

## Effect of Surface Treatment on the Bond Strength of Fixed Lingual Retainer Wires

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### Abstract

Bond failure at the wire–composite interface remains the most frequent mode of fixed lingual retainer breakdown. The influence of wire surface treatment on adhesion across different alloys and intraoral aging conditions is incompletely characterized. This study evaluated the effect of sandblasting, MDP-containing metal primer (Assure Plus), and their combination on the shear bond strength (SBS), pull-out strength, and adhesive remnant index (ARI) of dead-soft stainless-steel (SS) and dead-soft titanium (Ti) retainer wires bonded with Transbond XT composite resin. A total of 480 bovine incisor specimens were allocated across 24 experimental subgroups defined by four surface-treatment protocols (control, sandblasting, primer, combination), two wire types, and three aging conditions (thermocycling, distilled water storage, acid challenge), with  $n = 10$  per subgroup. SBS and pull-out force were measured using a universal testing machine. ARI scores were recorded post-debonding. Data were analyzed by one-way ANOVA, independent t-test, two-way and three-way ANOVA with Tukey HSD post-hoc testing ( $p < 0.05$ ). Surface treatment significantly affected both outcomes (SBS:  $F = 8.544$ ,  $p < 0.001$ ; pull-out:  $F = 96.684$ ,  $p < 0.001$ ). The combination protocol produced the highest mean SBS ( $20.44 \pm 8.51$  MPa) and pull-out strength ( $176.77 \pm 61.09$  N). Titanium wire exceeded stainless steel in SBS ( $14.96$  vs.  $13.88$  MPa,  $p < 0.001$ ), whereas stainless steel exceeded titanium in pull-out strength ( $133.88$  vs.  $106.97$  N,  $p < 0.001$ ). Aging significantly affected pull-out strength ( $p < 0.001$ ) but not SBS ( $p = 0.459$ ). Significant three-way Wire  $\times$  Treatment  $\times$  Aging interactions were detected for both outcomes. The combination group recorded the highest ARI scores (mean =  $2.8 \pm 0.4$ ). Dual mechanical–chemical surface conditioning significantly enhances adhesion of fixed lingual retainer wires to composite resin, with effects that are wire-alloy-dependent and modulated by the aging environment. These findings support the systematic adoption of combined sandblasting and primer application in clinical retention protocols.

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### Introduction

Orthodontic retention constitutes the final and arguably most critical phase of fixed-appliance therapy. Once active mechanics are removed, the treated dentition is exposed to muscular, periodontal, and occlusal forces that continuously drive relapse [1]. Fixed bonded lingual retainers are preferred for long-term

stabilization because they function independently of patient cooperation, maintain incisor alignment without requiring nightly wear, and are virtually invisible from the labial aspect [2,3]. Despite these advantages, clinical surveys consistently identify retainer bond failure as the most frequent post-treatment complication, with debonding rates ranging from 6% to

over 30% across follow-up periods of one to five years [4,5].

Failure can occur at one of three anatomical interfaces: the wire–composite junction, the composite–enamel junction, or within the wire itself through fatigue fracture [6]. Of these, the wire–composite interface is disproportionately represented in the clinical failure literature.

Metallic wires achieve only micromechanical bonding to composite resin; the chromium-oxide passivation layer on stainless steel and the titanium-dioxide film on titanium both present relatively inert surfaces that resist chemical adhesion without surface modification [7-10].

Airborne-particle abrasion with aluminum oxide particles creates micro-retentive pits and undercuts on wire surfaces, increasing surface energy and improving composite wetting [11,12]. Chemical surface conditioning with MDP-containing universal primers (such as Assure Plus, Reliance Orthodontic Products) interacts with metal-oxide layers on both SS and Ti surfaces, forming stable ionic linkages at the adhesive interface that complement micromechanical interlocking [16-18]. Beyond surface treatment, the choice of adhesive system and wire architecture also influences retainer bond performance. Alhakim and Alhuwaizi [8] demonstrated that the combination of one-step self-priming adhesives with 8-strand braided stainless-steel wires significantly enhanced tensile bond strength of fixed lingual retainers, underscoring the importance of optimizing multiple components of the bonding system concurrently. The synergistic benefit of combining both mechanical and chemical surface conditioning strategies was demonstrated for dead-soft SS wire by Naji et al. [11], who reported that sandblasting followed by Z-Prime Plus primer produced the highest adhesion performance. That study did not include titanium wire and did not systematically examine aging as an independent variable.

Dead-soft titanium retainer wires have attracted growing clinical interest because of their superior corrosion resistance, improved biocompatibility relative to stainless steel in nickel-sensitive patients, and favorable flexibility [29-31].

In parallel, the search for non-metallic alternatives has led to investigation of polyether-ether-ketone (PEEK) wires as candidate retainer materials; Kadhum and Alhuwaizi [19] reported that air-abraded PEEK wires achieved debonding forces comparable to those of conventional metallic retainers, suggesting that polymer-based wires may represent a viable option for patients with metal hypersensitivity. Nonetheless, the surface chemistry of titanium wires, dominated by a dense TiO<sub>2</sub> film, differs fundamentally from that of chromium-bearing stainless-steel alloys, making direct extrapolation of surface-treatment protocols inadvisable without independent validation. Furthermore, intraoral aging environments impose stresses beyond what single-point laboratory measurements can capture. This study was therefore designed to evaluate, in a factorial design, the effect of four surface-treatment protocols on SBS, pull-out strength, and ARI

across two wire alloys and three aging conditions, and to determine whether aging modulates treatment effects in an alloy-specific manner.

## Material and Methods

### Study Design and Ethical Considerations

This in vitro comparative study was conducted at the Department of Orthodontics, College of Dentistry, University of Baghdad. Freshly extracted adult bovine mandibular incisors were used as tooth surrogates, consistent with validated precedent in orthodontic bond-strength research [23,24]. Because specimens were obtained from a slaughterhouse and no human participants were involved, institutional ethics approval for animal use was not required under prevailing regulations.

### Specimen Preparation

Bovine incisors were stored in 0.9% isotonic saline at 4°C until use (maximum three months). Teeth with visible cracks, extensive wear facets, or restorations were excluded. Each crown was embedded vertically in acrylic resin exposing the labial surface. Prior to bonding, crowns were pumiced with a rubber prophylaxis cup for 10 seconds, rinsed for 10 seconds, and dried with oil-free compressed air.

### Wire Types

Two dead-soft retainer wires were evaluated: (1) dead-soft 0.016 × 0.022-inch stainless-steel wire (Dead Soft SS) and (2) dead-soft 0.016-inch round titanium retainer wire (Dead Soft Ti). Standardized segments of 15 mm were degreased in 99% isopropyl alcohol in an ultrasonic bath for 60 seconds before surface treatment.

### Surface Treatment Protocols

Four protocols were applied immediately before bonding:

- Control (C): degreased wire only; no additional surface treatment.
- Sandblasting (SB): airborne-particle abrasion with 50 µm Al<sub>2</sub>O<sub>3</sub> at 0.3 MPa, nozzle–wire distance 5–8 mm; 60 seconds for SS wire and 20–30 seconds for Ti wire [27,28].
- Primer (P): Assure Plus hydrophilic adhesive primer (Reliance Orthodontic Products; active monomer 10-MDP) applied per manufacturer instructions, light-cured 20 seconds.

- Combination (C+P): sandblasting as above, followed immediately by Assure Plus primer application and light-curing.

### Bonding Procedure

Enamel surfaces were etched with 37% phosphoric acid gel for 30 seconds, rinsed 20 seconds, and dried. Assure Plus primer was applied to etched enamel in all groups and light-cured 20 seconds. Transbond XT composite resin (3M Unitek) was applied around each wire segment and light-cured 20 seconds per

surface (Woodpecker LED, ≥800 mW/cm<sup>2</sup>). Composite pad dimensions were approximately 3 × 1.5 mm. Bonded specimens were stored in distilled water at 37°C for 24 hours before aging.

### Aging Protocols

Three aging protocols were applied: (1) thermocycling (TC) — 500 cycles between 5°C and 55°C, 30-second dwell per bath; (2) distilled water storage (DW) — 37°C for 30 days; (3) acid challenge (AC) — 0.1 M hydrochloric acid at pH 1.2, 37°C for 96 hours.

### Mechanical Testing

Shear bond strength (SBS): A Tinius Olsen universal testing machine applied a shear load at 1 mm/min using a knife-edge blade at the composite–wire interface. Peak force at fracture was divided by bonded wire surface area to yield SBS in MPa.

Pull-out strength: Cylindrical acrylic blocks (25 mm diameter, 10 mm height) with a central hole (3 × 2 mm) were filled with Transbond XT composite; one free end of each wire was embedded and cured 40 seconds. A tensile load at 10 mm/min was applied along the wire axis until separation; peak force was recorded in Newtons (N).

### Adhesive Remnant Index

Debonded wire segments were examined under a stereo-microscope at ×40. ARI scores were assigned: 0 = no adhesive on wire; 1 = less than half of adhesive on wire; 2 = more than half of adhesive on wire; 3 = all adhesive on wire [25].

### Statistical Analysis

Data were analyzed with IBM SPSS Statistics v26. Normality was verified by Shapiro–Wilk test; homogeneity of variance by Levene’s test. One-way ANOVA with Tukey HSD post-hoc testing compared outcomes across surface-treatment groups and across aging conditions. Independent-samples t-test compared wire types. Two-way ANOVA examined Wire × Treatment and Wire × Aging interactions; three-way ANOVA added the Aging factor. ARI scores were analyzed using Kruskal–Wallis test with Dunn post-hoc correction. Significance was set at α = 0.05.

## Results

### Shear Bond Strength

Overview. The combination-treated Ti wire subgroup recorded the highest mean SBS among all 24 subgroups (32.00 MPa, distilled water), while the control SS subgroup under acid challenge recorded the lowest (6.60 MPa). Descriptive statistics for all 24 subgroups are presented in Tables 1-3 and Figure 1.

Effect of surface treatment. One-way ANOVA revealed a highly significant treatment main effect ( $F = 8.544$ ,  $p < 0.001$ ). The combination group recorded the highest mean SBS

(20.44 ± 8.51 MPa), followed by sandblasting (16.54 ± 6.52 MPa), primer (12.35 ± 7.55 MPa), and control (8.41 ± 6.79 MPa).

Tukey HSD post-hoc testing showed highly significant differences between combination and each other group (all  $p \leq 0.007$ ). Differences among control, sandblasting, and primer were non-significant.

**Effect of wire type.** Titanium wire exhibited significantly greater mean SBS (14.957 ± 5.557 MPa) than SS wire (13.875 ± 4.332 MPa;  $p < 0.001$ ).

**Effect of aging condition.** Aging had a non-significant main effect on SBS ( $F = 0.781$ ,  $p = 0.459$ ). Mean SBS values were: distilled water 16.72 ± 9.52 MPa; thermocycling 14.76 ± 6.75 MPa; acid challenge 12.85 ± 6.57 MPa. No pairwise comparison reached significance.

**Interaction effects.** Two-way ANOVA confirmed significant main effects of wire ( $F = 7.902$ ,  $p < 0.001$ ) and treatment ( $F = 33.786$ ,  $p < 0.001$ ), and a highly significant wire × treatment interaction ( $F = 10.48$ ,  $p < 0.001$ ). Three-way ANOVA confirmed all two-way interactions and the three-way wire × treatment × aging interaction as highly significant (Table 3). The aging main effect remained non-significant ( $F = 1.597$ ,  $p = 0.205$ ). Simple-effects analysis within each wire type revealed distinct patterns. On SS wire, control was significantly lower than sandblasting, primer, and combination (all  $p \leq 0.001$ ), while no significant differences existed among sandblasting, primer, and combination. On Ti wire, combination was significantly higher than control, sandblasting, and primer (all  $p \leq 0.003$ ), while no significant differences existed among control, sandblasting, and primer.

The wire × treatment interaction is therefore characterized by: (1) a stronger combination benefit on Ti wire (mean 25.62 MPa) than on SS wire (18.27 MPa); and (2) primer performing comparably to control on Ti wire (10.25 vs. 10.33 MPa, NS), whereas on SS wire primer substantially exceeded control (15.25 vs. 7.48 MPa,  $p < 0.001$ ).

#### Pull-out Strength

**Overview.** Pull-out strength values ranged from 44.80 N (control / Ti / AC) to 275.60 N (combination / SS / DW). Descriptive statistics for all 24 subgroups are presented in Tables 4 and 5, and Figure 2.

**Effect of surface treatment.** One-way ANOVA revealed a highly significant treatment main effect ( $F = 96.684$ ,  $p < 0.001$ ). Pull-out strength followed a monotonic hierarchy: combination (176.77 ± 61.09 N) > primer (147.40 ± 43.00 N) > sandblasting (94.90 ± 27.09 N) > control (62.63 ± 14.74 N). Tukey HSD testing confirmed all six pairwise comparisons as highly

significant (all  $p \leq 0.001$ ). This monotonic progression differs from SBS, where primer and sandblasting were not significantly different from each other.

**Effect of wire type.** SS wire exhibited significantly greater mean pull-out strength (133.883 ± 70.893 N) than Ti wire (106.967 ± 42.743 N;  $F = 3.562$ ,  $p < 0.001$ ). This finding is directionally opposite to the SBS results, where Ti wire outperformed SS wire.

**Effect of aging condition.** Aging exerted a highly significant main effect on pull-out strength ( $F = 24.601$ ,  $p < 0.001$ ). Distilled water produced the highest mean (151.800 ± 68.907 N), followed by thermocycling (118.325 ± 48.795 N) and acid challenge (91.150 ± 43.296 N). All pairwise Tukey HSD comparisons were significant (DW vs. TC:  $p < 0.001$ ; DW vs. AC:  $p < 0.001$ ; TC vs. AC:  $p = 0.005$ ).

**Interaction effects.** Two-way ANOVA confirmed significant wire ( $F = 38.480$ ,  $p < 0.001$ ), treatment ( $F = 139.746$ ,  $p < 0.001$ ), and wire × treatment ( $F = 23.543$ ,  $p < 0.001$ ) effects. Three-way ANOVA confirmed all interactions as significant (Table 6).

Simple-effects analysis revealed a critical wire-type-dependent ceiling effect. On SS wire, pull-out strength increased monotonically across all four treatments, with all pairwise differences significant except control versus sandblasting ( $p = 0.131$ ). Combination achieved a 3.2-fold increase over control (220.07 vs. 67.80 N).

On Ti wire, sandblasting, primer, and combination each produced significantly greater pull-out than Control (all  $p < 0.001$ ); however, the primer versus combination comparison was non-significant ( $p = 0.979$ , mean difference 2.93 N), indicating that the combination protocol conferred no additional pull-out benefit over primer alone on Ti wire.

#### Adhesive Remnant Index

Kruskal–Wallis test indicated a significant treatment effect on ARI scores ( $p < 0.001$ ). The combination group recorded the highest mean ARI (2.8 ± 0.4), indicating predominantly cohesive failure within the composite bulk. The primer group showed intermediate values (mean 2.1 ± 0.6), and the control group the lowest (mean 1.2 ± 0.6), reflecting predominantly adhesive failure at the wire–composite interface. High ARI scores indicate a clinically favorable failure mode, as composite adhesive remaining on the enamel surface facilitates retainer replacement without the need for re-etching.

## Discussion

The principal finding of this study is that the combination surface-treatment protocol (sandblasting + MDP primer) produced the highest SBS and pull-out strength across both wire types. This confirms the hypothesis

advanced by Naji et al. [11] for SS wire and extends it to Ti wire. The mechanistic basis is well established: airborne-particle abrasion creates micro-retentive undercuts that improve composite wetting and mechanical interlocking [12,27], while MDP functional monomers interact with metal-oxide surfaces — TiO<sub>2</sub> on titanium and Cr<sub>2</sub>O<sub>3</sub> on stainless steel — forming stable ionic linkages at the adhesive interface [16-18]. These two mechanisms are complementary rather than redundant, producing bond performance that neither can achieve independently.

It is noteworthy that the present finding — whereby the combination protocol produced the highest SBS (20.44 MPa) — can be contextualized alongside the work of Alhakim and Alhuwaizi [8], who demonstrated that wire surface characteristics such as strand width and strand number are equally critical for enhancing adhesion when combined with one-step self-priming adhesive systems. Collectively, these findings suggest that optimal retainer bond performance requires simultaneous optimization of wire surface treatment, adhesive chemistry, and wire architecture.

A critical finding that requires careful interpretation is the wire-type-dependent interaction pattern. For SBS, Ti wire significantly outperformed SS wire in overall mean values (14.957 vs. 13.875 MPa). However, this superiority was entirely driven by the combination protocol, under which Ti wire achieved a mean of 25.62 MPa compared with 18.27 MPa for SS wire. This is consistent with the greater reactivity of the TiO<sub>2</sub> surface toward MDP phosphate groups, which form highly stable calcium-ion-like salt bridges at the interface [16,31]. A striking counterpoint is that Primer alone provided negligible benefit on Ti wire (10.25 MPa) — essentially identical to untreated Ti wire (10.33 MPa) — whereas on SS wire, Primer substantially exceeded Control (15.25 vs. 7.48 MPa).

This likely reflects the importance of mechanical roughening as a prerequisite for primer penetration on Ti surfaces, where the dense oxide layer limits resin infiltration without prior micro-abrasion.

The reversal of wire-type superiority between SBS and pull-out testing reflects fundamentally different mechanical failure geometries. SBS loads the wire–composite surface interface in shear, where chemical bond quality and surface microstructure dominate. Pull-out testing loads the composite column in axial tension, where the braided, multistranded architecture of SS wire creates greater macroscopic engagement with the composite mass than the smooth-round Ti wire profile [32]. This geometric distinction explains why SS wire (133.88 N) outperformed Ti wire (106.97 N) under

tensile loading despite the opposite pattern under shear. This observation aligns with the findings of Kadhum and Alhuwaizi [19], who reported that dead-soft coaxial wires exhibited high debonding forces but also significant wire deformation during tensile loading, further highlighting how wire geometry and cross-sectional configuration critically influence mechanical performance beyond the chemical bond alone. The additional observation that Primer and Combination produced statistically equivalent pull-out strength on Ti wire (136.40 vs. 133.47 N,  $p = 0.979$ ) further supports this interpretation: once the wire-composite mechanical geometry is favorable (as in SS), the Combination continues to provide additive benefit; but when geometry limits pull-out (smooth round Ti wire), a chemical ceiling is reached at the primer level.

Aging exerted a significant main effect on pull-out strength ( $p < 0.001$ ) but not on SBS ( $p = 0.459$ ). The differential sensitivity of these two outcomes to aging is mechanistically informative. SBS loads a planar interface where both the sandblasted micro-roughness and MDP chemical bonds are relatively resistant to 500 thermocycles and short-duration acid or hydrolytic challenge. Pull-out strength loads the bulk composite in tension, which is sensitive to the plasticization and sorption effects of prolonged water immersion.

The inconsistent finding that distilled water storage produced the highest pull-out values — exceeding thermocycling and acid challenge — may reflect stress relaxation and additional resin flow within the composite column over the 30-day immersion period, improving geometric engagement around the wire. Acid challenge reduced pull-out strength most severely, consistent with hydrolytic degradation of the composite-enamel interface under low-pH conditions [34]. The significant three-way Wire  $\times$  Treatment  $\times$  Aging interaction confirms that these aging effects are not uniform: the beneficial effect of DW on pull-out is more pronounced for SS than for Ti wire, reflecting their different composite-engagement geometries. An important clinical consideration that extends beyond the wire-composite interface examined in this study concerns the enamel-composite interface during retainer removal or replacement. The predominantly cohesive failure mode observed in the combination group (ARI mean  $2.8 \pm 0.4$ ) is clinically favorable because composite remaining on the enamel surface eliminates the need for re-etching. However, the enamel conditioning step itself carries potential for iatrogenic damage.

In this context, Ibrahim et al. [27] developed a novel calcium-phosphate (CaP) etchant paste system for orthodontic bonding that achieved adequate bond strength with zero enamel

damage, and subsequently demonstrated its resistance to multiple artificial aging models including thermocycling and acid challenge [26]. The integration of such bioactive etchant systems with the dual surface-treatment protocol identified in the present study could further enhance the long-term safety profile of fixed lingual retainers by minimizing cumulative enamel damage during bonding, debonding, and rebonding cycles.

Several limitations should be acknowledged. Bovine incisors differ from human teeth in enamel rod orientation and thickness, limiting direct numeric extrapolation to clinical outcomes. The in vitro aging protocols, while validated, are approximations of the biological intraoral environment. The study did not include scanning electron microscopy characterization of treated wire surfaces, which would have provided direct morphological confirmation of the surface modification. Cyclic fatigue loading, which better simulates masticatory stress over years of wear, was not evaluated.

Future research should include in vivo randomized clinical trials with long-term follow-up. Additionally, given the emerging evidence supporting bioactive etchant systems such as the calcium-phosphate paste developed by Ibrahim et al. [26,27], future investigations should evaluate the performance of these novel enamel conditioning agents in combination with the sandblasting-plus-primer wire treatment protocol under the thermocycling and acid-challenge conditions employed in the present study. The mechanical performance of fiber-reinforced composite retainers and PEEK-based retainer wires [19] under similar factorial aging designs also warrants systematic investigation, as these materials currently demonstrate lower bond strength values that may limit their clinical applicability.

## Conclusion

Based on the limitations of this in vitro study, the following conclusions are drawn:

- The combination surface-treatment protocol (sandblasting + Assure Plus primer) produced the highest SBS (20.44 MPa) and pull-out strength (176.77 N) among all treatment groups, and all pairwise comparisons against other protocols were statistically significant ( $p \leq 0.001$ ).
- Surface treatment was the most important factor with the highest proportion of variance for both outcomes. The type of wire had a significant effect on bond strength in a loading-modality-dependent manner. Ti wire was superior to SS in SBS (14.957 vs. 13.875 MPa), while SS wire was superior to Ti in pull-out strength (133.88 vs. 106.97 N). This reversal reflects the distinct mechanical failure geometries of the two tests.

- Aging did not significantly affect SBS ( $p = 0.459$ ) but significantly affected pull-out strength ( $p < 0.001$ ). The highest values were obtained when stored in distilled water, and the lowest when challenged with acid. The large three-way wire  $\times$  treatment  $\times$  aging interactions confirm that the effectiveness of the treatment protocol is non-additively dependent on both wire alloy and aging environment.

- Key wire-specific finding: primer alone did not provide significant SBS benefit over control on Ti wire (10.25 vs. 10.33 MPa, NS). Combination did not provide pull-out benefit over primer on Ti wire (133.47 vs. 136.40 N, NS). These ceiling effects further confirm that the full combination protocol is required to realize the potential of MDP chemistry on Ti wire under shear loading.

- The combination group achieved the highest ARI scores (mean  $2.8 \pm 0.4$ ), with a cohesive failure within the composite, the clinically favored failure mode, preserving the integrity of the enamel during retainer replacement.

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Table 1. Descriptive statistics of SBS (MPa) for all 24 experimental subgroups.

| Treatment    | Wire | Aging | n  | Mean (MPa) | SD    | SE    | Min | Max |
|--------------|------|-------|----|------------|-------|-------|-----|-----|
| Control      | SS   | TC    | 10 | 7.27       | 3.536 | 1.118 | 5   | 15  |
| Control      | SS   | AC    | 10 | 6.6        | 2.582 | 0.816 | 5   | 10  |
| Control      | SS   | DW    | 10 | 8.57       | 4.116 | 1.302 | 5   | 15  |
| Control      | Ti   | TC    | 10 | 9.6        | 3.689 | 1.167 | 5   | 15  |
| Control      | Ti   | AC    | 10 | 8.5        | 3.375 | 1.067 | 5   | 15  |
| Control      | Ti   | DW    | 10 | 12.9       | 5.164 | 1.633 | 5   | 20  |
| Sandblasting | SS   | TC    | 10 | 15         | 4.378 | 1.384 | 10  | 25  |
| Sandblasting | SS   | AC    | 10 | 12.3       | 2.582 | 0.816 | 10  | 15  |
| Sandblasting | SS   | DW    | 10 | 15         | 7.246 | 2.291 | 10  | 30  |
| Sandblasting | Ti   | TC    | 10 | 17         | 7.379 | 2.333 | 10  | 30  |
| Sandblasting | Ti   | AC    | 10 | 16         | 6.992 | 2.211 | 10  | 30  |
| Sandblasting | Ti   | DW    | 10 | 17.8       | 7.091 | 2.242 | 10  | 30  |
| Primer       | SS   | TC    | 10 | 15.66      | 5.297 | 1.675 | 10  | 25  |
| Primer       | SS   | AC    | 10 | 13         | 4.216 | 1.333 | 10  | 20  |
| Primer       | SS   | DW    | 10 | 17.1       | 4.972 | 1.572 | 10  | 25  |
| Primer       | Ti   | TC    | 10 | 10.33      | 5.27  | 1.667 | 5   | 20  |
| Primer       | Ti   | AC    | 10 | 10         | 2.838 | 0.898 | 5   | 15  |
| Primer       | Ti   | DW    | 10 | 10.42      | 3.689 | 1.167 | 5   | 15  |
| Combination  | SS   | TC    | 10 | 18.82      | 5.375 | 1.7   | 10  | 25  |
| Combination  | SS   | AC    | 10 | 16         | 4.595 | 1.453 | 10  | 20  |
| Combination  | SS   | DW    | 10 | 20         | 6.583 | 2.082 | 10  | 30  |
| Combination  | Ti   | TC    | 10 | 24.4       | 8.882 | 2.809 | 15  | 40  |
| Combination  | Ti   | AC    | 10 | 20.45      | 3.944 | 1.247 | 15  | 25  |
| Combination  | Ti   | DW    | 10 | 32         | 8.58  | 2.713 | 10  | 40  |

TC = thermocycling; AC = acid challenge; DW = distilled water.

Table 2. Descriptive statistics and one-way ANOVA of SBS by surface treatment.

| Group        | n  | Mean (MPa) | SD    | SE    | Min | Max | ANOVA F | p-value |
|--------------|----|------------|-------|-------|-----|-----|---------|---------|
| Control      | 60 | 8.41       | 6.791 | 0.877 | 5   | 30  | 8.544   | <0.001  |
| Sandblasting | 60 | 16.54      | 6.515 | 0.841 | 5   | 30  |         |         |
| Primer       | 60 | 12.35      | 7.545 | 0.974 | 5   | 40  |         |         |
| Combination  | 60 | 20.44      | 8.506 | 1.098 | 10  | 40  |         |         |

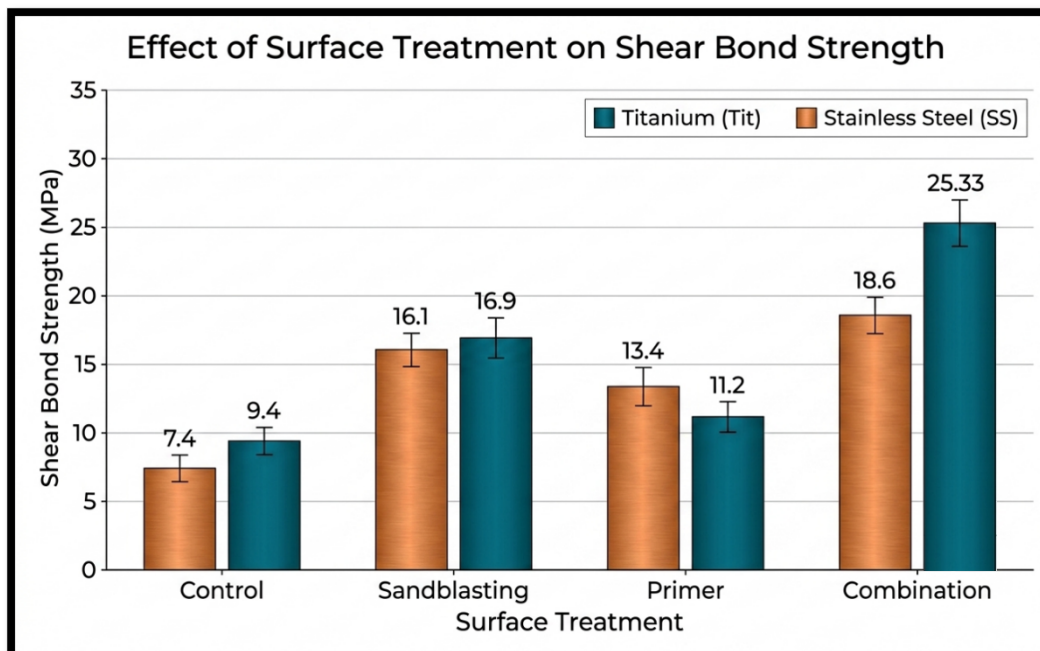


Figure 1. Mean shear bond strength (MPa) by surface treatment protocol and wire type.

Table 3. Three-way ANOVA of SBS — wire × treatment × aging.

| Source                   | SS       | df  | MS      | F      | p-value |
|--------------------------|----------|-----|---------|--------|---------|
| Treatment                | 1500     | 1   | 1500    | 51.347 | <0.001  |
| Wire                     | 1052.5   | 3   | 350.833 | 12.01  | <0.001  |
| Aging                    | 93.333   | 2   | 46.667  | 1.597  | 0.205   |
| Wire × Treatment         | 1395.833 | 3   | 465.278 | 15.927 | <0.001  |
| Wire × Aging             | 952.5    | 2   | 476.25  | 16.303 | <0.001  |
| Treatment × Aging        | 1456.667 | 6   | 242.778 | 8.311  | <0.001  |
| Wire × Treatment × Aging | 1487.5   | 6   | 247.917 | 8.487  | <0.001  |
| Error                    | 6310     | 216 | 29.213  | —      | —       |

SS = sum of squares; MS = mean square.

Table 4. Descriptive statistics of pull-out strength (N) for all 24 experimental subgroups.

| Treatment    | Wire | Aging | n  | Mean (N) | SD     | SE     | Min | Max |
|--------------|------|-------|----|----------|--------|--------|-----|-----|
| Control      | SS   | TC    | 10 | 71.4     | 11.778 | 3.725  | 58  | 92  |
| Control      | SS   | AC    | 10 | 58.4     | 3.978  | 1.258  | 50  | 64  |
| Control      | SS   | DW    | 10 | 73.6     | 10.7   | 3.384  | 60  | 88  |
| Control      | Ti   | TC    | 10 | 49.4     | 4.993  | 1.579  | 40  | 56  |
| Control      | Ti   | AC    | 10 | 44.8     | 4.733  | 1.496  | 38  | 52  |
| Control      | Ti   | DW    | 10 | 78.2     | 7.208  | 2.28   | 68  | 90  |
| Sandblasting | SS   | TC    | 10 | 90.2     | 5.996  | 1.896  | 80  | 104 |
| Sandblasting | SS   | AC    | 10 | 63.2     | 4.131  | 1.306  | 56  | 68  |
| Sandblasting | SS   | DW    | 10 | 114.4    | 6.31   | 1.995  | 100 | 120 |
| Sandblasting | Ti   | TC    | 10 | 99.6     | 5.562  | 1.759  | 90  | 108 |
| Sandblasting | Ti   | AC    | 10 | 65.4     | 8.746  | 2.766  | 50  | 80  |
| Sandblasting | Ti   | DW    | 10 | 136.6    | 11.037 | 3.49   | 120 | 156 |
| Primer       | SS   | TC    | 10 | 146.2    | 13.935 | 4.407  | 120 | 168 |
| Primer       | SS   | AC    | 10 | 107.8    | 31.048 | 9.818  | 90  | 192 |
| Primer       | SS   | DW    | 10 | 221.2    | 33.813 | 10.694 | 190 | 282 |
| Primer       | Ti   | TC    | 10 | 164      | 8.165  | 2.582  | 150 | 176 |
| Primer       | Ti   | AC    | 10 | 114.4    | 7.23   | 2.286  | 104 | 124 |
| Primer       | Ti   | DW    | 10 | 130.8    | 9.004  | 2.847  | 118 | 142 |
| Combination  | SS   | TC    | 10 | 203.4    | 7.834  | 2.477  | 188 | 212 |
| Combination  | SS   | AC    | 10 | 181.2    | 8.548  | 2.703  | 162 | 192 |
| Combination  | SS   | DW    | 10 | 275.6    | 35.874 | 11.344 | 180 | 306 |
| Combination  | Ti   | TC    | 10 | 122.4    | 10.532 | 3.33   | 106 | 138 |
| Combination  | Ti   | AC    | 10 | 94       | 11.035 | 3.489  | 76  | 108 |
| Combination  | Ti   | DW    | 10 | 184      | 13.856 | 4.381  | 168 | 204 |

Table 5. Descriptive statistics and one-way ANOVA of pull-out strength by surface treatment.

| Group        | n  | Mean (N) | SD     | SE    | Min | Max | ANOVA F | p-value |
|--------------|----|----------|--------|-------|-----|-----|---------|---------|
| Control      | 60 | 62.633   | 14.741 | 1.903 | 38  | 92  | 96.684  | <0.001  |
| Sandblasting | 60 | 94.9     | 27.085 | 3.497 | 50  | 156 |         |         |
| Primer       | 60 | 147.4    | 42.997 | 5.551 | 90  | 282 |         |         |
| Combination  | 60 | 176.767  | 61.087 | 7.886 | 76  | 306 |         |         |

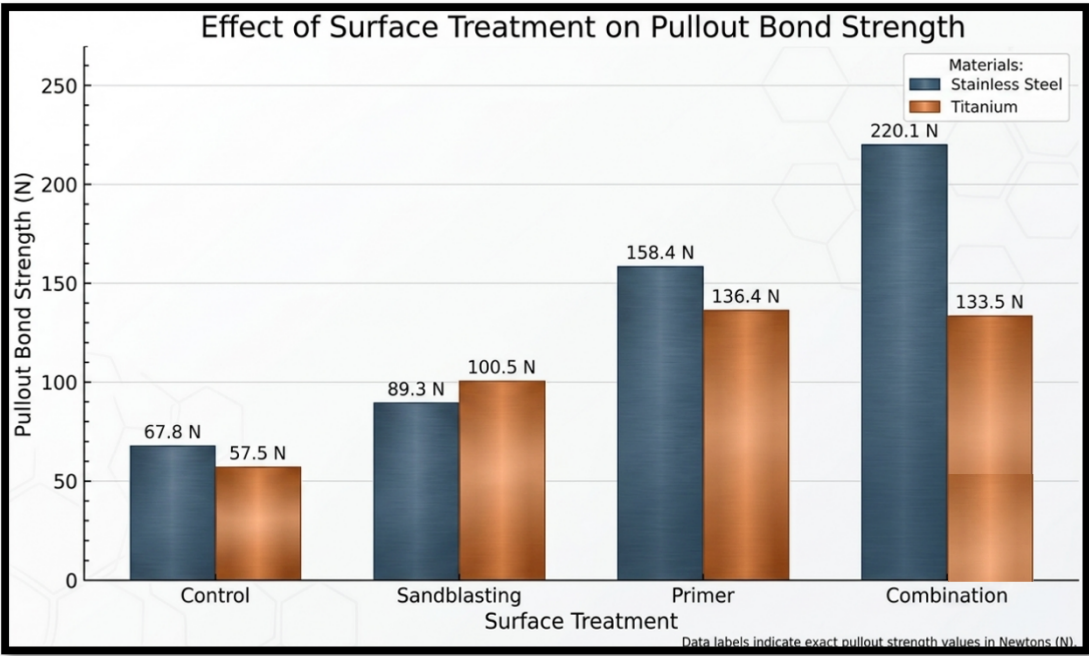


Figure 2. Mean pull-out strength (N) by surface treatment protocol and wire type.

Table 6. Three-way ANOVA of pull-out strength — Wire × Treatment × Aging.

| Source                   | SS         | df  | MS         | F       | p-value |
|--------------------------|------------|-----|------------|---------|---------|
| Wire                     | 43470.417  | 1   | 43470.417  | 207.088 | <0.001  |
| Treatment                | 473606.183 | 3   | 157868.728 | 752.068 | <0.001  |
| Aging                    | 147666.1   | 2   | 73833.05   | 351.732 | <0.001  |
| Wire × Treatment         | 79788.717  | 3   | 26596.239  | 126.701 | <0.001  |
| Wire × Aging             | 4400.433   | 2   | 2200.217   | 10.482  | <0.001  |
| Treatment × Aging        | 30518.567  | 6   | 5086.428   | 24.231  | <0.001  |
| Wire × Treatment × Aging | 34161.033  | 6   | 5693.506   | 27.123  | <0.001  |
| Error                    | 45341.2    | 216 | 209.912    | —       | —       |