

Preparation of Transparent and Thermal Resistant Zirconia Reinforced Epoxy Based Polymer Matrix Composite Coatings

Sanjay Kumar Awasthi¹, Kamal Sharma², Aayush Gupta^{3,*}

Abstract

This study investigates the thermal stability, structural characteristics, optical transparency, and surface wettability of epoxy-based composite coatings reinforced with 2 wt.% zirconia (ZrO₂) nanoparticles. The composite coatings were synthesized using a dip-coating technique on carbon fiber substrates and subsequently subjected to thermal treatments at 300 °C, 400 °C, and 500 °C. Structural analysis using X-ray diffraction (XRD) confirmed the amorphous nature of the epoxy matrix with no significant crystallization or phase change post-thermal exposure. Fourier-transform infrared (FTIR) spectroscopy validated the successful incorporation of ZrO₂ nanoparticles and revealed shifts in characteristic Zr–O and C=O vibrational bands, particularly at higher temperatures, indicating subtle chemical modifications. Surface wettability was assessed through static contact angle measurements, which showed consistent hydrophobic behavior (contact angles ~75°) across all thermal conditions, demonstrating the resilience of prepared coatings to thermal stress. Interestingly, optical transmittance in the visible region improved with increasing thermal treatment, rising from 7-10% in untreated and 300 °C treated samples to approximately 17% after treatment at 500 °C. This enhancement suggests temperature-induced microstructural changes favoring light transmission. Overall, the epoxy-ZrO₂ composite coatings maintained structural integrity, hydrophobicity, and exhibited improved transparency under elevated temperatures, making them suitable for advanced applications in harsh thermal environments such as aerospace, automotive, and electronics.

Keywords: Epoxy-ZrO₂ coatings, thermal stability, surface wettability, nanocomposite materials, optical transparency

INTRODUCTION

For engineering applications, hydrophobic surfaces have acquired attention of scientific community

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due to moisture repellent, corrosion resistive, and self-cleaning features [1–3]. Hydrophobicity of a material prevent the adherence of water molecules on the surface extending the lifespan and reducing maintenance costs with performance enhancement. Polymers such as epoxy resins are quite popular coating materials among scientific community due to decent mechanical strength, good durability, and excellent chemical resistance [4–7]. Unfortunately, the thermal stability or thermal oxidation limits the utility of epoxy based coatings at high temperatures. Prolonged exposure at high temperatures would deteriorate epoxy affecting its structural integrity. It restricts its engineering utility in industrial machinery, high-performance vehicles, and electronic components [8,9].

The incorporation of ceramic (such as metal oxides) particles could participate in enhancing the thermal performance of epoxy based coatings. With exceptional thermal resistance, chemical inertness, and superior mechanical properties, zirconia (ZrO_2) can be a promising choice as a filler materials for this application [10–12]. Due to high surface energy and nano-scale roughness, the addition of ZrO_2 nanoparticles exhibited the generation of hierarchical microstructure in epoxy based composite coatings [13,14]. The incorporation of ZrO_2 particles in epoxy based composite coatings enhances hydrophobicity by minimizing the contact area between water droplets and the coating. Such characteristics of composite coatings enables the their suitability in extreme environmental conditions including moisture and temperature [15–17]. Recently, Li et al. [18] reported polydopamine (PDA)- ZrO_2 /graphene oxide (GO) reinforced epoxy coating for corrosion resistant applications with 111.7° (maximum) as contact angle against water as compared 77.2° in case of pure epoxy based coating. Further, Alshammari et al. [19] reported a inconsistent (or decreasing from 50% to 40% in visible electromagnetic spectrum) optical transparency of 15 wt% ZrO_2 reinforced polyvinyl alcohol (PVA) coatings.

This study describes the synthesis of ZrO_2 (2 wt.%) reinforced epoxy based composite coatings and the effect of temperatures (up to $500^\circ C$) on its structural, optical characteristics and hydrophicity. By assessing factors such as structural integrity, wettability, transparency, and thermal stability, researchers can improve the formulation of ZrO_2 reinforced epoxy based composite coatings.

EXPERIMENTAL SECTION

To prepare ZrO_2 reinforced epoxy based composite coatings, a multi-step methodology was used. An ethanol based solution of epoxy resin (Araldite CY 230-1) was prepared followed by the addition of 2 wt.% ZrO_2 (purity 99%; 100-200 nm) nanoparticles in ethanol through sonication. The mixture was degassed to remove trapped air bubbles. A hardener (Aradur HY 951) was added to initiate the curing process, and the prepared coating solution was applied to aligned carbon fiber sheets using the dip-coating technique to achieve uniform coverage. The coated samples were then cured at room temperature for 24 hours, followed by a post-curing step at $80^\circ C$ for 2 hours. To assess the thermal resistance of the coatings, they were exposed to temperatures of 300, 400, and $500^\circ C$ in a controlled furnace environment. The samples were labeled as E-2Z (epoxy- ZrO_2 coating at room temperature), E-2Z300 (epoxy- ZrO_2 coating subjected to $300^\circ C$), E-2Z400 (epoxy- ZrO_2 coating subjected to $400^\circ C$) and E-2Z500 (epoxy- ZrO_2 coating subjected to $500^\circ C$). The dimensions of the synthesized samples were 10 mm in width and 50 mm in length. Characterization techniques, including XRD, FTIR, and contact angle measurements, were used to analyze the structural, chemical, and wettability properties of the coatings.

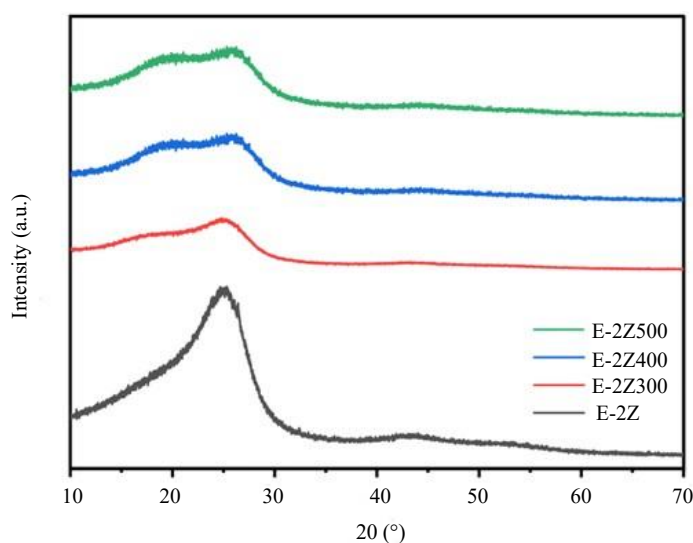


Figure 1. XRD patterns of epoxy- ZrO_2 coatings after thermal treatment at different temperatures (300 – $500^\circ C$).

RESULTS AND DISCUSSION

X-ray Diffraction (XRD) Analysis

XRD patterns of ZrO₂ reinforced epoxy based composite coatings were recorded to understand the structural modifications as a result of heat treatment at different temperatures (i.e., 300-500 °C). Figure 1 shows the XRD patterns suggesting a insignificant structural modifications upon heat treatment. The epoxy matrix maintained its amorphous structure after heating, indicating that the molecular arrangement of polymer did not experience crystallization or phase transitions [20,21]. XRD pattern of the prepared coating samples did not exhibit any signature of ZrO₂ phase. While, the confirmation of the inclusion of ceramic reinforcement has been discussed in subsequent section. Furthermore, a prominent peak at ~25° in all samples corresponds to the (002) crystallographic plane of the carbon fiber substrate, demonstrating that the underlying carbon fiber remained structurally stable and was not affected by the thermal treatments [22]. These results suggest that ZrO₂ reinforced epoxy based composite coatings are thermally resilient, maintaining their structural integrity and uniform nanoparticle distribution even at elevated temperatures.

FTIR Analysis

Figure 2 presents the molecular vibrations (FTIR spectra) of composite coating samples, confirming the successful integration of ZrO₂ and epoxy based polymer matrix. The FTIR spectrum of the epoxy-coated carbon fiber sheet shows no distinct peaks, indicating that the epoxy matrix does not significantly contribute to characteristic vibrational modes. For all the coating samples, vibrations in 500-900 cm⁻¹ range correspond to the stretching of Zr-O and Zr-O-Zr bonds confirming the presence of ceramic ZrO₂ [23,24]. Additionally, a strong vibration at 1050-1250 cm⁻¹ can be attributed to the bending of Zr-O bonds, further supporting the structural integrity of the composite samples. The spectral region (1000-1300 cm⁻¹) supported the stretching of C-O-C functional groups within the epoxy based polymer matrix, while an intense vibration was observed between 1600-1800 cm⁻¹ which is associated to the stretching of C=O bonds in the epoxy resin [20,21]. Interestingly, the a significant shift towards lower wave number has been observed for bending of Zr-O bond (at 1050-1250 cm⁻¹) along with the disappearance of 1400-1500 cm⁻¹ vibrations upon heat treatment at 400 and 500 °C.

Contact Angle Analysis

Contact angle measurements in Figure 3 reveal that the epoxy-ZrO₂ coatings maintained their hydrophobic properties with only slight variations after exposure to high temperatures. The initial contact angle of the unheated coating (E-2Z) was 74.80±0.11°, while coatings subjected to 300°C (E-2Z300), 400°C (E-2Z400), and 500°C (E-2Z500) exhibited contact angles of 74.96±0.21°, 75.41±0.20°, and 75.32±0.17°, respectively. These small changes indicate that the coatings resist significant alterations in surface wettability even under thermal stress.

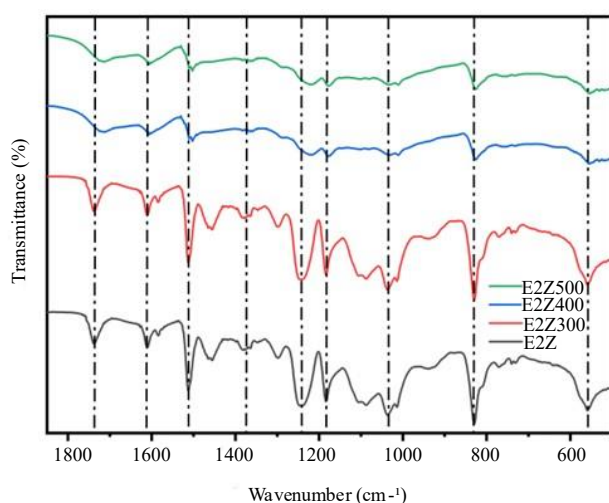


Figure 2. FTIR spectra of epoxy-ZrO₂ coatings after thermal treatment at different temperatures (300-500 °C).

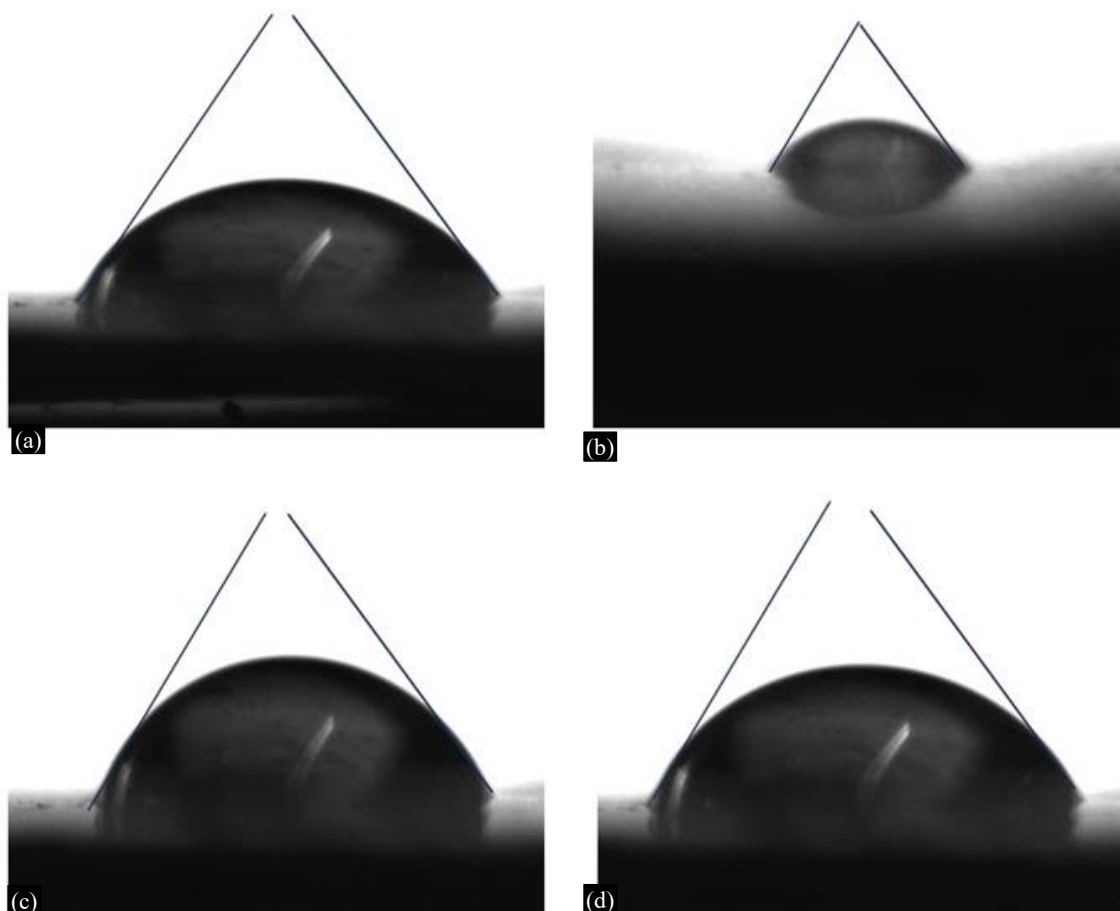


Figure 3. Front view of the water droplet to determine contact angle of (a) E2Z, (b) E2Z300, (c) E2Z400 and (d) E2Z500 as substrate.

This stability can be attributed to the strong interfacial interactions between the epoxy matrix and the ZrO_2 nanoparticles, which prevent degradation of the surface structure. Additionally, the uniform dispersion of ZrO_2 nanoparticles within the epoxy matrix likely contributes to maintaining a consistent surface roughness and chemical composition, further preserving the hydrophobic characteristics of the coatings across different temperature conditions [25–27].

Optical Transmittance Analysis

Optical transmittance study revealed that the prepared polymer-based coating was not completely transparent in the visible region of electromagnetic spectra. Figure 4 suggested that the heat treatment enhanced the transparent feature of the coating significantly. The substrate and E2Z exhibited 7-10% transmittance in the visible region. E2Z300 exhibited similar transparency in the same region as observed in the above-mentioned samples. While the optical transmittance ability of coatings was found to be directly proportional to the heat treatment temperature i.e. 400 (10% for E-2Z400) and 500 °C (17% for E-2Z500). Among these heat-treated samples, it was observed that treatment at 500 °C (E3A400) yielded maximum transmission (~17%) as compared to other E3A300 (<8%) and E3A400 (~11%) samples. Similarly, Alshammari et al. [19] reported optical transmittance characteristics of ZrO_2 reinforced PVA coating with varying concentration of ZrO_2 . They observed a decreasing transmittance with decreasing wavelength of radiation between 400 to 800 nm, while the present work describes a consistent transparency of the prepared coating samples within same range of electromagnetic spectrum. These observations opened a gate to explore the possibility of transmittance optimization for filler concentration and heat treatment of epoxy-based coatings.

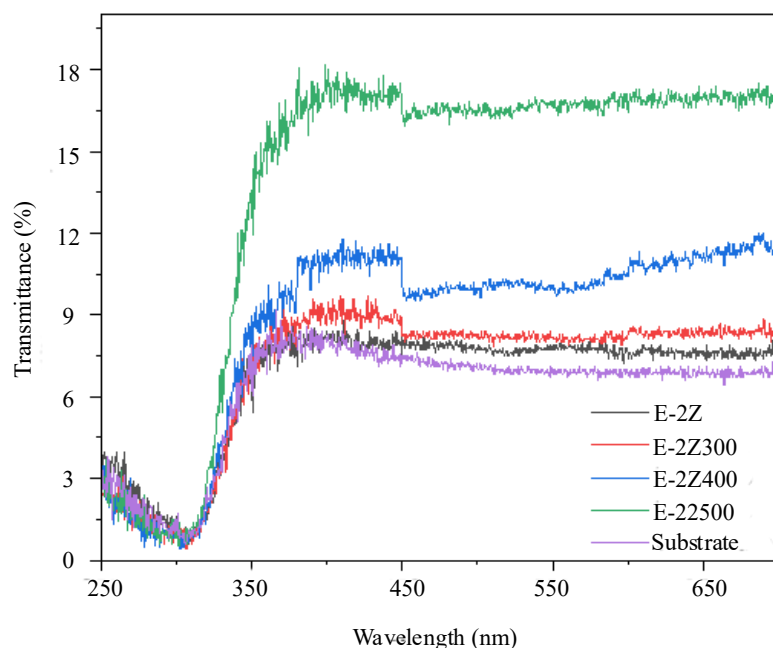


Figure 4. Effect of heat treatment at different temperatures (300-500 °C) on optical transmittance of epoxy-ZrO₂ coatings.

CONCLUSIONS

This study provides strong evidence that epoxy-ZrO₂ coatings exhibit excellent thermal stability, maintaining structural integrity and hydrophobic surface properties even at temperatures as high as 500 °C. The coatings showed minimal changes in their physical and chemical characteristics, with contact angle values consistently around 75°, confirming resilience against thermal degradation and retention of water-repellent behavior. However, while the coatings demonstrated promising performance under short-term thermal stress, their long-term durability and corrosion resistance under real-world service conditions remain to be fully explored. Additionally, the optical transmittance, although improved with temperature, is still relatively low for applications requiring high transparency. Future research should investigate the long-term environmental stability of these coatings, including resistance to moisture, UV radiation, and corrosive agents. Moreover, optimizing the nanoparticle loading and exploring hybrid fillers or surface treatments could enhance transparency and multifunctionality, broadening the scope of applications in aerospace, electronics, and protective surfaces.

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