# FIELD TESTS OF FURNACE MATERIALS

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One of the goals of SoTu-project was to test different potential cladding materials (3R12, San36Mo, San41 ja San63) in actual recovery boiler lower furnace conditions, but at higher than current temperatures, in order to determine what materials could be suitable for future high pressure recovery boilers. Field tests were meant to be performed in two boilers over the course of a year, but this objective had to be given up, when tests with air cooled probes failed. The executive board on SoTu decided in 2005 to give Boildec Oy task to design and build a liquid cooled probe, with which different types of cladding materials could be tested in recovery boiler furnace for 1000 hours so, that temperature of test materials is 400°C and 440°C.

The task was challenging, because there had not been built a liquid cooled probe in Finland before. The liquid cooled probes which have been built elsewhere, liquid had been circulated with pumps, while now the aim was to use natural circulation in probe cooling. Moreover the schedule was tight, because time reserved to build equipment and do the experiments was under a year.

# **Furnace Probe**

The experimental setup consists of natural circulation circuit which includes steam generator in which the test materials are placed (Figure 1). The steam generator is installed into liquor gun opening so that the front wall, consisting of the test materials (plate type), is at same level with furnace wall tubes.

The natural circulation circuit is cooled by air. Air flow is controlled so that the circuit pressure is kept constant. To the front wall of steam generator were installed thermal elements, with which both temperature of test materials underneath the surface of the test pieces and heat flux directed at front wall of the steam generator could be measured. Surface temperature of the test materials could be calculated on the grounds of the results. Based on one temperature measurement a fan was controlled, with which air was blown to cool the condenser. The control of the fan was aimed to be done so, that temperature of the test materials remained the same.

The heat transfer fluid used in the probe was organic heat transfer oil, the heat of evaporation of which is 257 - 425 °C in pressure of 1 - 14 bar. Due to this the

temperature of front wall of the steam generator could be adjusted to desired, while the pressure of the probe being under 10 bar.



Figure 1. Natural circulation probe

# **Performed tests**

#### <u>Test 1.</u>

First test was performed in March 2006. Test was begun, when load of the boiler was being raised. To ensure that the probe would not suffer excessive pressure, air fan was adjusted to full power during installation.

Heat flux and surface temperatures of the test pieces calculated on the grounds of temperature measurements are shown in figure 2.



Figure 2. Results of first test

Air cooling was enough to keep temperatures of test materials in desired range, when heat flux directed at front wall of the boiler was at most 200 kW/m<sup>2</sup>. During the night between March 8th and 9th the heat flux rose so high that the probe pressurized until safety valve opened. Through the safety valve escaped so much heat transfer oil that steam generator dried up and overheated. Overpressurization of the probe happened at maximum load of the boiler.

It was presumed in the design of the probe that maximum heat flux of the probe would be at most  $150 - 200 \text{ kW/m^2}$ . The probe operated when heat flux was in this range, but when heat flux went up the cooling power was not sufficient anymore; therefore structure of the probe had to be changed.

Functionality of the altered probe was decided to be ensured also in full load before continuing actual experiments by doing a test, in which all test materials are not used, but front wall of the boiler is built only of 3R12-steel.

### <u>Test 2.</u>

Second experiment was performed in April 2006. Results of the experiments are shown in figure 3.



Figure 3. Results of second test

Results of the experiment showed that probe works as expected when boiler is at full load. The maximum heat flux directed at probe was admittedly lower than in previous experiment, but on grounds of the results the control was obviously working, even if heat flux would be occasionally even higher.

Results showed also, that automatic control of probe temperature worked well in that respect, that the temperature of the lower part of the steam generator front wall was always very close to desired value, when heat flux directed at probe was large enough. On the other hand temperature difference between upper and lower parts of the steam generator was occasionally about 50°C. Temperature differences being so large different the test materials could not be installed to the front wall of steam generator in such a way, that temperature of the materials would be the same (400 or 440°C), therefore to fix this defect the structure of the probe had to be changed further. In the first experiment similar problem was not observed, therefore assumption was that necessary changes in probe structure to even out test material temperatures were implementable. Next experiment was thus decided to be performed by using test materials.

## Experiment 1.

The experiment was performed 26 June -7 July duration being 1006 h. Temperature control worked mainly fine, as can be seen in results of figure 3. In the figure is shown example of surface temperatures of the materials over duration of 24 hours.



Figure 4. Various surface temperatures of test materials in first experiment

The temperatures of different test materials are rather close to each other and target temperature of 400°C.

According to calculations surface temperature of San41-test piece, in which the temperature measurement used in control was located, varied during the experiment between 388 - 432°C over a course of 630 hours. In the periods concerned calculated average surface temperatures of test materials were 391°C (3R12), 405°C (San36Mo), 397°C (San 41) and 401°C (San63).

Unfortunately temperature control did not stay stable during the entire experiment, but temperatures of the test materials went four times over 500°C. Combined duration of the periods, when temperatures of the test materials were over 500 °C, was 2,5 h. Regarding to corrosion, the critical temperature is probably 660 °C, in which eutectic composition of nickel and sulphur is formed. Temperatures higher than this were measured in test pieces only for 10 minutes and only in one material (San63). Thus it's possible, that results are representative regardless of occasional excessive temperatures – possibly excluding San63.

Reason for aforementioned temperature spikes was occasional covering of steam generator with wet black liquor acting as a heat sink so long that the circuit pressure dropped below atmospheric, despite of the fact that the fans rotational speed was adjusted to minimum. The critical heat flux of liquid transfer fluid, ie. the heat flux, which liquid can receive without developing of uniform steam membrane between liquid and hot surface, which functions as insulator causing rise of temperature of the surface to several hundred degrees higher than temperature of the liquid, is dependent of pressure declining with lowering pressure. Once black liquor shower ends and the steam generator is again exposed to high heat flux, heat transfer liquid at these low pressures may sometimes evaporate vigorously resulting in heat transfer crisis and, consequently, test material temperature surge. As a result of which the surface temperature rose to about 600  $^{\circ}$ C.

To eliminate these surges the probe was equipped in the next experiment with electric heater, keeping the circuit pressurized by heating it during the periods when circuit pressure is temporarily low.

### Experiment 2.

The experiment was performed 1. - 29. September duration being 484 h. The experiment was interrupted earlier than planned, when liquid level of the probe was discovered to be lowered significantly and leakage point was not known. The lowering of liquid level did not have impact on temperatures of test pieces. Example from results of the experiment can be seen in figure 5.



Figure 5. Various surface temperatures of test materials in second experiment

Cooling of the probe was controlled according to temperature of San41-test piece so, that surface temperatures of the test pieces were about 440 °C. According to the results the temperature of San41-test piece was over 420 °C for altogether 354 hours, over which time averages of the surface temperatures were 431 °C (3R12), 437 °C (San36Mo), 444 °C (San41) and 432 °C (San63). Temperature of the test pieces was over 470 °C only for 6 minutes, greatest value being 509 °C.

The added electric heater in the probe was working until 16th of September, when it burnt causing also a fuse to blow. The probe measurements and the fan got their electricity from the same fuse circuit, hence the probe was without active cooling and its state is unknown for 47 h period of time. Safety valve limits the pressure of the probe so, that surface temperature of the test pieces can be at most about 490 °C. According to test results of the safety valve the safety has not opened during the experiment, hence it's possible that temperatures of the test materials have been lower than mentioned during power failure. This assumption is backed up by the fact that temperatures of the test pieces were about 440 °C at the moment when electric supply returned and for most of the duration of the power failure the load of the boiler was considerably lower than at the moment when electric supply returned.

## CONCLUSION

SoTu-project was successful in designing and building completely new kind of a probe, which can be used to test different potential cladding materials in actual recovery boiler lower furnace conditions but at higher than current temperatures. In the project the probe was used in two experiments, in which temperatures of the test materials were about 400 and 440 °C. Durations of the experiments were ca. 1000 and 500 h.

In the first experiment control of the temperatures of the test materials worked well excluding 2,5 hour period, when temperatures went over 500 °C. The temperature spikes resulted from probe covering with wet black liquor and liquor igniting abruptly when pressure of the probe was low.

To prevent temperature spikes, in the second experiment the probe was equipped with electric heater. Purpose of the heater was to maintain pressure in periods of time, when heat flux from furnace to probe is low.

Due to changes made in the probe the control of the temperatures of test materials worked excellently in the second experiment. Surface temperatures of the test materials were near target value of 440 °C and changes in the temperatures were low. Hardship in the experiment was, that following the burning of the electric heater the equipment of the probe were left without electricity for 47 hours, for which period of time measurement results are missing. The break in measurements and probe cooling caused by heater burning could have been prevented by installing other devices to different fuse circuits.

Normally the experiments probably would have been repeated to ensure results, but due to tight schedule of the project this was not possible this time. Due to this results are initial. If the experiments will be carried on, test materials could be tested also in different spots in the furnace or in higher temperatures, which correlate to cladding that has been in use for a long time.

The probe can be also used to measure the heat flux directed at walls of furnace of a recovery boiler. According to the measurements performed this time the heat flux directed at the wall on black liquor spraying level varies considerably from time to time. At maximum the flux was about 275 kW/m<sup>2</sup> (in experiment 1) the average flux in experiment 1 was 169 kW/m<sup>2</sup> and in experiment 2 123 kW/m<sup>2</sup>. Obvious reason for temporal variations of the heat flux is splashing of wet black liquor onto the probe.