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### Innovative 1000K Sodium Loop For Qualification Of New Materials For Applications In CSP Field

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Abstract. The present paper describes the 1000K sodium loop developed at the Karlsruhe Institute of Technology for creep fatigue and corrosion/erosion investigations of new materials for applications in the concentrating solar power plants. The construction of the high temperature loops is motivated also by the lack of experimental data for steels in flowing sodium at temperatures above  $\sim$ 650°C, as revealed by the literature review. The study discusses the loop operation procedures and the safety measures considered. Some of the experimental results obtained so far are presented, namely the loop operations at maximal specified temperature, at maximal specified flow rate, the calibration of the sodium flowmeter and the dynamic flow conditions in the loop. The new materials proposed at KIT for the receiver are briefly presented.

#### **INTRODUCTION**

In the aftermath of the newly proposed concept [1] of a CSP (concentrating solar power) power plant employing sodium as heat transfer fluid (HTF), various research topics have been underlined that require dedicated and thorough experimental and numerical investigations. The major topics identified range between experimental investigations of fundamental thermal-dynamics in CSP relevant geometries, development, investigation (e.g. creep-fatigue tests, corrosion and erosion studies) and qualification of new materials for high temperature CSP components (receiver, pipelines, heat exchangers) up to investigations of sodium based thermoelectric converters for DC generation. Several high temperature sodium facilities have been erected at KIT to fulfill these tasks [2]. Moreover, the experimental data will be used for qualification of the CFD (computational fluid dynamics) codes for liquid metals flows.

Although the CSP field can profit from numerous material studies performed in hot flowing sodium for nuclear liquid metal fast reactors, besides the experimental investigation and development of new materials, the lack of experimental data for creep fatigue and corrosion investigations above 700°C motivated the erection of the SOLTEC (SOdium Loop for TEst materials and Corrosion) facilities at KIT. An overview of the sodium effects on material corrosion and creep fatigue behavior is reported by Borgstedt [3]. Uniaxial creep-rupture and low-cycle fatigue (LCF) data for stainless steels 304 and 316 in sodium-exposed conditions at temperatures between 550 and 700°C are reported by Natesan et al. [4] for applications in the nuclear field. The LCF data obtained at 600°C on these

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stainless steels showed that thermal aging of the materials in either argon or sodium had a significant effect on fatigue life. The results on the cyclic stress-fatigue life indicated a greater strain-hardening rate for SS 316 than for SS 304 under all material conditions. Microstructural examination of the fatigue specimens showed a significantly smaller number of cracks after sodium exposure than in the annealed condition. LCF tests of steel SS 304 and SS 316L(N) in flowing sodium at 550°C were reported also by Borgstedt and Huthmann [5]. The observed corrosion phenomena affects the steel mechanical properties, if the surface to volume ratio might be large enough. The LCF life of steel SS 304 was longer in sodium compared to reference tests in air in all these tests. The LCF behavior of SS 316L(N) in sodium at 550°C was comparable to that of SS 304. In general, fatigue lives of 316L(N) specimens tested in sodium were found to be higher than those tested in air up to by a factor of ~4. However, for tests conducted under hold time conditions, fatigue lives in both the environments were comparable [6].

In Karlsruhe, SS 304 samples were cut from the test section of the sodium loop CREVONA that was operated for more than 80000h and analyzed by SEM/EDAX for microstructures and changes in chemical composition [7]. The tubes were operated for 60000h at 550°C and 20000h at 600°C at a sodium velocity of 1.5 m/s. Although the leaching rates of Cr and Ni were rather similar to other steels, the SS 304 develops porosities due to the exposure to sodium. Hence, the authors suggest that austenitic steels containing molybdenum are better compatible with sodium. Corrosion tests of T91, 15-15Ti and 316L(N) steels in liquid sodium at 550°C up to 1600h are reported by Courouau et al. [5] in the CORRONa loop. At this temperature, the authors report a corrosion rate of about 15  $\mu$ m/y for both 316L(N) and 15-15Ti and 27  $\mu$ m/y for T91. However, the authors mention that lower corrosion rates should be expected if the oxygen level would be controlled to a lower level as in the tests.

#### DESCRIPTION OF THE SOLTEC EXPERIMENTAL FACILITY

The SOLTEC family of test facilities are similar loops designed for a maximal operating sodium temperature of 720 °C at a maximal overpressure of 3.5 bar. The maximal sodium flow rate is specified at 300 kg/h and depending on the inner diameter of the test sample sodium velocities up to  $\sim$ 5 m/s can be reached in examined material probes.



FIGURE 1. Piping and instrumentation diagram of the SOLTEC-1/-2 facility

One innovative characteristics of the loop is the usage of the sodium tank not just for storage but also for the sodium expansion during operation, reducing the number of components and avoiding additional connections in the high temperature side. Another innovative feature is the combined sodium-sodium heat recuperator coupled to a sodium-air heat exchanger to ensure an efficient heat recuperation, a tight arrangement and to avoid the welding of different materials. Due to its compact configuration, namely  $1.2 \times 1.6 \times 1.9$  m<sup>3</sup>, the loop can be easily transported and integrated in a standard laboratory that is appropriately equipped for liquid metal experiments. A safety-by-design approach and several safety measures, such as a complete shielding (removed in Fig 1a), have been adopted, to provide the highest safety for the operating staff.

SOLTEC-1 and -2 are constructed with an adaptable connection to the test section, to ensure a large flexibility in the integration of the test section. Each facility has its own test section, namely SOLTEC-1 (see Figure 2b) for creep-fatigue and SOLTEC-2 for corrosion investigations (see Figure 2c). SOLTEC-3 is built also as a universal test loop for investigations of thermoelectrical converters and high temperature receiver prototypes for CSP plants.

SOLTEC-1 and -2 have an 8-shape configuration (see Figure 1) consisting of a low temperature side, up to 450°C and the high temperature side, where the test section is placed. The vacuum pump branch and the storage tank branch are attached to the low temperature side and are usually operated at low temperatures. All major components (sodium pump, sodium-air heat exchanger, sodium flowmeter, sodium pressure sensors) are placed in the low temperature side, to limit the number of welds and connections on the high temperature side. The argon circuit is connected to the vacuum pump branch and to the storage tank. Argon, as cover gas, is used to protect the sodium against oxidation, for filling and drainage procedures and pressure monitoring in the loop. All safety and overpressure valves are placed in the argon circuit and the connection between the sodium in the operating loop and the pressurizing argon in the storage tank/expansion tank is permanently ensured during normal operation procedures.





At the interface between the low temperature side and the high temperature side, a 27kW sodium-sodium heat recuperator has been integrated and directly connected to the 7.5 kW sodium-air heat exchanger to provide a compact and efficient heat recuperation after the test section. In the high temperature side a 6.3 kW heater has been installed.

The design of the facility followed a safety-by-design approach that included several safety measures. The emergency drainage is initiated at any critical malfunction. The high temperature heater and the sodium pump are to be stopped either by the programmable logic controller (PLC) or by sensors operating separately from the PLC. The sodium inventory in the loop is less than 12 liter, while only about 0.6 liter are in the high temperature side (heater and test section). Since no water-based components are used, the only possible scenarios leading to fire events are due to the sodium-air contact, as for example in the case of sodium leaking from cracks. As a safety measure for this possible scenario, the leaking detection system installed on the sodium pipelines and components will initiate the emergency drainage, so only a small amount of sodium can possibly exit the loop. Even in this case, the sodium will be first retained in the outer hull and the thermal insulation. If leakages occur in the storage tank, the sodium will drain in the metallic gathering tray below the loop. Any possible fire will be restrained to the encapsulated metallic housing of the facility and extinguished due to the lack of oxygen and reactant consumption. The low overpressure at which the loop is operated will limit also the amount of sodium that can potentially can exit the facility. Another safety measure is that any maintenance, overhaul and inspection actions are performed only at room temperature when the sodium is in solid state. Up to the present date, no leakage and fire incidents are to be reported.

Initially the sodium purification from SOLTEC was intended to be performed in the KASOLA facility [9] available at KIT. This option and the request to limit the dimensions of the SOLTEC loop have led to the decision to not include a cold trap in the loop. A sodium optical inspection in the storage tank revealed its shiny silvery aspect. However, prior to the experimental tests for corrosion investigations, a cold trap designed in the meantime will be integrated in the loop.

#### **EXPERIMENTAL RESULTS**

Prior to the tests of the planned materials, an extensive experimental campaign has been conducted in the frame of the facility set-into-operation. The campaign aimed the test of the facility functionality, coupling it with the optimization of the PLC controlling software, tests of all major operation states (loop filling with sodium, regular operation, loop drainage), operation tests at maximal temperature at different flow rates, tests of the instrumentation (e.g. calibration of the flowmeter) and tests of the safety systems.

Prior to the regular loop filling, the facility is evacuated both at room temperature (with sodium frozen) and in hot condition at about 200°C. Once the loop has been evacuated, the sodium pump is started and pressurized argon at about 200 mbar is applied on the storage tank, so that the loop can be filled with sodium. The loop filling process takes only a few minutes and is usually performed at a temperature in the loop of about 200°C. Once the loop has been filled, the high temperature heater is set to the desired test temperature and the sodium pump to the requested power. After the test has been performed, if the temperature in the high temperature side is still too high, i.e. more than 150°C difference to the temperature in the storage tank, then the sodium is further circulated in the loop and cooled in the sodium-air heat exchanger, while the high temperature heater is set to a lower temperature. To prepare the drainage, once a temperature gradient below 150°C has been reached between the maximal temperature in the loop and the temperature in the storage tank, the argon pressure on the storage tank is reduced to atmospheric pressure and the sodium pump is stopped. Two regular drainage modes can be applied, namely a faster one, with pressurized argon at about 100mbar applied on the argon transfer line on the NA-V-02 valve (see Figure 1) or by simply opening the argon transfer line between the sodium and the storage tank. In this case, sodium will flow by gravity in the storage tank, while the loop is filling with argon at atmospheric pressure.

#### **Operation at maximal temperature**

One of the first experimental tests performed after the loop proper functionality was achieved, was the test at the maximal temperature foreseen for the loop. An overview of the temperature distribution along the loop during the test is presented in Figure 3.

For this test, the storage tank is held at about 200°C, while the low temperature side, which is highlighted with the blue rectangle in Figure 3, is held at about 300°C. In the high temperature side, highlighted with the red rectangles, the high temperature heater heats up the sodium up to 720°C (see cyan rectangle). At a sodium flow rate of 0.04l/s, which represents ~40% from the maximal specified flow rate, the temperature gradient along the high temperature heater is ~166°C, while the temperature difference between the cold inlet and the hot outlet of the

sodium-sodium heat recuperator is ~255°C, meaning that about 79% of the heat is recuperated. At this flow rate, the estimated sodium velocity in a test sample having an inner diameter of 11mm is 0.42 m/s. Even at higher sodium flow rates of 0.051/s and 0.061/s, the heat recuperation remains rather constant at the same level mentioned above.



FIGURE 3. View of the PLC display during a test at the maximal temperature of 720°C reached in the high temperature heater. Blue rectangle designates the low temperature side, red rectangles designate the high temperature side. The red arrows indicate the sodium flow direction.



FIGURE 4. Temperature distribution along the high temperature heater during a temperature ramp test at a sodium volume flow rate of 0.04 l/s

The maximal operational temperature can be obtained also for a flow rate of 0.051/s and a temperature of  $\sim 300^{\circ}$ C in the low temperature side. For higher flow rates, the maximal temperature can be reached in the test section if the temperature in the low temperature side is also increased above  $300^{\circ}$ C.

The temperature ramp achieved at 0.04l/s flow rate is presented in Figure 4. Starting from 200°C, a temperature increase of 400°C in the high temperature heater has been reached in between 1h - 1h11min, which corresponds to a temperature ramp in the range 5.6 -  $6.5^{\circ}$ C/min. The maximal temperature of 720°C has been reached in between 1h41min - 2h10min, which corresponds to a temperature ramp in the range 4 -  $5.1^{\circ}$ C/min. At these rather fast temperature ramps the facility can be operated on a daily basis for fast transient tests, as well as for long term tests. The temperature ramps can be further shorten if the starting temperature is increased up to ~450°C, close to the maximal temperature in the low temperature side.

Once the stationary operation state has been reached, the trace heating on the low temperature side is automatically set to zero power by the PLC to limit the energy consumption, while only the high temperature heater remains active, however at reduced power.

#### Estimation of the sodium flow rate

An inductive electromagnetic flywheel flowmeter has been installed in the loop. The calibration of the flowmeter has been performed using the following formula, which is implemented in the PLC system:

$$\dot{V} = \dot{V}_0 \frac{\sigma_{GaInSn}(T_{cal})}{\sigma_{Fluid}(T)} + \left[1 + k \frac{\sigma_W(T)}{\sigma_{Fluid}(T)}\right] 2\pi r n A \tag{1}$$

where  $\dot{V}$  is the sodium volume flow,  $\dot{V}_0$  the reference volume flow rate,  $\sigma$  electrical conductivity,  $T_{cal}$  the calibration temperature, k is the device constant, r the average radius of the rotating disc at flowmeter (47.5 mm), n the rotational speed measured by the flowmeter and A the cross-section of the flow channel.



FIGURE 5. Distribution of the sodium flow rate at different power levels of the sodium pump

The distribution of the measured sodium flow rate function of the pump power is presented in Figure 5. Due to the inertness of the flowmeter, even small changes of the pump power levels are not accurately captured and the flowmeter needs a few minutes to reach the steady state value for a certain pump level. However, the focus of the facility relies on long term tests where stationary flow rates are of high interest. For these investigations, the inertness of the flowmeter is accepted. The calibration Equation 1, presented with black squares in Figure 5, is in good agreement with the experimental values measured with the flowmeter.

To validate the calibration of the flowmeter, the volume flow was determined with the heat equation at constant sodium flow rate:

$$\dot{Q} = \dot{m} c_p \,\Delta T \tag{2}$$

where  $\dot{Q}$  is the heat,  $\dot{m}$  is the mass flow rate,  $c_p$  is the specific heat capacity and  $\Delta T$  the temperature difference. Namely, with the exception of the high temperature heater, all trace heating systems were switched off and the power level of the high temperature heater was determined by measuring its voltage and current. The inlet and outlet temperatures at the high temperature heater were considered for the calculation. Considering several measurements and no heat losses through the thermal insulation, the volume flow rate evaluated with Equation 2 was found to be 2.4-2.5% smaller than the volume flow rate measured with the flowmeter.

#### Velocity and pressure distributions

The sodium velocity is not directly measured in the loop, however it can be easily estimated based on the measured flow rate, namely using the relation

$$\dot{V} = v A \tag{3}$$

where v is the sodium velocity and A the cross-sectional area of the pipe.

Presently, a U-Type pipe having an inner diameter of 11 mm has been mounted in SOLTEC-2 facility. For this test sample velocities up to 1.35 m/s can be achieved at the maximum specified sodium flow rate, as presented in Figure 6. However, significant larger velocities can be reached if the test samples have smaller diameter. In this case, the increased pressure loss due to the smaller cross-section will be compensated by the sodium pump. In the low temperature side, the velocity achieved at the maximal specified sodium flow rate is 1.13 m/s (red curve in Figure 6).



FIGURE 6. Distribution of the sodium flow rate at different power levels of the sodium pump

For a creep fatigue studies, the samples foreseen have an inner diameter in the range of 5 mm. For this diameter, at the maximal specified flow rate the sodium velocity will reach up to 5.4 m/s.

The pressure dependency on the sodium flow rate is presented in Figure 7. The pressure before the sodium pump is held almost constant at the level of the argon pressure applied in the storage tank, namely  $\sim 200$ mbar overpressure. The pressure after the pump has the most sensitive character, since the pressure sensor, which is situated in the close vicinity of the pump, is directly affected by the sodium flow column pumped upwards. The maximal values of the pressure are observed after the sodium pump and for the test sample considered (with ID

11mm) the pressure reaches values up to 1.38 bar a. The pressure prior to the entrance in the high temperature heater does not exhibit this transient character, since the flow oscillations are damped in the sodium-sodium recuperator.



FIGURE 7. Pressure distribution in the sodium at different sodium flow rates

#### NEW MATERIALS FOR HIGH TEMPERATURE CSP APPLICATIONS

Besides austenitic steels and nickel-chrome-based alloys, new materials, such as tungsten-copper composites [10] are planned to be tested in the SOLTEC facilities.

FIGURE 8. W-Cu laminates prototypes as candidate materials for a CSP receiver to be tested in SOLTEC facilities

As reported by Borgstedt [3], refractory metals such as tungsten W are very well compatible with sodium of very high degree of purity, even at high temperatures and flow velocities. Tungsten has the highest melting temperature of all metals in pure form and is an excellent absorber material with high thermal conductivity and high creep resistance. These properties make tungsten one of the most interesting candidates for high temperature applications. Among the drawbacks of tungsten are its low fracture toughness at room temperature and the high brittle-to-ductile transition temperature. For these reasons, pure tungsten is not used directly as structural material. To by-pass these

drawbacks Reiser et al. [10, 11] proposed the ductilization of tungsten through the synthesis of tungsten foil laminates and creation of a multilayer material made of ultra-fine grained tungsten foils. The procedure takes advantage of the unique property of tungsten that by cold-rolling the brittle-to-ductile transition temperature can be reduced. Hence, tungsten-copper laminates have been proposed [10] as candidate materials for high temperature applications, such as the receiver of a CSP plant (Fig. 8). Such laminated W-Cu pipes manufactured at KIT were already successfully tested for hours at the Plataforma Solar de Almería, Spain in a solar furnace under a heat load of 4.5 MW/m<sup>2</sup> [11].

#### CONCLUSIONS

The SOLTEC facility for corrosion/erosion investigations is at the end of its qualification phase. All major operation states (loop filling with sodium, regular operation, loop drainage) have been successfully tested. The maximal operation temperature has been achieved, the flowmeter has been calibrated and the flow rate measurement has been validated. The next step is the certification of the loop by the Technical Inspection Authority (TüV) in Germany, prior to begin of the foreseen experimental tests.

For the test sample currently mounted in the loop sodium velocities up to 1.35 m/s have been reached, which lead to a pressure increase up to ~0.4 bar a after the sodium pump. However, significantly higher velocities can be achieved since the sodium pump has sufficient power reserve and the loop is specified at a maximal overpressure that is almost 10 times higher than the pressure achieved in the tests reported here. The temperature ramps (200 to 720°C) achieved allow the use of the facility on a daily basis for short-term fast transients, without overnight operation. Due to the heat recuperator, after the test section a heat recuperation of almost 80% has been reached.

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