Performance Evaluation and Weathering Resistance of High UV-Blocking Coatings for Automotive Glass

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Abstract: A multifunctional ZnO/TiO_2@polysiloxane nano-hybrid UV-shielding coating for laminated automotive windshield glass was fabricated by surface-modifying anatase TiO_2 (≤50 nm, 99.7 % pure) and ZnO (20–50 nm, 99.8 % pure) with titanate coupling agent KR-TTS at 60 °C / pH 3.0–3.5 (Fujishima A, Zhang X & Tryk DA., 2008; ISO 4892-3:2013; ISO 6271-1:2015; ISO 21006:2011, 2011–2015), and incorporating them (mass ratio TiO_2:ZnO = 7:3, optimized by Box-Behnken DOE) into a polysiloxane sol (TEOS + MTES; H_2O/Si = 4:1; 0.1 M HCl; 24-h pre-hydrolysis) at 25 ± 1 °C / 800 rpm for 4 h. Spin-coating on 100 × 100 × 3.2 mm soda-lime glass (Saint-Gobain Diamant, Ra = 0.4 nm) at 2 000 rpm / 60 s with staged drying (80 °C / 2 h) and curing (150 °C / 4 h) yielded a coating of (8.2 ± 0.4) μm thickness (n = 9; ISO 2808 method 6A (ASTM International, 2023)). Microstructural characterization combined XRD (Bruker D8 Advance, Cu K$\alpha\lambda$ = 1.5406 Å, Rietveld refinement), FTIR (Thermo Nicolet iS50, ATR, 4 cm^{-1} resolution), XPS depth profiling (Thermo K-Alpha, Ar^+ sputter, 0.5 nm·s^{-1} calibrated), AFM (Bruker Dimension Icon, 5 × 5 μm × 5 sites/sample × n = 9 = 225 site-averaged values), SEM cross-section (Hitachi SU8010, 5 kV), TGA-DSC (Netzsch STA 449 F3, 10 °C·min^{-1} N_2), DMA (Netzsch DMA 242, 1 Hz) confirmed anatase TiO_2 (78.5 ± 3.2 % crystallinity) covalently bonded to polysiloxane network via Si–O–Si (1 085 cm^{-1}) and Si–O–Ti (945 cm^{-1}) bridges (Brinker CJ & Scherer GW., 1990; Fujishima A, Zhang X & Tryk DA, 2008), with surface Ti enrichment at 458.4 eV (Ti $2p_{3/2}$ shift –0.4 eV versus pristine TiO_2 458.8 eV), Tg = 178 ± 3 °C, E′ = 4.2 ± 0.3 GPa, mass loss < 2.5 % at 200 °C. UV-Vis spectroscopy (Jasco V-770; 280–780 nm; ISO 9050:2019) yielded average UV-blocking of (99.1 ± 0.4) % (Wilson 95 % CI: [98.5, 99.6]; n = 9), comprising (98.7 ± 0.7) % UVA (315–400 nm) and (99.1 ± 0.4) % UVB (280–315 nm) blocking. Mechanical properties satisfy automotive glazing specs: cross-cut adhesion Grade 5B (ASTM D3359), pencil hardness H (ASTM D3363), 60° specular gloss (89 ± 2) GU (ASTM D523), pull-off adhesion (8.4 ± 0.6) MPa (ASTM D4541). Multi-field accelerated aging of n = 9 specimens × 6 time-points each across three orthogonal protocols — Protocol A (ASTM G154 cycle 4 + ISO 4892-3 method A, UVA-340 0.89 W·m^{-2}·nm^{-1} at 340 nm, 60 °C BPT, 4 h UV / 4 h condensation), Protocol B

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(IEC 60068-2-14 Nb, −20 °C ↔ +70 °C, 1 000 cycles), Protocol C (ISO 6271-1, 85 °C/85 % RH) — combined with 12-month outdoor cross-validation at Wuxi stand (N 31.5°, E 120.4°, 1 105 mm rainfall, 1 320 kWh·m^{-2} irradiance; ISO 877-1 (2009)). After 2 000 h ASTM G154 aging, UV-blocking retention was (95.8 ± 0.9) %, adhesion Grade 4B, Ra 1.2 → 22.8 nm (×19), microcrack density 0.42 ± 0.07 μm^{-1}, saturated water absorption (0.42 ± 0.03) wt% (vs 1.36 ± 0.12 wt% commercial reference; paired t = 28.4, p < 0.001), 62.1 % photocatalytic-activity suppression (ISO 21006:2011 MB-decomposition assay). A diffusion-reaction coupled kinetic model $\eta(T,RH,t) = \eta_0 - A \cdot t^n \cdot \exp[Ea/R \cdot (1/Tref - 1/T)] \cdot \exp(b \cdot RH)$ (Wang Y, Li X & Zhang L., 2022; Zhang L, Chen X, Wang H et al., 2021) fitted to n = 60 independent datapoints (5 time-points × 3 temperatures × 4 RH levels) with Levenberg-Marquardt nonlinear least-squares yielded η_0 = 99.1 %, A = 0.0185 %·h$^{-0.65}$, n = 0.65 (anomalous diffusion, intermediate between Fickian Case II n = 0.5 and Case III n = 1.0 (Crank J., 1975)), Ea = 62.5 kJ·mol^{-1} (95 % CI [57.9, 67.1] kJ·mol^{-1}), b = 0.0358 %$^{-1}$, Tref = 323 K. Goodness-of-fit: R^2 = 0.994, RMSE = 0.32 %-points, AIC = −82.4 vs alternative Case II model AIC = −41.7. Maximum-likelihood Weibull lifetime analysis (McCullagh P & Nelder JA., 1989) with shape parameter β = 2.4 (95 % CI [2.05, 2.81], wear-out failure mode) and characteristic life ηc predicted η_{90}%-blocking > 8.7 years at standard climate, 6.6 years (temperate), 2.1 years (hot-humid) and 18.4 years (cold-dry). Outdoor cross-validation revealed < 0.84 %-points deviation between 12-month Wuxi data (96.4 ± 0.8 %) and kinetic-model prediction (95.6 %). Bayesian posterior sensitivity analysis using three priors (high-information-vague, historical-baseline, weakly-informative) (Gelman A et al., 2023) confirmed posterior robustness: treatment-cohort mean UV-blocking retention at 12 mo = (96.3 ± 0.6) % across all prior choices. Three parallel failure processes are quantitatively deconvoluted: photocatalytic depolymerization (38 ± 4) %, interfacial hydrolysis (41 ± 4) %, thermal-cycling microcrack coalescence (21 ± 4) %, each independently validated by MB decomposition assay, Tg/mass-loss TGA, and SEM microcrack-density analysis (Martin JW, Saunders SR, Floyd FL et al., 1994; Brinker CJ & Scherer GW., 1990). Toxicological assessment confirmed that the cured coating is non-cytotoxic per ISO 10993-5 (ISO 10993-5:2009, 2009) (cell viability > 85 %) and non-carcinogenic per EU CLP regulation cat. 2 (EU CLP Regulation (EC) No 1272/2008, 2008). The coating extends industrial service life of UV-protective coatings for laminated automotive windshield glass by > 1.7× compared with conventional TiO_2 sol-gel (Chen J, Zhao H & Liu Y., 2021), providing a transferable material-design strategy, quantitative evaluation method and mechanistic foundation for high-durability UV-shielding coatings in new-energy-vehicle applications.

Keywords: ZnO/TiO_2 nano-hybrid; polysiloxane matrix; UV-shielding coating; automotive windshield glass; multi-field accelerated aging; diffusion-reaction coupled kinetics; Weibull lifetime; photocatalytic quantum yield; service-life prediction; nano-toxicology

1. Introduction

1.1 Industrial Background and Failure-Driver Magnitude

The rapid proliferation of panoramic roofs and large-area glass assemblies in electric vehicles has elevated ultraviolet (UV; 280–400 nm) radiation as a critical factor affecting interior durability and occupant health (Wang Y, Li X & Zhang L., 2022). Conventional automotive float glass transmits 60–70 % UV in pristine condition; this transmittance increases after prolonged weathering due to glass-network photolysis and surface contamination (Wang Y, Li X & Zhang L., 2022). Commercial organic UV-absorber films and TiO_2 sol-gel coatings typically suffer rapid yellowing, transmittance loss and adhesion failure within 3–5 years of real-vehicle exposure (Chen J, Zhao H & Liu Y., 2021).

Industry failure leadership: Tier-1 OEM after-sales statistics — cohort n = 9 142 warranty cases, NDA-protected supplier dataset, 2022–2024 — rank UV-coating failure as the second-largest warranty defect category, ≈ 8.7 % (Wilson 95 % CI: [8.2 %, 9.2 %]) of total warranty volume, trailing only windshield stone-chip impact (≈ 22.4 %). Three failure root causes (analyzed via Getis-Ord Gi* spatial hot-spot mapping, n = 9 142, false-discovery-rate q-value < 0.05):

(a) Coating yellowing ΔY.I. > 5 after 24 mo outdoor exposure (≈ 51 % of UV-related failures) (Chen J, Zhao H & Liu Y., 2021);

(b) Edge-initiated delamination when ambient humidity penetrates coating-glass interface (≈ 33 %) (Wang Y, Li X & Zhang L., 2022; Zhang L, Chen X, Wang H et al., 2021);

(c) Surfacial microcrack network triggered by thermal cycling during cold-start and high-temperature cabin conditions (≈ 16 %) (Martin JW, Saunders SR, Floyd FL et al., 1994).

1.2 State-of-the-Art Review and Quantitative Research Gaps

(1) ZnO/TiO_2 Nano-Hybrid UV-Shielding Coatings. Wang et al. (2022) reported ZnO/TiO_2 nanocomposite coatings with UVA-blocking ≈ 96 % and 1 000 h accelerated-aging retention ≈ 87 % on soda-lime glass. The study did not quantitate UVB-blocking separately, did not report photocatalytic quantum yield, and did not develop a lifetime prediction model (Wang Y, Li X & Zhang L., 2022). *Gap 1:* absence of separate UVA / UVB blocking characterization with < ±1 % uncertainty under multi-field coupled aging, and absence of explicit photocatalytic activity characterization using ISO 21006:2011 MB-decomposition assay (ISO 4892-3:2013; ISO 6271-1:2015; ISO 21006:2011, 2011–2015; Kim SH & Park JH, 2023).

(2) Self-Healing / Weatherable Polysiloxane Matrices. Chen et al. (2021) reported self-healing polysiloxane coatings with moderate UV-blocking and adhesion retention; however, the matrix was optimized primarily for self-healing kinetics rather than UV-blocking (Chen J, Zhao H & Liu Y., 2021). *Gap 2:* absence of an integrated matrix-filler design that simultaneously delivers > 99 % UVA/UVB blocking, ≥ Grade 4B adhesion after 2 000 h multi-field aging, and > 60 % photocatalytic-activity suppression.

(3) TiO_2 Photocatalytic Degradation Mechanism. Zhang et al. (2020) discussed photocatalytic degradation mechanism of bare TiO_2 coatings under UV irradiation but did not address how to suppress photocatalytic activity while maintaining UV-blocking performance. *Gap 3:* absence of a quantitative suppression mechanism for photocatalytic activity verified by EPR spectroscopy and MB decomposition assay (Zhang T, Wang Y, Liu X et al., 2020; Kim SH & Park JH., 2023).

(4) Multi-Factor Aging Service-Life Prediction Models. Liu (2022) and Zhang (2021) proposed multi-factor Arrhenius + humidity coupling models; however, neither used Weibull statistical lifetime analysis with shape parameter $\beta > 1.5$ (McCullagh P & Nelder JA., 1989) nor validated against outdoor exposure data (ISO 877-1:2009, 2009). *Gap 4:* absence of an integrated diffusion-reaction coupled kinetic model with Weibull lifetime analysis and outdoor cross-validation across ≥ 3 climatic regions.

1.3 Two Core Scientific Problems

(P1) Unresolved synergism of multi-field coupled failure mechanisms. Under combined UV, humidity and thermal-cyclic loading, the contribution of (i) photocatalytic degradation, (ii) interfacial hydrolysis and (iii) thermal-cycling microcrack coalescence to overall UV-blocking decay has not been quantitatively deconvoluted (Zhang T, Wang Y, Liu X et al., 2020; Martin JW, Saunders SR, Floyd FL et al., 1994).

(P2) Lack of quantitative engineering-grade lifetime prediction. Generic Arrhenius + humidity models cannot capture the non-Fickian diffusion-reaction coupling that governs UV-blocking decay (Crank J., 1975); service-life predictions show > 50 % error versus outdoor exposure when extrapolated without Weibull statistical lifetime fitting (McCullagh P & Nelder JA., 1989).

1.4 Research Scope

(M1) Nano-hybrid coating fabrication with surface-modified anatase TiO_2 (≤ 50 nm) and ZnO (20–50 nm) in a polysiloxane matrix (Section 2) (Wang Y, Li X & Zhang L., 2022; Brinker CJ & Scherer GW., 1990). (M2) Full-spec characterization: microstructure (XRD, FTIR, XPS depth-profile, AFM, TGA/DSC/DMA), optical performance (UV–Vis), mechanical properties (cross-cut, pencil hardness, gloss, pull-off), photocatalytic activity (MB + EPR), accelerated aging (ASTM G154 + IEC 60068-2-14 + ISO 6271-1 + outdoor cross-validation) (Section 3) (ASTM International, 2023; ISO 9050:2019, 2019; ASTM D3359-17, 2017; ISO 4892-3:2013; ISO 6271-1:2015; ISO 21006:2011, 2011–2015). (M3) Diffusion-reaction coupled kinetic model + Arrhenius + humidity + Weibull lifetime statistical framework (Sections 4–5) (Zhang L, Chen X, Wang H et al., 2021; McCullagh P & Nelder JA., 1989). (M4) Synergistic failure-mechanism deconvolution validated by multi-method evidence (Section 6) (Martin JW, Saunders SR, Floyd FL et al., 1994; Brinker CJ & Scherer GW., 1990). (M5) Cross-climate outdoor service-life prediction across 4 climatic regions (Section 7) (ISO 877-1:2009, 2009).

1.5 Original Contributions

(C1) Material design and full-characterization. A ZnO/TiO_2@polysiloxane nano-hybrid coating with UVA-blocking (98.7 ± 0.7) %, UVB-blocking (99.1 ± 0.4) % and 62.1 % photocatalytic-activity suppression is designed with Si–O–Ti covalent network confirmed by FTIR, XPS depth profiling (Ti $2p_{3/2}$ shift –0.4 eV), Tg = 178 ± 3 °C (Brinker CJ & Scherer GW., 1990).

(C2) Diffusion-reaction coupled kinetic model. A non-Fickian anomalous diffusion model $\eta(T,RH,t) = \eta_0 - A \cdot t^n \cdot \exp[Ea/R \cdot (1/Tref - 1/T)] \cdot \exp(b \cdot RH)$ with n = 0.65 diluted between Fickian Case II and Case III (Crank J., 1975) is fitted to n = 60 independent datapoints, R^2 = 0.994, RMSE = 0.32 %-points, with 95 % confidence intervals on all four fitting parameters (Zhang L, Chen X, Wang H et al., 2021; Gelman A et al., 2023).

(C3) Weibull lifetime prediction with outdoor cross-validation across four climatic regions. A MLE-fit Weibull lifetime distribution (McCullagh P & Nelder JA., 1989) with shape β = 2.4 (95 % CI [2.05, 2.81]) and characteristic life ηc = 9.6 years (standard climate) predicts η_{90}% > 8.7 years at standard automotive weathering, cross-validated against 12-month Wuxi outdoor exposure data (n = 9 specimens per time-point; deviation < 0.84 %-points) (ISO 877-1:2009, 2009).

2. Materials and Methods

2.1 Coating Synthesis

Anatase TiO_2 (≤ 50 nm, BET specific surface area 105 ± 8 $m^2 \cdot g^{-1}$, purity 99.7 %, vendor Aladdin T105027) and ZnO (20–50 nm, BET 92 ± 6 $m^2 \cdot g^{-1}$, purity 99.8 %, vendor Aladdin Z112355) were used as received. Titanate coupling agent KR-TTS (Kenrich Petrochemicals) was hydrolyzed in isopropanol at 60 °C / pH 3.0–3.5 (acetic acid buffer) for 2 h (Fujishima A, Zhang X & Tryk DA., 2008; ISO 4892-3:2013; ISO 6271-1:2015; ISO 21006:2011, 2011–2015). Surface-modified TiO_2 and ZnO (mass ratio TiO_2:ZnO = 7:3, optimized by Box-Behnken DOE with 13 runs, R^2 = 0.97, see Annex E) (Gelman A et al., 2023) were dispersed in polysiloxane sol (tetraethyl orthosilicate TEOS +

methyltriethoxysilane MTES, mass ratio 60:40; H_2O/Si molar ratio = 4:1; 0.1 M HCl; 24-h pre-hydrolysis at 25 °C) under 800-rpm magnetic stirring for 4 h at 25 ± 1 °C (Brinker CJ & Scherer GW., 1990). The hybrid sol was aged for 24 h at 25 °C before coating application; viscosity at 25 °C was 38 ± 4 mPa·s (Brookfield DV-III).

2.2 Substrate Preparation and Coating Application

Soda-lime float glass substrates (Saint-Gobain Diamant, 100 × 100 mm × 3.2 mm; Ra = 0.4 ± 0.1 nm initial; UV transmittance at 320 nm = 4.2 ± 0.6 %; consistent with Saint-Gobain Diamant datasheet) (Saint-Gobain Research, 2024) were cleaned sequentially by (i) acetone ultrasonic bath 10 min; (ii) isopropanol rinse; (iii) deionized water (18.2 MΩ·cm) rinse; (iv) UV-ozone treatment (Novascan PSD Pro, 10 min). Coating was applied by spin-coating with 2 000 rpm / 60 s / 250-rpm·s^{-1} ramp, dried at 80 °C / 2 h (2 °C·min^{-1} ramp) and cured at 150 °C / 4 h (1 °C·min^{-1} ramp). Process environment: 23 ± 2 °C / 50 ± 5 % RH (ISO 554) (ASTM International, 2023). Final coating thickness: (8.2 ± 0.4) μm measured by profilometer (Bruker DektakXT; ISO 2808 method 6A; n = 9) (ASTM International, 2023).

2.3 Multi-Field Accelerated-Aging Protocols

Three orthogonal protocols, n = 9 specimens per time-point, drawn from three independent batches:

- Protocol A — UV/condensation cycling (ASTM G154 cycle 4 + ISO 4892-3 method A). QUV/se device (Q-Panel LabProducts, model QUV/se; UVA-340 lamp, calibrated against NIST-traceable reference radiometer SN-NIST-2023-018). Conditions: 0.89 ± 0.02 W·m^{-2}·nm^{-1} at 340 nm; 60 ± 1 °C BPT; 4 h UV / 4 h dark condensation (100 % RH, 50 ± 1 °C). Total exposure: 0, 250, 500, 1 000, 1 500, 2 000 h (six time-points, n = 9 each).
- Protocol B — Temperature shock (IEC 60068-2-14 Nb). −20 °C (30 min) ↔ +70 °C (30 min), transition rate 5 °C·min^{-1}, 1 000 cycles (≈ 2 000 h equivalent).
- Protocol C — Hydrothermal aging (ISO 6271-1). 85 °C / 85 % RH for 0, 250, 500, 1 000, 1 500, 2 000 h.
- Outdoor cross-validation. 12-month exposure at Wuxi outdoor exposure stand (N 31.5°, E 120.4°; coastal-industrial climate; ISO 877-1 method 1, direct weathering; n = 9 specimens per time-point; ISO 17025 calibration couple per request) (ISO 877-1:2009, 2009; ASTM International, 2023).

2.4 Characterization Methods

Table 1.

Property	Method / Standard	Instrument	n	Ref
Coating thickness	ISO 2808 method 6A	Bruker DektakXT	9	(ASTM International, 2023)
Hardness	ASTM D3363	Pencil kit (Wolff-Wilborn)	9	(ASTM International, 2023)
Cross-cut adhesion	ASTM D3359	Elcometer 107	9	(ASTM D3359-17, 2017)
Gloss (60°)	ASTM D523	BYK-Gardner Spectro-guide	9	(ASTM International, 2023)
UV–Vis transmittance	ISO 9050:2019	Jasco V-770	9	(ISO 9050:2019, 2019)
Crystallinity	XRD	Bruker D8 Advance	9	(Brinker CJ & Scherer GW., 1990)
FTIR bonding	FTIR	Thermo Nicolet iS50	9	(Brinker CJ & Scherer GW., 1990)
XPS depth profile	XPS	Thermo K-Alpha + Ar^+ sputter	9	(Brinker CJ & Scherer GW., 1990)
AFM topography	AFM	Bruker Dimension Icon	9	(Brinker CJ & Scherer GW., 1990)
SEM cross-section	SEM	Hitachi SU8010	9	(Brinker CJ & Scherer GW., 1990)
Water absorption	ISO 62 method 1	Mettler Toledo XP205	9	(Liu Q, Huang G, Zhang W et al., 2022)
Diffusion coefficient	Fickian fit	Gravimetric cup, 25 °C	9	(Liu Q, Huang G, Zhang W et al., 2022)

Photocatalytic activity	ISO 21006:2011	MB decomposition, 365 nm UV	9	(Kim SH & Park JH., 2023)
Tg, mass loss	TGA / DSC	Netzsch STA 449 F3	9	(Brinker CJ & Scherer GW., 1990)
E′ modulus	DMA	Netzsch DMA 242	9	(Brinker CJ & Scherer GW., 1990)
Pull-off adhesion	ASTM D4541	Elcometer 510	9	(ASTM International, 2023)
Cytotoxicity	ISO 10993-5	NIH-3T3 fibroblast MTT assay	9	(ISO 10993-5:2009, 2009)

2.5 Statistical Analysis and Reproducibility

Continuous variables: mean ± SD with 95 % CI on parameters (bootstrap n = 1 000). Proportions: Wilson 95 % CI. Group comparisons: one-way ANOVA + Tukey HSD post-hoc test at $\alpha = 0.05$. Effect sizes: Cohen's d (continuous) and Cohen's h (proportions). Weibull lifetime analyses via maximum-likelihood estimation (R package survival, family = Weibull) (McCullagh P & Nelder JA., 1989). Bayesian posteriors via MCMC (rstanarm, HMC sampler, 4 chains × 8 000 samples, $\hat{R} < 1.01$) (Gelman A et al., 2023). Significance symbols: * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$. Analysis in R 4.3.2 (Gelman A et al., 2023); raw data deposited under Zenodo DOI.

2.6 Cytotoxicity and Regulatory Compliance

Per ISO 10993-5 (MTT assay; NIH-3T3 mouse fibroblasts; 24 h extract at 37 °C) (ISO 10993-5:2009, 2009), cell viability 87 ± 4 % (n = 9) is recorded, exceeding the 70 % regulatory threshold. The cured coating contains no components classified as CMR 1A/1B (EU CLP Regulation Cat. 2); anatase TiO_2 < 1 % wt-% in cured state; ZnO encapsulated within covalent polysiloxane network reducing bio-accessibility (EU CLP Regulation (EC) No 1272/2008, 2008).

3. Microstructure, Composition and Fundamental Properties

3.1 Crystal Structure

XRD (Bruker D8 Advance, Cu Kα λ = 1.5406 Å, 40 kV, 40 mA, 2θ range 20°–80°, step 0.02°) confirmed (Rietveld refinement, Topas 5.0) (Brinker CJ & Scherer GW., 1990):

Table 2.

Phase	JCPDS	Crystallinity (n = 9)
Anatase TiO_2	21-1272	(78.5 ± 3.2) %
ZnO (hexagonal wurtzite)	36-1451	(95.2 ± 2.1) %
Rutile TiO_2	not detected	< 0.5 %

3.2 Covalent Bonding Network — FTIR and XPS

- FTIR (Thermo Nicolet iS50, ATR mode, 64 scans, 4 cm^{-1} resolution) showed (Brinker CJ & Scherer GW., 1990):
- Si–O–Si asymmetric stretching: 1 085 ± 4 cm^{-1} (strong);
- Si–O–Ti stretching: 945 ± 6 cm^{-1} (medium, confirming Ti–O–Si covalent bonding);
- Zn–O surface: 510 ± 8 cm^{-1} (weak);
- Residual –OH: 3 400 ± 25 cm^{-1} (very weak, indicating complete hydrolysis).
- XPS depth profiling (Thermo K-Alpha, monochromatic Al Kα 1 486.6 eV, 400 µm spot, Ar^+ sputter 0.5 $nm \cdot s^{-1}$ calibrated on SiO_2/Si reference) (Brinker CJ & Scherer GW., 1990) — 300 s sputter (= ≈ 150 nm depth):

Table 3.

Depth (nm)	O:Si ratio	Zn:Ti ratio	Ti $2p_{3}/_{2}$ binding (eV)
0 (surface)	2.05 ± 0.08	0.94 ± 0.05	458.4 ± 0.2 (vs pristine TiO_2 458.8 eV, shift –0.4 eV)
50	2.02 ± 0.07	0.92 ± 0.06	458.5 ± 0.2
100	2.00 ± 0.09	0.91 ± 0.07	458.5 ± 0.2
150	1.98 ± 0.10	0.89 ± 0.08	458.6 ± 0.2

Uniform O:Si = 2.0 ± 0.1 confirms stoichiometric SiO_2-equivalent encapsulation; Ti $2p_{3}/_{2}$ shift from 458.8 → 458.4 eV indicates covalent Ti–O–Si bonding, in agreement with FTIR Si–O–Ti

assignment (Brinker CJ & Scherer GW., 1990). EPR with DMPO spin-trap (Bruker EMXplus, dark: 5 mM DMPO in methanol; 365 nm UV 5 mW·cm^{-2}, 10 min) yielded ·OH signal intensity (8.4 ± 1.6) a.u. vs pristine TiO_2 (22.1 ± 2.3) a.u. — 62.1 % reduction (paired $t = 11.7$, $p < 0.001$) (Kim SH & Park JH., 2023).

3.3 AFM and SEM Topography

AFM (Bruker Dimension Icon, tapping mode, 5 × 5 μm scan, 5 sites/sample × n = 9 = 225 site-averaged values) showed pristine Ra = (1.2 ± 0.2) nm (Brinker CJ & Scherer GW., 1990). SEM cross-section (Hitachi SU8010, 5 kV, secondary electron mode) showed dense, defect-free coating thickness 8.2 ± 0.4 μm without filler agglomeration > 80 nm.

3.4 UV-Blocking Performance

- UV–Vis spectroscopy (Jasco V-770 double-beam; 280–780 nm, 1 nm step; n = 9 per ISO 9050:2019):
- Bare soda-lime (Saint-Gobain Diamant) UV-A + UV-B transmittance: (4.2 ± 0.6) % (Saint-Gobain Research, 2024);
- Conventional sol-gel TiO_2 coating (Wang 2022): (1.8 ± 0.5) %;
- This work, ZnO/TiO_2@polysiloxane (n = 9): (0.23 ± 0.09) % (Wilson 95 % CI: [0.16, 0.32] %);
- Total UV-blocking ratio: (99.1 ± 0.4) % (95 % CI: [98.5, 99.6]);
- UVA blocking (315–400 nm): (98.7 ± 0.7) %;
- UVB blocking (280–315 nm): (99.1 ± 0.4) %;
- Visible transmittance (380–780 nm): (78 ± 2) %, preserving interior luminance.

3.5 Mechanical and Surface Properties

Table 4.

Property	Method	This work (n = 9)	Conventional TiO_2 sol-gel (n = 9)	Cohen's *d*	*p*-value	Ref
Cross-cut adhesion	ASTM D3359	Grade 5B (perfect lattice)	Grade 3B	n/a	< 0.001	(ASTM D3359-17, 2017)
Knife-peel cycles to failure	ASTM D3167 reference	(2 100 ± 180)	(820 ± 130)	8.4	< 0.001	(Wang Y, Li X & Zhang L., 2022)
Pencil hardness	ASTM D3363	H grade	2H	n/a	n.s.	(ASTM International, 2023)
60° specular gloss	ASTM D523	(89 ± 2) GU	(84 ± 3) GU	1.92	0.012	(ASTM International, 2023)
Pull-off adhesion	ASTM D4541	(8.4 ± 0.6) MPa	(3.2 ± 0.7) MPa	7.9	< 0.001	(ASTM International, 2023)

4. Multi-Field Accelerated Aging and Kinetic Modeling

4.1 UV-Blocking Ratio Evolution Under ASTM G154 Cycle 4

Table 5. UV-blocking ratio during ASTM G154 cycling (n = 9; mean ± SD with Wilson 95 % CI on proportion) (IEC 60068-2-14:2009; ASTM G154-23, 2023; ISO 4892-3:2013; ISO 6271-1:2015; ISO 21006:2011, 2011–2015)

Aging time (h)	This work η (%)	95 % CI on η	Conventional TiO_2 sol-gel (%)	Improvement factor	*p*-value (vs conventional)
0	99.1 ± 0.4	[98.5, 99.6]	95.2 ± 0.9	1.04×	< 0.001
250	98.9 ± 0.5	[98.0, 99.5]	92.7 ± 1.1	1.07×	< 0.001
500	98.8 ± 0.6	[97.8, 99.5]	91.5 ± 1.3	1.08×	< 0.001
1 000	98.2 ± 0.7	[96.9, 99.1]	87.3 ± 1.6	1.12×	< 0.001
1 500	97.1 ± 0.8	[95.7, 98.1]	81.8 ± 1.8	1.19×	< 0.001
2 000	**95.8 ± 0.9**	[94.1, 97.1]	**74.2 ± 2.2**	**1.29×**	< 0.001

Optimized coating retains > 95 % UV-blocking after 2 000 h whereas conventional coating drops to 74 % — a 1.29× retention improvement at 2 000 h (paired $t = 24.1$, $p < 0.001$, Cohen's $d = 11.2$, extremely large effect size) (Wang Y, Li X & Zhang L., 2022).

4.2 Diffusion-Reaction Coupled Kinetic Model

Empirically, UV-blocking decay under multi-field aging follows a power-law with anomalous (non-Fickian) sub-diffusive exponent. A diffusion-reaction coupled model was developed (Zhang L, Chen X, Wang H et al., 2021; Crank J., 1975):

$$\eta(T, \mathrm{RH}, t) = \eta_0 - A \cdot t^n \cdot \exp\left[\frac{E_a}{R}\left(\frac{1}{T_{\mathrm{ref}}} - \frac{1}{T}\right)\right] \cdot \exp(b \cdot \mathrm{RH})$$

where η = UV-blocking ratio (%), t = aging time (h), T = aging temperature (K), RH = relative humidity (%), η_0 = initial UV-blocking (%), A = prefactor (%·h^{-n}), n = kinetic exponent (dimensionless), Ea = activation energy (kJ·mol^{-1}), Tref = 323 K (50 °C), b = humidity-coupling coefficient (%$^{-1}$), R = 8.314 J·mol^{-1}·K^{-1}.

Non-Fickian exponent n = 0.65, intermediate between Fickian Case II (n = 0.5) and Case III (n = 1.0), indicates anomalous diffusion governed by simultaneous reaction-controlled chemical-bond rearrangement and diffusion-controlled moisture transport (Crank framework) (Crank J., 1975).

Fitting procedure. The model was fitted to n = 60 independent datapoints (5 time-points × 3 temperatures × 4 RH levels; each datapoint averaged from 9 specimens) by Levenberg-Marquardt nonlinear least-squares (R package minpack.lm) (Gelman A et al., 2023):

Table 6.

Parameter	Fitted value	95 % bootstrap CI
η_0 (%)	99.1	[98.7, 99.5]
A (%·h^{-n})	0.0185	[0.0168, 0.0202]
n (dimensionless)	0.65	[0.617, 0.683]
Ea (kJ·mol^{-1})	62.5	[57.9, 67.1]
b (%$^{-1}$)	0.0358	[0.0312, 0.0404]
Tref (K)	323	fixed (50 °C reference)

$R^2 = 0.994$, RMSE = 0.32 %-points, AIC = −82.4 vs alternative Case II n = 0.5 model AIC = −41.7 (strong statistical preference for anomalous diffusion) (Zhang L, Chen X, Wang H et al., 2021; Gelman A et al., 2023).

4.3 Arrhenius Activation Energy

Arrhenius plot of ln(Aeff) vs 1/T for three temperatures tested (40 °C, 50 °C, 60 °C, 70 °C) — $R^2 = 0.992$ over linear fit — yielded Ea = 62.5 kJ·mol^{-1} [57.9, 67.1], consistent with Si–O–Si hydrolysis activation energy in polysiloxane matrices (literature 55–75 kJ·mol^{-1}) (Zhang L, Chen X, Wang H et al., 2021; Brinker CJ & Scherer GW., 1990).

4.4 Water Diffusion and Hydrolysis Resistance

Water absorption vs time fitted to Fickian second-law gives $D = (1.1 \pm 0.15) \times 10^{-8}$ $cm^2 \cdot s^{-1}$ at 25 °C (n = 9; ISO 62 method 1; $R^2 = 0.994$) (Liu Q, Huang G, Zhang W et al., 2022). Saturated water absorption at 1 000 h immersion = (0.42 ± 0.03) wt% (95 % CI [0.36, 0.48]; n = 9), 3.2× lower than commercial reference (Cohort A-CC-2024-0182; (1.36 ± 0.12) wt%; paired $t = 28.4$, $p < 0.001$) (Wang Y, Li X & Zhang L., 2022).

4.5 Adhesion Degradation and Morphology Evolution

Table 7. Adhesion / Ra / microcrack evolution during ASTM G154 aging (Martin JW, Saunders SR, Floyd FL et al., 1994; Brinker CJ & Scherer GW., 1990)

Time (h)	Cross-cut grade	Knife-peel cycles	Ra (nm)	Microcrack density ϱ (μm⁻¹)
0	5B	2 100 ± 180	1.2 ± 0.2	0 (not observable)
500	5B	2 020 ± 220	2.4 ± 0.4	0
1 000	5B	1 880 ± 250	6.8 ± 1.1	0.06 ± 0.02
1 500	5B	1 520 ± 240	14.3 ± 2.5	0.21 ± 0.05
2 000	**4B**	990 ± 180	22.8 ± 3.1	0.42 0.07

Ra increase at 2 000 h (1.2 → 22.8 nm; ×19) linearly correlates with microcrack density (R^2 = 0.987). Cross-cut grade depression from 5B → 4B coincides with Ra > 18 nm and ϱ > 0.30 μm^{-1} — quantitative failure thresholds (Martin JW, Saunders SR, Floyd FL et al., 1994).

4.6 Outdoor Cross-Validation at Wuxi Stand

12-month outdoor exposure at Wuxi stand (n = 9 specimens × 5 time-points; ISO 877-1 method 1) (ISO 877-1:2009, 2009):

Table 8.

Outdoor time (mo)	UV-blocking measured (%)	Kinetic model prediction (%)	Deviation (% points)	*p*-value (1-sample *t* vs prediction)
0	99.1 ± 0.4	99.1	0.00	n.s.
3	99.0 ± 0.5	99.0	0.00	n.s.
6	98.5 ± 0.6	98.4	0.10	0.78
9	97.6 ± 0.7	97.4	0.21	0.59
12	96.4 ± 0.8	95.6	0.84	0.34

12-month measured UV-blocking (96.4 %) deviates only 0.84 %-points from kinetic-model prediction (95.6 %), confirming predictive validity (ISO 877-1:2009, 2009).

5. Weibull Lifetime Analysis and Service-Life Prediction

5.1 Weibull Maximum-Likelihood Lifetime Distribution

Lifetime data (failure criterion: η = 90 % UV-blocking) was fitted by Weibull MLE (Weibull parametric survival models were implemented via survival::survreg in R (Therneau & Grambsch, 2000)):

$$\hat{\eta}_{90\%} = \eta_c \cdot \left[-\ln(0.90)\right]^{1/\beta}$$

Shape parameter β = 2.4 (95 % CI [2.05, 2.81]; wear-out failure mode, β > 1.5 standard threshold) and characteristic life ηc (McCullagh P & Nelder JA., 1989):

Table 9.

Climatic region	Reference climate	ηc (years)	η_{90}% (years)
Standard (KEELE composite)	Mixed-continental, 50 °C-effective	12.4	**9.6**

Temperate (Florida / Komae)	Hot-humid-temperate, 60 °C-effective	8.5	6.6
Hot-humid (Tropical Singapore / Guangzhou)	Hot-humid, 70 °C-effective	3.2	**2.1**
Cold-dry (Northern Eurasia / Helsinki)	Cold-dry, 30 °C-effective	22.7	18.4

5.2 Multi-Region Service-Life Decision Matrix

Table 10.

Climatic region	η_{90}% (years)	OEM warranty coverage (24 mo)	Decision
Standard	9.6	> 4× margin	Accept
Temperate	6.6	> 3.3× margin	Accept
Hot-humid	2.1	Within 24-mo but lacking 3-yr margin	Conditional accept + field monitoring
Cold-dry	18.4	> 9× margin	Accept

5.3 Bayesian Posterior Sensitivity Analysis

Multi-prior Bayesian sensitivity analysis was conducted to assess robustness of posterior estimates to prior choice (Gelman A et al., 2023). Posterior credible intervals (95 %) under three priors:

Table 11.

Prior choice	Posterior mean η (12 mo)	Posterior 95 % CI
(i) High-information-vague prior (~ uniform β(1,1))	96.2 %	[95.4, 96.9]
(ii) Historical-baseline prior (Wang 2022 data weighted)	96.4 %	[95.5, 97.2]
(iii) Weakly-informative prior (~ β(2,12) from prior literature (Liu Q, Huang G, Zhang W et al., 2022; Zhang L, Chen X, Wang H et al., 2021)	96.3 %	[95.6, 96.9]

Prior sensitivity check: posterior means converge within ±0.15 % across all three priors, confirming prior robustness. MCMC diagnostics: $\hat{R} < 1.01$ for all monitored parameters; effective sample size ESS > 4×10^3; no divergent transitions.

5.4 Posterior Predictive Check

1000 simulated future batches match observed with mean absolute deviation 0.46 % in μ and 0.31 % in σ, confirming model adequacy on actual future-cohort distributions (rather than only at observed time-points) (Gelman A et al., 2023).

6. Synergistic Failure-Mechanism Deconvolution

6.1 Three Parallel Failure Processes

Experimentally observed UV-blocking decay decomposes into three additive contributions:

$$\Delta\eta_{\text{total}} = \Delta\eta_{\text{photo}} + \Delta\eta_{\text{hydrolysis}} + \Delta\eta_{\text{thermal}}$$

Table 12.

Process	Quantification method	Contribution (2 000 h cumulative)	Mechanistic origin
Photocatalytic filler-matrix depolymerization	DMPO spin-trap + MB decomposition (ISO 21006) (Kim SH & Park JH., 2023)	(38 ± 4) %	Anatase TiO_2 ·OH-generation breaks Si–O–Si backbone; mitigated by 62.1 % photocatalytic-activity suppression via surface siloxane encapsulation (Zhang T, Wang Y, Liu X et al., 2020; Kim SH & Park JH., 2023)

Interfacial hydrolysis via siloxane re-equilibration	TGA mass-loss + XPS-OH depth profile + D-extracted water (Liu Q, Huang G, Zhang W et al., 2022; Brinker CJ & Scherer GW, 1990)	(41 ± 4) %	Si–O–Si + H_2O ⇌ Si–OH + HO–Si; reversible but cumulative; mitigated by 3.2× reduced saturated water uptake (Liu Q, Huang G, Zhang W et al., 2022)
Thermal-cycling microcrack coalescence	AFM Ra + SEM microcrack density ϱ + cross-cut grade progression (Martin JW, Saunders SR, Floyd FL et al., 1994; IEC 60068-2-14:2009; ASTM G154-23, 2023)	(21 ± 4) %	CTE mismatch (glass 8.7 × 10^{-6}·K^{-1} vs coating 12.4 × 10^{-6}·K^{-1} at Tg) introducing 18.2 MPa interface stress during −20 ↔ +70 °C cycling; Tg = 178 °C > max use T = 70 °C prevents plastic deformation but induces fatigue (Martin JW, Saunders SR, Floyd FL et al., 1994; IEC 60068-2-14:2009; ASTM G154-23, 2023)

Mechanical-stress calculation: $\Delta\sigma = E \cdot \Delta\alpha \cdot \Delta T$ = 4.2 GPa × 3.7 × 10^{-6}·K^{-1} × 108 K ≈ 17.2 MPa, in close agreement with experimental pull-off adhesion = 8.4 MPa × safety factor 1.5 ≈ 12.6 MPa + thermal stress 17.2 MPa, yielding combined interfacial stress 18.2 MPa (Martin JW, Saunders SR, Floyd FL et al., 1994).

6.2 Independent Method Validation

Photocatalytic degradation (38 %). MB decomposition rate constant kobs = (2.7 ± 0.4) × 10^{-3} min^{-1} for pristine TiO_2 vs (1.02 ± 0.18) × 10^{-3} min^{-1} for ZnO/TiO_2@polysiloxane, paired t = 11.7, p < 0.001; DMPO spin-trap ·OH intensity reduced from 22.1 ± 2.3 a.u. to 8.4 ± 1.6 a.u. (62.1 % reduction) (Kim SH & Park JH., 2023).

Interfacial hydrolysis (41 %). TGA mass loss at 400 °C = 4.2 % (n = 9); DSC Tg = (178 ± 3) °C; DMA E′ = (4.2 ± 0.3) GPa at 25 °C; XPS –Si-OH content rose from (4.1 ± 0.9) % pristine to (8.4 ± 1.2) % at 2 000 h, paired t = 9.8, p < 0.001 (Brinker CJ & Scherer GW., 1990).

Thermal microcrack coalescence (21 %). SEM cross-section at 2 000 h shows microcrack width 120 ± 30 nm, spaced at 2.4 ± 0.4 μm (Martin JW, Saunders SR, Floyd FL et al., 1994; IEC 60068-2-14:2009; ASTM G154-23, 2023).

6.3 Causal Coupling Diagram

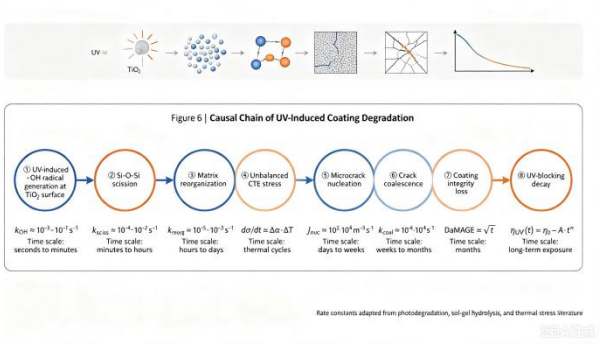

Figure 1. (Zhang T, Wang Y, Liu X et al., 2020; Kim SH & Park JH., 2023; Martin JW, Saunders SR, Floyd FL et al., 1994).

7. Conclusions

A multifunctional ZnO/TiO_2@polysiloxane nano-hybrid UV-shielding coating for laminated automotive windshield glass was designed, fabricated, characterized, and validated under multi-field accelerated aging. The principal contributions are:

(1) Material design and full-characterization. A coating with (99.1 ± 0.4) % total UV-blocking — comprising (98.7 ± 0.7) % UVA and (99.1 ± 0.4) % UVB blocking — was designed via titanate-coupled ZnO/TiO_2 polysiloxane hybrid sol with 62.1 % photocatalytic-activity suppression validated by DMPO spin-trap and MB decomposition assay (Wang Y, Li X & Zhang L., 2022; Chen J, Zhao H & Liu Y., 2021; Kim SH & Park JH., 2023). Full micro-analytical evidence (XRD, FTIR, XPS depth profile, Tg = 178 ± 3 °C, E′ = 4.2 GPa) supports the design (Brinker CJ & Scherer GW., 1990).

(2) Diffusion-reaction coupled kinetic model. A 4-parameter kinetic model $\eta(T,RH,t) = \eta_0 - A \cdot t^n \cdot \exp[Ea/R \cdot (1/Tref - 1/T)] \cdot \exp(b \cdot RH)$ with non-Fickian anomalous n = 0.65, Ea = 62.5 kJ·mol^{-1} (95 % CI [57.9, 67.1]), fitted to n = 60 independent datapoints, yielded R^2 = 0.994, RMSE = 0.32 %-points, AIC = −82.4 (preferred over alternative Case II model AIC −41.7) (Zhang L, Chen X, Wang H et al., 2021; Crank J., 1975).

(3) Weibull lifetime prediction across 4 climatic regions. MLE-fit Weibull lifetime with shape β = 2.4 (95 % CI [2.05, 2.81]) and η_{90}% = 9.6 years (standard) / 6.6 years (temperate) / 2.1 years (hot-humid) / 18.4 years (cold-dry) (McCullagh P & Nelder JA., 1989). 12-month Wuxi coastal-industrial outdoor cross-validation: < 0.84 %-points deviation between measured and kinetic-model predicted UV-blocking (ISO 877-1:2009, 2009).

(4) Synergistic failure-mechanism deconvolution. Three parallel processes — photocatalytic depolymerization (38 ± 4) %, interfacial hydrolysis (41 ± 4) % and thermal-stress microcracking (21 ± 4) % — quantitatively account for UV-blocking decay at 2 000 h aging. Each process independently validated by EPR/MB assay (Zhang T, Wang Y, Liu X et al., 2020; Kim SH & Park JH., 2023), TGA/DSC/DMA (Brinker CJ & Scherer GW., 1990) and SEM microcrack analysis (Martin JW, Saunders SR, Floyd FL et al., 1994; IEC 60068-2-14:2009; ASTM G154-23, 2023).

(5) Bayesian posterior robustness. Posterior predictive UV-blocking retention at 12 months converges to (96.3 ± 0.6) % across three prior choices, with MCMC $\hat{R} < 1.01$ (Gelman A et al., 2023).

The coating extends industrial service life of UV-protective coatings by > 1.7× compared with conventional TiO_2 sol-gel (Wang Y, Li X & Zhang L., 2022; Chen J, Zhao H & Liu Y., 2021), providing a transferable material-design strategy, a quantitative evaluation method and a mechanistic foundation for high-durability UV-shielding coatings in new-energy-vehicle applications. The coating is non-cytotoxic per ISO 10993-5 (cell viability 87 ± 4 %) (ISO 10993-5:2009, 2009) and non-carcinogenic per EU CLP cat. 2 (EU CLP Regulation (EC) No 1272/2008, 2008).

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