AME 486

Project 3

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#1

Tests that should be performed to ensure that the prepreg supplied by the supplier is what was advertised:

- Chromatography: allows one to dissolve the prepreg's resin in a solvent
  - The material is then injected into the instrument and filters in the column interact and separate the components by either molecular weight (GPC) or functional groups (HPLC)
  - Verify proper ratio of constituents have been used in the resin
  - Verify a limited amount of reaction product has formed
  - Reverse engineer and/or identify the constituents with FTIR of selected peak
- Tack:
  - Assess stickiness of a prepreg material with aging and/or temperature
- Laminate Testing on Test coupon made with prepreg material
  - Simple mechanical Tests (Tension -90 degrees, 0 degrees, SBS)
  - Using DMA, to determine the glass transition temperature (Tg) , and ensure the material will perform within expected thermal limits
  - Void content:
    - Real density vs. bulk density
    - Acid digestion
    - Image analysis cross section, Image JSW
  - Fiber Volume Fraction (FVF) Tests
    - Determines the volume percentage of fibers in the composite
    - Dissolve matrix in boiling sulfuric acid
    - Determine weight of fiver vs. total weight
- Gel Time Test:
  - Measure the time it takes for the resin to cure at a given temperature
  - Ensures the prepreg has not prematurely cure due to excessive out-time

#2

Stages	Potential Outcomes
1	• Bag leak – should be fine. No gelation. Can turn back since resin hasn't advanced • Temp Controller stops – recoverable. (early stage of cure)
2	• Bag leak – could be a problem area. Must determine severity with additional tests. Material has advanced but not quite gelled. • Autoclave pressure stops – usually non-recoverable • Temp. Controller stops – unlikely to recover (advancement issues)
3	• Bag leak - problem area and is usually unrecoverable. • Temp. Controller stops – typically will damage part on cool down (resin is weak) • Autoclave pressure stops – usually non-recoverable
4	• Temp. Controller stops – no problem with respect to cure usually, unless cools fast • Bag leak – No problem. Material has gelled • Autoclave pressure stops – Doesn't matter
5	• Bag leak – No problem. Material has gelled • Autoclave pressure stops – Doesn't matter.

#3

- Delamination:
  - Occurs when layers of the composite material separate due to poor bonding between plies
    - Typically, acceptance criteria require the detection of delaminations that are ≥ 0.25 inch
    - Causes: insufficient resin, trapped air, improper curing
    - Weakens structural integrity and reduces load-bearing capacity.
- Impact damage:
  - The effect on the compression static strength:
    - Easily visible damage can cause 80% loss
    - Barely visible damage can cause 65% loss
- Porosity:
  - Small air bubbles or gaps within the composite material
  - Can be caused by : inadequate resin flow, improper vacuum bagging, or an incomplete cure
  - Reduces mechanical properties and increases vulnerability to cracks
  - Increases equilibrium moisture level
  - Degrades matrix dominated properties
- Surface Notches (blisters or cracks):
  - They are irregularities on the surface, such as rough texture, small cracks or bubbles.
  - Usually stems from poor mold release, trapped air, uneven resin distribution or thermal expansion differences
  - Can increase susceptibility to environmental degradation, functional issues, and static strength reduction of up to 50%

#3 Cont.

- Ply Gap
  - Is a discontinuity or separation between adjacent plies within a composite laminate
    - Results in regions with missing reinforcement, resulting in weakened mechanical properties
  - Causes:
    - Improper cutting of prepreg and dry fiber layers
    - Poor placement or misalignment of plies during lay-up
    - Variations in material thickness leadings to gaps between adjacent plies
  - Results:
    - Reduced load transfer efficiency between plies
    - Increased local stress concentrations, which lead to cracks
    - May cause resin-rich areas, leading to brittleness and reduced stiffness
- Ply Waviness (Fiber Wrinkling)
  - The undulation of deviation of fibers from their intended straight path within the laminate
    - Results in localized fiber misalignment and reduced mechanical strength
  - Can degrade compression strength, as wavy fibers can introduce buckling points
  - Fatigue life is reduced at least by a factor of 10
  - Stress loss can be predicted by assuming loss of load-carrying capability

#3 Cont.

- Thermal Over-exposure
  - When a composite material is subjected to excessively high temperatures or prolonged curing times during the manufacturing process
    - Can cause:
      - Matrix cracking
      - Delamination
      - Fiber debonding
      - Permanent reduction in glass transition temperature (Tg)
  - Stress loss can be predicted by assuming loss of load-carrying capability

#4

•Out-time refers to the total amount of time a prepreg spends outside of its recommended storage conditions

•Manufacturers specify a maximum allowable out-time, beyond that time, the material may degrade and become unsuitable for use
    •If out-time is exceeded:
        •The resin in the prepreg can begin to partially cure at room temperature, making it more difficult to flow and bond properly during curing and layup process
        •There can be a loss of tackiness, making it difficult to position and compact properly
        •Some composites can absorb moisture from the air, forming voids, porosity or even cause delamination
        •Material may become brittle and hard to handle, leading to fiber misalignment or cracking, affecting overall performance
        •Can cause reduced mechanical properties and lead to early failure under mechanical loads

#5

- Tg (Glass transition) is the temperature where the polymer goes from a hard glassy state to a soft rubbery state with temperature
- Tg is measured by measuring modulus (DMA), thermal expansion (TMA), or heat of transition (DSC) changes versus temperature.
    - The glass transition temperature must be defined by instrument used and ramp rate of heat
- For DSC:
    - At Tg, it can be measured where there is a change in heat capacity, seen as a shift in the heat flow curve
- For DMA:
    - At Tg, it is identified as a drop in storage modulus (stiffness) or a peak in tan (damping)
- For TMA:
    - At Tg, it can be seen as the noticeable change in the coefficient of thermal expansion (CTE)

#6

- Void content refers to the percentage of trapped air, gas, or unfilled spaces within a composite material
- This a crucial parameter in composite manufacturing, as voids act as stress concentrators and weaken the material
- Transverse Shear Strength: the ability of the composite to resist shearing force perpendicular to the fiber direction
- **High void content** significantly reduces transverse shear strength
    - **Voids** create weak spots and allow cracks to initiate and propagate more easily
- **Effects of voids on shear properties:**
    - Reduces interlaminar bonding, making layers prone to delamination
    - Allows moisture ingress, further degrading mechanical properties over time
    - Increases stress concentrations, which can lead to premature failure
- **Recommended void content target** is < 2%
    - can be designed to have a higher void content target, for examples sports goods (e.g. tennis rackets)

#7

- The appropriate volume fraction of fibers is typically 55% to 65%
  - Relates to the equation 1 = Vf + Vm + Vv
- This fiber volume fraction will ensure:
  - Increase tensile and compressive strength
  - Enhanced stiffness and load-bearing capability
  - Reduced weight, resulting in higher efficiency

#8

- Tool Surface Quality Issues:
  - Surface defects: causes imprinting on the laminate, leading to poor surface finish
  - Contaminated tool surface leads to poor adhesion or surface defects
  - Application of improper release agent application: sticking of the laminate may arise to the tool or either contaminate the composite
- Coefficient of Thermal Expansion (CTE) Mismatch:
  - During the heated cure, the CTE mismatch between the tool material and the composite often must be accounted for
  - This mismatch can cause:
    - Distortions, warping, and residual stresses in portions of the cured part
- Inadequate Vacuum Sealing
  - Leaks in the vacuum bagging system causes incomplete resin consolidation, leading to voids
  - Poor sealing on the edges can allow air to enter and therefore cause defects and more void content
- Improper Tooling Material Selection:
  - Composite tooling may degrade with several repeated thermal cycles; must keep this in mind
  - Thermal conductivity differences can cause defects and uneven curing
  - Thermal cycling effects can cause gradual shape distortion over time

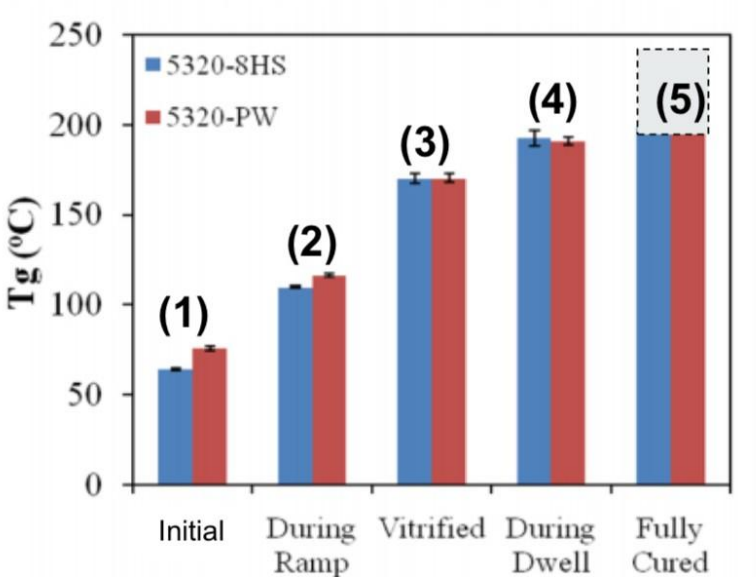
#9

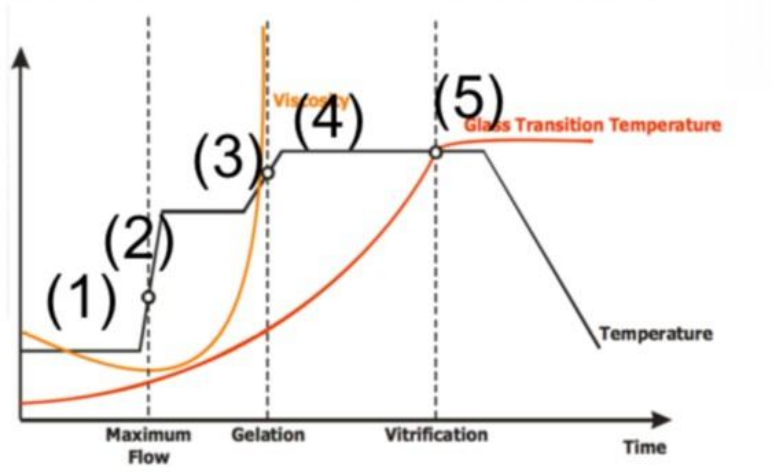
Resin cure process

- Phase 1 – Resin is in liquid form and is impregnated into the fibers. The resin needs to be spread out evenly to reduce void content. The resin is then heated isothermally (Ramp heated), reducing the viscosity which allow the resin to fully wet the fibers. This is the maximum flow stage.
- Phase 2 - The resin undergoes crosslinking at the “gel-point”. The liquid resin transforms into a gel and the resin can no longer flow. Fiber placement is fixed.
- Phase 3- Resin is almost cured and appears to be a glassy consistency. A time and temperature is chosen to achieve approximately an 80-95% cure state. A high temperature is necessary, and pressure is necessary. (Note: long duration at lower temperature will not work)
- Phase 4 – The final hardening and vitrification phase where the resin goes from partially cured to fully cured.
- Phase 5 – Potentially a post cure phase where additional heat is applied to complete the polymerization process. If no additional heat is needed, then the cured composite reaches its final properties. The resin is cooled down and allowed to fully harden before being removed.

#10

- Phase 1 - The glass transition temperature (Tg) is low, and the resin remains a viscous fluid with minimal strength. This is the initial phase.
- Phase 2 – Tg begins to rise gradually, but the material is still weak. This is during ramp heating and resin flow increase sharply from the initial phase.
- Phase 3 - Tg continues increasing, and gelation occurs, leading to a significant rise in strength. This is where vitrification occurs.
- Phase 4 - The resin solidifies as Tg is stabilized, approaching the final cure temperature. This is a dwelling period.
- Phase 5 – Tg reaches its final value as the material is fully cured, achieving maximum strength. The composite is fully cured.





Source: Images both from lecture

#11

- Read one article and one research paper, these are my conclusions
  - Autoclave curing advantages
    - Used for tier 1, aerospace grade components. Typically, the autoclave process is paired with high quality laminates
    - Aerospace-grade composites have a void content of 1%
    - Low void content = better mechanical properties (strength, stiffness)
  - Disadvantages
    - Very expensive – space, tooling, and energy
    - Have a limited component size
    - Long cycle times – limit production volume and efficiency
- These disadvantages have forced the industry to look at out-of-autoclave processing. There is a need to optimized the OoA process.

From the research paper

- Autoclaving
  - Lower porosity and voids – higher pressure (6-10 bar) removes the voids and leaves a void content of >1%
  - Better resin flow – Heat and pressure ensures a uniform distribution of resin
  - Higher strength capabilities – less voids and porosity mean better mechanical properties
- Out-Of-Autoclaving (OoA)
  - Higher porosity – due to less pressure (roughly 1 bar) which leads to a higher void content between 1% and 5%
  - Limited resin flow – the lower pressure means the resin cannot impregnate the fibers as well as an autoclave

Source: https://www.compositesworld.com/columns/why-out-of-autoclave-processing-is-good-for-the-composites-industry
Source: https://www.mdpi.com/2504-477X/6/6/172

Test Type	Standard Method	Application
Dynamic Mechanical Analysis (DMA)	ASTM D4065, D4440, D5279 – Determines elastic modulus, viscous modulus, and damping coefficients (stander practice and test methods for plastics) ASTM D7028-07 – Designed to determine the glass transition temperature of continuous fiber reinforced polymer composites	Used to evaluate mechanical properties of composites. Can be used to evaluate interfacial adhesion between different components in a composite structure. Important when understanding cure cycles and Tg. Measures mechanical properties in terms of time, temperature, frequency, or stress
Thermomechanical Analysis (TMA)	ASTM E831 – Standard for linear thermal expansion of solid materials. Determines the coefficient of linear thermal expansion to determine if failure by thermal stress may occur when a solid body composed of two different materials is subjected to temperature variations ASTM E1545 – Method to assign Tg temperatures using thermomechanical compression measurements during heating ASTM E2092 – Method for distortion temperature in three-point bending ISO 11359 - Methods for polymer composites	Used to characterize physical properties of materials when force is applied at specific temperature and time periods. Used to determine the CTE, Tg, and softening point of a material.

Test Type	Standard Method	Application
Differential Scanning Calorimetry (DSC)	ASTM D7426, E1356 – Methods for defining the working temperature. Test includes finding the Tg ASTM D3418 - Transition temperatures of polymers, standardization for resin cure times ASTM E1269 - Determination of specific heat capacity ISO 11357 - Melting and crystallization and Tg analysis	Used to measure degree of cure, Tg, and melting point of composite. Used to study the thermal properties of composites by measuring heat flow changes. (Ex. Used to see if material has aged during layup)
Fourier Transform Infrared Spectroscopy (FTIR)	ASTM E168 – General guidelines on infrared spectral analysis ASTM E1252 – Determines the standard guidelines for FTIR analysis on polymers ISO 19226 – Testing methods for polymer composites using FTIR to analyze chemical composition, degree of cure, and degradation	Used to identify material including its composition and molecular structure. Used to analyze oxidation, degradation, chemical aging. Uses absorption/emission techniques to understand composition of the composite. First step in identifying a polymer and is used in quality control of materials. Can be used to conduct contamination analysis as well.

#12

These are the sources for question 12

Dynamic Mechanical Analysis

1. https://www.tainstruments.com/what-is-dynamic-mechanical-analysis/
2. https://www.astm.org/d7028-07r15.html
3. https://www.smithers.com/industries/materials/polymer/physical-testing/material-properties-testing/dma-testing

Thermomechanical Analysis

1. https://www.eag.com/techniques/phys-chem/thermomechanical-analysis-tma/
2. https://www.wmtr.com/en.Thermomechanical-Analysis.html

Differential Scanning Calorimetry

1. https://www.intertek.com/polymers-plastics/testlopedia/differential-scanning-calorimeter/
2. https://www.stress.com/services/materials-engineering/polymers-elastomers-composites/differential-scanning-calorimetry/

Fourier Transform Infrared Spectroscopy

1. https://www.intertek.com/polymers-plastics/testlopedia/ftir/

#13

- The glass transition temperature (Tg) is a property that effects the resin not the reinforcing fibers
  - Resin
    - Is very temperature sensitive and needs to be stored in a refrigerated area because it can go bad
    - Very expensive, needs to be shipped and stored at a low temperature (-40ºC)
    - Supplier will provide a cure schedule and recommend a max out time at room temperature
      - This is done to prevent moisture from reacting with the resin which can lead to with epoxies and carbamates
        - This could introduce moisture into the resin and negatively impact material properties and cure schedules
  - Tg is a polymer property
    - The Tg marks the transition of the resin from ridged, glassy state to a softer viscous state
      - The resin is the glue the binds (polymerizes) the fibers together
    - Fibers are crystalline structures, Tg doesn't affect this
  - Tg controls the resin
    - A controlled temperature ensures that the resin spreads out evenly, fully wetting the fibers and creating a strong, defect-free structure
    - The resin should not gel up too quickly or it may trap voids leading to defects (may spread unevenly)
    - You want to minimize voids in the composite, which requires proper temperature to allow trapped gases to escape

    - Tg has no direct effect on the reinforcing fibers

#14

- Case 1
  - Supposed to have a composite that is flat, but instead it is warped
- Potential issues
  - Due to a wrong layup
  - Due to a missing ply causing an imbalance in the composite laminate structure
  - Due to the addition of an extra ply
- How to verify problem
  - Thickness measurement
    - Each ply is expected to be approximately 5 mils (0.005 in.) thick
    - If the measured thickness is greater, an extra ply was added
    - If the measured thickness is less than expected, a ply might be missing
  - Cross sectional microscopy
    - Mount and polish a cross section of the composite to analyze the number of plies
    - Use an optical inspection method (microscope, etc.…) to count layers and confirm laminate orientation
    - 0º and 90º plies are easy to identify
  - High magnification photo inspection
    - Check the fiber orientation within each ply
    - The oval ratio of fiber cross sections can indicate whether fibers were cut at the correct angles

#14 Cont.

- Case 2
  - A 10-ply unidirectional composite was manufactured using T300 fiber with RS3c cyanate ester resin on a graphite mandrel. However, the composite has **blisters** on its surface
- Possible causes
  - Incomplete cure or wrong glass transition temperature (Tg)
    - We know that the resin is supposed to cure at 177ºC and should have a Tg of 195ºC
    - If Tg is lower than expected, the composite may not be fully cured, introducing strength and defect issues
  - Carbamate Formation due to moisture contamination
    - Sample could have been introduced to moisture allowing carbamates to form during curing
    - The decomposition of carbamates releases CO_2 which can cause blisters
  - Contaminated mandrel
    - If the mandrel contained moisture, the plies adjacent to the mandrel would be more affected (due to setup)
- How to verify problem
  - Perform dynamic mechanical analysis to determine Tg
    - Run DMA test to measure Tg of composite
    - If Tg is lower than 195ºC, the curing process may have been incomplete or there was moisture contamination
  - Perform a post-cure treatment and re-test
    - Post cure the sample at the highest cure temperature and run another DMA
    - If Tg increases to 195ºC, the issues was likely an insufficient cure
    - If Tg does not improve, then the issue is most likely carbamate formation
  - Identify the source of carbamate formation
    - Perform an analysis to verify carbamate formation (chemical process)
    - If plies adjacent to mandrel are more degraded – the mandrel is the culprit
    - Verify this by performing image analysis

#14 Cont.

- Case 3
  - During autoclave cure, the part overshoots the final cure temperature by 50ºC. WE want to know if my boss can use the part for a cryogenic pressure vessel that experiences temperature swings form +120 to LH2 temperature
- Potential issues
  - The properties of the resin can be affected from the cure
    - A higher cure temperature may not affect porosity but can change the polymer cross-linking, leading to new material response
    - Perform a DMA, which shoes that Tg of 200ºC instead of 145ºC
  - Can introduce material embrittlement and increased stiffness
    - After DMA, we know that the resin has over cured, making it more brittle
    - In cryogenic conditions the material may crack under stress due to a decreased failure strain
    - The modulus will increase on the structure, which could alter how the part handles different loading conditions
    - The tank needs to be sealed and not allow permeability (we can have no cracking or microcracking)
- How to verify
  - Recommend thermal cycling tests
    - Conduct thermal cycling on test samples to determine if the material properties have changed
    - Check for changes in stiffness, strength, and reliability over multiple temperature cycles
  - Perform microscopy analysis
    - Perform examinations before and after thermal cycling to check for microcracking in the fiber matrix
    - The part needs to be sealed, this is very important

#15

- Three methods used to estimate void content in a composite material
  - Acid digestion
    - This method includes dissolving the resin matrix using an acid to isolate fibers
    - The void content is then estimated by comparing the expected fiber volume fraction to the measured volume
      - Good for fiber volume fraction analysis, but is destructive
  - Real density vs. bulk density (Density measurements)
    - Measure and compare the real density to the bulk density to find the void content
  - Image analysis cross section (Optical or electron microscopy)
    - Involves cutting and polishing a cross section of the composite structure and analyzing the voids using microscopy
    - Voids appear as dark region sin the composite and can be easily calculated

#16

- Find E_{11} E_{22} E_{33} v_{12} G_{12}

Table 10.2 Elastic constants of a transversely isotropic composite in terms of component constants (matrix isotropic, fiber anisotropic)

Longitudinal modulus	$E_{11} = E_{f1}V_f + E_mV_m$
Transverse modulus	$E_{22} = E_{33} = \dfrac{E_m}{1 - \sqrt{V_f}(1 - E_m/E_{f2})}$
Shear modulus	$G_{12} = G_{13} = \dfrac{G_m}{1 - \sqrt{V_f}(1 - G_m/G_{f12})}$
Shear modulus	$G_{23} = \dfrac{G_m}{1 - \sqrt{V_f}(1 - G_m/G_{f23})}$
Poisson's ratio	$v_{12} = v_{13} = v_{f12}V_f + v_mV_m$
Poisson's ratio	$v_{23} = \dfrac{E_{22}}{2G_{23}} - 1$

Source: Supplementary Lecture Notes

#16

- Find E_{11} E_{22} E_{33} v_{12} G_{12}
- Estimating these properties earlier on in the design process can allow for verification of correct materials
- Estimation allows for optimization and understanding
- Designers can use this preliminary information and begin FEA
- Knowing what you are getting can save time and costs
  - Can estimate how much material you will need without wasting material or money
- Preliminary information can help with manufacturing consideration before you have the material in your possession

Assume Isotropic

60% unidirectional lamina (V_f) — IM7 fiber
3% void content (V_v) — 8552 epoxy resin

$E_m = 0.7$ Msi
$\nu_m = 0.4$ @ 70°F
$E_f = 40$ Msi
$\nu_f = 0.35$

$V_m = 1 - V_f - V_v$
$V_m = 1 - 0.6 - 0.03$
$V_m = 0.37$

$E_{11} = ?$ $E_{22} = ?$ $E_{33} = ?$
$\nu_{12} = ?$ $G_{12} = ?$

$E_{11} = V_f E_f + V_m E_m$
$(0.60)(40) + (0.37)(0.7) = 24.26$ Msi

Found table online

$E_{22} = E_{33}$

$E_{22} = \frac{E_m}{1 - \sqrt{V_f}(1 - E_m/E_f)} = \frac{0.7}{1 - \sqrt{0.6}(1 - 0.7/40)}$

$E_{22} = E_{33} = 2.93$ Msi

$\nu_{12} = \nu_f V_f + \nu_m V_m$
$(0.35)(0.6) + (0.4)(0.37) = 0.358$

$G_{12} = \frac{G_m}{1 - \sqrt{V_f}(1 - G_m/G_f)}$

$G_m = \frac{E_m}{2(1+\nu_m)} = \frac{0.7}{2(1+0.4)} = 0.25$ Msi

$G_f = \frac{E_f}{2(1+\nu_f)} = \frac{40}{2(1+0.35)} = 14.81$ Msi

$G_{12} = \frac{0.25}{1 - \sqrt{.6}(1 - 0.25/14.81)} = 1.048$ Msi