

# Lowering Costs by Improving Efficiencies in Biomass Fueled Boilers: New Materials and Coatings to Reduce Corrosion

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Dissemination level			
PU	Public	٧	
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	Services)		
со	Confidential, only for members of the consortium (including the Commission		
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### VALIDATION

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### **DOCUMENT DATA**

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### **Executive Summary**

This deliverable includes the laboratory testing protocol defined and followed for biomass induced fireside and steam corrosion. The protocol has been designed and optimized along the project aiming to produce results that are comparable to pilot and real plant scale conditions.

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# 1. Corrosion test protocol under simulated biomass atmosphere and steam

Substrate coupons for lab scale testing were agreed to have the following dimensions: 20 mm x 10 mm x 2 mm. For all the experiments, the uncoated sample coupons are ground up to 800-SiC paper grit with intermediate steps at 180, 400, 600-SiC. To remove grease and dirt after the grinding or polishing, samples will be degreased with acetone following with an ultrasonic bath for 5 minutes. Finally, the samples will be cleaned with ethanol to remove any remaining acetone spots.

Prior to exposure, all samples are dimensioned with digital calliper and the weight is recorded by a precision balance of five decimals. Their mass is recorded before and after the test.

List of steps followed for fireside exposures:

1. Samples are weighed and measured before exposure.

Spray KCl saturated solution on the sample surface, dry and weight until 2 mg/cm<sup>2</sup> for time exposure up to 2000h while. 3mg/cm2 was sprayed for longer exposures. The furnace must be calibrated to determine the temperature profile so that a working zone with the test temperature +/-  $5^{\circ}$  C is localized..

- 2. Ramp the temperature of the furnace to 600 °C.
- 3. Adjust the flow rates of gases.
- 4. Insert the KCI-boat upstream from the samples.
- 5. Samples will be inserted in the furnace at temperature and removed at temperature.
- 6. The mass gain will be measured after exposure.
- 7. Cross-sectional investigations (mount/cut/polish) will be carried on one of each type of coated sample out using SEM-EDX, XRD and digital microscopy for macrographs which includes the full sample profile.

List of steps followed for steamside exposures at low pressure (1 atm):

- 1. Samples must be weighed and measured before exposure.
- 2. Roughness of the ground uncoated substrates must be measured (3 measures on each side).
- 3. The furnace must be calibrated to determine the temperature profile.
- 4. Samples will be inserted in the furnace at room temperature in the zone with the accepted temperature range (600 +/- 5).
- 5. Close the reactor, introducing  $N_2$  into the system and ramp the temperature of the furnace/s up to 600 °C.
- 6. Adjust the water flow rate.
- 7. At the test temperature, the  $N_2$  flow will be stopped and the water/steam will be introduced.
- 8. The reverse procedure will be used to remove the samples.
- 9. The mass gain will be measured after exposure.

10. Cross-sectional investigations (mount/cut/polish) will be carried on one of each type of coated sample out using SEM-EDX, XRD and digital microscopy for macrographs which includes the full sample profile.

### List of steps followed for steamside exposures at high pressure:

- 1. Roughness of the ground uncoated substrates must be measured (3 measures on each side).
- 2. All samples must be weighed and measured before exposure.
- 3. Sample preparation must be done just before the exposure and the polished samples are stored in desiccator.
- 4. Samples will be loaded in the autoclave and the temperature and pressure are ramped up according to typical operation procedure.
- 5. Temperature for the exposure will be 600 °C and pressure 250 bar, flow rate will be 5 ml/min which corresponds to linear gas velocity of 0.02 cm/s.
- 6. Samples are weighed and visually examined at 168 hours, 366 hours, 500 hours, 750 hours, and 1000 hours. Then every 250 h up to 2000 h.
- 7. Samples are removed for investigations after 500, 1000, and 2000 h.
- 8. Cross-sectional investigations (mount/cut/polish) will be carried on one of each type of coated sample out using SEM-EDXXRD and digital microscopy for macrographs which includes the full sample profile.

### 1.1. Fireside

### **1.1.1.** Parameters for fireside lab test

- Material: reference materials and coated samples (2 samples/coating; standing vertical and parallel to the gas flow)
- Polishing and Grinding: as-received for coated samples only
- Environment: 5% O<sub>2</sub> + 20% H<sub>2</sub>O + N<sub>2</sub> (bal.)
- Temperature: 600 °C
- Stops: Every 1000 h for gravimetric control. Selected times for further microstructural investigations.
- Salt deposition: KCl-boat plus KCl 2 mg/cm<sup>2</sup> sprayed up to 2000 h while 3mg/cm2 was sprayed for longer time.
- Flow rate: 0.1 cm/s

### 1.2. Steamside

### **1.2.1.** Parameters for steamside lab test – Low pressure

- Material: reference materials and coated samples (2 samples/coating; standing vertical and parallel of the gas flow).
- Polishing and Grinding: as-received for coated samples only.
- Roughness of the uncoated substrates must be measured (3 measures on each side)

- Environment: 100% H<sub>2</sub>O
- Temperature: 600 °C
- Pressure: atmospheric
- Stops: Every 1000 h for gravimetric control. Selected times for further microstructural investigations.
- Linear velocity: 7 cm/s
- Removal of 2 samples/system for characterising: 2000 hours, 10000 hours and 15000 hours.

### **1.2.2.** Parameters for steamside lab test – High pressure

- Materials: reference materials and coated samples (2 samples/coating).
- Polishing and Grinding: substrates up to 800–SiC grit (used in the RR tests) and as received for coated samples.
- Environment: 100% H<sub>2</sub>O (150 ppb O<sub>2</sub>)

Water flow rate: 5 ml/min

Temperature: 600°C

- Pressure: 250 bar
- Duration: at least 1000 h
- Stops for visual and gravimetric monitoring: 168 hours, 366 hours, 500 hours, 750 hours, 1000 hours. Then every 250 h up to 2000 h and/or until the end of the test
- Removal of 2 samples/system for characterising: 1000 hours, 2000 hours and/or 4 months before project completion or until the system failure.

# 2. Degree of Progress

This task has been completely fulfilled.

## 3. Dissemination Level

Public

## 4. References

Internal protocols and procedures belonging to the partners.

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Deliverable 3.1.

Deliverable 3.3

Deliverable 3.4