ELECTRON MICROSCOPY AND ELECTROCHEMICAL STUDIES OF NICKEL THIN FILMS FOR CORROSION PREVENTION IN H_2S ELECTROLYTE.

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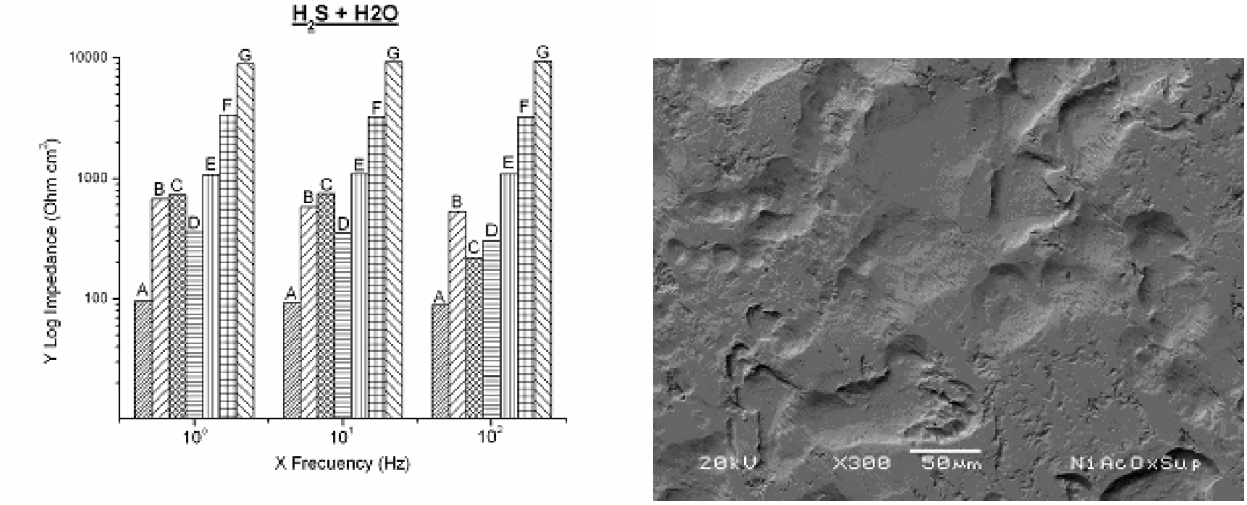
Nickel thin films with regular configuration and similar thickness were deposited by: magnetron sputtering[1,2] and electrolytic deposit[3]-on steel AISI 1018 (UNSG 10180). These thin film coatings (1300 nm) expect be an alternative method of protection for corrosion in a sulphur media. The aim of this work was to rank the deposition techniques used by spectroscopy impedance technique applied to the thin films coatings. Also a study of the morphology by scanning electronic microscopy (SEM) characterization was performed in order to identify topological and structural imperfections like porosities, terraces. kinks, etc. of the films surface and evaluate the protective properties when are in contact with the sulphur media[4]. Sputtering deposits present an homogeneous thin film coating better than the ones obtained by electrolytic technique. For the sulphur media a saturated H_2O+H_2S electrolyte was used.

Sulphur corrosion environments present a great interest to oil and gas industry. The presence of sulphides compounds in oil industry results in economical losses due to corrosion. Failures of pipeline and equipment due to wet cracking are frequently experienced in oil refining and production operations, in the literature [5] it is reported attempts to obtain better understanding of the mechanisms involved in corrosion processes. Metallic coatings can be used as a protection in sulphur media since their properties to form oxidized and reduced states; can promote resistance to oxidation processes [6]. The passive nickel thin film on the steel like a protective properties is other aim of the present work.

The substrate used is mild carbon steel AISI 1018 (UNS G 10180) sheets (2x2x0.1cm). The different deposits of nickel thin films was obtain by: electrolytic and magnetron sputtering deposition. The electrolytic thin film deposition use a typical Watt's bath [3].
Magnetron sputtering is the other nickel thin films deposition technique used. A nickel target (99.99% purity) with argon (99.99% purity) like working gas was used, the sputtering pressure is 34.6 pa. The deposit potential was 1000 volts. These values were for all the deposits. The deposition time was 15 minutes. Structural, morphological and topological studies in the samples surfaces were carried out with a JEOL 5600-LV. scanning electron microscope A GillAC potensiostat 4.2.9,. was used to electrochemical characterization with the Electrochemical Impedance Spectroscopy (EIS) mode for identify imperfections in the film and evaluate the protective properties in the sulphur media. The electrochemical measurements were carried out in H_2O+H_2S (pH=3) electrolyte: The impedance conditions are: max. frequency is 30.000 Hertz and the min.0.01 Hertz, the amplitude is 10 mili Volts, the number of integration are 10 cycles.

The spectra are presented in the forms of Nyquist and Bode diagrams. The Nyquist diagrams are too difficult to explain for the quantity of noise values and dispersed points, the same way for the Bode diagram, this shows various time constant mechanism for all the deposits and techniques, this difficult any kind of analysis for the results, even using the Faraday's cell, This work try to discuss the values obtained, employing Bode diagram values to plot them for an alternative presentation, this consist in arrangement between the modulus of the impedance versus the frequency. The Bode's values chosen, are for three zones with different frequency to explain diverse parts in the electrochemical process, the order of frequency proposed is 10^0, 10^1, 10^2 Hz.



a) b)

Figure 1 Graphic with the values of Log Impedance versus frequency for: **a)** A)Steel, B) Ni Bulk, C) Ni Bulk Oxide, D) Steel Ni Electrolyte, E) Steel Ni Electrolyte Ox., F) Steel Nickel Sputtering, G) Steel Nickel Sputtering Oxide in H_2O + H_2S (saturated) electrolyte and **b)** SEM micrograph of the Steel Nickel Sputtering Oxide system.

The nickel coatings applied by sputtering present homogeneity in the entire surface applied opposite thing to the obtained by the electrolytic deposit, this can be corroborated in the impedance results. The corrosion observed in the coatings of this work is classified as located corrosion type. Electrochemical Impedance Spectroscopy (EIS) technique seems to be suitable to evaluate the optimal thickness in the metallic thin film.

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