

## **Development of high temperature test facilities for material investigations in hot liquid metal flows**

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### **Abstract**

*At the Karlsruhe Institute of Technology three high temperature liquid metal test facilities have been developed in the frame of the Helmholtz Energy Material Characterization Platform (HEMCP). The motivation for the usage of liquid metals is based on the innovative concept proposed for Concentrating Solar Power. The requirements for the qualification of new materials for high temperature applications on a long time basis could not be fulfilled by a facility with a single test section. Therefore, several universal scientific infrastructures, the Sodium Loop for Test materials and Corrosion facilities (SOLTEC) have been designed, each facility having its specific test section.*

*The core facility consists of two regions, the high temperature side with temperatures up to 1000 K connected to the test section and the low temperature side (up to ~773 K), where the pump and the main components are located. Nickel based steels are foreseen for the high temperature side, while conventional AISI 316Ti stainless steel is used for the low temperature side. At the interface between both regions a heat recuperator has been combined with a Na-air heat exchanger, providing a compact arrangement. All facilities have an electromagnetic pump that drives the sodium into the test section at a maximal mass flow rate of 300 kg/h. Several safety measures have been considered from the design phase and all facilities are designed to operate autonomously with passive fail safe option, allowing a fast emergency drainage and even component failure does not lead to large fire damages.*

*SOLTEC 1 serves for a unique in situ low cycle fatigue investigation for new high temperature materials, such as tungsten laminated compounds. A universal test machine and a high temperature vacuum oven are considered to allow long term and fail safe experiments, even in the case of crack failures. SOLTEC 2 will be used for investigations of steel erosion and corrosion in flowing hot Na environment, while SOLTEC 3 facility will be employed for long term investigations of sodium based thermoelectrical devices. The design activities are finished and the SOLTEC facilities are currently in construction.*

## Introduction

Liquid metals (LM) are considered in the energy field as a promising alternative to conventional heat transfer fluids (HTF) such as solar salts and oils. Liquid metals have good thermodynamic properties (large thermal conductivity and large temperature range as liquids) making them efficient HTFs in a wide range of applications. They have been extensively investigated from the early phase of nuclear fission reactors, especially as coolant in fast neutron reactors. Currently, liquid metal technology is widely used in innovative nuclear systems such as MYRRHA or the GEN-IV systems based on liquid sodium (ASTRID) or lead (ALFRED).

Based on the operational experience gathered in the nuclear field and due to their excellent heat transfer properties LMs such as sodium were proposed as HTF for Concentrating Solar Power (CSP) systems even from the '80s [1]. Following the accident that occurred at Plataforma Solar de Almeria (PSA), Spain, in 1986 the technological interest in sodium diminished. However, in the last decade the interest in LM technology rose again significantly [2,3]. Conventional HTFs for CSP have reached their operating upper temperature limits. In parallel, the demand on higher electrical output and higher efficiency for thermal power plants increased. In this context several new innovative CSP concepts using LMs have been proposed [4] and comprehensive research projects have been started in Germany [5] and USA (SunShot Initiative) [6]. In Australia, after a successful test of a sodium based receiver, the construction of a 6 MWth Solar Thermal Pilot Station has been finished and commissioning tests are currently underway.

## Motivation

Among all liquid metals, sodium has been identified as probably the most appropriate heat transfer fluid for CSP plants [7,8]. Compared to other liquid metals and to conventional HTFs such as molten salts, sodium has several favourable properties that make it a very good heat transfer fluid. It has a large thermal conductivity ( $68.2 \text{ W K}^{-1} \text{ m}^{-1}$  at  $500 \text{ }^\circ\text{C}$ ) that allows a fast conductive thermal transport. Its temperature operating range ( $98\text{-}883^\circ\text{C}$  at atmospheric pressure) as liquid is significantly higher than for conventional HTFs and the low melting temperature ensures lower costs for trace heating. Further, the demands on the pumps are not large, since sodium has a low density ( $834 \text{ kg m}^{-3}$  at  $500 \text{ }^\circ\text{C}$ ). The main disadvantages of sodium are related to its reactive character with water and air. These issues can be tackled by appropriate safety measures and careful maintenance and procedures in order to completely suppress the sodium-water contact. Such an approach is based on international knowledge in handling sodium as well as the KIT expertise.

In the above mentioned context, a new concept for a CSP plant that uses sodium as HTF instead of conventional molten salt has been proposed in [4]. The plant proposed can be operated up to temperatures of  $\sim 900 \text{ }^\circ\text{C}$  at the level of the receiver. The fluctuations of the solar energy can be compensated if a thermal energy storage device is used, which can be appropriately dimensioned to allow a continuous operation on a 24/7 basis. The basis loop of the plant operates below  $\sim 600^\circ\text{C}$ , therefore conventional steels such as AISI 316Ti can be employed. The concept proposes the use of thermoelectrical converters as a topping cycle to harvest the peaks in the solar energy so that the solar heat available in the temperature range  $600\text{-}900^\circ\text{C}$  can be directly converted into DC electricity.

Such a concept raises several issues that need appropriate and dedicated investigation. Namely, several major tasks were identified:

- Investigation and qualification of appropriate materials for high temperature applications and pipelines able to endure such a high sodium temperature level under steady-state and transient conditions,
- Stress-corrosion cracking analysis as well as creep fatigue evaluation of the materials to be performed in hot sodium environment on a long time basis and under isothermal and temperature gradient conditions,
- Investigation of corrosion/erosion rate of high temperature steels in hot sodium environment under steady-state and transient conditions,
- Material qualification for thermoelectric converters in hot sodium environment,
- Long-term investigation of thermoelectric devices to assess the performance behaviour on a long time basis and under different operating conditions.

Such a wide range of experimental investigations cannot be performed using a single test section. Therefore it was decided that three independent facilities will be developed, each having its own test section [9]. The facility was named SODium Loop to TEst materials and Corrosion (SOLTEC).

### **Design of high temperature SOLTEC facilities (P&I diagrams)**

Several requirements were set for the loops and the main ones are further summarized. The loops should have:

- A universal and flexible configuration capable to be coupled to different high temperature test sections or host them,
- Be able to sustain temperatures well above 1000 K and to recuperate a significant part of the lost heat,
- Capability to operate under steady-state and different transient conditions on a long time basis,
- Unpretentious disassembling of the main components for maintenance and exchange of the test samples,
- A compact design with a certain degree of mobility,
- Safety oriented design with limited personnel actions required in case of fire,
- Very fast drainage in emergency cases,
- Low operating costs.

The SOLTEC facilities are 1000 K sodium loops presently under construction. Two similar facilities (SOLTEC-1 and -2) will be used for material investigations and the third facility (SOLTEC-3) is planned for long term tests of the thermoelectric converters. While the differences between SOLTEC-1 and SOLTEC-2 are limited to the test section and operating conditions, the SOLTEC-3 facility has a different configuration than the other two loops due to the different test conditions, see Figures 1 and 2.

For all facilities a very compact construction is considered and the entire loop considering all additional systems should have dimensions of  $1.2 \times 1.0 \times 1.6 \text{ m}^3$  (width  $\times$  depth  $\times$  height). All three facilities have a high temperature side, where the test section is located and where temperatures up to 1000 K can be reached. The main components of the loops are located in the low temperature side, where temperatures of up to  $\sim 773 \text{ K}$  can be reached, as highlighted in the piping and instrumentation diagrams (see Figure 1 and 2). Due to the high temperature level nickel based steels are planned for the high temperature side, where pipelines having dimensions of  $16 \times 2.5 \text{ mm}^2$  will be used. For the low temperature side stainless steel AISI 316Ti will be used. The maximal sodium mass

flow rate planned is 300 kg/h, while the maximal operating pressure planned is low, namely about 2.5 bar abs. At the maximal flow rate, the sodium velocity can reach 0.31 m/s in the low temperature side and 1.1 m/s in the high temperature side. The maximal velocity occurs in the test sample in SOLTEC-1, where velocities up to 4.8 m/s are expected. The total pressure loss in the loop at maximal flow rate is estimated to be about 1.2 bar.

Figure 1 Piping and instrumentation diagram for SOLTEC-1, -2

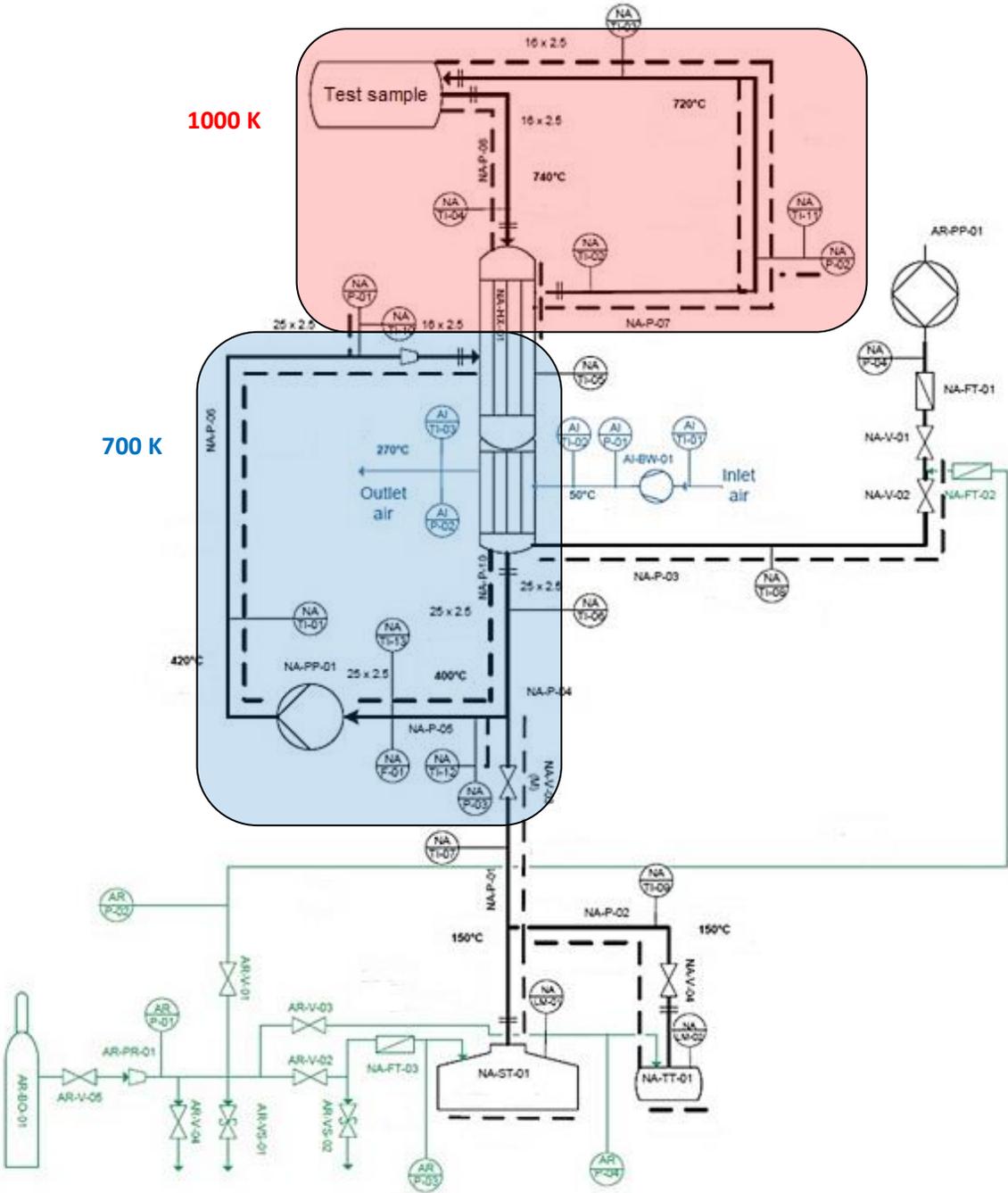
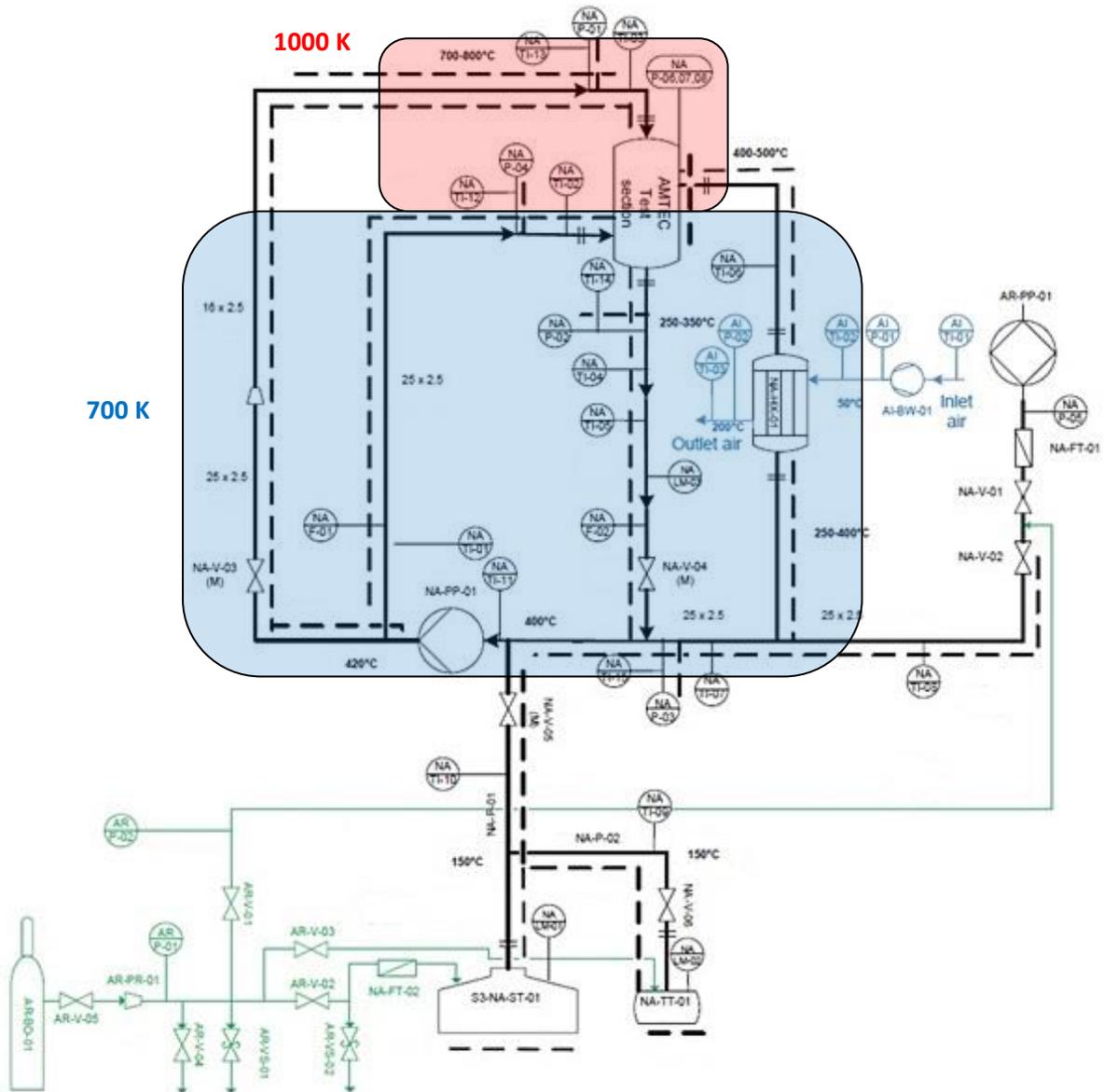


Figure 2 Piping and instrumentation diagram for SOLTEC-3



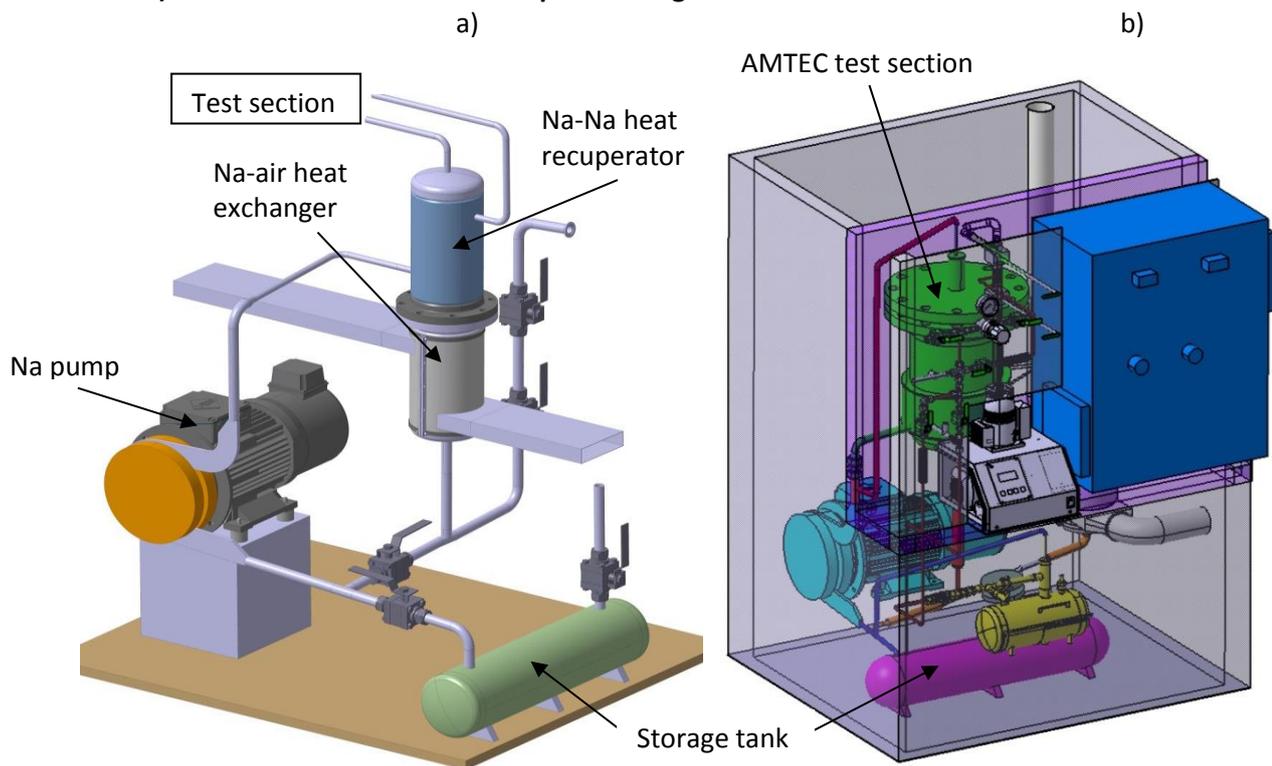
All facilities have the same pump type, namely a 3 kW permanent magnet pump able to operate up to 723 K. Argon is used as cover gas to protect sodium against oxidation and to facilitate the filling and drainage of the facility. All facilities have their own vacuum pump used to extract air and moisture that can potentially react with sodium. All major components (valves, pumps) have been placed in the low temperature side of the loop, where the thermal stresses are lower.

At the interface between the high temperature side and the low temperature side a combined Na-Na heat recuperator and Na-air heat exchanger have been placed for the SOLTEC-1 and -2 loops (see Figures 1 and 3a). This combined heat exchanger provides a compact and efficient solution to recuperate most of the sodium heat after the exit from the test section. For the SOLTEC-3 loop only a Na-air heat exchanger has been considered. Due to the expected temperature level, the Na-Na heat

recuperator will be manufactured from Inconel steel, while standard stainless steel AISI 316Ti will be used for the Na-air heat exchanger.

The safety concept is based on the experience gained in the design of other sodium loops [10, 11, 12] in KIT and relies on several constructive and operational measures. To ensure a safe and fast drainage of sodium in both cases, normal operation and emergency case, in less than 30 s, the sodium inventory in the loop is low (restricted for SOLTEC-1 and -2 to about 10 litres) and all relevant sodium valves have a normally open configuration. In the case of a leakage or sample rupture occurring in the test section, several measures were foreseen to automatically detect the situation and sodium will be immediately released in the storage tank. No water based systems are allowed in the loop and possible fire scenarios can occur only due to the contact of hot sodium to air, e.g. in case of cracks occurring in the pipelines. In this case, it is detected by the leak sensor and the drainage is initiated. Before leaving the thermal insulation a lot of sodium can be collected inside the outer hull. Therefore only a small amount of sodium can access the encapsulated atmosphere of the metallic housing of the facility. In the event of cracks occurring in the storage tank, sodium can drain in a collection tray integrated in the framework of the housing and the associated fire and smoke will be limited within the insulated metallic housing. As an operational safety measure, the loops will be operated at very low overpressure.

**Figure 3 a) 3D model of the SOLTEC-1 and -2 loops without test section**  
**b) 3D model of the SOLTEC-3 loop with integrated test section embedded in metallic box**



The 3D models of the loops are presented in Figure 3. For SOLTEC-1 and -2 the test section (not shown in the figure) shall be connected to the loop, while for SOLTEC-3 the test section is integrated in the loop. For visualization purposes the thermal insulation is not displayed. All control valves,

manometers and components to be operated manually have been gathered on the control panel, as can be seen in Figure 3b.

### *Operating modes of the loop*

The main operating modes of the facilities are filling, normal operation, drainage and emergency. The argon side of the loop will be used to drive the sodium from the 4 litre transport tank (NA-TT-01) into the 12 litre storage tank (NA-ST-01), see Figure 1. Prior to the filling of the loop with sodium, the facility will be evacuated using the vacuum pump (AR-PP-01), as schematically shown in Figures 1 and 2. The evacuation will be performed with the sodium side heated at about 150-200 °C, so that besides air the moisture rests can be also extracted. Once the loop has been evacuated, the sodium will be driven by pressurized argon in the loop, which is moderately heated at about 200 °C. As a difference to other sodium loops, the present facility does not contain a dedicated expansion tank. Such a tank should be placed in the highest location of the facility, i.e. in the high temperature side. Exactly in this zone no components are desired, in order to avoid the constructive complex measures associated to the connections that have to be welded in this high temperature side. Instead, the storage tank is used as an expansion tank in the following way: once the loop has been filled with sodium, it will be heated up to the operational temperature required. Since the connection between the loop and the storage tank will be kept open, the sodium will expand in the storage tank, where the pressure can be controlled by adjusting the argon pressure (green pipelines in Figure 1 and 2). Once the loop has reached its desired operating temperature, the valve situated at the interface between the loop and the storage tank (NA-V-04 in Figure 1 and NA-V-05 in Figure 2) can be closed. In this way the loop is constructively simplified and the operational safety is significantly increased. A small gas bubble trap is foreseen in the low temperature side.

The increase in the total volume due to thermal expansion can be estimated using the relation:

$$\Delta V = \gamma V_{initial} \Delta T \quad (1)$$

where  $\gamma$  [1/K] is the volumetric expansion coefficient,  $V$  [m<sup>3</sup>] the volume and  $\Delta T$  [K] the temperature gradient. For SOLTEC-1 and -2, the increase in the sodium volume from 293 K to 1000 K in the high temperature side is about 5 %, while for the sodium in the low temperature side, the total expansion from 273 K to 673 K is about 2.7 %.

For the normal operation state, the valves situated at the interface to the argon cover gas are closed, so that sodium can be circulated by the pump. Possible flow fluctuations induced by the pump can be damped by an appropriate adjustment of the argon pressure in the storage tank.

After the tests have been performed, the trace heating is reduced down to about 150 °C and the sodium is first drained in the storage tank by gravity. All pipelines have an inclination of a few degrees to allow the drainage of sodium under gravity. In the second part of this state, the sodium is driven in the storage tank by pressurized argon. For SOLTEC-3, a very small quantity of sodium will remain in the test section and will be neutralized when the test section will be opened for module replacement or maintenance.

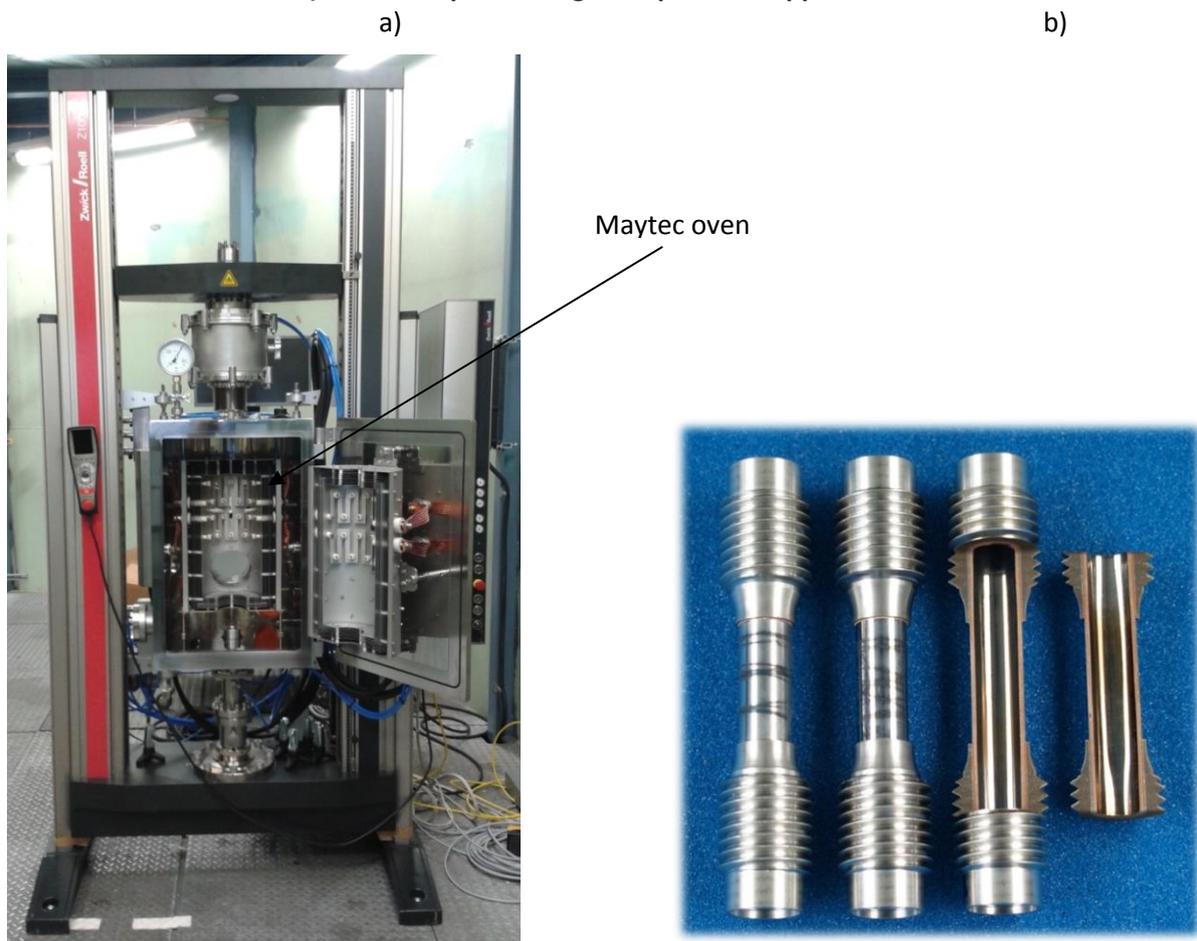
In all loops, the vacuum conditions set in the test chamber will be monitored. Therefore, when sample ruptures occur in the test sections, the pressure increase in the test chamber will be detected and the loop will be at once drained in the storage tank. Further, pressurized argon will be supplied in the test section to drive all sodium in the storage tank. Similarly, in case of a crack occurring in the

pipelines or in the components, the sodium will immediately be drained in the storage tank and the facility will be pressurized with argon.

### Test sections

The in situ low cycle fatigue investigation tests will be performed in a Zwick/Roell Z100 universal traction facility that has been installed in the rotunda building that houses the sodium loop KASOLA facility at KIT, as displayed in Figure 4a. In the traction facility traction and compressive forces up to 50 kN can be measured at a cycle time of max. 1000 Hz. A Maytec vacuum oven has been installed in the facility, where test samples can be heated up and tested at temperatures above 1000 °C in vacuum down to about  $10^{-5}$  mbar. Test samples up to about 10 cm in length and 6 cm in diameter can be tested. Among the materials planned to be investigated are conventional steels, such as AISI 316Ti, 1.4988 and 1.4970. Furthermore, new materials for high temperature applications, such as advanced PM2000, innovative W-Cu laminates and ceramic-metal joints for advanced thermoelectrical modules are planned for investigations.

**Figure 4 a) Zwick/Roell universal traction facility for low cycle fatigue tests (SOLTEC-1 loop);  
b) W-Cu samples for high temperature applications**

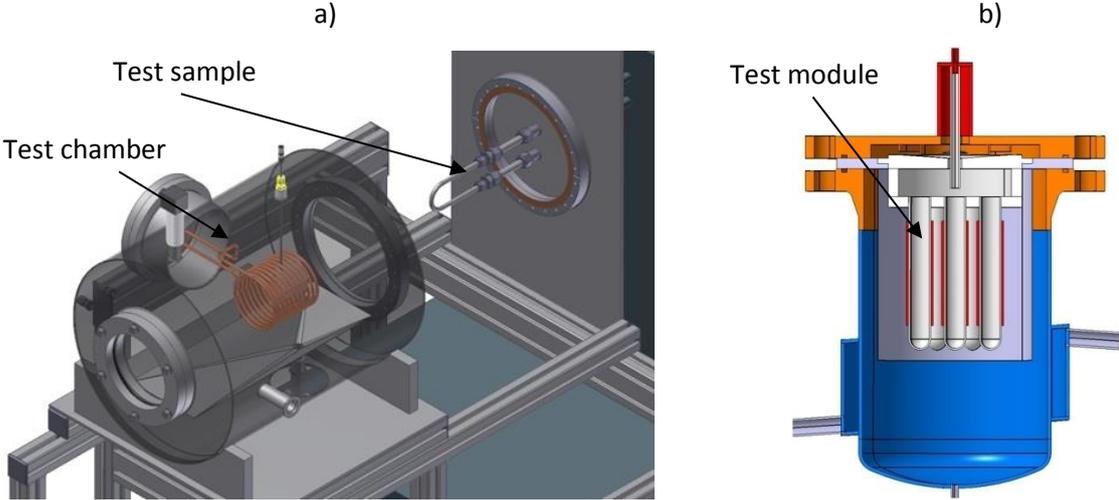


Prototypic samples of tungsten-copper laminates planned to be investigated in SOLTEC-1 in flowing hot sodium are displayed in Figure 4b. The aim is to employ them in high temperature receivers for CSP, as proposed in [13]. Tungsten has the highest melting temperature and is an excellent absorber material, therefore it is one of the best candidates for high temperature applications. However, tungsten has low fracture toughness at room temperature and high brittle-to-ductile transition temperature. Therefore, it cannot be used directly as structural material. As reported in [14], the tungsten-copper laminates have the potential to by-pass these drawbacks by cold working the tungsten, significantly lowering the temperature of the brittle-to-ductile transition below -120 °C. Hence, pipes made of tungsten laminates can extend the operational range of chrome steels and nickel-base steels, to meet the requirements of futures CSP plants.

Although some experimental data are available regarding corrosion of austenitic materials in hot sodium [15], there is a lack of experimental data regarding the effect of temperature excursions on structural materials at such high temperatures as planned in SOLTEC. While data are available for temperatures up to 550°C [16,17], for SOLTEC-2 the task is to generate reliable long term data based on a set of experiments in sodium at temperatures between 650-900 °C.

For the investigation of steel corrosion in sodium environment, the test section contains an U-type pipe made of different steels, which will be connected to the upper part of the SOLTEC-2 facility. The sample pipes will be introduced in the test chamber, as displayed in Figure 5, and additionally inductively heated. Among the materials planned for investigation are austenitic steels with variable chrome content, nickel-based steels, Inconel-based steels and tungsten-copper laminate pipes previously mentioned. The probes will be analysed by SEM (scanning electron microscope) method and the material surface will be characterized using a profilometer.

**Figure 5 a) High temperature test chamber for investigations of new steels in SOLTEC-2 loop  
b) Universal test section for investigations of different AMTEC modules in SOLTEC-3 loop**



Long term investigations of direct energy convertors AMTEC (Alkali-Metal Thermal-to-Electric Converter) are planned in SOLTEC-3. AMTEC convertors are thermoelectric devices that directly convert heat in the range 600-1000 °C into DC electricity at total efficiency of about 30 %. The technology relies on  $\beta''$ -alumina solid electrolyte (BASE) ceramics to allow the transport of Na ions, while blocking the flow of electrons. The key process is the ionization of sodium on the anode side of the BASE. The driving force of the sodium ions through the BASE is a thermodynamic potential

characterized by temperatures in the range 700-1000 °C and pressures of  $\sim 10^5$  Pa on the anode side and temperatures of 300-500 °C and pressures of about  $\sim 50$  Pa on the cathode side, where the sodium vapour formed is condensed and circulated back to the anode side, where the cycle can restart [18].

The experimental investigations and development of AMTEC cells has been restarted at KIT in the frame of the Helmholtz alliance on Liquid Metal TECHNOlogy (LIMTECH) and Helmholtz Energy Materials Characterization Platform (HEMCP). A dedicated test facility is intended for short term investigations [11,12]. The long term tests are planned in SOLTEC-3, where new AMTEC configurations will be also investigated. For this task, a universal test section (see Figure 5b) has been developed that can incorporate modules of different configuration with a limited number of parts to be replaced at every module test. Further, temperatures up to 1000 °C in the module can be sustained, due to the use of ceramic parts.

For SOLTEC-3, the sodium flow is divided in two branches after the pump, one side characterized by high temperature and low mass flow rate for the hot side of the AMTEC module and a second side having a lower temperature ranging between 250-450 °C and larger mass flow rate for the cooling of the AMTEC test chamber.

### Design of and choice of main components and instrumentation

Due to the high temperature level, Saas PMP 250 permanent magnet pumps that are able to sustain 450 °C have been acquired. The pumps have a 3 kW three-phase alternating current electric motor that drives two discs equipped with SmCo magnets. The flowing channel is placed between the two discs of the rotor. The magnets generate a rotating magnetic field perpendicular to the flowing channel, which induces an eddy current  $I$  and therefore a force on the fluid in the flowing channel. The maximal flow rate of the pump is 60 l/min and the maximal pressure allowed is 6 bar. The pumps can be regulated using a programmable logic controller (PLC) system or directly using a Danfoss FC 301 frequency converter.

Both Na-Na heat recuperator and the Na-air heat exchanger have a counter-current configuration for enhanced efficiency. The combined heat exchanger and its main geometric data are displayed in Figure 6b. The recuperator contains 35 inner Inconel pipes having dimensions  $\varnothing 16 \times 2.5$  mm<sup>2</sup>, while the Na-air contains 17 stainless steel pipes having dimensions  $\varnothing 20 \times 2$  mm<sup>2</sup>. For a temperature gradient of 260 °C the thermal output of the Na-Na heat recuperator at maximal flow rate of 300 kg/s is estimated as:

$$\dot{Q} = \dot{m} c_p \Delta T \sim 27 \text{ kW} \quad (2)$$

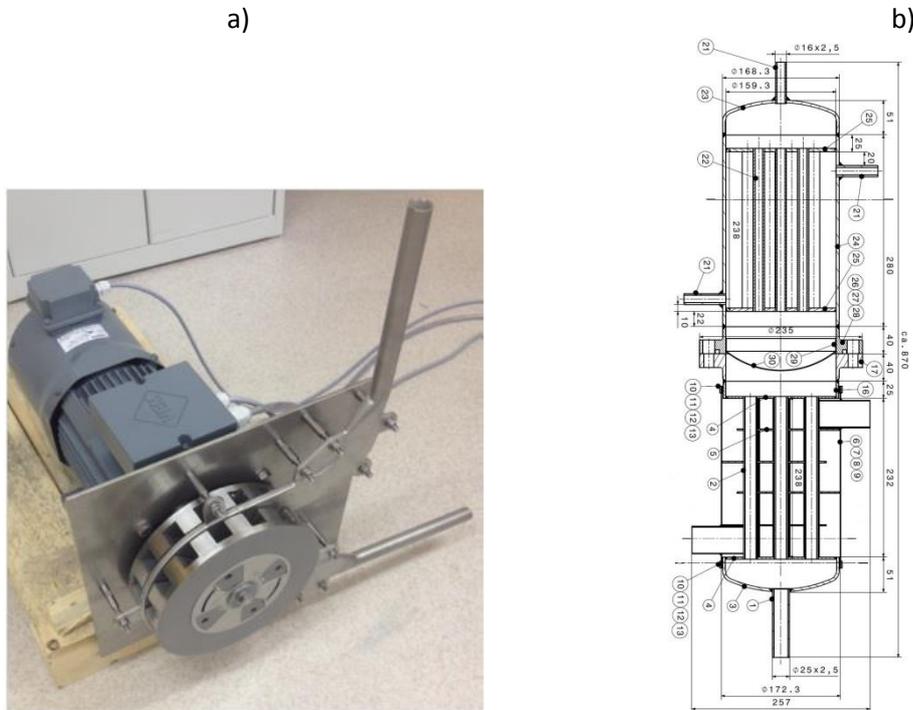
where  $\dot{Q}$  [W] is the thermal output,  $\dot{m}$  [kg/s] is the mass flow rate,  $c_p$  [J/kg K] is the specific heat capacity and  $\Delta T$  [K] the temperature gradient.

For the Na-air heat exchanger in SOLTEC-3, the same configuration as for the Na-air part from the combined heat exchanger has been kept in order to reduce the costs.

For the instrumentation of the loops, temperature, pressure, mass flow rate and oxygen level are to be measured. The instrumentation is based on the experience gained in the development of the KASOLA [19] and ATEFA loops [11,12]. The temperature is planned to be determined using temperature sensors placed on the outer side of the sodium pipeline. The pressure will be measured

locally using Kulite HEM-375M sensors. For the mass flow rate, flywheel electro-magnetical flowmeters are considered.

**Figure 6 a) Saas PMP-250 permanent magnet pump; b) Combined Na-Na-air heat exchanger**



The loops are presently in the final erection phase and will be assembled by the company Saas GmbH, Germany. The set-into-operation phase is planned for the beginning of 2017.

## Conclusions

The present paper discusses the design of three 1000 K sodium loops, currently in the final stage of erection at Karlsruhe Institute of Technology in Germany. The loops are planned to be used for investigations and qualification of innovative materials and steels in hot sodium environment for high temperature applications as well as long term tests of innovative thermoelectrical converters. Among the materials planned for investigations are W-Cu compounds, advanced PM2000, austenitic steels and nickel-based steels. The research is motivated by the need of material development for high temperature applications and by the lack of experimental data for in-situ experiments of creep fatigue in hot sodium.

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