

# Correlation between Corrosion Behaviour and Semiconductor Characteristics of Passive Film on Incoloy 825 in Ammonium Chloride Solution

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Received: 8 September 2019 / Accepted: 1 November 2019 / Published: 10 April 2020

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The correlation of the corrosion behaviour and semiconductor characteristics of a passive film on Incoloy 825 in 5%~30% NH<sub>4</sub>Cl solutions was investigated using potentiodynamic polarization scan, Mott-Schottky curves, scanning electron microscope(SEM) and the point defect model(PDM). The results indicate that Incoloy 825 is self-passivated and has a pitting tendency in all four concentrations of NH<sub>4</sub>Cl solution at 50°C. With the increasing NH<sub>4</sub>Cl solution concentration, the quantities of chloride ions absorbed in passive films on Incoloy 825 increase, which can increase the quantities of cation vacancies for the Mott-Schottky reaction, so the quantities of point defects in the passive film on Incoloy 825 increase and the anodic dissolution and pitting of Incoloy 825 becomes easier. Therefore, the passive current density of Incoloy 825 increases, and the pitting potential of Incoloy 825 shifts negatively with the increasing concentration of ammonium chloride.

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**Keywords:** Incoloy 825; corrosion resistance; Mott-Schottky curve; ammonium chloride

## 1. INTRODUCTION

In recent years, with the deterioration of crude oil properties, the contents of nitrides and chlorines have gradually increased in the feeds of catalytic cracking and hydrogenation units. Consequently, the fouling problems of the main fractionators in the catalytic cracking unit and reactor effluent systems in hydrogenation units caused by the deposition of ammonium chloride salts become increasingly serious[1~5]. Water washing is usually employed to eliminate the influence of ammonium

chloride deposition on the process. At the beginning of water washing, ammonium chloride will be dissolved in water to form an ammonium chloride solution with a high concentration, which will cause serious corrosion of the equipment and pipelines made of carbon steels and stainless steels in the units[6~8]. Incoloy 825 has been gradually used in the reaction effluent system of hydrogenation units because of its excellent corrosion resistance and good processing performance[9~10]. Zhu et al.[11] investigated the corrosion behavior of Incoloy 825 in a 5%~20%  $\text{NH}_4\text{Cl}$  solution at 60°C. The results indicate that chloride ions participate in the electrochemical reaction, and the corrosion of Incoloy 825 is mainly characterized by localized corrosion. The pitting sensitivity of Incoloy 825 increases with increasing  $\text{NH}_4\text{Cl}$  solution concentration. Duan et al.[12] investigated the corrosion resistance of Incoloy 825 in a 30%  $\text{NH}_4\text{Cl}$  solution at 80°C. The results show that the velocity has little influence on the corrosion resistance of Incoloy 825 in an ammonium chloride solution, and Incoloy 825 exhibits good corrosion resistance in both static and high velocity conditions.

In addition to the effect of the  $\text{NH}_4\text{Cl}$  solution concentration on the corrosion resistance of Incoloy 825, the semiconductor characteristics of the passive film on Incoloy 825 also needs attention. The Mott-Schottky curve has been widely used to research and characterize the semiconductor characteristics of passive films on nickel-based alloys[13,14]. Although the corrosion behavior of Incoloy 825 in ammonium chloride solution has been investigated by previous authors, the correlation of the corrosion resistance and semiconductor characteristics of Incoloy 825 in  $\text{NH}_4\text{Cl}$  solution is still unknown. However, this is a key to comprehending the corrosion mechanisms of alloys. Therefore, it is valuable to study the correlation of corrosion resistance and semiconductor characteristics of Incoloy 825 in  $\text{NH}_4\text{Cl}$  solutions to clarify the corrosion resistance of Incoloy 825 in  $\text{NH}_4\text{Cl}$  solutions and to provide guidance for the material selection for fractionation systems in catalyst cracking units and reactor effluent system in hydrogenation units. Since the temperatures of the equipment and pipelines of the fractionation system in a catalyst cracking unit and reactor effluent system in a hydrogen unit are mainly near or above 50°C, the corrosion behavior and semiconductor characteristics of Incoloy 825 in ammonium chloride at different concentrations at 50°C were investigated using potentiodynamic polarization curve, Mot-Schottky curve and scanning electron microscope in this paper.

## 2. EXPERIMENTAL

### 2.1 Electrodes and solutions

The chemical composition of the Incoloy 825 alloy investigated in this paper is shown in Table1. The bulk Incoloy 825 was cut into coupons with a size of 10 mm×10 mm×6 mm. First, the coupon was soldered by copper wire. Then, the coupon was encapsulated in epoxy resin, leaving only one of the surfaces exposed as the working surface of the research electrode. The effective working area of the research electrode was approximately 1cm<sup>2</sup>. The working surface of the coupon was abraded step by step by using abrasive paper with 150, 240, 320, 400, 500, 600, 800, 1000, 1200, 1500 and 2000 grades, cleaned with distilled water and acetone, and then dried in air before electrochemical measurements.

**Table 1.** Chemical composition of Incoloy 825 (wt%)

elements	Ni	Fe	Cr	Mo	Cu	Ti	C	S	P	Mn	Si	Al
content	41.25	29.11	22.90	3.21	1.67	0.96	0.017	0.005	0.015	0.61	0.17	0.08

All the electrochemical measurements were performed in aerated ammonium chloride solutions, and the solutions were prepared with analytically pure ammonium chloride and distilled water. The concentrations of the ammonium chloride solutions were 5%, 10%, 20% and 30%, and the pH values of the solutions were 5.44, 5.09, 4.39 and 4.16, respectively. The experimental temperature was  $50 \pm 1^\circ\text{C}$ .

## 2.2 Experimental methods

A potentiostat 273A and a lock-in amplifier 5210 were used to perform the electrochemical experiments, and the software Powersuite was used to record, reserve and analyze the experimental data. The experimental solutions were contained in a standard electrolytic cell K0047, and all the electrochemical measurements were performed with a three-electrode system in which Incoloy 825 was the working electrode, Ag/AgCl electrode was the reference electrode, and a pair of graphite rods was the auxiliary electrode.

The potentiodynamic polarization curves were used to investigate the corrosion behaviour of Incoloy 825. The potential scan rate was  $1 \times 10^{-3}$  V/s, and the potential was scanned from -0.25 V (versus corrosion potential) to the potential at which the polarization current density was approximately  $1000 \mu\text{A}/\text{cm}^2$ .

Mott-Schottky was used to investigate the semiconductor characteristics of Incoloy 825. The potential step rate was  $20 \times 10^{-3}$  V, and the potential was scanned from -0.50 V<sub>Ag/AgCl</sub> to 0.70 V<sub>Ag/AgCl</sub> with a frequency of 1kHz and an amplitude of  $10 \times 10^{-3}$  V. To compare the semiconductor characteristics of passive films on Incoloy 825 at the same film forming potential in ammonium chloride solutions with different concentrations, the coupons were polarized at 0 V<sub>Ag/AgCl</sub> for 7200 s before the Mott-Schottky measurements.

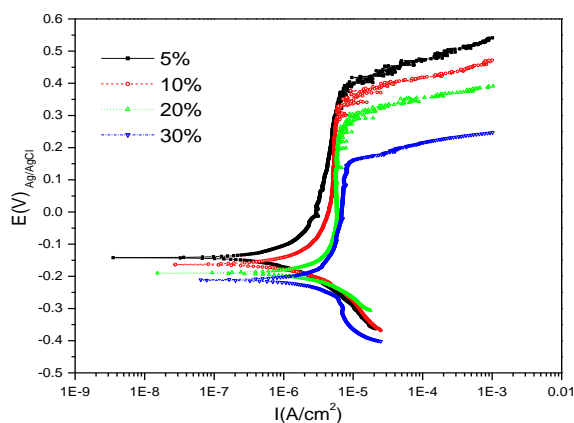
The surface morphologies of the coupons after electrochemical measurements were characterized by a Hitachi S-3400 scanning electron microscope.

## 3. RESULT AND DISCUSSION

### 3.1 Potentiodynamic scanning polarization curves

The potentiodynamic scanning polarization curves of Incoloy 825 in different concentrations of NH<sub>4</sub>Cl solution at 50°C was shown in Fig. 1. It can be seen from the figure that Incoloy 825 is self-passivated in all four concentrations of ammonium chloride solution, and the polarization curve has the characteristics of a typical self-passivation polarization curve. The electrochemical parameters of Incoloy 825 in different concentrations of NH<sub>4</sub>Cl are shown in Table 2. As shown in Table 2, with

increasing NH<sub>4</sub>Cl solution concentration, the corrosion potential ( $E_{corr}$ ) and pitting potential ( $E_{pit}$ ) shift negatively, while the passive current density ( $i_p$ ) increases. The more negative the corrosion potential is, the greater the tendency for corrosion is[15]. The greater the passive current density is, the greater the corrosion rate is[16]. Therefore, with the increasing NH<sub>4</sub>Cl solution concentration, the corrosion tendency of Incoloy 825 increases, and the corrosion resistance of Incoloy 825 decreases. The pitting potential of Incoloy 825 shifts negatively with increasing NH<sub>4</sub>Cl solution concentration, so the pitting sensitivity of Incoloy 825 increases with increasing NH<sub>4</sub>Cl solution concentration. This is consistent with the research results of Zhu et al.[11].



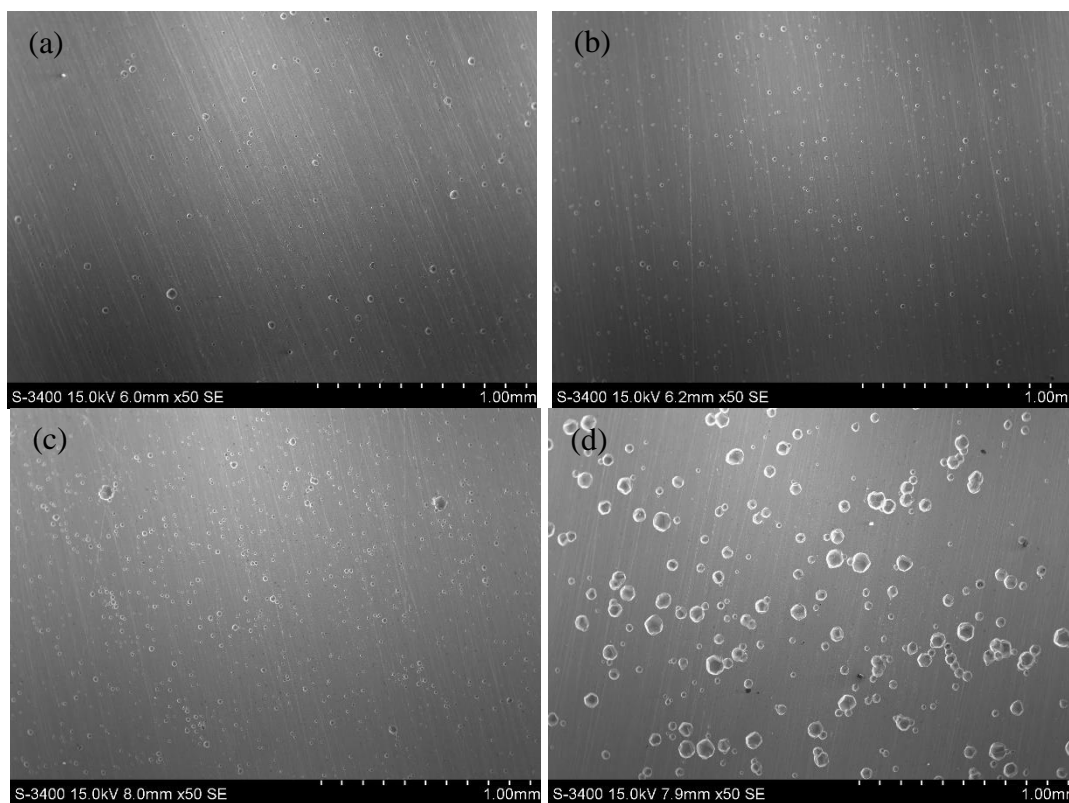
**Figure 1.** Polarization curves at  $1 \times 10^{-3}$  V/s for Incoloy 825 in 5%, 10%, 20% and 30% NH<sub>4</sub>Cl solutions at 50°C

**Table 2.** The electrochemical parameters of Incoloy 825 in NH<sub>4</sub>Cl solutions at 50°C

Concentration of NH <sub>4</sub> Cl (wt%)	5	10	20	30
Corrosion potential, $E_{corr}$ (V)	-0.142	-0.163	-0.190	-0.211
Pitting potential, $E_{pit}$ (V)	0.451	0.410	0.268	0.162
Passive current density, $i_p$ ( $\mu$ A/cm <sup>2</sup> )	4.05	4.68	5.02	6.37

\*: The average polarization current density in the passivation region was taken as the passive current density

Fig.2 shows the surface morphologies of Incoloy 825 in different concentrations of NH<sub>4</sub>Cl when the coupons were polarized to the potential at which the polarization current density was approximately 1000  $\mu$ A/cm<sup>2</sup> ( Incoloy 825 was polarized to 0.541, 0.472, 0.391 and 0.247 V<sub>Ag/AgCl</sub> in 5%, 10%, 20% and 30% ammonium chloride solutions, respectively). As shown in Fig.2, the polishing marks on the surfaces of Incoloy 825 coupons are clearly visible, and the surfaces are covered with nearly round and open pitting pits of different sizes.



**Figure 2.** The surface morphologies of Incoloy 825 after polarization in ammonium chloride solutions with different concentrations at 50°C (a) polarized to 0.541 V<sub>Ag/AgCl</sub> in a 5% ammonium chloride solution, (b) polarized to 0.472 V<sub>Ag/AgCl</sub> in a 10% ammonium chloride solution, (c) polarized to 0.391 V<sub>Ag/AgCl</sub> in a 30% ammonium chloride solution, (d) polarized to 0.247 V<sub>Ag/AgCl</sub> in a 30% ammonium chloride solution

### 3.2 Mott-Schottky curves

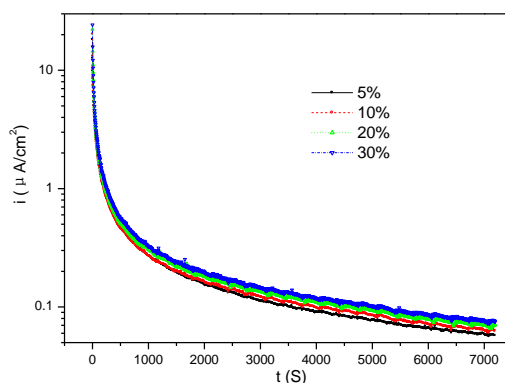
According to Mott-Schottky’s theory[17], the oxide film formed on the metal surface is an intrinsic n-type semiconductor or p-type semiconductor. The relationship between the space charge capacity (C) and applied potential (E) can be expressed by Equ. (1) and Equ. (2).

$$\frac{1}{C^2} = \frac{2}{\epsilon\epsilon_0 e N_D} \left( E - E_{fb} - \frac{kT}{e} \right) \quad \text{n-type (1)}$$

$$\frac{1}{C^2} = -\frac{2}{\epsilon\epsilon_0 e N_A} \left( E - E_{fb} - \frac{kT}{e} \right) \quad \text{p-type (2)}$$

Where C refers to the space charge capacity (F·cm<sup>-2</sup>); E<sub>fb</sub> refers to the flat band potential (V); ε refers to the relative dielectric constant of the passive films; ε<sub>0</sub> refers to the vacuum permittivity (8.85×10<sup>-12</sup>F·m<sup>-1</sup>); K refers to the Boltzman constant (1.38×10<sup>-23</sup>J·K<sup>-1</sup>); e refers to the electron charge (1.6×10<sup>-19</sup>C); T refers to the absolute temperature (K); N<sub>D</sub> refers to the donor density (m<sup>-3</sup>); and N<sub>A</sub> refers to the acceptor density (m<sup>-3</sup>). The dielectric constants of the oxides of the main alloy elements, Fe, Ni and Cr, are in the range of 11.9~14.2[18,19]. For this paper, the value of ε is 13.3[20].

Fig.3 shows the current density-time transients of Incoloy 825 polarized at 0 V<sub>Ag/AgCl</sub> in different concentrations of NH<sub>4</sub>Cl solution. As shown in Fig. 3, the polarization current density of Incoloy 825 decreases sharply at the beginning of the polarization, and then the polarization current density of Incoloy 825 gradually becomes stable as the polarization time increases. Therefore, the polarization current density at 7200 s is approximately regarded as the steady-state current density (*i<sub>ss</sub>*) of Incoloy 825, and the result is shown in Table 3. As shown in Table 3, the steady-state current density of Incoloy 825 increases with the increasing NH<sub>4</sub>Cl solution concentration. Therefore, with increasing NH<sub>4</sub>Cl solution concentration, the dissolution rate of the passive film on Incoloy 825 increases, and the corrosion resistance of Incoloy 825 decreases. This is consistent with the measurement result of the passive current density.

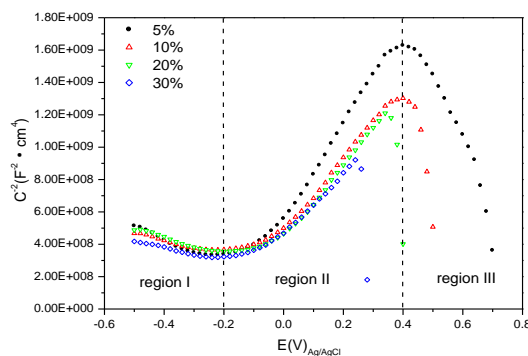


**Figure 3.** Current-time transients of Incoloy 825 in 5%, 10%, 20% and 30% ammonium chloride solutions at 50°C and polarized at 0 V<sub>Ag/AgCl</sub>

**Table 3.** Steady-state currents of Incoloy 825 in ammonium chloride solutions at 50°C and polarized at 0 V<sub>Ag/AgCl</sub>

Concentration (%)	5	10	20	30
<i>i<sub>ss</sub></i> (μA/cm <sup>2</sup> )	0.058	0.063	0.071	0.076

Fig.4 shows the Mott-Schottky curves of Incoloy 825 in NH<sub>4</sub>Cl solutions. As shown in Fig. 4, in all four concentrations of ammonium chloride, when the applied potential *E* is higher than the pitting potential *E<sub>pit</sub>*, the variation amplitude of *C*<sup>-2</sup> with *E* increases sharply. Thus, the jump phenomenon can be observed from the Mott-Schottky curves of Incoloy 825 (the applied potentials of Incoloy 825 in 5%, 10%, 20%, and 30% ammonium chloride solutions were approximately 0.62, 0.44, 0.36 and 0.26V<sub>Ag/AgCl</sub>, respectively).



**Figure 4.** The Mott-Schottky curves of Incoloy 825 in 5%, 10%, 20% and 30% ammonium chloride solutions at 50°C and polarized at 0 V<sub>Ag/AgCl</sub>

According to the variation trend of  $C^{-2}$  with  $E$  in Fig. 4, the Mott-Schottky curves of Incoloy 825 in 5% and 10% ammonium chloride solutions can be divided into three regions, and the Mott-Schottky curves of Incoloy 825 in 20% and 30% ammonium chloride solutions can be divided into two regions. In region I (-0.50~-0.20 V<sub>Ag/AgCl</sub>), the slope of the Mott-Schottky curve is negative, which means that the passive film on Incoloy 825 has a p-type semiconductor characteristic. In region II (-0.22~0.40 V<sub>Ag/AgCl</sub>), the slope of the Mott-Schottky curve is positive, which means that the passive film on Incoloy 825 has a n-type semiconductor characteristic. In region III ( $\geq 0.42$  V<sub>Ag/AgCl</sub>), the slope of the Mott-Schottky curve is negative, which means that the passive film on Incoloy 825 has a p-type semiconductor characteristic.

According to Equ. (1) and Equ. (2), the acceptor density ( $N_A$ ), donor density ( $N_D$ ) and flat band potential ( $E_{fb}$ ) were calculated and are shown in Table 4. As shown in Table 4, with increasing NH<sub>4</sub>Cl solution concentration, the  $N_A$  of the passive film on Incoloy 825 increases in region I, and the  $N_D$  of the passive film on Incoloy 825 increases in region II, while the  $E_{fb}$  of the passive film on Incoloy 825 decreases in both regions.

**Table 4.**  $N_D$ ,  $N_A$  and  $E_{fb}$  of Incoloy 825 in ammonium chloride solutions at 50°C and polarized at 0 V<sub>Ag/AgCl</sub>

Concentration (%)	Potential region	Slope	$N_D/N_A$ (m <sup>-3</sup> )	Intercept	$E_{fb}$ (V)
5	I	$-8.99 \times 10^8$	$1.05 \times 10^{20}$	$4.03 \times 10^8$	0.420
	II	$2.91 \times 10^9$	$3.26 \times 10^{19}$	$5.64 \times 10^8$	-0.222
10	I	$-5.99 \times 10^8$	$1.57 \times 10^{20}$	$2.46 \times 10^8$	0.383
	II	$2.27 \times 10^9$	$4.15 \times 10^{19}$	$4.77 \times 10^8$	-0.238
20	I	$-5.70 \times 10^8$	$1.65 \times 10^{20}$	$2.23 \times 10^8$	0.363
	II	$2.23 \times 10^9$	$4.22 \times 10^{19}$	$4.69 \times 10^8$	-0.238
30	I	$-4.96 \times 10^8$	$1.90 \times 10^{20}$	$1.83 \times 10^8$	0.341
	II	$1.88 \times 10^9$	$5.01 \times 10^{19}$	$4.59 \times 10^8$	-0.272

The location of defects in passive films is usually where the passive film breaks or pits initiate[21], and the lower the electron donor or acceptor density of a passive film is, the more stable the passive film is[22]. The greater the electron acceptor or donor density of a the passive film is, the more vulnerable the passive film is to destruction[23]. The electron donor density and acceptor density of Incoloy 825 increase with the increasing  $\text{NH}_4\text{Cl}$  solution concentration. Therefore, in region I and in region II, the stabilities of the passive films on Incoloy 825 in ammonium chloride solution decrease with the increasing  $\text{NH}_4\text{Cl}$  solution concentration.

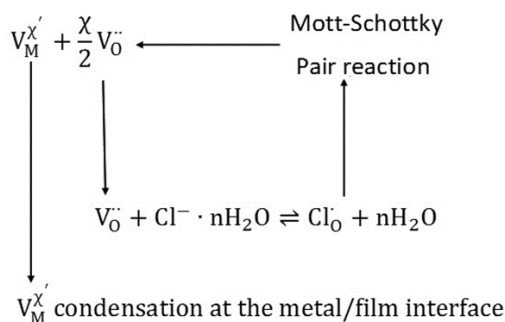
The Nerstian equation of a semiconductor flat band potential can be expressed as shown in Eq. (3)[24].

$$E_{fb} = -E_F^0/q + \Delta\phi_H \quad (3)$$

where  $E_F^0/q$  refers to the potential drop of the space charger layer; and  $\Delta\phi_H$  refers to the potential difference between the electrode surface and the outer Helmholtz layer. Because the value of  $E_F^0/q$  at the flat band is zero, the change of the flat band potential reflects the change in the potential distribution in Helmholtz layer[24]. The adsorption of anions on the surface of passive film will increase the quantities of negative charges on the surface of the passive film and lead to a decrease in  $\Delta\phi_H$ , which can cause flat band potential to move in the negative direction[25]. With increasing  $\text{NH}_4\text{Cl}$  solution concentration, the flat band potential of the passive film on Incoloy 825 shifts negatively in region I and region II. Therefore, with increasing  $\text{NH}_4\text{Cl}$  solution concentration, the quantities of anions adsorbed on the passive film increase in region I and region II.

On the basis of the point defect model (PDM)[26], the transmission of ions through a passive film occurs by vacancy motion. During film growth, cation vacancies are generated at the passive film/solution interface but are eliminated at the metal/passive film interface. Similarly, anion vacancies are generated at the metal/passive film interface but are eliminated at the passive film/solution interface. Therefore, the cation vacancy migrates from the passive film/solution interface to the metal/passive film interface, while the anion vacancy migrates from the metal/passive interface to the passive film/solution interface. The chloride ions in the solution can be absorbed by the oxygen vacancies at the passive film/solution interface. When a chloride ion is absorbed by a surface oxygen vacancy, the system responds to the loss of the oxygen vacancy and has a positive charge. This can result in the formation of oxygen vacancy/cation vacancy pairs by a Mott-Schottky pair reaction. The newly produced oxygen vacancy can in turn react with other chloride ions at the passive film/solution interface to produce more oxygen and cation vacancies, as shown in Fig. 6[25,26]. Therefore, the generation of cation vacancies is autocatalytic. If the annihilation reaction of cation ions is unable to consume the cation vacancies transferring at the metal/passive film interface, the excessive cation vacancies will accumulate and result in local stripping of the film from the alloy substrate. Therefore, if the local tensile stress is high enough and/or the film is dissolved locally, the barrier layer will break, resulting in the formation of a pit.





**Figure 5.** The reaction of an oxygen vacancy and chloride ion in the point defect model ( $V_M^{x'}$ , cation vacancy;  $Cl_O^{\bullet}$ , chloride ion in anion site;  $V_O^{..}$ , oxygen vacancy)

As mentioned above, with the increasing  $NH_4Cl$  solution concentration, the quantities of anions absorbed on the surface of the passive film on Incoloy 825 increase in region I and region II. The anions in the  $NH_4Cl$  solution are mainly chloride ions. Therefore, with the increasing  $NH_4Cl$  solution concentration, the quantities of chloride ions absorbed on the surface of the passive film on Incoloy 825 increase in region I and region II, and the Mott-Schottky reaction in Fig.5 is promoted, which can result in an increase in the quantities of cation vacancies. Consequently, the concentrations of excessive cation vacancies in the passive film increase, and the quantities of cation vacancies accumulated at the metal/passive film interface increase. Therefore, with the increasing  $NH_4Cl$  solution concentration, the stability of the passive film on Incoloy 825 decreases, the polarization current density and the passive current density of Incoloy 825 increase, and the sensitivity for pitting increases.

In summary, the PDM and the parameters of Mott-Schottky curves can be employed to interpret the corrosion behavior of Incoloy 825 in different concentrations of  $NH_4Cl$  solution. With the increasing  $NH_4Cl$  solution concentration, the quantities of chloride ions absorbed in passive films on Incoloy 825 increase, which can increase the quantities of cation vacancies by the Mott-Schottky reaction, so the quantities of point defects in the passive film on Incoloy 825 increase and the anodic dissolution and pitting of Incoloy 825 occur more easily. Therefore, the passive current density of Incoloy 825 increases, and the pitting potential of Incoloy 825 shifts negatively with the increasing concentration of the ammonium chloride solution.

#### 4. CONCLUSIONS

(1) Incoloy 825 is self-passivated and has pitting tendency in 5%~30%  $NH_4Cl$  solution at 50°C. With increasing  $NH_4Cl$  solution concentration, the corrosion tendency of Incoloy 825 increases, the stability of passive film on Incoloy 825 decreases, the corrosion resistance of Incoloy 825 decreases, and the pitting sensitivity of Incoloy 825 increases.

(2) The Mott-Schottky curves of Incoloy 825 in 5% and 10% ammonium chloride solutions have three regions. In region I (-0.50~-0.20  $V_{Ag/AgCl}$ ), the passive film on Incoloy 825 has a p-type semiconductor characteristic; in region II (-0.22~0.40  $V_{Ag/AgCl}$ ), the passive film on Incoloy 825 has a n-type semiconductor characteristic; in region III ( $\geq 0.42 V_{Ag/AgCl}$ ), the passive film on Incoloy 825 has a p-type semiconductor characteristic. The Mott-Schottky curves of Incoloy 825 in 20% and 30%

ammonium chloride solutions have only two regions. In region I (-0.50~-0.20 V<sub>Ag/AgCl</sub>), the passive film on Incoloy 825 has a p-type semiconductor characteristic; In region II ( $\geq -0.22\sim-0.40$  V<sub>Ag/AgCl</sub>), the passive film on Incoloy 825 has a n-type semiconductor characteristic. With the increasing NH<sub>4</sub>Cl solution concentration, the steady state current density, acceptor density and donor density of the passive film on Incoloy 825 increase, while the flat band potential of the passive film on Incoloy 825 decreases.

(3) The PDM and the parameters of the Mott-Schottky curve can be employed to interpret the corrosion behavior of Incoloy 825 in different concentrations of NH<sub>4</sub>Cl solution. With the increasing NH<sub>4</sub>Cl solution concentration, the quantities of chloride ions absorbed in passive films on Incoloy 825 increase, which can increase the quantities of cation vacancies by the Mott-Schottky reaction, so the quantities of point defects in the passive film on Incoloy 825 increase and the anodic dissolution and pitting of Incoloy 825 occur more easily. Therefore, the passive current density of Incoloy 825 increases, and the pitting potential of Incoloy 825 shifts negatively with increasing NH<sub>4</sub>Cl solution concentration.

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