Prepreg Process
Limitation Information

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Introduction:
Goals of Processing

- To achieve a finished structure with the desired chemical, physical, and mechanical state
  - This state should be the same as that achieved when creating the material properties database
- Strictly speaking, it is not correct to say that exceeding a property (lower bound), e.g. strength, is a ‘good’ thing
  - One should consider the underlying cause: why is the strength higher? Might it indicate a problem in an un-measured property?
  - Better to consider lower and upper bounds on properties
  - Best to consider underlying definitions of chemical, physical, and mechanical equivalence
    - e.g. degree of cure, volume fraction, and residual stress
- In practice, some interesting questions are
  - scaling (going from small test panels to actual structure)
    - Am I really getting the same material?
  - minimizing uncertainty at all stages (variability in all its forms)
    - If I change the cure cycle, and get equivalence at the test panel level, will I get equivalence at all scales?
Our Historical Background: Science-based Processing/Process Modelling

- First work that we are aware of goes back GD Convair division in the 60s/70s
- Significant industrial/government research focus over the years
- Oddly enough, comparatively little systematic academic research
  - Springer, Gutowski, Dave, … in the early years
- UBC group became interested in the mid-80s
  - We couldn’t make high-quality transparent panels for matrix cracking project
- Our applied involvement started in early 90’s with Boeing Seattle and the NASA-funded ATCAS program.
- Fortunate to have collaborated and been funded by many US organizations: Boeing, NASA, US Air Force, DARPA/Navair, …
- First major industrial application was support of 777 horizontal stabilizer in mid-90s
- Convergent Manufacturing Technologies created in 1997 as spin-off company from UBC to do applied/proprietary/technology work. Located on UBC campus, totally separate entity but with very strong links to UBC research group
- COMPRO 2D and 3D commercially available, current biggest application is Boeing 787 where significant use being made for all cure cycle design.
- Industrial application has shown the strong need for:
  - Hierarchy of solutions from graphical methods to closed form eqns to 1D to 2D to 3D
  - A structured approach, with all the checks and balances of validation, verification, documentation, etc., needed to introduce the methodology into the workflow
- In this presentation, we concentrate on the graphical methods
QUESTION: Are we testing the same at all levels:
• Material?
• residual stresses?
• Dimensions?

Both mean & variation?

Scaling from test panels to full structure

coupons (thousands)

elements, joints, small panels (hundreds)

• Dimensions
• Strength (inc residual stress)
• Modulus
• Toughness

large panels sub-components (dozens)

• Local temperature history
• local resin degree of cure
• local fibre volume fraction and orientation
• local residual stresses
• local void fraction

test box

full structure

• Cure cycle
• Part geometry: thickness and other details
• Tooling design (material/substructure/cauls…)
• Autoclave/oven performance
Cure Process

Temperature

Degree of Cure

Equivalence:
- Chemical
- Physical
- Mechanical
Cure Measurement & Modeling

DSC Experiments

Data Analysis

Data Reduction/Model Fitting

\[
\frac{d\alpha}{dt} = \frac{K\alpha^m (1-\alpha)^n}{1 + e^{C[\alpha-(\alpha_{c0}+\alpha_{cT}T)]}}
\]

\[
K = A\exp^{-E/RT}
\]

e.g.
Methods of Cure Cycle Selection

- 2D and 3D computer models incorporating cure kinetics (and more) are now fairly well established
  - Expensive and complex
- Significant interest in having simpler methods for evaluating and selecting cure cycles
  - No reason why such methods cannot be consistent and complementary with the more complex methods
- Graphical methods are ideal, as large amounts of data can be presented, trends identified, and information absorbed.
  - Best known approaches are those of Gillham & Colleagues:
    - TTT – Time-Temperature-Transformation
    - CHT – Cure-Heating-Transformation
    - TgTP – Conversion-Temperature-Property
Process Map Development:

$\alpha$-$T$ Map Concept

**Goals:**

1. To follow any cure cycle path on a single map
2. Evaluate material property development by overlaying the cure path on contours of constant property values
Isothermal Contours

\[ \alpha_i = \int_0^{t_i} \frac{d\alpha}{dt} \, dt \]
Dynamic Contours

\[ T = T_0 + \frac{dT}{dt} \cdot t \]
\[ \alpha_n = \alpha_{n-1} + \frac{d\alpha}{dt_{n-1}} \cdot \Delta t \]

Time contours start at 600 seconds with increments of 600 seconds.
Single Ramp/Hold Cycles

Comparison of different heating rates

Time contours start at 600 seconds with increments of 600 seconds.

- 0.55 °C/min (1 °F/min)
- 1.65 °C/min (3 °F/min)
- 2.75 °C/min (5 °F/min)
- 4 °C/min
- 6 °C/min
- 8 °C/min
- 10 °C/min

Residual Stress?
Viscosity Contours

Viscosity contours start at 0.05 Pa*s and increase by multiples of 2.

- 0.55 °C/min (1 °F/min)
- 1.65 °C/min (3 °F/min)
- 2.75 °C/min (5 °F/min)

Gelation

Residual Stress?
Cure Rate Contours

Cure rate contours start at 0.00003 /s, and end at 0.00053 /s and increase by increments of 0.00002 /s.

- Max cure rate for 0.55 °C/min = 0.00022 /s
- Max cure rate for 1.65 °C/min and 2.75 °C/min = 0.00049 /s
Plotting Multiple Ramp/Hold Cycles

- How does one move from a dynamic (ramp) segment to an isothermal (hold) segment, and vice-versa, in an arbitrary cycle with multiple segments?
  - As shown previously, any arbitrary \((\alpha, T)\) point can be achieved by an isothermal hold (within limits).
  - Similarly, we can generate a set of dynamic ramps with different starting values of \(\alpha\) and \(T\), such that any arbitrary \((\alpha, T)\) point can be achieved by a dynamic curve (within limits).
  - Thus an arbitrary cycle can be generated by jumping between contours.
  - Note that it is practical to only have one set of dynamic contours (one ramp rate) per map, as otherwise the map gets too busy.
Example: 2 Hold Cycle

Time contours start at 600 second with increments of 600 seconds.

\[
\frac{dT}{dt} = 3 \, ^\circ\text{C/min}
\]

3\(^\circ\text{C/min}\) Ramp to 140\(^\circ\text{C}\)

1 hour hold at 140\(^\circ\text{C}\)

140min hold at 180\(^\circ\text{C}\)

3\(^\circ\text{C/min}\) Ramp to 180\(^\circ\text{C}\)
Example: Cure Cycle Selection

MRCC for Material A

1. Heat at 1-3°C/min (2-8°F/min) to 110°C ± 5°C (230°F ± 9°F)
2. Hold at 110°C ± 5°C (230 °F ± 9 °F) for 60 minutes ± 5 minutes.
3. Heat at 1-3°C/min (2-8°F/min) to 180°C ± 5°C (356 °F ± 9 °F)
4. Hold at 180°C ± 5°C (356 °F ± 9 °F) for 120 minutes ± 5 minutes.
Material A MRCC

Highest Recommended Heating Rate

Time contours start at 600 seconds with increments of 600 seconds.

\( \frac{dT}{dt} = 3 \, ^\circ C/min \)

1 hour hold at 110\(^\circ\)C

3 \(^\circ\)C/min ramp to 180\(^\circ\)C

120 min hold at 180\(^\circ\)C
Material A MRCC

Lowest Recommended Heating Rate

Time contours start at 600 seconds with increments of 600 seconds.

\( \frac{dT}{dt} = 1 \, ^\circ \text{C/min} \)

1 hour hold at 110\(^\circ\)C

1 \(^\circ\)C/min ramp to 180\(^\circ\)C

120 min hold at 180\(^\circ\)C

Residual Stress?
Material B MRCC

• Apply the temperature ramp from ambient to $270 \pm 10 \, ^\circ F$ at a rate of $3.0 \pm 1.0 \, ^\circ F$ per minute.

• Maintain the cure temperature at $270 \pm 10 \, ^\circ F$ for 120 – 150 minutes.
Material B MRCC

**Draft**
Material B: Evaluation of effect of hypothetical additional hold

Additional contours + 5 min

Degree of Cure

Temperature (°C)

1.5 Cpm

300 s

200 s

100 s

**DRAFT**
Conclusions

- The last 20-30 years has seen the development of considerable processing science and modelling knowledge/technology
- Even a few years ago, this knowledge was only available in return for significant investment and technical sophistication
- Recently, our team (UBC/CMT) has been working on simple techniques which are consistent with the complex techniques and provide much of the information for a fraction of the cost
- The process maps presented here are a good example of this simple approach, and are valuable aids in process cycle selection.
- The aim is to overlay on the maps zones where chemical, physical and mechanical equivalence can/cannot be achieved.
- These maps can be generated easily for any material whose behaviour has been characterized.
- Maps can be used in conjunction with other simple techniques to evaluate tooling decisions, oven/heating profiles, etc., to minimize risk and uncertainty in meeting equivalence requirements