difference in the influence of dwell time on of the number of cycles to failure was observed between the lower (1 %) and the higher (2 %) strain range. The results show that at the lower strain range a longer dwell time gives a shorter life, but at the higher strain range the dwell time has none or small effect on the life. Furthermore, when the fatigue part is strain controlled and the creep part is load controlled the creep part consist no longer of stress relaxation but creep under constant load. From the results, it is shown that Sanicro 25 with comparably greater creep strength has better CF life compared to Esshete 1250. In addition, Esshete 1250 shows greater creep strains during the dwell time compared to Sanicro 25, that is due to a lower creep strength. Both alloys show CF damage.

Austenitic stainless steels and nickel-based alloys are considered to have satisfactory ductility at elevated temperatures but with lower stress-carrying abilities. AISI 316L and AISI 310 showed a drop in ductility already at intermediate temperatures compared to RT. The increase in ductility at 700 °C for AISI 310 should be treated with care since it represents one specimen. Sanicro 25 and Alloy 800HT showed increasing ductility with increasing temperature until it dropped at 650 °C and 600 °C, respectively, compared to RT. Alloy 617 showed increasing ductility with increasing temperature for all elevated temperatures compared to RT. The damage mechanisms found did not differ considerably between the alloys at elevated temperature, and cannot explain the differences in ductility. All materials in the present study have similar crystallographic structure and are stable at elevated temperatures. Thus, the differences in ductility may depend on chemical composition. In terms of ductility, the alloys rank in order of increasing ductility from: AISI 316L/AISI 310 -> Alloy 800HT/Sanicro 25 -> Alloy 617. This correlates reasonably well with the alloys' corresponding nickel content. It is interesting that deformation twins were found at elevated temperatures as a deformation mechanism. Deformation twinning usually does not occur in austenitic stainless steels at the tested elevated temperatures. However, the dominant deformation mechanisms during uni-axial tensile testing at elevated temperatures and moderate strain rates are dislocation driven planar slip and localised slip bands in one or multi-directions. When the strain rate is decreased, these deformation mechanisms are accompanied by strain rate dependent deformation and recovery mechanisms such as dynamic recovery and dynamic recrystallisation. From microstructure investigations evidence of damage from strain rate dependent deformation, similar to creep damages and cracking in Haynes 282 was observed. Therefore, it is proposed that the strain rate dependent deformation during SSRT should be considered as creep. The typical damage mechanisms during slow strain rate tensile testing at elevated temperatures are strain concentrations and micro-cracks at grain boundaries due to interaction between deformation and precipitates. When lowering the strain rate the materials show other damage mechanisms such as strain-rate dependent deformation during SSRT at elevated temperature.

Long term ageing affects the alloys differently depending on chemical composition which influence the precipitation of different particles as seen for Alloy 617 and Sanicro 25. In Sanicro 25 precipitation of Z-phase, Laves phase and $M_{23}C_6$ was observed both in the grains and at the grain boundaries and this is believed to reduce the toughness. Since Alloy 617 contain high amount of Al and Ti, γ' is the main precipitate. Besides γ', Mo-rich carbide (M_6C) and Cr-rich carbide ($Cr_{23}C_6$) have been observed. Mo-rich carbides are very large and have precipitated at grain boundary and in the grains.

The clear difference in TMF-life between the aged and the virgin specimens shows that microstructural evolution during ageing are crucial for the performance of the investigated materials. The aged materials became more brittle and experienced increased stress until failure.

Regarding the SRC evaluation more work is needed before any conclusion can be made.

In this work the relatively large number of materials studied has been an advantage since properties, microstructure and deformation mechanisms seem to act differently with respect to time, temperature and variations in the parameters used in testing like strain rate. This has made the project results more interesting due to the large ability to compare different commercial austenitic alloys and improved the analysis of the results. The results produced in this project will be of great importance for development of new materials and a useful source of information in design of future energy conversion plants.

6 Conclusions

Tougher operation conditions, to achieve increased efficiency of biomass-fired power plants and move towards more sustainable power production, will push austenitic stainless steels above the limit of their present design. Deeper understanding of the high-temperature behaviour of austenitic alloys is required to improve the performance of austenitic stainless steels through material development.

The focus of this project has been to evaluate:

- the creep and LCF interaction diagram of the boiler materials for material selection and integrity analysis since the boiler materials can undertake both creep and low cycle fatigue during the service (Goal a).
- the structure stability and the toughness of the materials after long term service at a temperature higher than 650°C and service time longer than 20 000 hours for safety analysis (Goal b).
- thermo-mechanical fatigue properties of the boiler materials for safety and life evaluation since the power plants can start/shutdown quite often during service for energy saving and flexibility purposes in the future (Goal c).
- the stress relaxation cracking behaviour of the boiler material. It is a critical problem for some boiler materials (Goal d).

By providing further understanding on these topics, it is believed that this project can contribute to the material development of improved, advanced heat resistant austenitic stainless steels. By doing so, this project also contributes to the general goals of KME (especially the goals: "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") and long term benefits for industry and society overall.

The creep–fatigue interaction behaviour of austenitic stainless steels in strain-controlled testing shows that dwell time causes a larger plastic strain range. The results also show that dwell time gives shorter life at a lower strain range, but has none or small effect on the life at a higher strain range. Sanicro 25 and Esshete 1250 both show creep-fatigue interaction damage. The dominant deformation mechanisms during uni-axial tensile testing at elevated temperatures and moderate strain rates are dislocation driven planar slip and localised slip bands in one or multi-directions as well as deformation twinning. When the strain rate is decreased, these deformation mechanisms are accompanied by time dependent deformation and recovery mechanisms such as dynamic recovery and dynamic recrystallisation. The typical damage mechanisms during slow strain rate tensile testing at elevated temperatures are strain concentrations and micro-cracks at grain boundaries due to interaction between deformation and precipitates. The predominant damage mechanism of the time dependent deformation is cavities at grain boundaries. Fracture occurs mainly intergranularly due to grain boundary embrittlement from precipitates and coalescence of micro-cracks.

After long term high-temperature ageing (up to 30 000 hours at 650 °C and 700 °C) it was found that the precipitation and growth of precipitates strongly influence the impact toughness depending on the chemical composition of the alloys. It was also found that the tested nickel-based alloys possess a better structural integrity and has an impact toughness less influenced by long term ageing at elevated temperatures.

Pre-aged (2 000 hours at 800 °C) samples showed a shorter TMF-life compared to virgin samples. The shorter TMF life for the pre-aged samples were attributed to an embrittling effect from precipitation.

Two SRC testing methods are used in the evaluation, one screening method and one mechanism evaluation method. More work is needed to be able to use these two methods for SRC evaluation.

Other conclusions made from the project regards thermal cyclic testing in a water vapour environment of austenitic stainless steels. The study shows the effects of detrimental chromium vaporisation, causing both outward oxide growth of a non-protective iron rich oxide and inward oxide scale growth of spinel oxides, consuming the bulk material. More heavily alloyed stainless steels, such as Sanicro 28, showed better resistance to oxidation in water vapor at 650 °C than less alloyed (such as AISI 304 and 316L). It was also found that α' martensite is formed at the surface of AISI 304 but not in the other tested stainless steels. α' martensite is related to thermal martensitic transformation and it influence the oxidation by decreasing the oxide growth. This is attributed to increased chromium supply from the bulk material by grain boundary diffusion due to the formation of small α' martensite grains that increase the number of grain boundaries at the surface.

The results produced within this project can be used in development of new materials and are useful sources of information in design of future energy conversion plants.

7 Suggestions for future research work

Within the work that underlies this project report, several different mechanical tests have been performed at elevated temperatures. In the research field of material used for biomass-fired power plants, much focus is on oxidation and corrosion since they are the most crucial limitations of the materials used today. However, the influence of both mechanical and thermal stresses cannot be overlooked; mechanisms such as creep and fatigue as well as structural integrity for long term service are important. Therefore, more effective test methods, closer to real operation conditions with interaction of chemical and mechanical material behaviour, are needed. Well designed, more effective and realistic methods can be used as tools for material development, directly by the industry and/or for advanced material modelling. By performing mechanical tests in a high-temperature environment, involving the environment found in biomass combustors, this could be achieved. Already existing methods for high-temperature material property evaluation, such as thermomechanical fatigue, creep–fatigue interaction and slow strain rate tensile testing, could, by adding a biomass-fired related environment, provide this more realistic testing.

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9 Publications

9.1 REFEREED JOURNAL PAPERS

Mattias Calmunger, Guocai Chai, Robert Eriksson, Sten Johansson and Johan Moverare
Characterization of Austenitic Stainless Steels Deformed at Elevated Temperature
Metallurgical and Materials Transactions A, 2017, (48), 4525-4538. (Open Access)

Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
Creep and Fatigue Interaction Behavior in Sanicro 25 Heat Resistant Austenitic Stainless Steel
Transactions of the Indian Institute of Metals, 2016, (69)2, 337-342.

Mattias Calmunger, Robert Eriksson, Guocai Chai, Sten Johansson and Johan Moverare
Surface Phase Transformation in Austenitic Stainless Steel Induced by Cyclic Oxidation in Humidified Air
Corrosion Science, 2015, (100), 524-534.

Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
Deformation behaviour in advanced heat resistant materials during slow strain rate testing at elevated temperature
Theoretical and Applied Mechanics Letters, 2014, (4)041004. (Open Access)

9.2 REFEREED CONFERENCE ARTICLES

Hugo Wärner, Mattias Calmunger, Guocai Chai and Johan Moverare
Creep-fatigue interaction in heat resistant austenitic alloys
FATIGUE 2018; Poitiers; France; 27 May 2018 through 1 June 2018
MATEC Web of Conferences, 2018, (165)05001. (Open Access)

Guocai Chai, Mattias Calmunger, Sten Johansson and Johan Moverare
Toughening Behavior in Alloy 617 with Long Term Ageing
MSMF8; Brno; Czech Republic; 27 July 2016 through 29 July 2016
Solid State Phenomena, 2017, (258), 302-305.

Mattias Calmunger, Robert Eriksson, Guocai Chai, Sten Johansson, Jan Högberg and Johan Moverare
Local Surface Phase Stability During Cyclic Oxidation Process
THERMEC 2016; Graz; Austria; 29 May 2016 through 3 June 2016
Materials Science Forum, 2017, (879), 855-860.

Guocai Chai, Mattias Calmunger, Sten Johansson, Johan Moverare and Joakim Odqvist.
Influence of Dynamic Strain Ageing and Long Term Ageing on Deformation and Fracture Behaviors of Alloy 617
THERMEC 2016; Graz; Austria; 29 May 2016 through 3 June 2016
Materials Science Forum, 2017, (879), 306-311.

9.3 CONFERENCE CONTRIBUTION

Hugo Wärner, Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
Thermomechanical fatigue behavior of aged heat resistant austenitic alloys
EUROMAT 2017; Thessaloniki; Greece; 17 September 2017 through 22 September 2017.

Mattias Calmunger, Robert Eriksson, Guocai Chai, Sten Johansson and Johan Moverare
Influence of cyclic oxidation in moist air on surface oxidation-affected zones
EUROMAT 2017; Thessaloniki; Greece; 17 September 2017 through 22 September 2017.

9.4 THESES WITHIN THE PROJECT

Sabari Raj
Development of screening method and evaluation of stress relaxation cracking in high temperature materials
M.Sc. Thesis 2018, ISRN: LIU-IEI-TEK-A--18/03003—SE.

Hugo Wärner
Thermomechanical fatigue behaviour of aged heat resistant austenitic alloys
M.Sc. Thesis 2016, ISRN: LIU-IEI-TEK-A--16/02485—SE.

Mattias Calmunger
On High-Temperature Behaviours of Heat Resistant Austenitic Alloys
PhD Thesis 2015, ISBN: 978-91-7685-896-7.

Mechanical behaviours of austenitic stainless steels in high-temperature environments

Increased knowledge about the mechanical performance of virgin and aged austenitic alloys for future application in the area of power generation has been achieved by using several mechanical testing methods, such as thermo-mechanical fatigue, creep/fatigue interaction and toughness tests, of austenitic alloys at elevated temperature and room temperature. The results show that the structural development during testing is affecting properties differently depending on alloy composition. This knowledge can lead to improvements of the performance of the materials.

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