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Carbon Transfer in Sodium System Under Thermal Cycling-Possibility of ‘‘Metal Dusting’’

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High temperature sodium systems encounter thermal cycling during operation. Under this condition, the behavior of carbon dissolved in sodium needs special attention. Carbon chemistry of sodium is very complex because of the existence of multiple carbon-bearing species. In addition, carbon exists both in the dissolved (‘‘active’’) and undissolved (‘‘inactive’’) forms. Under thermal cycling conditions, as the temperature is lowered, carbon may precipitate as sodium acetylide. The equilibrium carbon activity imparted by this species is high enough to cause the precipitation of iron carbide (Fe_3C) in ferrous alloys. The Fe_3C may be destabilized at lower activities of carbon in the environment (when the temperature is increased) and may decompose to a fine dispersion of metal and graphite. This phenomenon of ‘‘metal dusting’’ is extremely detrimental to the components of sodium systems. The article analyses the possibility of ‘‘metal dusting’’ in an operating sodium system.

Keywords carbon activity, high temperature sodium, iron carbide, metal dusting, sodium acetylide, thermal cycling

1. Introduction

Liquid sodium has been widely recognized as an efficient coolant due to its favorable thermo-physical properties. Coupled with the good neutronic properties it finds application as coolant in fast breeder nuclear reactors. Ferrous alloys of austenitic and ferritic steel grades are normally employed as construction material in sodium systems because of the favorable compatibility at high temperature. However, sodium possesses the capacity to dissolve the constituents of the steel and participate in a mass transfer phenomenon (Ref 1-10). The situation of corrosion becomes even more complex when non-metals such as carbon, hydrogen, and oxygen are present in sodium. These non-metals, especially oxygen can react with the metallic constituents of the steel and liquid sodium, promoting the generation of multi-component compounds (Ref 11, 12). Thus, the corrosion and mass transfer of elements would be enhanced particularly due to interaction with non-metals. This phenomenon has been comprehensively reviewed by the author in an earlier publication (Ref 13) and by other pioneering investigators (Ref 3, 5).

Both oxygen and hydrogen can be easily removed from the sodium by cold-trapping. The concentration of these elements can, thus, be maintained at a desired minimum. On the other hand, the behavior of carbon is quite complex. It exists both in dissolved form and as undissolved suspension. The rate and influencing factors with which the undissolved suspension gets converted into the dissolved form (‘‘active’’ carbon) is complex. Moreover, carbon is an important constituent of both austenitic and ferritic grade steels and any imbalance in the optimum concentration caused through transport would adversely influence the mechanical behavior. Removal of carbon from sodium requires the adoption of hot trapping techniques and even then the transport problem is seldom solved. Unlike in the case of oxygen and hydrogen, it is very difficult to specify an acceptable carbon level in sodium. Too low thermodynamic activity of carbon in the sodium is equally dangerous as too high carbon activity. In sodium systems the activity of carbon in sodium is influenced and even controlled by the structural material.

The following description is a thermodynamic assessment based on the established facts on carbon behavior and the operational philosophy of sodium systems. In a thermal cycling environment (normal operation and shut down conditions, when typically the temperature cycles between 823 and 473 K), the carbon activity may reach very high values to promote ‘‘metal dusting’’ which will seriously limit the life of sodium system components.

2. Dissolution of Carbon in Sodium

Even though carbon is readily transported through sodium, the dissolution of graphite when added to it is doubtful. Luner et al. (Ref 14) employed an isotope labeling method to study the solubility of graphite in sodium. They reported a value as low as 0.005 ppm at 723 K when graphite was equilibrated for 3000 h. The very high value of solubility reported by earlier workers was attributed by Luner et al. to the existence of carbon as fine insoluble suspension. Borgstedt and Pillai (Ref 15) observed very poor rate of dissolution of high purity charcoal (medical grade) even in sodium containing significant concentration of hydrogen. The incapacity of sodium to dissolve graphite is on account of the high heat of dissociation

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of the C-C bond in graphite. The dissociation energy of C-C bond in graphite (544 kJ/mol C) is higher than the heat of solution of carbon in sodium (108.5 kJ/mol C), estimated from the solubility expression of Thompson (Ref 13, 16). Realizing this situation, the other investigators employed an indirect method to dissolve carbon. They have employed carbon dissolved in nickel (Ref 17, 18) or in iron (Ref 19) as the source, where the element is present in a labile form. Carbon from such alloys equilibrated with sodium with favorable kinetics. Based on the results reported in Ref 17-19 and a thermodynamic technique of Johnson et al. (Ref 20), Thompson (Ref 16) proposed an averaged expression relating the solubility of carbon (S_c) and temperature (T) as

$$\log\ S_C\,(\text{wppm}) = 7.449 - 5858/T, \qquad \text{(Eq 1)}$$

where wppm in the bracket indicates the solubility, which is expressed in weight ppm.

Employing this equation, the solubility at 723 K is calculated to be 0.222. This value is significantly higher than that reported in Ref. 14.

3. Temperature Cycling in Sodium System and the Effect on Carbon Potential

At the highest temperature zone, the carbon activity and the concentration of "active" carbon in sodium would be, essentially, determined by the activity of carbon in the structural material. Naturally, in a carburizing environment the activity of carbon in sodium would be higher. Let us visualize an optimum situation where the activities of carbon in the structural steel and sodium have attained an equilibrium value. The activity of carbon in AISI 316 LN austenitic steel (a_C) is given by the Natesan and Rajendran Pillai equation (Ref 21-23)

$$\begin{aligned}\ln a_C = {} & \ln\,(0.048\%\text{C}) + (0.525 - 300/T)\%\text{C} - 1.845\\ & + 5100/T - (0.021 - 72.4/T)\ \%\ (\text{Ni} + \text{Mn})\\ & + (0.248 - 404/T)\,\%\ (\text{Cr} + \text{Mo})\\ & - (0.0102 - 9.422/T)\ \%\,(\text{Cr} + \text{Mo})^2\\ & + 0.033\,\%\ (\text{Cr} + \text{Mo}) \qquad \text{(Eq 2)}\end{aligned}$$

A typical composition of AISI 316 LN stainless steel (in wt.%) is shown in Table 1.

The carbon activity at 823 K (employing Eq 2 and composition shown in Table 1) is evaluated as 0.007125. When coupled with solubility of carbon (Eq 1) and assuming Henrian behavior, the concentration of carbon in sodium in equilibration with the stainless steel is 0.0153 ppm. If the sodium has been initially carburizing (possessing carbon activity higher than the structural material), the activity and concentration of dissolved carbon would be accordingly higher. When the temperature of the sodium containing 0.0153 ppm carbon is lowered to 632 K saturation occurs (limit of dissolution of graphite). At this solubility limit of graphite a solution of disodium acetylide exists in sodium (Ref 16, 18, 20). The solubility of disodium acetylide is given by (quoted in Ref 20)

Table 1 Composition (mass%) of 316 LN stainless steel (typical)

C	Mn	Si	P	S	Cr	Ni	Mo	N	Cu	Fe
0.03	1.8	0.05	0.035	0.025	17.5	12.25	2.5	0.07	1.0	Rest

$$\log_{10}\ S_{Na_2C_2} = 0.345 - 4289K/T \qquad \text{(Eq 3)}$$

where $S_{Na_2C_2}$ is the solubility expressed in mass of carbon by mass of sodium. When the carbon content is 0.0153 ppm, the solubility limit of disodium acetylide will be attained at 525.6 K and its precipitation is initiated. Thus, in a system with a maximum temperature of 823 K, deposition of sodium acetylide is likely to occur in the down-stream region which is maintained at temperatures lower than 525.6 K. The existence of sodium acetylide in sodium systems have been reported in the literature (Ref 16, 18, 20, 24-28). This would not cause any serious problem in the cold leg as the diffusion coefficient of carbon is very low at this temperature.

The penetration of carbon into the stainless steel matrix is governed by the diffusion coefficient (established by analyzing machined sections of sodium-exposed specimens) given by (Ref 29)

$$D\,(\text{m}^2\,\text{s}^{-1}) = 7.762 \times 10^{-8}\ \exp\,(-15297/T), \qquad \text{(Eq 4)}$$

where T is the temperature in K. By the above expression, it can be estimated that the diffusivity at 823 K is 6.57×10^{-16} m^2 s^{-1}, whereas at 723 and 623 K these values are, respectively, 5.027×10^{-17} and 1.684×10^{-18} m^2 s^{-1}. Thus, a decrease in the temperature by 100 K (from 823 to 723 K) lowers the rate of diffusion by 13 times and decrease of temperature by 200 K (from 823 to 623 K) by 400 times. This implies that even though the precipitation of sodium acetylide occurs at low temperature, it will predominantly be retained at the surface.

The situation is completely different when the component is cycled between two different temperatures. There are situations when a region in the sodium system experiencing 823 K is brought to temperatures lower than 525.6 K. In this case, the higher activity of carbon would promote the precipitation of sodium acetylide (Ref 18, 20). The formation of acetylide is further assisted by dissolution in sodium and the resulting solvation effect (Ref 20). On reheating the system to 823 K, the generated Na_2C_2 may undergo decomposition, reaction with steel matrix or simple leaching by the flow of sodium. The possibility of reaction with constituents of the steel is analyzed. The carbon equilibrium in sodium is given by

$$2\text{Na(l)} + 2\text{C}(s) \Leftrightarrow \text{Na}_2\text{C}_2(\text{s}) \qquad \text{(Eq 5)}$$

The Gibbs energy of formation of sodium acetylide at 823 K is obtained by linear interpolation of data reported by Johnson et al. (Ref 20). It is evaluated to be 21.53 kJ/mol.

The equilibrium constant is given by

$$K = \frac{a_{Na_2C_2}}{a_C^2 \cdot a_{Na}^2} \qquad \text{(Eq 6)}$$

When both sodium and sodium acetylide are present as separate phases, their activities are unity. Then

$$K = \frac{1}{a_C^2} \qquad \text{(Eq 7)}$$

$$\Delta G^{\circ} = -RT\ln K = -RT\ln\frac{1}{a_C^2} = 2RT\ln a_C \qquad \text{(Eq 8)}$$

By employing the above data of free energy of formation, the carbon activity is evaluated to be 4.8.

This very high activity of carbon would lead to the generation of carbides of high equilibrium activity. The carbides of chromium are generally encountered in sodium systems (even during normal operation) as the equilibrium activities required for their formation are lower than unity. Generation of these carbides does not pose any significant problem that cannot be surmounted in a system. However, the very high activity of carbon may promote the generation of metastable carbide of formula Fe_3C and the possibility is analyzed.

Consider the reaction

$$3\mathrm{Fe(s)} + \mathrm{C(s)} \Leftrightarrow \mathrm{Fe_3C(s)} \qquad \text{(Eq 9)}$$

The Gibbs energy of formation is given by (Ref 30)

$$\Delta G^{\circ} = 21359.3 - 19.895T\ (\mathrm{J/mol}) \qquad \text{(Eq 10)}$$

At a temperature of 823 K, $\Delta G^{\circ} = 4985.7$ J/mol

The equilibrium constant is given by

$$K = \frac{a_{\mathrm{Fe_3C}}}{a_{\mathrm{Fe}}^3 \cdot a_{\mathrm{C}}} \qquad \text{(Eq 11)}$$

When iron carbide is formed as a separate phase, its activity is unity. Iron is the major constituent of austenitic stainless steel and hence may be expected to show Raoultian behavior. The activity is taken as 0.7 (the mole fraction). Substituting these values in the equation $\Delta G^{\circ} = -RT \ln K$ the equilibrium activity of carbon is evaluated to be 6.04. Hence, the generation of Fe_3C is not expected, when we take into account the bulk composition for the thermodynamic treatment. However, the situation is different in a service environment. The carbon activity associated with the carbon content in the sodium and steel promotes the precipitation of $Cr_{23}C_6$, Cr_7C_3, and Cr_3C_2 at the surface and at grain boundaries. Molybdenum also is a carbide former and would partially replace chromium in forming the above carbides. The precipitation of carbide causes the depletion of chromium and molybdenum at the surface and grain boundaries (Ref 31). This amounts to a corresponding increase in the concentration of iron to an activity of nearly 0.9. The equilibrium carbon activity is calculated by using 0.9 as the activity of iron using Eq 11 and found to be 2.84. Hence, the generation of sodium acetylide would cause the generation of Fe_3C. It is also expected that part of the iron in Fe_3C is replaced by Cr, Ni, and/or Mo which will be formed even at lower activities of carbon. It is to be emphasized that in solid state reactions (governed by diffusion) it is the mixed carbide of the formula M_3C (where M is Fe partially replaced by Cr, Ni, and/or Mo) that is formed rather than the carbide of a single metal. Being stable carbide formers, the interaction of Cr and Mo would promote the formation of the carbide even at lower activities of carbon. The depletion of chromium and molybdenum in a carburizing environment and the consequent increase in activity of iron close to 0.9 leading to the generation of Fe_3C has been reported in the literature (Ref 32).

A measure of protection from "metal dusting" may be provided by an adherent oxide scale (Ref 33). However, in a sodium environment this scale is non-existent because of reaction with sodium and the formation of multi-component compounds (Ref 12).

The generated Fe_3C would decompose during sustained operation at 823 K and forms particles of carbon and metal (reverse of reaction 9, called "metal dusting"). In effect the alloy matrix disintegrates into a mass of metal and carbon powder, thus adversely affecting the mechanical integrity of this region. This phenomenon is reported to be a catastrophic (Ref 34-41) causing the failure of components. The above discussed phenomenon is schematically represented in the Fig. 1.

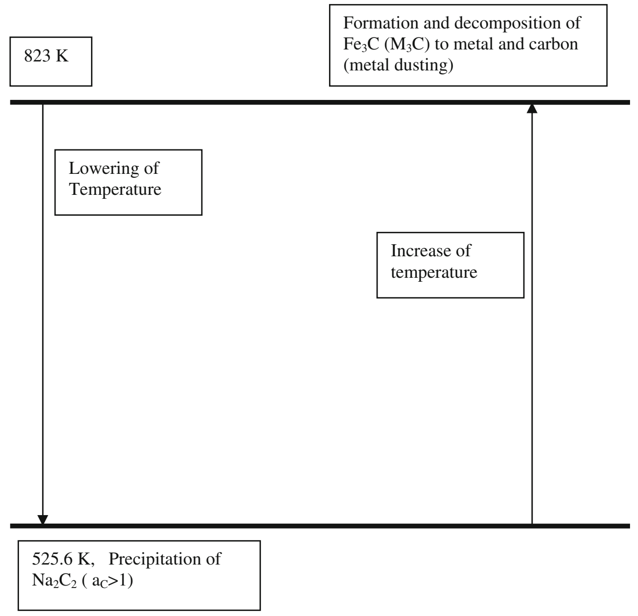

Fig. 1 Schematic representation of metal dusting due to thermal cycling

Table 2 Equilibrium carbon activities of Na_2C_2 and Fe_3C at 673 and 723 K

	Equilibrium carbon activity of:	
Temperature, K	**Na_2C_2**	**Fe_3C**
673	7.25	5.7
723	6.36	4.38

In many cases the activity of carbon in sodium will be higher than that determined by assuming equilibrium with the structural steel. In these cases the generation of sodium acetylide would occur even at temperatures higher than 525.6 K. The equilibrium carbon activity of disodium acetylide and that of Fe_3C (assuming activity of iron as 0.9) at two different temperatures of 673 and 723 K are evaluated and compared in Table 2. Thus, in both cases the generation of Fe_3C is possible resulting in subsequent "metal dusting." If due to some unforeseen circumstances in the sodium system such as entry of oil from the centrifugal pump, carbon activity will be extremely high contributing to accelerated "metal dusting."

4. Conclusion

1. Thermal cycling of sodium components may promote the deposition of sodium acetylide with thermodynamic activity of carbon higher than unity. The high carbon activity promotes the formation of Fe_3C (M_3C with iron

partially replaced by Cr, Mo, or Ni) which further decomposes to cause "metal dusting." Thermal cycling is normally an operational requirement in pool-type fast reactors.

2. Confidence has to be gained by carrying out experiments in sodium system simulating the envisaged temperature cycling. Methods to mitigate "metal dusting" have to be incorporated so that the design life of components (40 years) may be safely achieved. Development of alloys resistant to metal dusting also merits enhanced attention.

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