Rencontre GDR feux – Lille 2019

Anne-Lise Davesne
PhD Student

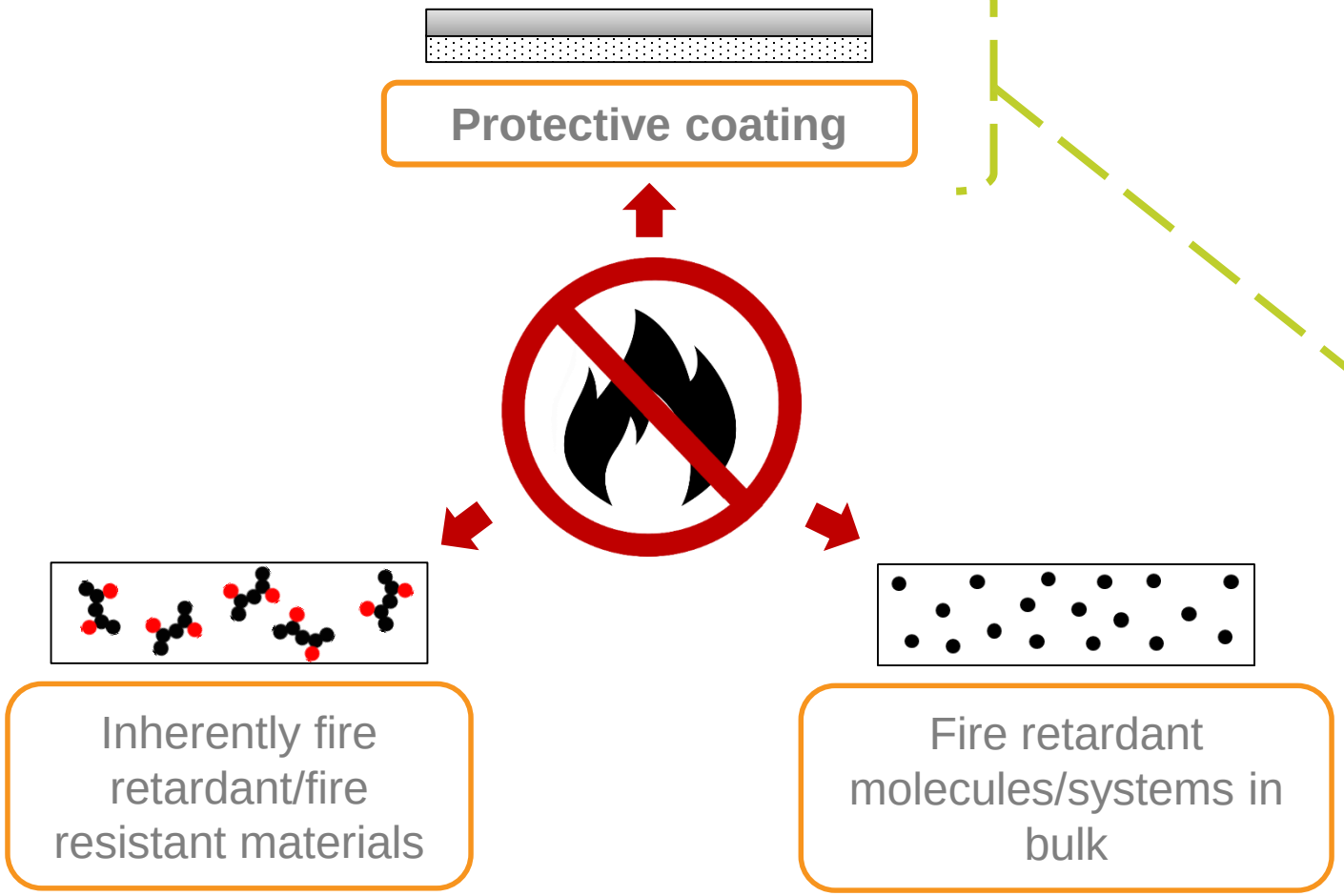
Low-emissivity metal/dielectric coatings applied to the fire protection of polypropylene and polyamide 6

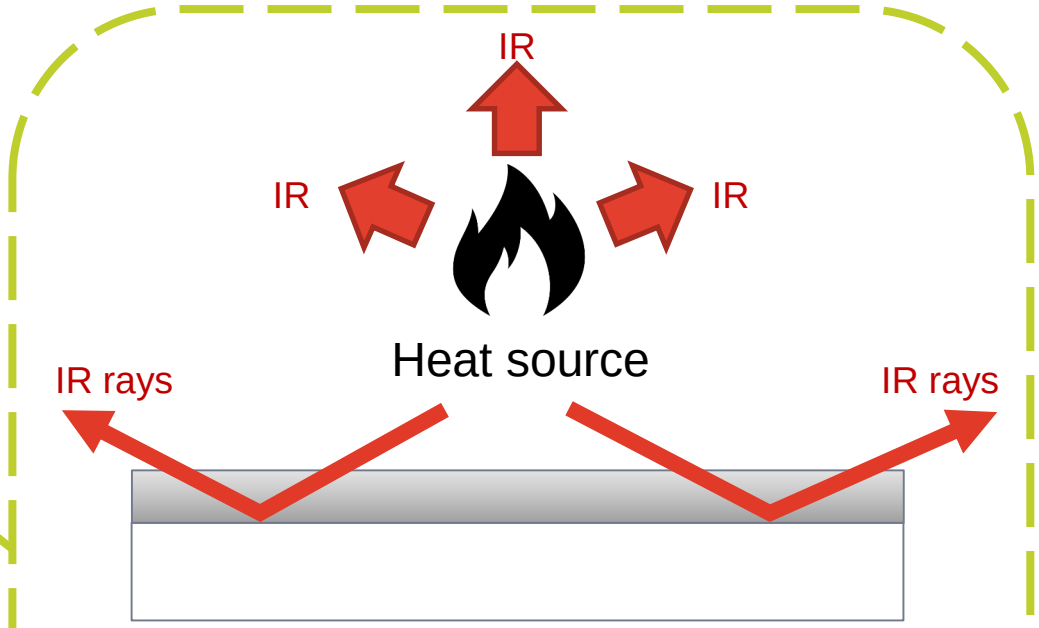
A-L. Davesne, T. Bensabath, J. Sarazin, S. Bellayer, F. Parent, F. F. Sanchette, F. Samyn, M. Jimenez, S. Bourbigot

Groupement De Recherche Feux 2864
cnrs
Université de Lille
utt université de technologie Troyes
FireBar Concept
erc European Research Council

Introduction

Fire protection of polymers:





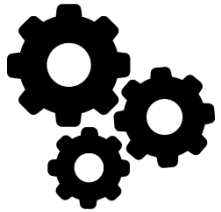
Radiative transfer dominant in medium to large scale fire

Radiative barrier = low-emissivity surface (high reflectivity)

J. F. Sacadura, Radiative heat transfer in fire safety science, *J. Quant. Spectrosc. Radiat. Transf.*, vol. 93, no. 1–3 SPEC. ISS., pp. 5–24, 2005

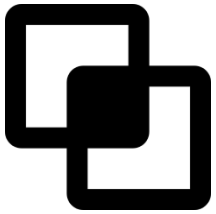
Overview

Introduction



Coating design and deposition

Proof of concept : cone calorimetry testing



Combination of bulk and surface mechanisms

Credit : flaticon, icomoon

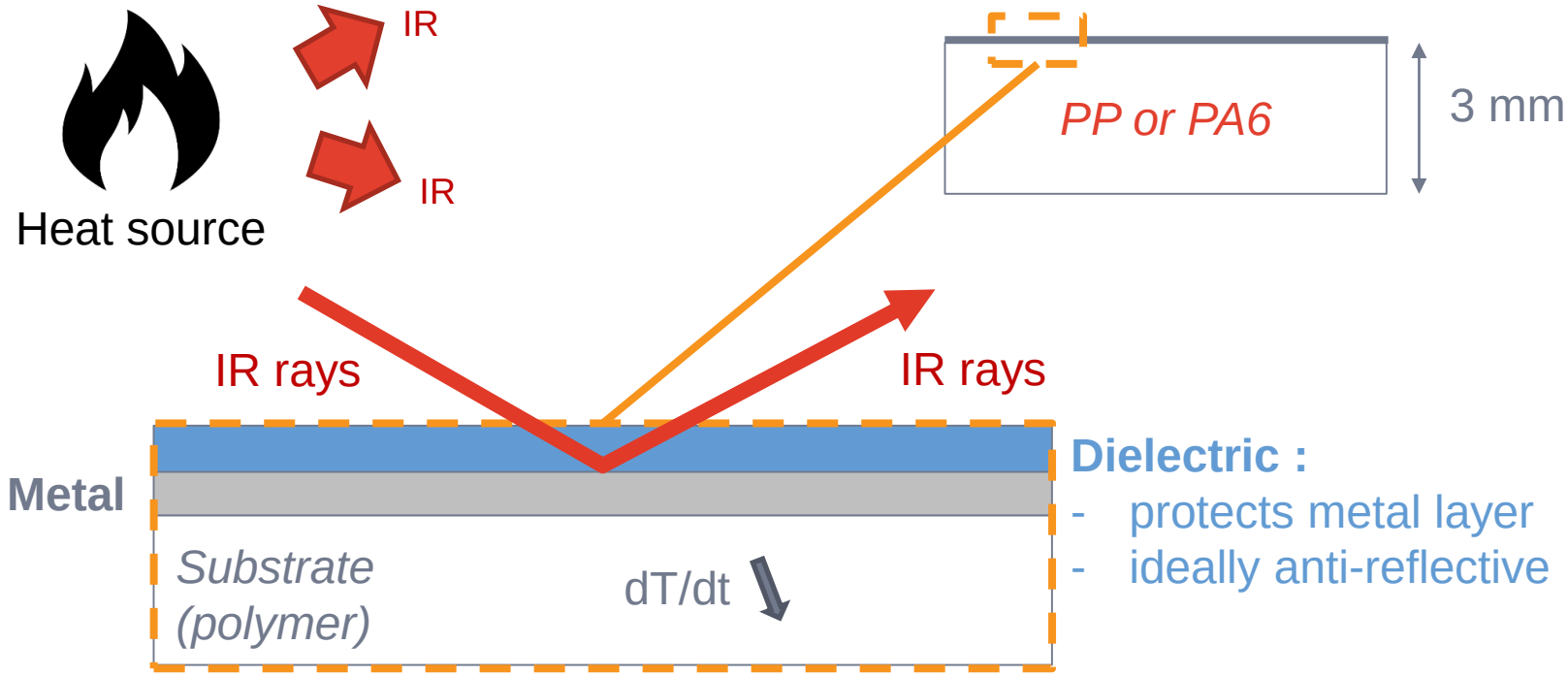
Strategy and choice of materials

2011 : Schartel et al [1]
Trilayer Cr/Cu/SiO_2
on PC and PA6,6

2014 : Försth et al [2]
ITO on PMMA
VO_2 on steel

2015 : Sonnier et al [3]
Semi-transparent PE+Al
flakes coating on PE

2017 : Försth et al [4]
VO_2 on glass



High density of free electrons ➔ low emissivity:
- High reflectivity (UV/visible-IR)
- Low absorptivity

[1] Schartel et al, "Sub-micrometre coatings as an infrared mirror: A new route to flame retardancy," *Fire Mater.*, vol. 36, pp. 671–677, 2012.
[2] Försth et al, "Spectrally selective and adaptive surfaces for protection against radiative heating: ITO and VO2," *Fire Mater.*, vol. 38, pp. 111–124, 2014.
[3] Sonnier et al, "Controlled emissivity coatings to delay ignition of polyethylene," *Materials (Basel).*, vol. 8, no. 10, pp. 6935–6949, 2015.
[4] Ochoterena and Försth, "The effect of thermochromic coatings of VO2on the fire performance of windows," *Fire Mater.*, no. December 2017, pp. 2–5, 2018.

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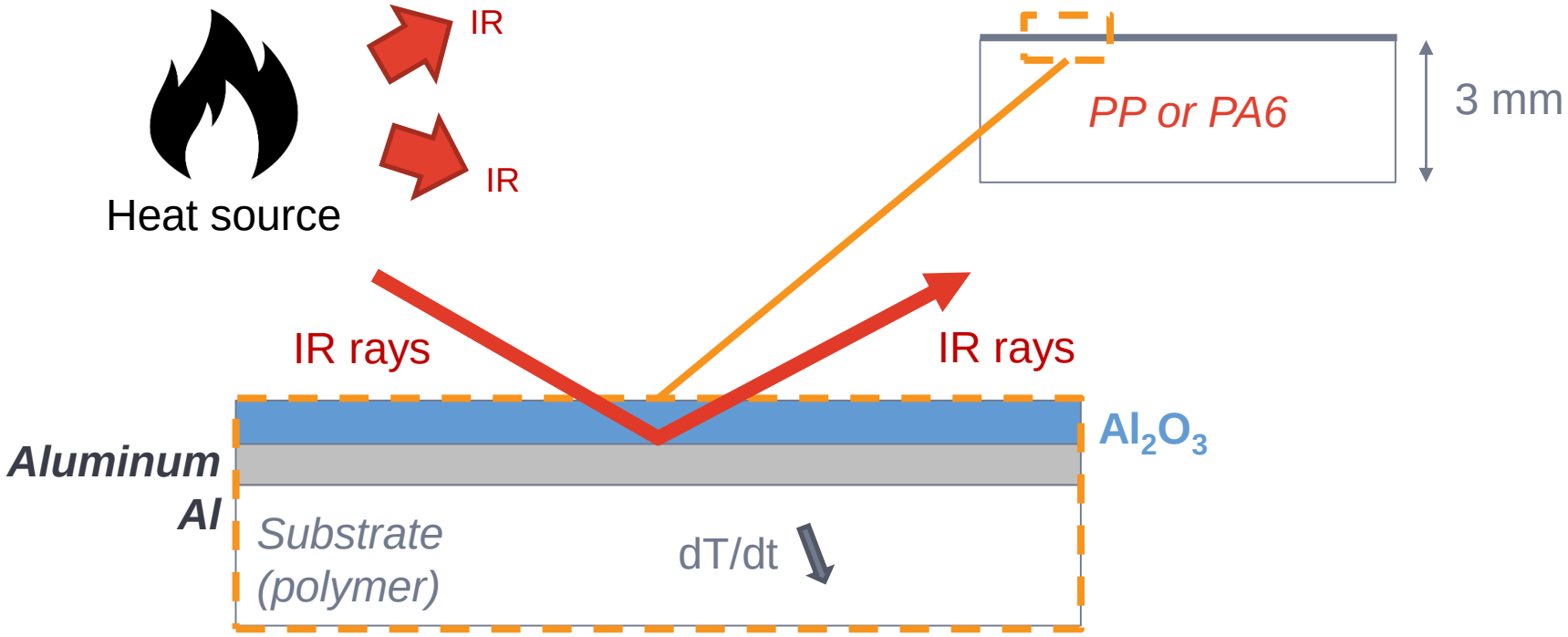

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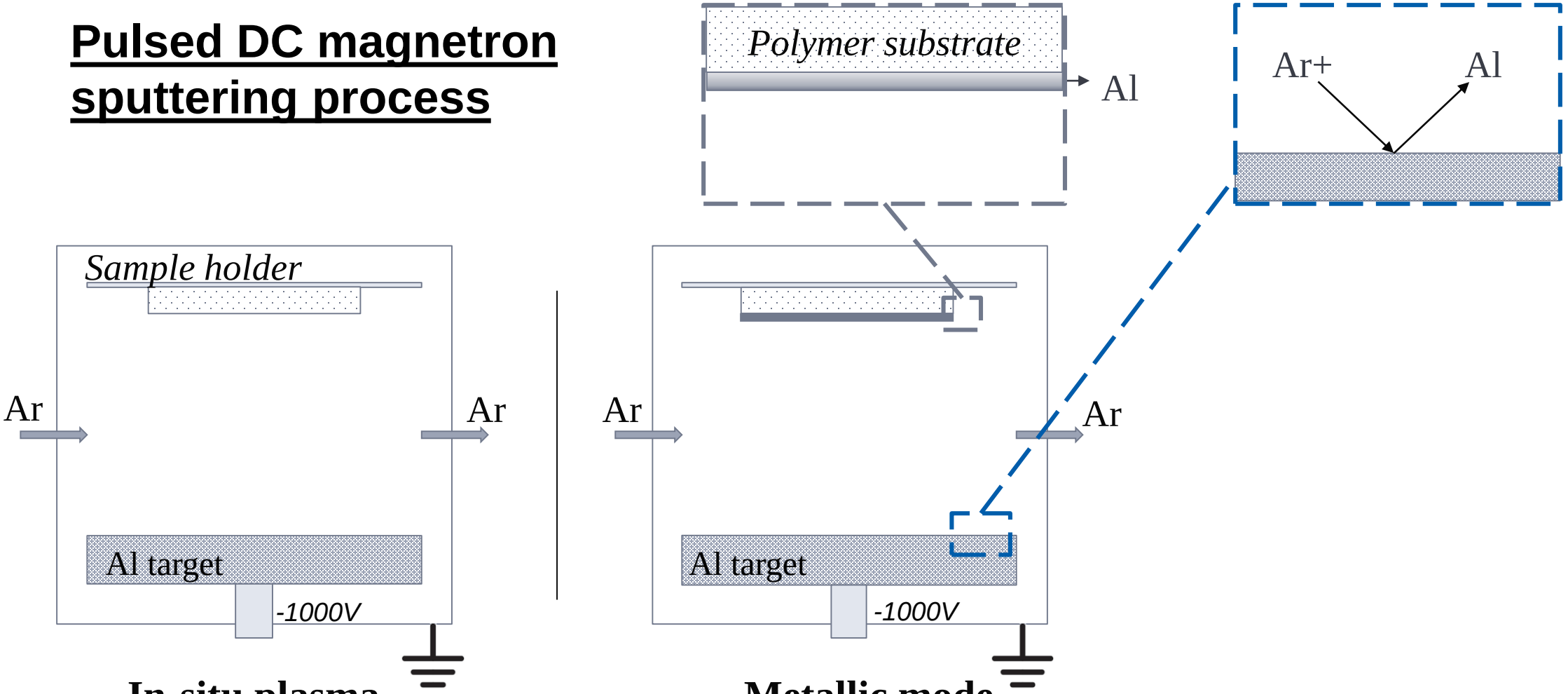
Coating deposition

Pulsed DC magnetron sputtering process



In-situ plasma pre-treatment

- Adhesion enhancement -

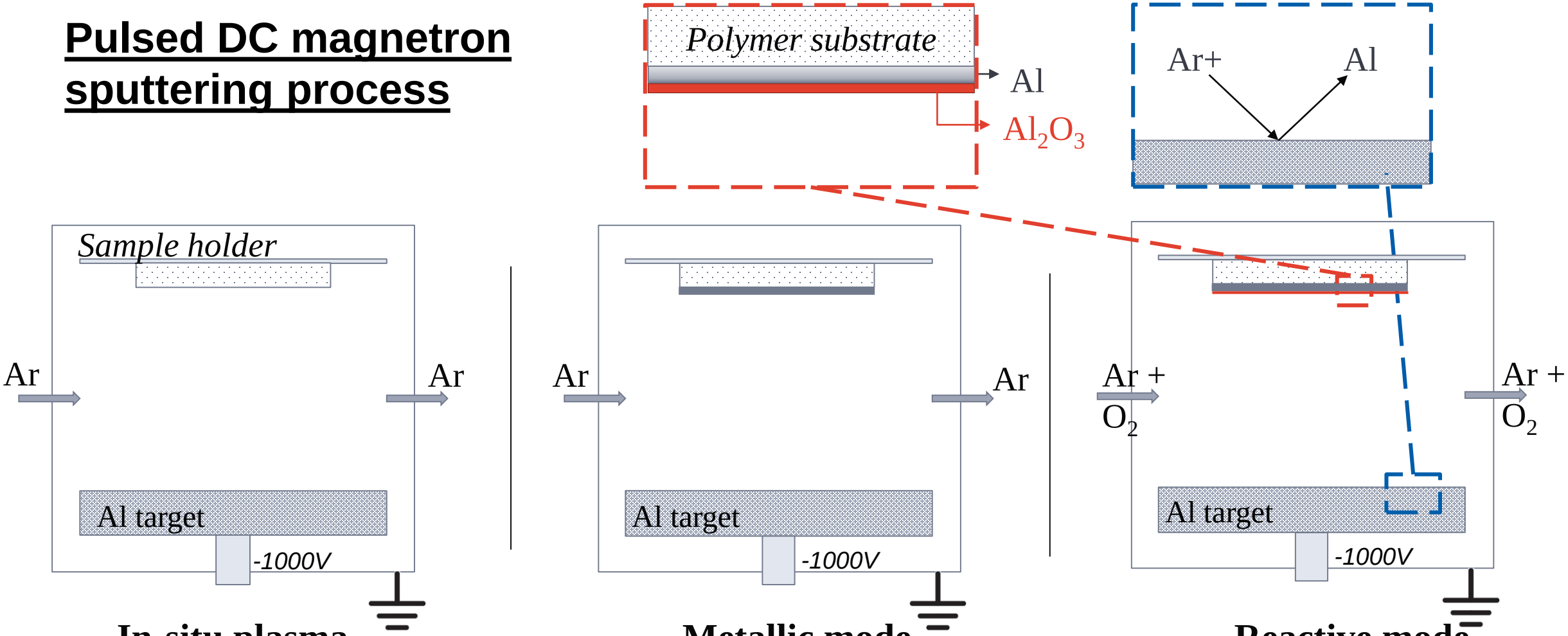
Metallic mode

- Aluminum deposition -

30 min

Coating deposition

Pulsed DC magnetron sputtering process



In-situ plasma pre-treatment

- Adhesion enhancement -

Metallic mode

- Aluminum deposition -

30 min

Reactive mode

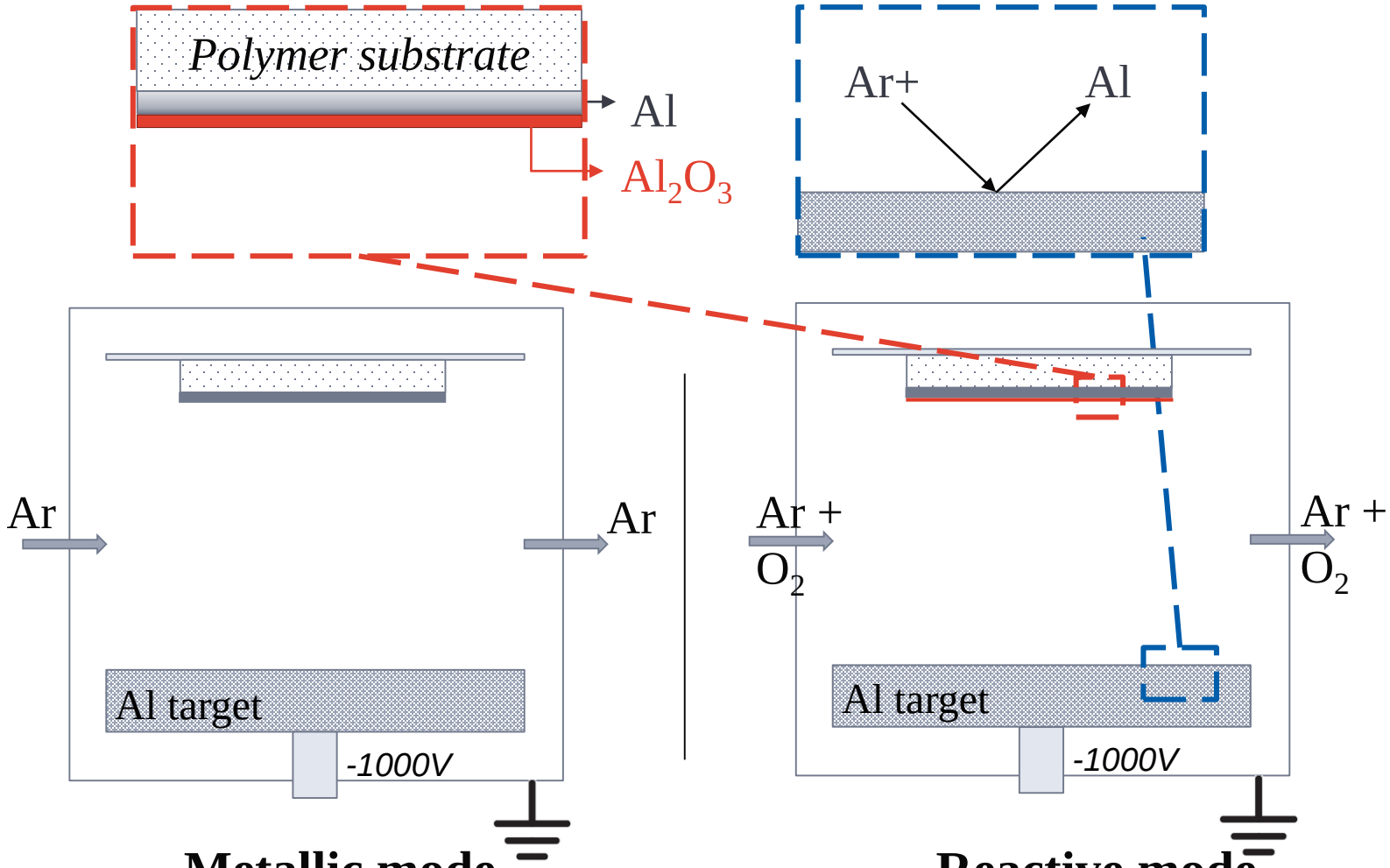
- Alumina deposition -

60 min

Coating deposition

Pulsed DC magnetron sputtering process

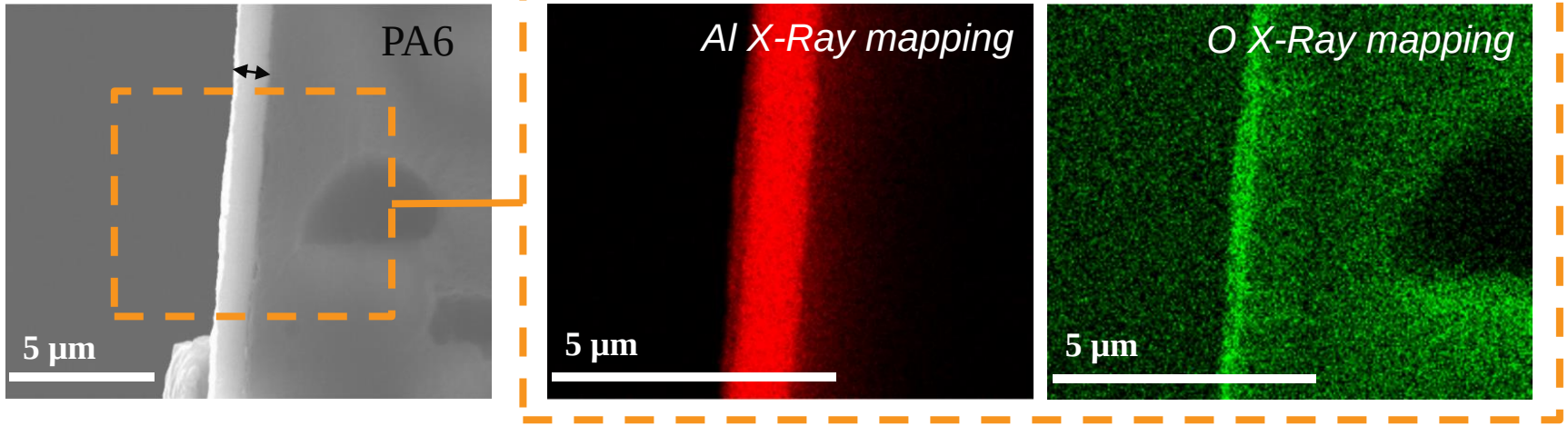
- **Low pressure** and **low temperature** deposition process
- **Condensation** of the vapor on the cold substrate



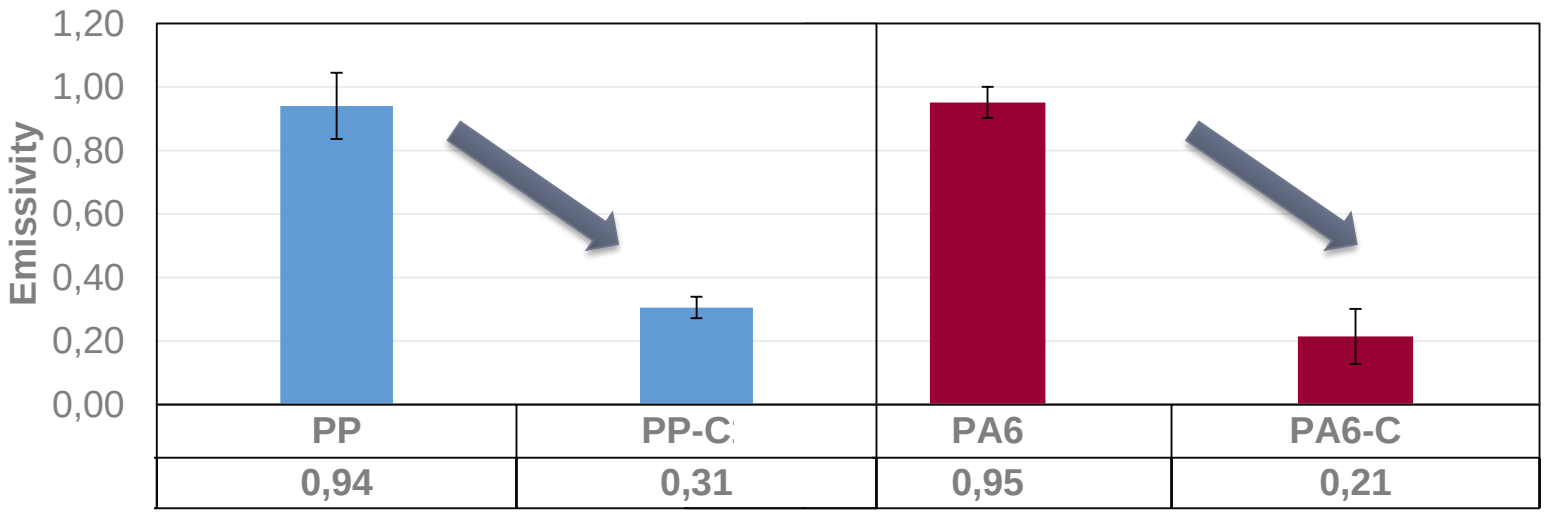
Metallic mode
- Aluminum deposition -
30 min

Reactive mode
- Alumina deposition -
60 min

Caracterizations



Total thickness : **1.02 ± 0.03 µm**
Al_2O_3 layer thickness : **0.08 ± 0.02 µm**



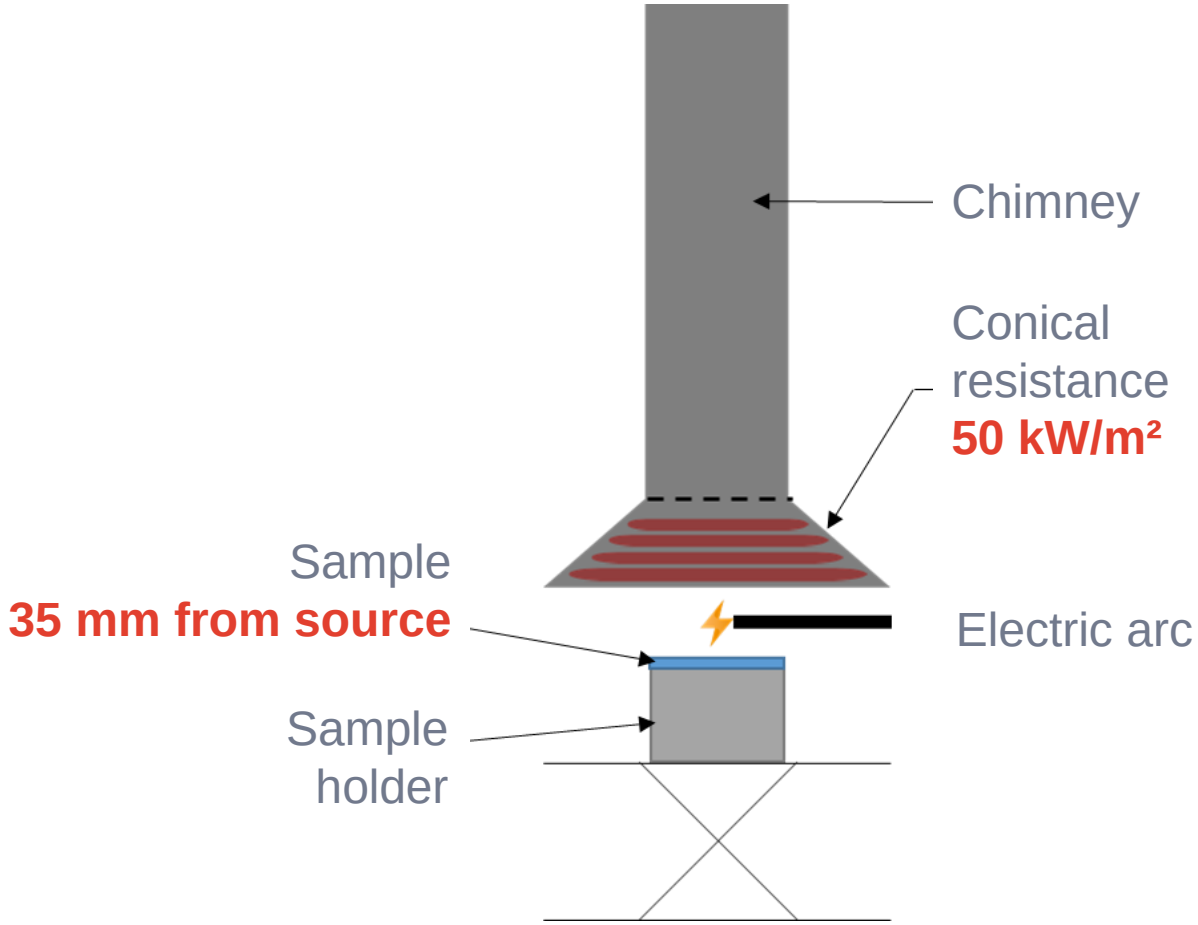
Emissivity measurements
(infrared diffuse reflectance measurement)
Bruker Vertex 70v FTIR spectrometer

$$\rho = 1 - \varepsilon$$

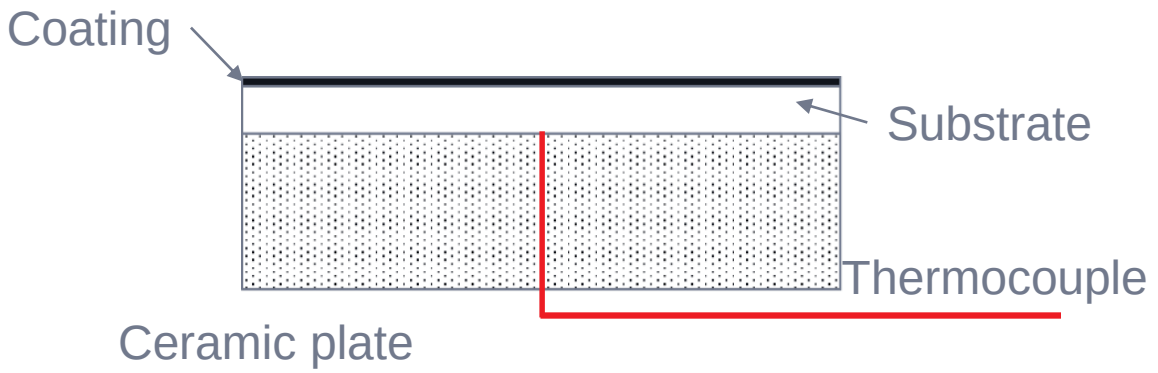
Reflectance of infrared rays enhanced

Experimental set-up

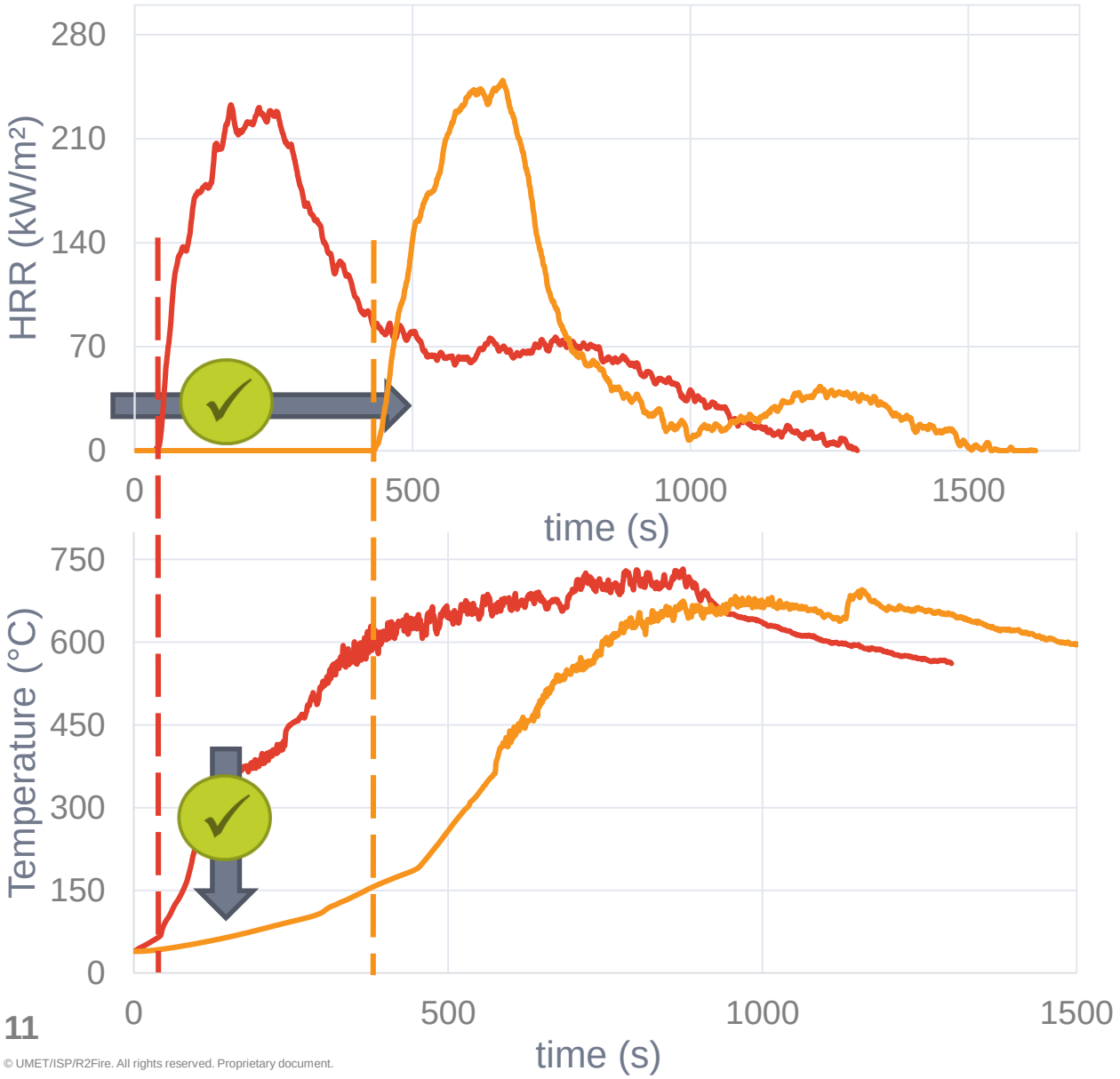
Mass loss cone
(sample size 5x5x0,3 cm³)

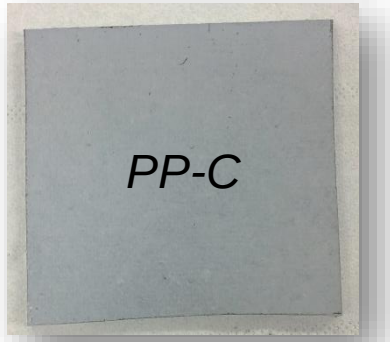


Evolution of temperature



Polypropylene substrate

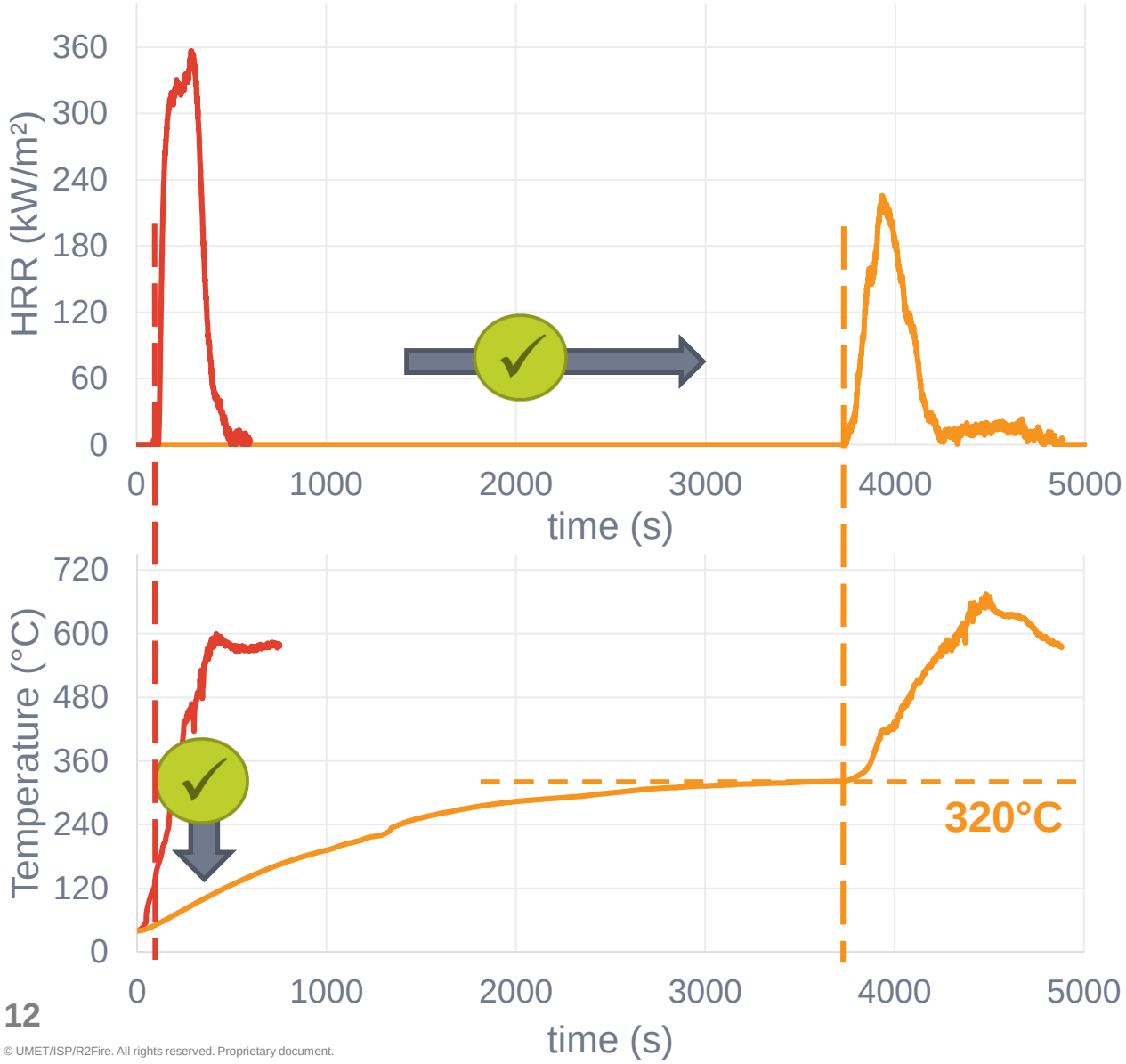


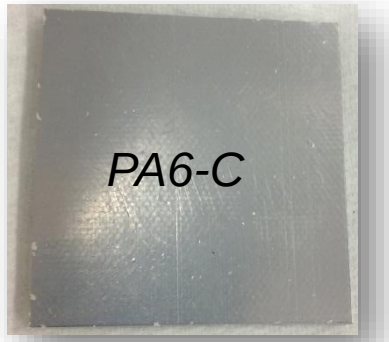


- Time to ignition 40s → ***7 min***
- Temperature rise ↘
- Burning behavior =

PP — PP-C

Polyamide 6 substrate



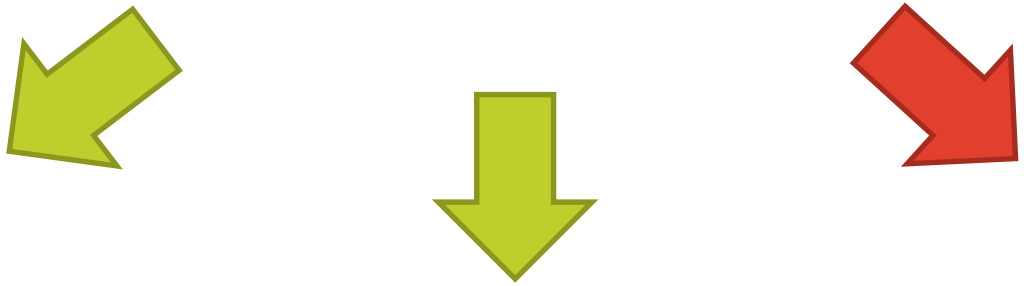


- Time to ignition 75s ➔ + ***1 h***
- Temperature rise ↘
- Burning behavior = ↘

PA6 — PA6-C

Conclusion

Proof of concept validated



Long ignition time

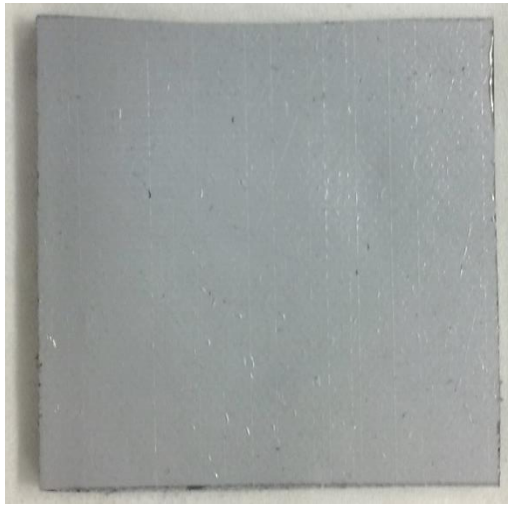
Heating rate slowed down

➔ PA6 : temperature stabilized to a plateau.

Burning behavior is not affected

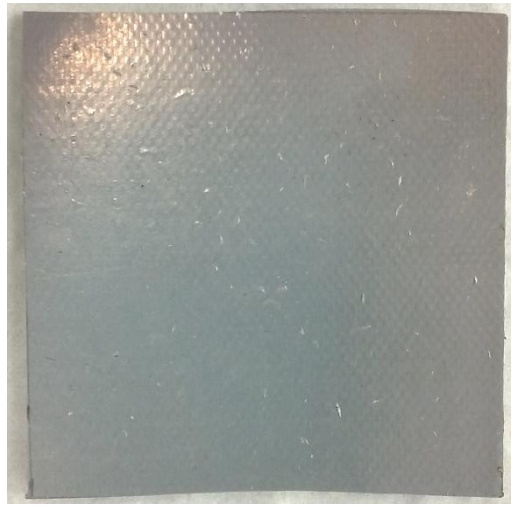
Objective and experimental

PP + **10% EG**



Expandable graphite
Physical intumescence
(« worms »)

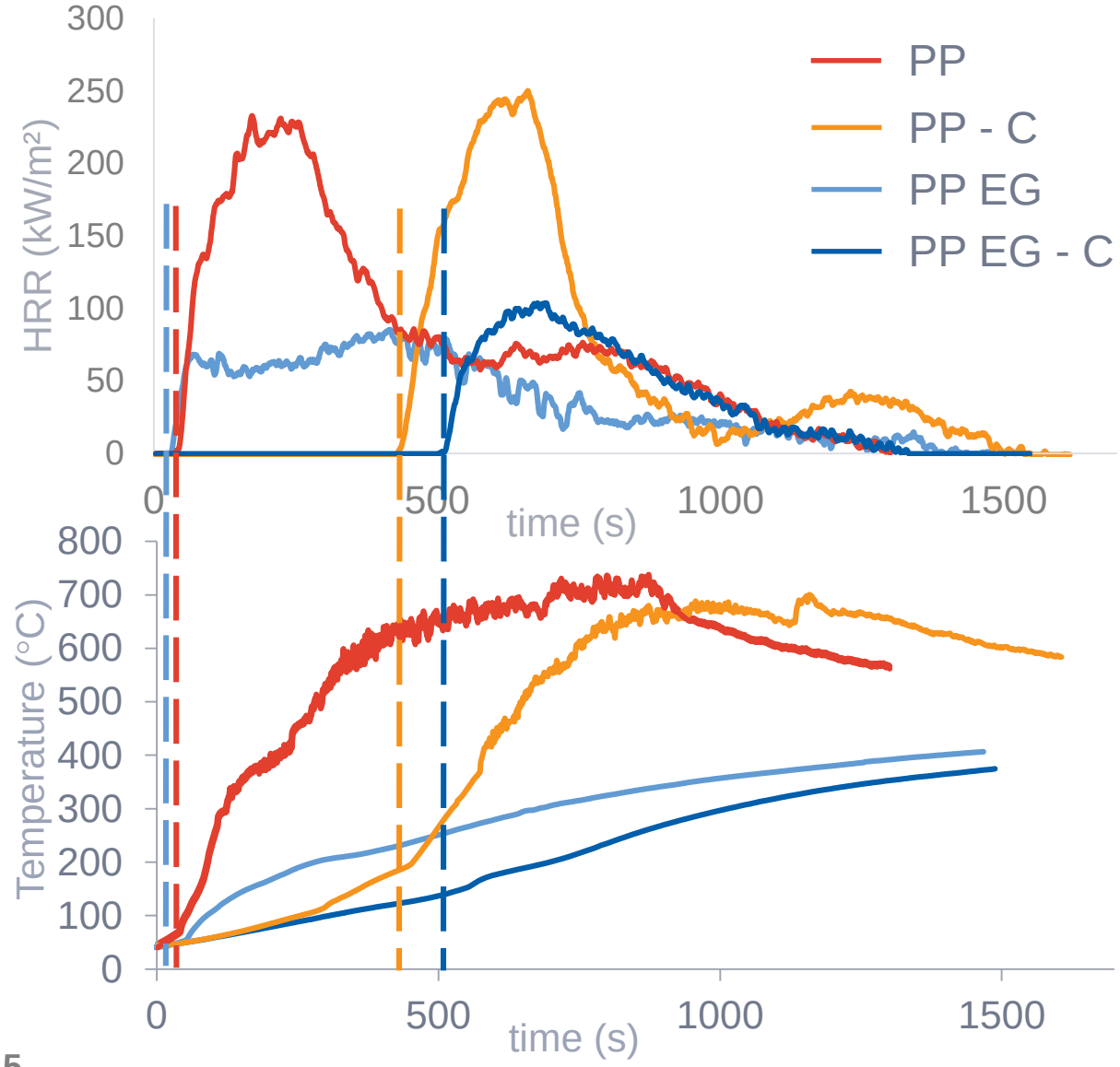
PA6 + **23% OP1311** (Exolit Clariant)

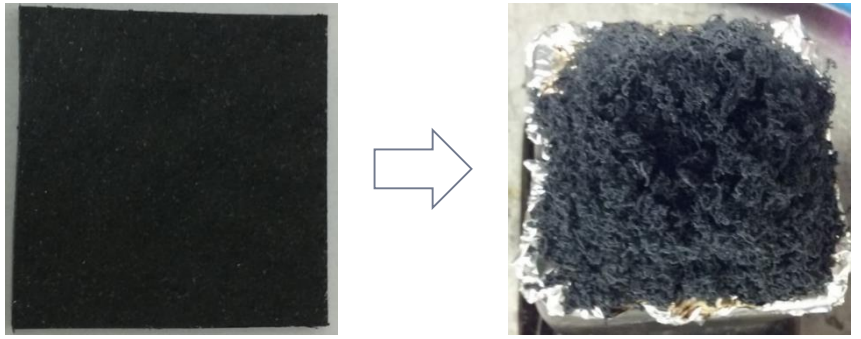


DEPAL + MPP
Chemical intumescence

Deposition conditions : time of deposition	
Al	**Al_2O_3**
30 min	60 min

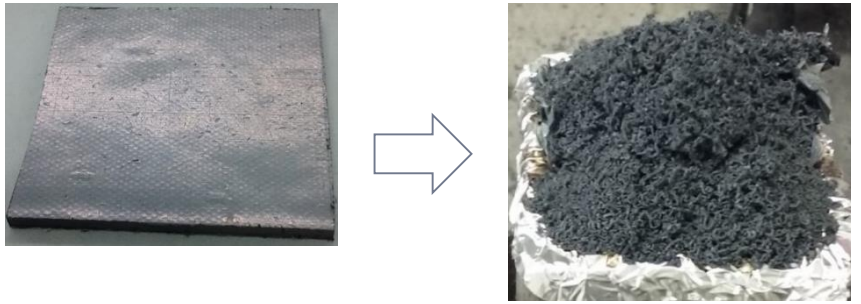
Polypropylene substrate





Low time to ignition ✗ PkHRR -50% ~ ✓

Low heating rate ✓ THR -50% ~ ✓



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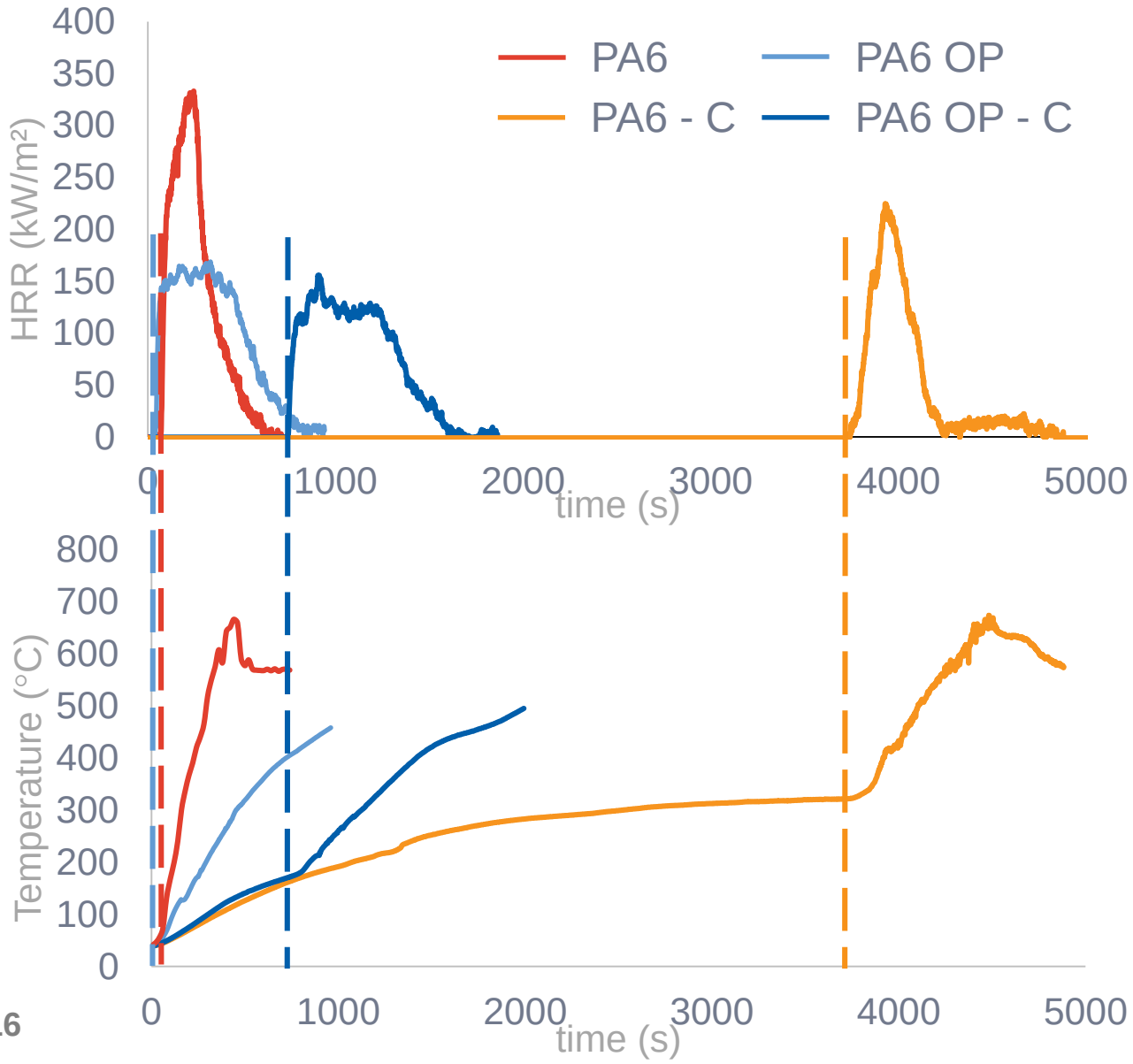
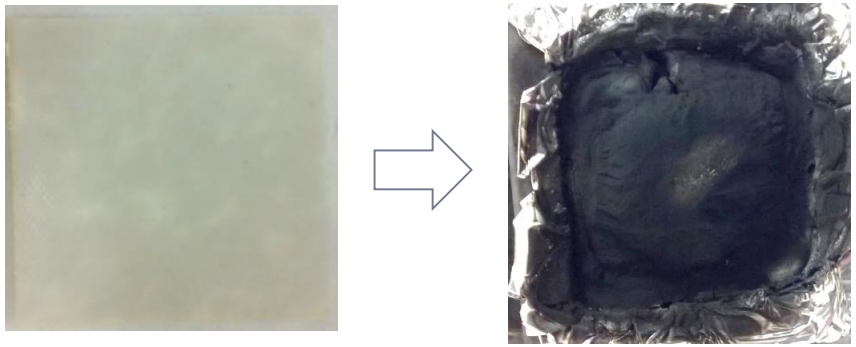
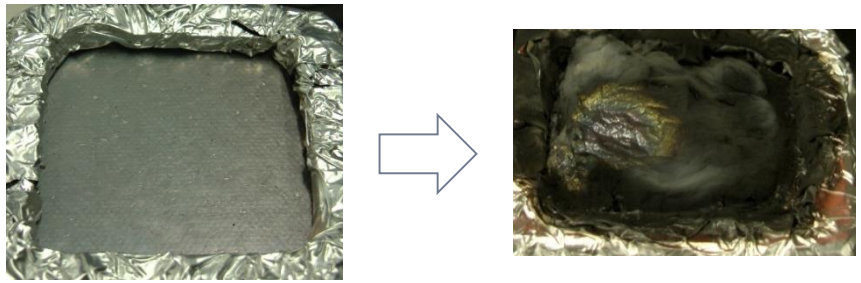
Polyamide 6 substrate





Low time to ignition ✗ PkHRR -50% ~ ✓

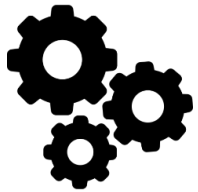
Low heating rate ✓ THR -10% ~



Long time to ignition ✓ PkHRR -50% ~ ✓

Low heating rate ✓ THR -10% ~

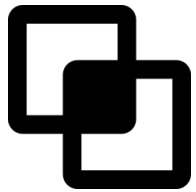
Conclusion



- **Ignition time** increased
- **Temperature rise** slowed down

Burning behavior not impacted

Combination with bulk fire retardant solutions successful



- Test other metals and fire retardant mechanisms
- Test other substrates → innovative 3D designs (Laura Geoffroy)

Acknowledgment

Thank you for you attention!

Pr. Serge BOURBIGOT

Pr. Maude JIMENEZ

Dr. Fabienne SAMYN

For collaboration on the project :
PVD deposition process

Pr. Frédéric SANCHETTE

M. Fabrice PARENT

(LASMIS, Université Technologique de Troyes, Nogent antenna)

For MEB imaging

Dr Séverine BELLAYER

For help with emissivity measurements

Dr Tsilla BENSABATH

M. Johan SARAZIN

…and everyone in the Firebar concept program

Université de Lille

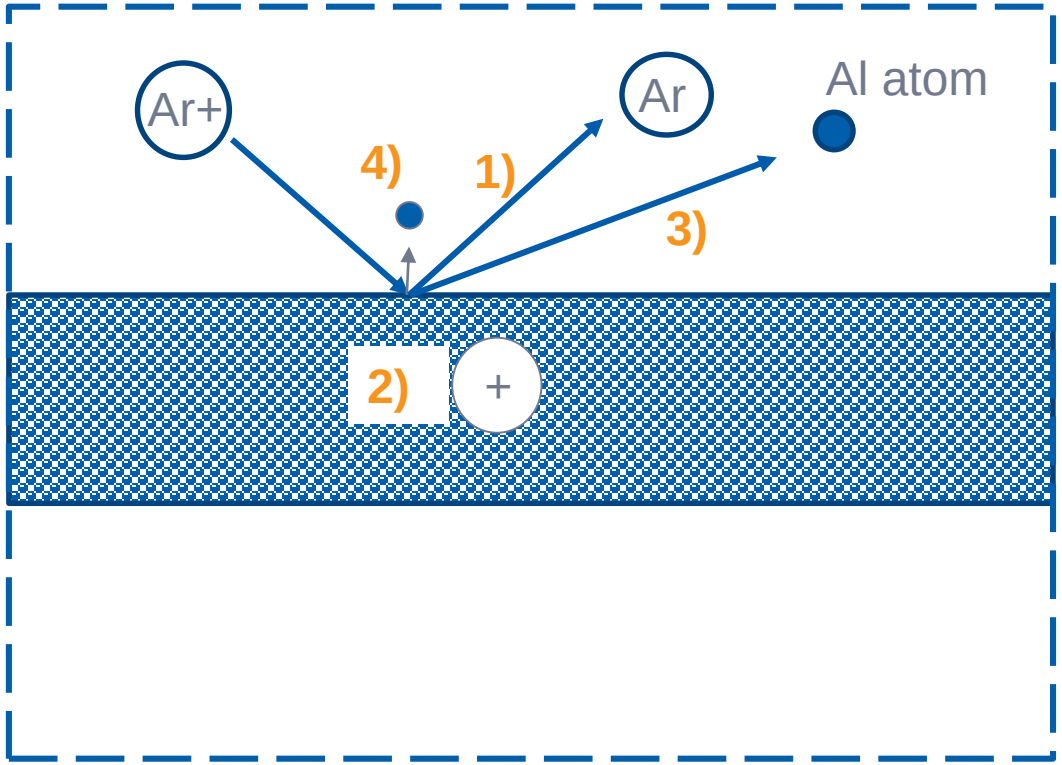
FireBar Concept

erc European Research Council

utt université de technologie Troyes

cnrs

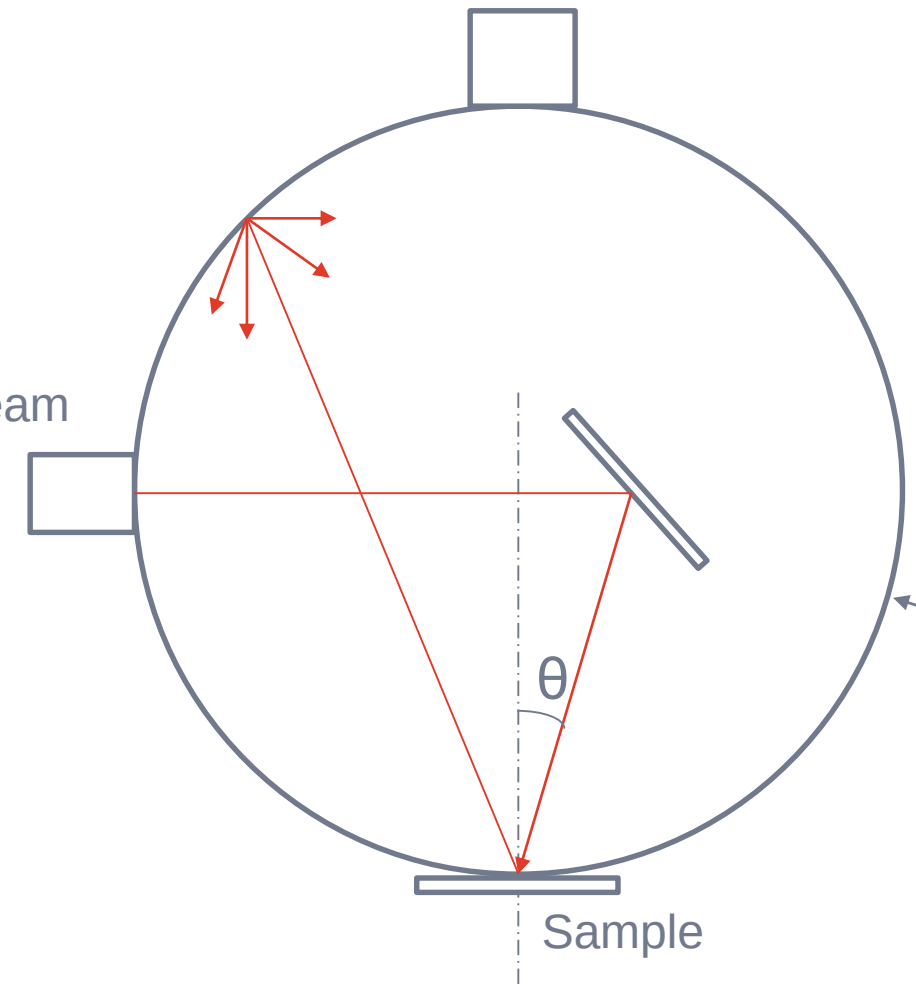
Pulsed DC magnetron sputtering



1) Elastic reflection of neutralized ion
2) Ion implantation
3) Aluminum atom sputtering
4) Secondary electron emission

infrared diffuse reflectance measurement : integrating sphere

Measurement of directional-hemisphérical reflectance



Reflectance of black body : R(BB)
Reflectance of sample : R(S)

At T (°C)

⇒ Emissivity of sample

Integrating sphere
(gold coated)

θ = 13°

PP – SEM observations