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Surface Coating by Ni and Ni/Si Co-deposition for Carbonaceous Steel Burners Protection from Corrosion at High Temperature

Pierre Jean-Marie Richard Dablé[a,*], **Désiré Martial Koutouan Abro**[b]

[a]*Institut National Polytechnique Félix HOUPHOUET BOINGNY-Yamoussoukro RCI – Départements de Génie Mécanique et Energétique – Laboratoire de Thermodynamique - Traitement et Sciences des Surfaces ; et Interfaces – Ingénierie et Physicochimie des Procédés - Mécanique et Matériaux; L2TSIPM.*
[b]*Institut National Polytechnique Félix HOUPHOUET BOINGNY-Yamoussoukro RCI - Laboratoire de Thermodynamique ; Traitement des Surfaces et Interfaces ; Ingénierie et Physicochimie des Procédés - Mécanique et Matériaux -2TSIPM*

***Abstract*:**
The present work has been undertaken to improve resistance to corrosion that carbonaceous steel sheets undergo when used as burner material. Coating by Ni and Co deposition of Ni/Si composite from a nickel electrolytic solution named WATT bath containing 30 to 50 g/L of silicon particles have been performed on carbonaceous steel patterns. Those patterns have than gone through a thermal treatment under burners' conditions. The efficiency of the coatings resisting corrosion has been discussed under the highlight of the quality of the coats, catch of mass and electrical resistance variation, compared to a naked witness pattern that has also supported the same thermal treatment. The results showed improved resistance to corrosion, which were strongly depending on the quality of the coating with preferential oxidation of Si into SiO_2.

***Keywords*:** Corrosion; Co deposition; Weight; Thermal treatment; Electrical resistance.

1. Introduction

In the undeveloped countries, the weakness of industrialization level leads the craftsmen to conceive articles for usual needs. For these purposes, gas burners are elaborated with carbonaceous steel sheet. These articles, because of the material used, don't support corrosion effects in the conditions of such use (Figure 1). Such defects of the articles present many consequences such as risk of accumulating unburned gas that can lead to explosion, a loss of gas due to a bad mixing with air and a cost for the renewal of the burners. Some researches need to be undertaken for ameliorating their resistance to corrosion in order to improve their day time and guaranty the safety of their using.

In the present work, surface treatment by coating has been chosen for investigations. This is motivated by all the works undertaken on metal and alloys corrosion by hot gas due to the technological advances and industrial requests.

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*Corresponding author:

Institut National Polytechnique Félix HOUPHOUET BOINGNY-Yamoussoukro, BP 1093 Yamoussoukro Côte d'Ivoire; pjmardable@yahoo.com / pierre.dable@inphb.ci.

The electrolytic coating of metal has been largely developed both for decorating [1], engineering [2], and to improve burners. For their protection against corrosion at high temperature, burners are generally elaborated in cast steel or special steel. Stainless steels based on iron, nickel and containing more than 12% of chromium, or 5% of aluminum per mass, are mostly used for burners' conception. This is to control high temperature corrosion. Under exposition to high temperatures a protective chromium oxide (Cr_2O_3) or alumina (Al_2O_3) oxide film is formed, creating a stable and effective barrier against outer environment. However, the cost of these alloys makes such approach not easily applicable [3].

The electrolytic coating presents the double advantage of being easy to undertake and to improve the properties of the surface treated [4]. Nevertheless, under high temperature situation, the immunity of the surface treated is not guarantee.

The electrodeposited composite coats of nickel with inert particles have been developed to reduce corrosion by hot gas phases [5-7]. These studies have pulled out dry anti corrosion properties and mechanical one to be strongly relied to the level of particles embedded in the metallic matrix. The basic concept consists in the embedment of micro or nano-sized solids particles from a suspension into a metallic matrix by electro deposition. Thus novel materials with better mechanical properties (i.e. hardness, tensile strength, wear resistance), corrosion resistance or lubrication effects can be easily developed [8]. The Nickel-Silicon carbide coatings have been used by Ostermann [9] to improve the wear-resistant in the WANKEL engine.

Many works have concerned the experimental factors susceptible to govern the uniformity and the quality of the composite deposit that's to say the chemical state of the electrolyte, the stirring of the bath, the density of the current applied, the pH of the solution, the kind of particle and the effect of surfactants [10-12].

According to Guglielmi [13], the codeposition takes place in two steps of adsorption. This model has permitted the prediction of mass percentage of particles embedded for several systems under the condition of varying only the applied current density or the particles' content.

Nowak *et al.* [14], has proposed a probability law of particles' embedded. A fraction k/K of particles is being effectively embedded by contact with the growing metal phase. On another hand, Celis [15], have tried an explanation of the bad silica particles embedding in metallic nickel matrix compared to SiC particles one by using the Electrochemical Impedance Spectroscopy technique (E.I.S.) Addition of a surfactant such as CTAB (Ethyl Methyl Ammonium Bromide) in the Ni/SiC electrolytic bath increases the level of particles in the coat [16].

Dablé *et al.* [17] in a hydrodynamic study using a jet cell, have pulled out that the embedding of particles depends on the flow mode. The turbulent flow appears to favor better particles embedding. Particles are adhered to the electrode surface and embedded by the growing metal phase. But for higher Reynolds numbers, the high speed of the flow, make them bounce at the surface and return into the electrolyte leading to a weak level of embedding.

Despite all those results, compiling optimal values of parameters and conditions don't lead to a perfect composite deposit. Many works have been developed either to determine conditions that insure a perfect coat, or to prove the efficiency of a coat to resist to dry corrosion process. The present work is undertaken to improve resistance to corrosion at high temperature for carbonaceous steel that is not formerly destined to be used under burner conditions.

Fig. 1. Damaged burner.

2. Material and method

Patterns of 8cm^2 have been cut from carbonaceous steel sheet and polished with abrasive papers of P60 and P600. They have than been cleaned by plunging into an oxalic acid 0.5 mol/L solution at 50°C during five minutes. This treatment was done to insure a perfect surface without any oxide. The patterns were weighted and the initial mass m_i determined. All weight measurements were undertaken with a SATORIUS BP 161S weight machine.

The coating has been realized in an electrochemical cell constituted by a glass container of 200ml where the electrodes are plunged into the electrolyte. The working electrode is constituted by the pattern to cathode polarization. The counter electrode is constituted by a stick of Ni elaborated by fritting pure nickel powder at 1200°C. The reference electrode is the calomel saturated one. The electrodes threesome is linked to a potentiostat-galvanostat D1048 monitored by the AUTOLAB software (Figure 2). The electrolyte used is a WATT bath of an industrial production of DALIC patent elaborated for nickel plating. Table 1 gives the composition of this bath. Three solutions containing 0; 30 and 50 g/L of silicon have been elaborated.

Fig. 2. Electro deposition apparatus.

Table 1
Composition of the electrolyte.

Reagent	Content (g/L)
$NiSO_4, 6H_2O$	250
$NiCl_2, 6H_2O$	90
H_3BO_3	30

The electrical deposition conditions were determined after a volta-amperometric sweep of Ni reduction from the particle free electrolyte presented (Figure 2). The registered graph shows a cathode pic at -0.9V; very closed to the value used to realize thin nickel deposit on different substrates [18]. This value was so retained to realize the deposits. Each surface of the patterns was present to coating during 10 minutes and the solution was magnetically stirred at 700 rpm.

After deposition, the patterns are weighted again to determine the final weight m_f. The deposit weight m_d is so determined according to Eq. (1); m_i being the mass of the pattern before the deposit:

$$m_d = m_f - m_i \quad (1)$$

$$\Delta m = m_j - m_f \quad (2)$$

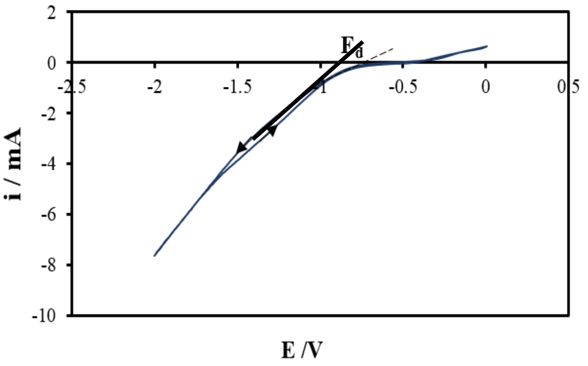

Fig. 3. The cyclic voltammetry registration for nickel reduction obtained on carbonaceous steel.

The patterns so characterized and one, not coated, serving as witness have undergone the thermal treatment under burner conditions. They were exposed to the blue flame of a burner fed with butane gas (Figure 4). The exposure time was fixed to 15 minutes per day for each pattern and exposed after to atmosphere. They were weighted again to determine the new weight m_j after exposure to atmosphere. The mass variation after each treatment is calculated with Eq. (2).

After the thermal treatment and exposure to atmosphere, the patterns have undergone an electrical test to determine their resistance variation. For this purpose, each pattern was introduced in a potentiometric circuit in with a fixe circulating current I (Figure 5). From the tension V at the bounds of the pattern and an applied constant current I, the application of the OHM law leads to calculation of the pattern resistance:

$$R = \frac{V}{I} \quad (3)$$

Fig. 4. Thermal treatment disposal.

The potentiometric circuit was constituted of a tension generator PROMAX FAC- 363B, and the resistance of the pattern is determined with a multimeter PROMAX PD-986.

The techniques applied, permit to characterize the quality of the deposit from exploitation of data gotten by the Eq. (1). Those gotten by Eq. (2), highlight the changes intervening after thermal treatment and exposure to atmosphere. The resistance variation permits the understanding of what happening at the electronic level in the material.

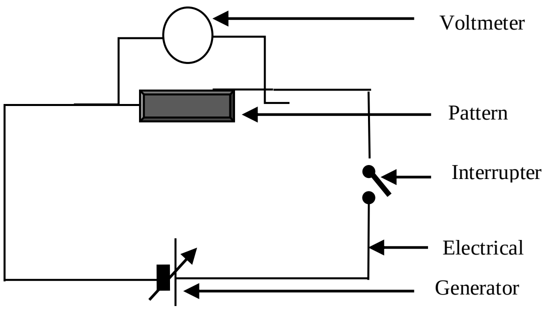

Fig.5. Resistance measurement apparatus.

3. Results and discussion

3.1. Quality of the coats

Eq. (1) applied to the experimental values gives data presented in Table 2.

It appears that; with the electrolyte solution containing 30g/L of silicon, under the same condition the deposit is more than twice important compared to the one coated with the particle free one. This shows out that embedding of particles has occurred. If one supposes that the metal deposition is not perturbed by particles in solution, the mass of particle embedded should be 0.074 g.

With the electrolyte solution containing 50g/L of silicon, the deposit is practically from half less than the one with the particle free electrolyte. This shows that the particles content perturbs the metal deposition.

Table 2
Values of weight of coat deposited on the patterns form each electrolyte solution.

Electrolyte	m_d value (g)
Ni^{2+}	0.0673
Ni^{2+} - Si 30g/L	0.1413
Ni^{2+} - Si 50g/L	0.0342

Under tension, metallic ions support an electric force that drives them to the cathode for their reduction. This force is expressed by the following relation:

$$F_{j;el} = z.F.E \tag{4}$$

With $F_{j,el}$ the electric force supported by a mole of metallic ions j, z the electric charge of the ion, F the Faraday constant and E the tension applied. This force is the one that governs migration of the nickel ions in the particles free electrolyte solution and is thereby proportional to the deposit weight.

In the case of deposition with the electrolyte containing 50 g/L silicon, this electric force is so weaker. This means that presence of 50 g/L of Si particles induces friction forces which are opposite to metal ions migration. This force is expressed by:

$$F_{j,fr} = -\left(\frac{1}{U_j}\right).V_j \tag{5}$$

With U_j the absolute mobility of the ion and V_j, its migration speed. Presence of particles may so reduce the mobility of the ions; as the friction force are becoming important. In a stationary state, the resultant of both forces is null. Regarding this, mobility of metallic ions in the 30 g/L of Si electrolyte may be less than the absolute one, because of some friction forces surely less than in the 50 g/L electrolyte solution but existing anyway.

Thereby, the deposed metal should surely be less than the 0.0673 g obtained with the particle free electrolyte implying that the weight of particles embedded is more than the supposed 0.074g. The deposit obtained with the 30 g/L electrolyte is more homogenous in particles translating a good composite coat.

3.2 Effect of thermal treatment

The experiments have been undertaken with the three patterns coated and a witness one. The weight variation versus duration time in days is presented Figure 6. The treatment has been suspended after Twenty days of treatment and renewed from the thirty eighth day. This to make sure that the weight varying was due to the treatment. It clearly appears that all patterns undergo an increasing of weight. After twenty days of treatment interruption the weights do not vary indicating that the catch of mass was effectively due to the treatment the patterns have undergone. All patterns see their weights increase and only interaction with the environment could be responsible.

During the thermal treatment, butane gas is oxidized according to the following equation:

$$C_4H_{10} + \frac{13}{2}O_2 \leftrightarrows 4CO_2 + 5H_2O \qquad (6)$$

The butane gas and oxygen are respectively, the combustible and the combustive, agents. Under such condition oxygen can’t reach the burner to cause oxidation. On the other hand, the products of reaction in the state of hot gas, move away of the hot point. So, as longer as combustion takes place the burner stays safe and only expands.

The dilatation coefficient of Fe and Ni that are respectively 1.2 10^{-5} m.K^{-1} and 1.3 10^{-5} m.K^{-1} are very close. So, the changes appearing on the patterns can't be due to expansion.

The temperature of the butane blue flame is around 1380°C, so when the thermal treatment is stopped, the metal cools by radiation. The atmosphere around receives the heat so lost making its constituents more reactive by the increasing of their kinetic energy. Under such condition the atmospheric oxygen penetrates the metallic network that still remains dilated, as the retractation stays slower than cooling. An internal oxidation takes place. In the case of the witness pattern, the metal concerned is Fe.

On this pattern, the repetitive thermal treatments lead to the following oxidation reactions:

$$2\ Fe + O_2 \leftrightarrows 2\ FeO \qquad (7)$$

$$6\ FeO + O_2 \leftrightarrows 2\ Fe_3O_4 \qquad (8)$$

$$4\ Fe_3O_4 + O_2 \leftrightarrows 6\ Fe_2O_3 \qquad (9)$$

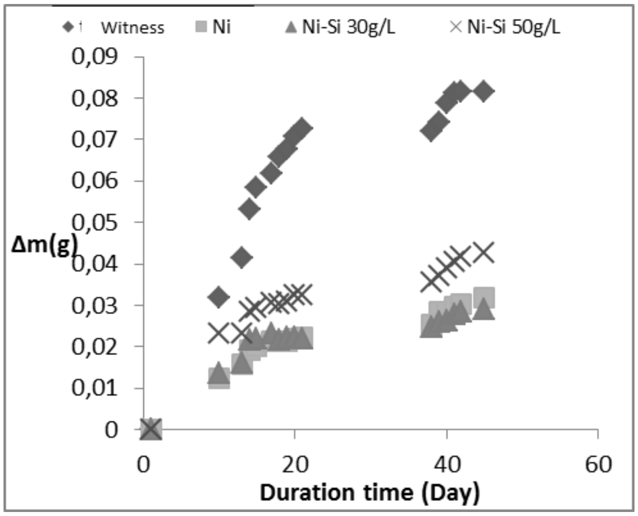

Fig.6. Catch of mass of the patterns after thermal treatment and exposure to atmosphere.

Formation of iron oxides in the metal network causes interfacial tension between the metal and the oxides. In order to reduce the interfacial energy between both phases, the surface in contact must be minimized. Both phases may so separate and this, leads to formation of puffiness that appear on the witness pattern (Figure 7). This state that weakens the material is responsible of degradation that the burner has undergone (Figure 1). The important catch of mass noticed for the witness pattern is due to the internal oxidation.

Concerning the coated patterns, the catch of mass is clearly less even if, the pattern one elaborated with the 50 g/L silicon solution stays higher compared to both others. This may be due to the bad quality of the deposit as has been noticed previously. Oxidation of the metallic phase occurs but less severely than on the witness pattern. Despite the bad quality of the coating, it reduces somewhat the oxygen penetration in the metallic network.

The patterns coated by the Ni-Si 30 g/L of silicon solution and the one without any particles, present the same catch of mass during the twenty first days. The very weak level of this catch of mass and its constancy during this period may be the fact of adsorption of gas molecules or water condensed from atmosphere. This appears for any pattern exposed under the same conditions on that period. The catch of mass on the first twenty days is around 0.02 g. Considering the surface of both sides of the pattern, this represents 1.25 10^{-3} g/cm^2. This value is close to adsorption of water in any area with 100% hygrometry.

Fig.7. Witness pattern aspect after thermal treatment.

Continuing the thermal treatment after the observing period, an increasing of the catch of mass appears for all the coated patterns. Only oxygen present in the atmosphere can react with the material to cause this catch of mass. Considering the nickel coated and the Ni-Si 30 g/L coated patterns, a particular regard to evolution of the catch of mass evolution let suppose a difference of process (Figure 8).

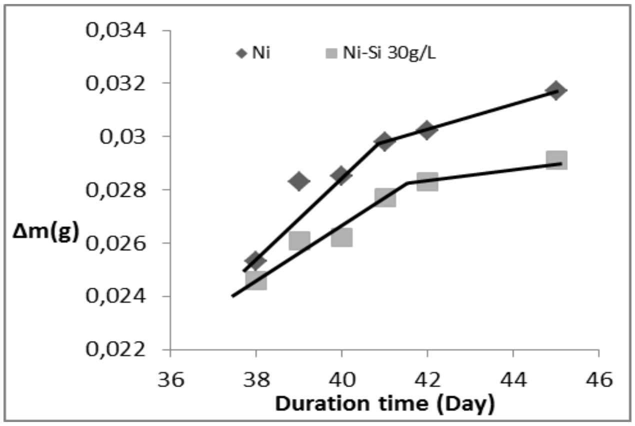

Fig. 8: Zoom on the catch of mass of the patterns coated with nickel and the Ni/Si 30g/L electrolytes from the thirty eights day.

Oxidation takes place during the cooling period. The energy available for the reactions is partly dissipated. So during each cooling a partial oxidation occurs so that the catch of mass increases anytime a thermal treatment is performed.

On Figure 8, it clearly appears that for the nickel coated pattern, the catch of mass increases more than the one coated with the Ni-Si 30g/L solution and this, on the same period.

Under the same conditions, if the catch of mass was due to the same process, the representations would have presented the same slopes. This is not observed here. Thereby, it is very probable that reactions leading to the catch of mass are different for both patterns.

The catch of mass that occurs for the nickel plated pattern should come from nickel oxidation according to Eq. (10):

$$Ni + \frac{1}{2} O_2 \leftrightarrows NiO \qquad (10)$$

The value of the Gibbs energy of the reaction is -188.1 kJ mol^{-1} at 1380 °C [19].

The nickel oxide NiO is ionic. During the process, the oxygen molecule in the network between nickel and dissociates. The oxygen atoms occupy the octahedral sites available. Then, by an internal diffusion of oxygen atoms to migrate from one octahedral site to another, thus promoting a high level of oxidation. This seems to hold for the first forty days where the slope of variation is the greater. After this period, the slope is low, indicating a reduction in oxygen diffusion. The system constituted by the atmosphere, the oxide NiO and metallic nickel tends to balance. The diffusion of oxygen is reduced and the ground terminal adopts a "gentle" slope.

As regards the solution with the coated silicon Ni-Si 30 g / L, the mass of rise remains less than that of nickel coated model. It is the same for the slope in the first forty days. Then after, the ground terminal is practically constant, which makes one suspect a stable weight of the specimen. Since only oxygen can be responsible for the increase in mass under these conditions, the oxidation of silicon occurs:

$$Si + O_2 \leftrightarrows SiO_2 \qquad (11)$$

The Gibbs enthalpy variation of this reaction at the same temperature is of - 627 kJ mol^{-1}. So, Eq. (11) is thermodynamically favored compared to (10). The catch of mass may be consecutive to silicon oxidation into silica. Even if this reaction is thermodynamically; the kinetic conditions due to the steric crowding of SiO_2 tetrahedral structure would reduce the oxygen approach on non-oxidized silicon.

This explains the low catch of mass. On the other hand, the oxidation concerns the silicon particles at the surface of the coat. As silica is a network oxide, there is no oxygen diffusion through the network. So neither the nickel matrix nor the embedded silicon can be oxidized.

The stability of the weight which can be noticed is consecutive to the oxidation of all the particles of silicon present at the surface. The surface then consists of silica. The structure of the oxide network of the silica does not allow the approach of any oxygen molecule. In such a state, there is more oxidation. The oxygen of the silica network pointing outwards would lower the oxygen gradient. This corresponds to what happens with the stainless steel forming Al_2O_3 or Cr_2O_3, which are oxides of the network phase protection against metal corrosion.

3.3 Resistance variation

Figure 9 presents resistance variation of the patterns. Electrical conductivity is due to presence of free electrons named FERMI gas, which stabilizes metallic nucleus forming the network.

When a tension is applied, the FERMI gas electrons are put into movement that creates a variation of electron flux giving a current:

$$I = \frac{dq}{dt} \quad (12)$$

So, the current is proportional to dq; the quantity of electrons put into movement determines the current.

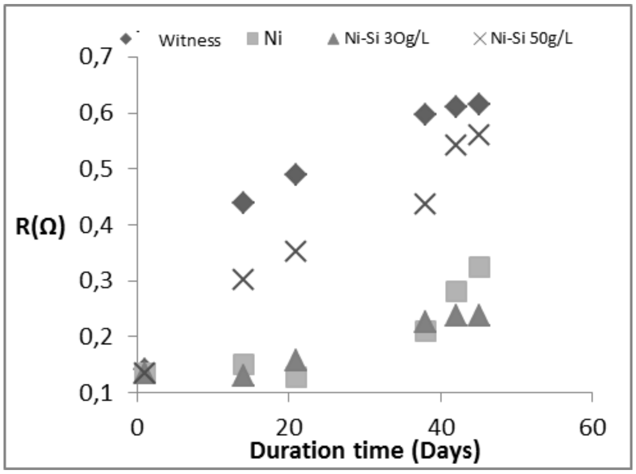

Fig. 9. Resistance variation versus the treatment duration.

Resistance is the opposition that the material presents in order to reduce the current. Considering the Eq. (12), this implies that dq diminishes. A resistance increasing is so consecutive to reduction of electrons in the FERMI gas. This permits understanding of the electronic process supported by the patterns.

Whereas the witness pattern, the molecular oxygen enters the network of iron and dissociates into atoms. The oxygen atom fixed two electrons torn from the Fermi gas to form oxygen anion:

$$1/2O_2 + 2e^- \leftrightarrows O^{2-} \quad (13)$$

Regarding Eqs. (7) to (9), the oxide forming implies a drop of the electrons level in the FERMI gas, inducing an increasing of the resistance. The pattern coated by the Ni-Si 50 g/L solution also presents an increasing resistance. This indicates the internal oxidation of the metallic phase. The bad state of the coating lets discovering the carbonaceous steel phase into which oxygen penetrates and causes the oxidation.

One can notice on Figure 11, the presence of puffiness confirming the same oxidation process with the witness pattern.

The patterns coated by the Ni-Si 30 g/L and Ni present an unvarying resistance value during the first twenty days of thermal treatment. Oxidation seems not to occur on both patterns, confirming the protective effect of the coats. After the first twenty days, the resistances start increasing. Changes so appear inside the material.

Figure 9 shows that the resistance of the Nickel coated pattern starts an exponential increasing while, the pattern coated by Ni-Si 30 g/L remains linear with a low slope and attains a constant level after forty day. The increasing of the resistance may be due to oxidation that reduces conductivity as previously indicated.

But the oxidation process which takes place is obviously different for the two modes. The exponential increase of the resistance indicates the formation of NiO. The ionic character of the oxide promotes the reaction.

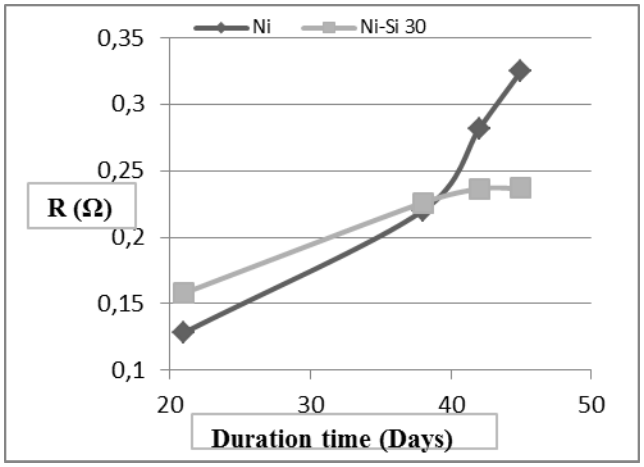

Fig. 10. Zoom on the resistance variation after the twentieth day of treatment.

The change in resistance of the coated structure of 30 g/L solution of Ni-Si is due to oxidation of the silicon particles at the surface of the layer. Silica is an oxide network with covalent bonds. There are no free electrons to ensure conductivity. Therefore, as the resistance is a bulk measurement, it takes into account the resistance of silica that has been formed at the surface. The constancy of the resistance which occurs after 40 days of heat treatment and exposure to the atmosphere indicated that the entire surface of the specimen is coated with silica. In these circumstances, the oxygen in the atmosphere cannot reach the ground; making protected against the effect of high temperature corrosion.

Figure 12 that represents the Nickel coated pattern presents shapes of metal oxide Fe_2O_3 at the surface, confirming oxidation state. Thermal treatment may have damaged the nickel oxide coat exposing the carbonaceous steel. The difference of standard redox tension between Ni and Fe respectively -0.25V/NHE and -0.47V/NHE could induce in the thermal condition, the galvanic corrosion that is supported by Fe. The surface oxidation occurs on the nickel coated pattern while the pattern coated with the Ni-Si 30 g/L solution (Figure 13) remains safe from corrosion. This shows that the Ni-Si 30 g/L coating composite is better protector form corrosion than the nickel coat.

Fig. 11. The Ni-Si 50 g/L pattern coated after the thermal treatment and exposure to atmosphere.

Fig. 12. The Ni coated pattern after the thermal treatment and exposure to atmosphere.

Fig. 13. The Ni-Si 30 g/L coated pattern after forty five days of thermal treatment and exposure to atmosphere.

4. Conclusion

The present work has pulled out much in formations that permit a better and clear understanding of the processes that intervene. The witness pattern undergoes corrosion by internal forming of iron oxides that weaken the material and causes its destruction. The internal oxidation is confirmed by the increasing of the resistance that translates a fall of electron level in the FERMI gas of the metal.

The Ni coated pattern presents a limited day time. In spite of the good protection the Ni is known to prevent from corrosion, under high temperature conditions this property fails. This may be due to mechanical instability of the deposit or a galvanic corrosion under thermal treatment. Some Interfacial tensions probably appear between the oxide NiO, and the metallic nickel. The interfacial energy weakens the material that undergoes corrosion.

The filing made with the Ni/Si with 30 g/L of silicon is much better protection then Ni. This assumes that the NiO formation is not involved here. The quality of the deposit depends on the particle content of the electrolyte. This value requires optimum value above which, the deposit becomes worse. The electrolyte Ni-30 g/L silicon coating provides a good while that containing 50 g/L silicon gives unstable surfaces that does not protect the sample from corrosion under the conditions studied. The protection capacity is due to the particles of silicon, which are oxidized into silica and covering the surface of the specimen.

The very low thermal expansion coefficient of the silicon in the nickel matrix can reduce expansion of the metal so that under condition of high temperature, in spite of the kinetic energy received by oxygen, it cannot enter Ni in the network. In addition, the silicon on the surface of the pattern is oxidized. The network of silica, by oxygen atoms, pointing outwards, reduces the oxygen gradient. This prevents oxygen from approaching the model; protecting thereby the pattern from any corrosion. The resistance appearing is to be attributed to silica formed. The variation of the catch of mass, and the resistance evolution appear on the same periods so that the explanations given here are in concordance.

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