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NITRIDING OF
STAINLESS STEEL REACTOR COMPONENTS
AND FUEL CLADDING
IN THE CRBRP

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~~APPLIED TECHNOLOGY~~

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ABSTRACT

A study was conducted to determine if nitriding will occur in stainless steel reactor components and fuel cladding. It was found that some nitriding of the reactor components at and above the sodium-argon interface will occur under service conditions, but that the depth of the nitride case (approximately 0.035 in.) will not be large enough to be detrimental to material properties. It was also determined that the fuel cladding and components below the sodium-argon interface will not be subject to nitriding. A recommendation is made that the nitrogen content of the primary system cover gases be limited to ≤ 1 vol %.

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1.0 SUMMARY

Nitride formation will occur under service conditions in the reactor components at and above the sodium-argon interface. However, the amount and depth of nitride formation [approximately 0.035 in. maximum in 30 years with 1 wt. % (1.43 vol %) nitrogen in the argon] will not significantly affect material properties except for thin-walled components.

Little or no nitriding will occur in the fuel cladding and components below the sodium-argon interface because very little nitrogen is in the sodium. Some carburizing of the fuel cladding may occur, caused by the carbon originally in the sodium, but not enough to cause problems.

A nitrogen content of ≤ 1 vol % is recommended for the primary system cover gases. This level of nitrogen in the argon will limit nitride case depths to less than 0.035 in. in 30 years.

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2.0 INTRODUCTION

A study was conducted to determine if nitriding will occur in austenitic stainless steel fuel cladding and reactor vessel components when the sodium "washes off" the steel's oxide coating.

The components have a 30-year design life and operate at temperatures up to 1020°F. The cladding has a 1-year design life and operates at a temperature of 1200°F. The cover gas will be argon at a positive pressure of 10 in. WG (approximately 0.025 atm). The nitrogen content of the argon delivered to the plant will be 15 vppm maximum [$P(N_2) = 1.538 \times 10^{-5}$ atm].[22] In-leakage of nitrogen from cell atmospheres may increase the nitrogen content of the recycled argon cover gases substantially above this concentration.

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3.0 COMPATIBILITY OF AUSTENITIC STAINLESS STEELS WITH NITROGEN

Nitriding is a process whereby a metal is case-hardened by the diffusion of nascent nitrogen into the surface of the steel, where it reacts to form nitrides.[21] Commercially, nitrided surfaces are applied by the high-temperature decomposition of ammonia gas or solid nitrate compounds. Nitriding temperatures for steels range from 925 to 1100°F.

Reports of nitriding occurring in the presence of nitrogen gas (N_2) at temperatures of 700 to 1700°F have been published.[11,29] These temperatures are well below those at which nitrogen normally dissociates. One source stated that dissociation is unimportant at temperatures less than 3000 K (4940°F).[10] Another, using an arc-heated channel flow system, found that significant dissociation does not occur until gas temperatures reach 10,000 K (17,560°F).[13] A steel surface, however, will act as a catalyst,[14,15,21] enabling some of the N_2 that adsorbs on the surface to dissociate. Some of the nascent nitrogen (N) diffuses into the steel. Because nascent nitrogen rapidly recombines ($2\ N \longrightarrow N_2$),[6] not all of it will be available for diffusion and subsequently for nitride formation.

The effect of a neutron environment was investigated to determine if radiation would dissociate N_2. It indicates that it is easier for gamma rays to knock out an electron to form an ion pair (N_2^+) than to dissociate the molecule. Even the number of ion pairs formed is small compared to the total number of nitrogen molecules.

Austenitic stainless steels nitride with difficulty to shallow case depths and low hardness because of the Cr_2O_3 film that normally covers it.[2] The oxide is an effective barrier to the diffusion of nitrogen into the steel.[3,19] Stainless steel parts must be perfectly clean and free of embedded foreign particles before being nitrided.[2] However, highly buffed or burnished surfaces retard nitriding. Nitriding adversely affects the corrosion resistance of stainless steels, but it provides a case with a lower coefficient of friction, thus improving abrasion resistance. The core material will remain soft. Nitride cases formed at higher temperatures are deeper, tougher, and less hard.

The normal nitrogen content of austenitic stainless steel is 0.003 to 0.02%.[7,23] Some stainless steels have up to 0.25% N deliberately added. Nitrogen in solution stabilizes austenite and increases strength. Nitriding increases the yield strength, creep and fatigue properties, and sometimes the tensile strength of carbon and stainless steels.[2,18,30]

The nitriding of stainless steel in nitrogen atmospheres at high temperatures is prevented by the addition of small percentages of oxygen. The oxygen produces a thin, impervious oxide layer. The continuous presence of oxygen provides a self-healing effect to the rupture of this oxide barrier. The presence of oxygen will not, however, block the formation of nitrides during creep. Creep-rupture specimens from tests conducted in air and nitrogen at 1500 and 1700°F showed nitride formations, mostly at the grain boundaries. Test durations ranged from 50 to 10,500 h.

The effect of oxygen additions on the nitriding of stainless steel has been studied as a function of time, temperature, and oxygen content. The results are presented in Table 1.

TABLE 1

EFFECT OF OXYGEN CONCENTRATION ON NITRIDING SS[9]

Exposure Time (months)	Oxygen Concentration	Change in N_2 Concentration (wppm) of SS Samples at Temperatures of		
		1000°F	1200°F	1400°F
1	500	Nil	-	160
	5000	Nil	-	Nil
3	500	Nil	-	255
	5000	Nil	-	Nil
6	500	Nil	136	410
	5000	Nil	-	Nil

In the absence of oxygen or in the presence of sodium, the development of nitride-case surfaces has been reported. One set of tests performed at AI was conducted in a sodium-nitrogen system at 1000°F. Specimens of Type 304 stainless steel tubing, located in the low-velocity leg of a dynamic sodium loop, were positioned so that a portion of each specimen protruded above the sodium into the nitrogen cover gas. The cover gas pressure was maintained at greater than 1 atm to keep out the air. Table 2 summarizes the results. The extent of nitriding was greatest at the sodium-nitrogen interface, although a nitride case also developed in the nitrogen cover gas. Similar results were observed in Type 347 stainless steel over the temperature range 900 to 1100°F. Little or no nitriding developed 18 in. below the interface. In an 850°F sodium system through which nitrogen was bubbled, Type 304 stainless steel did not nitride.

TABLE 2

NITRIDE CASE THICKNESS FOR TYPE 304 STAINLESS STEEL
IN 1000°F SODIUM-NITROGEN SYSTEM

Exposure Time (h)	Specimen Position (mils)			
	Sodium Side Interface		Nitrogen Side Interface	
	Slightly Below	3 in. Below	Slightly Above	3 in. Above
660	0.9	0.1	None	None
1140	1.5	0.1	0.1	0.1
1400	2.0	0.3	-	0.4

In a series of experiments performed at ANL,[29] 7-day tests were conducted in which Type 304 stainless steel foils were exposed to sodium at 1020 and 1200°F. The cover gases used were argon with 0.1 wt. % (0.143 vol %) and 1.0 wt. % (1.43 vol %) nitrogen. After exposure, microscopic examination revealed massive nitrides at the grain boundaries and smaller nitride precipitates within the grains. The depth of the nitrides at the grain boundaries near the sodium-exposed surface was less than 0.001 in. (1 mil). The degree of nitriding increased with temperature and nitrogen content of the cover gas. In addition, examination of a plug exposed at the sodium-argon interface in the tank suggests that nitriding at the sodium level may be more important than nitriding in the sodium.

The foregoing work shows the accelerated nitriding rates in the presence of sodium (and potassium) and their vapors. It is suspected that the liquid metal is a very effective getter of oxygen in the atmosphere used. However, the amount of nitriding also increases with time, temperature, strain rate, and nitrogen content of the atmosphere.[1,2,21,23]

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4.0 SOLUBILITY OF NITROGEN IN STAINLESS STEEL

Nitrogen is soluble in steel (and iron) only in the nascent form.[7,17,19,20,23,31] The factors affecting solubility are temperature, crystal structure, and nitrogen partial pressure.[23]

Figure 1 shows the effect of temperature and crystal structure. The austenitic (γ) section of the curve has been extrapolated back to 550°C (1020°F), the temperature we are concerned with. Austenitic iron has a face centered cubic (γ) lattice, which allows a higher nitrogen solubility than a body centered cubic (α or δ) lattice.[27] The austenitic stainless steel will have essentially the same lattice parameters as γ-iron, so the solubility of nitrogen in it will be nearly the same as that of γ-iron. The alloying elements (primarily nickel and chromium) will not significantly affect nitrogen solubility in this case.

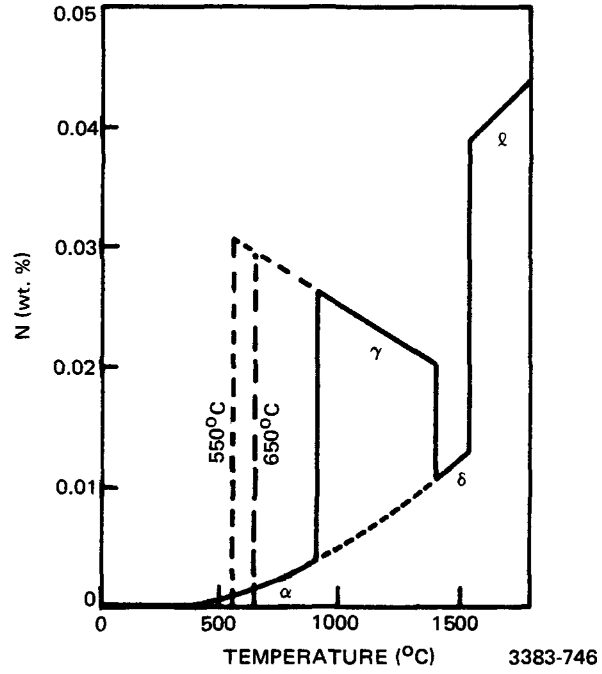

Figure 1. Solubility of Nitrogen Gas at 1 atm in Iron; Stable Equilibrium Diagram at 1 atm (Ref. 17)

The equilibrium solubility of nitrogen in γ-iron at 550°C can be estimated from Figure 1: at 550°C, $S = 0.031$ wt. %; at 650°C, $S = 0.030$ wt. %. The solubility (S) can also be calculated from the equation[23] $\log S = 450/T - 1.955$, where T is the absolute temperature. At 550°C, $S = 0.039$ wt. % N; at 650°C, $S = 0.034$ wt. % N. The equation was only meant to be used in the normal γ range, 910 to 1400°C, but the figures agree quite well. These figures are for a nitrogen partial pressure of 1 atm. It is reasonably safe to assume that under these conditions, the equilibrium solubility of nitrogen in stainless steel is approximately 0.035 wt. % at 550°C and approximately 0.032 wt. % at 650°C.

The solubility of nitrogen in steel obeys Sieverts Law,[7,12,17,31] $S = K\sqrt{P}$, where P is nitrogen partial pressure and K is a proportionality constant.

The equilibrium solubility of nitrogen in austenitic stainless steel will be calculated for several nitrogen partial pressures: the original argon gas with 15-vppm nitrogen and the argon cover gas assuming in-leakage of 0.1, 0.5, and 1.0 vol % from the nitrogen-inerted cells surrounding the reactor. The gas in-leakage contains 0.5 to 2.0 vol % O_2, but a content of 1.0 vol % will be assumed. This will give nitrogen partial pressures in the argon cover gas of 1.538×10^{-5}, 1.03×10^{-3}, 5.09×10^{-3}, and 1.015×10^{-2} atm, respectively.

Using $K = 0.035$ wt. % N/atm$^{1/2}$ at 550°C, the following solubilities are calculated:

No in-leakage $S = 0.035\sqrt{1.538 \times 10^{-5}} = 1.37 \times 10^{-4}$ wt. % N
0.1 vol % in-leakage $S = 0.035\sqrt{1.03 \times 10^{-3}} = 1.12 \times 10^{-3}$ wt. % N
0.5 vol % in-leakage $S = 0.035\sqrt{5.09 \times 10^{-3}} = 2.49 \times 10^{-3}$ wt. % N
1.0 vol % in-leakage $S = 0.035\sqrt{1.015 \times 10^{-2}} = 3.53 \times 10^{-3}$ wt. % N

Figure 1 shows that as the temperature increases, the solubility of nitrogen in austenitic steels decreases.

Because of the low solubility of nitrogen under the service conditions, almost all of the nitrogen gained by the steel will form nitrides rather than go into solution.

5.0 CASE FORMATION ABOVE THE SODIUM-ARGON INTERFACE

Most of the nitrogen originally in the steel (approximately 0.02 wt. %) will precipitate as nitrides at room temperature and above.[23] Only 10^{-4} to 10^{-3} wt. % N will remain in solution.[17,23] Most of the nitrides formed in stainless steel are chromium nitrides (Cr_2N and CrN).[1,2,3,28] Chromium is a strong nitride former; only aluminum has a greater tendency to form nitrides.[3] Little iron nitride will form because free energy considerations (see Table 3) will prevent it. Not much manganese nitride will form because there is not much manganese in the steel. Nickel nitride decomposes at approximately 400°C (790°F), so it will not be present under service conditions.[16]

Table 3 lists some of the more important reactions that will occur between the gases and metals. Those reactions that promote formation of chromium and iron oxides will retard nitriding. The reactions of oxygen and water vapor with sodium reduce the amount of oxygen and water vapor available to form chrome-iron oxides, thereby aiding the nitriding process. The sodium also reacts with chromium oxide to remove the protective coating. This provides a clean surface where adsorption and diffusion of nitrogen into the steel can occur. For dissociation to occur, the partial pressure of an impurity gas in the argon has to be less than the equilibrium pressure given. The partial pressures of cover gas impurities are usually greater than 10^{-6} atm, so the oxides and most of the nitrides will tend to form.

The equilibrium pressure is calculated from the equation $\Delta G^\circ_T = -RT \ln K$. The proportionality constant K is equal to P^n (gas), where n is the number of moles of gas involved in the reaction. Negative free energies (ΔG°) indicate that a reaction will occur spontaneously. However, they do not tell at what rate a reaction will occur; a reaction may have a highly negative free energy yet proceed very slowly.

TABLE 3
GAS/METAL REACTIONS

Reaction	Melting Point (m) or Decomposition (d) Temperature (°C) of Compound	ΔG° at 550°C (cal)	Equilibrium Pressure (atm)	Reference
$2\ Cr_2N = 4\ Cr + N_2$	(m) 1060	24,700	2.7×10^{-7}	34,35
$4\ CrN = 2\ Cr_2N + N_2$		23,000	7.6×10^{-7}	34,35
$Fe_4N = 4\ Fe + 1/2\ N_2$	(m) 672	-7,000	5.25×10^{3}	34,35
$Ca_3N_2 = 3\ Ca + N_2$	(m) 900	63,800	1×10^{-17}	34,35
$Ni_3N = 3\ Ni + 1/2\ N_2$	(d) 360 to 420			16
$Mn_5N_2 = 5\ Mn + N_2$	(m) 950	27,900	3.8×10^{-8}	4,17,20
$Mn_3N_2 = 3\ Mn + N_2$		16,300	4.7×10^{-5}	4,17,20
$Na_3N_2 = 3\ Na + N_2$	(m) 308			39
$Cr_2O_3 = 2\ Cr + 3/2\ O_2$	(m) 2775	216,300	4×10^{-39}	34,35
$Fe_3O_4 = 3\ FeO + 1/2\ O_2$	(d) 1538	50,020	2×10^{-27}	23,34
$3\ Fe_2O_3 = 2\ Fe_3O_4 + 1/2\ O_2$	(d) 1560	22,020	1.8×10^{-12}	23,34
$Na_2O = 2\ Na + 1/2\ O_2$	(m) 920	73,100	4×10^{-20}	34
$Na_2O_2 = 2\ Na + O_2$	(m) 675	124,860	6.3×10^{-34}	40
$Cr_{23}C_6 = 23\ Cr + 6\ C$	(m) 1800	104,000		17,34
$Cr_7C_3 = 7\ Cr + 3\ C$		46,000		17,34
$Fe_3C = 3\ Fe + C$	(m) 1600	-10,300		17,34
$2\ Cr + 3\ H_2O = Cr_2O_3 + 3\ H_2$				23
$Na + H_2O = NaOH + 1/2\ H_2$		-121,500		40
$Cr_2O_3 + 6\ Na = 2\ Cr + 3\ Na_2O$				41
$7\ Cr + 3\ CO = Cr_7C_3 + 3/2\ O_2$				
$Fe_2O_3 + 3\ CO = 2\ Fe + 3\ CO_2$				17
$6\ Na + 6\ CO = Na_6(CO)_6$				24,36
$Fe_3C + CO_2 = 3\ Fe + 2\ CO$				
$7\ Cr + 3\ CH_4 = Cr_7C_3 + 6\ H_2$				
$3\ Fe + CH_4 = Fe_3C + 2\ H_2$				
$Cr_7C_3 + 3\ CO_2 = 7\ Cr + 6\ CO$				

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Other reactions that may be of importance are those of CO, CO_2, and CH_4 with chromium and iron. Under service conditions, CO and CH_4 will have a slight carburizing influence[36] and CO_2 will have a decarburizing influence. It is difficult to determine which process will dominate. However, CO is unstable below 700°C and will combine with O_2 to form CO_2.[17] At 550°C, the equilibrium ratio of $CO_2/CO = 70/1$. Carbon and carbides in steel slow down the diffusion of nitrogen and thus the rate of nitride formation. In this report, it will be assumed that little or no carburizing will occur above the sodium-argon interface, since low partial pressures of CO and CH_4 are involved and CO_2 is present to counteract their effect.

Nitriding of the stainless steel will occur at and above the sodium-argon interface. More nitriding will occur at the interface than above it. It is difficult to calculate the depth of the nitride layer that will form in 30 years. It is dependent on several things:

- The amount of in-leakage of nitrogen from the atmosphere surrounding the reactor. Higher nitrogen contents will lead to more nitride formation and a deeper case.
- Temperature: the higher the temperature, the greater the degree of nitriding. Typically, the temperature of the cover gas will range from 400 to 1200°F. Below 850°F, nitriding should not occur.
- Time at temperature: because nitrogen is a diatomic gas, the depth of the nitride layer formed is directly proportional to the square root of time, assuming the other variables remain constant.
- The amount of sodium corrosion of the stainless steel. The amount of corrosion depends on several factors (such as temperature and oxygen content), but over 30 years, it may amount to approximately 0.005 in.[38] This means that some of the nitride case will be corroded away.
- The rate at which sodium and sodium vapor remove the protective chromium oxide coating from the stainless steel.

A rough idea of the maximum depth of nitriding to be expected in 30 years can be calculated by plotting the results from Table 1 (see Figure 2). The depth of nitriding is plotted against the square root of time (in hours). One slope gives a very small case depth in 30 years. Two of the slopes are approximately the same. They give a case depth of 0.056 in. (56 mils) in 30 years. Because these figures are derived from tests in a nitrogen atmosphere, they represent a maximum rate of nitriding. From the graph, it can be seen that nitriding does not begin immediately. There is an "incubation" period before nitriding starts that may be related to the rate at which sodium removes the Cr_2O_3 coating from the stainless steel.

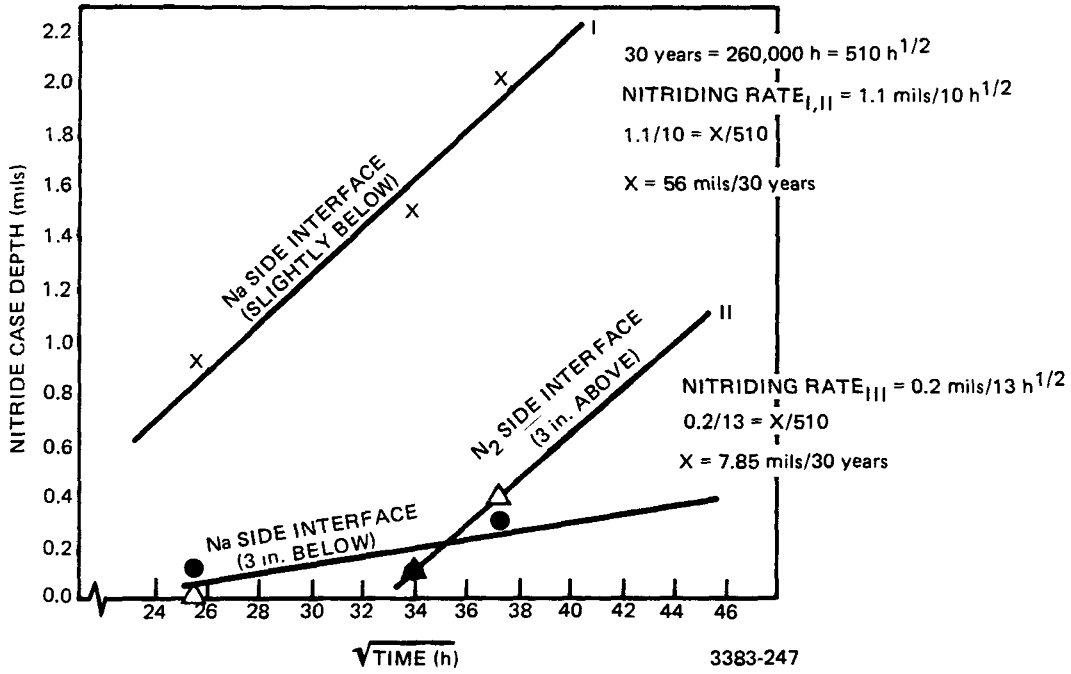

Figure 2. Nitride Case Depth versus Time for Type 304 SS Exposure to 1000°F Sodium-Nitrogen System

A more appropriate figure can be derived using the results of the 7-day (13 $h^{1/2}$) tests at ANL (mentioned above). In an argon atmosphere with 0.1 and 1.0 wt. % N, at 1200°F, nitrides formed to a depth of less than 1 mil. Setting up a ratio, 1 mil/13 $h^{1/2}$ = X/510 $h^{1/2}$ (30 years), X = nitride case depth = 39 mils. This figure is for the 1.0 wt. % (1.43 vol·%) N_2

atmosphere at 1200°F. The degree and depth of nitriding decreases with decreasing temperature and nitrogen content. The deepest penetration of nitrides is at the grain boundaries. Fewer nitrides form within the grains.

Assuming some sodium corrosion of the stainless steel, the depth of the nitride case after 30 years would be approximately 35 mils. This is also assuming argon with a nitrogen content of 1 wt. %, a constant temperature of approximately 1000°F, and a clean stainless steel surface available for nitride formation. Due to variations in these parameters over 30 years, the actual case depth will possibly be less than 35 mils. Also, the case depth will probably be smaller above the sodium-argon interface than at the interface. This case depth (35 mils maximum) will not significantly affect material properties over 30 years, unless components are thin walled.

It could not be determined what the final wt. % N of the case or the component would be. The final nitrogen content of the component would depend on its thickness.

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6.0 CASE FORMATION BELOW THE SODIUM-ARGON INTERFACE

The solubility of nitrogen in liquid sodium is barely measurable;[24,25] at 500°C (925°F) it is approximately 5.32×10^{-8} wt. % N. Nitrogen dissolves in liquid sodium in molecular form (N_2). If there is carbon in the sodium, nitrogen will be more readily absorbed, but only if the temperature is above 700°C.[25] In the presence of a few wppm calcium, nitrogen can be transported from the cover gas to a metal surface, causing nitriding.[24] The sodium will have a maximum calcium content of 10 wppm. Analysis of sodium samples from EBR-II and the high-temperature sodium facility showed that the calcium content was usually less than 1 ppm.[32,33] Small amounts of argon in the atmosphere inhibit the Ca-N_2 reaction. Several compounds of nitrogen and sodium form, but they melt or dissociate below 360°C (680°F).[5,20]

Since the fuel cladding is in the sodium, the idea of dissociation pressures cannot be directly applied. However, the concentration of nitrogen in the sodium (barring large amounts of calcium, and subsequent Ca-N_2-stainless steel reactions) is less than the dissociation pressures of the chromium nitrides. Therefore, it is doubtful whether nitrides will form.

Much more likely to occur is carburizing of the fuel cladding (and other stainless steel components below the sodium-argon interface).[36] The RDT Sodium Purchase Specification sets a maximum carbon content of 30 wppm in the sodium.[26] The solubility of carbon in sodium at service temperatures is less than 1 wppm, so most of it is in the form of graphite in a colloidal suspension. The rest is in the form of sodium-carbon compounds. Carbon can also be picked up from CO and hydrocarbon impurities (mainly CH_4) in the cover gas atmosphere.[24,36] Since the level of the hydrocarbons is low, there should be little carbon pickup from them.[36] CO is unstable below 700°C, so very little should be present for reaction with the sodium. Carbon transfer is heavily dependent on the concentration of oxygen in the sodium; reducing the oxygen level reduces the transport of carbon.

A surface carbon content of 0.5 wt. % in stainless steel will have negligible effects on mechanical properties.[36] Using this as a basis, 1200°F sodium containing 20-wppm carbon would be in equilibrium with stainless steel having a surface carbon content of 0.5 wt. % (see Figure 3). At lower temperatures, steel is less susceptible to carburizing. These figures assume a constant replenishment of the carbon in the sodium lost to the stainless steel. However, the only sources of carbon are the cover gas impurities, and they probably will not replace the carbon in the sodium as fast as it is lost to the stainless steel.

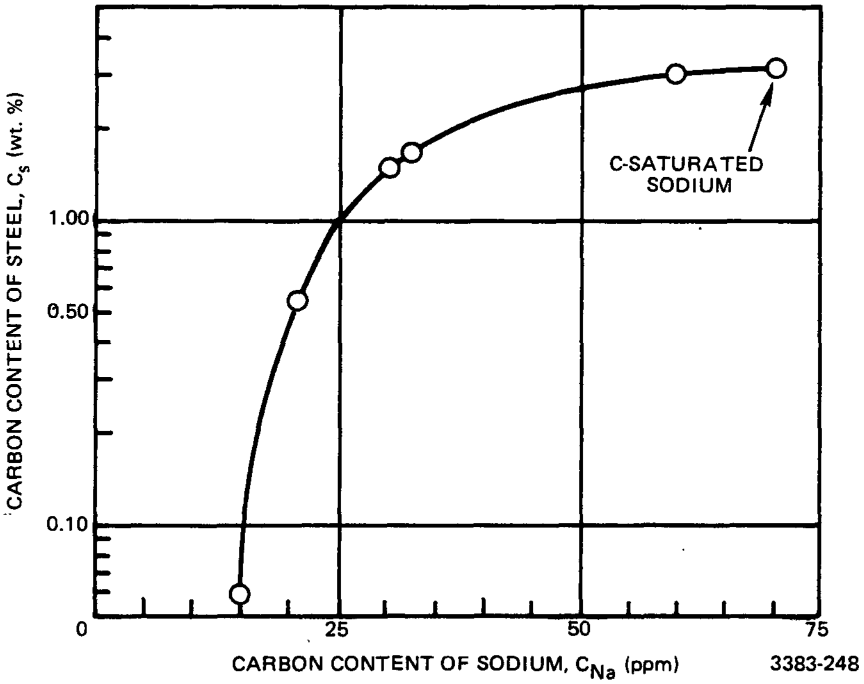

Figure 3. Equilibrium Carbon Concentrations of Sodium and Type 304 Stainless Steel at 1200°F

AI work shows[37] that carbide precipitation does not occur until carbon content is greater than 0.31 wt. %. These carbides are of the type $(Fe,Cr)_7C_3$, containing 75% Cr.

According to these reports, some carburization will occur at 1200°F if the carbon content of the sodium is greater than 15 to 18 wppm. However, the carburization of the fuel cladding should be slight, and problems should not occur.

Other components below the sodium-argon interface will also be subject to carburizing. But since they will be at lower temperatures, they will undergo less carburizing than the fuel cladding.

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7.0 CONCLUSIONS

1. Reactor components. Some nitride formation will occur at and above the sodium-argon interface under service conditions. However, due primarily to the partial pressures of nitrogen involved, the depth of the nitride case formed in 30 years will be small (<35 mils). This will not significantly affect material properties.

2. Fuel cladding. Little or no nitriding will occur in the fuel cladding and components in the sodium due to the extremely small amount of nitrogen in the sodium. A much more likely possibility is the carburization of the cladding caused by the carbon originally in the sodium. However, the amount of carburizing can be controlled by regulating the amount of carbon and oxygen in the sodium, and no problems should occur.

3. A nitrogen content of ≤ 1 vol % is recommended for the primary system cover gases. This level of nitrogen in the argon will limit nitride case depths to less than 0.035 in. (35 mils) in 30 years.

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