Thermal behavior of carbon fibers PEKK composites exposed to kerosene flame

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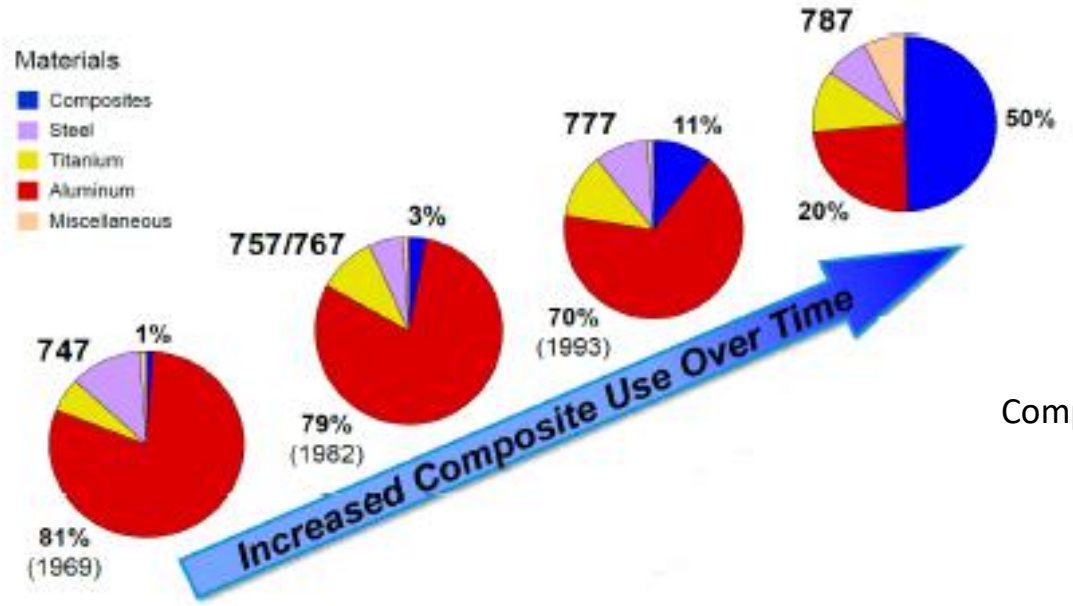
Introduction

What kind of composite materials for future aircrafts ?



Composite materials trend [1]

- Focus shifted from thermoset to thermoplastic (TP) based composites

[1] Roeseler WG, Sarh B, Kismarton MU. Composite structures: the first 100 years. *16th International Conference on Composite Materials. ICCM 16*, July 2007, Japan.

- Collaboration

CORIA (COmplexede Recherche Interprofessionnel en Aérothermochimie)

- Interaction between fire and composite structure

GPM (Groupe de Physique des Matériaux)

- Behavior of composite material under mechanical loading

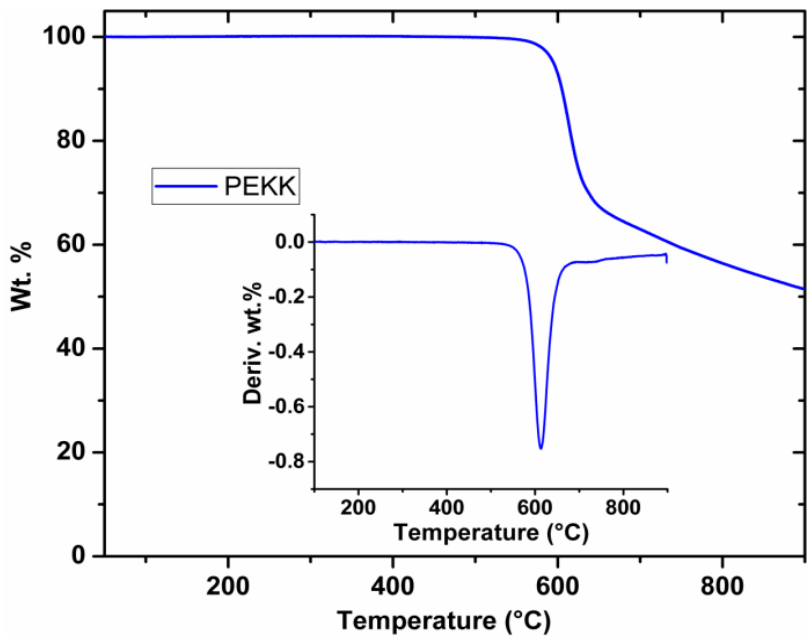
Motivation of study

- Thermoplastic (TP) composite: promising for aeronautical applications.

Thermoplastic (TP)composite: (e.g. PEEK, PEKK, PPS)			
Pros	Ability to be recycled	Cons	Lower mechanical properties
	Low cost processing/cost effectiveness		Poor adhesion at fiber/matrix interface
	Corrosion resistance		Higher processing temperature above glass transition temperature
	Fire resistance		
	Enhanced impact toughness		

TGA test conditions

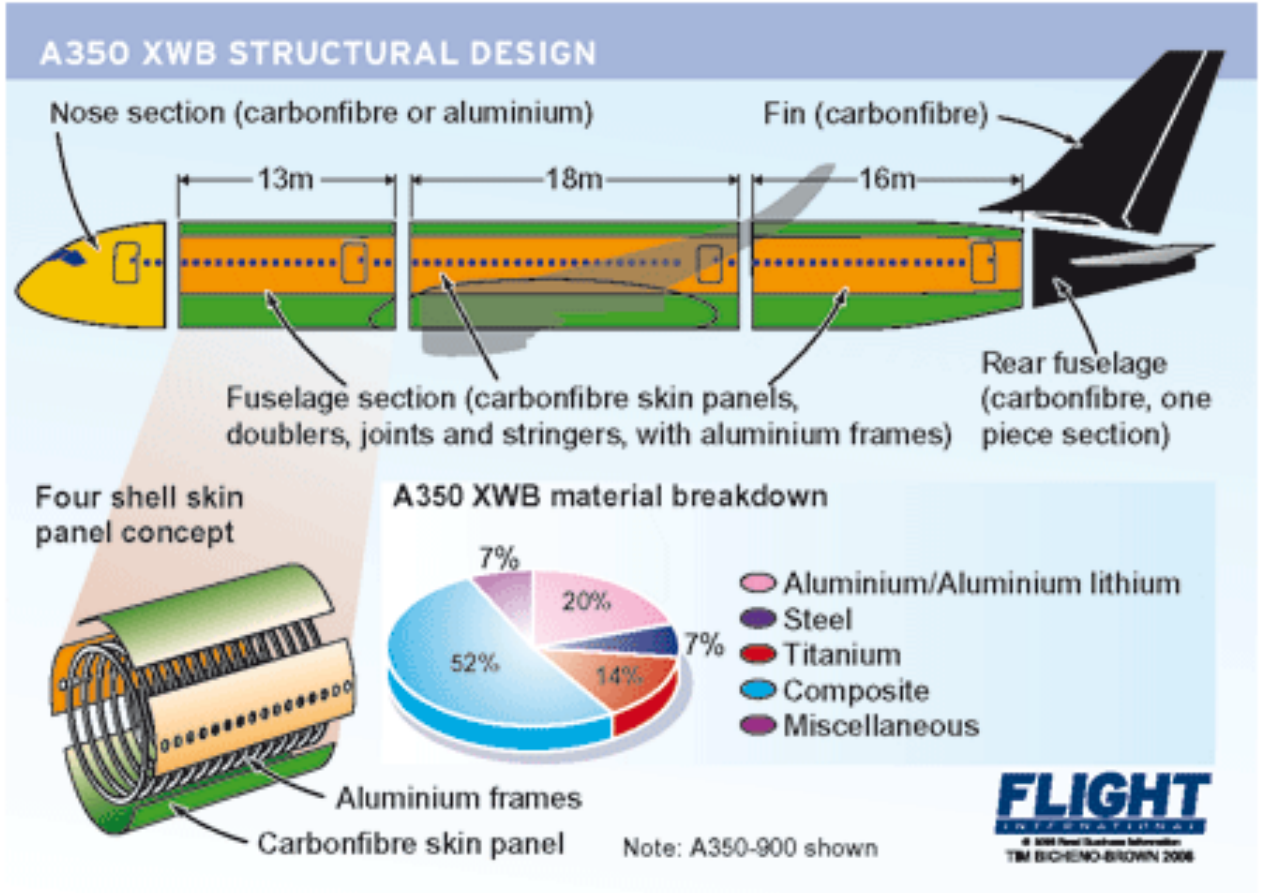
- Polyether Ketone Ketone (PEKK), (Kepstan® 7003PT from ARKEMA) having density (ρ) = 1,29 g/cm^3
- samples of 7 mg (±0.5 mg)
- heating rate of 20 K/min
- N_2 flow rate of 20 ml/min from 50°C to 900°C



TGA tests under N_2 environment: PEKK matrix

- PEKK - thermally stable up to 450-500°C
- Suitable material for high-temperature aeronautical applications.

➢ Examples of TP based composites parts for AIRBUS A350 [2]:



[2] https://www.flightglobal.com/airbuss-a350-vision-takes-shape-flight-takes-an-in-depth-look-at-the-new-twinjet/71030.article

Experimental Procedure

Developed standard tests by Federal Aviation Administration (FAA):

1) AC 20-135: Flux 106 kW/m^2 (engine)
2) AC 20-107B: Flux 182 kW/m^2 (wings, structure)

} Gas temperature 1100 $\pm$93°C

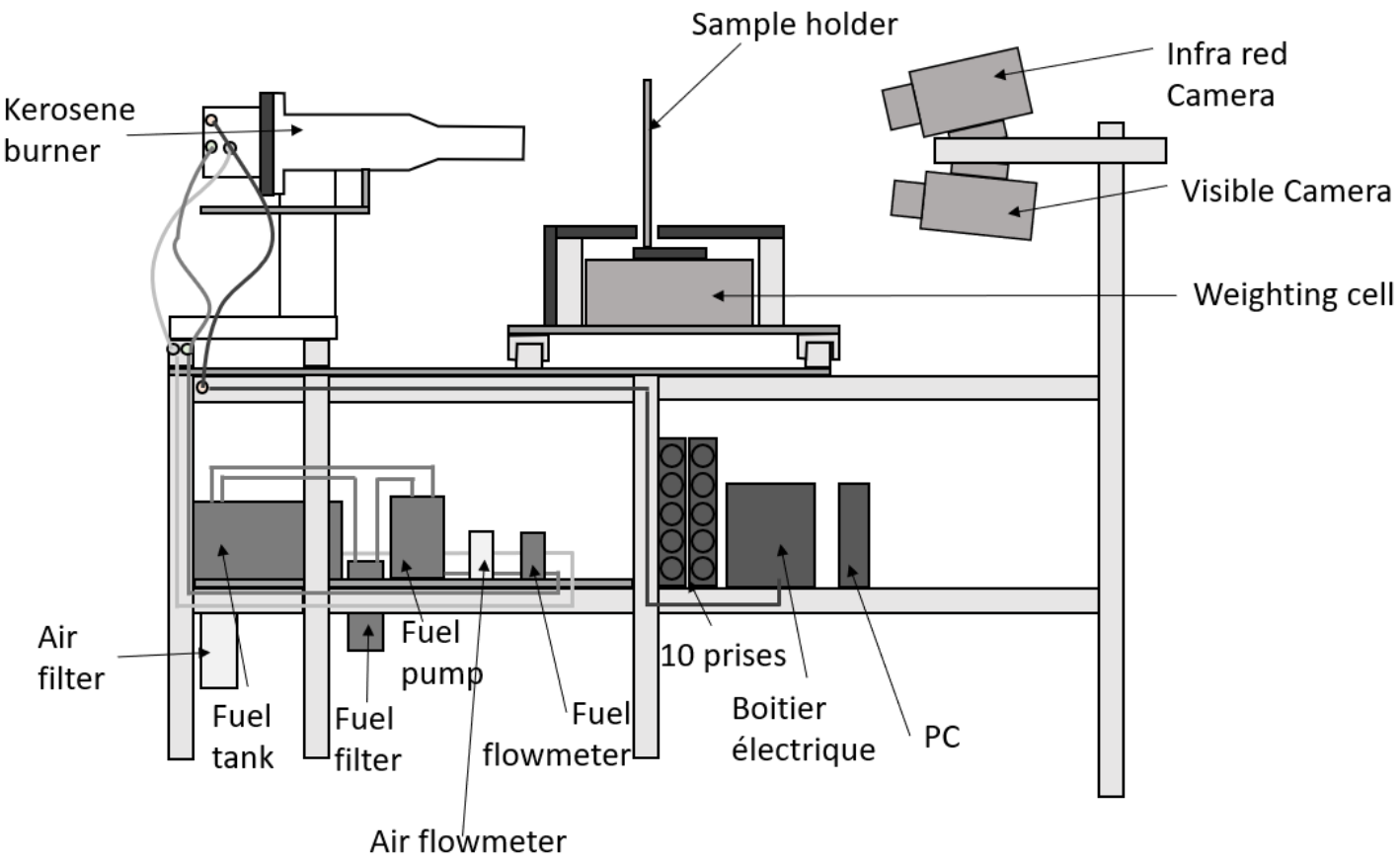
Small scale testing using cone calorimeter

- Sample size about 10 cm x 10 cm
- Heat flux in range of 30 – 150 kW/m^2
- Exposure time – 1 to 5 minutes

Large scale testing using NexGen burner

- Kerosene air diffusion flame
- Heat flux of 116 $\pm$10 kW/m^2
- Mean flame temperature of 1100 $\pm$93 °C
- Sample must withstand 15 min exposure

Experimental setup in present study



Scheme of the test bench

Tests conditions:

- Heat flux at stagnation point: 120 kW/m^2
- Temperature at exit of flame tube: 1100 °C

Heat flux on the sample:

- Measured with a fluxmeter (Gardon gauge type by Captec)

Diagnostics during tests:

- Mass loss rate
- Thermocouples
- Video camera
- Infra red camera (back side)

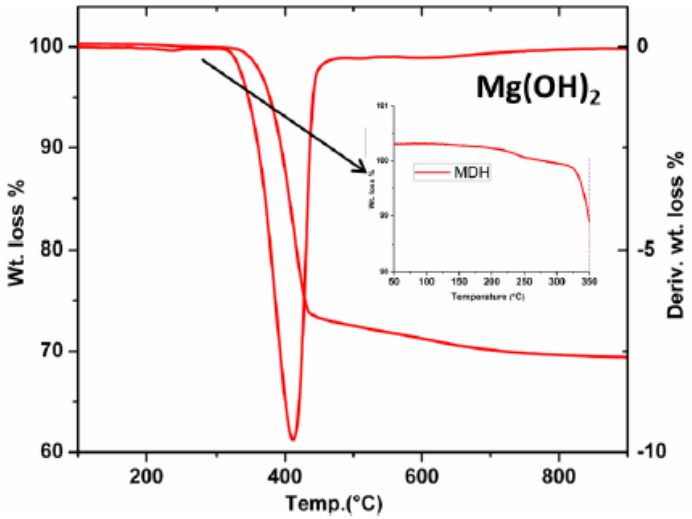
Composites specimen

- C/PEKK with orthotropic and quasi-isotropic fiber orientation
- C/PEKK with or without phosphorus based flame retardant additive (Exolit) (20 wt. % of PEKK)

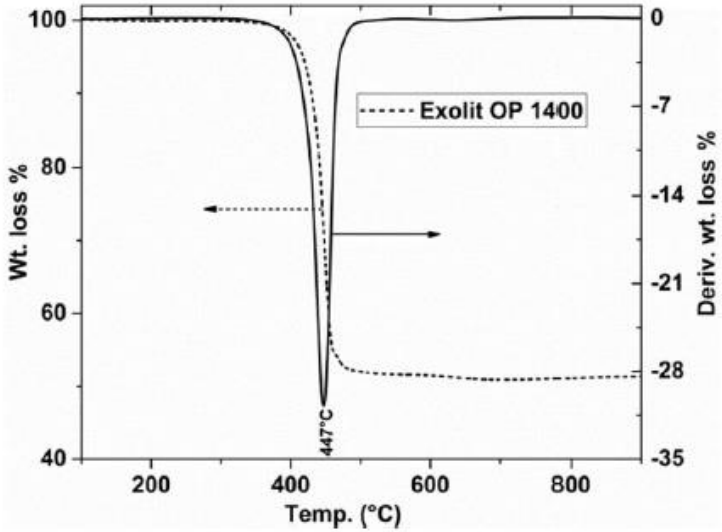
Choice of flame retardant additive

Magnesium Hydroxide ($Mg(OH)_2$) vs Exolit® OP 1400

- Exolit OP-1400 offers higher thermal stability compared to $Mg(OH)_2$



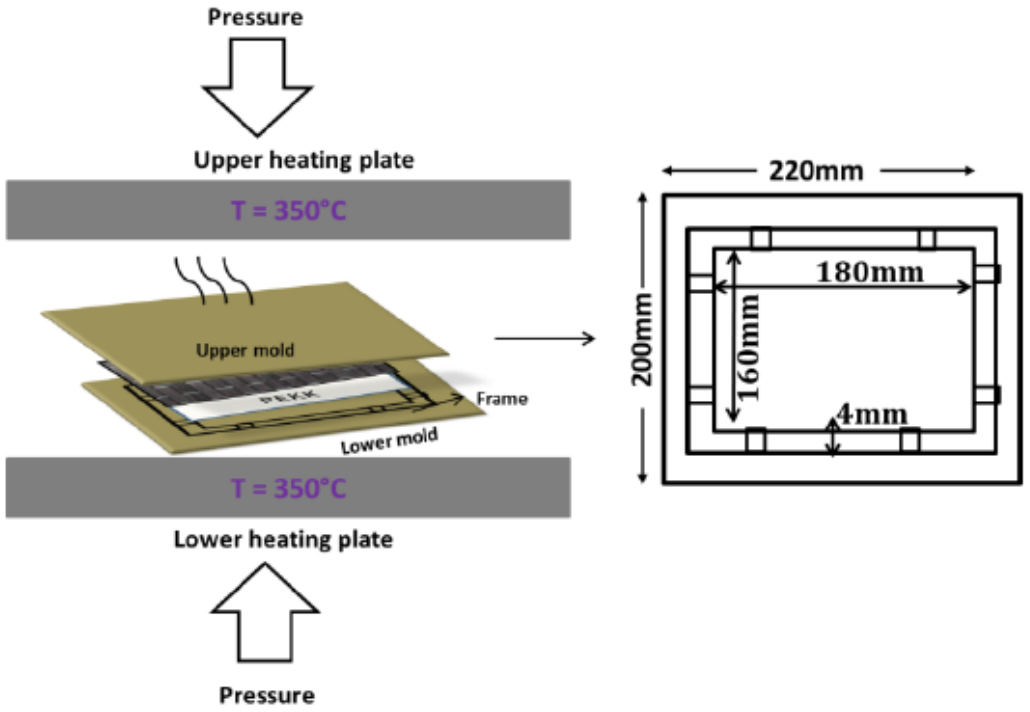
TGA curve of $Mg(OH)_2$ sample under N_2 environment



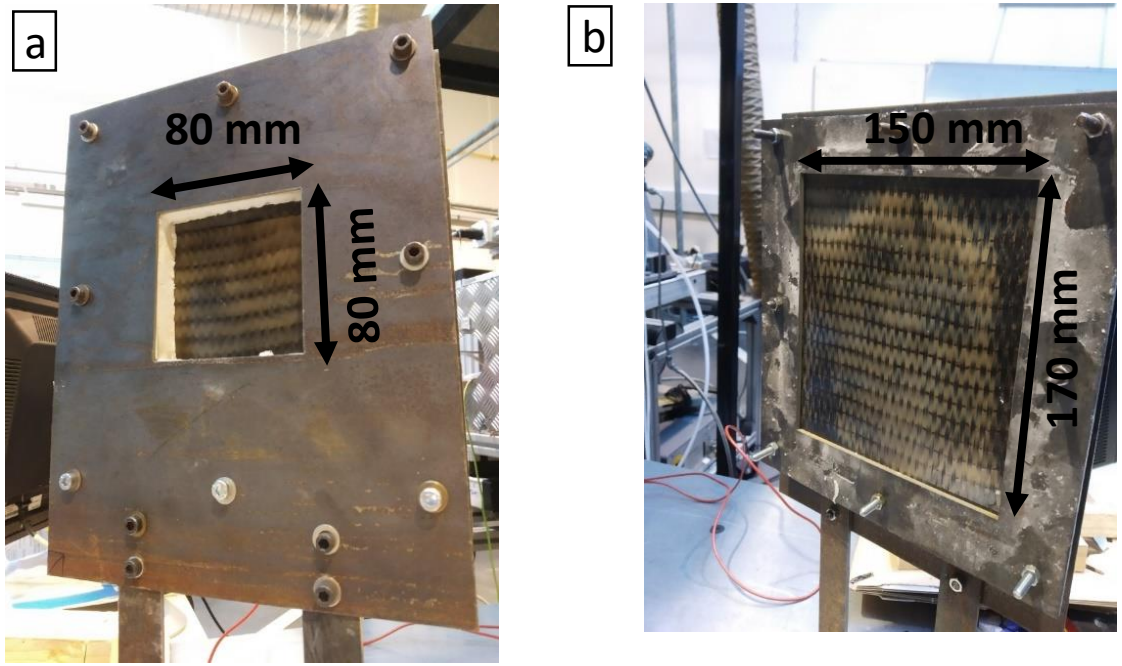
TGA curve of Exolit OP-1400 sample under N_2 environment

Composites sample fabrication

- Carbon fibres - dried at 80°C for 24 hours in an oven
- PEKK powder - dried at 120°C under vacuum during 16-18 hr
- Flame retardant additive (20 wt. % of PEKK)
- Samples – molded at 350°C, pressure applied 68 bar



Thermo compression moulding technique used for fabrication of composite in this work.



Pictures of the composite sample in the holder before the test. a: front side, b : backside.

Pictures of the composite sample in the holder after a test. a: front side, b : backside.

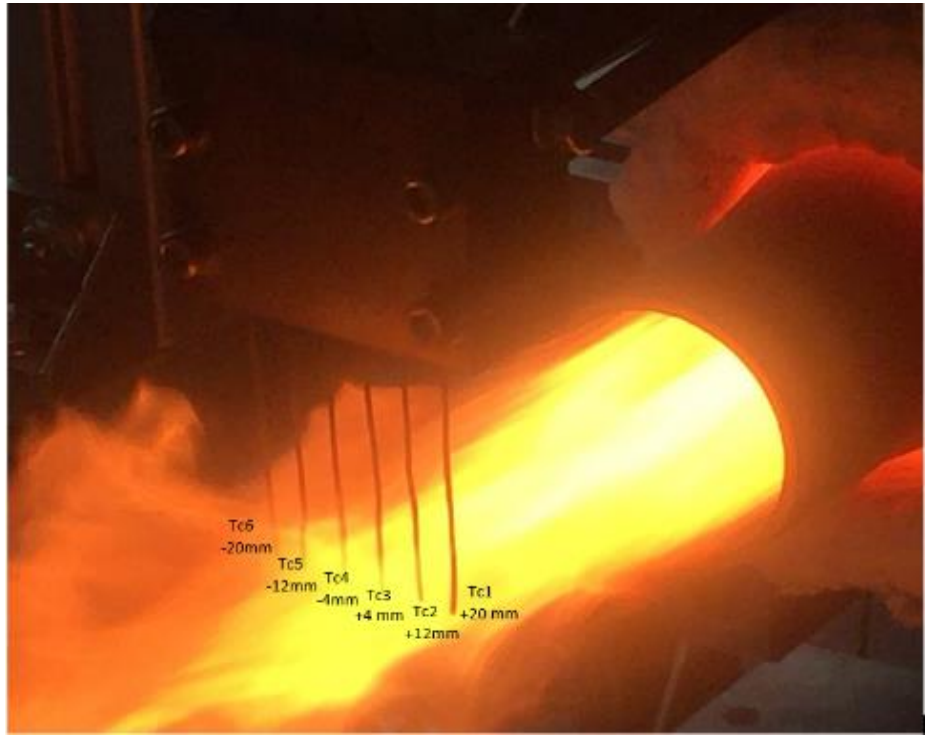
Nomenclature

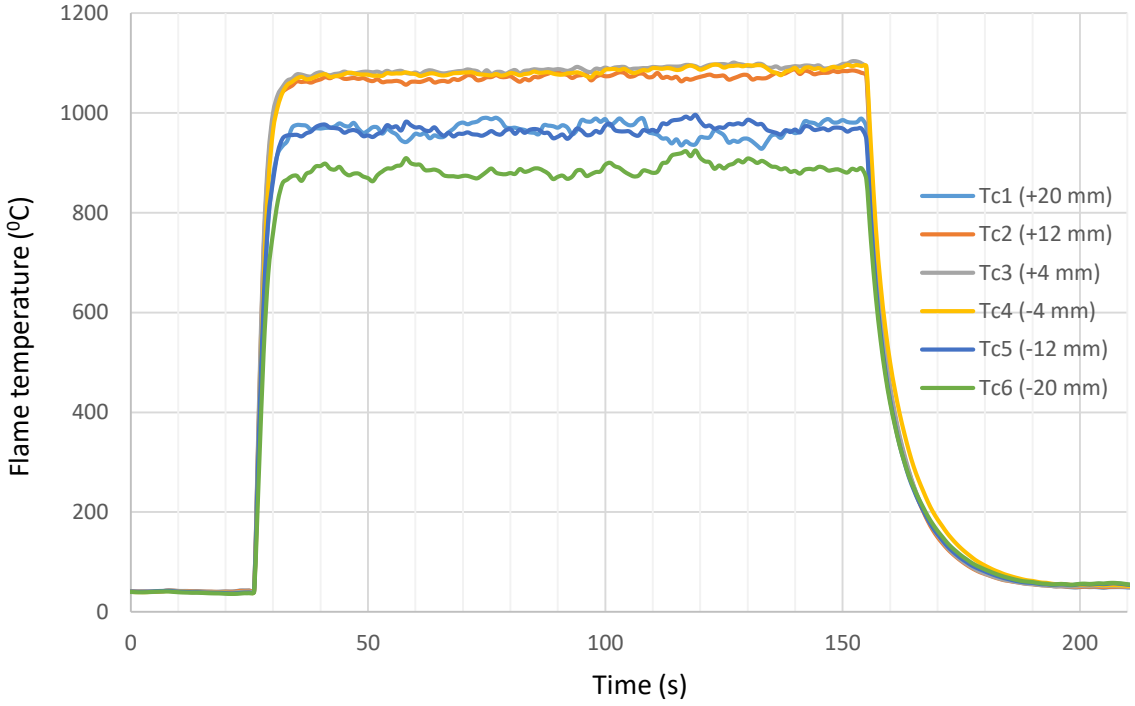
- Orthotropic without exolit (referred as PUD)
- Orthotropic with exolit (referred as PUDE)
- Quasi-isotropic without exolit (referred as PQI)
- Quasi-isotropic with exolit (referred as PQIE)

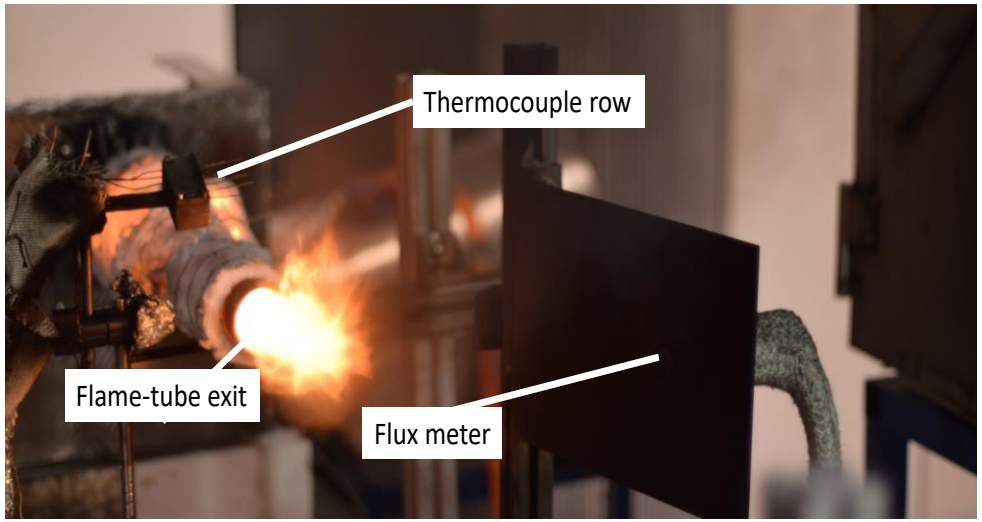
- In Quasi-isotropic, carbon fibers are stacked in sequence of [0,+45,-45,90]

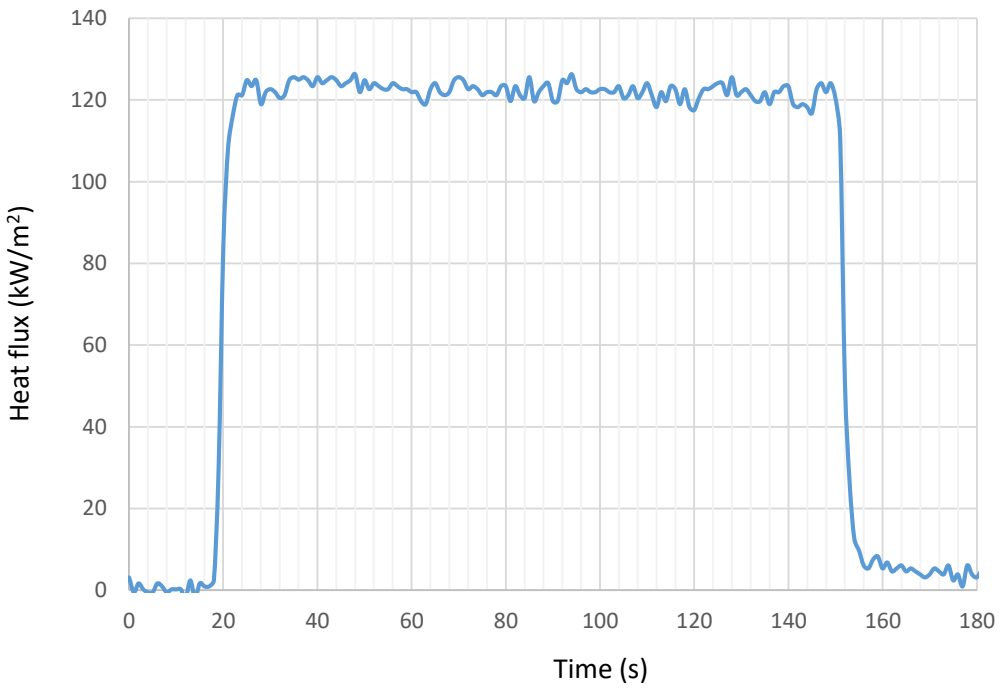
Test conditions

Flame exposure time 300 seconds at 120 kW/m²; 1100 °C





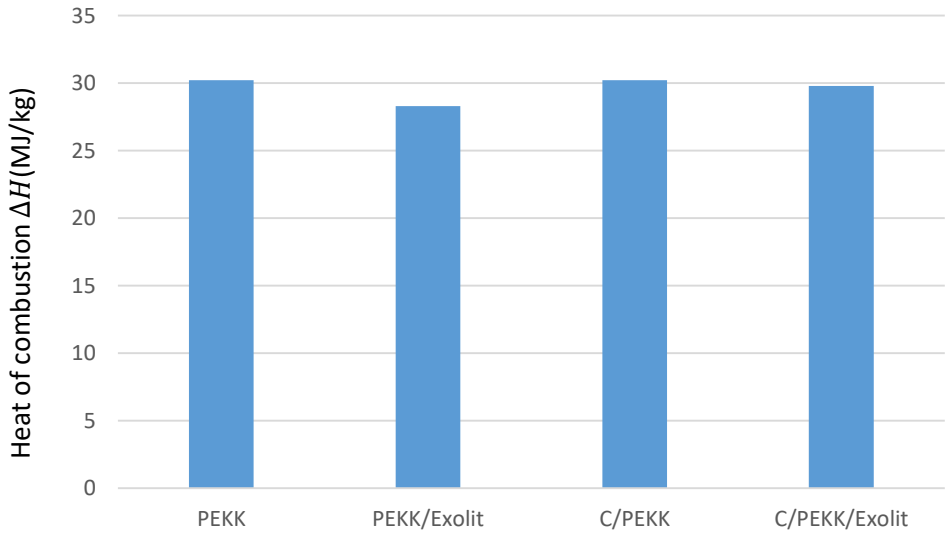

Thermocouple row
Flame-tube exit
Flux meter


Heat flux (kW/m²)
Time (s)
0
20
40
60
80
100
120
140
160
180

Material	Mass (g)	ΔH (MJ/kg)
PEKK/Exolit	0,7048	28,290
PEKK	0,7015	30,202
C/PEKK/Exolit	0,6438	29,800
C/PEKK	0,6876	30,230

Measurement of heat of combustion by bomb calorimeter

Material	PEKK (% mass)	Carbon (% mass)	Exolit (% mass)
PEKK	100	0	0
C/PEKK	50	50	0
PEKK/Exolit	80	0	20
C/PEKK/Exolit	40	50	10



- No significant effect of Exolit addition on heat of combustion

Experimental details

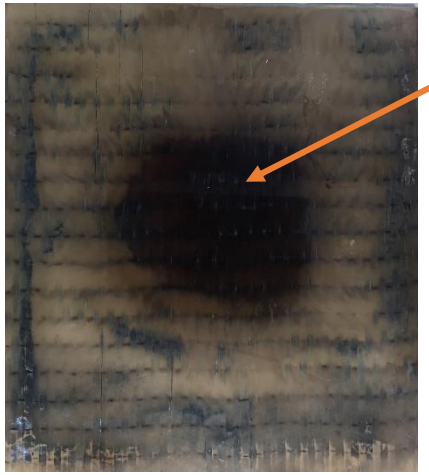
Specimen	Initial mass (g)	Final mass (g)	Sample initial mass (g)	Sample final mass (g)	Mass loss, sample (g)	Mass loss (%)
PUDK-300s_1	1870.54	1868.06	121.80	119.46	2.34	1.92
PUDEK-300s_1	1880.07	1877.21	132.35	129.50	2.85	2.16
PQIK-300s_1	1868.92	1866.71	121.36	118.83	2.53	2.09
PQIEK-300s_1	1884.26	1881.84	136.44	133.82	2.62	1.94
PQIK-300s_2	1877.12	1874.72	128.48	126.23	2.25	1.75
PQIK-300s_3	1873.00	1870.25	131.26	128.58	2.68	2.04

C/PEKK with or without phosphorus based flame retardant additive (Exolit)

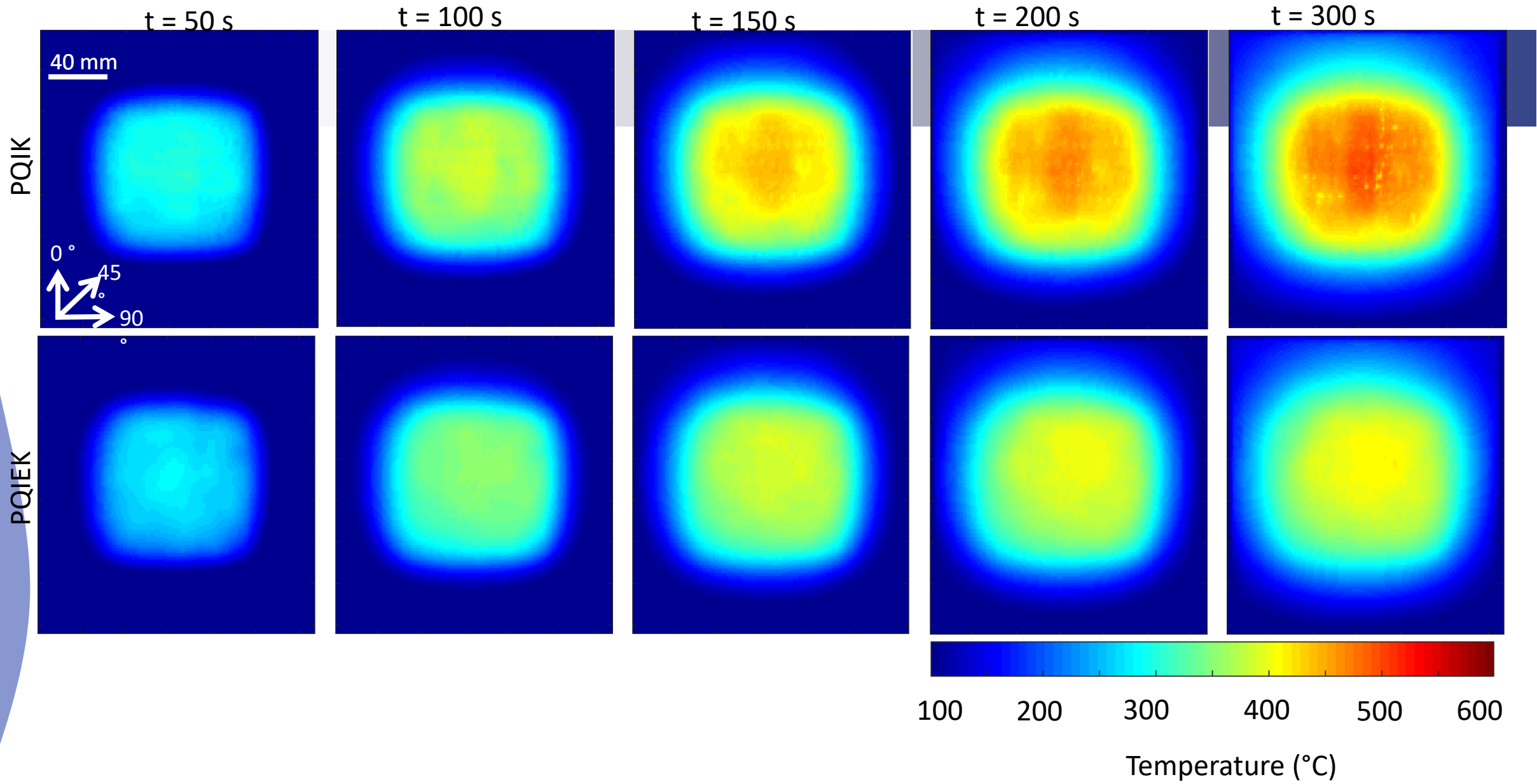
PQIK | **PQIEK**

Front face

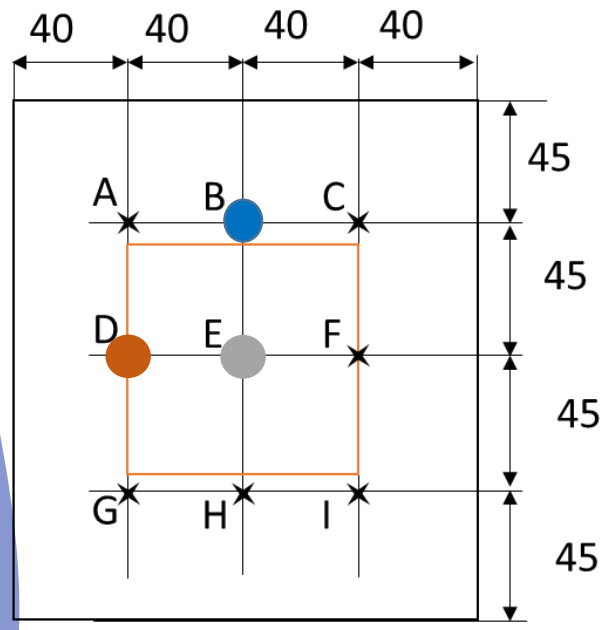
Back face

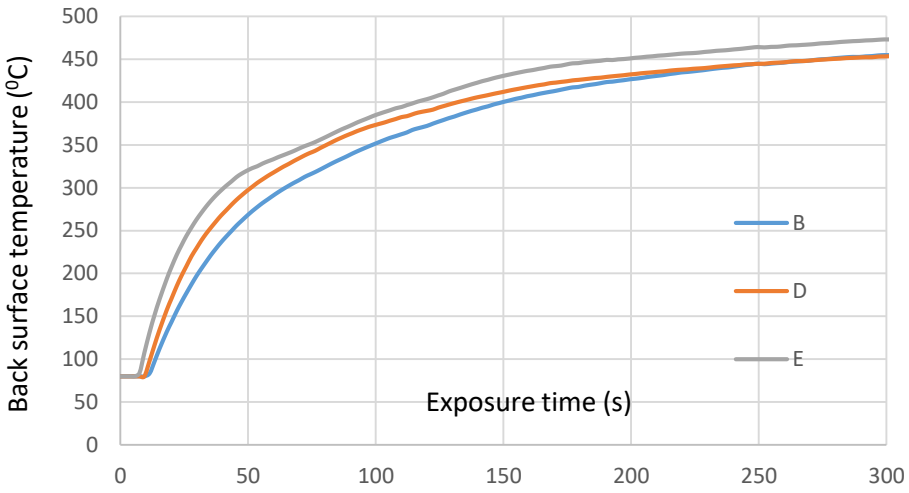


Pictures of the samples PQIK and PQIEK *after a 300s fire exposure.*

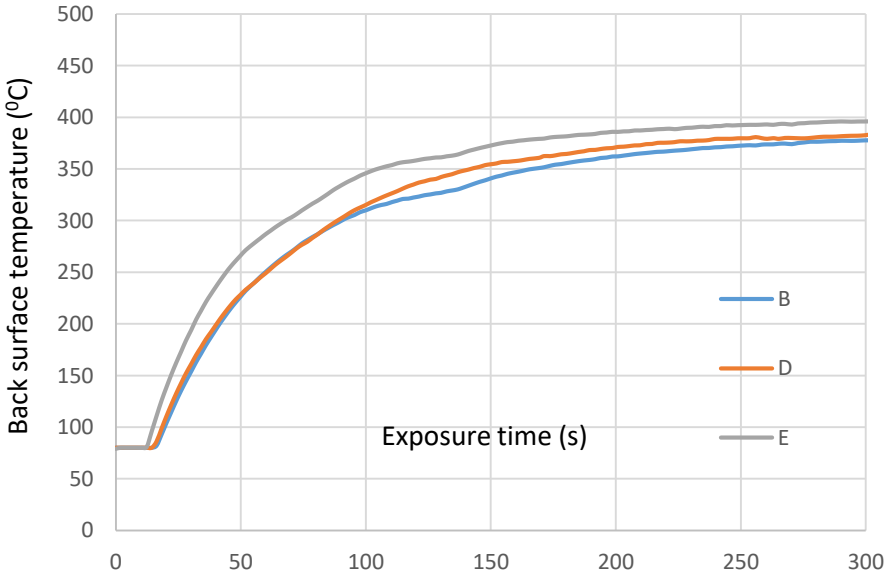


Back surface temperature during fire exposure for PQIEK and PQIK test.

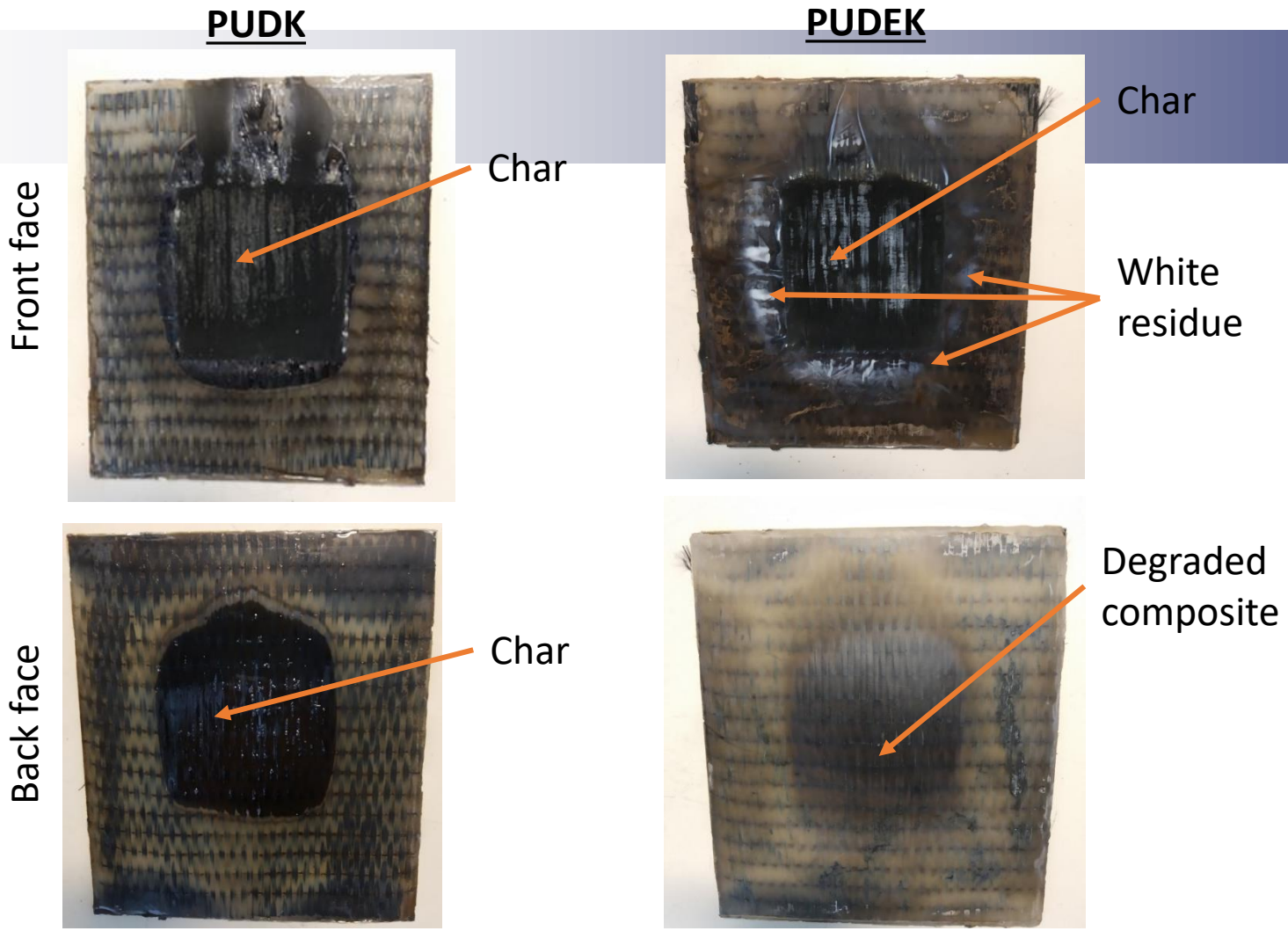




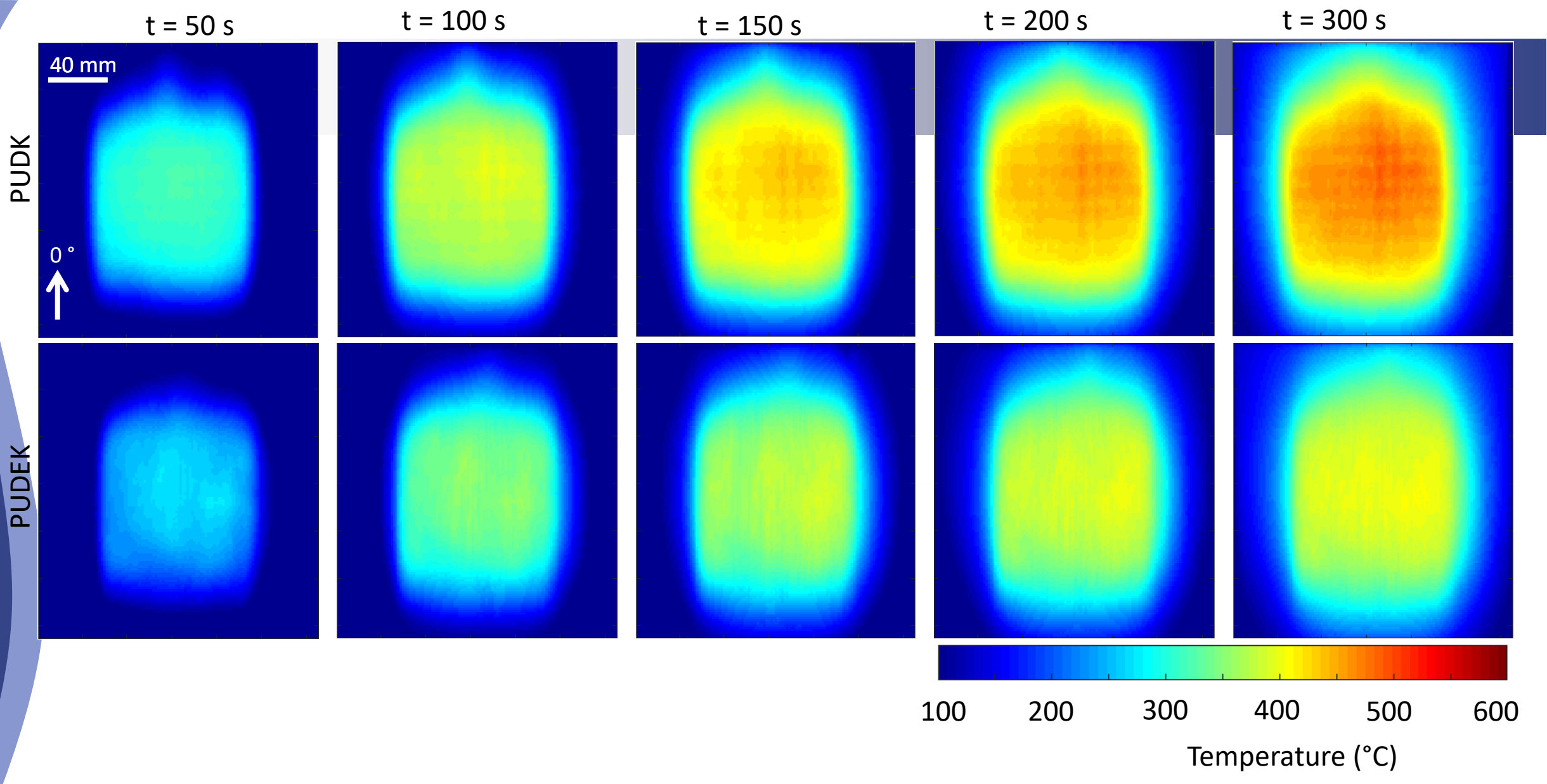
Back surface temperature during fire exposure for PQIK



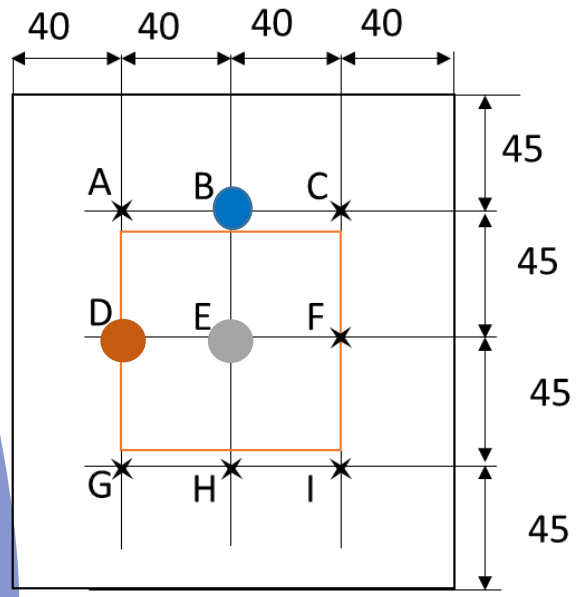
Back surface temperature during fire exposure for PQIEK

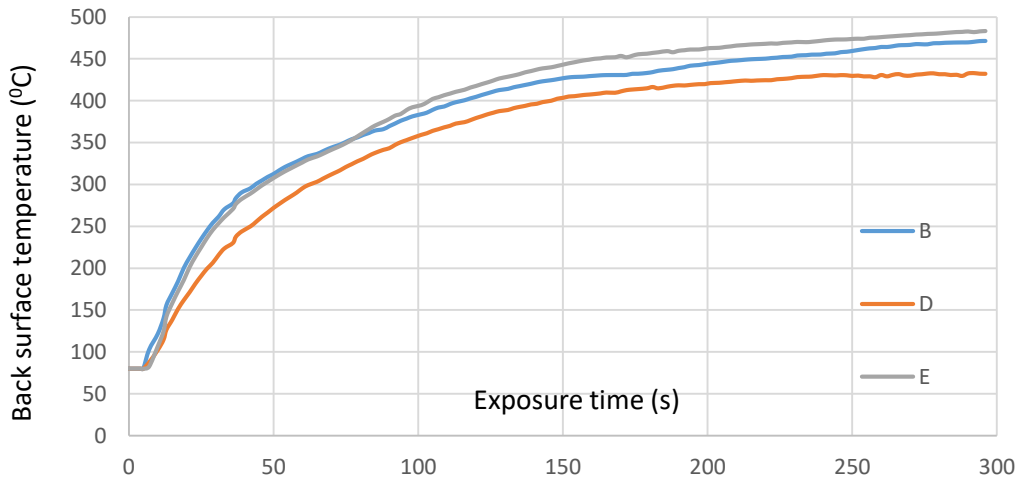


Pictures of the samples PUDK and PUDEK *after a 300s fire expusure.*

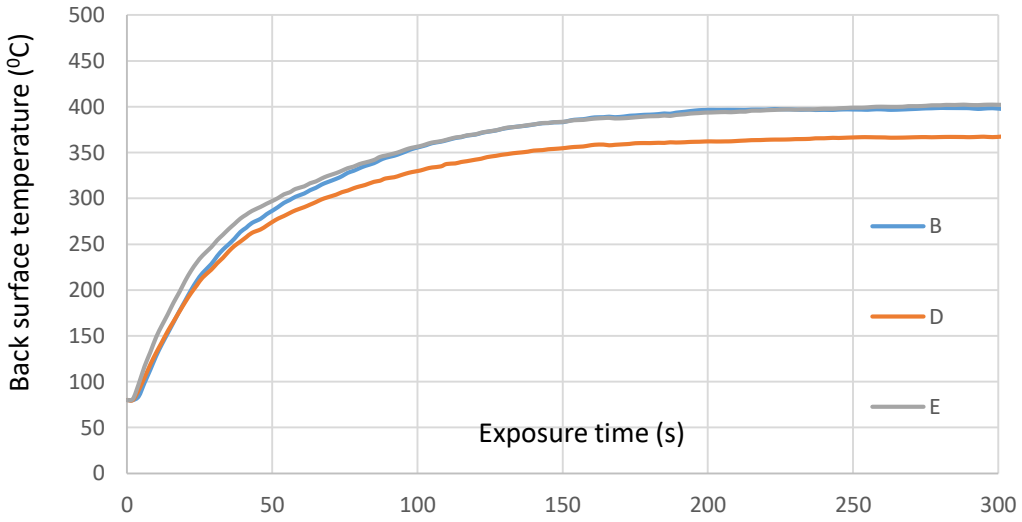


Back surface temperature during fire exposure for PUDEK and PUDK test.

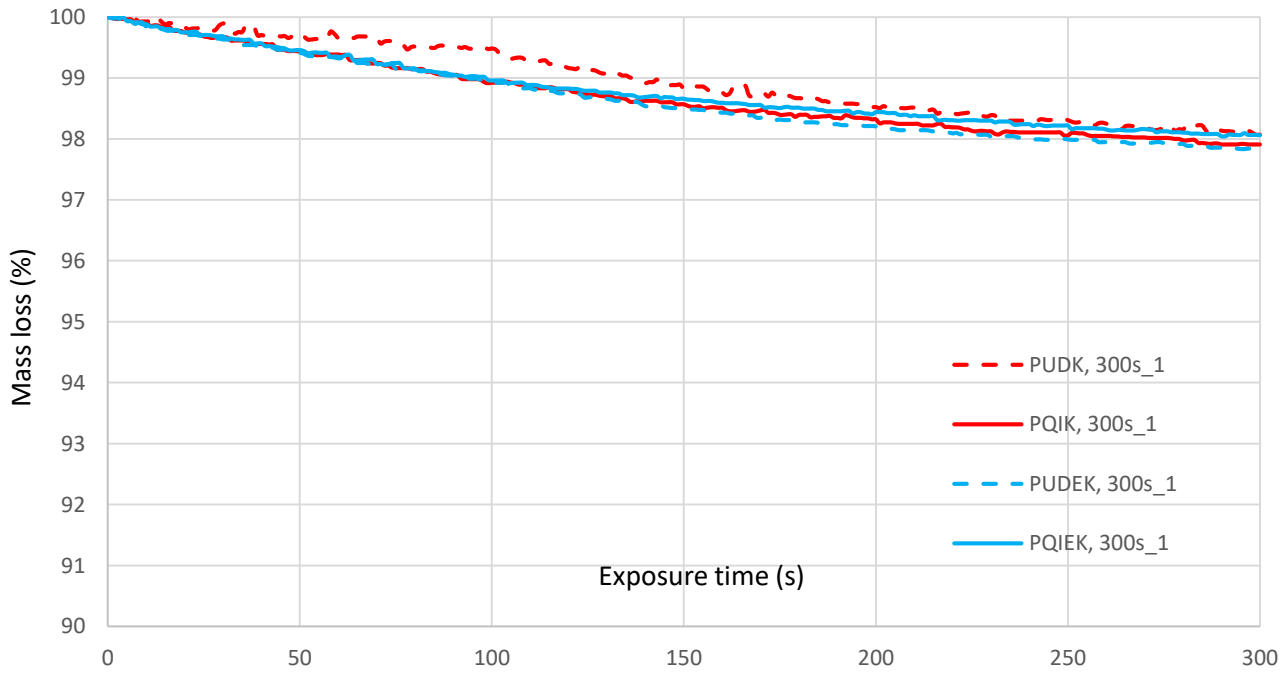




Back surface temperature during fire exposure for PUDK



Back surface temperature during fire exposure for PUDEK

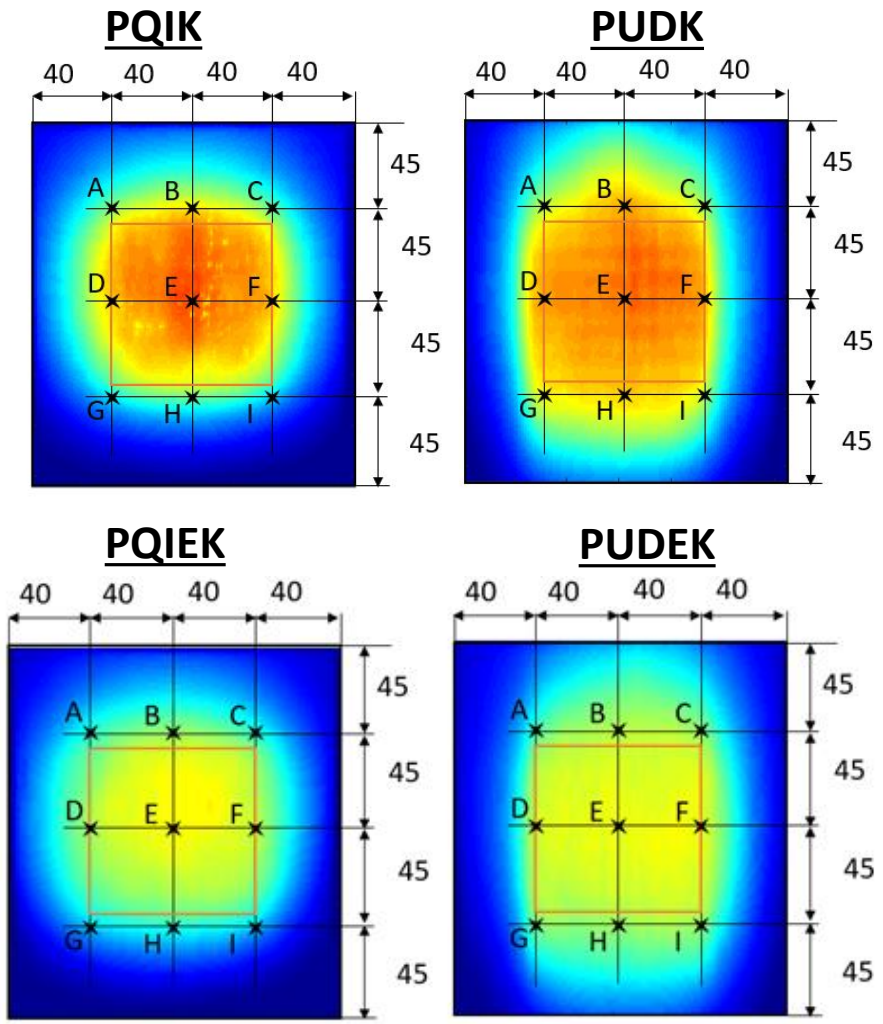


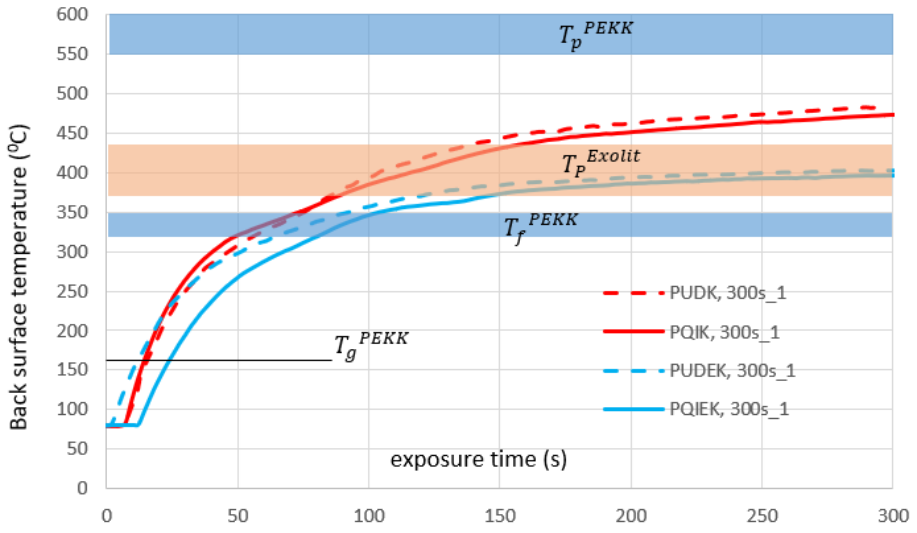
Comments

- No significant different in mass loss for C/PEKK with and without exolit
- Exolit serves the purpose of protecting resin matrix but degrades itself.

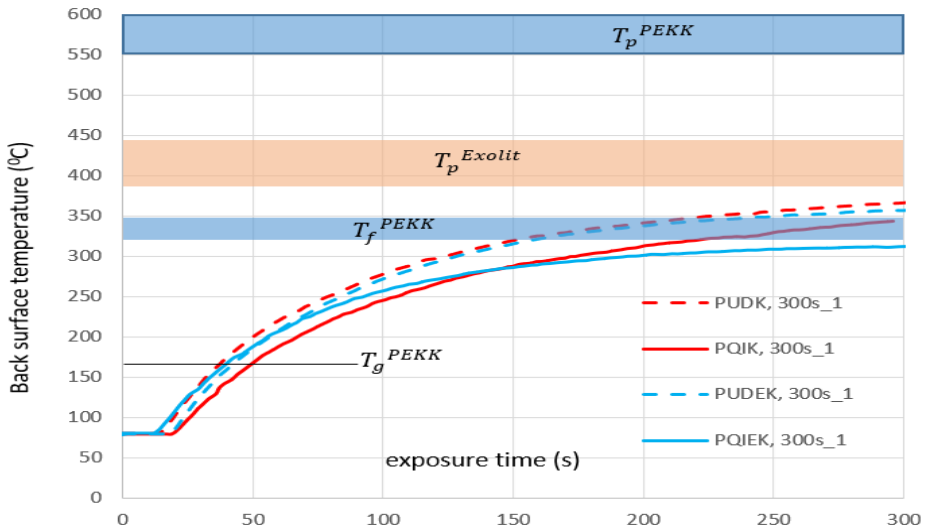
Without exolit

With exolit





Back surface temperature during fire exposure at the centre of the sample

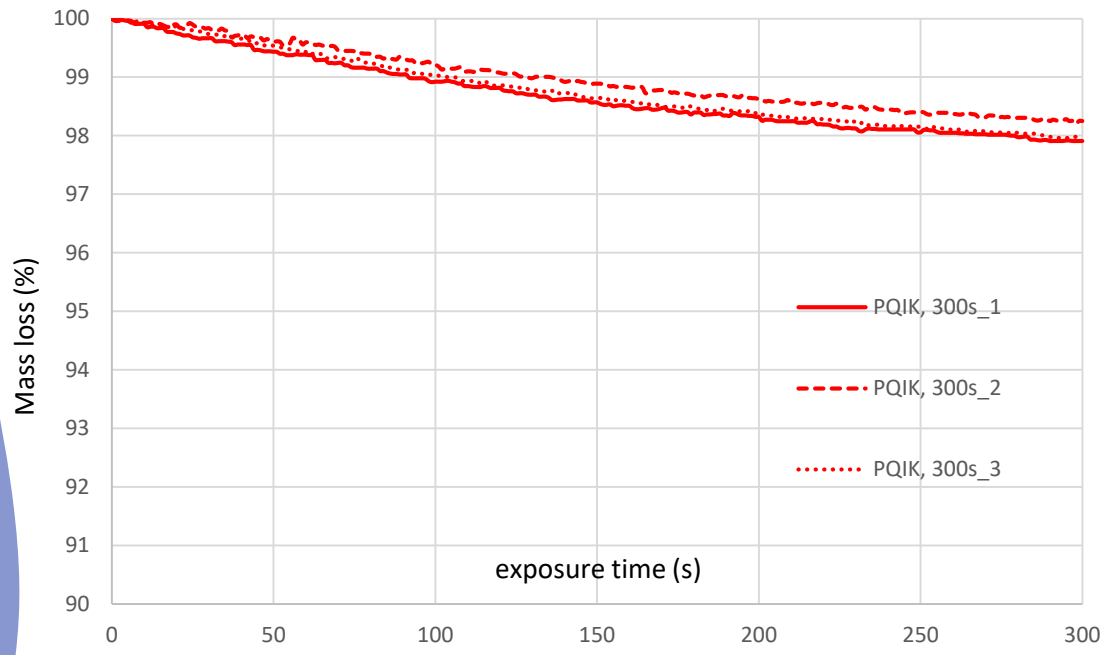


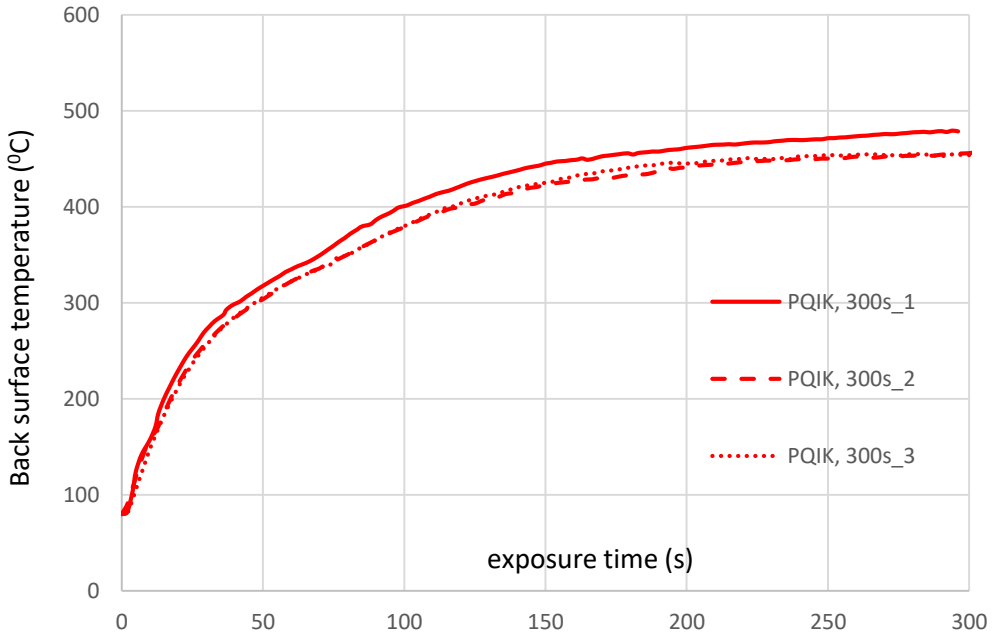
Back surface temperature during fire exposure at the bottom of the sample.

Comments

- Stacking sequence makes no difference close to the center zone
- Outside center zone, orthotropic sequence favors more heat conduction

Repeatability tests of PQIK





Conclusions

- Less thermal degradation for C/PEKK with exolit.
- Exolit seems to degrade prior to resin but also protects it.
- Different shapes of thermal stress for UD and QI.
- No effect of fibers stacking sequence on thermal behavior close to center zone.
- Outside center zone, UD (orthotropic stacking sequence) favors more heat conduction.
- Good repeatability for quasi-isotropic composite tests.
- Detailed study on effect of fibers stacking sequence on mechanical properties will be carried out in future work.

QUERIES ?