



# Damage resistance and anticorrosion properties of nanosilica-filled epoxy-resin composite coatings



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## ABSTRACT

Silica nanoparticles surface-capped with diglycidyl ether of bisphenol A were dispersed in a solution of epoxy resin, hardener and acetone. The resultant suspension was then coated onto the surface of duplex stainless steel of type DSS 2205 and cured with temperature, generating a 50  $\mu\text{m}$  thick silica/epoxy coating. Epoxy coating without nanosilica was also prepared as a reference in the same manner. Mechanical properties of these coatings were compared and characterized using the Vickers hardness test. Three-point bending test was performed in combination with acoustic emission to analyze the damage initiation and development in the coating. The effects of incorporating the silica particles on the surface characteristics and the corrosion resistance of the epoxy-coated steel were investigated with contact-angle and surface energy as well as by potentiodynamic polarization and electrochemical impedance spectroscopy in a 3.5 wt.% NaCl solution. Results indicate, that silica particles significantly improved the microstructure of the coating matrix, which was reflected in an increased damage resistance, reduced degree of delamination, increased surface roughness and induced hydrophobicity. The silica/epoxy coating was proven to serve as a successful barrier in a chloride-ion-rich environment with an enhanced anticorrosive performance, which was confirmed by the reduced corrosion rate.

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## 1. Introduction

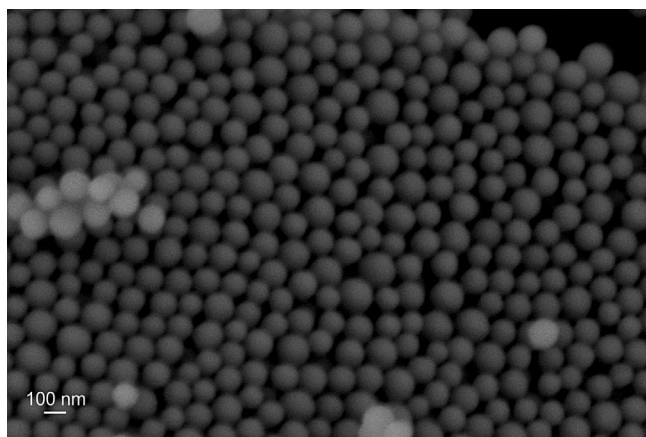
Polymer composites are subjected in many applications as adhesives and matrix resins, epoxy resin being one of the most common polymer matrices that are widely used to protect steel reinforcements in concrete structures [1,2]. It has excellent mechanical properties, chemical resistance, good electrical insulating properties and strong adhesion to heterogeneous substrates. Epoxy coatings not only reduce the corrosion of a metallic substrate by providing an effective physical barrier between the metal and the environment containing an aggressive species, such as an enhanced chloride-ion concentration,  $\text{O}_2$  or  $\text{H}^+$ , they also serve as a reservoir for corrosion inhibitors that help the steel surface to resist attack from aggressive species.

The practical use of epoxy coatings in industry, however, is seriously limited by poor impact resistance and stress cracking resistance due to a highly cross-linked structure [3] as well as by susceptibility to damage by surface abrasion and wear [4].

To overcome this drawback, researchers have made numerous attempts to improve the properties of epoxy by adding various nanofillers [5–13]. They studied the favourable effects of particle size, volume fraction and the quality of the dispersion on the mechanical response of the polymer composites [6,14–21]. In addition, a lot of attention has been paid to epoxy coatings containing nanoparticles that show a significantly improved barrier performance for corrosion protection by decreasing the porosity [22].

There is, however, still a lack of knowledge of the fracture mechanisms in particulate (i.e., silica)/epoxy composite coatings on various substrates under applied load. As commonly used surface-sensitive diagnostic methods like scanning electron microscopy (SEM) are insufficient tools for the detection of all possible failure sources and for establishing a direct correlation between the applied strain and delamination, a different approach is needed to gain a full insight into crack formation, progress and delamination. Acoustic emission (AE) analysis during tensile, fatigue or three-point bending provides a suitable method to obtain the strain dependence of crack formation and growth. So far AE studies mostly investigated damage resistance of fibre (i.e., carbon) or textile/epoxy composites and metallic coatings on metallic substrates. Here AE signals were used to determine the quality of coatings [23]

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**Fig. 1.** SEM image of synthesized 130 nm silica nanoparticles. The image was taken with FE-SEM JEOL JSM-6500.

and to relate the spectral signature of AE signals to a specific type of failure such as matrix cracking, fibre breakage, debonding, fibre pull-out or delamination [24].

In this study we evaluated the influence of incorporation of silica nanoparticles in an epoxy matrix on the damage resistance of 50  $\mu\text{m}$  thick silica/epoxy coatings adsorbed on duplex stainless steel (DSS 2205) substrate. Mechanical properties of coatings were studied with Vickers hardness test. To analyze the crack formation and delamination of the coatings, three-point-bending tests were performed together with acoustic emission analysis to detect transient stress waves propagating in the coating as a consequence of bending deformation. Contact angle/surface energy measurements gave us insight into the surface and wetting characteristics of silica/epoxy coatings in comparison to pure epoxy coatings. Finally, the anticorrosion behaviour of the coatings was evaluated with potentiodynamic measurements.

## 2. Experimental

### 2.1. Materials

Duplex stainless steel DSS 2205 (22.7% Cr, 5.7% Ni, 2.57% Mo, 1.37% Mn, 0.38% Si, 0.032% P, 0.03% C, 0.001% S in mass fraction) was used as a substrate.

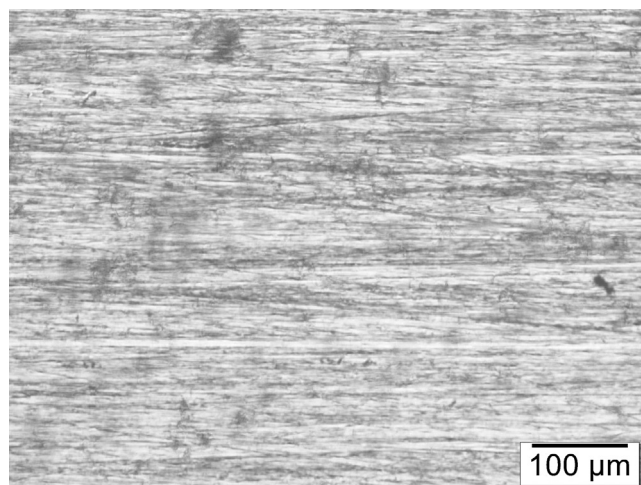
Epoxy resin (Epikote 828LVEL, Momentive Specialty Chemicals B.V.) was mixed with a hardener 1,2-Diaminocyclohexane (Dytek DCH-99, Invista Nederland B.V.) in the ratio 100: 15.2 wt.% and used as the matrix in the composite.

Composite reinforcing silica ( $\text{SiO}_2$ ) nanoparticles with a mean diameter of 130 nm were synthesized following the Stöber–Fink–Bohn method [25]. The specific details of our synthesis procedure are discussed in [26,27]. Fig. 1 shows the scanning electron microscopy (SEM) image of the synthesized 130 nm silica nanoparticles and indicates a homogeneous size distribution of the particles.

Diglycidyl ether of bisphenol A (Sigma–Aldrich) was used as the silica surface modifier to prevent agglomeration. Imidazole (Sigma–Aldrich) served as a reaction catalyst.

### 2.2. Surface modification of silica

Silica and diglycidyl ether of bisphenol A (modifying agent) were mixed in the weight ratio of 2:3 and dispersed in 50 mL of toluene in the presence of imidazole (25 wt.%). The mixture was then refluxed at 100  $^\circ\text{C}$  for 2 h. To remove the by-product (imidazole) it was centrifuged three times using acetone as a solvent. The remaining silica



**Fig. 2.** Optical micrograph of 50  $\mu\text{m}$ -thick 130 nm silica/epoxy coating on DSS 2205 substrate. The image was taken with Nikon Microphot-FXA.

was then dispersed in acetone and stirred at room temperature for 2–3 h. Finally, the acetone was removed and the silica was dried in an oven at 110  $^\circ\text{C}$  for a few hours.

### 2.3. Steel substrate preparation

The steel sheet with a thickness of 1.5 mm was cut into plates of 15 mm in width and 75 mm in length for the mechanical testing and into discs of 15 mm diameter for the electrochemical examination. Prior to the application of the coating the steel plates and discs were prepared by a mechanical procedure, initially ground with SiC emery paper down to 1200 grit, and then rinsed with distilled water.

### 2.4. Composite coating preparation

Epoxy-based composites were prepared by blending with 2 wt.% of 130-nm surface-modified  $\text{SiO}_2$  particles. To improve the dispersion of the silica particles in the coating, they were dispersed in epoxy resin using a solvent. Acetone was used as a solvent, since the Fourier-transform infrared spectroscopy (FTIR) and FT-Raman analysis [28] indicated that the processing in acetone did not chemically alter the epoxy network. Prior to the addition of the silica particles, both the resin and the hardener were separately diluted in acetone in the weight ratio of 1:1. The nanoparticles were then dispersed in the epoxy resin/acetone solution using ultrasonication for 20–30 min at room temperature. After adding the hardener/acetone solution in the next step, the mixture was manually stirred for a few minutes. Finally, 10–20  $\mu\text{l}$  of the silica/epoxy resin/hardener/acetone mixture was poured onto the steel substrate plates and a uniform film was then applied to the substrate using a wet film applicator. The prepared coatings were degassed under vacuum for 10–15 min to additionally remove the excess solvent. The composite coating was then cured in two steps. The composite coatings were first pre-cured at 70  $^\circ\text{C}$  for 1 h and then post-cured at 150  $^\circ\text{C}$  for another hour. The resulting coatings on the steel-substrate plates were 50  $\mu\text{m}$  thick. For comparison, neat epoxy coatings without silica fillers were also prepared and cured in the same process as the composites.

Prior to testing, the prepared coatings on DSS 2205 substrate were analyzed with light microscopy to indicate microscopic homogeneity. In Fig. 2 we present a typical light microscopy image of a 130 nm silica/epoxy coating indicating

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