

Research Article **High Temperature Corrosion of Nickel in NaVO₃-V₂O₅ Melts**

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Received 23 February 2017; Revised 12 May 2017; Accepted 25 May 2017; Published 22 June 2017

Academic Editor: Patrice Berthod

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Many alloys used at high temperature in industrial processes are Ni-based and many others contain it in appreciable quantities, so it is of interest to evaluate the performance of pure nickel in order to determine the behavior of its alloys once the elements responsible for their protection have been depleted due to accelerated corrosion processes in the presence of vanadium-rich molten salts. Due to this, this work presents the study of Ni behavior in NaVO₃-V₂O₅ mixtures at different temperatures. The behavior of pure nickel was determined by both electrochemical and mass loss measurements. The results show that the aggressiveness of the vanadium salts is increased by increasing both the V₂O₅ content and temperature. V₂O₅ addition considerably increases the current densities of the anodic and cathodic reactions. The corrosion process of Ni is modified due to the presence of its corrosion products, and its presence increases the activation energy by at least one order of magnitude. Although nickel shows a high reactivity in vanadium-rich salts, its reaction products are highly stable and protect it from the corrosive medium because the corrosion reactions trap the vanadium and block the migration of nickel ions.

1. Introduction

Many alloys for engineering applications at high temperature are Fe- or Ni-based. Their excellent mechanical properties and corrosion resistance make them useful for manufacturing components of industrial equipment such as industrial gas turbines and other energy conversion systems [1–6]. Corrosion resistance of alloys depends mainly on their chemical composition. In high temperature applications it is essential that the alloys be capable of developing protective oxides such as Cr_2O_3 , Al_2O_3 , or SiO_2 . Chemical and physical characteristics of this type of oxides, such as good adhesion, low gas permeability, high thermodynamic, and low diffusion coefficients, provide excellent protection to alloys in gaseous environments mainly [7–9].

However, the protection afforded by this type of oxide can be limited when molten phases are involved in the corrosion process. In these cases, the accelerated degradation of the materials may occur due to the so-called molten salt corrosion, hot corrosion, or high temperature corrosion. This type of corrosion is characterized by the accelerated degradation of the alloy when covered by a thin layer of molten salt at high temperature in an oxidant environment. In these cases, the integrity of any alloy will be compromised if its protective oxides do not show low solubility in the melt [1, 10, 11].

The solubility of the protective oxides is greater in the presence of vanadium-rich fused salts (sulfate-vanadate type) than in pure Na_2SO_4 ; this is because the vanadate ions increase the acidic fluxing of any oxide [10]. On the other hand in Na_2SO_4 - V_2O_5 mixtures the maximum solubility occurs in the interval 10 to 30% Na_2SO_4 [12]. In many industrial systems where the use of heavy fossil fuels is common, the species causing the catastrophic corrosion of the materials

are complex compounds of vanadium, β and γ sodium vanadates (Na₂O·V₂O₄·5V₂O₅ and 5Na₂O·V₂O₄·11V₂O₅, resp.) being the most corrosive vanadium species [6, 12–14].

When accelerated corrosion decreases the concentration of the element responsible for forming the protective oxide, corrosion will proceed catastrophically if the rest of the elements of the alloy are unable to form stable corrosion products. For example, in most alloys the element responsible for forming a protective oxide is Cr; however, in the presence of acidic vanadium salts it is not capable of forming a stable protective oxide [15].

Therefore, because many Ni-based superalloys show excellent corrosion resistance due to their ability to develop a protective oxide, Cr_2O_3 , this study evaluates the performance of pure nickel in the presence of NaVO₃-V₂O₅ mixtures from 600 to 900°C. This is a model system (few reaction products, NiO and vanadates) in order to determine if this element would be able to provide some protection to the alloy once the element responsible for protection has been depleted.

2. Experimental Procedure

2.1. Corrosive Salts. The corrosive medium employed consisted of different NaVO₃: V₂O₅ ratios, from pure NaVO₃ to pure V_2O_5 , and two intermediates ratios that promote the formation of the highly corrosive complex vanadates, namely, sodium β -vanadyl-vanadate (Na₂O·V₂O₄·5V₂O₅) and sodium γ -vanadyl-vanadate (5Na₂O·V₂O₄·11V₂O₅). In previous studies these complex vanadates have been synthesized from the following ratios: 2NaVO₃:5V₂O₅ and 10NaVO₃:7V₂O₅, respectively [15], where NaVO₃ is the vanadium salt with the highest basicity, V₂O₅ is the compound with the highest acidity, and 2NaVO₃:5V₂O₅ and 10NaVO₃:7V₂O₅ ratios are compounds with intermediate acidity and high oxygen adsorption capacity. Since the NaVO₃ can be synthesized from the Na₂O : V_2O_5 ratio, it is considered that by increasing the molar ratio of V₂O₅ the acidity of the resulting compound increases. For simplicity, hereafter the above-mentioned compounds will be identified according to the following nomenclature: NV is NaVO₃, NV3 is sodium γ -vanadyl-vanadate, NV6 is sodium β -vanadylvanadate, and V is V2O5, where N is Na2O. Its known melting points are 630°C, 627°C, 535°C, and 691°C, respectively [14].

2.2. Electrochemical Measurements. Electrochemical evaluations were performed using a three-electrode arrangement. As pseudo-reference electrode and counter electrode a platinum wire was used. Nickel samples were spot-welded with a Ni20Cr wire in order to get electrical connection. Ni20Cr wire was isolated with a ceramic tube and sealed with refractory cement. The test temperatures selected were 700, 800, and 900°C, which are above the melting point of the salts. The test temperature was measured constantly, using a type K thermocouple and was controlled to 2°C with respect to the test temperature. Before starting the electrochemical measurements, the electrode array is immersed in the molten salt and the open-circuit potential of the working electrode is measured until steady state (\pm 5 mV) is reached; in all cases this was achieved in about 30 minutes. In all experiments, the atmosphere above the melt was static air. Potentiodynamic polarization curves and linear polarization resistance (LPR) measurements were carried out. Potentiodynamic polarization curves were obtained polarizing the working electrode from -300 to 300 mV at scanning rate of 1 mV/s, and the electrochemical parameters (Tafel slopes, $E_{\rm corr}$ and $I_{\rm corr}$) were obtained from the active regions of the corresponding anodic and cathodic zones. LPR measurements were obtained applying a potential of ± 10 mV, and the variation in current intensity associated with that polarization was measured during 24 hr. An ACM Instrument potentiostat, model GillAC, controlled by a personal computer was used for the electrochemical experiments.

2.3. Mass Loss Corrosion Testing. Before mass loss tests, the specimens were cleaned with acetone and dried, subsequently weighed by means of an analytical digital scale with a precision of 0.00001 g, and then packed in the mixture of salts in porcelain crucibles with 500 mg/cm^2 of vanadium salts. The corrosion tests were carried out in electric furnaces in static air during 100 hours at temperatures of 700, 800, and 900°C. Four specimens were exposed to each test condition. After corrosion tests the corrosion rate was measured as mass loss. Three specimens of each condition test were descaled and chemically cleaned according to ASTM G1 standard, and the other specimen was mounted in thermosetting resin in cross section and polished to analyze the subsurface corrosive attack using a Karl Zeiss DSM-950 scanning electronic microscope (SEM) with an X-ray energy dispersive (EDS) analyzer. In short, the specimen descaling was realized mechanically with a cutter in order to remove the coarse and soft layers of products of corrosion; subsequently the heavily adhered corrosion products were removed by chemical cleaning in a solution of 15% hydrochloric acid (v/v)at room temperature with immersion times of 3 minutes, and cleaning by light brushing until the complete removal of the corrosion products (constant weight of the metal).

In all cases high purity Ni (99.9%) was used. Ni specimens were obtained from rectangular bars and cut in samples with dimensions of $10 \times 5 \times 5$ mm. In order to obtain a homogeneous and defined reaction area, specimens were polished with silicon carbide sandpaper to grade 600. Subsequently, the specimens were washed with distilled water and acetone before performing the corrosion tests.

3. Results and Discussion

3.1. Polarization Curves. Figures 1–3 show the potentiodynamic polarization curves for Ni in the different vanadium salts at 700, 800, and 900°C, respectively. It is observed that, in the presence of NV3, NV6, and V, at all test temperatures, the Ni polarization curves were shifted to higher current densities and nobler potentials than those observed in NV. No significant variation in $E_{\rm corr}$ values was observed in the presence of NV3, NV6, and V, at all test temperatures. In all cases, by increasing the vanadium content in the salt an increase in the current densities of the anodic and cathodic



FIGURE 1: Potentiodynamic polarization curves for Ni in NV, NV3, NV6, and V at 700 $^{\circ}$ C.



FIGURE 2: Potentiodynamic polarization curves for Ni in NV, NV3, NV6, and V at 800° C.

branches was observed. This difference is most noticeable as the temperature increases.

The observed behavior is associated with the conductive nature of the vanadium salts, because in the molten state the vanadium salts act as electronic conductors or ionic conductors [16]. The conductive nature of the vanadium salts depends on the Na₂O concentration, V^{5+}/V^{4+} ratio, and oxygen partial pressure, such that by increasing the Na₂O concentration the ionic conduction increases, and by increasing the V^{5+}/V^{4+} ratio and the partial pressure of oxygen, the electronic conduction increases. Because vanadium can coexist in different oxidation states (the most stable oxidation states are +5, +4, and +3), the aggressiveness of the vanadium salts increases by increasing the V^{5+} proportion (higher concentration of V_2O_5) and the partial pressure of oxygen (higher oxidation state of vanadium is favored); on the other hand, its aggressiveness is reduced by



FIGURE 3: Potentiodynamic polarization curves for Ni NV, NV3, NV6, and V at 900 $^\circ\text{C}.$

increasing the concentration of Na_2O , due to an increase in the proportion of V^{+4} and V^{+3} (increase in melt basicity) [11, 17, 18]. Therefore, the presence of these multivalent ions enhanced the charge transport through the melt either by counterdiffusion of multivalent cations or electron hopping [11].

From the current corrosion densities calculated from Figures 1–3, the effect of the NV : V ratio and temperature on the nickel corrosion rate was determined. Figure 4 shows the effect of NV: V ratio and temperature on Ni corrosion rate. Although the corrosiveness of the salts NV3 and NV6 shows little difference, it is clear that the aggressiveness of the melt increases by increasing its acidity (higher proportion of V^{+5}) and temperature, and the contrary happens by increasing its basicity (higher concentration of Na₂O). Similar trends have been observed in studies using chromium [15]. In particular, the NV3 and NV6 compounds have little difference in their V content, and they have been considered as the only vanadium species with the highest oxygen absorbing capacity [12, 19]. The increase in the aggressiveness of the molten salts by increasing the V concentration is congruent with previous reports indicating that the V2O5 catalyzes the oxidation reaction of Ni [20].

3.2. LPR Measurements. Figures 5–7 show the I_{corr} variation (from LPR measurements) with time for Ni in the different vanadium salts from 700 to 900°C. The observed trends are similar to all test temperatures; namely, there is a strong variation in the I_{corr} values in the first two hours of test and subsequently a pseudo-stationary state is reached. This behavior shows the high reactivity of Ni in the vanadium salts and that the corrosion products formed are highly protective. In all cases, the vanadium salts corrosiveness increases with both the melt acidity and temperature, and again, it is observed that the NV3 and NV6 species show little difference in their degree of aggressiveness. Figure 8 shows the appearance of the corrosion front of Ni evaluated in NV3



FIGURE 4: Effect of NV: V ratio (a) and temperature (b), on Ni corrosion rate.



FIGURE 5: $I_{\rm corr}$ variation versus time for Ni in V-rich molten salts at 700°C.

at 800°C. A similar behavior was observed at all temperatures and with the different vanadium salts.

In almost all cases a thin film rich in oxygen and nickel (NiO) onto Ni surface and a thick and dense layer of corrosion products rich in Ni-V-O onto the thin film were observed. Corrosion products showed a defined form of crystals growing either from the Ni surface or from the NiO film towards the melt. On the other hand, the presence of Ni was not observed in the vanadium-rich zone. All this is congruent with the tendency of the LPR values; that is to say, after the initial attack (first hours of immersion) the corrosion products formed a protective barrier that limited the access of the vanadium salts to the Ni surface.

In order to evaluate if there was a change in the corrosion process kinetics between the information obtained from the



FIGURE 6: $I_{\rm corr}$ variation versus time for Ni in V-rich molten salts at 800°C.

polarization curves and LPR measurements, the activation energies were determined based on the $I_{\rm corr}$ values obtained from both tests. In the case of the $I_{\rm corr}$ values obtained from the LPR tests, the average of the last 4 hours of test was considered. The dependence between corrosion rate and temperature can be obtained from the Arrhenius equation:

$$I_{\rm corr} = k \exp\left(-\frac{\Delta E}{RT}\right),$$

$$R \ln\left(I_{\rm corr}\right) = R \ln k - \frac{1}{T}\Delta E,$$
(1)

where ΔE (J mol⁻¹) is the activation energy, k is a constant, and R is the gas constant (8.314472 J/K-mol). Figure 9 shows the $R \ln(I_{corr})$ versus 1/T relationship of the



FIGURE 7: I_{corr} variation versus time for Ni in V-rich molten salts at 900°C.



FIGURE 8: Cross sectional aspect of Ni and element mapping, after LPR measurements in NV3 at 800°C for 24 hours.

 $I_{\rm corr}$ values obtained from the potentiodynamic polarization curves and the LPR measurements. In the first case the information derived from the potentiodynamic polarization curves corresponds to the interaction of the bare Ni surface with the molten salts, and in the second case due to the corrosion process of Ni with corrosion products adhered to its surface (NiO and nickel vanadates). In the case of a bare Ni surface, the activation energies determined were 193, 23, 22, and 21.5 kJ mol⁻¹ for NV, NV3, NV6, and V, respectively. These values show that the Ni corrosion rate increases with the vanadium content of the molten salts and that the corrosiveness of the NV3, NV6, and V is at least one magnitude order greater than that of the NV. In contrast, the activation energy values after 20 hours of corrosion varied between 181 and 202 kJ mol⁻¹ for the different vanadium salts. The corrosiveness of the NV did not change significantly,



FIGURE 9: $R \ln(I_{corr})-1/T$ relationship for Ni evaluated in vanadium salts. (a) Data from the potentiodynamic polarization curves. (b) Data from LPR measurements.



FIGURE 10: (a) Effect of the NV : V ratio and (b) temperature on Ni corrosion rate after 100 hours.

but it is clear that the reactivity of the other vanadium salts decreased by one order of magnitude. The drastic increase in the activation energy values shows that the corrosion products adhering to the Ni surface provide high protection.

3.3. Mass Loss Tests. Figure 10 shows the effect of the different vanadium salts on the corrosion rate of Ni, expressed in terms of weight loss (mg/cm^2) at different temperatures after 100 hours of immersion. From the figures it is observed that, at higher exposure times, the Ni behavior in the different vanadium salts shows the same tendency as observed in the previous tests; namely, the aggressiveness of the molten salts increases with the vanadium content and temperature.

Corrosiveness of NV3 and NV6 was similar, being slightly higher with NV6.

Figure 11 shows the cross sectional images of the Ni samples after being immersed in vanadium salts at 700, 800, and 900°C for 100 hours. In all cases similar characteristics are observed. At all temperatures onto Ni surface a nickel oxide layer was observed (in NV3, NV6, and V at 900°C it is not very evident), and on it a thick layer of corrosion products rich in V, Ni, and O. It is clear that nickel was rapidly corroded by the vanadium salts; however the thick layer of corrosion products formed can act as a barrier against the penetration of the corrosive agent.

Regardless of the molten salt, the X-ray diffraction analysis of the corrosion products showed that these were



FIGURE 11: Micrographs of the cross sections of Ni exposed to NV3 at 600, 700, 800, and 900°C, during 100 hours.

mainly composed of nickel oxide (NiO) and nickel vanadate (Ni₃ V_2O_8); similar observations have been made in other studies [21, 22]. This indicates the high corrosiveness of the vanadium salts because they increase the acidic solubility of

the metallic oxides [10]. According to the chemical composition of the vanadium salts used, the Ni degradation occurred according to the following reactions:

$$2Ni + O_2 \longrightarrow 2NiO$$
 (2)



FIGURE 12: Thermodynamic stability diagrams for Ni-Na-V-O at 900°C.

$$3Ni + 2NaVO_3 + \frac{1}{2}O_2 \longrightarrow Ni_3V_2O_8 + Na_2O$$
 (3)

$$3Ni + V_2O_5 + 2O_2 \longrightarrow Ni_3V_2O_8 + \frac{1}{2}O_2 \qquad (4)$$

$$3NiO + 2NaVO_3 \longrightarrow Ni_3V_2O_8 + Na_2O$$
 (5)

$$3NiO + V_2O_5 \longrightarrow Ni_3V_2O_8$$
 (6)

In the corrosion process by vanadium salts in molten state, the melt acts as n-type semiconductors where it is possible that oxygen diffuses as O^{-2} or VO_3^- , and the transfer of charge probably involves the redox reaction $V^{+5} \leftrightarrow V^{+4}$ [16, 20, 23]. Therefore, any modification to the equilibrium of these processes will affect the corrosiveness of the melt. The performance of a material in a highly corrosive medium depends on the chemical stability of both the metallic elements and their compounds such as oxides and vanadates [22]. Therefore, the observed decrease in the Ni corrosion rate as time elapses (LPR measurements) may be due to the high melting point of the nickel vanadate (1220°C), which is considered a refractory compound [24]. The presence of this compound may have limited the mobility of aggressive species towards the metal surface.

Because the Na₂O reduces the acidity of the molten salts, the increase in the corrosion rate observed by increasing the V concentration is consistent with the reactions mentioned above, since at higher V content the proportion of Na₂O formed is reduced. Based on the above reactions, in addition to the equilibrium, $2NaVO_3 = V_2O_5 + Na_2O$, the phase stability diagram shown in Figure 12 was constructed, according to the procedure indicated elsewhere [25].

According to the diagram, it is observed that, in a wide range of partial oxygen pressures equivalent to the melt-atmosphere interface (≈ 0.21 atm) and to the melt-metal

interface ($\approx 1 \times 10^{-4}$ to 1×10^{-12} atm), if acid vanadium salts (NV3, NV6, and V) are present the only reaction product will be $Ni_3V_2O_8$ and, on the contrary, in low acidity salts (NV) the additional presence of NiO is possible. This is congruent with the micrographs shown in Figure 11. In these it is observed that only in the presence of NV at 900°C a NiO thick layer is evident onto Ni surface. According to other studies the low aggressiveness in NV melts is due to the fact that during its reaction with Ni or NiO, in addition to forming the metallic vanadate, it also generates Na₂O as a byproduct; this further reduces the acidity of the melt and affects the charge transport because the V^{5+} : V^{4+} ratio is modified [11]. In contrast, the reaction of Ni or NiO with V only generates the metallic vanadate without any modification in the acidity of the melt. In the case of the NV3 and NV6 species the decrease in their corrosiveness, compared to that of the V, is due to the presence of NV which reduces the acidity of the mixture. However, its aggressiveness is much greater than that shown by NV, because these complex vanadates in molten state also have the ability to absorb a large amount of oxygen which provides a rapid transport of oxygen to the metal surface [12, 19].

The high initial reactivity of Ni in the presence of vanadium salts has a beneficial effect because it forms a highly stable refractory phase, $Ni_3V_2O_8$, which reduces the corrosiveness of the melt by trapping the vanadium and blocking the migration of nickel ions [3, 23, 26], and the stability of the refractory phase still prevails in the presence of Na_2SO_4 [2]. The results obtained are congruent with other studies where NiO has been proposed as a corrosion inhibitor in vanadium-rich molten salts [2, 3, 24], or Ni-derivatives to remove vanadium compounds [26]. Notwithstanding the above, in the case of nickel-based alloys with a multielement chemical composition the results may be different due to synergistic dissolution reactions between the different oxides developed [11, 27–29].

4. Conclusions

From the results of this work, it can be concluded that Ni shows a high reactivity in vanadium molten salts. An increase in both the acidity and temperature of the melt causes a noticeable increase in the current densities of the anodic and cathodic reactions due to an increase in the transport of charge by the presence of vanadium in multivalent state (V^{5+}) , V⁺⁴). Corrosion products form a dense and protective barrier onto Ni surface which limits the access of the vanadium salts since no presence of Ni into vanadium melt was observed. The corrosion products increase the activation energy of the corrosive process by at least one order of magnitude. The initial reactivity of the nickel forms a highly stable refractory phase that reduces the corrosiveness of the melt by trapping the vanadium and blocking the migration of nickel ions. The corrosiveness of the vanadium salts increased in the order NV < NV3 < NV6 < V. Low corrosiveness of the NV is due to the fact that during the corrosion process the Na₂O is released as a reaction byproduct, and the melt basicity is increased affecting the charge transport and decreasing the $V^{5+}: V^{4+}$ ratio. On the other hand, the high corrosiveness of V is due to the fact that its corrosion reactions only form metallic vanadates which do not affect the melt acidity.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

Financial support from Consejo Nacional de Ciencia y Tecnología (CONACYT, México) (Project 159898) is gratefully acknowledged.

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