**CORROSION RESISTANCE AND BEHAVIORAL CHARACTERISTICS OF METALS
EXPOSED TO 70 PERCENT BY WEIGHT SULFURIC ACID AT
ELEVATED TEMPERATURES**

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Abstract

The development of a concentrated acid hydrolysis process may necessitate the storage, handling, and processing of concentrated solution of sulfuric acid at temperatures in excess of 70°C. Due to the corrosivity of the sulfuric acid at elevated temperatures, a series of corrosion tests was conducted to determine the corrosion performance and behavior of various construction materials using immersion and electrochemical techniques. Test results showed that among the stainless steels tested, only Carpenter 20Mo-6 performed satisfactorily up to 70°C. It passivated spontaneously and corroded at a rate less than 40 $\mu\text{m}/\text{yr}$ (1.6 mpy). Among numerous nickel-based alloys tested, only Hastelloy B-2 had excellent corrosion resistance up to 100°C with a corrosion rate less than 50 $\mu\text{m}/\text{yr}$ (2 mpy), although the alloy did not passivate. Zirconium alloy Zr 702 provided excellent corrosion resistance to 100°C. The alloy passivated spontaneously, but its passive range decreased, evidently with increase in temperature. Tantalum and KBI-40 provided excellent corrosion protection at all test temperatures. The materials passivated spontaneously with a wide passive range.

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Introduction

The Tennessee Valley Authority's Environmental Research Center and the Polymer Science Department of the University of Southern Mississippi are developing an ion-exclusion chromatography process to separate sulfuric acid from solutions generated when hydrolyzing pentosans and cellulose to monomeric sugar. One process option being considered consists of removing the acid from the hydrolyzate and then concentrating it to approximately 70 percent by weight (wt%). The concentrated acid could then be used in conjunction with an extruder (Dunning and Lathrop 1945) to mechanically impregnate the acid into the cellulose, which results in higher cellulose conversion efficiencies at lower acid loadings.

Although simple in concept, acid evaporation presents a difficult problem for evaporator designers because of the corrosivity of acid at temperatures exceeding 70°C. Currently, fiberglass-reinforced plastics (such as polyester and vinyl-ester laminates), polyvinyl chloride, polyethylene, and carbon steel (at limited dynamic conditions) offer simple and inexpensive means for storage and transportation of acid. However, use of these materials is restricted to near ambient temperature. As the temperature of the acid is increased, corrosion aggressiveness increases. Therefore, with introduction of additional variables of elevated pressure, temperature, and mechanical abuse, some equipment, such as spent acid evaporators, heat exchangers, and extruders, require materials with more effective chemical and mechanical characteristics.

A series of corrosion tests was conducted on various construction materials exposed to 70 wt% sulfuric acid at temperatures of 70, 80, 90, and 100°C. The objective was to determine corrosion performance of the test materials within a certain temperature range. Data may be used as a reference for process and design engineers in equipment materials selection for exposure to 70 wt% sulfuric acid solution. Corrosion performance of test materials was

determined using standard weight-loss tests. Behavior was characterized using DC potentiodynamic polarization scan tests. Materials tested included stainless steels (Type 316L, Type 904L, Carpenter 20Cb-3, Carpenter 20Mo-6, E-Brite, and 7-Mo Plus), nickel-based alloys (ALLCORR and Monel 400), Hastelloys (B-2, C-22, C-276, and G-30), and refractory metals (Zirconium 702, tantalum, and KBI-40; 40% Nb-60% Ta).

Experiment

Immersion tests conformed to the National Association of Corrosion Engineers (NACE) Test Method TM-01-69 (NACE 1992) and to Muscle Shoals Corrosion Laboratory Test Method MSCL-TM-01-92 (MSCL 1992). Each test unit included a disc specimen 5 cm (2 in) in diameter and 0.32 cm (1/8 in) thick with a 120-grit surface finish suspended in a test tube containing 750 mL of the test solution. The tests were conducted in temperature-controlled thermal blocks. During the tests, a water-cooled condenser was fitted on top of each test tube to prevent water loss and change in the test solution concentration due to evaporation. Prior to the tests, specimens were measured, cleaned, ultrasonically degreased in acetone, dried, and weighed. The test solution was neither agitated nor aerated during testing. Following 7-day exposure, the specimens were removed, mechanically and ultrasonically cleaned, and weighed. Surfaces were examined for peculiar corrosion attack. Corrosion rates were calculated in micrometers/year ($\mu\text{m}/\text{yr}$) based on weight-loss data using the following equation.

$$\text{Rate, } \mu\text{m}/\text{yr} = [(9.271 \times 10^6)(M_i - M_f)][A \cdot d \cdot t]^{-1}$$

where,

M_i = specimen initial mass, g

M_f = specimen final mass, g

d = material density, g/cm^3

A = specimen surface area exposed, cm^2

t = test duration, days

Electrochemical test procedures conformed with ASTM Standard G 3-81 (ASTM 92). The cylindrical test specimen was 0.95 cm (3/8 inch) in diameter and 1.27 cm (1/2 inch) in height with a 600-grit surface finish. The exposed surface area was approximately 5.3 cm². Prior to testing, specimens were ultrasonically degreased in acetone 5 minutes and dried. Test specimens were then immersed in a glass cell containing approximately 800 mL of the test solution. Corrosion potential or open-circuit potential (E_{cor}) was allowed to become relatively stable. The DC potential polarization scan tests were performed by scanning specimen surface potential from -300 mV versus E_{cor} to 1,600 mV versus a saturated calomel electrode (SCE) or to a current density of approximately 10 $\mu\text{A}/\text{cm}^2$. Scan rate was 0.17 mV/s or 0.6 V/hour. Test results are presented in the form of E vs i , which is specimen surface potential E (mV) versus specimen current density i ($\mu\text{A}/\text{cm}^2$) in semilog scale. Typical equipment used in DC potential polarization scan tests includes a potentiostat, electrochemical software, an IBM personal computer, and electrochemical cells. Behavioral characteristics were determined from passivity, critical passive current density (i_c), passive current density (i_p), primary passivation potential (E_{pp}), and breakdown potential (E_b).

Results and Discussion

Table 1 gives the results of the weight-loss tests. Tables 2 and 3 give the results of the DC potential polarization scan tests. Figures 1 and 2 show the DC potentiodynamic polarization diagrams of the test materials.

Immersion Tests

Immersion test results indicate that Type 316L stainless steel corroded extensively at 70 and 80°C. The average 7-day corrosion rate was approximately 2,500 $\mu\text{m}/\text{yr}$ (100 mpy). Observations of the coupon surface revealed that corrosion was severe on regions that had not been cold worked. In cold worked areas, corrosion was less. Due to severe corrosion at lower temperatures, no immersion tests were

performed on Type 316L at temperatures of 90 and 100°C.

Type 904L exhibited good corrosion resistance at 70 and 80°C with rates of 100 $\mu\text{m}/\text{yr}$ (4 mpy) and 125 $\mu\text{m}/\text{yr}$ (5 mpy), respectively, and fair resistance at 90°C with a corrosion rate of 225 $\mu\text{m}/\text{yr}$ (9 mpy). However, general corrosion of Type 904L increased substantially to a rate exceeding 12,000 $\mu\text{m}/\text{yr}$ (480 mpy) at 100°C. Test coupon surfaces were heavily etched and end-grain attack was also observed.

Corrosion of Carpenter 20Cb-3 and 20Mo-6 also increased proportionally with temperature. At 70 and 80°C, Carpenter 20Cb-3 corroded at rates of 76 $\mu\text{m}/\text{yr}$ (3 mpy) and 100 $\mu\text{m}/\text{yr}$ (4 mpy), respectively; and Carpenter 20Mo-6 corroded at rates of 50 and 100 $\mu\text{m}/\text{yr}$ (2 and 4 mpy), respectively. However, the corrosion rate of these alloys increased extensively at higher temperatures. The corrosion rate of Carpenter 20Cb-3 was approximately 25,000 $\mu\text{m}/\text{yr}$ (1,000 mpy) at 90 and 100°C, and rates of Carpenter 20Mo-6 were 225 $\mu\text{m}/\text{yr}$ (9 mpy) at 90°C and approximately 4,500 $\mu\text{m}/\text{yr}$ (180 mpy) at 100°C.

Ferric stainless steels also corroded excessively in 70 wt% sulfuric acid at high temperatures. E-Brite and 7-Mo Plus (25Cr-5Ni) corroded at rates in excess of 50,000 $\mu\text{m}/\text{yr}$ (2,000 mpy) at 70 and 80°C. Because these stainless steel specimens completely disappeared after 2-day exposure, no immersion tests were conducted at 90 and 100°C.

ALLCORR corroded extensively at all test temperatures with preferential attack to the weld. Corrosion rates were 1,400, 6,600, 24,000, 38,000 $\mu\text{m}/\text{yr}$ (56, 264, 960, and 1,520 mpy) at 70, 80, 90, and 100°C, respectively.

Hastelloy B-2 exhibited excellent corrosion resistance at all test temperatures. Corrosion rates of Hastelloy B-2 approximated 25 $\mu\text{m}/\text{yr}$ (1 mpy) at 70, 80, and 90°C; and 50 $\mu\text{m}/\text{yr}$ (2 mpy) at 100°C. No pitting or crevice corrosion was observed.

Table 1. Conventional Immersion Tests of Various Construction

Materials Exposed to 70 wt% H₂SO₄ at 70, 80, 90, and 100°C

Materials tested	Corrosion Rate, $\mu\text{m/yr}$			
	70°C	80°C	90°C	100°C
Stainless steel				
316L	25,000	25,000	-a	-a
E-Brite	10,000	50,000	-a	-a
7-Mo Plus	50,000	50,000	-a	-a
904L	100	3,750	350	12,500
Carp. 20Cb-3	100	100	12,500	12,500
Carp. 20Mo-6	50	100	225	4,500
Nickel-based				
ALLCORR	1,400	6,600	-a	-a
Hastelloy B-2	25	25	25	50
Hastelloy C-22	225	375	6,250	20,000
Hastelloy C-276	125	250	1,300	17,750
Hastelloy G-30	75	125	22,700	25,000
Monel 400	50	125	4,900	23,500
Refractory metals				
Zr-702	<2.5	<2.5	2.5	25
KBI-40	<2.5	<2.5	<2.5	<2.5
Ta	<2.5	<2.5	<2.5	<2.5

-a Due to the severity of the corrosion at 70 and 80°C, no tests were performed at this temperature.

Table 2. Results of DC Potential Polarization Scan Tests

on Construction Materials Exposed to 70 wt% Sulfuric Acid

Matl tested	E_{cor} mV/SCE	E_{pp} mV/SCE	E_p mV/SCE	i_c $\mu A/cm^2$	i_p $\mu A/cm^2$	Spontaneous Passivity
Stainless Steels						
316L	-289	-188	1,000	13,000	50.0	No
904L	-199	63	1,000	3,000	50.0	No
20Cb-3	-178	-52	979	1,000	4.0	No
20Mo-6	104	-a	967	-a	1.5	Yes
Nickel-Based Alloys						
B-2	88	-b	-b	-b	-b	No
C-22	75	-104	958	-	4.0	Yes
C-276	-75	17	967	100	3.0	No
G-30	194	-50	983	-	3.0	No
Monel	-123	-b	-b	-b	-b	No
Refractory Metals						
Zr 702	-329	-a	283	-a	1.0	Yes
KBI-40	0	-a	-a	-a	2.0	Yes
Ta	-417	-a	-a	-a	1.0	Yes
At 80°C						
904L	-185	-83	930	2,000	60.0	No
20Cb-3	-175	-63	1,000	4,000	15.0	No
20Mo-6	-153	-42	1,000	2,000	5.0	No
Nickel-Based Alloys						
B-2	34	-b	-b	-b	-b	No
C-22	-171	-104	850	300	10.5	No
C-276	-115	0	950	2,000	40.0	No
G-30	-117	-33	950	1,500	3.0	No
Monel	-122	-b	-b	-b	-b	No
Refractory Metals						
Zr 702	-349	-a	250	-a	2.0	Yes
KBI-40	-70	-a	-a	-a	2.0	Yes
Ta	-159	-a	-a	-a	2.0	Yes

-a Can not be measured because it is outside of test range.
 -b No Passivity observed.

**Table 3. Results of DC Potential Polarization Scan Tests
on Construction Materials Exposed to 70 wt% Sulfuric Acid**

Matl tested	E_{cor} mV/SCE	E_{pp} mV/SCE	E_p mV/SCE	i_c $\mu A/cm^2$	i_p $\mu A/cm^2$	Spontaneous Passivity
At 90°C						
Stainless Steels						
904L	-153	0	1,000	10,000	100.0	No
20Cb-3	-136	33	1,000	8,000	10.0	No
20Mo-6	-131	0	1,000	5,000	7.0	No
Nickel-Based Alloys						
B-2	115	-b	-b	-b	-b	No
C-22	-131	-20	1,000	1,000	10.0	No
C-276	-125	0	1,000	1,000	10.0	No
G-3	-116	0	1,000	2,500	6.0	No
Monel	-175	-b	-b	-b	-b	No
Refractory Metals						
Zr 702	295	0	62	-a	3.0	Yes
KBI-40	-29	-a	-a	-a	2.0	Yes
Ta	-80	-a	-a	-a	2.0	Yes
At 100°C						
Stainless Steels						
904L	-125	-83	930	2,000	60.0	No
20Cb-3	-126	-63	1,000	4,000	15.0	No
20Mo-6	-102	-42	1,000	2,000	5.0	No
Nickel-Based Alloys						
B-2	101	-a	-a	-a	-a	No
C-22	-167	-106	850	2,000	100.0	No
C-276	-96	-35	920	2,000	100.0	No
G-30	-109	0	1,000	7,000	100.0	No
Monel	-122	-a	-a	-a	-a	No
Refractory Metals						
Zr 702	-276	-a	49	-a	2.0	Yes
KBI-40	-70	-a	-a	-a	2.0	Yes
Ta	-159	-a	-a	-a	2.0	Yes

-a Can not be measured because it is outside of test range.
-b No Active-passive behavior observed.

Unlike the nickel-molybdenum alloys (Hastelloy B-2), the nickel-chromium-molybdenum alloys corroded significantly. At 70°C, Hastelloy C-22 corroded at a rate of 225 $\mu\text{m}/\text{yr}$ (9 mpy); Hastelloy C-276 at 125 $\mu\text{m}/\text{yr}$ (5 mpy); and Hastelloy G-30 at 75 $\mu\text{m}/\text{yr}$ (3 mpy). At 80°C, Hastelloy C-22 corroded at a rate of 375 $\mu\text{m}/\text{yr}$ (13 mpy); Hastelloy C-276 at 250 $\mu\text{m}/\text{yr}$ (10 mpy); and Hastelloy G-30 at 125 $\mu\text{m}/\text{yr}$ (5 mpy). However, corrosion shifted to severe at 90 and 100°C. These nickel-based alloys corroded at rates of 1,250 $\mu\text{m}/\text{yr}$ (50 mpy) to excess of 12,500 $\mu\text{m}/\text{yr}$ (>500 mpy). The majority of these specimens were unrecognizable following the test series due to attack severity.

Monel 400 (30% Cu-70% Ni) showed excellent corrosion resistance at 70°C and good corrosion resistance at 80°C. The alloy corroded at rates of 50 and 125 $\mu\text{m}/\text{yr}$ (2 and 5 mpy) at 70 and 80°C, respectively. However, corrosion increased significantly at 90 and 100°C with rates exceeding 4,500 $\mu\text{m}/\text{yr}$ (180 mpy) at 90 and 25,000 $\mu\text{m}/\text{yr}$ (1,000 mpy) at 100°C.

Refractory metals tested include zirconium alloy Zr 702, tantalum, and KBI-40 (40% Nb-60% Ta). The refractory materials exhibited excellent corrosion resistance at the test temperatures. Tantalum had corrosion rates of less than 2.5 $\mu\text{m}/\text{yr}$ (<0.1 mpy) at all test temperatures. At the four test temperatures corrosion rates of KBI-40 were in the range of 25 to 50 $\mu\text{m}/\text{yr}$ (1-2 mpy) and corrosion rate of Zr 702 was less than 25 $\mu\text{m}/\text{yr}$ (<1 mpy). Although, the surface of Zr-702 coupons turned dark in color, no evidence of chemical attack was observed.

DC Potentiodynamic Polarization Scan Tests

Due to corrosion severity during 7-day immersion tests, E-Brite, 7-Mo Plus, and ALLCORR were eliminated from the electrochemical series. Test results on Type 316L stainless steel showed nonspontaneous passivation at all test temperatures. Passivity occurred only when surface potential polarized toward the noble direction at 70°C. Additionally, corrosion behavior of Type 316L appeared

complex with multiple corrosion potentials (-289, 98, and 395 mV vs SCE) on the anodic polarization curve (Fig 1a) as potential scanned more positive. Passivity is established by potentiostatically polarizing corrosion potential in the passive range (300 to 900 mV vs SCE). However, corrosion protection is not feasible due to an excessively high i_p value (50 $\mu\text{A}/\text{cm}^2$). At 80°C, Type 316L also showed unusual passivity. Instead of a classical S-shape relationship between E and $\log i$ (normal stable passivation behavior), the anodic polarization diagram exhibited irregular behavior. At 90 and 100°C, evidence of passivity was unclear.

At all test temperatures, Type 904L exhibited active-passive behavior when the surface potential was polarized. The passive range encompassed approximately 900 mV. However, alloy passivity was nonspontaneous since corrosion potential (-199 mV vs SCE) was more active than primary passive potential (63 mV vs SCE). As with Type 316L, passivity of Type 904L was not very protective with passive current density values of 50 to 100 $\mu\text{A}/\text{cm}^2$. Thus, anodic protection is not feasible. Unlike Type 316L, Type 904L corroded at one corrosion potential at each test temperature (Fig 1b). The potential shifted to more noble values as temperature was increased.

Carpenter 20Cb-3 also showed active-passive behavior (Fig 1c) with nonspontaneous passivity. However, good corrosion protection can be achieved at 70°C by potentiostatically shifting corrosion potential in the passive range (0 to 900 mV vs SCE). Passive current density of the alloy was approximately 4 $\mu\text{A}/\text{cm}^2$. Thus, the general corrosion rate was less than 50 $\mu\text{m}/\text{yr}$ (2 mpy) if the corrosion potential is polarized potentiostatically in the passive range. At 80°C and above, passivity of Carpenter 20Cb-3 was less protective than at 70°C. The passive range narrowed and passive current density was slightly high (10-15 $\mu\text{A}/\text{cm}^2$).

Of the stainless steels tested, only Carpenter 20Mo-6 showed spontaneous passivity at 70°C (Fig 1d). The corrosion potential (104 mV vs SCE) was more noble than the primary

passivation potential (more active than the low range potential). The passive current density was very low ($1.5 \mu\text{A}/\text{cm}^2$). At 80°C and above, passivity was nonspontaneous (Fig 1d). However, it remained stable and protective due to the width of the passive range (about 900 mV) and low passive current densities ($5\text{--}7 \mu\text{A}/\text{cm}^2$).

No passivity was exhibited of nickel-based alloys Monel 400 and Hastelloy B-2. The potential-current plots showed classical Tafel behavior (Fig 2a). Although there was no evidence of passive film formation on the surface, corrosion of Hastelloy B-2 appeared to be controlled by diffusion of cathodic species (high slope segment on the cathodic polarization diagram). Slow diffusion of the cathodic species to the corroding surface causes rapid polarization of the cathodic site toward the cathodic direction in order to replenish the cathodic species. As a result, a high slope segment appears on the cathodic diagram (Fig 2a, Hastelloy B-2).

Test results on Hastelloys C-22, C-276, and G-30 showed nonspontaneous passivation at the test conditions. Corrosion potentials (Tables 2 and 3) were more active than the E_{pp} . Passivity of these alloys appeared only when surface potential was polarized toward the noble direction. At 70°C , passivity of all three alloys was protective, with a wide passive range (approximately 1000 mV) and low passive current densities ($3\text{--}4 \mu\text{A}/\text{cm}^2$). When test temperature was increased to 80°C , the passivity of Hastelloy G-30 still showed evidence of corrosion protection with a passive current density of $3 \mu\text{A}/\text{cm}^2$ (Fig 2b). However, no protective passivation appeared on Hastelloy C-22 and C-276 at 80°C . Passive current densities were high (approximately $10 \mu\text{A}/\text{cm}^2$). When temperature was increased to 90 and 100°C , no protective passivation was observed because i_p was high (approximately $100 \mu\text{A}/\text{cm}^2$).

Tests on zirconium alloy 702 showed interesting results. Although the alloy passivated spontaneously at low passive current densities ($1\text{ to }2 \mu\text{A}/\text{cm}^2$) at all four test temperatures, the

passive range was surprisingly narrow compared to stainless steels and nickel-based alloys. Although the corrosion potential did not change significantly with increase in temperature, the breakdown potential of Zr 702 shifted toward to more active values. As a result, the passive range decreased significantly as temperature increased (Fig 2c). At 70 and 80°C , the passive range was about 600 mV, but was about 320 to 350 mV at 90 and 100°C . Because the resistance to pitting and crevice corrosion of alloys is dependent on the potential distance between E_{cor} and E_b , for passive alloys, E_b approaching E_{cor} means the alloys are likely to be attacked by pitting or crevice corrosion, even though the general corrosion rate is still low. Thus, localized corrosion may be a problem when alloy Zr 702 is exposed to 70 wt% sulfuric acid at high temperatures.

As predicted, the test results showed that both tantalum and KBI-40 (40% Nb-60% Ta) passivated spontaneously and no breakdown was observed at any of the test temperatures (Fig 2d). These materials corroded in the passive range. The passive range was extremely high and passive current density was low (approximately $1\text{ to }2 \mu\text{A}/\text{cm}^2$). Neither of these test quantities were affected by the by the test temperatures.

Conclusions

1. Among stainless steels tests, only Carpenter 20Mo-6 is suitable for exposure to 70 wt% sulfuric acid at 70°C . If an anodic protection is provided, Carpenter 20CB-30 and 20Mo-6 can be employed to 70 and 80°C , respectively. Otherwise, Type 316L, 904L, 7Mo-Plus, and E-Brite are not suitable for service in 70 wt% sulfuric acid at the test temperatures.
2. Among nickel-based alloys tested Monel is suitable to 70°C . Up to 100°C , Hastelloy B-2 is a suitable. Nickel-chromium based alloys tested are not suitable due to high general corrosion rate and nonspontaneous passivity.
3. Tantalum and KBI-40 (40% Nb-60% Ta alloy) had excellent corrosion performance, but

their high cost is a limiting factor. Zirconium alloy ZR 702 alloy is suitable for service in 70 wt% sulfuric acid. However, pitting or crevice corrosion may occur because the passive range of the alloy decreases with temperature increase.

Recommendations

Taking into account the corrosion performance, cost, and availability, Carpenter 20Mo-6 is recommended for service in 70 wt% sulfuric acid at temperatures up to 70°. Up to 100°C, Hastelloy B-2 is a suitable material. Zirconium Zr 702 can be considered, but localized corrosion is highly possible at high temperatures.

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Figure 1. Potentiodynamic polarization of stainless steels.

Figure 2. Potentiodynamic polarization of nichol-based alloys and refractory metals.

Figure 1 - Potentiodynamic polarization of Stainless Steels Exposed to 70-90% Sulfuric Acid at Medium Temperatures. Test at a Scan Rate of 0.47 mV/s.

Figure 2 - Potentiodynamic polarization of Nickel-Based Alloys and Refractory Metals Exposed to 70-100% Sulfuric Acid at Medium Temperatures. Test at a Scan Rate of 0.47 mV/s.