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## **THE INFLUENCE OF COMPOSITION UPON SURFACE DEGRADATION AND STRESS CORROSION CRACKING OF THE Ni-Cr-Mo ALLOYS IN WET HYDROFLUORIC ACID**

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### **ABSTRACT**

At concentrations below 60%, wet hydrofluoric acid (HF) is extremely corrosive to steels, stainless steels and reactive metals, such as titanium, zirconium, and tantalum. In fact, only a few metallic materials will withstand wet HF at temperatures above ambient. Among these are the nickel-copper (Ni-Cu) and nickel-chromium-molybdenum (Ni-Cr-Mo) alloys. Previous work has shown that, even with these materials, there are complicating factors. For example, under certain conditions, internal attack and stress corrosion cracking (SCC) are possible with the Ni-Cr-Mo alloys, and the Ni-Cu materials can suffer intergranular attack when exposed to wet HF vapors.

The purpose of this work was to study further the response of the Ni-Cr-Mo alloys to HF, in particular their external corrosion rates, susceptibility to internal attack and susceptibility to HF-induced SCC, as a function of alloy composition. As a side experiment, one of the alloys was tested in two microstructural conditions, i.e. solution annealed (the usual condition for materials of this type) and long-range ordered (this being a means of strengthening the alloy in question).

The study of external corrosion rates over wide ranges of concentration and temperature revealed a strong beneficial influence of molybdenum content. However, tungsten, which is used as a partial replacement for molybdenum in some Ni-Cr-Mo alloys, appears to render the alloys more prone to internal attack.

With regard to HF-induced SCC of the Ni-Cr-Mo alloys, this study suggests that only certain alloys (i.e., those containing tungsten) exhibit classical SCC. It was also discovered that high external corrosion rates inhibit HF-induced SCC, presumably due to rapid progression of the external attack front.

With regard to the effects of long-range ordering, these were only evident at the highest test temperatures, where the ordered structure exhibited much higher external corrosion rates than the annealed structure.

Keywords: nickel alloys, hydrofluoric acid, stress corrosion cracking, corrosion rates, internal attack

## INTRODUCTION

Of the common halogen acids, hydrofluoric is the most difficult to deal with. Not only is it extremely hazardous, but also it attacks glass, titanium, zirconium, and tantalum, in addition to ferrous alloys. Nickel alloys are among the few metallic material options for wet hydrofluoric acid service. The mechanism by which the nickel alloys are protected is not well understood, but is believed to involve the formation of insoluble fluoride salts on their surfaces.<sup>1-3</sup>

However, it is now well known that nickel alloys and austenitic stainless steels are susceptible to Environmentally Assisted Cracking (EAC) when exposed in wet hot hydrofluoric acid.<sup>4</sup> Yet nickel alloys are less susceptible than stainless steels to cracking and all nickel alloys are not equally susceptible.<sup>5</sup> Among the different types of corrosion mechanism which the alloys may encounter are: uneven corrosion or serration of the surface, stress corrosion cracking, internal attack, transgranular or intergranular attack, dealloying, internal voids, pits or cavities, and so forth, depending on the environment conditions (concentration, temperature, velocity, impurities, and liquid or vapor state of the acid) and also on the chemical composition of the alloy. Also, it is worth mentioning that the internal attack is unique to HF since other mineral acids, such as hydrochloric, sulfuric, nitric, phosphoric, etc., do not promote internal attack in the alloys. Moreover, this internal attack is unique in the sense that each alloy suffers a completely different pattern of attack from HF.

Several studies have been made of the resistance of nickel alloys to cracking in HF environments using U-bend specimens.<sup>6</sup> Tests using C-276 (N102276) U-bends were carried out in 1% HF and 10% HF at 24°C and 50°C and in 10% HF at 76°C for 48 hours. It was reported that none of the N10276 specimens suffered EAC (environmentally assisted cracking). In another series of tests, U-bend type of specimens of alloys N10276, C-22 (N06022) and 686 (N06686) alloys were exposed up to 100 hours to a HF gas stream both at 50°C and at 70°C.<sup>7</sup> The HF concentration is not known. None of these three alloys suffered EAC under the test conditions. The general corrosion rate at 50°C was higher than at 70°C, probably because more condensation occurred. Other U-bend specimens of a Ni-Cr-Mo alloy (N06059) were tested for resistance to EAC in aerated and deaerated 20% HF at 25° and 50°C.<sup>8, 9</sup> It was reported that none of the U-bend specimens showed any indication of EAC after an exposure time of 200 hours.

In previous work by the authors, the effects of test time, acid concentration, acid temperature, and stress upon the corrosion performance of HASTELLOY<sup>®</sup> C-2000<sup>®</sup> alloy<sup>(A)</sup> (N06200) and MONEL<sup>®</sup> 400 alloy<sup>(B)</sup> (N04400) were determined, both in liquid hydrofluoric acid and in hydrofluoric acid vapors.<sup>4, 10</sup> In liquid hydrofluoric acid, the corrosion rate of alloy N04400 (Ni-Cu) increased with test time, while test time was less influential in the case of N06200 (Ni-Cr-Mo). Corrosion rates (the measure of external attack) increased with concentration and temperature, more so for N04400 alloy than N06200 alloy.

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<sup>(A)</sup> HASTELLOY<sup>®</sup> and HAYNES<sup>®</sup> alloys are registered trademark of Haynes International, Inc.

<sup>(B)</sup> MONEL is a registered trademark of Special Metals Corporation.

At the two highest test temperatures, 79°C (175°F) and 93°C (200°F), in 20 wt. % hydrofluoric acid over 240 hours, the Ni-Cu alloy suffered appreciable intergranular attack, especially in the vapor phase. At the same two temperatures, C-2000 alloy suffered significant internal attack, defined as “lace-like”.

The suggested mechanism of the internal attack is a type of localized corrosion probably caused by specific chemical reactions between the small size of fluoride ions and the metal elements. This reaction (or series of reactions) may create inside the metal an environment that is different from the bulk external environment. This internal environment may become disengaged from the outside environment and be self sustained in an auto catalytic type of process that is known at this time. To study the effects of stress in the previous same work, U-bend samples of N04400 and N06200 were tested alongside three other nickel alloys, in 20 wt.% hydrofluoric acid at 66°C (150°F), 79°C (175°F) and 93°C (200°F), over 240 hours. There was a strong effect of temperature, and while most of the alloys were resistant to SCC in liquid and vapor at 66°C, most exhibited cracking, either in the liquid, vapor, or both at 93°C.<sup>4</sup> A review of HF corrosion, including an overview of industrial processes using HF and problems concerning materials selection, has been published recently.<sup>11, 12</sup>

The objective of this new study was to gain a greater understanding of how the Ni-Cr-Mo alloys respond to wet hydrofluoric acid. In particular, it was hoped that the effects of compositional variations might be ascertained, and that more knowledge might be gained concerning the stress corrosion cracking phenomenon. The alloys chosen for study are listed in Table 1.

To maximize their resistance to corrosion, most Ni-Cr-Mo materials are slightly overalloyed, and therefore prone to microstructural changes (the precipitation of so-called second phases). For this reason, wrought products are generally supplied in the solution annealed and rapidly quenched condition, to “lock in” the fully fcc structure. However, thermal exposure induces the initiation and growth of second phases, such as the following (in N10276):

- $A_2B$ , by long-range ordering in the approximate range 300-650°C (a slow, homogeneous reaction, with no preferential precipitation at the grain boundaries and twin boundaries).
- $M_6C$ , in the approximate range 650-1040°C (a rapid, heterogeneous reaction with preferential precipitation at the alloy grain boundaries and twin boundaries).
- $\mu$  phase, in the approximate range 760-1100°C (also a rapid, heterogeneous reaction).<sup>13</sup>

C-22HS alloy is unusual in that it was designed to take advantage of the homogeneous, long-range ordering reaction. Its yield strength can be doubled by a two-step aging treatment which induces the precipitation of  $A_2B$ .<sup>14</sup>

As to the role of the chromium and molybdenum in the Ni-Cr-Mo alloys, chromium encourages the growth of protective (passive) surface films, in the presence of oxygen, as it does in the stainless steels. Molybdenum enhances the corrosion resistance of nickel in the absence of oxygen, strengthens the fcc nickel solid solution, and is known to help with passivation.<sup>15</sup>

Other additions to these alloys include tungsten, which can be used as a partial replacement for, and has similar properties to, molybdenum. Copper, which is present in N06200, is known to be of benefit in reducing acids. Niobium, an important addition to HAYNES<sup>®</sup> 625 alloy (N06625), is known to enhance solid solution strength.

## EXPERIMENTAL PROCEDURES

Two types of specimen were used in this work, plain sheet specimens of approximate dimensions 3.2 mm x 25.4 mm x 25.4 mm and U-bend specimens made according to the recommendations of ASTM Standard G 30.<sup>16</sup> The plain sheet specimens were used to determine corrosion rates and to study internal attack, over wide ranges of acid concentration (in the range 1 to 30 wt. %) and temperature (in the range 24° to 93°C). The U-bend specimens, also prepared from 3.2 mm thick sheet, were used to determine the susceptibility of the alloys to SCC, both immersed and in the vapor space, at three specific combinations of concentration and temperature (1%/79°C, 20%/52°C and 20%/79°C).

Tests were carried out in kettles made of N10276 and lined with PTFE (polytetrafluorethylene). The U-bend specimens tested in the vapor space were suspended by means of PTFE thread. The kettles were open to the atmosphere of the laboratory scrubber through a small orifice at the top of a PTFE-lined condenser, i.e. the ingress of air into the kettles was not restricted. Tests were conducted at the free potential and the evolution of the corrosion potential of the specimens during the test was not monitored. The reported acid concentrations and test temperatures correspond to the conditions of the liquid solution.

All tests involved a period of 240 hours, without interruption. This test duration was based on previous work, which indicated good reproducibility of results over this test time. Two specimens of each alloy were tested at each concentration/temperature combination, and the corrosion rates averaged. One specimen of each alloy tested at each combination was studied metallographically after test, by sectioning, mounting in epoxy resin, and polishing.

## RESULTS AND DISCUSSION

The corrosion rates by weight (mass) loss of the test alloys at twenty two combinations of acid concentration and temperature are given in Table 2. The effects of acid concentration at 52°C are illustrated in Figures 1 and 2. The effects of temperature at an acid concentration of 20 wt. % are shown in Figures 3 and 4.

Figures 1 and 3, which include all of the test alloys, reveal considerable differences between the low (<10 wt. %) and high (>10 wt. %) molybdenum alloys (see Table1). Indeed, the low molybdenum alloys exhibit such high corrosion rates at higher concentrations and temperatures that separate graphs excluding these materials (Figures 2 and 4) were necessary to illustrate the differences in performance between the high molybdenum alloys.

From Figures 2 and 4, according to weight-loss measurements, it can be deduced that:

1. Copper (present in N06200) appears to be a beneficial addition to the Ni-Cr-Mo alloys, as indicated by lower corrosion rates at acid concentrations of 5% and 10% in Figure 2, and at temperatures of 66°C and 79°C in Figure 4.
2. The aging of C-22HS alloy is detrimental to its resistance to 20% HF at temperatures in excess of 52°C.

With regard to internal attack, the results of the metallographic studies on plain non-stressed specimens are given in Table 3. In many cases, there was no discernible internal attack, but in others, notably with N06022 and N10276 at higher concentrations and temperatures, the attack was significant. From this, it can be deduced that tungsten (which is present in these two alloys, but not the others) has a detrimental effect upon resistance to internal attack in HF.

Photomicrographs illustrating the nature of the internal attack on plain non-stressed specimens in 20% HF at 79°C are shown in Figures 5 to 8. The internal attack of annealed C-22HS alloy (Figure 5) was nondescript, with no apparent relationship to the microstructure. The internal attack of N06022 (Figure 6), N10276 (Figure 7), and N06200 (Figure 8), however, was whisker-like, with apparent relationships to the microstructure. Figure 6 is a medium magnification photomicrograph. Figures 7 and 8 are high magnification photomicrographs, which reveal more detail. The specimens shown in Figures 5 to 7 were unetched. The N06200 specimen in Figure 8 was very lightly etched in a mixture of hydrochloric and oxalic acids, to reveal the alloy microstructure and enhance the contrast of the internal attack. From Figure 8, it is apparent that the twin boundaries are among the favored sites for internal attack. Other metallographic work revealed some preference for internal attack along alloy grain boundaries. Also, it is assumed that the wave-like appearance of the near-surface attack is related to cold work, introduced during preparation (grinding) of the specimens.

The results of immersion and vapor space testing of stressed (U-bend) specimens are given in Table 4. The depths of internal features were determined by metallographic examination of longitudinal apex sections. The outer surface is that exposed the highest tensile stresses; the inner surface is that exposed to the highest compressive stresses. These results indicate that both concentration and temperature of the acid are important to the susceptibility of these alloys to cracking or internal attack, under stress. Internal effects were only significant at the highest test concentration and temperature (20% HF at 79°C) and only with three alloys, namely N06022, N10276, and N06200. The absence of internal features in N06035 and N06625 is attributed to their rapid rates of external attack, which would overwhelm any internal effects.

Comparison of the depth values for the outer surfaces of the N06022, N10276, and N06200 specimens immersed in 20% HF at 79°C with the depths of internal attack of the same alloys (under the same conditions) in the absence of stress indicates a significant influence of tensile stress. Furthermore, it appears that compressive stresses reduce or nullify the susceptibility of these alloys to internal attack.

Comparing Figure 6 with Figure 9, it is evident that the tensile stresses induce some form of cracking in N06022, since the latter photomicrograph indicates some directionality and approximates to the "branching transgranular" pattern commonly associated with chloride-induced SCC of the austenitic stainless steels. The same conclusions may be drawn about N10276 (Figures 7 and 10). The copper-bearing, tungsten-free N06200 (Figures 8 and 11), on the other hand, exhibits a different response to stress. Indeed, the near surface features in Figure 11 are hardly resolvable by optical metallography and appear to be enhanced internal attack rather than cracking. From this, it may be deduced either that tungsten has a negative effect upon the resistance of these alloys to HF-induced SCC, or that copper has a positive effect, or both.

As to the differences in outer surface depth values between the liquid and vapor samples, it is evident that immersion is much more damaging to N06022, N10276, and N06200 than vapor space suspension.

## CONCLUSIONS

- ❖ The molybdenum contents of the Ni-Cr-Mo alloys are important to their resistance to HF; those alloys with molybdenum contents less than 10 wt. % corrode rapidly at higher concentrations and temperatures.
- ❖ Copper, which is present in N06200, is beneficial to the performance of the Ni-Cr-Mo alloys in HF.
- ❖ The aging of C-22HS alloy is detrimental to its resistance to 20% HF at temperatures in excess of 52°C.
- ❖ Tungsten, which is present in N06022 and N10276, has a detrimental effect upon resistance to internal attack in HF.
- ❖ Grain boundaries and twin boundaries are favored sites for internal attack in HF.

- ❖ Tensile stresses induce cracking of a branching, transgranular nature in N06022 and N10276.
- ❖ N06200 exhibits a different response to stresses, best described as enhanced internal attack.
- ❖ Immersion in HF is more damaging to the Ni-Cr-Mo alloys than vapor space suspension.

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TABLE1  
NOMINAL COMPOSITION OF TEST ALLOYS (WT. %)

Alloy	UNS	Ni	Cr	Mo	Fe	W	Cu	Mn	Si	C	Al	Ti	Other
C-22	N06022	56	22	13	3	3	0.5*	0.5*	0.08*	0.01*	-	-	V 0.35*
C-22HS	-	61	21	17	2*	1*	0.5*	0.8*	0.08*	0.01*	0.5*	-	-
C-276	N10276	57	16	16	5	4	0.5*	1*	0.08*	0.01*	-	-	V 0.35*
C-2000	N06200	59	23	16	3*	-	1.6	0.5*	0.08*	0.01*	0.5*	-	-
G-35	N06035	58	33.2	8.1	2*	0.6*	0.3*	0.5*	0.6*	0.05*	0.4*	-	-
625	N06625	61	21.5	9	2.5	-	-	0.2	0.2	0.05	0.2	0.2	3.6 (Nb+Ta)

TABLE 2  
CORROSION RATES (mm/y) OF NON-STRESSED SPECIMENS IMMERSSED IN HF FOR 240HOURS

CONC. wt.%	TEMP. °C	N06022	C-22HS ANNEALED	C-22HS AGED	N10276	N06200	N06035	N06625
1	52	0.03	0.04	0.03	0.13	0.03	0.01	0.05
1	66	0.10	0.09	0.13	0.33	0.08	0.03	0.13
1	79	0.21	0.31	0.35	0.40	0.18	0.15	0.31
1	93	0.26	0.23	0.25	0.29	0.16	0.46	0.21
5	38	0.04	0.04	0.04	0.15	0.02	0.02	0.09
5	52	0.15	0.19	0.20	0.34	0.09	0.10	0.70
5	66	0.47	0.45	0.51	0.51	0.33	0.73	1.91
5	79	0.58	0.60	0.74	0.59	0.57	1.91	3.54
10	24	0.03	0.03	0.03	0.07	0.02	0.01	0.06
10	38	0.09	0.10	0.11	0.22	0.06	0.05	0.56
10	52	0.33	0.32	0.34	0.41	0.22	0.24	2.23
10	66	0.64	0.53	0.78	0.73	0.56	2.28	4.48
10	79	0.78	0.81	0.72	0.68	0.58	5.23	10.62
20	24	0.07	0.06	0.06	0.08	0.04	0.19	0.40
20	38	0.22	0.20	0.21	0.29	0.21	0.31	1.55
20	52	0.53	0.45	0.59	0.48	0.48	3.49	4.33
20	66	0.95	0.81	4.17	0.84	0.68	8.52	10.25
20	79	1.65	1.87	3.53	1.07	0.72	16.65	23.75
30	24	0.09	0.08	0.08	0.10	0.05	0.49	0.64
30	38	0.34	0.21	0.26	0.35	0.25	1.86	2.55
30	52	0.64	0.62	0.72	0.72	0.62	5.55	6.25
30	66	1.26	1.26	5.79	1.12	1.61	13.56	16.31

TABLE 3  
 MAXIMUM DEPTHS OF INTERNAL ATTACK (mm) OF NON-STRESSED SPECIMENS  
 IMMERSSED IN HF FOR 240 HOURS

Conc. wt. %	Temp. °C	N06022	C-22HS ANNEALED	C-22HS AGED	N10276	N06200	N06035	N06625
1	52	N	N	N	N	N	N	N
1	66	N	N	N	N	N	N	N
1	79	N	N	N	N	N	N	N
1	93	N	N	N	0.04	N	N	N
5	38	N	N	N	N	N	N	N
5	52	N	N	N	N	N	N	N
5	66	N	N	N	N	N	N	N
5	79	N	N	N	N	N	N	N
10	24	N	N	N	N	N	N	N
10	38	N	N	N	N	N	N	N
10	52	N	N	N	N	N	N	N
10	66	N	N	N	0.05	N	N	N
10	79	0.08	N	N	0.10	0.08	N	N
20	24	N	N	N	N	N	N	N
20	38	N	N	N	N	N	N	N
20	52	N	N	N	N	N	N	N
20	66	0.08	N	N	0.09	N	N	N
20	79	0.22	0.10	N	0.23	0.13	N	N
30	24	N	N	N	N	N	N	N
30	38	N	N	N	N	N	N	N
30	52	0.04	N	N	0.06	N	N	N
30	66	0.13	N	N	0.13	N	N	N

N = No discernible internal attack

TABLE 4  
 MAXIMUM DEPTHS OF CRACKING OR INTERNAL ATTACK (mm) OF U-BEND SPECIMENS

Alloy	Phase	1%, 79°C		20%, 52°C		20%, 79°C	
		Outer	Inner	Outer	Inner	Outer	Inner
N06022	Liquid	N	N	N	N	0.33	N
	Vapor	N	N	N	N	0.10	0.05
C-22HS ANNEALED	Liquid	N	N	N	N	N	N
	Vapor	N	N	N	N	N	N
C-22HS AGED	Liquid	N	N	N	N	N	N
	Vapor	N	N	0.03	0.01	N	N
N10276	Liquid	N	N	N	N	0.43	N
	Vapor	N	N	N	N	0.19	0.10
N06200	Liquid	N	N	N	N	0.18	0.08
	Vapor	N	N	N	N	0.06	0.05
N06035	Liquid	N	N	N	N	N	N
	Vapor	N	N	N	N	N	N
N06625	Liquid	N	N	N	N	N	N
	Vapor	N	N	N	N	N	N

N = No discernible internal attack

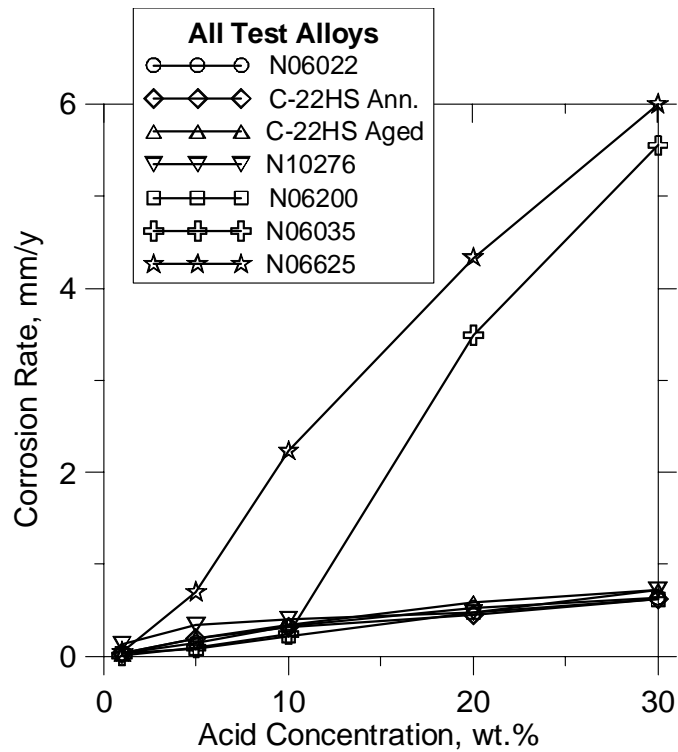


FIGURE1- Corrosion Rate vs. Acid Concentration at 52°C  
-All Test Alloys-

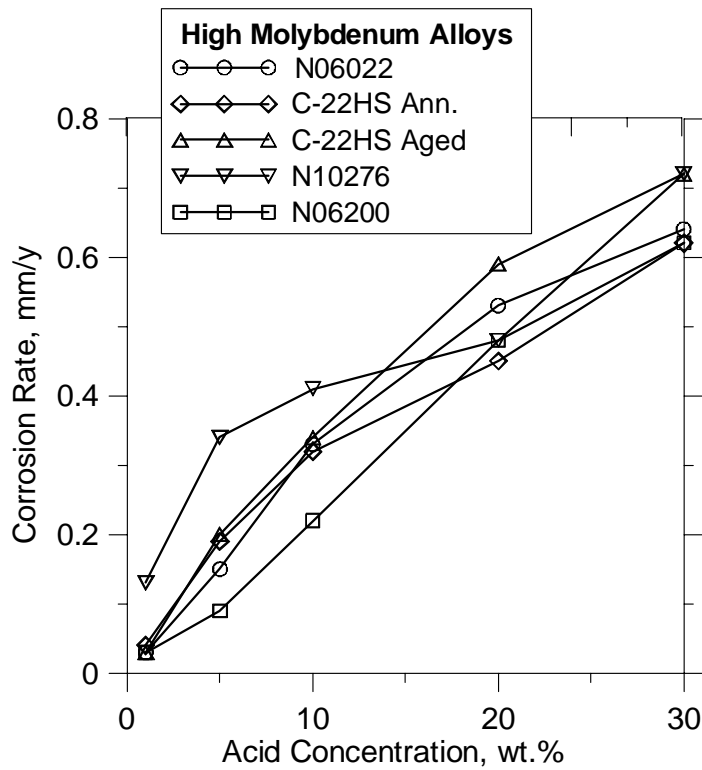


FIGURE 2- Corrosion Rate vs. Acid Concentration at 52°C  
-High Molybdenum Alloys-

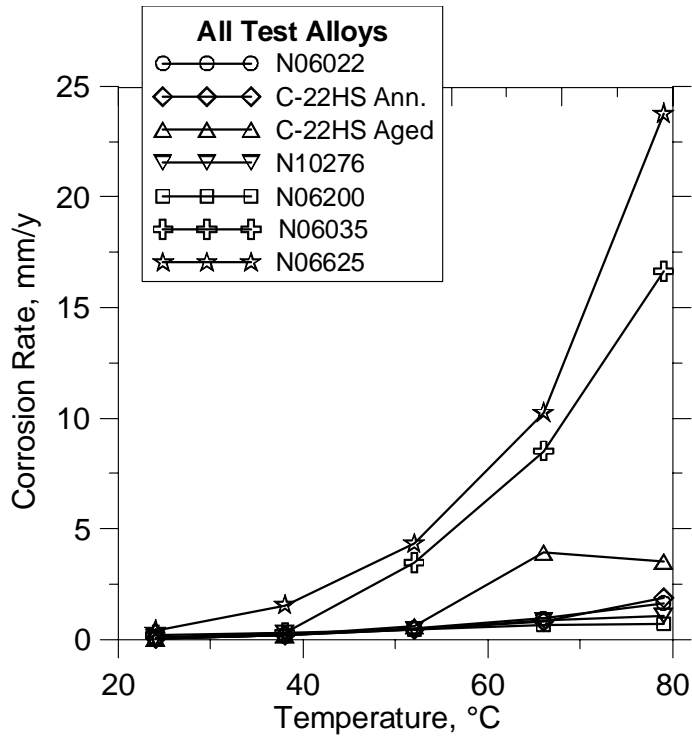


FIGURE 3- Corrosion Rate vs. Temperature in 20% HF  
-All Test Alloys-

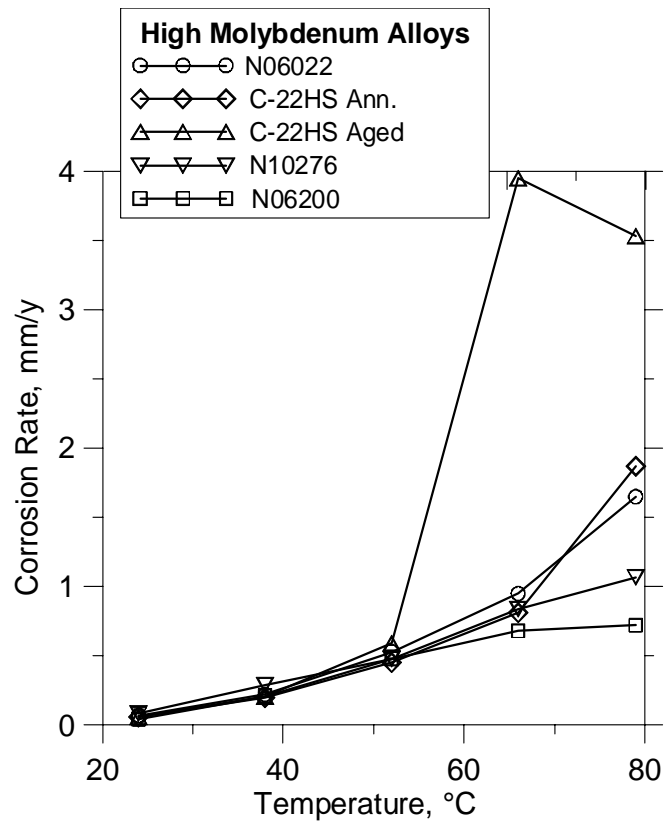


FIGURE 4- Corrosion Rate vs. Temperature in 20% HF  
-High Molybdenum Alloys



FIGURE 5 - Internal Attack of Non-Stressed Annealed C-22HS Alloy in 20% HF at 79°C  
(Medium Magnification-Unetched)

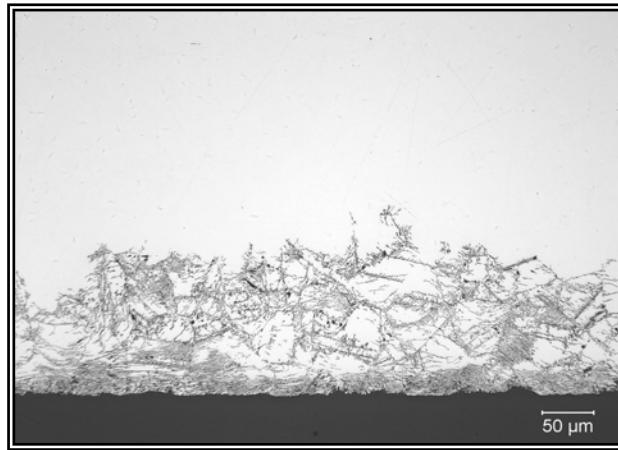


FIGURE 6 - Internal Attack of Non-Stressed N06022 in 20% HF at 79°C  
(Medium Magnification-Unetched)

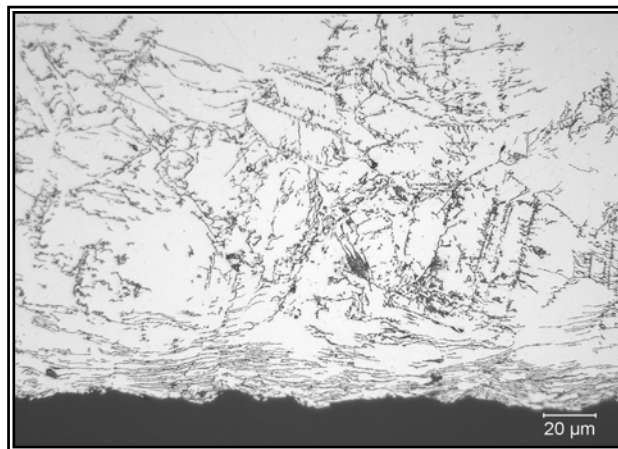


FIGURE 7 - Internal Attack of Non-Stressed N10276 in 20% HF at 79°C  
(High Magnification-Unetched)

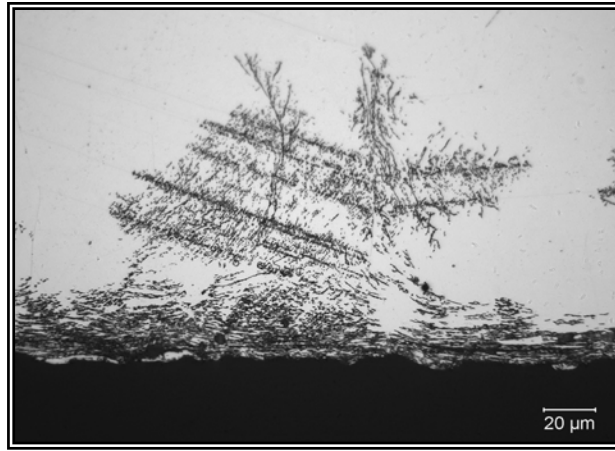


FIGURE 8 - Internal Attack of Non-Stressed N06200 in 20% HF at 79°C  
(High Magnification-Etched)



FIGURE 9 - Internal Features of Stressed N06022 in 20% HF at 79°C  
(Medium Magnification-Unetched)

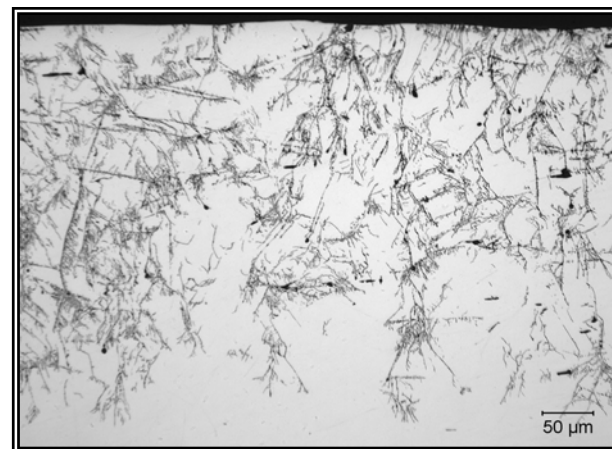


FIGURE 10 - Internal Features of Stressed N10276 in 20% HF at 79°C  
(Medium Magnification-Unetched)

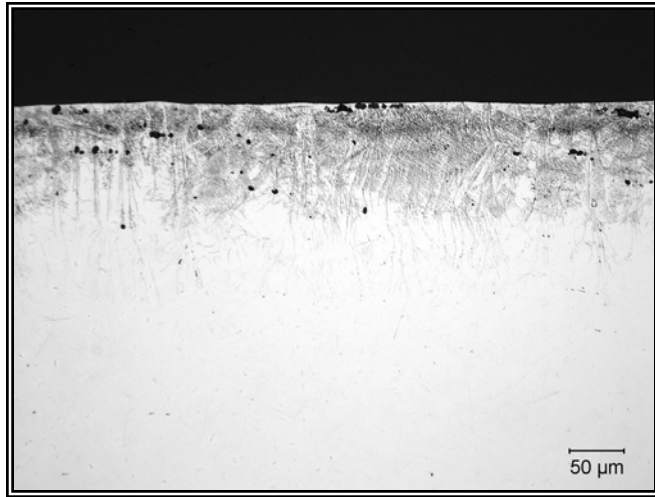


FIGURE 11 - Internal Features of Stressed N06200 in 20% HF at 79°C  
(Medium Magnification-Unetched)