

# Effect of anti-block particles on oxygen transmission rate of SiO<sub>x</sub> barrier coatings deposited by PECVD on PET films

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A collaboration between:



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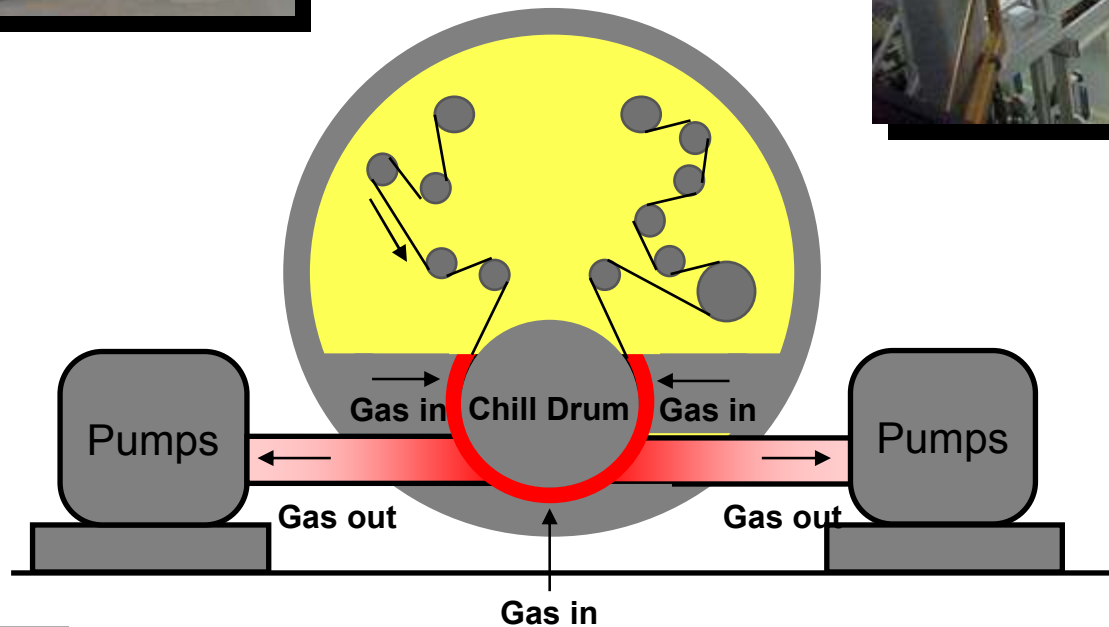
# Industrial PECVD Equipment at Tetra Pak



Typical OTR-Values:

Plain PET 12  $\mu\text{m}$   
 $120 \text{ cm}^3/\text{m}^2/\text{day}/\text{atm}$

PET/SiO<sub>x</sub> (15 nm)  
 $< 3 \text{ cm}^3/\text{m}^2/\text{day}/\text{atm}$



**Plasma** = Electrical Power + Gas mixture (Disiloxane, Oxygen)

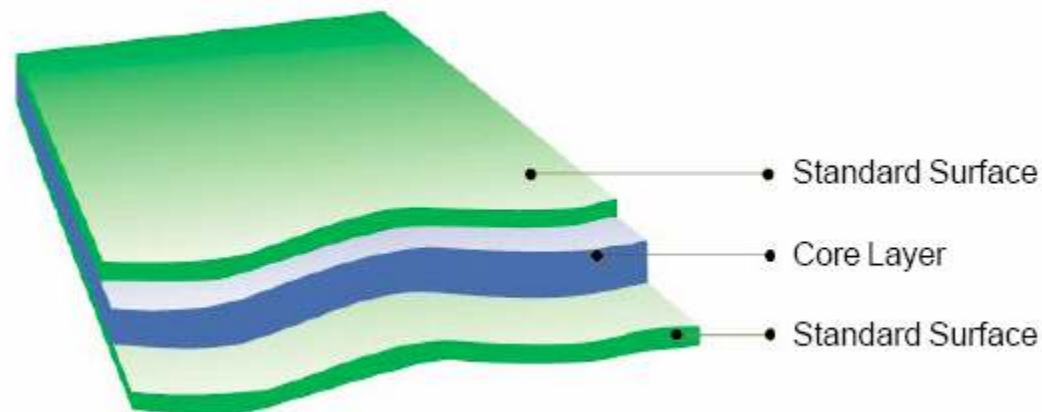
# ®Hostaphan RNK



Universal film with low haze

®Hostaphan RNK is a highly transparent, biaxially oriented, co-extruded film, made of Polyethylene Terephthalate (PET) and characterized by out-standing physical properties.

Layer structure ®Hostaphan RNK



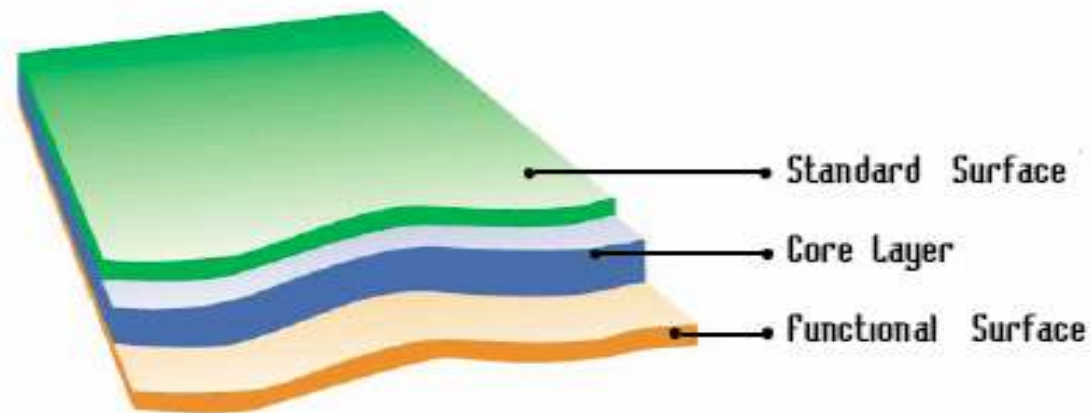
# ®Hostaphan RD



Film with one very smooth and glossy surface

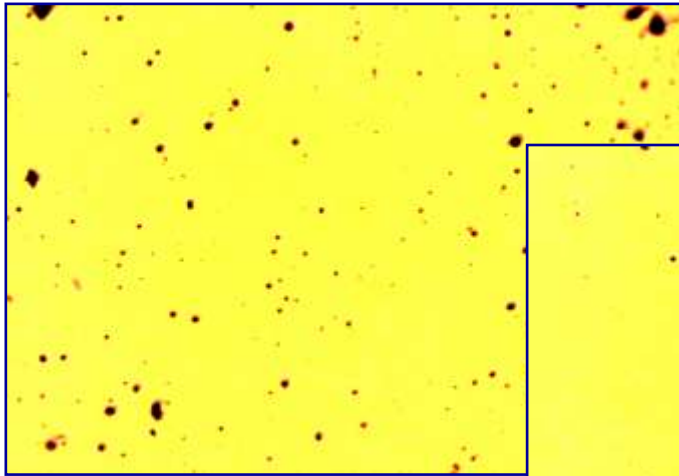
®Hostaphan RD is a highly transparent, biaxially oriented, co-extruded film, made of Polyethylene Terephthalate (PET) with different topography of the two surfaces. While the surface structure of one surface is the same as a standard PRT film, the functional surface side displays an extremely regular surface structure with very low roughness.

Layer structure ®Hostaphan RD

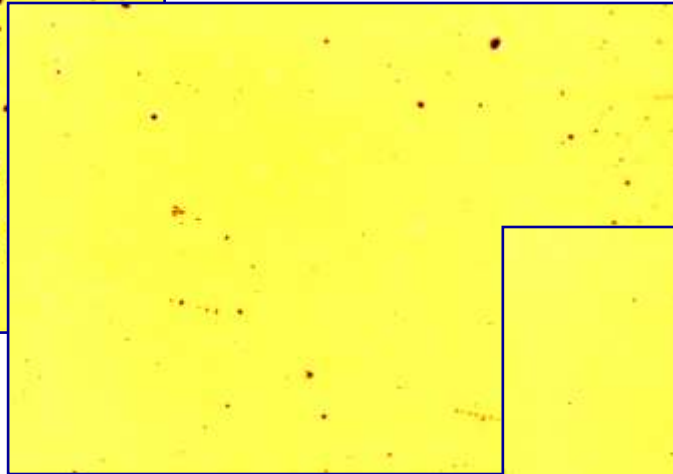


# PET Film Characterisation

*Reflexion Optical Microscopy (490x330  $\mu\text{m}^2$ )*



**RNK12**



**RD12**

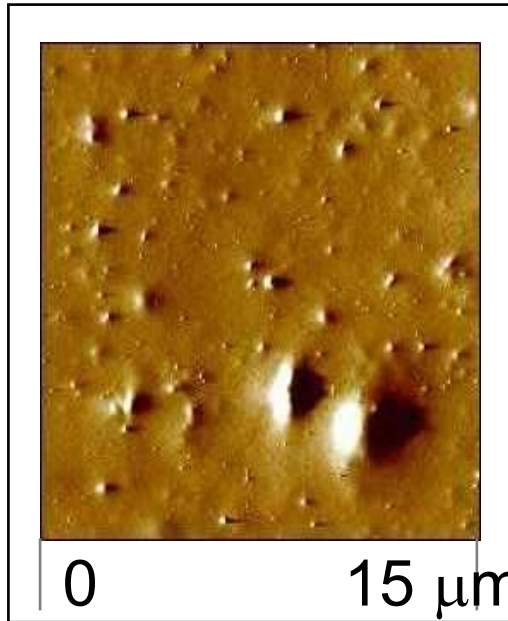


**RDO12**

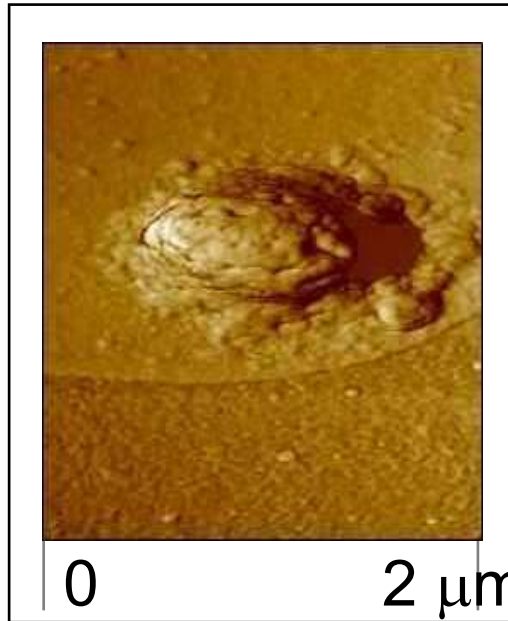
# Defects on PET Films

## *Size dispersion of anti-blocking silica and dust particles*

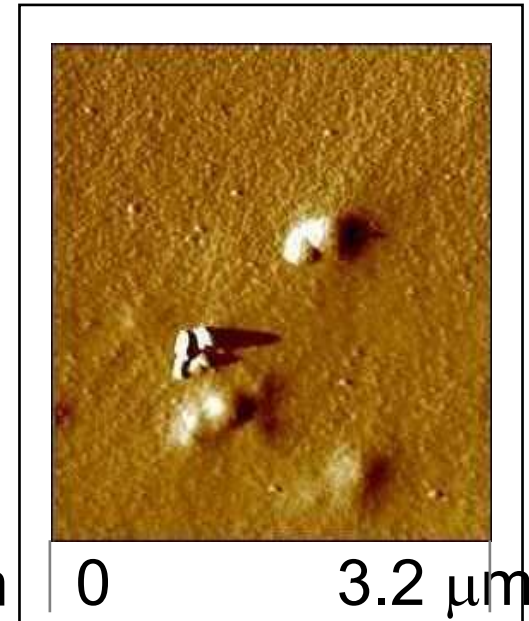
Defects of various sizes



Silica Anti-blocking

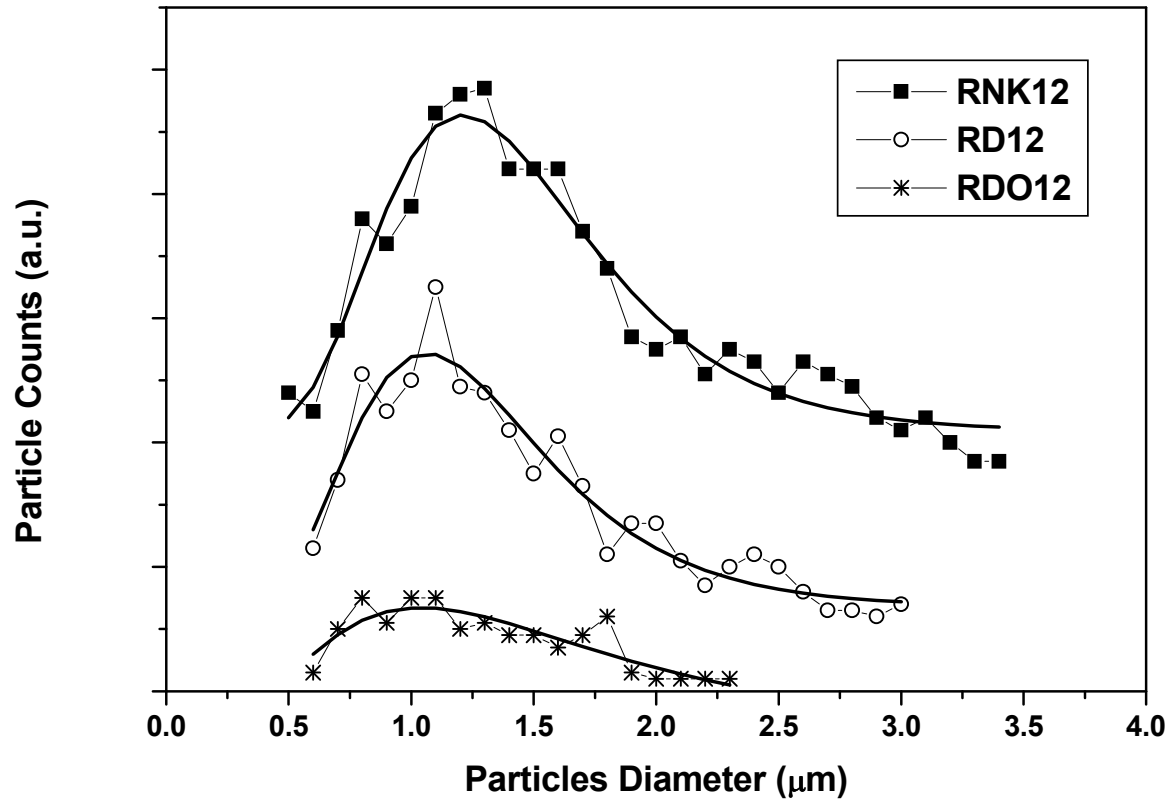


Dust particles

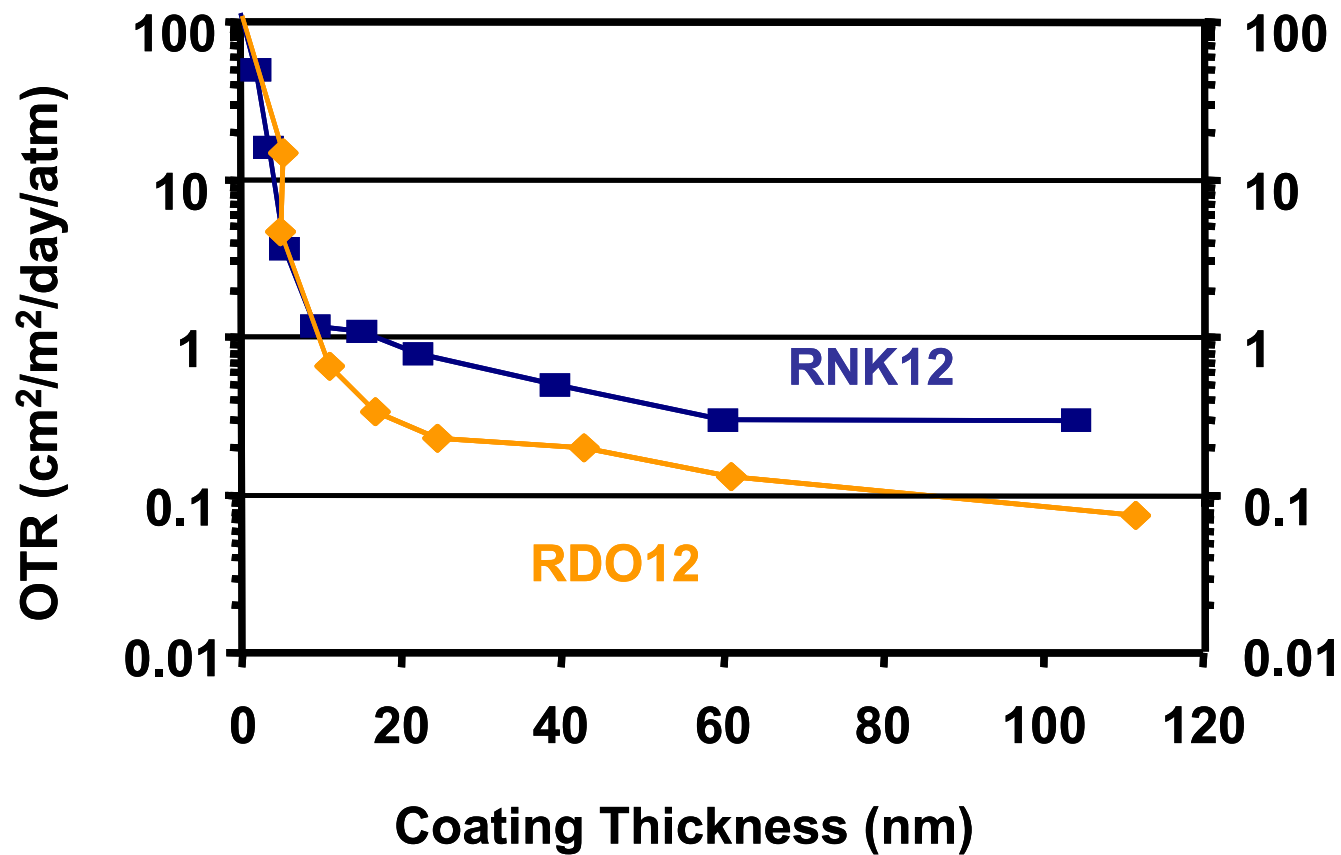


Data from tapping AFM: Amplitude

# Size Distribution of Anti-blocking particles



# OTR as a Function of Coating Thickness





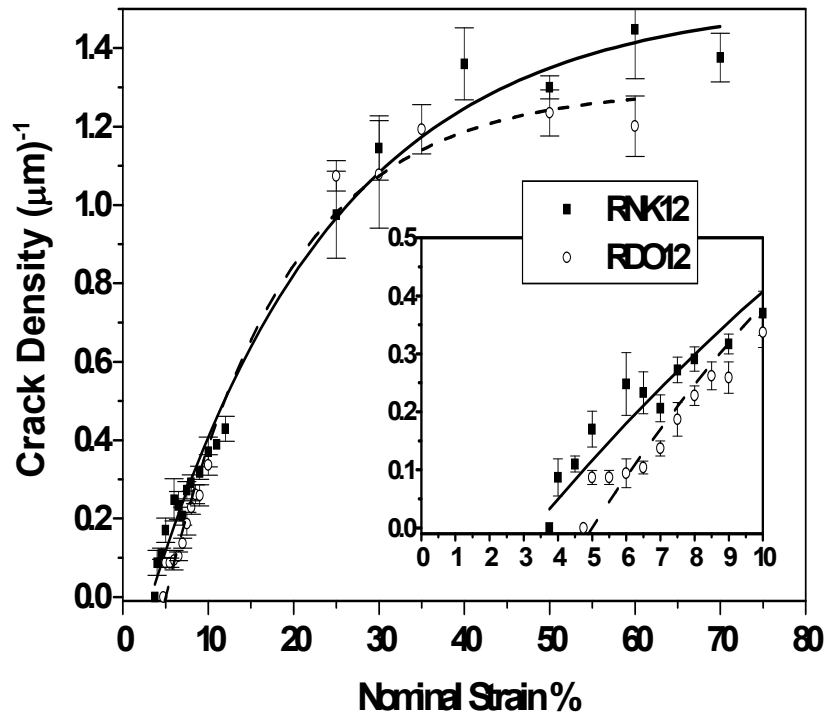
# Effect of Anti-blocking Particles on Oxygen Transmission Rate (OTR) for 40-nm SiO<sub>x</sub>

Film	Particles per mm <sup>-2</sup>	OTR* (cm <sup>3</sup> /m <sup>2</sup> /day/atm)
RNK12	1710	0.58 ± 0.1
RD12	860	0.39 ± 0.1
RDO12	290	0.21 ± 0.1

\* 23 °C, 50% RH

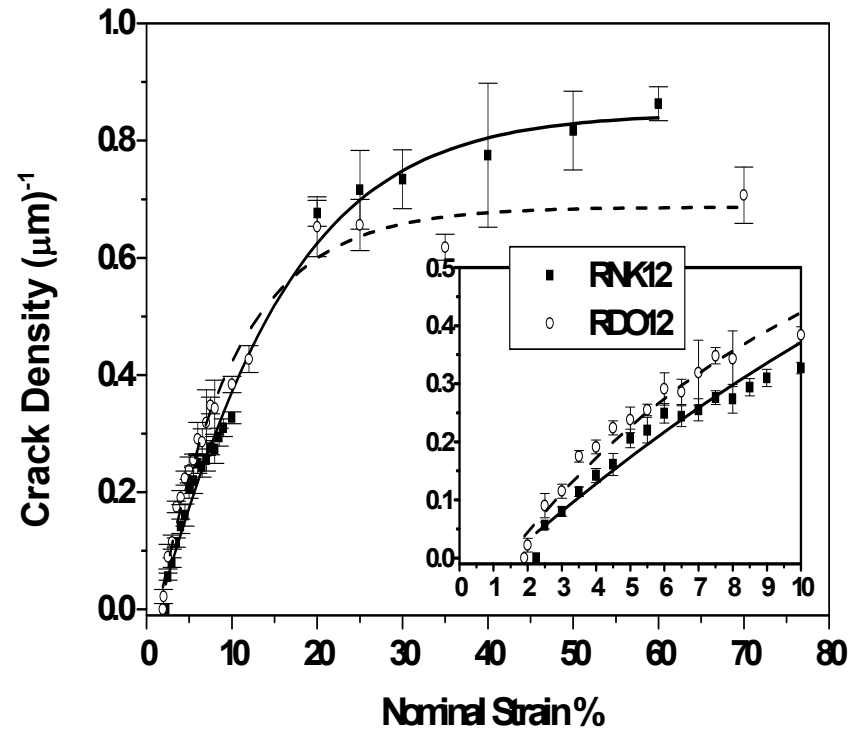
# Fragmentation Tests for SiOx Coatings

## 9-nm Thick



**COS: 3.8% RNK12  
4.8% RDO12**

## 40-nm Thick



**COS: 2.3% RNK12  
1.9% RDO12**

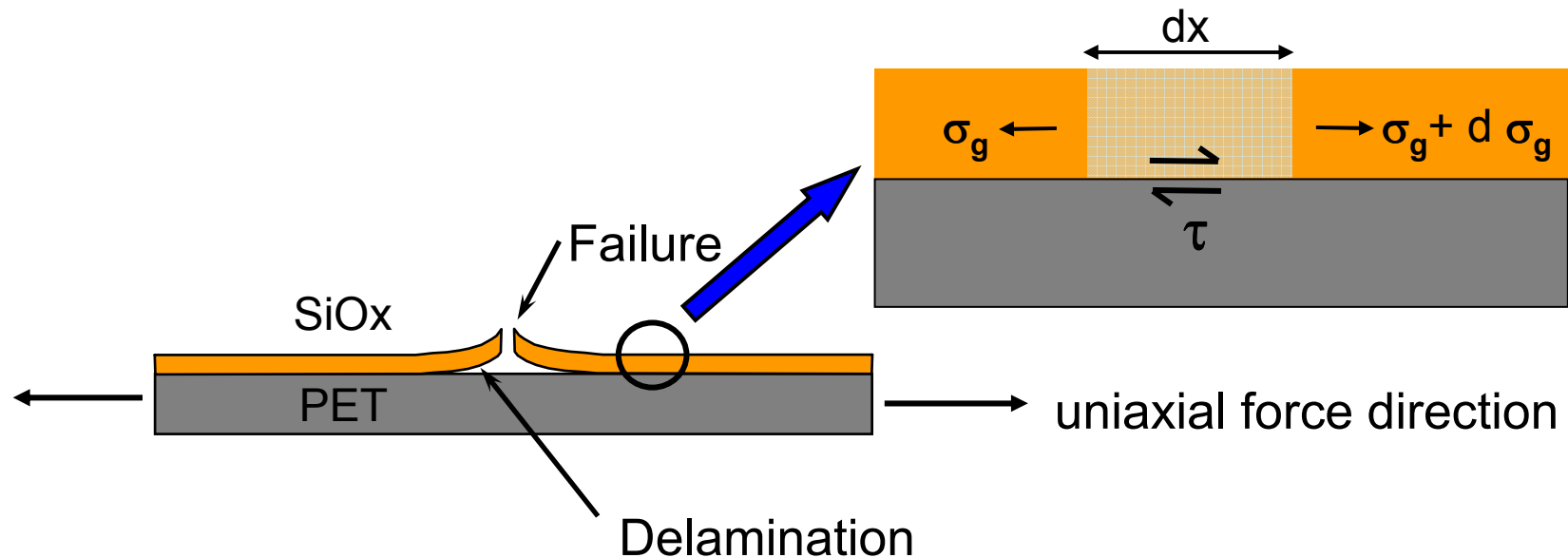
# Durability of SiOx Coated PET Films

The durability of the coating relies on :

Failure controlled by the coating cohesion strength

Delamination controlled by adhesion to substrate

Stress transfer at the coating/substrate interface



# Interfacial Shear Strength (IFSS) or Adhesion Strength

The coating's fragmentation process provides the mean fragment length at saturation,  $l_s$ , and the coating tensile strength  $\sigma_{\max}$  at critical length  $l_c = (2/3)l_s$ , from which is modeled the interfacial shear strength :

$$\tau = 1.337 \left( \frac{h}{l_s} \right) \sigma_{\max} (l_c)$$

# Cohesive Strength

The coating strength is modeled from a two parameters Weibull distribution :

$$\sigma_{\max}(l_c) = \beta \left( \frac{l_c h}{l_0 h_0} \right)^{-1/\alpha} \cdot \Gamma \{1 + 1/\alpha\}$$

The coating crack onset is derived from the theory of linear elastic fracture mechanics :

$$\varepsilon_f = \frac{C}{\sqrt{h_c (h_s + h_c)}}$$

# Mechanical Properties

## *9-nm Thick SiO<sub>x</sub>*

Film	COS (%)	$\sigma_{max}$ (GPa)	$\tau$ (MPa)
RNK12	3.8 ± 0.05	9.5	137
RDO12	4.8 ± 0.05	7.4	128

## *40-nm Thick SiO<sub>x</sub>*

Film	COS (%)	$\sigma_{max}$ (GPa)	$\tau$ (MPa)
RNK12	2.3 ± 0.05	2.8	94
RDO12	1.9 ± 0.05	4.7	196

# Conclusion

1. Oxygen transmission rate (OTR) and crack onset (COS) of PECVD SiO<sub>x</sub> depend on the density of anti-block particles at bi-oriented PET film surfaces.
2. OTR is proportional to the surface density of anti-blocking particles.
3. COS increased up by 20% for substrate with the lower anti-blocks density.
4. Micro-mechanical modeling of fragmentation process of SiO<sub>x</sub> coatings provided evaluation of their cohesive and adhesion strengths on PET.
5. Cohesion force of 9-nm thick SiO<sub>x</sub> is twice higher than 40-nm coatings.
6. Interfacial shear strength is as high as the bulk shear stress reflecting the presence of covalent bonds at the interface.

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