ANL-GenIV-163

Corrosion Performance of Advanced Structural Materials In Sodium

Nuclear Engineering Division

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Corrosion Performance of Advanced Structural Materials in Sodium

K. Natesan, Y. Momozaki, Meimei Li, and D. L. Rink
Nuclear Engineering Division
Argonne National Laboratory

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ABSTRACT

This report gives a description of the activities in design, fabrication, construction, and assembling of a pumped sodium loop for the sodium compatibility studies on advanced structural materials. The work is the Argonne National Laboratory (ANL) portion of the effort on the work project entitled, "Sodium Compatibility of Advanced Fast Reactor Materials," and is a part of Advanced Materials Development within the Reactor Campaign. The objective of this project is to develop information on sodium corrosion compatibility of advanced materials being considered for sodium reactor applications. This report gives the status of the sodium pumped loop at Argonne National Laboratory, the specimen details, and the technical approach to evaluate the sodium compatibility of advanced structural alloys. This report is a deliverable from ANL in FY2010 (M2GAN10SF050302) under the work package G-AN10SF0503 "Sodium Compatibility of Advanced Fast Reactor Materials."

Two reports were issued in 2009 (Natesan and Meimei Li 2009, Natesan et al. 2009) which examined the thermodynamic and kinetic factors involved in the purity of liquid sodium coolant for sodium reactor applications as well as the design specifications for the ANL pumped loop for testing advanced structural materials. Available information was presented on solubility of several metallic and nonmetallic elements along with a discussion of the possible mechanisms for the accumulation of impurities in sodium. That report concluded that the solubility of many metals in sodium is low (<1 part per million) in the temperature range of interest in sodium reactors and such trace amounts would not impact the mechanical integrity of structural materials and components.

The earlier report also analyzed the solubility and transport mechanisms of nonmetallic elements such as oxygen, nitrogen, carbon, and hydrogen in laboratory sodium loops and in reactor systems such as Experimental Breeder Reactor-II, Fast Flux Test Facility, and Clinch River Breeder Reactor. Among the nonmetallic elements discussed, oxygen is deemed controllable and its concentration in sodium can be maintained in sodium for long reactor life by using cold-trap method. It was concluded that among the cold-trap and getter-trap methods, the use of cold trap is sufficient to achieve oxygen concentration of the order of 1 part per million. Under these oxygen conditions in sodium, the corrosion performance of structural materials such as austenitic stainless steels and ferritic steels will be acceptable at a maximum core outlet sodium temperature of ≈550°C. In the current sodium compatibility studies, the oxygen concentration in sodium will be controlled and maintained at ≈1 ppm by controlling the cold trap temperature.

The oxygen concentration in sodium in the forced convection sodium loop will be controlled and monitored by maintaining the cold trap temperature in the range of 120-150°C, which would result in oxygen concentration in the range of 1-2 ppm. Uniaxial tensile specimens are being exposed to flowing sodium and will be retrieved and analyzed for corrosion and post-exposure tensile properties. Advanced materials for sodium exposure include austenitic alloy HT-UPS and ferritic-martensitic steels modified 9Cr-1Mo and NF616. Among the nonmetallic elements in sodium, carbon was assessed to have the most influence on structural materials since carbon, as an impurity, is not amenable to control and maintenance by any of

the simple purification methods. The dynamic equilibrium value for carbon in sodium systems is dependent on several factors, details of which were discussed in the earlier report. The current sodium compatibility studies will examine the role of carbon concentration in sodium on the carburization-decarburization of advanced structural materials at temperatures up to 650°C. Carbon will be added to the sodium by exposure of carbon-filled iron tubes, which over time will enable carbon to diffuse through iron and dissolve into sodium. The method enables addition of dissolved carbon (without carbon particulates) in sodium that is of interest for materials compatibility evaluation. The removal of carbon from the sodium will be accomplished by exposing carbon-gettering alloys such as refractory metals that have a high partitioning coefficient for carbon and also precipitate carbides, thereby decreasing the carbon concentration in sodium.

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1 Introduction

The objectives of the Fuel Cycle Research & Development are to expand the use of nuclear energy to meet the increasing global energy demand and to address the nuclear waste management. Reactor campaign is part of the Fuel Cycle Research & Development to develop liquid metal reactor technologies for use in a closed nuclear fuel cycle in the United States. The campaign is focusing on the sodium-cooled reactor concept because of its technical maturity. The current needs are for larger, more reliable, more economical reactors suitable for commercial nuclear power generation. Research and development focuses on three areas including advanced materials, innovative components and systems, and computer models and simulation. Inherent in this research and development is the selection and adequate long-term performance of reactor components that are directly exposed to the sodium coolant at elevated temperatures.

Figure 1 shows a schematic diagram of a sodium-cooled fast reactor. In these reactors, heat is generated from fission reactions in a nuclear-fueled core. The heat is removed by liquid sodium flowing through the core, the heat is transferred to a steam system, and the steam is used to drive a turbine. Because of excellent heat transfer properties, liquid sodium has been selected as the coolant and heat transport medium. In general austenitic stainless steels are used for the fuel cladding, in-core structural components, piping, and intermediate heat exchanger. Ferritic steels are, at present, used for the steam generator in the secondary coolant system. The selection of a particular material and combinations thereof that will be used in both primary and secondary circuits is based upon a number of factors. For in-core applications, the stability of the material in the fast-neutron environment and compatibility with the sodium coolant and the fuel (including possible fission-product interactions) are of primary concern. In the case of the steam generator, the resistance of the material to aqueous chloride and caustic cracking as well as minimization of corrosion wastage rates due to sodium-water reaction products in the event of a tube failure have important bearing on the material selection.

In general, structural materials can undergo a variety of interactions upon exposure to liquid sodium. The extent of interaction depends upon the conditions of temperature, temperature gradient, sodium velocity and purity, the materials of construction, and external sources and sinks for the impurities. The interactions can be broadly classified into either metallic or nonmetallic element mass transfer. Metallic element mass transfer usually establishes a go/no-go type of evaluation of an alloy for use in sodium environment, based upon material wastage or component section loss. A classic example of this is the unacceptable performance of vanadium base alloys in sodium of normal reactor purity. Upon selection of an alloy for sodium service, corrosion allowances that account for metallic element transfer can be incorporated into the design of specific components. Nonmetallic elements, such as oxygen, carbon, nitrogen, and hydrogen, are known to migrate in structural material/sodium systems (under both isothermal and non-isothermal conditions) as a result of chemical activity differences. In a previous report (Natesan and Meimei Li, 2009), we addressed the thermodynamics and kinetics of metallic and nonmetallic impurity elements in sodium and their role in the corrosion of structural materials such as austenitic stainless steels and ferritic steels. The report also presented the available data for the impurity elements and experience on control and maintenance of the impurities in sodium systems. The report also presented the impact of various impurity elements, especially nonmetallic elements, on the corrosion performance of various structural components in sodium-cooled reactors.

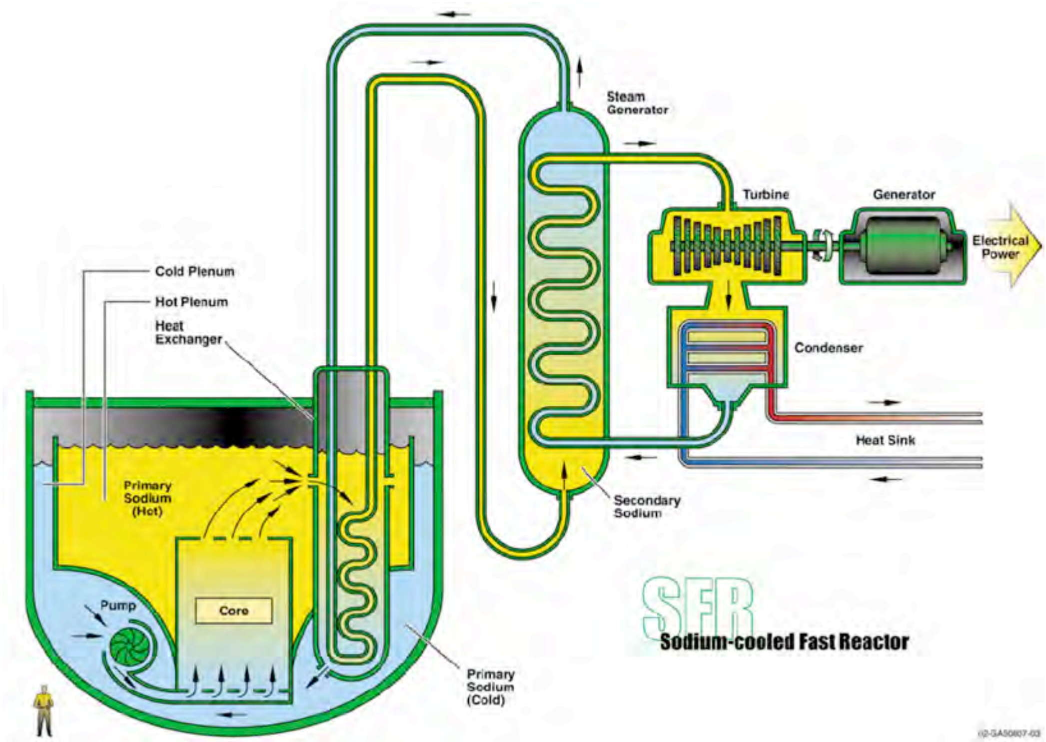

Figure 1. A schematic of sodium-cooled fast reactor.

In a subsequent report (Natesan et al. 2009), we gave a description of ongoing activities in the design, fabrication, construction, and planned testing of advanced structural materials in a forced convection sodium flow loop. The work is the Argonne National Laboratory portion of the effort on the work project entitled, "Sodium Compatibility of Advanced Fast Reactor Materials," and is a part of Advanced Materials Development within the Reactor Campaign. The objective of this project is to develop information on sodium corrosion compatibility of advanced materials being considered for sodium reactor applications. This report gives the status of the sodium pumped loop at Argonne National Laboratory, the specimen details, and the technical approach to evaluate the sodium compatibility of advanced structural alloys. This report is a deliverable from Argonne National Laboratory in FY2010 (M2GAN10SF050302) under the work package G-AN10SF0503 “Sodium Compatibility of Advanced Fast Reactor Materials.”

2 Background

A solid metal dissolves in a liquid metal by the diffusion of atoms from the lattice of the solid metal into a boundary layer, followed by transfer of the dissolved atoms from the boundary layer into the bulk liquid. In general, the rate of dissolution depends on the solubility of the solute element in the liquid metal, temperature, and hydrodynamic conditions of the solid/liquid interface. Apart from the atomistic dissolution of metallic elements into sodium, mass transfer can also occur by formation of corrosion products on the metallic surfaces due to interaction between reactants in sodium with the metals to form corrosion products that subsequently erode/spall into the flowing sodium. These corrosion products generally consist of binary or complex oxides of various metals.

The dissolution of metallic elements is generally unavoidable especially in systems with temperature gradients, and needs to be minimized to control the metal wastage in regions at elevated temperatures, limit the deposition of corrosion products in the low temperature regions of the system, and also to control the amount of radioactive corrosion product transport in the primary sodium circuit of the fast reactor. The approach to minimize the problem is to select materials that have 1) relatively low solubility in sodium, and 2) solubility values with small temperature dependence. Metallic element mass transfer usually establishes a go/no-go type of evaluation of an alloy for use as a structural material for service in liquid sodium environment.

Nonmetallic elements such as oxygen, carbon, nitrogen, and hydrogen are known to migrate in structural materials/sodium systems as a result of chemical activity differences that occur in isothermal or non-isothermal systems. Transfer of these elements also occurs in systems where combinations of materials of different composition are used. By examining the partitioning of nonmetallic elements between the structural alloy and sodium, the extent to which oxygen, carbon, and nitrogen interact in the structural material-sodium systems can be assessed. An earlier report addressed the thermodynamics of nonmetallic impurity elements in sodium with emphasis on purification, and examined the compatibility of structural materials from the sodium purity standpoint (Natesan and Meimei Li, 2009).

From the operation of the sodium-fast-reactor standpoint, the oxygen impurity in sodium can be controlled to ≈1 ppm which would result in acceptable corrosion of austenitic and ferritic steels and nickel-base alloys at a typical maximum operating temperature of 600°C for the coolant. The solubility of nitrogen in sodium is low in the reactor anticipated operating temperatures and its effect on the materials performance may not be significant. On the other hand, observations by various investigators, that materials of nominally the same composition with different degrees of decarburization/carburization in sodium loops operating over similar temperature ranges, have resulted in questions regarding the extrapolation of results obtained in small scale sodium loops to large reactor systems. Since carbon concentration in a given system is established by a dynamic equilibrium between the carbon sources and carbon sinks present in the system, it could vary from system to system as well as over time within a system. It is necessary to evaluate the influence of carbon in sodium on the microstructural and mechanical properties of advanced structural materials.

3 Sodium Forced Convection Loop

A small sodium loop was constructed at Argonne National Laboratory for testing of advanced structural materials. The construction of the loop has been completed and is currently undergoing shakedown. The loop consists of a sample tank with a heat exchanger, one electromagnetic pump, two electromagnetic flow meters, economizer, and cold trap. Each component is connected by 1/2-inch stainless steel tubing. The loop is placed over a stainless steel drip pan for spill control, and placed in a small containment made from sheet metal for smoke control in case of leak. The system is designed to circulate sodium under an argon atmosphere through the sample tank and the associated loop without an operator for an extended period of time (see Figure 2).

The total amount of sodium in the loop is approximately 10 kg. The nominal operating pressure and the temperature for the sample tank in the loop are 3 psig and 550°C, respectively. As shown in Figure 2, sodium flows from the sample tank and reduces its temperature as it flows through the economizer 1. The electromagnetic pump, EP021 pushes sodium and some goes through the electromagnetic flowmeter, FM141. This sodium flows back to the tank through the economizer 1. Remaining sodium flows through the electromagnetic flow meter, FM 241 and the economizer 2 while further reducing the temperature. The sodium then flows into the cold trap, in which the sodium purity is maintained. The clean sodium flows back through the economizer 2 and warms up. Finally, the clean sodium further increases the temperature in the economizer 1 and flows back into the tank.

An isometric view of the sodium loop is shown in Figure 3. The sample tank is constructed from 8-in, schedule 40, stainless steel pipe and a heat exchanger made of 1-1/4-inch, schedule 40, stainless steel pipe is attached to it. The volume of the tank is approximately 10 liters. The tank has three penetrations at the top for loading samples as well as small penetrations for a level probe, thermocouple and Argon/vacuum line. Each of the three penetrations for sample loading has a ball valve and a gate valve. The penetration is vertical and the ball valve is located above the gate valve. The ball valve isolates the inside of the tank from outside atmosphere and the gate valve protects the ball valve from sodium condensation. The Argon/vacuum line is fitted with a pressure gauge.

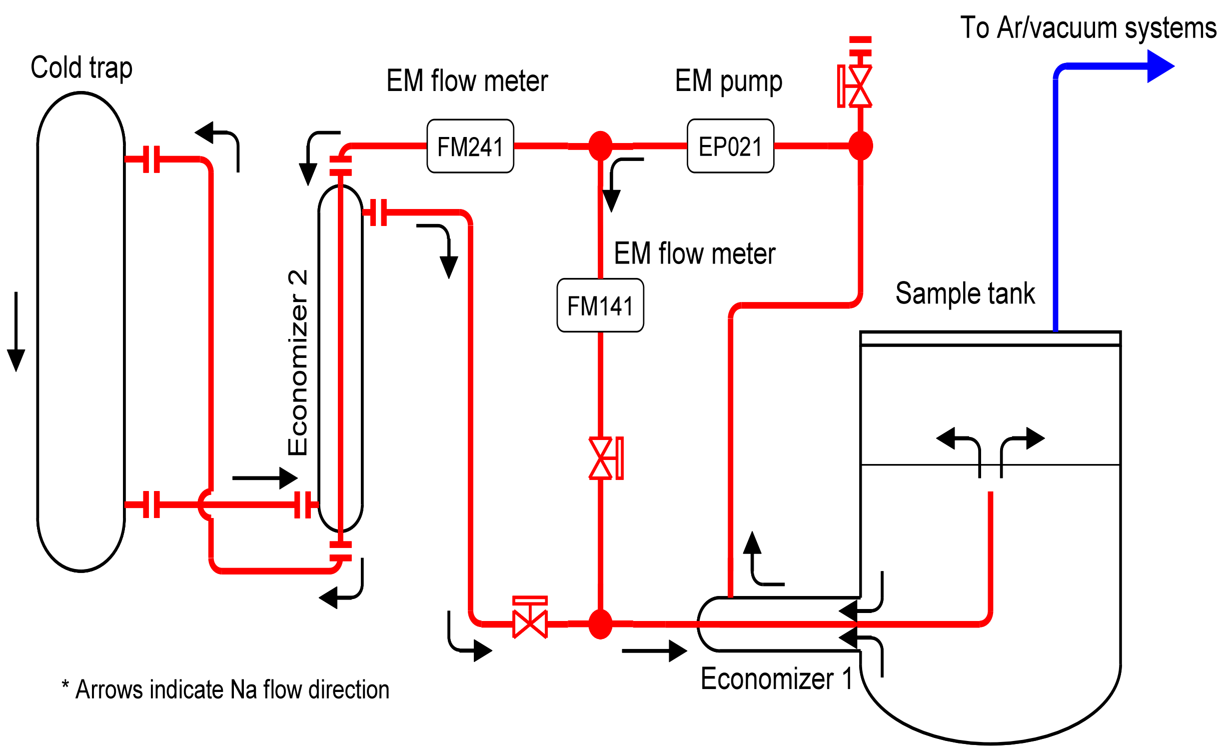

Figure 2. Schematic diagram of the loop.

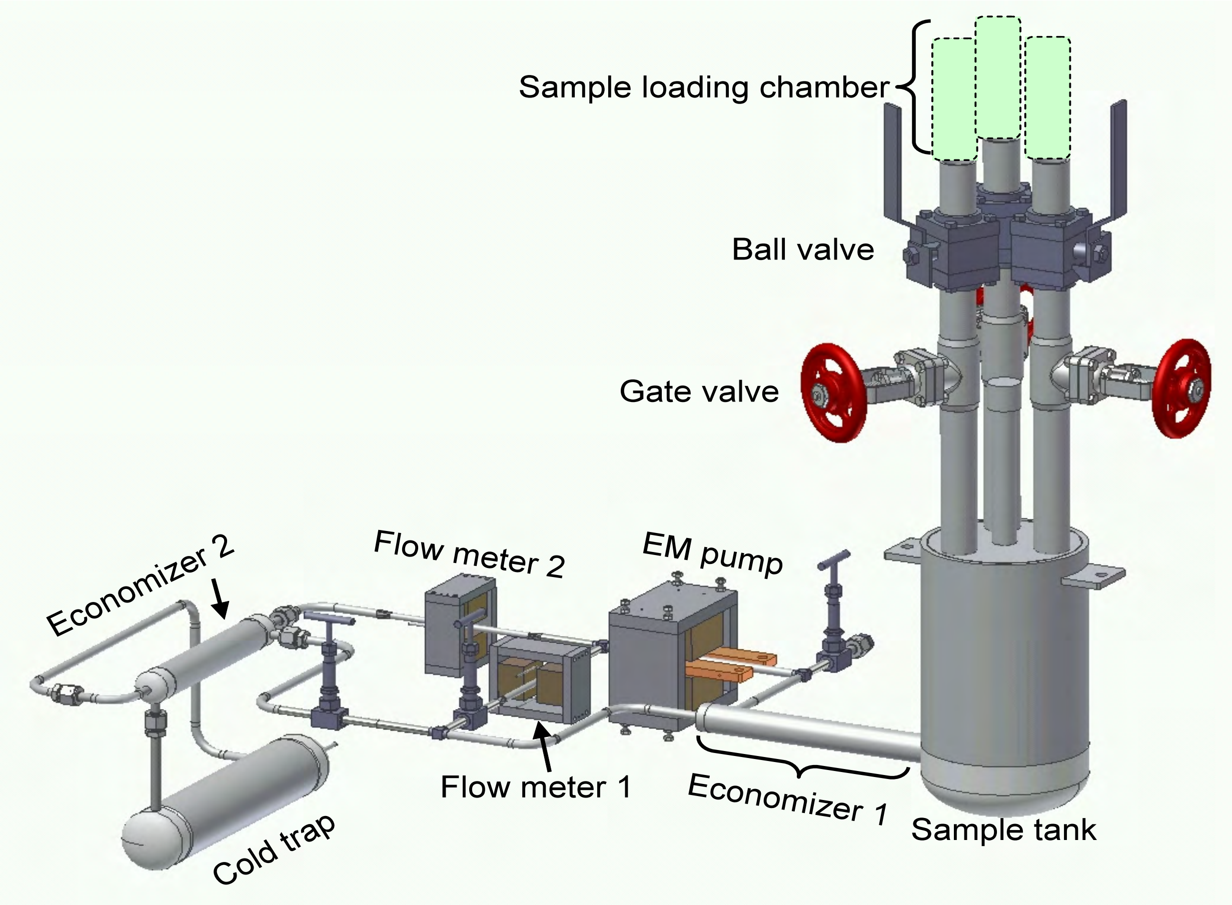

Figure 3. Isometric view of forced circulation sodium loop.

The cold trap and economizer are constructed from 3-inch and 1-1/2-inch, schedule 40, stainless steel pipe, respectively. The cold trap is packed with stainless steel mesh, providing a large surface area to facilitate precipitation of impurities. The cold trap is equipped with its own heaters, air blower, and Type-K thermocouples for precise temperature control, particularly at low temperatures in the range of 100-150 °C (see Figure 4). The temperature of the cold trap is controlled by means of feedback using a commercially available temperature controller (Omega Engineering, CN7500 series controller). The economizer is a tube-in-tube type heat exchanger with the inner tube being 1/2-in. stainless steel tubing. The economizer 1 is also equipped with heaters, air blower, and Type-K thermocouples while the economizer 2 is equipped with heaters and Type-K thermocouples, but without an air blower (see Figures 5 and 6). The temperature of the economizers is also controlled by means of temperature feedback using a commercially available temperature controller. The loop sodium is partially cooled while exchanging heat with outgoing sodium from the cold trap as it flows through the economizers. The partially cooled sodium enters the cold trap and is further cooled. The cold sodium leaving the cold trap is heated as it flows through the economizer.

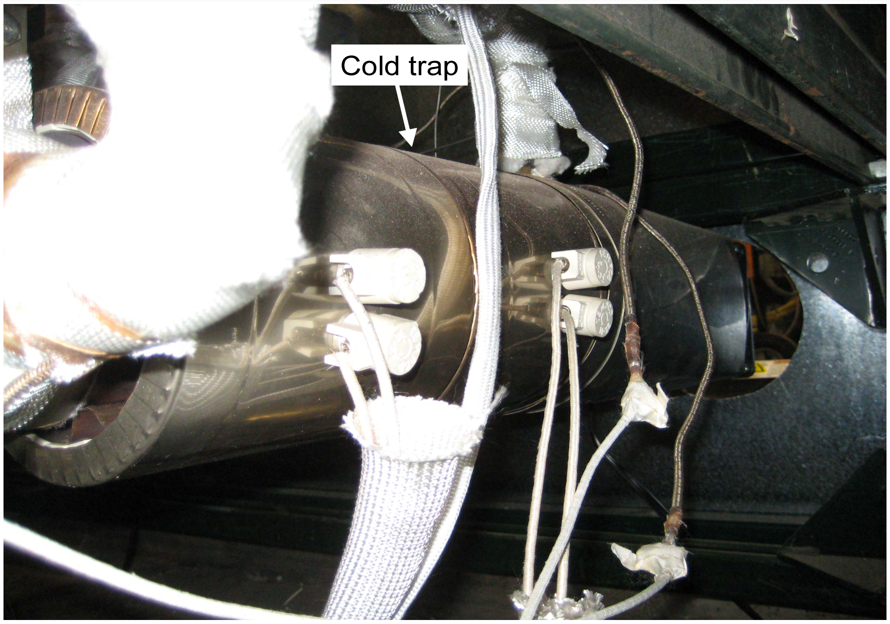

Figure 4. Cold trap with band heaters.

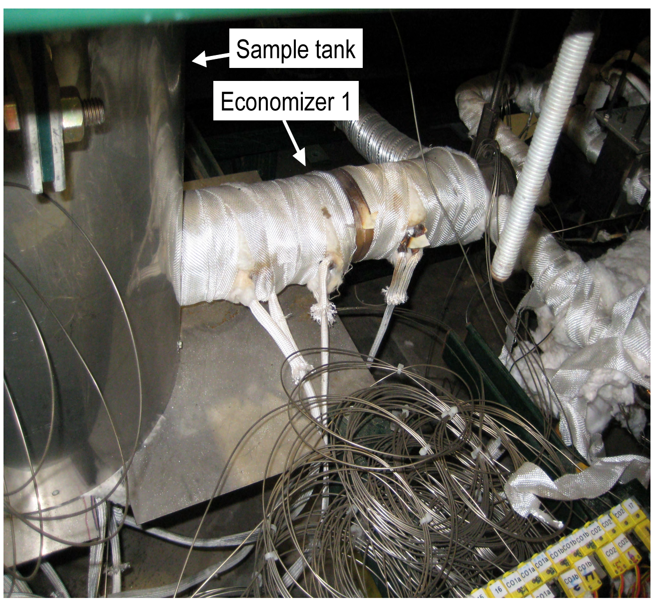

Figure 5. Economizer 1 covered with insulator.

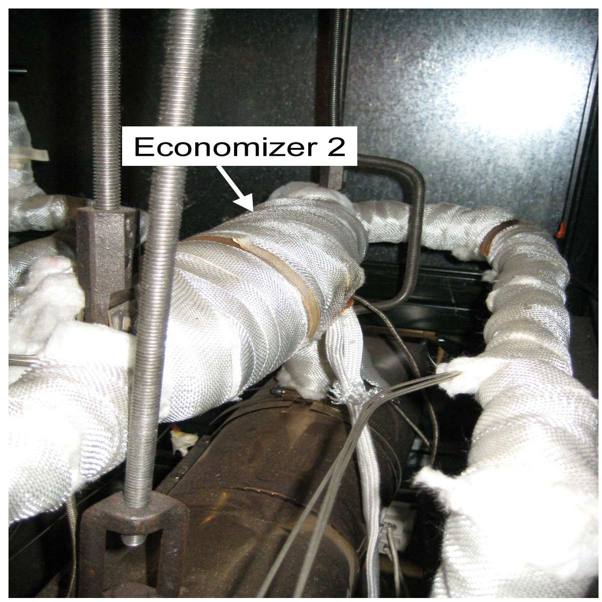

Figure 6. Economizer 2 covered with insulator.

The electromagnetic pump is a conventional, direct current, conduction electromagnetic pump with a yoke consisting of two permanent samarium-cobalt (SmCo) magnets (see Figure 7). The pump is powered by a direct current power supply (Hewlett Packard, HP 6681 A) that can deliver up to 580 A of direct current. The electromagnetic flow meters are also a simple direct current electromagnetic device with a yoke consisting of two SmCo magnets (see Figure 8).

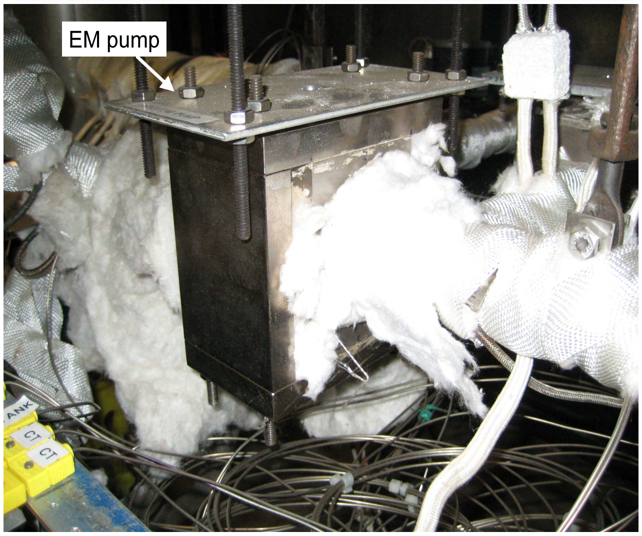

Figure 7. Electromagnetic pump.

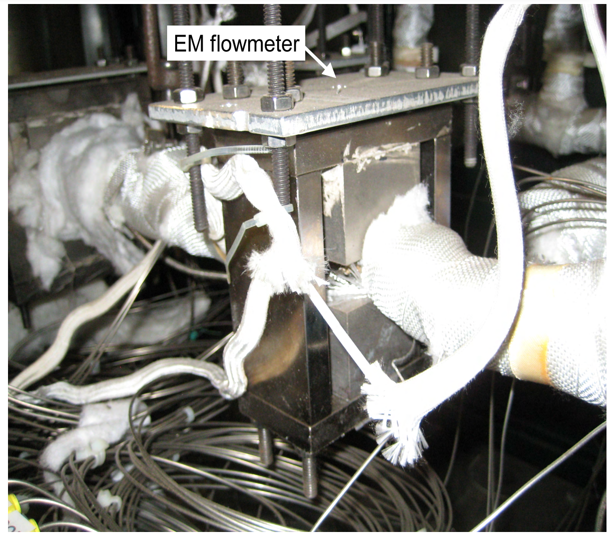

Figure 8. Electromagnetic flowmeter.

To prevent undue stresses in the loop, which may arise due to differential thermal expansion, the loop legs and components are hung from a Unistrut support frame, or otherwise supported loosely on the Unistrut frame.

The loop is heated by a number of ceramic band heaters attached to the loop components. Also several heating wires are used to heat the tubing. Many Type K thermocouples whose tolerance is within 2.2°C at ≤293.3°C or 0.75% at higher temperatures are attached to the loop at various locations to monitor temperatures, and some of them are used to control the heaters. All thermocouples used in the system are stainless steel sheathed, ungrounded type thermocouples. The heaters are grouped into 8 different zones to regulate heating of each zone as well as power distribution. Each heating zone is controlled by a solid-state relay based control system. For redundancy to ensure the safe operation of the loop each control system contains two independent temperature controllers and each controller has its own thermocouple for temperature monitoring. One controller is to accurately control the temperature around the set point using the solid-state relay and another controller acts as a circuit breaker using an electro-mechanical relay to turn off the power to the zone when the reading from the thermocouple exceeds the over-temperature set point. This latter heater controller is to provide an extra over-temperature protection to the heater control system (see Figure 9). Eight thermocouple bundles, each corresponding to each zone, are employed (Figure 9). Each thermocouple bundle contains three Type K thermocouples. The first of the three thermocouples is to provide an input to a temperature controller, the second is to provide separate input to another temperature controller for independent over-temperature protection, and the third is a spare. Figure 10 shows a schematic of the circuit diagram of heater control system.

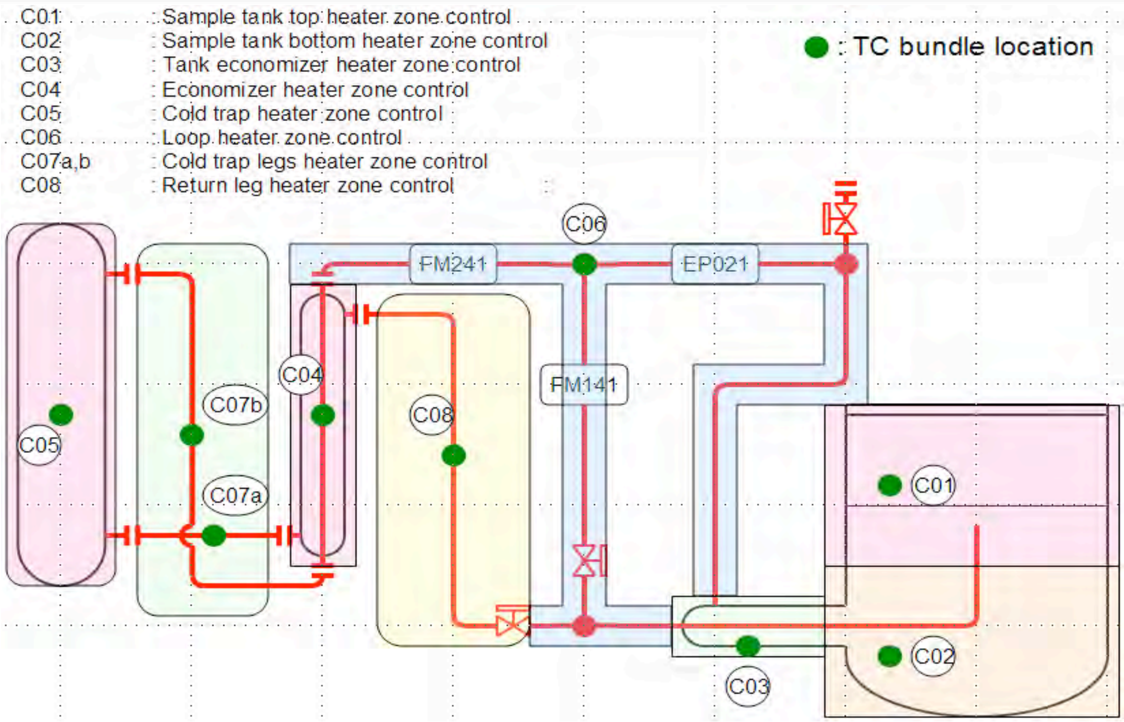

Figure 9. Heater control thermocouple bundle locations.

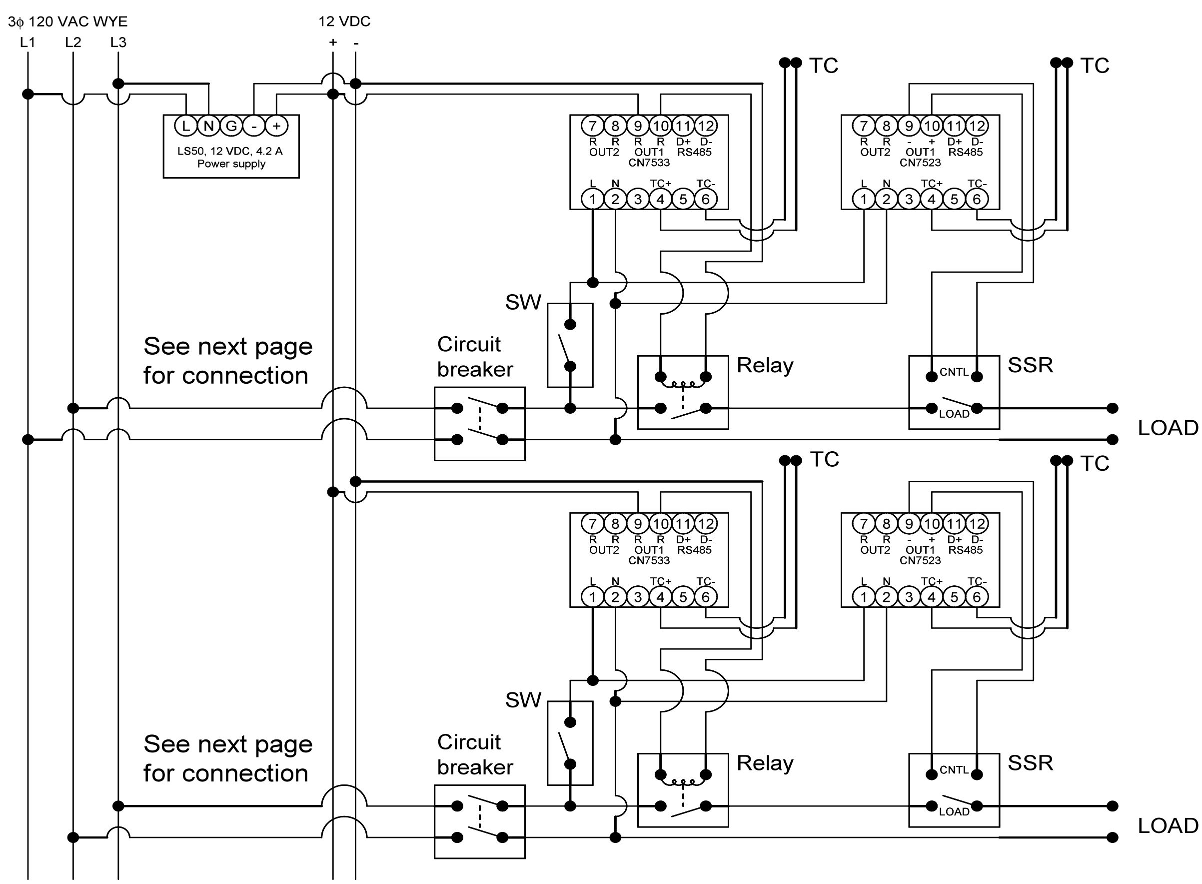

Figure 10. Circuit diagram of heater control system (2 zones are shown).

The temperatures of the various parts of the apparatus are monitored by type K thermocouples (Figure 11) of the loop. All the tips of these thermocouples attached to the outside wall of the loop were mounted using high temperature, high thermal conductive adhesive to ensure good thermal contact. Two thermocouples are embedded in the cold trap. A thermocouple is also inserted in the sample tank to better monitor the sodium temperature, to which the samples are exposed. The thermocouples monitoring various loop temperatures are connected to a data acquisition system, and collected data are sent to the computer for display and storage.

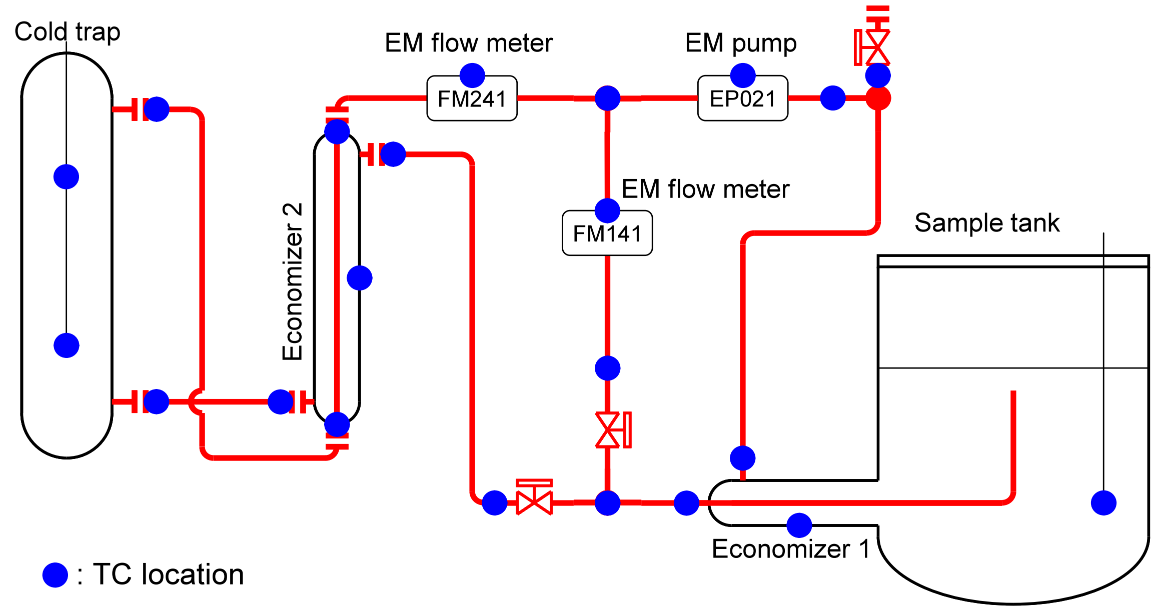

Figure 11. Monitoring thermocouple locations.

4 Loop Preparation for Material Exposure

After all the loop components were connected, the loop was He-leak tested to check the tightness of the system. All welds and connections below the sodium level indicated undetectable level of He leak ($<10^{-9}$ atm-liter/sec). In addition, the loop was leak tested at 50 psig and passed per ASME B 31.3, 345.

After all thermocouples and heaters were installed and wired, the loop was heated for shakedown of the heater control system as well as for bake-out to clean the system, while the loop was continuously evacuated by a dry scroll type vacuum pump at ≈30 microns ($\approx 3\times10^{-2}$ Torr). The sample tank was heated to 675°C while the rest of the system was heated to 525°C, at which the loop was maintained for a couple of days.

Subsequent to the loop bake-out, the loop was brought to 200°C for sodium loading. Sodium was supplied in a 55-gallon drum from Mine Safety Appliance and it was connected to the loop via 1/2 inch tubing (see Figure 12). Heating wires were installed along the connecting tube, which was thermally insulated by glass fiber insulator to keep the tube well above the melting point of sodium during the sodium transfer. The drum was heated at ≈120-130°C and was pressurized to ≈3-6 psig, while the loop was kept at ≈200°C under vacuum

(≈3×10^{-2} Torr). Then the isolation valve was slowly opened while monitoring temperatures along the loop legs and the level probe signals. The sodium was loaded in 2 steps. In the first step, the tank was filled to ≈50% level.

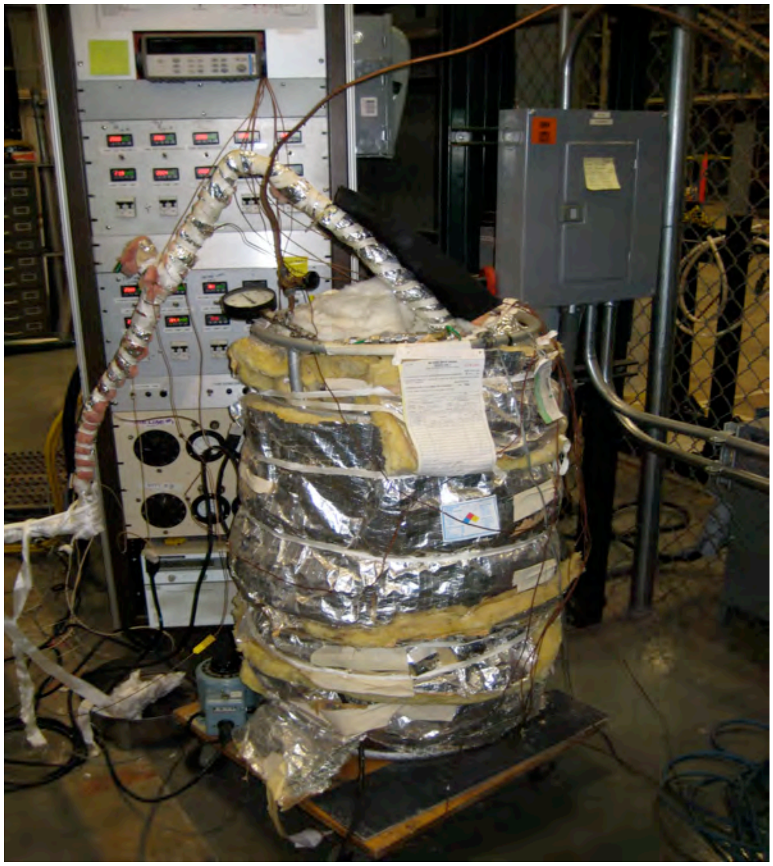

Figure 12. Sodium drum from Mine Safety Appliance.

Then the loop temperature was raised to ≈350°C to facilitate sodium settling as well as wetting of the inside wall of the loop, especially in the electromagnetic pump. Over 1 hr after the initial Na loading, temperature changes along the loop, especially around the economizer and the cold trap area, were observed, supposedly indicating gradual filling of each component as sodium flows and wets the inner surfaces of the loop. A small drop of sodium level was also observed during this settling period. Initially, the electromagnetic pump did not drive the sodium when the direct current power supply provided some current, indicating complete lack of wetting in the pump tubing. The heating wire for the electromagnetic pump was brought to the maximum power level and the maximum amount of the direct current (580A) was delivered to the pump to raise the pump tubing temperature. The combination of heat from the heating wire and the Joule heating raised the pump temperature from 120 to ≈360°C over a few hours of period, at which sudden temperature changes around the pump was observed. This sudden temperature changes were due to starting of sodium flow and immediately the pump current was reduced to ≈50A, at which stable sodium flow was confirmed and maintained. After wetting, the second sodium fill was performed and the total of ≈10 liters of sodium was loaded into the system. The argon cover gas pressure was then brought to ≈2 psig.

Prior to the loading of the specimens into the sodium vessel, the electromagnetic pump was operated at various feed-currents (0-100A) and surface waves of the sodium in the sample tank was observed to check if sufficient movement of sodium is obtained for satisfactory replenishment of sodium in the tank (see Figure 13). Apparently even at 40A of the electromagnetic pump current, clearly visible disturbances were observed in the sample tank.

A method to estimate the performance of an electromagnetic flow meter has been developed at Argonne National Laboratory and the performance of the electromagnetic flow meters used in this system has been calculated (See Figure 13). The range of the circulation frequency for the loop is set at 5-10 cycles per hour (or the time constant of 360 to 720 seconds). Since the inventory of the sodium in this system is approximately 10 liters, this circulation frequency corresponds to a flow rate of about 0.015-0.03 liters/second. The total flow rate of 0.015 mV (reading from FM 141 plus FM241) was achieved at the pump current of 60A. The initial material testing has started at the sodium temperature of 550 °C in the sample tank and the pump current of 60A.

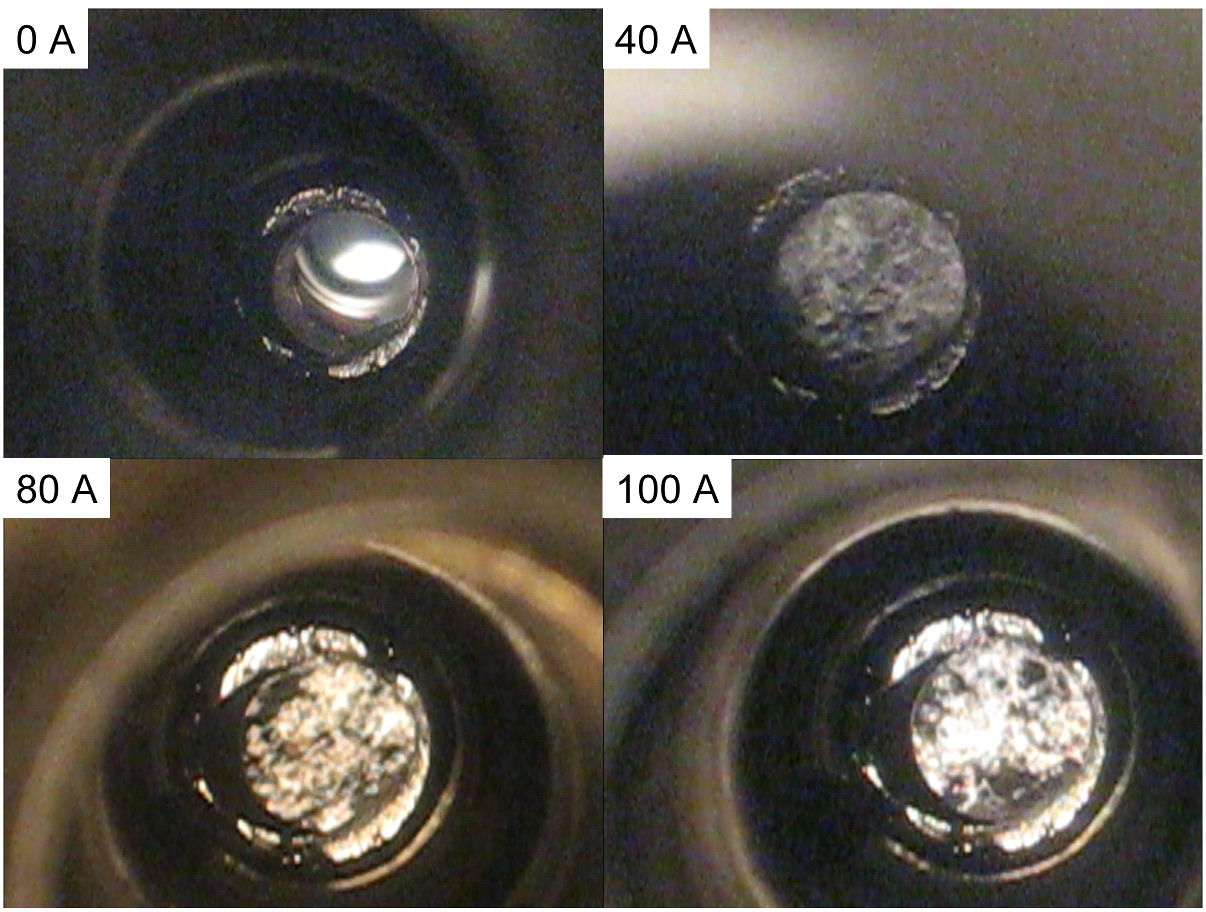

Figure 13. Sodium surface waves at various EM pump currents.

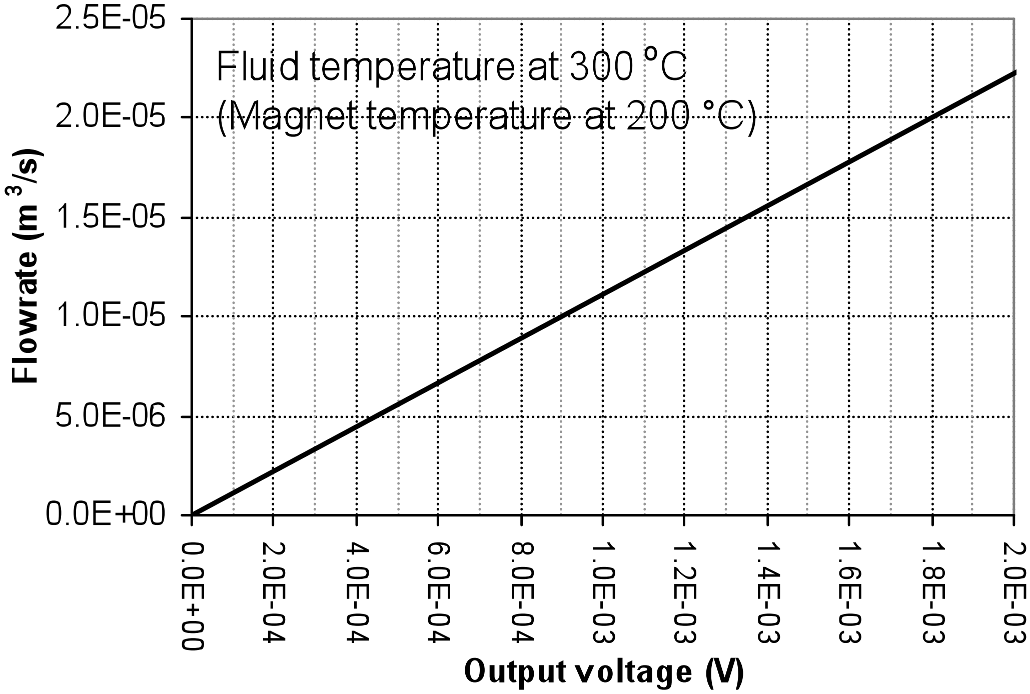

Figure 14. Estimated performance of the flowmeter.

5 Materials

The development of advanced structural materials with improved mechanical properties will enable improved reactor performance. This may be attained in the form of improved economics, greater flexibility, longer lifetimes for components, and larger operating safety margins. The development of advanced structural materials for use in sodium-cooled fast reactors has been initiated as part of the Advanced Structural Materials program under Advanced Fast Reactor Campaign (Busby et al. 2007, Busby et al. 2008).

The advanced materials to be examined include two ferritic steels, NF616 and modified 9Cr-1Mo, and austenitic stainless steel, HT-UPS. The conventional grade of Type 316 stainless steel will also be included for comparison of corrosion performance between the conventional and advanced austenitic alloys. The Oak Ridge National Laboratory provided the advanced alloys for use in this test program. The modified 9Cr-1Mo steel was normalized at 1050°C for 1 h and tempered at 760°C for 1 h. NF616 alloy is also used in the normalized and tempered conditions, the temperatures for which will be established in the future. The HT-UPS material will be annealed at 1200°C for 1 h in vacuum or will be in 1200°C hot-rolled condition. Figure 15 shows macrophotographs of uniaxial tensile specimens of the three advanced alloys that have been fabricated in accordance with ASTM specifications. Figure 16 shows a magnified view of two of the NF616 specimens. Figure 17 shows a macrophotograph of two assembled specimen holders prior to exposure in sodium.

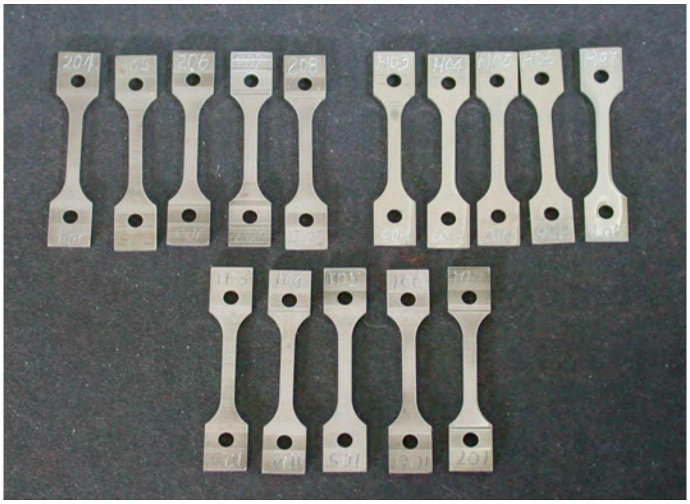

Figure 15. A macrophotograph of uniaxial tensile specimens of three advanced alloys, fabricated according to American Society for Testing Materials specifications.

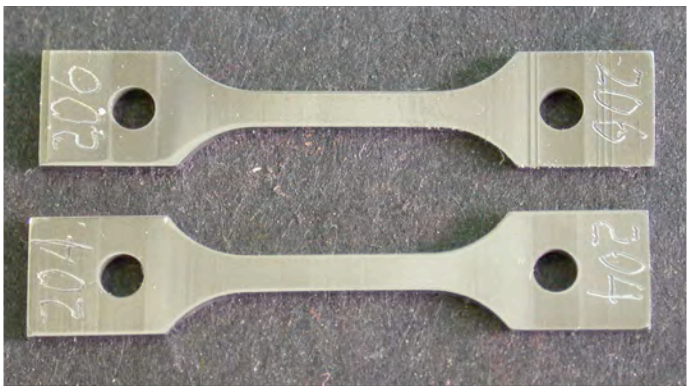

Figure 16. A macrophotograph of two uniaxial tensile specimens of advanced ferritic/martensitic alloy NF616, fabricated according to American Society for Testing Materials specifications.

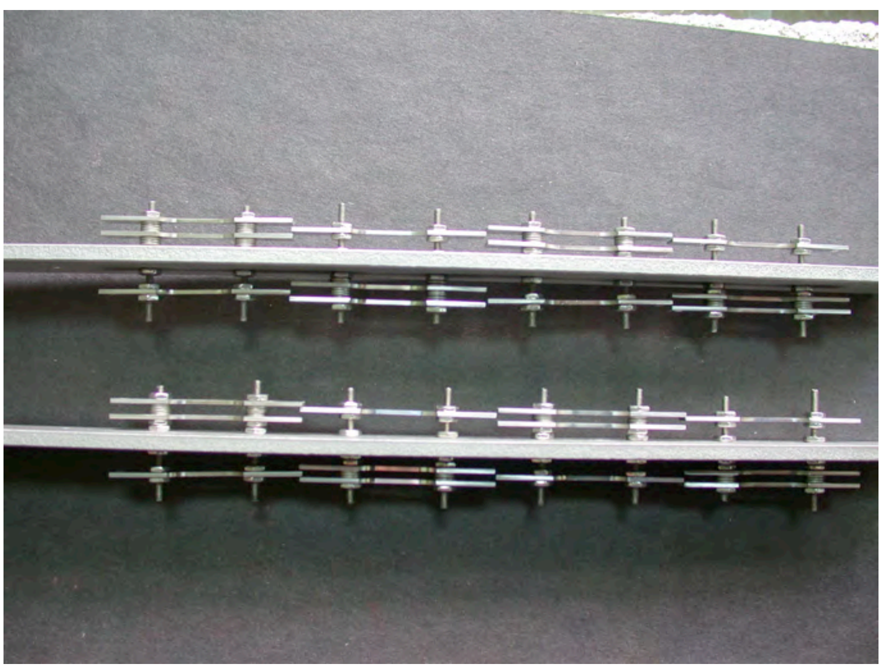

Figure 17. A macrophotograph of two specimen holders with assemblage of uniaxial tensile specimens of advanced alloys prior to sodium exposure.

6 Summary

This report gives a description of activities in the design, fabrication, construction, and planned testing of advanced structural materials in a relatively small (10kg sodium) forced convection sodium flow loop. The work is the Argonne National Laboratory portion of the effort on the work project entitled, "Sodium Compatibility of Advanced Fast Reactor Materials," as a part of Advanced Reactor Concepts Program of the Fuel Cycle Research & Development Reactor Campaign.

The ANL pumped sodium loop was designed, components were fabricated, and assembled. This report presents the details on loop components, their fabrication, and construction features. Sodium has been transferred to the loop and the loop components were conditioned to achieve adequate wetting by sodium. Among the nonmetallic elements present in liquid sodium, oxygen is deemed controllable and its concentration in sodium can be maintained in sodium for long reactor life by using cold-trap method. The use of cold trap is sufficient to achieve oxygen concentration of the order of 1 part per million. Under these oxygen conditions in sodium, the corrosion performance of structural materials such as austenitic stainless steels and ferritic steels will be acceptable at a maximum core outlet sodium temperature of ≈550°C. In the current sodium compatibility studies, the oxygen concentration in sodium will be controlled and maintained at ≈1 ppm by controlling the cold trap temperature.

Among other nonmetallic elements in sodium, carbon was assessed to have the most influence on structural materials since carbon, as an impurity, is not amenable to control and maintenance by any of the simple purification methods. The dynamic equilibrium value for carbon in sodium systems is dependent on several factors, details of which were discussed in an earlier report. Based on the assessment of carbon behavior in sodium systems, the current sodium compatibility studies will examine the role of carbon concentration in sodium on the carburization-decarburization of advanced structural materials at temperatures up to 650°C. Carbon will be added to the sodium by exposure of carbon-filled iron tubes, which over time will enable carbon to diffuse through iron and dissolve into sodium. The method enables addition of dissolved carbon (without carbon particulates) in sodium that is of interest for materials compatibility evaluation. The removal of carbon from the sodium will be accomplished by exposing carbon-gettering alloys such as refractory metals that have a high partitioning coefficient for carbon and also precipitate carbides, thereby decreasing the carbon concentration in sodium.

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Nuclear Engineering Division
Argonne National Laboratory
9700 South Case Avenue
Argonne, IL 60439

www.anl.gov