-

Mechanical Characterization of Wood-Adhesive Interphase with an Improved Nanoindentation Technique

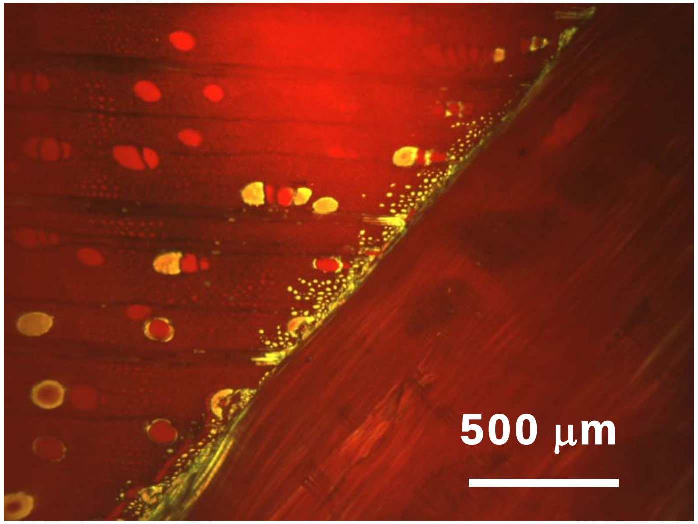
YU Yan [1,2] , JIANG Zehui [1]
C.R. FRIHART [2] , J.E. JAKES [2]

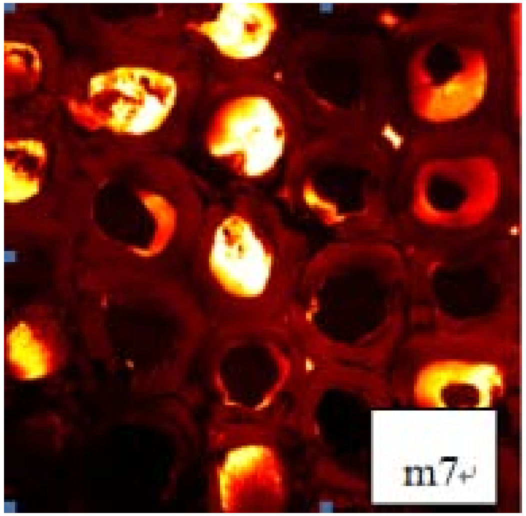
[1]International Center for Bamboo and Rattan, Beijing, China

[2] Forest Product Lab, USDA FS, Madison, WI, USA

1. Introduction

- **Adhesive penetration into wood structure is believed to be vital to the durability of wood-adhesive bondlines (Frihart, 2009).**
- **Flowing into the micron-scale cavities of wood**





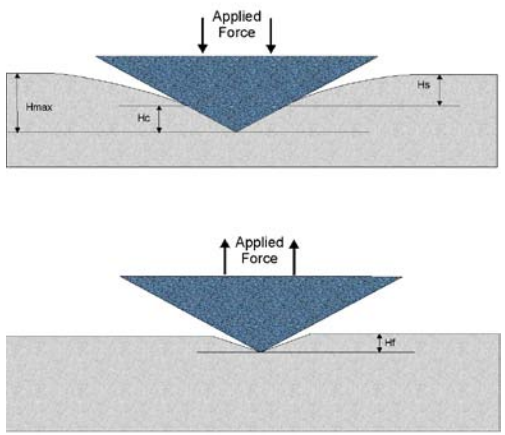
Fluorescence microscope and CLSM can directly show the distribution of adhesive in wood at micron scale

- **Infiltrating into the cell walls.**

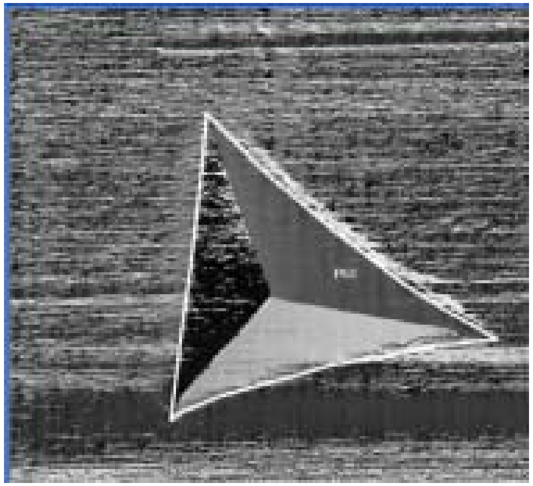
SEM-EDAX, UV microscopy, Thermal AFM et al

1. Introduction

- Understanding how infiltrated adhesives affect wood cell wall properties is critical to understanding how to make durable wood-adhesive bondlines.
- Nanoindenatation was recently used to characterize wood-adhesive bondlines, which is based on the reasonable assumption that the infiltration of adhesives into wood cell walls might change their mechanical properties.



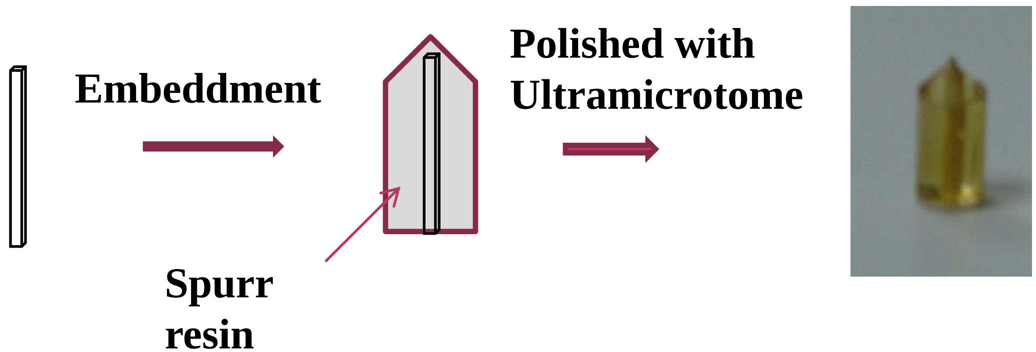
Nanoindentation testing



Scan Size: 4 μm *4 μm

1. Introduction (cont)

◆ Problem 1: sample preparation



- Long time (2-3 day);
- Cell wall may be chemically modified
- embedment medium might interfere with the adhesive distribution in the bonding area

1. Introduction (cont)

Problem 2: suitability of Nanoindentation

Oliver-Pharr method (standard method):

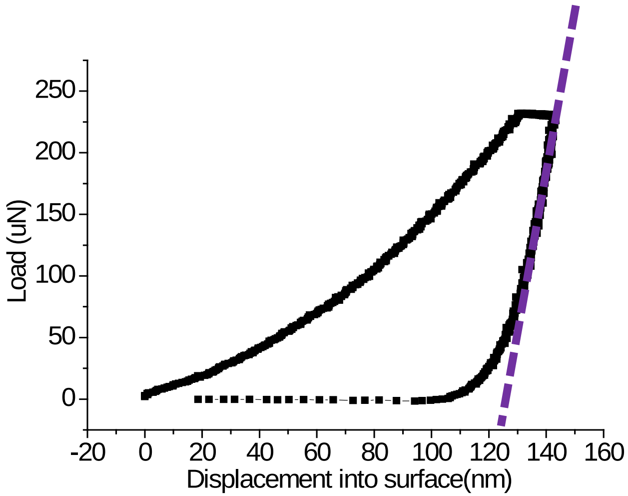
Assumptions:

- **homogeneous half spaces;**
- **isotropic**
- **rigidly supported in the testing machine**

$$E_r = \frac{\sqrt{\pi}}{2 * C_p} \frac{1}{\sqrt{A_c}}$$

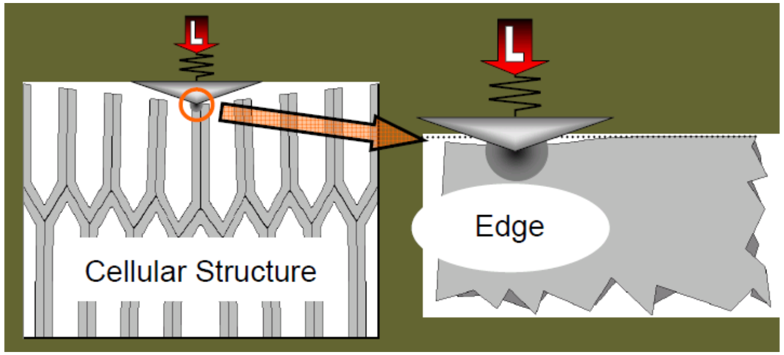
$$H = \frac{P_{\max}}{A_c}$$

$$C_p = C_t - C_m$$ (constant)



- **C_p: actual unloading compliance**
- **Ac: projected area of indents, obtained by calculation.**

1. Introduction (cont)



(From Jakes)

Assumptions might be violated in the nanoindentation testing of wood cell wall or adhesive bonding

Improved nanoindentation test **(Jakes et al , 2008; 2009)**

$$C_p = C_t - C_m - C_s$$

A_c: measured with AFM

- C_s: **Structural compliance induced by the heterogeneity, free edge, cracks, pores of samples**

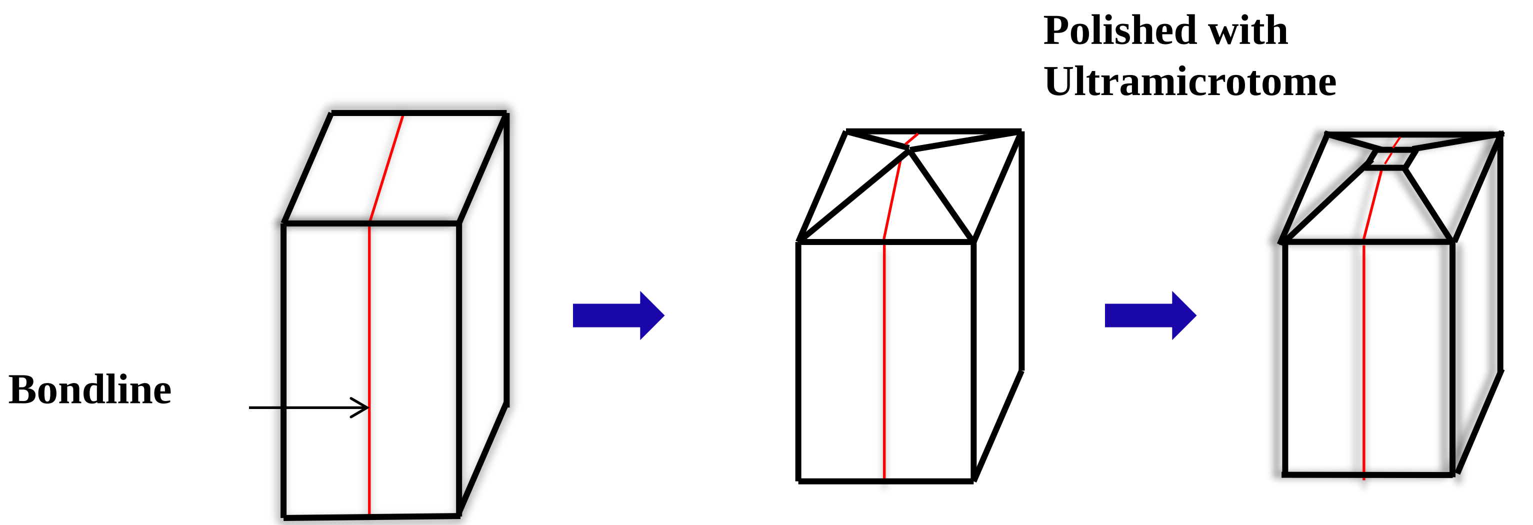
2. Materials and Methods

◆ **Southern pine veneers with a thickness about 5 mm were bonded together with PF, UF, epoxy and EPI adhesives with the bonding parameters shown in Table 1.;**

	Temperature (°C)	Time (Min)	Pressure (MPa)	Application Rate (g/m^2)
PF	158	6	1.2	80
UF	125	5	1.4	150
Epoxy	Room temperature	180	0.86	174
EPI	Room temperature	Overnight	1.4	180

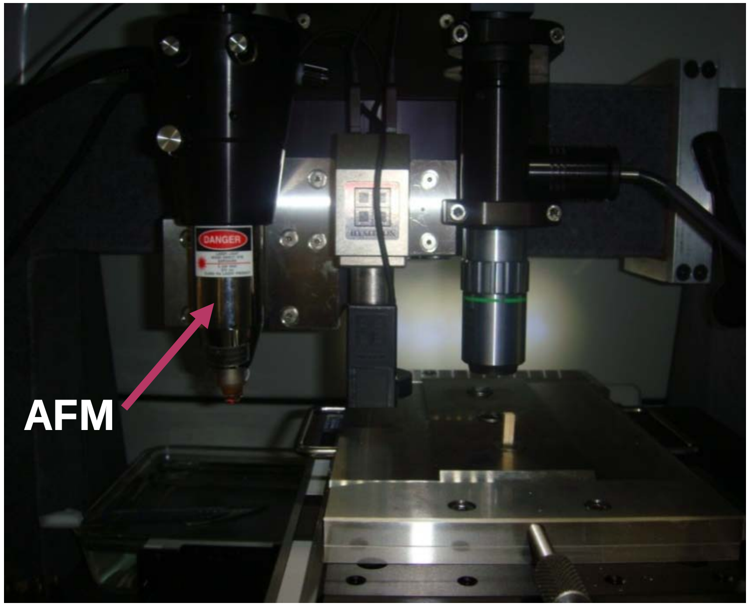
Sample preparation

◆ **Unembedment method** **(Jakes et al, J Mater Res, 2008)**



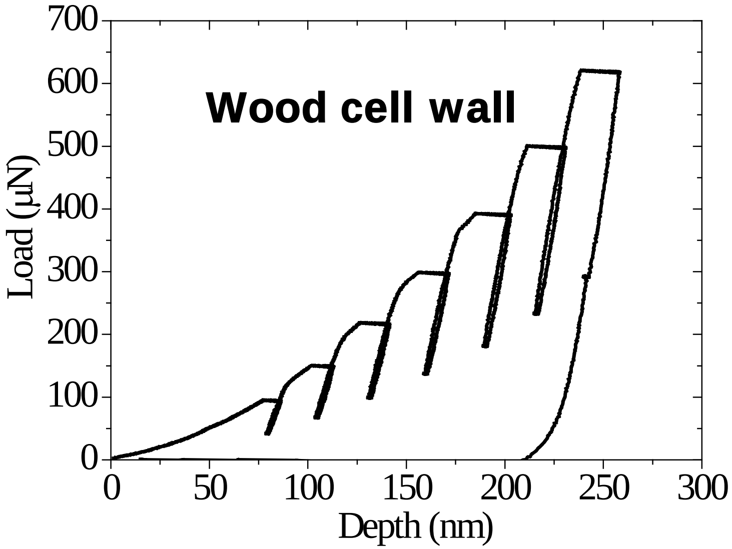
- **1 h**
- **Cell wall remains natural state**

Equipment



Triboindenter (Hysirton, USA)

Cs calculated from SYS plots



Typical multiload curves

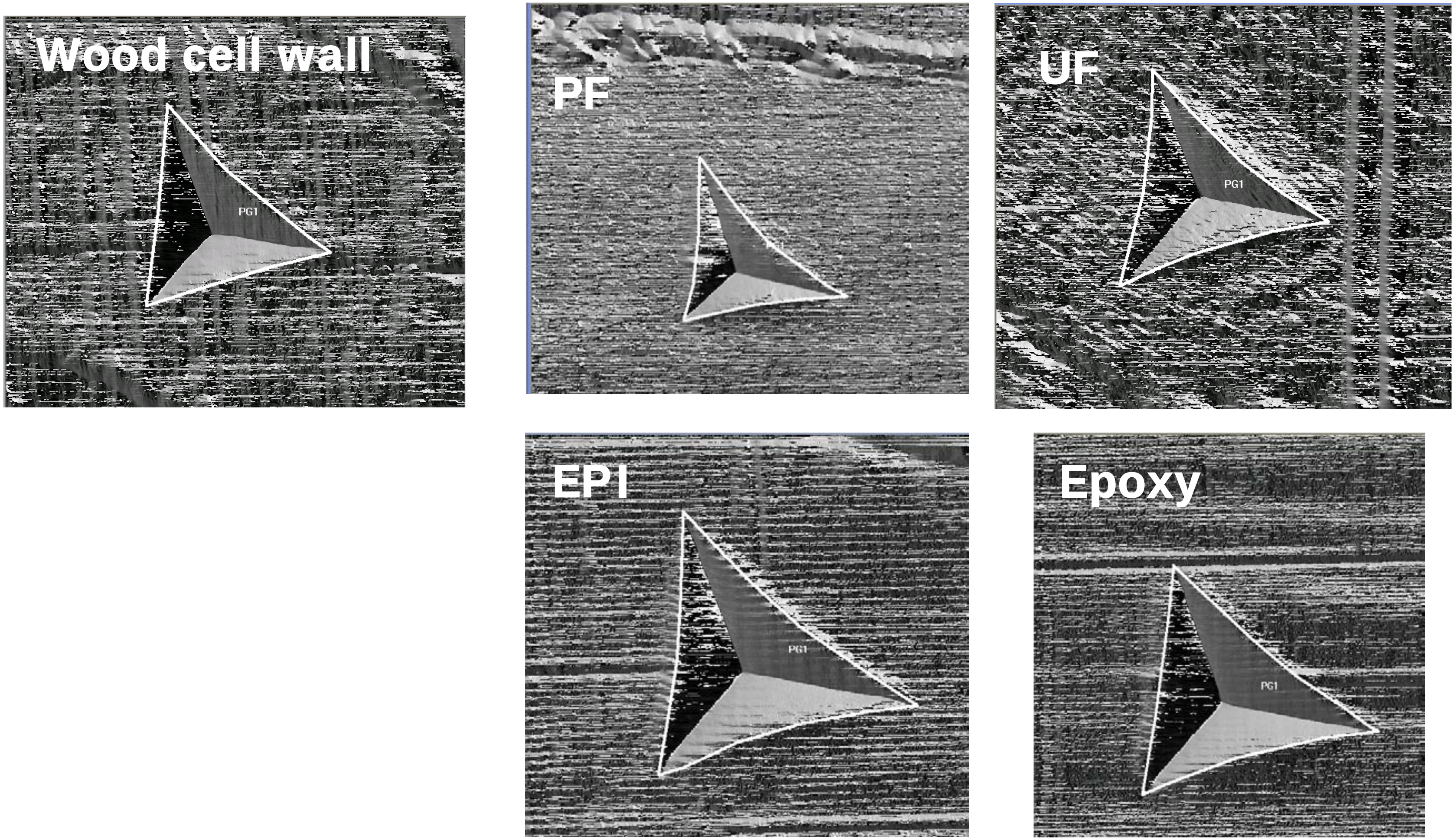
SYS plots

$$C_t L_{max}^{1/2} = \left(C_m + C_s\right) L_{max}^{1/2} + J_0^{1/2}$$

Dependent variable

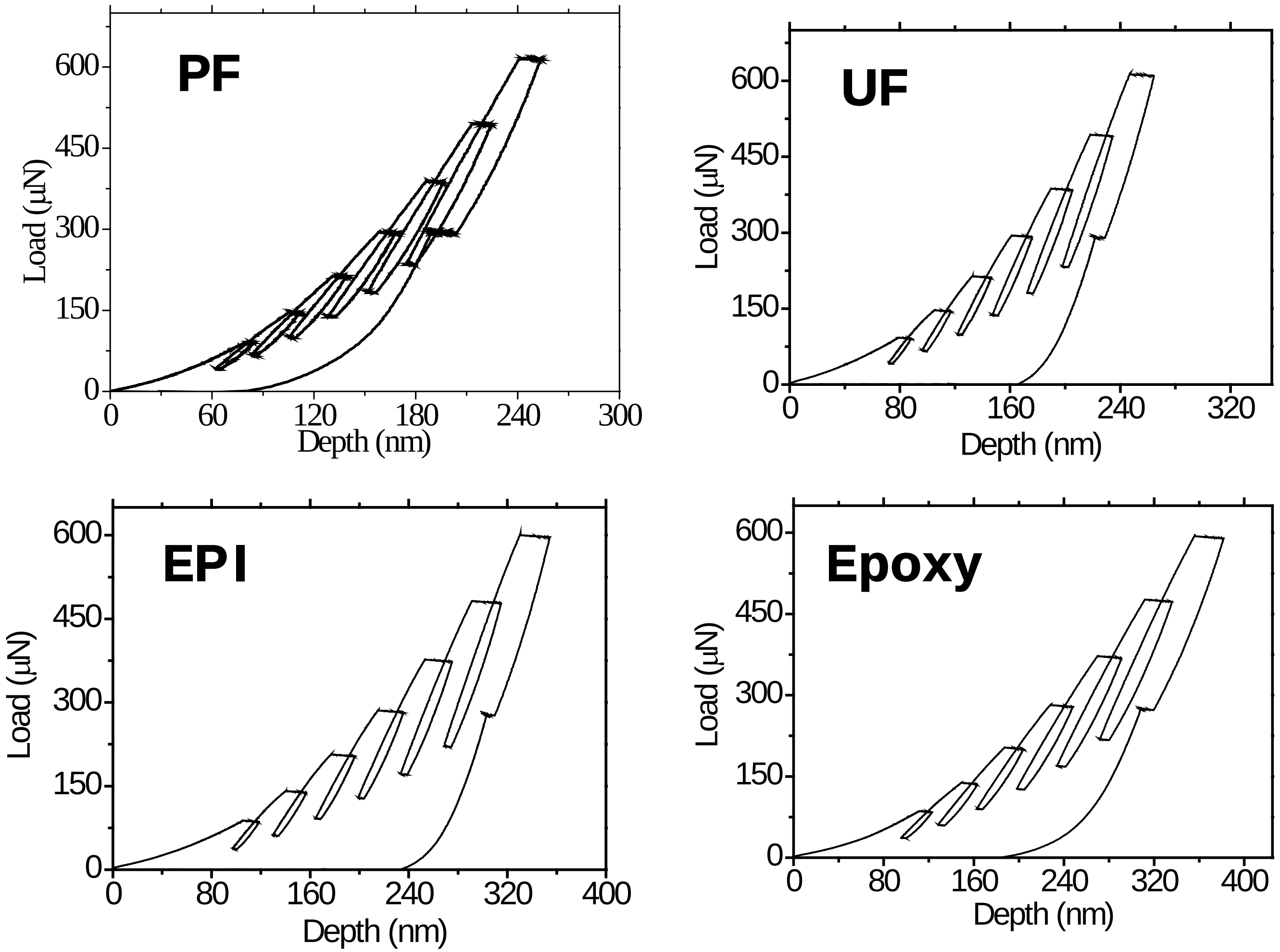
Variable

Projected area measured with AFM

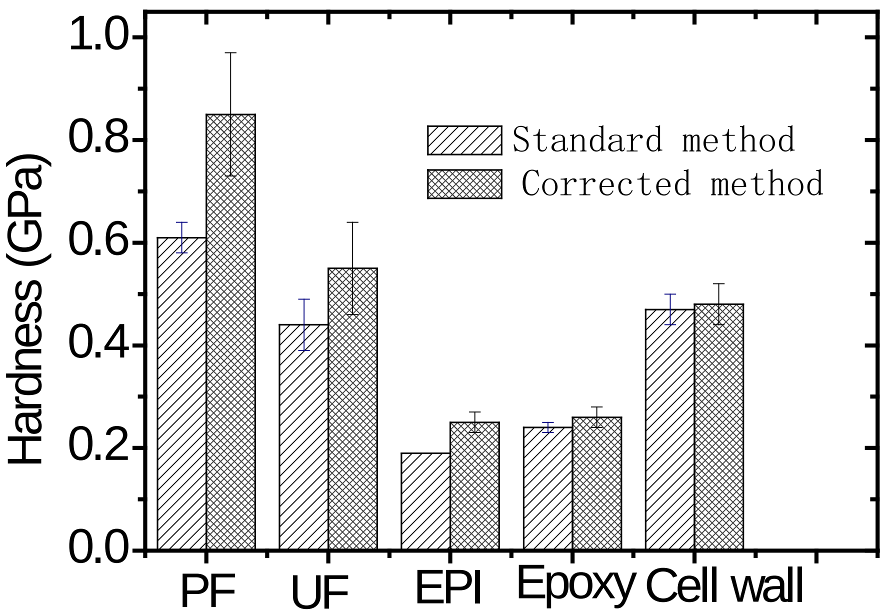


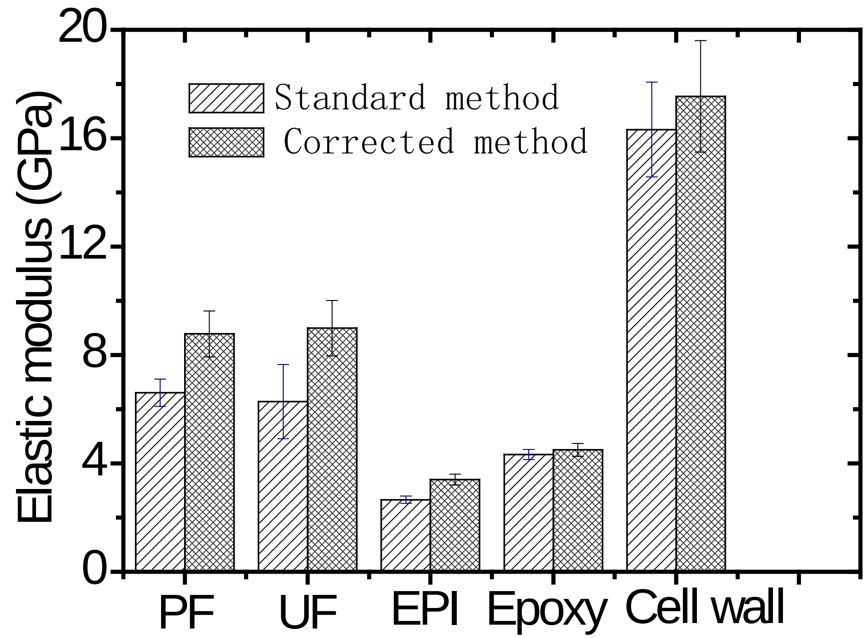
Scan Size: 4 μm *4 μm

3. Results and discussion



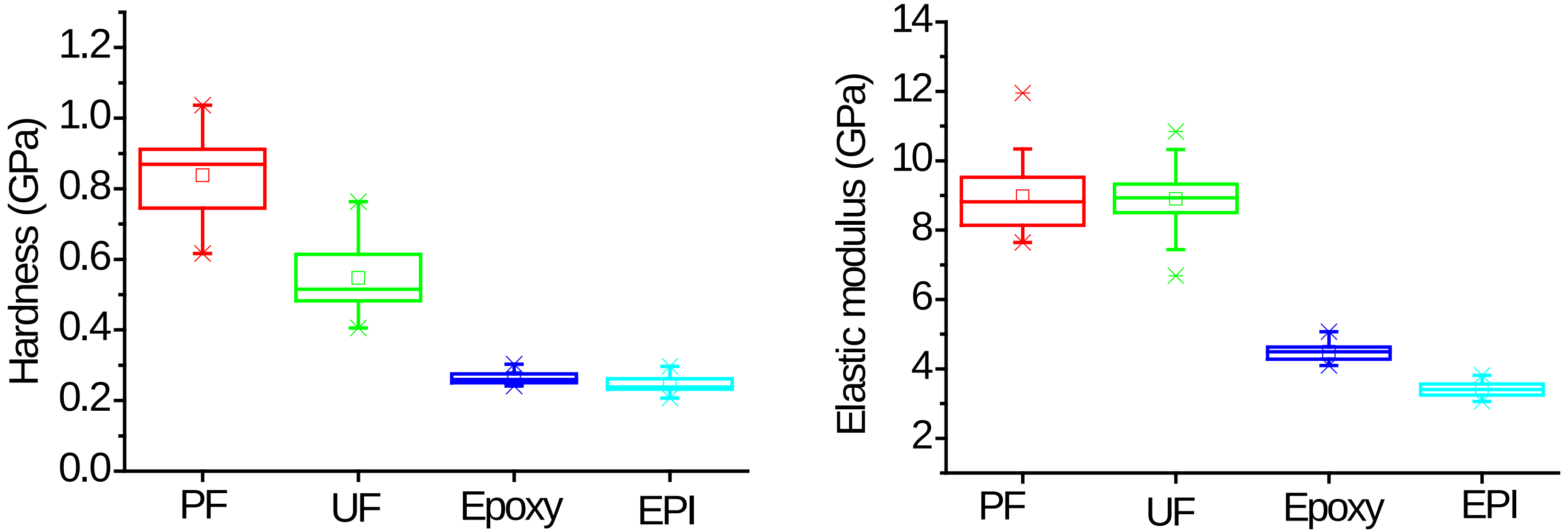
Typical load-depth curves of multiload indents on the four adhesives





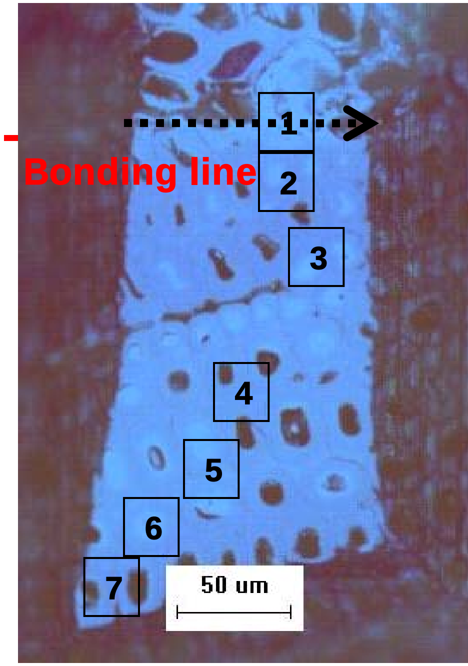
Comparison between standard method and corrected method in MOE and hardness

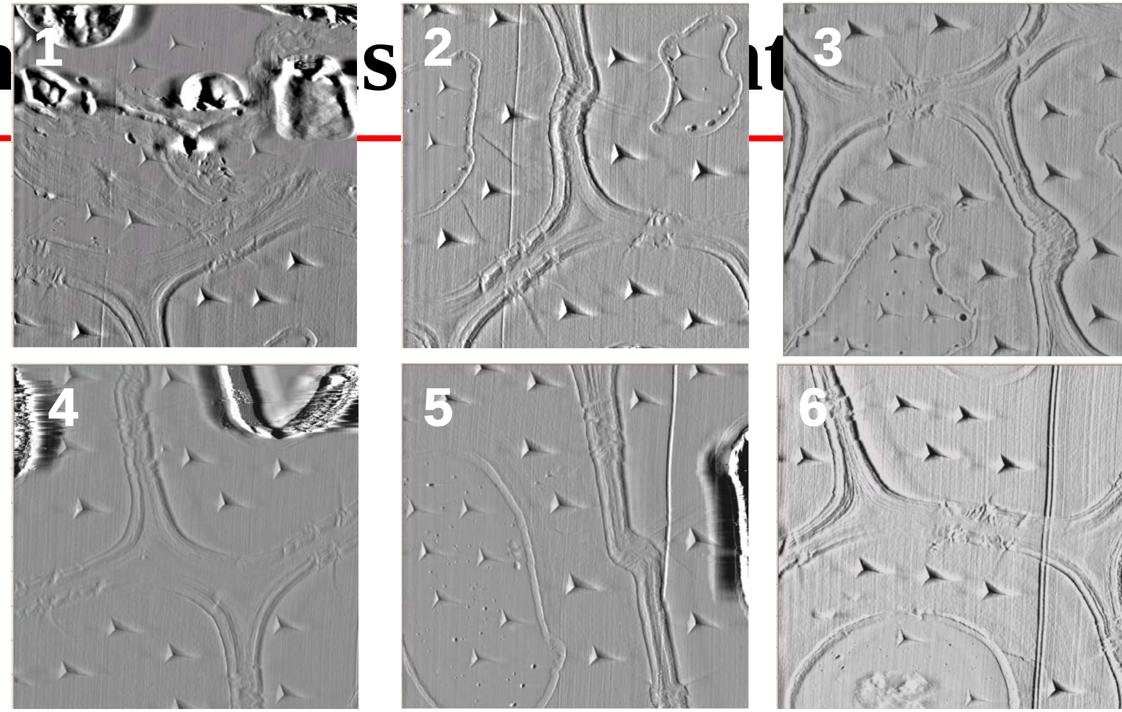
3. Results and discussion (cont)

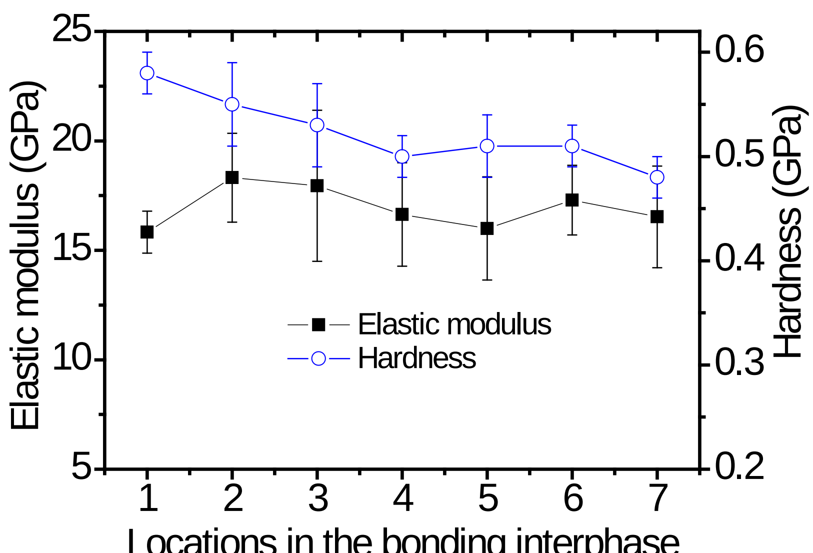


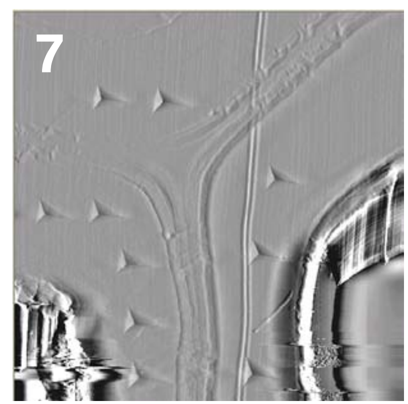
The elastic modulus and hardness of the four wood adhesives

PF bonding



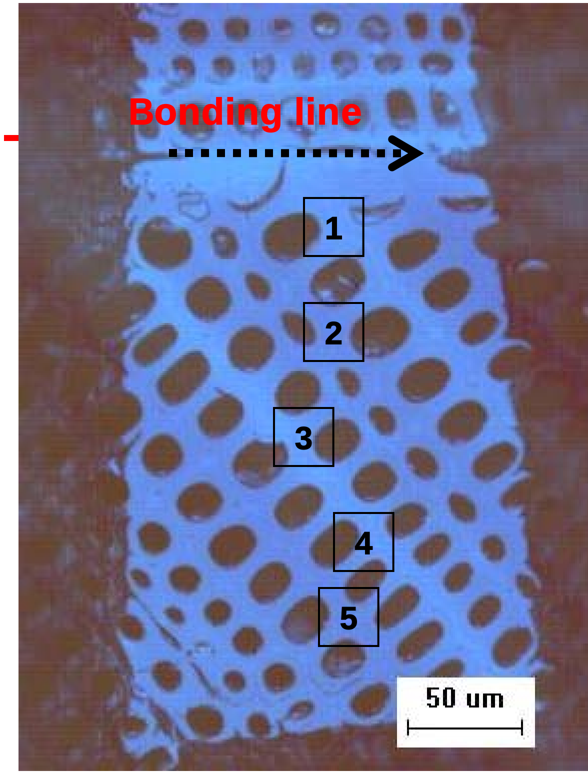


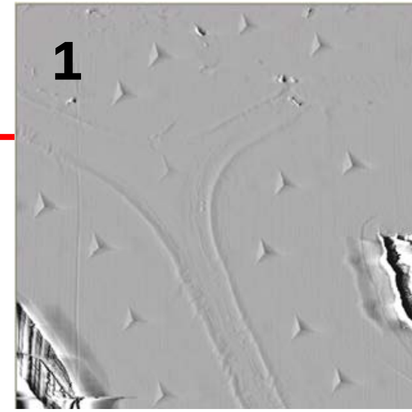


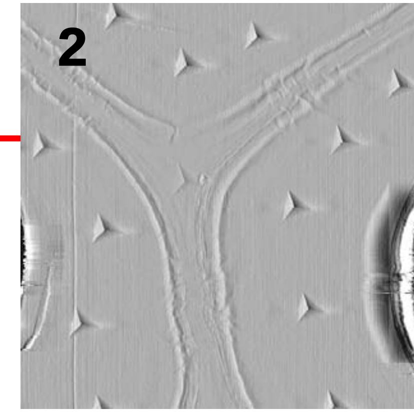


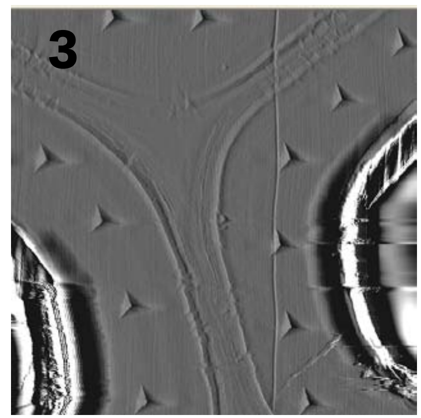
The effect of distance to PF adhesive line on the elastic modulus and hardness variation of wood cell wall

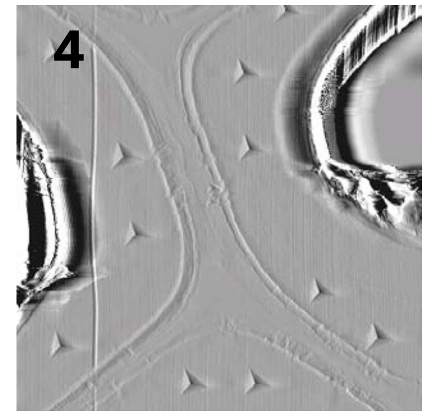
UF bonding

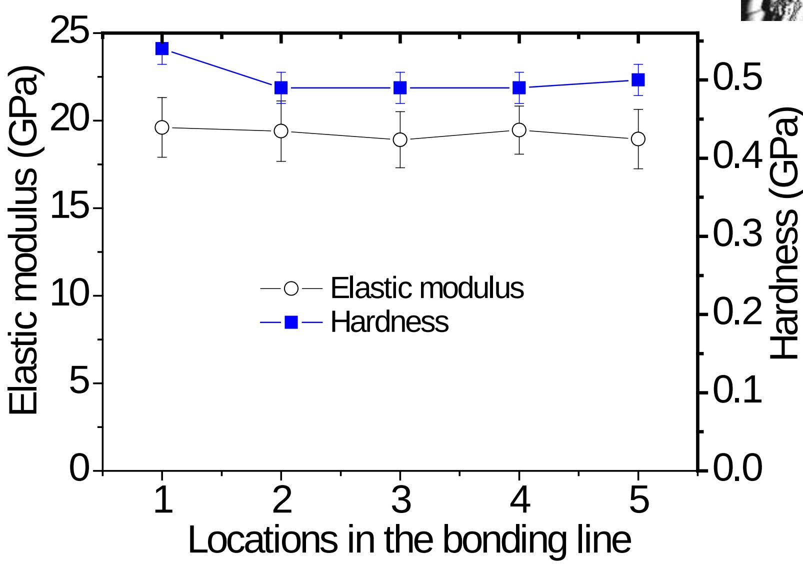

Bonding line
1
2
3
4
5
50 um

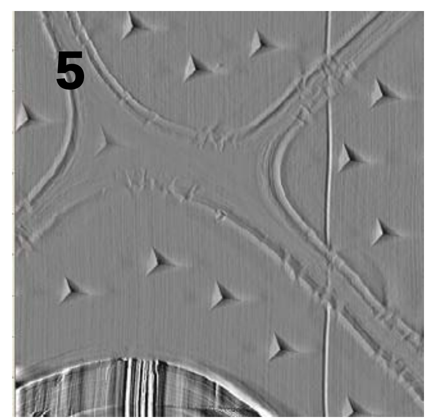

1


2

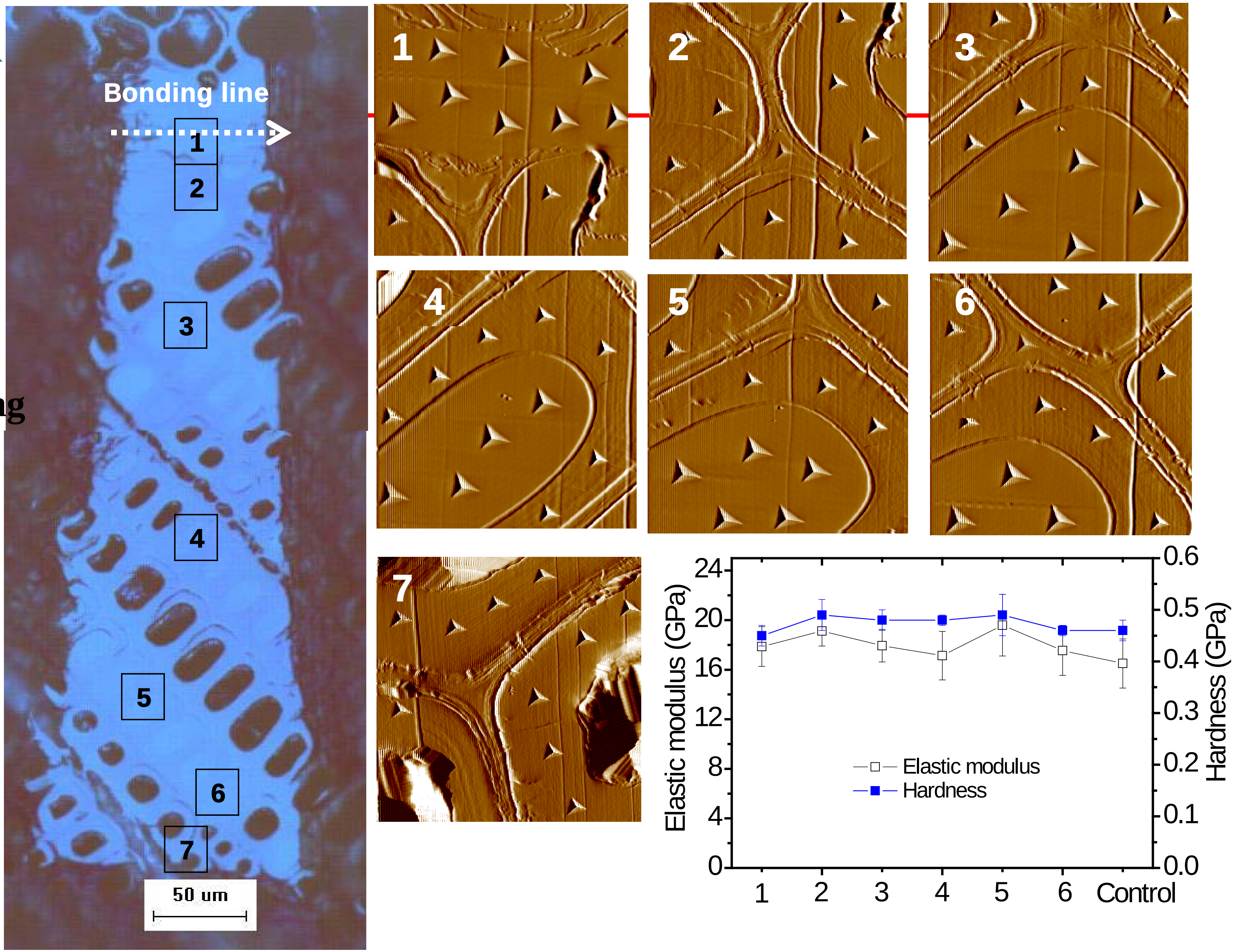

3

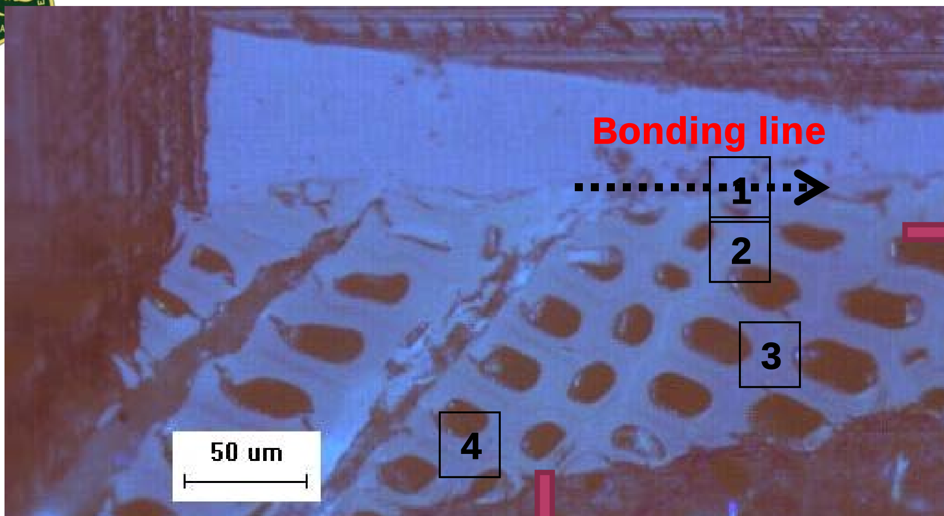

4

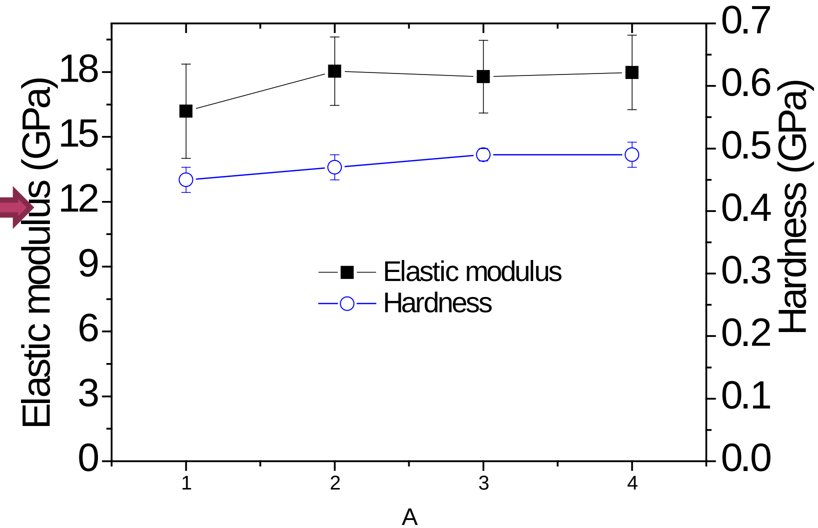

Elastic modulus (GPa)
Hardness (GPa)
Locations in the bonding line
Elastic modulus
Hardness
0
5
10
15
20
25
0.0
0.1
0.2
0.3
0.4
0.5
1
2
3
4
5

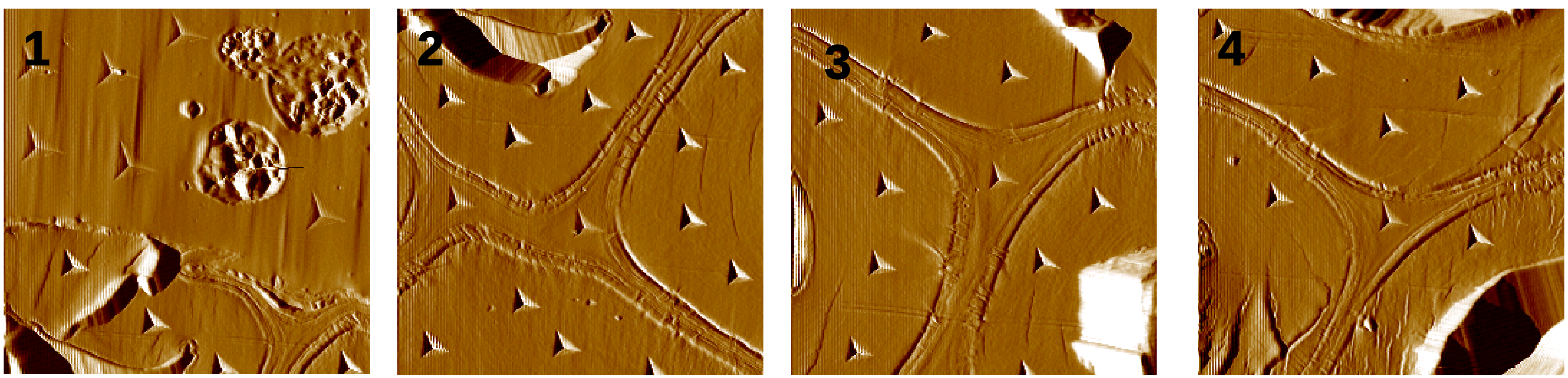

5

Epoxy bonding




Bonding line
1
2
3
4
50 um


Elastic modulus (GPa)
Hardness (GPa)
0
3
6
9
12
15
18
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
1
2
3
4
A
Elastic modulus
Hardness


1
2
3
4

EPI bonding

4. Conclusion

- **Of the wood adhesives tested, PF has the highest elastic modulus and hardness, followed by UF, epoxy and EPI in turn.**
- **Wood cell walls near UF and PF bondlines had increased hardness, but the elastic modulus was not modified.**
- **Mechanical properties of wood cell walls near epoxy and EPI bondlines were not modified.**
- **The durability and strength of wood-PF bondlines is likely because of the high mechanical properties of the PF itself and its ability to infiltrate and strengthen wood cell walls near the bondline.**

This is ICBR, welcome to visit

Any question?