

A COMPARATIVE STUDY ON AGING OF HIGH TEMPERATURE POLYMER COMPOSITES REINFORCED BY CARBON FIBRE THIN-PLIES AND SATIN WEAVES

Patrik Fernberg Luleå University of Technology, Sweden





- Background
 - Temperature resistant composites in aeronautics applications
- Objective
- Materials
- Results
- Summary and conclusions



POLYMERIC COMPOSITES IN AERO **ENGINE APPLICATIONS**



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COMPOSITES CONSTITUENT PROPERTIES

	Carbon fibers	Polymer matrix
Stiffness	Stiff (~ 230-300 GPa)	 Rigid (~ 5 GPa) when below glass transition temperature, T_g Soft (~ 50 MPa) above T_g
Density	Light (~ 1800 kg/m ³)	Light (~ 1300 kg/m ³)
Thermal stability	Stable in inert environments and within typical use- temperature range	 Softens at elevated temperature (above T_g) Tendency to degrade in presence of air at elevated temperature
Thermal expansion coefficient	-0.1.10 ⁻⁶ K ⁻¹ (in fiber direction)	50·10 ⁻⁶ K ⁻¹



















OVERALL RESEARCH OBJECTIVE & GOAL

Objective:

- Develop and mature high temperature carbon fibre composites for use in future generation aero-engines

Goal:

- Investigate how the thin-ply effect can play a positive role for high temperature composites



THIN-PLY COMPOSITES





CURE RESIDUAL STRESSES

 At cure temperature

 "stress free"

 "stress free"

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After cooling by ∆T to room temperature ^{°free ply} ^{°actual} stresses[°]

↓ ↑ ↓

↓ σ₂₂ Linear elastic laminate theory Residual stress depends on

- α different between layers
- *E* stiffness differences
- ΔT cooling from cure temperature

Assume a quasi-isotropic layup:

- [-45/45/90/0]_{2S}
- $\Delta T=300 \text{ K}$

Residual transverse thermal stress

- σ₂₂ ~ 75 MPa
- UD strength ~ 53 MPa





OXIDATIVE AGEING



Weight loss:

- Accelerating over time
- Experimental variability
- "Surface governed"

Micro-defects could explain variability



Thin-ply composites to mitigate

- occurrence of manufacturing induced cracks
- thermal oxidative ageing

from MSc thesis H McLaren, LTU 2015

MATERIALS

Baseline laminates:

- Resin: NEXIMID® MHT-R
 - Thermosetting formulation for RTM
 - 6F-dianhydride (6-FDA) backbone
 - 4-(Phenylethynyl)Phthalic Anhydride (4-PEPA) end-group crosslinker
 - ethynyl bis-phthalic anhydride (EBPA) main chain cross-linker
 - Tg ~ 370°C
- Composites
 - [(-45/+45)/(90/0)]_{2s}
 - 370 g/m² 8-Harness Satin weave based on Cytec Thornel T650/35 (Sigmatex)
 - 3 mm thick
 - V_f 58%
 - CPT ~0.375 mm

Thin-ply laminates:

- Resin: NEXIMID® MHT-R
 - Thermosetting formulation for RTM
 - 6F-dianhydride (6-FDA) backbone
 - 4-(Phenylethynyl)Phthalic Anhydride (4-PEPA) end-group crosslinker
 - ethynyl bis-phthalic anhydride (EBPA) main chain cross-linker
 - Tg ~ 370° C
- Composites
 - [(-45/+45)/(90/0)]_{6s}
 - 82 g/m² spread-tow biaxial fabric based on IMS65 Toho Tenax fibres (Oxeon)
 - 2 mm thick
 - V_f 51%
 - CPT ~ 0,083 mm









TRANSVERSE CRACKING DURING TENSILE LOADING



Quasi-static tensile loading – unloading test

- Examine specimen edges for occurrence of cracks after each loading cycle
- Beneficial thin-ply effect
 - <u>No cracks after manufacturing</u>
 - <u>Cracking onset strain shifted</u>
 - From about 0.3% to around 0.6%



THERMAL OXIDATIVE STABILITY – WEIGHT LOSS @ 320°C

Sample dimensions: 75 mm x 11 mm x 2-3 mm





THERMAL OXIDATIVE STABILITY

weight loss relative to matrix weight @ 320° C





THERMAL OXIDATIVE STABILITY



- Initial weight loss (1%) of moisture
- Accelerating trend (as previous)
- Large variations between batches
- Small variation within batches
- 5-8% matrix mass loss after 500 h
 - Corresponding to 2-3 % composite sample weight loss
- <u>No noticeable improvement from</u> <u>thin-plies</u>



BASELINE – BATCH 1 VS. 2



the ra

Presence of voids and cracks



THIN-PLY VS BASELINE BATCH 2



STT a

"potentially micro-pores"



SUMMARY AND CONCLUSIONS

- Thin-ply composites samples based on spread-tow biaxial carbon fibre fabric and high temperature polyimide were successfully manufactured
- A thin-ply effect was observed in terms of
 - Close to zero manufacturing induced transverse cracks for thin-ply composites
 - Onset of transverse cracking in tensile tests shifted towards higher strains (from 0.3% (*Baseline*) to 0.6% (*Thin-ply*))



SUMMARY AND CONCLUSIONS

- No detectable positive thin-ply effect with respect to thermal oxidative ageing
 - Difference in mass degradation rate appears to be dominated by presence of defects
 - Difference in void content and cracks between two different Baseline batches
 - Presence of what appears as micro-defects between individual fibres in *Thin-ply* laminates
- Mechanisms governing difference in degradation rate remains to verify and deserves more research
 - Influence of fibres (stiffness, sizing), interfaces
 - Stress relaxation and/or build up, evolution of matrix properties over time, ...



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THANK YOU FOR THE ATTENTION





MECHANICAL PROPERTY KNOCK-DOWN-BASELINE MATERIALS





COMPOSITE MANUFACTURING

Stage	T [°C]	Time [min]	P [Bar]
Melt and homogenize	240	30	1
Degass under vacuum	240	10	<0.005
Inject resin in to mould	320	~ 5	≤12
Isothermal cure	320	60	12
Heating of tool	320 - 370	30	12
Isothermal cure	370	120	12
Cooling of tool	370 - 80	720	1
Demould	80		1



RTM-facilities for high temperature RTM at RISE SICOMP. Ability to process liquid resins with temperature up to 300°C.

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THIN-PLY COMPOSITES



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"potentially micro-pores"

