High Temperature Corrosion and Abrasive Wear of Boiler Steels

JELENA PRISS
Dissertation was accepted for the defence of the degree of Doctor of Philosophy in Engineering on May 9, 2014

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Defence of the thesis: June 20, 2014

Declaration:
Hereby I declare that this doctoral thesis, my original investigation and achievement, submitted for the doctoral degree at Tallinn University of Technology has not been submitted for doctoral or equivalent academic degree.

/Jelena Priss/

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ISSN 1406-4758
Katlateraste kõrgtemperatuurse korrosiooni- ja kulumisuuuringud

JELENA PRISS
**Abbreviations and Symbols**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC²T</td>
<td>Austrian Centre of Competence for Tribology</td>
</tr>
<tr>
<td>CFB</td>
<td>Circulating Fluidized Bed</td>
</tr>
<tr>
<td>DTE</td>
<td>Department of Thermal Engineering</td>
</tr>
<tr>
<td>EC</td>
<td>Erosion–Corrosion</td>
</tr>
<tr>
<td>EDX</td>
<td>Energy Dispersive X–ray</td>
</tr>
<tr>
<td>EO</td>
<td>Erosion–Oxidation</td>
</tr>
<tr>
<td>HHT</td>
<td>Hot Hardness Test</td>
</tr>
<tr>
<td>HT</td>
<td>High Temperature</td>
</tr>
<tr>
<td>HTC</td>
<td>High Temperature Corrosion</td>
</tr>
<tr>
<td>HT–CIA</td>
<td>High Temperature Cyclic Impact–Abrasion</td>
</tr>
<tr>
<td>HTE</td>
<td>High Temperature Erosion</td>
</tr>
<tr>
<td>LHV</td>
<td>Low Heating Value</td>
</tr>
<tr>
<td>LTC</td>
<td>Low Temperature Corrosion</td>
</tr>
<tr>
<td>NPP</td>
<td>Narva Power Plant</td>
</tr>
<tr>
<td>OM</td>
<td>Optical Microscopy</td>
</tr>
<tr>
<td>PF</td>
<td>Pulverized firing</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>TUT</td>
<td>Tallinn University of Technology</td>
</tr>
</tbody>
</table>

*Vol. %*  Volumetric percentage  
*Wt. %*  Weight percentage
Introduction

Wear in its various modes is the most important issue in many industrial applications. Correct steel selection could allow to increase the durability and operating reliability of components in boilers. Combustion of Estonian oil shale containing inorganic matter produces several chemically active compounds leading to both fouling and accelerated high temperature corrosion of heating surfaces. The research of high temperature corrosion of different new steels in order to reveal the most corrosive–resistant and most suitable one for operation in the conditions of high temperature corrosion under influence of oil shale ash deposits is always of high importance. The ability to predict accurately the remaining life of the heating surfaces tubes subjected to intensive corrosion would help to reduce expenses of tubes replacement. This study focuses on the relations between the properties of low and high alloyed steels and their corrosion and wear behaviour.

The Main Objectives of the Thesis:
• to study boiler steels durability in the conditions of corrosion, erosion and impact–abrasion at high temperatures;
• to rank tested steel grades according to their resistance to corrosion;
• to obtain data for analysis of the applicability of investigated steels for manufacturing of different components of boiler on the basis of carried out tests results.

Scientific Novelty
Scientific novelty of the doctoral thesis includes:
• New data of four boiler steel grades resistance to high temperature corrosion and abrasive wear have been obtained.
• New methodology of high temperature corrosion (HTC) testing has been proposed and realised in newly developed HTC tester.

List of Publications:


Other publications (not included in the thesis):


**Author’s Own Contribution**

This section describes the author’s contribution to the papers included in the thesis.

[PAPER-I] I am the main author of the paper, responsible for the performance of tests, analysis of results, drawing the conclusions, literature review and data collection and had a major role in writing the paper.

[PAPER-II] I am the main author of the paper, responsible for the performance of tests, analysis of results, drawing the conclusions, literature review and data collection and had a major role in writing the paper.

[PAPER-III] I took part in the analysis of results.

[PAPER-IV] I am the main author of the paper, responsible for the analysis of results, drawing the conclusions, literature review and data collection and had a major role in writing the paper.

[PAPER-V] I am the main author of the paper, responsible for the analysis of results, drawing the conclusions, literature review and data collection and had a major role in writing the paper.

**Approbation:**

Presentations at different international conferences:

- 22nd DAAAM World Symposium, 23–26th November 2011, Austria Center Vienna, Vienna, Austria;
- 23rd International DAAAM Symposium in 2012 in Zadar, Croatia;
Acknowledgements
Firstly I would like to express my gratitude to my supervisors Prof. Ivan Klevtsov and Ph.D Andrei Dedov from the Department of Thermal Engineering of TUT for their excellent guidance and support during this research. Special gratitude goes to Ph.D Maksim Antonov for support, discussions and advises. I appreciate Ph.D Renno Veinthal, Ph.D Kristjan Juhani, Ph.D Ahto Vallikivi from the Department of Material Sciences of TUT for support. My gratitude goes also to colleagues at Austrian Centre of Competence for Tribology Ph.D Ewald Badisch, Ph.D Horst Winkelmann, MSc Christoph Mozelt, Dipl.-Ing Christian Katsich, Markus Flasch, Harald Rojacz, Markus Varga, Sten Vinogradov, Werner Tschirk and to the technical personal who has in one way or another contributed to this work.
I would like to thank Maaris Nuutre from the Department of Thermal Engineering of TUT for support in chemistry laboratory and Ph.D Toomas Lausmaa for helping with thesis writing. I wish to thank Mykola Semeyuk for fabrication of specimens for experiments.
I would like to thank Ph.D Dmitri Vinnikov for giving me opportunity to be a member of “Doctoral School of Energy and Geotechnology II” and my doctoral school friends: Victoria Bashkite, Anton Rassõlkin, Ph.D Marina Kostina, Ph.D Lauris Bisenieks, Julia Gulevich and Andrei Tjurin.
I am thankful to my colleagues from AS Sweco Project: Priit Lepmets, Igor Habarov and Liis Jääger for forbearance during research processes. I would like to thank all organizations for financial support of this study:

- European Social Fund (Project „Doctoral School of Energy and Geotechnology II“) Archimedes Foundation.
- ESF DoRa T6 and DoRA T8 Funding, Archimedes Foundation.

Finally, I would like to thank my family for their support, Estonian and Austrian “climbing” friends for encouragements and optimism during my studies and thesis writing.

Jelena Priss
1 REVIEW OF THE LITERATURE

1.1 Fundamental mechanisms of wear

The wear is usually defined as the process of material removing from the surface due to interaction with tribological elements. The most typical mechanisms of wear are presented in Figure 1.1.

In present research abrasive wear of boiler steels were investigated.

Corrosion, corrosive wear and oxide formation at high temperatures

The corrosion of the metal is spontaneous process between the metal and its surrounding environment, during which damage and destroy of the material happens.

Material removing from the surface due to interaction with tribological elements in corrosive media, the resultant wear is called corrosive wear. The most dominant corrosive element in air is oxygen and wear in air is called oxidative wear [1].

Typical materials for power plant components are ferritic or austenitic heat resistance steels or super alloys that show oxidation resistance at high temperatures (500–800 °C) [1].
Alloyed steel changes the picture of the oxide formation, especially since chromium and nickel in the steel will be involved in the oxidation. Figure 1.2. presents a schematic view of the oxidation of a metal surface. The diffusion depends on the metal/alloy type, temperature and the atmospheric conditions [1].

The reaction between a metal and oxygen may be represented by the following equation [1]:

\[ aM + \left(\frac{b}{2}\right)O_2 = M_aO_b, \]

where \( M \) – oxidizable metal;
\( O \) – oxygen;
\( a, b \) – stoichiometric coefficients.

As the result of oxide formation, many possible ways of boiler metal wear exist. Firstly, it is a high- and low-temperature corrosion (HTC/LTC), when formed oxide scales influence the thermal transfer in boiler. Many parameters determine the course of high-temperature corrosion [2]. It is influenced mainly by the structure and stability of the oxide layer on the metal. External forces like cleaning cycles, thermal stresses, that factors result damage to the oxide layer on the tube metal.

**Erosive wear**

Erosive wear is loss of material from a solid surface due to relative motion. Material loss due to erosion is measured as the ratio of the mass of removed material with respect to the mass of erosive particles. Illustration of the erosive wear is shown in Figure 1.3. Mechanisms of erosion can involve both plastic deformation and brittle fracture [3].
H. Uuemõis and I. Kleis [4] has found a number of parameters influence erosion: impact velocity, impact angle, particle size, particle concentration, hardness ratio of the abrasive and the material subjected to wear, geometry of abrasive particles, strength of abrasive particles, test temperature, corrosiveness of environment, content of additives in the abrasive (fluid or solid). The most important parameter of erosion is impact angle. The maximum wear rate of ductile materials is at an impact angle between 30° and 60°, while the maximum wear rate of brittle materials occurs approximately at the impact angle of 90° [5].

**Abrasion**

Abrasion is the most common form of wear in industry. It is defined as: “the wearing away of any part of a material by frictional action of hard particles through the surface of a softer material” [6]. When one surface (usually harder than the second) cuts material away from the second, two body abrasion occurs. This mechanism very often changes to three body abrasion as the wear debris then acts as an abrasive between the two surfaces. Explanation of abrasion is presented in Figure 1.4 [3].
1.2 Modes of wear in boilers

Gas – side processes in boilers

According to A. Ots [2] the purpose of a boiler is to transfer the energy released during fuel combustion (expressed by flue gas temperature) to flue gas and, thereafter, to the medium to be heated. The latter is usually water, steam, or air. For gas–side processes, the main equipment in a boiler is a heat exchange surface. Gas-side processes in a boiler represent a set of interrelated and consecutive chemical and physical phenomena, which ultimately determine the rate of heat transfer from flue gas to the heated medium, the lifetime of the boiler tubes, and the design and arrangement of the heated exchange surfaces. The nature of gas–side processes is determined by fuel properties and combustion technology.

The typical conditions of fire side corrosion of tubes of power plant steam boiler heating surfaces are presented in Figure 1.5.

![Figure 1.5. Fireside corrosion and wear.](image)

Conditions in fluidized–bed boilers (e.g., elevated temperatures, oxidizing atmospheres and impacts by fluidized sand and ash particles) may cause significant wearing of some boiler components, such as heat exchanger tubes, by a combination of oxidative attack and erosive wear [2, 7, 8]. M. Antonov [9] describes that erosion–corrosion is a result of material wastage rates in power generation industry. Boiler components, such as superheater and reheater in fluidized bed boilers are suffering from fly-ash erosion. Major constitutes of the fly–ash are SiO₂, Al₂O₃, and Fe₂O₃. To reduce the wear rates there is imposed limits for the flue gas velocities (12–13 ms⁻¹ for erosive ash having high silica content and 18–20 ms⁻¹ for less–erosive ash), but also boiler efficiency should be considered.
Wear problems in circulating fluidized bed boilers

Oil shale is a sedimentary rock containing organic matter [2] and the main source in Estonia. Around 90% of the electricity in the country is produced from oil shale at the Narva Power Plants (NPP). The lower heating value (LHV) of oil shale used for electricity production is 8.3–8.4 MJ/kg [10]. Oil shale is characterized as a fuel with a high content of alkali metals and chlorine. Burning that kind of low-grade fuel is associated with several problems: dissociation of carbonate minerals, high CO₂ emission levels, ash handling and storing [2].

For minimizing the environmental impact of power production, the application of modern Circulating Fluidized Bed (CFB) technology at the NPP (Figure 1.6) provides advantages in utilization of upgraded oil shale with the LHV at up to 11.0 MJ/kg [10]. CFB of solid fuels, a technology that is still in the development stage, is the newest trend in thermal power plants [2]. In CFB combustion boiler, the inert bed material is in the fast fluidization mode. Particles move upward (ash particles by burning of oil shale), get separated there and return to the bottom of the furnace. A circulating contour is created [2].

Scheme of CFB boiler in NPP is presented in Figure 1.6 [11]. The potential problems of HTC, LTC and erosion in NPP CFB boiler parts are summarized at Table 1.1.

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Figure 1.6. Scheme of oil shale-fired CFB boiler [11]: 1 – Raw fuel silo, 2 – Fuel feeder, 3 – Secondary fuel crusher, 4 – Grate, 5 – Furnace chamber, 6 – Separating chamber, 7 – Fluidized bed internal heat exchange (Intrex), 8 – Separator of solids, 9 – Convective superheater and reheater, 10 – Economizer, 11 – Air preheater, 12 – Electrical precipitator.
Table 1.1. NPP CFB boiler components potential wear [2, 8, 12].

<table>
<thead>
<tr>
<th>Position</th>
<th>Potential wear</th>
<th>HTC</th>
<th>LTC</th>
<th>Erosion</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>Fuel supply system (plates)</td>
<td></td>
<td></td>
<td>X</td>
</tr>
<tr>
<td>4</td>
<td>Furnace chamber</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Separating chamber</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Fluidized bed internal heat exchanger</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Convective superheater and reheater</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Air preheater (APH)</td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Economizer</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>7</td>
<td>Intrex Superheater III</td>
<td>X</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Header; inlet</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Header; outlet</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Reheater</td>
<td>X</td>
<td></td>
<td>X</td>
</tr>
</tbody>
</table>

1.3 Corrosion of boiler tubes and waste incineration systems

Gas-side corrosion

According to A. Ots [2] “Gas-side” corrosion develops in an environment defined by the interaction of flue gas with the ash deposits found on the boiler tube surfaces. In boiler design, it is customary to divide the flue gas corrosion processes into those taking place at low and high temperatures, noting the differences in the course of these processes. Low-temperature corrosion is linked to the condensation of sulphuric acid and/or water vapour from the flue gas onto the heat transfer surface. High-temperature corrosion develops at higher temperatures than during the low-temperature processes, but there is no definite temperature limit. Formation of aggressive chlorides and sulphur dioxides in flue gases in industrial boilers is the result of incineration of biomass and waste [13]. Table 1.2 presented the number of surface phenomena in waste incineration systems. According to J. Adamiec [13] research corrosion processes in the combustion chamber of waste incineration systems have an oxidizing nature, connected with the presence of nitric and chromium acids, iron and copper salts, nitrites, chromates, sulphuric, hydrochloric and phosphoric acids, organic acids, solution of alkaline salts and halides. As the result, the presence of a mixture of the above mentioned compounds and increased temperature may lead, among others, to crevice or pitting corrosion.
Table 1.2. Typical surface phenomena in waste incineration systems [13].

<table>
<thead>
<tr>
<th>Corrosion processes connected with the presence of humidity</th>
<th>Corrosion processes as a result of increased temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Surface corrosion</td>
<td>- Sulfiding</td>
</tr>
<tr>
<td>- Crevice corrosion</td>
<td>- Carbonization</td>
</tr>
<tr>
<td>- Stationary pitting corrosion</td>
<td>- Formation of metal-dusted areas (dusting)</td>
</tr>
<tr>
<td>- Stress corrosion</td>
<td>- Nitriding</td>
</tr>
<tr>
<td>- Intercrystalline corrosion</td>
<td>- Sulfiding</td>
</tr>
</tbody>
</table>

The influence of oil shale ash on metal corrosion

According to A. Ots investigation [2] in oil shale boilers, in addition to the very heavy coverage of heat transfer surfaces by ash deposits, another important problem occurs: the high-temperature corrosion of the same boiler tubes. This corrosion is quite acute for the super heater metal. Corrosion-activating components form during the process of fuel (oil shale) properties but also to the combustion technology.

There have been studied [14] the impact of oil shale ash deposits on tubes made from the steels rich in nickel (Ni – 12.16 %, Cr – 16.03 %) and tubes covered with coatings (Ni – 73 %, Cr – 21 %, Ni/Cr – 1.5). The results clearly demonstrate the influence of the steel composition on the corrosion rate. A more detailed investigation of the corrosion rate factor of austenitic steels demonstrated that its value correlates well with the ratio Ni/Cr in metal. This can be seen on Figure 1.7, where the influence of the ratio Ni/Cr on the corrosion rate factor of austenitic steels is presented.

![Figure 1.7. Influence of the ratio Ni/Cr on the corrosion rate factor during the corrosion of austenitic steels under the impact of a stable oil shale ash deposits at 580 °C [14].](image-url)
The figure illustrates that the value of \( n \) decreases under the impact of a chlorine-containing deposit with a decrease in the chromium content or in the Ni/Cr ratio of the metal. Therefore, the superheater tubes of an oil shale boiler are more resistant to corrosion when made of a steel in which the ratio Ni/Cr exceeds 1 [14].

**High temperature corrosion in the presence of chlorine**

A. Ots [2] describes that an important factor in alloy corrosion can be the reaction between the oxides formed from the different alloy components. As a result of these reactions, a stable composite oxide, with completely new characteristics and with a diffusion-resistant intricate structure, may form on the alloy surface. Depending on the alloy composition and on the external parameters, a composite-oxide might include not all, but only a certain number, of the alloy components. The diffusion rate of the reacting components in this type of composite composition oxide layer is generally much slower than in a pure metal oxide. These types of oxides are found in corrosion-resistant alloys.

High-temperature corrosion in an atmosphere containing chlorine is complex. It leads to the formation of scale with an exemplary structure presented in Figure 1.8 [15]. This illustration was derived according to Y. Kawahara [15] analyses of corrosion products and thermodynamic consideration.

![Figure 1.8. Structure of scale formed in Ni-Cr-Mo-(Nb, Fe) alloy as a result of high-temperature chlorine corrosion [15].](image)

**1.4 Erosive – corrosive processes in boilers**

**Erosion-oxidation and erosion–corrosion mapping**

There are a number of factors which were mentioned before [4] that influence the erosion–oxidation (EO) of the materials, one of them is the oxidation kinetics, which is affected by the temperature and the alloy [5] and should be considered as well.

The erosion–oxidation process is divided into different regimes [5], ranging from pure erosion to pure oxidation.

Laboratory testing seemed to indicate erosion, erosion–corrosion and pure corrosion processes according to weight loss and temperature increase [5].
Figure 1.9 illustrated the increasing of wear rates with an increase in temperature to some peak and then decrease with a further increase in temperature.

![Figure 1.9. Diagram showing the erosion–corrosion response of metals to temperature changes [16].](image)

Erosion–oxidation mapping (EO maps) and more recently erosion–corrosion (EC maps) is a popular way [17] to represent the system behavior under different EO and EC conditions. This is the best option to select the material, to understand of materials response under wide ranges of conditions [18]. EO maps are useful to determine what will happen when conditions change. Figure 1.10 represent the example of EO map.

More recently, such mapping has been further developed into modelling of materials selection maps [19]. Figure 1.11 represent the example of such mapping.

![Figure 1.10. EO map evaluated by Stack’s simulation program [20].](image)
Erosion processes in boilers

The wear of boiler’s heat transfer surface consists of the thinning of the tube wall over time [2]. Hard particles, either directly from the fuel or from the ash that forms after combustion, can cause erosive damage to boiler components [21].

According to A. Ots [2] the increase in diffusion resistance of the oxide layer over time is a significant factor in the boiler tube corrosion process. Therefore, any damage to the oxide layer on the boiler tubes and other similar phenomena that reduce the diffusion resistance of the oxide layer will accelerate the rate of metal corrosion. For example, fly ash erosion refers to damage caused by ash particulate entrained in high-speed flue gases striking metal surfaces [21].

A. Ots [2] declared, that the term, erosive wear, is used to define the wear of the material caused by a force acting periodically on a metal surface. If the erosive impact of the force on the metal damages the protective oxide layer, the process is defined as corrosive-erosive wear, and the periodic crumbling of the oxide layer on the metal surface accelerates the corrosion process.

Erosive damage can be attributed to a number of different sources, such as particles in fly ash [21], temperature fluctuations, falling slag and soot blowing. The fact that erosion–oxidation problems still inconvenience power generation, for example, in fluidized–bed boilers, after a few decades of practical and theoretical experience, reflects that the phenomena are complicated [19].

Basic problems of abrasive impact wear

Wear caused by the abrasive impact wear action of hard particles [21] is dominant problem in many industrial processes. One of the wastage mechanisms is impingement of particles on the surface [22], which causes abrasive wear.

The abrasive wear theory assumes that abrasive particles leave a wear area of the same cross-sectional shape [6]. Specific mechanical and geometrical properties of abrasive particles which are getting in contact with the wearing surfaces could restrict the life time of costly machine parts [23].
Many researches are done to understand of the response of various materials exposed to this type of wear [24 – 25].

1.5 Research process

The investigation of problem existed in some boiler components could be prepared according to the scheme (Figure 1.12) proposed by M. Antonov [26]. Corrosion and wear investigation have two options for process determination. The first method is industrial testing and the second is laboratory testing. The laboratory test is performed according to standards, usually involves test specimens and could be executed in short time [26]. The test samples are smaller than the real component and of simple geometry that makes the test more convenient and enables to raise the level of control of conditions and of tracking the tribosystem behaviour [26].

Problem statement

Laboratory testing

Understanding of the mechanism

Development of theory and mathematical model

Real industrial tests

Theory verification

Parametric study

Improved boiler components

Figure 1.12. A schematic overview of the researching process to improve the performance of the boiler parts [26].

Tribotests

Tribology (from the Greek τριβος – rubbing or attrition) is defined as “the science and technology of interacting surfaces in relative motion” [3]. Tribotester is the machine or apparatus for simulation the wear in laboratory conditions.

Tribotests could be classified by rising of the degree of realism (how closely they imitate the conditions of real application) as laboratory test, simplified component, component, sub-system test [26]. The comparison between laboratory testing and industrial testing with more evaluation aspects is given in Table 1.3.
Table 1.3. Comparison between laboratory testing and industrial testing [26].

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Laboratory testing</th>
<th>Industrial testing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Degree of realism</td>
<td>Lower</td>
<td>Higher</td>
</tr>
<tr>
<td>Duration of the test</td>
<td>Several hours</td>
<td>Several months</td>
</tr>
<tr>
<td>Cost of the test</td>
<td>Lower</td>
<td>Higher</td>
</tr>
<tr>
<td>Level of the conditions control</td>
<td>Higher</td>
<td>Lower</td>
</tr>
<tr>
<td>Ability to study individual, isolated wear mechanisms</td>
<td>Higher</td>
<td>Lower</td>
</tr>
</tbody>
</table>

**Laboratory tests of Department of Thermal Engineering**

The corrosion tests of steels (X12CrNiTi18-9, X10CrMoVNB9-1) in laboratory of Department of Thermal Engineering (DTE) have been carried out in accordance with standard techniques [27, 28]. These tests have been performed in electrically heated vertical tube-type furnaces with an inner diameter of 40mm (Figure 1.13). The combustion products of natural gas have been directed into the furnaces by the pipe header. The combustion gas velocity in the furnaces has been 15–18 cm/s. In order to avoid condensation of water vapour the pipe header has been heated by electrical heating coils. The temperature in the furnaces has been maintained at 540 °C, 580 °C and 620 °C with accuracy ± 2 °C [29].

The corrosion tests have been performed on flat polished specimens with dimensions 3x10x40 mm, which have been cut from original boiler tubes. Prior to the tests all specimens were degreased, precisely measured and weighed. They were then coated with a mixture of oil shale ash and ethyl alcohol for imitation of oil shale on-tube deposits. Electrical precipitator ash, with chlorine content of about 0.5 %, was used in the laboratory experiments [29].
Figure 1.13. The scheme of laboratory tester for investigation of steels high temperature corrosion: 1 – electrical furnaces; 2 – tools for temperature measurement and control; 3 – test specimens; 4 – thermocouples; 5 – combustion chamber; 6 – fan [29].

The removal of corrosion and oxide scales from the post-test specimens was performed in the environment of liquid sodium by blowing ammonia [30]. The quantity of corroded material was determined as the difference of mass of clean specimens before and after testing. The accuracy of weighing was ± 0.1mg. On the basis of mass difference the corrosion depth was calculated (ΔS, mm):

\[
\ln \Delta S = \alpha - \beta T^{-1} + (\gamma + \varepsilon T) \ln \tau,
\]

where

- ΔS – the corrosion depth [mm];
- τ – the test duration, [h];
- T – metal temperature, [K];
- \( \alpha, \beta, \varepsilon \) and \( \gamma \) – coefficients which depend on the ash characteristics, the particular fuel, the steel grade and temperature of the metal.

The binomial term \( (\gamma + \varepsilon T) \) is usually defined as an exponent of the corrosion process [31–34].

**Experiments**

Typical materials for power plant components are ferritic, austenitic heat resistance steels or super alloys. For present research four high temperature resistance steels used at Narva Power Plants were investigated in the HTE, HTC and HT–CIA testers, Table 1.4. Additionally, HHT measurements at room and elevated temperatures were done for steels characterization. All samples for tests were cut from original boiler tubes in laboratory.
HTC, HTE and HT – CIA tests have been performed for better understanding steels behaviour under HT corrosion – abrasion conditions. All testers are accelerators and test conditions do not agree with real conditions in industrial boiler processes. The temperatures 500 and 600 °C for all tests were chosen for maximal simulation combustion gases temperature in boiler. In HTC test oil shale ash were used for simulation deposits and pure HCl for getting maximal corrosion under such extreme conditions. Erosion (HTE) at such a high speed (80 m/s) was selected to reduce possible role of oxidation during erosion-oxidation process. Erosion tests were conducted at impact angles of 30° and 90°, respectively. HT-CIA test was done for investigation steels behaviour. Test conditions are extremely high and could not appear in boiler in the same type, but different variations of it could be found in fuel handling systems.

Table 1.4. Testing matrix.

<table>
<thead>
<tr>
<th>Hot hardness test (HHT)</th>
<th>High temperature corrosion (HTC) test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range: RT, 100 – 800 °C</td>
<td>Temperature range: 500, 600 °C Conditions: HCl, oil shale ash</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>High temperature erosion (HTE) test</th>
<th>High temperature cyclic impact abrasion (HT-CIA) test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range: 500, 600 °C</td>
<td>Temperature range: 500, 600 °C Conditions: 0.8 J, abrasive 3 g/s, 45°</td>
</tr>
<tr>
<td>Conditions: 80 m/s, 30°, 90°</td>
<td>Conditions: 0.8 J, abrasive 3 g/s, 45°</td>
</tr>
</tbody>
</table>
1.6 Steels properties

The chemical compositions of all steels (Table 1.5) are given in Table 1.6 [PAPER I].

Table 1.5. Investigated steel grades

<table>
<thead>
<tr>
<th>Investigated steels</th>
<th>Equivalent steels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel Grade</td>
<td>Standard</td>
</tr>
<tr>
<td>A</td>
<td>12X18H10T</td>
</tr>
<tr>
<td>B</td>
<td>X10CrMoVNb9-1</td>
</tr>
<tr>
<td>C</td>
<td>12X1MФ</td>
</tr>
<tr>
<td>D</td>
<td>TP347HFG</td>
</tr>
</tbody>
</table>

Table 1.6. Chemical composition of investigated steels in wt. % [PAPER I].

<table>
<thead>
<tr>
<th>Tested steel</th>
<th>Composition, wt. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel Grade</td>
<td>Standard</td>
</tr>
<tr>
<td>A</td>
<td>12X18H10T</td>
</tr>
<tr>
<td>B</td>
<td>X10CrMoVNb9-1</td>
</tr>
<tr>
<td>C</td>
<td>12X1MФ</td>
</tr>
<tr>
<td>D</td>
<td>TP347HFG</td>
</tr>
</tbody>
</table>

In order to avoid variant reading the steels grades in this thesis were designated according to DIN and EN standards.

Material A is an austenitic, corrosion-resistant steel X12CrNiTi18-9. This steel has a high temperature resistant microstructure Cr – 18 %, Ni – 10%, which improves the resistance against corrosion in reducing environments and stress corrosion cracking [35]. Additionally titanium is added (0.52 %) to stabilize the carbon and the chromium in the matrix and increasing the fracture toughness by precipitating intergranular TiC [36].

Martensitic steel B (X10CrMoVNb9-1) is a chromium alloyed high strength steel with increased creep resistance. At the chromium content of 9 % and the added molybdenum (1.08 %) the pitting and crevice corrosion resistance is increased [37, 38]. Steel C is a pearlitic steel 13CrMoV42 with a chromium content of 1.44 %.

The austenitic steel X8CrNiNb16-13 (D) has homogeneous structure in bimodal distribution of the grain size. In this steel Nb is added to increase the strength and the resistance against different corrosive attacks [39].

Metallographic cross sections of all investigated steels in AC²T [PAPER I] can be seen in Figure 1.14. Steel A, as seen in Figure 1.14a reveals austenitic microstructure, with intergranular TiC-precipitations and TiN inclusions. In Figure 1.14b the martensitic steel B can be seen, revealing its typical microstructure. The pearlitic structure of 13CrMoV42 can be seen in Figure 1.14c. Steel D (Figure 1.14d) reveals austenitic structure with twin grain boundaries and intergranular precipitations (mostly NbC).
1.7 Hardness at room and elevated temperatures

Hardness is the resistance of a material to plastic deformation. The hardness is higher for material which is more difficult to plastically deform under load. Hardness is one of the most important and easily measurable properties which has a major effect on abrasive wear. By abrasive wear, material wear rate depends on material hardness— if it is lower than abrasive hardness, microcutting of the surface may take place. If material hardness is higher than abrasive hardness, clear removal of the material usually does not take place and the entire process has the nature of fatigue [40].

The hardness test at room and elevated temperatures was done for steels investigated. The tests were performed in Austrian Centre of Competence for Tribology (AC²T). Figure 1.15 displays the hot hardness tester.

The room temperature hardness was determined by standard Vickers hardness testing procedure. Average hardness values were calculated from 10 indents per specimen.

The hot hardness test (HHT) is based on the Vickers HV10 method extended for elevated temperature up to 800 °C. A load of 10 kg was chosen to measure the compound hardness of steels. As prevention against oxidation, the whole test is performed under low vacuum condition (5 mbar). Sample’s position can be set by an actuator and variation of hardness over temperature curves can be measured on one sample. Three indents at each temperature were done for statistical evaluation. The diagonals of the indent marks were measured by
means of optical microscopy after cooling down and Vickers hardness was calculated. Figure 1.16 presents example of HHT indents. For this investigation the hardness was measured at room temperature (RT), 100, 300, 500, 600, 700 and 800 °C, respectively [PAPER II].

Steels have an initial HV10 Vickers hardness of ~221 HV10 (A); 235 HV10 (B); 146 HV10 (C); 180 HV10 (D) at RT which is slowly declining with increasing the temperature, as seen in Figure 1.17 martensitic steel B shows linear decrease up to 500 °C due to annealing processes – exceeding this temperature the hardness drops more rapidly due to further microstructural changes. Ferritic-pearlitic steel C reveals softening due to the formation of globular Fe₃C [PAPER II].

Figure 1.15. Hot hardness tester (HHT): a – scheme of apparatus, b – image [41].

Figure 1.16. Image of HHT indents of steel (D) austenitic steel X8CRNINB16-13.
Figure 1.17. Hot hardness of steels investigated: (A) austenitic steel X12CrNiTi18-9, (B) martensitic steel X10CrMoVNb9-1, (C) pearlitic steel 13CrMoV42, (D) austenitic steel X8CrNiNb16-13 [PAPER II].
2 HIGH TEMPERATURE CORROSION TESTING

2.1 Corrosion tester and experiments

The high temperature corrosion tests of all steels investigated were carried out within the newly developed HTC tester. The test rig is mainly based on a furnace with a thermal protection, a gas feed and cleaning unit. A detailed scheme for the HTC tester is given in Figure 2.1. The test rig is based on a horizontal furnace equipped with a ceramic working tube. This furnace enables gaseous corrosion testing up to 1500 °C with randomly mixable gasses. The temperature and flow of corrosive gases are automatically controlled. The gasses are fed into the furnace through a thermal protection, where the samples are embedded in crucibles. At the other side of the heated tube, there is also a thermal protection including a gas cleaning unit, for high process reliability and workspace security. To ensure highest security for operators and the surrounding laboratories, the whole test rig is implemented in a protection chamber with an alarm system [PAPER I].

Within this study experiments were performed at 500 °C and 600 °C with an exposure time of 19 h. The corrosion tests have been performed on grinded specimens (25x15x6 mm), which have been cut from original boiler tubes. Prior to the tests all specimens were ultrasonically cleaned with ethanol, precisely measured by a calliper and weighed. After that, the samples were coated by manual brushing with a suspension of oil shale ash and ethanol for the optimal simulation of oil shale ash on-tube deposits for the corrosion tests. The tests were carried out in the temperature-controlled horizontal tube furnace of the HTC tester. For the experiments HCl was passed through the chamber at a velocity of 1,9 cm/s. The samples were positioned vertically and parallel to the flow direction. After the test procedure the samples were weighed, determining the mass gain for the calculation of the corrosion rates. For the experiments in oxidative environment, the samples were given into a furnace at 500 °C and 600 °C for 24h in synthetic air atmosphere (O₂~20 %, N₂~80 %). The oxidation tests have been done and the characterization was studied quantitatively by determining the samples mass increase [PAPER I].
Figure 2.1. The HTC tester illustration.

Figure 2.2. The HTC tester [PAPER I].
2.2 Properties of the Estonian oil shale ash

For the lab tests oil shale ash was processed by co-firing oil shale and biofuel in a Narva Power Plant with a CFB boiler, as seen in Figure 1.6. The fusion temperature of this ash strongly depends on the chemical and mineralogical composition of the ash. The main components of the Estonian oil shale ash which influence the ash fusibility are CaO, SiO₂ and Al₂O₃. Mineral matter in oil shale matter is a heterogeneous system containing minerals of carbonatic and sandy-clay nature. Due to the heterogeneity of different mineral matter in oil shale different fusion temperatures can occur [2]. In general, the fusion temperature for oil shale ashes and their components is higher than 1000 °C [39].

After the firing/processing an electrostatic precipitator (Figure 1.6 – pos. 11) was used to separate oil shale ash with a particle size from 45–60 µm. These oil shale ash particles were used for the corrosion tests in the HTC tester [42].

The chemical composition of the ash, analyzed by methods of analytical wet chemistry presented in Table 2.1:

<table>
<thead>
<tr>
<th>Components</th>
<th>%</th>
<th>Components</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO₂</td>
<td>5.11 %</td>
<td>CaO₉</td>
<td>10.80 %</td>
</tr>
<tr>
<td>C.elem (CO₂)</td>
<td>1.37 (5.04) %</td>
<td>CaO</td>
<td>29.40 %</td>
</tr>
<tr>
<td>SO₃ common (Scom)</td>
<td>6.58 (2.63) %</td>
<td>MgO</td>
<td>5.63 %</td>
</tr>
<tr>
<td>S sulphate</td>
<td>1.86 %</td>
<td>K₂O</td>
<td>4.10 %</td>
</tr>
<tr>
<td>S sulphite</td>
<td>0 %</td>
<td>Na₂O</td>
<td>0.25 %</td>
</tr>
<tr>
<td>SiO₂</td>
<td>35.72 %</td>
<td>Cl</td>
<td>0.50 %</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>5.33 %</td>
<td>Unburned parts</td>
<td>5.60 %</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>8.47 %</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2.1. Chemical composition of the ash [PAPER I].
2.3 Results and discussion

Mass change due to high temperature HCl corrosion in Estonian oil shale ash and oxidation

The mass gain depends on types of formed oxides (FeO, Fe₂O₃, Fe₃O₄, etc.) so the precise corrosion rate based on the determined mass gain is not possible without determination of above mentioned compounds and may be used for preliminary ranking of steel HTC resistance. For a more precise assessment of the steels HTC resistance, oxide layer was removed with an etchant and after the complete removal according to [42] the total mass loss was determined. Therefore a methodology using a 20 % aqueous Di-ammonium hydrogen citrate [(NH₄)₂HC₆H₅O₇] solution was used to remove the oxide layer from all surfaces. Etching was conducted at 80 °C with several repetitions. Every etching cycle had a duration of 30 minutes. Figure 2.4 shows consistent decrease of the samples mass due to the oxide layer removing process. The mass gain of tested samples before etching the samples and the total mass loss due to HTC are presented in Figure 2.5.
Figure 2.4. Relative mass changes of the steels investigated under HCl corrosion at 500 °C and 600 °C after corrosion layer removing. 1.1–1.3, 2.1–2.2, 3.1–3.3, 4.1–4.4 are number of the samples [PAPER I].
Figure 2.5. Mass loss and mass gain of the steels investigated under HCl corrosion at 500 °C and 600 °C after oxide layer removing [PAPER I].

One specimen of each steel was chosen for SEM-investigations – Therefore the sample was embedded and grinded/polished for optimal examination of the built-up corrosion products. Metal mass loss of specimen divided by specimen area allows obtaining the corrosion depth according to the following equations 2.1 and 2.2 [43]:

\[ \Delta S = 1.28 \cdot \Delta m \ [\text{mm}] \] for carbon steels and medium-alloy pearlitic steels \ (2.1)  
\[ \Delta S = 1.25 \cdot \Delta m \ [\text{mm}] \] for high-alloy martensitic and austenitic steels \ (2.2)  

Corrosion depths of all samples in HCl atmosphere and in Estonian oil shale ash are presented in Table 2.2 and Figure 2.6.

Table 2.2. Corrosion depth of the samples in HCl atmosphere and oil shale ash; Values given in [mm] [PAPER I].

<table>
<thead>
<tr>
<th>Steel type</th>
<th>500 °C</th>
<th>600 °C</th>
<th>Steel type</th>
<th>500 °C</th>
<th>600 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.00163</td>
<td>-</td>
<td>C</td>
<td>0.00492</td>
<td>0.00764</td>
</tr>
<tr>
<td></td>
<td>0.00378</td>
<td>0.00390</td>
<td></td>
<td>-</td>
<td>0.00782</td>
</tr>
<tr>
<td>B</td>
<td>0.00550</td>
<td>-</td>
<td>D</td>
<td>0.00367</td>
<td>0.00860</td>
</tr>
<tr>
<td></td>
<td>0.01550</td>
<td>-</td>
<td></td>
<td>0.00782</td>
<td>0.01100</td>
</tr>
</tbody>
</table>
To compare the results of laboratorial methods determining the high temperature corrosion of steels, which were performed at the Department of Thermal Engineering, Tallinn University of Technology (DTE TUT) [31–34] results of both test series are given in Figure 2.6. As seen in this graphs, the results of DTE TUT laboratory tests are presented as kinetic diagrams of corrosion depth in coordinates $\ln \Delta S - \ln \tau$. In logarithmic coordinates the kinetic lines of high temperature corrosion are straight lines and usually expressed by following empirical correlation (2.2) [31–34].

Coefficients of graphs 2.6a and 2.6b are determined experimentally, at graph 2.6c is used coefficients specified in [43].

As it could be seen from Figure 2.6a the results of present experimental of steel A (12X18H10T) are in good agreement with the results of DTE TUT laboratory tests of this steel. Compared to steel A the corrosion depths of steels B, C and D are much higher [PAPER I].

In Figure 2.6b are presented steel grade B (X10CrMoVNb9-1) AC$^2$T experimental HTC results in comparison with the results of DTE TUT [44]. It is seen that corrosion depth at temperature 600 °C (AC$^2$T experiments) is a little higher than it was measured in DTE TUT at temperature 580 °C and 620 °C. Corrosion depth at temperature 500 °C (AC$^2$T experiments) is lower than DTE TUT at temperature 540 °C. So in case of steel grade X10CrMoVNb 9-1 it could be found good agreement with previous results as well.

In Figure 2.6c are presented steel grade C (13CrMoV42) AC$^2$T experimental HTC results in comparison with the PTM empirical correlation [43] based on...
HTC tests of this steel under effect of oil shale ash provided in DTE TUT. It is seen that corrosion depth at temperature 500 °C is in good agreement with empirical correlation and at temperature 600 °C is significantly lower.

In conclusion of comparative analysis of HTC experimental results it should be emphasized that the results of HTC experiments, which have been carried out in laboratory AC²T are in a good agreement with results obtained from the tests conducted in DTE TUT for three investigated steels. The HTC tests in AC²T have been carried out in the media of gaseous HCl and oil shale ash, while the tests in DTE TUT have been performed in the media of natural gas combustion products and periodically renewable oil shale ash. Thus a good agreement of the results allows to consider used in AC²T technique for HTC investigation as a valid one and the results to be reliable. Moreover the AC²T technique has an advantage in comparison with DTE TUT technique because it does not require periodical interrupting of the experiment for oil shale ash renewal. HTC laboratory tests of investigated boiler steel grades in the presence of sulphur and potassium containing oil shale ash have shown that the best corrosion resistant is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, pearlitic steel grade (C) 13CrMoV42 and martensitic (B) X10CrMoVNb9-1. That sequence represents the corrosion ranking of the tested steel grades for their application as high-temperature heating surfaces in boilers firing oil shale which contain chlorine, sulphur and potassium in outer deposits. Obtained ranking and HTC tests results could be used for selection of steel for boiler components designed for operation in similar conditions.

In order to make more precise life time assessment of component operated in HTC conditions in the presence of oil shale ash deposits the long term field tests are required.

In high temperature oxidative environment all steels show mass increase due to the formation of oxide surface layer. The mass changes of all steels investigated are given in Figure 2.7.

![Figure 2.7. Mass changes of the steels investigated in oxidative environment at 500 °C and 600 °C for 24h [PAPER I].](image)

As seen in the figure, steel C show the highest mass increase due to the lowest amount of alloying elements like chromium and nickel, which can decrease
oxidation due to the formation of dense layers [PAPER I]. The ranking of steel grades resistance in high temperature oxidative environment (air) in the laboratory conditions was as follows: the best resistant is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, martensitic (B) X10CrMoVNb9-1 and pearlitic steel grade (C) 13CrMoV42.

2.4 Qualitative analysis of the formed corrosion layer using scanning electron microscopy

In the following chapter the effects of high temperature corrosion of the investigated steels are shown and discussed. Qualitative analyses were performed to understand high temperature corrosion of the presented steels using a scanning electron microscope (SEM) “Zeiss FESEM Gemini Supra VP” and energy dispersive X-Ray spectroscopy (EDX) [PAPER I].

Prior to the removal of the built-up scales the surface of the corroded samples were analyzed by SEM/EDX. Metallographic cross sections of the corroded samples were prepared to analyze formed layers and the corrosive behavior of different phases from all steels investigated. In Figure 2.9 and Figure 2.10 analysis of a surface of the samples after exposure are observed after the tests at 500 °C and 600 °C. In Figure 2.8 and in Figures 2.11 – 2.18 cross-sectional analysis of the steels investigated are given. The oxide layer formed after 500 °C and 600 °C corrosion test for 24h is different for austenitic, pearlitic and martensitic steels [PAPER I].

As mentioned above, the corrosion layer thickness strongly depends on chemical composition of steels. Steels (A) austenitic X12CrNiTi18-9 alloy and (D) austenitic X8CrNiNb16-13alloy consist of 18.48 % / 18.64 % of chromium (Cr) and 10.39 % / 11.19 % of nickel (Ni) [PAPER I].

Steel A Figure 2.11, 2.15 shows a well visible sublayer at the boundary between the base metal and the oxide layer which is damaged by pores and cracks formed according to above described corrosion process. Chlorides which penetrate through the base material at 500 °C cause a porous sublayer at the boundary. The thickness of this porous sublayer may be estimated as 5 µm, at the temperature of 600 °C increases up to 7 – 9 µm – locally the sublayer disappeared completely [PAPER I].

Steel B, a martensitic steel X10CrMoVNb9-1 alloy consist 8.86 % of chromium and 1.08 % of molybdenum. As seen in Figure 2.12, 2.16 the boundary between the base metal and the oxide layer is a well visible sublayer damaged by pores and cracks. The uneven surface of the base metal indicates to a different corrosion rate along the surface of the sample. At 500 °C the thickness of the porous sublayer can be estimated to ~10 µm, at 600 °C increases up to 20-25 µm [PAPER I].

Steel C, Figure 2.13, 2.17 the pearlitic steel 13CrMoV42 did not built-up any protective oxide layer at both temperatures (500 °C and 600 °C) because of low contents of heat-resisting alloying elements such as chromium (1.44 %). Porous sublayer between the base metal and the oxide layer cannot be detected, but the corrosion develops quite uniformly along the sample surface [PAPER I].
The influence of volatile chlorides, especially chromium chlorides, on the corrosion mechanism in this case is at a minimum wage due to the low chromium content in the bulk. The corrosion rate of this low alloyed steel grade in HCl environment is comparable to the corrosion of higher alloyed austenitic steels and lower than those for martensite, due to the same low resistance against gaseous HCl at elevated temperatures due to the lack of protective layers which can resist the chlorine attack [PAPER I].

Steel D Figure 2.14, 2.18: At 500 °C the thickness of the porous sublayer can be estimated as 5 – 7 µm, at 600 °C increases up to 10 – 15 µm. In this sublayer many pores are visible throughout the whole samples. Those significant difference in the HTC rate between two austenitic steel grades (having about the same content of Chromium and Nickel but with different elements used for stabilization of the structure) can be pointed out to the formation of less resistant layers at steels D and the higher amount of nickel, which more easily forms chlorides than chromium and the resulting formation of porous sublayers [PAPER I].
Figure 2.8. Scanning electron microscopy (SEM) surface cross-sectional analysis of the oxide layer formed after corrosion tests at 500 °C (1) and 600 °C (2) for 24h on steels: (A) austenitic steel X12CrNiTi18-9, (B) martensitic steel X10CrMoVNb9-1, (C) pearlitic steel 13CrMoV42, (D) austenitic steel X8CrNiNb16-13.
Figure 2.9. Scanning electron microscopy (SEM) surface analysis of the sample after corrosion tests at 500 °C for 24h on steels: (A) austenitic steel X12CrNiTi18-9, (B) martensitic steel X10CrMoVNb9-1, (C) pearlitic steel 13CrMoV42, (D) austenitic steel X8CrNiNb16-13 [PAPER I].

Figure 2.10. Scanning electron microscopy (SEM) surface analysis of the sample after corrosion tests at 600 °C for 24h on steels: (A) austenitic steel X12CrNiTi18-9, (B) martensitic steel X10CrMoVNb9-1, (C) pearlitic steel 13CrMoV42, (D) austenitic steel X8CrNiNb16-13 [PAPER I].
Figure 2.11. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 500 °C: (A) austenitic steel X12CrNiTi18-9 [PAPER I].

Figure 2.12. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 500 °C: (B) martensitic steel X10CrMoVNb9-1 [PAPER I].
Figure 2.13. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 500 °C: (C) pearlitic steel 13CrMoV42 [PAPER I].

Figure 2.14. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 500 °C: (D) austenitic steel X8CrNiNb16-13[PAPER I].
Figure 2.15. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 600 °C: (A) austenitic steel X12CrNiTi18-9 [PAPER I].

Figure 2.16. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 600 °C: (B) martensitic steel X10CrMoVNb9-1 [PAPER I].
Figure 2.17. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 600 °C: (C) pearlitic steel 13CrMoV42 [PAPER I].

Figure 2.18. Scanning electron microscopy (SEM) cross-sectional analysis of the corrosion layer after the high temperature corrosion tests at 600 °C: (D) austenitic steel X8CrNiNb16-13 [PAPER I].
Influence of the ash deposits on the corrosion of boiler steels

The protective properties of deposits, arising on a superheater surface, strongly depend on the properties of the surface, the content of alloying elements in a formed oxide layer, its porosity, the presence of crack and the character of cohesion of a layer with the base metal. In general alloying elements migrate from the base metal into the oxide layer, and their content in a layer is correlated with its content in the base metal and face temperature, remaining constant along the thickness of oxide layer [34, 44]. However, if external deposits on tubes contain chlorine compounds the corrosion processes and the resulting phenomena essentially are changed. The compounds of chlorine force a chemical reaction with both, chrome and iron. A counter migration takes place; chlorine migrates from the surface into the oxide layer and further to the base metal of the tube. Alloying elements migrate in an opposite direction [30].

The main components in an oxide layer, which decreases the velocity of the corrosion process in steels, is chromium oxide Cr₂O₃ and the mixed phases like NiCr₂O₄ and FeCr₂O₄ which at the presence of chlorine will form compound CrCl₃ volatilize from the tube surface [46]. Compounds like FeCl₃, CrO₂Cl₂, CrO₃, NiCl₂ etc. similarly volatilize. Thereby the oxide layer becomes porous, with low anti-corrosive protective properties, and KCl can easily diffuse through the pores to more cold layers of tube. Further, the present chlorides react with the gaseous combustion atmosphere and the oil shale, which both are sulphur containing. The elevated temperatures during the combustion process can turn the built-up chlorides into sulphates [31].

In the present experiments hot corrosion involves corrosion reactions between molten salts, oxidation, sulphatation and gaseous corrosion. In environment like air, the normal oxide scale which forms at the surface of an alloy acts as an effective barrier to further oxidation [15].

In practice, the oxide scale often becomes porous and cracked, allowing much faster transport along these channels. When molten salts are present, the salt wets the surfaces of the oxides and enables the corrosive anions to penetrate deep into and through the pores and cracks by capillary action [15].

In presented corrosion experiment no oxygen (O₂) was added into the gas flow, pure hydrogen chlorine (HCl) was reacted with steels covered with oil shale ash. The reaction with gas (HCl) and ash, containing sulphides leads to salts formation. Under high temperature melting salts localized molten salts corrosion is the main damaging mechanism, which can be present in all phases occurring in a material [45].

On the basis of conducted experiments have been found:

- HTC tests of investigated boiler steel grades in the presence of sulphur and potassium containing oil shale ash in HCl environment have shown that the best corrosion resistant is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, pearlitic steel grade (C) 13CrMoV42 and martensitic (B) X10CrMoVNb9-1. That sequence represents the corrosion ranking of the tested steel grades for their
application as high-temperature heating surfaces in boilers firing oil shale which contain chlorine, sulphur and potassium in outer deposits.

- The ranking of steel grades resistance in high temperature oxidative environment (air) was as follows: the best resistant is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, martensitic (B) X10CrMoVNb9-1 and pearlitic steel grade (C) 13CrMoV42.

**Performance maps**

In order to make reasonable selection of relevant steel grade in definite laboratory conditions the corrosion-oxidation performance maps (Figure 2.19, 2.20) have been proposed.

![Performance map of studied steels at 500°C.](image)

![Performance map of studied steels at 600°C.](image)
3 HIGH TEMPERATURE ABRASIVE WEAR TESTING

3.1 Study of high temperature erosion

*Erosion testing method*
Solid particle erosion tests have been performed in a centrifugal four-channel accelerator (HTE) in which up to 20 specimens can be treated simultaneously under identical testing conditions (Figure 3.1). Prior to the tests all specimens were ultrasonically cleaned with ethanol, precisely measured by a calliper and weighed. The abrasive particles used in this work were rounded silica particles. Investigation of steady state erosion rate was made as a function of the impact angle at the abrasive particles velocity of 80 m/s [19]. Erosion at such a high speed was selected to reduce possible role of oxidation during erosion-oxidation process. Erosion tests were conducted at impact angles of 30° and 90°, respectively. The duration of each erosive test was approximately 1 hour. In each test, specimens of the size of 20×15×4.5mm were treated simultaneously. Parameters of the tests are given in Table 3.1 [46]. After the test procedure the samples were weighed, determining the mass loss.

*Table 3.1. Testing parameters used in erosion testing [46].*

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Impact velocity</td>
<td>80 m/s</td>
</tr>
<tr>
<td>Impact angles</td>
<td>30°, 90°</td>
</tr>
<tr>
<td>Erodent</td>
<td>Silica sand</td>
</tr>
<tr>
<td>Total weight of erodent</td>
<td>6 kg</td>
</tr>
<tr>
<td>Test temperatures</td>
<td>500 and 600 °C</td>
</tr>
</tbody>
</table>
Results of high temperature erosion of steels

The volumetric wear rate \( K_{\text{vol}} \) was determined as wear rate \( K \) of the target sample divided by steel density (Figure 3.2, 3.3).

\[
K_{\text{vol}} = \frac{K}{\rho} \quad [\text{mm}^3/\text{kg}] \tag{3.1}
\]

where wear rate \( K \) is calculated as following:

\[
K = \frac{\Delta m}{G \cdot v} \quad [\text{mg/kg}], \tag{3.2}
\]

where \( \Delta m \) – specimen weight loss [mg];
\( G \) – the quantity of sand [kg];
\( v \) – is share of sand per one specimen for elevated temperature tester \((v = 0.0187)\).

An accuracy of 0.1mg was obtained for the target mass loss measurements.

The investigated steels show a change of the wear in the temperature range from 500 °C to 600 °C. Considering steels B, C and D volumetric wear rate for both tests increases with increasing temperature. Opposite behaviour can be detected for austenitic steel A which exhibits better wear performance at higher testing temperature of 600 °C in test with impact angle of abrasive 30° [46]. That may be explained by particle sticking (Figure 3.4 and 3.5).
Analyzing steels wear rate accordingly to impact angle of abrasive particle influence, it should be mentioned that wear rate is lower with impact angle of 90° for steels investigated [46].

![Figure 3.2. Volumetric wear rate of steels at elevated temperatures under the impact angles of 30° [45].](image1)

![Figure 3.3. Volumetric wear rate of steels at elevated temperatures under the impact angles of 90° [46].](image2)

For better understanding of steels behaviour in erosive conditions the SEM analysis of samples after test was performed. A typical SEM images of steels are given at Figure 3.4 and 3.5. It can be clearly seen that abrasive SiO₂ particles embed into wear surface [46]. The worn surfaces of steel tested under 30° and 90° impact angles at elevated temperatures indicates a highly plastically deformed surface. The highest volumetric wear rate among the tested materials shows steel (C) 13CrMoV42 that may be explained by low hot hardness.
Figure 3.4. Worn surfaces of eroded steels at temperature 600 °C of impact angle 30°: (A) X12CrNiTi18-9, (B) X10CrMoVNb9-1, (C) 13CrMoV42, (D) X8CrNiNb16-13[46].

Figure 3.5. Worn surfaces of eroded steels at temperature 600 °C of impact angle 90°: (A) X12CrNiTi18-9, (B) X10CrMoVNb9-1, (C) 13CrMoV42, (D) X8CrNiNb16-13[46].
3.2  **Study of high temperature abrasion wear**

*Cyclic-impact abrasion tester and experiments*

For the high temperature cyclic impact abrasion (HT – CIA) testing apparatus (Figure 3.7a) of the Austrian Centre of Competence for Tribology (AC²T) was used to determine the behaviour of steels at elevated temperatures. Test principle is based on potential energy (0.8 J) which is cyclically turned into kinetic energy by free fall. Samples are fixed at 45° and are periodically hit by the plunger with 1.8 – 2.0 Hz frequency with a constant flow of abrasive (3 g/s, silica sand with particle size of 0.4 – 0.9 mm, Fig. 3.7c) between plunger and sample (Figure 3.7b) [24]. Tests were done at 500 °C and 600 °C. Prior to the tests all specimens were ultrasonically cleaned with ethanol, precisely measured by a calliper and weighed. After the test specimens were ultrasonically cleaned again and weighed. Wear was determined by mass loss and wear volume was calculated via steels density. The plunger material used for these tests was a Co-rich high-speed steel. Test duration was set constant to 1 h. The testing parameters are summarized in Table 3.6. The characterization of wear behaviour was done by determining the weight loss (accuracy 0.1 mg). Qualitative analysis were performed [PAPER II] by standard optical microscopy (Figure 3.9, 3.10) and by confocal microscopy Leica DCM 3D (Figure 3.12).

*Table 3.6. Testing parameters of high-temperature cyclic impact abrasion [PAPER II].*

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<td>Impact angle</td>
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<tr>
<td>Frequency</td>
<td>1.8 – 2 Hz</td>
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<tr>
<td>Number of testing cycles</td>
<td>6900 – 7200</td>
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<tr>
<td>Abrasive, size, shape and hardness</td>
<td>Silica sand; 0.4 – 0.9 mm; angular; 1000 – 1200 HV</td>
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<tr>
<td>Abrasive flow</td>
<td>3 g/s</td>
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<tr>
<td>Test temperature</td>
<td>500 and 600 °C</td>
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Figure 3.7. High-temperature cyclic impact abrasion (HT – CIA) test: a) view of the tester; b) testing principle; c) micrograph of abrasive particles [24].

Results of high-temperature impact-abrasion of steels

Qualitative analysis was performed by standard optical microscopy (Figure 3.9, 3.10) and by confocal microscopy Leica DCM 3D (Figure 3.12). The results of the HT – CIAT tests are displayed in Figure 3.8. The investigated steels show a significant change of the wear in the temperature range from 500 °C to 600 °C. Considering steels A, B and D that exhibit higher hot hardness values than steel C, volumetric wear increases with increasing temperature. It can be assumed that the volumetric wear increases due to softening effects of the microstructure. Opposite behaviour can be detected for the ferritic – pearlitic steel C which exhibits better wear performance at higher testing temperature of 600 °C. In order to deeper understand this effect SEM analyses of the wear scars in the different regions were performed for all steels. A typical SEM analysis of the worn surface of steel B is present in Figure 3.11a. It can be clearly seen that abrasive SiO₂ particles stuck in the wear surface in impact zone of the sample. EDS line scan analysis was done additionally to detect incorporate abrasive particles in the surface and near-surface region (Figure 3.11b). Low hot hardness of steel C allows a strong interaction of abrasive SiO₂ particles with the ductile surface zone within the impact region, resulting in a pronounced formation of a mechanically mixed layer which enables the protection of the material and reduction of the wear rate [PAPER II].
Figure 3.8. Volumetric wear of steels at elevated temperature under HT – CIA [PAPER II].

Figure 3.9. Macro images of typical wear scar of steel tested at 500 °C temperature: (A) X12CrNiTi18-9, (B) X10CrMoVNb9-1, (C) 13CrMoV42, (D) X8CrNiNb16-13.
Figure 3.10. Macro images of typical wear scar of steel tested at 600 °C temperature: (A) X12CrNiTi18-9, (B) X10CrMoVNb9-1, (C) 13CrMoV42, (D) X8CrNiNb16-13.

Figure 3.11. SEM micrographs of typical worn surfaces of steel B (martensitic steel X10CrMoVNb9-1) after cyclic impact/abrasion testing at 600 °C: (a) Impact zone; (b) Formation of mechanically mixed layer (MML) [PAPER II].

Additionally, qualitative analysis were performed by confocal microscopy Leica DCM 3D for steel (A) X12CrNiTi18-9. The topography and longitudinal and transverse profile of wear dimple has been measured, see Figure 3.12.
Figure 3.12. The topography and profiles of wear dimple of steel (A) X12CrNiTi18-9.
**Volumetric wear maps**

To summarize the volumetric wear maps (Figure 3.13, 3.14) have been proposed for the comparison of the investigated steels behaviour in different operating conditions.

Figure 3.13. Volumetric wear map of studied steels at 500 °C.

Figure 3.14. Volumetric wear map of studied steels at 600 °C.
4 GENERAL CONCLUSIONS

The corrosion, erosion and impact-abrasion of four boiler steels have been tested at high temperatures in laboratory conditions.

On the basis of the experiments tested steel grades have been ranked as follows:

- **HTC**: the best corrosion resistant material is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, pearlitic steel grade (C) 13CrMoV42 and martensitic (B) X10CrMoVNb9-1.
- **Oxidation**: the best resistant material is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, martensitic (B) X10CrMoVNb9-1 and pearlitic steel grade (C) 13CrMoV42.

The results have shown that the ranking of steels according to their resistance to high temperature corrosion and oxidation does not agree with the ranking on the basis of tribotests.

In order to make reasonable selection of relevant steel grade for boiler components designed for operation in definite conditions the corrosion-oxidation and abrasive wear performance maps have been proposed. The real operation conditions of the component should be taken into account.

**Scientific Novelty**

Scientific novelty of the doctoral thesis includes:

- New data of four boiler steel grades resistance to high temperature corrosion and abrasive wear have been obtained.
- New methodology of high temperature corrosion (HTC) testing has been proposed and realised in newly developed HTC tester.
**Future work**

The thesis proposed high temperature corrosion, oxidation and two types of material wear analysis: high temperature erosion and high temperature cyclic impact-abrasion.

The major interest for future researches provides high temperature corrosion in aggressive gas and ash media. It makes interest to conduct cyclic test with the kinematical growth of the corrosion layer, to measure elements with XRD-analysis in it and to find chlorine impact on corrosion velocity in gas environment.

It would be interesting to evaluate erosion-oxidation, erosion-corrosion mechanism during the tests and to plot the EO, EC maps. These maps could help to identify the regime in which a system will be operating and determine the degree of material wastage that can be expected for a given system.

In order to make more precise life time assessment of component operated in HTC conditions in the presence of oil shale ash deposits the long term field tests are required.

The EU Framework Programme for Research and Innovation Horizon 2020 [47] will provide fellowships for individual researches, which could be considered as major source of financial support.
References


Abstract

Investigation of Corrosion and Wear of Boiler Steels at Elevated Temperatures

Thesis is focused on the boiler steels investigation under corrosion, erosion and abrasion impact on the basis of laboratory tests. In order to analyze the boiler steels with relation to corrosion and wear at elevated temperatures the number of experiments has been performed.

High temperature corrosion tests have been carried out in AC2T laboratory using new testing technique proposed by the author of the given thesis. According to this technique the HTC tests have been carried out in the media of gaseous HCl and oil shale ash. Some of the tested steel grades have been previously tested in DTE TUT using another technique namely the tests have been performed in the media of natural gas combustion products and periodically renewable oil shale ash.

The comparative analysis of HTC experimental results has shown that the results of HTC experiments, which have been carried out in laboratory of AC2T are in a good agreement with results obtained from the tests conducted in DTE TUT for three investigated steels. Thus a good agreement of the results allows to consider used in AC2T technique for HTC investigation as a valid one and the results to be reliable. Moreover the AC2T technique has an advantage in comparison with DTE TUT technique because it does not require periodical interrupting of the experiment for oil shale ash renewal.

HTC tests of investigated boiler steel grades in the presence of sulphur and potassium containing oil shale ash have shown that the best corrosion resistant is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, pearlitic steel grade (C) 13CrMoV42 and martensitic (B) X10CrMoVNb9-1. That sequence represents the corrosion ranking of the tested steel grades for their application as high-temperature heating surfaces in boilers firing oil shale which contain chlorine, sulphur and potassium in outer deposits. Obtained ranking and HTC tests results could be used for selection of steel for boiler components designed for operation in similar conditions. Right steel selection could help to increase the durability and operating reliability of boiler components.

The oxidation rate of the steels in high temperature oxidative environment (air) was also investigated and the ranking of steel grades resistance in these conditions was as follows: the best resistant is austenitic steel grade (A) X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, martensitic (B) X10CrMoVNb9-1 and pearlitic steel grade (C) 13CrMoV42.

Solid particle erosion tests have been performed in TUT laboratory. The abrasive particles used in this work were rounded silica particles. Investigation of steady state erosion rate was made as a function of the impact angle. Erosion tests were conducted at impact angles of 30° and 90°. It was found that the best high temperature erosion resistance shows austenitic steel grade (A)
X12CrNiTi18-9, then are going austenitic steel (D) X8CrNiNb16-13, martensitic (B) X10CrMoVNb9-1 and pearlitic steel grade (C) 13CrMoV42. The volumetric wear rate of the steels B, C and D increases with temperature increasing. Opposite behaviour can be detected for austenitic steel A which exhibits better wear performance at higher testing temperature of 600 °C in test with impact angle of abrasive 30°. That may be explained by particle sticking. It was also found that the volumetric wear rate is lower with impact angle of 90°. All of investigated steels have been subjected to high-temperature cyclic impact abrasion testing as well. The tests have been performed in AC²T laboratory. The specimens were fixed at 45° and periodically hit by the plunger at 1.8-2.0 Hz frequency with a constant flow of abrasive. Tests have been carried out at the temperatures 500 °C and 600 °C. The characterization of wear behaviour has been done by determining the weight loss. It was found that the best wear resistance shows pearlitic steel grade (C) 13CrMoV42. The volumetric wear rate of the steels A, B, and D increases with temperature increase. Opposite behaviour can be detected for pearlitic steel grade (C) which exhibits better wear resistance at higher testing temperature of 600 °C. It could be explained by the sticking of the abrasive particles to the specimen surface that decreases the rate of steel wear. This conclusion has been made on the basis of SEM analysis of the tested specimen surface.

The results of carried out investigations of boiler steels X12CrNiTi18-9, X10CrMoVNb9-1, 13CrMoV42 and X8CrNiNb16-13 have shown that the ranking of steels according to their resistance to high temperature corrosion and oxidation does not agree with the ranking on the basis of tribotests. Thus in order to select relevant steel grade for particular boiler component the real operation conditions of the component should be taken into account. To summarize the corrosion-oxidation-wear rate maps have been proposed for the comparison of the investigated steels behaviour in different operating conditions.
Kokkuvõte

Katlateraste kõrgetemperatuurilise korrosiooni- ja kulumisuuringud

Väitekiri on keskendunud katlamaterjalide korrosiooni, erosiooni ja abrasiooni laboratoorsetele uuringutele. Katlamaterjalide korrosiooni ja kulumise uurimiseks kõrgetel temperatuuridel tehti rida katsid.


Kõrgetemperatuurilise korrosioonikatsete tulemuste võrdlus näitas, et kolmele terasemargile AC²T laboratooriumis saadud tulemused on heas kooskõlas varem TTÜ STI-s saadud tulemustega. Tulemuste hea kokkulepe näib, et AC²T-s kasutatud meetod toimib ja on usaldusväärne. Veelgi enam, AC²T-s kasutatud meetodil on TTÜ STI meetodi ees mõningad eelised, sest puudub katsetada põlevkivi tuha uuendamiseks.

Katlateraste kõrgetemperatuurilised katsed väävlist ja kaaliumit sisaldava põlevkivi tuha juuresolekul on näidanud, et kõige suurem vastupanu korrosioonile on austeniitterasel (A) X12CrNiTi18-9. Talle järgneval korrosioonikindluse kahanevas järjekorras austeniitteras (D) X8CrNiNb16-13, perliitteras (C) 13CrMoV42 ja martensiitteras (B) X10CrMoVNb9-1. See järjestus annab katsetatud terastest põletamisel, kus gaasipoolsed sadestused sisaldavad kloori, väävlist ja kaaliumit.

Saadud katlateraste järjestust ja kõrgetemperatuuriliste korrosioonikatsete tulemusi saab kasutada sarnastes tingimustes töötavate katlakomponentide materjalide valikus. Materjalide õige valik aitab tõsta katlakomponentide tööga ja töökindlust.

Samuti uuriti materjalide korrosioonikindlust kõrgetemperatuurilises kõrgetemperatuurilises oksüdeerivas keskkonnas (õhk) ja saadud korrosioonikindluse järjestus on järgmine: parim on austeniitteras (A) X12CrNiTi18-9, millele järgneval austeniitteras (D) X8CrNiNb16-13, martensiitteras (B) X10CrMoVNb9-1 ja perliitteras (C) 13CrMoV42.

Tahkete osakestera erosioonikatsed tehti TTÜ laboris, kasutades abrasiivsete osakste ümmargusi räniosakesi. Statsionaarset erosioonikirust uuriti lõõginurga funktsioonina, valides lõõginurkadeks 30° ja 90°. Leiti, et suurimate vastupanu kõrgetemperatuurilisele erosioonile osutab auteniitteras (A) X12CrNiTi18-9, millele järgneval austeniitteras (D) X8CrNiNb16-13, martensiitteras (B) X10CrMoVNb9-1 ja perliitteras (C) 13CrMoV42. Materjalide B, C, ja D mahuline kulumine suureneb temperatuuri tõusuga.
Vastupidist käitumist võib täheldada austeniitterase A korral, mis näitab paremat kulumiskindlust kõrgemal temperatuuril 600 °C juures 30° lööginurga korral. Seda võib seletada osakeste kleepuvusega. Ühtlasi tuvastati, et 90°-se lööginurga korral on mahulise kulumise kiirus väiksem.
Kõiki uuritud materjalide katsetati ka kõrgel temperatuuril tsüklisti abrasiooni tingimustes. Katsed viidi läbi AC²T laboratoriumis. 45°-se nurga all kinnitatud katsekehti pommitati sagedusega 1,8÷2,0 Hz konstantse abrasiivmaterjali joaga. Katsed viidi läbi temperatuuridel 500 °C ja 600 °C. Kulumise iseloomu määrati kaalukao järgi.
Tuvastati, et parimat kulumiskindlust näitas perliitteras (C) 13CrMoV42. Vastupidiselt käitub perliitteras C, mis näitab suuremat kulumiskindlust kõrgemal temperatuuril 600 °C juures. Seda võib seletada abrasiivosakeste kleepumisega katsekeha pinnale, mis vähendab sel viisil kulumise kiirust. Selline järeldus tehti katsekehade pinna skaneeriva elektronmikroskoobiga analüüsimise tulemusel.
Katlaterastega X12CrNiTi18-9, X10CrMoVNb9-1, 13CrMoV42 ja X8CrNiNb16-13 läbiviidud uuringud näitasid, et nende järjestus kõrgetemperatuurilise korrosiooni- ja oksüdeerumiskindluse järgi ei lange kokku nende järjestusega tsüklisti kulumiskindluse järgi. Seega tuleb igale katla-komponendile sobiva terasemargi valikul võtta arvesse tegelikke töötööningimisi. Uuritud katlateraste korrosiooni-oksüdeerimis-kulumiskindlusest täielliku ülevaate saamiseks pannakse töös ette võrrelda nende materjalide käitumist erinevates töötööningimustes.
Elulookirjeldus

Isikuandmed

Ees- ja perekonnanimi          Jelena Priss
Sünniaeg ja -koht              16.05.1983, Eesti
Kodakondsus                   Eesti
E-posti aadress               jelenapriis@gmail.com

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<td>2014</td>
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---|---
2012- 12th International Symposium „Topical Problems in the Field of Electrical and Power Engineering“ (5 days) | Doctoral School of Energy and Geotechnology, Kuressaare, Estonia.
2012- Välisõppe 5 kuud | AC²T, Wiener Neustadt, Austria

4. **Teenistuskäik**

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5. **Teadustegevus**


Curriculum Vitae

Personal data

Name: Jelena Priss  
Date and place of birth: 16.05.1983, Estonia  
E-mail: jelanapris@gmail.com

1. Education

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<td>Pärnu Russian Gymnasium</td>
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<td>Tallinn University of Technology (TUT)</td>
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<td>B. Sc., Thermal Engineering</td>
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5. Scientific work


27. **Sergei Tisler.** Deposition of Solid Particles from Aerosol Flow in Laminar Flat-Plate Boundary Layer. 2006.


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82. **Heiki Tiikoja.** Experimental Acoustic Characterization of Automotive Inlet and Exhaust System. 2014.