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Nano-Coating Technology

Mechanical properties of SiOx coated PET films characterized by AFM equipped with micro tensile stage

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BIOPHY RESEARCH

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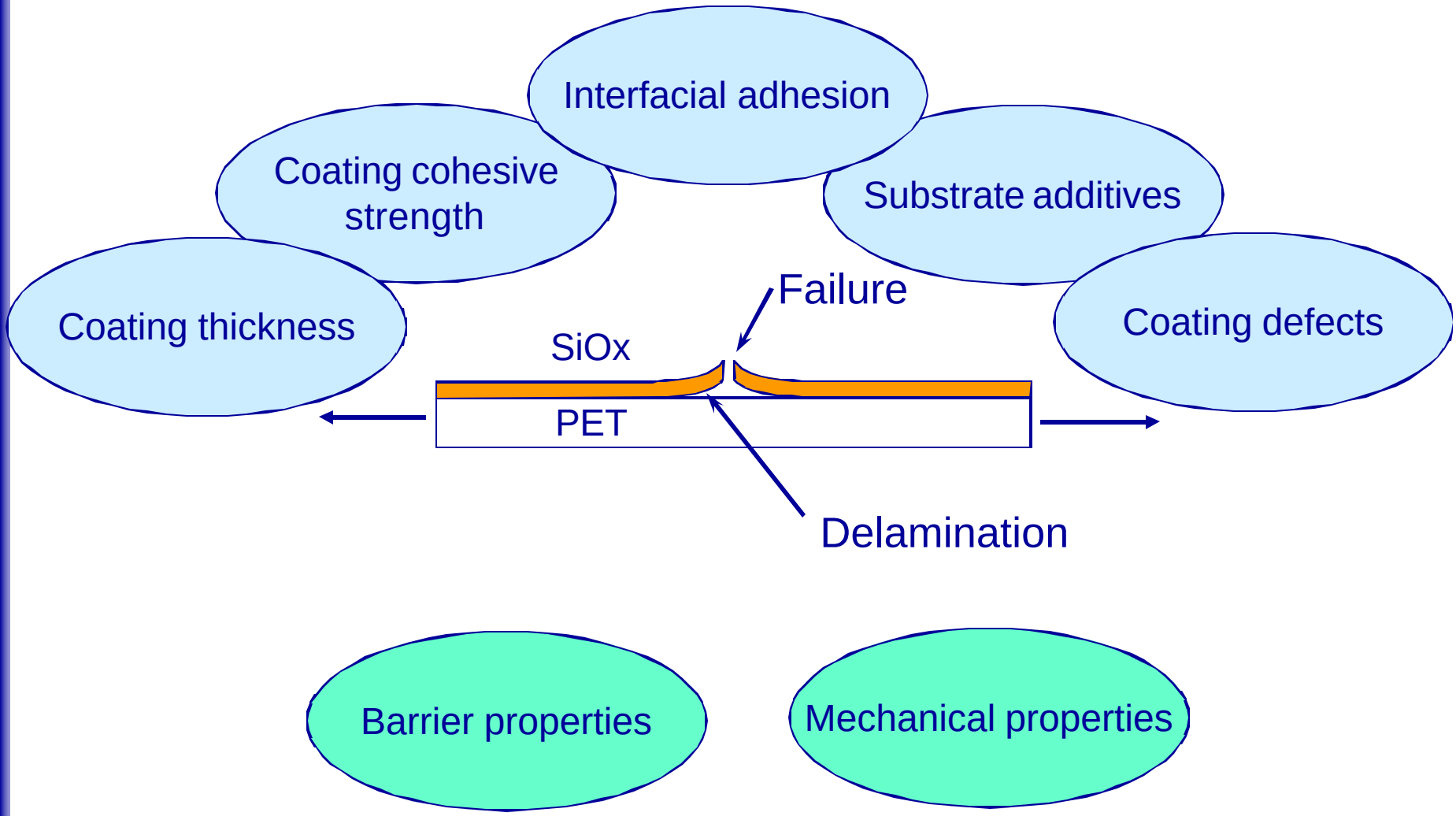
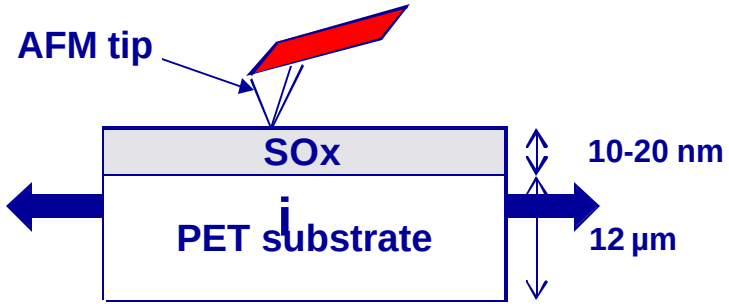
Introduction



Experimental setup

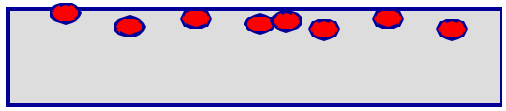
PET / SiOx sample is strained on a micro-tensile stage, and *in-situ* fragmentation process is evaluated by AFM (tapping mode)



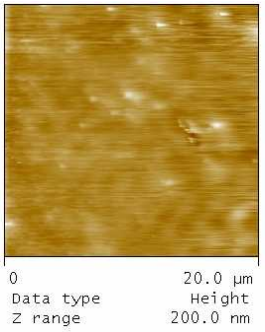
Materials

10 and 20 nm thick SiOx coatings deposited by Plasma Enhanced Chemical Vapor Deposition on 3 different PET substrates (*Mitsubishi Polyester Films*)

RNK, 12 μm

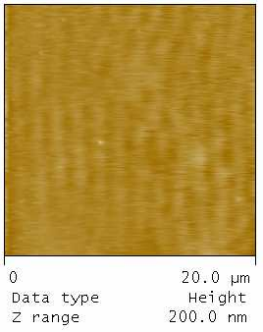


PET with high density of anti-block additives



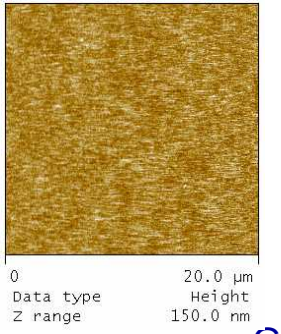
RDO , 12 μm

PET, low density of additives



RHB , 12 μm

PET/PEN, no additive



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Fragmentation process

Main stages of coating fragmentation :

- **1st stage: random cracking**

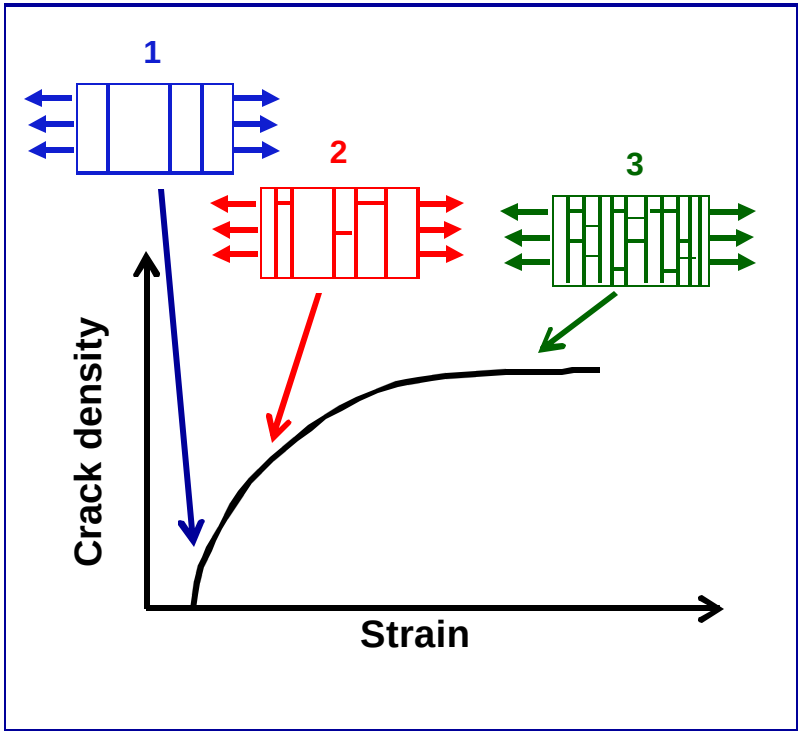
First cracks develop at defect sites on the underlying polymer substrate

- **2nd stage: mid-point cracking**

Decrease in the fragmentation rate and observation of transverse buckling failures

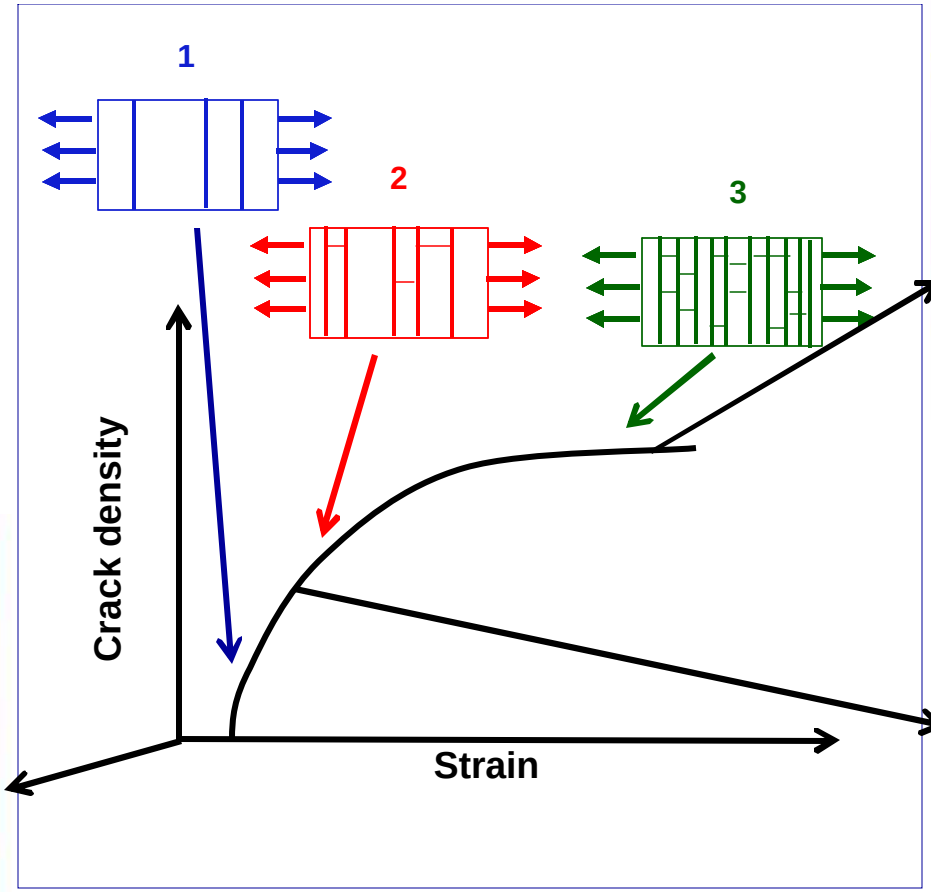
- **3rd stage: saturation stage**

No more cracks are formed as the strain is increased

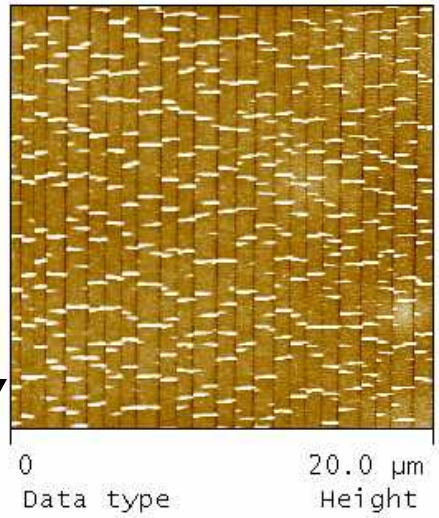


Fragmentation process

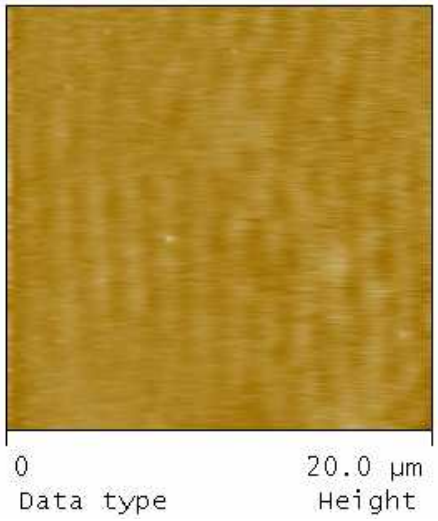
10 nm SiOx / RDO12



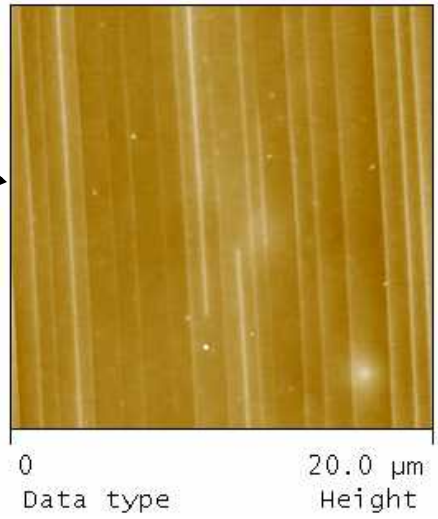
15% strain



0% strain



5% strain



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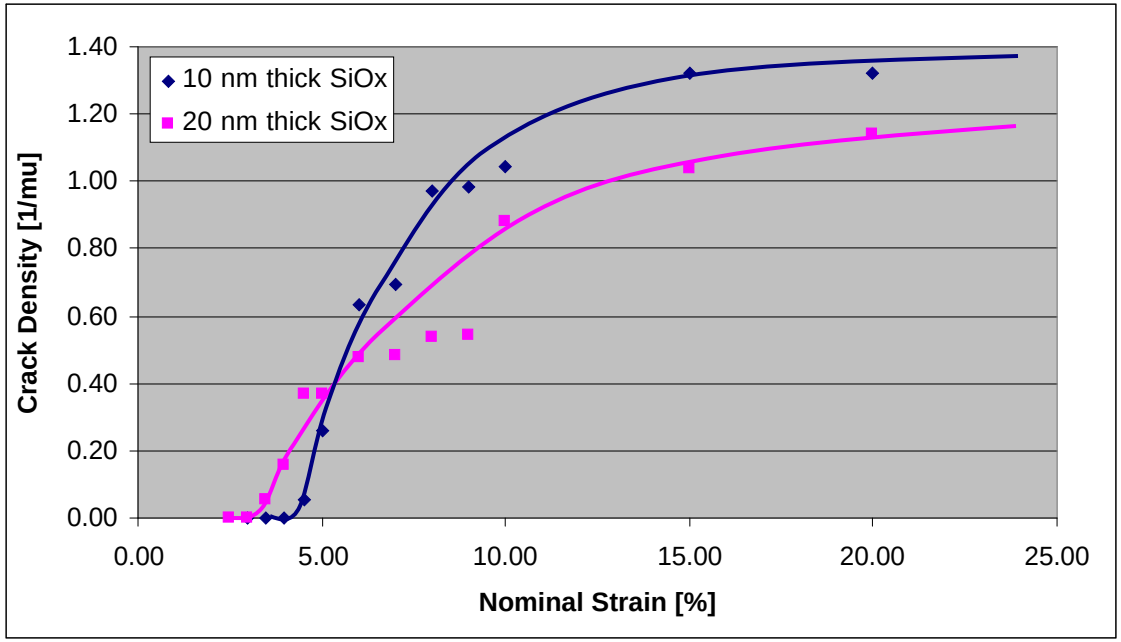
Effect of coating thickness

Thicker coatings crack at lower strain compared to thinner ones, and present larger crack density at saturation



Sample	SiOx thickness [nm]	Crack Onset Strain [%]
SiOx / PET (RDO12)	20	3.5
	10	4.5

What before fragmentation?

In the pre-fragmentation stages, different effects have been observed in previous works(*):

- partial damage of the coating (increased number of defects, micro-cracks);

- slight loss of barrier properties.

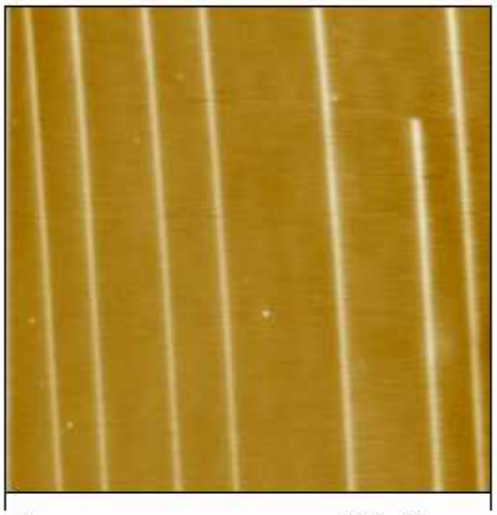
(*)Czeremuszkin and Latrèche, 43rd SVC Annual Technical Conference, 2000

Question: What is really happening before the development of the very first cracks?

What before fragmentation?

AFM allows to see what has been up-to-now hidden be the other used methods

3% strain

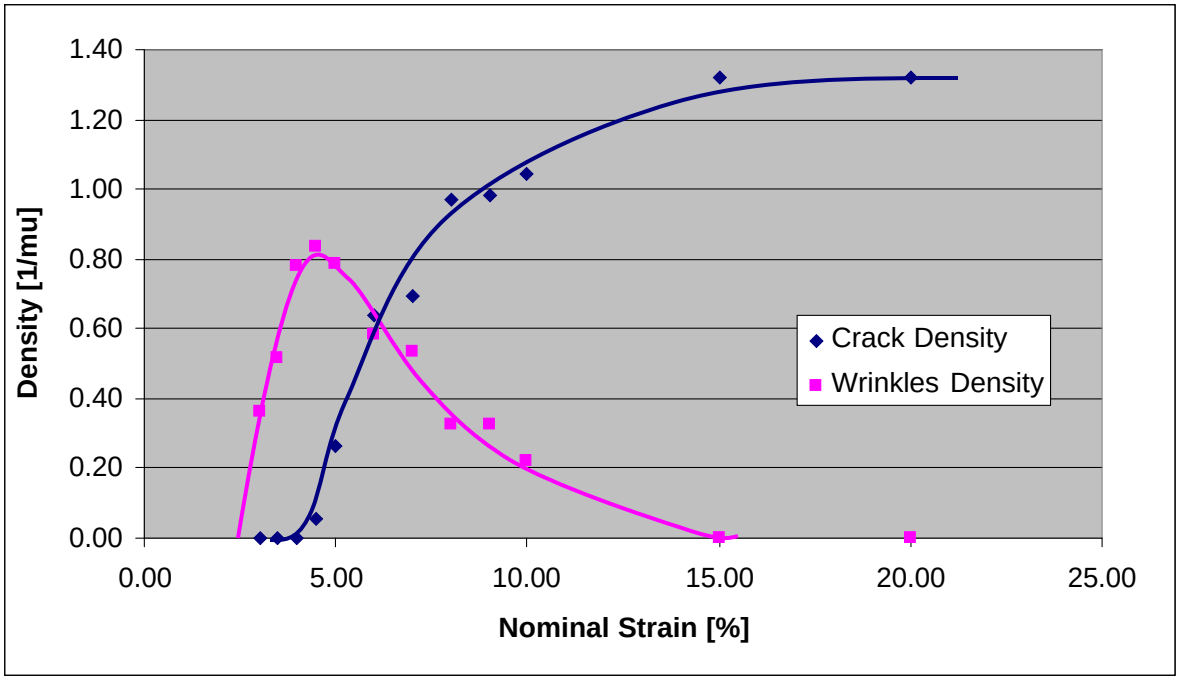


10 nm SiOx / RDO12

No explanation currently exists for the development of such “wrinkles” at the film’s surface

What before fragmentation?

The density of wrinkles increases before the initiation of fragmentation, and then decreases until it vanishes



10 nm SiOx / RDO12

Effect of substrate surface quality

Comparatively to what had been observed using SEM, the presence of anti-block particles at the substrate surface has been proven to decrease the coating resistance, and thus the crack onset strain by up to 25%

Sample	Crack Onset Strain [%]
20 nm SiOx / RNK12	3.0
20 nm SiOx / RDO12	3.5
20 nm SiOx / RHB12	3.6

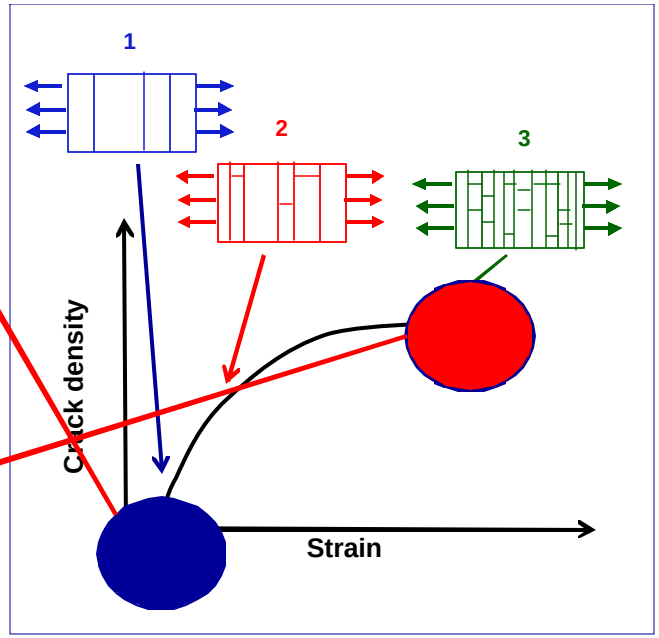
Micromechanical evaluation

Coating cohesive strength

$$\sigma_{max}(l) = \beta \cdot (l / l_0)^{-1/\alpha} \cdot \Gamma(1 + 1/\alpha)$$

Interfacial shear strength

$$\tau = \frac{2 \cdot h_c \cdot \sigma_{max}(l_c)}{l_c}$$



Sample	SiOx thickness (nm)	Cohesive strength [GPa]	IFSS [MPa]
SiOx / RDO12	20	3.6	90.2
	10	4.5	119.2

- The coating cohesive strength is higher for thin coating
- Coating/substrate adhesion is observed to be similar for all systems studied

Conclusions

AFM coupled with a micro-tensile stage is a versatile non-destructive technique allowing to:

- follow *in situ* cracks initiation and growth in inorganic coatings deposited on polymer substrates;
- determine mechanical properties and influence of surface morphology

Advantages compared to other techniques:

- Operation at atmospheric pressure
- No sample preparation needed
- High resolution in height

Conclusions

Results obtained have been shown to be comparable to the ones determined via SEM analyses in previous works. In particular:

For a given substrate:

- thick coatings are more brittle, and present lower cohesive strength
- the oxide/substrate adhesion strength is not related to coating thickness neither to substrate morphology, and denotes the presence of covalent bonds at the interface

For a given coating thickness:

- additives at the polymer surface induce local defects in the coating, decreasing the crack onset strain by up to 25%

Thank you for your attention!