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Monitoring of thermoset composites curing by SHM sensors

Presentation number 1451

ONERA – The French aerospace lab

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ICCM 23 BELFAST 2023

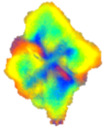
ICCM 23 – Belfast

Context: Control of the health state

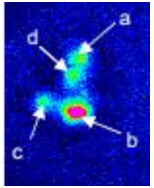
Different methods for the control of structures:

→ **NDT methods** to control parts after the manufacturing step

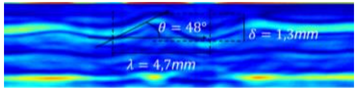
Ex: thermography pulse, ultrasonic methods



lightning strikes in UD composites detected by pulse thermography



mechanical impacts
Detected by lock-in thermography

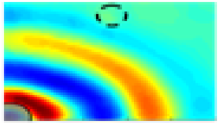


Out-of-planes in composites detected by ultrasonic controls

→ **SHM methods**:

Embedment of sensors into the structure to monitor the health state and to trigger targeted maintenance operations

PZT sensors network on a stiffened panel

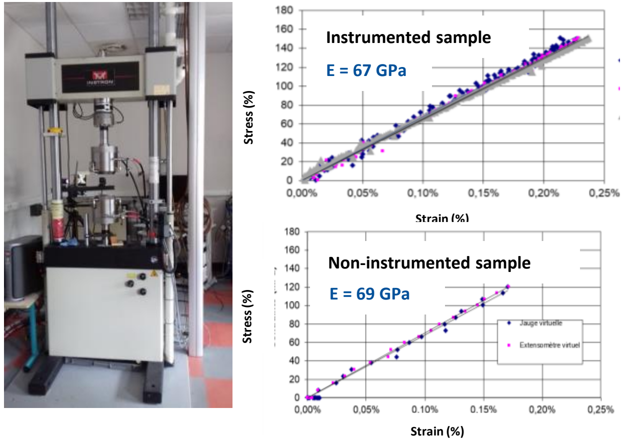


Lamb waves propagation in composites

Control of structure thermomechanical stresses by FBG

→ Opportunity of the composites: Embed **sensors into the core of the composite**

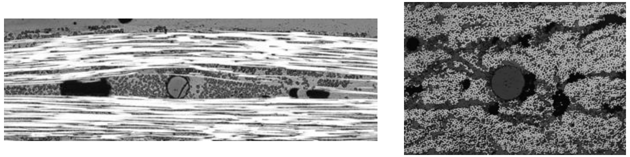
Embedment of sensors into the core of the composites



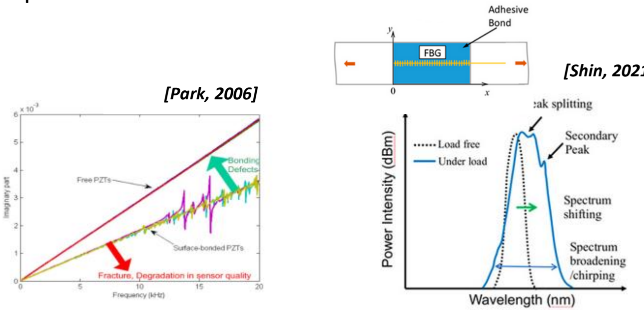
Mechanical influence of the FBG embedment into the core of a composite

Break of their integration:

- ➢ Resistance of sensors to manufacturing conditions (temperature, pressure…)
- ➢ Intrusiveness of sensors on composite properties



Embedment of a FBG into the core of a thermoset composite

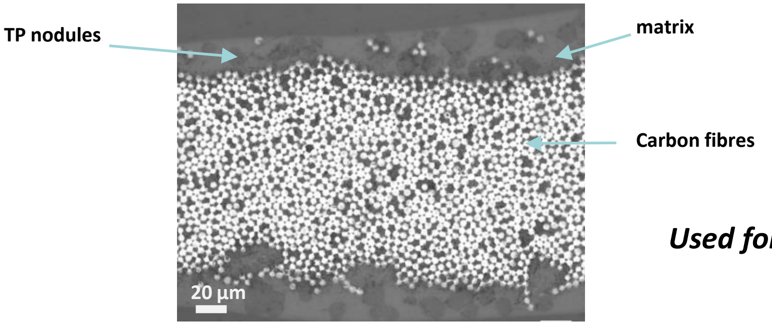


Influence of the sensors debonding on their response

Goals/advantages:

- ➢ Getting knowledge of material's core behaviour
- ➢ Monitoring health state of material from manufacturing step
- ➢ Freeing from bonding issues (bonding quality and repeatability, bonding aging…)
- ➢ Protection of sensors from environment conditions

Definition of the system



Microscopic observation of the prepreg

Material of interest:

- Thermoset UD prepreg: **IMA/M21ev**
Thermoplastic nodules at the interfaces
Thickness ~ 185 µm, Vf ~ 56%

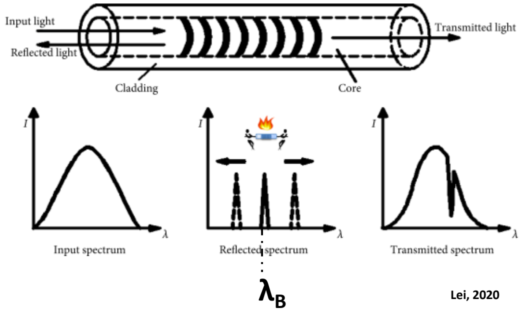
Used for aircraft wings: high strength and toughness

Fibre Bragg Grating (FBG):

- Optical sensor sensitive to temperatures and strains

$$\frac{\Delta\lambda_B}{\lambda_B} = K_T.\Delta T + K_\varepsilon.\Delta\varepsilon + \cancel{K_P.\Delta P}$$

- Gauge length = 5mm
- Diameter = 250µm with acrylate coating, 125µm without coating

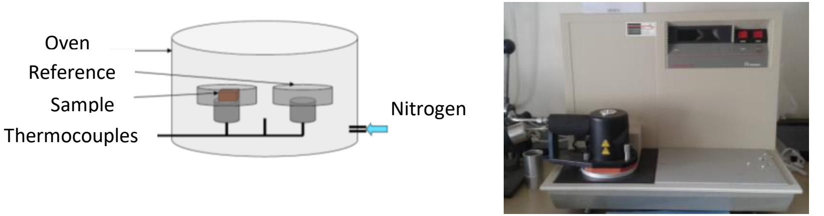


FBG operating principle schema

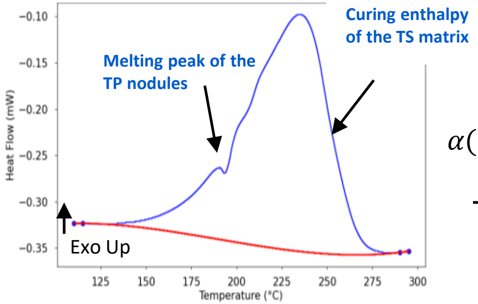
Characterisation of the prepreg curing kinetic (1/2)

Anisotherm characterisation by DSC

- Monitoring of the curing during heat phase of the prepreg
- Identification of the melting temperature of the TP nodules

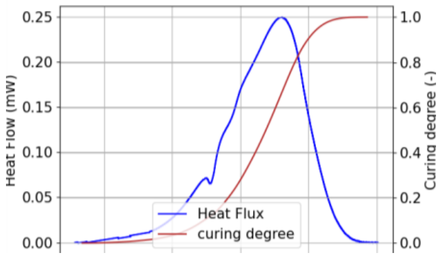


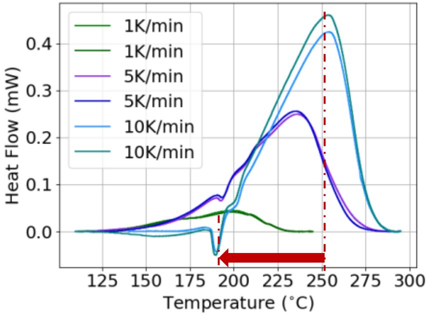
Differential Scanning Calorimetry set-up



Heat rate: 5K/min

$$\alpha(t) = \frac{1}{\Delta Htot}\int H(t)dt$$





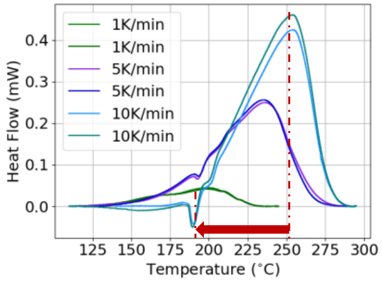
- Characterisation with different heating rates: Shift to lower temperatures

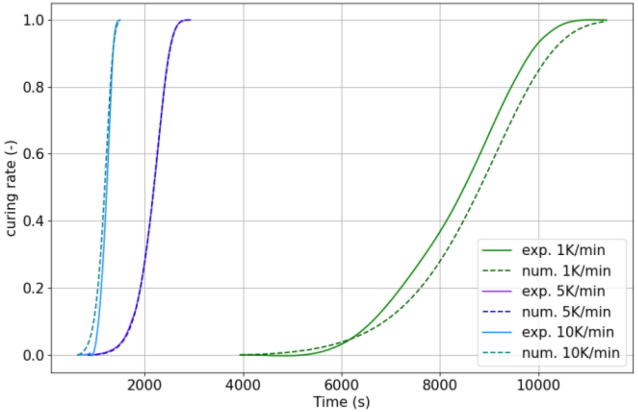
Characterisation of the prepreg curing kinetic (2/2)

Identification of a curing kinetic law

- Selection of a Kamal-Sourour model $\frac{d\alpha}{dt} = (k_1 + k_2\alpha^m)(1-\alpha)^n \quad k_i = A_i \exp(\frac{-E_{ai}}{RT})$

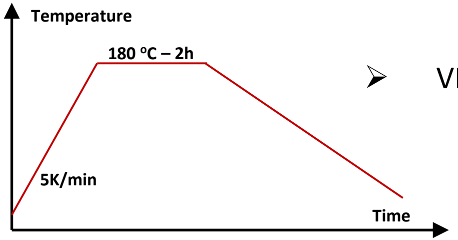
- Identification of the model parameters by a Nelder-Mead minimisation method





A_1 (s^{-1})	A_2 (s^{-1})	E_{a1} (kJ/mol)	E_{a2} (kJ/mol)	m	n
46.5.106	255.7	100	53.2	0.29	1

Instrumentation of the manufacturing process



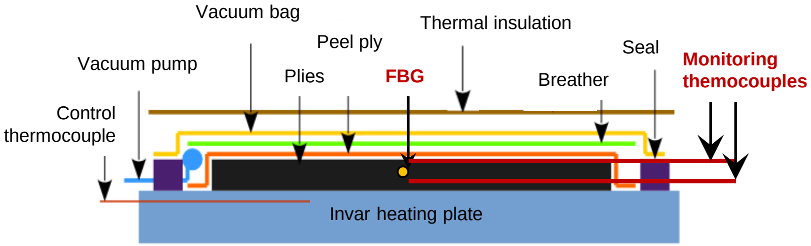
Temperature cycle of the laminate curing

Process conditions:

- VBO (*Vacuum-Bag-Only*) process: Pressure only applied by a vacuum bag → Pmax = 1bar
- Heat set-up: heating plate→ temperature gradient through the thickness
- Laminate: $[0]_8$, 100x100mm^2

Instrumentation:

- **1 FBG in the core** of the laminate at half-thickness, oriented perpendicular to the carbon fibres
- **2 monitoring thermocouples**:
  - 1 in the core of the laminate at half-thickness
  - 1 on the top of the laminate



Lay-up for the curing monitoring

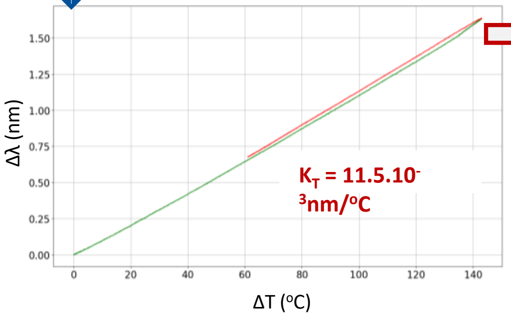
Preliminary results

Data monitored during the curing cycle:

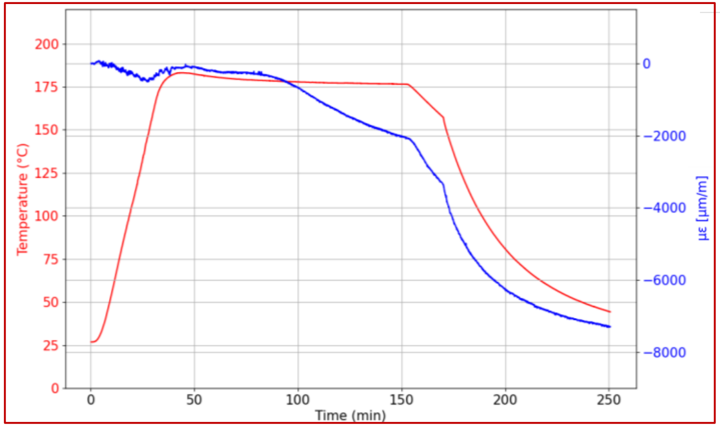
- Evolutions of λ_B and temperatures (in the core and on the surface) throughout the cycle
- Need to take into account the effect of temperature on the sensor response
- Need to decouple temperature and strain effects

$$\frac{\Delta\lambda_B}{\lambda_B} = \boxed{K_\varepsilon.}\Delta\varepsilon + \boxed{K_T.}\Delta T$$

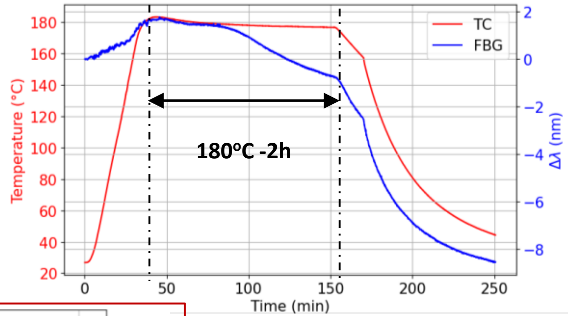
K_ε ~ 1.2 10^{-3} nm/µdef
[Demirel, 2009]



FBG calibration → Evolution of K_T with temperature



Evolutions of temperature and strain during the curing cycle



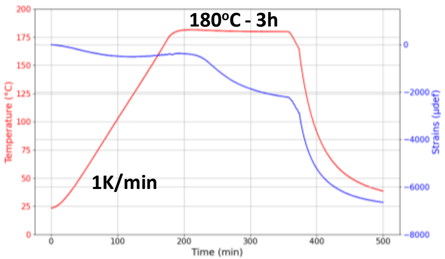
Raw data recorded during the curing cycle

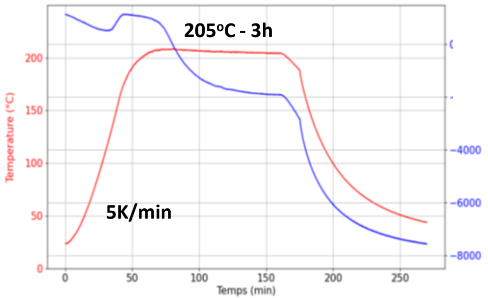
Influence of process parameters on sensors responses

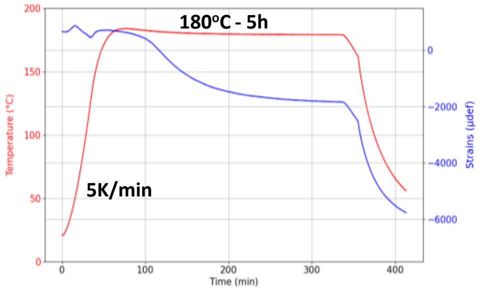
Evolution of curing parameters:

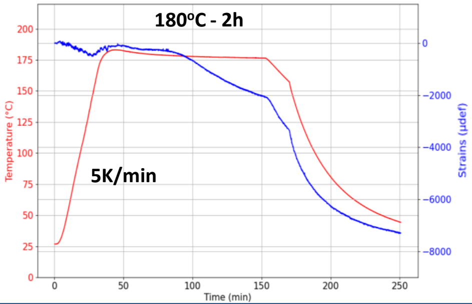
- Influence of 3 parameters: Heating rate, temperature of the plateau, plateau duration
- Good tests repeatability:

  slight differences during the heating phase BUT similar behaviour at the plateau and during the cooling phase
- Strain variations between 6000 and 8000 µdef (in function of the curing conditions)

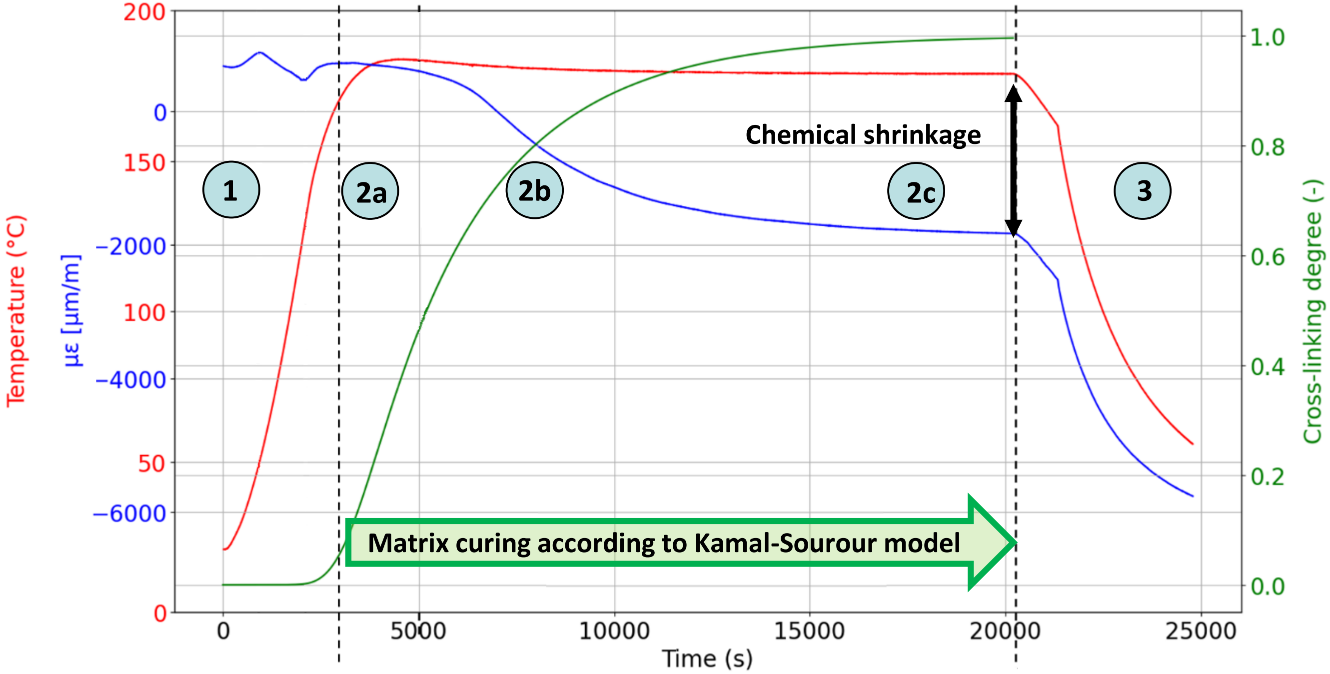








Study of curing phenomena



Different identified steps:

1: Before curing
Low adhesion of the FBG

2a: Start of curing
Compensation of thermal expansion and chemical shrinkage

2b: Important chemical shrinkage

2c: Stabilisation of the chemical shrinkage

3: Thermal shrinkage

Evolution of the chemical shrinkage (1/2)

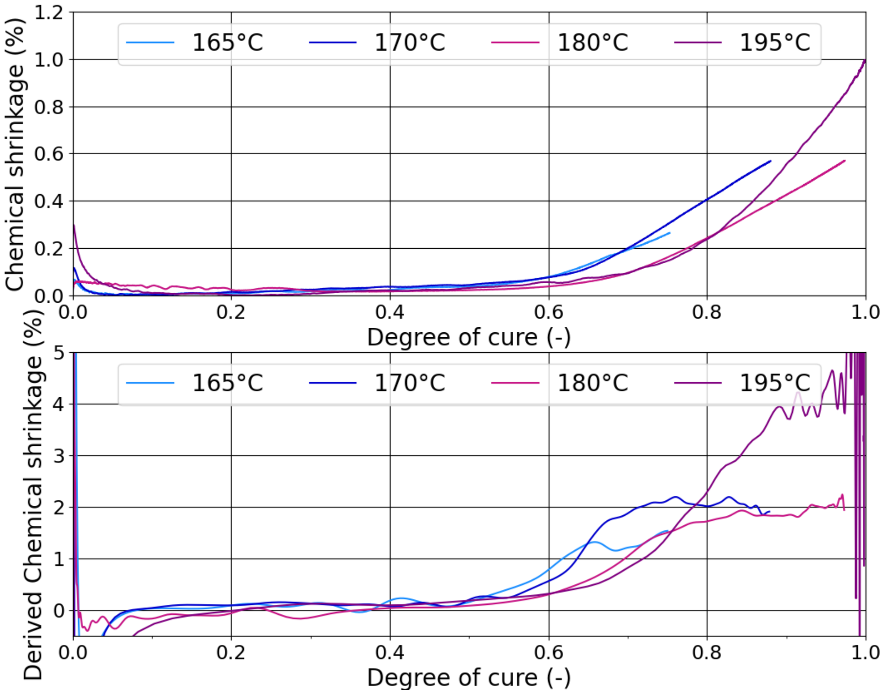
Strains and curing cycle:

For different manufacturing conditions (at **every moment** of the cycle):

- ➢ Strains measured by the FBG
- ➢ Curing degree estimated by the Kamal-Sourour model

Nonlinearity between the cure degree and the chemical shrinkage

- ➢ **Threshold value** of the cure degree **around 0.5**



Evolution of the chemical shrinkage (2/2)

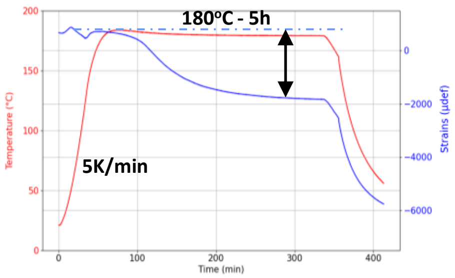
Study of the final chemical shrinkage:

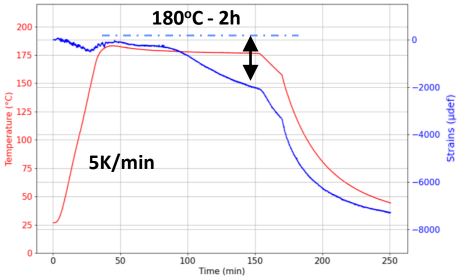
For different manufacturing conditions (**at the end of the plateau**):

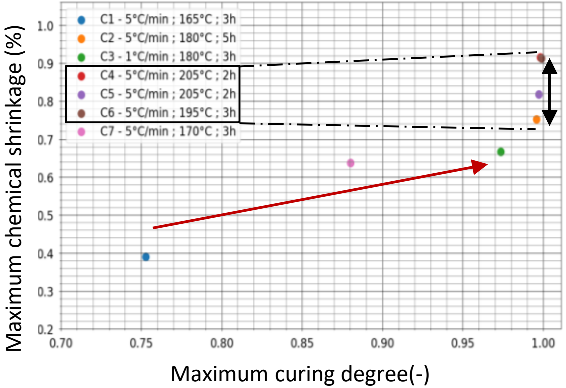
- Maximum chemical shrinkage considered
- Maximum curing degree estimated by the Kamal-Sourour model

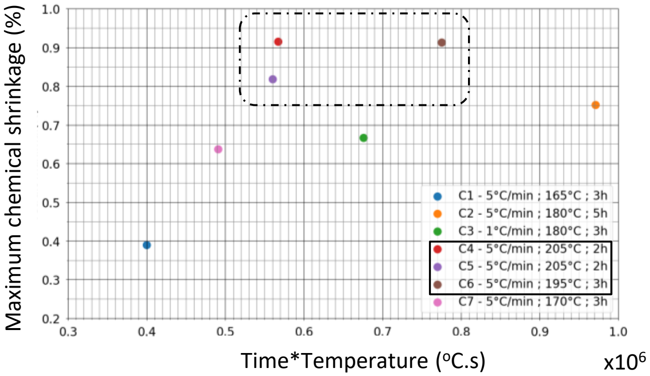
Singular points: data dispersion and high chemical shrinkages

- Hyp: Influence of TP nodules melting ($T_{plateau} > T_{m\ nodules}$)









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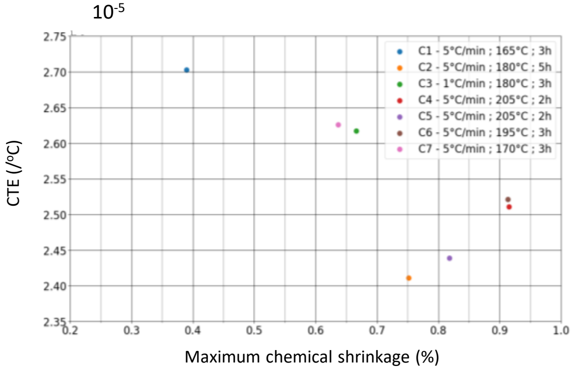
Conclusions and Perspectives

Conclusions:

- Reliable and repeatable monitoring of a thermoset composites curing by FBG
- Identification of the different curing phases
- Possibility to estimate the end of the curing from strains monitoring
- Establishment of the link between the chemical shrinkage and the curing degree (non-linear)



Influence of chemical shrinkage on the composite coefficient of thermal expansion

Perspectives:

- Improve the estimation of the curing degree from the strains measurement
- Study the influence of thermoplastic nodules melting on the strains measurement
- Validation of this method on another thermoset prepreg
- Impact of curing degree on composite properties

Thank you for your attention!

Any question?