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Thermal Hydraulic Static Operation of a Chloride Molten Salt Shut-Off Valve

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Abstract. The Sandia National Laboratories (SNL) National Solar Thermal Test Facility (NSTTF) conducted efficacy testing on a shut-off isolation valve for use with molten ternary chloride salt. A ball valve was tested under controlled N_2 ullage gas pressure and connected with flanged fittings that featured a spiral-wound gasket. The valve assembly consisted of boronized nickel coated SS316 components, with design features that greatly reduce the cost of overall valve assembly. Testing results showed that the valve did not leak, and post-test analysis demonstrated that the ball, seat, packing, and body all survived both the heat loads and the relative corrosive environment. Spiral-wound gaskets for flanged connections used in the system also functioned nominally, with no leaks or signs of failures during post-test analysis. However, testing was ultimately forced to rapidly stop after testing between 500-530°C as the actuator used on the valve failed in the heat, preventing the valve from sealing in the closed position. In addition, salt plugs and salt vapor plating also prevented the test from continuing.

INTRODUCTION

In the development of a Gen 3, 1 MW_{th} pilot-scale system for the U.S. Department of Energy (DOE) Gen 3 Liquid-Pathway project, to increase concentrated solar power (CSP) operating temperatures above 700 °C, ternary chloride salt (20%NaCl/40%MgCl2/40%KCl by mol wt. %) has been researched extensively as a potential heat transfer fluid [1],[2]. A primary concern of molten chloride salt flow systems is the prohibitive cost of designing isolation ball valves, as these valves would require expensive materials that are challenging to forge. High-nickel content alloys are commonly considered the best candidates to construct valves for high-temperature and corrosive halide salt applications [3]. Typically considered candidates are chosen in terms of strength and corrosion resistance, with top contenders including Haynes 230 (H230) and Inconel 625 [4]. Unfortunately, these alloys are far more expensive than conventional materials such as stainless steel (SS316). To reduce the cost of isolation valves for ternary chloride salt use, a boride layer for corrosion protection is being considered for commercial production, which is used as a coating over stainless steel materials with a cost less than nickel alloys. This boride layer has shown good corrosion resistance to ternary chloride salts [5]. A particular formulation and boronizing manufacturing process has been used to design boronized SS316 valve components with the potential to operate beyond 700ºC. This valve design significantly reduces the cost per isolation valve by an estimated $1/5^{th}$. This valve design also utilizes flanged connections with multi-material spiral wound gaskets. The gaskets were constructed of graphite layers on the salt-side and thermiculite layers on the air-side to meet both high-temperature and corrosion resistance demands and preventing degradation previously found with single material designs. The ability to use flanged fittings instead of welded connections significantly reduces the cost of valve installation. To test the efficacy of this design, a prototype 1.25 nominal pipe size (NPS) valve was fabricated and sent to the NSTTF for thermal-mechanical characterization with

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particular emphasis on evaluation of leak mitigation properties. The valve was installed into a small molten chloride salt piping system to test the functionality of both the valve and the multi-material gaskets at temperatures between 500°C and 550°C.

VALVE EFFICACY TESTING SETUP

The prototype boronized-nickel ball valve was placed in a piping system made of Inconel 625. The valve was oriented vertically in the piping system with segments of Inconel 625 above and below the valve. The segments of Inconel 625 pipe were schedule 40 and 1.25 NPS. The piping segments were attached to the valve using Inconel 625 raised face class 300 ANSI flanges. This piping system was assembled with the multi-material spiral wound gaskets placed at the interface between the flanged connections. The ends of the piping segments were sealed with welded ¼ inch Inconel 625 plates. The FIGURE 1 setup shows the valve in between the piping segments, supported in position by a steel frame.

FIGURE 1. Prototype Valve Mounted in Piping

Ternary chloride molten salt was first purified and inventoried into a vessel below the valve test assembly. Once purified, ternary chloride salt cannot be exposed to air. Exposure would result in the formation of oxides that can alter corrosion and material compatibility, and impurities raise the melting the temperature of the salt [5],[6]. To allow salt flow into the valve while maintaining salt purity, a test process flow design, Fig. 2, was created which consisted of a nitrogen ullage gas system, a vacuum pump, an air compressor, a salt containment vessel, the valve and attached piping, heaters, and controllers.

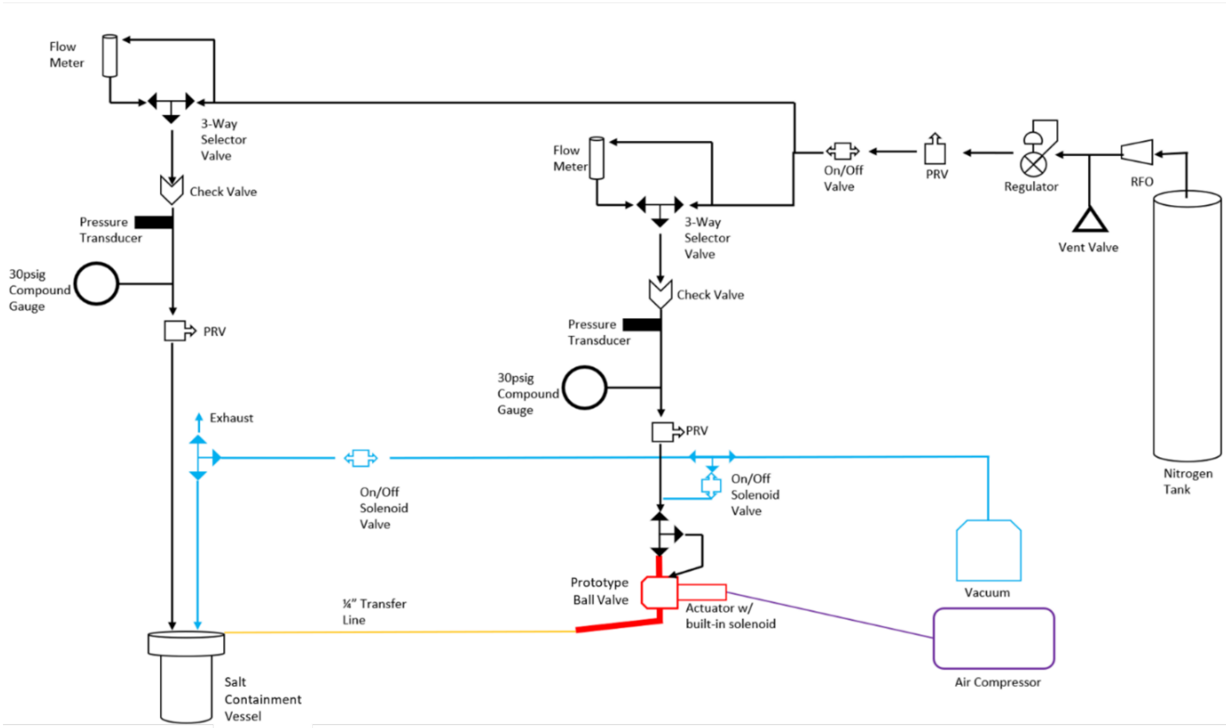

FIGURE 2. Test System Process Flow Design

The containment vessel, which contained approximately 1 gallon of salt, was comprised of a 6 NPS schedule 10 stainless steel 316 pipe, sealed with a welded end plate and a class 150 ANSI flange. The vessel had attached port connections for vacuum lines, nitrogen lines, and a salt transfer line. The vessel was filled with purified ternary chloride salt at the National Renewable Energy Laboratory (NREL) under a glove box and filled with argon cover gas. It was then shipped to SNL where the containment vessel was placed in a ceramic fiber heater and connected to nitrogen cover gas supply.

FIGURE 3. Containment Vessel SolidWorks Model

To move the salt from the containment vessel to the valve without exposure to air, a salt transfer line was connected between the vessel and the lower piping segment attached to the valve. An exhaust piping system was also added to the top of the valve and piping. To move salt on demand between the containment vessel and the valve, pressurization of the containment vessel was facilitated with nitrogen while leaving the exhaust valve open. This resulted in a pressure driven transfer of salt into the valve and piping. Before transferring, a vacuum pump was used to facilitate a cycle purge of any contaminant air from the system. A ceramic fiber-heated furnace was used to heat the salt containment vessel, all other wetted components were wrapped in heat trace. All heated test components were subsequently wrapped in insulation. Fig. 4 shows the completed test setup of the valve test system.

FIGURE 4. Photograph of the completed test setup

To cycle the valve safely and remotely for functionality and leak testing at varying operational temperatures, the valve was designed to open and close with a pneumatic actuator. The yellow actuator can be seen attached to the side of the valve in Fig. 4. To deliver air to the pneumatic actuator a compressed air system was added, supplied by an air compressor, and controlled with a solenoid valve. Various equipment was used to monitor test conditions including pressure transducers, flow meters, and thermocouples. A scale was placed under the containment vessel to monitor changes in mass while moving salt between the vessel and valve. A data acquisition (DAQ) system was built to monitor all sensors and allow for remote actuation of the solenoid valve. All heaters were connected to limit controllers which allowed set point programing, thermal hold times, and high limit cut-off temperatures.

EFFICACY TESTING RESULTS

The planned procedure included valve filling along the vertical stand pipe, and observation of the valves thermal-mechanical performance while actuating at incremental temperatures between 500°C and 550°C. Prior to testing, all components of the system were heated to 100°C to bake out moisture. At each respective operational temperature, the system was cycle purged with vacuum and ultra-high purity nitrogen. Heat controllers were then set to bring all components to 500°C. To avoid thermally shocking the valve or flanged fittings, no testing could be conducted until the salt and all test components were at similar temperatures. The ceramic fiber heater rapidly heated the salt to 500°C within the vessel. To help support the heating of the large amount of thermal mass within the valve assembly, a second layer of heat trace on top of the first layer of insulation was added, followed by a second layer of insulation to act as a thermal brake and mitigate heat losses. The addition of the thermal brake solved the heating issue, though heating of the valve assembly took 9 hours. When the valve reached 480°C, it was determined that testing could proceed without thermally shocking the valve. To initialize the transfer, the valve was first actuated into the open position to facilitate flow through the valve. An exhaust valve above the Inconel 625 piping was opened to allow for displacement of gas as the system filled with salt. The salt containment vessel was then pressurized to 7psig with nitrogen, immediately resulting in a flow of salt from the vessel into the valve. The valve and attached piping systems completely filled with salt, as indicated by thermocouples and a decrease on the scale below the containment vessel. Almost 10lbs of salt was transferred into the valve and attached piping. With salt in the valve, a 24volt signal was delivered to the solenoid valve, triggering pneumatic actuation. The valve was cycled between open and closed position 10 times. Fig. 5 shows the temperatures at the time of testing.

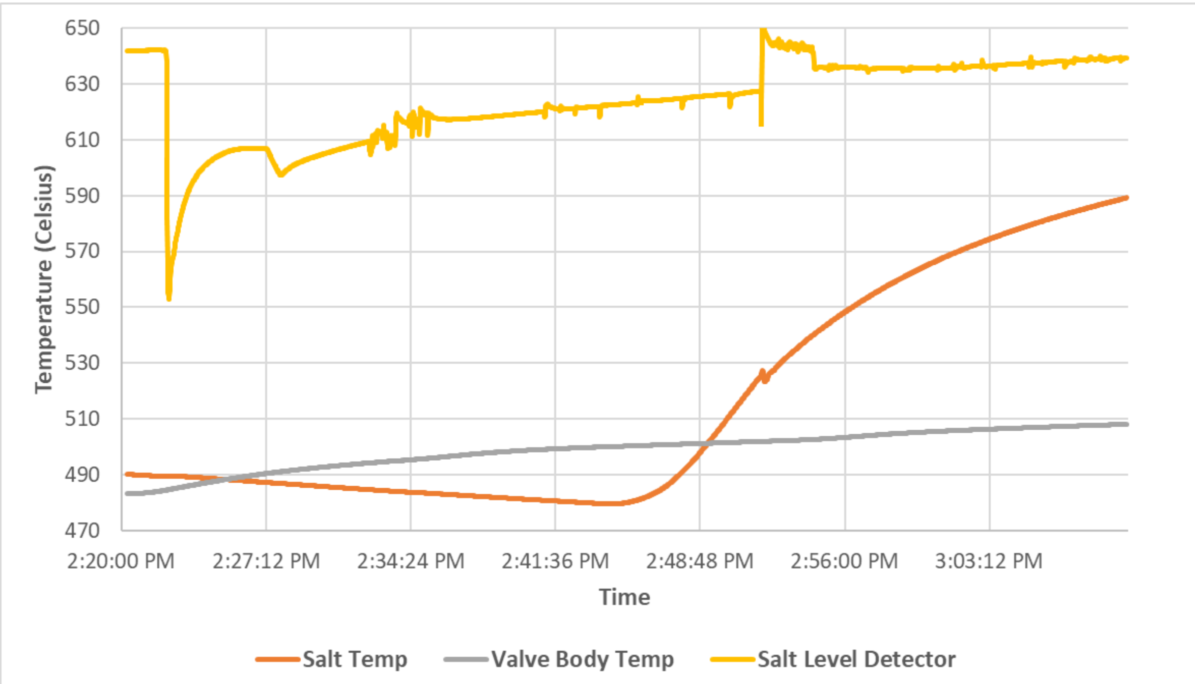

FIGURE 5. Graph of Test Temperatures and Pressures

The Fig. 5 graph shows three temperatures as recorded by thermocouples: the salt temperature, the external valve temperature, and the temperature of a thermocouple which was used as a salt level detector. As can be seen in the figure, at 2:20pm when the transfer was initiated the valve temperature was near 480°C, the salt temperature was at approximately 490°C, and the level detector thermocouple was at approximately 640°C. The level detector was at high temperature due to close proximity to a heat trace line. The proximity of this thermocouple to the heat trace line did make its temperature measurements inaccurate, however this thermocouple was only used for indicating that salt had reached the level needed for testing. The thermocouple was placed at the desired salt level to record the temperature drop that would coincide with 500°C salt contacting the thermocouple in the 640°C vapor space above the valve. The thermocouple did this successfully. At 2:22pm, the salt reached the desired level as indicated by a sharp drop in temperature at the level detector thermocouple. By 2:27pm the external valve temperature had increased passed 490°C. Subsequently, the valve was cycled open and closed. The valve successfully actuated while containing salt without leakage.

After 10 cycles, the valve was left in the closed position to hold the salt level in the vertical standpipe segment assembly. Set point temperatures on the heater controllers were increased for preceding elevated temperature testing. However, it was noticed that salt was draining back into the containment vessel. Within 30min., all 10lbs of salt had drained back. Some drain back was to be expected, but the valve had been left in the closed position so all salt on the outlet side of the valve should have remained in the piping system. This indicated that the valve had failed to close completely and was allowing salt to leak through. Subsequently, the test was shut down.

After salt exposure the valve system was disassembled, cleaned with water, and the valve was analyzed and subjected to the required valve inspections and tests as described in the API 598 standard [7]. As part of the API 598 standards, the shell must be tested to 1.5X the operating pressure. To meet this standard, a 720psi shell test was conducted on the prototype valve which showed no leakage, indicating no failures of the packing or body/bonnet seals. A low-pressure seat test was also conducted as required by the API 598 standards, which also yielded no leakage, indicating that the ball and seat were not warped or damaged by the heat or the ternary chloride salt. Qualitatively, the valve showed no signs of damage. In addition, the flanged fittings and spiral wound gaskets did not appear to have warped or leaked after exposure to the salt and elevated temperatures. At room temperature the valve was still exhibiting signs of jerkiness upon actuation. A seal test was conducted on the actuator which showed leakage past the seals. The seals had been damaged and the actuator could not produce enough torque to effectively cycle the valve.

CONCLUSIONS

Between the operational test temperatures of 500-530°C, the boronized valve cost reduction was a success. All methods of valve cost reduction employed in this valve design performed well and showed no signs of failure. Most importantly, the boronized SS316 shell was not damaged by the heat or by exposure to chloride salt. The 720psi shell test clearly indicated no change in the shell, packing, or body seal performance before and after exposure to the chloride salt. In addition, low-pressure testing showed that the ball and seat, which saw heavy salt exposure while under mechanical stress, were not altered or damaged by the chloride salt in any way. At 500°C, the valve performed exceptionally. This initial test indicates that the use of boronized stainless steel ball valves could be a viable option for isolation valves in ternary chloride salt flow loops. In addition, the test indicates that costs can be further reduced with the use of flanged fittings and spiral wound gaskets. The raised faced flanges and spiral would gaskets did not leak or show any signs of damage after exposure to the salt. The use of compressed graphite on the interior of the gasket provided good resistance to the chloride salt. The overall multi-material spiral-wound gaskets composition of graphite and thermiculite seems to have worked well at holding both the salt and nitrogen pressures up to 15psig. Though the valve was only exposed to the salt for a single day of testing, no signs of damage or corrosion were found in the boride layer upon analysis, confirming the expected performance as was predicted by previous corrosion studies on boronized materials [5]. Longer duration testing is desired to further study if chloride salt will corrode the boronized stainless steel surface. Ultimately, the only failure in the system was on the actuator. The actuator sourced was rated to operate at high temperature. Yet in an unexpected result, upon filling the valve with salt, heat transferred through the internals of the actuator to seals, raising actuator temperatures above 300°C. This resulted in the failure of some internal seals in the actuator. A solution to this problem has already been developed in which higher temperature seals will be used along with an extended bracket with heat sinks on the coupler to mitigate the high heat transfer into the actuator. These changes will allow for continued testing at higher temperatures to confirm the results of this test at 500°C and to confirm the efficacy of the valve at higher temperature. Further testing at higher-temperatures (>700°C) and for extended heat soak durations is planned for confident reliability.

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