LESSONS LEARNED FROM OPERATION OF A FORCED CONVECTION CHLORIDE MOLTEN SALT LOOP

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Abstract

We have designed, built, and operated a molten salt loop for corrosion testing constructed by SS316. The experiments are conducted using Magnak ($MgCl_2$-NaCl-KCl) salt at 650°C with a flow rate at 0.45 l/s (1.35 m/s). This paper will share the experiences and lessons learned from three tests lasting between 50 to 100 hours, including design modifications and operational procedures. The loop consists of several components, including tanks, a pump system, a sample insertion column, and measurement systems. The presentation will detail the loop's design and operational procedures, including gas systems, heating and isolation systems, temperature and flow measurement systems, seal technologies, and molten salt valves. It will also discuss experiences such as salt loading, purification, transportation, circulation, and drainage. We highlight lessons learned during salt circulation, including issues with chlorine-based gas hazards and vapor, which can inform future designs of high-temperature chloride FCLs.

Introduction

The Molten Salt Reactor (MSR) shows promise as a next-gen nuclear reactors due to molten salt's exceptional properties: high boiling point, low vapor pressure, high heat capacity, and high thermal conductivity. The existing corrosion databases rely on static capsule or crucible tests, where corrosion reaches equilibrium, making them inadequate for explaining corrosion in dynamic molten salt environments. Conducting corrosion tests under flowing molten salt is important, as it simulates real- or near-real-world conditions in which structures are exposed to molten salt. The significance of this type of testing was initially demonstrated by the Oak Ridge National Laboratory (ORNL), during the Aircraft Reactor Experiment (ARE) and Molten Salt Reactor Experiment (MSRE) programs[1,2].

In recent years, a few MSLs have been constructed and operated by many research groups. However, from the limited MSL publications available, nature convection loops (NCL) studies dominate due to their simple structure and ease of operation. Forced convection loops (FCL) investigations are scarce due to the significant expenses associated with building and operating these loops, as well as compatibility challenges with critical components such as valves, flanges, pumps, and sealants. An FCL was operated in Japan since 1998 to investigate molten salt heat transfer using LiF-BeF_2 (Flibe) as a coolant. However, instead of using Flibe, a surrogate salt (Hitec salt (KNO_3: $NaNO_2$: $NaNO_3$, 53-40-7 mol%)) was used. This research yielded insights into Hitec-related phenomena, including melting behavior, invasion into gas lines, residuals at welding joints, and non-uniform pipe heating[3,4,5]. Sabharwall et al.[6] presented a comprehensive design for FCL experiments with LiF-NaF-KF (FLiNaK) and $MgCl_2$-KCl salts, offering four molten salt flow measurement methods. Sohal et al.[7] proposed a conceptual design for FCL heat transfer testing. Yoder Jr. et al.[8] reported on the construction of an FCL at the ORNL. The loop is capable of circulating FLiNaK salt and operating at temperatures up to 700°C. Arora et al.[9] (2021) conducted a study focused on improving flow and temperature measurements in a 600°C FCL. Their research, funded to enhance thermal-hydraulic parameter measurement in FLiNaK, utilized the FLEXIM ultrasonic flowmeter, NaK-filled pressure sensor to validate flow rate measurements across a wide range. Head et al.[10] published their experience testing an FCL, similar in construction to the FCL developed by Arora et al.[9], but using a eutectic mixture of Hitec molten salt at a temperature of 200°C.

It is worth noting that the majority of MSL tests conducted to date have focused on fluoride salt. Despite several FCLs having been tested or operated in the past 25 years, none have been able to match the achievements of ORNL in the 1970s, with significantly lower operation temperatures and durations. This article aims to share our operational experiences and lessons learned from our three tests, lasting 50, 80, and 100 hours, on an FCL utilizing Magnak salt ($MgCl_2$-KCl-NaCl) at 650°C.

Molten Salt Loop System

A forced convection molten salt loop was designed and constructed in Nuclear Material and Fuel Cycle Center at Virginia Tech. The loop consists of an SS316 auxiliary (Aux.) tank, an SS316 storage tank, a centrifugal sump-type pump system, an SS316 loop, a vertical sample insertion column, an SS316 drain line, a cover gas and pressurizing system, heater zones on tanks, trace heating system for all pipes, and temperature and flow measurement system. The 3D drawing and real images are given in FIG.1. The loop specifications are listed in TABLE *1*. At a temperature of 650°C, approximately 40 kg of Magnak salt flowed through an 8-meter-long pipe system at a velocity of 1.35 m/s.

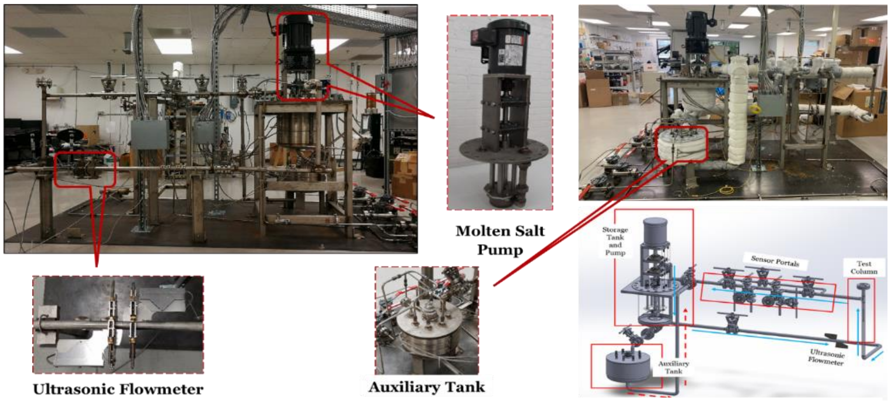

FIG. 1. 3D drawings and real images of initial design of the loop.

TABLE 1 MOLTEN SALT LOOP SPECIFICATION.

Capability	Parameters	Capability	Parameters
Flow rate	0.45 L/s	Salt volume	30 L (8 gallons)
Testing temperature	650 °C	Main piping	1-inch sch. 160
Main heater power	15 kW_{th}	Pump	3 HP 0-60 Hz
Trace1 heater power	10.8 kW_{th}	Loop length	8 m
Trace 2 heater power	3.6 kW_{th}		

Molten Salt Loop Operation experience and Lessons

Salt loading: The Magnak salts are acquired individually in powder form and are separately weighed and pre-mixed in a bucket. The mixture is then loaded into the Aux. tank via a 2" diameter port, using a funnel. The weighing and loading of the salt are carried out in the ambient environment while wearing appropriate personal protective equipment. Once loaded, the port is sealed with a threaded lid. Since the salts used are non-toxic and non-hazardous, we did not encounter any issues in this step. A total of 40 kg of salt is purified in two separate batches of 25 kg and 15 kg, respectively, filling the Aux. tank to 50-75% capacity with powder salt.

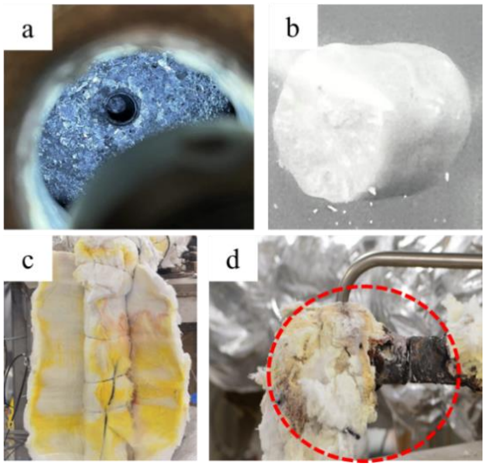

FIG. 2 a) Auxiliary tank after first batch of salt transportation. b) Magnak salt after purification. c) Yellow products on glass fiber isolation. d) loop failure.

Salt purification and solid salt pre-melt: The initial step in our process is to purify the salt received from the vendor, which is done in the Aux. tank. The purification process involves thermal purification with argon flow. To begin, the tank is evacuated and refilled with argon gas five times to replace the air from the open-air salt loading process. Then the salt was heated up at a rate of 1 °C/min combining 5-10 SCFH argon flow purging at 1 psi. After the salt has melted, it is kept at a temperature of 500 ºC and bubbled with argon flow at a constant rate for one hour. Once this process is completed, the exhaust line is turned off and the salt is heated to a temperature of 600 ºC for transportation. The powdery $MgCl_2$ has fine particles and a fluffy texture, making it prone to becoming airborne. In the purification process, the tank cover gas is kept at around 2 to 3 inches thick. Purging flow, carrying mainly $MgCl_2$, leads to some powder settling at the far end of the exhaust line. To prevent this, consider using a taller tank or implementing vacuum heating before melting. Purification was a one-time operation, with subsequent use of solidified salt. The solidified salt had low thermal conductivity, posing a risk of tank overheating with automated set point control. Future designs should consider this for large molten salt batches. The salt picture after purification is shown in FIG. 2 (b). The melting point of the salt by DSC is 394.67 ºC, which is slightly deviated from the theoretical melting point of 385 ºC. The ICP-MS analysis shows that the salt composition closely matches the target, with the major elements in mole percentage being $MgCl_2$-KCl-NaCl, 45-26.6-28.3. Concentrations of corrosion products after purification were also measured, including 2.6×10^{-4} of Cr, 3.27×10^{-4} of Mn, 1.48×10^{-2} of Fe, and 6.49×10^{-4} of Mo, all in mole percentage.

Salt transportation: This step involves transferring pre-melted salt from the Aux. tank to the upper storage tank, spanning a 3.5 ft (1.12 m) height difference. To accomplish this, two pre-steps are followed. First, the storage tank gas are exchanged from air to argon atmosphere. Second, the storage tank and pipes are heated to 550 ºC. Molten salt is transported between tanks using pressure, with pressure needed to reach a certain height estimated via the hydraulic head equation, $H = pgh$. The approximate pressure requirement for this process is 2.6 psi (18 kPa). The pressure difference results in the molten salt being pressed from the Aux. tank to the storage tank. The process becomes complicated when the Aux. tank inlet flow rate increases rapidly, reaching the upper limit of the flowmeter. FIG.2 (a) illustrate the images of the Aux. tank subsequent to the transportation of salt into the storage tank. The salt transfer process was highly effective, as evidenced by the presence of black crystals, which indicate MgO deposition, and minimal salt residue.

Salt circulation: The first step involves pre-heating the loop body above 300 °C. During the pre-heating process, it was observed that the temperature distribution was highly non-uniform, as shown in FIG. 3 from A to D. Then activating the pump with a frequency of 35 Hz. The temperature and flow rate on the loops must be monitored closely. Once all the TCs show uniform readings, at point D, the start-up is considered a success. From D to E refers to the heating up process to the target temperature. After point E, the flowmeter reading was given as 0.45 l/s. As expected, leaks were encountered throughout the operation. 1) The first issue that arose was related to the flange connections. It was addressed by replaced with welded connections. 2) The persistent issue of ball valve leaks has proven to be a significant challenge, requiring considerable effort before a solution was finally found during the final test. The persistent issue of valve leaks has led to damage to the heaters in the past. The potential danger of this problem was underscored by two severe accidents caused by a short circuit, which led to the electrolysis of chloride salt. Following several hours of loop operation, a noxious odor was detected, and subsequently, heat failure was observed. The odor was extremely potent and caused significant irritation. Fortuitously, both incidents occurred over the weekends when the building was unoccupied. Upon recognizing that the odor could not be mitigated, and that additional heaters and temperature controllers had failed, we promptly shut down the loop. We also observed the presence of a green coating on the equipment adjacent to the loop, likely indicating corrosion from chlorine gas. Upon disassembling the leak section, we observed a significant accumulation of yellow substance on the white insulation, FIG. 2 (c), likely a gas byproduct. Interestingly, a complete failure of one welding joint was also detected, FIG. 2 (d), with the pipe separating into two sections and salt leaking out and solidified around the failure. Notably, the flow meter did not detect any flow rate change, demonstrating the potential benefits of using molten salt to address coolant loss incidents in boiling water reactors.

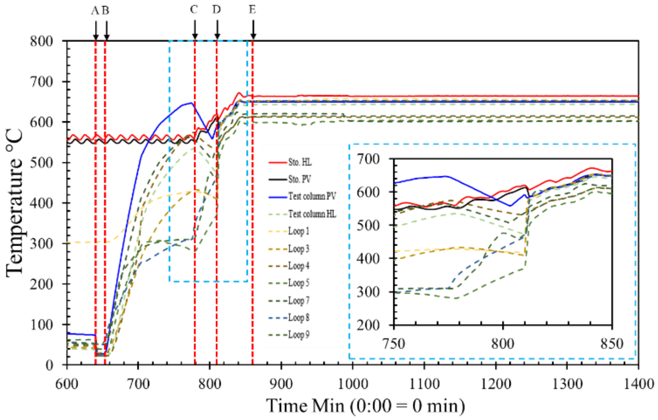

FIG. 3 Temperature profile of the loop body and storage tank on the day of salt circulation. Red line: storage tank process temperature. Black line: storage tank high-limit temperature. Blue line: test column temperature. Other color dashed lines: loop body temperature.

Salt drainage: The process of salt drainage occurs after each salt circulation, or in case of incidents requiring the loop to be shut down. This is facilitated by the use of an Aux. tank, which provides a reliable seal. Before turning on drain valve, the Aux. vent line is opened. This allows the salt to be driven by the pressure of the cover gas and gravity, draining from the storage tank to the Aux. tank. After the salt has been drained, the system to naturally cool to room temperature. It is important to note that the Aux. tank gas system should be kept on avoiding the formation of negative pressure in the tank due to the shrinking gas volume as the temperature decreases.

Conclusion

In conclusion, the development and use of a forced convection chloride molten salt test loop has deepened our understanding of material compatibility in molten chloride settings. We've confirmed the benefits of molten salt as a coolant, notably its ability to reduce coolant leaks due to its self-sealing properties. However, it is important to be cautious about the potential electrolysis of the chloride salt, as the current heating source for the test loop is electricity, which may generate hazardous gases. A major challenge is creating effective seals for connections and valves in high-temperature applications. The importance of reliable seals under these conditions cannot be overstated. This research offers valuable insights into material compatibility in molten chloride environments, setting the stage for future advancements.

Acknowledgements

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