



# The failure behaviour of a commercial highly pigmented epoxy coating under marine alternating hydrostatic pressure

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## ABSTRACT

A commercial epoxy coating, incorporated with inert pigments, was aged in a sodium chloride (3.5 wt.%) solution under alternating hydrostatic pressure (AHP), compared with that under atmospheric pressure (AP). By the fitting analysis of the impedance data with equivalent electric circuits (EEC), the failure process was accelerated and got complicated under AHP. The evolution of water absorption by gravimetric tests indicated that water diffusion deviated from typical Fickian diffusion to case II diffusion. A leaching process of pigments was observed by scanning electron microscopy (SEM) and no shift of chemical bonds was monitored by Fourier transform infrared spectroscopy (FT-IR). The AHP accelerated the coating physical failure, and the chemical structures of the coating did not change during the experiment.

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## 1. Introduction

The metallic equipment is prone to be attacked by aggressive species such as water, oxygen and ions when applied in the corrosive marine environments. The epoxy resin based organic coating has been widely used in the corrosion protection of metals in marine environments [1–3]. The organic coating acts as a barrier film between the metallic equipment and the aggressive environments, isolating the metals from corrosive species [4–7]. To enhance the barrier performance of the coating, various kinds of anti-corrosion pigments have been incorporated into the polymer matrix [8–12]. The inactive or inert pigments, which are non-soluble in water and non-toxic to environment, have gained a lot of attentions recently [13–15]. Micaceous iron oxide, red iron oxide, titanium dioxide, glass flake, mica and talc are the typical examples of the inert pigments. These pigments mainly play a physical role on the barrier behaviour of coatings by increasing the length of the diffusional pathways for water and oxygen and thereby decreasing the coating's permeability [16,17].

In recent years, extensive knowledge has been gained on the influence of pigmentation on coatings' protective ability and water permeability. Hu et al. [16] studied the corrosion electrochemical characteristics of red iron oxide pigmented epoxy coatings on aluminium alloys. Santana et al. [17] studied the resistance of

metallic substrate protected by an organic coating containing glass flakes. Most of the previous works emphasize on the optimizing of the pigmentation or the degradation of pigmented coatings under atmospheric environments. However, only few works have been reported on the failure of the coatings in deep sea environments. Among the environmental parameters in the marine, the change in hydrostatic pressure with depth is the most significant and may greatly affect the corrosion behaviour of materials applied in deep sea [18–20]. Recently, Liu et al. [21] studied the failure behaviour of a nano-SiO<sub>2</sub> filler epoxy coating under hydrostatic pressure. Li et al. [22] studied the failure mechanism of an epoxy glass flake coating under high hydrostatic pressure. Liu et al. [23] studied the impedance models and water transport behaviour of epoxy coating at hydrostatic pressure. It revealed that the high hydrostatic pressure weakened the barrier performance of the inert pigments and accelerated the penetration of water into the coatings. In fact, the equipment applied in deep sea, such as transfer-vessels, usually serves in an alternating environment between the deep sea and shallow sea, exposing to the alternating hydrostatic pressure (AHP). However, the available information remains insufficient for proper understanding of the failure mechanisms of the inert pigmented organic coatings in the real marine environments. Therefore, it is very important and urgent to study the failure behaviour of the organic coating incorporated with inert fillers under AHP environment.

In this work, a commercial highly pigmented epoxy coating was immersed in a sodium chloride (3.5 wt.%) under AHP environment. The electrochemical impedance spectroscopy (EIS), gravimetric

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method, pull-off adhesion test, scanning electron microscopy (SEM) and in situ Fourier transform infrared spectroscopy (FT-IR) were applied and the results were compared with that under atmospheric pressure (AP) environment. The influences of AHP on the deterioration process of the coating were discussed based on the experimental results.

## 2. Experimental

### 2.1. Sample preparation

The substrate metal material was a kind of steel commonly used in marine environments, with the following composition (in wt.%): 4.67 Ni, 0.60 Cr, 0.46 Mo, 0.065 Al, 0.54 Mn, 0.076 C, balance Fe. The dimensions of the steel specimens were 20 mm × 20 mm × 2 mm for EIS test and 30 mm × 30 mm × 2 mm for pull-off test. The EIS specimens were mounted in curing epoxy resin with an exposure area of 4 cm<sup>2</sup>. All steel specimens were ground to 600 grit size using SiC paper, degreased with acetone and ethanol, and then put into a desiccator for future use.

A commercial, two-components (A and B), epoxy-polyamide, heavy-duty coating applied in marine environments (H44-61, Xiamen Sunrui Ship Coating Co., Ltd., China) was used in this study. HX-501 was used as lacquer diluent, and it is a mixture of xylene and *n*-butanol with the ratio of 7:3. Component A was dissolved with HX-501 at the ratio of 10:3. Then component A was mixed with component B at the ratio of 7.1:1. After thorough mixing of the two liquid components, the coating was applied by hand brushing. Two different kinds of coating samples were prepared for the corresponding measurements: (1) the coating/steel sample, in which the coating was applied on the steel by a hand brush. In order to obtain a uniform coating, the samples were cured in an oven under the following conditions: 40 °C for 24 h and room temperature (25 °C, 30% RH) for 168 h (7 days). These samples were for EIS measurements, pull-off tests and morphology observation. The average coating thickness was measured by a handheld electronic gauge (PosiTector 6000 of Defelsko, United States) according to ISO 2808-1997 [24]. The thickness of the coating was controlled to 250 ± 10 μm. (2) The free film sample, in which the coating was brushed on a silica gel plate. After being cured in an oven with 40 °C for 24 h, the film was peeled off from the plate and cut into the dimension of 20 mm × 20 mm × 0.2 mm for gravimetric experiment. Then the films were put at room temperature (25 °C, 30% RH) for 168 h (7 days). Prior to all the tests, the samples were stored in a desiccator to keep dry and to avoid any changes in properties due to adsorption of moisture from the atmosphere.

### 2.2. Simulation of the marine AHP environment

Two testing experiments were applied for the measurement: (1) the testing experiment conducted under AP; (2) the experiment conducted under AHP, this was carried out in an Automatic Deep Ocean Simulation System (shown in Fig. 1). The system includes autoclave, hydrostatic system, pneumatic system, thermostatic system and monitoring system. The high hydrostatic pressure was obtained by pushing nitrogen into the autoclave of the system, which was controlled by a pressure valve. The high hydrostatic pressure in the autoclave was kept at 60 atm (which is equivalent to 600 m depth hydrostatic pressure in the ocean) and the low pressure was kept at 1 atm. One alternating pressure cycle contained high hydrostatic pressure for 6 h and atmospheric pressure for 6 h (60 atm, 6 h – 1 atm, 6 h). The whole experimental process lasted for 240 h (10 days), consisting of 20 alternating cycles. The 3.5 wt.% NaCl solution was applied as the test solution, and then the whole system was sealed completely before pressurizing. The solution temperature was controlled by a thermostatic bath to

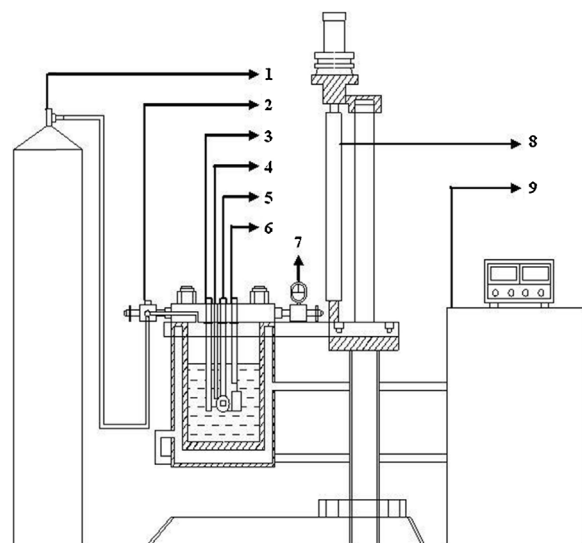


Fig. 1. Schematic diagram of deep ocean simulation device: (1) nitrogen cylinder; (2) valve; (3) solid reference electrode; (4) thermocouple; (5) working electrode; (6) counter electrode; (7) pressure metre; (8) automatic elevator and (9) temperature controller.

room temperature (25 ± 1 °C). With this experimental setup, in situ electrochemical measurements and other immersion experiments could be conducted.

### 2.3. EIS measurements

An AUTOLAB electrochemical station (Metrohm, Switzerland) was used for EIS measurements under both AP and AHP environments. A conventional three-electrode system was used, i.e. with the coating/steel sample as the working electrode, platinum plate (20 mm × 20 mm) as the counter electrode, and the saturated calomel electrode (SCE) and the solid Ag/AgCl electrode as the reference electrode under AP and AHP, respectively. The solid Ag/AgCl electrode (saturated with KCl,  $E_{(vs. SCE)} = -0.157$  V) was adopted because SCE might be broken during the pressurization and depressurization process. Preliminary experiments showed that these two different reference electrodes had no effects on the EIS test. A sinusoidal AC perturbation of 50 mV (rms) amplitude coupled with the open circuit potential was applied to the metal/coating system. All EIS tests were performed in the frequency range from 100 kHz to 10 mHz. The EIS data were analyzed with ZSimpWin software.

### 2.4. Gravimetric tests and adhesion tests

Gravimetric data were obtained by measuring the mass of the free film samples at stipulated time intervals under AP and AHP, respectively. The samples were removed from immersion, quickly dried with filter paper and mass measures were performed. The maximum time out of testing solution was 1 min. The gravimetric tests were carried out using a Sartorius BS124S microbalance (1 μg resolution). The experiments were carried out three times to ensure reproducibility of the results. The water absorption ( $Q_t$ ) was then calculated according to the following equation [25]:

$$Q_t(\%) = \frac{m_t - m_0}{m_0} \times 100 \quad (1)$$

where  $m_t$  is the mass of free coating at time  $t$ ;  $m_0$  is the mass before immersion; and  $Q_t$  is the water absorption (%) at time  $t$ .

The adhesion tests were conducted by PosiTest Pull-Off Adhesion Tester according to ASTM D4541-02 [26], which was a testing method for assessing the intensity of the outer paint to separate

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