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# A Study on the Material Reliability of a Ni Alloy Electrodeposition

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## 1. Introduction

Due to the occasional occurrences of a localized corrosion such as a SCC and pitting in steam generator tubing(Alloy 600), the degraded tube is removed from service and fixed by a plugging leading to a significant economical loss. Otherwise, a degraded steam generator tube is repaired for re-use. Apart from the typical sleeving method by introducing welding and mechanical expansions which causes a residual stress in the parent tube, an electrodeposition inside a tube does not induce a parent tube deformation and hence a significant residual stress.[1]

For a successful electrodeposition inside a tube, many processes should be developed. Among these processes, an anode to be installed inside a tube, a degreasing condition to remove any dirt and grease, an activation condition for a surface oxide elimination, a strike layer forming condition to be adhered tightly between the electroforming layer and the parent tube and a condition for an electroforming layer should be established. Through a combination of these various process parameters, the desired material properties can be accomplished. For an application in a plant, it should be noted that a material reliability of the electrodeposit concerned with a variation of a material property as a function of the electrodeposit position in the vertical direction of a tube is also very important.

It is natural that a Ni alloy electrodeposition is selected as a proper electrodeposition system because Alloy 600 is mainly composed of nickel and a nickel electroplating has been widely studied to improve a corrosion resistance, and the mechanical and magnetic properties[2,3]. Moreover, a Ni alloy electrodeposition process can be used for a PWSCC mitigation of various components including a steam generator tube because a Ni alloy electrodeposit shows an excellent SCC resistance[4].

This work is related to an anode development for an electrodeposition inside a tube and a variation of material properties of the Ni alloy electrodeposits formed through the use of the anode.

## 2. Experimental

For a plate specimen, Ti plate coated with Pt and a stainless steel plate with a surface area of $3x10cm^2$ were used as an anode and a cathode, respectively. For a tube specimen, the developed anode was installed inside the tube by using air pressure and then a solution was circulated by using a solution pump at a flow rate of about 100ml/min.

Strike layer was formed in an aqueous solution including nickel chloride of 1.6 mol and boric acid of 0.65 mol with 5% hydrochloric acid. Temperature of strike layer bath and layer thickness were controlled to be about $40^{o}C$ and 5 μm, respectively.

Regards to an electrodeposition layer, Ni sulphamate, phosphorus acid, Fe sulphamate and DMAB(dimethyl amine borane) are used as a Ni source, a P source, an Fe source and a B source, respectively. The bath was composed of Ni sulphamate of 1.39mol and boric acid of 0.65mol. Contents of the P, Fe and B sources were controlled to be 0.0035~0.007, 0.0025 and 0.001mol, respectively. The pH and temperature of the prepared bath were controlled to be 2 by using sulphamic acid and 60°C, respectively.

During the electrodeposition, the applied average current density and duty cycle were $100mA/cm^2$ and 50%, respectively. Duty cycle(%) is defined as the ratio of on-time to one period(on-time + off-time) and the one period is constant at 10msec in this study.

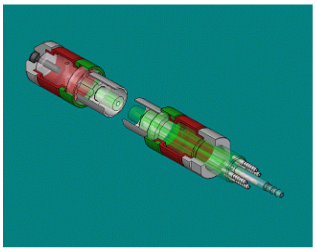
Alloy composition analysis of the deposit was performed by using an ICP analyzer(Model JY80C(Jobin Yvon)). Hardness was measured by applying a 50g load for 10sec, 10 times. The average was used as the hardness value. Stress-strain curve for the specimens prepared by EDM(electro discharge machining) was obtained with a strain rate of 1mm/min by using Instron 8872.

## 3. Results and discussion

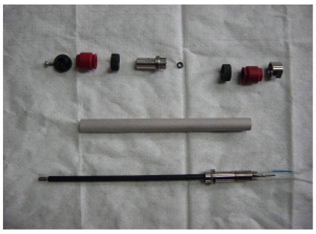
Figs. 1(a) and (b) show the schematic design and actual parts for an anode probe. The anode can be positioned at a desired location and insulated from the cathode(Alloy 600) by using two seals expanded by the air provided through an air line. Through two inlets and three outlets, the solution is refreshed continuously. Pt coated tube inside the anode probe and an Alloy 600 tube are used as the anode and the cathode, respectively. This anode can be installed to a multiple electrodeposition system as shown in Fig. 2. Multiple electrodeposition system was assembled to form three electrodeposits simultaneously. For this, 3 anodes, power supplies, solution pumps and air pressure controllers can be controlled independently. Temperature of six baths for cleaning, degreasing, activation, strike layer and electrodeposition can also be controlled independently.

Table 1 presents the Vickers hardness values for pure Ni, Ni-P-Fe and Ni-P-B as a function of the electrodeposit position in the length direction. The upper parts of the electrodeposit showed higher hardness values than those for the lower parts.

Fig. 3 shows the stress-strain curves for a pure Ni electrodeposit as a function of the electrodeposit position in the length direction. Similar to the hardness results of Fig. 3, yield strength and tensile strength of the upper parts were larger than those of the lower parts. On the other hand, an elongation of the upper parts was smaller than that of the lower parts. However, it can be conceived that the variation of the material properties along the electrodeposit length is acceptable when considering the process margin. In spite of a relatively small variation of the material properties, it is presumed that an improvement of the variation should be attempted. It is plausible that a variation occurrence can be composed of 3 causes. First, hydrogen evolution reaction occurs on the cathode surface(Alloy 600) generating hydrogen gas which moves toward the upper part. Hydrogen gas can operate as a nucleation site leading to a hardness increase caused by a grain refinement. Secondly, oxygen evolution reaction occurs on the anode surface(Pt) vividly generating oxygen gas. Oxygen gas can also operate as a nucleation site at the upper part. Thirdly, a variation can occur by an intrinsic factor such as a local and micro current distribution or other unknown factors.



(a)



(b)

Fig. 1. Schematic design for an anode probe and actual parts assembled into an anode probe.

### 4. Summary

A material reliability of the an electrodeposit concerned with a variation of a material property was evaluated as a function of the electrodeposit position in the vertical direction of a tube. It can be conceived that the variation of the material properties along the electrodeposit length is acceptable when considering the process margin. For an improvement of the reliability of a material property, the causes for a variation occurrence were presumed and an attempt for a variation improvement was conducted. A Ni alloy electrodeposition process can be suggested as a PWSCC mitigation method of various components including a steam generator tube because the Ni alloy electrodeposit formed inside a tube by using the installed assembly shows proper material properties as well as an excellent SCC resistance.

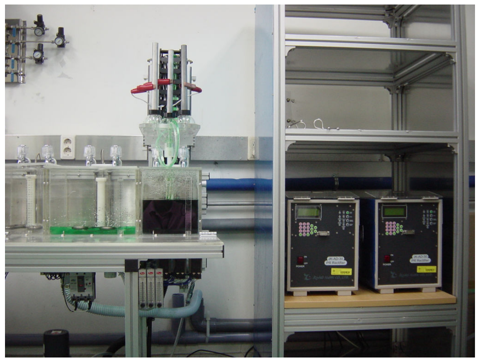


Fig. 2. Multiple electrodeposition system.

Table 1. Vickers hardness values for pure Ni, Ni-P-Fe and Ni-P-B as a function of the electrodeposit position in the length direction.

| **Hardness** | **Pure Ni** | **Ni-P-Fe** | **Ni-P-B** |
|---|---|---|---|
| **High** | **166** | **200** | **220** |
| **Middle-high** | **165** | **208** | **213** |
| **Middle-low** | **146** | **168** | **179** |
| **Low** | **148** | **167** | **218** |

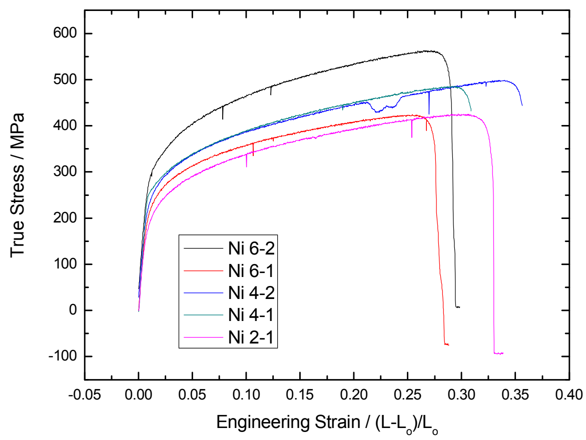



Fig. 3. Stress-strain curves for pure Ni electrodeposit as a function of the electrodeposit position in the length direction.

## REFERENCES

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