

THE EFFECT OF NIOBIUM ON THE CORROSION RESISTANCE OF NICKEL-BASE ALLOYS

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Abstract

The role of niobium in enhancing the mechanical properties of nickel-base alloys is well established in the technical literature. Its effect in improving the corrosion behavior of nickel-base alloys is less well documented and understood. This paper reviews some of this literature pertaining to the corrosion resistance of niobium containing nickel-base alloys and presents some in-house data on corrosion performance of certain nickel-base alloys with varying additions of niobium. Primary focus will be on the contribution of niobium to the enhancement of sulfidation resistance in high-temperature sulfur-containing environments such as is found in chemical, petrochemical, power and petroleum refining plants. In addition to the use of niobium in high temperature alloys, the effect of increasing niobium content on aqueous corrosion resistance with an emphasis on localized corrosion resistance will be explored.

To aid in explaining the mechanism(s) by which niobium is effective in enhancing corrosion resistance, key observations from a search of the literature will be summarized. The paper will report on two important factors that tend to explain the role of niobium. First, it has been suggested that niobium decreases the time required for an alloy to form its protective scale, such as Cr_2O_3 , in an oxidizing/sulfidizing environment. Secondly, niobium additions may tend to form a blocking precipitate within the external scale that acts as a barrier for the inward diffusion of sulfur through scale defects.

Then, corrosion data on several commercially significant Nb-containing alloys in typical industrial environments will be shown. These applications include petrochemical and chemical uses in both aqueous and high temperature environments and in both waste-to-energy and coal-fired boilers. To complete the paper, niobium's role in Ni-base alloys for aqueous service and the development of a Nb enhanced alloy will be discussed.

Introduction

Niobium is recognized as a key alloying element in a number of major nickel-base wrought alloys. It is the purpose of this paper to examine the role that Nb can and does play in enhancing corrosion resistance in certain alloys and environments. To clearly define the enhancement achieved by the addition of Nb, an aqueous and two high temperature Nb-free corrosion resistant alloys were selected and the improvement in corrosion resistance evaluated as a function of additional Nb content. Earlier literature studies have suggested that Nb improves corrosion resistance in high temperature sulfidizing environments so this has become one focus of our study. Microstructural examination of the scales formed at high temperature is studied to aid in defining the role of Nb. Additional literature shows that Nb may enhance the localized corrosion resistance of nickel-base alloys. To examine the effects of Nb additions on

aqueous corrosion, a commercial Ni-Cr-Mo-W alloy (INCONEL[®] alloy 622) was selected and the effects of Nb additions on critical pitting (CPT) and crevice corrosion temperature (CCT) in two severe environments were determined. In addition to these laboratory studies, data on Nb-containing nickel-base alloys in commercial applications is presented to correlate with and add to the laboratory studies. It seems appropriate to begin by examining the relevant properties of Nb as they pertain to niobium's use in nickel-base alloys.

The Elemental Properties of Niobium

Niobium, a body-centered cubic (BCC) Group VA element, is one of the four major refractory elements used in nickel-base alloys along with Mo, W and Ta. Niobium, as a refractory element is of relative low modulus ($100 \text{ n/m}^2 \times 10^6$), relative low melting point (2468°C), relative low density (8.4 g/cm^3) and large atomic size (2.852 \AA). Niobium is the least soluble refractory in Ni and Ni-Cr alloys. The atomic mismatch of Nb with nickel and iron is the greatest of the refractory elements. Mismatch contributes to limiting solubility. Niobium is also the most electropositive of the refractory elements. This electropositive characteristic defines why Nb has a strong affinity for the formation of A_3B -type TCP phases, such as, γ' (Ni_3Nb), the body-centered tetragonal (BCT) strengthener phase in several nickel-base superalloys. The electropositive nature of Nb favors NbC ($-\Delta F = 30 \text{ Kcal/g-atom}$) and NbN ($-\Delta F = 38 \text{ Kcal/g-atom}$), usually present to some extent as primary or secondary phases in such alloys as 625, 706 and 718. Carbon and nitrogen can combine to form primary and secondary Nb(C,N) as well. Niobium has a moderate affinity for oxygen forming NbO, NbO₂ and Nb₂O₅ [$1/5 \text{ Nb}_2\text{O}_5$ ($-\Delta F = 38 \text{ Kcal/g-atom}$)]. Data on the sulfides and chlorides of Nb are not available.

The Effect of Niobium on the Sulfidation Resistance of Nickel-Base Alloys

Earlier studies have attributed a benefit to Nb in high temperature sulfidizing environments^{1,2}. Because of this and since sulfur is one of the most common corrosive contaminants in high temperature industrial environments³, it was elected to select two high temperature alloys and study the effect of Nb additions on the rates of corrosion and attempt to define the role of Nb using Scanning Auger Microscopy (SAM) and the Electron Microprobe (EM).

The rate of sulfidation of pure Nb has been established as being extremely low^{4,5}. Investigators have studied the sulfidation of Ni-Nb^{6,7} and Co-Nb⁸ and found the rates of sulfidation lower than those of pure Ni and Co but not approaching that of pure Nb. Baxter and Natesan studied the effects of Nb in experimental Fe-Ni-Cr alloys and showed that additions of Nb up to 6wt% aided in the formation of a protective Cr₂O₃ scale under oxidizing/sulfidizing conditions at 875°C ^{1,2}. These authors attributed the improvement in sulfidation resistance in their alloys to two factors. First, that the Nb decreased the time required for these alloys to form a protective Cr₂O₃ scale. Secondly, that Nb formed an oxide within the external Cr₂O₃ scale and thus acted as a barrier for the inward diffusion of sulfur through possible defects in the scale.

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Experimental Procedure for Defining the Role of Nb in High Temperature Sulfidation

The chemical composition of two variants of INCONEL[®] alloy 693 (nominal base composition of Ni-30wt%Cr-5wt%Fe-3.3wt%Al) and two variants of INCOTHERM[®] alloy TD (Ni-15wt%Cr-1.4wt%Si) are shown in Table 1 as alloys HV5660, HV5661 and HV9601, HV9602 respectively. Machined pins of these alloys (7.62 mm in diameter x 17.78 mm in length) with a 32 micro-inch surface finish were exposed to a gaseous environment consisting of 54%H₂-45%CO₂-1%H₂S at 816°C. During the test, the samples were removed from the electric furnace (mullite tube with end caps) at regular intervals, weighed, and placed back into the environment for additional exposure. At the completion of the testing, Harry Meyer III and Larry Walker of the Oak Ridge National Laboratory examined the surface morphology of each sample with a scanning electron microscope (SEM) and the surface chemistry of the corrosion product using SAM. After surface analysis, the samples were cross-sectioned and examined with EM.

Table 1 – Composition (wt %) of the Alloys Used to Study the Role of Nb in Sulfidation

Heat #	C	Fe	Si	Ni	Cr	Al	Nb
HV5660	0.006	5.80	0.12	Bal	30.0	3.3	0.54
HV5661	0.009	4.30	0.14	Bal	30.0	3.3	2.04
HV9601	0.030	--	1.38	Bal	15.1	--	0.88
HV9602	0.015	--	1.43	Bal	15.4	--	1.90

Results of the Variants of INCONEL alloy 693

Figure 1. Weight change results for the alloys presented in Table 1 exposed to 54% H₂ - 45% CO₂ - 1% H₂S at 816°C

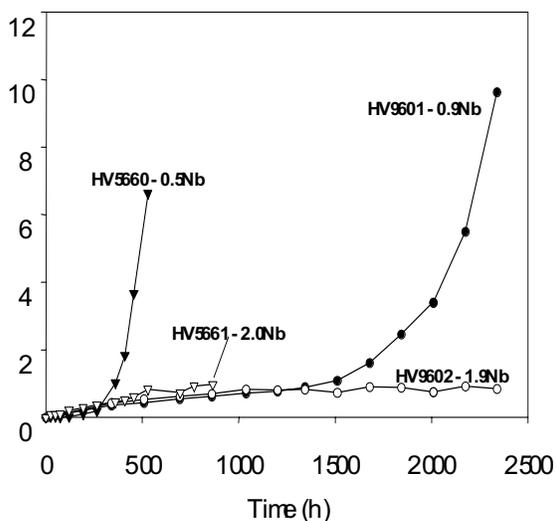


Figure 1 shows the weight change exhibited by each of the variants of INCONEL alloy 693 during exposure to a 54%H₂-45%CO₂-1%H₂S gas mixture at 816°C. As shown in this graph, alloy HV5661, containing the greatest Nb content, exhibited the lowest rate of sulfidation as demonstrated by its lower weight gain. SEM revealed the two alloys exhibited different Cr₂O₃ scale morphologies. The oxide scale morphology of the leaner Nb alloy (HV5660) is relatively smooth, in contrast to the rough fine-grained scale grown on alloy HV5661. Chemical analysis of the surface regions where accelerated attack occurred on HV5660 showed the presence of Fe, Cr, Al, O and traces of S. In

contrast, the region of accelerated attack on HV5661 showed high levels of S, Cr and O with some Ni present as seen in Figure 2. Backscatter electron (BE) imaging of the cross-section of alloy HV5661 shows the external scale to consist of Cr, O with Nb present. Beneath the external scale, at the scale/alloy interface, S was present and the internal precipitates were rich in Si, O and S. Elemental analysis was also conducted just beneath the scale/metal interface in order to determine the extent of elemental enrichment and/or depletion in this area. The results

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in Table 2 show little change in the compositional gradient of HV5660 while HV5661 exhibits depletion of Cr at the scale/alloy interface and a significant enrichment of Nb.

Table 2 – Electron Microprobe Concentration Data for HV5660 and HV5661

Heat #	Composition Beneath Interface		Composition in Bulk Alloy	
	Cr	Nb	Cr	Nb
HV5660	30.0	0.5	30.0	0.6
HV5661	22.1	2.2	28.0	1.7

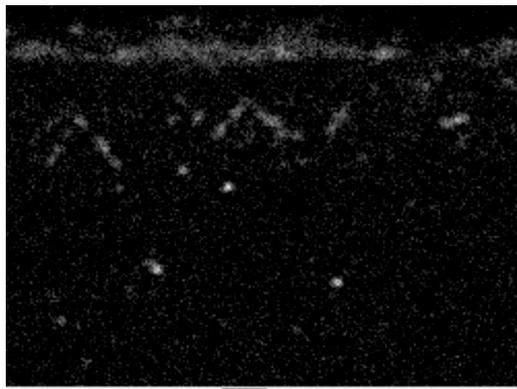
Results of the Variants of INCOTHERM alloy TD

Figure 1 (above) depicts the weight change exhibited by each of the variants of INCOTHERM alloy TD during exposure to a 54% H_2 -45% CO_2 -1% H_2S gas mixture at 816°C. As shown in this graph, alloy HV9602, containing the higher level of Nb, exhibited the lowest rate of sulfidation as demonstrated by its lower weight gain. However, sulfide nodules were found on both HV 9601 and to a far less extent on HV9602. Chemical analysis of the surface regions where accelerated attack occurred on HV9601 showed high levels of Cr, O and small amounts of Ni. In contrast, the region of accelerated attack on HV9602 showed high levels of Cr, O, and Nb present. Backscatter electron (BE) imaging of the cross-section of alloy HV9601 shows the external scale to be rich in Cr and O (probably Cr_2O_3). Beneath the external scale, at the scale/alloy interface, S was present and the internal precipitates were rich in Cr and S (probably CrS). Like HV5661, HV9602 exhibits significant Nb present in the Cr_2O_3 scale and Nb, Cr, O and S all present beneath the scale/alloy interface.

Elemental analysis was also conducted just beneath the scale/metal interface in order to determine the extent of elemental enrichment and/or depletion in this area. The results are shown in Table 3 with depletion of Cr at the scale/alloy interface in both alloys HV9601 and HV9602 and a significant enrichment of Nb at the scale/alloy interface in alloy HV9602. The BE images for HV9602 are shown in Figure 3.

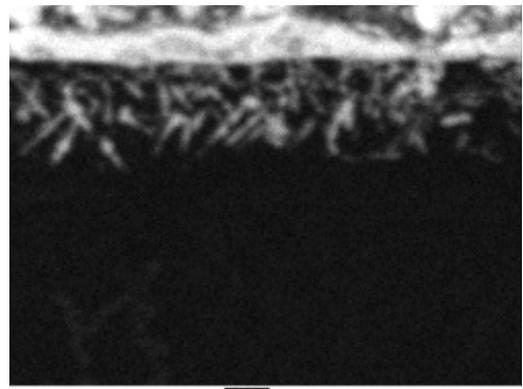
Table 3 – Electron Microprobe Concentration Data for HV9601 and HV9602

Heat #	Composition Beneath Interface		Composition in Bulk Alloy	
	Cr	Nb	Cr	Nb
HV9601	11.6	0.6	15.3	0.8
HV9602	10.4	3.1	15.3	2.0



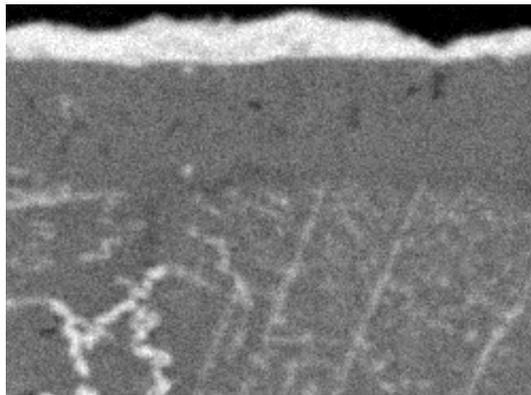
S EZ04553F 10µm

a)



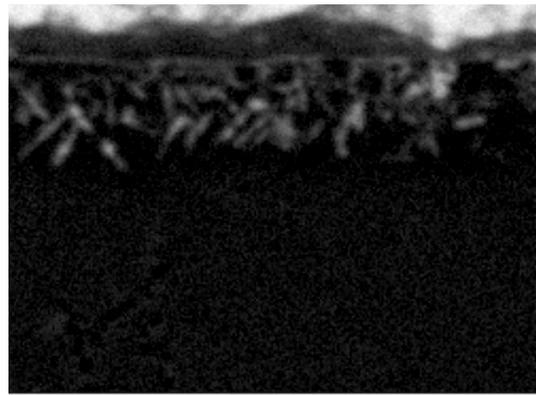
O EZ04553D 10µm

b)



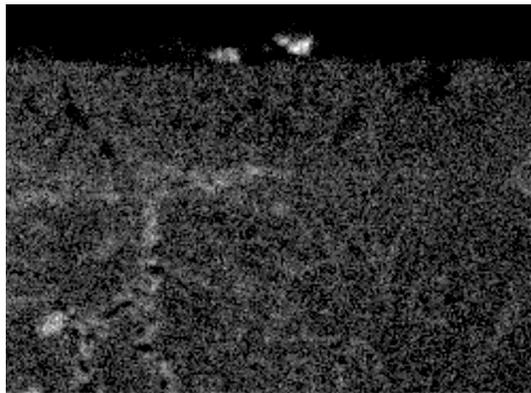
Cr EZ04553H 10µm

c)



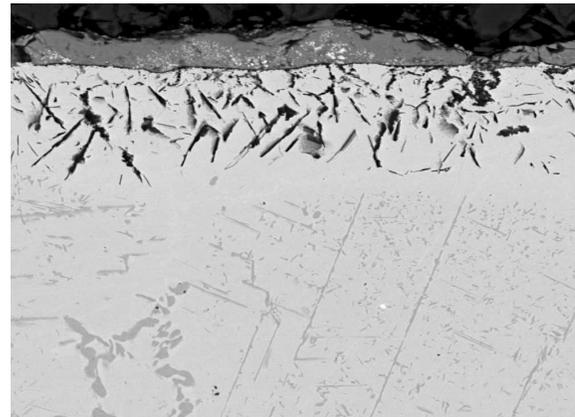
Si EZ04553G 10µm

d)



Nb EZ04553E 10µm

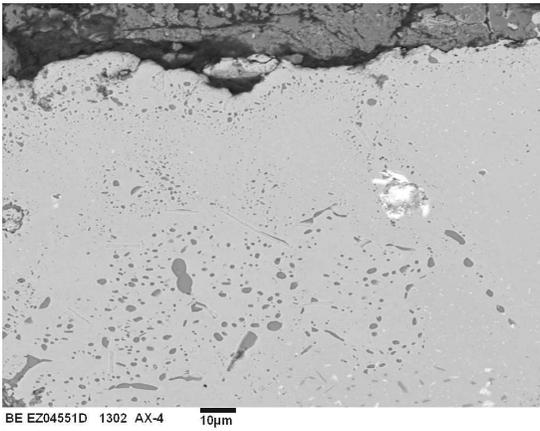
e)



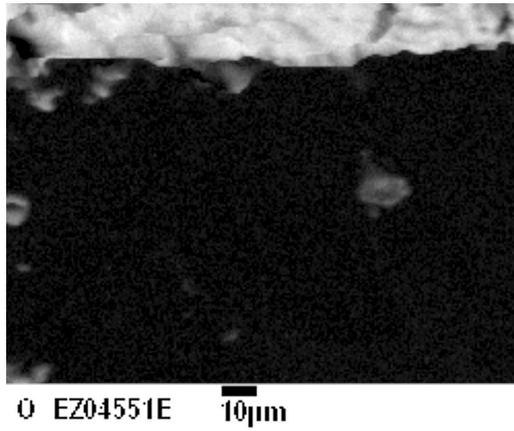
BE EZ04553C 1302 AX-2 10µm

f)

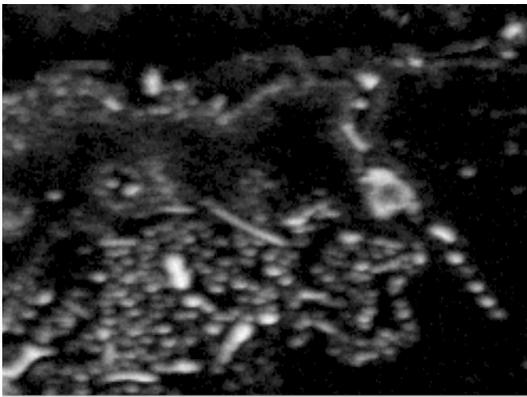
Figure 2. a-e: Elemental dot maps of region shown in Figure 2(f) (HV5661).



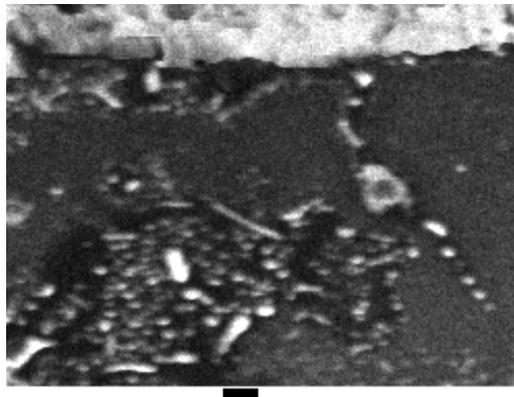
a)



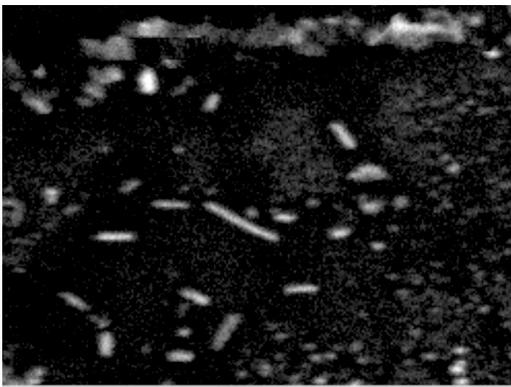
b)



c)



d)



e)

Figure 3. (a) Backscattered electron image of the cross-section of sample HV9602 and (b)-(e) associated elemental dot maps.

Discussion of the Role of Nb in Nickel-Base Alloy Sulfidation Resistance

The weight changes results plotted in Figure 1 clearly show the advantage of Nb additions to two different Ni-base alloys. Baxter and Natesan studied the effect of Nb additions on the sulfidation resistance of an Fe-12wt%Cr and an Fe-20wt%Ni-25wt%Cr alloy by adding 1,3 and 6 weight percent Nb to these base compositions^{1,2}. In their studies, the addition of Nb improved the sulfidation resistance by increasing the initial rate of protective scale formation thus reducing the level of S incorporated into the scale and within the alloys⁹. These authors also postulated the addition of Nb enhanced oxide nucleation resulting in a finer oxide scale grain size. This result is also consistent with the SEM results observed for HV5661 as compared to HV5660 in this study. In addition, this study has also confirmed that Nb is incorporated within the external scale, thus possibly aiding in blocking the inward diffusion of S through the scale and into the alloy where detrimental Cr-sulfides would form. Based on these results, it is apparent that judicious additions of Nb can be beneficial for enhancing the sulfidation resistance of Ni-base alloys.

The Role of Niobium in Aqueous Environments

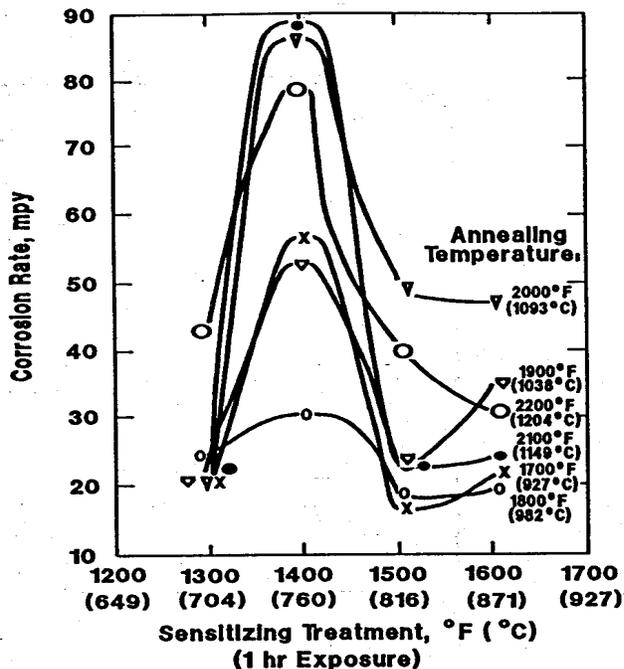


Figure 4: Effect of heat treatment on corrosion rate of 625 in boiling 65% Nitric acid¹⁴

niobium carbides¹¹. Thus, the carbon is tied up and subsequent exposure, either through welding or service, at temperatures between 700 and 900 °C does not cause chromium carbides to form at grain boundaries. In an unstabilized alloy such as 304 SS or INCOLOY alloy 800, chromium carbides would precipitate at grain boundaries during exposure around 800 °C causing a chromium depleted region around the grain boundaries. This phenomenon, called sensitization, renders an alloy susceptible to severe intergranular attack due to the chromium depletion. For example, if 625 does not receive an anneal that precipitates niobium carbides, its susceptibility to IGA increases dramatically as seen in Figure 4.

Like other refractory elements, such as Ta, W, and Mo, elemental niobium has excellent resistance to many common aqueous environments, including most mineral and organic acids. For example, niobium resists most concentrations and temperatures of HCl and nitric acid extremely well. One notable exception is niobium's resistance to HF, which is poor¹⁰. Niobium's impact on nickel-base alloys designed for resistance to aqueous media, however, has been minor compared to Mo. Several well-known alloys such as INCONEL[®] alloy 625 and INCOLOY[®] alloy 020 utilize Nb. Alloy 020, which contains 0.5% Nb, takes advantage of niobium's previously mentioned propensity to form carbides. Both 020 and 625 are stabilized against intergranular attack (IGA) through an annealing step that precipitates

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Applications that operate at high temperatures can also benefit from Nb’s stability enhancing properties. Alloys 625 or 347 SS are often specified for hydrotreating units where hydrogen is used to remove sulfur and nitrogen from fuels. Upon shutdown of these units, alloys 800 or 304 SS would often crack due to polythionic acid (PTA) stress corrosion cracking¹². The stabilization through formation of niobium carbides prevents the PTA SCC that plagues unstabilized alloys. Alloy 625 is also used in a variety of aqueous applications. To examine the effect of niobium on the corrosion resistance of 625, a quick comparison with INCONEL alloy 617 is in order. As seen below in Table 4, the compositions of 625 and 617 are similar with three exceptions. Alloy 617 contains no niobium, but it contains cobalt at the expense of nickel. Alloy 617 also has more carbon than 625 for higher strength. In spite of these differences, niobium’s influence on general corrosion may be loosely examined as seen in Table 5.

Table 4: Nominal compositions of alloys 625 and 617

Alloy	C	Fe	Ni	Cr	Mo	Nb	Co	Al
625	0.02	2.9	Bal (~62)	21.8	9.6	3.5	-	0.3
617	0.09	1.1	Bal (~53)	22.0	9.0	0.1	12.5	1.1

Table 5: General corrosion comparison of 625 and 617 in several acidic media^{17, 18}

Environment	Temperature (°C)	625 rate (mpy) ¹⁷	617 rate (mpy) ¹⁸
50% H ₂ SO ₄	80	17	94
50% H ₃ PO ₄	Boiling	5	31
10% H ₂ SO ₄	Boiling	17	28
40% H ₂ SO ₄	80	5	40
60% H ₃ PO ₄	Boiling	14	50

These brief examples illustrate how niobium has been used in aqueous environments in the past both as a stabilizer and to enhance general corrosion, but Nb also plays a role in localized corrosion resistance.

Niobium’s effect on localized corrosion resistance

Some work has been done in the past that suggests that niobium has a detrimental effect upon the localized corrosion resistance of nickel base alloys¹⁵. This work suggests that Nb additions up to 0.8% in a Ni-Cr-Mo-W alloy has no effect while additions above 0.8% are detrimental. No explanation was given although the author noted that in high carbon alloys niobium ties up the carbon allowing chromium to increase the pitting resistance. No data on the condition of the test pieces or other elements was presented in this work though. Other work has shown that the addition of niobium to 625 enhances crevice corrosion up to about 2.5%, but neither helps nor hinders after that point¹⁶. This work however was performed on as-cast samples and thus niobium segregation upon solidification may have skewed the results.

In general, several methods exist for ranking the localized corrosion resistance (both crevice and pitting) of stainless steels and nickel-base alloys. The most common and widely accepted ranking system is a numerical ranking called the pitting resistance equivalency number (PREN). The PREN is a number that is calculated based on the weight percent of certain alloying elements in the metal. This number also varies depending on which alloying system is used. For example, Hibner presented a crevice corrosion equivalency number (CCEN) as $CCEN = \%Cr + 2.6(\%Mo) + 4.4(\%Nb) + 6(\%W)$ ¹⁹. For nickel-base alloys, the PREN can also be calculated as $\%Cr + 1.5[(\%Nb) + (\%Mo) + (\%W)]$ ²⁰. So, based on empirical data, niobium

has been included as an element that is beneficial in pitting and crevice corrosion resistance. Of course, being in the same family as Mo, this inclusion makes sense, but very little experimental work has been performed to isolate niobium's effect on the pitting or crevice corrosion resistance of nickel-base alloys. In spite of evidence that Nb is helpful to localized corrosion resistance, the most resistant nickel base alloys from the Ni-Cr-Mo family generally do not include niobium. Thus, experimentation was conceived and carried out to determine the true effect of Nb on localized corrosion resistance.

Results of the Variants of INCONEL alloy 622

To determine niobium's effect on the localized corrosion resistance of a commercial Ni-Cr-Mo-W alloy, four 100-pound heats of a modified Ni-Cr-Mo-W were melted. In these heats, all elements except nickel and niobium were held constant. Additions of 0, 1.5, 3.0, and 4.5% niobium were included at the expense of nickel. The actual compositions of the experimental heats are listed below in Table 6.

Table 6 – Composition of Alloys Used to Study the Role of Nb in Localized Corrosion

Heat #	Composition (wt%)							
	C	Fe	Ni	Cr	Mo	W	Nb	Al
HV0165	0.008	2.52	Bal	20.69	14.52	3.26	0.006	0.34
HV0166	0.008	2.57	Bal	21.14	14.69	3.30	1.42	0.34
HV0167	0.017	2.52	Bal	20.69	14.5	3.24	2.90	0.36
HV0168	0.0024	2.51	Bal	20.67	14.54	3.25	4.32	0.38
622	0.004	2.3	Bal	20.5	14.3	3.2	0.004	0.26

After hot working, cold working, and annealing, samples from each heat approximately 5 cm x 2.5 cm x 0.3 cm thick were cut for testing. All samples were dry sanded to a 120-grit finish. Samples for crevice corrosion tests were then drilled with a 0.7 cm drill bit. All samples were scrubbed with pumice and washed to ensure the surface had no contaminants. Crevice corrosion samples were fitted with slotted Teflon washers and torqued to 40 in-oz with titanium bolting. Test pieces were then checked to ensure electrical isolation of the sample to prevent galvanic corrosion. Tests were then placed in a Teflon autoclave with Green Death (11.9% H₂SO₄ + 1.3% HCl + 1% FeCl₃ + 1% CuCl₂) solution and isolated in a lab furnace at the desired temperature for the prescribed length of time. Other tests were placed in an acidified 6% ferric chloride (ASTM G48C) solution and tested in a constant temperature water bath. After testing, the samples were evaluated for either pitting or crevice corrosion and depending on the result, the temperature was increased or lowered until the critical pitting (CPT) or crevice temperature (CCT) was isolated. Both of these solutions are highly corrosive and stainless steels will show attack below room temperature. The Green Death solution mimics a harsh environment encountered in flu gas desulfurization. Table 7, below, shows the results of this testing.

Table 7 Results for Localized Corrosion Tests on Nb enhanced Ni-Cr-Mo-W alloy

Heat	Green Death CPT	Green Death CCT	G-48 C CCT	% Nb	PREN
HV0165	130 °C	125 °C	>85	0	47.4
HV0166	> 140 °C	140 °C	>85	1.4	50.2
HV0167	> 140 °C	> 140 °C	>85	2.9	51.7
HV0168	> 140 °C	> 140 °C	>85	4.3	53.8
622	125 °C	125 °C	>85	0	46.8

PREN is calculated as $Cr + 1.5(Mo+W+Nb)$ using each element's weight percent

As seen by the above results, niobium additions, like molybdenum additions, greatly enhance the localized corrosion resistance of a Ni-Cr-Mo alloy. Although this research shows no detrimental effect of increased Nb additions, thermodynamic modeling has demonstrated increased secondary phase formation as Nb increases. The microstructure of heats HV0166 (1.4% Nb) and HV0168 (4.3% Nb) are presented in Figures 5 and 6.

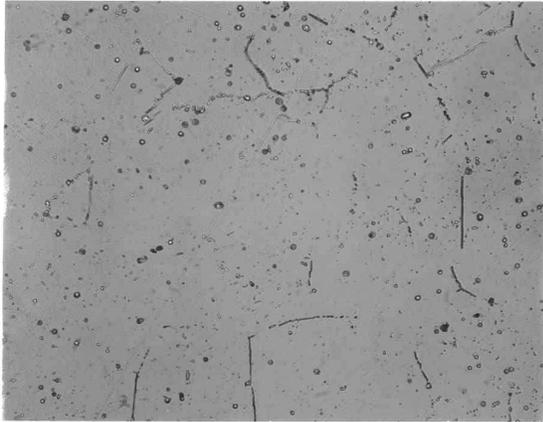


Figure 5: Microstructure of 1.5% Nb enhanced 622. Image is of 1175 °C annealed plate and is 100x.

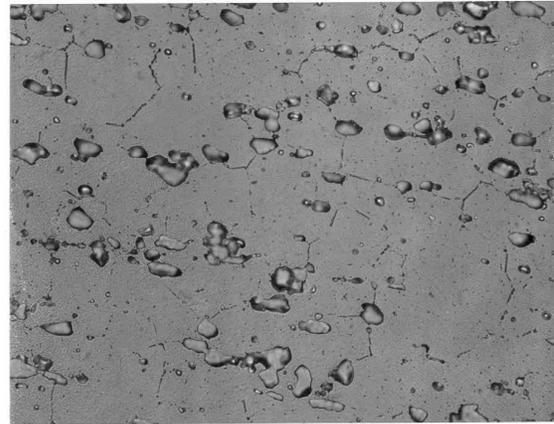


Figure 6: Microstructure of 4.5% Nb enhanced 622. Image is of 1175 °C annealed plate and is 100x.

As seen in HV0166, most of the secondary phase is gone, while HV0168 has un-dissolved intermetallics throughout the microstructure. The increasing Nb leads to larger and more prevalent precipitates that cannot be dissolved through standard mill annealing. Finally, the addition of niobium decreases the predicted melting point of the alloy dramatically. This significantly narrows the hot working temperature window. The effect of niobium on the secondary phase solvus temperature and the liquidus temperature are shown in Table 8.

Table 8 Effect of Nb on thermodynamic equilibrium temperatures of a Ni-Cr-Mo-W alloy

Heat	Intermetallic solvus temperature* (°C)	Liquidus temperature* (°C)	%Nb
HV0165	1080	1350	0
HV0166	1115	1305	1.4
HV0167	1150	1260	2.9
HV0168	1180	1215	4.3

*Temperatures calculated with JMatPro thermodynamic modeling software

Although these precipitates do not seem to hinder the localized corrosion resistance of the alloy, more work must be done to determine their effect on the general corrosion resistance and mechanical properties. Based on thermodynamic modeling, however, about 2% niobium appears to provide a substantial boost to pitting resistance while at the same time, remaining relatively benign in the microstructure. It is further expected that the Nb will behave as a stabilizer to decrease the propensity for IGA in these alloys as previous work on 0.75% Nb Ni-Cr-Mo-W alloy demonstrated improved stability under these conditions¹³.

Conclusions

The effect of Nb on the high temperature sulfidation resistance of two Ni-base alloys was studied. The results show:

- 1) The addition of Nb can improve the sulfidation resistance of a Ni-30wt%Cr-5wt%Fe-3wt%Al and a Ni-15wt%Cr-1.4wt%Si alloy.
- 2) Electron microprobe chemical analysis shows the presence of Nb within a Cr-rich external oxide scale and, at least in certain alloys, a niobium enhancement beneath the scale/alloy interface.

The effect of Nb on the localized corrosion resistance of a Ni-Cr-Mo-W alloy was studied. The results show:

- 1) The addition of Nb enhances the pitting and crevice corrosion resistance of a Ni-21wt%Cr-15wt%Mo-3wt%W alloy significantly, contrary to a previous study.
- 2) The addition of more than three weight percent niobium to this alloy results in intermetallic secondary phases that cannot be feasibly solution annealed.
- 3) Niobium's role in aqueous corrosion utilizes both its propensity to form carbides and its status as a refractory element to increase resistance to IGA, PTA SCC, as well as localized corrosion.

Acknowledgment

The authors are indebted to Harry M. Meyer III and Larry R. Walker of the Oak Ridge National Laboratory for performing the microstructural analysis of the alloys exposed to the high temperature sulfidation atmospheres. Their efforts are to be published in greater detail in the near future.

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