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Inorganic Porous Materials Based Epoxy Self-Healing Coatings

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The long-term stability of protective coating for metal is critically important for structural applications [1, 2]. Selfhealing ability extend the service life of protective coatings leading to a significant reduction in maintenance cost for oil and gas pipe lines and structural parts in civil and construction industry. Recently, the self-healing technology based on healing agent loaded containers has been receiving attention [3, 4]. The incorporation of self- healing agent loaded containers into polymer matrix can be carried out using existing blending techniques. Hence, this technology facilitate large-scale application of self-healing materials [5]. Different micro or nano containers has been used for the storage and release of self-healing agents upon specific corrosion triggering conditions (e.g. on pH change) or upon mechanical damage [6]. Polymer capsules, polymer nanofibers, hollow glass bubbles, hollow glass fibers etc. were used by the researchers to load the healing agent inside their cavity. The inorganic particles with nano cavity offers large surface area, high pore volume and good stability favorable for the storage of the healing agents. Moreover, the usage of inorganic nanomaterials as reservoirs for healing agent can eliminate the tedious encapsulation process. The present study aims to use inorganic nanotubes and mesoporous silica as containers for healing agents in epoxy coating. The ability of Halloysite nanotubes (HNT), titanium dioxide (TiO₃) nanotube and mesoporous silica to load and release the healing agents are investigated and compared their performance. Among them, Halloysite nanotubes are naturally occurring clay mineral. Meanwhile, TiO₂ nanotube and mesoporous silica are synthesised in laboratory and characterised using scanning electron microscopic (SEM), transmission electron microscopic (TEM) techniques and Brunauer-Emmett-Teller (BET) surface area analysis. The morphology of the nanotubes and mesoporous silica are shown in Figure 1 (in supporting file). In this study, the epoxy pre-polymer and hardener are used as healing agents. Containers loaded with epoxy and hardener can provide a repair system with matching chemical entity with host epoxy coating. Both epoxy encapsulated nanotubes (either Halloysite or TiO₂ nanotubes) and amine immobilized mesoporous silica are incorporated into epoxy, followed by the addition of diethylenetriamine curing agent. The mixture is coated on the metal with an average thickness of 300 μm. The controlled epoxy coatings are also prepared without nanotube and mesoporous silica. Epoxy coating loaded with encapsulated Halloysite

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nanotubes and immobilized mesoporous silica is abbreviated as 'EP/HNT/SiO₂' and the one loaded with encapsulated TiO₂ nanotubes and immobilized mesoporous silica is abbreviated as 'EP/ TiO2/SiO2'. The self-healing ability of the scratched coatings is monitored by electrochemical impedance spectroscopy (EIS) in definite time intervals for 5 days. Both EIS bode plots and tafel polarization curves are analysed to observe the self-healing ability of the coatings. For the scratched controlled epoxy coating, after an immersion time of 24 hours, the impedance curve drop to its minimum value over the entire frequency range and on further immersion period the impedance curve remains its minimum value. However, in the case of self-healing coatings, the initially declined impedance value recovers in successive days. The recovery in low frequency impedance values (at 0.01 Hz), which is a direct reflection of the recovery of corrosion resistance of the coating are evaluated. While EP/TiO₂/SiO₂ coating recovered 57% of its anticorrosive property, the EP/HNT/SiO₂ coating recovered only 0.026%. This results suggest that the nature of the nanotubes affect the amount and rate of healing agent released into the scratched area from the tube lumen which itself affect the self-healing ability of the coating. SEM is also used to observe the healed scratches on the coatings. After 96 hours of immersion in 3.5 wt% NaCl solution, the scratches in EP/TiO₂/SiO₂ self-healing coatings are found to be almost covered. The results confirm the effective self-healing ability of the EP/TiO₂/SiO₂ coating in which the released epoxy pre-polymer from nanotube lumen get contact with the amine hardener immobilized in mesoporous silica and cross-link to cover the scratch.

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