

Material Studies Related to the Use of NaK Heat Exchangers Coupled to Stirling Heater Heads

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Abstract

NASA has been supporting design studies and technology development that could provide power to an outpost on the Moon, Mars, or an asteroid. Technology development efforts have included fabrication and evaluation of components used in a Stirling engine power conversion system. Destructive material evaluation was performed on a NaK shell heat exchanger that was developed by the NASA Glenn Research Center (GRC) and integrated with a commercial 1 kWe Stirling convertor from Sunpower Incorporated. The NaK Stirling test demonstrated Stirling convertor electrical power generation using a pumped liquid metal heat source under thermal conditions that represent the heat exchanger liquid metal loop in a Fission Power Systems (FPS) reactor. The convertors were operated for a total test time of 66 hr at a maximum temperature of 823 K. After the test was completed and NaK removed, the heat exchanger assembly was sectioned to evaluate any material interactions with the flowing liquid metal. Several dissimilar-metal braze joint options, crucial for the heat exchanger transfer path, were also investigated. A comprehensive investigation was completed and lessons learned for future heat exchanger development efforts are discussed.

Introduction

Stirling engine power convertor technology is one option being explored for generating space-mission power. Fission power offers robust, environment-independent flexibility and adaptability for a broad range of space mission power requirements. The Fission Power Systems (FPS) Power Conversion Materials Risk Reduction team has been tasked with reviewing the materials and fabrication options for the free-piston Stirling power conversion candidate engine design. The Fission Surface Power System (FSPS) concept developed for the Moon and Mars would provide a net power of 40 kWe with a fullpower service life of 8 years (Fission Surface Power System Initial Concept Definition, 2010). Although the hot-end temperatures are lower for a candidate FPS Stirling convertor than those used in other successful systems, the relatively large heater head size and the NaK heat transfer fluid pose distinct challenges. Recently two P2A (formerly known as EG-1000) 1 kWe free-piston Stirling power convertors, purchased from Sunpower Incorporated, were retrofitted with experimental heater heads featuring NaK heat exchangers designed by the NASA Glenn Research Center (GRC) to allow the convertors to be interfaced with a pumped NaK loop at the NASA Marshall Space Flight Center (MSFC) (Mason, 2009). For this design, type 316L stainless steel (SS316L) is used as the exclusive containment material for the NaK. The convertors were operated at hot-end temperatures ranging from 673 to 823 K, cold-end temperatures from 303 to 343 K, and piston amplitudes ranging from 6 to 11 mm. The total NaK

mass flow to both convertors ranged from 500 to 900 g/s. The NaK heat exchangers were operated for 66 service hours, met all experimental objectives, and the test units operated as expected (Briggs, Geng, and Robbie, 2010). One of the two heat exchanger units was dissected to investigate potential material interactions and joint integrity.

In addition to evaluating a specific technology development component, a second aspect of this investigation concentrated on the evaluation of joining options for several designs of the power conversion system hot-side heat exchanger (HX) components. Dissimilar metal joints are required to impart both mechanical strength and thermal path integrity for a FPS-type heater head. Type 316L stainless steel is the expected containment material for the flowing liquid metal NaK, but does not have the mechanical strength desired for a thin-wall pressure vessel. Inconel 718 (IN718) is a stronger alloy that has been used in previous heater head designs, but would need to be bonded to stainless steel to maintain a mono-metallic NaK flow loop. Either heater head envelop material probably would be bonded to a Cu heat acceptor to increase the surface area in contact with flowing He. This paper reports on the initial evaluation of nickel-based, gold-based and copper-based braze materials to join the various combinations of these component materials options.

Materials Inspection of NaK Heat Exchanger

Examination of the Dome Section

The integrated liquid metal heat exchanger / P2A heater head / convertor configuration for a single convertor is shown in Figure 1 (Briggs, Geng, and Robbie, 2010). The original fabrication design for the experimental stainless steel heater head called for the various joints to be fabricated by electron beam (EB) welding. However, due to some fabrication issues and deadlines, most welds of the component were fabricated by gas tungsten arc welding (GTAW). Figure 1(b) shows the welded sections investigated after the NaK exposure. The initial sectioning for inspection of the heater head unit after the NaK exposure was done by wire electron discharge machining (EDM). The inlet and outlet pipes (cuts #1 and #2) were removed initially (Fig. 2(a)). The second major sectioning (cut #3) was done by splitting in half the main dome of the heat exchanger head (Fig 2(b)).



Figure 1.—(a) Experimental NaK heater head installed on 1-kWe Stirling power convertor. (b) NaK heater head welded sections investigated.



Figure 2.—Initial sectioning by wire EDM of the NaK heat exchanger.



Figure 3.—(a) One-half section of the heater head showing assembly defects observed at the jacket loop/outlet pipe welding joint. (b) View of the gap left between the pipe insert and the heater head loop.

The first noticeable shortcoming was observed at the joining section where the outlet pipe connects with the jacket loop of the heater head as observed in the one-half section of the heater head (Fig. 3(a)). A gap (Fig. 3(b)) is clearly noticeable near the joint between the insert and the jacket wall. It is possible that the insert, used to connect the pipe to the jacket, may have pressed and deformed the jacket wall, or perhaps the TIG welding operation may have distorted the thin jacket wall. This gap could trap the liquid metal and possibly create a crevice corrosion issue. "Metal bubbles", sometimes described as nuggets, created during the welding operation, can also be observed in the interior of the jacket wall. Figure 4 shows more detail of the metal bubbling features and the gap left between the insert and the wall jacket.

A different fabrication situation occurred at the joining section where the inlet pipe connects to the heater head dome (Fig. 5). In this location, the pipe insert extended 1 to 2 mm beyond the head shell dome; this extension of the inlet pipe into the open section of the heat exchanger dome might create a flow disturbance in the liquid metal during operation, which could affect the heat transfer efficiency.



Figure 4.—Different view of the welding defects observed at the loop/outlet pipe joint. "metal bubbles" (nuggets) left from the welding process and open gap are visible.



Figure 5.—NaK heat exchanger used for the 1 kWe Stirling test at MSFC showing the pipe insert extending 1 or 2 mm into the dome channel.

Examination of the Internal Surfaces of the Connecting Pipes

Figure 6 shows an incomplete filled butt weld joint clearly visible by the presence of a groove or channel inside the pipe. Localized internal oxidation near the heat affected zone (Fig. 6), revealed by the change of surface coloration, will be discussed later in detail. Hot spot metal nuggets that can disturb the liquid metal flow are also observed in some regions of the butt weld.

Imaging and measurement of the groove depth observed in these joints is reported and presented in Figure 7. The depth of the groove/channel is approximately $110 \mu m$ and the width near the pipe surface is approximately $300 \mu m$.

A socket type weld which corresponds to an overlap of a pipe with an elbow, as shown in Figure 8, has left a step and an opening under the step, where liquid metal could penetrate and get trapped producing a situation of localized or crevice corrosion. If this overlap assembly is needed, a second weld should be made at end of the step to eliminate the possibility of any liquid metal concentration in the overlap region.



Figure 6.—Internal view of connecting pipe butt weld showing incomplete filling of the joint and the presence of hot spot metal nuggets on the inside surface of the pipe.



Figure 7.—Imaging and measurement of the groove/channel depth observed in the butt weld after GTAW.



Figure 8.—Section of the socket type weld that may require a second weld to eliminate the possibility of trapping liquid metal between the pipe and elbow walls.

It was also noted that the welding of the support plates (Fig. 9), used to keep in place the pipes with respect to the heater head, has penetrated inside the pipe, leaving a slightly bulging surface on the internal surface of the pipe. These bulged surfaces might create non-desirable flow turbulence.

Electron microscopy imaging and chemical analyses of regions near some of the incomplete filled welds revealed the presence of various oxide scales (Fig. 10) with varying degrees of detectable Na concentration. The Energy Dispersive Spectrum (EDS) presented in Figure 11, clearly reveals the presence of oxygen O and Na in the vicinity of the groove/channel (Fig. 10). Semi quantitative chemical spot analyses in a region near the butt welded groove/channel revealed consistently the presence of Na in the oxidized region.



Figure 9.—The welding of support plates for the pipes produced bulging surfaces inside the pipe.



Figure 10.—Backscattered electron microcopy image showing the groove, a partial view of a nugget and oxide scales that formed in the vicinity of the butt weld. Area used for the Energy Dispersive Spectroscopy (EDS) analysis presented in Figure 11 is indicated.



Figure 11.—EDS chemical analysis of the oxide scale of region indicated in Figure 10, showing the presence of O, Na, Fe, and Cr.

Figure 12 shows a field emission scanning electron microscopy (FESEM) image of a polished cross section of a region near the butt weld joint where a $\sim 8 \mu m$ thick complex oxide scale is revealed. EDS analyses (Fig. 13) indicates the presence of higher levels of Na, Cr, and O in the dark phase compared to the white phase which seems to be enriched in Fe and Ni and, to a lesser degree, in Na. During the GTAW, the welded area (external region of the tubes) was protected from the environment by a shield of inert gas (normally argon). Most likely, the inside of the tubes during welding did not benefit from the outside gas shielding which resulted in a heat affected zone that oxidized during the joining operation. The presence of oxygen in liquid metals has been the subject of much research in the past (Hoffman, 1967; Zimmerman, 1965; Hiltz, 1970). For many elements, the corrosion rate increases with the quantity of oxygen present in the alkali metal, and corrosion may be thought of as the interaction of the metal alkali oxide (i.e., Na₂O) with the metal. In this fabrication assembly, oxide scales only formed in regions at or near the welds. No oxide scales were observed in regions away from the welded joints. Therefore, as NaK was introduced to the system, the oxide scales that formed in the vicinity of welds reacted with the flowing NaK, probably resulting in the formation of complex oxides such as NaCrO₂ and Na₄FeO₃ (Barker and Wood, 1971).



Figure 12.—(a) Low and (b) High magnification FESEM cross section images showing the complex oxide scale that formed near a butt weld joint.



Figure 13.—EDS analyses of the complex oxide scale phases that formed on the surface of the stainless steel tube.

Brazing of Dissimilar Metals

A successful dissimilar-metal joint in this component application must provide mechanical integrity, thermal conductivity, and stability for the expected mission life at elevated temperatures. Brazing is frequently a good approach for joining dissimilar metal in net-shape or near-net-shape configurations. Following is a summary of ongoing research on using commercially available braze alloys to join the various dissimilar metals that might be part of the heat exchanger. This summary focuses on down-selecting candidate alloys based on physical integrity and chemical interactions. Future results will further discuss long-term stability. Joint configurations were selected based on expected heater head designs. For example one possible design, as shown in Figure 14, would require a joint between components made of SS316L and IN718, and a secondary joint between IN718 and copper. A different design would require a good bond between SS316L steel and copper.

Nickel- and gold-based braze alloys (Table 1) were selected to join the stainless steel and the Inconel 718 superalloy in response to the first heater head design requirement; a second group of brazes (Table 2) were selected to join the type SS316L stainless steel to oxygen free high conductivity (OFHC) copper simulating an internal acceptor. Microstructural and mechanical properties results indicated that gold-based brazes resulted in minimal reaction and property degradation with either base metal SS316L or IN718 (Locci, Bowman, and Gabb, 2009). The phase reactions were observed to be more extensive in all the Ni-based brazes that were studied.

The initial brazing work was done using a small brazing furnace at GRC. This furnace was used to define the brazing parameters for the initial trials. Various exploratory runs were done simultaneously at the American Brazing Company (Willoughby, Ohio), using one of their larger brazing furnaces. This study considered braze alloys in paste, tape, and foil form. Figure 15 shows examples of ring segments prepared with paste and foil braze material. Material coefficients of expansion and braze reservoirs must also be considered in the design. For example, a foil can be pre-placed between the faying surfaces prior to the initiation of the heating cycle if a gap exist or can be machined. Extra foil (Fig. 15(b)), extending above the rings, might need to be added to fill completely the gap that will be created at the brazing temperature between the alloys.



Figure 14.—Possible liquid metal heat exchanger assembly design.

Brazes	Composition (wt.%)	Form	Braze Temp (K)
AMDRY 790	Ni-1.74B-3.22Si	Paste	1403
AMDRY 108	Ni-23Cr-11.5Fe-4.2P-6.4Si	Paste	1403
AMDRY 775	Ni-15.38Cr-3.8B	Paste	1403
Nioro	82Au-18Ni	Foil (0.0254)	1258 or 1273
Palniro-7	70Au-22Ni-8Pd	Foil (0.0254)	1323

TABLE 1.—BRAZE ALLOYS EVALUATED TO JOIN SS316L AND IN718

TABLE 2.—BRAZE ALLOYS EVALUATED TO JOIN SS316L AND CU

Alloy/Braze	Composition (wt.%)	Form (mm)	Braze Temp (K)
AMDRY 936	Ni-19Mn-4Cu-6Si-1B	Paste	1270
AMDRY 930	Ni-23Mn-5Cu-6Si	Таре	1311 or 1293
AWS-BNi-2	Ni-7Cr-3Fe-3.2B-4.5Si-0.06C	Foil (0.0508)	1311
Nioro	82Au-18Ni	Foil (0.0254)	1253
35%Au-65%Cu	Au 34.70-Cu 65.30	Foil (0.0254)	1293
40%Au-60%Cu	39.58Au-60.42Cu	Foil (0.0254)	1283
50%Au-50%Cu	49.67Au-50.33Cu	Foil (0.0254)	1253



Figure 15.—(a) Nioro paste applied to chamfer sections of three concentric rings (SS316L, IN718, and Cu) and (b) Nioro braze foil inserted in the gap left between the two concentric rings (SS316L and IN718), extra foil extends beyond height of the metal rings.

Another braze fabrication comparison used Nioro foil and paste to join the IN718 ring (outside ring) to SS316L (inside ring) or SS316L (outside ring) to IN718 (inside ring) as unveiled in Figure 16(a) and (b). In some instances, as contrasted in Figure 16(c) and (d), small unfilled gaps can be observed on some cross sections at the top of the rings, where the paste braze filler was originally applied. This gap may be the result of volume lost as binders are burnt out of the braze paste. Re-brazing, to fill completely leftover gaps, is an option and a common practice in the brazing industry. Higher magnification images of a sound Nioro-based joint with minimum chemical reaction are presented in Figure 17. Low and high magnification field emission scanning electron microscopy (FESEM) backscattered images, unveiled the complete braze coverage and the resultant microstructure after brazing for 10 min at 1273 K (1832 °F) in vacuum. An example of a poor braze bond is shown in Figure 18, where again Nioro was used to bond SS316L to OFHC copper, which resulted in limited wetting of the copper surface and discontinuous bonding.



Figure 16.—Examples of foil (a) and paste filler (b) Materials used to braze dissimilar alloys; transverse cross sections contrasting completely filled brazed region (c) Versus a region revealing an unfilled gap (d) at the top of the ring where the braze was originally dispensed.







Figure 18.—Stainless steel brazed to copper using Nioro braze alloy showing poor bonding.

Another example of reactions between the elements present in the braze alloy, in this case AMDRY 936, and two of the base metals, SS316L and Cu, is visible in the FESEM cross section image presented in Figure 19. On the stainless steel side (box A), grain boundaries are visible in the microstructure due to the precipitation of chrome borides. The tying of the Cr into borides can cause the localized depletion of Cr in the nearby regions of the alloy and change locally the corrosion resistance property of the stainless steel. Boron was present in the braze alloy, while Cr is available from the stainless steel. On the copper side and within 60 µm of the interface, multiple micron size Mn-Cu rich phases can be observed (box B). The Mn is present in the braze alloy, and since copper and manganese form a complete solid-state solution, diffusion of Mn during the brazing process must have occurred to form the fine particulates. The brazed section contains eutectic-like regions where one of the solid-state solution regions (dark gray) is enriched mainly in Ni, Si and Mn, while the other region (light gray) is mainly enriched in Cu and to lesser degree with Ni. The use of a different braze, AMDRY 930, that does not contain B, resulted in no Cr rich boride phase formation near the braze/stainless steel interface. However, Mn-enriched particles did form at the braze/copper interface as observed when using AMDRY 936.

A group of gold-copper brazes were explored as an option to join the stainless steel component to the OFHC copper. The three braze alloys were selected to minimize possible reactions with the components to be joined and also because the different levels of Au dictated their melting points and therefore, their brazing temperatures. All the three brazes wetted and spread evenly on both alloys which resulted in a good bond. Further study on the stability of these brazes is recommended if these alloys may be considered to join type 316L stainless steel to copper. Table 3 summarizes the brazing results for the multiple systems studied.



Figure 19.—Type 316L stainless steel brazed to copper using AMDRY 936 braze alloy showing reactions at or near brazed interfaces.

Metal 1	Metal 2	Braze Alloy	Wetting	Phase Formation w/ Metal 1	Phase Formation w/Metal 2	Compatibility with Base Metals	Recommended
SS316L	IN718	AMDRY 790	Good	Cr-Boride at G. Bds	Nb/Mo at G. Bds	Reactions at Braze/Metal Interfaces	No
SS316L	IN718	AMDRY 108	Good	minimal	Nb or/and P at G. Bds	Reactions at Braze/Metal Interface	Probably No
SS316L	IN718	AMDRY 775	Good	Cr-Boride at G. Bds	Nb/Mo at G. Bds	Reactions at Braze/Metal Interfaces	No
SS316L	IN718	Nioro	Good	minimal	minimal	OK	Yes
SS316L	IN718	Palniro 7	Good	minimal	minimal	OK	Yes
SS316L	Cu	AMDRY 936	Good	Cr-Boride at G. Bds	Mn-Cu	Reactions at Braze/Metal Interfaces	Probably No
SS316L	Cu	AMDRY 930	Good	minimal	Mn-Cu	ОК	Yes
SS316L	Cu	AWS-BNi-2	Good	Cr-Boride at G. Bds	Cr-Silicide	Reactions at Braze/Metal Interfaces	No
SS316L	Cu	Nioro	Poor	-	-	-	No
SS316L	Cu	35Au-65Cu	Good	minimal	minimal	Probably OK	More Research
SS316L	Cu	40Au-60Cu	Good	minimal	minimal	Probably OK	More Research
SS316L	Cu	50Au-50Cu	Good	minimal	minimal	Probably OK	More Research

TABLE 3.—SUMMARY OF BRAZING RESULTS

Conclusions

The NaK heat exchangers fabricated for the integrated Stirling convertor with pumped NaK loop testing performed as expected for the 66 hr test program and met all the experimental objectives. However, inspection of the NaK heater head revealed several fabrication and design shortcomings that could be problematic in longer-term testing. Electron microscopy analyses revealed the presence of oxidized regions with detectable levels of Na near the incompletely filled butt-weld joints. No other material interactions in the heat exchanger assembly with the NaK liquid metal were observed after the 66-hr test at a maximum temperature of 823 K. Inert gas shielding was probably not applied inside the tubes during the GTAW joining operation which resulted in an internal oxidized heat affected zone. If GTAW is the joining process selected, a shielding cover gas should be applied inside the tubes during the joining operation to minimize any internal oxidation. Other joining techniques such as electron beam or laser welding done under argon, should be explored to minimize oxidation and/or to eliminate the partial filling of the welds. It is recommended that like-components are inspected after the fabrication or joining of the heat exchanger components. Inspection of the welds by a non-destructive technique is also encouraged.

NASA GRC, collaboratively with industry, examined paste, tape, and foil brazing materials. Procedures and brazing methods were developed to join various samples and prototypical Stirling heat exchanger concentric ring configurations. The results of these examinations are summarized in Table 3. Based on the lowest impact on the base metal strength and the minimal chemical interaction, Nioro is the leading braze candidate to join the two dissimilar metals, stainless steel and Inconel 718. Careful metallurgical evaluation of brazed joints between 316L stainless and copper with B containing Ni-based alloys revealed varying degrees of chemical interactions. Some of these interactions, such as the formation of chromium boride precipitates, could affect the long term structural stability of the stainless steel. Based on these results, brazes that do not contain B and the copper-gold braze alloys should be explored further to join stainless steel to copper components.

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