KME

CONSORTIUM MATERIALS TECHNOLOGY for demonstration and development of thermal energy processes

Long term high temperature behaviour of advanced heat resistant materials

Långtidsegenskaper hos avancerade högtemperaturmaterial

Sten Johansson

KME-501

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Preface

The project has been performed within the framework the fifth stage of the material technology research programme KME.

KME, Consortium Materials technology for demonstration and development of thermal Energy processes, was established 1997 on the initiative of the Swedish Energy Agency. In the consortium, the Swedish Energy Agency, seven industrial companies and 18 energy companies participate. The programme stage has been financed with 60.2 % by participating industrial companies and with 39.8 % by Swedish Energy Agency. The consortium is managed by Elforsk.

The programme shall contribute to increasing knowledge to forward the development of thermal energy processes for various energy applications through improved expertise, refined methods and new tools. The programme shall through material technology and process technology developments contribute to making electricity production using thermal processes with renewable fuel more effective. This is achieved by

- Forward the industrial development of thermal processes through strengthen collaboration between industry, academy and institutes.
- Build new knowledge and strengthen existing knowledge base at academy and institutes
- Coordinate ongoing activities within academy, institutes and industry

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

Abstract

Increased knowledge about the mechanical performance of austenite materials for future application in the area of power generation has been achieved by using slow strain rate tensile deformation testing of austenitic materials at elevated temperature. The results show that the structural development during testing is affecting properties differently depending on alloy composition and applied strain rate.

Sammanfattning

Den världsomfattande ökningen i energianvändning och därmed sammanhängande ökning i koldioxidemission vid förbränning har skärpt kraven på energileverantörer och användare att i större utsträckning använda 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 leder till att anläggningar måste stoppas och startas betydlig oftare än vad som nu är vanligt vilket skapar behov av provningsmetoder som tar hänsyn till variationer med tiden i laster och töjningar. 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 mikrostrukturinducerade hittills inte beaktade deformationsmekanismer såsom tvillingbildning kan förstås och utnyttjas.

I detta projekt har inverkan av långsam deformation vid höga temperaturer på deformation, skadeuppbyggnad och brottegenskaper studerats. Kopplingar till utvecklingen av mikrostrukturer har undersökts med ökad förståelse för hur styrka och duktilitet påverkas. Även om den huvudsakliga plastiska deformationen är dislokationsstyrd så samverkar ett antal fenomen som dynamic strain ageing (DSA), tvillingbildning (TWIP) och olika tidsberoende fas-utskiljningsförlopp. Dessa fenomen kan leda till både förbättring och försämring av materialens prestanda beroende på ett antal faktorer som utreds. Så snart inverkan av legering på dessa fenomen är till fullo klarlagd kan nya och bättre material för användning i olika applikationer utvecklas.

Projektets mål har med god marginal kunnat nås tack vare avdelningen Konstruktionsmaterials tidiga utnyttjande av nya undersökningsmetoder i SEM där Electron Chanelling Contrast Imageing (ECCI) och Electron Back Scattering Diffraction (EBSD) har varit utmärkta redskap.

Samverkan med industriföretaget får i detta projekt anses vara mycket tillfredsställande och antalet publikationer är åtta.

Nyckelord: Dynamisk töjningsåldring, biobränsleanläggning, austenitiska rostfria stål, tvillingar, skada.

Summary

A worldwide increase in use of energy connected to an increase in carbon dioxide emission during combustion has increased the demand on energy producers and consumers to use more biomass fuels and also 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 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. This in turn leads to a need for increased knowledge about how of deformation mechanisms not so often accounted for like twin formation can be better understood and used to improve material properties.

In this project, the influence of low deformation rate 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 strength and ductility are affected. Despite the fact that the plastic deformation is generally dislocation induced, other phenomena like dynamic strain ageing (DSA), twin induced plasticity (TWIP) and different precipitation phenomena are also acting. Those phenomena can lead to both improvements and deterioration of the performance of the materials depending on how they combine, so increased knowledge in this area is important. As soon as the influence of alloying and treatment on these phenomena is wholly understood new materials with better properties can be developed.

The goal fulfilment is high and above expectations due to the use of the modern metallographic methods EDSD and ECCI.

The cooperation with the industry partner is to be considered extremely good and The number of publications produced within the frame of this project is eight.

Keywords: Dynamic strain ageing, biomass power plant, austenitic stainless steel, twins, damage.

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1 Introduction

1.1 Background

Global increase in energy consumption and global warming require increased energy production and lowered CO_2 emission. Increase in efficiency of energy production is an effective way of solving this problem [1]. This can be realised by increasing boiler temperature and pressure in a power plant. By an increase in material temperature of 50°C, efficiency in a biomass fuel power plant can be increased significantly and the CO_2 emission can be greatly reduced. However, the materials used for the future biomass fuel power plants with higher temperature and pressure are required to display improved properties such as higher yield strength, creep strength and high temperature corrosion resistance. Recently, a new advanced heat resistant material: Sanicro25 has been developed [2]. This material shows very high creep strength and high corrosion resistance. Sanicro28 is another advanced heat resistant material with high creep strength and the highest temperature and corrosion resistance among available heat resistant materials [3]. On the other hand, safety and reliability in these new power plants and consequently the materials used become a major concern. This also requires new evaluation methods. Recently, stress relaxation cracking testing and very slow strain rate deformation testing have been proposed to evaluate the long term high temperature behaviour of advanced heat resistant materials. Stress relaxation cracking is a degradation mechanism in austenitic components operating between 550ºC and 750ºC [4-7]. Many austenitic materials appeared to be susceptible to relaxation cracking, especially in welded condition and after cold deformation. So far, stress relaxation cracking behaviour is not well understood since components that has failed by relaxation cracking fully meet the requirements of standard room and high temperature mechanical tests. Increased knowledge about relaxation cracking in austenitic high temperature components is therefore required.

It has been found that very slow strain rate tensile tests data also could be presented in a Larson-Miller (LMP) diagram [8]. Low strain rate tensile tests show features that can lead to possibility to perform analysis similar to the one applied to creep tests at high stresses. This method can also be used to evaluate rupture time and creep rate [9]. In this study, these two methods will be used to evaluate the long term high temperature behaviour of advanced resistant materials. The influence of long term ageing on the structure integrity and safety will also be evaluated.

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-base 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.

Austenitic stainless steels generally have large coefficients of thermal expansion which restricts their use to applications where this is acceptable. In boilers they are primarily found as super heater tubes and sometimes as corrosion resistant barriers in composite tubes at the furnace walls, especially in waste fired boilers and black liquor recovery boilers.

The austenitic stainless steels studied in this project ranges from AISI 304 and 316L, which are thoroughly investigated and well suited for experiments considering new methods or mechanisms. They have been surpassed in strength by higher alloyed steels like AISI 310, and corrosion resistance by for example Sandvik Sanicro™28 that is used in composite tubes in both super heaters and furnace walls, and has interesting mechanical properties in its own at temperatures reaching 650°C. This is also about the maximum temperature for the super heater materials, Super 304H and HR3C, in use today in the most efficient ultra super critical coal fired boilers presently running. At the high end Sandvik Sanicro™25 is capable of use at 700°C material temperature together with the reference nickel base Alloy 617M, intended for steam temperature 700°C in next generation advanced ultra super critical boilers.

Alloy 800HT is a nickel base grade not primarily intended for use in boilers, but rather at other applications such as chemical reactors at temperatures approaching 1000°C. It was included in this project because it has under certain conditions shown some damage mechanisms of interest for this study, notably relaxation cracking.

Some of the materials mentioned above are used in the biomass power plants of today and some are potential materials for the next generation biomass power plant [10]. Biomass is the largest global contributor to renewable energy and has a great potential to expand in the production of heat and electricity [11]. It is a sustainable fuel because it gives no net contribution of CO_2 to the atmosphere and it can be considered endless [1, 11]. However, the global increase in energy consumption and the increase in emissions of greenhouse gases (e.g. CO_2) causing global warming, is pointing out needs for both an increase in energy production and a reduction of greenhouse gas emission [11, 12]. One way to accomplish this is to increase the efficiency of biomass power plants, which could be reached by increasing temperature and pressure in the boiler sections [13]. The requirements of more energy production is thus met and since biomass has no net contribution of CO_2 to the atmosphere smaller emissions of greenhouse gases is the result.

Both groups of austenitic alloys show good mechanical and chemical properties at the operation temperatures of today's biomass power plants [10]. However, materials to be used for 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-base alloys are already operating under such conditions in other applications and the group of nickel-base alloys is a possible option [10, 14]. The nickel-base alloys are more expensive than the austenitic stainless steels and improved austenitic stainless steels are therefore an interesting option to investigate as a material for future biomass power plants. The austenitic stainless steels are of main concern in this work and the nickel-base alloys are so far mainly acting as reference materials.

1.3 Research task

In the general goals of KME's program period 2010-2013 it is stated that:

"The program will contribute to the conversion to a sustainable energy system by development of more effective energy processes."

The purposes of this research project is to gain improved knowledge regarding tensile deformation and cracking behaviour during very slow deformation rate and the influence of long term ageing and tough environments, such as biomass fuel, on structural integrity and safety for advanced heat resistant materials.

Moreover, an evaluation of the degradation mechanism in stress relaxation cracking [4, 5, 15, 16] is aimed to be achieved. In addition, the obtained knowledge will facilitate the development of new materials for the new generation power plants with better performance with respect to temperature, pressure and environmental conditions.

1.4 Goal

The aims of this research are to increase the knowledge concerning:

1. Tensile deformation, damage and cracking behaviour during very slow deformation.

2. Influence of long term ageing on toughness of austenitic alloys for the future power plants.

3. Stress relaxation cracking behavior.

1.5 Project organisation

The project has been run in close cooperation between Linköping University division of Engineering materials and Sandvik Materials Technology (SMT). All activities from project planning, experiments and publishing have been shared activities in most cases. The reference group has included local participants and an external member, Göran Sjöberg and Joel Andersson GKN.

The total budget contribution from KME to LiU is 2.781Mkr.

2 Project acquisition

The project has been run in close cooperation between LiU and SMT and the different tasks have been performed according to schedule. The different research activities have been performed either in Linköping or Sandviken or both and the researchers have been operating either in Sandviken or Linköping depending on facilities used.

In Linköping new equipment for slow strain rate tensile testing was purchased by the division of Engineering Materials. The running in of the equipment was fast and the production of data could start very smoothly. In addition to that new techniques (ECCI) developed in Linköping could be applied directly to the microstructures studied and also applied very quickly in Sandviken. This resulted in a balanced work load between LiU an SMT in experimentation. This also resulted in an increased transfer of knowledge between LiU and SMT. The project has also benefitted from the fact that analysis and discussion of data and writing of publications has been performed in close cooperation with SMT thanks to the possibility of having one of the SMT representatives also acting as a co-supervisor and as an adjunct Professor with regular supervising and project discussions once a week during the project period. The fact that the graduate student quickly became acquainted with the people and equipment in Sandviken made work run very smoothly. All this and the common interest in the technical and scientific matters made the project run nicely according to plan but also allowed for interesting deep diving more deeply into scientific studies. The possibility go to conferences to present and discuss the work together with colleagues and supervisors has been of great importance. Also the participation by the graduate student in the graduate school AGORA MATERIAE (https://www.liu.se/forskning/afm/agora-materiae?l=sv) has been very fruitful.

3 Results and analysis

This section gives a short description of the work that has been performed. More comprehensive presentations of section 3.1 to 3.6 can be found in the licentiate thesis [17] written within this project. Also ongoing research is presented under section 3.7.

3.1 Deformation and damage behaviours of austenitic alloys in the dynamic strain ageing regime

The purpose of this investigation was to investigate the deformation and damage mechanisms related to dynamic strain ageing (DSA) in three austenitic stainless steels (AISI 310, AISI 316L and Sandvik Sanicro™ 25) and two nickel-base alloys (Alloy 617 and Alloy 800HT). The materials were subjected to tensile testing at different elevated temperatures (400 °C to 700 °C). One of the materials was low cycle fatigue (LCF) tested at 650 °C using a strain range of 1,2 %. Since DSA is present in different temperature ranges depending on alloy composition, a scanning electron microscope (SEM) investigation was mainly performed on the specimens tested at 650°C and 700 °C where all five materials displayed DSA. The influence of strain rate can be clearly seen in figure 1 a as an influence on general stress- strain behavior as well as microscopic behavior reflected by the serrations on the curves. The strength and ductility is clearly affected and b,c. Possible origin of the effects on a micro level can be evaluated from the ECCI pictures in figure 2.

As expected deformation in the DSA regime is planar slip and slip bands in single and multi-direction. Local damage has been connected to interaction between slip bands and/or interaction between twins and grain boundaries.

Ductility is affected differently by temperature depending on alloy tested.

DSA is not always related to low ductility of the material, it is therefore suggested that DSA may introduce a phenomenon similar to twinning induced plasticity (TWIP) that could increase the ductility.

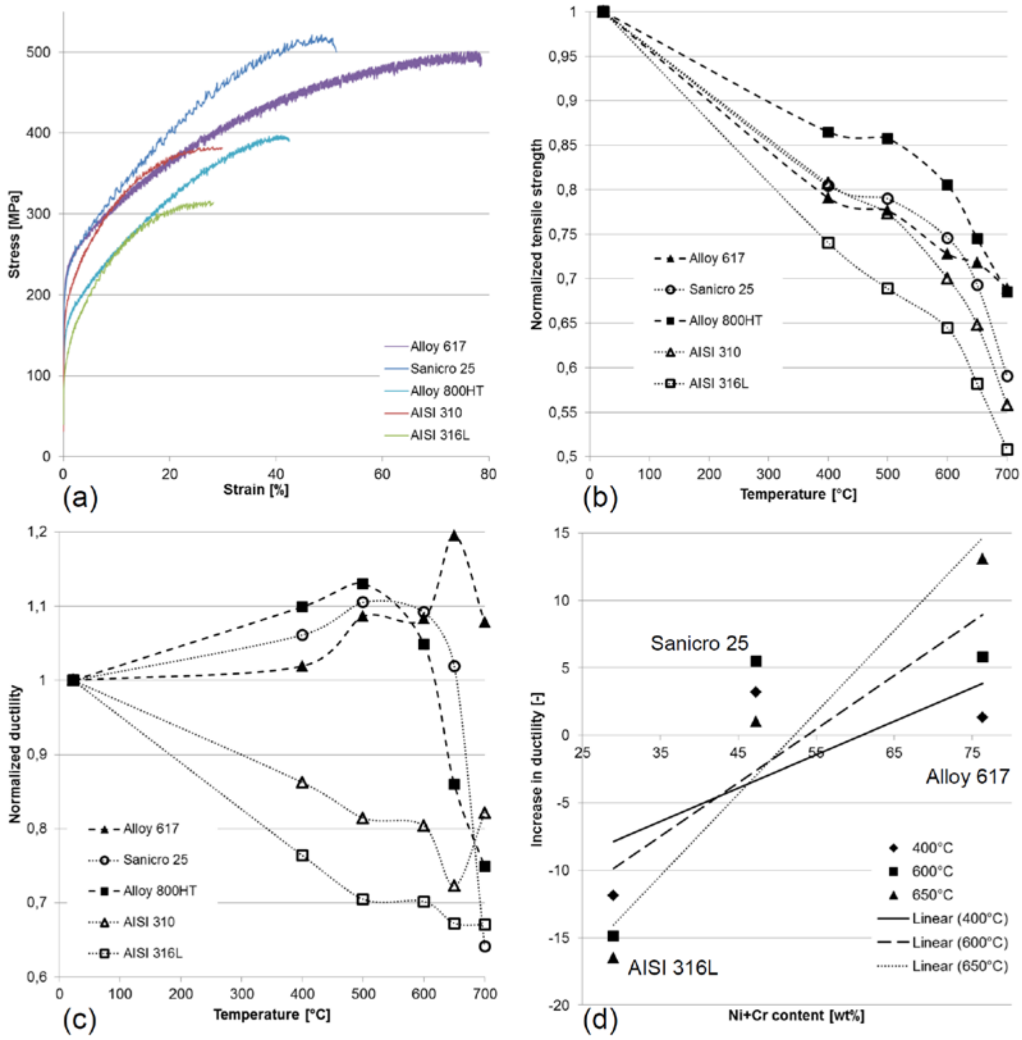


Figure 1 : Mechanical properties of the tested materials, engineering stress-strain curves at 650 °C (a), temperature influence on tensile strength (normalized tensile strength is determined by the tensile strength at elevated temperatures divided by the tensile strength at RT for each alloy) (b), temperature influence on ductility (normalized ductility is determined with the same method as normalized tensile strength) (c) and influence of nickel and chromium on ductility at elevated temperature (increase in ductility is determined by the ductility at RT subtracted from the ductility at the elevated temperatures) (d).

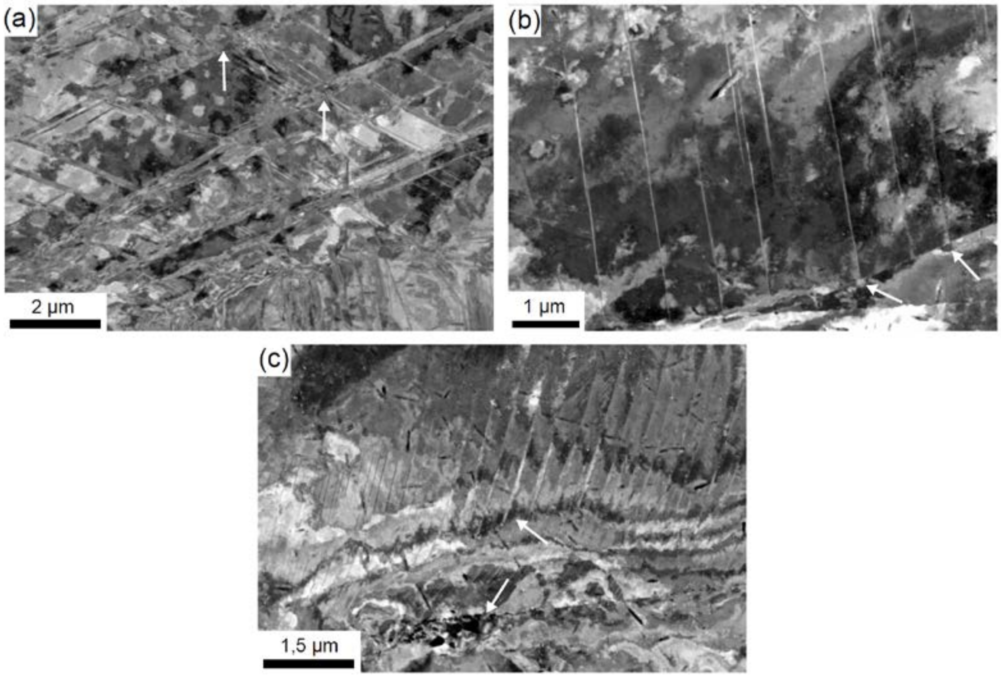


Figure 2. Deformation and damage behaviour in AISI 310 during the tensile testing, at
RT formation and interaction of DB’s (a), interaction of twins and GB at 600 °C (b) and interaction of twins or SB’s and GB at 650 °C (c) generates damage.

3.2 Influence of deformation rate on mechanical response of an AISI 316L austenitic stainless steel

This study is mainly concerned with high-temperature behavior during uniaxial slow strain rate tensile testing of an AISI 316L material, commonly used for components in power plants. Uniaxial slow strain rate tensile testing (SSRT) has been performed at different temperatures up to 700 °C using strain rates down to 10^{-6} s^{-1}. An investigation of the microstructure was conducted on the deformed and fractured materials using mainly the scanning electron microscopy methods, electron channeling contrast imaging (ECCI) and electron backscatter diffraction (EBSD), to capture the microstructural mechanisms coupled to the mechanical behaviors seen in engineering stress-strain curves.

It was found (Figure 3) that DSA occurs in AISI 316L during tensile testing at temperatures of 650 °C and 700 °C when using a strain rate of $10^{-6}\,s^{-1}$. The strength decreases with increasing temperature and decreasing strain rate. Total elongation on the other hand increases with decreasing strain rate applied at the same temperature, this has been observed at both room temperature (RT) and elevated temperature (up to 700 °C). Dynamic recrystallization

(DRX) can occur during tensile deformation at elevated temperature. At a low strain rate ($10^{-6}\,s^{-1}$) DRX are more homogeneously spread in the microstructure. The presence of dynamic recovery together with dynamic recrystallization can be seen (Figure 4) to effect the appearance of

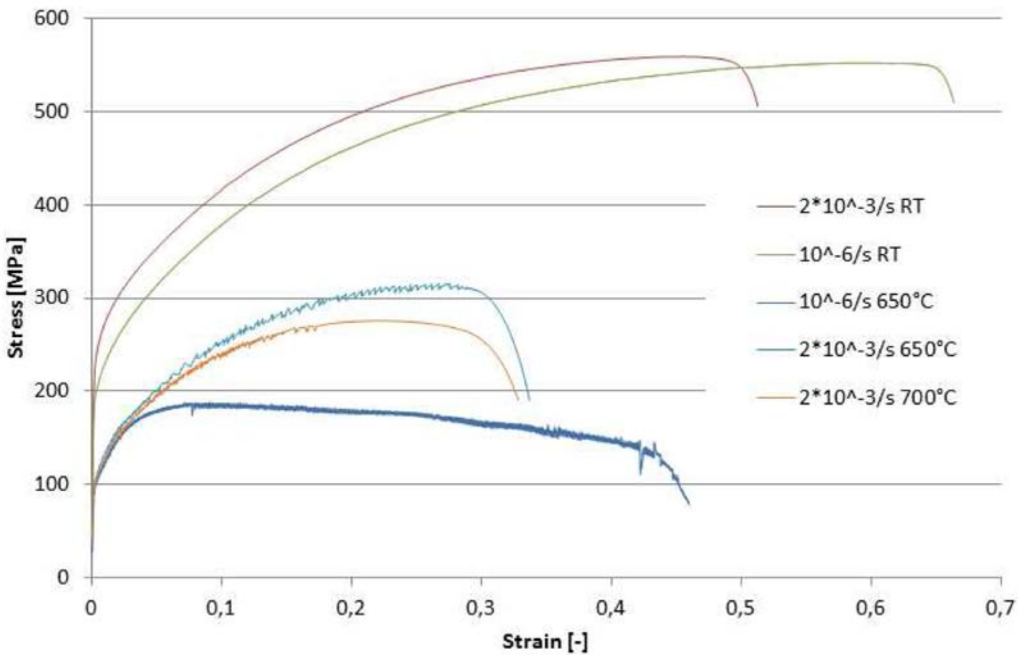


Figure 3. Tensile behaviours of AISI 316L at different temperatures and strain rates (engineering stress and strain).

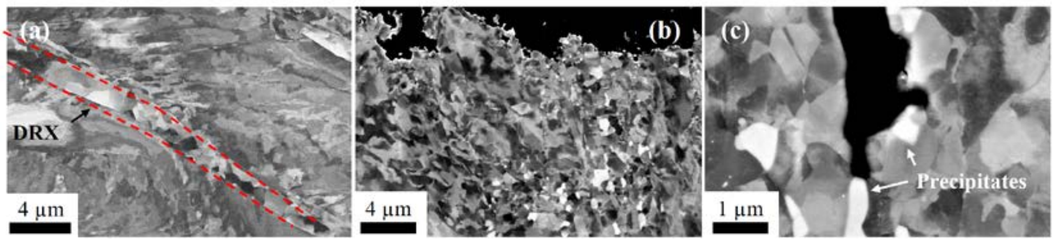


Figure 4. ECCI pictures at 650 °C with longitudinal load directions, (a) DRX band, using a strain rate of $2*10^{-3}\,s^{-1}$ and (b) homogeneously spread DRX near fracture surface, (b) small crack in recrystallized microstructure with precipitates, using a strain rate of $10^{-6}\,s^{-1}$ in both (b) and (c).

3.3 Mechanical behaviors of Alloy 617 with varied strain rates at high temperatures

Focus is here put on the deformation and damage mechanisms in Alloy 617. The alloy is deformed using low strain rates (down to 10^{-6} s^{-1}) at elevated temperatures (650 °C and 700 °C) by uniaxial slow strain rate tensile testing SSRT and microstructure evaluation using scanning electron microscopy techniques as electron channeling contrast imaging. In Figure 5 the influence of strain rate on the tensile properties is shown.

Dynamic strain ageing (DSA) can occur in Alloy 617 at temperature 650 °C and 700 °C with strain rate from 10^{-2} s^{-1} down to 10^{-6} s^{-1}. Twin induced plasticity is one of the mechanisms for a high elongation during DSA. Both strength and elongation increase with decrease of strain rate down to 10^{-4} s^{-1}, and then both decrease with further decrease of strain rate. Micro and nano dynamic recrystallization (DRX) can occur during the tensile deformation using very low strain rates. Repeated DRX can lead to the formation of damage in the material.

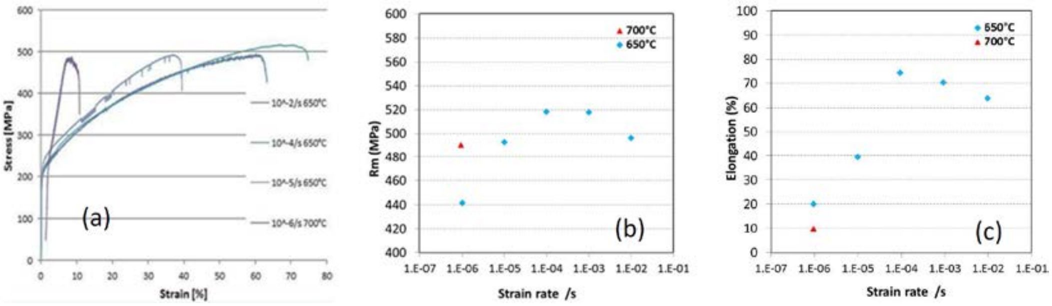


Figure 5.Influence strain rate on the tensile properties of Alloy 617 at elevated temperatures. (a). Stress versus strain curves, (b). Tensile strength, Rm, versus strain rate, (c). Elongation versus strain rate.

3.4 Damage and Fracture Behaviours in Aged Austenitic Materials During High-Temperature Slow Strain Rate Testing

The purpose of this study was to investigate damage and fracture mechanisms of high-temperature long term aged austenitic materials (the austenitic stainless steel AISI 304 and the nickel-base alloy Alloy 617) during uniaxial low strain rate tensile testing (SSRT) at room temperature (RT) and elevated temperature (Figure 6). The role of precipitation from high-temperature ageing and the long deformation process is evaluated by microscopy and coupled to the damage and fracture behaviour.

The investigation showed (Figure 7) that SSRT caused intergranular cracking in both high-temperature long term aged AISI 304 and Alloy 617 at both RT and 700 °C when using a strain rate of (10^{-6} s^{-1}). At RT the fracture is caused by cracks initiated due to stress concentration formed by the precipitates from the ageing process in the grain boundaries (GB's) for both alloys. Alloy 617 also exhibit crack initiation and propagation by slip band interaction with the small GB precipitates. At 700 °C the fracture is caused by GB precipitates formed during both the ageing process and the tensile deformation. Elongation to fracture decreases for both the aged stainless steel and the aged nickel-base alloy when a lower strain rate is used compared with a higher strain rate at 700 °C.

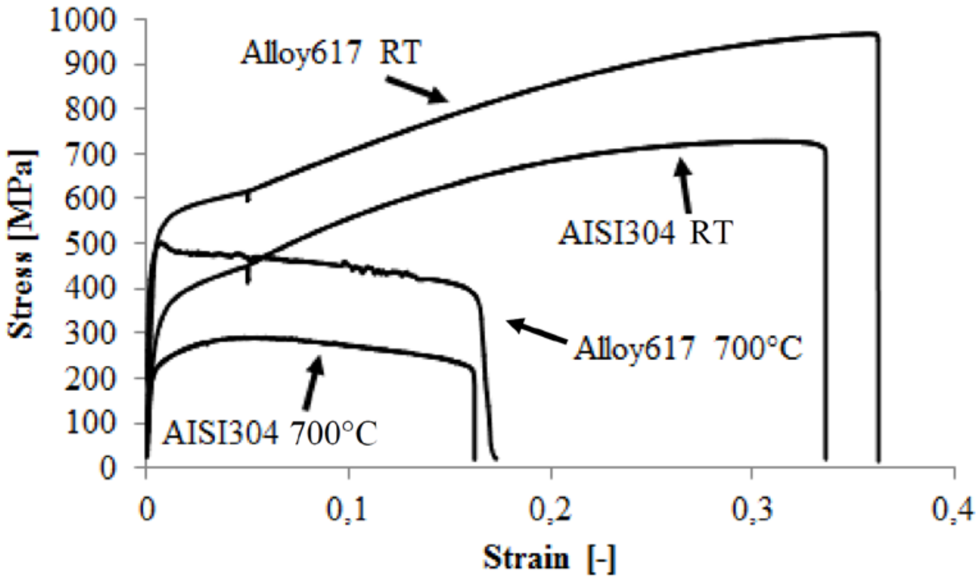


Figure 6.Engineering stress-strain curves show AISI 304 and Alloy 617 aged and SSRT

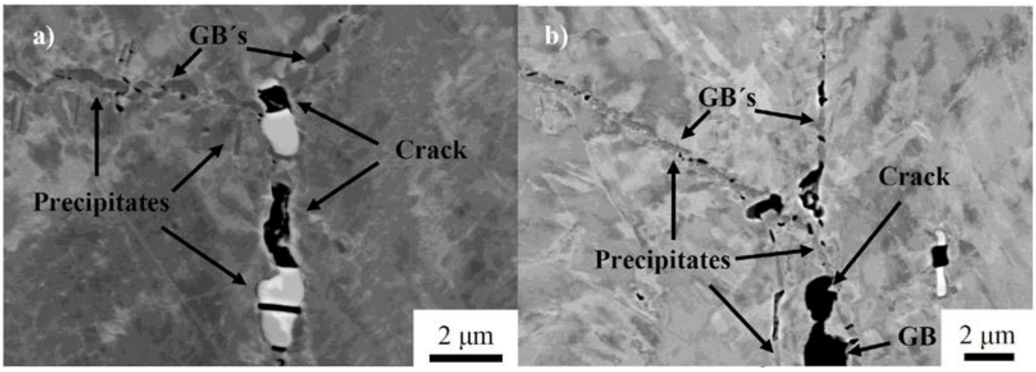


Figure 7. AISI 304 tensile tested a) at RT and using a strain rate of 10^{-6}/s, displaying cracks in GB due to stress concentrations formed by precipitates, both primary and from the ageing process and b) show a crack propagated in the GB due to stress concentrations formed by precipitates when tested using a strain rate of $2*10^{-3}$/s at 700°C.

3.5 Advanced Microstructure Studies of an Austenitic Material Using EBSD in Elevated Temperature In-Situ Tensile Testing in SEM

In this study in-situ tensile testing was performed on Sanicro 25 at two different temperatures. An investigation of the influence of temperature on the deformation behaviour was performed using the EBSD technique. Fracture behaviour will also be discussed.

The analysis from in-situ tensile test at room temperature (RT) and 300 °C in a SEM together with EBSD in Sanicro 25 has shown that, larger grains may tend to accumulate more local plastic strain for the same macroscopic strain values at both temperatures (Figure 8). Somewhat higher plastic strains at grain level can be obtained at RT than at elevated temperature for the same macroscopic strain value. Cracks initiate and propagate along the slip system(s) with the highest Schmid factor at RT.

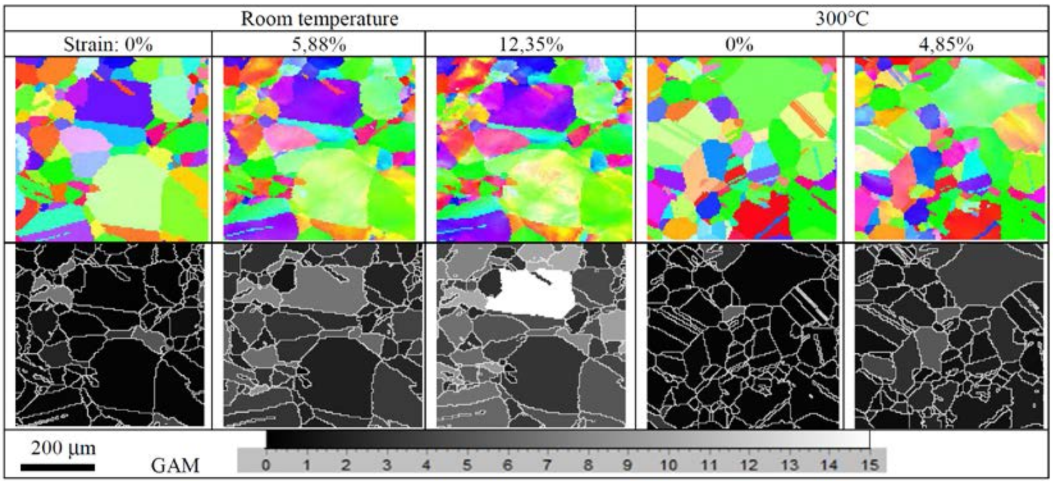


Figure 8. IPF (row 1) and grain average misorientation maps (row 2) with scale bars (row 3). The loading axis lies horizontally

3.6 Influence of High Temperature Ageing on the Toughness of Advanced Heat Resistant Materials

The influence of precipitation and growth of precipitates on toughness due to different chemical compositions and high-temperature treatment is investigated. The experiments were conducted on two austenitic stainless steels (AISI 304 and Sandvik Sanicro™ 28) and one nickel base alloy (Alloy 617). Impact toughness test have been performed and the fracture surface and cross-section have been investigated using microscopy (Figure 9-10). Thermo-Calc have been used to predict possible precipitates.
From the study it was found that the fracture initiation and propagation in the aged austenitic stainless steel is very local. The initiation and propagation of fracture behave differently in these materials due to different chemical compositions affecting nucleation, growth and shape of precipitates.
The brittle σ-phase can appear in the austenitic stainless steel after 1 000 hours at 650 °C and then increases in amount. The amount and shape have strong effect on the fracture behaviour, where needle shaped σ-phase which mostly appear at high temperature (700 °C) after longer ageing time (3 000 hours) lead to a low impact toughness and brittle fractures both locally and on a macro-level in the specimen (Figure 9). The nickel base alloy show higher impact toughness with increasing ageing temperature and time (Figure 10).

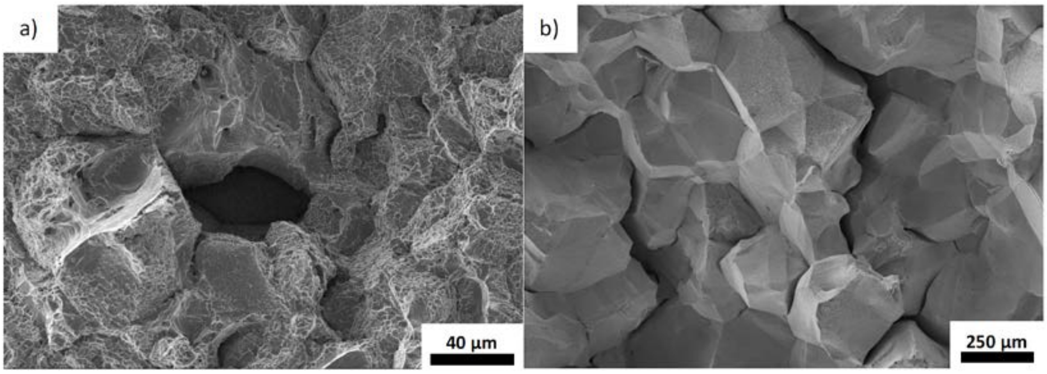


Figure 9. Pictures showing fracture surfaces from the impact tested samples, a) AISI 304 and b) Sanicro 28, both materials have been aged at 650°C for 3000h.

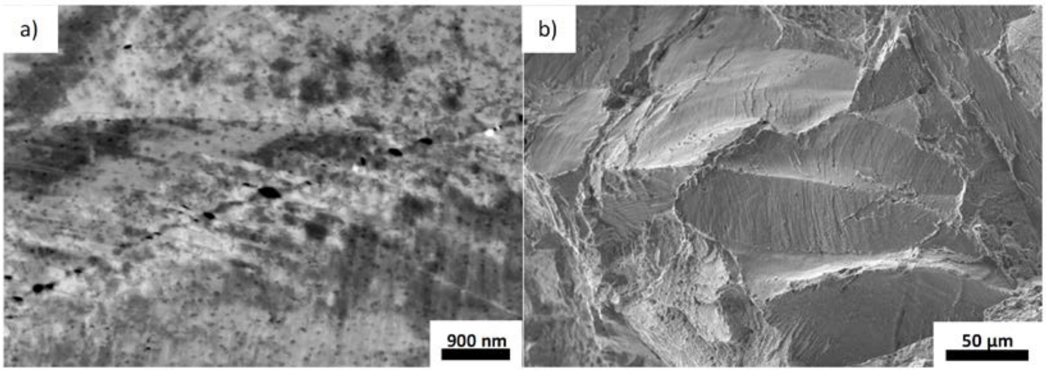


Figure 10. Precipitates in the aged Alloy 617 material at 700°C for 3000h and b) fracture after the impact toughness testing

3.7 Ongoing research

Three research's series have been started but not yet been finished or analysed. Following section will describe the testing method and what will be done to complete the testing series.

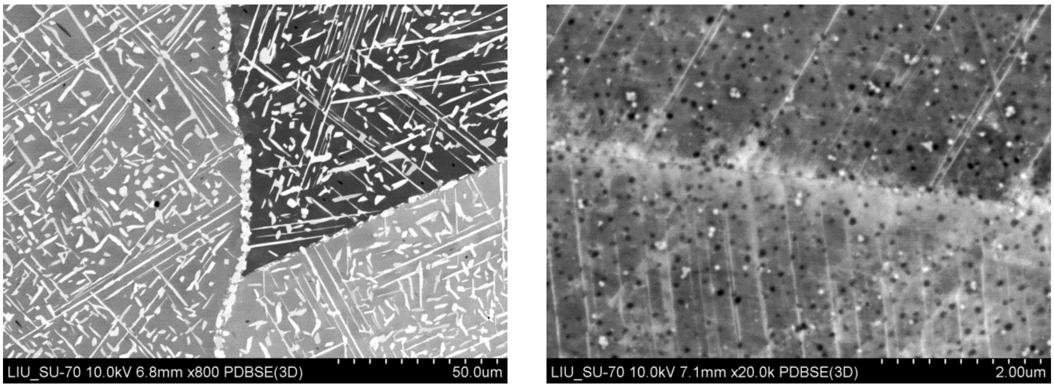
3.7.1 Long term high-temperature ageing

Within the project an investigation of the influence of long term ageing on structural integrity and safety of advanced heat resistant materials has been performed. The objective was to investigate the toughness of material that had been aged for up to 30 000 hours (roughly 3,4 years), at present material have been aged for 1 000, 3 000 and 10 000 hours at 650 °C and 700 °C and then been tested and analysed. The aged materials have been tested using impact toughness and fracture toughness methods, using Charpy V and CTOD respectively. Some results are showed in section 3.6, some results have not yet been published but will be be presented soon in a conference and some results are not yet decided when to be published. A combination of long term ageing (500 hours at 700 °C) and slow strain rate tensile testing have been performed and the results are presented in section 3.4. The results excluded in sections 3.4 and 3.6 not yet published results are presented below.

3.7.2 Influence of long term ageing on impact toughness

Parts of this research have been accepted for presentation on the International Conference on Fracture and Damage Mechanics in the summer of 2014. The influence of a 10 000 hours ageing process on impact toughness performance was investigated and a comparison with material aged for 1 000 and 3000 hours.

It was found that at room temperature brittle precipitate σ-phase is detrimental for the austenitic stainless steels (AISI 304 and Sandvik Sanicro™ 28). These austenitic stainless steels suffer greatly from embrittlement due to the σ-phase and the amount of σ-phase increases with increasing ageing-time and –temperature, creating a more brittle material with time. However, the investigated nickel-base alloy (Alloy 617) shows a much better impact toughness performance after 10 000 hours ageing at both 650 °C and 700 °C. Impact toughness value is higher for 700 °C than 650 °C. This could be attributed to the evenly distributed and small precipitates in the microstructure (figure 11). Since the Al-content is low these precipitates are more likely $M_{23}C_6$ carbides rather than gamma prime.



(a) (b)

Figure 11. Precipitation after 10 000 hours at 700 °C, (a) Sanicro 28 and (b) Alloy 617.

3.7.3 Influence of long term ageing on crack propagation mechanism

The findings from the fracture toughness testing on long term aged austenitic materials have been presented at the KME-conference. There are differences between the tested alloys concerning crack propagation mechanisms. The different performances have been connected to a different microstructural development. A material with high CTOD shows plastic deformation by twinning and dislocation slip. A formation of a severe plastic deformation (SPD) zone near the crack is observed, followed by crack propagation in the SPD zone (figure 12 (a) and (d)). Material with relative high CTOD shows formation of slip or shear band and then crack propagation (figure 12 (b) & (e)). For materials showing low CTOD the crack initiates at precipitates at grain boundaries (GB), and then propagates along the grain boundaries (figure 12 (c) and (f)).

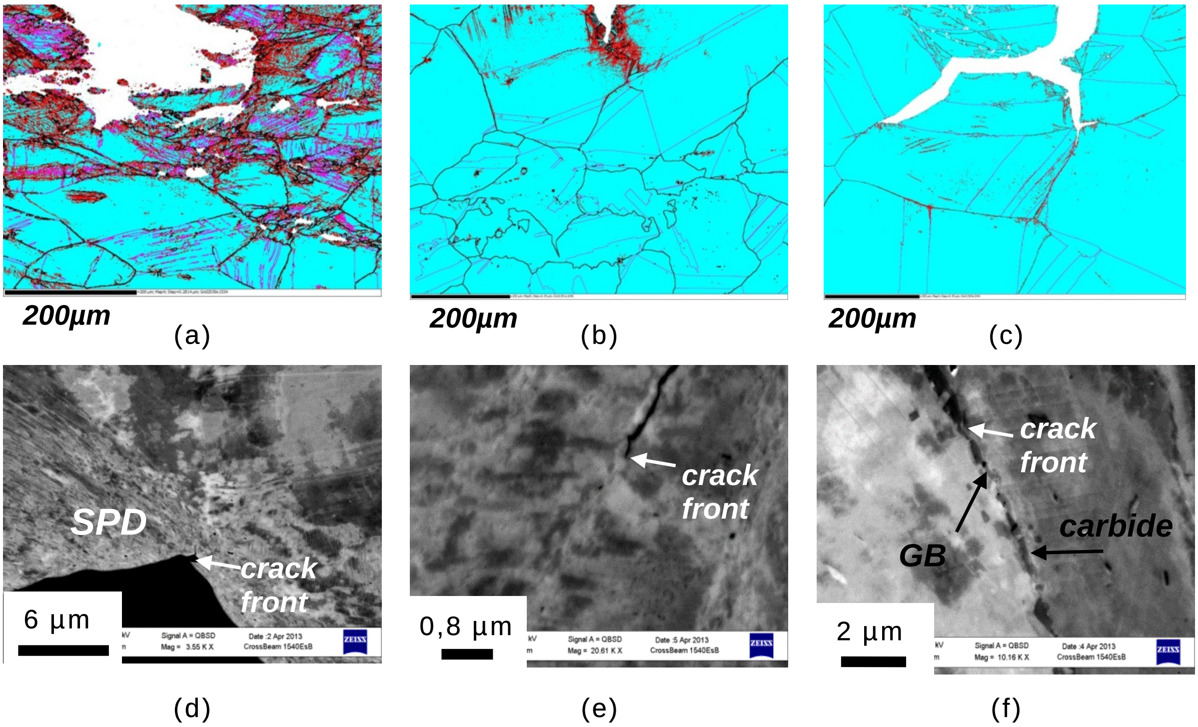


Figure 12. (a) & (d) Alloy 617 CTOD=2,42 mm aged at 650 °C for 3 000 hours, (b) & (e) Sanicro 2 8 CTOD=0,65 mm aged at 650 °C for 3 000 hours and (c) & (f) Sanicro 28 CTOD=0,15 mm aged at 700 °C for 3 000 hours.

3.7.4 Stress relaxation cracking

Stress relaxation cracking (SRC) testing is performed both in Sandviken (SMT) and Linköping (LiU).

Screening test

A screening test was designed to find out how susceptible to stress relaxation cracking a number of selected alloys are. The samples were plastically deformed in bending to resemble for example the bending of tubes in a superheater, and then aged at two temperatures and three strain levels during three different times.

The test samples were taken from tube walls. The dimensions were chosen such that the same geometry could be achieved from all alloy tubes, 5,0x5,0x70 mm. The samples were then bent by pressing a cylindrical stamp over the sample into a die (figure 13).

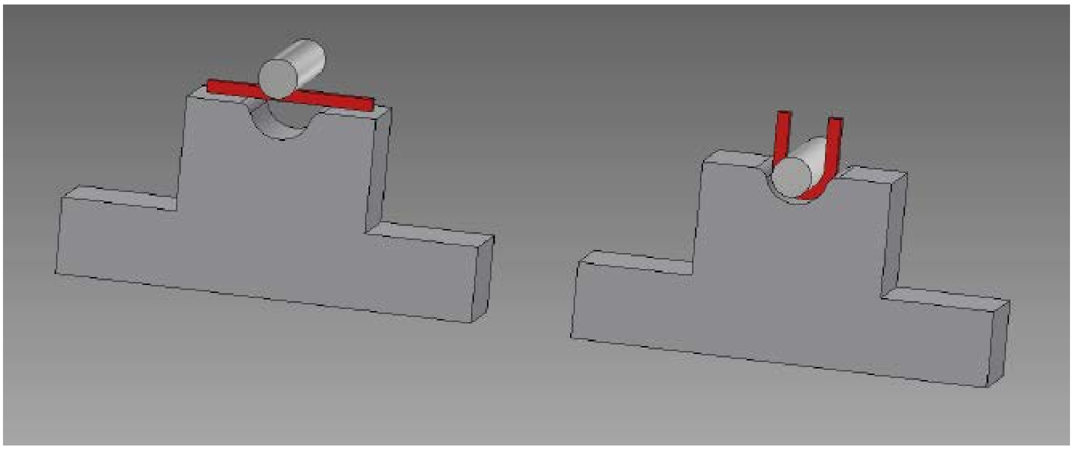


Figure 13. Bending of test pieces from tube walls.

Under the assumptions that there is compression at the inner radius and strain ε in the outer radius after bending, and from symmetry there is a neutral layer at radius R at half the sample thickness t (figure 14), strain in the outermost fibre can be approximately calculated according to

$$\varepsilon = \frac{t}{2R} \qquad (1)$$

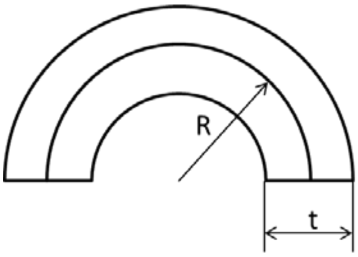


Figure 14. Geometry of bent sample.

The tools were designed to give the neutral layer of the samples the radii 50mm, 16,7mm and 8,3mm corresponding to a strain of 5%, 15% and 30% in the outermost fibre of the sample (figure 15).

Figure 15. Bent samples exposed at 650°C for 24h.

The expected result is to find relaxation cracks in the strained area of the samples if susceptible to SRC. The various exposures give a ranking of the susceptibility between the different alloys.
Few results have been produced and some patterns that may be cracks were documented however it is necessary to section the test pieces to examine the interior to decide if it is cracks or something else creating the pattern on the surface. In figure 16, there is a wide horizontal mark, along the sample, that may be a scratch from the bending procedure, and there are smaller vertical patterns, cross the sample, that may be cracks.

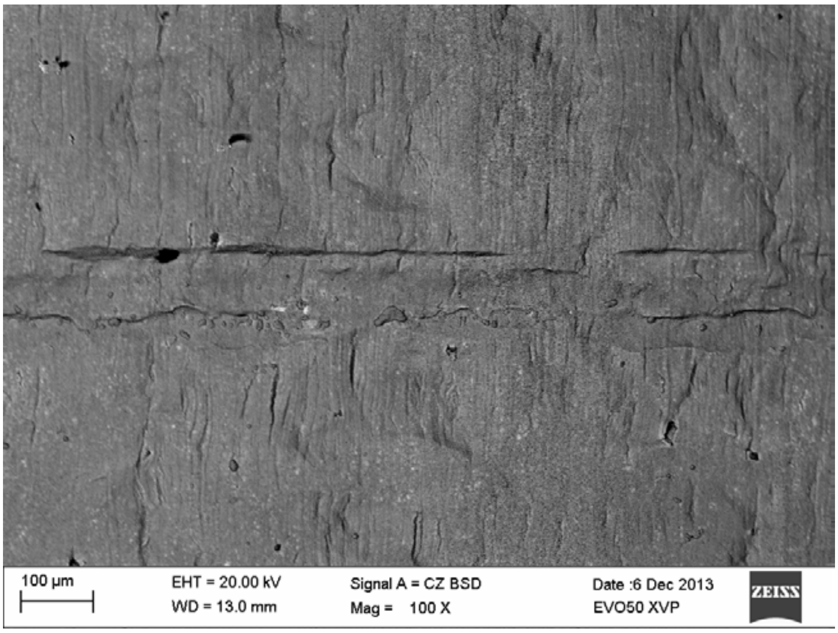


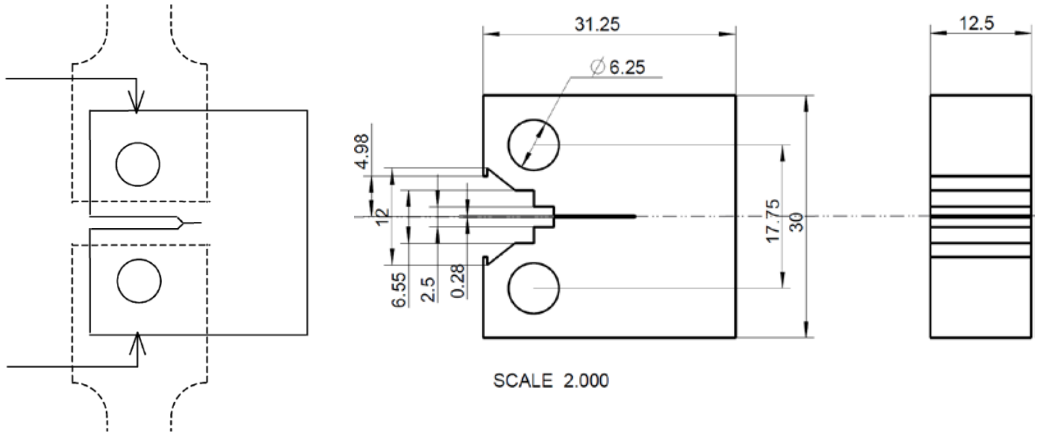
Figure 16. Outer radius surface of bent sample of Alloy 800HT, radius 8,3mm, 650°C, 1000h.

Tools for further evaluating the samples include LOM and SEM to identify cracks, and EBSD for a deeper study of the interaction between crack and matrix.

Useful knowledge regarding the screening method have been gained, it seems to be advantageous to use a sample design where the sample cannot be able to flex back when heated.

Mechanism evaluation method
A method to capture the mechanisms behind SRC is under development. A CT-specimen and tensile test machine with potential drop (PD) (figure 17) is used. A crack is propagated giving a plastic zone. Different plastic zones are used to represent different deformation degrees. The plastic zones are calculated with only the load range variable. Then the specimens will be tensile loaded up to the maximum load from the crack propagation and fixed at that position. Then the load will be monitored by the load cell and the crack propagation will be monitored using a PD-method. Right now the PD-equipment's are under calibration and testing, after this the testing can be stared directly. The tests will be performed at 650°C and 700°C for at

maximum 500 hours. This research will give information about the crack propagation as propagation rate and also the amount of stress relief. Fracture surface and microstructure will be analysed using SEM techniques as ECCI and EBSD. Sanicro 25, Alloy 800HT and AISI 347 will be used in these tests.



(a) (b)

Figure 17: (a) schematic drawing of CT-specimen with PD and (b) drawing of the CT-specimen that will be used.

3.7.5 Cyclic corrosion testing

Testing in corrosive environment was performed at 650°C in a cyclic rig (figure 18). The specimens were placed on a stationary ceramic table and the thermal cycling was accomplished by lowering and rising a furnace over the specimens. During the hot part of the cycle, the furnace was lowered and rested over the specimens for 96 h; during the cold part of the cycle, the furnace rose and a lid closed the furnace to minimize heat loss. The furnace remained in that position until the specimens had reached a temperature of 100°C through natural cooling; this took 18 min.

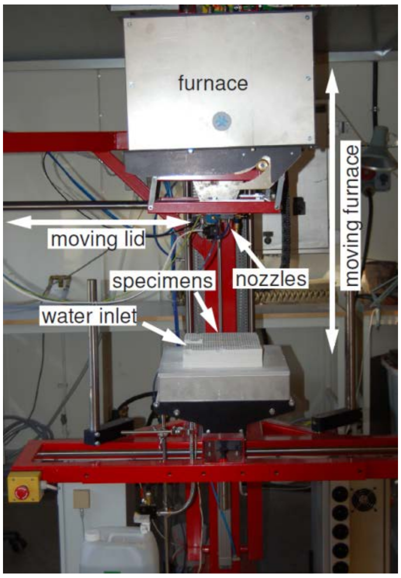
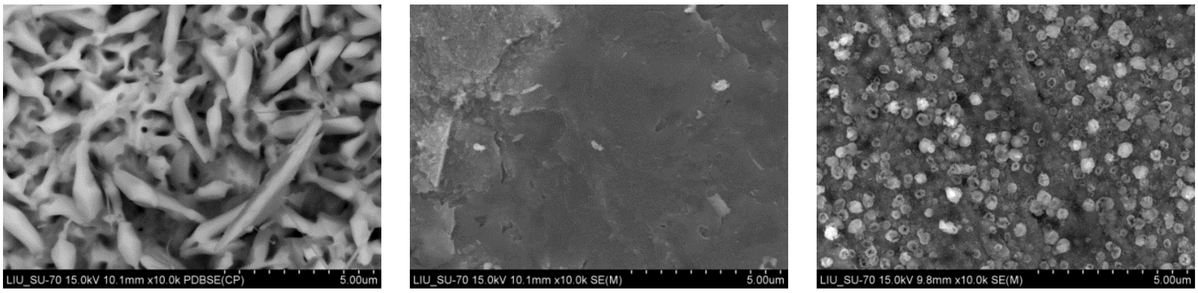


Figure 18: Cyclic furnace used for cyclic corrosion testing.

During the hot part of the cycle, water was introduced as an atomized air-water mist which was sprayed into the furnace (not directly onto the specimens). The atomized air immediately evaporated as it was sprayed into the furnace and increased the water vapor content in the furnace chamber. The amount of water vapor was controlled by the water-to-air ratio in the water mist and was adjusted to 15 mol%. The furnace was flushed through every 3rd min. Solution heat-treated specimens with dimensions 10x10x5 mm and ground surfaces (500 SiC paper 30 µm) were used. All alloys within the project are tested for 20, 50, 100, 200 and 300 cycles. Also static oxidation testing has been performed at 650°C for 1000 and 3000 hours, as reference materials. At this moment the last specimens have been taken out of the furnace, some of the samples have been analysed using XRD, EDS and WDS. Selected specimens will be analysed using XRD, EDS and WDS for oxide composition and Light optical microscopy (LOM) and SEM for oxide kinetics. Also simulation using ThermoCalc and Dictra will be performed to simulate the corrosion and oxidation evolution. This research will give additional information about corrosion properties, kind of oxides and oxide growth kinetics of the different alloys within the project, see figure 19.



(a) (b) (c)

Figure 19: (a) FeO, (b) Fe and Cr oxide probably a spinel on AISI 304 and (c) granular Ni and Cr spinel and NiO on Alloy 617 after 100 corrosion cycles.

4 Discussion of results

A study of austenitic materials and their behaviour at high temperature in all five austenitic stainless steel and two Ni-base alloys. Deformation, damage and fracture behaviour during slow strain rate deformation together with long term ageing has been focused on.

The results have been presented and discussed in five different published papers dealing with deformation and damage behaviour and dynamic strain ageing, deformation rate of AISI 316, mechanical behaviour of Alloy 617, behaviour of aged materials, detailed studies of in situ microscopic behaviour, ageing and toughness and stress relaxation cracking.

Although austenitic materials have been studied for decades with focus on deformation, temperature and microstructure this work has revealed a number of thing that were perhaps hidden in all detailed results from testing and TEM-studies. In this study 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 it more easy to discuss the results. This has also shown that it is possible to observe and discuss possible interaction between not only dislocation based deformation mechanisms but also between phenomena like dynamic strain ageing, twin formation and damage mechanisms. The use of new techniques like Electron Channelling Contrast Imaging and Electron Back Scattering Diffraction as a possible replacement for Transmission Electron Microscopy and X-ray analysis has made it possible to perform more direct studies going from a micro level to macro level to get a better overview. This has also made discussion about the influence of microscopic phenomena on macroscopic properties more reliable. The idea of systematic variation in strain rate during testing has proven to useful not only because the user profile tend to be more cyclic but also for more scientific reasons. The results produced in this project will probably be of great importance for development of new materials and a useful source of information in design of future energy conversion plants.

5 Conclusions

The presented research within this work deals with high-temperature behaviour of austenitic alloys, five austenitic stainless steels and two nickelbase alloys, with focus on deformation, damage and fracture behaviour during slow strain rate deformation and the influence of long term ageing.

It was found that the main deformation mechanisms in the austenitic alloys are planar dislocation deformation, such as planar slip, slip bands in single and multi-direction. Twinning has also been observed and related to the dynamic strain ageing phenomenon, dynamic strain ageing occurred in all tested materials but at different temperature ranges. The ductility performance of the austenitic alloys is influenced differently by temperature when subjected to tensile deformation. Dynamic strain ageing may not always lead to a lower ductility of the material, it is suggested that dynamic strain ageing may introduce a phenomenon similar to twinning induced plasticity that could increase the ductility. However, the ductility performance of some of the tested austenitic alloys increased when subjected to a slow strain rate tensile deformation process at elevated temperatures compared when using a higher deformation rate at the same temperature. While others showed an increase in ductility when decreasing to a certain deformation rate and then the ductility performance decreased with decreasing deformation rate.

During these tests recovery phenomena, like dynamic recovery and dynamic recrystallization appeared in some of the tested alloys, both in stainless steel and nickel-base alloys. In aged conditions the tested austenitic alloys showed only a decrease in ductility with decreasing deformation rate. This is attributed to the formation of precipitates in the grain boundaries (GB) creating stress concentration that causes intergranular fracture to appear. Also, in some of these specimens dynamic recrystallization was observed.

The analysis from electron backscattered diffraction and in-situ tensile test in a scanning electron microscope at room temperature and elevated temperature in Sanicro 25 has shown that, larger grains may tend to accumulate more local plastic strain for the same macroscopic strain values at both temperatures. Somewhat higher plastic strains at grain level can be obtained at room temperature than at elevated temperature for the same macroscopic strain value. At room temperature cracks initiate and propagate along the slip systems with the highest Schmid factor.

Local damage has been connected to an interaction between slip bands and/or interaction between twins and grain boundaries. Repeated dynamic recrystallization may also lead to the formation of damage.

It has also been found that the fracture initiation and propagation in the aged austenitic stainless steel is very local during impact testing. The initiation and propagation of fracture behave differently in the austenitic alloys due to different chemical compositions affecting nucleation, growth and shape of precipitates. The brittle σ-phase can appear in the austenitic stainless steels AISI 304 and Sanicro 28 after 1000 hours at 650°C and then increases in amount. The amount and shape have strong effect on the fracture behaviour, where needle shaped σ-phase which mostly appear at high temperature (700°C) after longer ageing time (3000 hours) lead to a low impact toughness and brittle fractures both locally and on a macro-level in the specimen. However, the tested nickel base alloy showed higher impact toughness with increasing ageing temperature and time.

6 Goal fulfilment

The purposes of this proposal are to evaluate stress relaxation cracking behaviour, tensile deformation and cracking behaviour with very slow strain rate for advanced heat resistant materials. The influence of long term ageing and rough environments such as biomass fuel on the structure integrity and safety will also be evaluated by using failure assessment diagram. The work has been divided into three parts with tasks and subtasks.

6.1 Part One: Stress relaxation cracking behaviour

6.1.1 Task 1-1 Theoretical and physical considerations of the testing method

Testing and analysis partly performed but the modelling part not yet performed.

6.1.2 Task 1-2 Stress relaxation cracking behaviour of heat resistant materials

Testing and analysis performed. Extended SEM analyses are still left of the screening tests. Testing have been performed by the new mechanism evaluation method described in section 3.7.2, it is under development and analyses and more testing to evaluate the new method and SRC are needed.

6.1.3 Task 1-3 Correlation between the stress relaxation cracking behaviour and safety/reliability

No correlation has been performed but some useful results about the screening testing have been gained and a new method is under development. The results so far can be used as tools for further investigations.

6.2 Part Two: Very low strain rate tensile testing SSRT (10^{-5} to 10^{-6}/s)

A large number of tests and analysis have been made (section 3.1-3.5) and all materials have been tested.

6.2.1 Task 2-1 Development of some new testing method by simulation

Data produced to allow simulation in further work.

6.2.2 Task 2-2 Characterization of the SSRT behaviour of heat resistant materials

SEM techniques as STEM, ECCI, EBSD, EDS and in-situ EBSD have been used to characterise the tested materials (section 3.1-3.6). Mostly as-received material has been tested but also tests on few aged materials have been performed (section 3.4) but no test have been performed on cold deformed material due to lack of time.

6.2.3 Damage and fracture mechanisms of SSRT

The damage and fracture mechanisms have been studied and mapped for the different austenitic materials. Several publications (section 3.1-3.6) have been written on the subject.

6.3 Part Three: Structural integrity of long term aged (1 000 to 30 000 hours) advanced heat resistant materials

6.3.1 Task 3-1 Influence of long term ageing on microstructures

Thermo-Calc has been used to simulate the effect of long term ageing regarding precipitation. All materials have been aged at 650 °C and 700 °C and this far for 10 000 hours. SEM techniques as ECCI and EDS have been used to analyse precipitates and their amount, location and size.

6.3.2 Task 3-2 Influence of long term ageing on ductility and fracture toughness

All materials have been aged at 650 °C and 700 °C and up until now below 10 000 hours and impact and fracture toughness tested, using Charpy V and CTOD method respectively. SEM techniques as ECCI, EBSD and EDS have been used to analyse the influence of precipitates on the impact and fracture toughness.

6.3.3 Task 3-3 Structure integrity evaluation

As mentioned above all materials have been aged at 650 °C and 700 °C and this far for 10 000 hours and impact and fracture toughness tested, using Charpy V and CTOD method respectively. The structural integrity has been evaluated and is more in detail covered in section 3.6 and 3.7.1.

6.4 Summary

The goal fulfilment is considered to be complete provided the ongoing research reported is included. The project tasks have been performed only with minor modifications of the tasks and the time plan.

7 Suggestions for future research work

Suggestion of future work is mainly studies of the influence of high-temperature environments on the mechanical behaviour of austenitic stainless steels.

The ongoing long term ageing experiment that is planned to last for 30 000 hours investigates its influences on structure integrity and safety. By using evaluation method as impact toughness and fracture toughness tests.

A continued and deeper study of stress relaxation cracking behavior of austenitic stainless steel in tough high-temperature environment is also suggested since the mechanism influence the biomass power plant life.

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9 Publications

9.1 Journal papers

1. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
   Deformation and damage behaviours of austenitic alloys in the dynamic strain ageing regime
   Conditionally accepted at Engineering Fracture Mechanics.

9.2 Conference Articles

1. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
   Mechanical Behaviours of Alloy 617 with Varied Strain Rate at High Temperatures
   THERMEC '2013, International Conference on Processing & Manufacturing of Advanced Materials. Processing, Fabrication, Properties, Applications. December 2-6, Las Vegas, USA, 2013.
2. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
   Influence of deformation rate on mechanical response of an AISI 316L austenitic stainless steel
   THERMEC '2013, International Conference on Processing & Manufacturing of Advanced Materials. Processing, Fabrication, Properties, Applications. December 2-6, Las Vegas, USA, 2013.
3. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
   Damage and Fracture Behaviours in Aged Austenitic Materials During High-Temperature Slow Strain Rate Testing
   Seventh International Conference on Materials Structure & Micromechanics of Fracture (MSMF7) 1-3 July 2013, Brno, Czech Republic, 2013. Key Engineering Materials, 2014, 592-593, 590-593.
4. Mattias Calmunger, Ru Peng, Guocai Chai, Sten Johansson and Johan Moverare
   Advanced Microstructure Studies of an Austenitic Material Using EBSD in Elevated Temperature In-Situ Tensile Testing in SEM
   Seventh International Conference on Materials Structure & Micromechanics of Fracture (MSMF7), July 1-3, Brno, Czech Republic, 2013. Key Engineering Materials, 2014, 592-593, 497-500.
5. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
   Damage and Fracture Behaviours in Advanced Heat Resistant Materials During Slow Strain Rate Test at High Temperature
   13th International Conference on Fracture (ICF13), June 16-21, Beijing, China, 2013.
6. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
   Influence of High Temperature Ageing on the Toughness of Advanced Heat Resistant Materials
   13th International Conference on Fracture (ICF13), June 16-21, Beijing, China, 2013.

7. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
Influence of Dynamic Strain Ageing on Damage in Austenitic Stainless Steels
19th European Conference on Fracture (ECF19), Fracture Mechanics for Durability, Reliability and Safety, August 26-31 2012, Kazan, Russia, 2012.

8. Mattias Calmunger, Guocai Chai, Sten Johansson and Johan Moverare
Long term high-temperature environmental effect on impact toughness in austenitic alloys
Accepted for presentation at the 13th International Conference on Fracture and Damage Mechanics (FDM13), 23-25 September, São Miguel Island, Portugal, 2014.

9.3 Licentiate Thesis

Mattias Calmunger
High-Temperature Behaviour of Austenitic Alloys: Influence of Temperature and Strain Rate on Mechanical Properties and Microstructural Development
2013.

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