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Development of high temperature liquid metal test facilities for qualification of materials and investigations of thermoelectrical modules

A Onea^{1,4}, W Hering¹, J Reiser², A Weisenburger³, N Diez de los Rios Ramos¹, M Lux¹, R Ziegler², S Baumgärtner² and R Stieglitz¹

¹Institute for Neutron Physics and Reactor Technology, Karlsruhe Institute of Technology (KIT), Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

²Institute for Applied Materials (KIT-IAM),

³Institute for Pulsed Power and Microwave Technology (KIT-IHM),

⁴Alexandru.Onea@kit.edu

Abstract. Three classes of experimental liquid metal facilities have been completed during the LIMTECH project aiming the qualification of materials, investigation of thermoelectrical modules, investigation of sodium transitional regimes and fundamental thermo-dynamical flows in concentrating solar power (CSP) relevant geometries. ATEFA facility is dedicated to basic science investigation focussed on the alkali metal thermal-to-electric converter (AMTEC) technology. Three SOLTEC facilities are aimed to be used in different laboratories for long term material investigation sodium environment up to a 1000 K temperature and for long term tests of AMTEC modules. The medium scale integral facility KASOLA is planned as the backbone for CSP development and demonstration.

1. Introduction

Liquid metals have very good thermodynamic properties (large thermal conductivity, large temperature range as liquids), which makes them efficient heat transfer fluids (HTF) for a wide range of heat transfer processes [1, 2]. They are considered to extend the application range of concentrating solar power to temperature up to 1000 °C, the temperature with the maximum efficiency of central tower systems. With that objective, the operation range exceeds single phase liquid metal application of sodium and two phase flows are to be considered. The same is true for the Alkali Metal Thermal-to-Electric Converter technology, which can operate in two modes focused on the fluid conditions on anode and cathode side: vapor/liquid and vapor/vapor. The AMTEC technology has been proposed as topping cycle for sodium operated CSP facilities [3].

AMTEC cells are thermoelectric devices that directly convert heat in the range 600-1000 °C into DC electricity at total efficiency of ~ 30 % [4, 5]. The technology relies on β'' -alumina solid electrolyte (BASE) ceramics to allow the transport of Na ions, while blocking the flow of electrons. Three issues have to be solved: 1. materials to operate at these high temperatures on a long time basis 2. electric isolations and feedthroughs 3. processes to combine different materials so that their noteworthy properties for high temperature applications can be used. Since the proof of concept has been achieved several years ago, the next step was initiated to profit from modern manufacturing developments and to build a new AMTEC prototype.



The present paper aims to give an overview of the liquid metal test facilities erected at KIT within the frame of the LIMTECH project. Financial support for the construction of the facilities has been also provided by the Helmholtz Energy Materials Characterization Platform (HEMCP).

2. New concentration solar power concept

Among all liquid metals, sodium has been identified as probably the most appropriate heat transfer fluid for CSP plants [1, 2]. Compared to other liquid metals and to conventional HTFs such as molten salts, sodium has several favourable properties that make it a very promising 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 ($98 - 883 \text{ }^\circ\text{C}$ 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 low, since sodium has a low density (834 kg m^{-3} at $500 \text{ }^\circ\text{C}$) and a viscosity in the range of water. 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 knowledge base in handling sodium as well as on 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 [3]. Depending on the chosen material for the pipelines of the receiver, the proposed plant 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 are compensated by a thermal energy storage device, which can be appropriately dimensioned to allow a continuous operation on a 24/7 basis. The basis loop of the plant operates below $\sim 550 \text{ }^\circ\text{C}$, therefore conventional steels such as AISI 316Ti can be employed. The peaks in the solar energy occurring in the range $600 - 900 \text{ }^\circ\text{C}$ are planned to be used by a cluster of thermoelectrical converters that can convert directly the heat into DC electricity.

3. Experimental capabilities established within Helmholtz AMTEC Center (HAC)

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

- Experimental investigation of fundamental thermo-dynamical aspects of sodium flows in relevant geometries (e.g. flows in a CSP receiver, flows in channels with sudden expansion).
- Generation of an experimental database for sodium flows to qualify and improve the anisotropic heat flux and momentum models in simulation codes.
- 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 (e.g. creep fatigue investigations).
- 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.
- Short-term and long-term investigations of thermoelectric devices to assess the performance behavior under different operating conditions, as well as on a long time basis.

For such a wide range of experimental investigations, several experimental facilities have been developed as summarized in table 1.

3.1. ATEFA facility

The AMTEC TEst FAcility (ATEFA) [6, 7] is intended for short-term tests of single AMTEC cells and targets the investigation of the cell performances, the coating materials and of the ceramic-metal brazing. Temperatures up to $1000 \text{ }^\circ\text{C}$ can be achieved locally in the ceramic at a maximal overpressure of 0.5 bar. Sodium is directed by pressurized argon from a tank into the test cell, where it is heated up

at the required test temperature. After diffusion through the ceramic, the sodium is condensed and collected in a second tank. Taking into consideration the low amount of sodium of only ~3 liters, the facility can operate for approx. 36 h until the tanks can be exchanged. The argon cover gas controls the system pressure and the sodium flow rate and is divided in a supply branch and a pressure branch. The first branch controls the sodium flow towards the cell, while the second one controls the pressure in the cell.

Table 1. Overview of the liquid metal experimental facilities erected at KIT.

Facility	Description and experimental tasks	Fluid/ Amount	Facility size	Operating conditions (Max. temperature, max. gauge pressure)
ATEFA	Small scale facility for short term AMTEC single cell tests	Na (~ 3 liter)	0.8 × 1.0 × 2.0 m ³	< 1000 °C, < ~0.5 bar
SOLTEC-1	Small scale facility for material qualification (low cycle fatigue; long term tests)	Na (~ 14 liter)	1.2 × 1.6 × 1.9 m ³	< 720 °C, < ~3.5 bar
SOLTEC-2	Small scale facility for investigation of steel corrosion in hot sodium (long time tests)	Na (~ 14 liter)	1.2 × 1.6 × 1.9 m ³	< 720 °C, < ~3.5 bar
SOLTEC-3	Small scale facility for long time tests of modular AMTEC cells	Na (~ 14 liter)	1.2 × 1.6 × 1.9 m ³	< 950 °C, < ~2.5 bar
KASOLA	Versatile large scale infrastructure	Na (7 tonnes)	Ø 7.6 m Height 12 m	< 550 °C, < ~3 bar

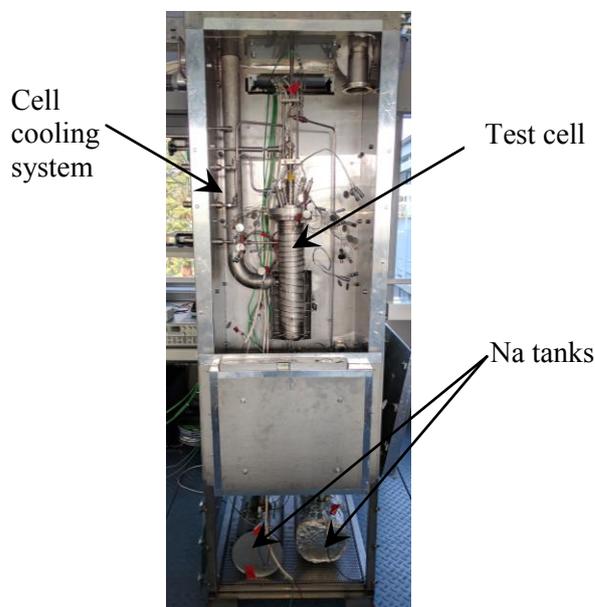


Figure 1. Frontal view of the ATEFA facility.

The sodium side consists of the sodium pipeline, the test cell, a sodium-air spiral heat exchanger and two sodium tanks. The entire sodium side is in-housed in a thermally insulated metallic frame separated from the argon system and the control system. Additional information about the test facility is reported in [6] and [7].

An overview and analysis of the candidate materials for the coating of the BASE (beta"-alumina solid electrolyte) ceramic is reported in [8]. Based on this analysis, the coatings performed up to now are molybdenum (Mo), titanium nitride (TiN) and titanium carbide (TiC) [6].

3.2. SOLTEC facilities

For the SOLTEC (SODium Loop to TEST materials and Corrosion) facilities it was decided that three independent facilities will be developed, each having its own test section. The SOLTEC-1 and -2 are similar facilities for material investigations having a maximal operating temperature of 720 °C at maximal 3.5 bar overpressure. SOLTEC-3 loop is planned for long term tests of thermoelectric converters to be operated at maximal 950 °C up to maximal 2.5 bar overpressure. 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 2 and 3.

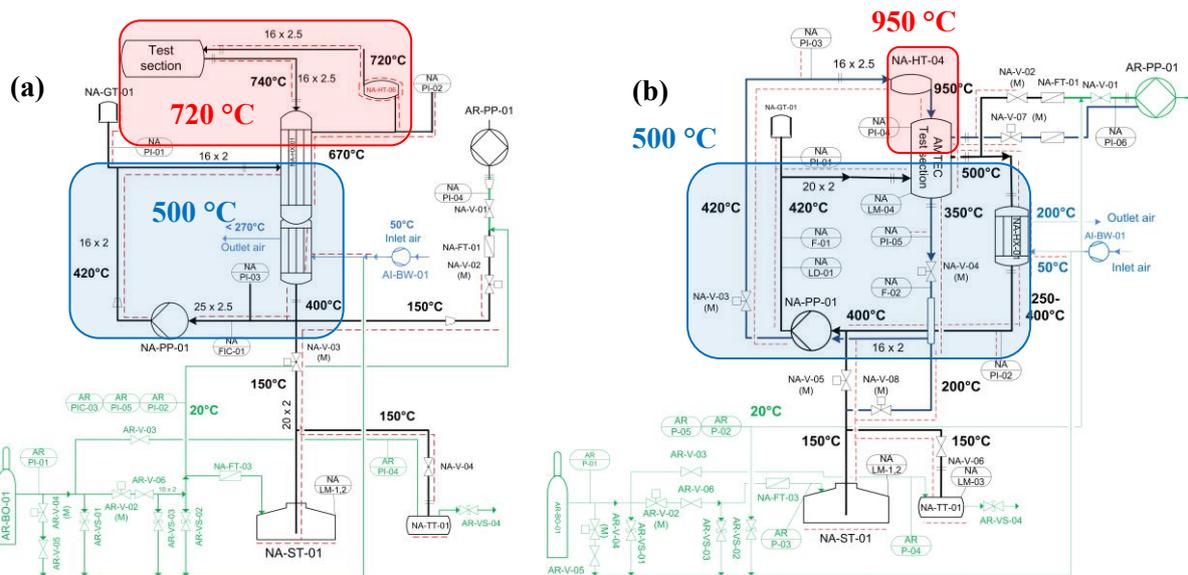


Figure 2. Piping and instrumentation diagrams of SOLTEC-1,-2 (a) and SOLTEC-3 (b).

For all facilities, a very compact construction is considered, as presented in table 1. For SOLTEC-1 and -2, the test sections are placed outside of the facility, while SOLTEC-3 integrates the test section within the framework. All three facilities have a high temperature side, where the test section is located. The main components of the loops are located in the low temperature side, where temperatures of up to ~500 °C can be reached, as highlighted in the piping and instrumentation diagrams in Figure 2. Due to the high temperature level nickel-based superalloys are used for the high temperature side, while for the low temperature side stainless steel AISI 316Ti is used. At the maximal sodium mass flow rate planned, i.e. 300 kg/h, 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 can be reached. For SOLTEC-1 and -2 the total pressure loss in the loop at maximal flow rate is estimated to be about 1.2 bar. All facilities have the same pump type, namely a 3 kW permanent magnet pump able to operate up to 450 °C. 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 to avoid their potential reaction with sodium. All major components (valves, pumps) have been placed in the low temperature side of the loop, where the thermal stresses are lower.

For SOLTEC-1 and -2 a combined 27 kW Na-Na heat recuperator and 7.5 kW Na-air heat exchanger has been placed at the interface between the high temperature side and the low temperature side. This combined heat exchanger provides a compact and efficient solution to recuperate most of the sodium heat after 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 is used for the Na-air heat exchanger.

The safety concept is based on the experience gained in the design of other sodium loops [9, 10] in KIT and relies on several constructive and operational measures. To ensure a safe and fast drainage of the sodium from the hot side in less than 30 s, the sodium inventory in the loop is low (restricted for SOLTEC-1 and -2 to about 12 liters) and all relevant sodium valves have a normally open configuration. In case of a leakage or test 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, the leak sensor placed on the pipelines will detect the sodium leaving the crack and initiate the drainage. Before leaving the thermal insulation most of the 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 bottom part of the framework of the housing. In this case, the associated fire and smoke will be limited within the insulated metallic housing. As an operational safety measure, the loops will be operated at low overpressure.

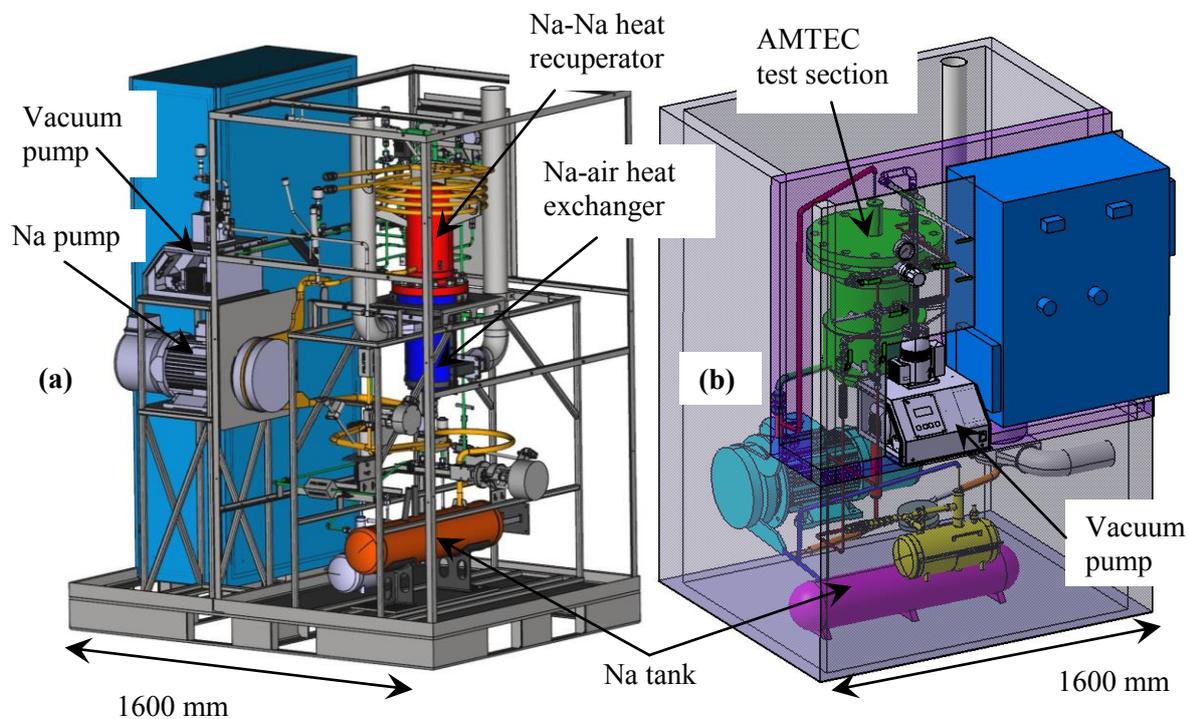


Figure 3. 3D-models of SOLTEC-1 and -2 (a) and SOLTEC-3 (b).

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 liter transport tank (NA-TT-01) into the 14 liter storage tank (NA-ST-01), see Figure 2. Prior to the filling of the loop with sodium, the facility will be evacuated using a vacuum pump (AR-PP-01). The evacuation will be performed with the sodium side heated at about 150-200 °C, so that besides air, 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. 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 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 2a and NA-V-05 in Figure 2b) can be closed. In this way the loop is constructively simplified and the operational safety is significantly increased. A small gas bubble trap is considered in the low temperature side.

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 4. In the traction facility traction and compressive forces up to 50 kN can be measured at a cycle frequency 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.

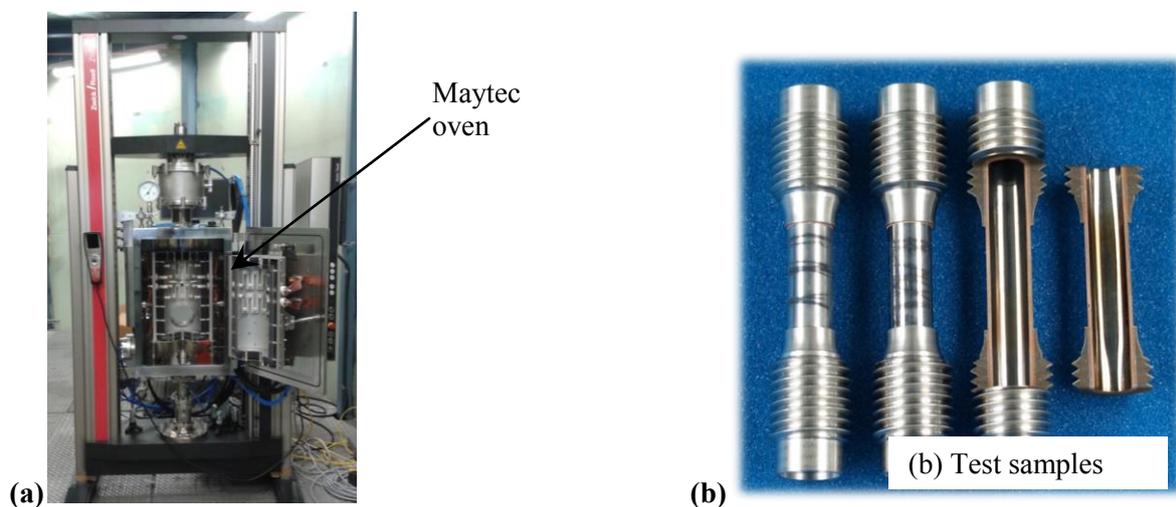


Figure 4. (a) Zwick/Roell universal traction facility for low cycle fatigue tests (SOLTEC-1 loop); (b) W-Cu samples for high temperature applications.

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 [11]. Tungsten has the highest melting temperature of all metals in pure form 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 [12], 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 ~ 125 °C. Hence, pipes made of tungsten laminates can extend the operational range of chrome steels and nickel-based superalloys, to meet the requirements of future CSP plants.

Although some experimental data are available regarding corrosion of austenitic materials in hot sodium [13], 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 [14, 15], for SOLTEC-2 one of the tasks is to generate reliable long term data based on a set of experiments in sodium at temperatures above

650 °C. The second task addresses thermal cycling of receiver tubes that in real operation are caused by day/night and cloud shadowing. For the investigations of steel corrosion in sodium environment at constant temperature and thermal cycling, the test section contains a U-type pipe made of materials to be investigated, which will be connected to the upper part of the SOLTEC-2 facility. During the experiments hot Na leaving the loop with up to 720 °C will flow through the tube sample and will be heated by an inductive heater. The inductive heater cooled by a thermal oil to minimize any risk is placed in a vacuum vessel (see Figure 5a). The oxygen and moisture content in the vacuum chamber will be permanently surveyed and if required adjusted. A pressure sensor controls any pressure increase, which can result from a leakage of the vacuum vessel or by a rupture of the U-type tube specimen, and gives signal for fast draining of the loop and the shutdown of all heaters.

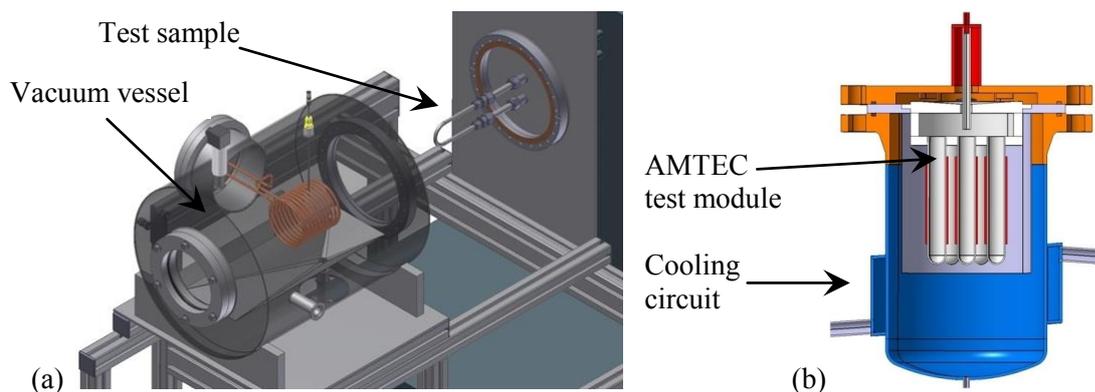


Figure 5. (a) High temperature test chamber for investigations of new steels in SOLTEC-2 loop
(b) Universal test section for investigations of different AMTEC modules in SOLTEC-3 loop.

Among the materials planned for investigation are austenitic steels with variable chrome content, nickel-based superalloys, Inconel-based superalloys and W-Cu laminate pipes previously mentioned. The probes will be analysed by scanning electron microscope (SEM) method and the material surface will be characterized using a profilometer.

Long term investigations of AMTEC modules in the range of weeks are planned in SOLTEC-3. The loop has a universal test section (see Figure 5b) that can incorporate modules of different configuration with a limited number of parts to be replaced at every module test. Temperatures up to 950 °C in the module can be sustained, due to the use of ceramic parts. For SOLTEC-3, the facility consists of two independent loops, the test loop being characterized by high temperature and low mass flow rate, while the cooling loop has a lower temperature ranging between 250 – 450 °C and larger mass flow rate. The cooling loop is used for the cooling of the AMTEC test chamber.

The design of the facility and the numerical calculations for the layout of the components and of the facilities have been performed by KIT. The loops are presently in the final stage of construction by the firms SAAS GmbH and Sowec GmbH & Co. KG, Germany. The first firm developed the instrumentation and control technology, while the second firm performed the mechanical and thermodynamical calculations of the heat exchangers, optimized the 3D-model and manufactured the main components. The set-into-operation phase is planned for the mid 2017.

3.3. Integral facility KASOLA

The KARlsruhe SODium LABORatory experimental research facility is a versatile sodium loop, able to embed test sections up to 6 m height. It contains a base loop with a purification system that incorporates a cold trap with integrated heat recuperator [16], the test ports for experiments and the 7m³ sodium storage tank situated in a neighboring dedicated building. Argon is used as cover gas to protect sodium from oxidation, to maintain the pressure balance in the loop and to facilitate the filling and drainage of the loop. The entire facility is located in a three floor steel containment, approximately

12 m in height. The maximum operational temperature foreseen is 550 °C. A 75 kW magneto hydrodynamic annular linear induction pump provides a maximum liquid sodium mass-flow rate of app. 150 m³/h within the base loop. Almost all main components of the base loop are gathered in the machine room, while the test section is located above the MHD pump.

The scientific program of the experiments foreseen in the KASOLA facility is reported in [17], while additional information about the facility can be found in [7]. The numerical investigations of the sodium flows in KASOLA performed with TRACE are reported in [18-20].

To assess the applicability of a sodium operated thermal energy storage (TES) tank, a prototype sodium TES is foreseen to be investigated in KASOLA. The thermocline tank considered has a total volume of about 500 liters and a thermal energy storage capacity of 50 kWh. It contains a floatable plate that separates sodium in cold and hot regions, avoiding therefore heat losses by conduction and convection. The proposed design can be appropriately dimensioned to meet the requirements set in [3], in order to allow overnight plant operation and cooling capabilities for the AMTEC cluster.

4. Summary

The present paper gives an overview of the experimental facilities erected at KIT in the frame of the Helmholtz Alliance on Liquid Metal Technology LIMTECH. The construction of the facilities was financially supported by the Helmholtz Energy Materials Characterization Platform.

The ATEFA facility is used for basic short-term investigations of thermoelectrical converters. The SOLTEC loops are planned to be used for long term investigations and qualification of innovative materials and steels in hot sodium environment for high temperature applications as well tests of innovative thermoelectrical converters. Among the materials planned for investigations are W-Cu compounds, advanced PM2000, austenitic steels and nickel-based superalloys. 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. The versatile medium size KASOLA facility will integrate prototypical components for future CSP plants to test a scaled CSP demonstrator.

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