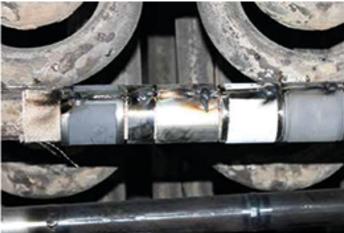
KME PROJECT STATUS REPORTS, 1/2017







CONSORTIUM MATERIALS TECHNOLOGY for thermal energy processes





Reporting period: July 2016 - December 2016

Status report KME

Project title: Influence of high-temperature environments on the mechanical

behaviours of high-temperature austenitic stainless steels

Project no: KME-701 (EM 39297-1)

Project manager: Mattias Calmunger

PhD:s/lic:s: (working in the project and degree of activity)

Hugo Wärner (Lic., 100%) started as a PhD-student 2016-07-01.

Project goals:

The main purposes of this project are to evaluate the mechanical behaviours for structure safety and integrity analysis, namely:

- 1. To evaluate the creep and LCF interaction diagram and integrity analysis since the boiler materials can undertake both creep and low cycle fatigue during the service.
- 2. To evaluate the structure stability and the toughness after long term service at an elevated temperature for safety analysis.
- 3. 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.
- 4. To evaluate the stress relaxation cracking behaviour of the boiler material. It is critical problem for some boiler materials.

Progress in relation to project goals:

Project tasks 1.1 is in progress relating to the main purpose and the first goal by performing and later evaluating specified tests, where the testing is completed. In relation to task 2 the mechanical testing of CTOD and some analysis is ongoing. Task 3.2 is in progress in accordance to the project time

schedule and is related to the third goal. The stress relaxation cracking method development and testing related to task 4 is ongoing. Hugo Wärner was employed as a PhD-student 2016-07-01 which contributing to the overall goal of training technical doctors and licentiates within the program.

Summary over results and work so far (max 1/2 A4-page):

Long-term ageing in Alloy 617 can lead to the precipitation of γ' , Mo-rich and Cr-rich carbides as shown in Fig.1 (a). This causes a reduction of toughness of the material (Fig.1 (b)). However, the material still has a high toughness even after an ageing at 700 °C for 20 000 hours as shown in Fig.1 (b).

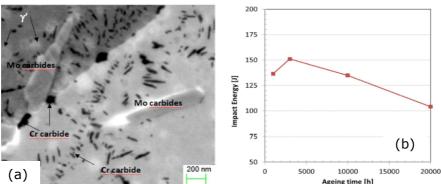


Figure 1: Microstructure and toughness performance of Alloy 617. (a) Precipitates in long term aged material at 700°C for 20 000 hours. (b) Impact toughness over ageing time at 700 °C.

Besides dislocation slip, twinning is another toughening mechanism for a long-term aged material during toughness testing as shown in Fig.2. These observations show that twinning is another toughening mechanism besides dislocation deformation. As mentioned previously, twinning that creates an extra ductility can compensate plasticity exhaustion after dislocation deformation or slipping. This may be an explanation why the long-term aged Alloy 617 material still can have high toughness although it contains very dense precipitates.

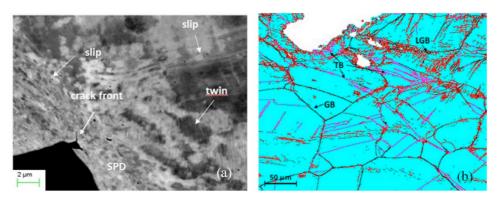


Figure 2: Deformation, damage and fracture behavior of specimen aged at 700°C for 3 000 hours during the CTOD test. (a) ECCI image, microstructure near the crack front. (b) EBSD image, grain structure near the crack front, black line: grain boundary (GB); red line, low angle grain boundary<10° (LGB), pink line: twin boundary (TB).

ELFORSK AB 3 (3)

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

The costs are according to the plan and no deviations to report. The schedule is mainly according to the plan, deviations on Task 1.1 otherwise no deviations to report. The deviations are related to broken test equipment's but this will not affect the project from deliver according to the project goals.

Number project reference group meetings held until report date:

A reference group meeting was held 20th of April 2016 at Swerea KIMAB with all project participants represented. Next reference group meeting is planned to the middle of the autumn 2017.

Planned activities:

Toughness tests after long-term ageing (task 2.1) and evaluation (task 2.2) and evaluation of creep/LCF interaction tests by establish the diagram of creep and LCF interaction (task 1.1).

Date 2017-03-30

Reporting period: July 2016 - Dec 2016

Status report KME

Project title: LCF and TMF crack growth in cast nickel-based superalloys

Project no: KME-702 (EM project no 39279-1)

Project manager: Johan Moverare

PhD:s/lic:s: (working in the project and degree of activity)

Christian Busse (80%), Frans Palmert (50%)

Project goals:

The aim of the project is to validate materials for future industrial gas turbines where there is a strong need for high fuel flexibility, availability and efficiency, as well as cyclic operation. This will require materials with high corrosion resistance and good resistance to TMF crack growth.

The key objectives of the project are:

- 1. Validate a TMF crack growth test method that can be used to generate high quality data for cast nickel-based superalloys, including single crystals.
- 2. Generate high quality test data for TMF crack growth in conventionally cast IN792 and the single crystal superalloy STAL15.
- 3. Improve the knowledge regarding the mechanisms that controls the crack growth rate for conventionally cast superalloys as well as for single crystal superalloys (e.g. influence of crystal orientation, phase shift and temperature-strain history).
- 4. Develop TMF crack growth models and life prediction methodologies that will reduce the need high safety margins. Today the safety of single crystal crack growth is in the order of three decades. The idea is to bring this order of magnitude down by one decade.
- 5. Validate the models for component near conditions.

Progress in relation to project goals:

• The isothermal testing to support goal (1) to (3) has been finalized. Test data is now available for 20°C, 500°C and 750°C.

- Stress controlled thermo-mechanical fatigue (TMF) crack propagation testing to support goal (1) to (3) are ongoing. IP and OP TMF between 100-750°C have been considered so far. Both STAL15 and IN792 are under investigation.
- Microstructure investigations to support goal (3) has been performed.
- Modelling related activities to support goal (4) and (5) is ongoing.

Summary over results and work so far (max 1/2 A4-page):

Since last status report isothermal crack propagation testing has been performed on DCT-specimens with an increased thickness 9.0mm instead of 4.5mm as used in the previous test. Aim is to investigate if the stress state plane-stress versus plane strain influence the transition from mode I to transcrystalline crack growth. The preliminary results indicate that there is no significant influence of sample thickness on the crack propagation rate as shown in Fig. 1.

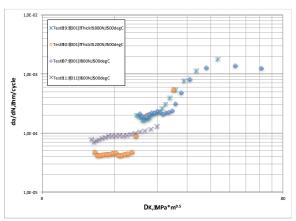


Fig 1: Crack propagation rates in STAL15 at

Regarding the modelling part of the project; A fracture mechanics tool is developed, which enables the possibility to calculates anisotropic Stress Intensity Factors resolved on a crystallographic slip plane in a three-dimensional context. This methodology makes it possible to analyze complex finite element-models, which are more application-near than traditional two-dimensional models. It is investigated which crystallographic slip plane experiences the highest stresses and is therefore most likely to show crystallographic cracking. When comparing to the experiments, the developed resolved shear stress intensity factor parameter is able to predict the correct slip plane on which crystallographic failures occurred for all evaluated tests on both DCT and KB specimens. The results have been summarized in a journal paper.

ELFORSK AB 3 (3)

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

Schedule and costs according to plans. No deviations

Number project reference group meetings held until report date:

Five project group meetings and two reference group meetings.

Planned activities: (3-4 lines)

- Continued TMF crack propagation testing.
- Long term ageing of specimen for TMF crack propagation has been started
- Investigate hold time influence and compare different specimen geometries.
- Continued investigations of the crack appearance and the deformation and damage behaviour
- Further develop the model for predicting crystallographic crack growth as well as transition between cracking modes (mode I and crystallographic)
- Modelling thermos-mechanical fatigue including viscoplastic effects

Reporting period: July 2016 - December 2016

Status report KME

Project title: Durable MCrAIX Coatings for demanding applications in gasturbines

Project no: KME-703 (EM project no 39296-1)

Project manager: Ru Peng

PhD:s/lic:s: (working in the project and degree of activity)

Till 2015-02 PhD Kang Yuan 100%

From 2015-05 PhD student Pimin Zhang 80%

Project goals:

The main aims of the project are:

- 1. To develop new durable MCrAIX coatings with improved performance for applications in medium size industrial gas turbines operating on a flexible base in terms of fuel and operation mode
- 2. To contribute to increased understanding of correlations between chemical composition, coating process, microstructure and performance of MCrAIX coatings
- To study the influence of applications of MCrAIX coatings on the behavior of superalloys.

Progress in relation to project goals:

Tasks 1-2 and 4-7 related to goals 1 and 2: Composition refinement by simulation for all proposed coating systems are complete. Characterization of TCF and oxidation resistance as well as post analysis is complete for two Nibase and one Co-base coating systems. Corrosion tests are continued. Selective coatings were tested in TBC for their performance as bond coat.

Tasks 3 directly related to goal 3 is ongoing with DBTT and TMF tests.

Summary over results and work so far (max 1/2 A4-page):

Chemical composition refinements/optimization by simulation were done on 4 groups of coatings and test samples for three groups, namely Ni-base with and without Fe-addition (for cost reduction) and Co-base, were made.

All the Ni-base coatings except one performed better than the reference coatings in TCF test and oxidation test up to 8000 hrs. For long term oxidation at $1000\,^{\circ}$ C for $11000\,$ hrs, one Fe-containing coating had the best performance and several others were also better than two of the three reference coatings.

TCF and isothermal oxidation tests showed a positive effect of Ru on resistance to coating degradation by interdiffusion but negative effect of Ce on surface oxidation for the Ni-base system. The addition of Fe to Ni-base coatings for coating-cost reduction did not impose a negative effect on TCF behaviour and oxidation resistance. Preliminary result of hot corrosion test with salt deposition did not show reduced corrosion resistance of the Fecontaining Ni-base coating.

The refined Co-base systems did not show improvement in oxidation behaviour, which is considered to be related to the addition of Hf to some coatings and a poor control of coating composition and thickness by the sample supplier.

DBTT and TMF tests on selected coating systems are underway. Preliminary results indicated low ductility with the Ni-base coating showing the best oxidation resistance. Analysis is underway to understand the behaviour.

Selected coatings used as bond coat in SPS TBC systems showed comparable or better TCF life than a widely used commercial reference bond coat.

Extensive simulations to understand the interdiffusion behaviour and coating life prediction have been performed. In particular, the condition for the formation of a beta-layer at the substrate-coating interface was analysed. In addition, the use of Al-activity instead of the beta-phase depletion as a life criterion of coating has been explored.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

Time schedule and costs follow in general the project plan and budget.

Number project reference group meetings held until report date:

1 reference group meeting.

Planned activities: (3-4 lines)

- 1. Continuing DBTT and TMF test of coated specimens
- 2. Continuing hot corrosion test of the Ni-base and Co-base specimens
- 3. Post analysis of the tested specimens from DBTT, TMF and corrosion tests to understand the observed behaviour
- 4. Planning for component test

Reporting period: 2016 09 30 - 2017 03 30

Status report KME

Project title:

Project title: MoSi₂ matrix composites for combustion components

exposed to high temperature oxidation and hot corrosion

Project no: KME-705

Project manager: Yiming Yao

PhD:s/lic:s: no

Project goals:

To produce high corrosion resistance MoSi₂ based composites for furnace and gas-turbine combustion chamber components aiming at operation temperatures ≥ 1200 °C.

Progress in relation to project goals:

- As sintered (AS) MoSi2+SiC, AS (Mo,Al)Si₂+SiC, and final sintered (FS) MoSi₂+ZrO₂ composites were isothermally oxidized at 1400C for 1000 h
- Hardness (HV10) and fracture toughness (Kc) of above materials were measured before and after isothermal oxidation.
- Microstructure of the isothermally oxidized samples was characterized.

Summary over results and work so far (max 1/2 A4-page):

- Thin uniform protective SiO_2 scales were formed on AS $MoSi_2+SiC$ materials at 1400°C. The oxidation behaviour of $MoSi_2+SiC$ materials is similar to that of KS1700 and FS and base $MoSi_2-ZrO_2$ materials.
- MoSi₂+SiC composites (MoSi₂+10 vol.% SiC) have high creep resistance comparable to Kanthal HT material in sagging test at 1700°C in air, superior to MoSi2 + ZrO₂ composite.

• Under to the protection of the SiO_2 layer, further sintering and densification in $MoSi_2$ + SiC composites occurred during long time exposure at 1400°C, especially for lower SiC additive contents (<10 vol.% SiC), which resulted in HV and Kc increasing.

- SiC additives decomposed at 1700°C. Glass bubbles and pores on the surface were formed, due to the released gases from SiC decomposition. The MoSi₂+SiC material is suitable for oxidation corrosion components under 1600°C.
- PLSed Mo(Si,Al)₂ materials were brittle in HT oxidation test due to the formation of Al-oxides in the base material and thick (Al,Si)- and Al-oxide scales on the surface. It is indicated that Mo(Si,Al)₂-based composites are not suitable for applications in HT oxidation environments. Mo(Si,Al)₂-based composites are more promising for applications in reducing atmospheres, under high gas velocities (Al₂O₃ not volatile such as SiO₂), and potentially in combustion gases.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

Yes.

Number project reference group meetings held until report date:

One meeting has been booked on 09/05 2017.

Planned activities: (3-4 lines)

- Prepare a new batch of MoSi₂-SiC materials.
- High temperature mechanical test at 1200°C for AS MoSi₂-SiC composites.
- Pitting corrosion tests at medium temperature at 500 700°C for AS MoSi₂-SiC and FS MoSi-ZrO₂ composites.
- Conference papers.

Reporting period: July-December 2016

Status report KME

Project title: Weldability of nickel-base superalloys for energy applications

Project no: KME-706 (EM project no 39283-1)

Project manager: Lars Nyborg (+46 31 772 12 57, lars.nyborg@chalmers.se)

PhD:s/lic:s: A PhD student (Fabian Hanning) is working in the project.

Project goals:

The continuation project aims to investigate and clarify the fundamental cause of formation of weld cracking, or more specifically strain age cracking. As basis for this overall aim, the goal is also to establish a test procedure for the assessment of the susceptibility towards strain age cracking of precipitation hardening Ni-based alloys.

Progress in relation to project goals:

The project proceed according to project the goals.

Summary over results and work so far (max 1/2 A4-page):

Background knowledge have been established during 2016 more focus will now be laid on experimental research. The repair welding study that has been analysed during 2016 has been submitted to Welding in the World. A large scale experimental study on the hot deformation behaviour of Haynes 282 is planned. This involves simulative testing by using the newly installed Gleeble machine at University West in Trollhättan. The aim of the study is to provide information on strain age cracking susceptibility as a function of material condition, *i.e.* the amount of hardening precipitates. Preliminary results of this study will be presented at the IIW annual assembly in subcommittee IX-H in June 2017. An in-depth microstructural characterisation is planned subsequent to Gleeble testing to relate mechanical performance to the materials microstructure, which is planned to be finished until Q3 2017. Following these investigations, further testing is planned on different

materials such as Waspaloy and 718Plus to enable the comparison of different alloys.

In parallel, an extensive material analysis with focus on repair welding of cast 718Plus is carried out. This study provides insight into the material behaviour under conditions close to reality and is aimed to be used as reference for further simulative testing on this new type of alloy. Results of this investigation will be submitted for presentation at the 2018 Superalloy 718 and derivatives conference in Pittsburgh, USA.

It is further planned to have the licentiate seminar in December 2017, which should then include results from the literature review presented at the SPS conference in Lund 2016, the repair welding study on Haynes 282 and the Gleeble study mentioned above. Until the end of 2017 it is hence anticipated to have a total of 3 articles published of which 2 are journal papers. Another conference paper will be submitted for presentation in 2018.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

The budget is according to plan both at the university and at the industry.

Number project reference group meetings held until report date:

Ten meetings have been held at GKN so far.

Planned activities: (3-4 lines)

- 1. Gleeble testing of Haynes 282
- 2. Microstructural characterization of Haynes 282
- 3. Analyses of repair welded cast ATI 718Plus
- 4. Licentiate defence

Reporting period:

Status report KME

Project title: Increased steam temperature in grate fired boilers - Steamboost

Project no: KME-709

Project manager: Torbjörn Jonsson, Chalmers

PhD:s/lic:s: T. Jonsson (25%), J. Eklund (30%), L. Paz, J (40%). Liske (5%), K. Hellström (5%) (until 2016-02-01), N. Israelsson (10%) (until 2015-07-01)

Project goals:

- Higher steam parameters & high electrical efficiency
- Development of novel solutions where steam is superheated in the furnace
- Develop improved material solutions including alumina formers

Progress in relation to project goals:

The first set of Steamboost material installed in the boiler has been removed and analysed. A second set is being exposed. A part of the un-cooled FeCrAl tube has been cut and analysed. Probe exposures (initial corrosion/deposit test) has been performed for a large number of boiler settings. All the deposits have been analysed and a preferred setting have been selected. Short/long time probe corrosion tests have been performed and some of the samples analysed. All of the exposed materials have now been analysed regarding material loss and selected materials more in detail with IC and SEM/EDX.

The laboratory studies are running as planned. We have received a large number of model alloys that are being exposed in parallel to commercial alloys. One of the model alloys have in addition been exposed during the probe test in the boiler. A new method of depositing KCl have been developed together with the HTC project 1a. The initial investigation of the model alloys shows that an increased Cr content and Si content may very be beneficial on the corrosion resistance of FeCrAl alloys in an KCl rich environment.

The half way report where the work has been summarized and a strategy for the second part of the project have been approved.

Summary over results and work so far (max 1/2 A4-page):

The aim with the first stage of the project was to generate knowledge about:

- CFD calculations deposit formation.
- The corrosion behaviour of the different materials (stainless steels and FeCrAl alloys) at the different temperatures focus on corrosion mechanisms.
- The corrosion properties of FeCrAl material by a tube consisting of FeCrAl inside the furnace near the Steamboost.
- The need for a ceramic shield.

The first part of the project has been successful and a large number of samples have been generated both through the extensive field exposures and laboratory work. The main results acquired so far have been compiled in order to determine the direction for the second stage of the project.

The laboratory studies have been focused on pre-oxidization and we have now also started to investigate different amount of alkali salt at 600 °C. Two presentations/papers have been submitted/shown on "Impacts of Fuel Quality on Power Production" conference, held 19-23 September 2016 in Prague.

Two parameters (Cr and Si) has been studied on FeCrAl model alloys in the lab. Both Cr and Si has a beneficial effect on the corrosion resistance of FeCrAl alloys. The Si effect on FeCrAl alloys will be presented in a poster at the conference Microscopy of Oxidation in England 3-5/4.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations: Yes, however some companies are slightly behind with in-kind reporting.

Number project reference group meetings held until report date:

We held reference group meetings (2015-22/10 and 2016-12-06).

Planned activities: (3-4 lines)

ELFORSK AB 3 (3)

The field exposures performed within the commercial waste-to-energy boiler AffaldPlus will focus on long time exposures of a new set of fixed installed material without a ceramic shield. The FeCrAl tube will be cut and analysed in detail. More analysis is in addition needed on the large set of samples generated in the first stage of the project. The work will focus on linking the different exposures results.

The laboratory work will focus on model alloys provided by Sandvik Heating Technology in KCl rich environments where more detailed analyses is required. We will in addition perform complementary probe exposures of new model alloys in the waste-to-energy boiler AffaldPlus. This exposure will link the lab results (beneficial effect of Si and Cr on FeCrAl alloys in KCl rich environment) to the field exposures and the complex environment.

Reporting period: 2016-10-01-2017-03-31

Status report KME-710

Project title: Design of a new generation of 12 % chromium steel

Project no: KME-710 (EM project no 39286-1)

Project manager: Associate Prof. Fang Liu (Deputy Manager 2014-11-01-

2015-05-31 was Prof. Hans-Olof Andrén)

PhD:s/lic:s: (PhD student Masoud Rashidi, 90%. Licentiate seminar 30th

September, 2015)

Project goals:

- 1. Based on the results obtained from the previous KME project and the parallel EU project, design three new test steels with fine-tuned chemical composition, aiming for improved creep resistance compared to the ones that have been designed and investigated in the previous KME 510 project;
- 2. Optimize heat treatment conditions for test steels;
- 3. Perform mechanical and creep testing on promising test steels with optimized heat treatment;
- 4. Understand the sophisticated effects of carbon addition on the precipitation reaction sequences in the Z-phase strengthened steels;
- 5. Understand effects of small addition of B on the Z-phase strengthened steels;
- 6. Understand special effects of Ta in combination with Nb;
- 7. Understand evolution of microstructure and its influence on the creep mechanisms.

Progress in relation to project goals:

Goals 1 and 2 are fulfilled with the production and heat treatment of the series ZU 1-4. Regarding goal 3, creep testing on ZU1, ZU2, and ZU3 has all been started. ZU4 has also been started after last report. Regarding goal 4, 5,

6, and 7, the work is ongoing. The main focus for the moment is Goal 6 and 7 – to study the coarsening rate of Ta-containing and Nb-containing Z-phase precipitates.

Summary over results and work so far (max 1/2 A4-page):

Currently, we focus on using TEM to perform quantitative study on the coarsening rate of Z-phase. (The chemical composition of these trial steels is provided in Table 1 in Appendix.)

- 1. For goal 5: We have obtained the volume fraction of BN in Z1. We found that quite some B has been consumed in these primary inclusions.
- 2. For goal 6 and 7: We found that: 1) Nb-containing Z-phase precipitates coarsen much faster than Ta-containing Z-phase. 2) We have finished most of measurement on the Z-phase size using TEM.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

We follow cost and time schedule plans.

Number project reference group meetings held until report date:

Seven project meetings:

- 1. At Chalmers, August 28, 2014; present Lennart Johanson, Siemens; John Hald, DTU; and Masoud Rashidi, Fang Liu and Hans-Olof Andrén, Chalmers.
- 2. At Siemens, Finspång, December 4, 2014; present Lennart Johanson, Torsten Kern and Arne Karlsson, Siemens; John Hald, DTU; and Masoud Rashidi and Hans-Olof Andrén, Chalmers.
- 3. At DTU, April 9, 2015; present Lennart Johanson and Torsten Kern, Siemens; John Hald and Frank Niessen, DTU; and Masoud Rashidi and Hans-Olof Andrén, Chalmers.
- 4. At Chalmers, September 3, 2015; present Lennart Johanson and Torsten Kern Siemens; John Hald, Irina Fedorova and Chitta Ranjan Das, DTU; and Masoud Rashidi, Fang Liu and Hans-Olof Andrén, Chalmers.
- 5. At Siemens, Finspång, December 17, 2015; present Lennart Johanson, Torsten Kern, Siemens; John Hald, Irina Fedorova, and Chitta Ranjan Das, DTU; and Masoud Rashidi, Hans-Olof Andrén, and Fang Liu Chalmers.
- At DTU, April 14, 2016; present Lennart Johanson and Torsten Kern, Siemens; John Hald, Kristian Vinter Dahl, Irina Fedorova, Chitta Ranjan Das, and Frank Niessen, DTU; and Masoud Rashidi, Robert Lawitzki, and Hans-Olof Andrén, Chalmers.
- 7. At Chalmers, September 13, 2016; present Lennart Johanson and

ELFORSK AB 3 (5)

- Torsten Kern Siemens; John Hald, Irina Fedorova and Chitta Ranjan Das, DTU; and Masoud Rashidi, Fang Liu and Hans-Olof Andrén, Chalmers.
- 8. At Finspång, December 17, 2016; present Lennart Johanson and Torsten Kern Siemens; John Hald, Irina Fedorova and Chitta Ranjan Das, DTU; and Masoud Rashidi, Fang Liu and Hans-Olof Andrén, Chalmers.
- 9. At Chalmers, March 20, 2017; present Lennart Johanson and Torsten Kern Siemens; John Hald, DTU; and Masoud Rashidi, Fang Liu and Hans-Olof Andrén, Chalmers.

Planned activities: (3-4 lines)

We will continue to analyse data of the evolution of the size of Z-phase precipitate. The exact coarsening rate will be available in next report. In addition, we will study the evolution of the precipitates, particularly the pathway of Z-phase transformation using combined XRD and TEM.

Appendix

Table 1. Chemical composition of ZU1-3 trial steels in weight and atomic % (Fe in balance).

	Ni	Co	Cr	W	Ta	Nb	C	В	N	Si	Mn	Cu
ZU1(wt%)	0.18	2.7	11.1	2.1	0.39	-	0.037	0.0048	0.053	0.35	0.13	2.0
ZU1(at%)	0.17	2.57	12.0	0.64	0.12	-	0.17	0.025	0.21	0.70	0.13	1.77
ZU2(wt%)	0.17	3.1	11.1	2.1	0.22	0.15	0.04	0.0054	0.053	0.33	0.14	2.0
ZU2(at%)	0.16	2.96	11.99	0.64	0.07	0.09	0.19	0.028	0.21	0.66	0.14	1.77
ZU3(wt%)	0.20	3.1	11.2	2.47	0.38	-	0.06	0.0045	0.056	0.22	0.15	-
ZU3(at%)	0.19	2.96	12.14	0.76	0.12	-	0.28	0.023	0.23	0.44	0.15	-

Reporting period:

Status report KME

Project title: Combating superheater corrosion by new materials and testing procedures - Corrosion experiments in the waste fired CFB boiler P15 at Händelö.

Project no: KME-711

Project manager: Jesper Liske, HTC/Chalmers

PhD:s/lic:s: Dr. T. Jonsson (5%), Dr. J. Liske (20%), Dr. L. Paz (40%), A. Olivas (40%)

Project goals:

The overall goal of the project is to improve plant economy by enabling an increased green electricity production and optimum material selection. The material matrix includes commercial steels available today as well as future materials developed for this type of environment. This will be achieved by generating new knowledge about the following topics:

- To correlate the corrosivity of the flue gas with the flue gas temperature in respect to the material temperature.
- To verify and quantify the corrosion rates for different superheater materials in superheaters with a horizontal design.
- Verify and compare the corrosion properties of commercial superheater materials as well as state-of-the-art stainless steels and FeCrAl alloys.
- Verify and compare the corrosion properties of coatings performed with the new generation coating technology HVAF (High Velocity Air Fuel).

ENERGIFORSK AB 2 (3)

• Compare different corrosion testing methods (i.e. probes, coils/tubes and clamping) with respect towards their complexity, cost and plant availability risk.

Project goals in relation to KME goals

This project proposal contributes to the following KME goals:

- Verifying novel solutions in boiler design with respect towards corrosivity.
- Increased steam parameters and thereby higher electrical efficiency.
- Test improved material solutions including alumina forming alloys and coatings.

Progress in relation to project goals:

• Verifying novel solutions in boiler design with respect towards corrosivity.

This goal is primarily directed towards investigation of the corrosion attack of a horizontal SH bank. We have in the project installed several clamp samples and tube samples in this section whereas some has already been removed from the boiler. The clamp samples included also FeCrAl model alloys. The exposed clamp samples have been evaluated by Chalmers and AmecFosterwheeler. The results show that the corrosion rate is rather low, which was expected since the material temperature has been rather low (360 °C and 380 °C). This was also true for low alloyed steels, i.e. the corrosion rate was low regardless of material.

- Increased steam parameters and thereby higher electrical efficiency.
- Test improved material solutions including alumina forming alloys and coatings.

The clamp samples has been exposed at different material temperatures, however, the difference in temperature between different samples was lower than expected, only 20 °C. Thus, the comparison between different temperatures for long term testing may be slightly vague. Clamp samples of model FeCrAl alloys has been analysed after long term exposure. The selection of model alloys has been done in close collaboration with Sandvik Heating technology as well as KME709 and HTC 1a "Critical corrosion phenomena". In addition, both HVOF and HVAF coated samples has been exposed as clamp materials.

The short term testing has been performed successfully and according to plan. The evaluation of the exposed samples are ongoing as well as compiling and evaluation operational data from the exposures. In addition to the "ordinary" probe corrosion test also an in-situ corrosion probe has been exposed. Furthermore, thermodynamical modelling of the exposures and IACM measurements are currently being performed and/or evaluated. The aim of these three different approaches (i.e. TD modelling, IACM and in-situ

ENERGIFORSK AB 3 (3)

corrosion probe) is to evaluate their accuracy in predicting corrosion. In addition to these tests, also new alloys were tested during these short term exposures. This included both Sanicro33 and a FeCrAl model alloy. These alloys will be compared to the low alloyed steel T22 and the stainless steel 347H. The later samples were doublet samples of which one of the samples had undergone a pre-oxidation prior to the exposure in the boiler. The aim with the pre-oxidation was not to obtain a protective oxide. Rather, the pre-oxidation aimed to destroy the initial, normally protective oxide. By doing so in a well-controlled way the aim is to improve the knowledge of how chlorine is able to penetrate the oxide scales. This has been performed in collaboration with the HTC project "Critical corrosion"

Summary over results and work so far (max 1/2 A4-page):

The analysis of the first set and second set of clamp samples has been finalized and the conclusion is that the corrosion rate is very low during this exposure. The reason for the low corrosion rate is explained by the low temperature of the superheaters in the boiler (and thus, low temperature of the clamp samples).

The effect of start-up sequence of probe exposures were tested, analysed and reported during 2015. The results were presented by Chalmers on the 22nd FBC conference held in Tampere Finland in June 2015. The results indicated that the start-up of probe exposures can be performed as they are done today, i.e. inserted directly into a hot boiler.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

Yes, on the project as a whole. However, some companies is behind in reporting their in-kind contribution whereas some companies are showing more in-kind contribution that compared to budget. On the project as a whole, the in-kind contribution is larger than budget. Chalmers costs are according to budget. Chalmers estimates that the overall budget will be fulfilled to the end of the project.

The time schedule is according to plan.

Number project reference group meetings held until report date:

Two reference group meetings has been held within this project, 22/10 2015 and 6/12 2016, both at Chalmers. This activity is synced with the KME projects KME709, 711 and 714, which has been very appreciated by the reference group members.

Planned activities: (3-4 lines)

We plan to withdraw the third set of clamp samples from the boiler during the spring 2017.

The short term tests (corrosion probes, in-situ alkali measurement, in-situ corrosion probe, deposit probe and TD modelling) is currently being analysed and will be finalized and evaluated.

Reporting period:

Status report KME

Project title: Sulfur recirculation and improved material selection for high temperature corrosion abatement - Investigating different aspects of corrosion memory

Project no: KME-714

Project manager: Torbjörn Jonsson, Chalmers

PhD:s/lic:s: T. Jonsson (20%), J. Phother (30%), L. Paz, J (30%), J. Liske (10%),

Project goals:

- Increase green electricity production from combustion of biomass and waste.
- Improved fuel flexibility.
- Improved material design/selection

Progress in relation to project goals:

Måbjergverket has changed owners during the project period. This resulted in an updated time plan that has been approved by SEA. The work follows the updated project plan and the following tasks has been performed during the period:

- Sulfur recirculation installed
- Fixed installed material in the boiler
- Corrosion probe exposures performed
- Lab investigation of microstructure performed at DTU

Investigation of field samples at DTU linking microstructure to corrosion attack

Summary of results and work so far (max 1/2 A4-page):

Summary of the activates at Måbjergverket:

The sulfur recirculation installation at MEC line 1 in Holstebro has been successfully in operation since October 2016. Line 2 has been in operation with the same fuel as line 1, according to the plan.

- 1) Corrosion memory tests with corrosion probe exposures were performed according to the plan, with 2 x 24 h and 2 x 1000 exposures, each involving 4 corrosion probes. 2 probes were stationary and 2 probes were switched after half the exposure resulting in ref-ref, rec-rec, ref-rec and rec-ref exposures, where ref = reference = line 2 and rec = Sulfur Recirculation = line 1
- 2) Superheater test materials installed in 4 superheater positions per line in October 2016 in order to enable comparison of the corrosion rates. Half of these samples will be cut out from the superheaters and replaced with conventional superheater tubes in April 2017 (line 1) and May 2017 (Line 2). The sample thickness will be measured on site and the materials will be sent to Chalmers for further investigations.

Activates at DTU: Corrosion tests on heat treated steel with Cr rich precipitates (700°C for 3000h) vs untreated samples has been undertaken. The material tested consists of 23.8 Cr, 22.0 Ni, 2.77 Cu, 2.46W, rest Fe.

The specimens have been exposed for 600° C and 168 hours in an atmosphere of 15 % H20, 5 % O2 and balance N2, with a coating of KCl or partially submerged in KCl deposit.

Results confirm the initial theory that there is more attack of the aged material, and microscopy reveals preferential attack at or within the precipitates.

The laboratory work is supported by thermodynamic assessments using ThermoCalc revealing the stability of phases.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations: The project follows the updated project plan/budget. However, some companies are slightly behind with in-kind reporting.

Number project reference group meetings held until report date:

A reference group meeting was held (2015-22/10 2016-12-06).

Planned activities: (3-4 lines)

Short/long time probe corrosion tests have been performed and the samples will be analysed (mass loss and SEM/EDX investigations on selected samples). Fixed installed material will be removed and analyzed.

To understand corrosion attack of carbide precipitates observed in the laboratory, corrosion testing of chromium carbide coated steel will be conducted and more advanced analysis of the exposed samples conducted.

Reporting period: 1st July 2016 - 31st Dec 2016

Status report KME

Project title: Intermediate temperature corrosion in used-wood fired boilers – the influence of lead, zinc and their chlorides

Project no: KME-717 (EM project no 40892-1)

Project manager: Rikard Norling

PhD:s/lic:s: Annika Talus (20% activity)

Project goals:

A specific goal of the project is to find out if lead, zinc and their chlorides causes serious corrosion problems in the temperature range 150- 420°C in boilers firing used wood, and if the attack is worsened by the use of additive that reduce alkali chloride corrosion on superheaters at higher temperatures. Based on the knowledge acquired by full-scale probe testing and the results of modelling and laboratory testing solutions for minimizing potential problems will be suggested.

Progress in relation to project goals:

The progress follows the project plan.

Summary over results and work so far (max 1/2 A4-page):

The work follows the project plan.

Short term and long term series of full-scale probe testing have been performed. Analysis of the samples is ongoing. The amount and composition of the deposits on the samples are shown to vary with and without the use of ammonium sulphate additive. However, when studying the key elements with respect to corrosion (Pb, Zn, K, Na, Cl, S) the difference appears limited.

Modelling of the test conditions has been performed. The modelling indicates that the amount of Pb and Zn in the gas phase is similar with and without the use of ammonium sulphate additive.

Laboratory testing at Åbo Academy started during autumn 2016 and first rounds of specimens have been analysed. The tests has shown that PbCl2+KCl salt mixture is much more corrosive than PbCl2 salt alone for the tested conditions. Addition of SO2 to the gas phase resulted in a decrease in the corrosion rate, which appears to be connected to sulphatation of the salt mixture.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

No deviations.

Number project reference group meetings held until report date: $\boldsymbol{1}$

A combined project and reference group meeting was held 2nd June 2016.

Planned activities: (3-4 lines)

Continuation of laboratory testing at Åbo Academy.

Continuation of analysis of the field test samples.

Reporting period: 1st July 2016 - 31st Dec 2016

Status report KME

Project title: High temperature corrosion in used-wood fired boilers – fuel additives and coatings

Project no: KME-718 (EM project no 41515-1)

Project manager: Rikard Norling

PhD:s/lic:s: Annika Talus (20% activity)

Project goals:

The overall aim of the project is to reduce operation and maintenance costs in heat and power boilers that burn predominantly used (recycled) wood by the use of additives to the fuel and the use of new coating materials. The effort will be directed towards furnace walls. In general this will result in increased fuel flexibility and make biomass a more attractive and financially competitive energy source.

Specifically the project aims at acquiring new knowledge from longer term studies on the effect of using sewage sludge as fuel additive on furnace wall corrosion. It further aims at finding a sludge that can be used as an alternative to sewage sludge and investigate the effects of it and evaluate whether it is suitable for longer term use.

The project aims at acquiring in-depth knowledge on the corrosion behaviour of materials typically used for furnace walls both when fuel additives are used and when firing the reference fuel. It also aims at investigating the corrosion properties of some new coating materials and their related performance with respect to furnace wall protection.

The academic goal of this project includes one licentiate (Annika Talus), as well as giving additional contribution to her PhD thesis (estimated 2018). The results will be published in one to two scientific papers.

Progress in relation to project goals:

The progress follows the project plan.

Summary over results and work so far (max 1/2 A4-page):

A 2-weeks exposure with sewage sludge has been performed. Two corrosion probes at furnace wall position and one super heater deposit probe have been exposed. The super heater deposit probe has been evaluated with respect to deposit growth and deposit composition. The chlorine content in the furnace probe deposits has also been evaluated. The addition of sewage sludge looks promising with lower deposit growth rate and low chlorine concentration in the deposit.

Field exposures of coated materials have been made as corrosion probe tests at the furnace wall. Specimen analysis is ongoing.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

No deviations.

Number project reference group meetings held until report date: ${f 1}$

A combined project and reference group meeting was held 7th June 2016.

Planned activities: (3-4 lines)

Analysis of samples from test campaigns with two different sludges.

Analysis of coated samples from probe testing.

A coated wall tube has been installed in the Blackburn Meadows Plant in UK as part of KME-708. It will be evaluated after a long-term exposure (>1 year) within KME-718.

Date 2017-04-18

Reporting period: July-December 2016

Status report KME

Project title: Development of weldability assessment and understanding of hot cracking in boiler and gas turbine materials

Project no: KME-719 (EM project no 40893-1)

Project manager: Lars Nyborg (+46 31 772 12 57, lars.nyborg@chalmers.se)

PhD:s/lic:s: A PhD student (Sukhdeep Singh) has been recruited and is now fully engaged in the project.

Project goals:

The overall goal of the project is to increase the overall understanding of hot cracking mechanisms as well as to develop weldability testing capability towards hot cracking in a reliable way. At the end of project, we expect to have a testing methodology both from theoretical and practical point of view that can be used as generic means of assessing weldability of high temperature materials.

Academic goals

The academic goals are the following:

- 1. To accomplish a licentiate engineer
- 2. To develop a unified theory for hot cracking
- 3. To develop a testing rationale for hot cracking

Industrial goals

The industrial goals are the following:

- 1. To generate weldability test data on materials relevant to the boiler industry
- 2. To recommend materials and parameters for improved weldability
- 3. To establish a testing methodology that can be used for assessing weldability with respect to hot cracking susceptibility

Progress in relation to project goals:

The project proceed according to outlined project goals.

Summary over results and work so far (max 1/2 A4-page):

The review paper titled "Review of hot cracking phenomena in austenitic stainless steels has been published and presented at the Swedish Production Symposium 2016 in Lund. The work related to the hot cracking in cast alloy 718 has been concluded, the study has been submitted in form of journal report to Science and Welding and Joining Technology (SWIT)

The new work is focused on hot cracking studies in ATI[®] 718 PlusTM. Preliminary results have been presented at the Intermediate Meeting for IIW (International Institute of Welding) in Trollhättan. The final work is planned to be presented at the annual meeting in Shanghai in June 2017. The work focuses on crack measurement analysis and more in depth microstructural characterization of the Varestraint tested samples.

Related to the austenitic stainless steels, the testing plates have been received from the project partner Amec Foster Wheeler (new name Sumitomo). Varestraint testing of the received material is planned in the autumn.

Concerning to the Graduate studies, the Licentiate defence is planned by the end of year 2017

The Varestraint weldability testing machine have been moved to a laser secured room enabling both arc and laser welding options.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

The budget is according to plan both at the university and at the industry.

Number project reference group meetings held until report date:

Several project meetings have been held with GKN as well as with Sumitomo (Amec Foster Wheeler). Dong Energy have also participated at one project meeting.

Planned activities: (3-4 lines)

- 1. Finish the analyses of the Varestraint tested 718 Plus-samples
- 2. Publish the 718Plus work as a journal article
- 3. Varestraint Testing of the Austenitic Stainless Steels
- 4. work on a new testing rationale for Gleeble testing and Varestraint weldability testing
 - a. make an upgrade of the Varestraint testing machine
- 5. Licentiate defence by Dec. 2017

Reporting period:

Status report KME

Project title: The effect of increased fractions of waste wood on water walland superheater corrosion - Combating corrosion by new materials and improved material selection

Project no: KME-720

Project manager: Jesper Liske, HTC/Chalmers

PhD:s/lic:s: Dr. T. Jonsson (5%), Dr. J. Liske (20%), Dr. L. Paz (30%), J. Eklund (5%)

Project goals:

The overall goal of the project is to improve plant economy by enabling an increased green electricity production and optimum material selection. The material matrix includes commercial steels available today as well as future materials developed for this type of environment. This will be achieved by generating new knowledge about the following topics:

- To verify and quantify the corrosion rates for different superheater materials in superheaters with a horizontal design.
- Verify and compare the corrosion properties of a biomass fired boiler (Örtoftaverket) and waste fired boiler (Händelöverket in KME711/EM39299-1)
- Investigate how the corrosion performance of water walls is affected by a stepwise increase of the waste wood fraction in the fuel mix.

Project goals in relation to KME goals

The goals of this project is direct related to the following goals stated in the program description:

ENERGIFORSK AB 2 (3)

Goal 3:

This project will explicitly examine several materials and their corrosion resistance when the fuel mix changes. The results will contribute to improve the material selection of superheaters and water walls so that materials with extended lifetime can be selected alternatively an extended fuel flexibility can be achieved. The results may also be used by material manufacturers to produce steel with optimized microstructural characteristics which better prevents the corrosion caused by the corrosive environment.

Goal6:

This project will investigate how the corrosion rate of several different steels/alloys changes with different fuels. With this knowledge, fuel flexibility and/or availability may be increased. Through an improved understanding of how corrosion is influenced by environmental and material-specific parameters new boiler designs, operating parameters and tools can be proposed.

Progress in relation to project goals:

We have since the last status report analysed the first sets of clamp samples from the boiler. The analysis has primarily been material loss evaluation. The results show that the stainless steels/highly alloyed steels as well as coated samples show negligible corrosion. The corrosion of the low alloyed steels are slightly higher, where the most corroded sample showed a material loss corresponding to 0.3mm/year. Further analysis of the clamp samples are currently ongoing at Chalmers. However, no results from these analyses has been presented yet.

Summary over results and work so far (max 1/2 A4-page):

The work so far has primarily been constituted towards planning, installation and removal of clamp samples. The removed clamp samples are being analysed by means of corrosion attack. The second year clamp samples were installed in the boiler during 2016 and will be removed during the summer revision 2017. The clamp samples exposures and analysis is following time and cost plan.

In addition, planning/discussions of the upcoming short term corrosion probe testing in the superheater region and planning of the water wall corrosion probe testing has been performed. The plan is to proceed with waterwall probe exposures in October/November 2017 (preliminary start is in w43 or 44). The plan is that the exposures will last 168 hours (one week) and that the fuel mix is varied from $20\% \rightarrow 40\% \rightarrow 60\%$ fraction of waste wood. During each of these levels, two water wall probes (with the "Vattenfall" design, i.e. going in between two water wall tubes) will be exposed for 1 week, i.e. 3 exposures are planned.

Is time schedule and costs according to plan/budget? Comments over reasons if deviations:

As mentioned in earlier status reports, the original time plan has been slightly postponed since the manufacturing of a new measurement hole in the boiler was delayed. However, the project is now again on track and the

ENERGIFORSK AB 3 (3)

measurement hole is planned to be performed during the summer revision 2017. The planned exposures will be executed in October/November 2017.

This delay has resulted in lower costs at Chalmers compared to budget as well as lower in-kind contribution for some of the companies. Also, not all companies has reported their in-kind contribution to this date. The project manager is reminding these companies. The estimation is that the project will fulfil budget and in-kind contributions until the project ends.

Number project reference group meetings held until report date:

Two reference group meetings has been held within this project, 22/10 2015 and 6/12 2016, both at Chalmers. This activity is synced with the KME projects KME709, 711 and 714, which has been very appreciated by the reference group members.

Planned activities: (3-4 lines)

We are performing corrosion analysis of the removed clamp samples. The last set of clamp samples are to be removed from the boiler in the summer of 2017. Water wall probe tests and short term probe tests with varying fractions of waste wood are planned for in October/November 2017.