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The Influence of the Solvent on the Corrosion Resistance of Vinyltrimethoxysilane Based Coatings Deposited on X20Cr13 Steel

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Abstract. The paper presents the results of research on the anti-corrosion properties of coatings based on vinyltrimethoxysilane, alcohol, and acetic acid in sulfate solutions with or without chloride ions (pH = 2). The experiment investigated the influence of a solvent (methanol, ethanol, butanol, propanol) on anti-corrosion properties, surface morphology, and adhesion to the steel substrate. The coatings were deposited on X20Cr13 stainless steel using the sol-gel dip method.

Introduction

Stainless steel is used in many industries: from the production of heavy machinery and energy to precision mechanics and electronics [1, 2]. The protective mechanism of stainless steel relies on a naturally generated (passive) oxide layer that prevents corrosion in mildly corrosive environments. In the presence of aggressive halogen ions (Cl-, Br-, I-), the oxide layer undergoes pitting corrosion [3-5]. In order to increase the protection against corrosion, coatings, e.g. chromate or phosphate, are used. The toxicity and carcinogenicity of chromium and phosphorus limited the use of this type of anti-corrosion coating [6-8]. The search for newer, better, more environmentally friendly coating systems has begun. One of the best alternatives in technology is the sol-gel method [9, 10].

The sol-gel process involves hydrolysis and condensation of M (OR) n metal alkoxides. Tralkoxysilanes such as VTMS vinyltrimethoxysilane is well known as coupling agents and crosslinkers. These are compounds that have such functional groups as vinyl, amino, carboxyl, glycidyl, hydroxyl, or acrylic. The speed of the hydrolysis and condensation reactions depends on the parameters: pH, temperature, and concentration of the reagents. Changing these parameters may change the structure and properties of the silane layer to a large extent [11-15].

The precursors of the synthesis reaction in the sol-gel method are various metal alkoxides, salts, or nitrates [16]. The preparation of sol solutions is based on dissolving the appropriate alkoxide in an organic solvent, most often anhydrous alcohol [17]. The simplest alcohols such as methanol, ethanol, propanol, or butanol are widely used raw materials in chemical synthesis [18, 19]. The hydrolysis and condensation (polycondensation) reactions take place simultaneously in the entire volume of the solution. The speed of the sol-gel process can be controlled, for example, by using a suitable catalyst (acid or basic) [20]. In this study, the influence of the solvent used as a component of the VTMS / Alkohol / AcOH coating on its adhesion and corrosion protection of X20Cr13 steel was assessed. Over the last dozen or so years, many articles have been written on the protection of metal surfaces with silanes, and these publications do not deal with the issue of the influence of the solvent on the process of protecting metals covered with silane coatings against corrosion. The paper presents research on alcohol: methyl, ethyl, butyl, and propyl alcohol.

The results presented in this article may be of interest to those industries where there is a risk of corrosion [21-24] and biocorrosion [25-28]. In the case of using special coatings [29-31], especially those subjected to laser treatment [32-34], the issue of texture may be important so that the anti-corrosion layer has proper adhesion. Improvement of anti-corrosion properties will significantly affect the risk of occurrence of particular failure scenarios [35-38] and thus modify the quality management schemes [39, 40]. Undoubtedly, it will also have an impact on the design of new devices and machines [41], especially those operating in difficult conditions [42-44] or military treatment [45-47], taking into account the increased corrosion resistance in accordance with the Robust Design concept [48, 49], and thus will be an inspiration for data analysis methods [50-52] in the field of production engineering and quality management [53].

Materials and Methods

Materials. The chemical composition of X20Cr13 stainless steel (in wt.%) was as follows: C-0.17; Cr-12.6; Si-0.34 ;, Ni-0.25; Mn-0.30; V-0.04; P-0.024; S <0.005; the rest is Fe. The following reagents were used: vinyltrimethoxysilane VTMS (by Sigma Aldrich), methanol (by Chempur), ethanol (by POCH Basic), propanol (by Sigma Aldrich), butanol (by Chempur), acetic acid AcOH (by Chempur) with the analytical purity grade and deionized water. The volume ratio of the resulting VTMS: Alcohol: AcOH coatings was 4.84: 2.16: 3.0.

Measurements were carried out on electrodes made of X20Cr13 stainless steel samples. Metal with a diameter of 5 mm was mounted in polymethyl methacrylate frames. The initial treatment of metal samples consisted of wet polishing with polishing papers (600, 1000, 2000), rinsing with deionized water and ethyl alcohol, and then drying at room temperature. In order to decrease the surface of the samples, each time before applying the coating, they were washed with acetone.

Preparations of VTMS/Alkohol/AcOH Coatings. Preparation with the use of a solvent was included in the publication [54]. Four sols were prepared according to the following procedure: 3.16 mol dm^{-3} VTMS was dispersed in alcohol:

1) methyl MtOH,
2) ethyl EtOH,
3) propyl IPOH,
4) butyl BtOH.

The amount of 0.1 mol dm^{-3} AcOH was gradually added to the solutions. The solutions were continued to mix for 2 days. After the solution had changed its consistency, the samples were immersed for approximately 20 minutes. The methodology of applying silane coatings was developed in the publication [55]. The coated samples were dried for 1 day at room temperature in a silica gel desiccator.

Experimental Conditions. The electrochemical tests were carried out using the CHI 706 measurement station (CH Instruments, Austin, Texas, USA) in a three-electrode system, in which the auxiliary electrode was a platinum electrode and the reference electrode was a saturated calomel electrode NEK. In order to determine the protective properties of VTMS/Alcohol/AcOH coatings, potentiodynamic polarization curves of uncoated and coated steel electrodes were recorded in the following solutions: 0.5 mol dm^{-3} Na_2SO_4 (pH = 2) and 0.5 mol dm^{-3} Na_2SO_4 + 0.5 mol dm^{-3} NaCl (pH = 2), the potential range was from -0.8 V to 1.6 V, the polarization rate was 10 mVs^{-1}. The surface appearance of the coatings deposited on the tested steel was assessed using the Olympus GX41 optical microscope. The adhesion tests were carried out using a simple Scotch^{TM} tape sticking and peeling test after each coating application.

Experimental Part

Fig. 1 shows the morphology of VTMS/Alkohol/AcOH coatings deposited on the surface of X20Cr13 steel. The morphology of all shells is smooth and transparent. The coatings evenly cover the entire surface of the electrodes, with no visible pits when the coating is removed. The adhesion to the substrate of X20Cr13 stainless steel was checked using Scotch^{TM} tape, immediately after the deposition of the VTMS/Alcohol/AcOH coatings. The produced coatings are characterized by good adhesion to the steel substrate.

In order to characterize the anti-corrosion properties of the produced VTMS/Alcohol/AcOH coatings against general corrosion, potentiodynamic curves were recorded in an acidified solution of 0.5 mol dm^{-3} Na_2SO_4 at pH = 2, in the potential range of -0.8 ÷ 1.6 V for X20Cr13 steel. uncoated and coated. As can be seen from Fig. 2, the produced coatings inhibit anodic processes and shift the corrosion potential of the steel by approx. 0.5 V towards positive values. The addition of the solvent leads to a reduction (1 - 4 times) of the cathode and anode currents density (Fig. 2c).

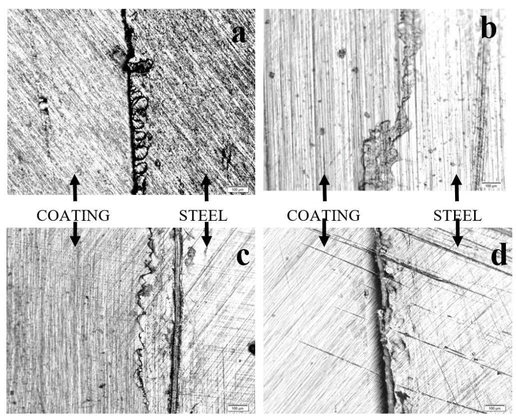

Fig. 1. *VTMS/Alcohol/AcOH coating with a concentration of 3,16 mol dm^{-3} VTMS in solution and containing alcohol: methyl MtOH (a), ethyl EtOH (b), propyl PyOH (c), butyl BtOH (d). Olympus GX41 (x100)*

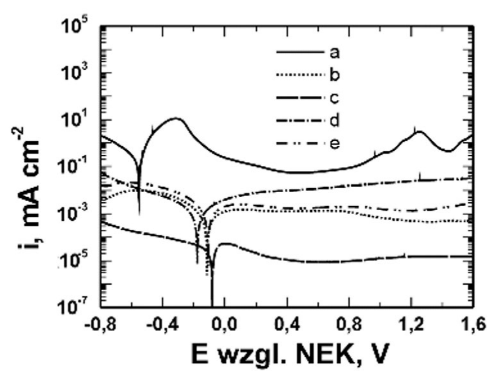

Fig. 2. *Potentiodynamic polarization curves recorded in a solution of 0.5 mol dm^{-3} Na_2SO_4 pH = 2 for X20Cr13 steel not coated (a) and coated with VTMS/Alcohol/AcOH coatings with a VTMS concentration of 3.16 mol dm^{-3} and containing alcohol: methyl MtOH (b), ethyl EtOH (c), propyl PyOH (d), butyl BtOH (e). Polarization speed 10 mVs^{-1}, solutions in contact with air, 24 ° C.*

In order to assess the inhibition capacity of the local corrosion coatings produced, analogous potentiodynamic curves were performed for the sulfate solution containing the addition of 0.5 mol dm^{-3} chloride ions (Fig. 3). The corrosion potential of X20Cr13 steel covered with VTMS/Alcohol/AcOH coatings is shifted towards the positive values by approx. 0.5 V (Fig.3c) in relation to the value of the corrosion potential recorded for the uncoated steel (E_{cor} = -0.597 V) (Fig. 3a). Lower values of cathode and anode current densities were also observed for steels covered with these coatings. For all VTMS/Alcohol/AcOH coatings deposited on X20Cr13 steel, no breakthrough potential of the passive layer (pitting nucleation potential) was observed. Thus, VTMS/Alcohol/AcOH coatings effectively protect the steel against the penetration of a solution containing Cl^- ions and inhibit pitting nucleation processes. Fig. 4 shows the surface photos of the samples after corrosion tests

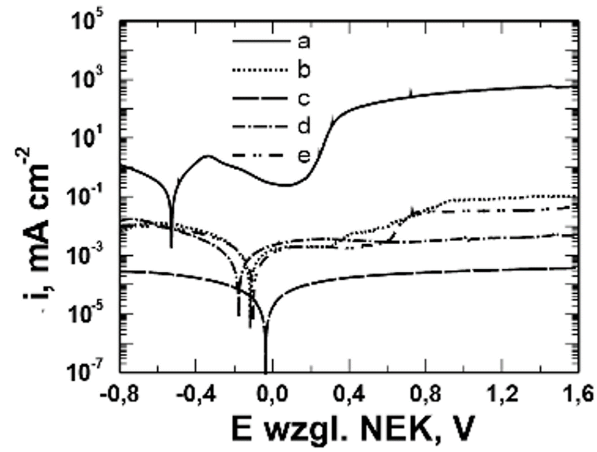

Fig. 3. *Potentiodynamic polarization curves recorded in a solution of 0.5 mol dm^{-3} Na_2SO_4 + 0.5 mol dm^{-3} NaCl pH = 2 for X20Cr13 steel not coated (a) and coated with VTMS/Alcohol/AcOH coatings with a VTMS concentration of 3.16 mol dm^{-3} and containing alcohol: methyl MtOH (b), ethyl EtOH (c), propyl PyOH (d), butyl BtOH (e). Polarization speed 10 mVs^{-1}, solutions in contact with air, 24°C.*

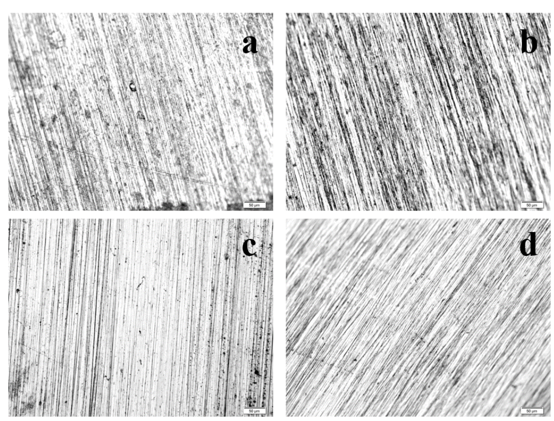

Fig. 4. *Morphology of the sample surface after corrosion tests, coating: VTMS/MtOH/AcOH (a), VTMS/EtOH/AcOH (b), VTMS/PyOH/AcOH (c), VTMS/BtOH/AcOH (d). Olympus GX41 (x200)*

Fig. 4 shows the morphology of samples with VTMS/Alcohol/AcOH coating after corrosion tests in a solution of 0.5 mol dm^{-3} Na_2SO_4 + 0.5 mol dm^{-3} NaCl pH = 2. The photos show steel surfaces without pitting. Thus, the obtained coatings effectively inhibit the corrosive processes.

Conclusions

The tests of VTMS/Alcohol/AcOH coatings have shown that the selection of an appropriate solvent has a significant impact on the anti-corrosive properties of VTMS coatings. The produced coatings show a homogeneous surface without visible defects in the structure, good adhesion to the substrate, and extend the time of steel resistance to the action of chloride and sulfate ions in an acidic environment. The VTMS/EtOH/AcOH coating shows the best ability to block the transport of chloride ions responsible for pitting corrosion of steel.

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