Optimizing interfacial strength between steel and polymer through plasma and wet chemistry treatments

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Use of Coupling Agents

No Coupling Agent

Low interfacial strength
Use of Coupling Agents

No Coupling Agent

With Coupling Agent

Polymer Matrix

Steel

Coupling Agent
Use of Coupling Agents

3 HO-Si-OH

Y R Y R

HO-Si-O-Si-O-Si-OH
Use of Coupling Agents

Monolayer → Best performance
Silane Application
Two Different Technological Approaches

Plasma

PlasmaZone®

Time efficient
Avoid solvents
High throughput/Easy integration
High (investment) cost
Silane Application
Two Different Technological Approaches

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Time efficient
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Wet Chemistry

Use of solvents
Low cost
Molecular control

SIM
innovating together
1. **Steel Cleaning**
   - Physical
   - Chemical
   - Mechanical

**Silane Application**

Wet Chemistry
2. Solution Preparation
- Type of Silane
- Solvent (ratio)
- Concentration
→ ‘Working window’
2. Solution Preparation
- Type of Silane
- Solvent (ratio)
- Concentration
→ ‘Working window’

(a1) Silane hydrolysis, (a2) Self-condensation & (b) Reaction to steel surface
2. Solution Preparation
- Type of Silane
- Solvent (ratio)
- Concentration
→ ‘Working window’

(a1) Silane hydrolysis, (a2) Self-condensation &
(b) Reaction to steel surface
3. Silane Deposition
- Dipping Time (Silane adsorption)
- Rinsing Time (Remove excess)
Silane Application
Wet Chemistry

4. Oven Conditions
- Vacuum/no vacuum
- Time
- Temperature
→ Condense silane to surface
→ Remove solvent residue

Wet Chemistry
Silane Application
Plasma Technology

Plasma → PlasmaZone®
Silane Application

Plasma Technology

Plasma → PlasmaZone® → Coated Steel

Silane Precursor

Carrier Gas → Ceramic → Coated Steel

HV

SIM
Silane Application
Plasma Technology

1. **Precursor Flow**
   - Type of chemistry
   - Amount of Silane
Silane Application

Plasma Technology

2. **Plasma Power**
   - As low as possible
   → Reduce precursor fragmentation
3. **Number of Passes**
- Thickness silane Layer
Silane Application

Plasma Technology

Post treatment (cfr. Wet Chemistry)

1. Oven
   - Tunable parameters
   - Low cost

2. Plasma
   - Fast
   - Combination with deposition → Investment cost ↓
   - In line
Determine Best Conditions

Pull-off testing (Dolly-plate-dolly)

Resin: EPIKOTE 828 LVEL + 1,2 Diamino Cyclohexane

Dolly-plate-dolly sample

Stress at break (MPa) = \( \frac{\text{Load at break (N)} \times 4}{\pi \times D^2 \text{ (mm)}} \)

* Plate thickness: 0.8 mm
* No of samples tested: 6
Optimization: Wet Chemistry

**Condition:**

- Silane solution concentration
- Dipping condition
- Rinsing Condition
- Condensation condition (oven condition: Temperature and Time)
Optimization: Wet Chemistry

Condensation condition

Optimum condition: 1.5 hr at 70°C in Vacuum

30 sec dip
Brief rinse
Optimization of rinsing condition and silane solution concentration

Optimum condition: Rinsing – 1 min, silane concentration: 2%
Optimization of dipping condition

**Optimum condition**: 2% silane solution, 30 sec of dipping, 1 min rinse in ethanol, condensation: 1.5 Hr@70°C (VAC)
Optimization: Plasma Technology

**Condition:**
- Precursor
- Plasma power
- No of passes
- Plasma post treatment
Optimization: Plasma Technology

Plasma Precursor: Pure APS vs APS+1% Water

- Plasma power: 350W
- No of passes: 6
- Condensation of silane: 4hr at oven (100°C)

![Graph showing Stress at break (MPa) for Blank, APS, and APS+1% water]
Optimization: Plasma Technology

Plasma Power

- Plasma precursor: APS+1% water
- No of passes: 6
- Condensation of silane: 4hr at oven (100°C)

Plasma Power: 200 W
Optimization: Plasma Technology

Effect of no of passes:

Optimum condition: APS+1% water (precursor), 200 W (power), 4 passes

- Plasma precursor: APS+1% water
- Plasma power: 250W
- Condensation of silane: 4hr at oven (100°C)
Optimization: Plasma Technology

Plasma post treatment

Stress at break (MPa)

- Blank
- Thermal condensation
- Plasma post treatment

* More details will be found at the Poster (Da Ponte Gabriella, VITO)
Comparison: Atmospheric plasma vs Wet treatment

![Graph showing comparison of stress at break (MPa) for different treatments.](image-url)

- **Blank**
- **Wet-1.5hr@70°C(VAC)**
- **Plasma-NF_Z_10-4hr@100°C**

Target value
Conclusions

• Optimize deposition conditions
  ➢ Increase interfacial strength – more than 300% improvement, better than the target value.
  ➢ Increase ‘product’ strength for both steel plate & steel fiber composites

• With both
  ➢ Plasma technology
  ➢ Wet chemistry application
  ❏ Each having their own advantages
Thank you