

# Economical Production of Corrosion and Temperature Shielding Coatings for Deep Water Applications

Patchamatla Satyanarayana Raju<sup>1</sup>, Ajjarapu Venkata Narayana Lakshmi Sharma<sup>1</sup> and Allaka Gopichand<sup>2</sup>

<sup>1</sup>Department of Mechanical Engineering, GIET University, Gunupur, Odisha, India

<sup>2</sup>Department of Mechanical Engineering, Swarnandhra College of Engineering and Technology, Narsapur, Andhra Pradesh, India

## Correspondence to:

Patchamatla Satyanarayana Raju  
Department of Mechanical Engineering,  
GIET University,  
Gunupur, Odisha, India.  
E-mail: [p.satyanarayanraju@giet.edu](mailto:p.satyanarayanraju@giet.edu)

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## Abstract

In this study, we present the inexpensive fabrication of titania and silica thin films on an aluminum substrate to enhance corrosion resistance, as well as the ability to reflect infrared radiation and protect the temperature, extending the substrate's useful life using a sol-gel dip coating approach. The coating crystal structure was studied using an X-ray diffraction (XRD), which revealed that the titania was anatase and the silica was amorphous with their characteristic peaks at Bragg angle 25° and 22°, respectively. Fourier-transform infrared spectroscopy (FTIR) studies on the titania and silica-coated alumina sandwich pipes demonstrated the formation of Ti-O-Ti and Si-O-Si functional bonds at wave numbers 520 cm<sup>-1</sup> and 980 cm<sup>-1</sup>, respectively. The surface morphology of the coating was investigated using the field emission scanning electron microscopy (FESEM) and found uniformity of the coating without any cracks and any other defects. The EDX (energy dispersive X-ray spectroscopy) analysis on the coating endorsed the existence of Ti, and Si elements of the coating with a percentage of 33.5% and 33%, respectively. The investigation of ultraviolet-visible and near-infrared spectroscopy (UV-Vis-NIR) in the diffused reflectance mode of the coating indicated that the coating may reflect infrared radiation and shield the sandwich pipe, extending the coating's life. Furthermore, corrosion resistance tests on the coating using ASTM B117 standards revealed that the coating can withstand corrosion for more than 72 h when tested with 5% NaCl (sodium chloride) solution.

## Keywords

Sol-gel synthesis, Dip-coating, Titania, Silica, Corrosion resistant, Temperature resistant

## Introduction

A thin layer of coating on the desired material has the power to increase the functionality and performance of the as-deposited material. Nanotechnology has transformed the above-mentioned purpose of coating and its versatile use [1-3]. Coating is the most common way for protecting the surface of various metals and alloys from external conditions and improving the material's functionality. Corrosion of metals and alloys may cause material deterioration and necessitate repair and replacement, which also plays a role in the safety of many applications. Ceramic coatings are currently used practically everywhere in industrial applications to increase corrosion resistance, heat resistance, erosion resistance, and wear resistance. Titania and silica-based coatings are always under extensive research to apply them in various fields of science and technology [4, 5]. With a thin layer of covering, these coatings can increase surface qualities and protect the target substance from salt solution and environmental contaminants. These coatings can

be developed via either expensive processes like physical vapor deposition [6], chemical vapor deposition [7] or cost-effective sol-gel based procedures including dip-coating [8], spin coating [9], and spray pyrolysis [10]. Physical vapor deposition and chemical vapor deposition procedures need extensive processing times and high fabrication costs, but sol-gel processes are simple and economical. The sol-gel method is a wet-chemical process that uses a precursor, a solvent, and a catalyst to create the required gel to deposit on a substrate. The major crucial use of the sol-gel based coating is its use on a wide range of substrates with varying compositions, without regard to the size or shape of the substrate's geometry.

Dip-coating is one of them, and technique is utilized for homogenous deposition on diverse substrates by simply dipping the appropriate substrate in the produced gel, which provides a thickness of less than 2  $\mu\text{m}$ . In the aforementioned method, the thickness of the coating may be regulated by altering the parameters dip rate and duration, the longer the dip time, the thicker the desired materials. Ceramic coatings with very low electronic conductivity like  $\text{TiO}_2$  [11],  $\text{SiO}_2$  [12],  $\text{ZrO}_2$  [13], and  $\text{Al}_2\text{O}_3$  [14] are the important materials that are reported for anti-corrosive treatment. Titania and silica are user-friendly and simple to synthesis, and they are increasingly employed for corrosion prevention due to their environmental friendliness. By applying them to the chosen material, silica coatings have the capacity to increase hardness while decreasing wear rate. Due to the additional thermal energy generated and the harsh exposure to seawater, aluminum sandwich pipes in deep seas frequently corrode and have decreased life duration. The coating's life may be extended by depositing a thin layer of titania or silica on the aluminum pipe; the as-deposited coating has the capacity to reject warm radiation generated by infrared radiation; the coating is also corrosive resistant and can display its multifunctional nature. Liang et al. fabricated nano- $\text{SiO}_2$  coating on the galvanized steel substrate that can act as both hydrophobic and corrosion-resistant by a one-step immersion technique [15]. When a silica coating was applied to the substrate, it exhibited a  $160^\circ$  contact angle with 1 wt.%  $\text{SiO}_2$  during interaction with the water droplet, as well as a decrease in contact angle up to  $120^\circ$  and an increase in corrosion efficiency up to 99.6% in 3.5 wt.% NaCl solution. Kania et al. deposited titania thin films on the magnesium alloy using atomic layer deposition approach to test the anti-corrosion behavior [16]. The electrochemical and immersion experiments in Ringer's solution were performed in a physiological environment at  $37^\circ\text{C}$  and showed that the titania covered magnesium alloy successfully protected it against corrosion. Jabri et al. used dip-coating to create polyaniline-based titania coatings on oil pipelines to prevent corrosion [17]. The coating's potentiostatic polarization investigation supported improved corrosion inhibition performance on the mild steel surface with titania of 0.5 g. Hamzah et al. coated a range of eco-friendly nanocomposites on a mild steel substrate by precipitating varying weight percentages of  $\text{CoAl}_2\text{O}_4$  on the surface of titania, followed by a calcination procedure [18]. The impact of  $\text{CoAl}_2\text{O}_4$  wt.% on anti-corrosion treatment was tested using electrochemical impedance in 3.5% NaCl solution, and the Tafel plot revealed

the best anti-corrosion capabilities of 99.99%. Bhuvaneshwari et al. prepared pure xV-titania samples using precipitation method for the anti-corrosion applications [19]. According to the findings of the doping investigation, vanadium doping increased the phase transition temperature of anatase to rutile and also endorsed that the V-titania as an alternative for the chromium-based corrosion inhibitor and lifetime enhancement of the product. Hu et al. fabricated silica coatings on magnesium alloys using a home-made immersion technique to prevent corrosion [20]. Electrochemical impedance spectroscopy and potentiodynamic experiments in 3.5% NaCl were used to study the corrosion resistance of the as-deposited coating, which revealed increased corrosion resistance on the coated surface. Nano-silica coatings are also often regarded as anti-corrosion protection coatings due to their high hardness, mechanical strength, and chemical stability. They can also serve as hydrophobic materials when properly dispersed into the organic matrix pores. Liang et al. employed the immersion approach for producing a superhydrophobic, corrosion-resistant coating using silica on a galvanized steel substrate [21]. The as-fabricated coating performed the corrosion protection efficiency test with 99.5% effectiveness, and the charge transfer resistance recorded  $1.54 \times 10^3 \Omega \cdot \text{cm}^2$ . Additional testing of the coating's water contact angle revealed a  $120^\circ$  contact angle, and this was recommended as the industrial process for creating hydrophobic, corrosion-resistant coatings.

In this work, we report the anti-corrosion and thermal shielding coatings fabricated by titania and silica gels using dip-coating technique. Section-2 of the draft deals with the experimental process of sol-gel synthesis and dip-coating process. The titania and silica coatings were examined with XRD, FTIR, FESEM, and UV-Vis-NIR, for their structural, morphological, and optical properties and presented in section 3. The corrosion test on the aluminum substrate was performed using ASTM standards and also included in section -3. Finally, the entire research was concluded in section-4 of the draft.

## Materials and Methods

### Chemicals involved in the synthesis process

The precursors in the synthesis method were titanium isopropoxide (TTIP) and tetraethyl orthosilicate (TEOS) from Sigma Aldrich. Ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ) from Changshu Hongsheng Fine Chemicals and distilled water from GK Life Sciences were utilized as solvents, and hydrochloric acid (HCl) from Sisco Research Laboratories was employed as a catalyst to speed up the process. All the reagents involved in the synthesis of titania and silica gel were of analytical grade and required no further purification.

### Sol-gel synthesis

#### Synthesis of titania and silica gels

Titania gel was synthesized utilizing the sol-gel method by combining the previously described precursor (TTIP), solvent ( $\text{C}_2\text{H}_5\text{OH}$ ), distilled water, and catalyst (HCl) in the molar ratios of 1:15:1:0.1. Similarly, silica was synthesized using the same method, with the precursor (TEOS), solvent (ethanol), distilled water, and catalyst (HCl) at molar ratios of

1:10:3:0.01, respectively as shown in figure 1. The titania gel may be utilized immediately for dip coating, but the silica gel requires 2 h of stirring and 24 h of ageing.

## Deposition

The aluminum substrates were ultrasonically cleaned with deionized water and ethanol after being extensively washed with lab detergent prior to the deposition procedure. Sol-gel derived titania and silica gels were dip coated onto the aluminum substrates by maintaining dip rate and dip time constant at 5 cm per 30 s and drawn at the same speed. After the titania and silica gels have been dipped into the aluminum substrates, they are allowed to drain at room temperature before being dried with a hot air blower for 10 min. Later, the films were sintered for one hour in a furnace at 500 °C as shown in figure 1 to eliminate excess solvent residues and achieve crystallinity of the deposited films.

The obtained films were then examined using XRD (Bruker D8 Advance, Germany) to determine the phase of the as-deposited films, FTIR (Bruker vertex, Germany) to show the functional groups in the coating, FESEM (ZEISS, Germany) to determine the surface morphology, EDX to determine the elemental composition, and UV-Vis-NIR (UV 1800, Shimadzu, Japan) for reflectance analysis. Furthermore, the coating is subjected to corrosion resistant experiments in accordance with ASTM standards to validate the coating's anti-corrosion performance.

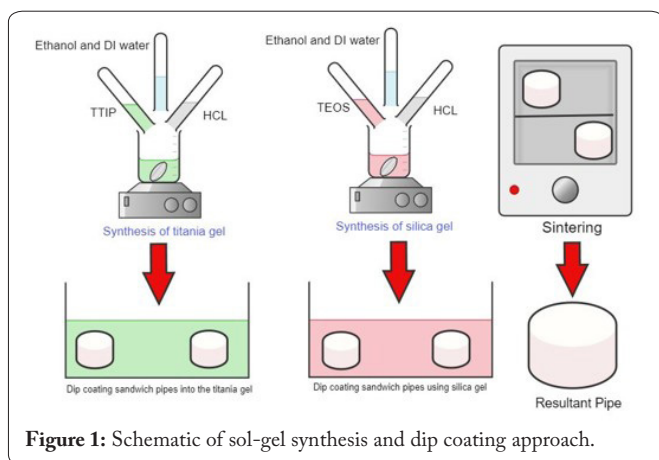


Figure 1: Schematic of sol-gel synthesis and dip coating approach.

## Results and Discussion

After the fabrication of titania and silica dip thin films on to the aluminum substrates, the coatings were investigated for structural properties using XRD and presented in figure 2a and 2b. The XRD pattern of the coating is recorded between 10° and 70° Bragg angles, as illustrated in figure 2a and 2b. Figure 2a shows an XRD pattern of titania thin films on aluminum sandwich pipes, with a digital picture inset. The broad peaks in the presented XRD pattern are due to the deposition of titania thin films onto the aluminum substrate. The produced coating accounts for the absence of a prominent peak in the exhibited XRD pattern of titania thin films, which shows a typical peak at Bragg angle 25°. Titania thin film deposition on aluminum substrates also decreased roughness by filling the surface holes on the substrate. The peaks seen at the Bragg an-

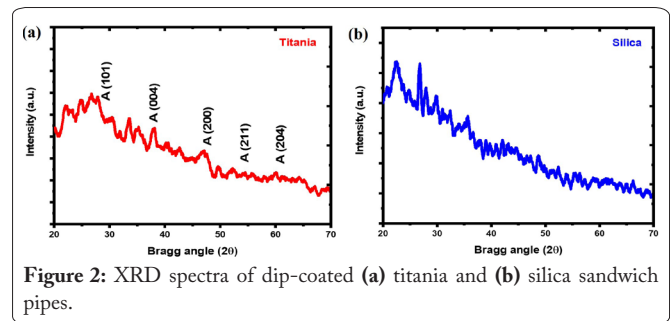


Figure 2: XRD spectra of dip-coated (a) titania and (b) silica sandwich pipes.

gles 25°, 38°, 47°, 55°, and 60°, respectively indicate the (101), (004), (200), (211), and (204) planes of anatase titania and are consistent with the published literature [22, 23]. The pattern also shows that there are no rutile peaks in the pattern. Due to the presence of 35.5% Ti and 64.5% oxygen in the coating, the EDX supports the structural pattern shown in figure 2a. Figure 2b demonstrates the XRD pattern recorded between Bragg angles 10° to 70° of the dip coated aluminum sandwich pipe. The pattern also shows noise, which is comparable to the XRD pattern of titania shown and may be linked to the aluminum substrate. Furthermore, the spectrum shows that only a distinctive peak at 22° Bragg angle was identified, indicating that the silica coating is amorphous, which is consistent with the published literature [15]. EDX study of the coating shows 33% Si content, which validates the structural research performed by XRD.

To identify and confirm the elements of the dip coated titania and silica coated sandwich pipe FTIR study was carried out and presented in figure 3a and 3b. The spectrum in figure 3a shows that the dip coated aluminum sandwich pipe has titania on the surface. The presence of stretching vibrations of O-H molecules in the coating is supported by the occurrence of peaks at wavenumber 3500 cm<sup>-1</sup>. The coating also shows the existence of carbonyl and hydroxyl groups, with their vibration bonds visible at 2300 cm<sup>-1</sup>. Furthermore, at wavenumber 520 cm<sup>-1</sup>, the Ti-O-Ti functional linkages in the coating are shown, demonstrating the presence of titania on the aluminum sandwich pipe. The FTIR spectra of the silica coated alumina sandwich pipe is supported by figure 3b. At wavenumber 3600 cm<sup>-1</sup>, the coating shows the development of O-H stretching bonds. At wavenumber 2400 cm<sup>-1</sup>, carboxyl, hydroxyl, and alkane linkages were found, as with the titania covered alumina sandwich pipe. The interaction of Si bonds with stretching bonds of O-H, resulting in Si-OH, was discovered at wavenumber 110 cm<sup>-1</sup>. Finally, the distinctive peak at wavenumber 980 cm<sup>-1</sup> supports the development of a silica coating on the aluminum sandwich pipe and is in good agree-

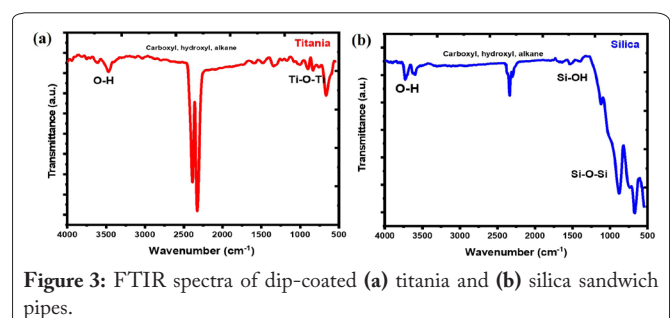


Figure 3: FTIR spectra of dip-coated (a) titania and (b) silica sandwich pipes.

ment with the published research [24, 25].

The morphological study of the titania and silica coated sandwich pipe was performed using FESEM and is shown in figure 4a and 4b, with their EDX analysis shown in figure 4c and 4d. Figure 4a and 4b show the surface morphology of the coating, demonstrating its homogeneity with no signs of fractures or other imperfections. The roughness of the aluminum surface was also reduced, and the surface was found to be smooth when titania and silica were deposited on the aluminum sandwich pipe. Figure 4c and 4d show the elemental analysis on the coating done using the EDX spectrum. The EDX analysis in figure 4c shows that the titania coating contains 35.55% Ti and 64.5% O, and the silica coating demonstrated in figure 4d on the alumina sandwich pipe contains 33% Si and 40.4% O, confirming that the titania and silica coatings are uniformly deposited.

The application of the as-fabricated titania and silica coatings on the alumina sandwich pipes as thermal shielding and corrosion resistant was demonstrated in figure 5. Figure 5 shows the results of a reflection examination on titania and silica coated alumina sandwich pipes using UV-Vis-NIR. The reflectance spectra show that the coating may reflect near-infrared radiation up to 59% and 65%, respectively, from the silica and titania coatings. When used in deep water applications, the developed coating can deflect radiation, boosting the pipe's performance and longevity. Furthermore, the anti-corrosion ability of the coating was tested in a closed chamber according to ASTM B117 standards by spraying a 5% NaCl solution in an inclined position for 72 h. Compared to immersion or other methods, the salt spray was chosen to do the corrosion resistant test since it creates a thin layer of oxygen-rich electrolyte in the aluminum substrate all the time, giving us real-time query data. The above-mentioned test on titania and silica coated aluminum sandwich pipes exhibited no surface corrosion or white rust for the whole period. The amount of time it takes to develop white rust depends on the corrosion resistance of the coating; coatings with a higher corrosion resistance retard the formation of white rust. After 72 h salt spray test on titania coated aluminum sandwich pipes and silica coated sandwich pipes, very little white rust was observed on the edges of the sandwich pipe. The indication of corrosion on the sandwich pipe's edge is attributed to the fabrication technology used, namely dip coating. This type of coating may not only deflect heat radiation from the sandwich pipe but also extend its life by making it anticorrosive. The research study is very original, and the type of multifunctional application that may block radiation while also functioning as corrosion resistance has not been discussed previously.

## Conclusion

Sol-gel synthesis was chosen as the synthesis method because of its low cost and ease of usage. The aforementioned processes were used to synthesize titania and silica gels, and the gel requires an optimum period of 24 h of ageing to continue the deposition process. The titania and silica coatings on the aluminum sandwich pipe were fabricated using a dip coating method, and the coated sandwich pipes were sintered in a muffle furnace to achieve their crystallinity. XRD investi-

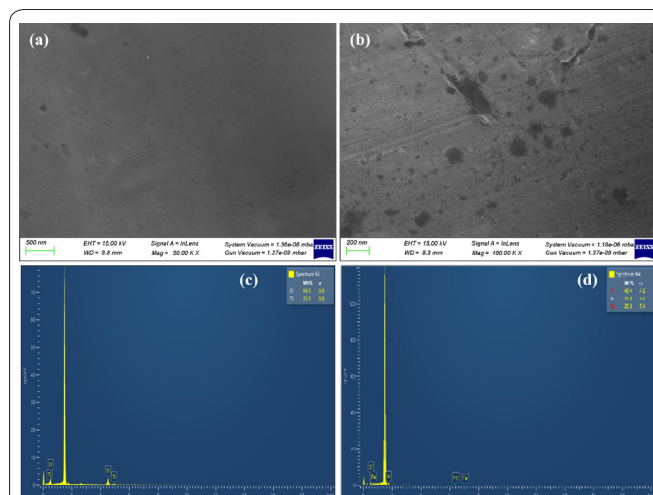


Figure 4: FESEM image of (a) titania and (b) silica and EDS analysis of (c) titania and (d) silica of aluminum sandwich pipes.

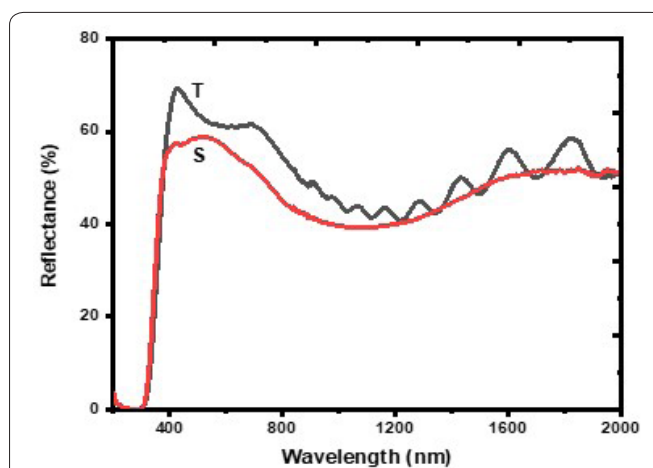


Figure 5: Reflectance spectra of the dip-coated titania and silica sandwich pipes.

gations on the coating revealed anatase titania and amorphous silica with a noise that may be attributed to the substrate aluminum. FTIR examination of titania and silica coated aluminum sandwich pipe reveals the presence of functional linkages Ti-O-Ti and Si-O-Si, confirming coating deposition. The coating's morphology was studied using FESEM, which confirmed the homogeneity of the coating with without any defects, and elemental analysis using EDX confirmed 33.5% Ti and 33% Si in the coating. Furthermore, the application of titania and silica coatings as thermal shielding and corrosion resistant demonstrates that the coating can reflect infrared radiation up to 59% and 65%, respectively, and that no white rust formed after exposing coated pipes to a salt fog atmosphere. Thus, coating can be recommended for aluminum sandwich pipes to deflect heat radiation and prevent the sandwich pipe from corrosion, which can increase the sandwich pipe's life and performance.

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## Conflict of Interest

The authors declare no conflict of interest.

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## Data Availability

Raw data were generated at NIT Warangal, India. Derived data supporting the findings of this study are available from the corresponding author P. Satyanarayana Raju on request.

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