Development of Process for Cleanup of Sodium-CO₂ Reaction Products

Nuclear Science and Engineering Division

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Development of Process for Cleanup of Sodium-CO₂ Reaction Products

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ABSTRACT

This report describes Argonne's recent experience in cleaning several kilograms of sodium mixed with solid reaction products formed by sodium-CO₂ interaction. One of the reaction products is carbon monoxide (CO) and precautions have to be taken to protect personnel from exposure to CO. Ethanol was an effective method to clean and dispose of small amounts of residual reaction products embedded in a sodium matrix. Ideally, the residual sodium should be melted out of the parts as possible in order to reduce the amount of time it would take to clean with ethanol since this process is slow.

Table of Contents

Abstract	i
List of Figures	iv
List of Tables.....	v
1 Introduction	6
2 Motivation	7
3 Carbon Monoxide Evolution in Reaction Products and Handling Requirements.....	9
4 Procedure Used to Remove Channel and Clean Reaction Products	10
5 Procedure for Cleaning Dump Tank Contaminated with Sodium-CO ₂ Reaction Products.....	13
6 Observations of Sodium Reaction Product Behavior in Ethanol	13
7 Observations of Sodium Reaction Product Behavior in Air	14
8 Observations of Sodium Reaction Product Behavior with Superheated Steam and Water in the Sodium Passivation Stall	14
9 Summary	15
Acknowledgements	15
References	15

LIST OF FIGURES

Figure 1: Drawing of bottom injection hub/nozzle/channel assembly. Part in red box was removed with the test vessel in place in the SNAKE enclosure. The part in blue dashed box remained embedded in sodium inside the test vessel and was removed using a process described below in the B308 scrubber. Approximately a 50 cm (19.6”) section of nozzle tube and channel remained embedded inside the test vessel.....	8
Figure 2: Test vessel attached to cleaning framework that will be used to direct bottom outlet of vessel into the scrubber pans.....	11
Figure 3: Pictures of burning sodium/sodium-CO ₂ reaction products in burn trays in the B308 Scrubber.	12

LIST OF TABLES

Table 1: Several options for removing channel and passivizing sodium and sodium reaction products 9

1 Introduction

The Department of Energy's Office of Nuclear Energy (DOE-NE) Advanced Reactor Technologies (ART) program is focused on the development of clean, safe, and affordable nuclear power. The ART program contains a spectrum of experimental facilities to support R&D on advanced reactor concepts including the SNAKE (S-CO₂ Na Kinetics Experiment) apparatus at Argonne National Laboratory (Argonne).

The supercritical carbon dioxide (sCO₂) Brayton cycle, coupled with a Sodium-Cooled Fast Reactor (SFR), has been identified as an advanced conversion technology that could contribute to meeting ART objectives especially in terms of improved economics and safety. One appealing feature of the sCO₂ Brayton energy conversion system is the smaller footprint that the hardware requires relative to the traditional superheated steam cycle, which is in part due to the small size of the turbomachinery as well as the use of compact diffusion-bonded heat exchangers (DBHEs) as the heat source heat exchanger (sodium-to-CO₂) as well as the recuperator and cooler modules.

The DBHEs have a high degree of structural integrity; however, the potential for leaks to develop between the sodium and CO₂ coolant channels in the secondary sodium heat exchanger cannot be ruled out. This type of failure would lead to discharge of high pressure CO₂ into the secondary coolant circuit. Due to the robustness of the DBHE design, catastrophic failure leading to CO₂ jet blowdown into the intermediate sodium loop is not deemed likely. Rather, small cracks (or micro-leaks) may develop in which CO₂ will bleed into the secondary system at a relatively low rate and chemically react with the sodium.

In recognition of the anticipated failure mode for a DBHE sodium-to-CO₂ heat exchanger, an Argonne experiment program, SNAKE, was initiated in Fiscal Year 2010 (see Farmer et al., 2010; Gerardi et al. 2011, 2012a, 2012b, 2013a&b, 2014, 2015, 2016 and 2017). The purpose of the SNAKE program was to investigate the reaction characteristics between sodium and CO₂ under micro-leak conditions. Specifically, the objective of the SNAKE facility was to determine the fundamental nature and extent of the chemical reactions that occur when high-pressure CO₂ is injected into liquid sodium from a micro-leak across a stainless-steel pressure boundary as a function of the sodium pool temperature and inlet CO₂ flowrate. SNAKE was also designed to examine the potential for the micro-leak to seal itself as a result of blockage formation from the chemical reaction byproducts of the sodium-CO₂ reaction, or as a result of oxide layer buildup on the crack faces.

Twelve sodium-CO₂ tests have been performed to-date. Two of these tests experienced flow-blockage of the CO₂ injection site due to rapid production of solid sodium-CO₂ reaction products. Ten tests sustained CO₂ injection flow for several minutes, all under an hour, until the flow of CO₂ was manually terminated. These ten sustained injection tests generated quantities of solid reaction products, in some cases substantial amounts. Reaction products generated included sodium carbonate, (Na₂CO₃), sodium oxide (Na₂O), carbon (C) and sodium oxalate (Na₂C₂O₄).

In the event of an sCO₂ leak in the DBHE of an operating SFR, it will be important to isolate the affected DBHE unit(s), locate and seal the leak site(s), clean the units and return them to service. The solid reaction products can block flow passages and cause corrosion or carburization which

could further damage high-capital cost DBHEs. This memo-report discusses Argonne's recent experience cleaning several kilograms of sodium mixed with solid reaction products. This report is not intended to be a comprehensive review of cleaning methodologies or to recommend an approach for use in an SFR; however, some of the lessons learned discussed here may be useful for future engineers that aim to develop procedures for cleaning SFR components contaminated with sodium-CO₂ reaction products.

2 Motivation

In Fiscal Year 2017, a sodium-CO₂ injection experiment was performed that involved injection of CO₂ into a sodium-filled DBHE single channel mock-up. CO₂ was injected over the course of 0.56 hrs and substantial quantities of solid reaction products were generated. The details of the test setup and results were reported in Gerardi et al. (2017). Removal of the injection hub/nozzle/channel assembly was initiated following cooldown of test section. The hub itself was successfully removed but the nozzle tube and channel were found to be embedded in a solid mass of sodium and sodium reaction products that made mechanical removal impossible. The nozzle tube was mechanically separated from the hub. The injection hub was replaced with a new blind hub while the nozzle tube and channel were left inside the test vessel embedded in the solid sodium/reaction product matrix. It was desired to extract the channel and clean all residue of sodium and sodium-CO₂ reaction products so that the channel could be physically and mechanically inspected.

Approximately a 50 cm (19.6") section of nozzle tube and channel remained embedded inside the test vessel, as depicted in Figure 1.

The solid reaction products mixed with solid sodium were in the form of a solid foam and were very porous and crumbly. Due to the porosity of the reaction products, the reaction rate with air of the remaining sodium and air-reactive products was much faster and more vigorous than the reaction of dense solid pure sodium with air. This means that the reaction products self-warm and produce hydrogen and possibly carbon monoxide more rapidly than pure sodium. Carbon monoxide production has been observed when the reaction products have been placed in contact with air. Good ventilation is required when the reaction products are exposed to air because the production of hydrogen and carbon monoxide can lead to combustion hazards.

Several methods, listed in Table 1, were considered for removal of the channel assembly and for disposal of the reaction products. The list is not all inclusive but are some of the most common methods used to handle and dispose of bulk sodium. It was desirable to remove the channel with minimal physical damage. This meant that the standard approach used at Argonne to clean sodium-contaminated parts, applying superheated steam to burn the sodium in a controlled manner, was not appropriate since this method would likely heat the channel up considerably and potentially obfuscate physical evidence from the sodium-CO₂ reaction test.

Figure 1: Drawing of bottom injection hub/nozzle/channel assembly. Part in red box was removed with the test vessel in place in the SNAKE enclosure. The part in blue dashed box remained embedded in sodium inside the test vessel and was removed using a process described below in the B308 scrubber. Approximately a 50 cm (19.6") section of nozzle tube and channel remained embedded inside the test vessel.

Table 1: Several options for removing channel and passivizing sodium and sodium reaction products

Method	Potential for damage to channel and test data	Simplicity	Length of cleaning process
Mechanically push channel and reaction products out of test vessel, scrape majority of sodium and reaction products off and clean channel with ethanol *	Minimal	Simple	< 1 day
Passivize sodium and reaction products in-place in the test vessel using superheated steam in a sodium passivation booth	High	Simple	< 1 day
Passivize sodium and reaction products in-place in the test vessel using a controlled ethanol drip/wash	Minimal	Challenging setup and have H ₂ production mitigation requirements	Weeks
Passivize sodium and reaction products in-place in the test vessel using a carbonation process with wet CO ₂	Moderate	Relatively challenging setup for bulk sodium	Days

* Selected Method

The method that was selected was to mechanically push the channel, along with any attached bulk sodium or reaction products, out of the test vessel. This method did not require any local heating of the channel, which minimized damage to any remaining evidence of the sodium-CO₂ reaction. The work was completed in the Building 308 sodium passivation unit (scrubber) at Argonne with the unit on at all times to ensure that workers were not exposed to any CO or toxic dusts. Once removed from the test vessel, the channel was cleaned of residue of sodium and reaction products by scraping them off. The channel was then placed in an ethanol bath and then rinsed with distilled water to fully clean it before examination. Bulk quantities of sodium and reaction products were burned off in the sodium passivation unit after the channel was extracted.

3 Carbon Monoxide Evolution in Reaction Products and Handling Requirements

Carbon monoxide production from the reaction products has been observed when the reaction products were in contact with air for several hours. Significant production of carbon monoxide is unlikely but theoretically possible. Two mechanisms for the production of CO were theorized. The first was that carbon monoxide was trapped in the sodium reaction product foam pores during the sodium-CO₂ reaction test and the breakdown of the porous structure when in contact with air caused the release of the trapped CO. The second possible mechanism is the reaction of a reaction product with air or humidity to form CO. None of the known reaction products (Na₂O,

Na₂CO₃, Na₂C₂O₄ or C) were expected to react with air or water to form CO, so such a reaction would be unexpected and point to a previously unidentified reaction product being present. The most likely source of CO was CO from the sodium-CO₂ reaction trapped in the porous solid structure.

The possibility of CO release when handling the reaction products required specialized procedures and precautions. Personal carbon monoxide detectors were used when handling the reaction products. These detectors were located in the breathing zone of workers and were set to alarm at 25 ppm CO. Additionally, exhaust ventilation was operational at all times during reaction product handling. In the case of the channel removal, the scrubber air handling equipment remained on at all times while the reaction products or the test vessel's interior were exposed to air or moisture.

4 Procedure Used to Remove Channel and Clean Reaction Products

The procedure below was used to successfully remove the channel without significant damage and to clean and dispose of the sodium-CO₂ reaction products.

1. Personnel donned PPE appropriate for burning sodium including fire retardant clothing, leather aprons, leather gloves, leather spats and face shields.
2. Personnel turned on their personal carbon monoxide detectors and attached them to their clothing in their breathing zone.
3. The test vessel was supported at approximately a 45 degree angle and bolted to a temporary unistrut structure. It was placed just outside the B308 scrubber door with approximately 50 cm clearance above the burn trays (see Figure 2). The bottom opening of the test section was located inside the scrubber.
4. The scrubber blower fan was turned on and left on during all operations where the reaction products were exposed to air.
5. The bottom and top test vessel hubs were removed.
6. Originally, a threaded plunger was intended to be used to push the reaction products out of the test vessel. However, the thermowells that were embedded in the sodium and running the length of the test vessel prevented the plunger from moving downward. Instead, a thick-walled 1.5" diameter pipe was inserted into the top of the test vessel and a sledgehammer was used to drive the pipe into the reaction products which, over time, successfully pushed the reaction products and channel out of the bottom of the test vessel into the burn trays. Note: This method would not have worked if the material inside the test tube was pure sodium which is far too soft and sticky to push like this.
7. Removed 2-3 samples of reaction products near the nozzle and saved for post-test examination.
8. Placed the channel assembly in an ethanol bath in a hood (or scrubber) in Bldg. 206 to react off residual sodium and reaction products in a controlled manner. After all material was observed to have reacted, then cleaned channel assembly with DI water and initiated mechanical and visual inspection.
9. Burned off the remaining bulk sodium and reaction products with the dry steam, wet steam and finally liquid water.

Figure 2: Test vessel attached to cleaning framework that will be used to direct bottom outlet of vessel into the scrubber pans.

If the above method was not as successful as it was to remove the reaction products and sodium from the test vessel, several backup plans were prepared in advance. These methods risked damaging/destroying some of the sodium-CO₂ reaction data so were only to be used if mechanical removal did not work. These steps were not needed but are documented here in case they are needed in the future:

1. If the sodium provides too much resistance, then it may require some heating to soften the sodium and enable the extraction of the interior materials.
2. Place heater tape on the exterior of the test vessel for possible use during part removal.

3. Heat the test vessel slowly, up to 110 °C, with 90 °C as a hold point, from the exterior with resistive heating elements (heater tape) until the sodium lining the test vessel wall softens enough that manual pushing of the reaction products can be improved. This is considered to be the least intrusive and least destructive method to remove the channel intact. If heating does not work proceed to next step; if it did work then skip the next step.
4. If the resistive heating element method does not work after a maximum setpoint of 110 °C, then remove the heaters and perform the following steps:
 - a. Place vessel fully in scrubber.
 - b. Use torch on exterior of test vessel to heat vessel.
 - c. Spray the exterior of the test vessel with dry steam to heat the vessel. Try to push the tube/channel assembly with the screw assembly.
 - d. If the dry steam does not work, then stop the steam and remove the screw hub assembly. Place the entire test vessel inside the scrubber at an angle and, with an acetylene torch, melt the sodium and the tube/channel slides out of the test vessel.
5. Let all materials cool if possible. Mechanically remove as much residual sodium and reaction products from the tube and channel assembly as possible.
6. Continue with Step 7 in previous procedure.

Photos of the sodium/sodium-CO₂ reaction product burning process are included in Figure 3. Partial views of the personnel protective equipment such as leather aprons and spats are shown in these photos. The sodium and sodium-CO₂ reaction products were contained within burn trays that were inside the scrubber burn stall area. The massive amount of smoke generated and low flame are typical of a sodium fire.

Figure 3: Pictures of burning sodium/sodium-CO₂ reaction products in burn trays in the B308 Scrubber.

5 Procedure for Cleaning Dump Tank Contaminated with Sodium-CO₂ Reaction Products

The vertical 8" dump tank was full of sodium that also contained some quantity of sodium-CO₂ reaction products. The 2017 experiment was partially drained into this dump tank. This cleaning process was very simple and took place like a standard sodium dump vessel cleaning:

1. Attach manual valve and extension line to dump tank siphon tube port.
2. Place dump tank just outside of scrubber door and extension line 2"-4" from burn tray located inside of the scrubber.
3. Heat dump tank, valve and extension line to 150 °C.
4. Personnel donned PPE appropriate for burning sodium including fire retardant clothing, leather aprons, leather gloves, leather spats and face shields.
5. Personnel turned on their personal carbon monoxide detectors and attached them to their clothing in their breathing zone.
6. The scrubber blower fan was turned on and left on during all operations where the reaction products were exposed to air.
7. Apply < 5 psi argon pressure to the dump tank headspace.
8. Open manual valve and allow sodium to flow into the burn tray(s) until sodium flow stops.
9. Close manual valve, allow to cool and roll dump tank out of area.
10. Allow sodium to burn in the tray which may require occasional stirring.
11. Rinse tray with dry steam, wet steam and then liquid water.

6 Observations of Sodium Reaction Product Behavior in Ethanol

Ethanol is an effective way to passivize small quantities, i.e. hundreds of grams, in a very controlled manner. The reaction rate is slow enough that a part contaminated with residual internal sodium can simply be placed in a bath of ethanol and left there for hours or days to allow the reaction to slowly take place. This process should be performed without a lid and in a fume hood since hydrogen is generated and ethanol vapors should be controlled. Periodically, the part will need to be gently disturbed to break up any layers of sodium ethoxide or sodium oxide that form on the surface in order to ensure that the ethanol can reach and react with remaining clean sodium. Once no hydrogen bubbles are observed, then the part can be removed from the ethanol bath and rinsed with distilled water to react any sub-gram quantities of sodium that remain and cleanse of any residual sodium-ethoxide.

This same process is effective to react off the metallic sodium that is mixed with the solid sodium-CO₂ reaction product foam matrix after a test. Sodium oxide will also react with ethanol to form sodium ethanolate and water. Sodium carbonate, sodium oxalate and carbon do not react with ethanol. However, the water that is formed by the reaction of ethanol and sodium oxide will dissolve sodium carbonate and sodium oxalate. Thus, ethanol is an effective way to passivize small amounts of sodium contaminated with sodium-CO₂ reaction products in a slow and controlled manner. Once the sodium and sodium oxide has reacted, there will remain some amount of sodium carbonate, oxalate and carbon. These can be rinsed of the component using water which will dissolve some of the carbonate and oxalate.

Some observations Argonne researchers have made when cleaning sodium-CO₂ reaction products with ethanol include:

- The carbon material does not react or dissolve with ethanol so the reaction products turn the ethanol solution a grayish color.
- No substantial difference in reaction rate or behavior other than color.

7 Observations of Sodium Reaction Product Behavior in Air

Due to their porosity, the reaction rate with the remaining sodium and air-reactive products is much faster and more vigorous than the reaction of solid pure sodium with air. This means that the reaction products self-warm and produce hydrogen and possibly carbon monoxide more rapidly than pure sodium. At room temperature, the reaction products mixed with sodium will vigorously bubble and slowly turn from dark black to a gray color. Sodium is hygroscopic and the reaction products themselves may be as well. Some CO production has been observed, at very low concentrations, when the reaction products were exposed to air.

8 Observations of Sodium Reaction Product Behavior with Superheated Steam and Water in the Sodium Passivation Stall

During the test vessel cleaning in the scrubber, the following observations were made:

- Reaction products were able to be hammered from tube with force, sodium would not have been able to be removed in such a matter.
- Reaction products stuck to tools in clumps requiring careful cleaning of tools.
- Clumps of reaction product could be ignited with simple application of flame (this burned with the vigor of crumpled paper).
- Once bulk material was removed from pipe, the pipe was heated for some time using a torch, eventually the remainder fell out in a mix of liquid and solid clumps.
- The solid clumps were easily ignited by applying flame to one of the more solid foam clumps.
- Reaction was self-sustaining.
- Application of steam would reveal covered pockets of pure sodium resulting in pops and cracks.
- No changes currently being considered for future work, especially since it is not anticipated that parts will need to be salvaged in the future which will make disposal simpler since such care will not need to be taken.

During the dump tank cleaning in the scrubber the following observations were made:

- There did not appear to be a substantial amount of reaction products in the dump tank, mostly pure sodium.

- Small amounts of reaction products did come out with the flow of sodium, they auto ignited at this temperature when exposed to air. Note that pure sodium generally does not auto ignite at this temperature in air.

9 Summary

This report described Argonne's recent experience cleaning several kilograms of sodium mixed with solid reaction products. This report is not intended to be a comprehensive review of cleaning methodologies or to recommend an approach for use in an SFR. However, some of the lessons learned that have been discussed here may be useful for future engineers that aim to develop procedures for cleaning SFR components contaminated with sodium-CO₂ reaction products.

In the event of an sCO₂ leak in the DBHE of an operating SFR, it will be important to isolate the affected DBHE unit(s), locate and seal the leak site(s), clean the units and return them to service. The solid reaction products can block flow passages and cause corrosion or carburization which could further damage high-capital cost DBHEs. This report shows that ethanol was an effective method to clean and dispose of small amounts of residual reaction products embedded in a sodium matrix. Ideally, the residual sodium should be melted out of the parts as possible in order to reduce the amount of time it would take to clean with ethanol since this process is slow. The small passages of a DBHE, coupled with the potentially tortuous pathways and inaccessible interior can make access to the internal parts very challenging. DBHE's should be designed with draining of sodium and even sodium contaminated with solid reaction products as a design requirement. This may require the ability to flip the DBHE to drain from either end, if solid reaction products plug the central region, for example.

Post-test results of the channel-mockup that was extracted will be discussed in a future report.

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