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Influence of the Time of Permanence in the Hydrolysed Solution of a Tetrasulfide Bis-silane Dopped with Cerium (IV) on Corrosion Behaviour of Coated Galvannealed Steel.

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Abstract – For metallic materials an important issue in delaying corrosion advance is the role of corrosion inhibitors or barrier films. Inhibitors can be added to polymeric films in order to improve their corrosion resistance. In addition, corrosion inhibitors should be non-toxic, of low cost and easy to handle, as cerium salts. In this work, the influence of time of permanence in the hydrolysed solution of a bis-1,2-[triethoxysilylpropyl]tetrasulfide (BTESPTS)+50 ppm Ce^{4+} on corrosion behavior of coated galvannealed steel was studied. Electrochemical results showed the adequate time of permanence to obtain BTESPTS +50 ppm Ce^{4+} film to protect the substrate against corrosion is 15 minutes.

Galvannealed steel is a corrosion resistant hot dip zinc coated steel with a further annealing treatment leading to specific intermetallic Fe-Zn phases which make this steel largely employed in the automotive industry. In order to get lower costs and lower environmental damages arising from the disposal of such processes effluents, silane films appear as a promising alternative to replace phosphatation and/or chromation treatments for galvannealed steel [1-2]. The silanes are organofunctional molecules known by acting as coupling agents; adhesion promoters in paintings and can also supply protection against corrosion [3]. The Ce^{4+} action in silane films is not completely known but it is known that Ce^{4+} can promote a homogeneous film, which can interact mainly in silanes reactions, increasing the length of the chain. The addition of Ce^{4+} ions can enhance the corrosion resistance of silane films, probably due to a complex formation with the silanol making the reticulation an easy and effective process [4-5]. The aim of this work is to study the influence of the time of immersion in the hydrolysis solution to obtain the bis-1.2-[triethoxysilylpropyl]tetrasulfide BTESPTS film dopped with ions Ce^{+4} in the attainment of a polysilane film for protection against corrosion of a galvannealed steel.

Galvannealed steel samples were coated by a dip-coating process (10cm/min entrance and exit rate) and permanence during 2, 5, 15 and 30 min in the hydrolyzed solution; coated samples were cured at 150°C for 40 minutes. The solvent was a water/ethanol (50/50w%) solution with higher water content than it is commonly found in the literature, in order to make the process safe to be used in the industrial practice (liquid stream with a higher flash point). The preparation conditions were 3% BTESPTS in 97%(50%water/ethanol) solution, pH=6.5 and, 135 min of hydrolysis. The coated samples corrosion resistance was evaluated by open-circuit potential (E_{oc}), electrochemical impedance spectroscopy (EIS), linear polarization resistance (R_p) and polarization curves after three hours of immersion in aerated 0.1 mol. L^{-1} NaCl. The real impedance value (at 30 mHz) and polarization resistance values showed that longer immersion times were needed to obtain barrier homogeneous silane films to promote corrosion resistance for galvannealed steel. The reason is that the the galvannealed steel surface morphology presents porous (consequence of a mutant eta phase) of hard wettability and it takes a longer time to get all the interstices completely coated. was obtained for sample prepared using 15 minutes of immersion in the hydrolized solution. The longest immersion time tested (30min) contributed to the undesired silanols condensation leading to a less protective film. The Z_{real} values at 30 mHz to all sample are presented in table 1.

Table 1: Z_{real} value obtained at 30 mHz and Rp value obtained after 3 hours of immersion in the electrolyte.

Time of immersion	2 min	5 min	15 min	30 min
Z_{real}	3.9 KΩ.cm²	8.5 KΩ.cm²	38.5 KΩ.cm²	18.9 KΩ.cm²
R_p	3.6 KΩ.cm²	3.9 KΩ.cm²	14.7 KΩ.cm²	10.3 KΩ.cm²

Polarizations curves and liner polarization resistance data are in close agreement with EIS data showing the best results for sample prepared using 15 minutes of immersion time. This study showed that to efficiently coat galvannealed steel it is necessary to hold the metal for a longer time (15min) in the hydrolyzed solution in order to get its surface totally wet to form an homogeneous and protective silane film.

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