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Cover Page Footnote

Firstly, I would like to thank my parents Dan and Jenny Kok for their unrelenting support in all my endeavors. I would certainly not be where I am as a person or as a scholar without them. They were even gracious enough to allow me to use their own backyard for my testing during this study, and for that alone I am deeply grateful. I would also like to thank Professor David Ibrahim, my project mentor, for his help and guidance throughout this entire project. Beginning and finishing this project would have been infinitely more difficult without his unwavering confidence in me and expertise in the field of nuclear engineering. In the same way I thank Dr. John Tatarko for his expertise and input into my project. His freely given knowledge of chemistry and material science allowed me to solidify the actual testing procedure for this study and gave me the strong foundation to discuss my findings. Thank you to Dr. Dan Sharda for his help in refining my thesis and presentation in addition to supporting me throughout the project. My thesis would be in a state of disarray without his guidance. I would also like to thank the Olivet Nazarene University Honors Program and all its faculty for providing the funding and opportunity to complete this project. Finally, a special thanks goes to the students of Honors Cohort 9 for their support and camaraderie for the duration of this project.

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To my brother Nate.
I will continue to keep you in my heart until we meet again.

ACKNOWLEDGEMENTS

Firstly, I would like to thank my parents Dan and Jenny Kok for their unrelenting support in all my endeavors. I would certainly not be where I am as a person or as a scholar without them. They were even gracious enough to allow me to use their own backyard for my testing during this study, and for that alone I am deeply grateful. I would also like to thank Professor David Ibrahim, my project mentor, for his help and guidance throughout this entire project. Beginning and finishing this project would have been infinitely more difficult without his unwavering confidence in me and expertise in the field of nuclear engineering. In the same way I thank Dr. John Tatarko for his expertise and input into my project. His freely given knowledge of chemistry and material science allowed me to solidify the actual testing procedure for this study and gave me the strong foundation to discuss my findings. Thank you to Dr. Dan Sharda for his help in refining my thesis and presentation in addition to supporting me throughout the project. My thesis would be in a state of disarray without his guidance. I would also like to thank the Olivet Nazarene University Honors Program and all its faculty for providing the funding and opportunity to complete this project. Finally, a special thanks goes to the students of Honors Cohort 9 for their support and camaraderie for the duration of this project.

ABSTRACT

The desire to reduce pollution caused by electricity production has led to a call for the replacement of conventional fossil fuel power plants. In order to fulfill this goal, a large amount of new nuclear reactors is required, and this provides the opportunity to put new and innovative reactor designs into production. The Molten Salt Reactor (MSR) is one of the most promising concepts, but a suitable combination of molten salt and container material needs to be found to reduce the potential for corrosion before the concept can be put into production. FLiNaK molten salt and the nickel-based alloy Hastelloy N have been identified as prime candidates for this function. The severity of FLiNaK corrosion in Hastelloy N requires study before this combination can be used in the operation of an MSR. In this work, Hastelloy N samples were submerged in FLiNaK molten salt for varying periods over a 55-hour interval and then hardness tested at twenty points on each sample in order to observe the progression of corrosion over time. Regression showed a statistically significant linear decrease in the hardness of the Hastelloy N samples over 55 hours submerged in the FLiNaK molten salt. When combined with previous studies, these findings indicate that Hastelloy N and FLiNaK are not a suitable pair to be used in the MSR.

INTRODUCTION

The world of energy production faces an uncertain future. It is projected that world energy consumption will increase by 34 percent in the period between 2013 and 2035 (British Petroleum, 2017). Fossil fuels have dominated the energy production market since its inception, with coal being the fuel of choice in the industrial revolution. This dominance continues in the modern world, with 81% of global energy coming from fossil fuel sources in 2011 (Khatib, 2012). The projected increase in energy consumption looks as if it will serve only to increase dependence on fossil fuels because they are the least costly means of generating the large amount of power required to meet this new demand (Shafiee & Topal, 2009). However, concerns about this dependence have arisen because of the pollution released into the atmosphere when fossil fuels are burned, and this has led to steps such as the Paris Agreement being taken to halt the climate change brought on largely by this pollution. The Paris Agreement arguably represents world opinion, and the only way to achieve those goals is to decrease dependence on fossil fuels (Nieto, Carpintero, & Miguel, 2018). However, in order to decrease dependence, an increase in the production of alternative sources of power is required.

Baseload power is a continuous supply of electricity necessary to maintain a steady, reliable power grid. It is currently produced by mainly fossil fuel plants, with nuclear contributing a small portion as well. As the world moves toward clean energy, a replacement will still be needed for the baseload power provided by fossil fuels (Reichenberg, Hedenus, Odenberger, & Johnsson, 2018). Though renewable energy sources such as wind and solar are very promising, they do not have the capacity to provide this baseload power. The inherent variability and unpredictability in the energy production from renewable sources prevent them from being able to function effectively as a baseload power source (Suman, 2018). This is illustrated by the capacity factors shown in Table 1. Capacity factors are calculated by dividing the actual production by the maximum potential production. Solar and wind power generators will run whenever possible because the "fuel" for operation is essentially free, so capacity factors are a good measure of reliability for these power sources. This means that the low capacity factors for solar and wind power indicate that they cannot be trusted to provide baseload power because of their unreliability.

Table 1

Capacity factors for different power plant types

Nuclear	Coal	Solar	Wind
92.2%	53.70%	25.7%	34.6%

Note: These capacity factors are averages taken over the year 2017. Values provided by EIA (Tyra, Cassar, Liu, Wong, & Yildiz, 2019).

Nuclear power is shown to be a very reliable source of power by its high capacity factor in **Table 1**. This makes it an ideal baseload power source, but despite this advantage the nuclear power industry has yet to increase its global energy production share to above 7.5% because of economic and safety concerns (British Petroleum, 2017). The hazardous nature of nuclear power was demonstrated by accidents at Three Mile Island (1979), Chernobyl (1986), and Fukushima (2011). These costly reactor meltdowns around the world decreased confidence in nuclear reactors, slowing the growth of nuclear power (Gu, 2018). The perceived danger of nuclear reactors also prompted more strict safety precautions, causing the cost of constructing a nuclear power plant to increase significantly to $5148/kW compared to coal's $3584/kW (Tyra, Cassar, Liu, Wong, & Yildiz, 2019). This prompted many corporations and governments to decide that nuclear power plants were not worth the risky investment (Lovering, Yip, & Nordhaus, 2016). However, reactor development has continued, and the recent call for carbon-free energy has reignited interest in nuclear energy.

In addition to the increased demand for energy, many currently operating reactors are outdated, with the average age of currently operating nuclear reactors at 29.9 years as of September 2018 (Schneider et al., 2018). The aging reactors will need to be replaced with new ones, and this opens the door for previously unemployed reactor concepts to be put into use. One of the most promising designs is the molten salt reactor (MSR) because of the advantages that it has over the reactor designs currently in use. It is both safer and more efficient than currently operational reactors, and its thorium fuel is more abundant than the uranium used in other nuclear reactor designs (Uhlíř, 2007). Though the older generation of nuclear reactors uses steam or another gas to transfer heat, the MSR has the advantage of using molten salts to transfer heat from the nuclear fuel. Molten salts can sustain temperatures as high as 1600°C without boiling, which is much higher than the 400°C maximum temperature of the pressurized water used in the common pressurized water reactor. The MSR also has a safer design than currently operating reactors, which is very desirable in a world where safety is a primary concern for any new nuclear reactor that is built. One inherent safety feature is that the molten salt coolant is capable of operating at barely above atmospheric pressure and remains liquid at very high temperatures, which removes the threat of an explosion caused by high pressure (Humphrey & Khandaker, 2018). Another inherent safety feature is that the reaction at the core is self-regulating because the rate of nuclear fission decreases as the core temperature increases (Elsheikh, 2013).

The significant advantages of MSRs also come with disadvantages though, and one of the largest disadvantages of the MSR is the corrosive nature of the molten salt in contact with the metal pressure vessel material (Serp et al., 2014). This corrosion has been observed in numerous experiments where metal samples are exposed to a molten salt coolant. These experiments were performed with the purpose of limiting corrosion in an MSR, as this is very important to the safety and cost of running an MSR. Significant corrosion will increase the operation cost and decrease the efficiency of the reactor. For the MSR to be a viable design, the corrosion must be reduced enough that it can compete with other reactor designs. It is important to consider the composition of the salt and of the container material when seeking to reduce corrosion.

In an MSR design, there are two separate molten salt coolant loops used to transfer heat from the fuel. The primary loop takes heat directly from the fuel and then transfers it to the secondary coolant loop, which in turn transfers its heat to water. Evaluating coolant for both loops is necessary, but the focus of this research is on the secondary loop. Many salts have been tested for this role, but a LiF-NaF-KF (46.5-11.5-42 mol %) mixture (FLiNaK) has been distinguished as the leading candidate because of its very good heat transfer properties, low vapor pressure, and small moderating ratio (Williams, Toth, & Clarno, 2006). The small moderating ratio means that FLiNaK salt is not an effective moderator, making it a less than optimal choice for the primary coolant loop where it would be expected to moderate the neutrons in a nuclear reaction. However, the heat transfer properties and low vapor pressure make it an excellent secondary loop coolant (Guo, Zhang, Wu, & Zhou, 2018). It was also found to be less corrosive than other molten salts with similar properties (V. Ignatiev & Surenkov, 2016).

Many materials have also been considered for molten salt coolant loops. The ideal material needs to be strong without being brittle and must be able to withstand high temperatures as well as corrosion from the salt (Ren, Muralidharan, Wilson, & Holcomb, 2011). Studies such as those by Olson et al. and Ignatiev & Surenkov have been conducted to find the material that best satisfies these requirements. It has been demonstrated that a nickel-based alloy is the most suitable material for containing FLiNaK because of the combination of strength and corrosion resistance (Ignatiev & Surenkov, 2013), and Hastelloy N has been shown to be more corrosion-resistant at a high temperature than other nickel-based alloys (Olson, Ambrosek, Sridharan, Anderson, & Allen, 2009). These properties make it the most promising candidate for use in an MSR.

FLiNaK and Hastelloy N is a promising combination for the secondary coolant loop, and as such, it has been the subject of several studies. Ouyang et al. compare the mass loss of Hastelloy N with another nickel-based alloy in their 2014 study (Ouyang, Chang,

& Kai, 2014), whereas another study found the migration tendencies of chromium and molybdenum in a similar nickel-based alloy exposed to FLiNaK by using a nuclear microprobe (Lei et al., 2017). Another study by Ye et al. examines what the corrosion of Hastelloy N in FLiNaK looks like at the microscopic level using several techniques such as x-ray diffraction and scanning electron microscopy (Ye et al., 2016). Studies such as these show the nature of FLiNaK corrosion in Hastelloy N, but they fail to determine how this affects the properties of the material. A more macroscopic study would be useful to acquire data in this area.

One property that can be used to judge the extent of corrosion is the hardness of the material. Hardness in this context is a material's ability to resist plastic deformation. This property has been used previously in studies such as those by Li and Yavas et al. to observe the progression of corrosion on a metal surface (Li, 2017; Yavas et al., 2018). The results of these studies show that a reduction in hardness can be used as indicator of corrosion, and that the hardness can be tested to observe the effect of FLiNaK corrosion on Hastelloy N. In addition, this hardness reduction can be associated with a decrease in yield strength, which is an important mechanical property considered when testing the materials used in a nuclear reactor (Cahoon, Broughton, & Kutzak, 1971).

In the experiment outlined in this paper, Hastelloy N samples were submerged in FLiNaK for varying times over a 55-hour period prior to hardness testing in order to assess the effect of FLiNaK corrosion on the hardness of Hastelloy N.

METHODS

Materials

14.059" x .75" x 2.75" Hastelloy N samples were purchased from Haynes International, Inc. and were machined down using a milling machine according to specifications (**Figure 1**). Fifty-eight grams of LiF, twenty-four grams of NaF, and 118 grams of KF were combined to meet the weight percent requirements of FLiNaK salt (Olson et al., 2009). A crucible with a 52-cubic inch capacity was used to hold the FLiNaK mixture and Hastelloy N samples for heating.

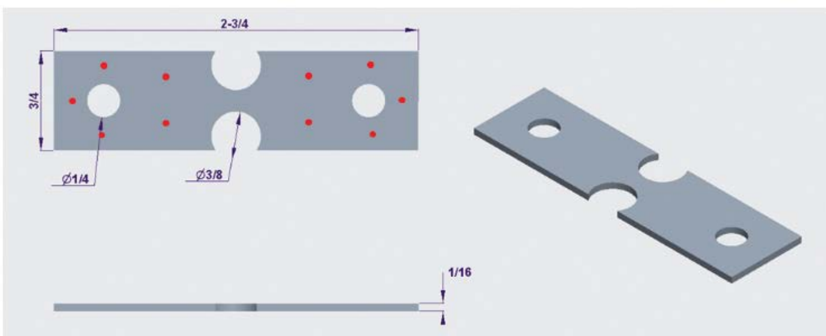

Figure 1: Dimensions (in inches) of machined Hastelloy N samples. These dimensions are based on measurements of an existing sample used for testing on the Rockwell Hardness Tester used in this study. The dots in the figure represent the approximate location that the hardness was tested (ten on both sides of the sample).

A custom stainless-steel lid was used to isolate the contents of the crucible from the outside environment, and a stainless-steel stir rod was used to mix the molten FLiNaK. Charcoal was used to provide heat for the experiment, and a custom-made apparatus was used to contain and concentrate this heat. This apparatus consisted of a hollow plaster cylinder with a small hole in the side for air flow, which was provided by a vacuum attached to this hole. A Rockwell Hardness Tester was used to find the hardness of the samples.

Procedure

The LiF, NaF, and KF were placed in the crucible together and heated to 993°C, which is the highest melting point of the three individual salts. The air flow to the charcoal was maximized to bring the contents of crucible to this high temperature. The stirring rod was then used to mix the molten salt and ensure that no solid salt remained in the crucible. Because FLiNaK is a eutectic mixture, meaning that the melting point is lower than that of its components, the melting point of the mixture dropped to its 454°C eutectic point after the initial melt. This allowed the air flow to the charcoal to be reduced in order to decrease the rate of charcoal burning.

This reduced air flow was maintained for the remaining duration of the experiment with charcoal being added every hour to ensure that the heat supplied to the crucible was constant. Eleven of the Hastelloy N samples were then submerged in the molten salt, and a single sample was taken out every five hours with the final sample being taken out after being submerged in the FLiNaK mixture for 55 hours. After the samples were removed from the molten salt, they were allowed to cool for one hour in the open air and then were cleaned in preparation for testing using an aluminum nitrate cleaning solution and subsequently water.

These eleven samples and three control samples with no exposure to the FLiNaK salt were then tested on a Rockwell hardness tester, which used a 1/16" indenting ball and a major load of about 981 N. This means that the machine measures hardness on the Rockwell B scale, which is a scale used to judge the hardness of metals. The hardness was tested on each sample at the same twenty locations shown in Figure 1, and the average of these tests was then taken as the hardness of the sample. A regression analysis was then performed on these average hardness values to determine the statistical significance of the data.

RESULTS

Hastelloy N samples were submerged in FLiNaK for varying times over a 55-hour period prior to hardness testing in order to assess the effect of FLiNaK corrosion on Hastelloy N. The average hardness calculated from twenty hardness readings on each sample is shown in Table 2 and Figure 2 for each exposure time. Whereas control samples maintained an average hardness of 80.89 ± 5.74 HRB, a progressive linear decrease in hardness was observed in the samples over the 55 hours exposure time (**Table 2 and Figure 2**). Furthermore, regression analysis revealed a statistically significant loss of 0.12% per hour in hardness ($p<0.05$).

Table 2

Hastelloy N hardness values decrease upon FLiNaK exposure

Exposure Time [hrs]	Hardness [HRB]	Standard Deviation
0	85.99	4.47
5	78.69	5.94
10	83.48	4.57
15	79.53	5.03
20	80.57	6.17
25	80.50	6.04
30	75.80	5.53
35	81.63	6.08
40	82.07	3.86
45	77.33	7.61
50	79.63	6.86
55	75.22	6.73

Note: The average hardness shown in this table is the average of the 20 hardness readings from each sample, and the standard deviation is for these 20 data points.

The HRB units represent the hardness measured on the Rockwell B Scale, which provides a meaningful comparison to other materials measured on this scale. For this study, they are simply used in comparison to the control sample.

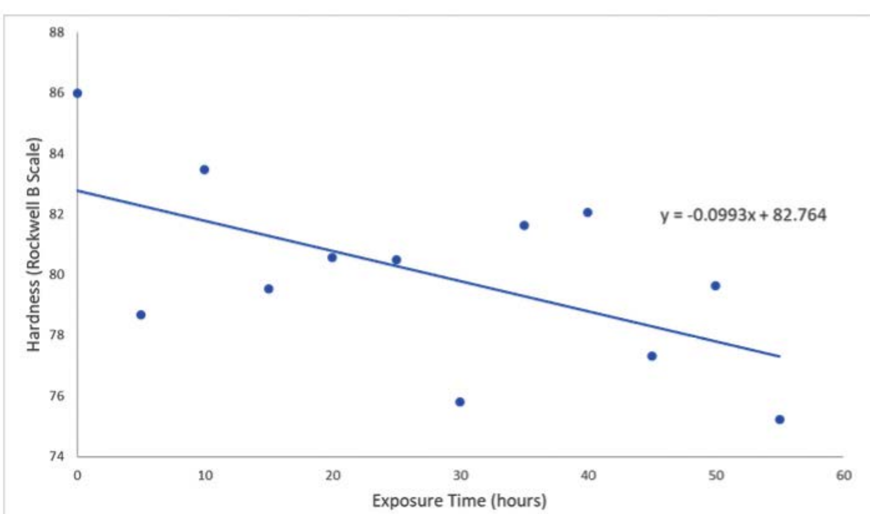

Figure 2: Hastelloy N samples hardness decrease upon increasing exposure to FLiNaK. The hardness values displayed on this plot are the average of twenty hardness readings from each of the fourteen Hastelloy N samples. A linear trend line fitted to this data is shown along with its representative equation.

As shown in Figure 2, the highest average hardness of 85.99 ± 4.47 HRB is observed in the control sample while the lowest average hardness of 75.22 ± 6.73 HRB is seen in the sample with the highest exposure time. Regression analysis revealed a negative trend representing a decrease in hardness as time exposed to FLiNaK increases, with an R square value (0.335) produced by this analysis demonstrating that approximately 33.5% of the variation in the data is accounted for by the linear model shown in Figure 2. The low p-value (0.0486) and high F-value (5.04) confirm the statistical significance based on a 90% confidence interval.

A residual plot was also produced to determine whether a linear model is the correct fit for the data (**Figure 3**). The equal distribution on either side of the x-axis of this plot indicates that the linear model shown in Figure 2 is correct.

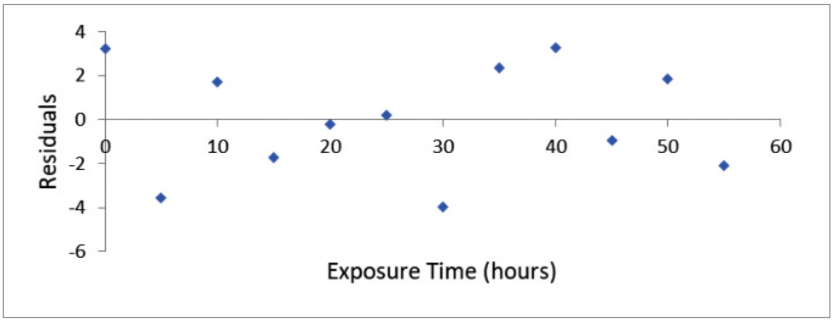

Figure 3: Plot of residuals vs the time submerged in FLiNaK molten salt. The residuals show the deviation of each average hardness value from the expected values calculated by the regression analysis, which means that ideal linear data would lie entirely on the x-axis. The exposure time is the duration that the Hastelloy N sample was submerged in the molten FLiNaK salt.

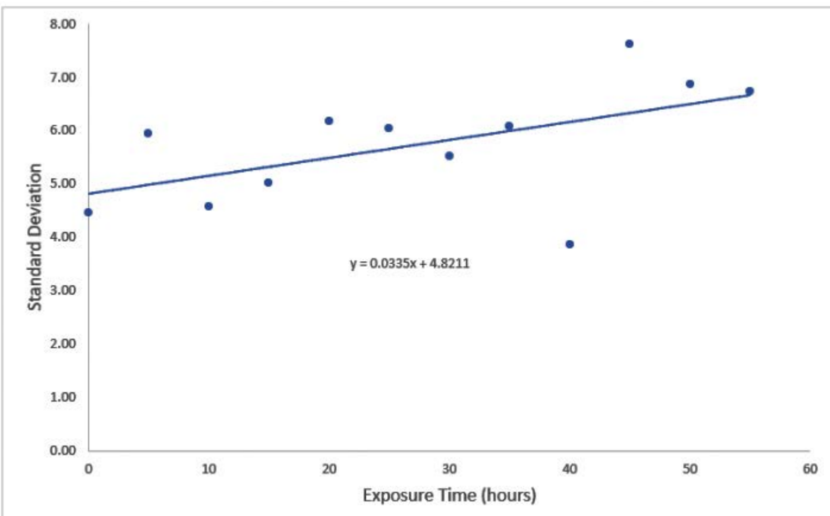

Figure 4: Standard deviation in hardness increases upon increasing exposure time. The standard deviation displayed on this plot are based on the twenty hardness readings from each of the fourteen Hastelloy N samples. The exposure time is the time that the sample corresponding to each standard deviation value was submerged in the FLiNaK molten salt. A linear trend line fitted to this data is shown along with its representative equation.

We also wanted to determine if variation of material hardness increased with exposure time. As shown in **Figure 4**, standard deviation in hardness did increase with exposure time, indicating that variation hardness in individual samples increased with increasing exposure time.

DISCUSSION

Next generation nuclear reactors, such as the molten salt reactor, are susceptible to corrosion, and thus research is needed to identify optimal materials that would allow this technology to be actualized. Here, we sought to determine if Hastelloy N samples would corrode in hardness when exposed to molten salt. Fourteen Hastelloy N samples were submerged in FLiNaK for varying amounts of time in order to determine the effect of corrosion on the hardness of the samples. This resulted in a linear decrease (-0.0993 HRB/hr) in the hardness of the samples over 55 hours of testing, including a high standard deviation in the hardness of the individual samples.

When combined, the regression analysis and residual plot demonstrate that the hardness of the Hastelloy N samples decreased in a linear fashion over the 55 hour time period. However, the standard deviation for the average hardness values is very high, with the average deviation being 5.74 HRB. This is troublesome when considering that the average hardness decrease is only 5.46 HRB over the 55 hours of the experiment. This calls into question the accuracy of the average hardness values, which, in turn, calls into question the validity of the findings from the regression analysis shown previously. In order to account for this variance, the results can be compared to those of previously conducted studies.

Having only one sample per time point prevents assessing the variation between individual samples, but the variation within samples shows an increase in standard deviation with an increase in the time submerged in molten salt (Figure 4). Hastelloy N, like all metals, is made up of grains, and the interface between the individual grains are called grain boundaries. It has been demonstrated that corrosion is most prevalent at the grain boundaries of Hastelloy N, and this is a possible explanation for the high standard deviation seen in the average hardness of the samples (Olson et al., 2009; Ouyang et al., 2014). This corrosion increases the prominence of the grain boundaries in the Hastelloy N, and it is possible that some of the locations where the hardness was evaluated were on the grain boundaries, resulting in a significantly lower value than would otherwise be observed. The measurements showing the decreased hardness at the grain boundary would increase the standard deviation in the average hardness by increasing the spread of data, and thus accounting for the greater deviation upon increased FLiNaK exposure.

Hastelloy N has chromium depletion 50 micrometers deep and a mass loss of about 2 milligrams per square centimeter of surface area after being submerged in FLiNaK molten salt for 500 hours, which indicates a progression of FLiNaK corrosion on the surface of the Hastelloy N sample over this span of time (Olson et al., 2009). The study conducted by Olson et al. indicates that the corrosion manifests primarily on the surface of the sample, making the surface weaker than the deeper material. A Rockwell hardness tester analyzes the surface of the material, so this corrosion at the surface would be expected to cause a noticeable decrease in the measured hardness of the sample. The results of this study would agree with the findings of Olson et al. in that a decrease in hardness would be expected to accompany the surface corrosion previously demonstrated.

When analyzing the use of Hastelloy N and FLiNaK in an MSR, there is a subjective aspect of determining whether it suits the needs of those designing the reactor. However, the decrease in hardness shown in this study paired with the findings of mass loss and microscopic studies done previously indicates that the rate of corrosion could be cause for concern (Olson et al., 2009; Ouyang et al., 2014). On the other hand, studies of corrosion in other materials indicate that the rate of corrosion decreases over time (Gomes et al., 2019), so additional investigation should be done on the long term FLiNaK corrosion of Hastelloy N. Previous studies of long term corrosion have used ranges of 400 hours (Rhee, McNallan, & Rothman, 1986) and 3000 hours (Froitzheim et al., 2012; Gomes et al., 2019), so somewhere in this range would be acceptable. The samples should be removed at varying times, as in the current study, and then be tested for tensile strength and hardness in order to gain an understanding of how the mechanical properties of Hastelloy N change over time while submerged in FLiNaK molten salt.

It is nearly impossible to make a definite conclusion on the merits of Hastelloy N and FLiNaK for use in an MSR without considering the effect that the accompanying corrosion has on the yield strength of the material. A significant reduction in yield strength could be the cause of a failure in the reactor, so it is of paramount importance for testing. There are connections that have been drawn between hardness and yield strength, and these relations can potentially be used to find the decrease in yield strength resulting from corrosion (Yang et al., 2018). Examining this connection is not within the scope of this project, but it is an important factor that should be the subject of future studies.

CONCLUSION

The results of this study indicate that the hardness of Hastelloy decreases linearly at a rate of 0.12% per hour. This concurs with previous studies which found that FLiNaK molten salt corrosion occurs at the surface of the sample, with the corrosion being most pronounced at the grain boundaries of the material. It is possible that the lesser hardness at the corroded grain boundaries of the sample caused the high standard deviation seen in the average hardness of the samples, which is shown to be likely by the increase in standard deviation over time submerged in FLiNaK. This short-term study, when combined with previous findings, indicate that Hastelloy N and FLiNaK should not be used together in an MSR. However, further study of Hastelloy N in FLiNaK salt is required before a definitive recommendation can be made on its potential use in a Molten Salt Reactor. Specifically, an experiment that allows the study of long-term effects of FLiNaK corrosion on Hastelloy N rather than the short-term effects shown in this study is needed. Additionally, a study should be conducted to determine the decrease in yield strength that coincides with the reduction in hardness demonstrated in this study. This can be used to get a more accurate representation of the effectiveness of Hastelloy N in a nuclear reactor after exposure to FLiNaK corrosion.

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