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Dissolution of a Can Carrier Pin in Concentrated Nitric Acid Solutions

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PREFACE OR ACKNOWLEDGEMENTS

The authors gratefully acknowledge the contributions of the following people for their assistance in the completion of these tests: T-T. Truong and D. T. Herman for consultation on initial ideas and concepts for the tests. T. E. Skidmore and B. C. Miller for providing laboratory space and materials for the tests. The laboratory space also included facilities to neutralize and dispose of the concentrated nitric acid solutions. M. L. Restivo, H. W. Baggett, and V. L. Bush for assistance in setting up the representative tests and for setting up cameras to monitor the progress of the tests. T. H. Murphy also provided laboratory support during the tests.

EXECUTIVE SUMMARY

H-Canyon will be dissolving Fast Critical Assembly (FCA) fuel from the Japan Atomic Energy Agency (JAEA). The FCA fuel will be dissolved in the electrolytic dissolver in a solution that is 50 wt.% nitric acid (HNO_3), 0.5 g/l gadolinium (Gd), and 0.05 M potassium fluoride (KF). The nitric acid concentration is expected to decrease during the batch process from 10.3 to 7 M. The temperature of the nitric acid solution may be as low as 15 °C. The fuel can will be placed in the dissolver basket insert utilizing a re-usable charging device. The charging device is a coated stainless-steel rod with a clevis design. The charging device employs a linchpin to secure the FCA fuel can as it is being charged into the H-canyon dissolver. Prior to beginning the electrolytic dissolution process, the pin will be dissolved, the fuel can will remain in the dissolver basket insert and the charging device will be removed. Currently the time necessary for complete dissolution of the pin is unknown and thus the timing of the removal of the charging device cannot be planned. This process is to be performed remotely and therefore complete dissolution of the pin will not be able to be visually determined. The pin will be made of a material that dissolves in the concentrated nitric acid solution in the dissolver. The facility desired to know the time that the pin would be dissolved so that the charging device for the FCA can could be removed from the dissolver. In particular, the pin dissolution time as a function of the nitric acid concentration and the temperature was desired. The facility would like to ensure that the solution environment was such that the charging device could be removed within an operational shift (8-12 hours).

A series of scoping tests were performed to determine the optimal pin material. Representative tests were then performed to determine the dissolution time as a function of nitric acid concentration and temperature. The following conclusions were drawn from the results of the tests.

- Carbon steel or zinc plated carbon steel experienced a passivation mechanism in concentrated nitric acid. The formation of a ferric nitrate layer on the surface of the metal decreased the rate of the dissolution reaction. The dissolution time for pins made of either of these materials will likely exceed an operational shift.
- On the other hand, 360 brass pins dissolved rapidly in the concentrated nitric acid solutions. If the temperature and nitric acid concentration are maintained above minimum values, the pin will dissolve within an operational shift. Section 5.0 provided recommendations for minimum concentration and temperature requirements for the dissolver solution.
- Based on the scoping tests and the representative tests, the presence of KF and $Gd(NO_3)_3$ had no significant effect on the dissolution rate of carbon steel or brass.
- The functional failure of the brass pin assembly was accelerated by localized corrosion mechanisms. Contact between brass and a dissimilar metal (e.g., carbon steel or stainless steel) and/or the formation of a crevice between an inert material (i.e., Teflon) or the dissimilar metal resulted in the brass corroding preferentially and at an accelerated rate. These two mechanisms, galvanic and crevice corrosion, will likely be operable in the dissolver. That is, the carbon steel ring on the pin and the stainless-steel ball and spring will likely fall to the bottom of the basket insert prior to complete dissolution of the pin. The carbon steel ring will eventually chemically dissolve while the stainless-steel parts will not chemically dissolve. These pin components will dissolve electrolytically in nitric acid.
- Functional failure of the pin assembly is expected to occur in the dissolver by a similar sequence and mechanisms. However, the facility will not be able to visually monitor the functional failure of each piece of the assembly. If the time for functional dissolution of the pin assembly from these tests is utilized, there is a risk of underestimating the optimal time for removing the charging device. Thus, it is recommended that the time based on final dissolution of the pin be utilized to guide the facility in the decision as to when to remove the charging device. This time should provide a

conservative estimate, likely 2 or more hours, for the criterion for the time at which the charging device may be removed from the dissolver.

The following recommendations for operations are made based on these test results.

- 360 brass is the recommended material for the linchpin that is used for the charging device for the FCA can.
- If the bulk temperature is greater than or equal to 25 °C, and the nitric acid concentration is greater than or equal to 8.5 M, allow a minimum of 2 hours to ensure complete dissolution of the pin.
- If the bulk temperature is greater than or equal to 25 °C, and the nitric acid concentration greater than or equal to 7 M, but less than 8.5 M, allow a minimum of 8 hours to ensure complete dissolution of the pin.
- If the bulk temperature is less than 25 °C, and the nitric acid concentration is greater than or equal to than 8.5 M, allow a minimum of 6 hours to ensure complete dissolution of the pin.
- If the bulk temperature is less than 25 °C, and the nitric concentration is less than 8.5 M nitric acid, dissolution of the pin may not occur within a 12-hour shift. Therefore, either the bulk temperature or nitric acid concentration should be increased such that pin dissolution occurs within 12 hours.

This material and these conditions should minimize the risk of removing the charging device from the dissolver too early.

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LIST OF ABBREVIATIONS

FCA	Fast Critical Assembly
JAEA	Japan Atomic Energy Agency
SRNL	Savannah River National Laboratory
SEM-EDS	Scanning electron microscopy-electron dispersion spectroscopy
SS	Stainless steel

1.0 Introduction

H-Canyon will be dissolving Fast Critical Assembly (FCA) fuel from the Japan Atomic Energy Agency (JAEA) as described by a flowsheet developed by Savannah River National Laboratory (SRNL) [1]. The FCA fuel will be dissolved in the electrolytic dissolver in a solution that is 50 wt.% nitric acid (HNO_3), 0.5 g/l gadolinium (Gd), and 0.05 M potassium fluoride (KF). The nitric acid concentration is expected to vary during the batch process from 7 to 10.3 M. The temperature of the nitric acid solution may be as low as 15 °C. The fuel carrier can will be placed in the dissolver basket insert utilizing a re-usable charging device (see Figure 1-1). The charging device is a coated stainless-steel rod with a clevis design. The charging device employs a linchpin to secure the FCA fuel can as it is being charged into the H-canyon dissolver. Prior to beginning the electrolytic dissolution process, the pin will be dissolved, the fuel can will remain in the dissolver basket insert and the charging device will be removed. Currently the time necessary for complete dissolution of the pin is unknown and thus the timing of the removal of the charging device cannot be planned. This process is to be performed remotely and therefore complete dissolution of the pin will not be able to be visually determined. A linchpin is used to secure the FCA fuel can as it is being charged into the H-canyon dissolver. The pin will be made of a material that dissolves in the concentrated nitric acid solution in the dissolver. The facility desired to know the time that the pin would be dissolved so that the charging device for the FCA container could be removed from the dissolver. In particular, the pin dissolution time as a function of the nitric acid concentration and the temperature was desired. The facility would like to ensure that the solution environment was such that the charging device could be removed within an operational shift (8-12 hours).

H-canyon provided three pin alternatives for testing [2-4].

- A zinc-plated carbon steel bent clevis pin;
- A carbon steel bent clevis pin;
- A 360 brass ring grip quick release pin.

A brief literature review indicates that the dissolution of zinc and carbon steel is relatively rapid in nitric acid environments [5-7]. For example, steel corrosion rates as high as 13 inches/yr have been observed at 6 M nitric acid [7]. However, this rate decreases to nearly nil at concentrations greater than 13 M nitric acid due to passivation of the surface by a ferric nitrate compound [7, 8]. Thus, it is expected that the dissolution rate may decrease as the nitric acid concentration increases from 7 to 10.3 M. Corrosion rates of carbon steel in nitric acid typically increase with temperature. For example, as the temperature was increased from 25 °C to 35 °C in 1 M nitric acid, the corrosion rate doubled [9]. Thus, the bounding temperature of 15 °C would tend to reduce the corrosion rate. On the other hand, the contact between the pin and the charging device creates a crevice, which has been shown to disrupt the passivation process and increase the rate of steel dissolution [8]. Furthermore, contact between the zinc and iron also results in a galvanic effect that may increase the dissolution rate [5]. The effect of the Gd and KF has not been quantified for steel, however, the presence of the halides (chlorides or fluorides) may increase or decrease the corrosion rate of iron alloys in nitric acid environments in this concentration range [10]. These confounding factors make it necessary to perform tests in a representative environment, rather than rely solely on literature values to predict the time to complete dissolution.

Figure 1-1. Charging Device with Zinc Coated Carbon Steel Pin.

Brass is an alloy material fabricated from copper (~60 wt.%) and zinc (~35% wt.%). These transition metals are also expected to dissolve rapidly in nitric acid environment [8]. Dezincification of brass has been reported in the literature which causes preferential zinc leaching resulting in porous/fragile structure that is prone to mechanical failure [11]. However, in this case passivation of the surface does not occur. Thus, the time for dissolution of the pin will likely be primarily influenced by nitric acid concentration and temperature. The pin assembly has components that are made of dissimilar metals (i.e., carbon steel and stainless steel (SS)) and tight crevices will exist at the carrier handle and the pin. These features were expected to accelerate corrosion locally.

Two series of tests were performed. The first series of tests were referred to as scoping and the objective of these tests was to determine the optimal pin material. The second series were referred to as representative and the objective of these tests was to determine the pin dissolution time as a function of nitric acid concentration and temperature. Two recommendations were made from the tests:

- The optimal pin material;
- The minimum temperature and nitric acid concentration that will ensure that the pin has dissolved within an operational shift (8-12 hours).

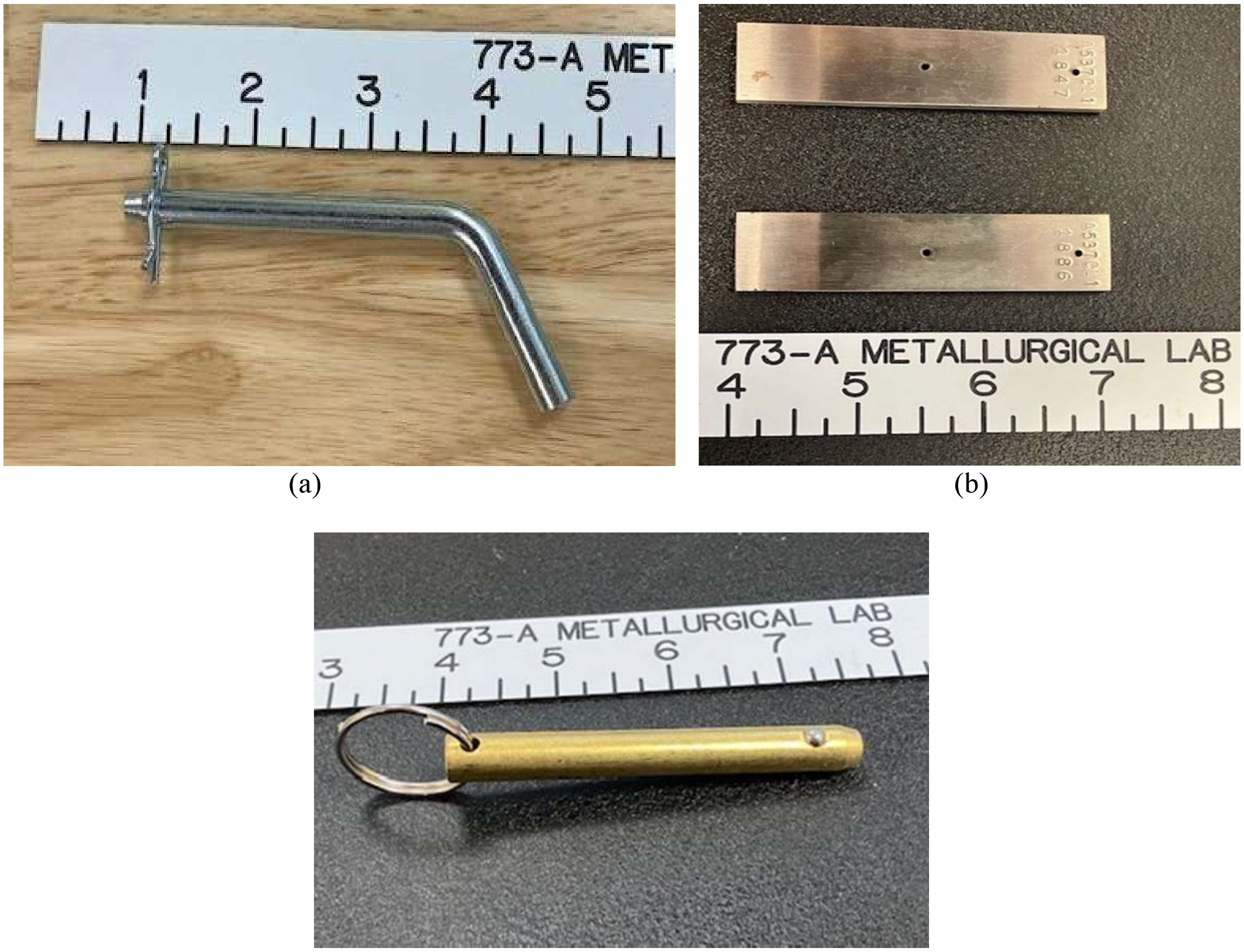
2.0 Experimental Procedure

2.1 <u>Scoping Tests</u>

The objective of the scoping tests was to determine the optimal material for the linchpin. The materials were tested at possible nitric acid concentrations in the dissolver. No attempt was made to control the temperature in these tests, although it was measured as a means of monitoring the intensity of dissolution. The materials tested, the solutions employed, and the test set-up are described next.

2.1.1 Materials Tested

Figure 2-1 shows the materials that were tested during the scoping tests. Photograph (a) shows a zinc-plated carbon steel bent clevis pin [2]. Photograph (b) shows ASTM A537 carbon steel coupons [12]. The facility also has access to carbon steel bent clevis pins that are not zinc plated. The coupons were used to represent the material for these pins. Photograph (c) shows the ring grip quick release pin that was fabricated from 360 brass [4].



(a) (b)

(c)

Figure 2-1. Materials for Initial Scoping Tests (a) Zinc Plated Carbon Steel Pin, (b) ASTM A537 Carbon Steel Coupon, and (c) Leaded Brass Pin.

The chemical composition for the pin materials is shown in Table 2-1. The AISI 1045 carbon steel and the A537 carbon steel show minor differences in the chemical constituents. Most of these differences (e.g., carbon concentration) influence the mechanical properties of the steel. For example, the higher carbon concentration for the AISI 1045 steel would indicate that the material has greater strength than the A537 steel. Nevertheless, these differences would not have a significant influence on the corrosion properties of the steel in an acidic environment. The brass pin material primarily consists of copper and zinc. A small quantity of lead (Pb) is present in the alloy to improve machinability of components. It should also be noted that the brass pin contained a carbon steel ring as well as a stainless-steel ball and spring mechanism.

Table 2-1. Pin and Can Materials Tested (wt.%)

Material	C	Mn	P	S	Fe	Cr	Mo	Ni	Cu	Zn	Pb
1045 Carbon Steel	0.42-0.5	0.6-0.9	≤0.04	≤0.05	98.51-98.98	-	-	-	-	-	-
ASTM A537 Carbon Steel	0.24	0.15-0.5	0.7-1.35	0.035	97.17-98.17	0.025	0.08	0.25	0.35	-	-
360 Brass	-	-	-	-	0.35	-	-	-	61.5	35.5	3

2.1.2 Solutions Tested

Six solutions were proposed for the scoping tests. Nitric acid was present in all test solutions. The nitric acid concentrations covered the range of anticipated concentrations in the dissolver [2]. There was an option to utilize solutions that did not contain either KF or $Gd(NO_3)_3$. These solutions would be utilized to determine if these small quantities of chemicals influenced dissolution of a material. It should be noted that only complete simulants (i.e., Solutions 4-6) were utilized for the representative tests. Throughout the discussion in the text of this report, the solution numbers from this table will be referenced.

Table 2-2. Solution Composition Test Alternatives

Solution Condition	7 M HNO_3	8.5 M HNO_3	10.3 M HNO_3	0.0009 M KF*	0.5 g/l $Gd(NO_3)_3$
1	X				
2		X			
3			X		
4	X			X	X
5		X		X	X
6			X	X	X

*See discussion below Table 2-2

The intent of this program was to utilize 0.05 M KF for these tests [2]. Approximately one month after the principal investigator completed the draft report, but prior to issuing the report, they noticed a typographical error in the directions used to prepare the 1 L solutions. Rather than 0.05 M (or 2.9 g/l) KF, the directions read 0.05 g (or 0.0009 M). All solutions were prepared in this manner. To determine the significance of this error, a representative test (see Section 2.2) in Solution 6 with a KF concentration of 0.05 M at a bulk temperature of 25 °C was performed.

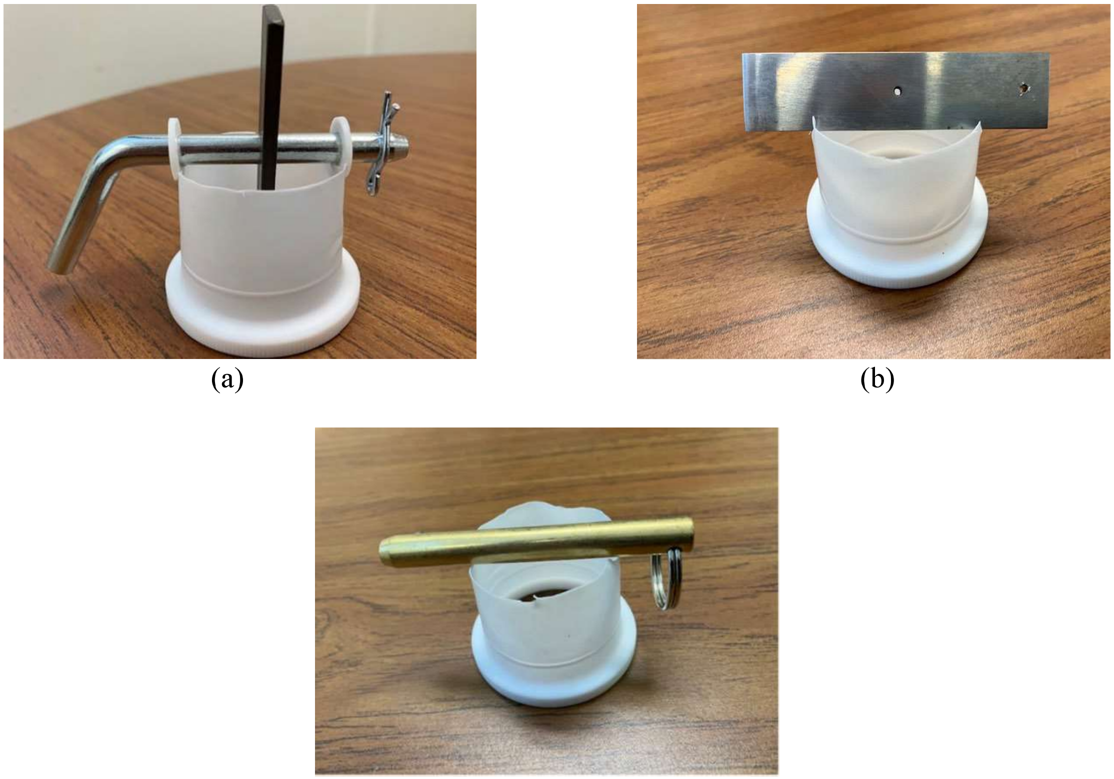
2.1.3 Test Set-up and Execution

The test configuration or pin assembly for the scoping tests mirrored some of the conditions in the dissolver, but not completely. Table 2-3 and Figure 2-2 describe and illustrate the pin assembly for the scoping tests. The zinc plated carbon steel pin (i.e., configuration 1) was set-up in a configuration that was like that in the dissolver. The 410 stainless-steel [13] coupon is the same material that was used to fashion the can carrier handle that will rest on the pin. The Teflon washers and platform are representative of the inert polymer coating that covers the charging device and is in contact with the pin.

The selection of the configuration for the coupon and the brass pin was influenced by the scoping test results for the zinc-plated carbon steel pin (see Section 3.1 for discussion). Therefore, in these tests, the objective was primarily to observe how the material itself reacted with the concentrated nitric acid without considering crevice or galvanic corrosion effects. The platform was used simply to suspend the coupon in the solution, while minimizing contact with the metal surface.

Table 2-3. Material Configuration in Test Set-up.

Material Configuration	Zinc Plated Carbon Steel Pin	ASTM A537 Carbon Steel Coupon	360 Brass Pin with Nickel Plated Ring and Stainless-Steel Ball	Teflon Washer	Teflon Platform	410 Ferritic Stainless Steel
1	X			X	X	X
2		X			X	
3			X		X	



(a)

(b)

(c)

Figure 2-2. Materials Configuration for Initial Scoping Tests (a) Zinc Plated Carbon Steel Pin, (b) ASTM A537 Carbon Steel Coupon, and (c) 360 Brass Pin.

The scoping tests were conducted in a 2-liter beaker. Approximately 1 liter of the test solution was prepared so that the sample would be completely immersed in the acid. Figure 2-3, Figure 2-4, and Figure 2-5 show the test configuration for each material condition. A thermocouple was placed next to the pin to monitor the local temperature during the test. No attempt was made to control the temperature during the test, but temperature data was recorded to assess the intensity of the reaction.

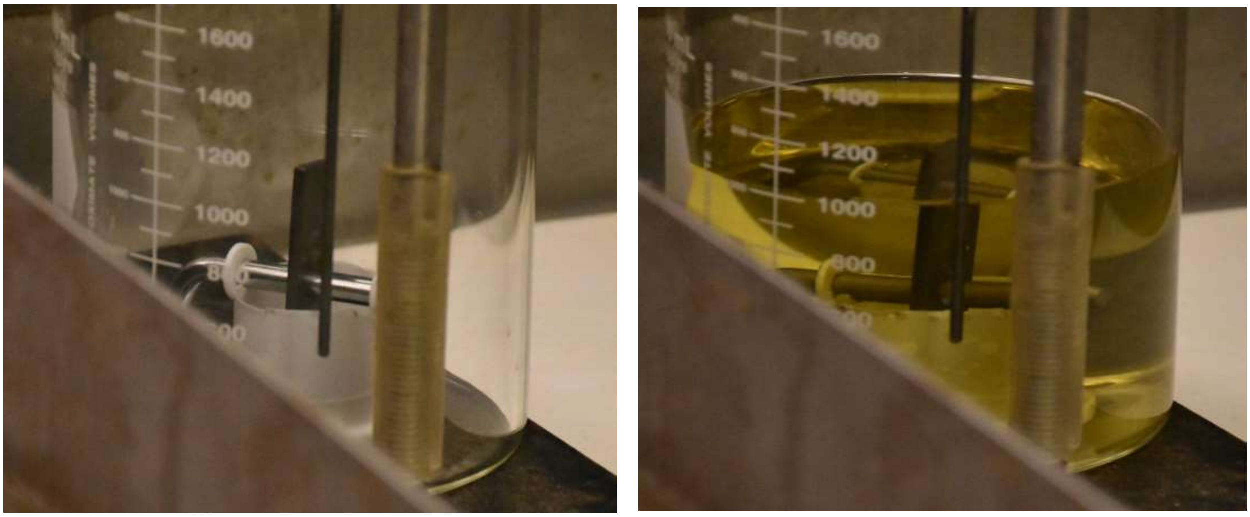

(a) (b)

Figure 2-3. Test Configuration with Zinc Plated Carbon Steel Pin

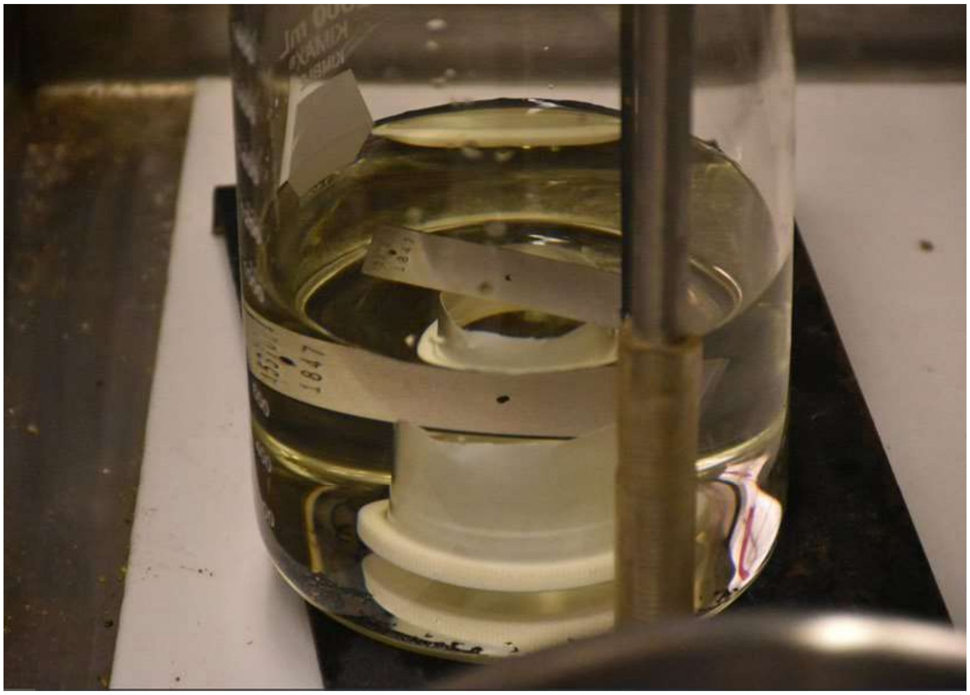


Figure 2-4. Test Configuration with A537 Carbon Steel Coupon

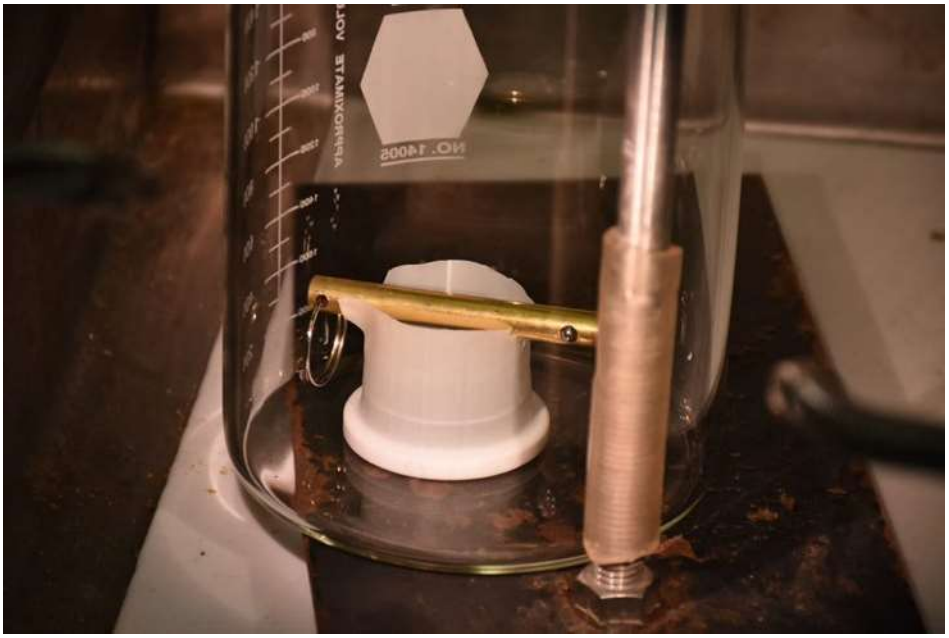


Figure 2-5 Test Configuration with 360 Brass Pin

2.2 Representative Tests

The objective of the representative tests was to determine the pin dissolution time as a function of nitric acid concentration and the bulk solution temperature. The materials were tested at possible nitric acid concentrations in the dissolver. Contact with dissimilar metals and creviced areas were designed into the pin assembly configuration. The bulk test solution temperature was controlled during the test, while local fluctuations in temperature near the dissolving pin were monitored. The materials tested, the solutions employed, and the test set-up are described next.

2.2.1 Material Tested

The scoping tests identified 360 brass as the optimal pin material. Therefore, all representative tests were performed with the ring grip quick release pin. The pin assembly also contained a carbon steel ring and a stainless-steel ball and spring mechanism. A Teflon holder was designed to suspend the pin and represents the inert polymer coating on the charging device. Details on specific pieces of the pin assembly are illustrated in Figure 2-6. This figure will be referred to frequently in Section 3.2 during the discussion of results.

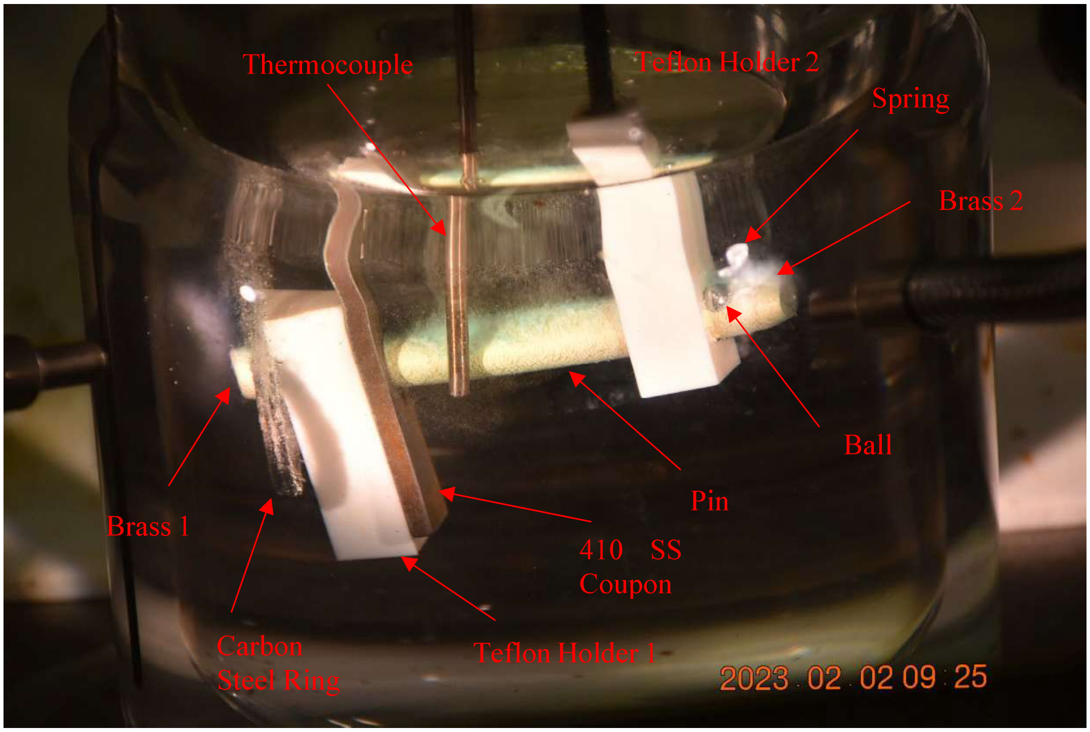


Figure 2-6. Brass Pin Assembly

2.2.2 Solutions Tested

Full simulants were utilized for the representative tests (i.e., Solutions 4, 5 and 6 from Table 2-2). Each solution was tested at a bulk temperature of 15 °C, the anticipated minimum temperature of the dissolver solution, and at a bulk temperature of 25 °C, a realistic ambient temperature for the dissolver.

2.2.3 Test Set-up and Execution

The representative tests were conducted in the vessel-in-vessel apparatus shown in Figure 2-7. The pin assembly and test solution were placed in the inner vessel. The pin was suspended in the test solution by an all-thread rod that was screwed into Teflon Holder 2 (See Figure 2-6 and Figure 2-8). The all-thread rod was supported by a ring stand. A pressure gauge dial was utilized in the system to detect movement of the pin assembly. This movement would be coincident with changes in the weight of the pin assembly due to pin dissolution. Cooling water was circulated through the jacket surrounding the inner vessel to maintain the bulk temperature of the test solution. The thermocouple located in the jacket is considered to be representative of the bulk solution temperature. The chiller utilized to circulate the water is shown in Figure 2-9. Two thermocouples were utilized. One thermocouple was placed in the jacket area and was utilized to monitor the cooling water temperature. The second thermocouple was placed adjacent to the pin assembly (See Figure 2-6) to monitor local temperature. A temperature read-out was connected to the thermocouples so that the temperatures could be recorded.

Figure 2-7. Test Vessel for Representative Tests

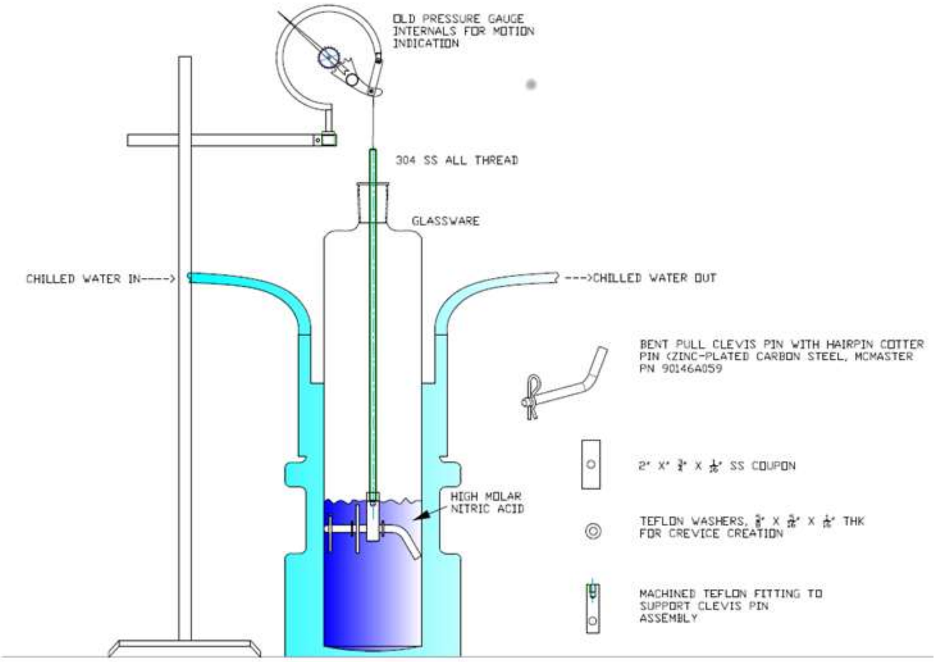


Figure 2-8. Schematic of Test Set-up

Figure 2-9. Chiller Utilized for Representative Tests

The tests were initiated by starting the chiller and circulating water through the jacket at either 15 or 25 °C. Once the bulk temperature was established, approximately 1 liter of the test solution was added to the vessel. This solution was then allowed to equilibrate with the water in the jacket until the solution was within 1 degree of the temperature of the water in the jacket. The pin assembly was then lowered into the solution to begin the test.

Two cameras were utilized to visually monitor the progress of the test. The first camera was positioned to take pictures of the pin while it was suspended in solution. The camera captured time-lapse photographs to show when significant changes in the pin assembly occurred. Once the pin assembly failed (i.e., all parts of the pin assembly were on the bottom of the vessel), the final stages of pin dissolution was monitored by a hand-held camera. The times at which various pieces of the pin assembly fell to the bottom of the vessel and the time for complete pin dissolution were recorded. General visual observations of the test solution and gas above the solution were also recorded. Finally, the local temperature near the pin and the bulk temperature of the water circulating in the jacket were recorded.

2.3 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

Data for this task was recorded in the electronic laboratory notebook system, notebook number Y7834, Experiment 00590-01. Details on the calibration of balances, thermocouples and calipers were recorded in this notebook. Directions for the tests and observations of results were also placed in this system.

3.0 Results and Discussion

The results from the scoping and representative pin dissolution tests are summarized in this section. Based on the results of these tests the pin material was selected and the time for pin dissolution was estimated [2].

3.1 Scoping Tests

3.1.1 Zinc Plated Carbon Steel Pins

The zinc plated carbon steel bent-pull clevis pin was tested initially [2]. In addition to the pin, the 410 SS coupon, the Teflon washers and platform were included in the assembly (see Figure 3-1). Solution 3 from Table 2-2, 10.3 M nitric acid, was utilized for the test. Approximately 5 minutes after the pin had been immersed in the nitric acid, the solution began to turn a yellowish green as shown in Figure 3-1. Shortly after that a few bubbles were noticed beneath the Teflon washers, however, no brown gas, which is indicative of NO_x, was observed. After approximately 20 minutes of exposure the pin began to lose its initial luster or "shininess" and become a brown or orangish color. Small "filaments" of solid material were observed floating above the sample. During the first 30 minutes of testing, the temperature increased approximately 9 °C, however, after that began to decrease slowly back to the original temperature of 19.6 °C. No significant changes in the sample or solution appearance were observed over the next 5 hours.

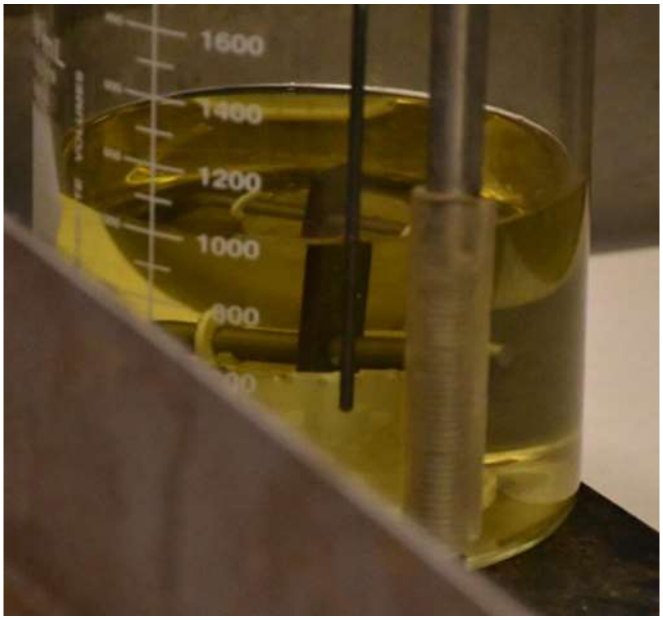


Figure 3-1. Zinc Plated Pin in 10.3 M Nitric Acid

At the completion of the test, visual observations and photographs of the pin were made (see Figure 3-2). In general, the shiny zinc electro-plated layer had disappeared, and the pin had areas that were either dull gray or orangish brown in appearance. Areas near the Teflon washer appeared to be more discolored (orangish-brown) than most of the pin, indicating perhaps more attack in this region although it was clearly limited. No galvanic or crevice corrosion was observed on the pin adjacent to the 410 SS coupon. The 410 SS coupon was also unattacked.

Figure 3-2. Post-Test Condition of Zinc Plated Carbon Steel Pin.

The lack of NO_x generation, the mottled appearance of the sample, the solution color change, and the absence of a significant temperature increase suggested that there was an additional barrier to the zinc and steel surfaces and that passivation of the carbon steel had occurred. The drawing for the pin states that the pin was plated or rather electroplated in a zinc bath. A note on the drawing also states that the pin has good strength and "some corrosion resistance". A review of ASTM standard B633 indicates that the zinc electroplated surface may receive a "supplementary treatment" after the plating process [14]. These treatments include chromate conversion coatings, passivation coatings, and phosphate coatings. Phosphate coatings are black and were therefore most likely not utilized. Chromate and passivation coatings may be colorless per the ASTM practice.

A pin in the as-received condition was examined by scanning electron microscopy and electron dispersion spectroscopy (SEM-EDS) to detect the surface layer. The SEM micrograph and the EDS spectra are shown in Figure 3-3. A chromium elemental peak was observed indicating that the supplementary treatment was most likely a chromate conversion coating.

The yellowish-green appearance of the solution and the mottled appearance of the pin suggested that the coating had begun to break down. Although neither the solution or the small "filaments" were analyzed, the color of the solution and the appearance of what appears to be protected areas and slightly corroded areas (i.e., mottled appearance) provides a degree of confirmation of this hypothesis. The orangish-brown color is reminiscent of ferric nitrate compounds that passivate carbon steel in concentrated nitric acid solutions [5]. Both the chromate coating and iron passivation appear to retard the dissolution of the pin. Therefore, the customer requested other pin materials be tested [4].

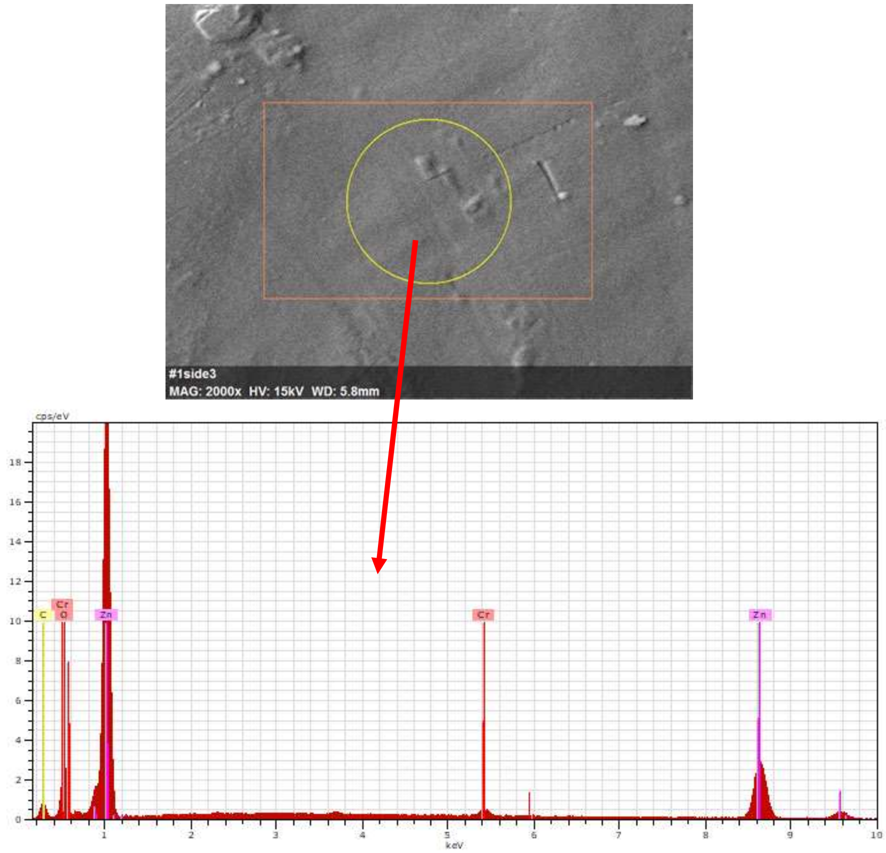


Figure 3-3. SEM-EDS of As-received Zinc Plated Carbon Steel Pin.

3.1.2 Carbon Steel Coupons

A carbon steel clevis pin was also available to the customer as an alternative [3]. To confirm that passivation of carbon steel occurred in the concentrated nitric acid during the tests with the zinc plated carbon steel, scoping tests with ASTM A537 carbon steel were conducted. Two coupons were tested. One coupon was immersed in Solution 3, and the second was immersed in Solution 6, which contains KF and $Gd(NO_3)_3$ at the concentrations prescribed in Table 2-2. The coupon was positioned on a Teflon platform in the vessel as shown in Figure 3-4. A stainless-steel thermocouple was placed in the beaker to monitor temperature change. Teflon washers and a 410 SS coupon were not utilized in these tests. The Teflon platform does however create a crevice region on the coupon.

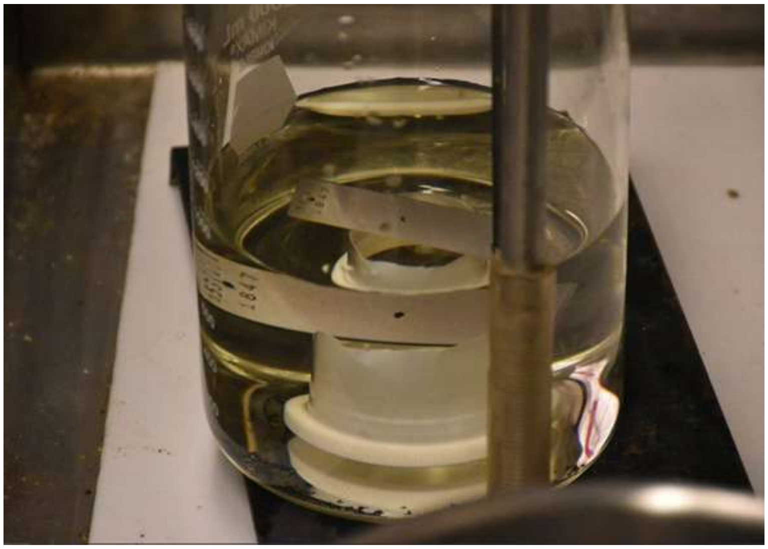


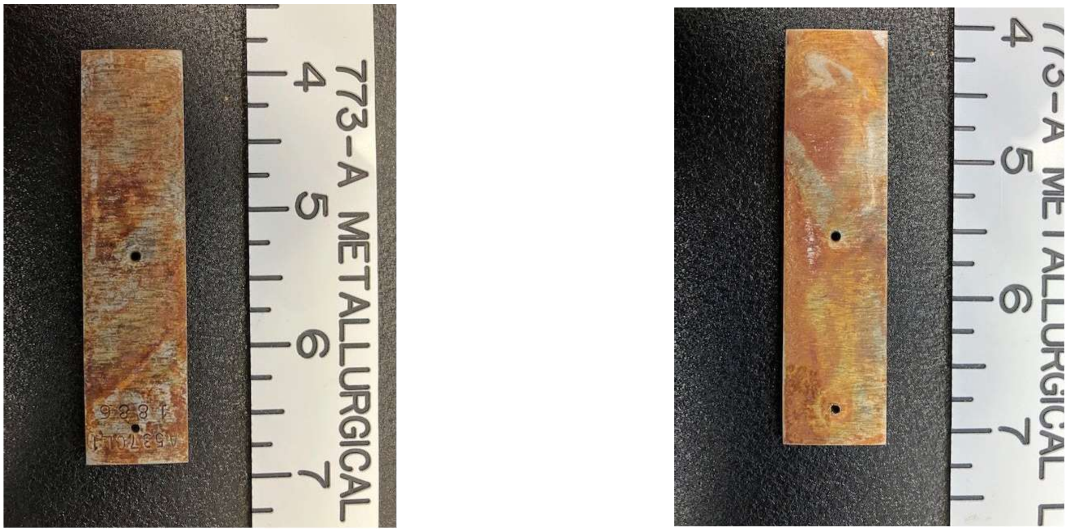
Figure 3-4 ASTM A537 Carbon Steel Coupon in 10.3 M Nitric Acid.

The test in Solution 3 was conducted for 8 hours. During that time the solution remained clear, there was no discernable local temperature change, and no evolution of a brown gas. The coupon surface gradually changed from a bright, silver color, to a dull gray and to ultimately orangish-brown (see Figure 3-5). The initial polishing marks and engraved number on the coupon were still present after the test, which suggested limited attack. The orangish-brown color also suggested that the passive ferric nitrate compound had formed on the surface.

a) b)

Figure 3-5. Post-test Condition of Carbon Steel Coupon Tested in Solution 3: (a) front and (b) back.

The test in Solution 6 was conducted for 5 hours. As with the test in Solution 3, the solution remained clear, there was no discernable local temperature change, and no evolution of a brown gas. The coupon surface gradually changed from a bright, silver color, to a dull gray and to ultimately orangish-brown as before. The initial polishing marks and engraved number on the coupon were still present after the test, which suggested limited attack. Qualitatively it appears that the addition of KF and $Gd(NO_3)_3$ had no effect on the rate of corrosion of the carbon steel at these low concentrations. The orangish-brown color also suggested that the passive ferric nitrate compound had formed on the surface.



a) b)

Figure 3-6. Post-test Condition of Carbon Steel Coupon Tested in Solution 6: (a) front and (b) back.

The coupons were weighed after the test so that the corrosion rate for each may be calculated. Given the weight change of the coupon during the test, the test time, the surface area of the coupon and the density of the steel, the corrosion rate may be calculated [15]. The corrosion rate for the coupon immersed in Solution 3 was 0.228 in/yr, while the corrosion rate for the coupon immersed in Solution 6 was 0.258 in/yr. Although the corrosion rate was slightly higher for the coupon in the solution with the KF and $Gd(NO_3)_3$, the difference may not be statistically significant given that only a single test was performed. The literature indicated that for 6 M nitric acid the corrosion rate is approximately 13 in/yr, while at 13 M nitric acid the corrosion rate decreases to 0.02 in/yr due to passivation. The corrosion rates measured in these tests are consistent with the literature observation and show the influence of passivation.

As an exercise, these corrosion rates were utilized to estimate the time to dissolve a carbon steel pin of the same dimensions as the zinc plated carbon steel pin. If the pin is 3/8" diameter, and it is assumed that uniform dissolution occurs, it would take approximately 265-300 days to dissolve the pin. Given that it was desired that the pin dissolve within an 8–12-hour shift, a carbon steel pin would not give the desired service.

3.1.3 360 Brass Pins

A 360 brass pin is also available to the customer [4]. Two 360 brass pins were tested separately with the same set-up as the carbon steel coupon (Figure 3-7). The first pin was tested in Solution 6, while the second pin was tested in Solution 4 (see Table 2-2). In addition to nitric acid, the KF and $Gd(NO_3)_3$ were added to the solutions.

The first test was performed in Solution 6. Initially the local temperature was 21.8 °C. Immediately upon immersion of the sample in the acid, bubbles formed on the sample and the solution began to turn green (see Figure 3-7). Within seven minutes of initiating the test, the solution test temperature had increased to 24.6 °C and the stainless-steel ball and spring had fallen to the bottom of the vessel (see Figure 3-8). Neither the spring nor the ball were corroding. Additionally, the carbon steel ring did not appear to be corroding significantly either. On the other hand, the brass pin continued to dissolve at a rapid rate as evidenced by the steady stream of bubbles and the darker green solution. After 15 minutes of exposure, the local temperature of the solution had increased to 34.7 °C and the intensity of the bubbles increased, and the first clear indication of a brown gas (NO_x) was observed.

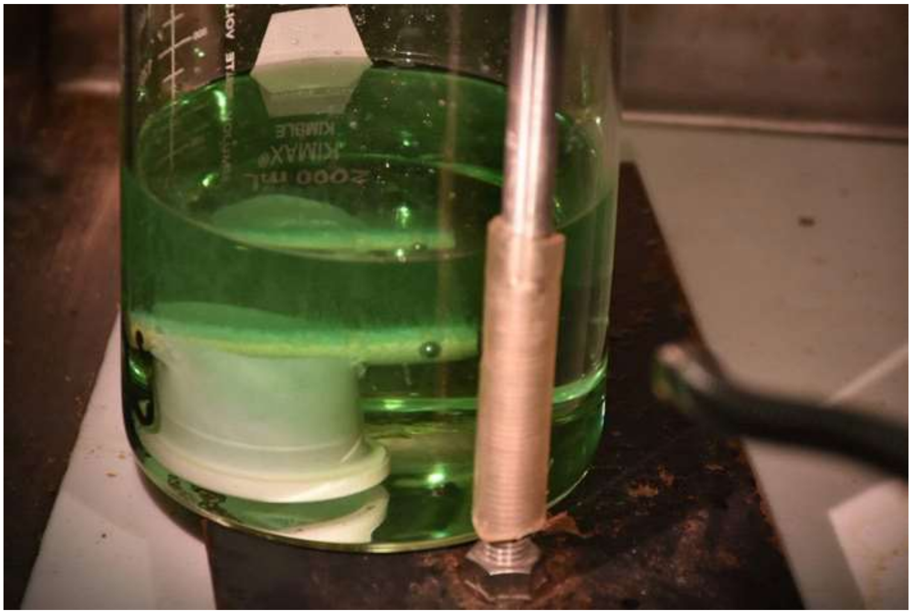


Figure 3-7. 360 Brass in 10.3 M Nitric Acid Initial Immersion.

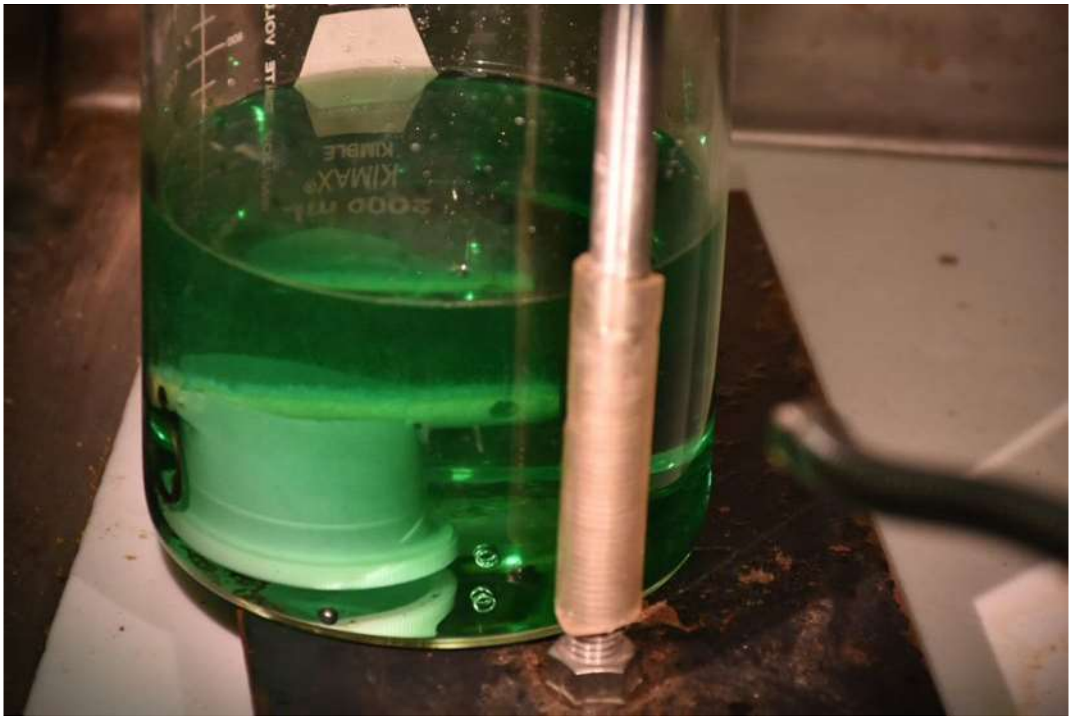


Figure 3-8. 360 Brass in 10.3 M Nitric Acid; Ball and Spring Fall to Bottom of Vessel.

After 21 minutes, a piece of the pin that had extended over the edge of the Teflon platform fell to the bottom of the vessel next to the stainless-steel ball (see Figure 3-9). Dissolution of the pin and piece was rapid as evidenced by the violent bubbling and the formation of a thick layer of brown gas above the dark green solution. The local temperature of the solution reached its maximum value of 50.2 °C. Shortly after this picture was taken, the carbon steel ring fell to the bottom of the vessel. Violent bubbling continued for another six minutes until the pin had completely dissolved. The absence of bubbles and foam at the surface was the indication that the pin had completely dissolved (see Figure 3-10). Notably the carbon steel ring did not dissolve significantly, once again displaying passivity at the concentrated nitric acid conditions.

Figure 3-9. 360 Brass in 10.3 M Nitric Acid; Piece of Pin Dissolving Next to Ball.

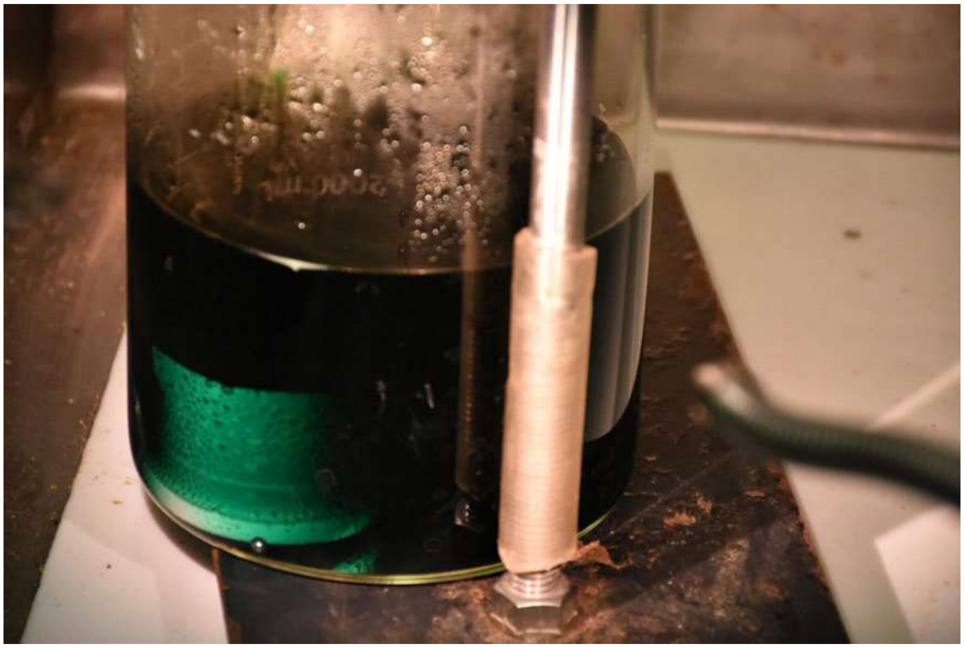


Figure 3-10. 360 Brass in 10.3 M Nitric Acid; Test Complete as Indicated by no Bubbling.

The test performed on 360 brass in Solution 4 followed a similar pattern, only the kinetics were slower as expected for the lower nitric acid concentration. For example, the ball and spring fell to the bottom of the vessel after 13 minutes of exposure vs. 7 minutes of exposure for Solution 6. Qualitatively, the gas generation, as evidenced by the bubble formation, was observed to be slower and the rate at which the solution became a dark green was slower. Another measure of the decrease in the reaction rate was the rate at which the local temperature increased during the test. Figure 3-11 shows that the initial rate of increase in the local temperature for Solution 4 is significantly less than that for Solution 6. This trend is indicative of less heat being given off due to a slower reaction rate. The brass pin dissolved in 50 minutes for the Solution 4 or at a rate approximately half that of the solution with the higher nitric acid concentration.

Finally, the carbon steel ring corroded more severely in Solution 4 than in the more concentrated Solution 6. Bursts of bubbles that circled the ring followed by flakes of iron oxide being expelled from the surface of the ring were evident. The higher rate of attack of the carbon steel at the lower nitric acid concentration is consistent with the literature, which indicates that there is less passivation of iron at the lower nitric acid concentrations.

The dissolution rate at both nitric acid concentrations met the time criteria for pin dissolution of less than a shift. Thus, 360 brass was selected as the pin material. Tests at representative dissolver conditions were performed next to determine time to pin dissolution.

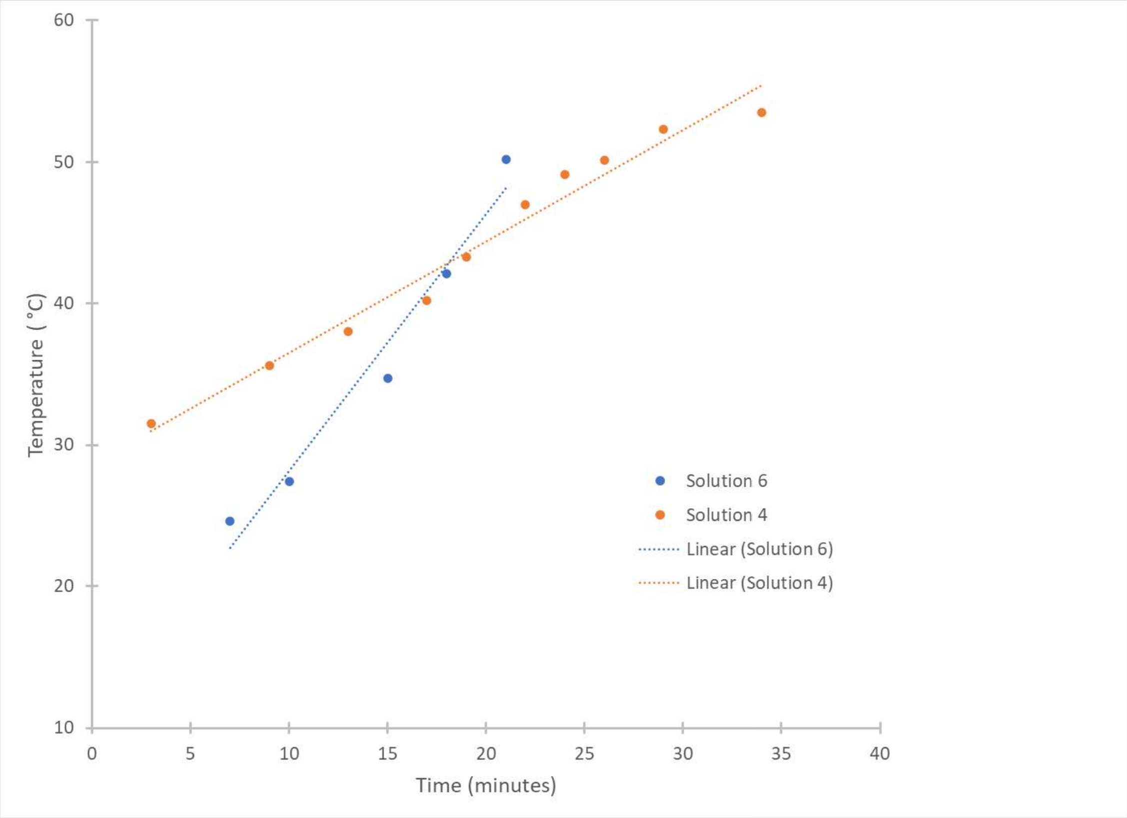


Figure 3-11. Comparison of Solution Temperature for Tests in Solution 4 and Solution 6.

3.2 Representative Tests

Table 3-1 is the test matrix for the representative tests in terms of the test solution and the bulk temperature. Tests at three nitric acid concentrations and two bulk temperatures were performed. The table shows only the nitric acid concentrations, but 0.0009 M KF and 0.5 g/l Gd were also added to each solution. (Note: A confirmatory test with 0.05 M KF was also performed. These results will be compared to the solution with lower KF concentration). Up to three tests were performed at each condition to assess variability. Two failure times were monitored during the test: 1) Functional failure of the pin assembly, and 2) Complete dissolution of the pin. Functional failure was defined as the time at which the pin and all the parts (e.g., ring, ball, spring, coupon, etc.) had fallen to the bottom of the vessel. During the test, the time at which each part fell to the bottom was recorded as well. Complete dissolution was determined when the bubbles from the pin were no longer visible. This visual assessment was correlated with the disappearance of the pin.

Table 3-1. Test Matrix for Representative Tests

Nitric Acid Concentration (M)	Temperature (°C)	
	15	25
7 (Solution 4)	X	X
8.5 (Solution 5)	X	X
10.3 (Solution 6)	X	X

See Table 2-2 for reference to Solutions. All solutions contain 0.0009 M KF and 0.5 g/l Gd.

Functional failure of the pin assembly was accelerated by crevice and/or galvanic corrosion and generally followed the same sequence for all the tests. Figure 3-12 through Figure 3-18 illustrate these two localized mechanisms. The dislodging of the ball and spring occurred first and was due to the dual mechanism of crevice and galvanic corrosion (See Figure 3-12). The ball and spring are fabricated of stainless steel and the dissimilar metal contact (galvanic) preferentially accelerated dissolution of the brass. The depressed area in which the ball rested created a tight crevice where liquid could seep in between the ball and pin. This action accelerated corrosion in the area beneath the ball.

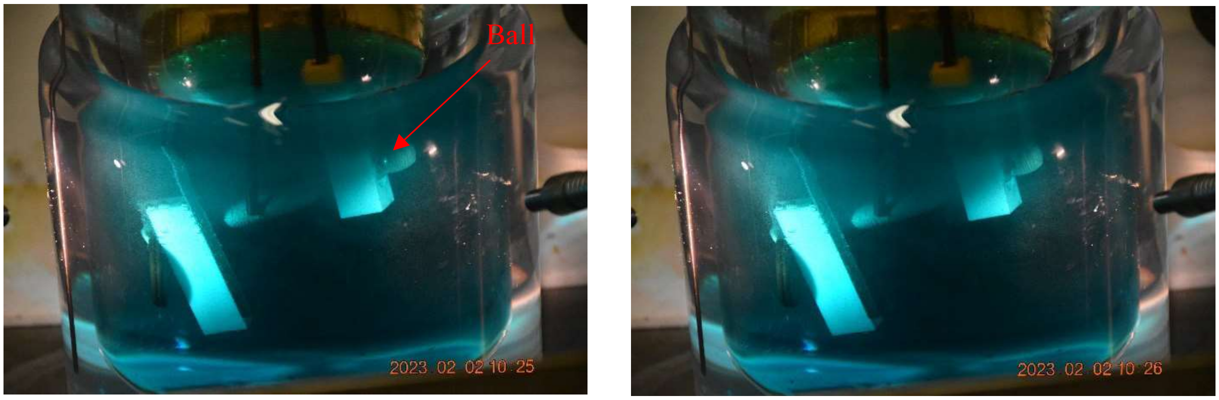


Figure 3-12. 360 Brass in 8.5 M Nitric Acid at 15 °C; Ball and Spring Falling to Bottom of Vessel.

The second piece of material that fell to the bottom of the vessel was the piece of brass (Brass 2 in Figure 2-6). This event is shown in Figure 3-13. This piece failed due to the corrosion induced by the ball and spring, and crevice corrosion between Teflon Holder 2 and the brass pin. This piece likely fell off first because the diameter of the pin decreased in this area. Once on the bottom of the vessel, general dissolution of the brass continued until the piece disappeared.

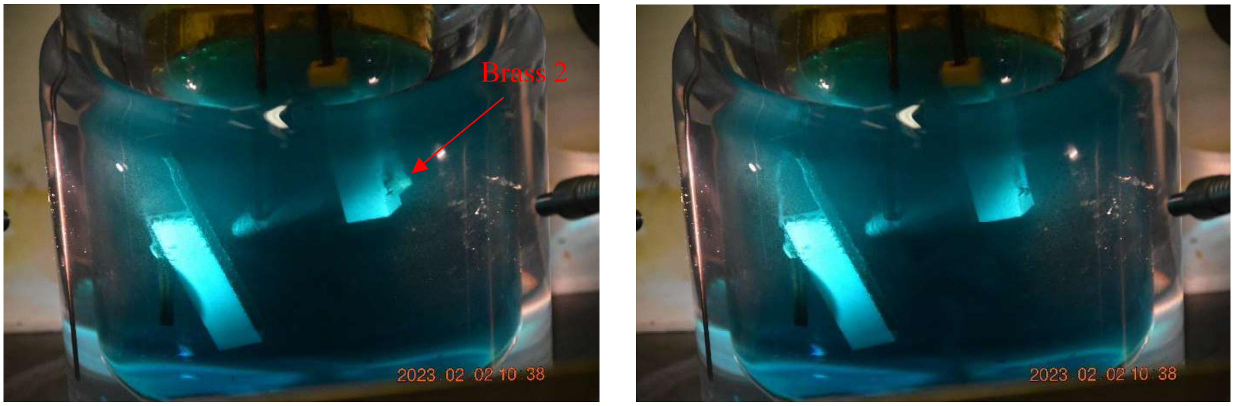


Figure 3-13. 360 Brass in 8.5 M Nitric Acid at 15 °C; Brass 2 Piece Falling to Bottom of Vessel.

The next piece of the assembly to fall to the bottom of the vessel was the carbon steel ring (See Figure 3-14). This piece also failed due to a combination of galvanic and crevice corrosion, which preferentially "sawed" through the end of the brass pin (Brass 1 in Figure 2-6). Thus, not only did the ring fall to the bottom of the vessel but a piece of the brass also fell to the bottom of the vessel. In addition to brass dissolution, the carbon steel ring also corroded. As was observed in the scoping tests, the degree of corrosion increased as the concentration of nitric acid decreased. This phenomenon is also explained by passivation.

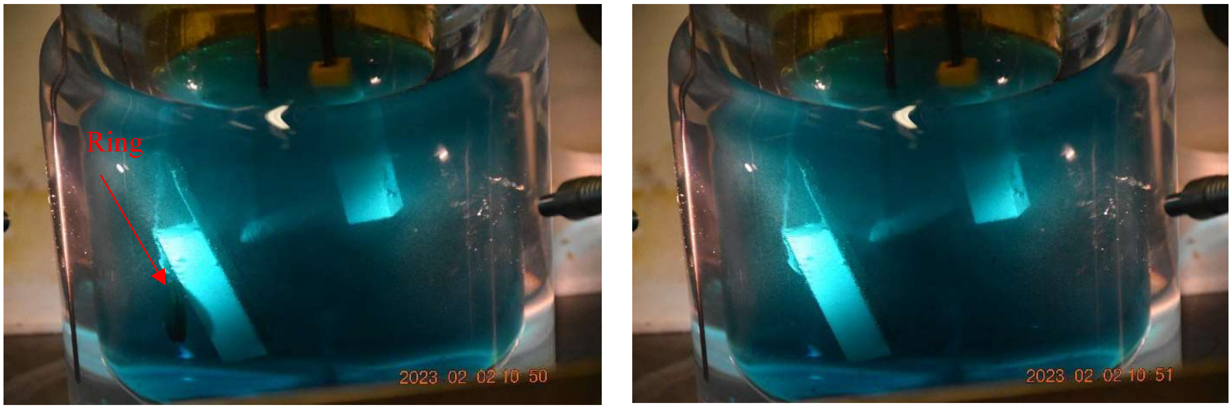


Figure 3-14 360 Brass in 8.5 M Nitric Acid at 15 °C; Carbon Steel Ring Falling to Bottom of Vessel.

Teflon Holder 1 (see Figure 2-6) was the next piece of the assembly to fall to the bottom of the vessel (See Figure 3-15). The crevice created by the contact between the holder and the pin accelerated corrosion of the brass. Teflon is chemically inert in this environment and did not degrade. Eventually, the diameter of the pin decreased such that the holder slid off the pin and fell to the bottom of the vessel.

Figure 3-15. 360 Brass in 8.5 M Nitric Acid at 15 °C; Teflon Holder Falling to Bottom of Vessel.

The 410 stainless-steel coupon also fell to the bottom of the vessel due to the combination of crevice and galvanic corrosion of the brass. Figure 3-16 shows that severe necking of the pin had occurred where the coupon was located. Likely, the presence of the carbon steel ring and Teflon Holder 1 prevented the coupon from sliding off the pin earlier. The coupon represents the handle of the FCA can carrier.

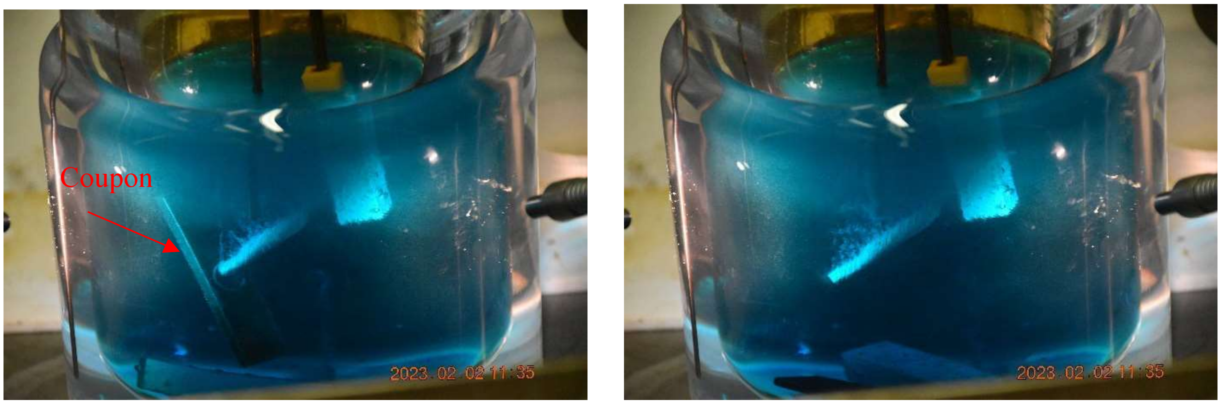


Figure 3-16. 360 Brass in 8.5 M Nitric Acid at 15 °C; 410 SS Coupon Falling to Bottom of Vessel.

The final piece of the assembly to fall to the bottom of the vessel was the pin itself. Figure 3-17 shows that the pin begins to tilt downward in almost a vertical orientation. Crevice corrosion between Teflon Holder 2 and the pin preferentially corrodes the brass until it slides out of the holder and falls to the bottom of the vessel.

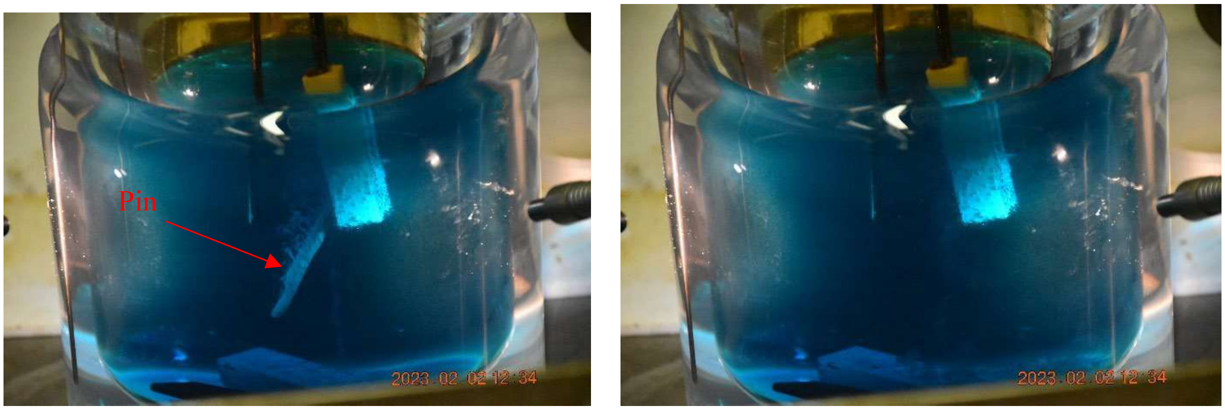


Figure 3-17. 360 Brass in 8.5 M Nitric Acid at 15 °C; Brass Pin Falling to Bottom of Vessel.

The dissolution of the pin proceeds uniformly over the surface of the pin at a rate that is dependent upon the concentration and bulk temperature of the solution. Figure 3-18 shows the uniform dissolution of the pin as it rests on the bottom of the vessel. The disappearance of the bubbles coincided with the complete dissolution of the pin.

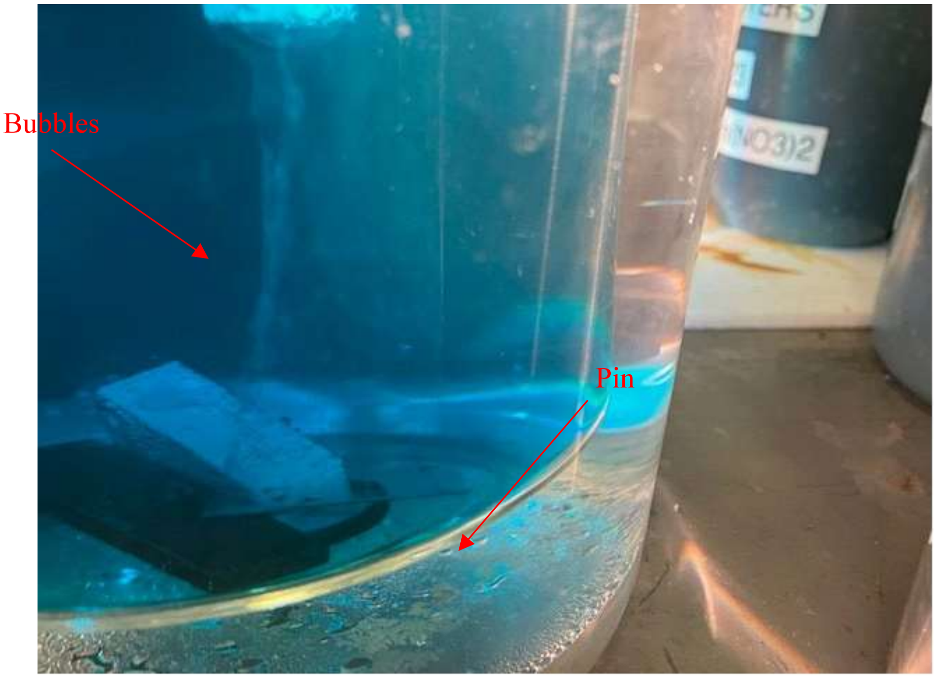


Figure 3-18. 360 Brass in 8.5 M Nitric Acid at 15 °C; Brass Pin Dissolving on Bottom of Vessel.

Figure 3-19 shows the triplicate tests that were performed in Solution 5 at a bulk temperature of 15 °C and illustrates the time at which each piece of the assembly fell to the bottom of the vessel. The sequence and time of "failure" for each piece of the pin assembly were remarkably consistent and led to a relatively small spread in the time to functional failure for the pin (i.e., this time is the same as the failure time for the pin in Figure 3-19) and ultimately the final dissolution time.

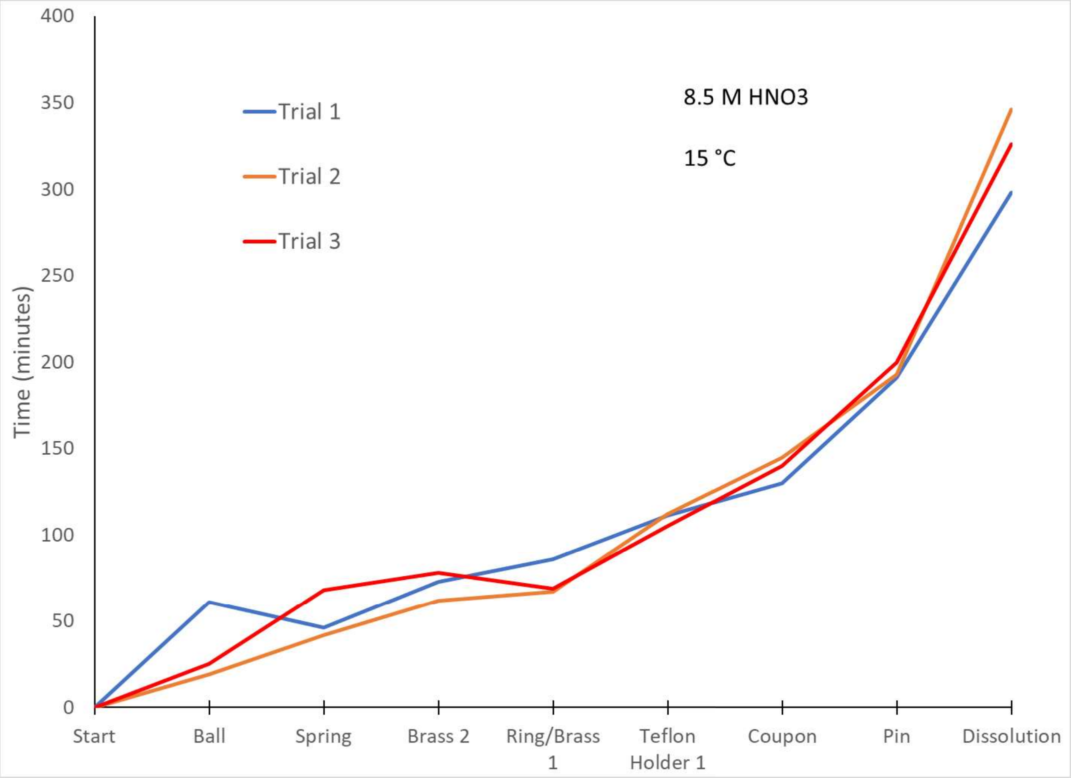


Figure 3-19. Failure of Pin Assembly Chronology for 360 Brass in 8.5 M Nitric Acid at 15 °C.

Table 3-2 shows the times to functional failure of the pin assembly and pin dissolution as a function of nitric acid concentration and temperature. In general, the time decreased as the nitric acid concentration and bulk temperature increased. The effect of the bulk temperature is clearly observed by a comparison of the Solution 5 results at 15 and 25 °C. The times had decreased by a factor of 3 to 4 for both the functional failure of the pin assembly and the pin dissolution as the temperature increased (Note: the average times for multiple tests were considered in this calculation). A similar decrease in the times with an increase in bulk temperature from 15 to 25 °C also appears to occur for the second test in Solution 6.

The effect of nitric acid concentration is seen by comparing the test results for Solutions 5 and 6 at a bulk temperature of 15 °C. Both the functional failure time and the pin dissolution time decreased by a factor of approximately 1.3 to 2.5 as the concentration of nitric acid increased. (Note: the average times for multiple tests were considered in this calculation). At a bulk temperature of 25 °C, the decrease in functional failure time and pin dissolution is by a similar factor for Solutions 5 and 6 (Note: the times for the test without an asterisk for Solution 6 were used for this comparison.). For Solutions 4 and 5, the decrease in the times with an increase in nitric acid concentration appears to be slightly greater on the order of a factor of 4 to 5. Nonetheless, clearly functional failure of the pin and pin dissolution times decrease as the nitric acid concentration increases.

The result from the additional test performed in Solution 6 with 0.05 M KF added at a bulk temperature of 25 °C is also shown in Table 3-2. When compared to the second test at this condition, no significant change in either the time for functional failure of the pin assembly (i.e., 12 minutes vs. 20 minutes) or complete dissolution of the pin (i.e., 1 hour and 23 minutes vs. 1 hour and 6 minutes) was observed. The change in the dissolution times due to the change in the KF concentration (0.0009 M to 0.05 M) is small compared to the effects of the nitric acid concentration and the bulk temperature. This result was expected given the relatively low bulk temperatures and the low concentrations of fluoride that were tested. Therefore, the dissolution times are considered representative for the 0.05 M KF concentration as well.

Table 3-2. Pin Dissolution Time as a Function of the Bulk Temperature and Nitric Acid Concentration

Temperature	15 °C		25 °C	
Nitric Acid Conc.	Functional Failure of Pin Assembly (Hr:Min)	Complete Dissolution of Pin (Hr:Min)	Functional Failure of Pin Assembly (Hr:Min)	Complete Dissolution of Pin (Hr:Min)
7.0 M (Solution 4)	10:30<t<17:30*	10:30<t<17:30*	4:12 4:38 5:12	6:32 7:08 7:23
8.5 M (Solution 5)	3:11 3:13 3:20	4:58 5:46 5:26	1:06	1:28
10.3 M (Solution 6)	1:46 1:11 1:09	3:53 3:58 4:09	0:04* 0:20 0:12**	0:22* 1:06 1:23**

* See discussion of test below.
** Test performed at 0.05 M KF.

The effect of the concentration may also be observed by examining the measurement of the local temperature adjacent to the pin as a function of time. Figure 3-20 shows that the local temperature is significantly different than the bulk temperature in the outer vessel at the outset of the test. As the test progressed, the local temperature plateaued and the temperature decreased. By the end of the test, the thermocouple measuring the local temperature and the thermocouple measuring the bulk temperature in the outer vessel were nearly the same. The decrease in temperature likely occurred because the pin had fallen to the bottom of the vessel and therefore was no longer adjacent to the local thermocouple.

As observed during the scoping tests, the initial rate at which the local temperature increases correlates well with an increase in the dissolution rate of the brass pin (see Figure 3-11). The initial rate of increase in the local temperature was compared for each concentration at a bulk temperature of 25 °C. Figure 3-20 shows that as the solution concentration increased, the rate at which the local temperature increased became greater (i.e., as judged by the slope of the lines). This observation correlated with a visible increase in the dissolution rate (i.e., more vigorous bubbling near the pin, darker greenish blue solution, and more NO_x gas evolution above the solution) and shorter dissolution times as the nitric acid concentration increased.

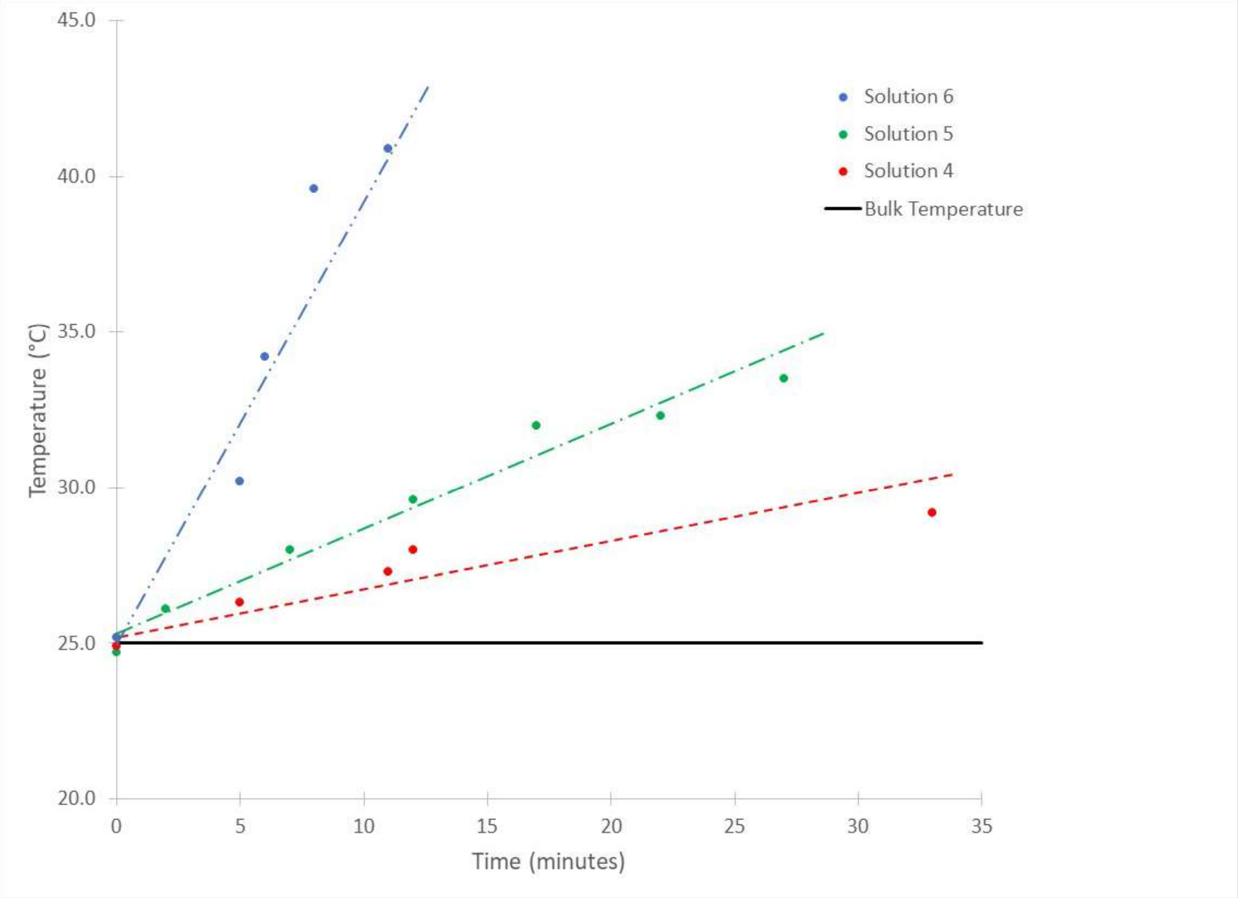


Figure 3-20. Initial rate of increase in local temperature as a function of nitric acid concentration. Note: Solution 4 is 7 M nitric acid; Solution 5 is 8.5 M nitric acid; Solution 6 is 10.3 M nitric acid.

Two tests, as indicated in Table 3-2 did not go as planned, however, the results appear to be consistent with the other tests. For Solution 4 at a bulk temperature of 15 °C, exact failure and dissolution times were not recorded as the test went on past midnight and exceeded the time at which the camera recorded the events. At the time the camera stopped recording, Teflon Holder 1 was still hanging on to the pin. Seven hours later, when the vessel was observed, the pin assembly had failed, and the pin had dissolved completely. Therefore, the functional failure and dissolution times were reported as a range. The tests for Solution 4 at a bulk temperature of 25 °C can be used to estimate a time for functional failure and pin dissolution. The decrease in temperature would be expected to increase these failure times by a factor of 3 to 4. Assuming a factor of 3 and using the average failure times for Solution 4 at a bulk temperature of 25 °C, the estimated times for functional failure and pin dissolution at 15 °C are 16 hours and 21 hours, respectively. Thus, functional failure would be within the range of times observed, while complete dissolution occurred a few hours later than the range of times. Although this does not provide actual time estimates, it does suggest that for Solution 4 at 15 °C, it would be expected that pin dissolution time would exceed an operational shift (i.e., 8-12 hours).

The first test in Solution 6 at a bulk temperature of 25 °C also did not go as planned. Originally the test was intended to be conducted at 15 °C. The cooling water in the jacket was controlled at 15 °C. As this was the first test in the series, a fresh batch of Solution 6 was prepared. Prior to allowing the solution to cool, it was added to the vessel such that the pin assembly was immediately immersed. The initial local temperature near the pin was measured at 30 °C. The local temperature increased to 35 °C and remained above 25 °C until the pin was dissolved. The dissolution was so violent that the whole assembly, including both Teflon Holders, fell to the bottom of the vessel within 4 minutes. Pin dissolution was observed after 22 minutes. The temperature profile during the test suggests that this was the most aggressive test. Therefore, the observation that this condition produced the fastest functional failure and dissolution times for this series of tests is consistent. Subsequent to this initial test, the solution in the vessel was always cooled to within 1 °C of the cooling water temperature before immersing the pin in the solution. This step provided more consistent bulk temperature control. For Solution 6 at a bulk temperature of 25 °C, it is recommended that the functional failure and pin dissolution times for the second and third tests at this condition be utilized.

Functional failure of the pin assembly is expected to occur in the dissolver by a similar sequence and mechanisms. However, the facility will not be able to visually monitor the functional failure of each piece of the assembly. If the time for functional dissolution of the pin assembly from these tests is utilized, there is a risk of underestimating the optimal time for removing the charging device. Thus, it is recommended that the time based on final dissolution of the pin be utilized to guide the facility in the decision as to when to remove the charging device. This time should provide a conservative estimate, likely 2 or more hours, for the criterion for the time at which the charging device may be removed from the dissolver.

These tests also provide a guide for the minimum temperature and concentration of the solution that will reduce the risk of attempting to remove the charging device too early. As an example, ensuring that the bulk temperature is greater than 15 °C and the nitric acid concentration is greater than 8.5 M, should allow the facility to remove the charging device from the dissolver within a shift (8-12 hours) with minimal risk. If the nitric acid concentration is lower (e.g., 7 M), the minimum temperature should be increased to at least 25 °C to maintain a low risk of early removal.

4.0 Conclusions

A linchpin is used to secure the FCA fuel can as it is being charged into the H-canyon dissolver. The pin will be made of a material that dissolves in the concentrated nitric acid solution in the dissolver. The facility desired to know the time that the pin would be dissolved so that the charging device for the FCA container could be removed from the dissolver. In particular, the pin dissolution time as a function of the nitric acid concentration and the temperature was desired. The facility would like to ensure that the solution environment was such that the charging device could be removed within an operational shift (8-12 hours).

A series of scoping tests were performed to determine the optimal pin material. Representative tests were then performed to determine the dissolution time as a function of nitric acid concentration and temperature. The following conclusions were drawn from the results of the tests.

- Carbon steel or zinc plated carbon steel experienced a passivation mechanism in concentrated nitric acid. The formation of a ferric nitrate layer on the surface of the metal decreased the rate of the dissolution reaction. The dissolution time for pins made of either of these materials will likely exceed an operational shift.
- On the other hand, 360 brass pins dissolved rapidly in the concentrated nitric acid solutions. If the temperature and nitric acid concentration are maintained above minimum values, the pin will

dissolve within an operational shift. Section 5.0 will provide recommendations for minimum concentration and temperature requirements for the dissolver solution.

- Based on the scoping tests, the presence of KF and $Gd(NO_3)_3$ had no significant effect on the dissolution rate of carbon steel or brass.
- The functional failure of the brass pin assembly was accelerated by localized corrosion mechanisms. Contact between brass and a dissimilar metal (e.g., carbon steel or stainless steel) and/or the formation of a crevice between an inert material (i.e., Teflon) or the dissimilar metal resulted in the brass corroding preferentially and at an accelerated rate. These two mechanisms, galvanic and crevice corrosion, will likely be operable in the dissolver. That is, the carbon steel ring on the pin and the stainless-steel ball and spring will likely fall to the bottom of the dissolver basket insert prior to complete dissolution of the pin. The carbon steel ring will eventually chemically dissolve, and the stainless-steel parts will likely not chemically dissolve. Both pin components will electrolytically dissolve in nitric acid.
- Functional failure of the pin assembly is expected to occur in the dissolver by a similar sequence and mechanisms. However, the facility will not be able to visually monitor the functional failure of each piece of the assembly. If the time for functional dissolution of the pin assembly from these tests is utilized, there is a risk of underestimating the optimal time for removing the charging device. Thus, it is recommended that the time based on final dissolution of the pin be utilized to guide the facility in the decision as to when to remove the charging device. This time should provide a conservative estimate, likely 2 or more hours, for the criterion for the time at which the charging device may be removed from the dissolver.

5.0 Recommendations

The following recommendations for operations are made based on these test results.

- 360 brass is the recommended material for the linchpin that is used for the charging device for the FCA can.
- If the bulk temperature is greater than or equal to 25 °C, and the nitric acid concentration is greater than or equal to 8.5 M, allow a minimum of 2 hours to ensure complete dissolution of the pin.
- If the bulk temperature is greater than or equal to 25 °C, and the nitric acid concentration greater than or equal to 7 M, but less than 8.5 M, allow a minimum of 8 hours to ensure complete dissolution of the pin.
- If the bulk temperature is less than 25 °C, and the nitric acid concentration is greater than or equal to than 8.5 M, allow a minimum of 6 hours to ensure complete dissolution of the pin.
- If the bulk temperature is less than 25 °C, and the nitric concentration is less than 8.5 M nitric acid, dissolution of the pin may not occur within a 12-hour shift. Therefore, either the bulk temperature or nitric acid concentration should be increased such that pin dissolution occurs within 12 hours.

This material and these conditions should minimize the risk of removing the charging device from the dissolver too early.

6.0 References

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