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Building, Testing, and Post Test Analysis of Durability Heat Pipe #6

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Timothy A. Moss
Solar Thermal Technology

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Abstract

The Solar Thermal Program at Sandia supports work developing dish/Stirling systems to convert solar energy into electricity. Heat pipe technology is ideal for transferring the energy of concentrated sunlight from the parabolic dish concentrators to the Stirling engine heat tubes. Heat pipes can absorb the solar energy at non-uniform flux distributions and release this energy to the Stirling engine heater tubes at a very uniform flux distribution thus decoupling the design of the engine heater head from the solar absorber. The most important part of a heat pipe is the wick, which transports the sodium over the heated surface area. Bench scale heat pipes were designed and built to more economically, both in time and money, test different wicks and cleaning procedures. This report covers the building, testing, and post-test analysis of the sixth in a series of bench scale heat pipes.

Durability heat pipe #6 was built and tested to determine the effects of a high temperature bakeout, 950°C, on wick corrosion during long-term operation. Previous tests showed high levels of corrosion with low temperature bakeouts (650-700°C). Durability heat pipe #5 had a high temperature bakeout and reflux cleaning and showed low levels of wick corrosion after long-term operation. After testing durability heat pipe #6 for 5,003 hours at an operating temperature of 750°C, it showed low levels of wick corrosion. This test shows a high temperature bakeout alone will significantly reduce wick corrosion without the need for costly and time consuming reflux cleaning.

Acknowledgements

The heat pipe program at Sandia required a team effort. The author wishes to thank the many contributors to its success both in the program and for this test. I am but a cog in a wheel making this effort happen.

Scott Rawlinson did thermal stress analysis on the durability heat pipe design for the long term testing, the high temperature bakeout, and the stresses encountered during different warm-up strategies.

Patricia Cordeiro and Jim Grossman put together the data acquisition system and wrote the program for this test. The many people at the NSTTF who put together the test cells that were used for the first time, J. Kelton, Daniel Ray, and K. Chisman to name a few. Daniel Ray also did all the welding of the heat pipe and assisted in its bakeout as well as the post-test removal of the sodium from the heat pipe.

Chuck Andraka, being the project leader, for his assistance in writing the software, the data downloading and plotting, and for his general stewardship of the heat pipe program.

Steve Showalter for all his previous work on metal felt wicks determining the sintering time and temperature and the type of wick to use.

Jim Moreno for his invaluable technical assistance.

Considerable post test analysis support was given by Gary Zender and Paul Hlava in SEM pictures and electron microprobe analysis.

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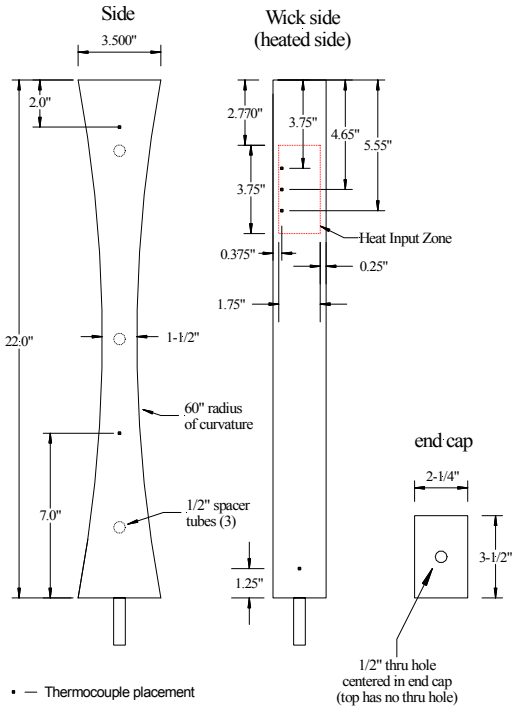
Introduction

Durability heat pipe number 6 was the sixth heat pipe following the durability bench test heat pipe design. A sketch of the heat pipe design is shown below in figure 1. Previous tests showed a significant reduction of wick contamination and corrosion when the heat pipe was baked out at a high temperature and reflux cleaned with sodium prior to testing. The objective of this test was to determine the effect of a high temperature bakeout alone on felt wick contamination and corrosion after 5,000 hours of operation at 750ºC.



The particulars of the heat pipe are:

Wick: 3 layers of 316LSS Bekaert 8/300 felt (8µm fiber diameter) compressed and sintered to 4.5 mm thickness

Perf-metal: 0.035" 304 SS with 60% open area

Wick substrate: 0.065" Haynes alloy 230

Side walls: 0.065" Haynes alloy 230

End caps: 0.065" Haynes alloy 230

Process connection: ½" Haynes alloy 230 tube 0.049" wall for both vacuum and sodium filling

Valve: SS-8U-TW-HT

Sodium: 500 cc from lot #25109-S

Figure 1: Drawing of the durability heat pipe #6

Heat pipe assembly

A felt wick consisting of 3 layers of 316LSS fiber manufactured by Bekaert Fibre Technologies (Marietta, Ga) as Bekipor® WB 8/300, where 8 stands for fiber diameter (8 µm) and the 300 refers to the mass of one square meter of the material (300 grams/m^2), 2" wide and 22⅛" long was sintered on the two curved sides of the heat pipe opposite each other. To keep the felt wick from moving during initial vacuum pumping in the vacuum furnace for sintering Krylon 1301 clear paint was sprayed on the substrate and between each layer of felt. The clear paint acts as an adhesive but burns away during sintering without leaving a residue. Both felt wicks had a perf-metal overlay. The intent of having the felt wick sintered to the perf-metal is to prevent the wick from compressing over time during use. Previous tests showed a felt wick made from Bekaert 8/300 felt compresses during testing due to the capillary pressure of liquid sodium. The felt wicks with the perf-metal overlay were sintered in a vacuum furnace for one hour at 1150ºC. During sintering neither felt wick sintered to the perf-metal along the entire length, particularly at the top end. It was felt that this was not important since it did not affect the objective of the test. The wick with the best perf-metal/wick bond was used as the heat input side. The wick was

kept 1/8" away from the edges of the substrate to prevent any interference with the fit-up and prevent any of the wick from burning during welding of the heat pipe.

Before any welding all the edges of the heat pipe parts were sanded with emery cloth to remove any contamination. The parts were cut from sheet stock with an EDM machine using brass wire. Copper, from the brass, is left behind along the cuts. If the copper is not removed the welds will have cracks and will not be leak tight. The heat pipe was welded using a TIG welder and 0.035" Haynes alloy 230 filler rod while maintaining an Argon purge. The ½" tubing was welded on from the inside of the end cap. The three ½" 316SS spacer tubes are tack welded on the inside of one of the hourglass shaped sides. The end caps were then welded on to the two hourglass shaped sides. The two curved sides with the wick sintered on are the last to be welded on. The spacer tubes are to prevent the sides from collapsing in while the two curved sides with the wicks were welded on. Care was taken to insure the welds were close to full penetration as possible, even though none of the individual weld areas were machined with bevels to aid in full penetration welds.

Heat Pipe Bakeout

Before bakeout, the heat pipe along with the fill can, the sodium can, and the interconnecting tubing were evacuated using a Balzers 60 ℓ/sec turbo-molecular vacuum pumping station to mid 10^{-5} Torr or less and leak checked using a VG Micromass 386 quadrupole residual gas analyzer (RGA) and spraying helium along the outside of the heat pipe and all vacuum connections. The schematic for the vacuum system is shown in figure 2. It was determined to be leak tight when no leaks greater than $8x10^{-6}$ Pa ℓ/s ($6x10^{-8}$ Torr ℓ/s) were found (based on an assumed pumping speed of 60 ℓ/sec on the quadrupole and a minimum RGA detection limit of $1.0x10^{-9}$ Torr). The heat pipe was heated using an oven made by Zircar Ceramics in Florida, NY consisting of two half cylinders with an inside diameter of 7.5" made with refractory ceramic fibers with imbedded wire resistance elements. Everything else was outside the oven. The interconnecting tubing between the vacuum pump, the fill can, and the sodium can were heat traced with cloth heaters and temperature controlled to 200ºC. The fill can and the ½" tubing from the fill can to the heat pipe, including the ½" valve on the heat pipe, were heat traced with In600 sheathed heaters and temperature controlled to 700ºC. The heat pipe and all interconnecting tubing were pumped on overnight before any heaters were turned on. The following day the ½" valve on the heat pipe was closed and all the heaters, except those for the heat pipe and the sodium container, were turned on. These heaters were left on for approximately 24 hours to insure the majority of the gasses being driven off were going to be pumped out rather than going into the heat pipe. After the 24 hour period, the ½" valve on the heat pipe was opened and the oven was turned on and initially set for 200ºC for two reasons. The first was to let the oven stabilize at a lower temperature since the oven heaters heat up much faster than the heat pipe and this allows the heat pipe to catch up to the oven at a lower temperature. The second reason is to keep the vacuum pressure at the RGA below 10^{-2} Torr. With a 60 ℓ/sec vacuum pump, four ½" valves, and about three feet of ½" tubing to pump through, the pumping speed on the heat pipe is not very high so the pressure inside the heat pipe is much higher than the indicated pressure at the RGA. At these temperatures water vapor is the main gas, by far, being outgassed from the internal surface areas. With two sides of the heat pipe having a metal felt wick there is a lot of internal surface area and, therefore, a lot of water vapor is being outgassed. Time is needed to pump out the water vapor while maintaining a good vacuum so as not to cause contamination with the materials inside.

The oven was held at 200ºC for about ½ hour and then allowed to reach 950ºC using an on/off temperature controller and held there for two hours. After two hours at 950ºC the total pressure gauge started decreasing in pressure after remaining fairly constant since first reaching 950ºC. At this point it was decided most of the outgassing had occurred and more time at this temperature was no longer necessary.

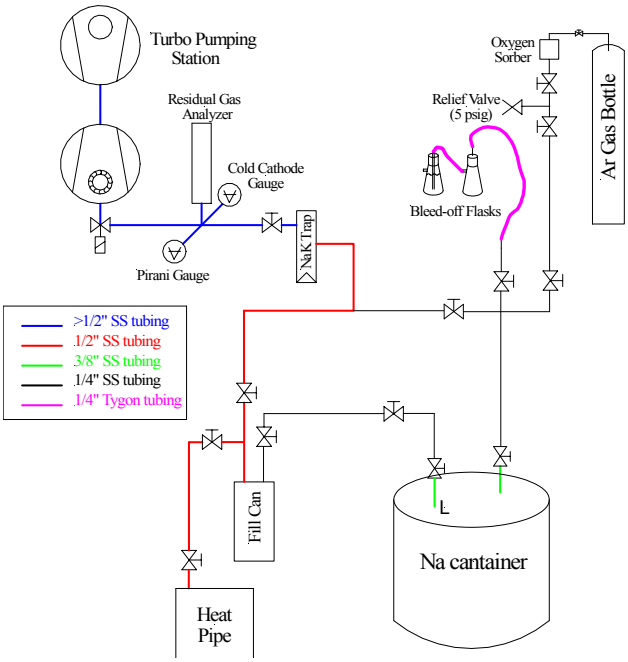


Figure 2: Schematic of the vacuum system to bakeout and fill of durability heat pipe #6

Filling the Heat Pipe With Sodium

After two hours of bakeout of the heat pipe at 950ºC, the drain can and the heat pipe are allowed to cool down to 300ºC. All heaters on the interconnecting tubing are turned off except between the sodium container and the drain can. After the heat pipe and drain can have cooled down to 300ºC all the valves are closed. Before transferring any sodium, the sodium container was heated to 150ºC for 24 hours to ensure a standard, or consistent, distribution of contaminates in the sodium. The most important of these contaminates is the level of dissolved oxygen in the sodium. This is to make sure the sodium is not getting either purer or more contaminated as the sodium is removed from the can for each fill operation. Following the SOP for transferring sodium, the sodium is transferred from the sodium container, lot #251079-S, to the fill can. The fill can is heated to a higher temperature than the sodium container to know when the sodium starts to fill the fill can by when its temperature drops due to the cooler sodium. This is the only way to know that the interconnecting tubing and valves are open and hot enough to allow sodium to transfer from the sodium container and the fill can. The fill can was sized to hold 500 cm^3 of liquid sodium, which will create about a 2½" pool depth with both wicks fully saturated.

The sodium was transferred from the fill can to the heat pipe by heating the fill can to 600ºC and the heat pipe to 300ºC and evaporating the sodium into the heat pipe. The orientation of the heat pipe and drain can was such that no liquid could move between the drain can and heat pipe. The sodium is evaporated into the heat pipe to ensure the highest possible purity of sodium going into

the heat pipe by leaving behind contaminates, such as sodium oxide, that may have formed during the filling process or was present as an impurity in the sodium from the container.

After the heat pipe was filled with sodium all the heaters were turned off and the heat pipe and its ½" valve were cooled to room temperature. After it had cooled, the fill can was removed. The heat pipe was wrapped with heaters and insulation and was laid down flat on the floor. It was heated to 600ºC for two hours so the sodium could wet the felt wick. The sodium has to remove the oxide layer on the felt wick fibers for the sodium to wet it. This is standard procedure for all heat pipes with felt wicks and has proven to be reliable.

Testing of the Heat Pipe

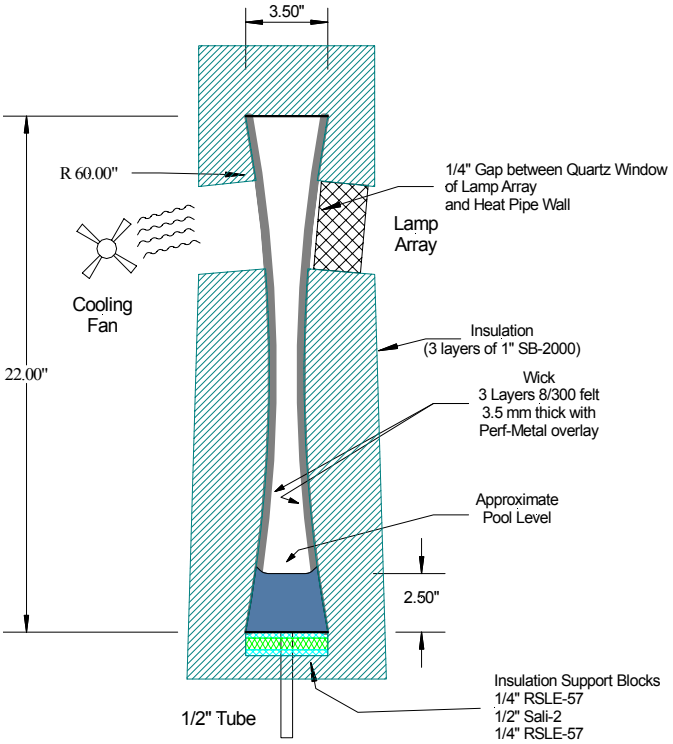


Figure 3: Durability heat pipe #6 test setup schematic

The heat pipe had 11 type K thermocouples spot-welded on the outside of the heat pipe to monitor temperature during testing. The thermocouple placement is also shown in figure 1. Figure 3 is a schematic of the durability heat pipe test system. The three thermocouples in the heat input zone are intrinsics to gain a better response time and surface temperature measurement, which is important in detecting a wick dry-out before irreversible damage is done to the heat pipe wall. All other thermocouples are 1/16" In600 sheathed thermocouples spot-welded on using 0.002" SS shim stock.

The heat pipe is mounted in a test stand with the ½"valve on the bottom, as shown in figure 3. The heat pipe is supported by resting the bottom end cap on a uni-strut angle bracket. To keep heat transfer between the heat pipe and the angle bracket to a minimum there is a ½" thick piece of Sali-2 with a piece of ¼" thick RSLE-57 on either side of it placed between the heat pipe and the angle bracket. The heat pipe is insulated with three layers of 1" thick SB-2000 insulation with aluminum foil between each layer, with aluminum foil being the last layer. Sali-2, RSLE-57 and SB-2000 are all products of Zircar Ceramics in Florida, NY. This type of insulation is fairly transparent in the infrared and the aluminum acts as a reflector for the infrared. A hole in the insulation is cut opposite the heat input area to act as the condenser area to release heat. The size of the hole is approximately the size of the heat input zone but it will vary depending on how well the heat pipe is insulated. If it is very well insulated with few air leaks the size of the opening would be increased. If the insulation is a little looser and has a few more air leaks than the size will be smaller.

The heat pipe was heated using a Multi-Zone load test heater model 5075 lamp array made by Research Inc. in Minneapolis, MN. This heater uses six 1000 watt quartz lamps to generate an average absorbed flux of about 60 watts/cm^2 over an area of 9.52 cm x 4.44 cm (3¾" x 1¾") for an approximate throughput of 2,500 watts. A small fan is positioned to blow directly into the hole created in the insulation for cooling. The size of the hole is determined such that with the fan on and the lamps at 5/6th full power the heat pipe maintains its operating temperature of 750ºC, as measured by the top thermocouple on the condenser side, which is above the hole and well insulated. The fan turns on only when the heat pipe is within 10ºC of the operating temperature and all the lamps are on. The control software can detect when a bulb burns out. If a bulb burnout is detected, the fan will not turn on and the remaining bulbs are operated at full power. This way the heat pipe will still maintain a 750ºC operating temperature so the test can then continue without interruption and it allows the bulb to be replaced at a convenient time.

To begin the testing, the heat pipe is initially heated using two ⅛" In600 metal sheathed heaters 62" long (Aerorod BXX heaters) that are spot welded to the hour glass shaped sides of the heat pipe. It was found that heating the heat pipe from a cold start using only the lamp array caused very high thermal stress between the side being heated and the sides that are still cool. The lamps first turn on when the lowest measured temperature is above 125ºC and then only at the lowest power level. When the lowest measured temperature is above 450ºC the lamp array is ramped up to full power. The metal-sheathed heaters are turned off when the lowest measured temperature is above 600ºC. The lamp array's power level is controlled to maintain the set operating temperature of 750ºC as measured by the top thermocouple on the condenser side. Typically the intrinsic thermocouples, which are in the heat input zone, are about 30ºC higher than the control thermocouple. Figure 4 shows a plot of a typical startup.

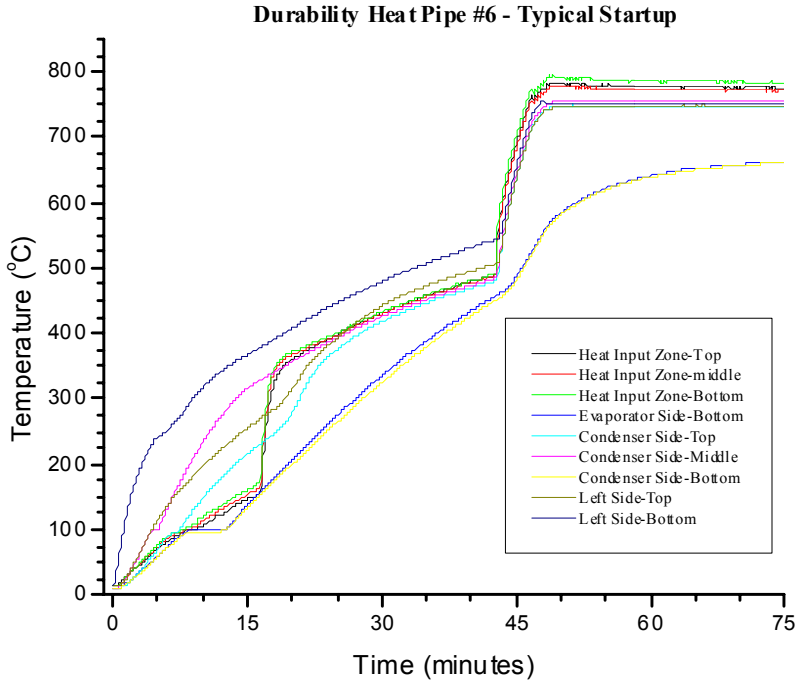


Figure 4: A plot showing a typical startup of the durability heat pipe #6. Notice the sharp increase in the heat input zone thermocouples at about 16 minutes when the lamps are first turned on at the lowest power setting and the second one after about 44 minutes when the lamps are fully turned on.

The heat pipe successfully completed 5,003.9 hours of testing at the operating temperature of 750ºC between 6/9/2000 and 4/23/2001. There were 14 cold restarts (heat pipe was less than 200ºC) and 3 hot restarts (heat pipe >300ºC) during the testing period. Most of the cold restarts were caused by power outages. X-rays were taken of the top third of the heat pipe after 286 and 1085 hours of operation, as shown in figure 5. Notice the wick compression on the right side of each image shown in figure 4. The x-rays taken after 1085 hours of operation showed the wick was compressing and detaching from the perf-metal farther down over time. But as mentioned earlier, this was not a concern since the test was to determine the effects of a high temperature bakeout for reducing wick corrosion and not wick adherence to the perf-metal

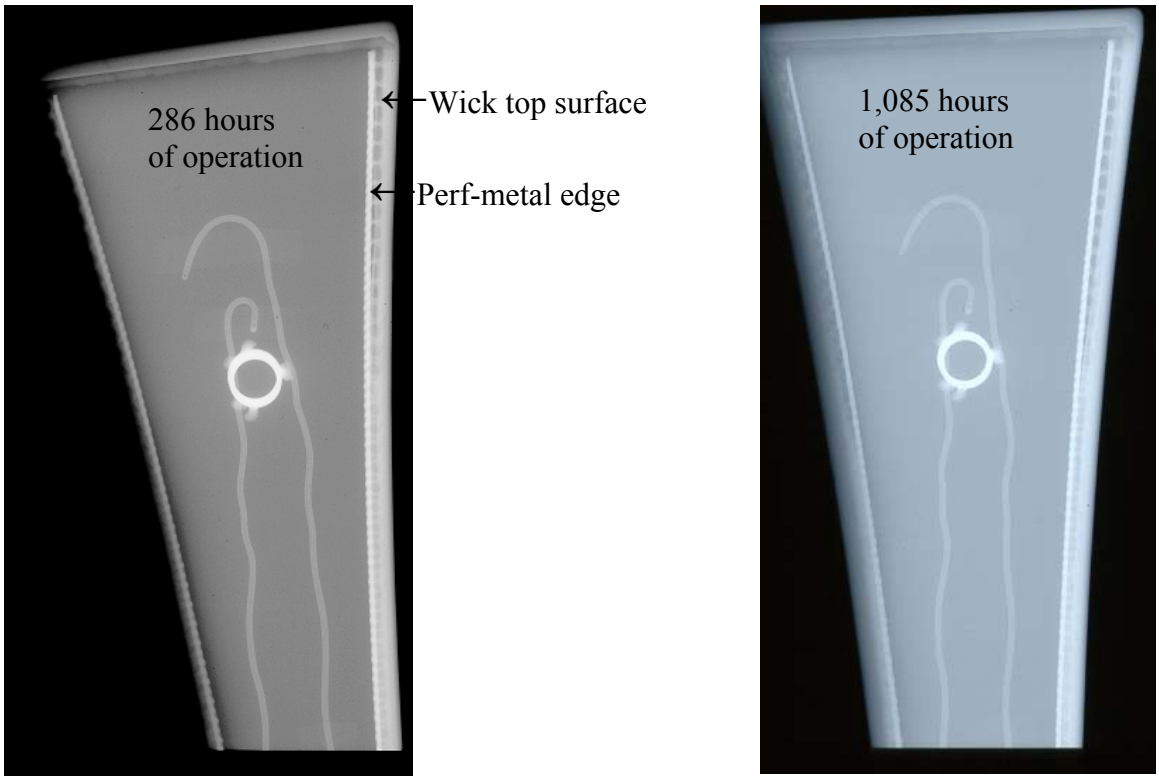


Figure 5: Two X-ray images of the top section of durability heat pipe #6, the left one after 286 hours and the right one after 1085 hours of operation. The two fishhook like objects in each picture are the metal-sheathed thermocouples spot-welded to the flat hourglass shaped sides. The round object in the center of each is the ½" tube welded in the inside to prevent the heat pipe from collapsing during welding. For both pictures the evaporator side is the right side of the heat pipe.

Post-Test Analysis of the Heat Pipe

After completing the testing, the heat pipe was removed from the test stand and a drain can was welded onto the heat pipe at the 1/2" valve. The heat pipe is put in a vertical position with the drain can at the bottom. The drain can was evacuated and baked out at 600ºC for two hours to remove any contaminates. After the two hour bakeout all valves were closed and the drain can was allowed to cool to 150ºC. The heat pipe was then heated to 700ºC to evaporate the sodium

out of the heat pipe into the drain can. When the heat pipe temperature reached 200ºC as it was heating up, the ½"valve between the drain can and the heat pipe was opened and the liquid sodium pool was drained into the drain can. The temperature of the drain can was monitored to determine if the connecting ½" tubing and valve were not plugged by a slug of solid sodium. When the liquid first entered the drain can it warmed it up since the liquid temperature was higher than the can temperature. When the temperature of the heat pipe went above 450ºC the drain can temperature increased again because the sodium vapor was condensing in the drain can. A good estimate of when most of the sodium has been removed from the heat pipe into the drain can is when the heaters are needed to maintain the temperature of the drain can since the condensation has ended. When the heaters were needed to maintain the temperature of the drain can, the heat pipe was maintained at 700ºC for another 2 hours to make sure all the sodium had been removed. After these two hours the heaters are turned off and the ½" valve on the heat pipe was closed. When the heat pipe and drain can are cooled down to near room temperature the insulation is removed. The drain can is lightly tapped to get an approximate idea of how much sodium is in the drain can. This is done to roughly verify all the sodium has, indeed, been removed from the heat pipe. The ½" valve is then cut off the heat pipe.

The heat pipe was cut open using a slit cutter with no lubricants in Sandia's machine shop. The cut was made down the center of the hourglass shaped sides lengthwise and through the end caps to preserve the two sides with the wicks. No lubricant was used as a safety measure in case sodium was still present in the heat pipe and also not to contaminate the wick on the two curved surfaces. Figure 6 shows a picture of the two halves of the heat pipe after being cut open. In figure 6, the bottom half is the evaporator side and the left side corresponds to the top. The area of discoloration on the left side bottom half is the heat input zone and is caused by contamination. This is the only area showing visible signs of contamination.

Figure 6: Picture of the two halves of durability heat pipe #6 just after being cut open. The left side is the top and the bottom halve is the evaporator side. The discolored area on the left side bottom half is the heat input zone.

After the heat pipe was cut open wick thickness measurements were taken down the center of the wick along the full length of both the evaporator and condenser wicks. The results are shown in figure 7. Note how the wick is compressed in the heat input zone. Both wicks were also completely separated from the perf metal starting from the top to about the middle of the heat pipe.

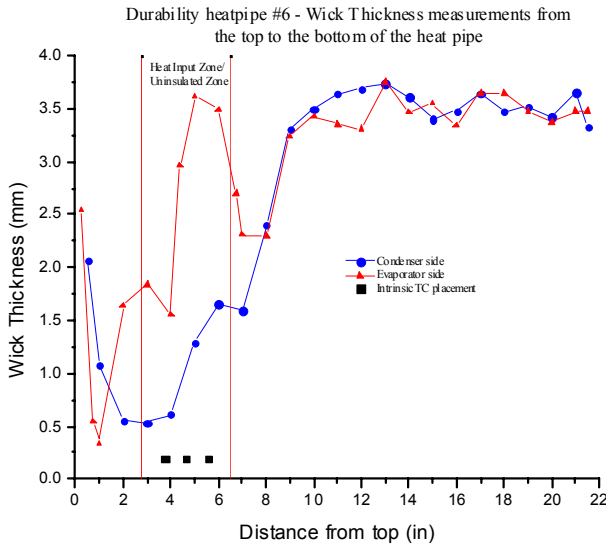


Figure 7: Graph showing the wick thickness of both wicks along the length of the heat pipe

Sections of the wick and substrate at the top, middle, and pool locations were cut out using a dremel tool and ceramic cut-off wheels. The location of these samples taken out of the heat pipe is shown in figure 8. These samples were sent to Gary Zender, org. 1822, for SEM pictures of the wick samples, which are shown in appendix A. These pictures show very little contamination and very little corrosion of the fibers. For comparison, samples of as received felt were also looked at.

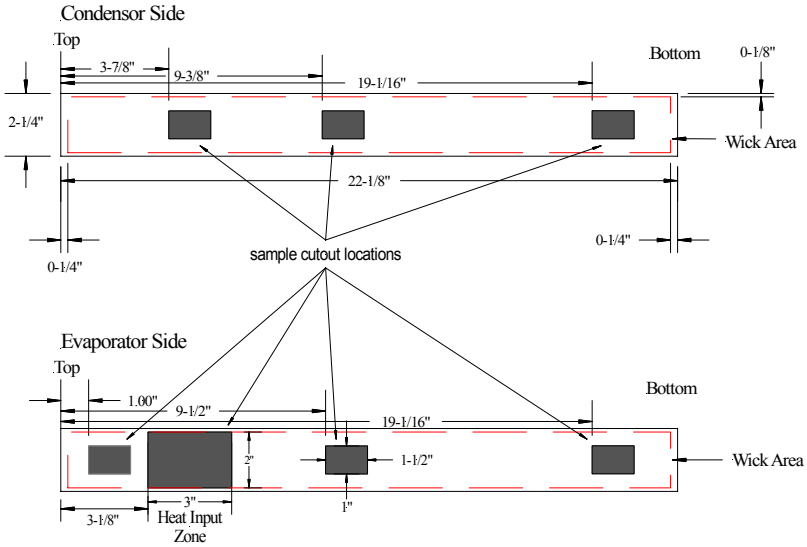


Figure 8: Drawing showing the location of the wick sample cut outs

Fiber thickness measurements of the wick samples were made using the SEM and are shown in appendix B. Fiber thickness of as-received fibers using the SEM was also made. These measurements show very little loss of fiber diameter after 5,000 hours of testing.

Some SEM pictures were also taken showing the bonds between fibers and are shown in appendix C. Again, these pictures show very little corrosion or material removal over the test period of the heat pipe.

The entire heat input zone was removed from the heat pipe, as shown in figure 8, and was analyzed by Paul Hlava in organization 1822 using the SEM and an electron bean micro-probe. This analysis is covered in appendix D. In short, the wick in the lower half of the heat input zone is similar to the other wick samples examined in appendix A in that there is very little corrosion and contamination. The upper half has a white powder filling some of the open pores of the wick that was mostly Ca and aluminum sulfate contamination. Both of these substances are not very corrosive so there was very little corrosion. Copper was also found fairly evenly dispersed inside the wick in the upper half. Again, copper is not a corrosive material and its presence did not seem to affect the operation of the heat pipe. None of these contaminates appeared to affect the bond strength of the wick to the substrate or to itself. Sitting on top of the wick on the side opposite where the top intrinsic thermocouple was placed was a deposit consisting mostly of $Na_2H_2O_3$ with some calcium and silicon. This deposit did not cause any fiber degradation and was not firmly attached to the wick.

Conclusion

In conclusion, there is still some wick contamination and it is still concentrated in the heat input zone. The high temperature bakeout of the heat pipe does significantly reduce wick corrosion compared to previous heat pipe tests. The corrosion in the heat pipe was not enough to cause wick failure in the 5,003 hours of operation it had seen and there was very little corrosion of the fibers themselves. In a Sandia report, SAND97-8248, by Goods et. al. 316LSS was analyzed after 10,000 in a NaK pool boiler at 700C. Their analysis showed no evidence of intergranular attack or enhanced grain boundary dissolution. Their data indicates dissolution is occurring uniformly but very slowly. This is supported with the data in appendix B that shows the wick fiber thickness exhibiting very little change after 5,000 hours of operation. The problem of wick contamination seems to be largely solved by using just the high temperature bakeout. Reflux cleaning of the heat pipe using sodium does help but does not appear to be cost effective compared to using just a high temperature bakeout. For example, reflux cleaning has reduced the amount of copper seen in the wick of a heat pipe after testing, but we have never had a heat pipe fail because of a copper nugget forming in, or on, a wick.

Appendix A- SEM pictures of the wick samples

Pictures using the SEM were taken of the wick samples (top, middle, and bottom) removed from durability heat pipe #6 (shown in figure 5 in the main text). The labeling of the pictures shown in all the figures below has two letters. The first is either a "c" or an "e", where "c" means the wick sample is from the condenser side and "e" is from the evaporator side. The second letter is a "t", "m", or "b" meaning top, middle, and bottom (or pool) respectively. For example, "em" means the wick pictured is from the evaporator side, middle location. Figures 1 through 23 show SEM pictures of the wick samples looking down at the top. In some of the pictures, such as figure 4, the hole in the perf-metal is seen. For comparison, figures 24 and 25 show SEM pictures taken of as-received virgin Bekipor® WB 8/300 metal felt. The as-received fibers have a striation to them caused by the drawing process used to make the fibers. This pattern disappears during the sintering process, not by any effects from being exposed to sodium in the heat pipe. Typically four pictures are taken of each wick sample at different magnifications. These pictures show there is slightly more intergranular notching on the condenser side than on the evaporator side. There also appears to be slightly more and slightly larger particles, or crystals, on the fibers themselves on the condenser side than on the evaporator side. Analysis of these crystals, as shown in figure 23 of the evaporator top at 1000x, shows it to be high in sulfur, aluminum, and silicon. Analysis of the fiber between the crystals shows the elements of 316LSS detected at about the same concentrations as given by the manufacturer. A rigorous examination was not undertaken because it did not seem necessary since very little corrosion was taking place outside the heat input zone (see appendix D). This indicates selective etching of the fibers is not occurring. Note also the overall cleanliness of the fibers and the empty space in between the fibers. There is no sign of any deposit buildup between fibers in any of the wick outside the heat input zone.

Some SEM pictures were also taken from the side of the wick samples, as shown in Figures 26 thru 31, from the condenser middle and condenser bottom locations. The orientation of these pictures is with the perf-metal on top and the substrate on the bottom, as can be seen in figure 26. The larger particles seen in these pictures are from the cutting tool used (a Dremel tool with a ceramic cut-off wheel) to remove the sample from the heat pipe. These pictures show the prevailing orientation of the fibers is parallel to the substrate with a few of the fibers perpendicular to the substrate. The bottom left hand side of figure 30, however, shows most of the fibers perpendicular to the substrate. This may have been the result of cutting the sample out rather than the fibers being orientated in this manner before sintering.

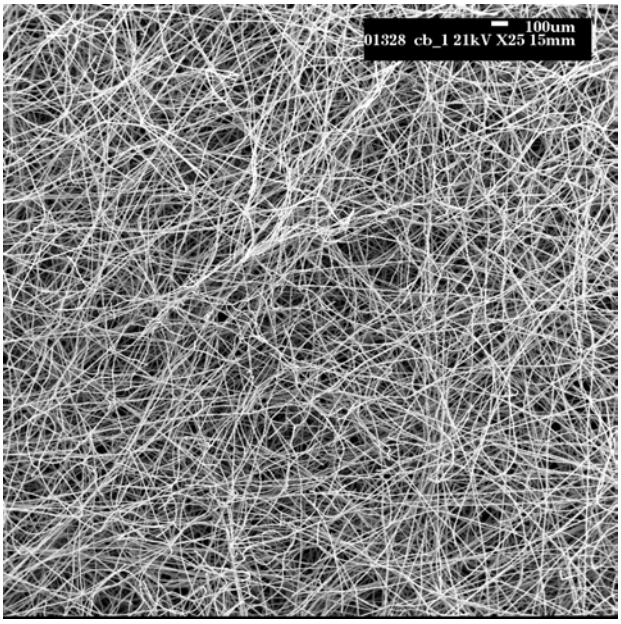


Figure A1: SEM picture of the condenser bottom wick sample at 25x (from the top)

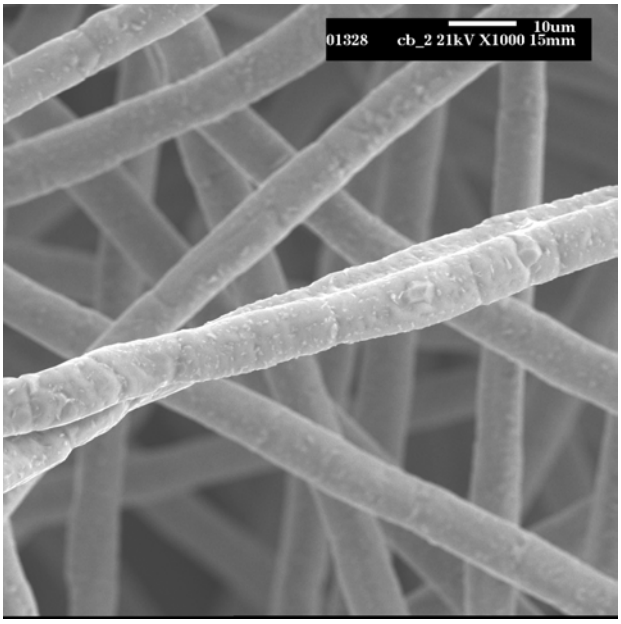


Figure A2: SEM picture of the condenser bottom wick sample at 1,000x (from the top)

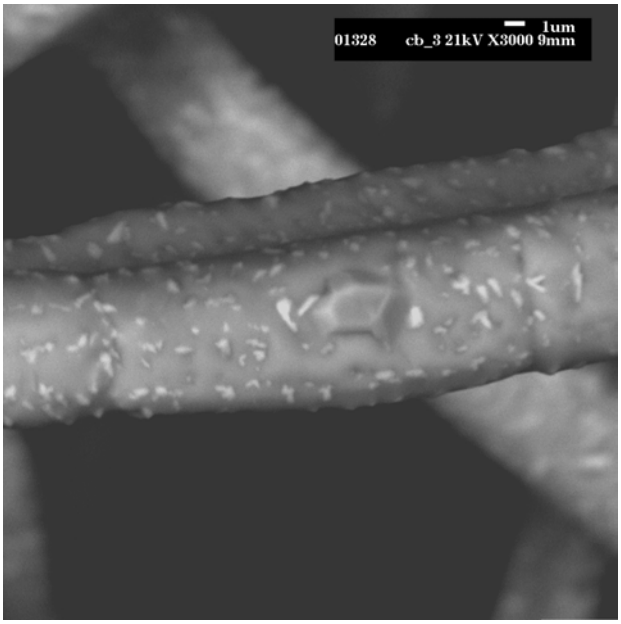


Figure A3: SEM picture of the condenser bottom wick sample at 3,000x (from the top)

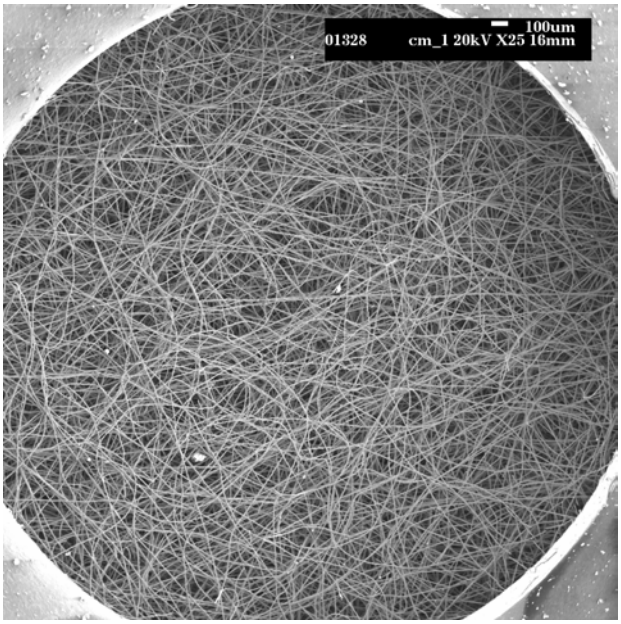


Figure A4: SEM picture of the condenser middle wick sample at 25x (through a hole in the perf-metal)

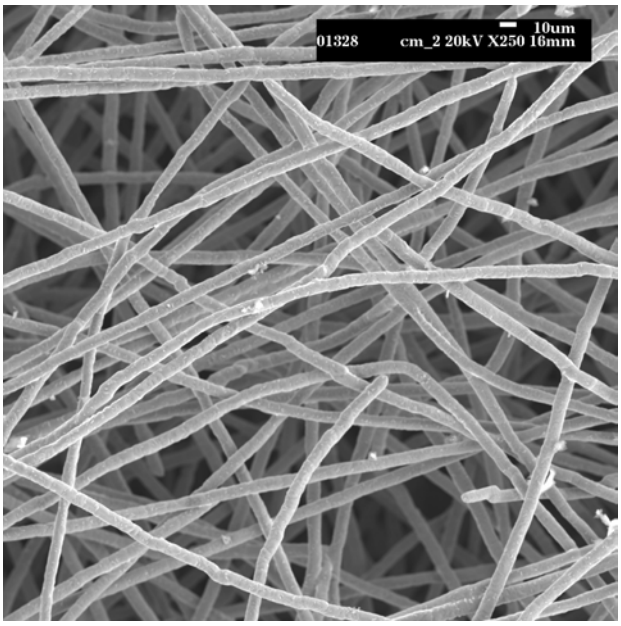


Figure A5: SEM picture of the condenser middle wick sample at 250x (from the top)

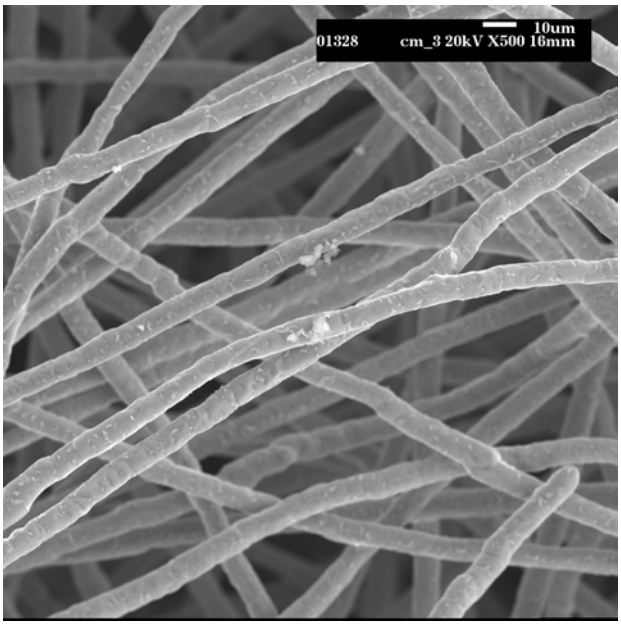


Figure A6: SEM picture of the condenser middle wick sample at 500x (from the top)

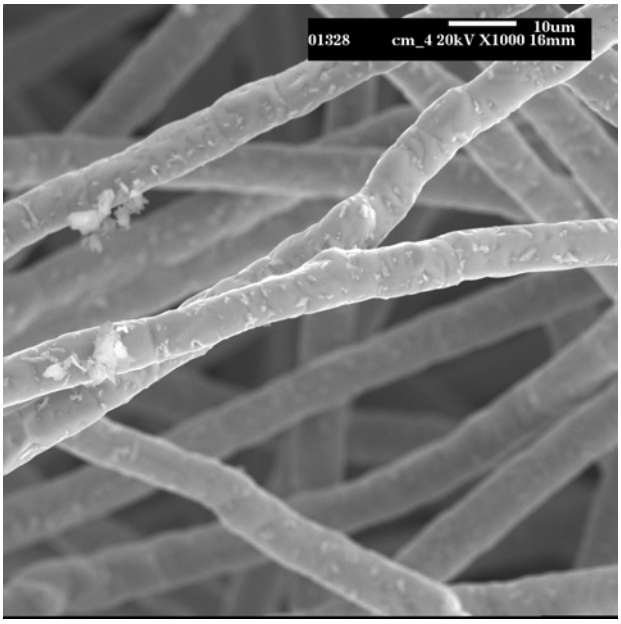


Figure A7: SEM picture of the condenser middle wick sample at 1,000x (from the top)

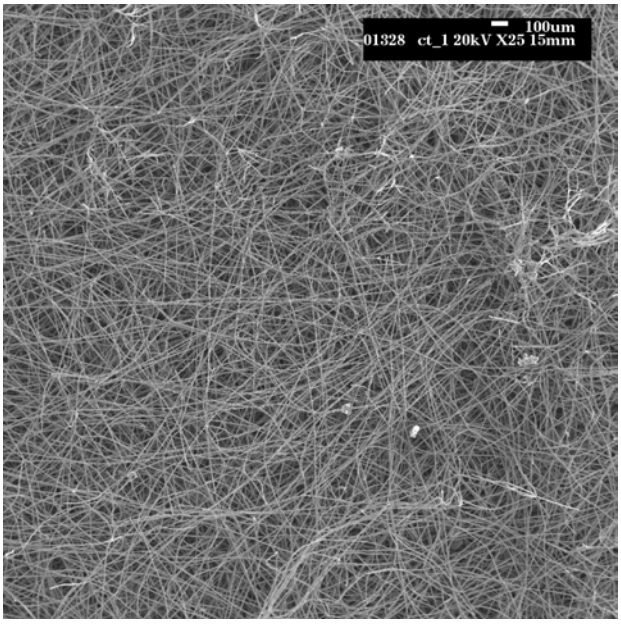


Figure A8: SEM picture of the condenser top wick sample at 25x (from the top)

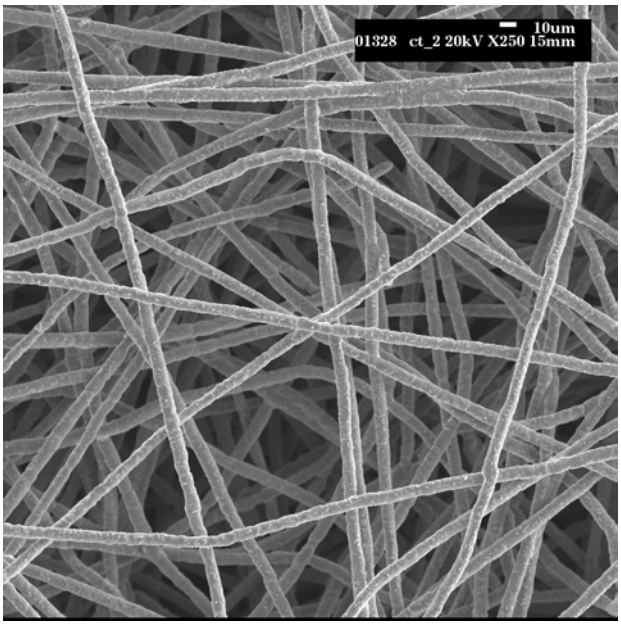


Figure A9: SEM picture of the condenser top wick sample at 250x (from the top)

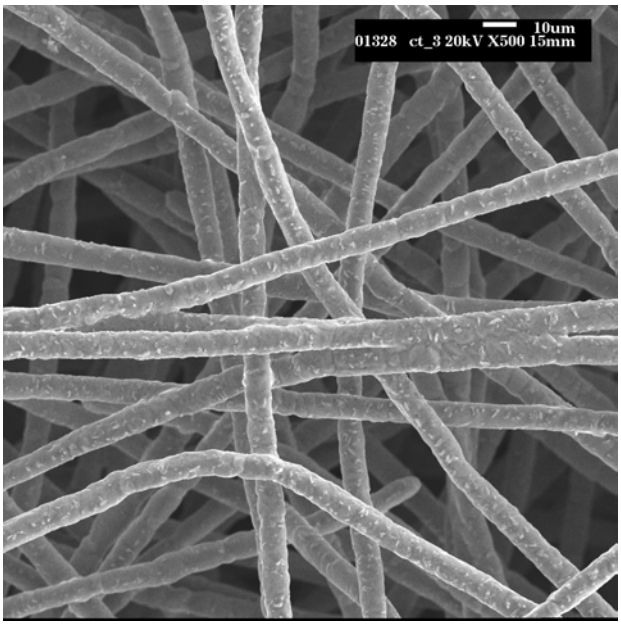


Figure A10: SEM picture of the condenser top wick sample at 500x (from the top)

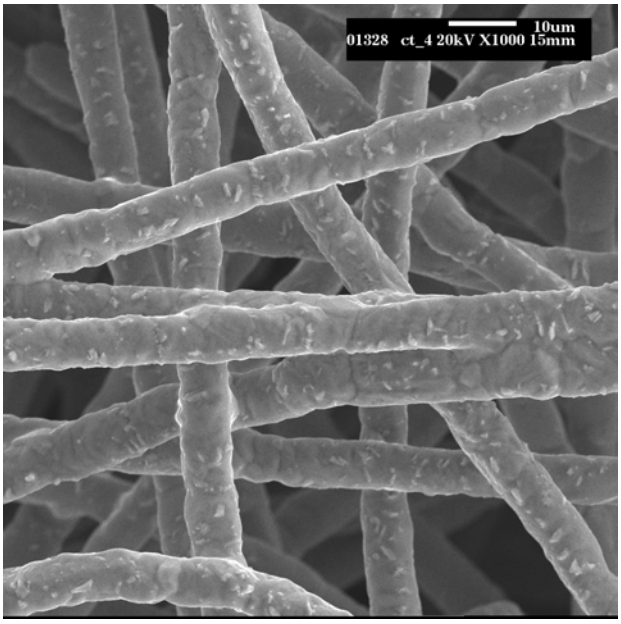


Figure A11: SEM picture of the condenser top wick sample at 1,000x (from the top)

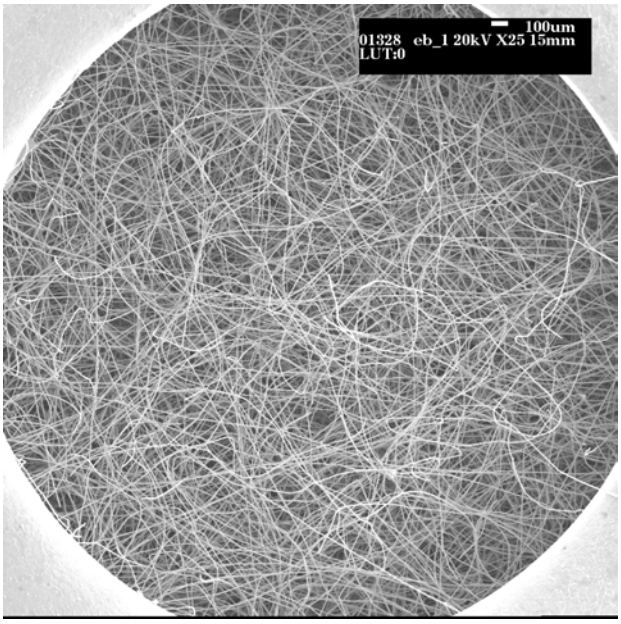


Figure A12: SEM picture of the evaporator bottom wick sample at 25x (though a hole in perf-metal)

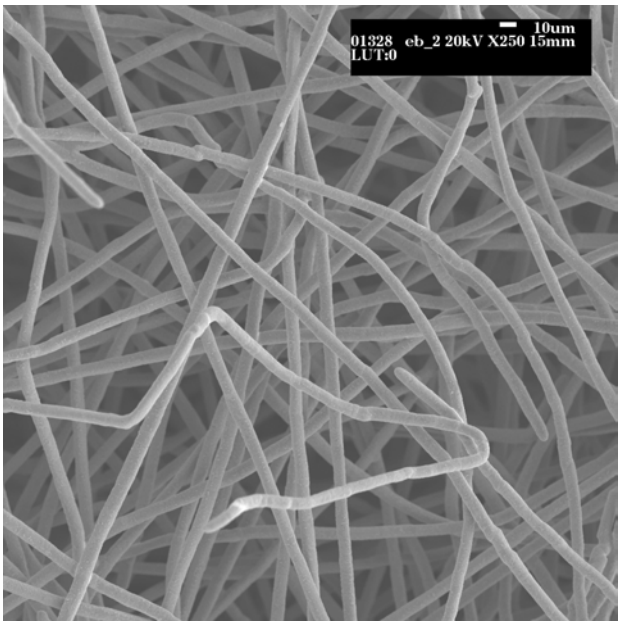


Figure A13: SEM picture of the evaporator bottom wick sample at 250x (from the top)

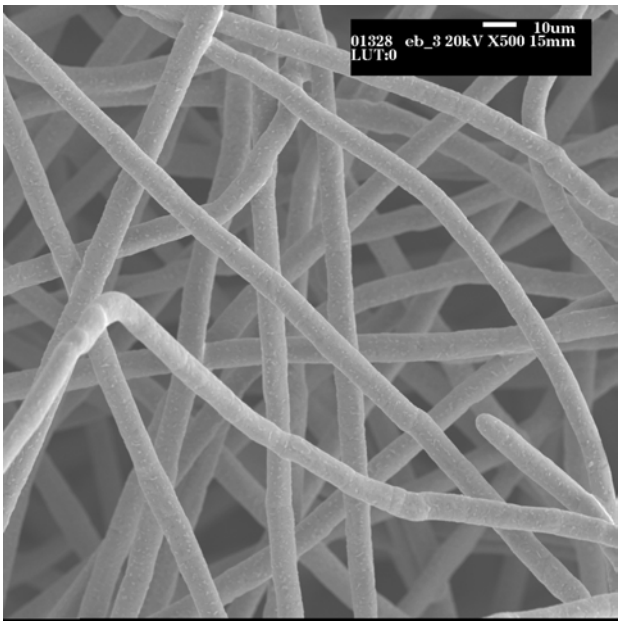


Figure A14: SEM picture of the evaporator bottom wick sample at 500x (from the top)

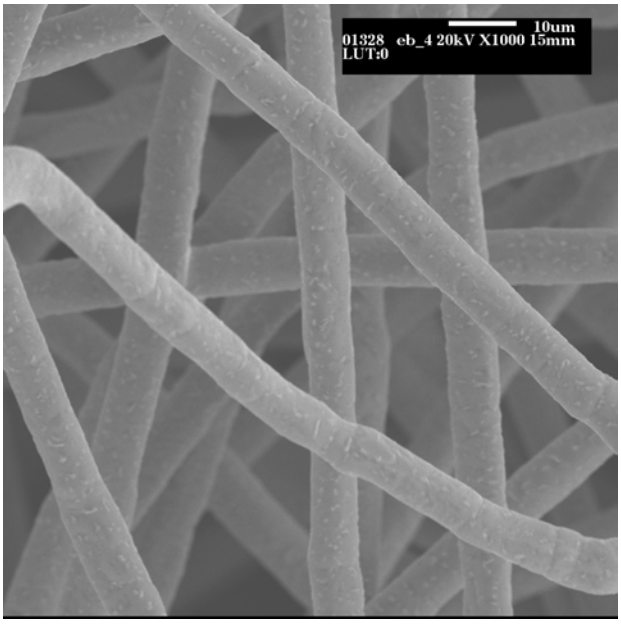


Figure A15: SEM picture of the evaporator bottom wick sample at 1,000x (from the top)

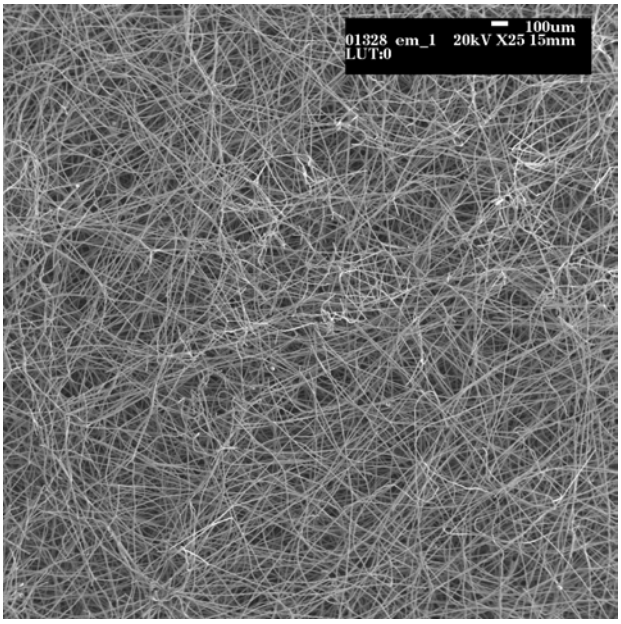


Figure 16: SEM picture of the evaporator middle wick sample at 25x (from the top)

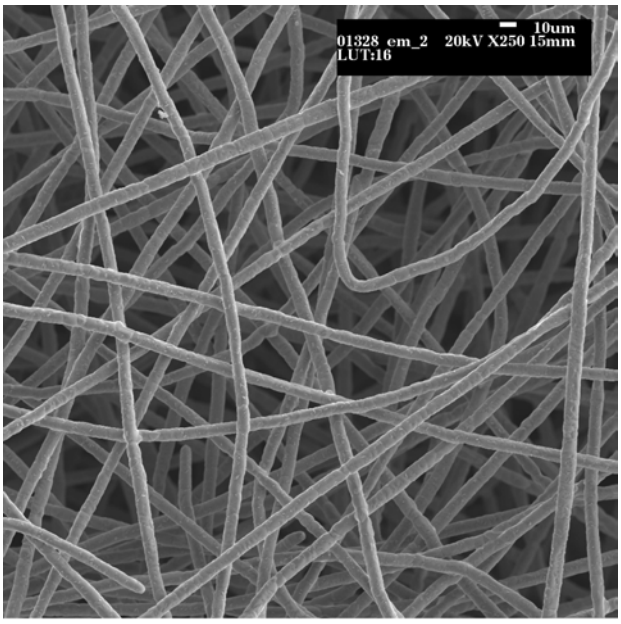


Figure A17: SEM picture of the evaporator middle wick sample at 250x (from the top)

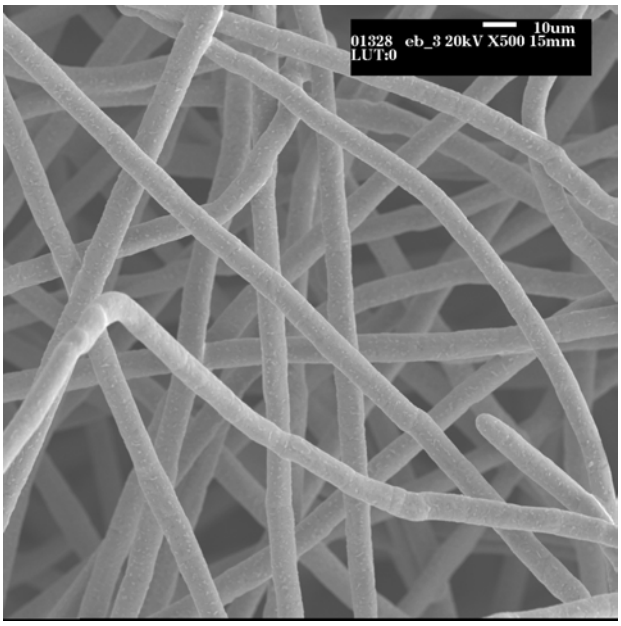


Figure A18: SEM picture of the evaporator middle wick sample at 500x (from the top)

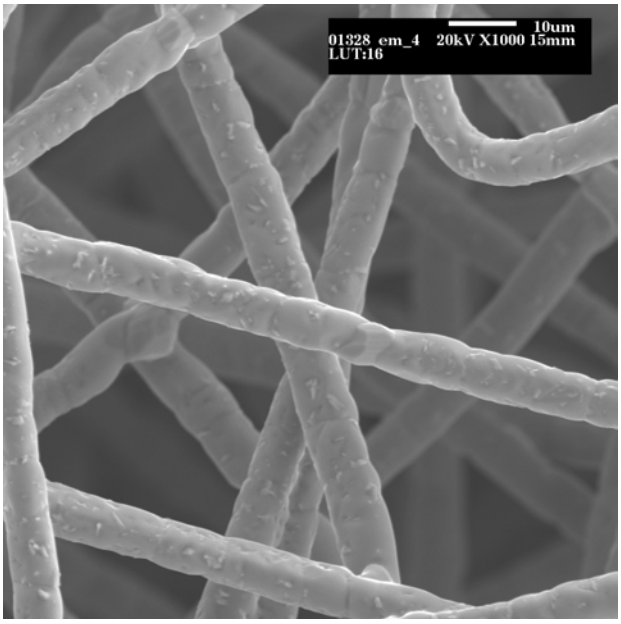


Figure A19: SEM picture of the evaporator middle wick sample at 1,000x (from the top)

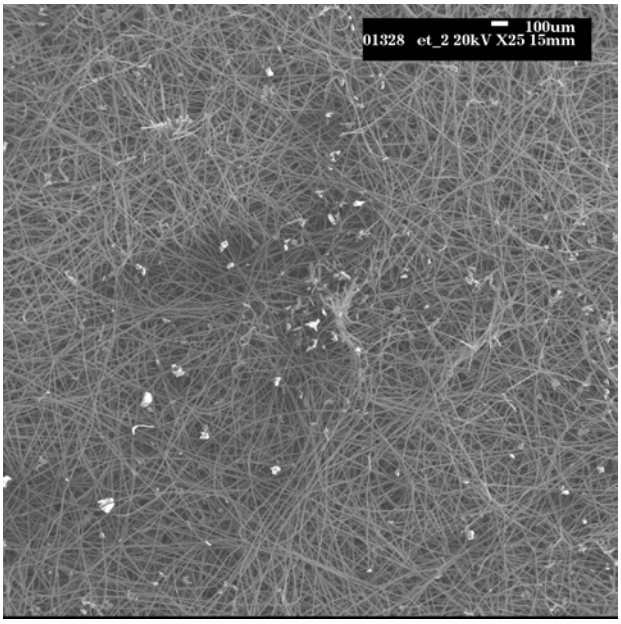


Figure A20: SEM picture of the evaporator top wick sample at 25x. The particles seen are from cutting out the sample from the wick.

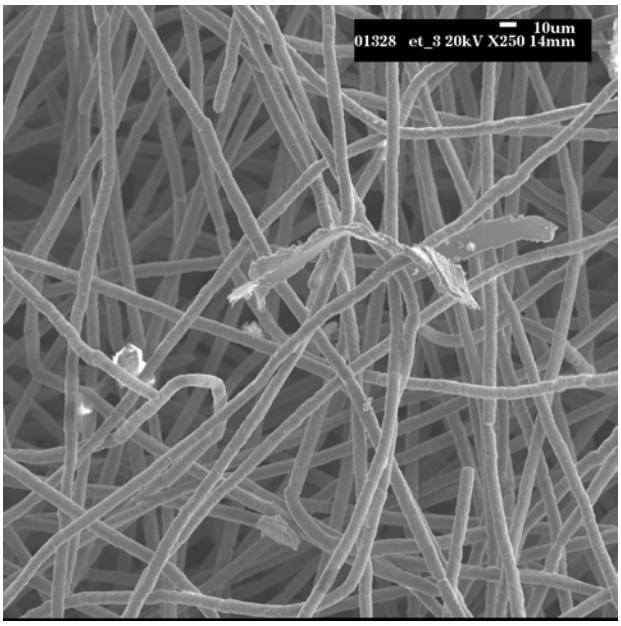


Figure A21: SEM picture of the evaporator top wick sample at 250x. The particles seen are from cutting out the sample from the wick.

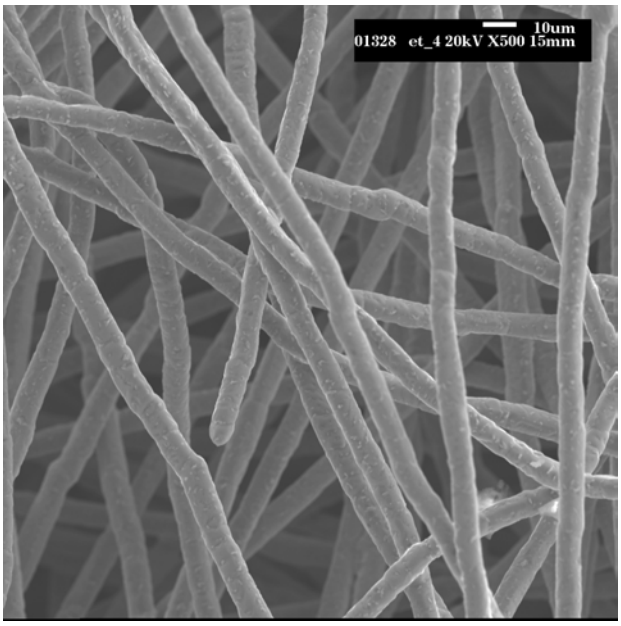


Figure A22: SEM picture of the evaporator top wick sample at 500x (from the top)

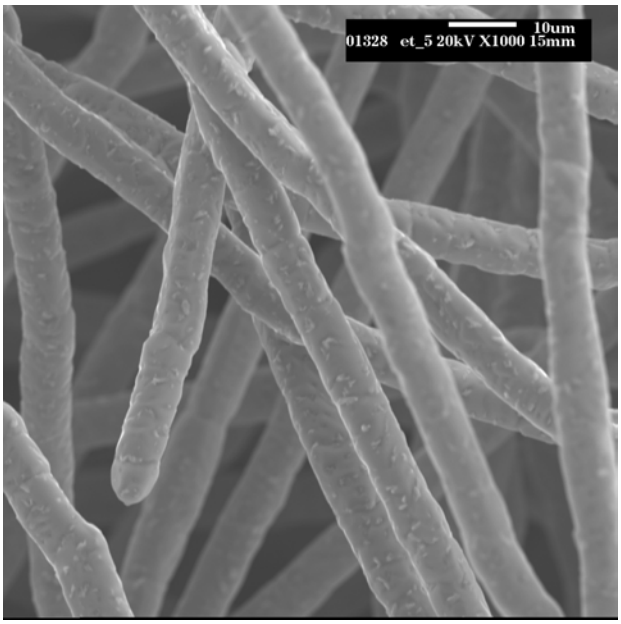


Figure A23: SEM picture of the evaporator top wick sample at 1,000x (from the top)

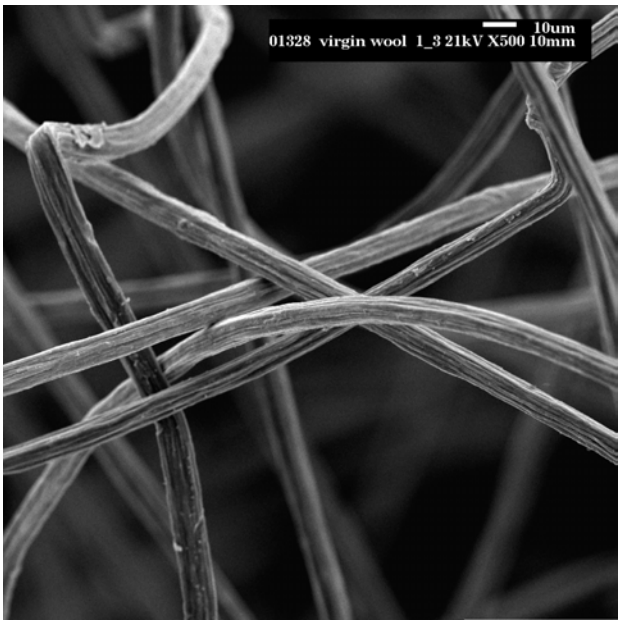


Figure A24: SEM picture of the as-received metal felt at 500x magnification

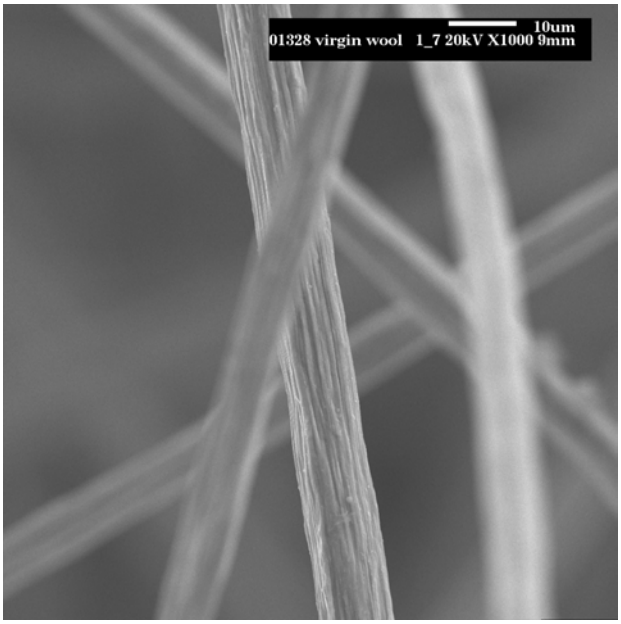


Figure A25: SEM picture of the as-received metal felt at 1,000x magnification

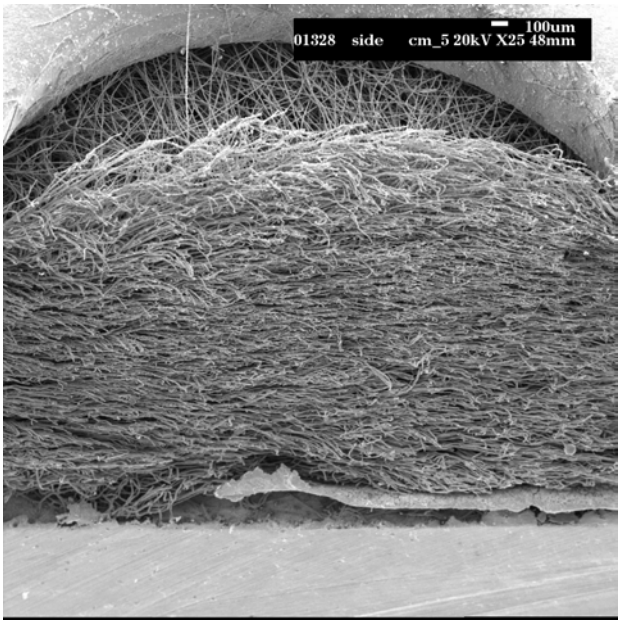


Figure A26: SEM picture of the condenser middle wick sample from the side (perf-metal on top and substrate on the bottom) at 25x magnification

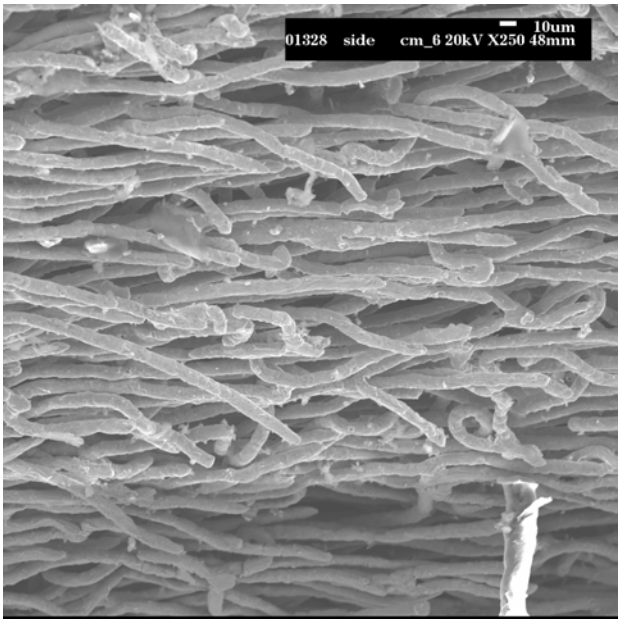


Figure A27: SEM picture of the condenser middle wick sample from the side (perf-metal above and substrate below) at 250x magnification

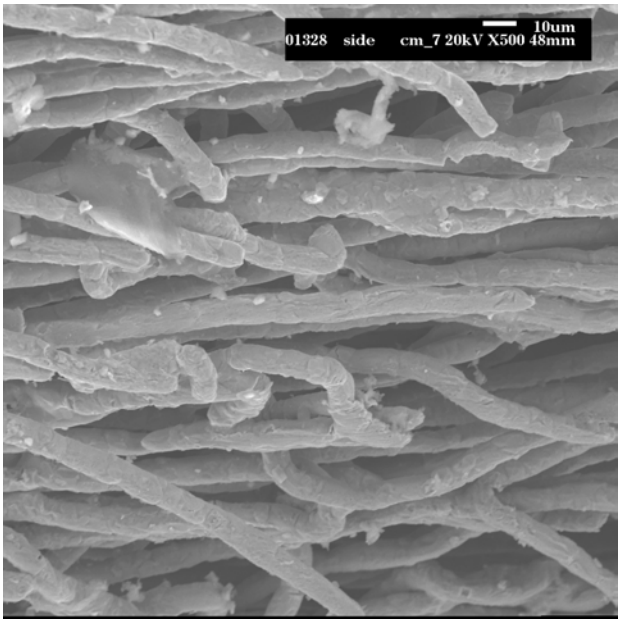


Figure A28: SEM picture of the condenser middle wick sample from the side (perf-metal above and substrate below) at 500x magnification

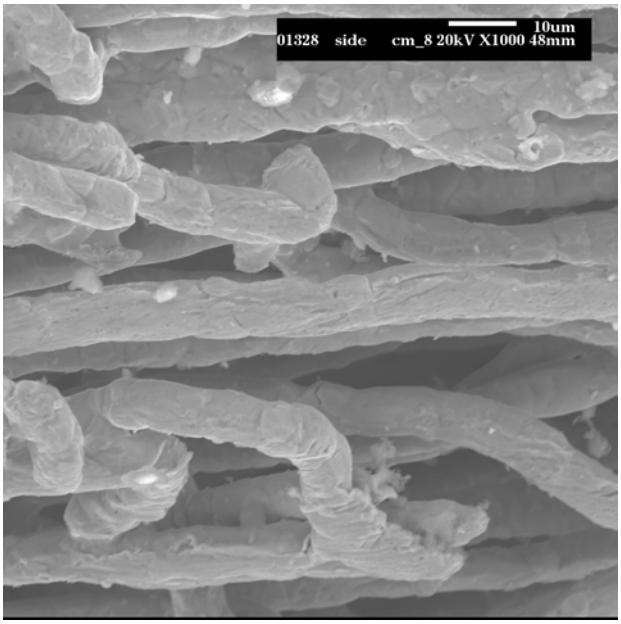


Figure A29: SEM picture of the condenser middle wick sample from the side (perf-metal above and substrate below) at 1,000x magnification

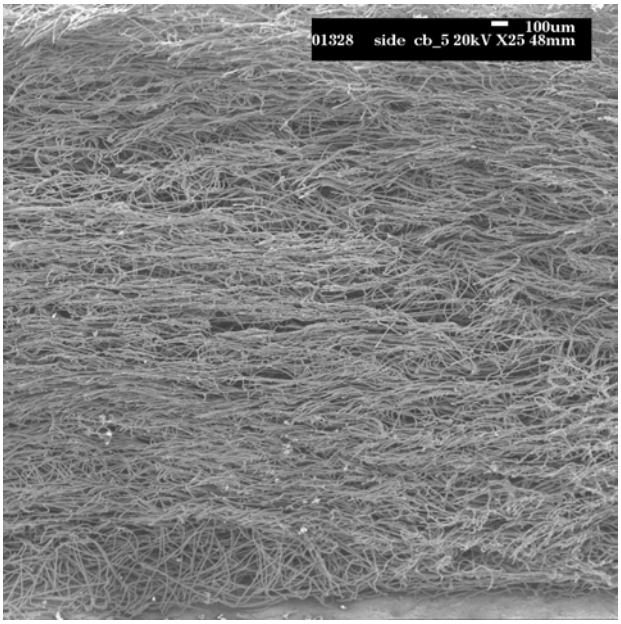


Figure A30: SEM picture of the condenser bottom wick sample from the side (perf-metal above and substrate below) at 25x magnification

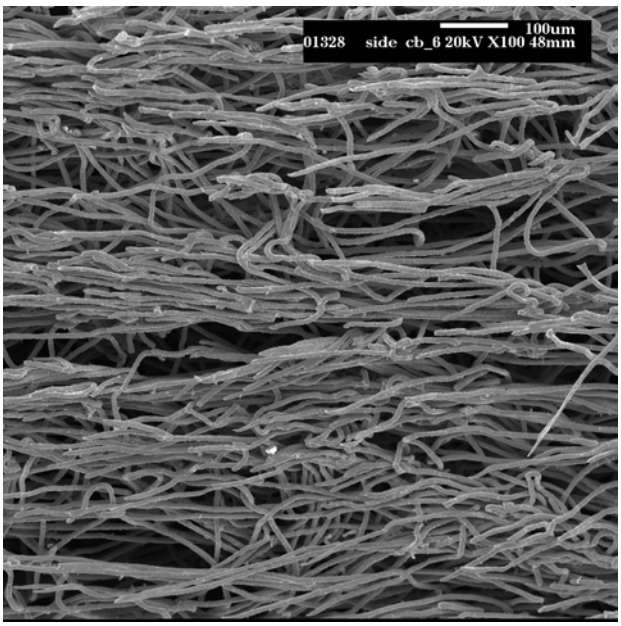


Figure A31: SEM picture of the condenser bottom wick sample from the side (perf-metal above and substrate below) at 100x magnification

Appendix B - Measurements of the wick fiber thickness

Using the SEM, measurements of the fiber thickness were taken of several fibers at multiple places from the three wick samples (top, middle, and bottom) removed from the evaporator and condenser side of durability heat pipe #6. Figures 1 thru 6 show the measurements taken using the SEM. For comparison, SEM measurements, shown in Figure 7, also measured fibers from the as-received 8/300 felt. The as-received fibers have a striation to them caused by the drawing process used to make the fibers. This pattern disappears during the sintering process, not by any effects from being exposed to sodium in the heat pipe. Figure 8 shows a plot of the average measured fiber thickness for each wick location, the average of the measured fiber thickness for all locations, and the average measured fiber diameter for the as-received felt. This plot shows there is very little difference in fiber thickness from the different locations in the heat pipe. For example, the average fiber thickness from all measurements on the condenser side is 7.5 ± 0.5µm and on the evaporator side it is 7.2 ± 0.5µm. The average measured fiber thickness from all locations in the heat pipe is 7.3 ± 0.5µm. The average measured fiber diameter for the as-received felt is 8.0 ± 0.5µm. The average fiber thickness in the heat is about 10% less than the as-received fiber thickness, but within one standard deviation, there is no difference between the as-received fibers and the fibers that had seen 5,000 hours in sodium at 750°C. The conclusion is there is very little corrosion of the wick fibers during the 5,000 hours of operation. This is a marked improvement over previous durability heat pipe tests with similar wicks that were made without a high temperature bakeout or reflux cleaning.

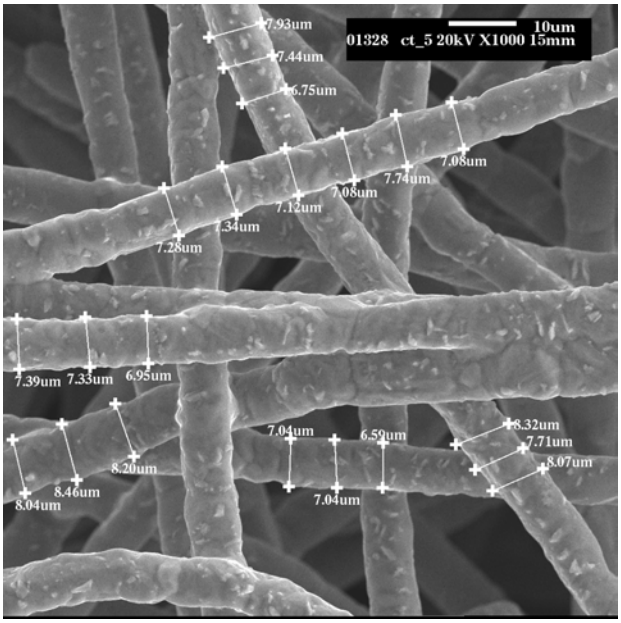


Figure B1: Wick sample from the condenser side, top location at 1,000x magnification

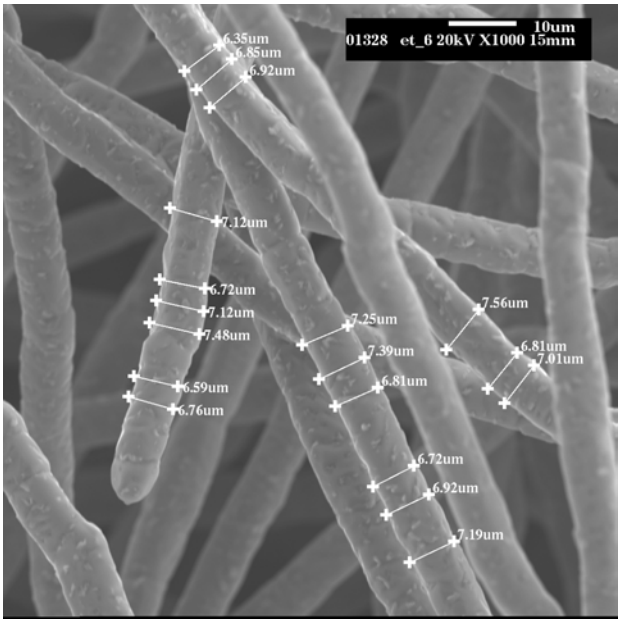


Figure B2: Wick sample from the evaporator side, top location at 1,000x magnification

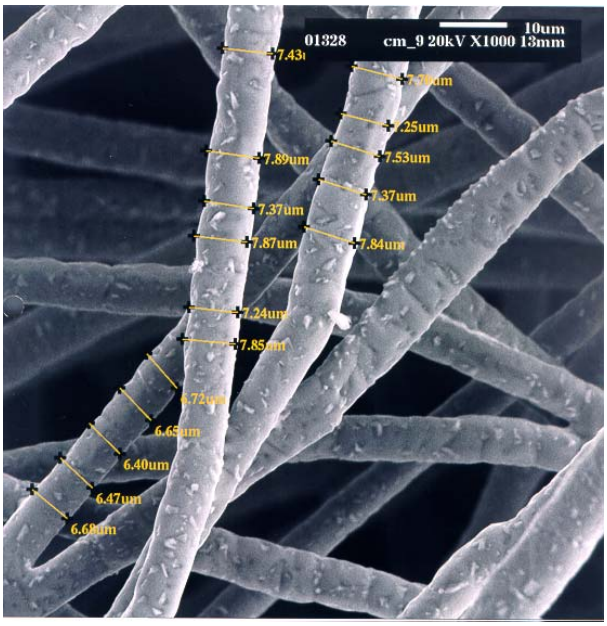


Figure B3: Wick sample from the condenser side, middle location at 1,000x magnification

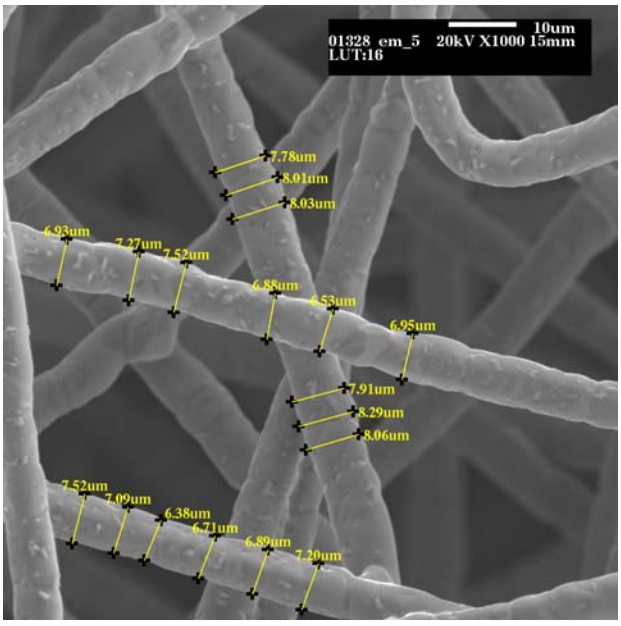


Figure B4: Wick sample from the evaporator side, middle location at 1,000x magnification

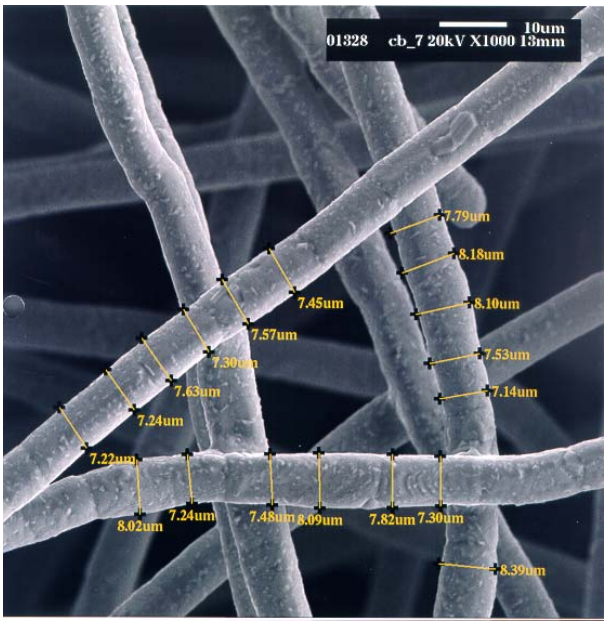


Figure B5: Wick sample from the condenser side, bottom location at 1,000x magnification

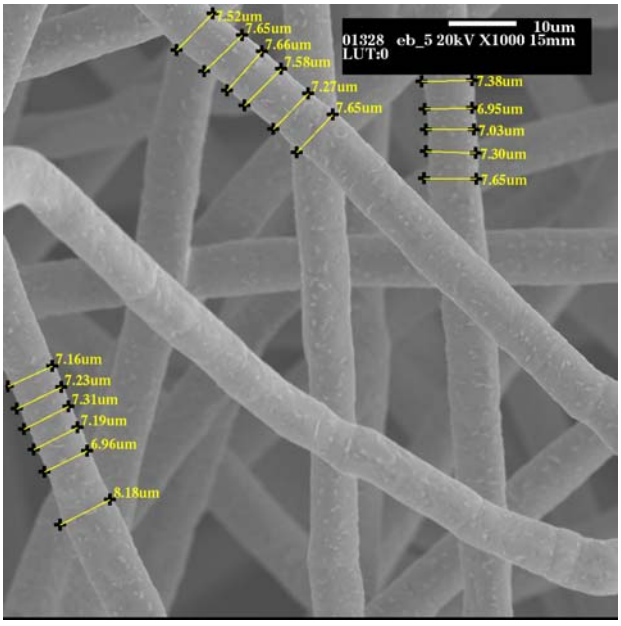


Figure B6: Wick sample from the evaporator side, bottom location at 1,000x magnification

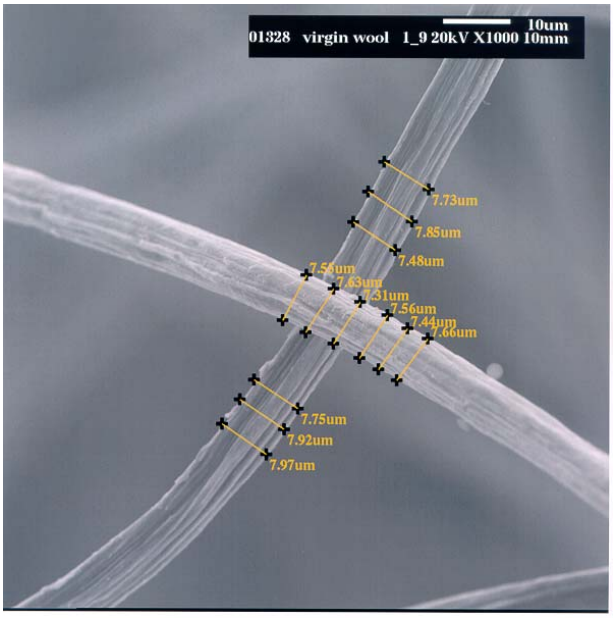


Figure B7: As-received 8/300 felt at 1,000x magnification

Figure B8: As-received 8/300 felt at 1,000x magnification

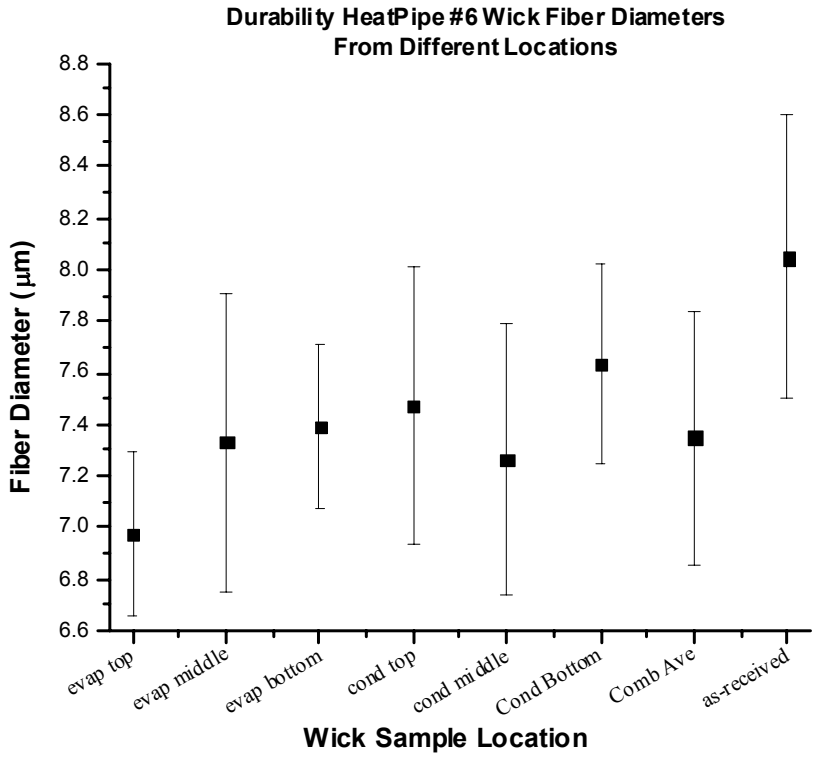


Figure B9: Plot showing the measured fiber thickness at each location showing one standard deviation

Appendix C- A look at the sintering bonds between wick fibers

The sintered bonds between fibers were examined using the SEM. The figures below show the pictures taken of the wick bonds from the three wick samples (top, middle, and bottom) from the evaporator and condenser sides of the heat pipe. These pictures show the condition of bonds between fibers initially formed during the sintering process. Note that these pictures show that the sintering bonds between the fibers do not appear to be affected any more or less than the rest of the fibers after 5,003 hours in sodium at 750°C. The first 4 figures are from the condenser side top and bottom locations with the rest are from the evaporator side. There is very little difference between the condenser side and the evaporator side as far as sintering bonds are concerned. The last figure, figure 13, shows the bonds formed during the sintering process for 4/150 felt. This particular wick was never put inside a heat pipe. No pictures were found of 8/300 felt after sintering but before use inside a heat pipe. This picture shows the same type of sintering bonds as the other pictures, figures 1-12, for the 8/300 felt. The comparison shows that the material removal rate caused by the sodium is about the same for the sintering bonds and the fiber itself.

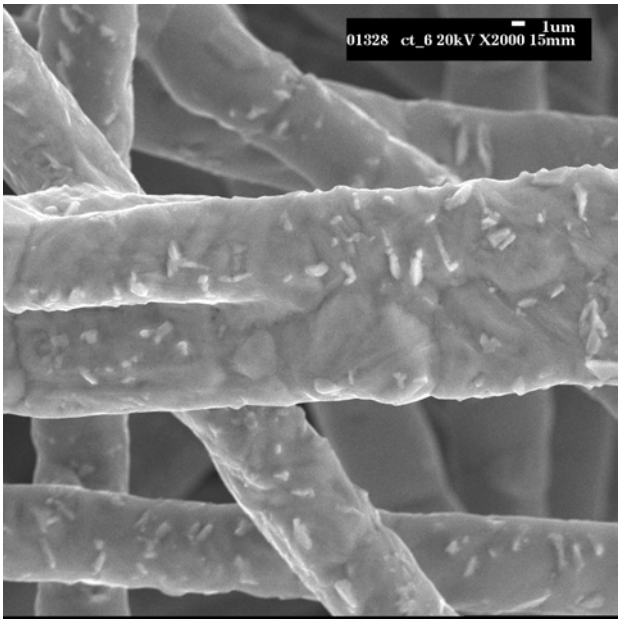


Figure C1: Wick section on condenser side, top location, at 2,000x magnification

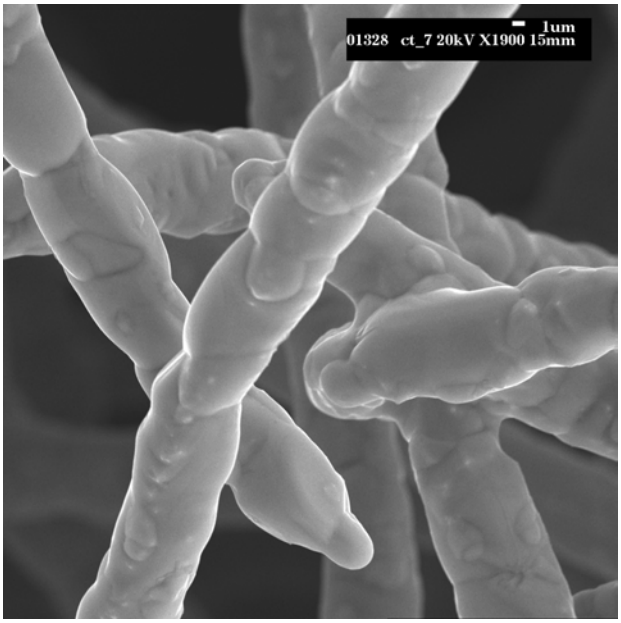


Figure C2: Wick section on condenser side, top location, at 1,900x magnification

Figure C3: Wick section on condenser side, bottom location, at 1,000x magnification

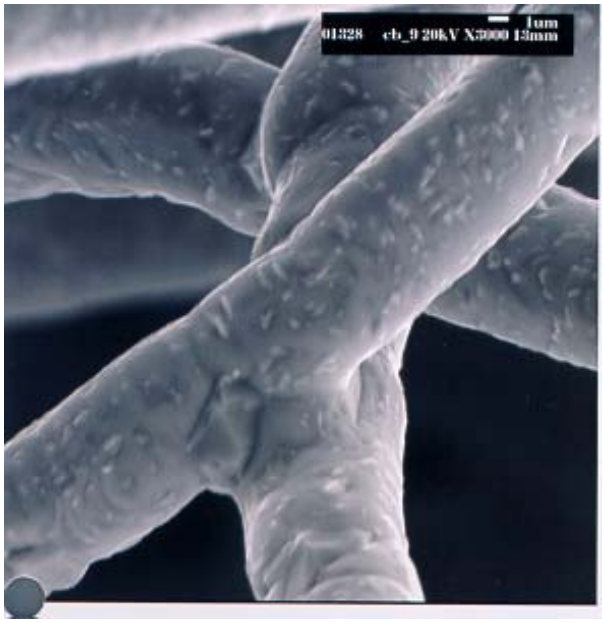


Figure C4: Wick section on condenser side, bottom location, at 3,000x magnification

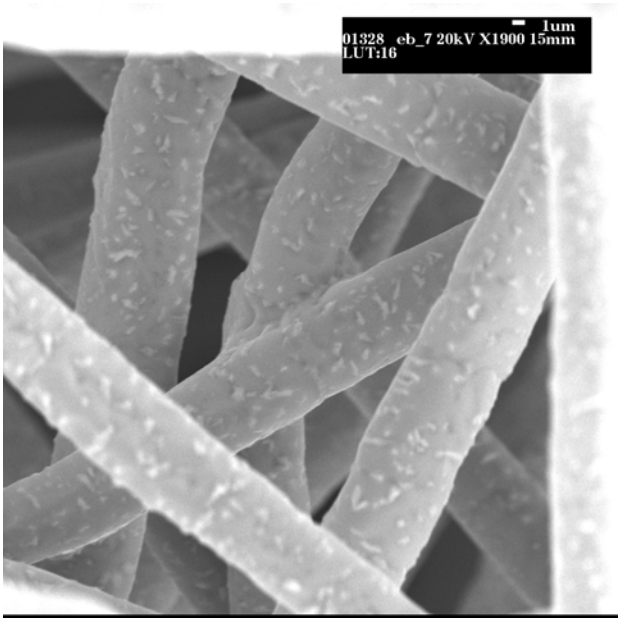


Figure C5: Wick section on evaporator side, bottom (pool) location, at 1,900x magnification

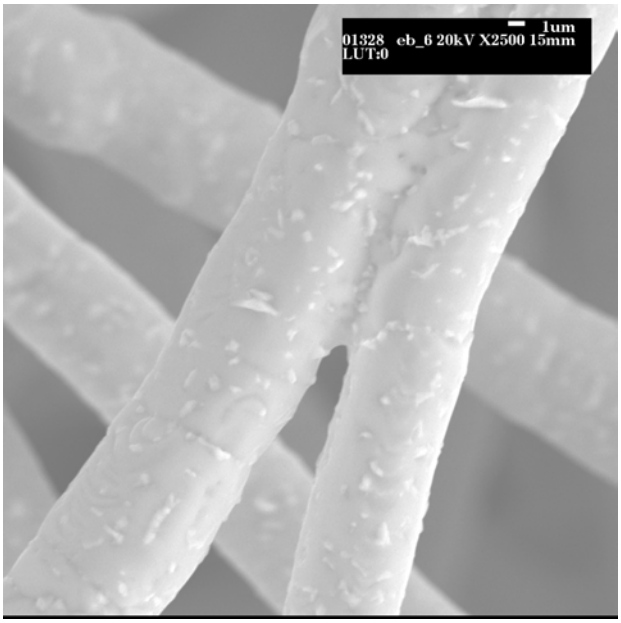


Figure C6: Wick section on evaporator side, bottom (pool) location, at 2,500x magnification

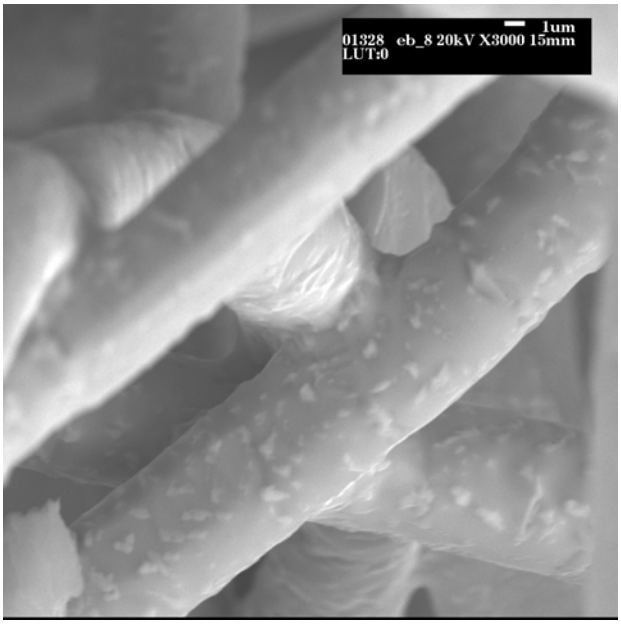


Figure C7: Wick section on evaporator side, bottom (pool) location, at 3,000x magnification

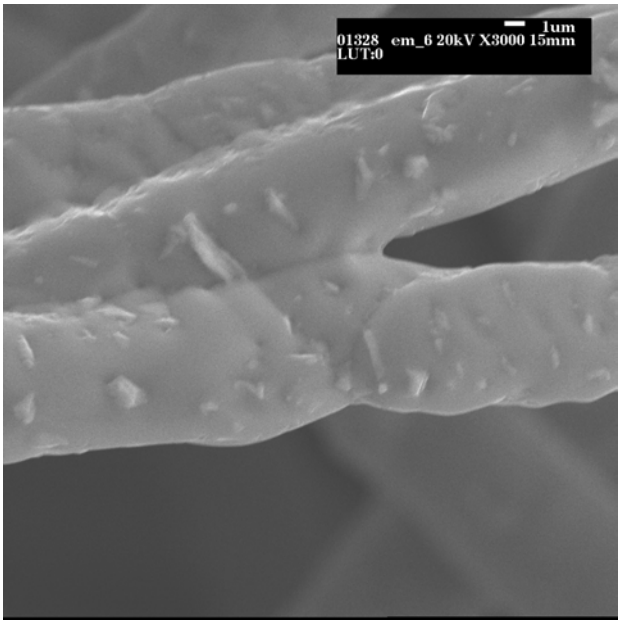


Figure C8: Wick section on evaporator side, middle location, at 3,000x magnification

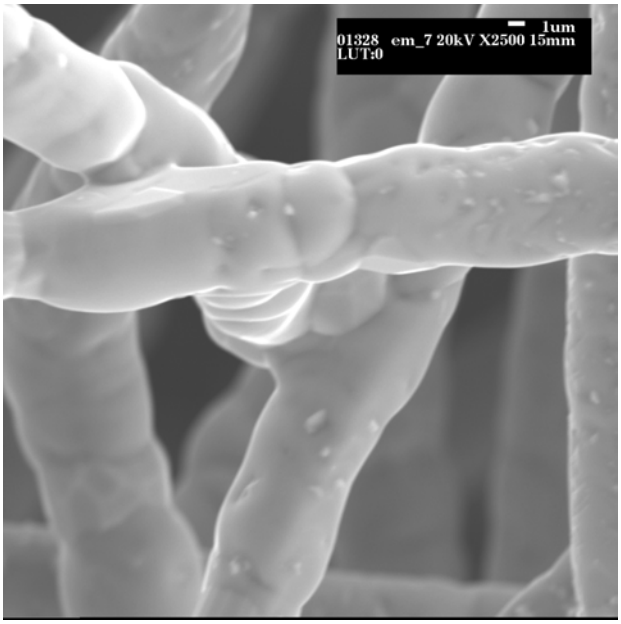


Figure C9: Wick section on evaporator side, middle location at 2,500x magnification

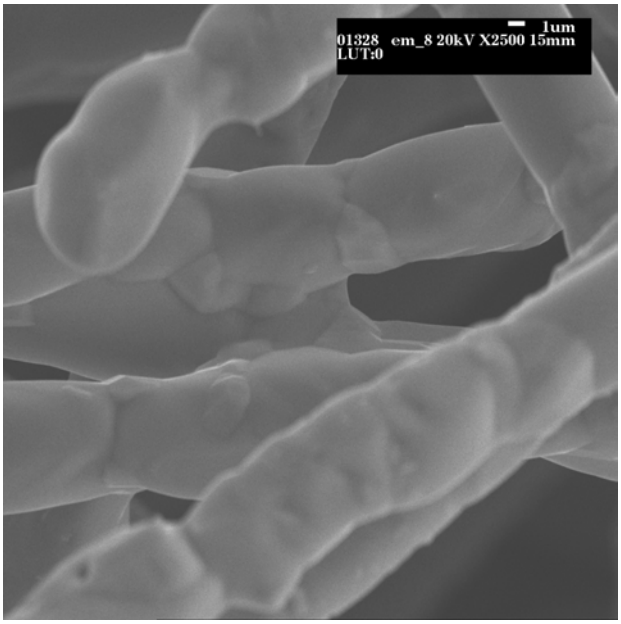


Figure C10: Wick section on evaporator side, middle location at 2,500x magnification

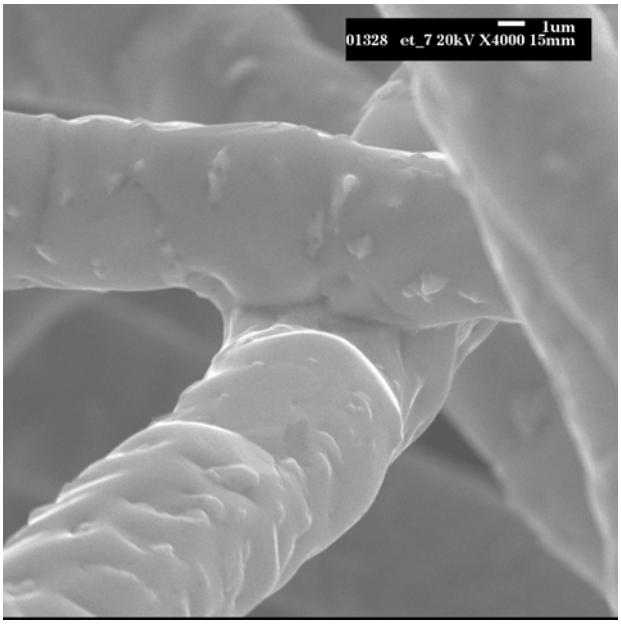


Figure C11: Wick section on evaporator side, top location, at 4,000x magnification

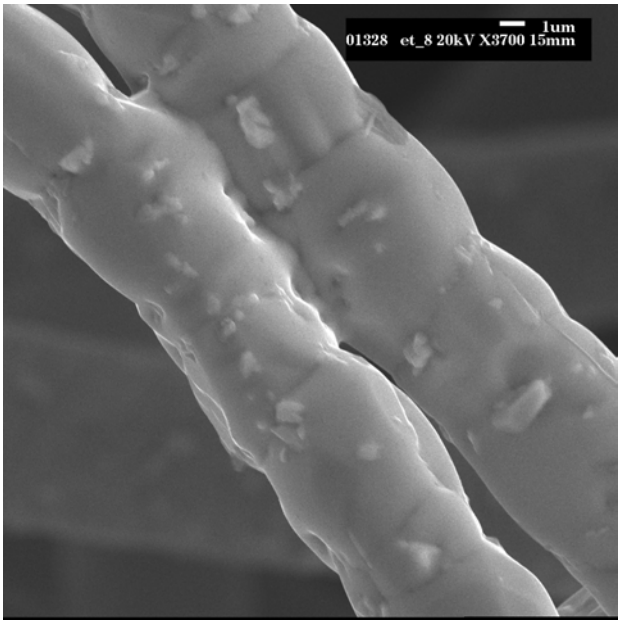


Figure C12: Wick section on evaporator side, top location, at 3,700x magnification

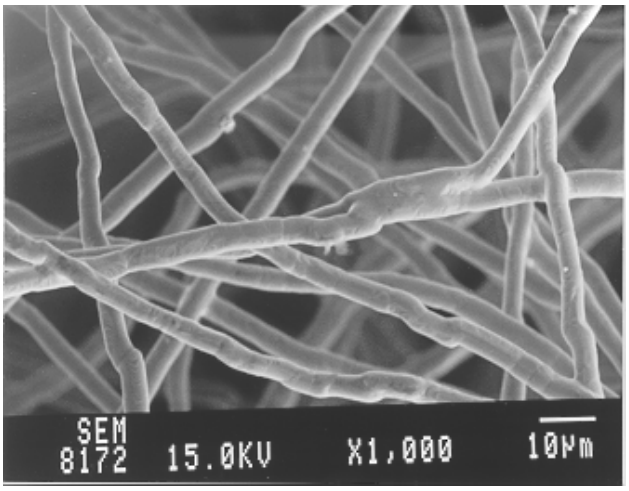


Figure C13: 4/150 wick section after sintering only, at 1,000x magnification

Appendix D - Analysis of the heat input zone

The entire heat input zone was removed from the durability heat pipe #6 for analysis using SEM and electron microprobe. Figures 1 and 2 show the entire heat input zone, the area directly in front of the lamp array, with the top being on the right side and the bottom of the heat input zone on the left. Figure 2 is the same as figure 1 but the perf-metal overlay was removed. The felt wick was not attached to the pert-metal except along the sides. The deposit on the top of the felt wick is more easily seen in figure 2. Figures 3 and 4 show the deposit at different magnifications. The actual location of the deposit is slightly above and on the opposite side of the intrinsic thermocouple used to monitor the surface temperature in the top part of the heated zone during testing. Using the electron microprobe, the deposit is mostly sodium and calcium oxide with some copper. The felt wick immediately around the edges of the deposit is discolored from the rest of the felt wick. The discoloration is caused by the surface of the fibers being enhanced with Fe and Cr, as compared to 316LSS. Previous analysis of as-received metal felt showed the fibers are within manufacture's specifications for 316LSS.

The deposit sitting on top of the wick contained one calcite crystal and some Si, but most of it was NaO that converted to $Na_2H_2O_3$ (sodium hydroxide) after being exposed to air. The Si comes from all the materials present in the heat pipe, Haynes alloy 230 (0.25-0.75 weight%), 316LSS (1 weight%), and sodium (13ppm). When the concentration of NaO is 100ppm or greater it becomes very corrosive. The deposit sitting on top of the felt wick was stuck to the wick, but not firmly attached since it separated from the felt wick without leaving anything behind and not taking any of the fibers with it. The fibers underneath the deposit did not show any more corrosion or reduction in fiber thickness than any other area in the heat pipe. This indicates the NaO concentration was below 50ppm, or there was a buffer layer between the NaO and the felt wick such as the Ca and aluminum sulfate, described in the next paragraph, inhibiting corrosion of the felt wick from NaO.

During analysis, the wick was peeled back from the substrate throughout the heated zone. The wick was firmly adhered to the substrate and to itself as it was hard to peel back and much of the wick was left behind on the substrate. Inside the wick starting just below the midpoint and extending about 1" vertically upward (in the direction of liquid flow), which includes the deposit on top of the wick, the wick was filled with a white powder. The electron microprobe showed this white deposit consisted of Ca and aluminum sulfate ($Al_2(SO_4)_3$). The Ca most likely came from the sodium (13 ppm) since it is not listed as a component of any other material in the heat pipe. The sulfur most likely came from the 316LSS (0.3 weight%) since it is not listed in any of the other materials in the heat pipe. Ca and aluminum sulfate are not corrosive materials and no indication was found that these deposits affected the wall/wick bond or the sintering bonds between fibers. No deposits were found in the bottom half of the heated zone, just as in all the other wick samples from the heat pipe.

Copper was found spread out in the heat input zone inside the wick close to the substrate starting at about the midpoint and extending vertically up (in the direction of liquid flow) the wick about ½" forming thin crystals to a coating on the felt fibers. No copper nugget was found, however, either on top of or inside of the wick as was found in previously tested bench test heat pipes and capsule tests. There is some copper in the as-received sodium (10 ppm) but the largest source of

copper is from the as-received metal felt as a result of the process used in making the fibers. Stainless steel fibers can be drawn individually down to only 30μm. To achieve smaller diameters, such as 4, 8, or 12 μm, the fibers are encased in a copper matrix and then drawn. When the desired fiber diameter is achieved the copper is acid etched away, but some copper is inevitably left behind on the surface and some in the bulk of the fiber. Previous analysis of as-received 8/300 metal felt showed 0.588 and 0.597% Cu by weight using atomic absorption spectroscopy. Copper is easily dissolved by the sodium in the heat pipe and is redeposited where the sodium evaporates. Since most of the evaporating sodium occurs in the heat input zone this is where most of the dissolved copper is redeposited, which is also true for most of the contaminates dissolved in the sodium. The 8/300 felt weighs about 0.19 gm/in^2. In the heat pipe there are three layers of 8/300 felt 1¾" wide and 22⅛" per side for a total of 232.3in^2. Using the average of 0.593% Cu by weight for the fibers this comes to 0.26gm of total Cu for the heat pipe. If all the copper in the felt was totally dissolved into the sodium and redeposited in the heated zone as one solid chunk it would be a cube roughly 3mm on a side. A single nugget of copper was not found probably because there wasn't a single point in the heat input zone where most of the sodium was evaporating from but was spread out into a larger zone. This is supported by the aluminum sulfate found in the wick being similarly spread out inside the wick instead of concentrating in one small area.

Figure D1: The entire heat input zone removed from durability heat pipe #6 with perf-metal overlay intact. In this picture the top of the heat input zone is the right side and the bottom is the left side.

Figure D2: The entire heat input zone removed from durability heat pipe #6. This is the same as figure 1 but with the perf-metal overlay removed.

Figure D3: Close up of deposit on top of the felt wick underneath the perf-metal.

Figure D4: Higher magnification of the deposit sitting on top of the felt wick. This picture shows how the deposit does not appear to penetrate into the wick but is sitting on top of it.

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