# Adhesion Properties of Wood Plastic Composite (WPC) Surfaces Using Atomic Force Microscopy

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**SIXTH INTERNATIONAL SYMPOSIUM ON CONTACT ANGLE, WETTABILITY AND ADHESION**

July 15, 2008

# Overview

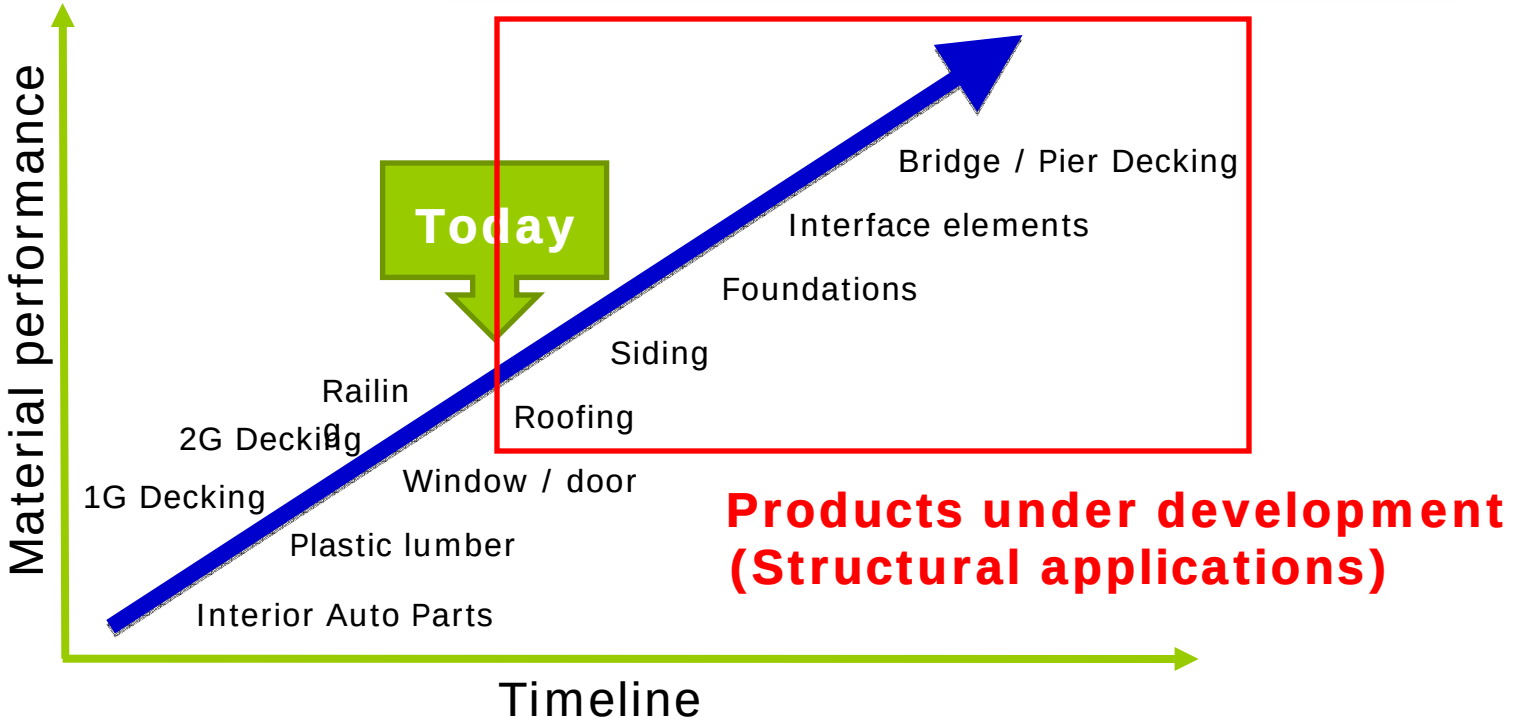
- Introduction
- Overall goal
- Methods
- Results
- Discussion & Conclusions

## Wood Plastic Composites (WPCs).

- Thermoplastic resins (30-80%).
  PE, PVC, PP
- Reinforcing fiber (30-70%).
  Wood, Hemp, Straw, etc.
- Additives (0-5%).
  Pigments, lubricants, coupling agents, UV inhibitors.

# WPCs next generation.

First generation WPC products




M. Wolcott, P.Smith and J. Hermanson, 8th International Conference on Woodfiber-Plastic Composites-Madison, WI (2006).

INTRODUCTION

- Limitations to use WPC as a Structural Material
  - Stiffness
    - Low stiffness (about half of typical structural lumber)
    - Decrease in stiffness in wet environments
  - Susceptible to Creep
    - Loss of stiffness over time with constant load
  - Creep Rupture
    - Material Failure over period of time with constant load

## • WPCs next generation. Challenges

- Material science issues related to efficient transfer of stress between the fiber and the matrix will need to be improved upon.
- A better knowledge of the interaction between the components present in WPC formulations.
- Improve the WPC adhesive bondability to any other material that can improve its mechanical performance (i.e. Fiber Reinforced Plastic).

- Ways to improve surface energy.

  - Chemical treatments.
  - Mechanical treatments.
  - Energetic treatments.

INTRODUCTION

## Surface and bondability characterization.

- Thermodynamic (Contact angle-Surface energy).
- Chemical (ATR-IR, XPS).
- Mechanical (Shear Strength, Fracture toughness)
- Microscopic (SEM, AFM)

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# Overall goal

To understand the contribution of the individual components of Wood Plastic Composites (WPCs) and WPC surface treatments on the final wettability and adhesive bondability using Atomic force microscopy as a complimentary tool for surface evaluation.

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METHODS

## Raw materials

- Thermoplastic resin (40%):

  Polypropylene (SE 29-31 mJ/m2) (BP Amoco, Houston, TX, USA).

- Reinforcing fiber (50%)

  Pine wood flour (40 mesh, American Wood Fibers (Schofield, WI, USA)).

- Additives (10%)

Polybond 3200 coupling agent (Chemtura, Middlebury, CT, USA).
TPW 113 lubricant (Struktol, Stow, OH, USA).
Gray colorant (Clariant, Lewiston, ME, USA).

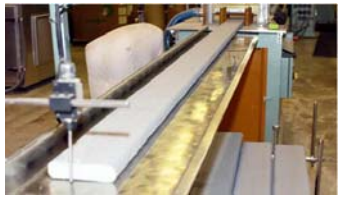
- Adhesives:

  Epoxy (Pro-Set® M1013 resin with M2017 Pro-Set® hardener manufactured by Gougeon Brothers Inc. (Bay City, MI).

METHODS

## WPCs production

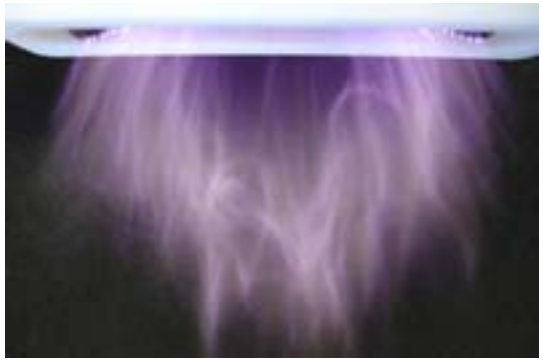
Figure 1: Davis-Standard WT94 Twin Screw Extruder. To produce WPC boards about 14 cm (wide) and 4 cm (thick). Rate of production: 4 feet/min



1. WPC boards were mechanically knife planed and sanded using abrasive papers of three different grit sizes, P60, P100 and P220.
2. WPC not planed.

METHODS

## Surface treatments



- Energetic treatment:

  Forced air plasma treatment (FAPT)

  - Using a Lectro Treat III Forced air plasma surface treater, LTIII (15kV, one head).
  - Varying LTIII pass (1, 5 and 10 passes and two lengths of discharge projected from the gun head (1” and 2.5”).
  - Bonding WPC boards with epoxy resins.

# cterization

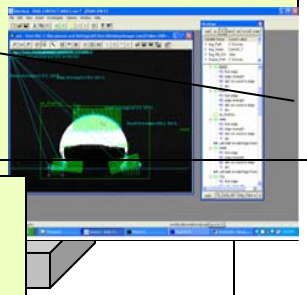
Dioodomethane
Water
Ethylene glycol
12 droplets (5µm)

***vOCG approach***

$$(1+\cos\theta)\gamma_L = 2[(\gamma_s^{LW}\gamma_L^{LW})^{1/2} + (\gamma_s^{+}\gamma_L^{-})^{1/2} + (\gamma_s^{-}\gamma_L^{+})^{1/2}]$$

$$\gamma^{p} = 2(\gamma^{+}\gamma^{-})^{1/2}$$

Labview
Matlab
Sherlock

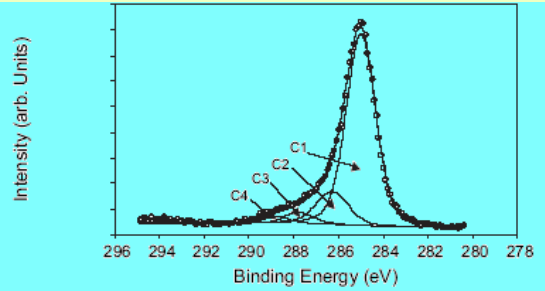


- Contact angle (sessile drop)-3 liquids.
- Surface energy- 1 approach.
- X-Ray photoelectron spectroscopy (XPS).
- Atomic force Microscopy.

Low resolution spectrum:
from 0 to 1100 eV

High resolution spectrum:
of the C1s region from 280 to 300 eV

***Ratio C/0***
***C unoxidided/ C oxidized***




***Topography***
***Adhesive forces***
***(Tapping & contact mode Silicon tip).***

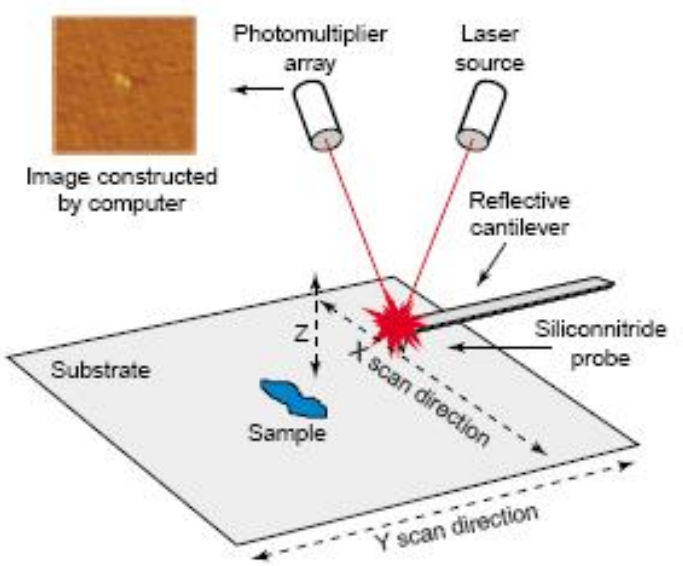
***Spring constant:42N/m***
***Resonance Frequency: 300kHz***

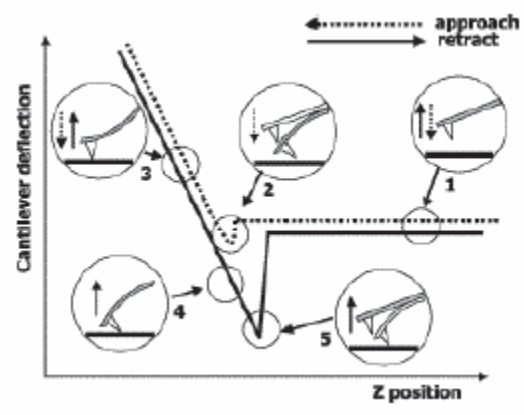
METHODS

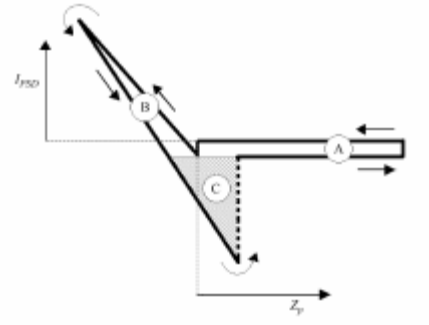
# Surface characterization

- Atomic force microscopy, AFM

  (Asylum AFM-MFP3D)



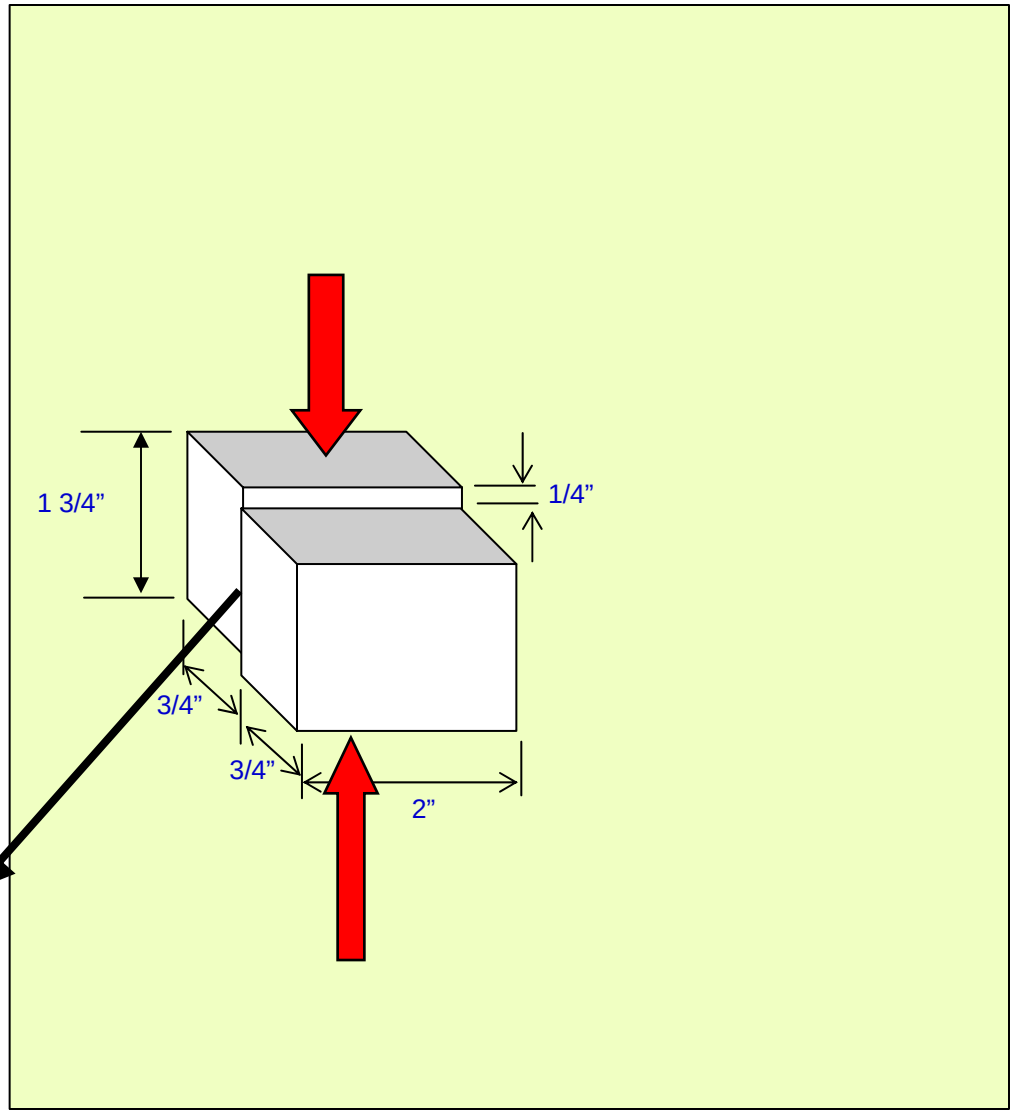



METHODS

# ❑ Bond performance

**Shear strength**

ASTM D 905
Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading.



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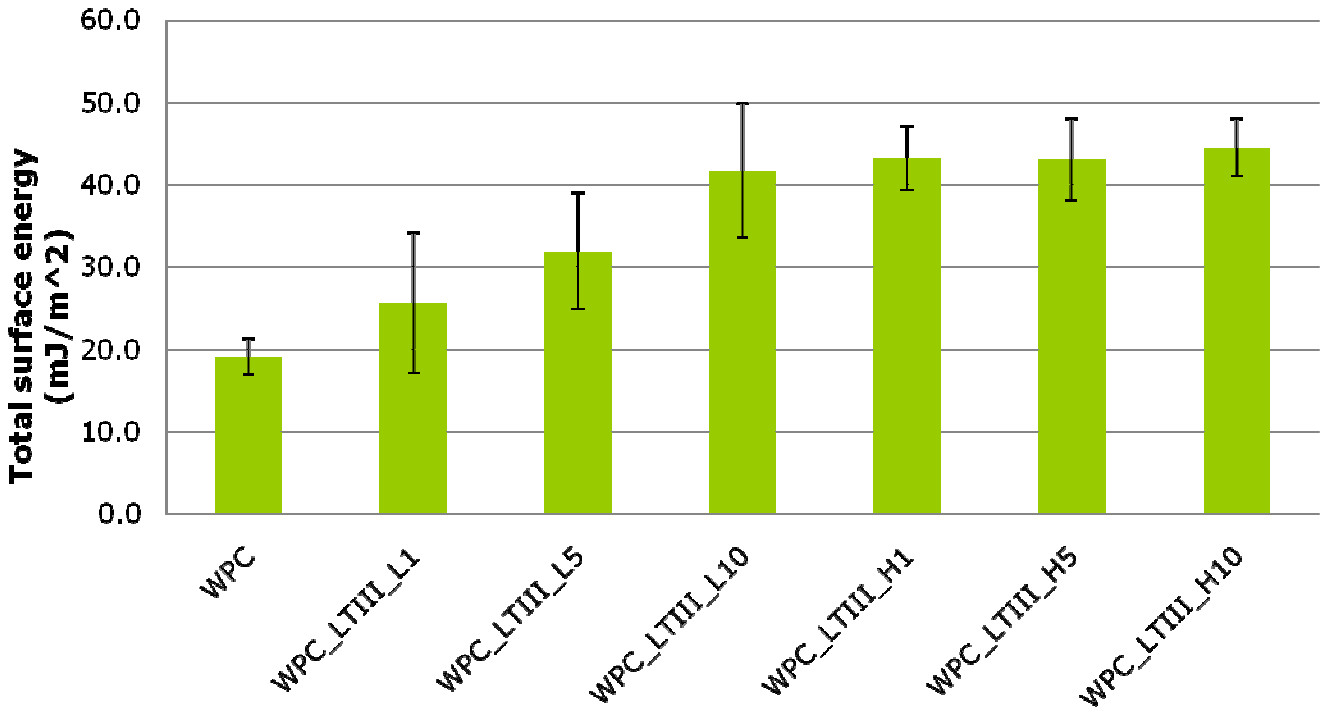
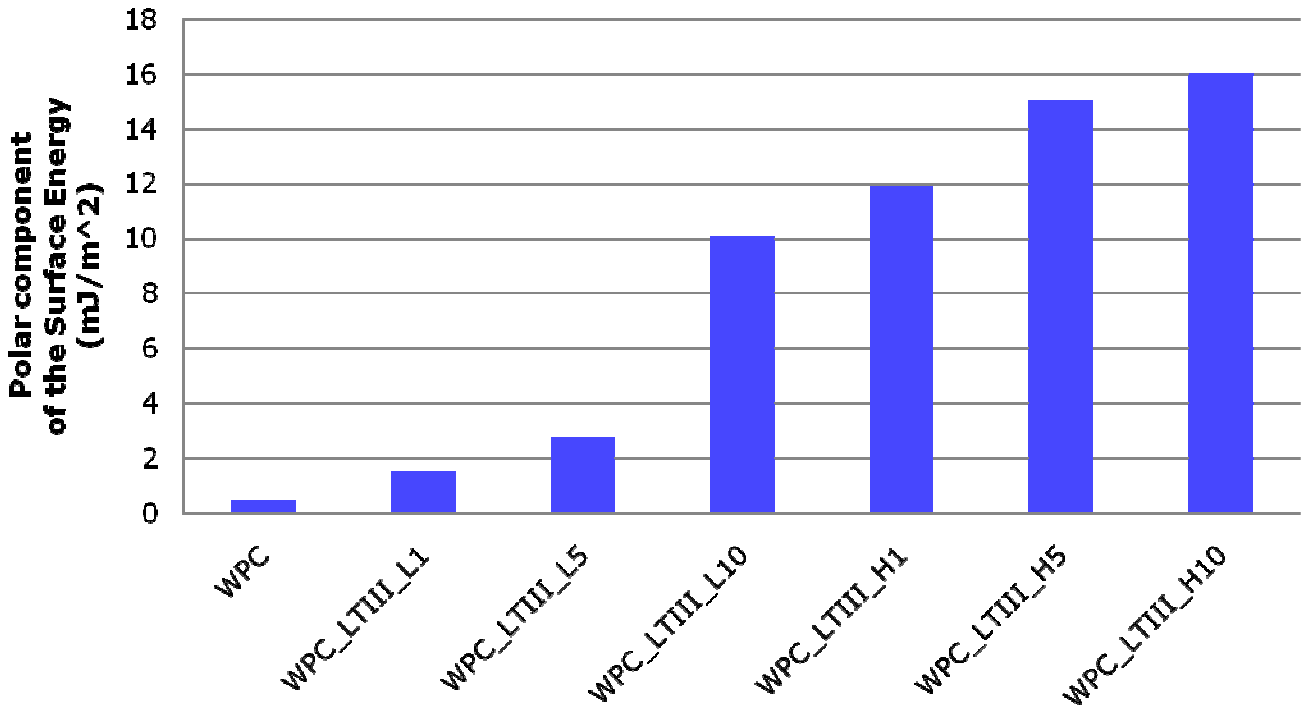
## Surface energy




WPC: Wood Plastic Composite planed and sanded.
LTIII: Lectro Treat III Forced air plasma surface treater.
L: low level discharge length (1");1,5, 10 passes. H: high level discharga length (2.5");1,5 and 10 passes.
*Using epoxy adhesive.

RESULTS

# Polar component of the surface energy.

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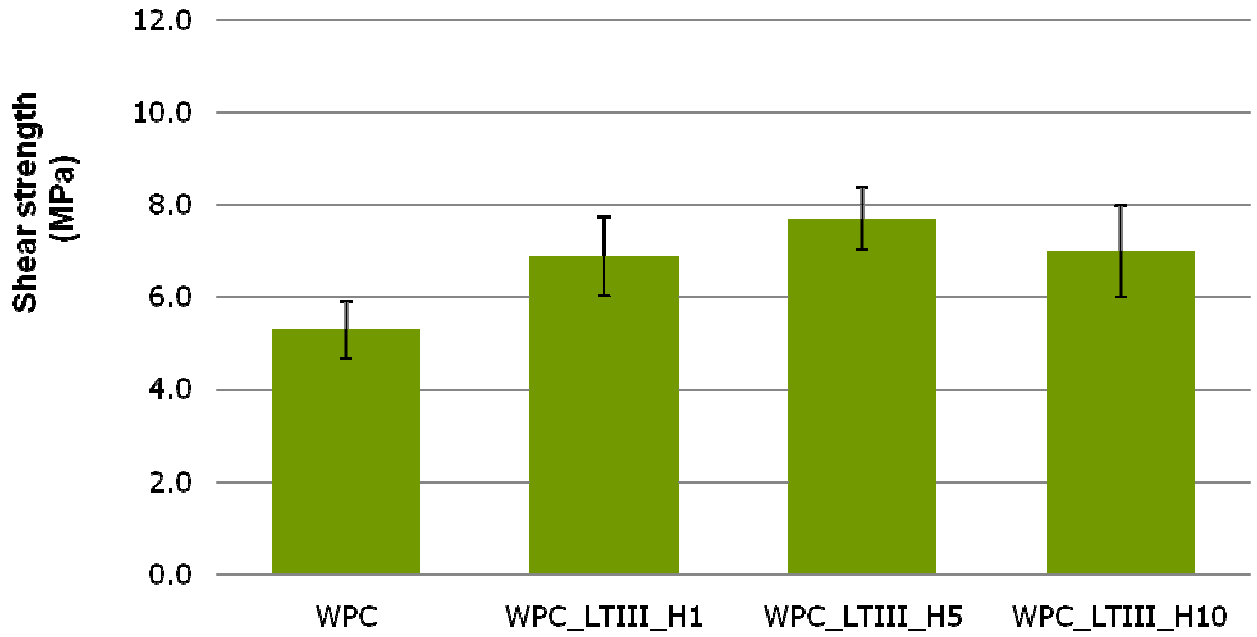
*Using epoxy adhesive.

RESULTS

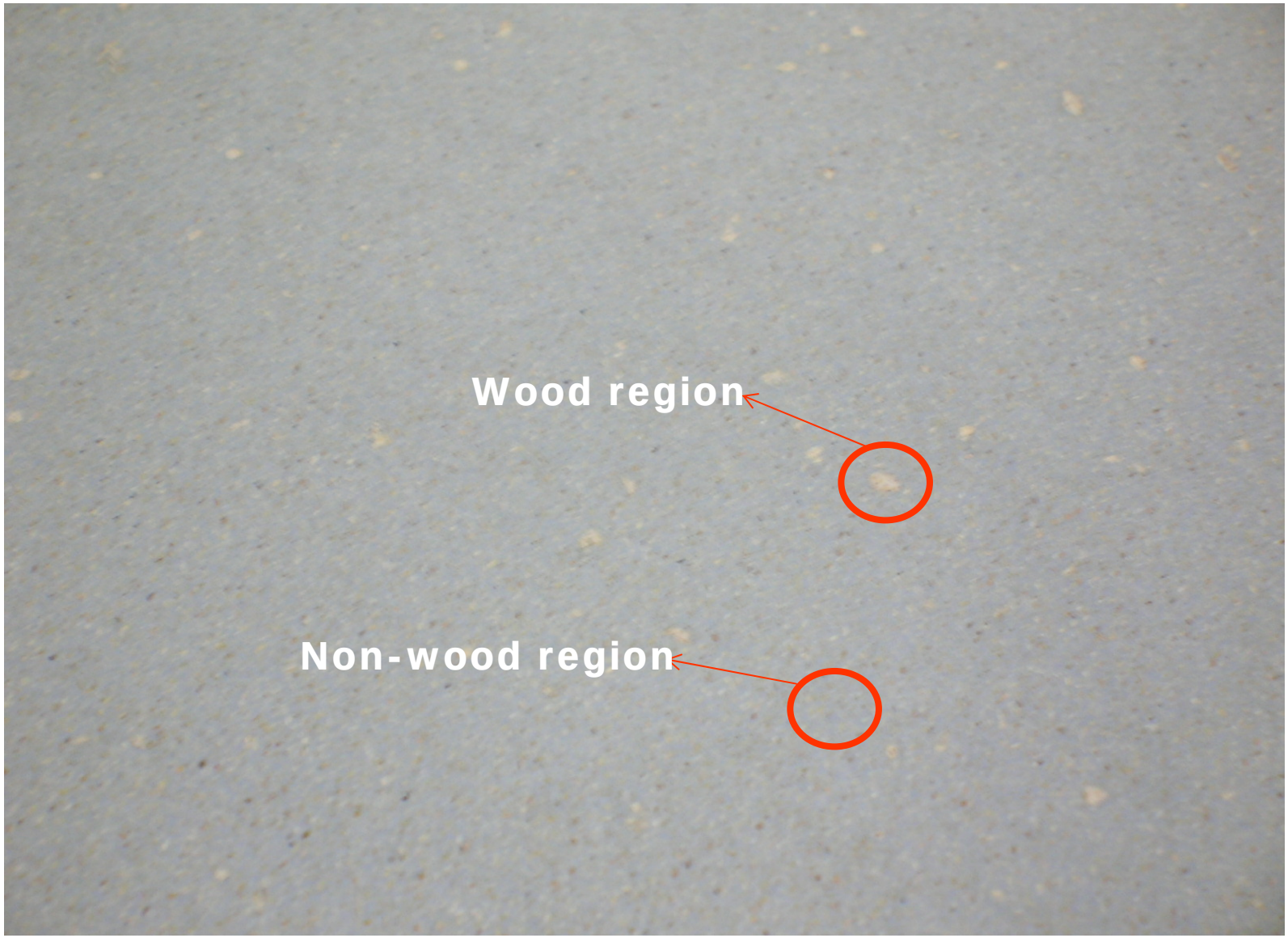
## Shear strength after varying the discharge passes




WPC: Wood Plastic Composite planed and sanded.
LTIII: Lectro Treat III Forced air plasma surface treater.
H: high level discharge length (2.5");1,5 and 10 passes.
*Using epoxy adhesive.

RESULTS

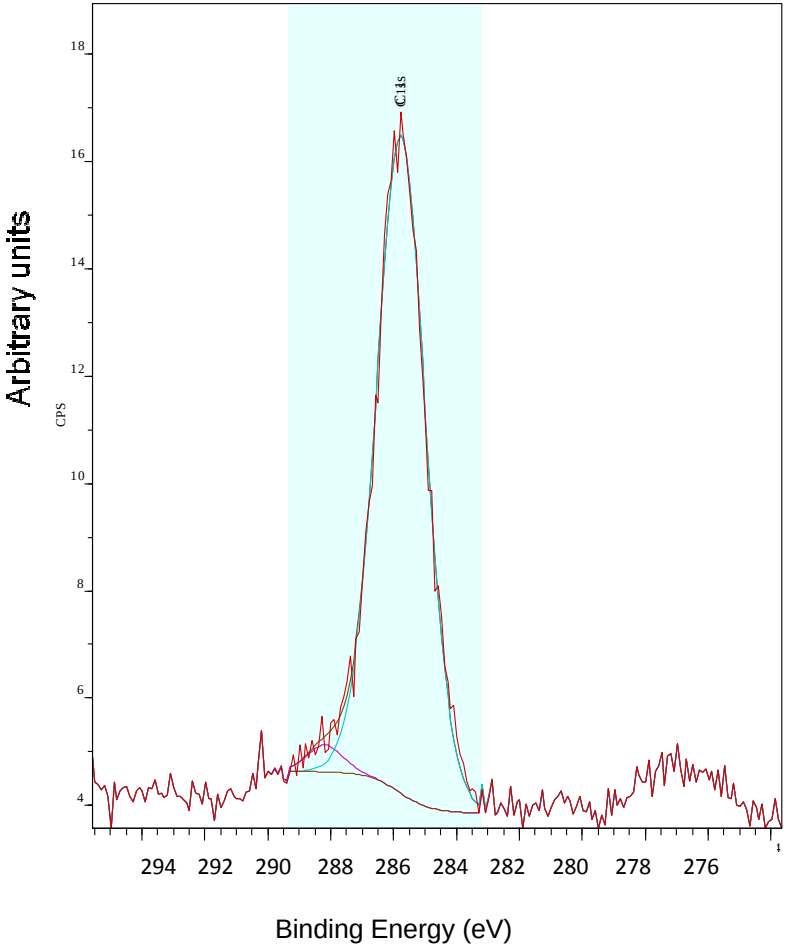
## XPS-Carbon spectra (WPC planed and sanded)



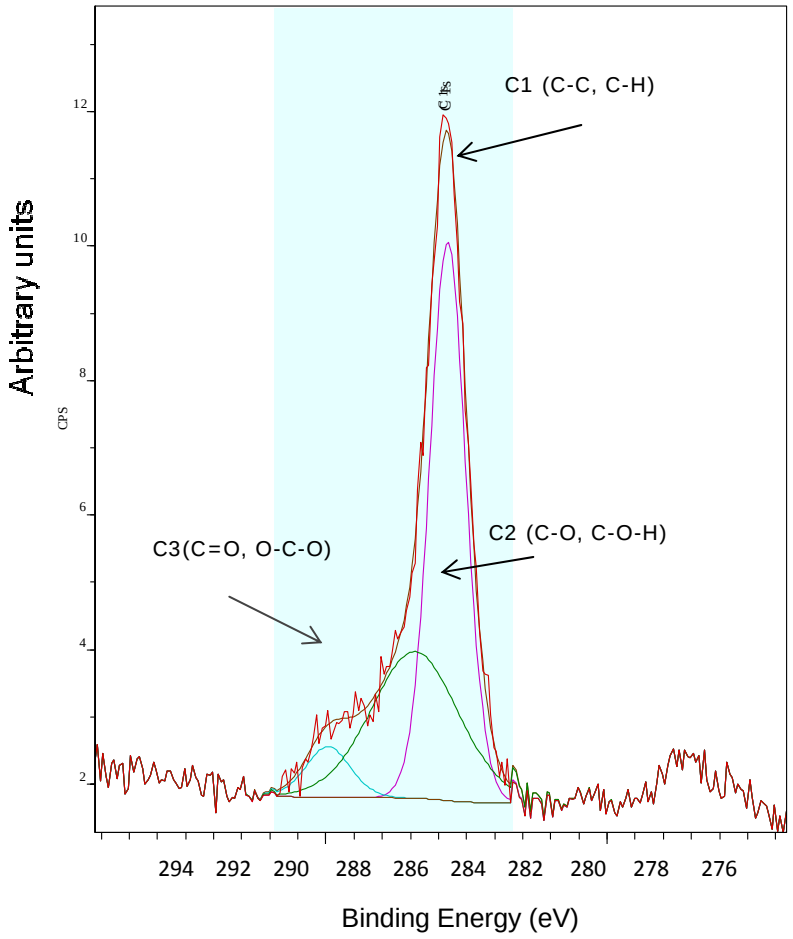
RESULTS

# XPS-Carbon spectra (WPC planed and sanded)

WPC_wood region




WPC wood region_LTIII_H5

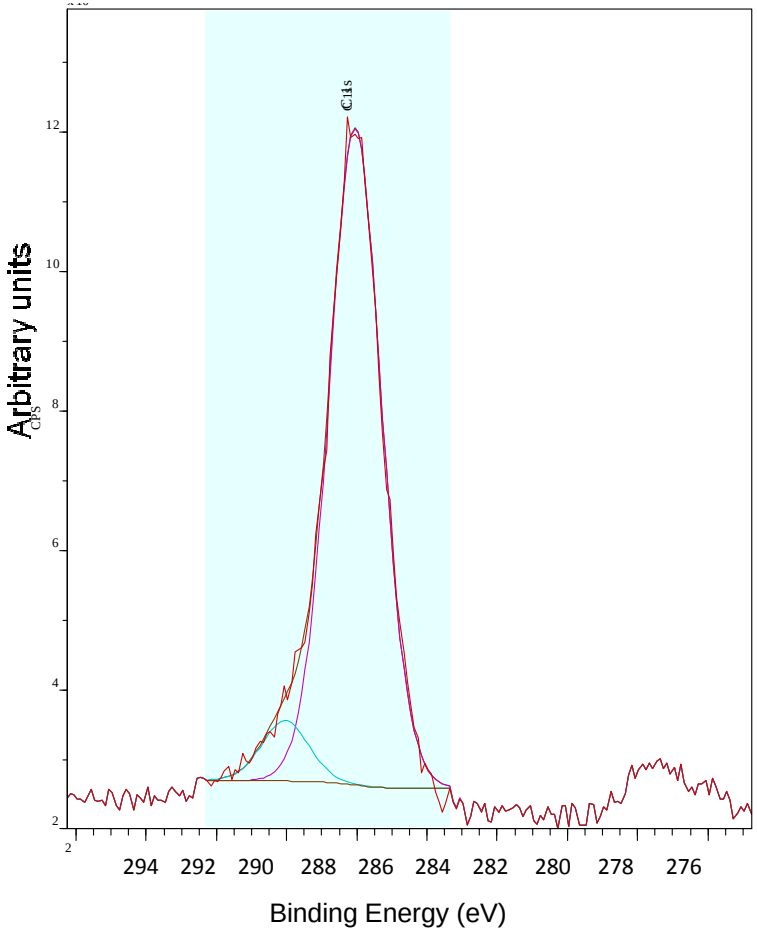



XPS : X-ray photoelectron spectroscopy
WPC: Wood Plastic Composite planed and sanded
LTIII: Lectro Treat III Forced air plasma surface treater.
H: high level discharge length (2.5”);5 passes.

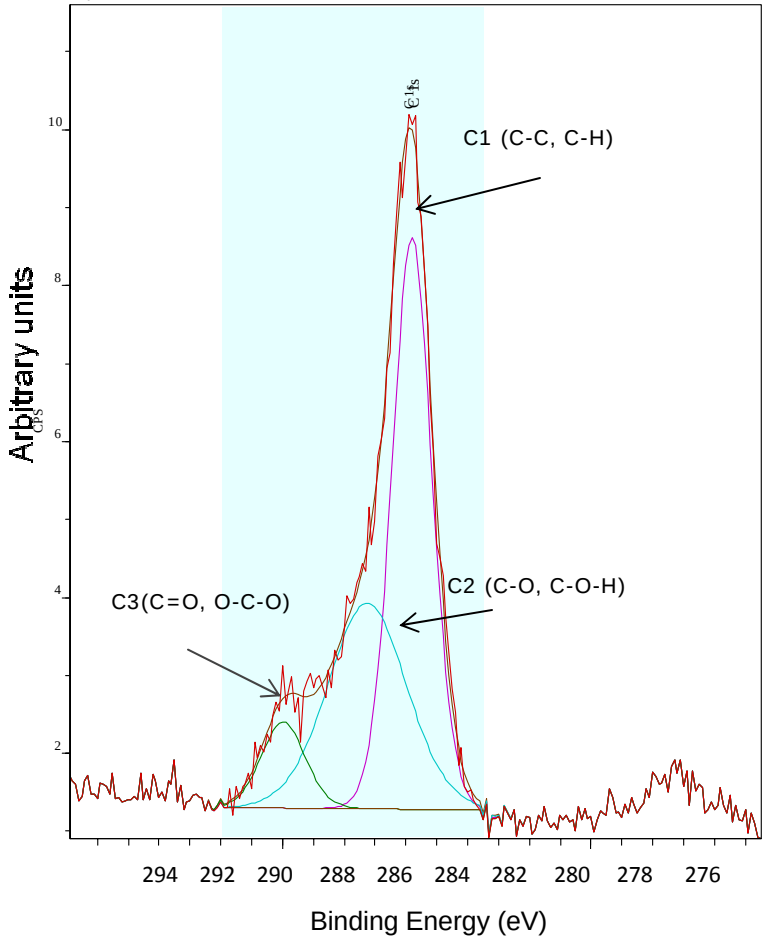
RESULTS

# XPS-Carbon spectra (WPC planed and sanded)

WPC_non wood region



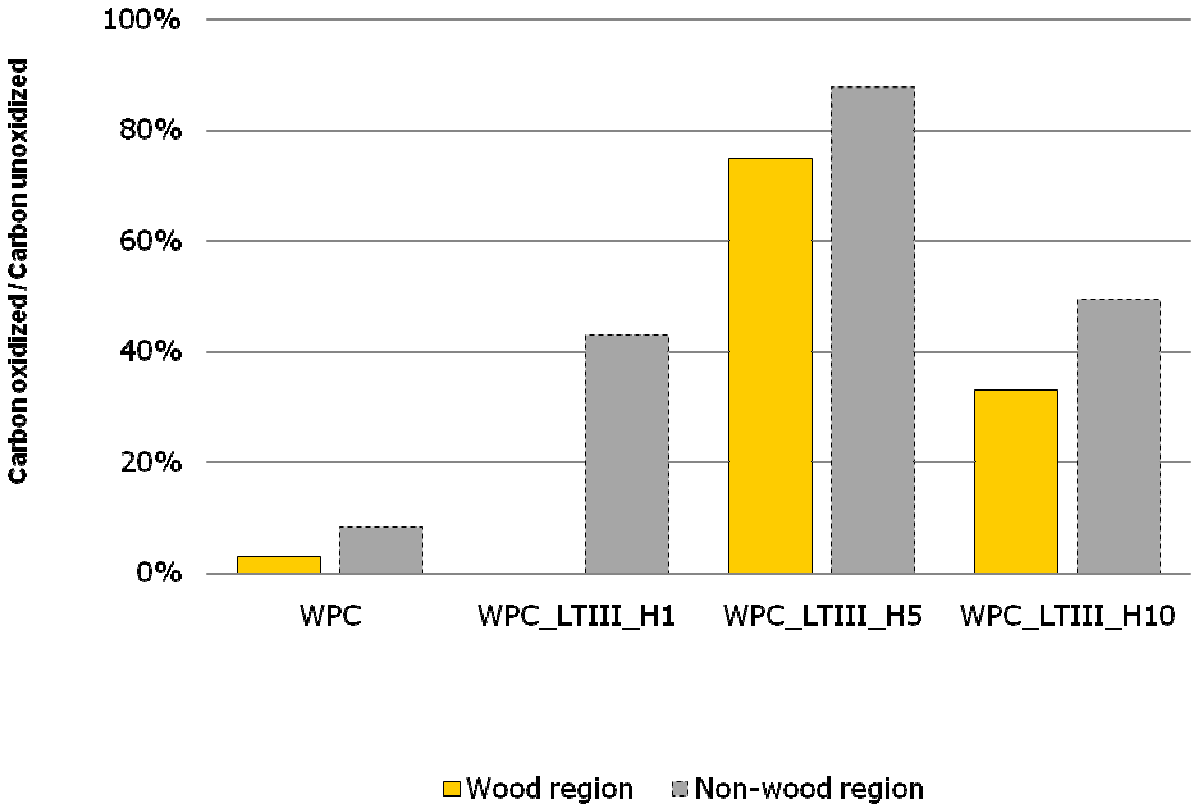

WPC non-wood region_LTIII_H5




XPS : X-ray photoelectron spectroscopy
WPC: Wood Plastic Composite planed and sanded
LTIII: Lectro Treat III Forced air plasma surface treater.
H: high level discharge length (2.5”);5 passes.

RESULTS

# XPS-Carbon spectra




XPS : X-ray photoelectron spectroscopy
WPC: Wood Plastic Composite planed and sanded
LTIII: Lectro treater (forced air plasma treatment)
H1, H5, H10: High level 1, 5 and 10 passes

RESULTS

## Atomic Force Microscopy Characterization WPCs

### Topography & Roughness

Topography was obtained in AMF Tapping Mode.
- ❖Resonance frequency: 300 kHz
- ❖Scanning rates: 0.3-0.6 Hz
- ❖Scan size: 5µm x 5µm

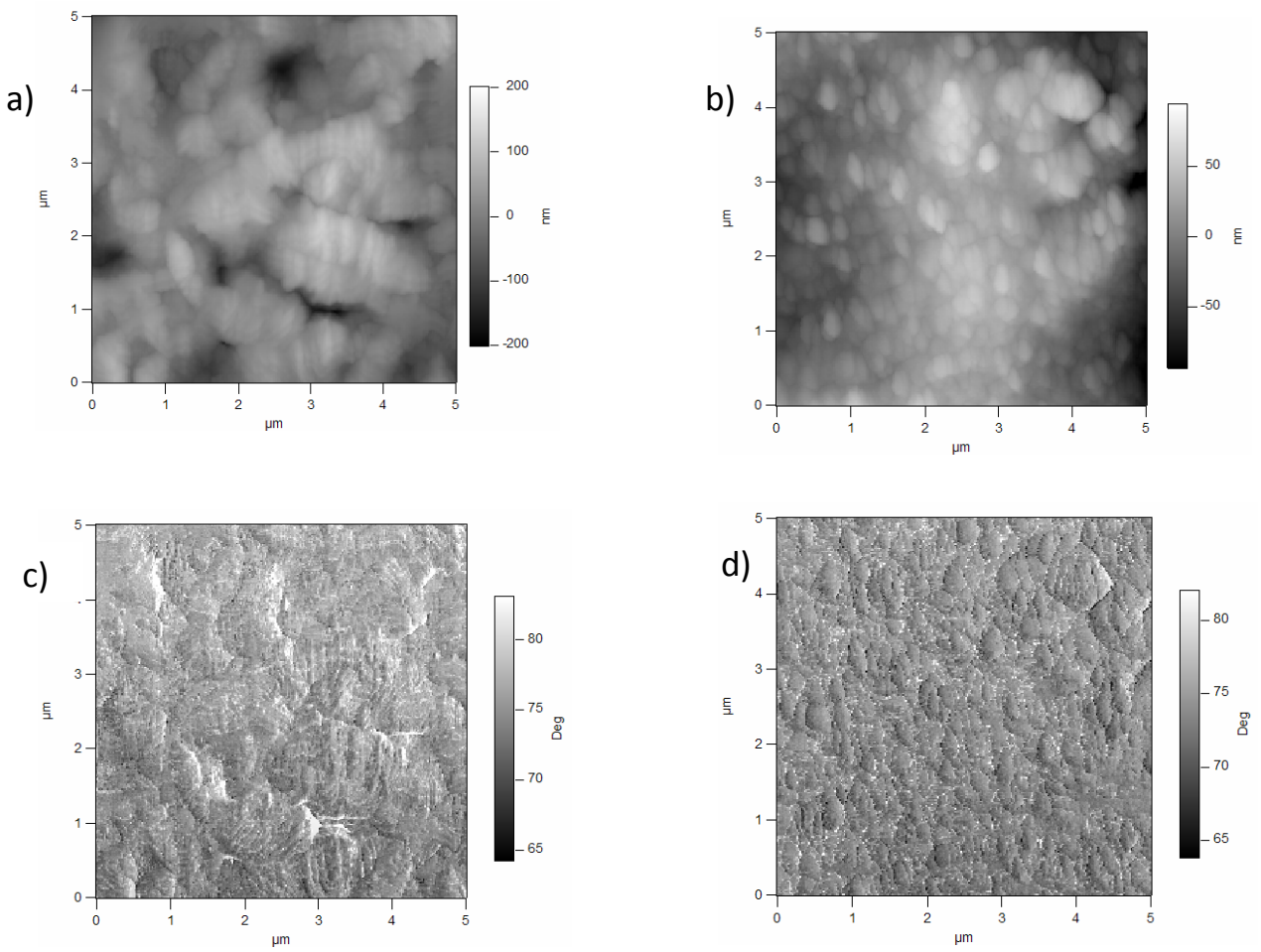
Tip characteristics:
- ❖Silicon tip
- ❖Mean spring constant: 42 N/m
- ❖Length: 160 µm
- ❖Tips end (diameter): 5 nm

1. Height, Amplitude and Phase images acquisition (per each sample).
2. Roughness determination (based on the analysis of 5 samples).

* Room temperature, room humidity, ambient pressure

RESULTS

# Atomic Force Microscopy Characterization WPCs components
## POLYPROPYLENE

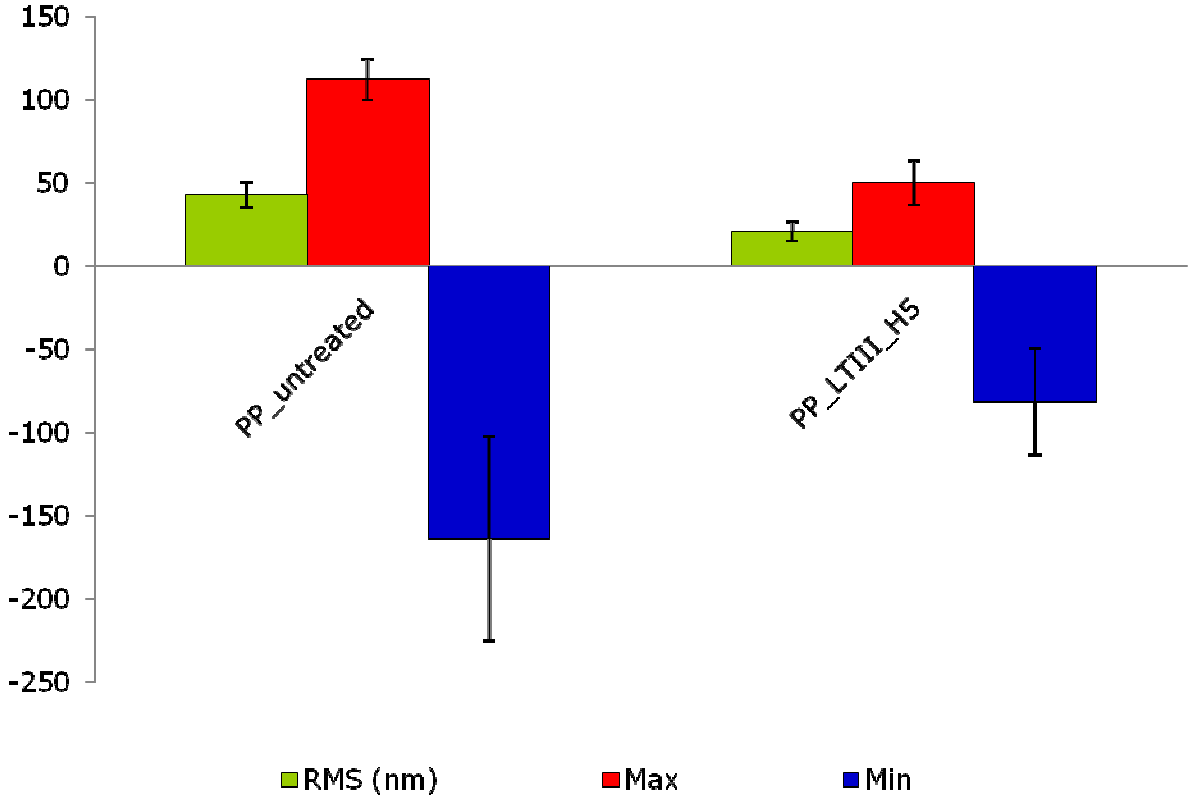



Polypropylene (height images), a) untreated, b) treated (LTIII_H5)
Polypropylene (phase images), c) untreated, d) treated (LTIII_H5)

RESULTS

# Atomic Force Microscopy Characterization WPCs components

## POLYPROPYLENE

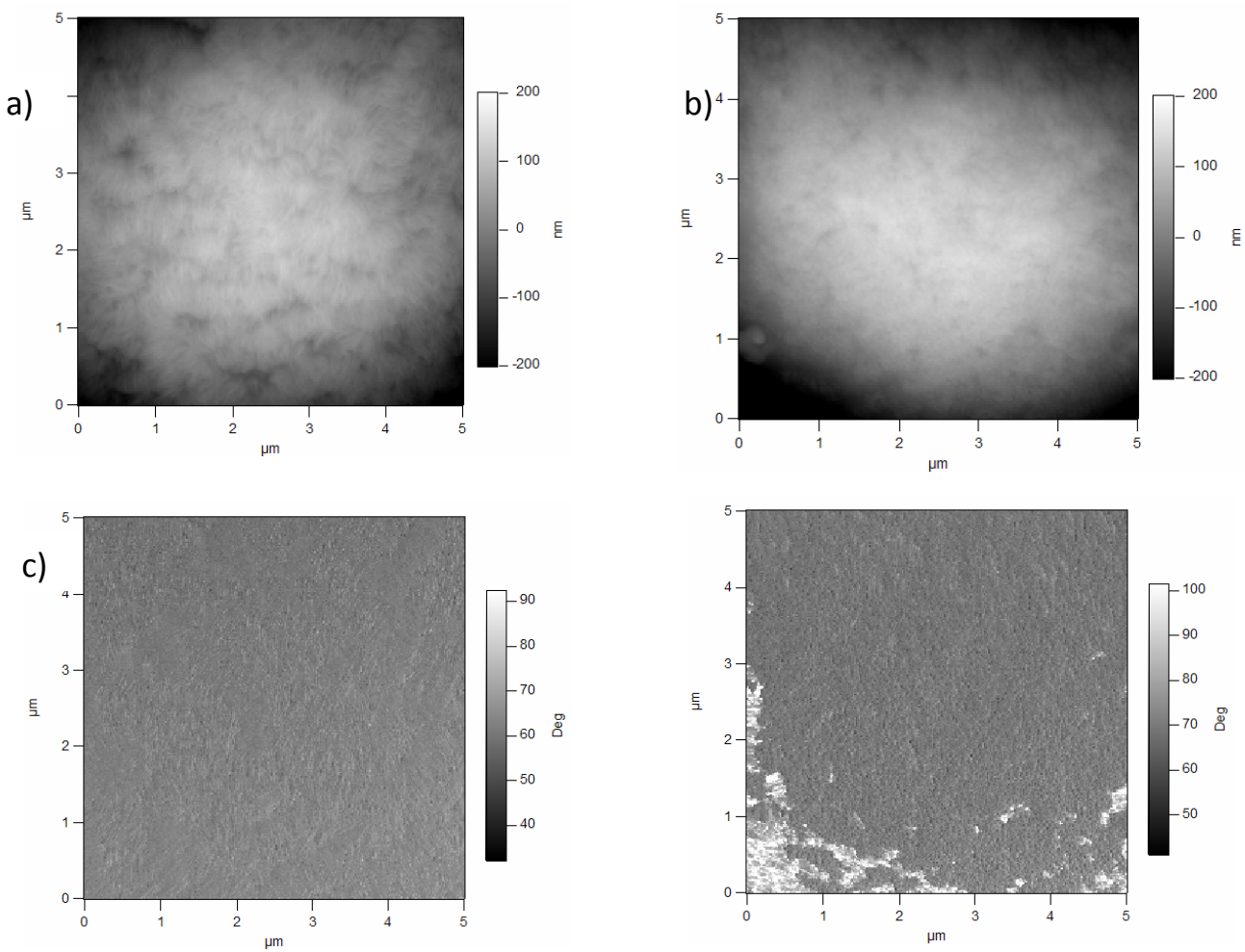



RMS: root-mean-square surface Roughness
LTIII: Lectro treater (forced air plasma treatment)
H5: High level discharge length; 5 passes

RESULTS

# Atomic Force Microscopy Characterization WPCs components

## COUPLING AGENT

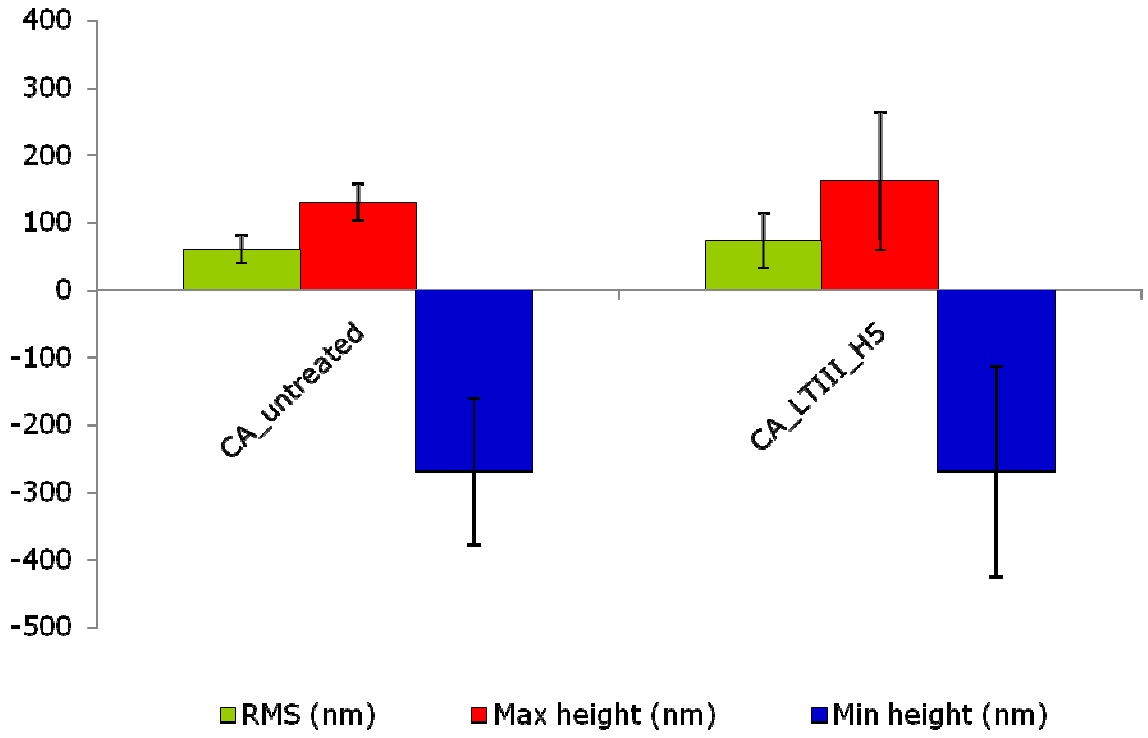



Coupling agent (height images), a) untreated, b) treated (LTIII_H5)
Coupling agent (phase images), c) untreated, d) treated (LTIII_H5)

RESULTS

# Atomic Force Microscopy Characterization WPCs components

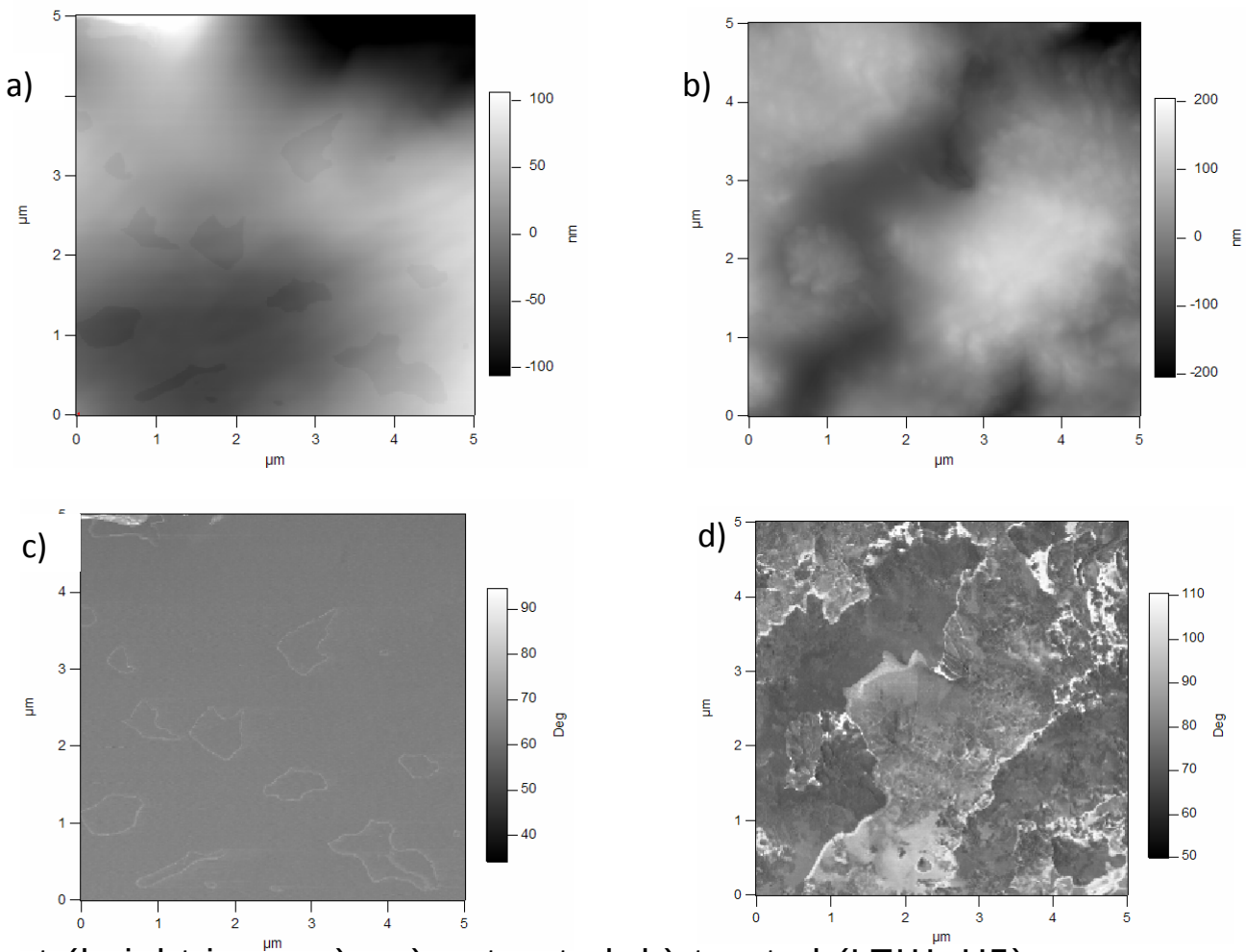
## COUPLING AGENT




RMS: root-mean-square surface Roughness
LTIII: Lectro treater (forced air plasma treatment)
H5: High level discharge length; 5 passes

RESULTS

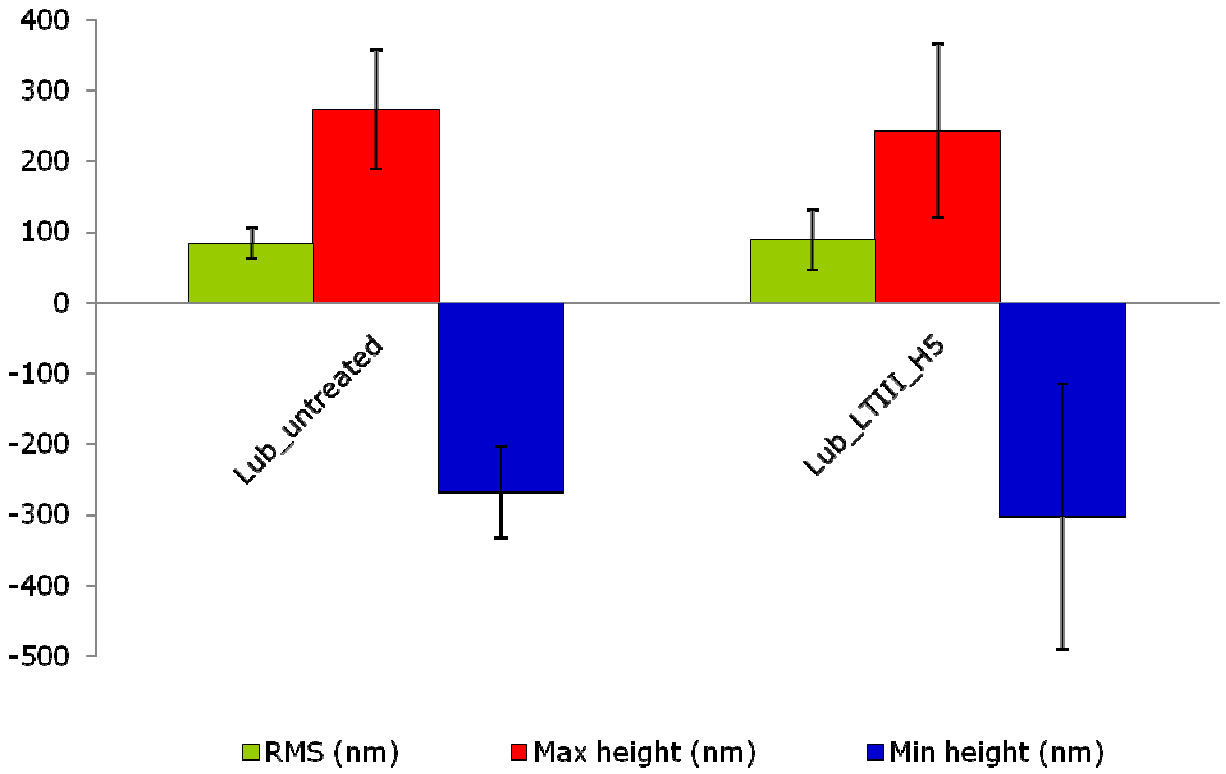
# Atomic Force Microscopy Characterization WPCs components
## LUBRICANT




Lubricant (height images), a) untreated, b) treated (LTIII_H5)
Lubricant (phase images), c) untreated, d) treated (LTIII_H5)

RESULTS

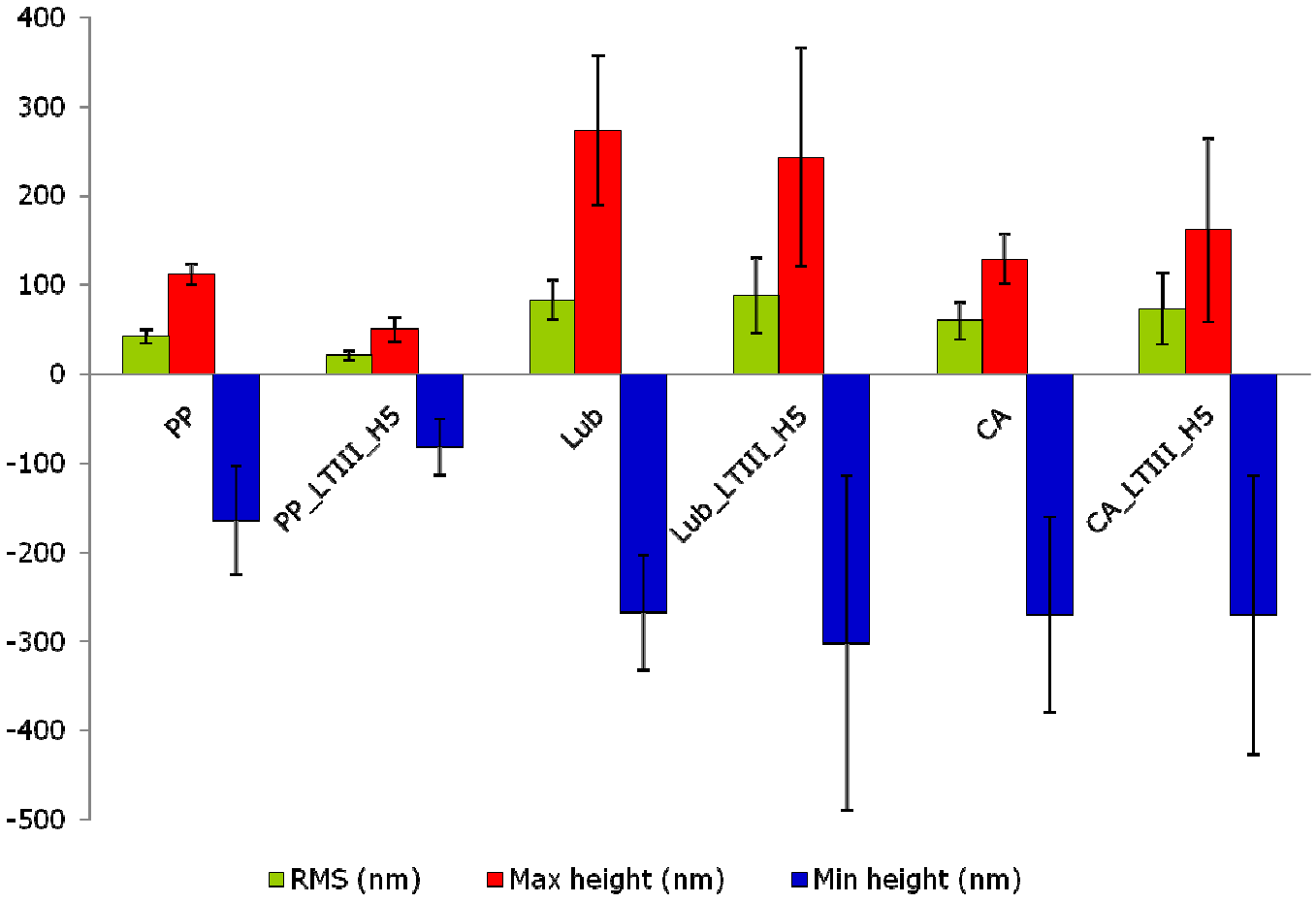
# Atomic Force Microscopy Characterization WPCs components
## LUBRICANT




RMS: root-mean-square surface Roughness
LTIII: Lectro treater (forced air plasma treatment)
H5: High level discharge length; 5 passes

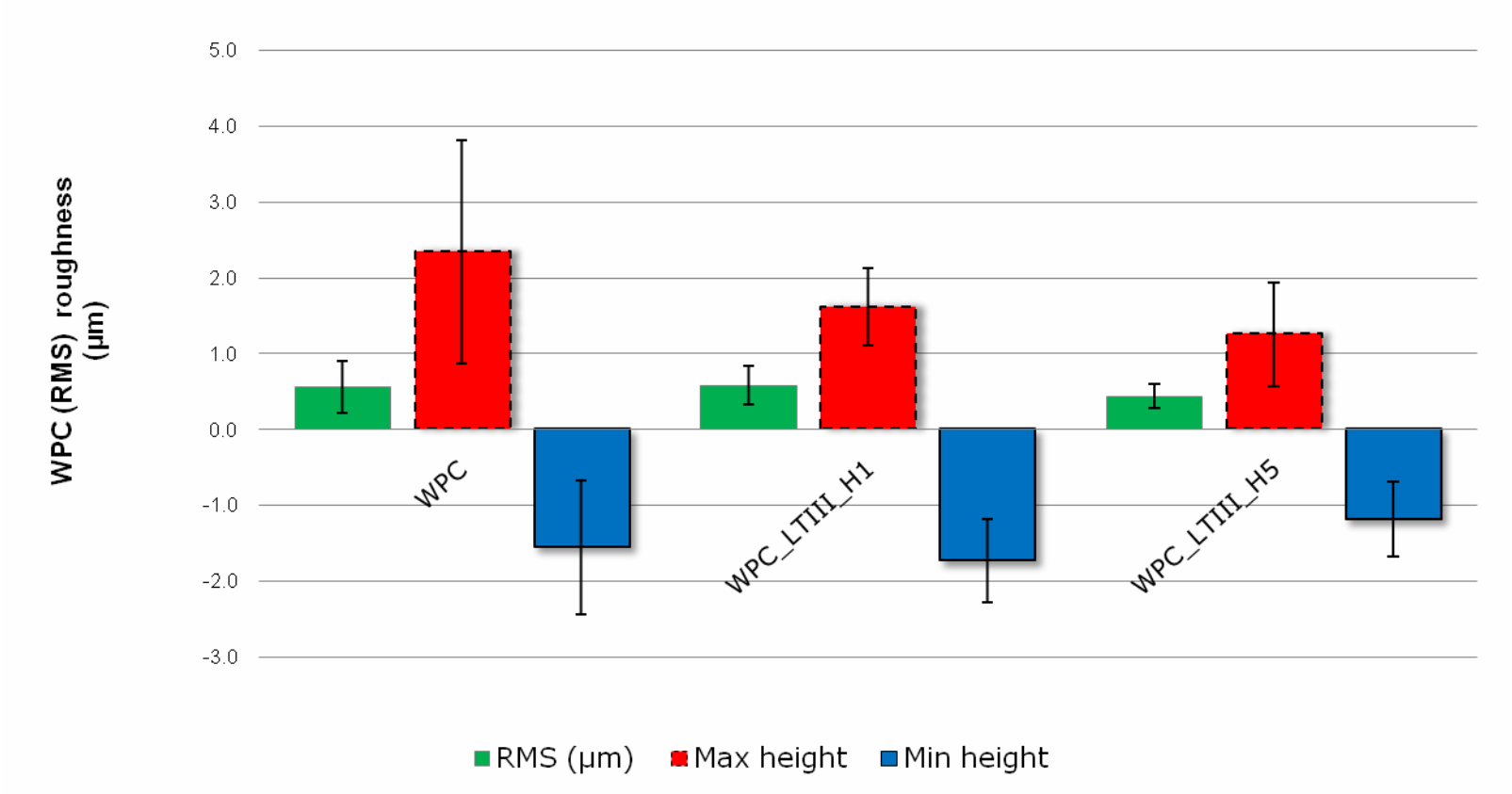
# Atomic Force Microscopy Characterization WPCs components

## ROUGHNESS SUMMARY



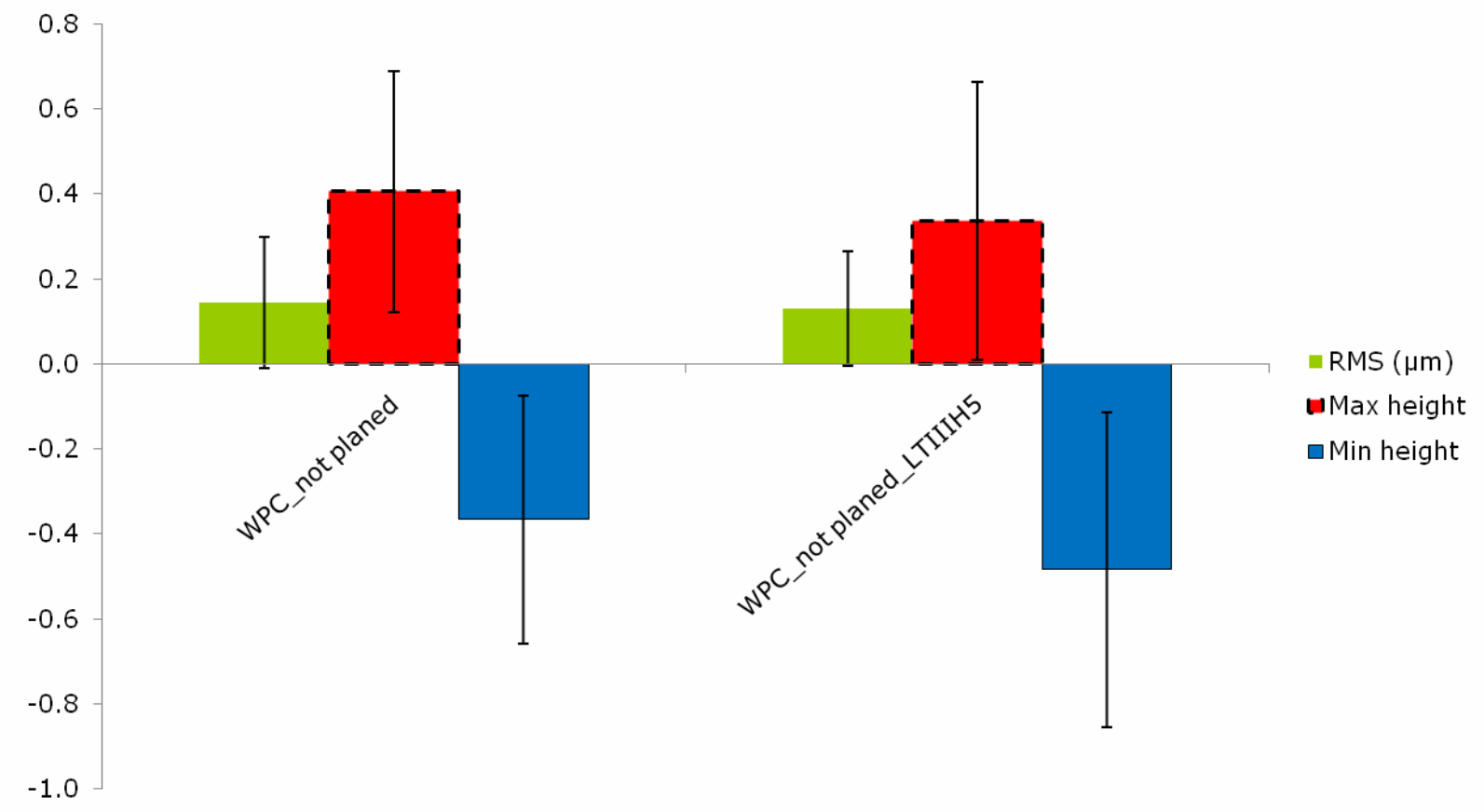
RESULTS

# Atomic Force Microscopy Characterization
## WPC PLANED & SANDED




RMS: root-mean-square surface Roughness
WPC: Wood Plastic Composite planed and sanded
LTIII: Lectro treater (forced air plasma treatment)
H1, H5: High level discharge length; 1, 5 passes

# Atomic Force Microscopy Characterization
## WPC NOT PLANED




RMS: root-mean-square surface Roughness
WPC: Wood Plastic Composite not planed
LTIII: Lectro treater (forced air plasma treatment)
H5: High level discharge length; 5 passes

RESULTS

# Adhesive forces (using AFM contact mode)

## Adhesive force determination

1.Spring constant determination.

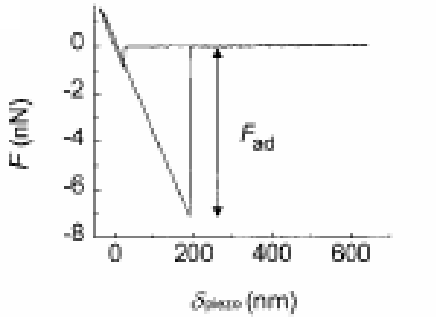
1.1 determine the slope of the contact region of a force curve to determine the sensitivity of the lever (in nm/V);
1.2 perform a thermal tune to determine the resonant frequency of the cantilever. An algorithm was subsequent used to compute the spring constant.

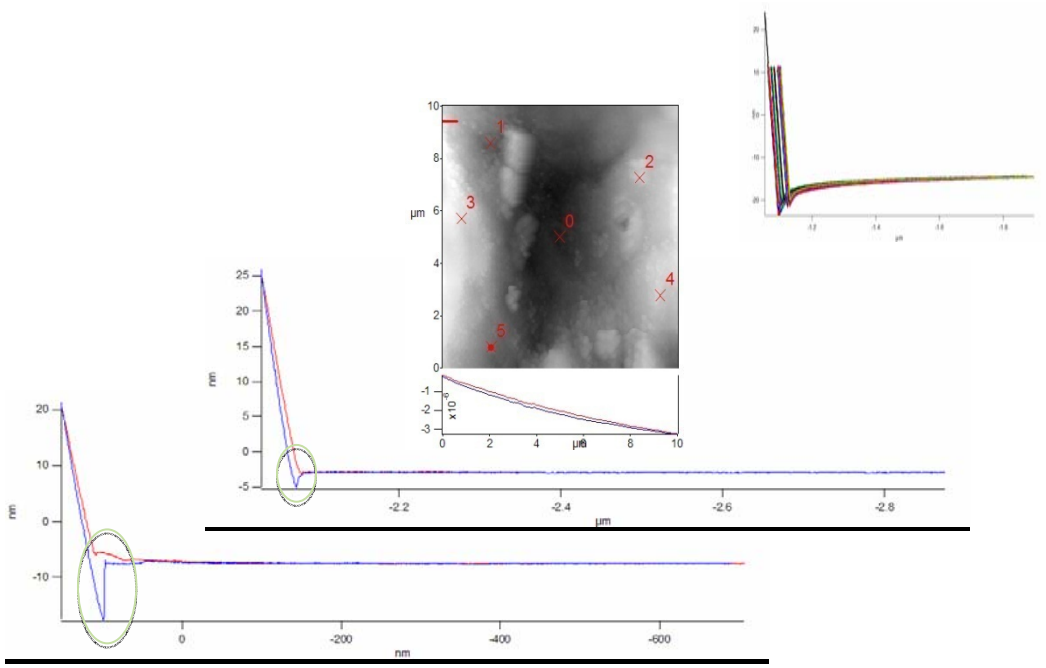
1.Force plots acquisition (more than hundred per each sample).
2.Histograms generation (representing number of force plots versus adhesive forces).

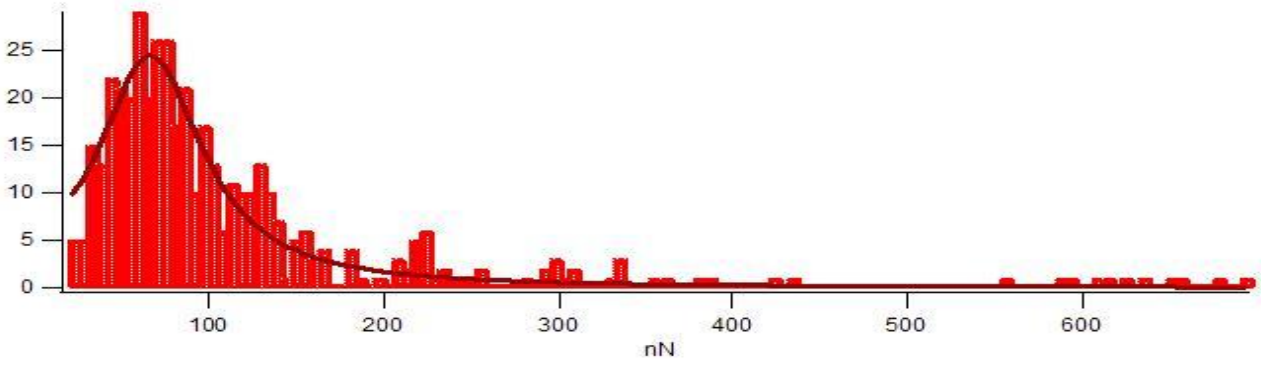
Adhesives forces were obtained in AMF Contact Mode.

RESULTS

- Adhesive forces



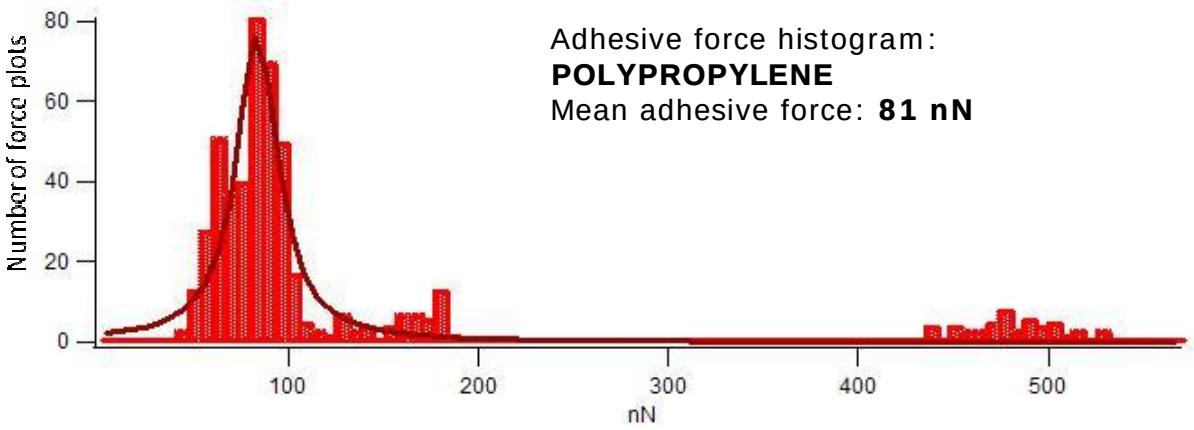



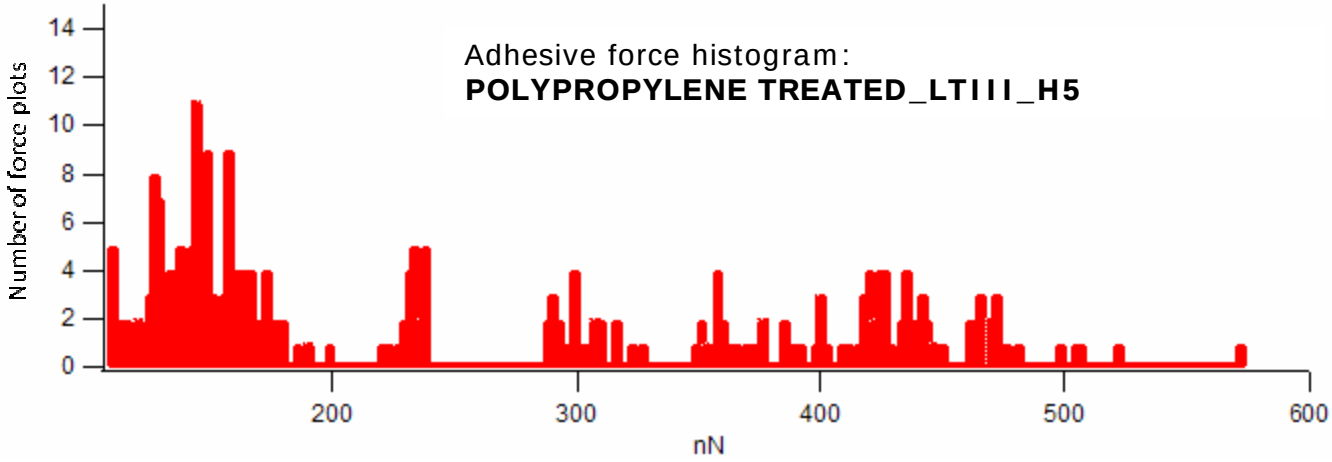
RESULTS

# AFM Adhesive forces WPCs components

## POLYPROPYLENE




Adhesive force histogram:
**POLYPROPYLENE**
Mean adhesive force: **81 nN**

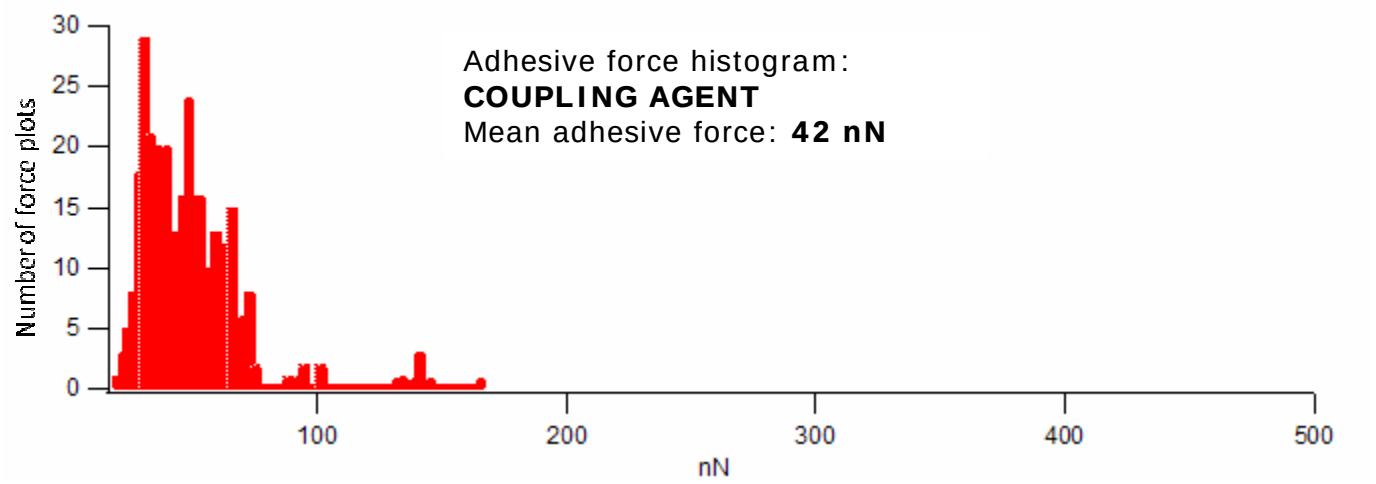


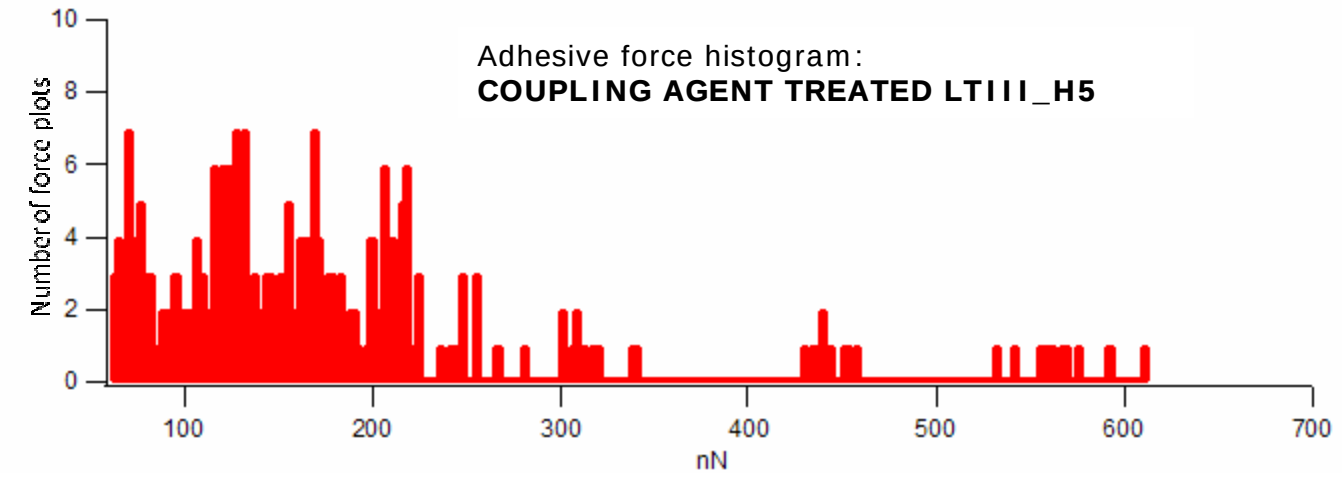

Adhesive force histogram:
**POLYPROPYLENE TREATED_LTIII_H5**

RESULTS

# AFM Adhesive forces WPCs components

## COUPLING AGENT

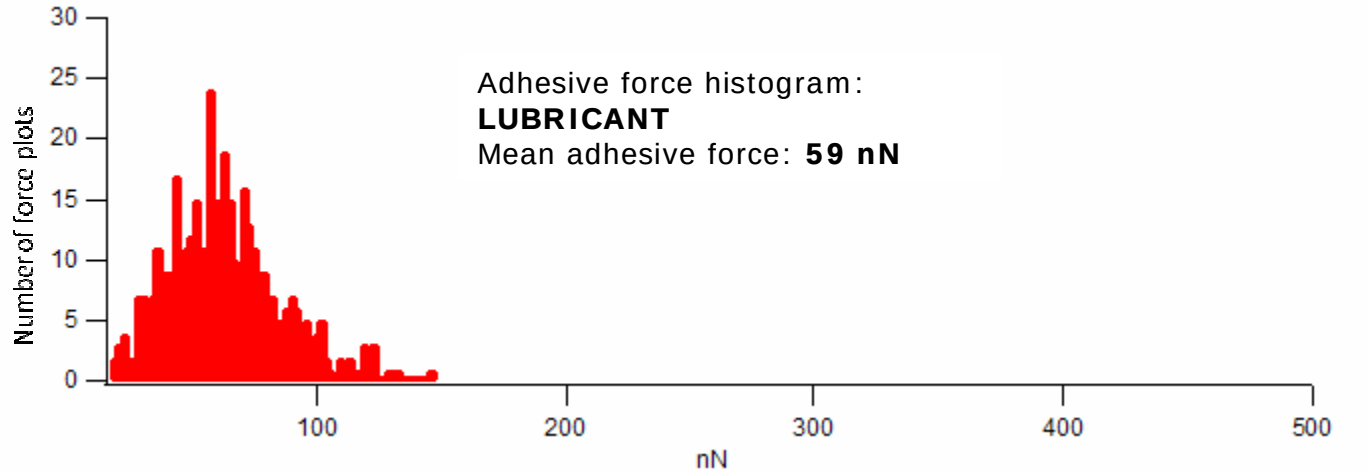


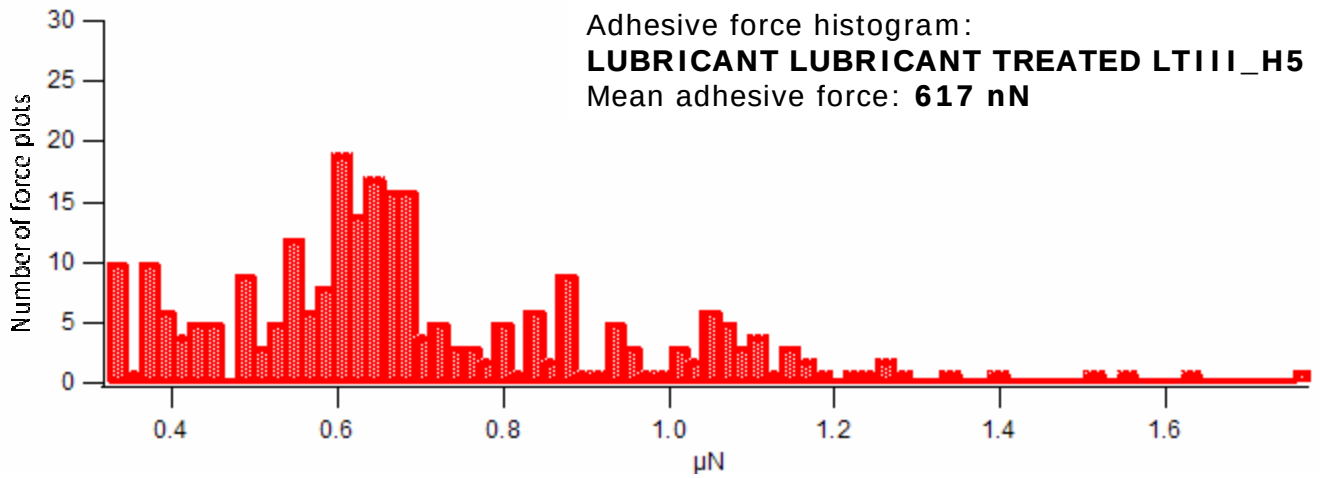
RESULTS

# AFM Adhesive forces WPCs components

## LUBRICANT



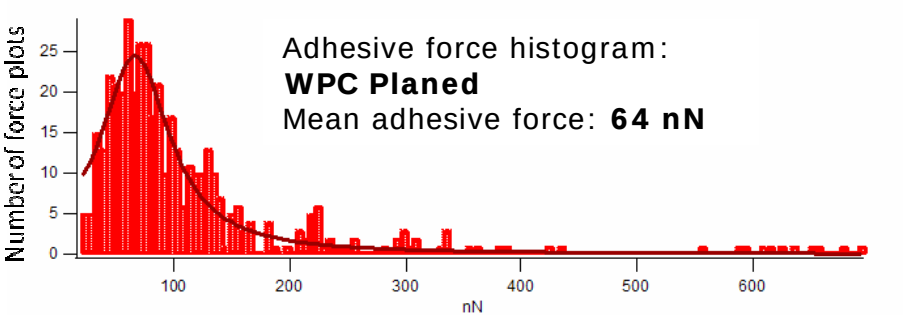

Adhesive force histogram:
**LUBRICANT**
Mean adhesive force: **59 nN**



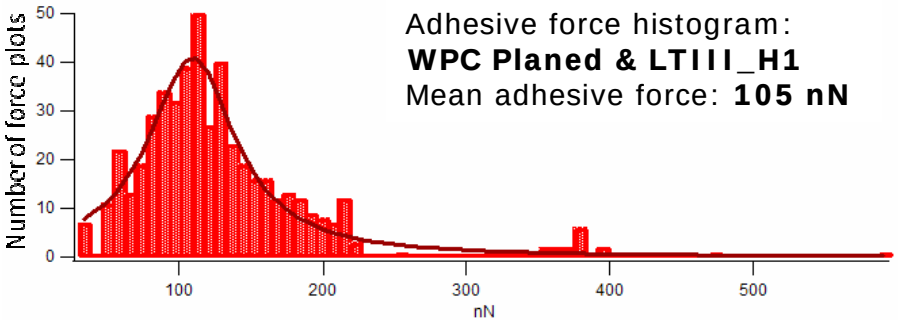

Adhesive force histogram:
**LUBRICANT LUBRICANT TREATED LTIII_H5**
Mean adhesive force: **617 nN**

RESULTS

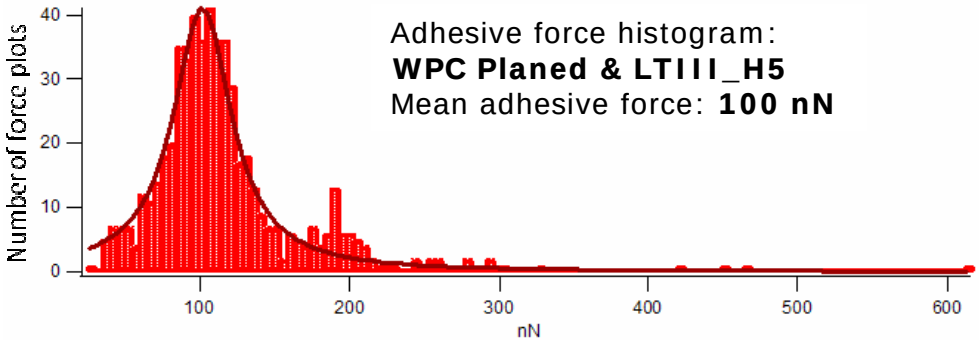
# Atomic Force Microscopy Characterization WPCs PLANED




Adhesive force histogram:
**WPC Planed**
Mean adhesive force: **64 nN**




Adhesive force histogram:
**WPC Planed & LTIII_H1**
Mean adhesive force: **105 nN**

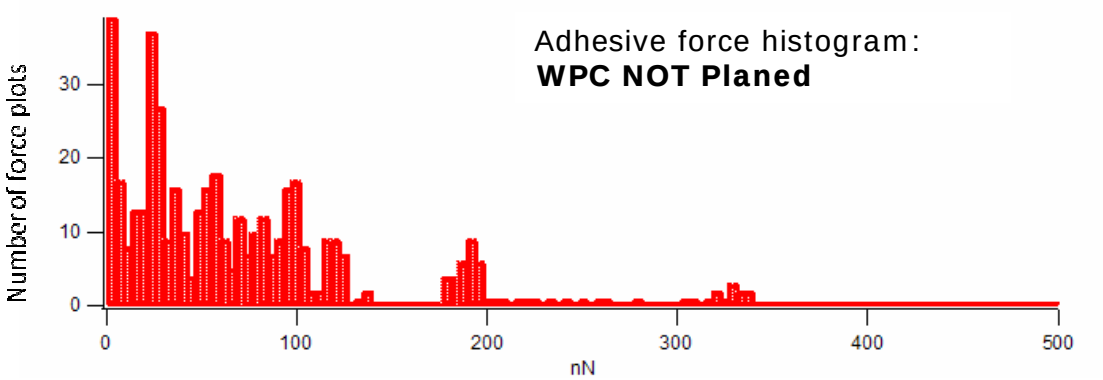


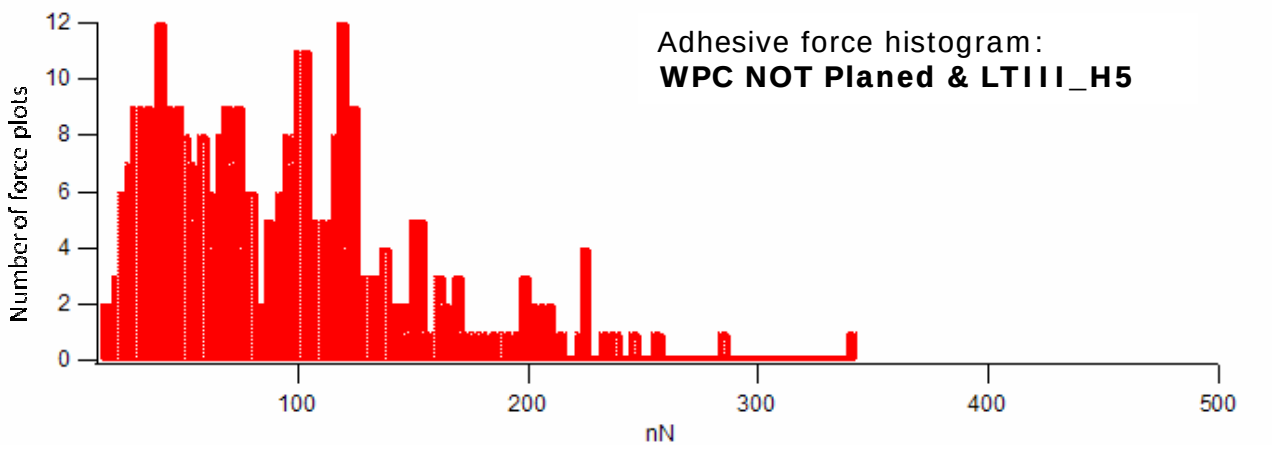

Adhesive force histogram:
**WPC Planed & LTIII_H5**
Mean adhesive force: **100 nN**

WPC: Wood Plastic Composite planed and sanded
LTIII: Lectro treater (forced air plasma treatment)
H1, H5: High level discharge length; 1, 5 passes

RESULTS

# Atomic Force Microscopy Characterization WPCs NOT PLANED



RESULTS

# Atomic Force Microscopy Characterization WPCs

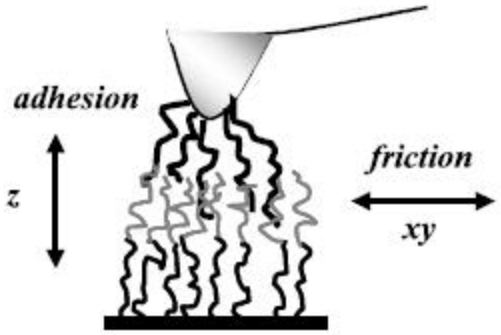
| Surface analyzed | Mean Adhesion force * (nN) |
|---|---|
| Polypropylene | 81 |
| Coupling agent | 42 |
| Lubricant | 59 |
| WPC planed | 64 |
| WPC planed_LTIII_H1 | 105 |
| WPC planed_LTIII_H5 | 100 |
| WPC not planed | |
| WPC not planed_LTIII_H5 | |

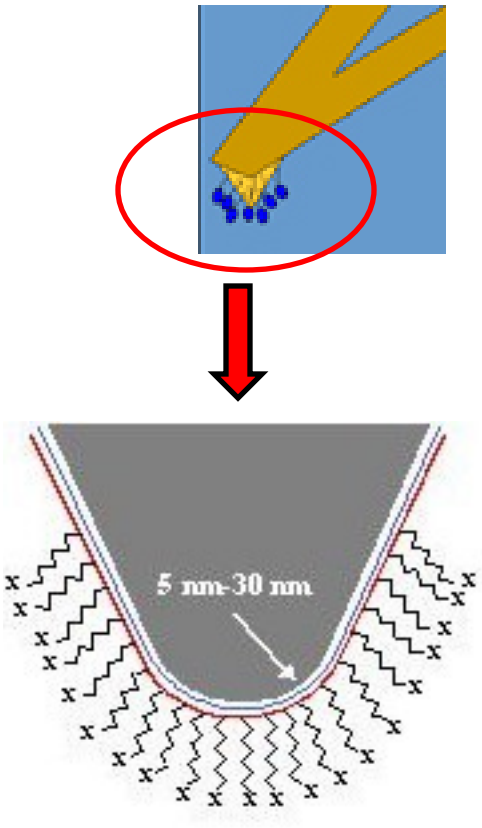
WPC: Wood Plastic Composite planed and sanded
LTIII: Lectro treater (forced air plasma treatment)
H1, H5: High level discharge length; 1, 5 passes

RESULTS

- Adhesive forces

using AFM & tips functionalized

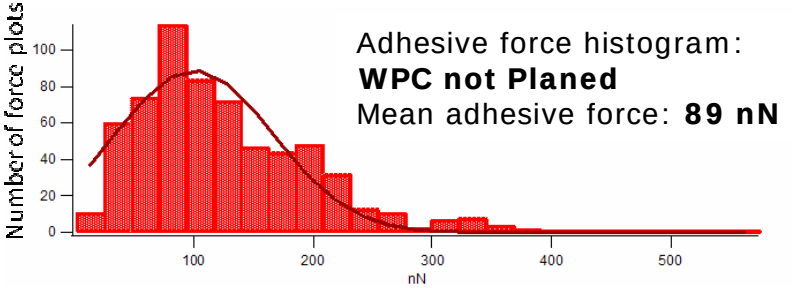


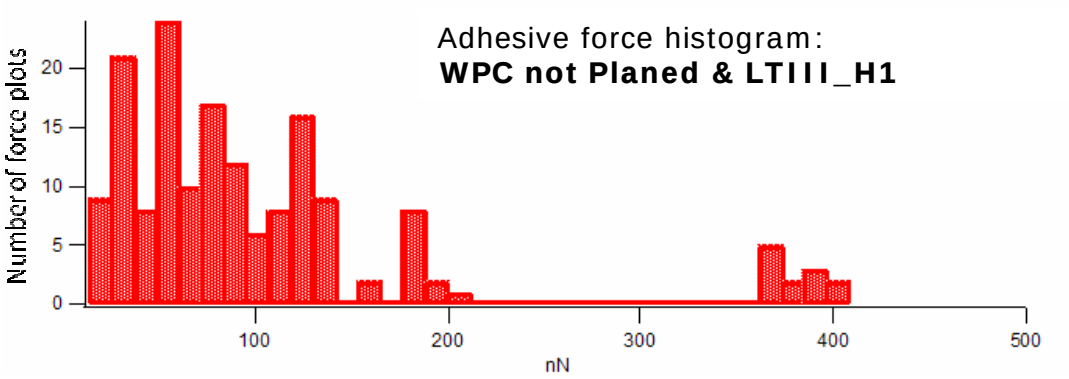

Functionalized tips used:
hydroxyl and methyl groups.

RESULTS

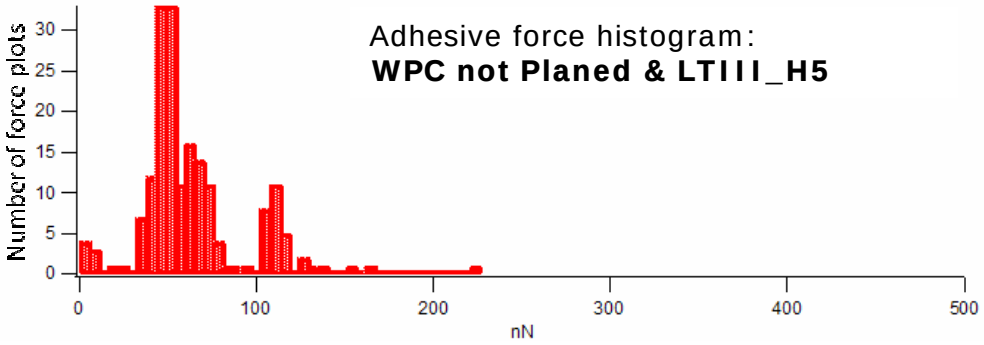
# Adhesive forces WPCs NOT PLANED_OH groups




Adhesive force histogram:
**WPC not Planed**
Mean adhesive force: **89 nN**



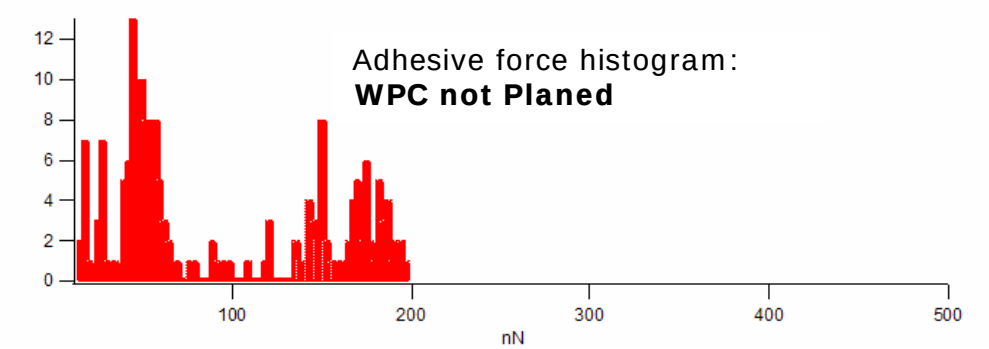

Adhesive force histogram:
**WPC not Planed & LTIII_H1**

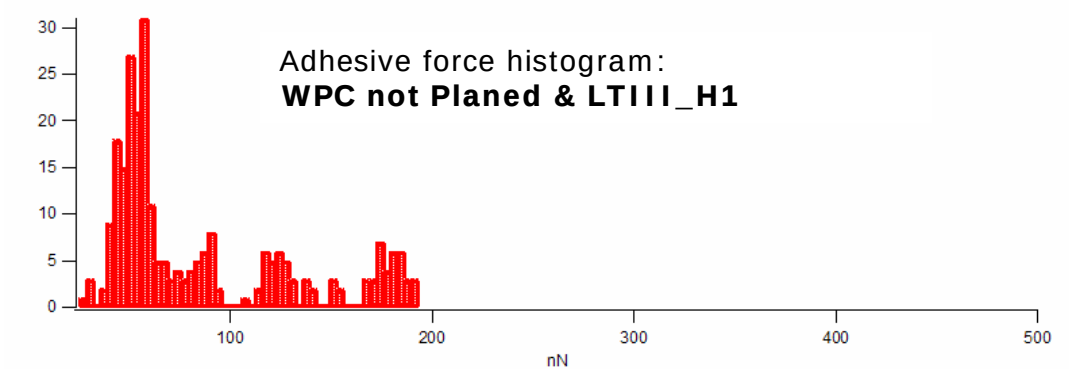


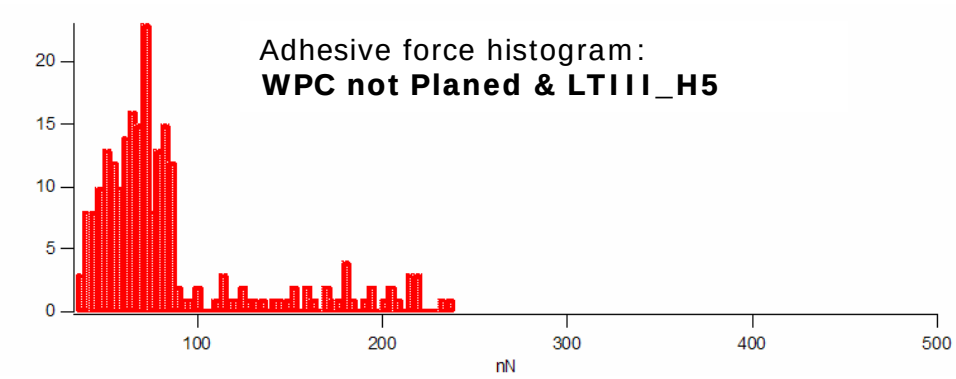

Adhesive force histogram:
**WPC not Planed & LTIII_H5**

RESULTS

# Adhesive forces WPCs NOT PLANED_CH3 groups



# Overview

- Introduction
- Overall goal
- Methods
- Results
- Discussion & Conclusions

# Discussion & Conclusions

- The use of a silicon AFM tips allows to distinguish different level of adhesion forces between the individual components of WPC and WPC treated surfaces.

- AFM results (using silicon tips) are comparable with those obtained for surface energy determinations (sessile drop method), XPS and shear strength for WPCs.

- Plasma treatment chemically modifies the planed WPC surfaces as opposed to making physical modifications.

- A deep evaluation of the main causes for hysteresis in the adhesion zone must be addressed. Especially for the lubricant component.

- Differences can be appreciated using functionalized AFM tips on WPC surfaces, however more work it is necessary to perform to evaluate and quantify adhesive forces.

# Questions & Comments