Non-Cr(VI)
Surface Pre-Treatment Evaluation

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Introduction

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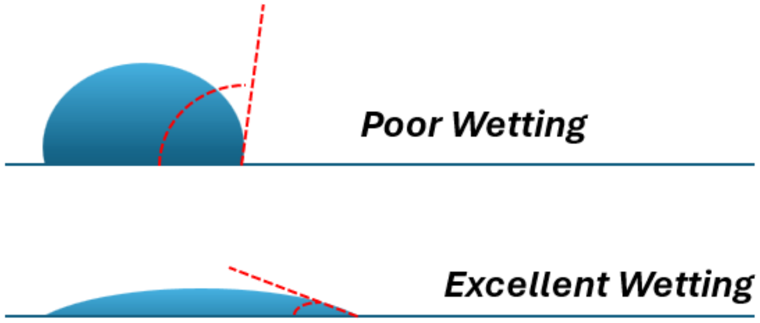
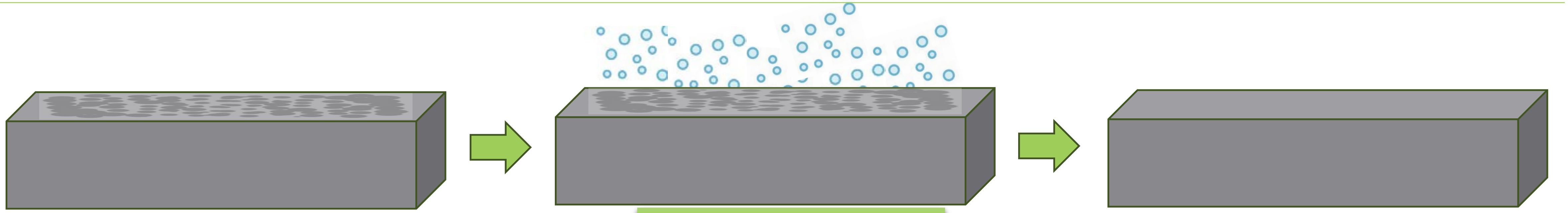
- Proper surface preparation protocols are crucial in preventing material failures and re-work
- In this study several surface pretreatments were evaluated to quantify impact via:
  - Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)
  - Surface Tensiometry
  - Water Break Free
- Insufficient surface pretreatments may cause:
  - Low Surface Free Energy (SFE)
  - Surface contaminants remaining
    - Correlates to poor adhesion, material failures and re-work



Improper surface preparation causes poor adhesion due to poor surface wetting leading to material failures and re-work

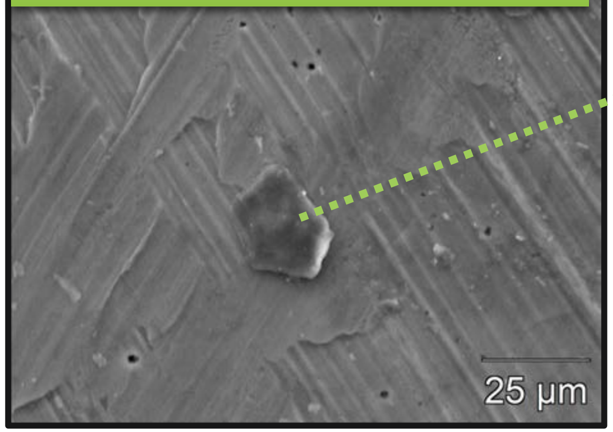
Step 1 – Wash to Decontaminate Surfaces

1. Wash → 2. Etch → 3. Anodize



Soiled Surface

SEM Microscopy



EDS Analysis

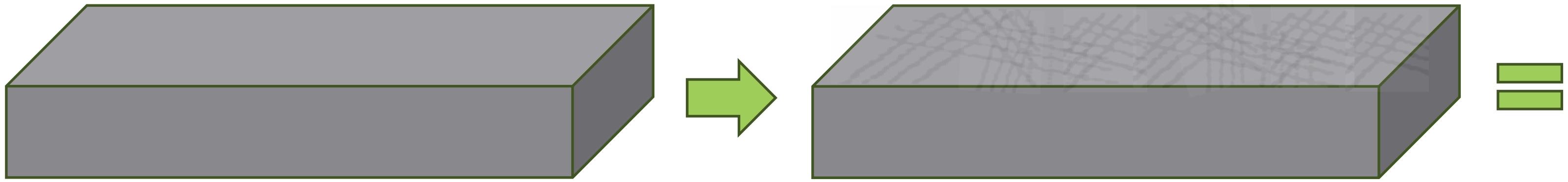
Element	Atom %
Carbon	73.83
Sodium	0.31
Magnesium	0.40
Aluminum	24.14
Silicon	0.44
Chlorine	0.43
Potassium	0.46

Contaminants Removed – Ready for Etch Step

Un-treated aluminum contains surface contaminants that disrupt bonding efficacy

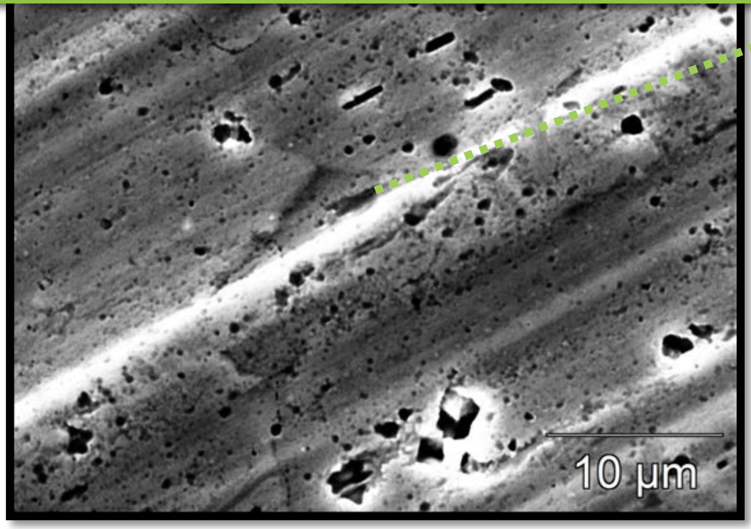
Step 2. Etch to Increase Surface Area

✓Wash → **Etch** → Anodize



Cross Section of Etched Surface

SEM Microscopy Post Acid Etch



EDS Analysis

Element	Atom %
Carbon	2.80
Magnesium	0.74
Aluminum	95.93
Oxygen	0.53

Alloying elemental residue from etching step, de-oxidize/de-smutting to remove prior to anodizing step

Mechanical or chemical etching increases surface area which is needed for effective bonding

Step 2. Etch to Increase Surface Area Continued

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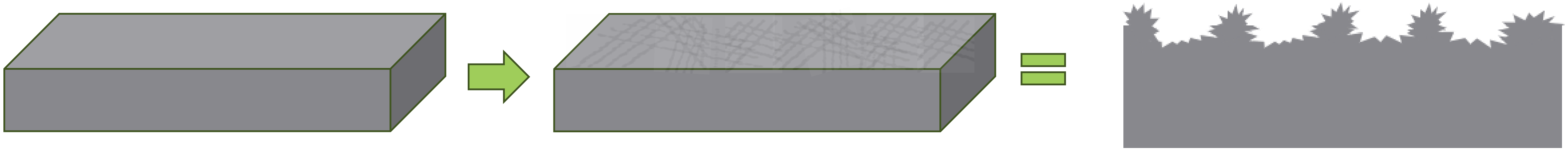
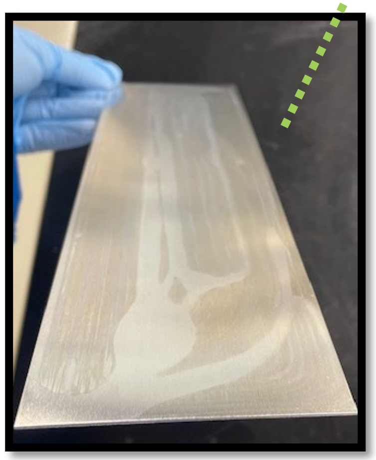
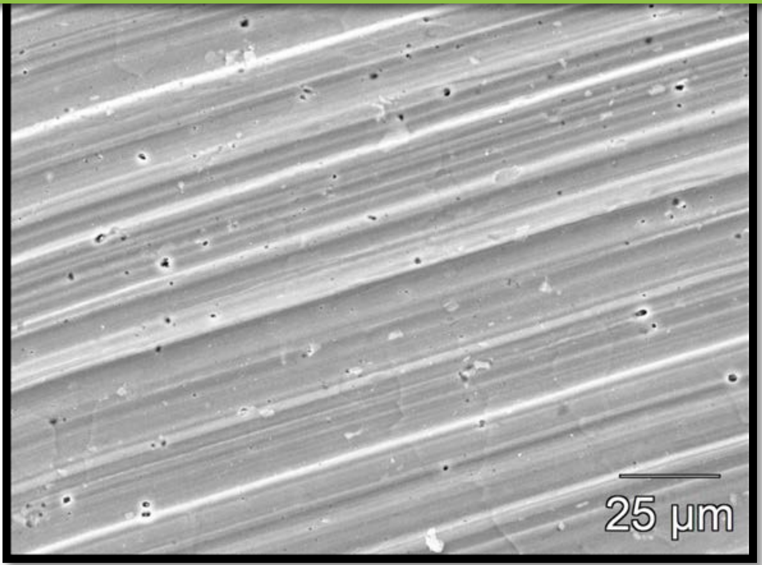
✓Wash → Etch → Anodize

Cross Section of Etched Surface

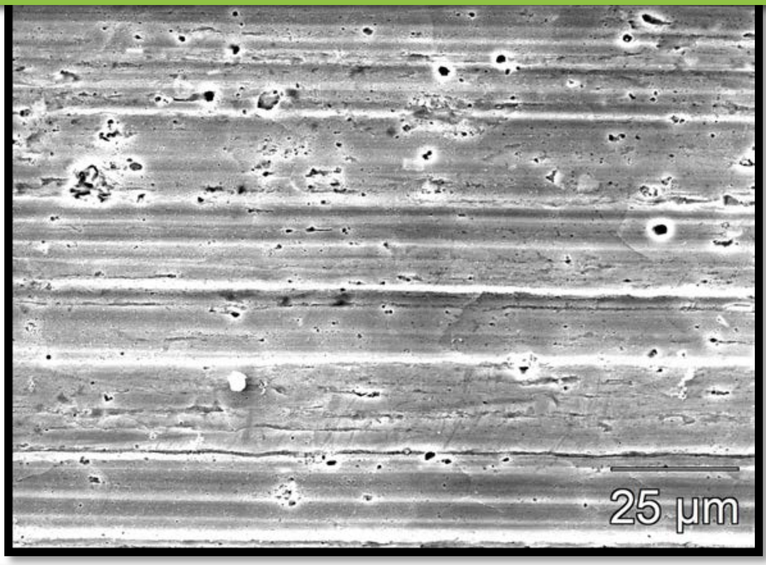


Insufficient Application of Acid Etch





Sufficient Dwell Time for Acid Etch

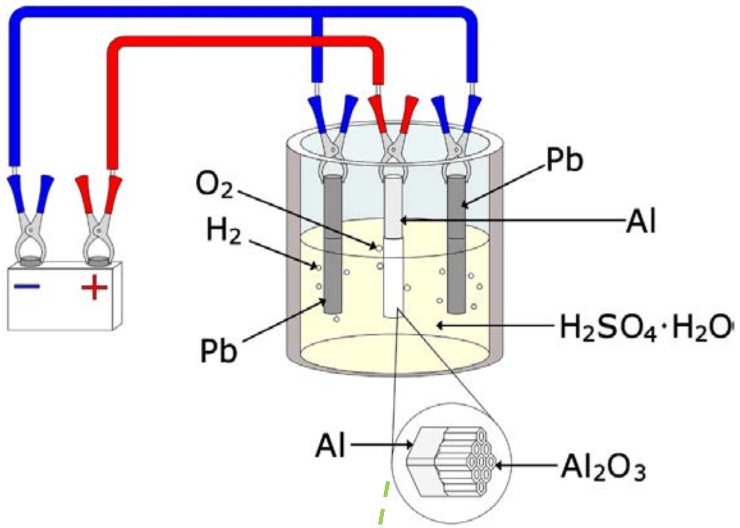


✓Increased surface area for bonding

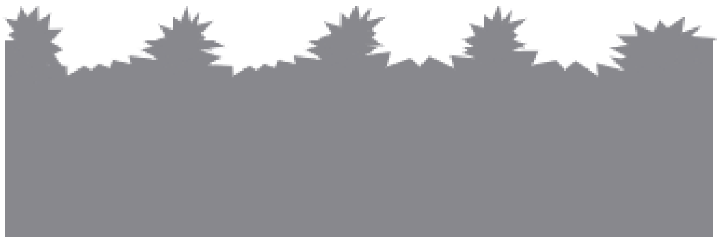
Mechanical or chemical etching increases surface area which is needed for effective bonding

Step 3. Anodize

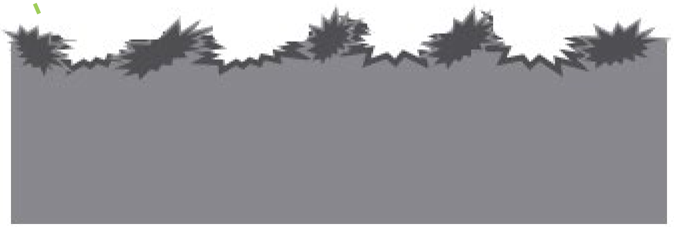
✓Wash → ✓Etch → **Anodize**

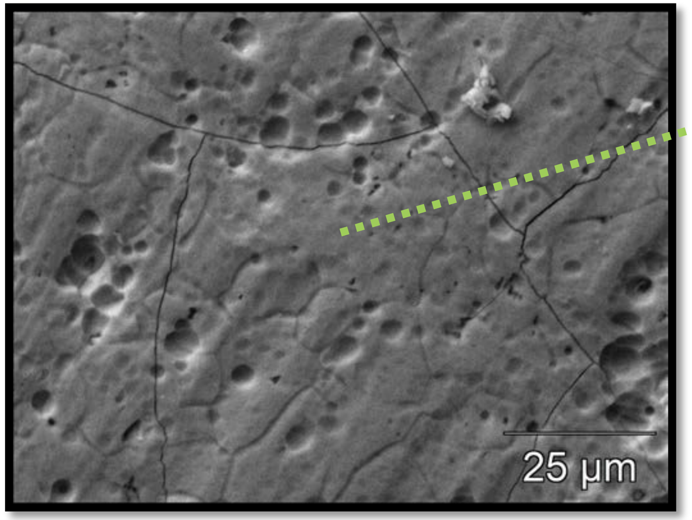


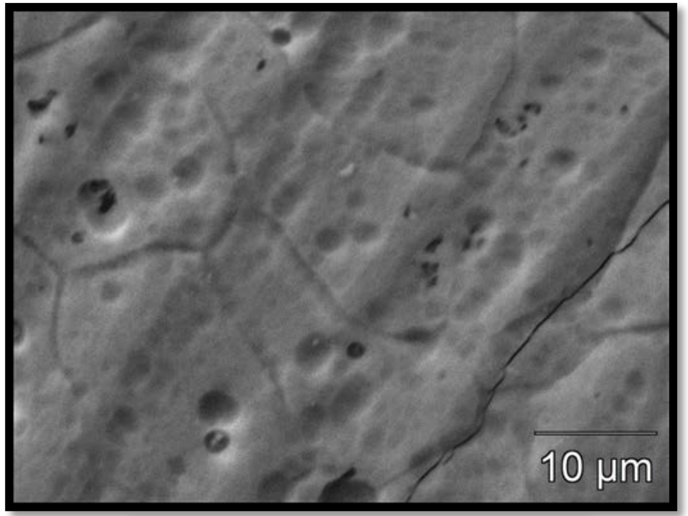
Cross Section of Etched Surface



Formed Al_2O_3







EDS Analysis

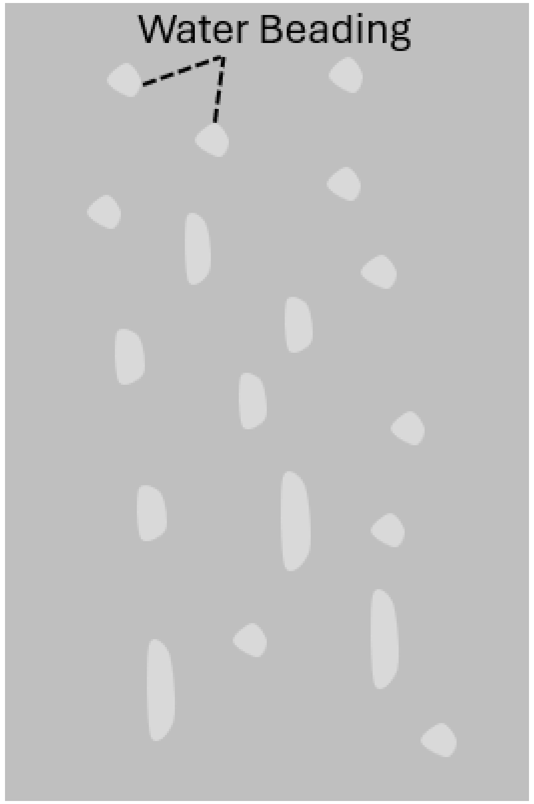
Element	Atom %
Oxygen	27.66
Aluminum	61.17
Sulfur	11.17

****Cracks from machining samples down to size***

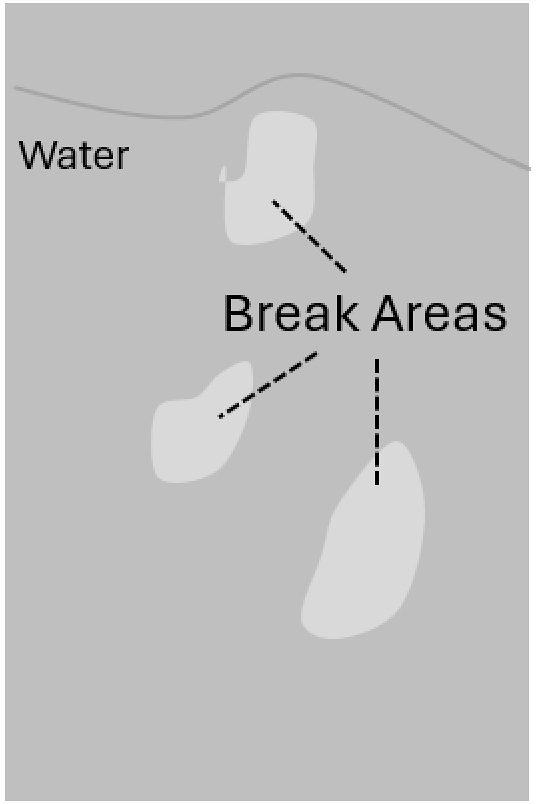
Anodizing promotes dense and durable protective oxide layer formation through electrolysis

Water Break Free – In Field Process Verification Check

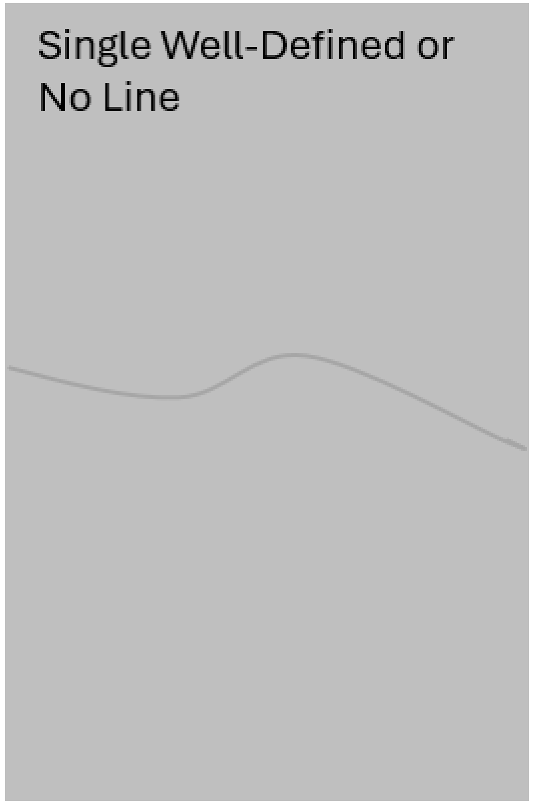
Highly Contaminated



Blotchy Areas – Variable Contamination



Water Not Beading = Clean

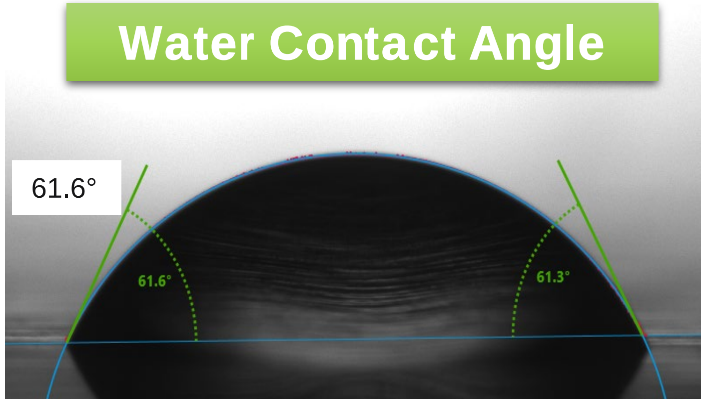


Water-break free testing provides a real-time indication of proper surface preparation

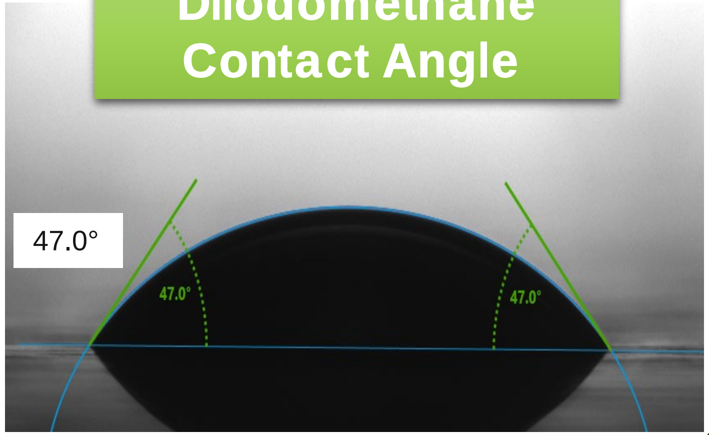
Surface Wettability Assessment via Contact Angle

Un-Treated 6061-T6 Aluminum

Water Contact Angle

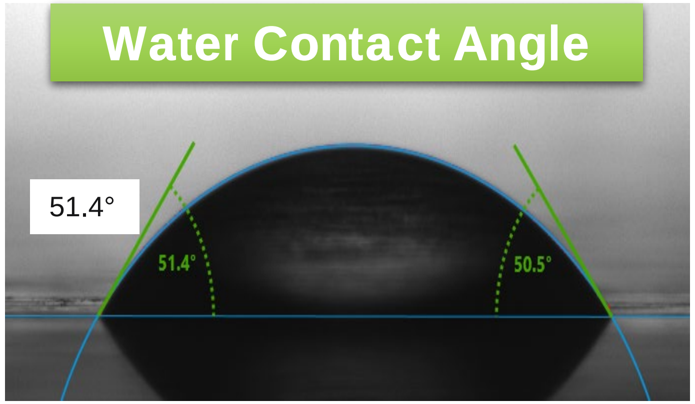


Diiodomethane Contact Angle

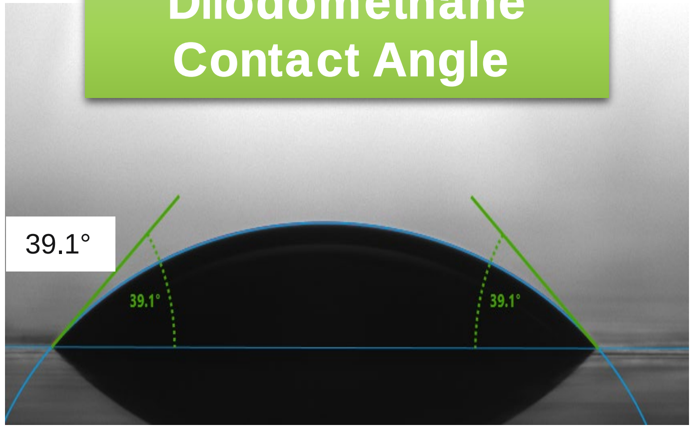


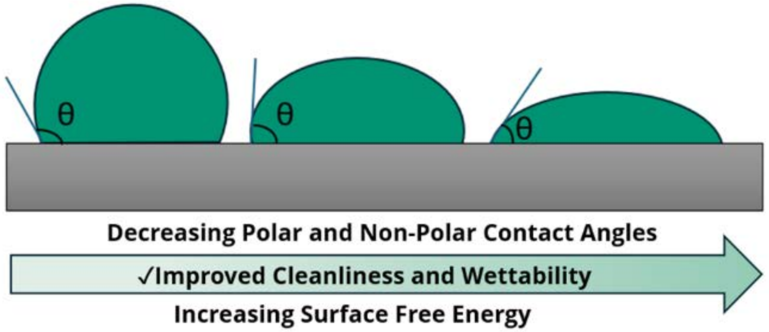
6061-T6 Aluminum Dry MEK Wipe

Water Contact Angle



Diiodomethane Contact Angle

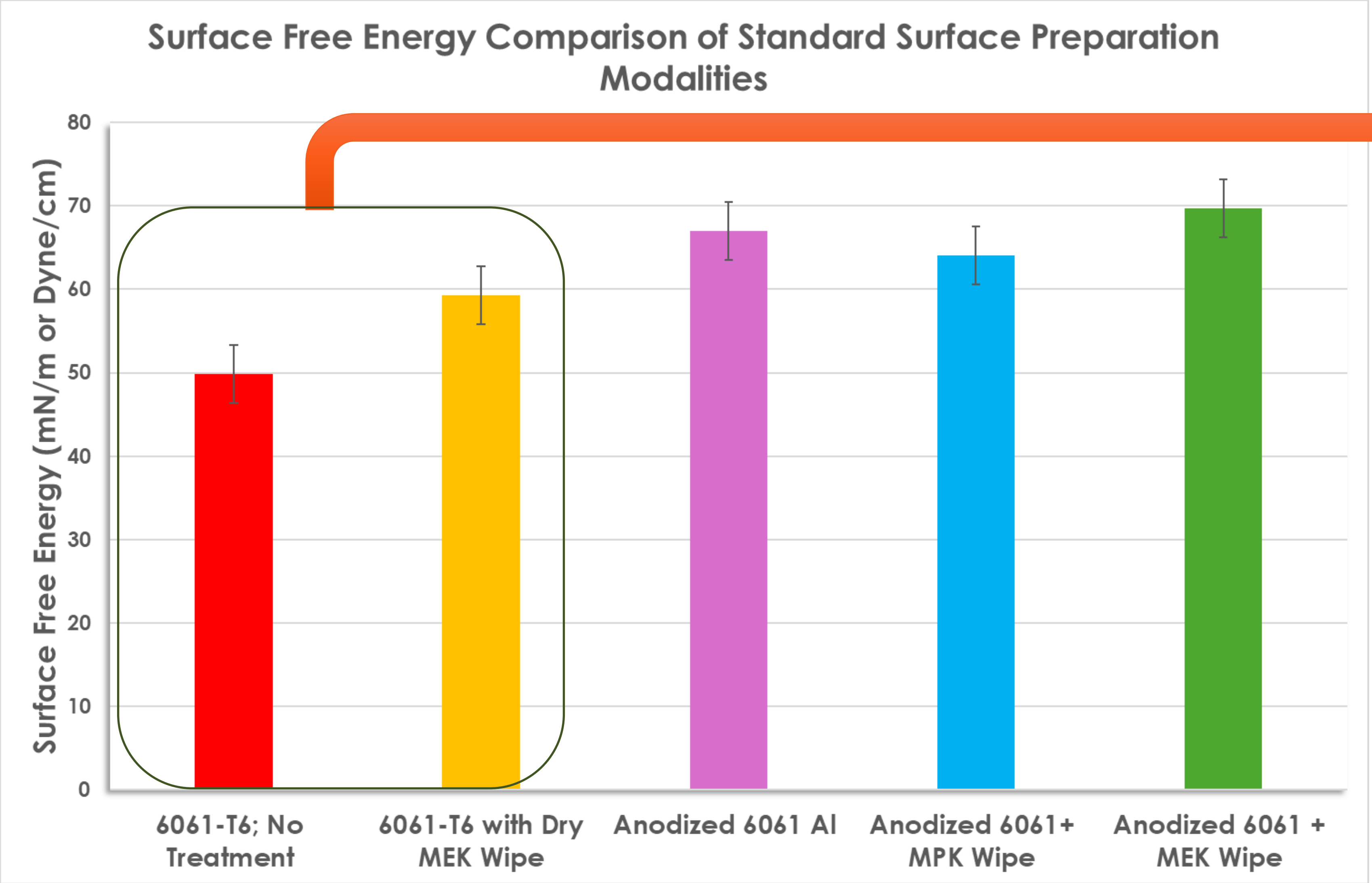




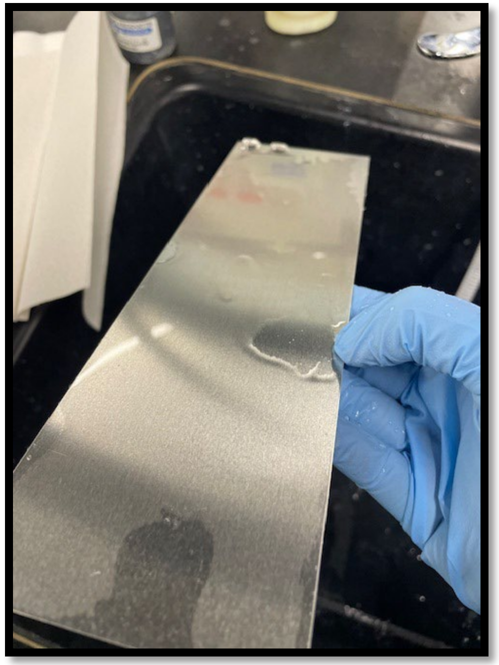
- MEK dry wipe protocol increased surface wettability by 17%
- Insufficient improvement from starting wettability as quantified by SFE

Improper surface preparation inhibits surface wettability needed for effective bonding

Impact of Surface Preparation on SFE



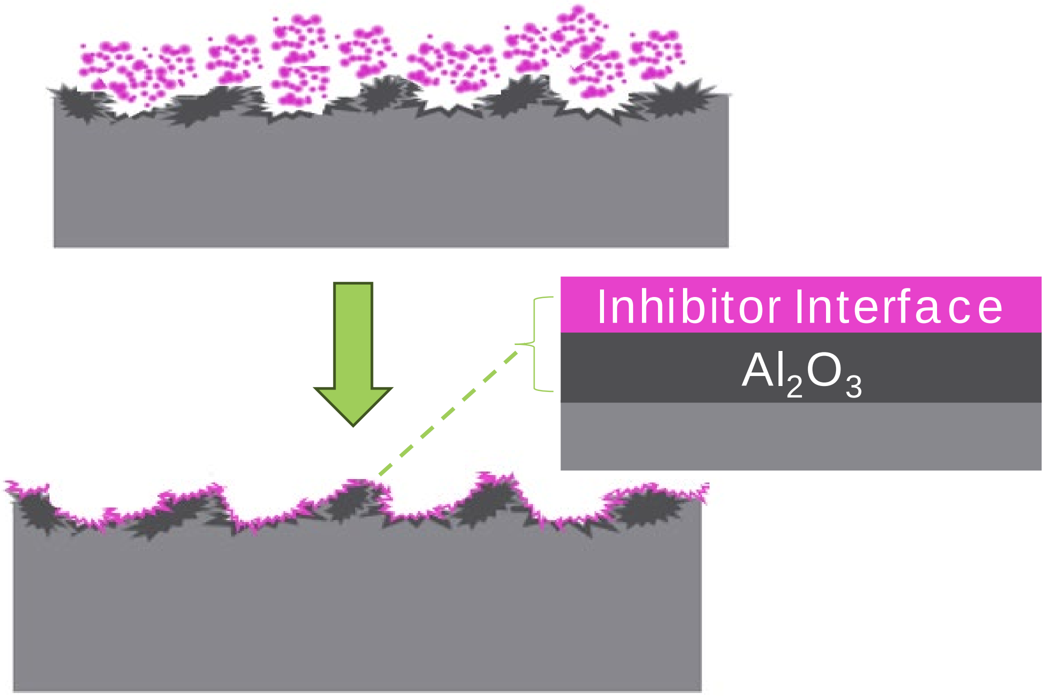
Low Surface Free Energy Quantifies Insufficient "Water Break Free" Condition

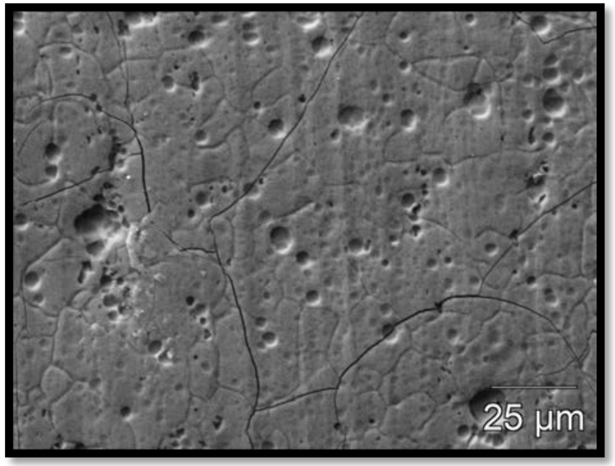


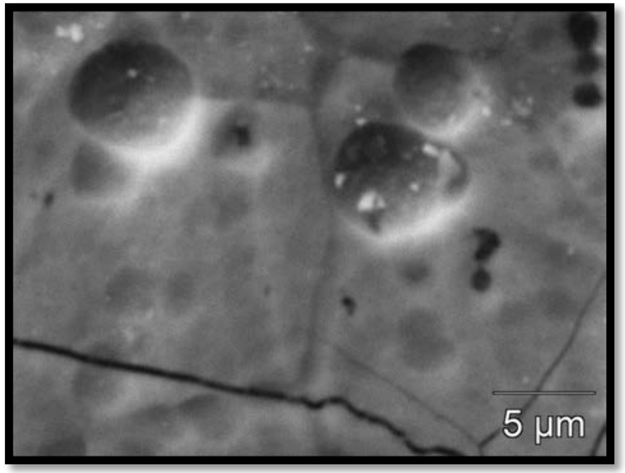
$$\sqrt{\gamma_{sv}^{d}\gamma_{lv}^{d}} - 2\sqrt{\gamma_{sv}^{p}\gamma_{lv}^{p}} = 0.5\gamma_{lv}(1 + cos\theta_{\gamma})$$

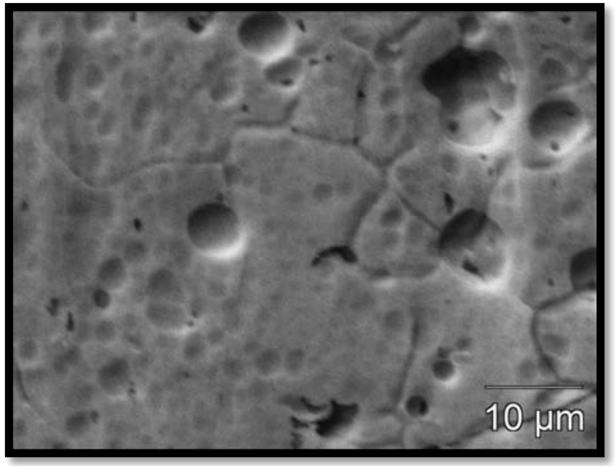
Step 4. Additional Non-Cr(VI) Surface Pre-Treatment(s)

Immersion or Spray Application of Non-Cr(VI) Inhibited Surface Adhesion Promoter









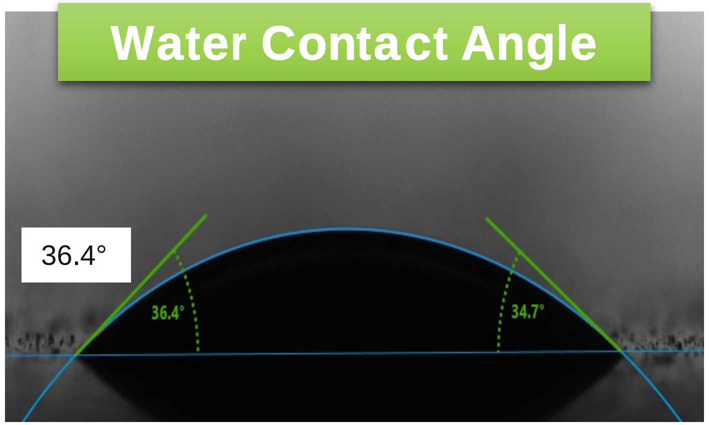
**Cracks from machining samples down to size*

Non-Cr(VI) inhibited surface adhesion promoter removes remaining surface contaminants and adds inhibitor constituents to Al_2O_3 interface for increased corrosion protection

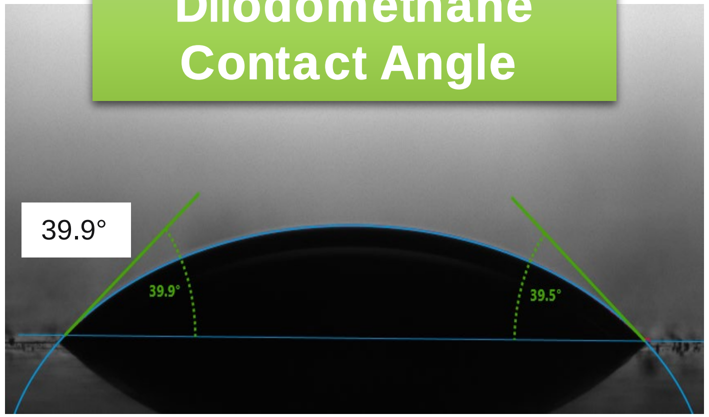
Contact Angle Comparison of Anodizing vs Additional Non-Cr(VI) Pre-Treatment

Sulfuric Acid Anodized 6061-T6 Aluminum

Water Contact Angle



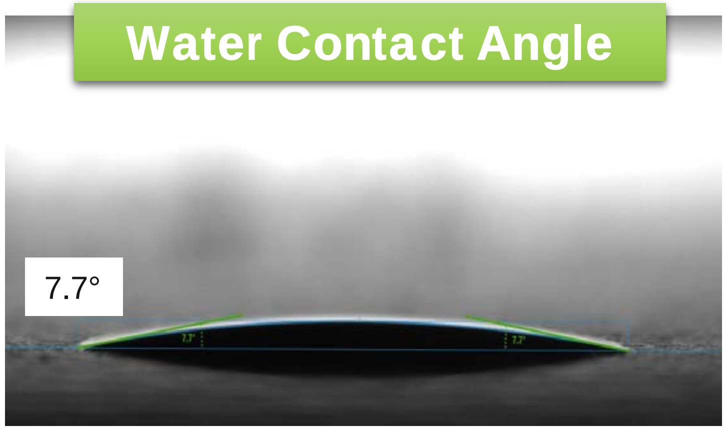
Diiodomethane Contact Angle



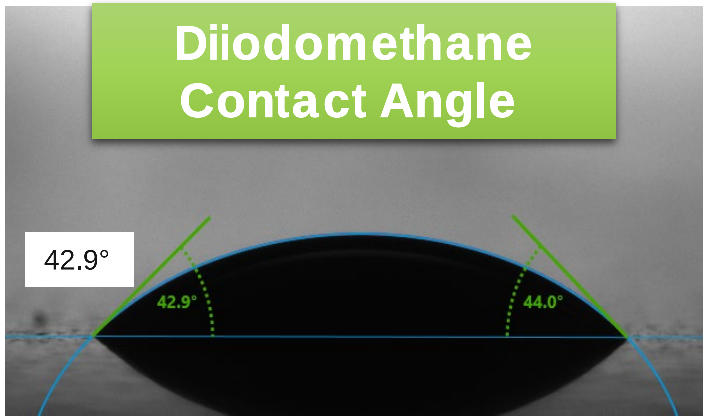
15 min Immersion treatment with Non-Cr(VI) Inhibited Surface Adhesion Promoter →

Sulfuric Acid Anodized 6061-T6 Aluminum

Water Contact Angle



Diiodomethane Contact Angle



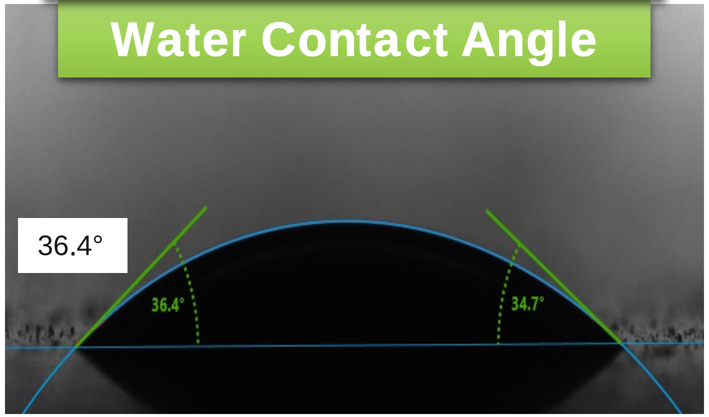
- ✓ **Polar surface wettability improved by 79%**

Treatment of anodized Al with non-Cr(VI) inhibited surface adhesion promoter significantly improves polar surface wettability

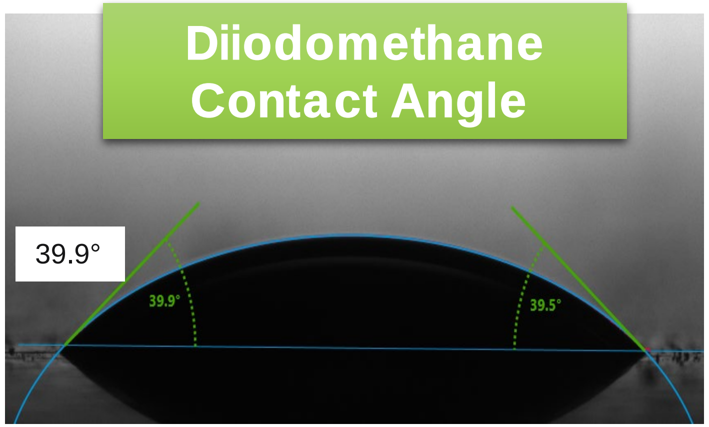
Contact Angle Comparison of Anodizing vs Additional Non-Cr(VI) Pre-Treatment

Sulfuric Acid Anodized 6061-T6 Aluminum

Water Contact Angle



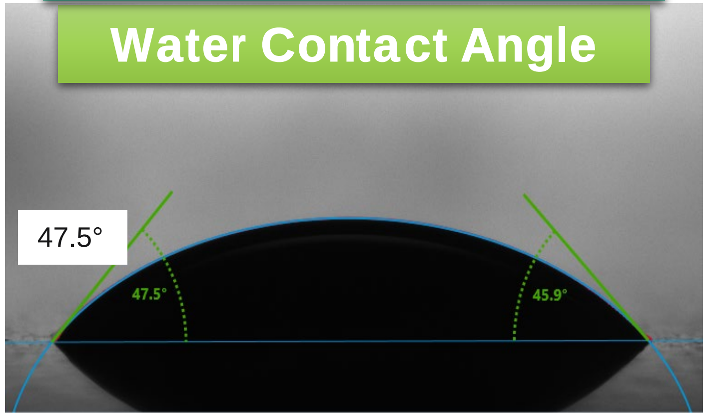
Diiodomethane Contact Angle



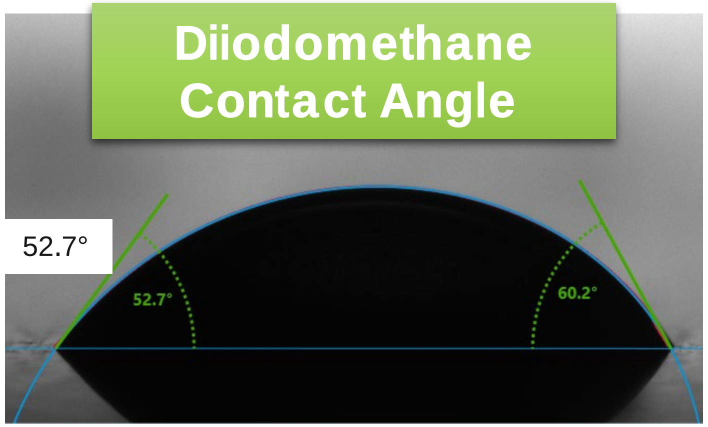
Application of an Un-Inhibited Surface Treatment (2x Scuff Application and Water Rinse)

Sulfuric Acid Anodized 6061-T6 Aluminum

Water Contact Angle

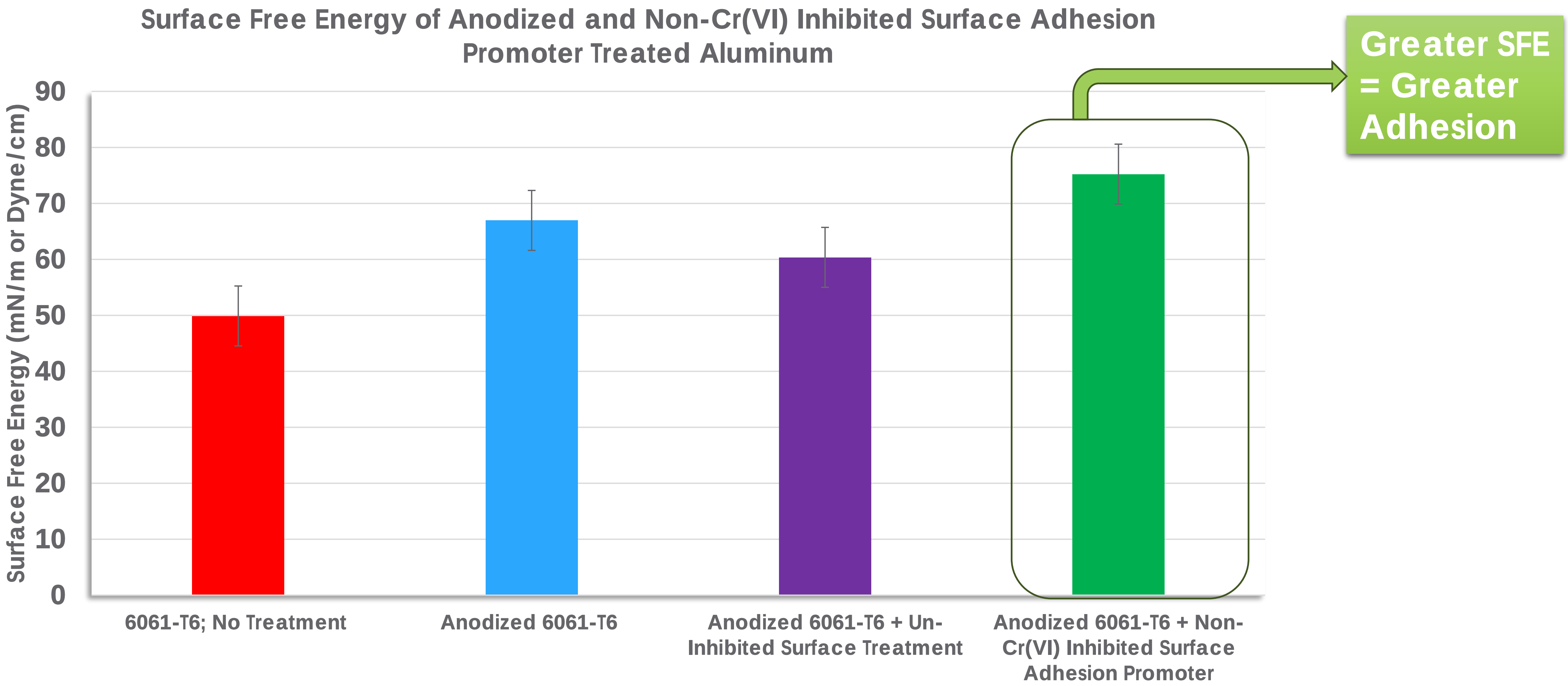


Diiodomethane Contact Angle



Treatment of anodized Al with an un-inhibited surface treatment did not enhance surface wettability because no surface functionalization occurred

Impact of Anodizing and Other Surface Pre-Treatments on SFE



SFE increases 12% with non-Cr(VI) inhibited surface adhesion promoter compared to anodized sheet stock

Summary

- Proper surface preparation has been shown to be critically important to prevent material failures and re-work
- The impact of surface pre-treatments was quantified in this study using:
  - Surface tensiometry
  - SEM/EDS Analysis
  - Water break free
- It was determined that the use of a non-Cr(VI) inhibited pre-treatment resulted in the following:
  - 79% increase in polar surface wettability
  - 12% increase in SFE
    - When compared to un-treated sulfuric acid anodized aluminum
      - Sharp contrast to an un-inhibited surface pre-treatment which did not increase surface wettability
- Phase 2 of this study will include adhesive strength determination and corrosion resistance testing

Improper surface preparation causes poor adhesion due to poor surface wetting, leading to material failures and re-work

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