UNIVERSITÉ DE NANTES

Institut de Recherche en Génie Civil et Mécanique

ANR-10-IED-0006-08

Water diffusion characterization in the bonded joint interphase by fiber optic sensor based on Fresnel reflection

EUROMECH – 29 August 2019

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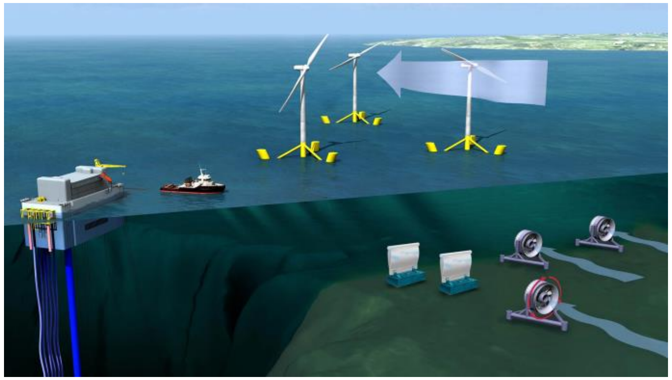
Context

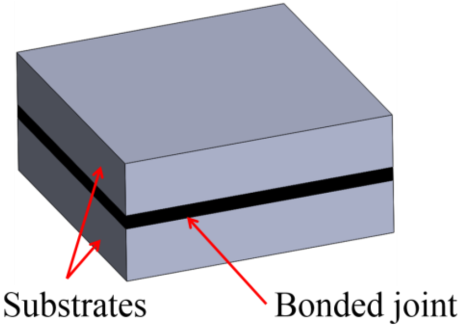
➢ Marine renewable energies

Kinetic energy (winds and currents)
Potential energy (tidal amplitude)
Mechanical energy (waves)
Thermal potential (temperature gradients)
Osmotic pressure (salinity gradients)



➢ Used materials

Metallic materials
Composite materials
Polymer materials (bonded joint)



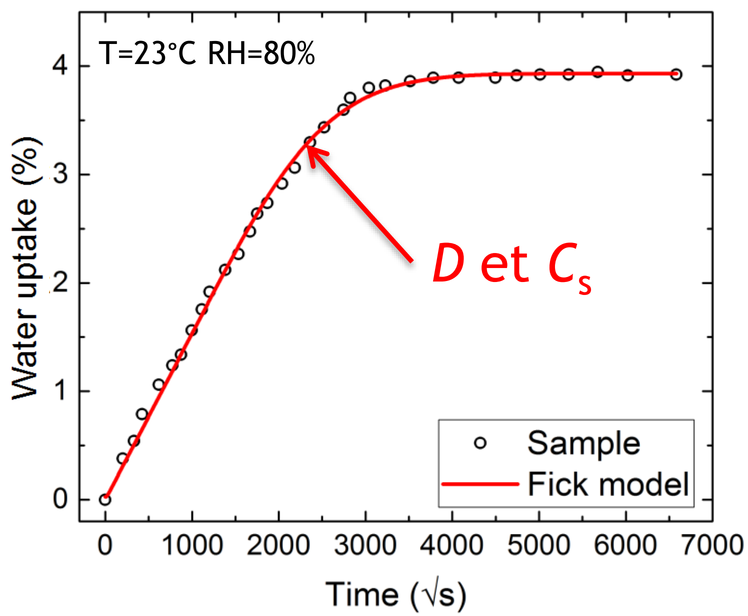
➔ These structures are subject to a humid environment: need to study durability

Context

- Polymer are hydrophilic

Water absorption can be characterized by gravimetric tests (mass monitoring)

Polyamide (Obeid, 2006)

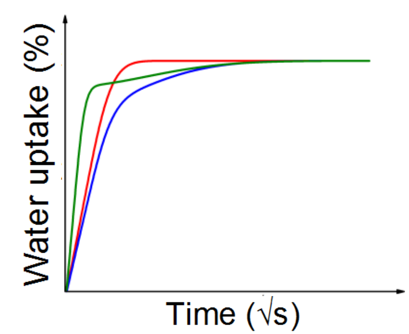


- Mathematical models

Fick model
D, C_s

Dual Fick model
D_1, C_{s1}, D_2, C_{s2}

Langmuir model
D, C_s, a, β



➔ Water diffusion parameters on a bulk polymer

Context

- Water diffusion front in a bulk adhesive

<u>Example for Fick model</u>:

D : diffusion coefficient ($mm^2.s^{-1}$)
C_s : Saturation water content (%)

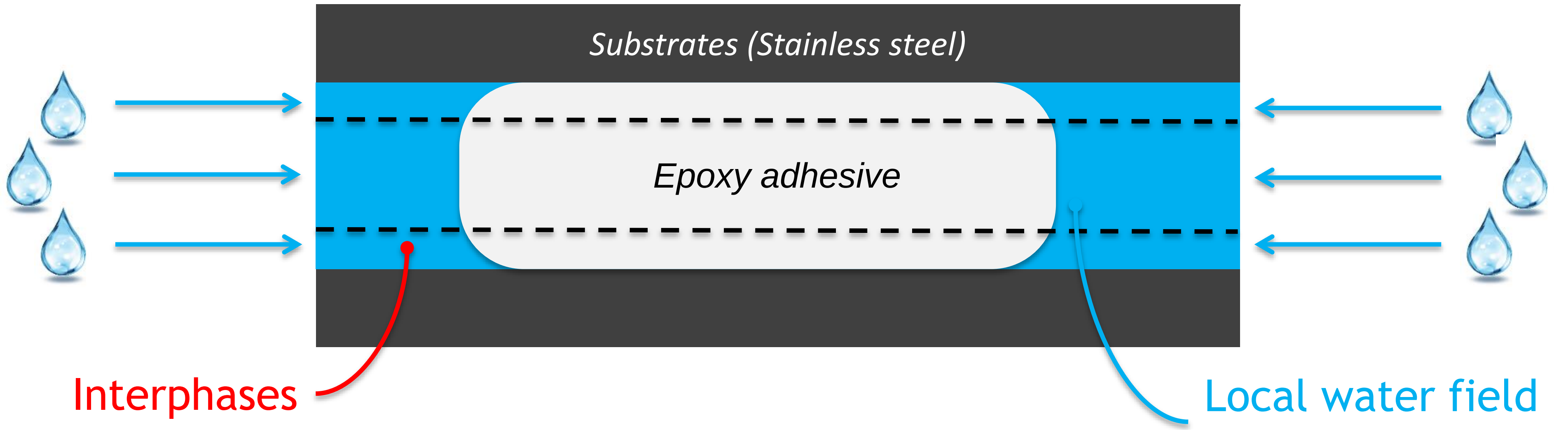
Calculation of the local water content

➔ Constant water diffusion front

- What happens when the adhesive is in a confined state ? (in bonded assembly)

Context

- What happens when the polymer is in a confined state ? (in bonded assembly)



➔ Non constant water diffusion front (Bruneaux, 2004)

- Presence of interphases *(Scientific lock)*

« Interface » ≠ « Interphase »

Context

- Presence of interphases *(Scientific lock)*: Two hypotheses

Interphases

Local water field (Bruneaux, 2004)

➔ Non constant water diffusion front

Capillary effect

Presence of a preferential way that accelerates water diffusion
(Zanni-Deffarges, 1995, Vine,2001)

(<u>hypotheses</u>: adhesive/substrate interactions, microcavities, cracks, surface energy…)

Under-crosslinking

(Hong, 1992, De Parscau, 2016)

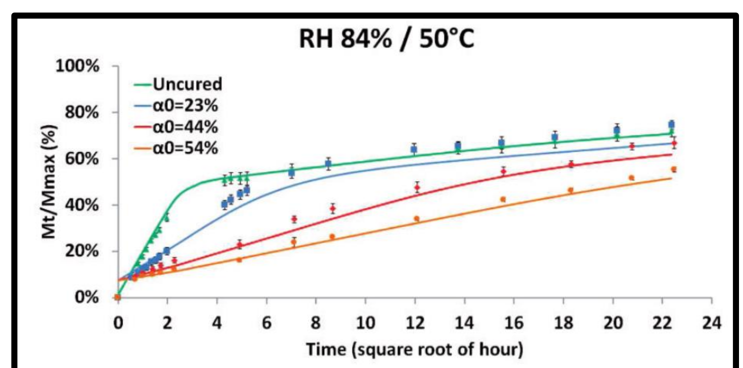
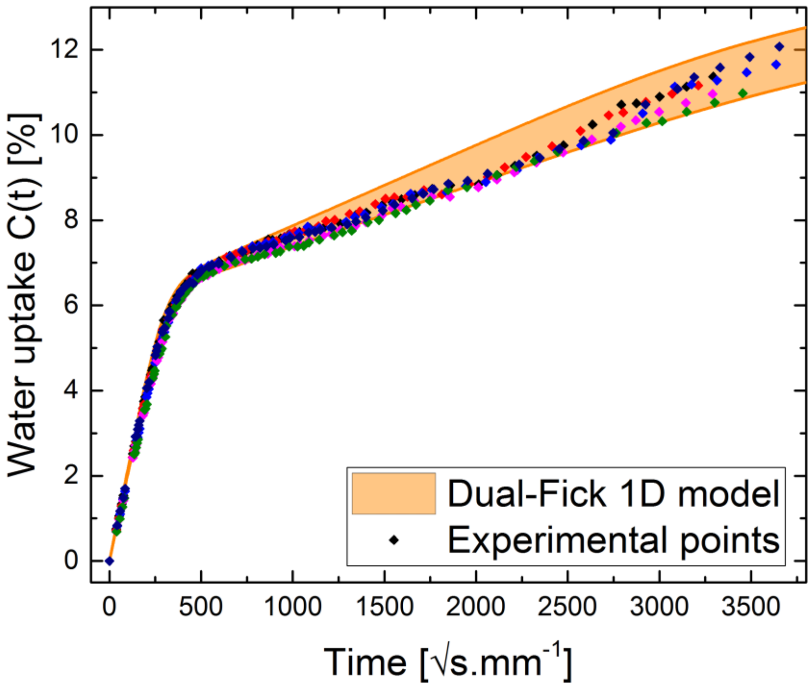


Table of content

1. Diffusion kinetics in a bulk polymer

- Gravimetric tests: immersion in water at 40 °C on discs (70 mm in diameter and 2 mm thickness)



Dual Fick 1D modeling, parameters identification:

D_1 [$mm^2.s^{-1}$]	$(1.74 \pm 0.04).10^{-6}$
C_{s1} [%]	5.95 ± 0.03
D_2 [$mm^2.s^{-1}$]	$(9.01 \pm 1.10).10^{-9}$
C_{s2} [%]	7.23 ± 0.45

Analytical solution of the Dual Fick 1D model: (Placette et al., 2011) (Crank, 1975)

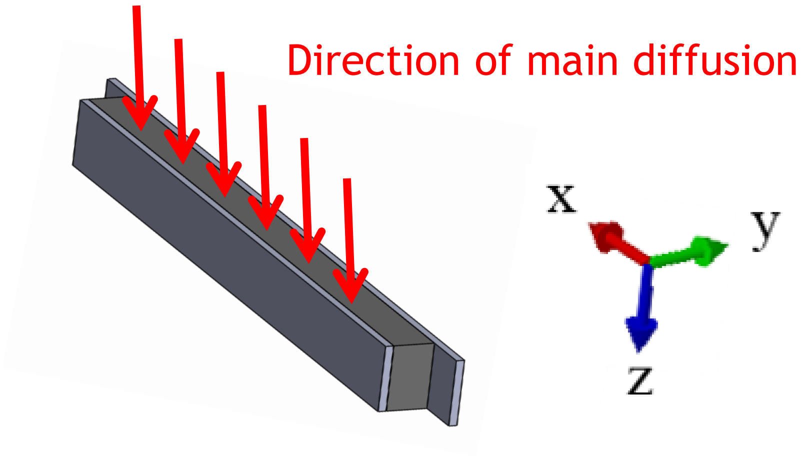
$$C(t) = \sum_{i=1}^{2}\left[c_{s_i}\left(1 - \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2} exp\left[-D_i(2n+1)^2\pi^2\frac{t}{e^2}\right]\right)\right]$$

2. Diffusion kinetics in a bonded joint

➢ Gravimetric tests on a bonded joint

<u>Geometry</u>: Parallelepiped samples (width 10 mm ; length 70mm ; thickness 1/2/5mm)

➔ Geometry chosen in order to be able to measure a significant variation in the mass of the bonded joint during immersion

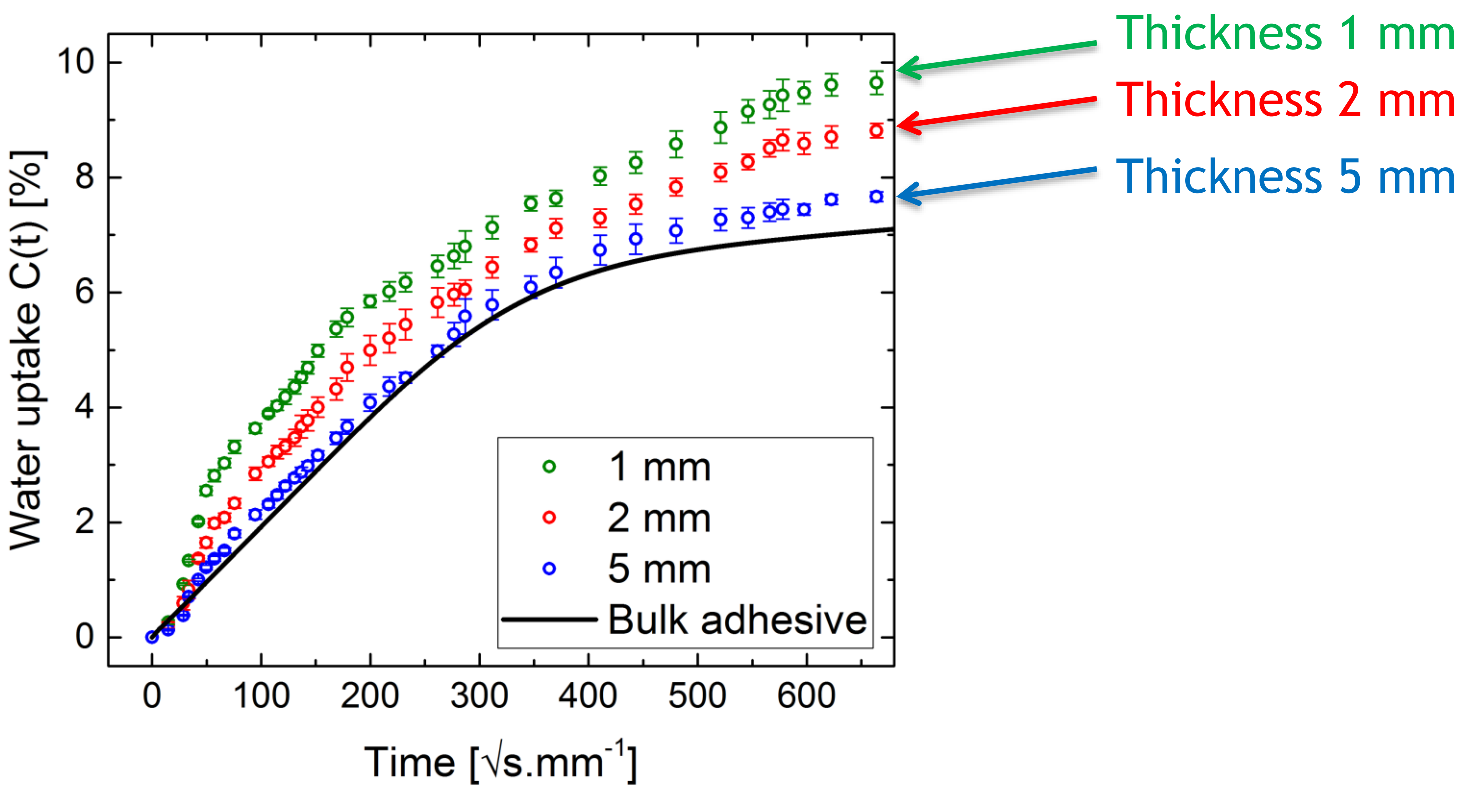


➢ What is the behavior of the adhesive in the case of a bonded assembly?

2. Diffusion kinetics in a bonded joint

➢ Gravimetric tests on a bonded joint

Results:



✓ The presence of interphases accelerates water diffusion

✓ Different interphase ratios depending on the total thickness of the bonded joint

3. Characterization of interphases

a) Approach with gradient model

Instrumentation of a bonded assembly with Fresnel sensor

↓

Optical characterization of the interphase during water diffusion

↓

Local water characterization of the interphase

↓

Water diffusion parameters of interphase

↓

Calculation of the macroscopic water content of bonded joint

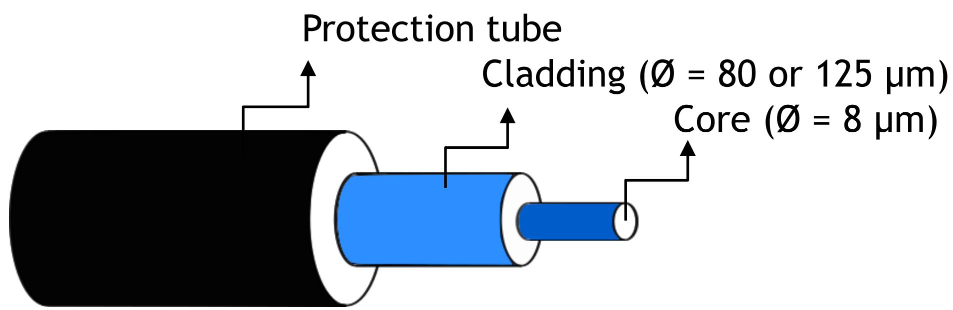
3. Characterization of interphases

b) Instrumentation of a bonded assembly with Fresnel sensor

The Fresnel sensor (Cusano, 2000)

It is a fiber optic sensor that allows the local measurement of the refractive index of a material

Optical fiber:

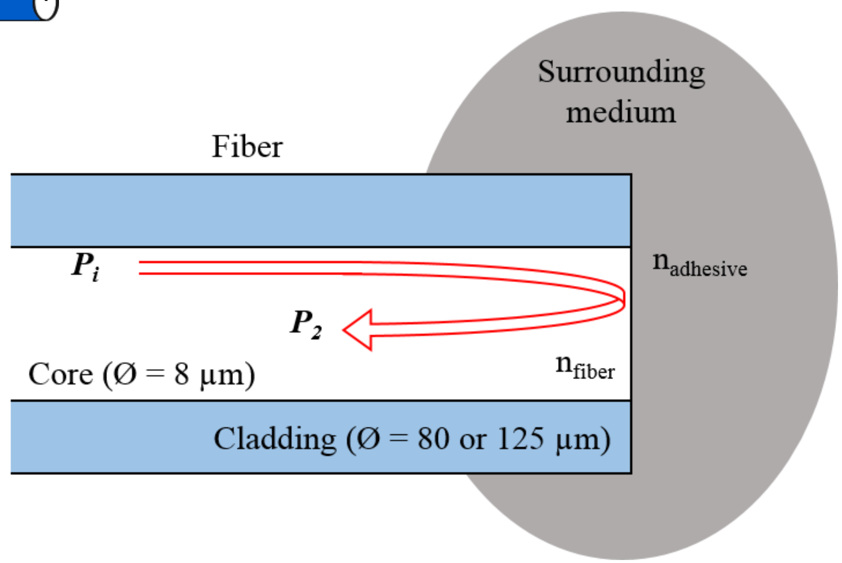


Fresnel sensor: Cleavage of an optical fiber

Measurement of the reflected power

↓

Calculation of the refractive index of the adhesive



3. Characterization of interphases

b) Instrumentation of a bonded assembly with Fresnel sensor

Refractive index

It's a dimensionless number that describes how light propagates through that environment

→ n = f (materials, wavelength, temperature, pressure, crosslinking rate, local water content)

(Cusano, 2000, Aduriz, 2007, Grangeat, 2019)

In this study during immersion: (Cusano, 2000, Aduriz, 2007, Grangeat, 2019)

→ n = f (~~materials~~, ~~wavelength~~, ~~temperature~~, ~~pressure~~, ~~crosslinking rate~~, local water content)

Unchanging   Unchanging 1550 nm   Negligible

Unchanging   Unchanging

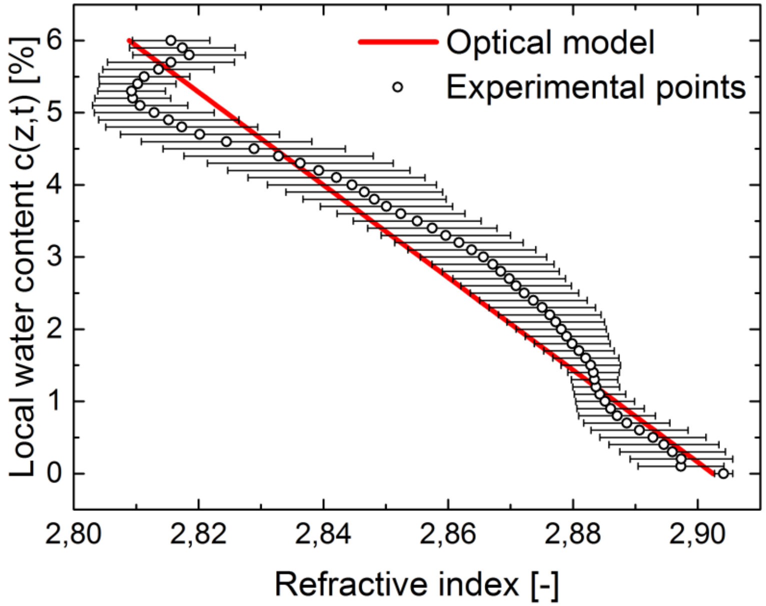
3. Characterization of interphases

b) Instrumentation of a bonded assembly with Fresnel sensor

Relationship between the refractive index and local water content

In the previous study (article in progress):

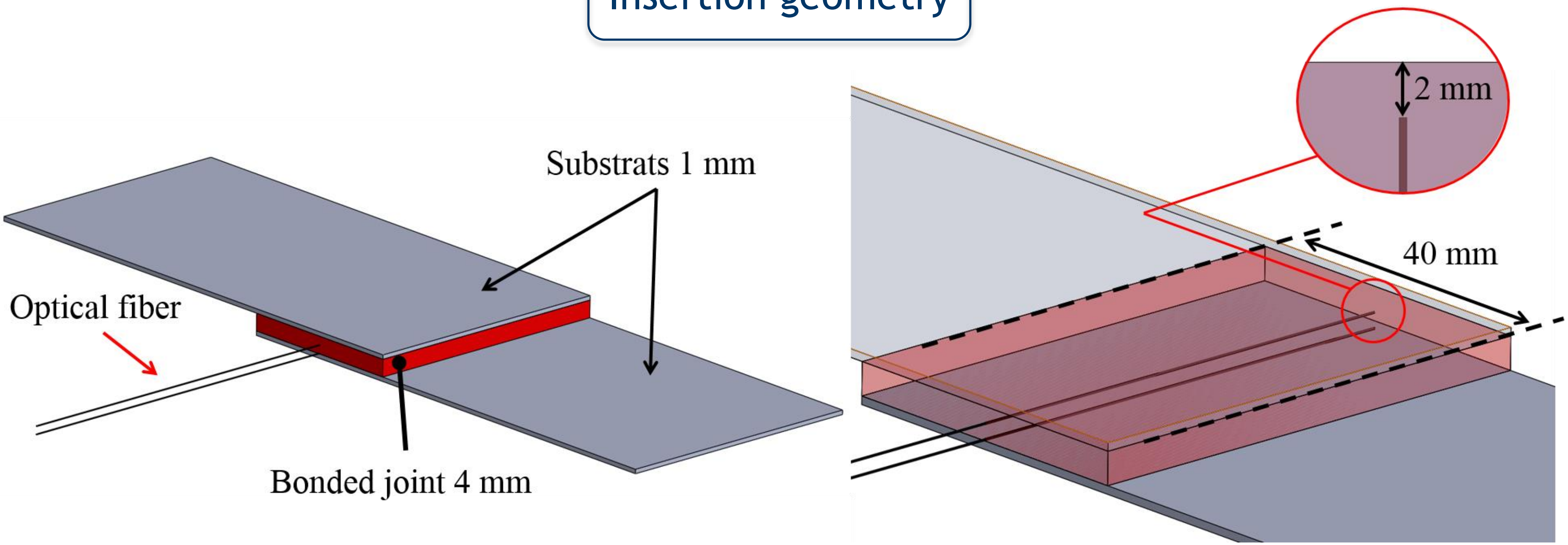
➔ Implementation of an optical model (Maxwell-Garnet model (Markel, 2016)) to link the refractive index of the adhesive to the local water content



b) Instrumentation of a bonded assembly with Fresnel sensor

Objective: Measure local water content in the interphase of a bonded joint

Insertion geometry

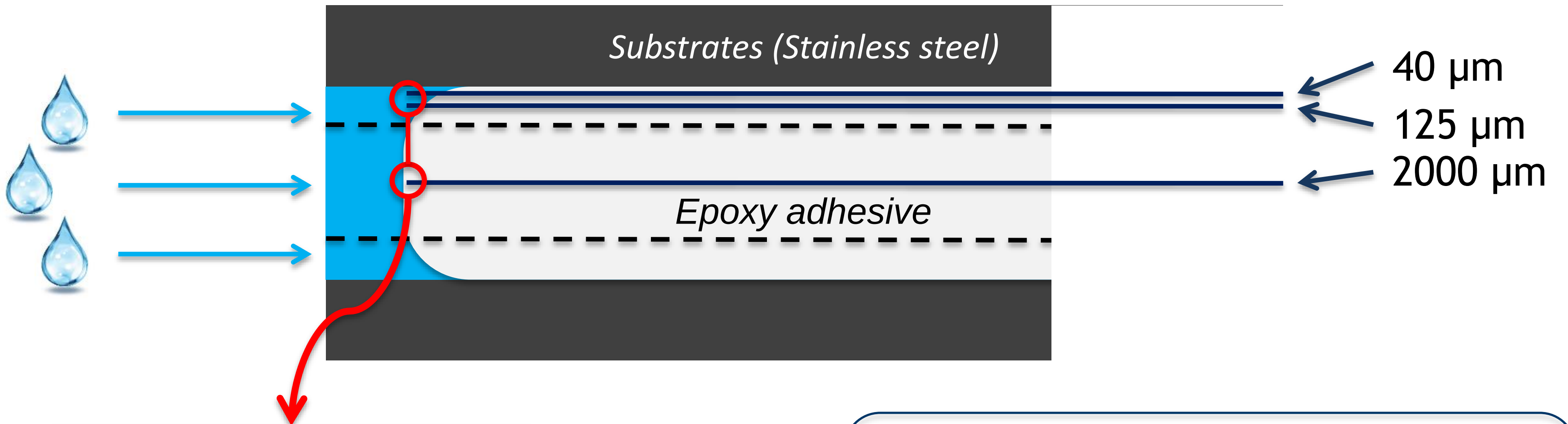


3. Characterization of interphases

b) Instrumentation of a bonded assembly with Fresnel sensor

Objective: Measure local water content in the interphase of a bonded joint

Insertion geometry



Measurement of local water content with Fresnel sensor

Water diffusion parameters:

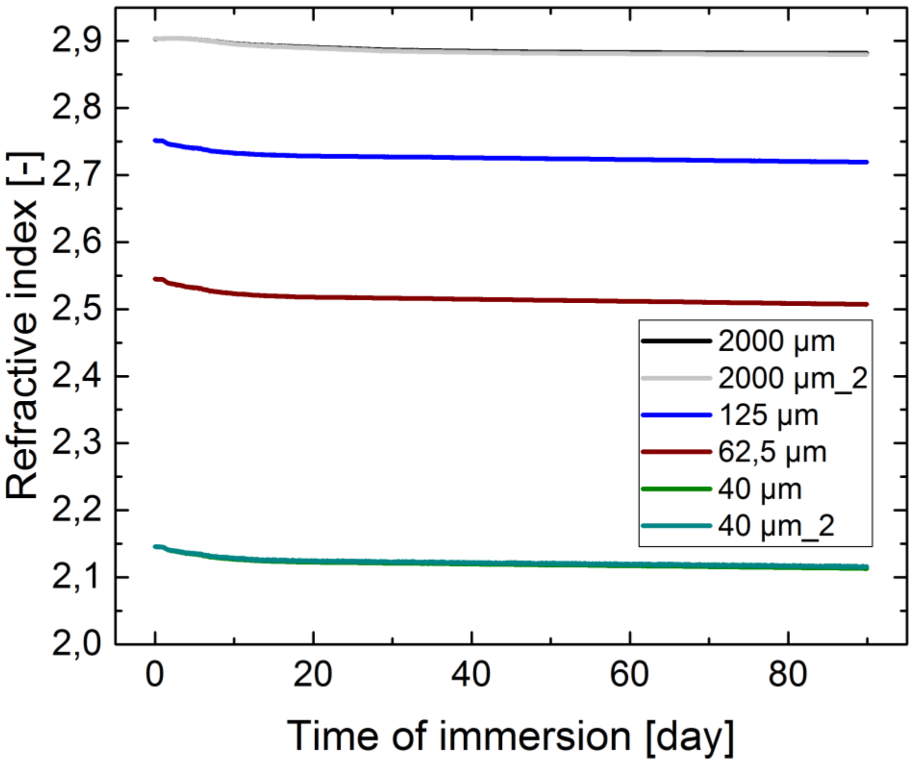
D : diffusion coefficient($mm^2.s^{-1}$)
C_{sat} : Saturation water content (%)

➔ Water diffusion parameters depending on the distance from the substrate?

3. Characterization of interphases

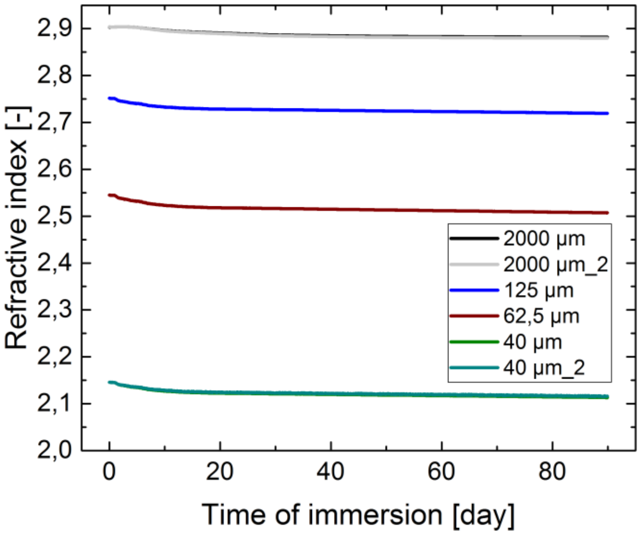
c) Optical characterization of the interphase during water diffusion

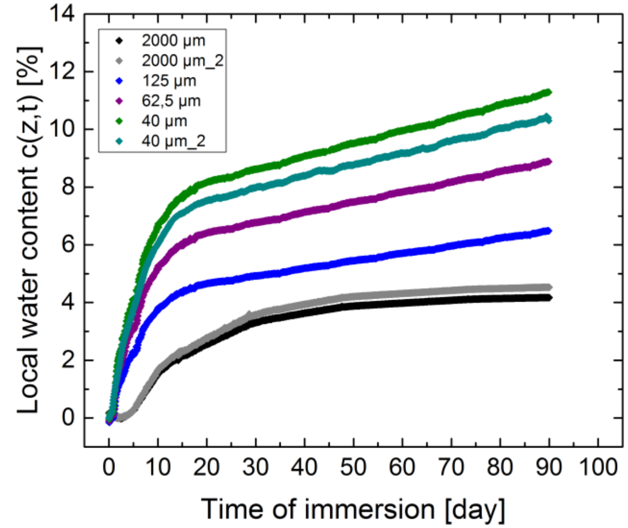
Results:



- ✓ Different refractive index at the beginning of the immersion due to under-crosslinking of the interphase (Grangeat et al., 2019)
- ✓ Decrease in refractive index during water diffusion

d) Local water characterization in the interphase





Adhesive refractive index before immersion

<u>Maxwell-Garnett model:</u>

$$c(z,t) = \left(\frac{V_a \cdot \rho_w}{m_{t0}}\right)\left(\frac{n_{a/w}^2(t) - n_a^2}{n_w^2 - n_a^2}\right) \cdot \left(\frac{n_w^2 + 2n_a^2}{n_{a/w}^2(t) + 2n_a^2}\right)$$

Local water content

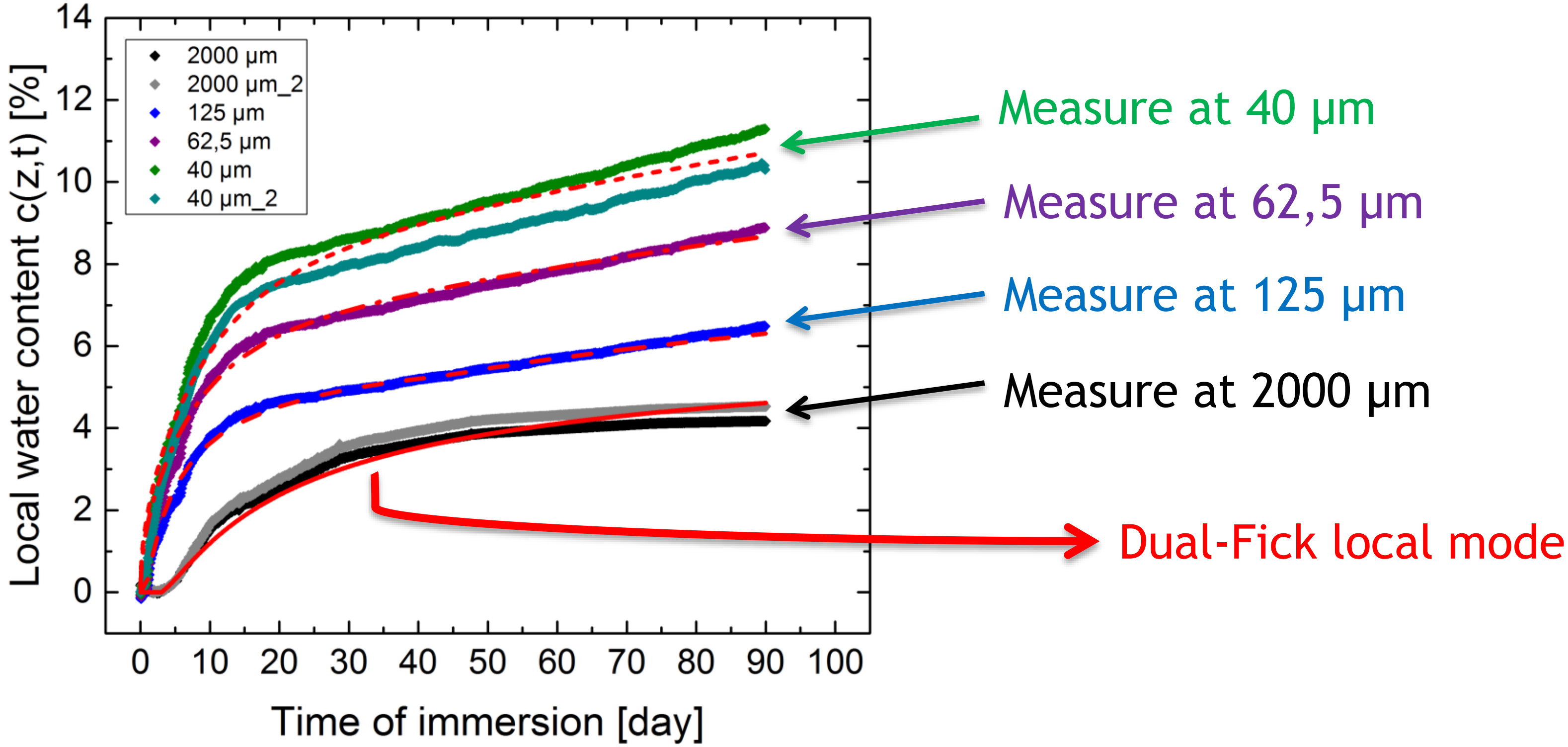
Water volume fraction

Water refractive index

Adhesive refractive index during immersion

d) Local water characterization in the interphase

<u>Results</u>:

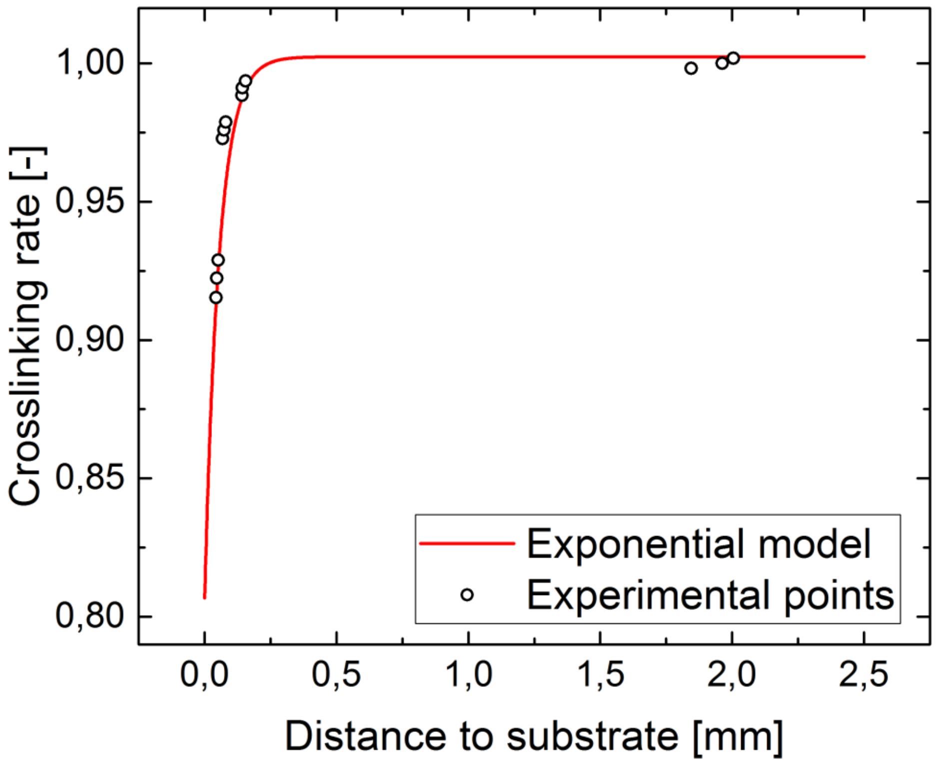


Parameters	Bulk	*d* = 2000 µm	*d* = 125 µm	*d* = 62,5 µm	*1.22* = 40 µm
D_1 (mm²/s)	(1,74 ± 0,04).10^{-6}	1,74.10^{-6}	5,68.10^{-6}	9,01.10^{-6}	(3,20 ± 1,22).10^{-5}
D_2 (mm²/s)	(9,01 ± 1,10).10^{-9}	9,01.10^{-9}	9,27.10^{-8}	1,15.10^{-8}	(4,05 ± 0,30).10^{-7}
C_{s1} (mm²/s)	5,95 ± 0,03	5,95	6,95	7,80	9,56 ± 0,73
C_{s2} (mm²/s)	7,23 ± 0,45	7,23	5,61	7,31	7,07 ± 0,48

3. Characterization of interphases

e) Water diffusion parameters of interphase

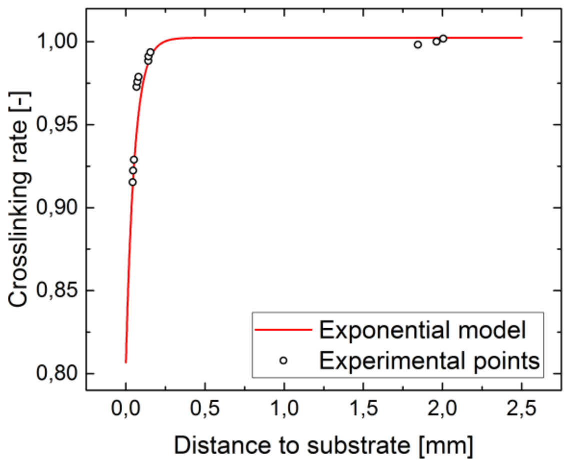
<u>Results</u>: Crosslinking gradient (Grangeat et al., 2019),

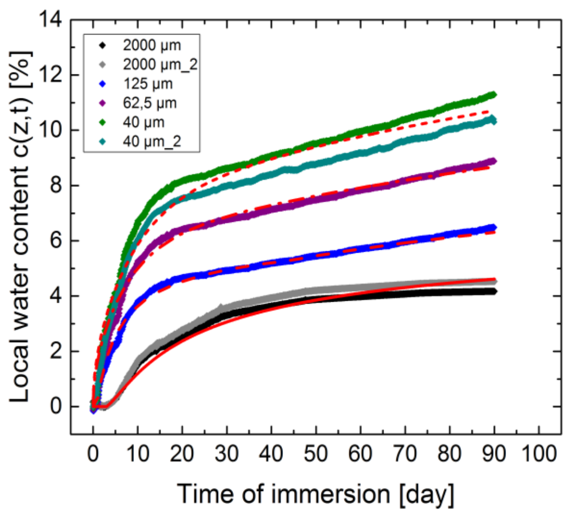


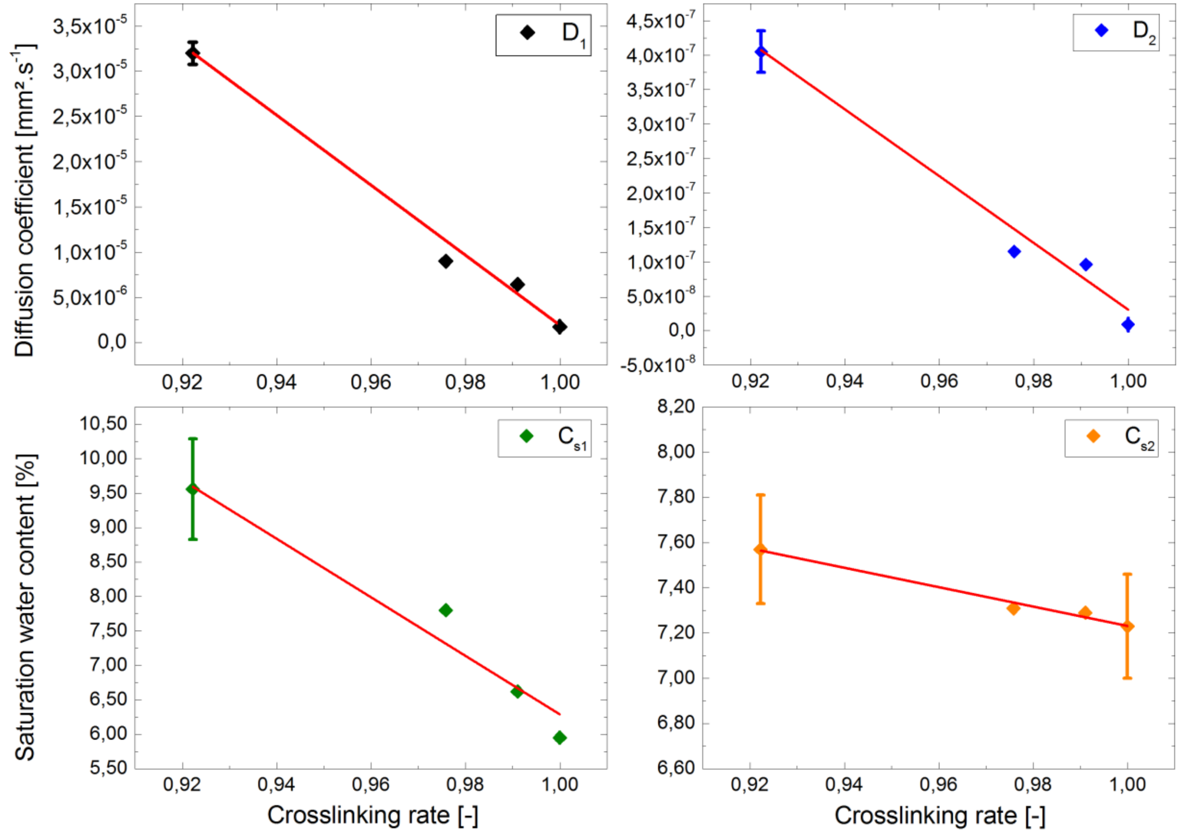
- The crosslinking rate was determined by measuring the refractive index using the Fresnel sensor in a bonded joint

3. Characterization of interphases

e) Water diffusion parameters of interphase



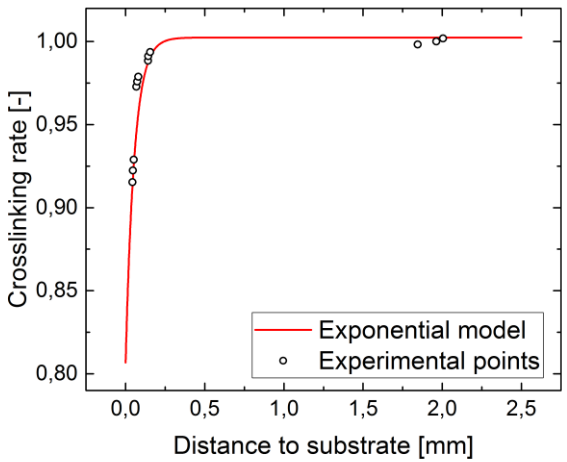


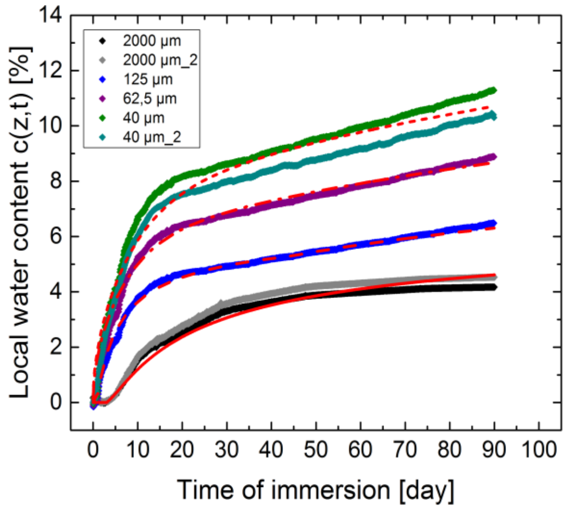


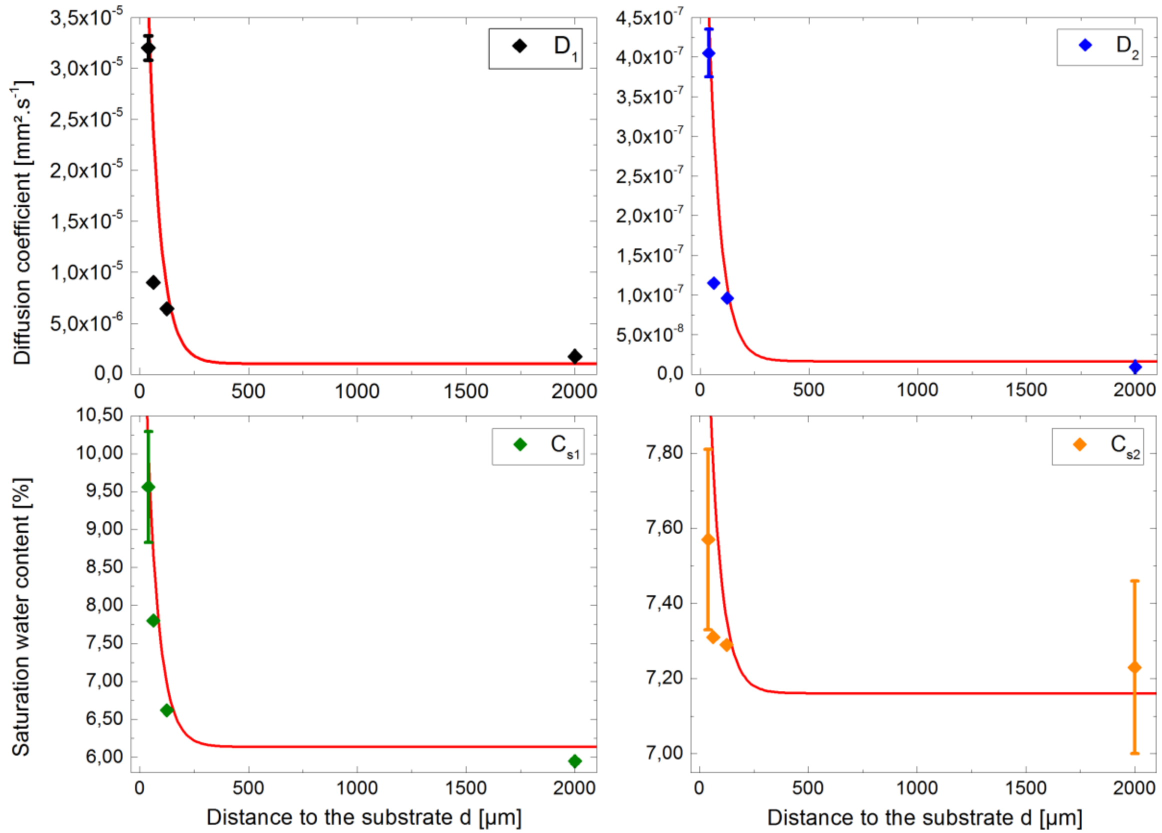
✓ Linear relation between diffusion parameters and crosslinking rate (De Parscau du Plessix et al., 2016)

3. Characterization of interphases

e) Water diffusion parameters of interphase





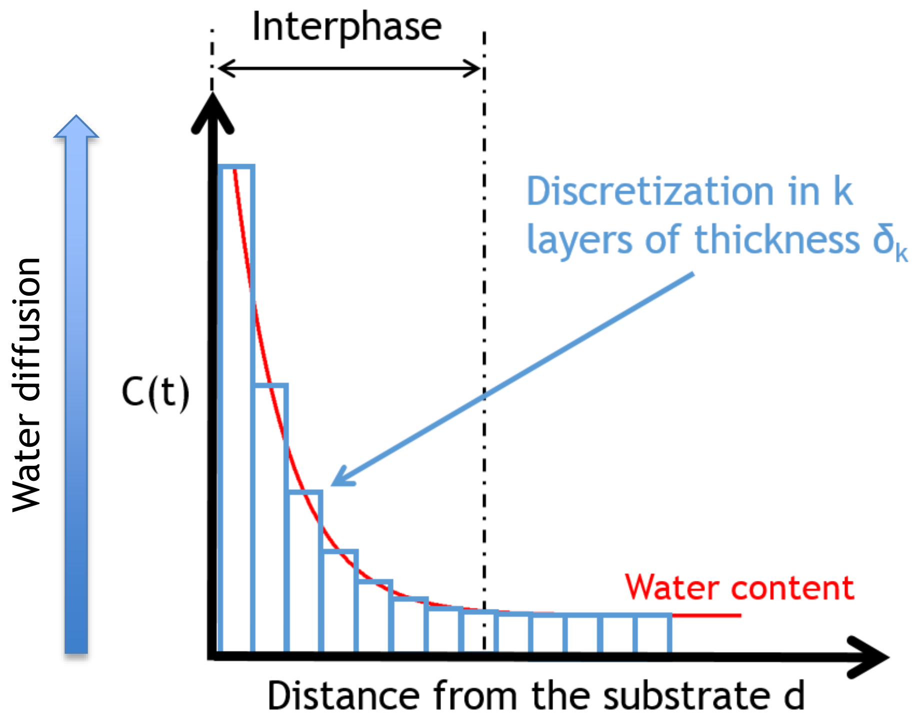


- ✓ Characterization of the diffusive properties of the interphase

3. Characterization of interphases

f) Macroscopic water uptake of bonded joint

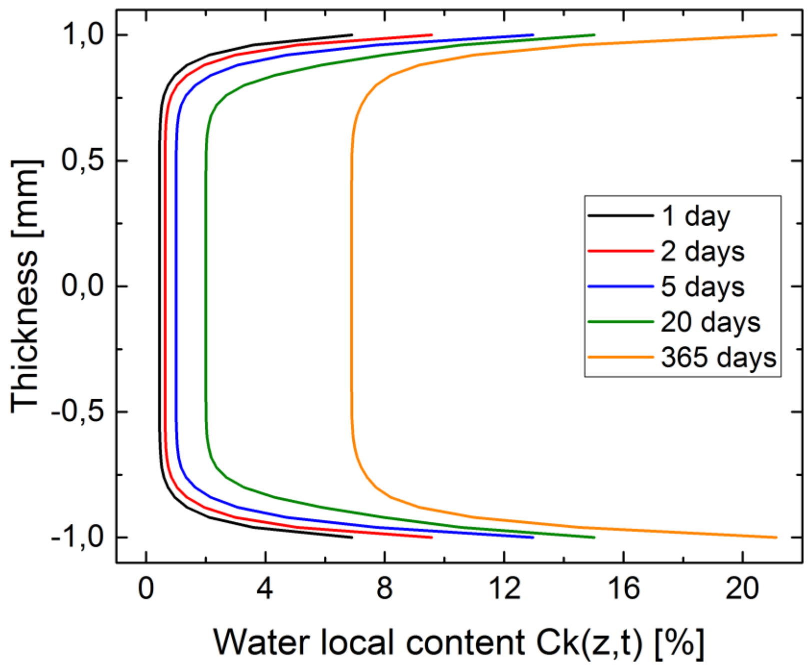
- Discretization in k layers of thickness δ_k
- For each layer : D_1, D_2, C_{s1}, C_{s2} (average of the properties on the layer k)
- Calculation of the water content $C_k(t)$ (Dual Fick 1D model) for each layer

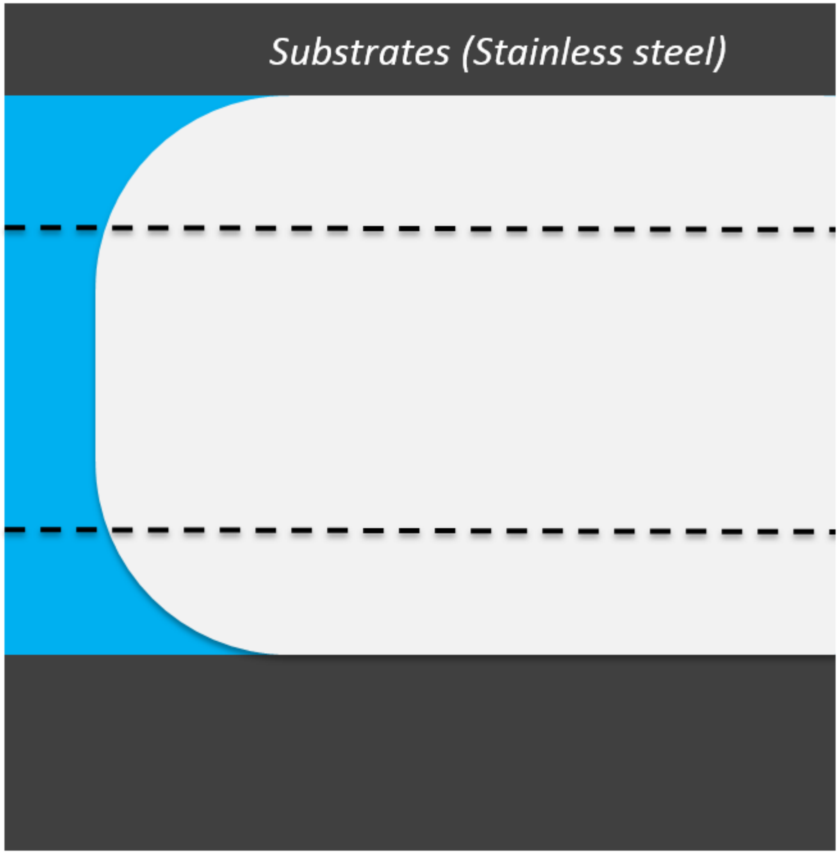


3. Characterization of interphases

f) Macroscopic water uptake of bonded joint

Results: Water diffusion parameters gradient



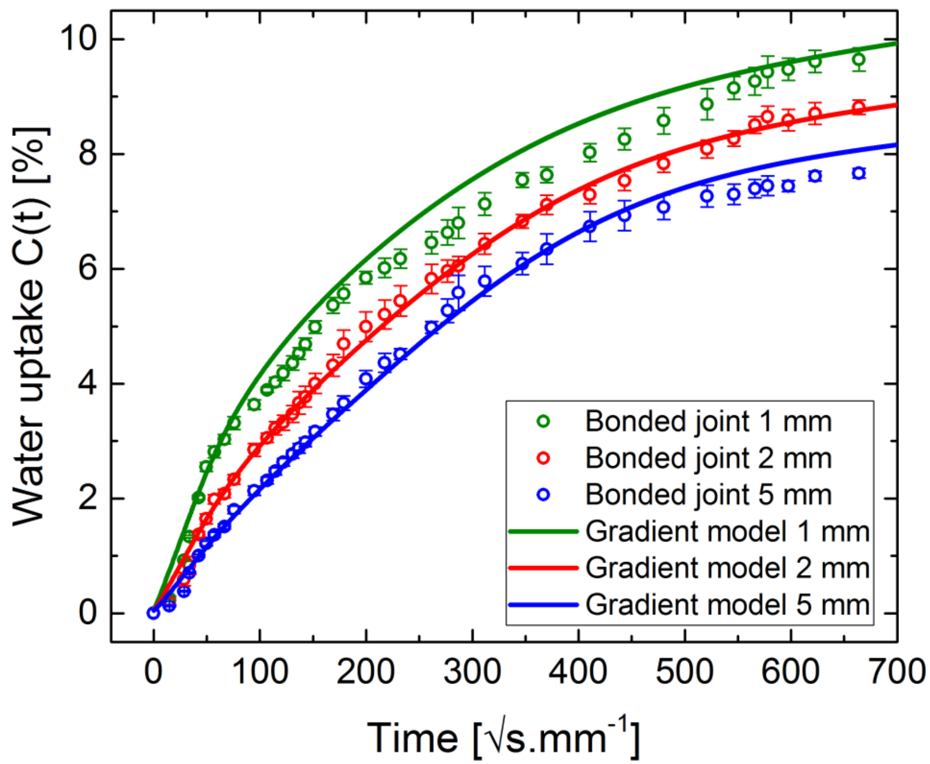


✓ Gradient of water content in the interfacial zone

3. Characterization of interphases

f) Macroscopic water uptake of bonded joint

Results: Comparison with gravimetric tests



✓ Good correlation between the water content simulated using the gradient model and gravimetric tests

4. Conclusion and perspectives

Conclusions

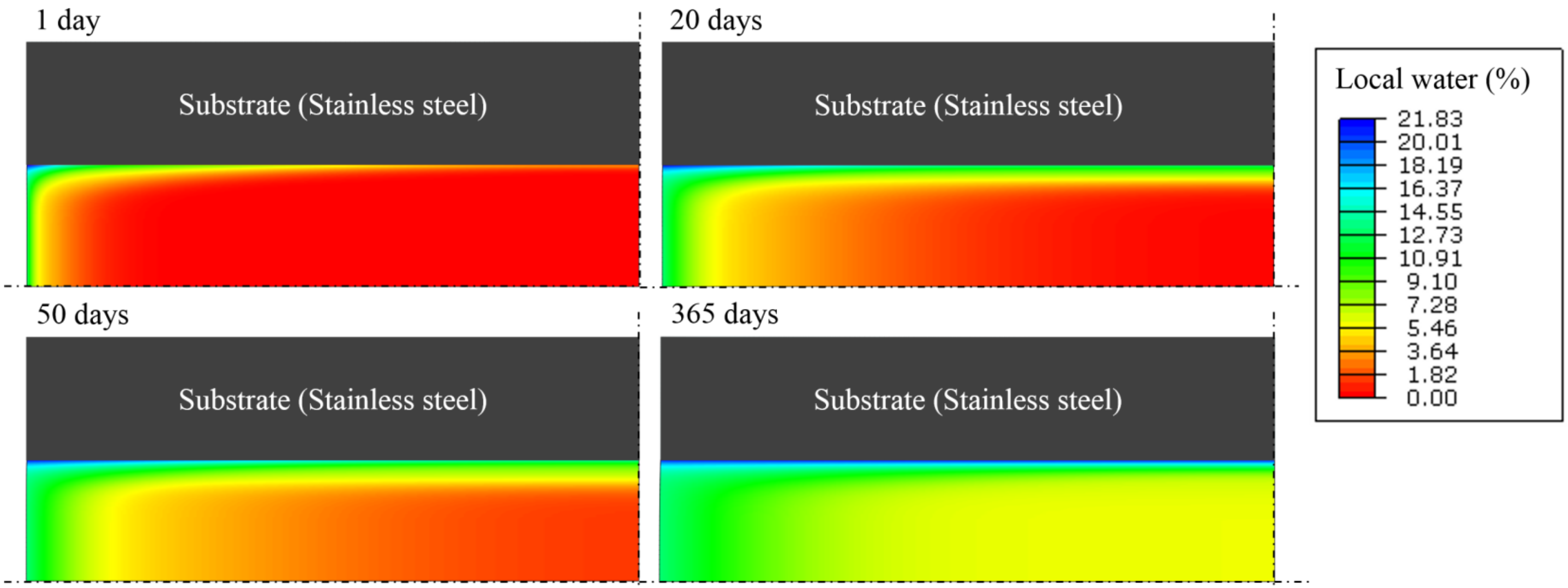
- ✓ Revealing of the interphase that accelerates water diffusion
- ✓ Characterization of the diffusive properties of the interphases (gradient model)
- ✓ Modeling of the water content field in bonded joint (gradient model)

4. Conclusion and perspectives

Perspectives

- Implement a finite element calculation model under Abaqus™: Simulate the mechanical field induced by the hygroscopic swelling of the bonded joint confined by the substrates (in progress)

First results: Simulation of water diffusion in a 2 mm thick bonded assembly (2D modeling of a quarter of a specimen)



Acknowledgement

Thank you for your attention !

This work benefited from France Energies Marines and State financing managed by the National Research Agency under the Investments for the Future program bearing the reference ANR-10-IED-0006-08.

ANR-10-IED-0006-08

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