



CURE MONITORING OF HIGH-TEMPERATURE RESINS FOR ENHANCING THE MANUFACTURING OF ADVANCED COMPOSITES

Dr N. Pantelelis, Synthesites SNC, Belgium

W. Gerrits, R. Klomp-de Boer, NLR, The Netherlands

A. Johnston, S. Wilson, A. McKibbin, Bombardier Aerospace, UK

C. Brauner, F. Schadt, L. Amirova, M. Grob, FHNW, Switzerland

Companies



R&D Centres and Universities



University of Applied Sciences and Arts
Northwestern Switzerland



On-going

Recotrans: (2017-2020) Multimaterial recyclable manufacturing for the transportation industry
RTM and pultrusion, Glass fibre, reactive thermoplastic for automotive and rail applications

Partners: Aimplas (CO), Fraunhofer, Daimler, Far UK, Stadler, INEA, Istanbul University, Arkema

SuCoHS: (2018-2020) Sustainable and Cost Efficient High Performance Composite Structures
demanding Temperature and Fire Resistance

Partners: DLR (CO), Bombardier, Aernnova, NLR, ONERA, Apodius, Rockwell Collins

Completed

Ecomise: First-time right composites manufacturing (2013-2016)

Partners: DLR (CO), Faser I., Bombardier, Hutchinson, Airborne, Loop, Dassault Systemes, NLR
RTM and RTI, Glass and carbon fibre, epoxy

Coaline: Injection pultrusion with microwave curing and injection of coatings (2013-2017)

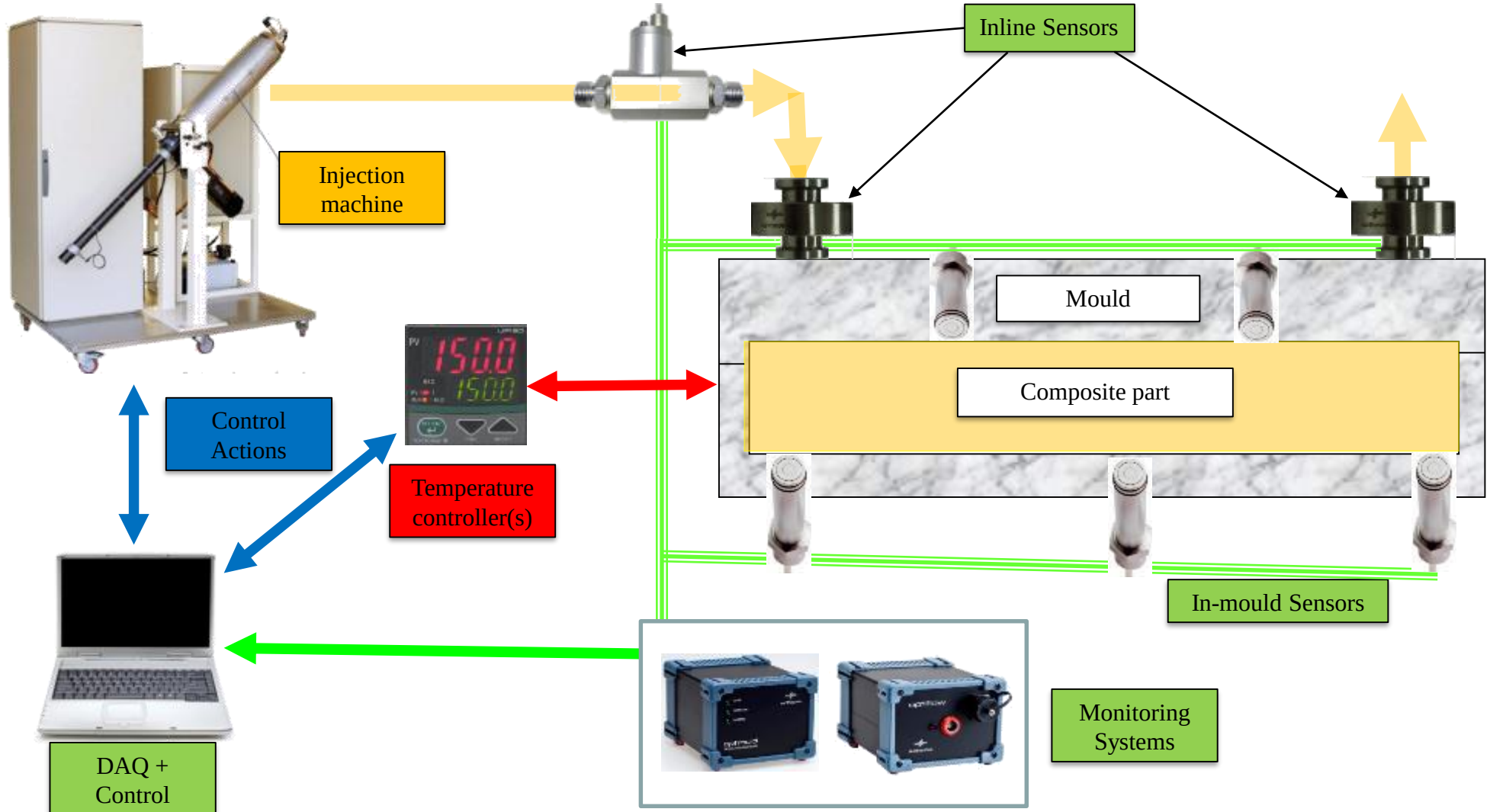
Fraunhofer ICT, Aimplas, Resoltech, Rescoll, Acciona

iREMO: intelligent Reactive Moulding (2009-2012)

RTM, Light RTM and Infusion, Glass and carbon fibre, epoxy and polyester

MAC-RTM: Microwave curing (2011-2013), Fraunhofer ICT and Aimplas

- **Check resin quality and adjust process accordingly**
- **Detect accurately resin arrival at critical locations**
 - **Open/close valves based on sensors' feedback**
- **Monitor viscosity changes and decide when start heating**
- **Identify minimum viscosity and decide about pressure**
- **Detect unexpected events and follow alternative routes**
- **Improve simulation accuracy and design intelligent strategies**
- **Real-time decision of the cure cycle based on T_g and degree of cure (depends on the resin) rather than time**



- 4 temperature and resin arrival sensors
- Resistance-based measurements and RTD temperature
- Continuous connection checking
- One relay output for process automation



In-mould
Durable



- flat areas
- possible mark

Gate
sensor



- ideal for vacuum infusion in oven/ autoclave (gates, pipelines, pots etc.)

Flexible
disposable



- Curved surfaces
- In the laminate for development
- Over the peel-ply
- Suitable for very long parts
- no extra protection for Carbon Fibre Preforms

FloWire
sensors



New
Curved
Durable



Real-time measuring of

- Resin's electrical resistance (from 0.1 MOhm up to 50 TOhm)
- temperature (pt100 sensor with 0.1°C accuracy)

Input of external signals e.g. pressure sensors

process monitoring sensor = electrical resistance + RTD sensors

Durable
sensor



High Temp RTM

- Resin arrival
- Viscosity rise
- Gelation
- End-of-cure

Flexible
sensor



VI and RT cure

- Resin arrival
- Viscosity rise
- Gelation
- End-of-cure

Inline sensor



- Avoid pipe cleaning
- Adjust cycle
- Mixing ratio check

Pot sensor



- Mixing ratio
- Resin Quality
- Resin aging
- Adjust cycle



New

CF In-mould
Durable

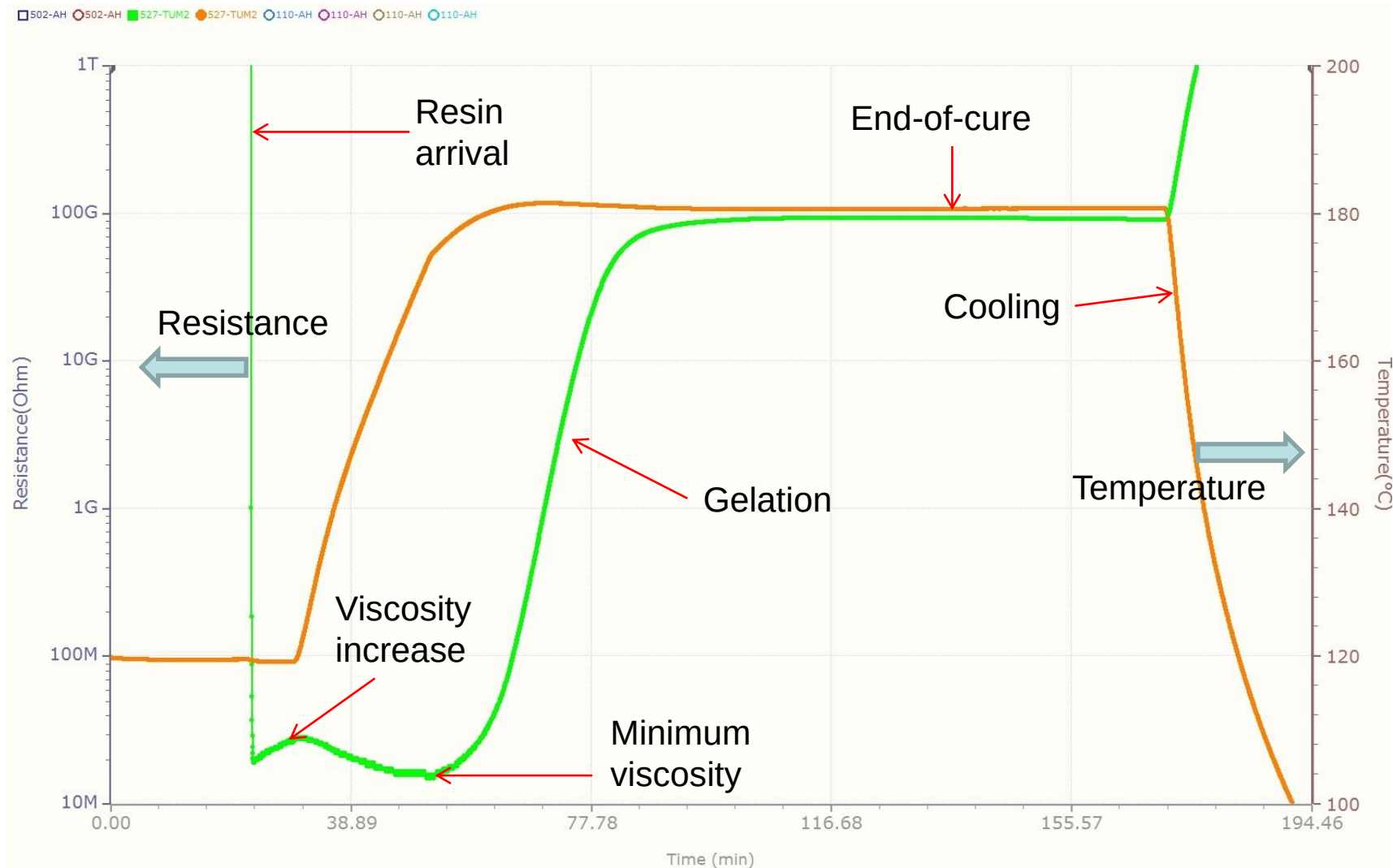


New

Vacuum Bag
Sensor



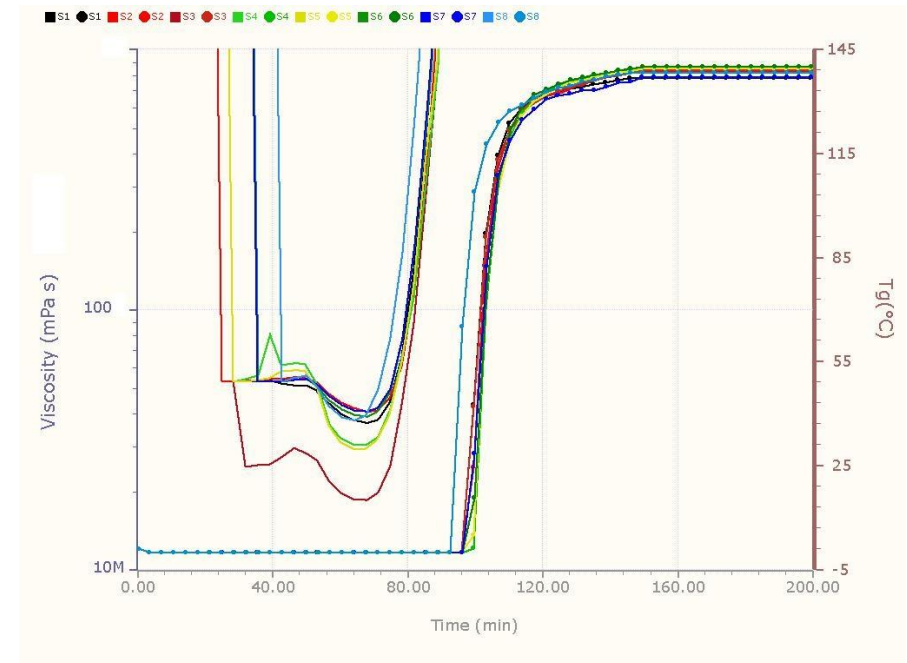
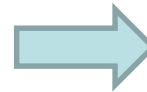
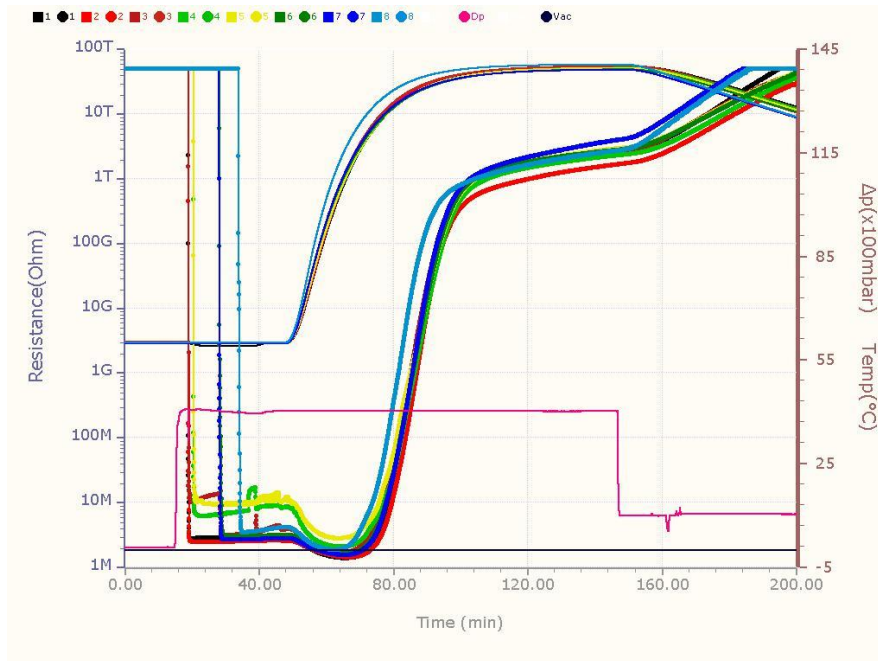
A typical RTM6 cure cycle as measured with Optimold



From Resistance and Temperature

to

Online viscosity and Tg estimation



More than 25 resins have been calibrated for the whole range of advanced composites manufacturing

Overview of the T_g estimated online with the ORS software and T_g measured right after demoulding by DSC and the difference between them for several isothermal and realistic test cases which shows that

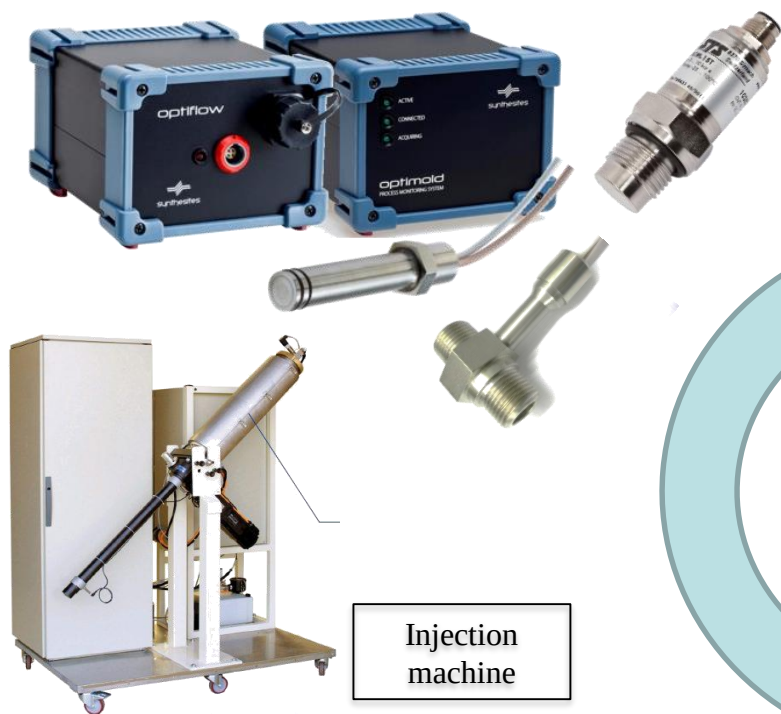
the T_g online estimation is within the DSC accuracy

Trials and DSC performed by

CARBON ROTEC
COMPOSITE TECHNOLOGY

published at SAMPE Journal,
v.53/6, Nov/Dec 2017, pp. 6-10

	Trial	Duration [h]	T _g -ORS (°C)	T _g -DSC (°C)	Difference (°C)
Isothermal	80DV1	3	73.17	73.34	-0.17
	80DV3	2.5	70.30	70.91	-0.61
	80DV4	2.5	73.45	72.49	0.96
	80-120'	1.92	66.96	66.02	0.94
	80-90'-1	1.50	62.04	61.80	0.24
	80-90'-2	1.50	65.52	65.21	0.31
	80-D2-2	1.50	61.88	60.59	1.29
	60-260'	4.33	55.02	56.51	-1.49
	70-190'	3.17	64.92	65.39	-0.47
Isothermal cases, mean difference					1.61
Isothermal cases, standard deviation					2.42
Non-isothermal	TEB1-1		61.37	59.54	1.83
	TEB1-2		69.36	70.93	-1.58
	TEB2-1		60.00	58.64	1.36
	TEB2-2		70.02	70.30	-0.28
	LESW1-1		76.97	74.35	2.62
	TESW1		71.34	69.18	2.16
	Shell1-1		80.36	78.92	1.44
	Shell1-2		75.72	77.83	-2.12
	Shell2-1		79.60	77.70	1.89
Non-isothermal cases, mean difference					2.15
Non-isothermal cases, standard deviation					1.26



Temperature controller(s)

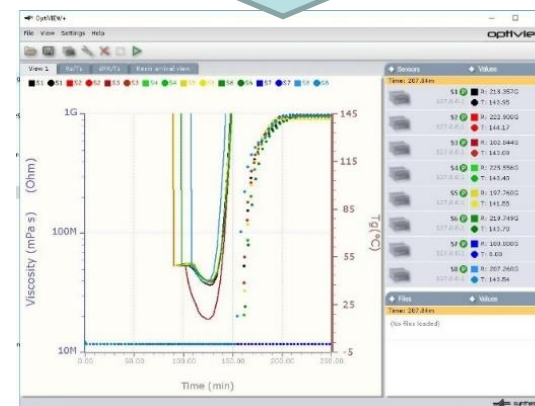
Measuring resistivity, temperature, pressure

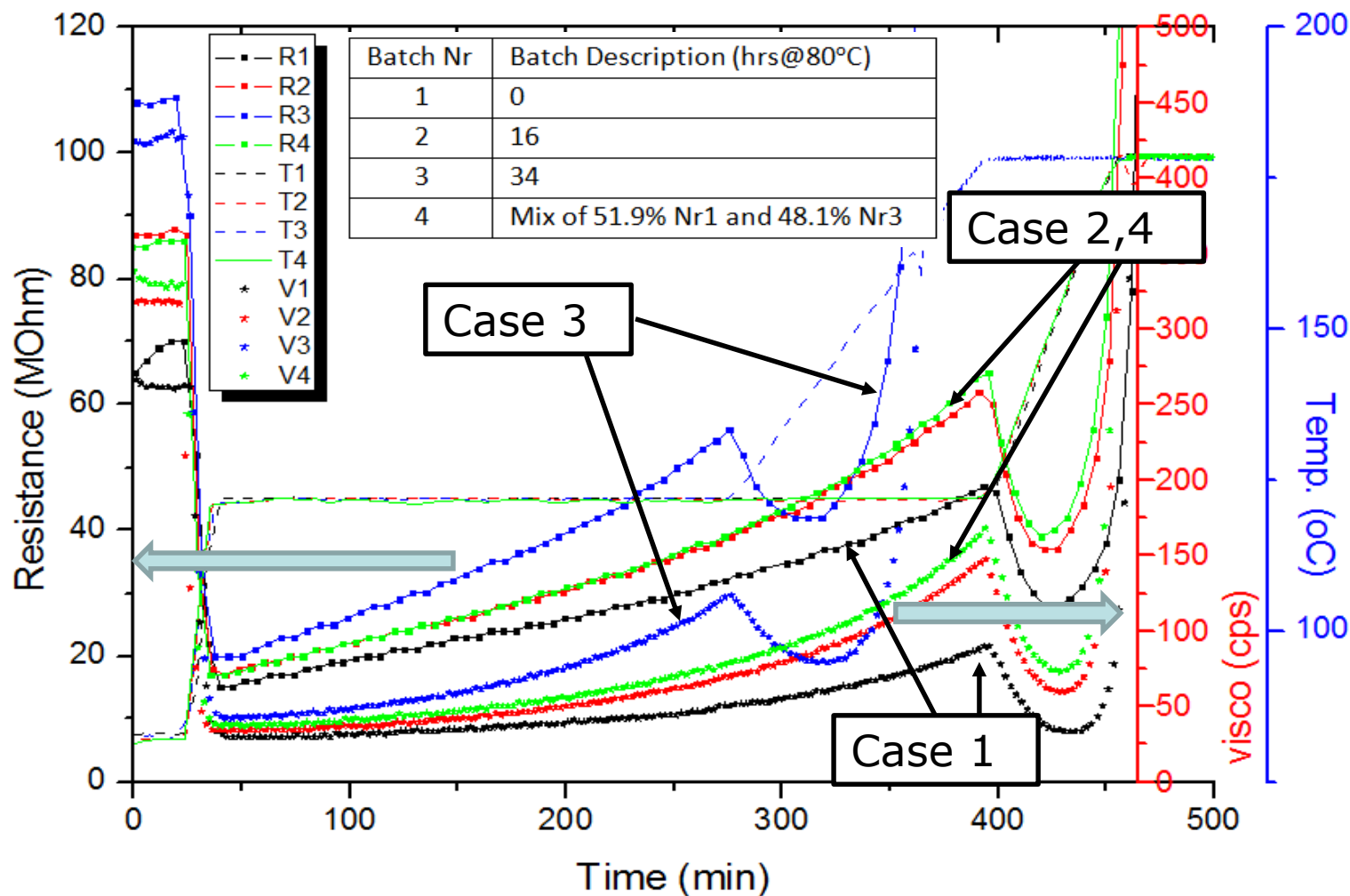
Intelligent Process Control

Controlling temperature, demoulding time etc.

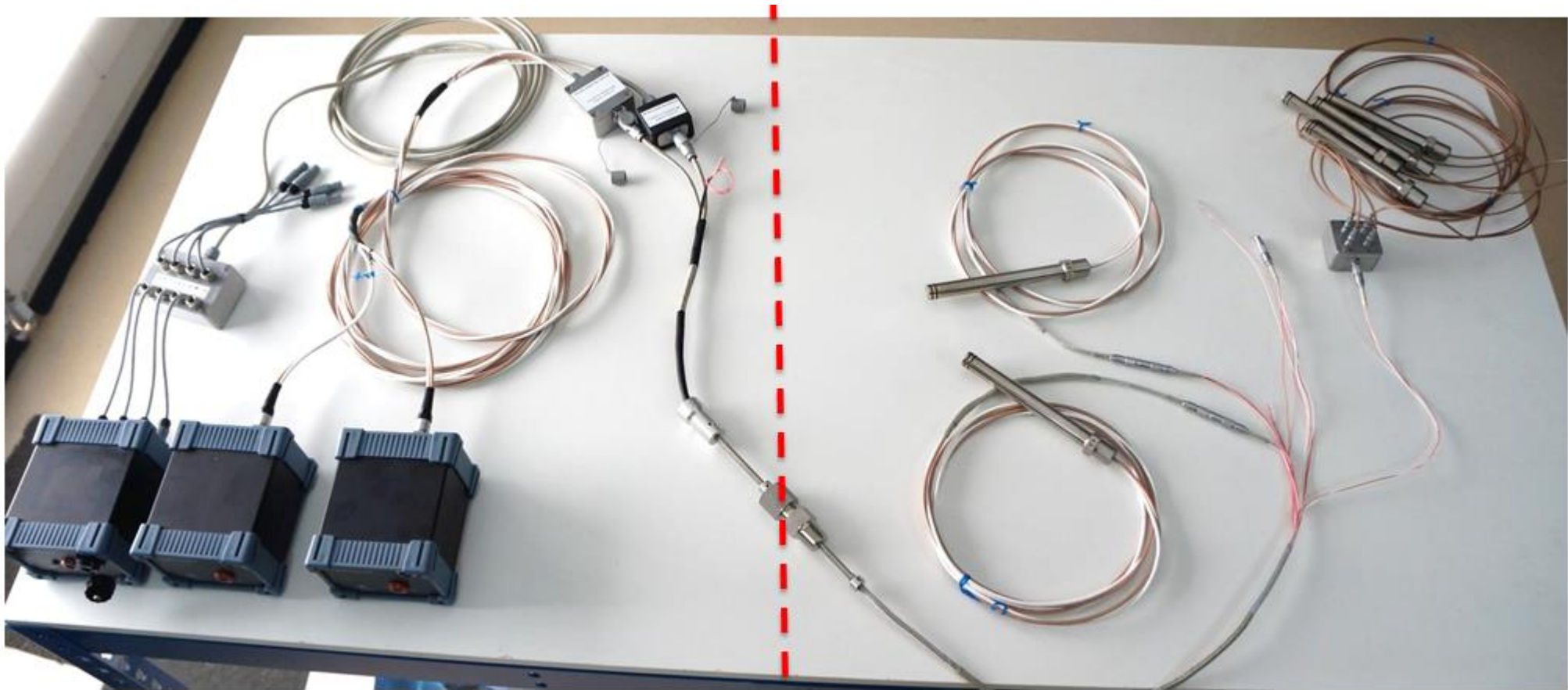


Online Estimating Tg, viscosity, degree of cure





Viscosity, Resistance and temperature vs. time for 4 resin batches of Cycom 890



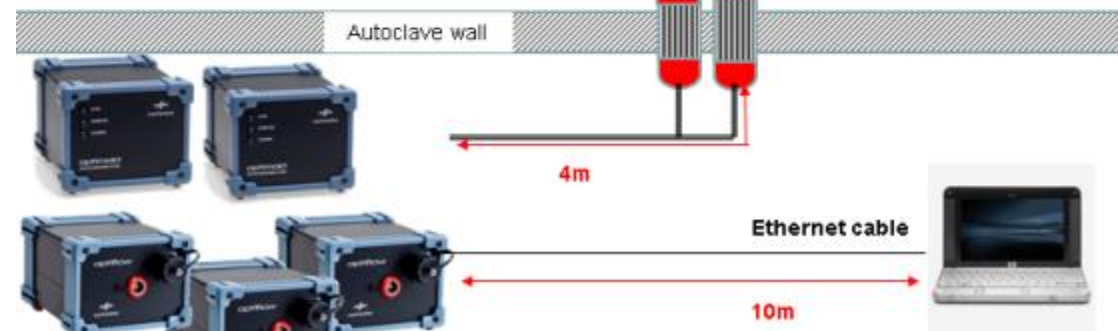
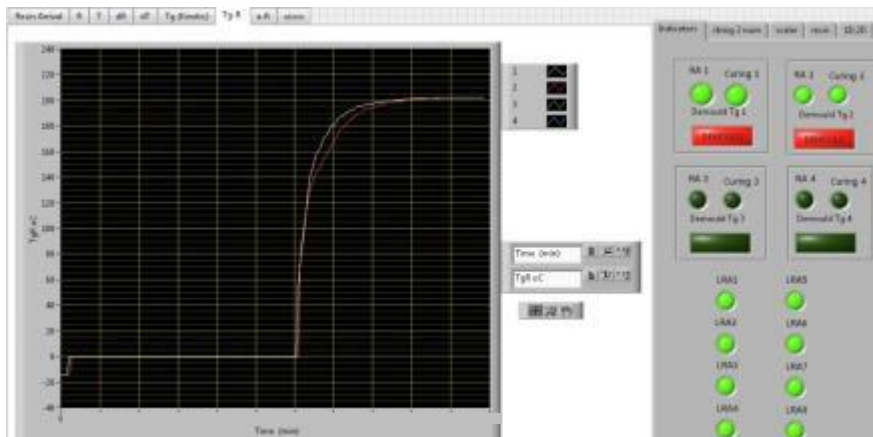
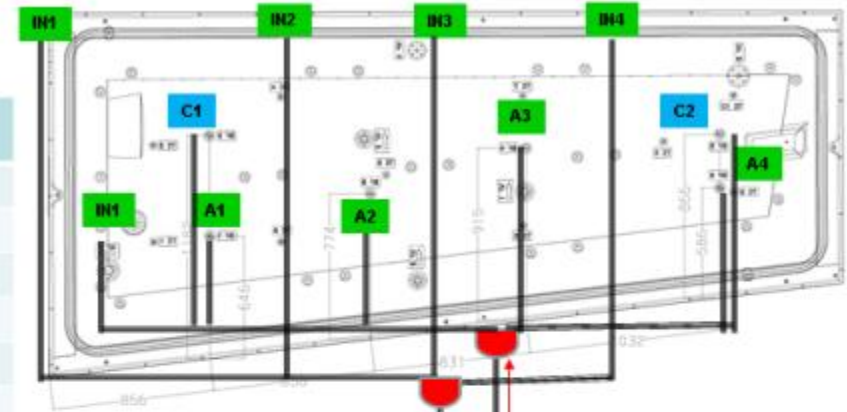
Outside of the autoclave

Inside of the autoclave

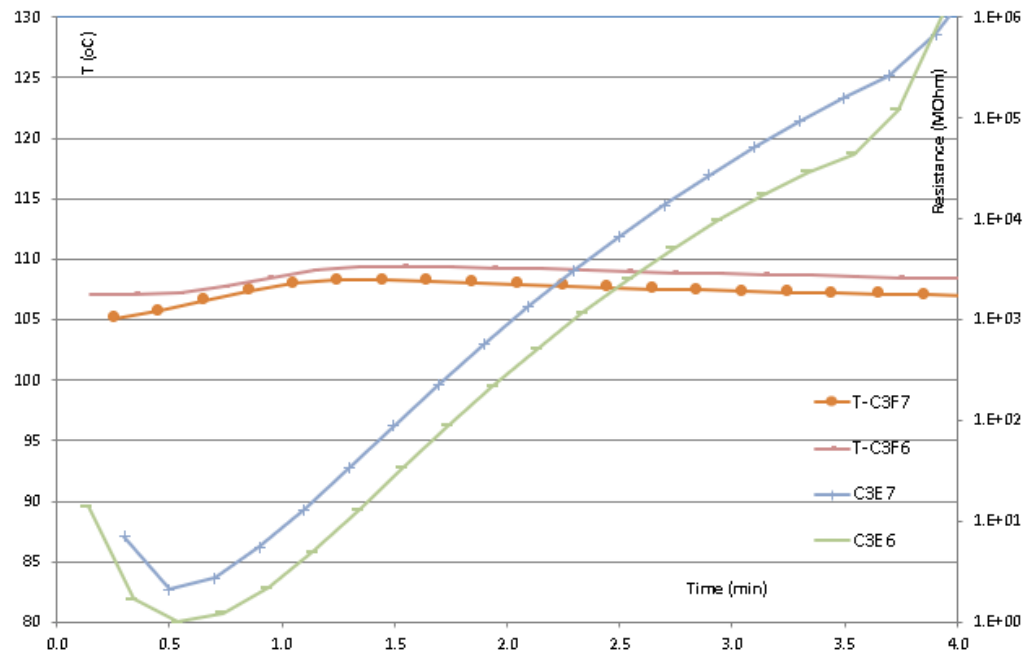


Sensor Location	Sensor Type
SP1	A1
SP2	C1
SP3	A2
SP4	A3
SP5	C2
SP6	A4

C = Cure sensor
A = Resin arrival and Temperature sensor



Real-time Tg prediction and demoulding decision based on targeted Tg.

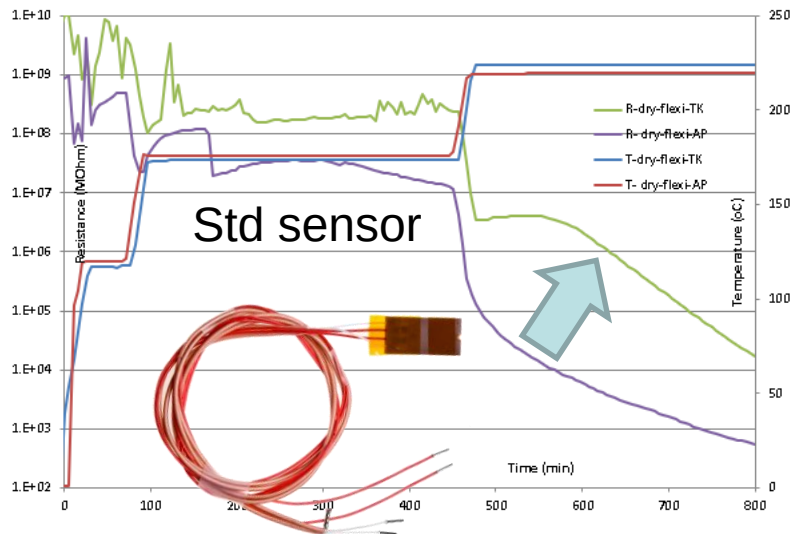


A new durable sensor was developed to allow to measure CFRP production without the need of any extra protection e.g. glass fibre

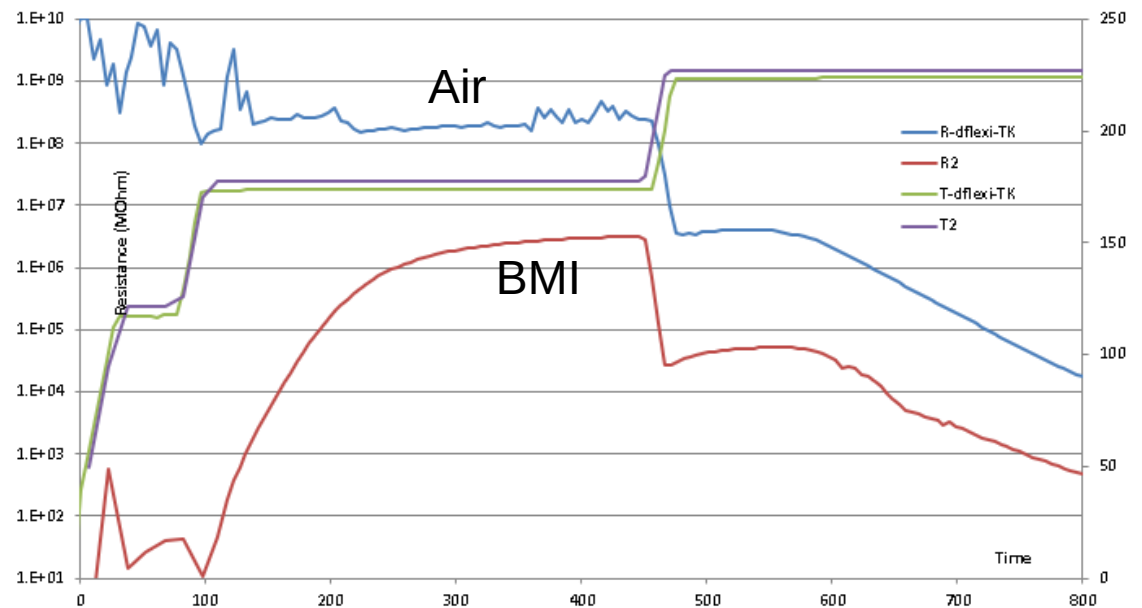
- Due to the unique surface measurement of the DC-based sensor no coating but specially shaped electrodes were used

Extensive trials in a C-HPRTM application have shown very good results confirming that the use of this sensor in CFRP production is feasible

@Air



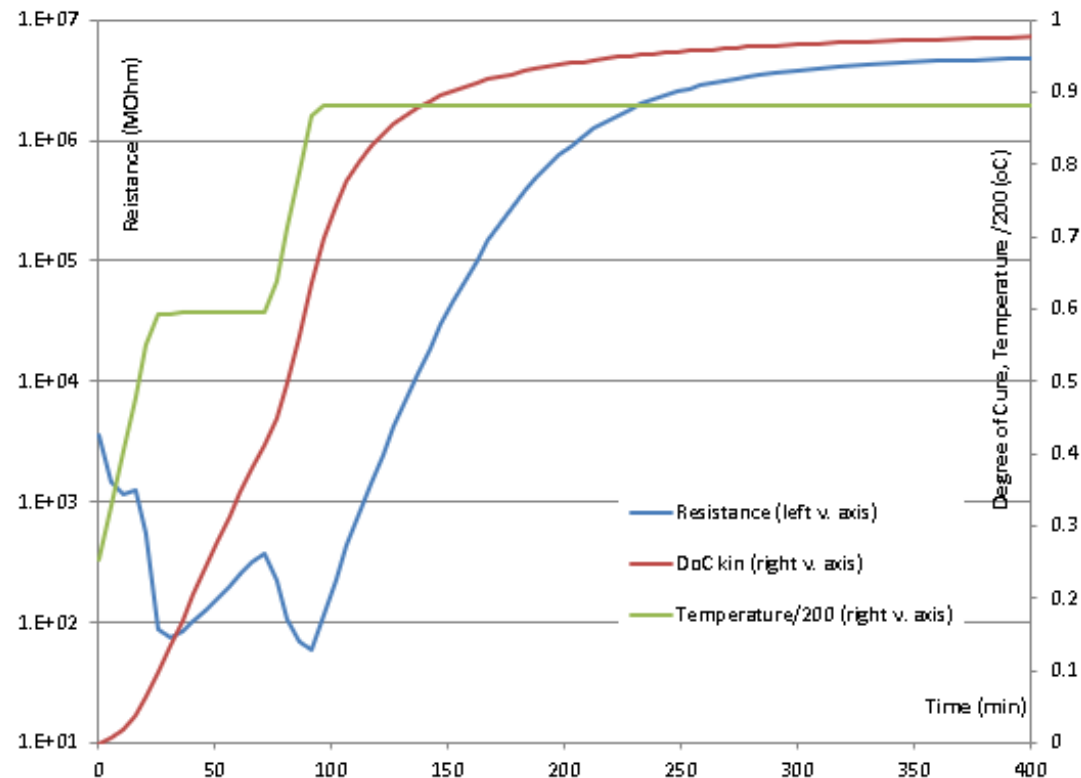
Air vs. BMI



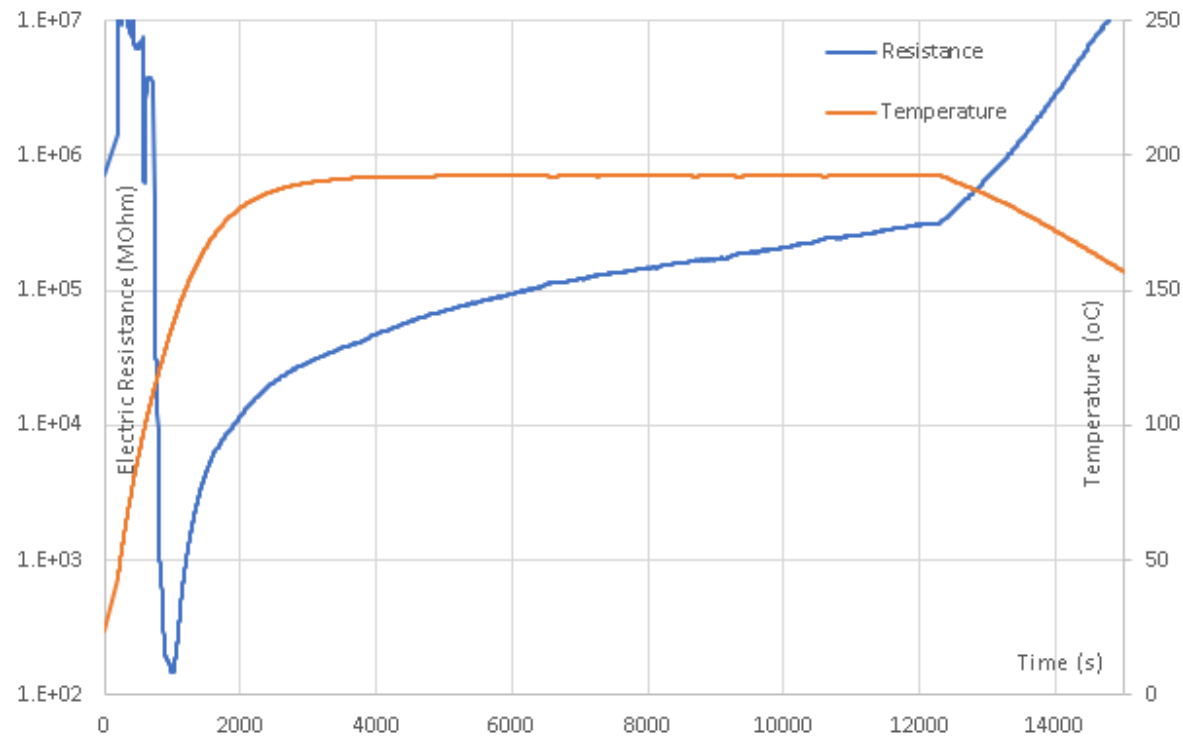
Resistance and temperature

A new higher-temp material was introduced in the disposable sensor to improve the high-temp performance of the sensor

First trials with a prepreg BMI sample sent by BAB demonstrated the improved performance of the sensor



BMI Cytec 5250: Correlation between the expected Degree of Cure as simulated by a kinetic model and the recorded resistance during for the recommended temperature profile.



Within SuCoHS, Cyanate ester resins were also monitored for the first time successfully by FHNW

- ✓ The online cure monitoring and quality control for high temperature resins was applied and verified successfully.
- ✓ The development of new high temperature sensors and calibration methods will lead to significant reduction of the curing time of advanced aerospace resins ensuring cure quality.
- ✓ Further development with SuCoHS project will allow disposable and durable sensors to work at temperatures up to 300°C (first step) and ultimately at 350°C.



SuCoHS

SUSTAINABLE & COST EFFICIENT
HIGH-PERFORMANCE COMPOSITE STRUCTURES
DEMANDING TEMPERATURE
AND FIRE RESISTANCE



This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N° 769178.



AERnova



apodius
quality in automation

BOMBARDIER



Collins Aerospace



ONERA
THE FRENCH AEROSPACE LAB



TUHH
Technische Universität Hamburg

www.sucohs-project.eu



<https://www.linkedin.com/company/sucohs-project/>

Belgium

SYNTHESITES SNC

Av. du Lycée Français 5, Bte9
1180, Uccle
Phone: +32 (0) 472 201 382
e-mail: be@synthesites.com

Greece

**SYNTHESITES INNOVATIVE
TECHNOLOGIES Ltd**

Kyprion Agoniston 33,
GR-185 41, Piraeus
Phone: +30 210 42 12 274,
e-mail: info@synthesites.com

UK

SYNTHESITES UK LTD

31 Arden Close,
Bristol, BS32 8AX
Phone: +44 333 01 2468 1
e-mail: uk@synthesites.com

www.synthesites.com