Production Control Effect on Composite Material Quality and Stability

John Tomblin, Yeow Ng, Beth Clarkson, Allison Crockett

The Joint Advanced Materials and Structures Center of Excellence

Production Control Effect on Composite Material Quality and Stability

- Motivation and Key Issues
  - Quality control tests on prepreg or lamina (i.e. receiving inspection or acceptance tests) may not always detect material defects
  - Material suppliers (e.g. fiber, resin, prepregger, etc.) and part fabricators need to have an understanding of each others' roles and responsibilities
- Objectives
  - Develop essential information on the nature of the controls required at various producer levels to assure the continuation of stable and reliable composite raw material for aerospace usage
    - Develop and clarify requirements
  - Provide guidance to NASA's National Center for Advance Materials Performance (NCAMP)
- Approach
  - Develop production control guidelines
  - Use NCAMP as the proofing ground
  - Document what works and what doesn't
  - Define "aerospace grade"

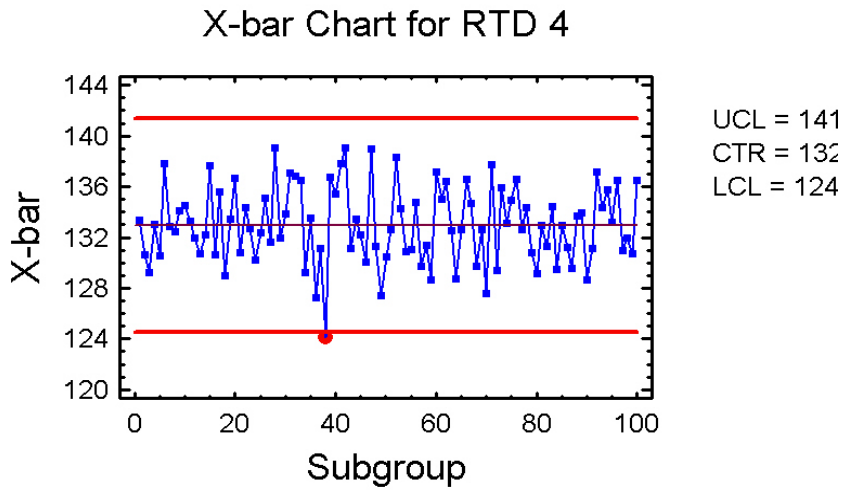
FAA Sponsored Project Information

- Principal Investigators & Researchers
  - John Tomblin, Yeow Ng, Beth Clarkson, Allison Crockett
- FAA Technical Monitor
  - Curtis Davies
- Other FAA Personnel Involved
  - Larry Ilcewicz, Peter Shyprykevich (retired), Lester Cheng, Evangelina Kostopoulos, and David Ostrodka
- Industry Participation
  - 40 Aircraft Companies & Tier-1 Suppliers
  - 16 Material Suppliers
- Other Partners
  - CMH-17 (formerly MIL-17), SAE P-17, SAE PRI Nadcap, SAE PRI QPL, ASTM D30
  - University of British Columbia and Center for Nondestructive Evaluation at Iowa State University

Material Key Characteristics (KC) and Key Process Parameters (KPP) Monitoring

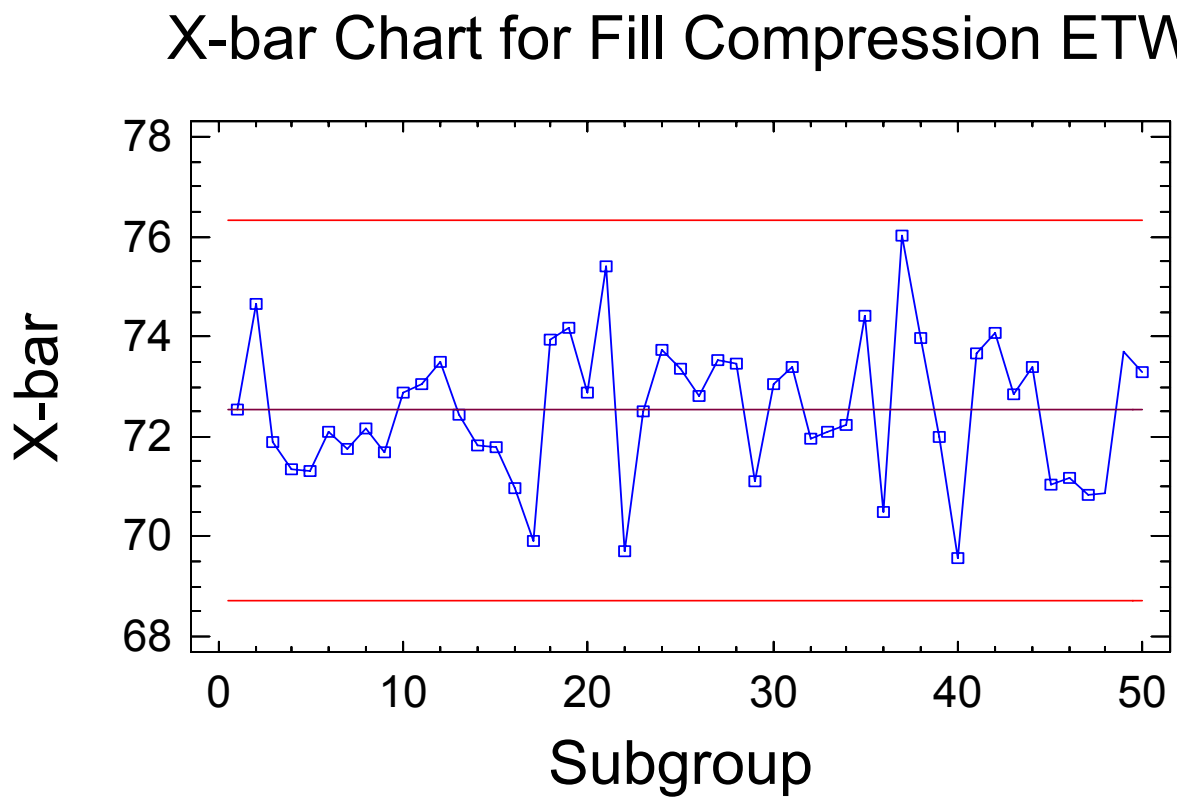
- Developed protocols to monitor KC and KPP variations over time

Control Chart



- Everything varies at least a little bit. So how do you tell when you are just experiencing normal variation versus when something out of the ordinary is occurring? Control charts were designed to make that distinction
- As long as all points lie inside the upper and lower control limits, the variation is presumed to be normal or a common cause variation. When a data point falls outside those limits, it's time to look for a reason for the variation
- Two-sided monitoring for all properties including strength

Example Control Chart

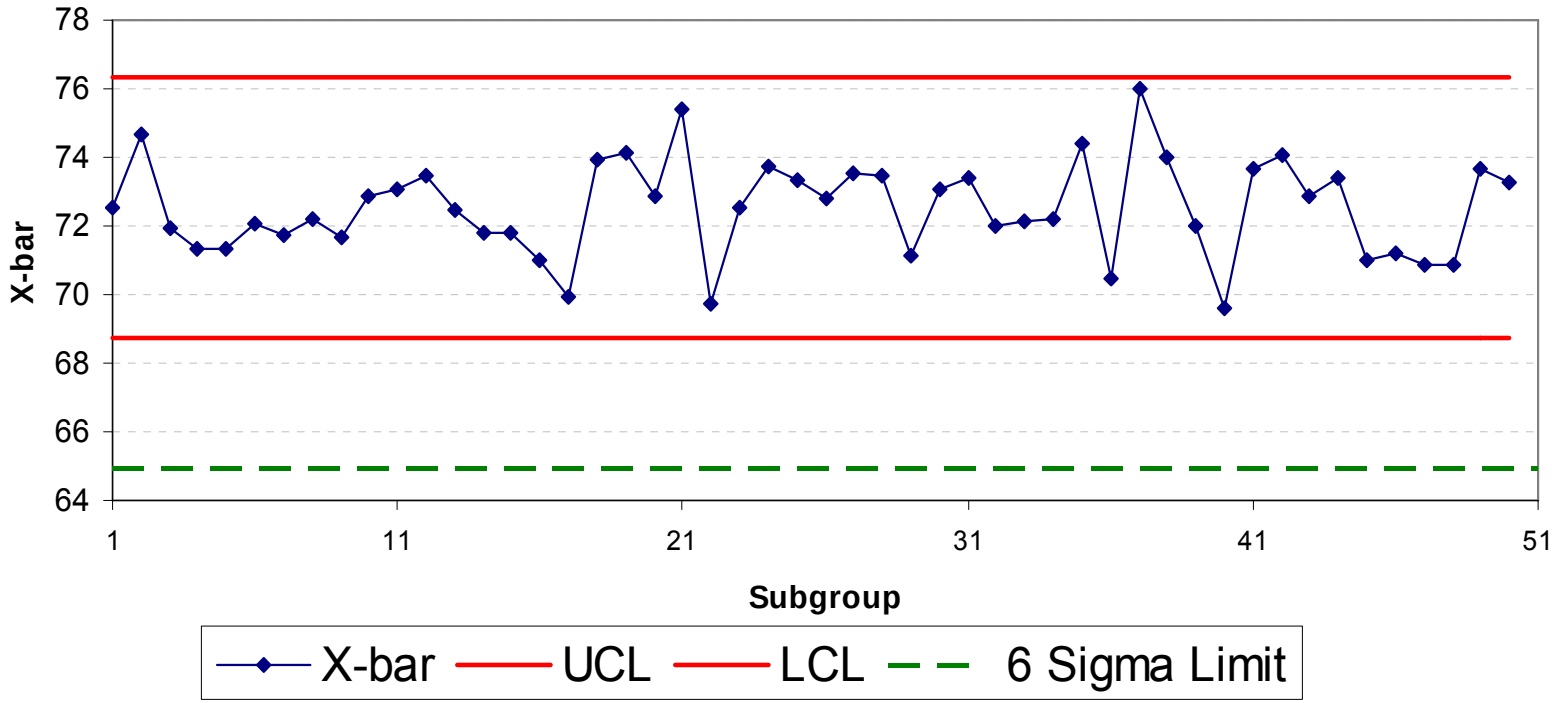


UCL = 76.34
CTR = 72.53
LCL = 68.72

Advantages of Control Charts

- Ideally the control limits lie inside the spec limits.
- Then the manufacturer can respond to the any out-of-control condition PRIOR to any out-of-spec product being produced.

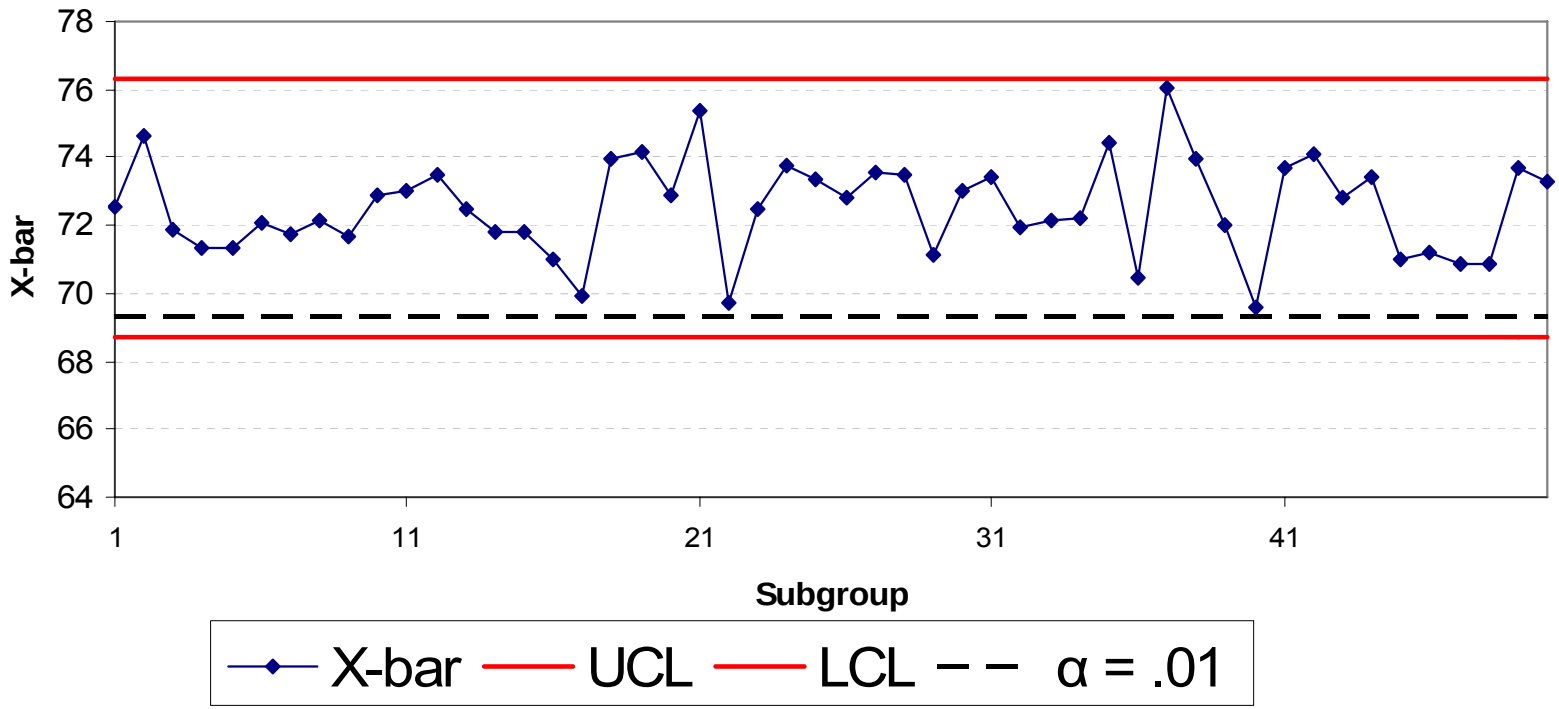
X-Bar Chart for Fill Compression ETW with 6 sigma spec limit added



Control Limits & Acceptance Criteria

- Acceptance Criteria is set such that the producer's risk is 0.01 (per DOT/FAA/AR-03/19)
- Control limits are set at ±3 standard deviations, corresponding to producer's risk of 0.003
- Thus, the acceptance limit is set tighter than the traditional control chart limits.
  - The use of modified CV (larger than measured CV) to establish specification limits may minimize this problem (i.e. reduce false alarm rate)
  - Retest may minimize the problem (but retest is not typically used in SPC)
  - **Should producer's risk be reduced from 0.01 to 0.001?**
  - More industry experience is needed to devise a solution!

X-Bar Chart for Fill Compression ETW with acceptance limit added



Investigate the Applicability of Western Electric Rules

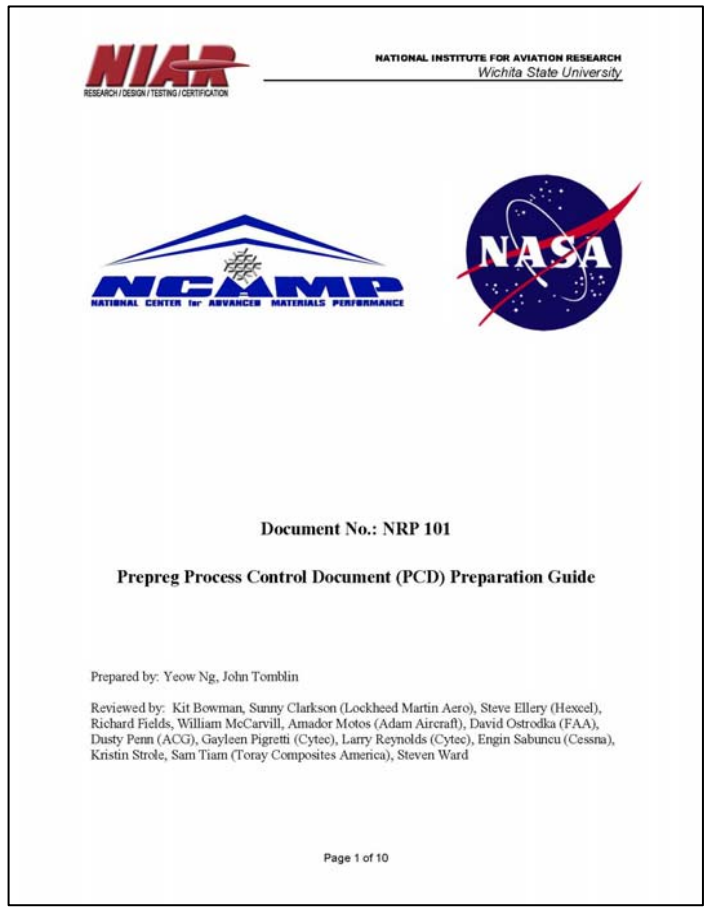
- **Rule 1: A single point falls on or outside the 3 sigma limit.**
- **Rule 2: Two out of three consecutive points fall in zone A or beyond—all on the same side of the center line.**
- **Rule 3: Four out of five consecutive points fall in zone B or beyond—all on the same side of the center line.**
- **Rule 4: Eight consecutive points fall in zone C or beyond—on the same side of the center line.**
- **Rule 5: A long series of points (about 14) are high, low, high, low, without any interruption in the sequence. (Systematic variables.)**

Investigate the Applicability of Western Electric Rules

- **Rule 6: Seven consecutive points all increasing or all decreasing. (monotonically is a stricter requirement)**
- **Rule 7: Fifteen or more consecutive points fall in zone C either above or below the centerline. (Stratification)**
- **Rule 8: Eight consecutive points fall on both sides of the centerline, with none falling in zone C. (Mixture pattern)**

Prepreg PCD Preparation Guide

- A guide for prepreg material suppliers to prepare a prepreg process control document (PCD)
- A prepreg PCD is a "recipe" for the production of prepreg
  - Defines qualified raw materials, equipment, production parameters, etc.
  - HIGHLY PROPRIETARY



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Wichita State University

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Prepreg Process Control Document (PCD) Preparation Guide

Prepared by: Yeow Ng, John Tomblin

Reviewed by: Kit Bowman, Sunny Clarkson (Lockheed Martin Aero), Steve Ellery (Hexcel), Richard Fields, William McCarvill, Amador Motos (Adam Aircraft), David Ostrodka (FAA), Dusty Penn (ACG), Gayleen Pigretti (Cytec), Larry Reynolds (Cytec), Engin Sabuncu (Cessna), Kristin Strole, Sam Tiam (Toray Composites America), Steven Ward

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Prepreg PCD Preparation Guide

- PCD is used to control the prepreg material properties and to manage changes
- Reviewed by the FAA, CMH-17, SAE P-17, material suppliers and major aircraft companies
- *Matured* guidelines will be incorporated into the final report of this project

Co-efficient of Variation (CV)

- CV is the standard deviation divided by the mean.

$$CV = \frac{S}{\bar{x}}$$

  - If CV is erroneously high – we will accept too many “bad” materials
  - If CV is erroneously low – we will reject too many “good” materials

Modified C.V. Approach

- The variation inherent to all processes can be partitioned into several sources. Two potential sources of variation that are NOT included in the qualification sample:
  - Variation due to the testing process; all samples are tested within a short period of time *(unrelated to material property variation)*
  - Variation due to the prepreg production process; all 3 or 5 batches of qualification prepreg are produced within a short period of time *(related to material property variation)*

- Modified CV is used to compensate for variation not captured in material qualification process

Effects of Modified CV

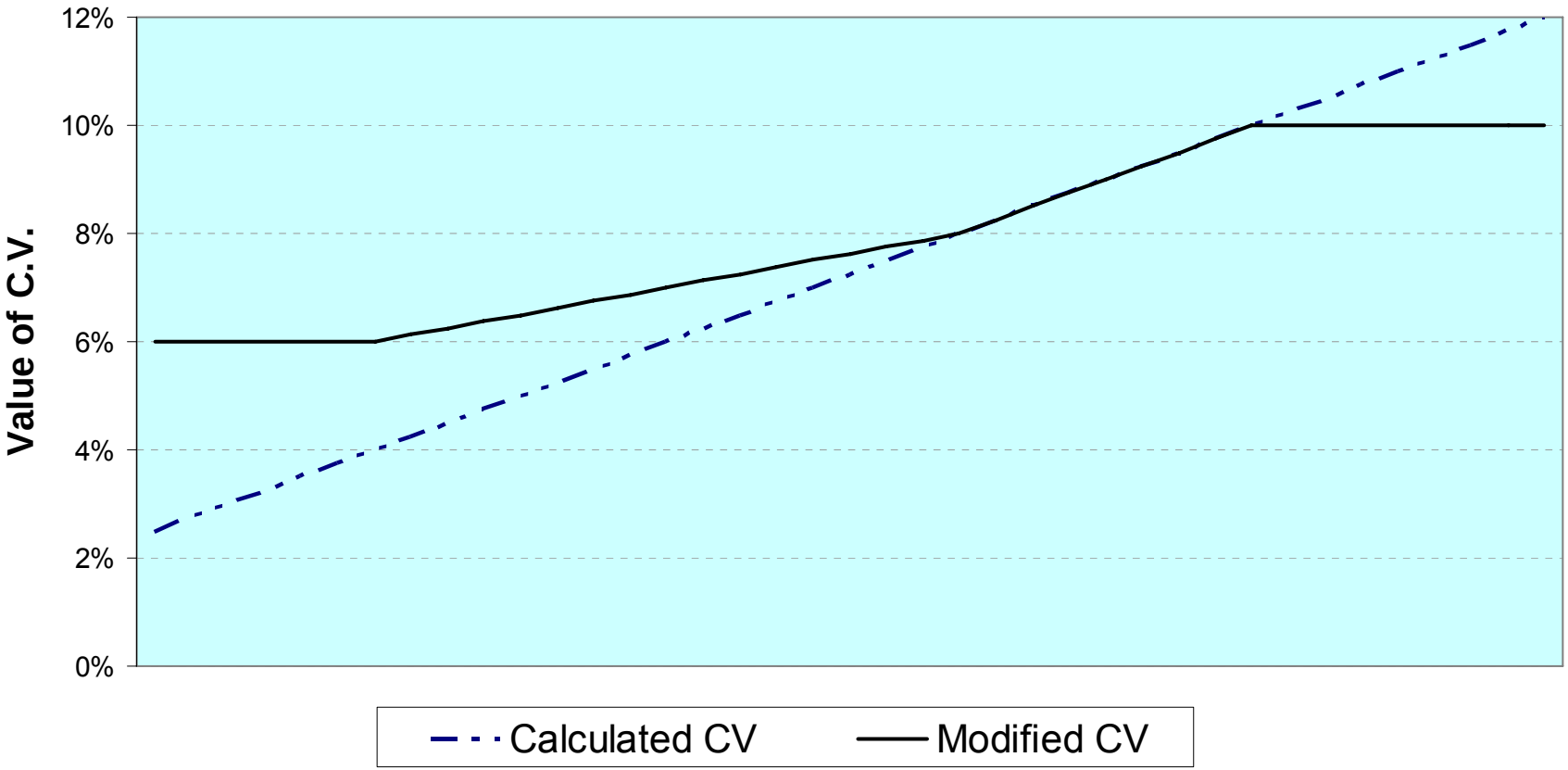
- In order to compensate for the additional variation, the CV is increased PRIOR to computing the A and B basis values.

- The effect of this will be:
  - Slight decrease in the computed A-basis and B-basis values, thereby increasing the margin of safety when designers use those values.
  - Slight decrease in pass/fail limits, thereby decreasing the number of failures during equivalence and material acceptance.

CV Modification Formula

- If CV < 4%:
  - Modified CV = 6%
- If CV is between 4% and 8%, adjust upward according to the following formula:
  - Modified CV = ½ CV + 4%
- If CV is between 8% and 10%:
  - Modified CV = CV
- If CV > 10%:
  - Modified CV = 10%

Comparison of CVs



Investigate the Effects of Modified CV Approach

- Currently approved for short-term use only
- What are the effects of long-term use?
  - Desirable or undesirable?
  - What are the implications on material property control?
  - What are the implications on building block approach?
  - What if material suppliers & users agree to use modified CV forever?
- Need balance between tolerable level of rejection rate and acceptable level of quality

Prepreg Aging Study

- Age may affect producibility and part quality
- Modern parts are larger and require longer handling life and staging life
- A reliable and simple method of measuring prepreg "age" is highly desirable

Prepreg Aging Study

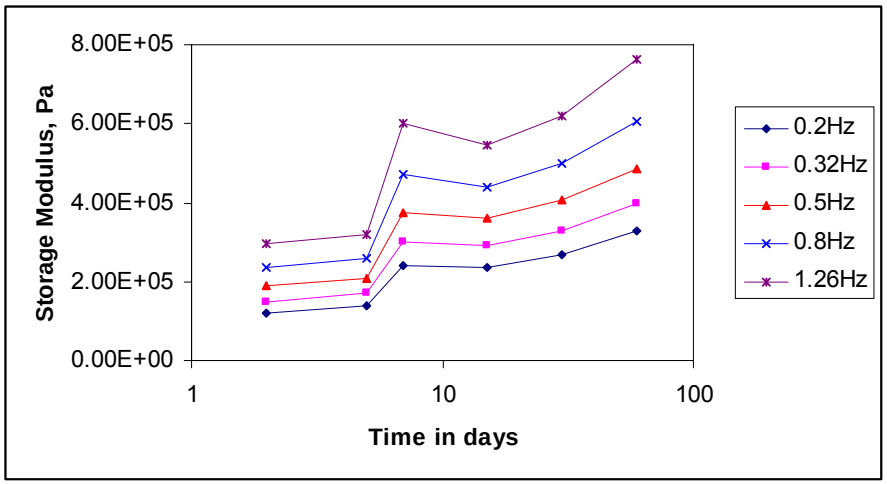
- Collaboration with University of British Columbia, Toray Composites America, Avpro, and Center for Nondestructive Evaluation at Iowa State University
- Prepreg aged (exposed to out-time) at Wichita State University and sent to collaborating partners in dry ice
- PLAN: Incorporate the effects of aging into process modeling

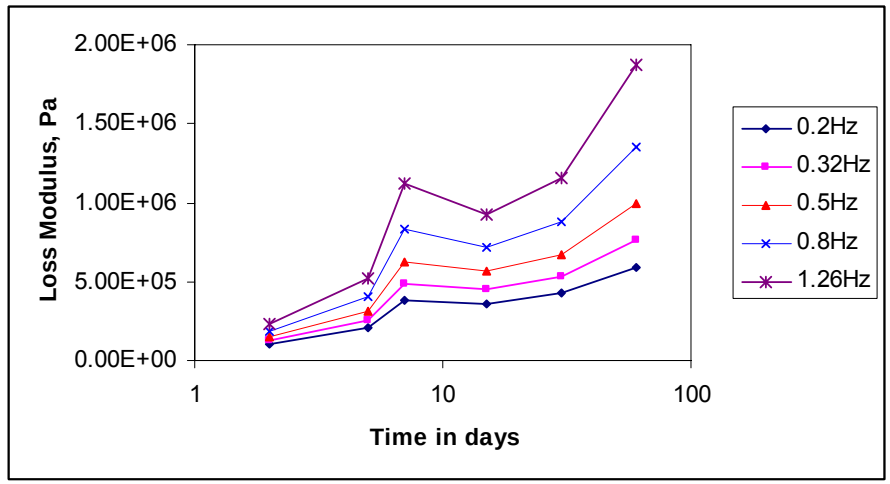
Aged prepregs were sent to participating members in dry ice (Prepreg: Toray T700SC 2510 PW)

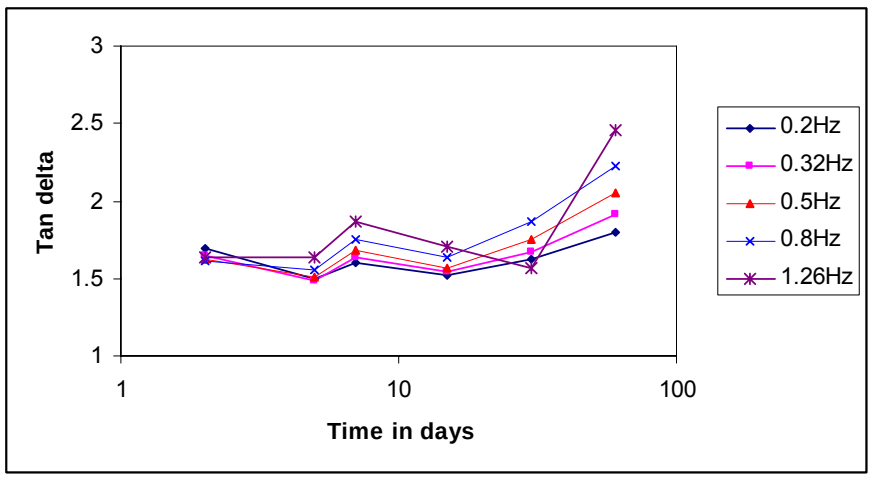
Freezer Storage Time <10°F	Out Time at 70° ± 10°F							
	< 2 day	3 days	5 days	7 days	10 days	12 days	15 days	18 days (Note 1)
See Note 2	✓ ??/1	✓ ??/3	✓ ??/5	✓ ??/7	✓ ??/10	✓ ??/12	✓ ??/15	✓ ??/21

Property	Method/Condition	# Replicates	Members
Short Beam Strength	ASTM D 2344-00, RTD	8	NCAMP
Gel Time	ASTM D3532-99	3	Toray
Tack	NCAMP Test Plan	3	NCAMP
Drape	NCAMP Test Plan	3	NCAMP
HPLC	SACMA SRM 20R-94	2	Toray
DSC	SACMA SRM 25R-94	2	Toray
Photomicrography and void content (and C-scan)	MIL-HDBK-17-1F, sec 6.6.7.3	As needed	NCAMP
Photoacoustic Infrared Spectrum	CNDE Procedures	As needed	CNDE
Tests related to process modeling	As needed	As needed	UBC
APA 2000 or similar tests	As needed	As needed	Avpro

Rheometric Dynamic Analyzer (RDA-III), Frequency Sweep

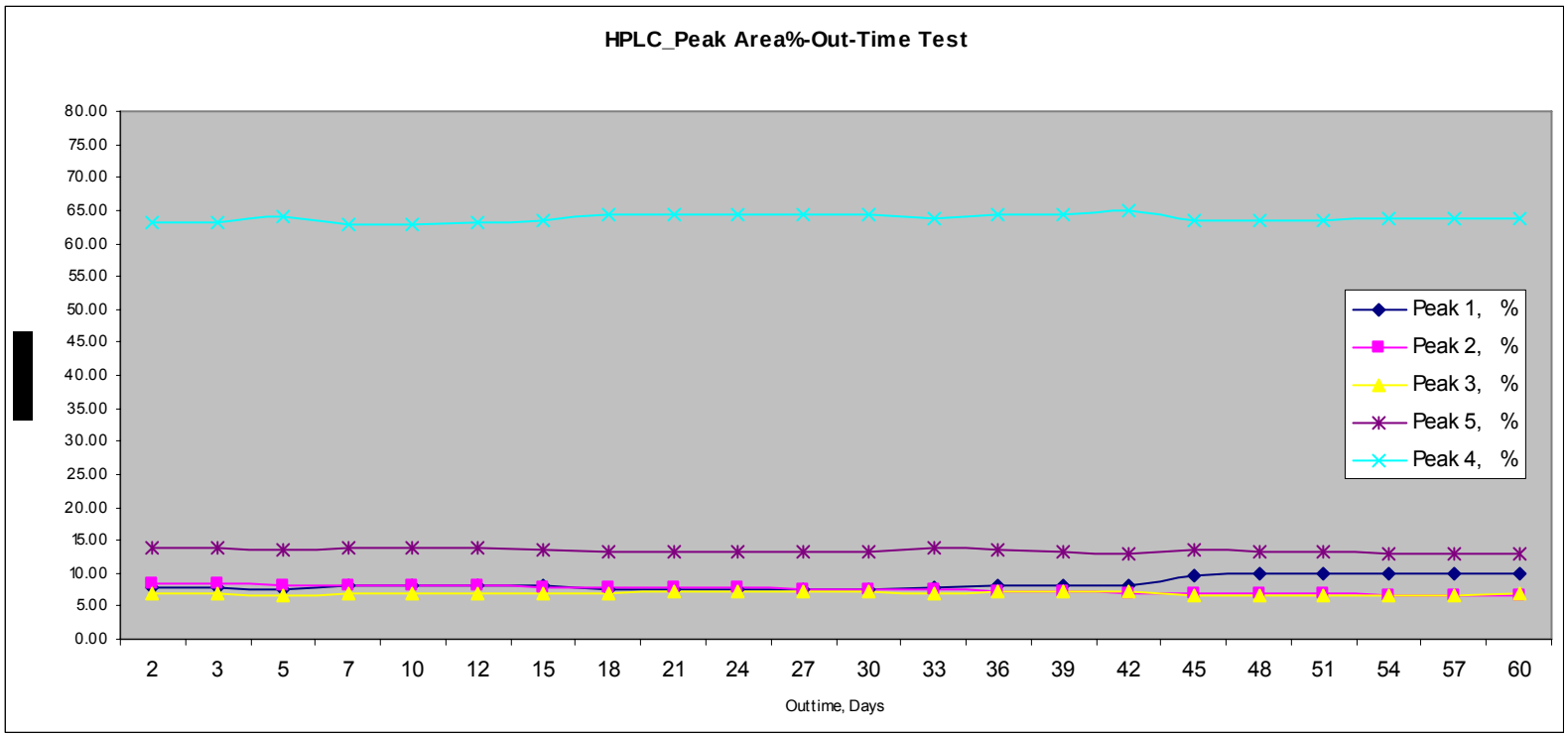






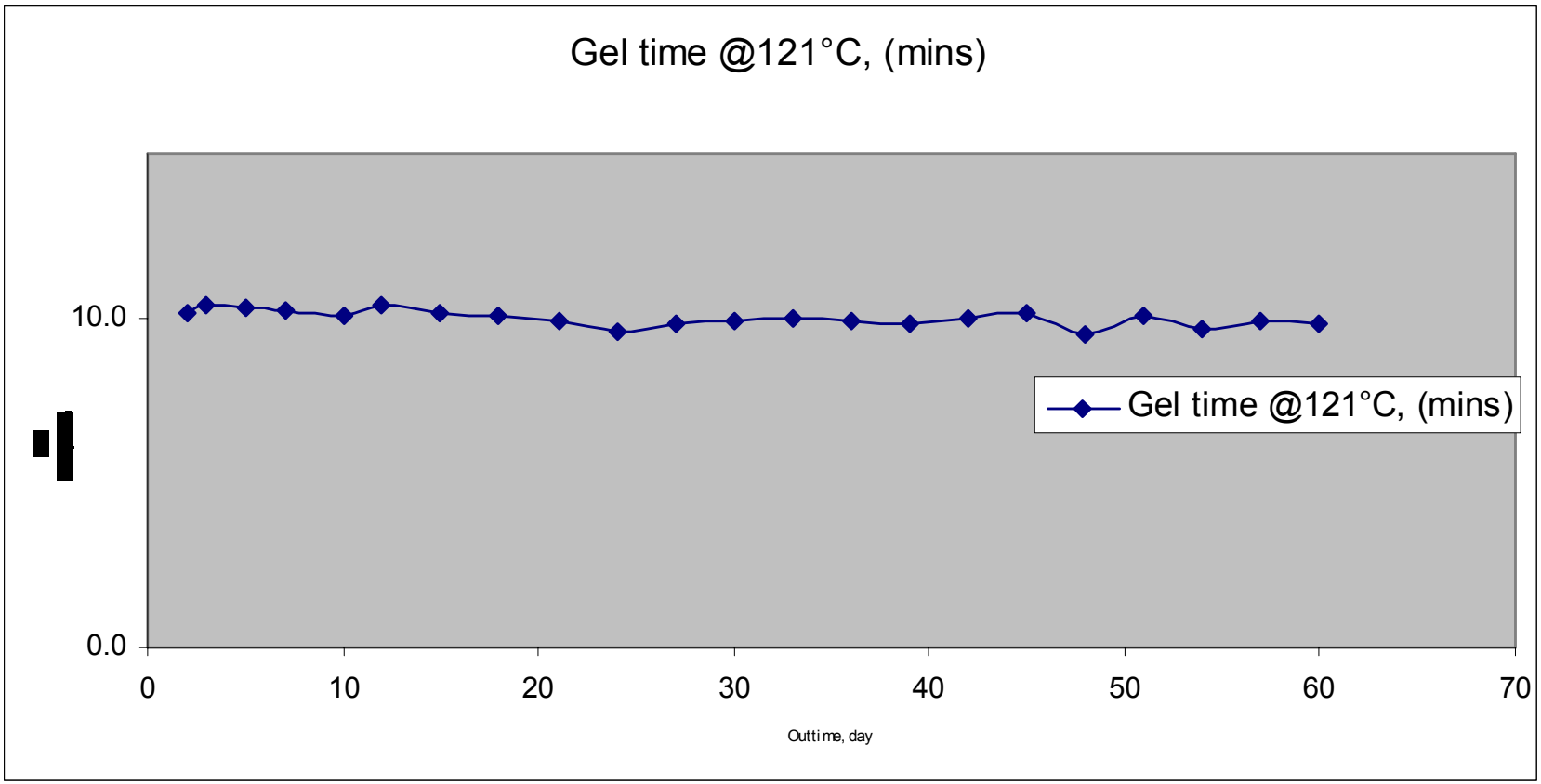
- Experiment performed by Avpro (Tom Rose)

HPLC per T700SC-12K-50C/#2510, F6273C-07M Specification



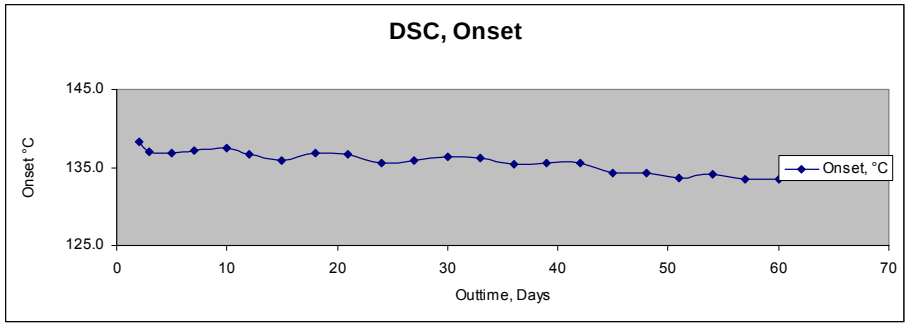
- Experiment performed by Toray Composites America

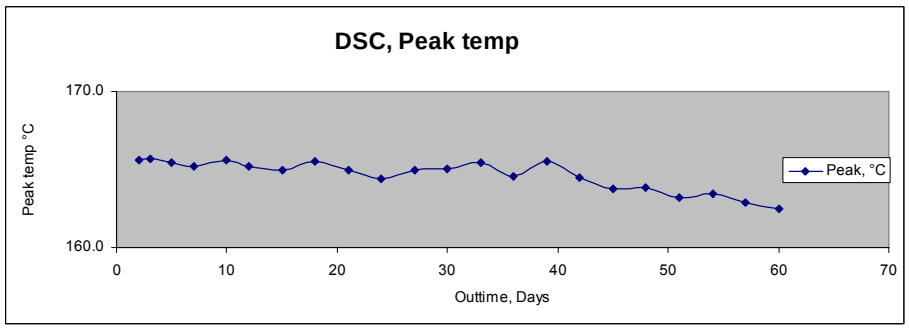
Gel Time per T700SC-12K-50C/#2510, F6273C-07M Specification

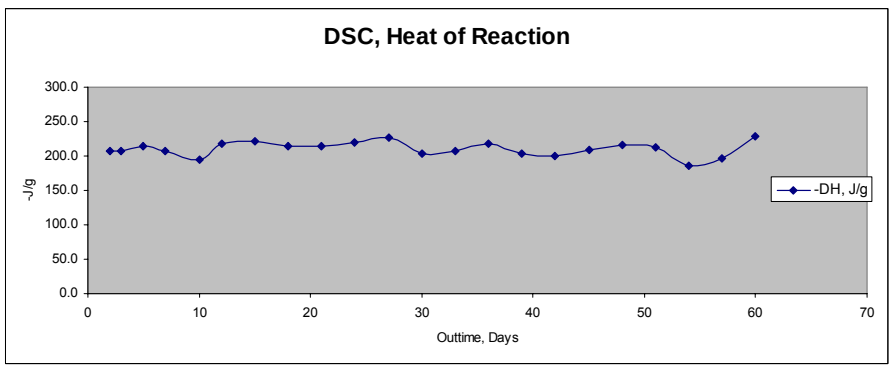


- Experiment performed by Toray Composites America

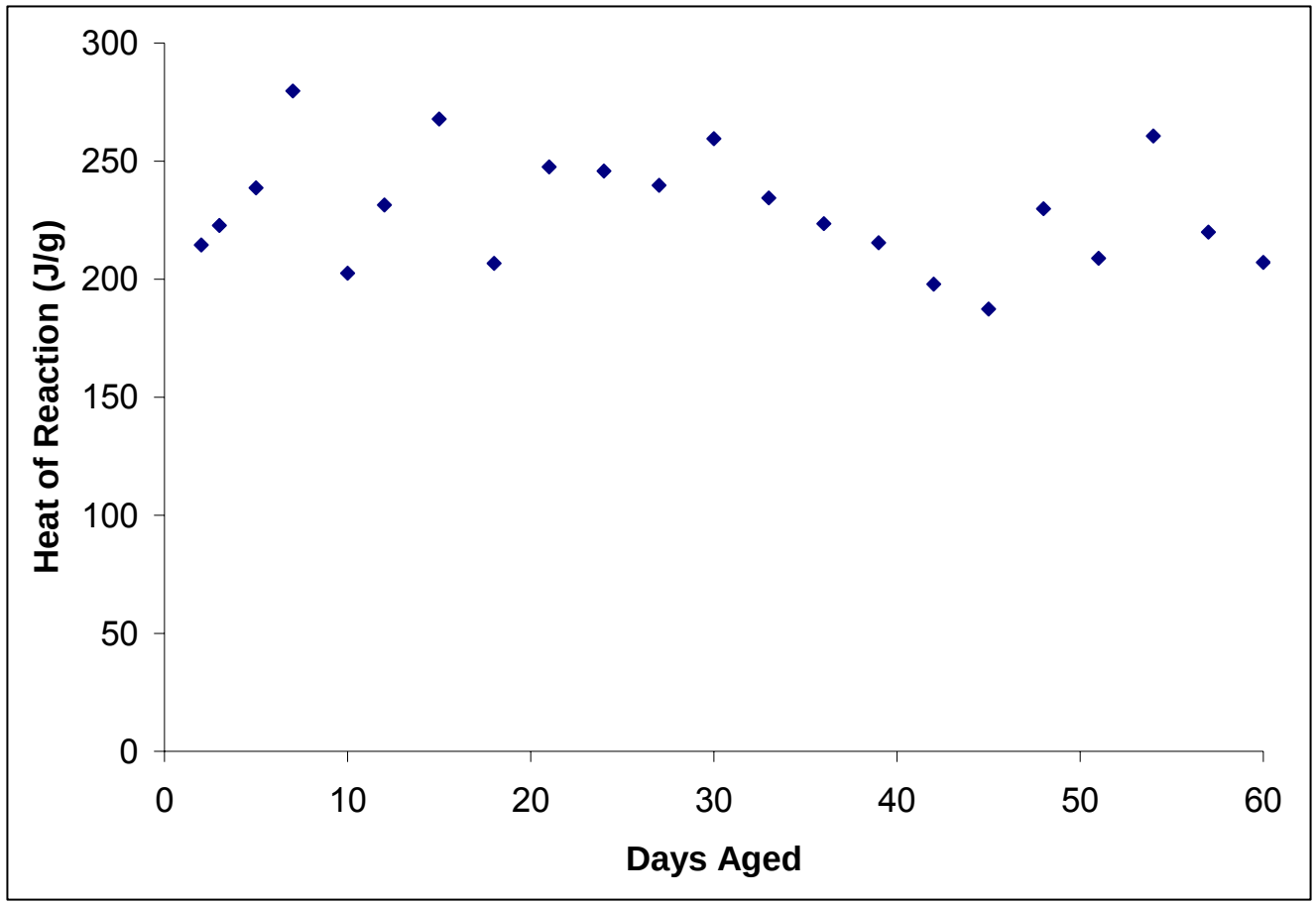
DSC per T700SC-12K-50C/#2510, F6273C-07M Specification





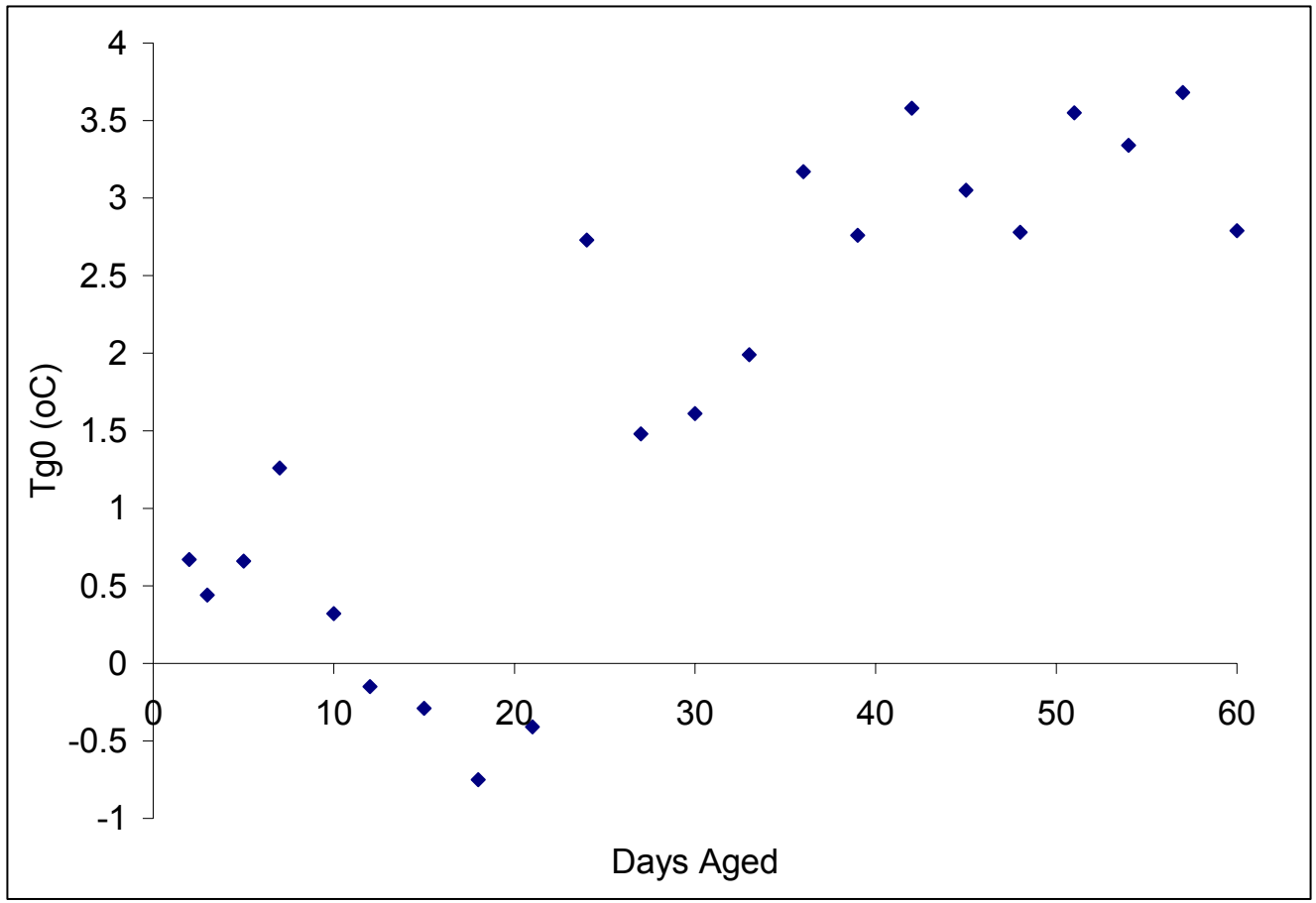


Differential Scanning Calorimetry (DSC) Analysis of Resin



- Experiment performed by UBC

Differential Scanning Calorimetry (DSC) Analysis of Resin



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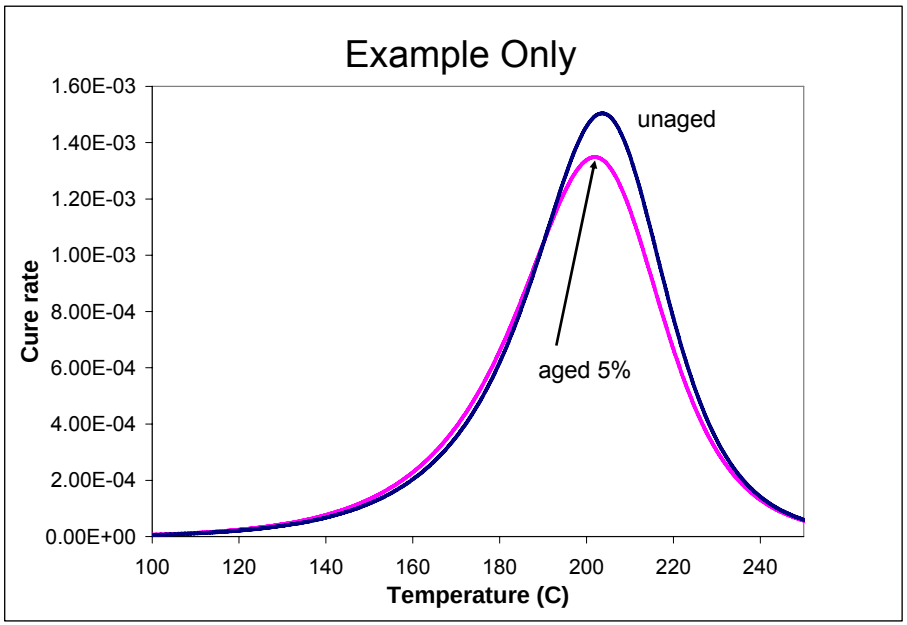
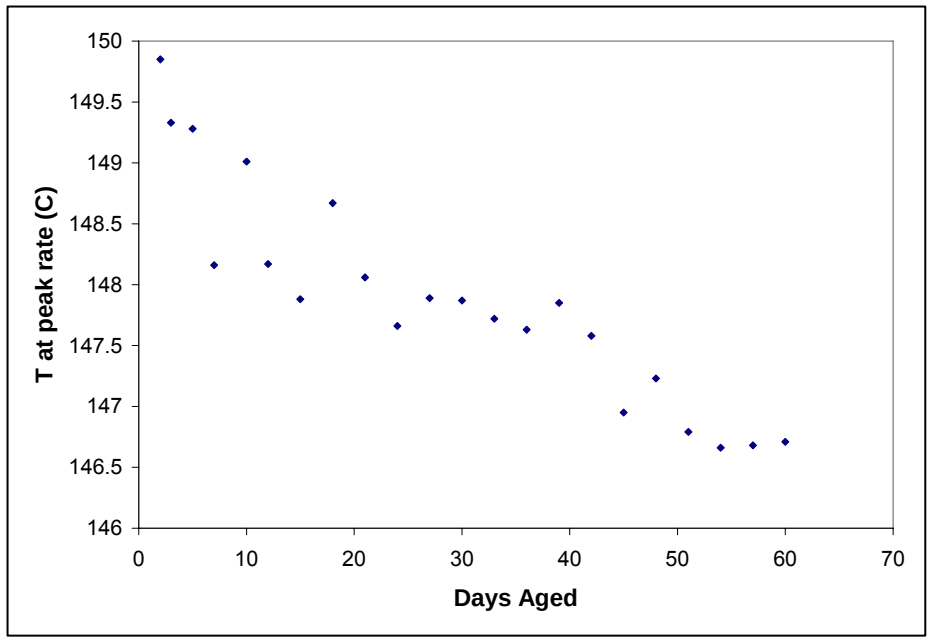
Differential Scanning Calorimetry (DSC) Analysis of Resin

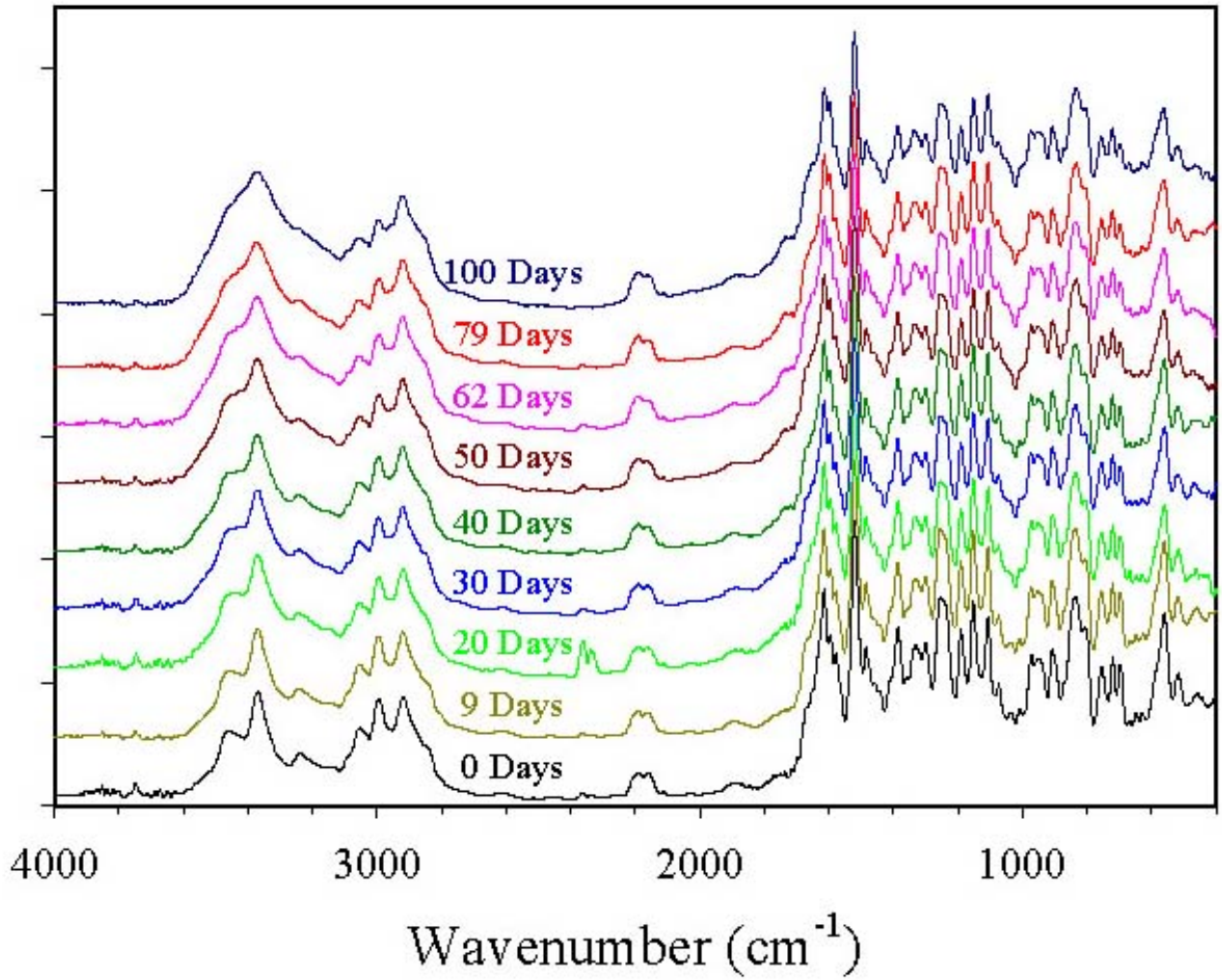




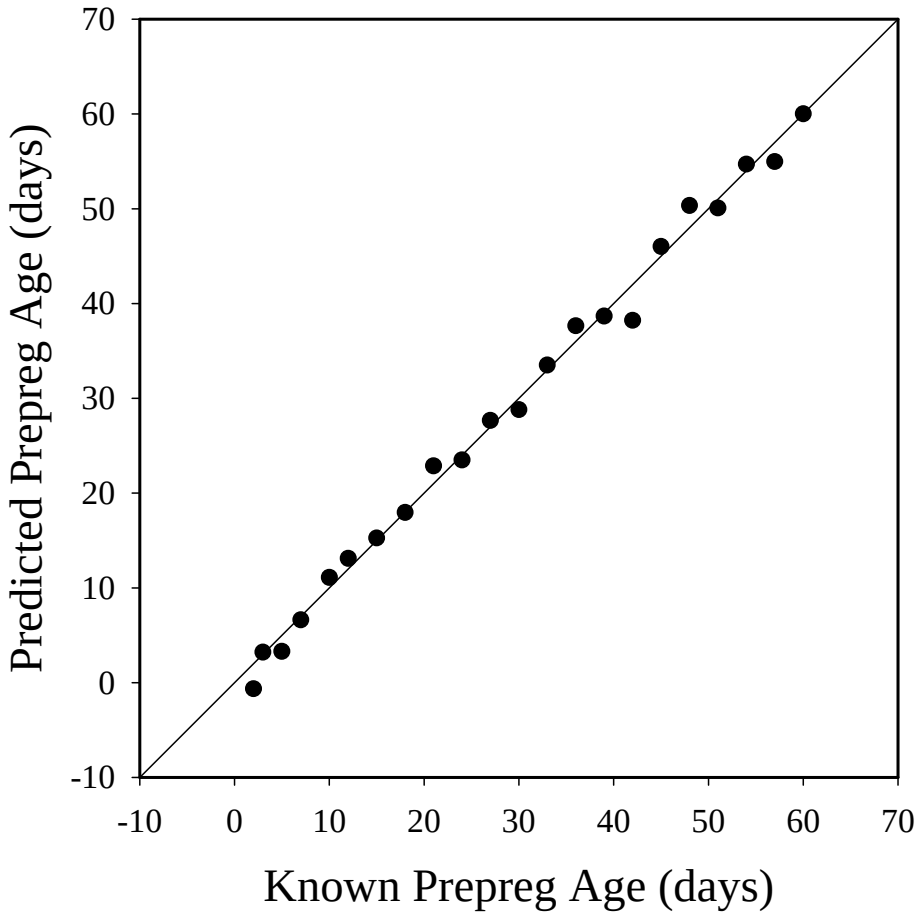
Experiment performed by UBC

Photoacoustic Infrared Spectroscopy Study of Out-Life Aging

John McClelland and Roger Jones
Center for Nondestructive Evaluation, Iowa State University



PAS Prediction



After the 33rd day, the specimens had become stiffer and lost some of its tackiness

Infrared Photoacoustic Spectroscopy (PAS) Technology

- Infrared spectroscopy is a proven technique to measure aging of epoxy prepreg, but is too tedious for routine use
- PAS can analyze the intact prepreg, allowing virtually all sample preparation to be skipped
- PAS works by sensing the infrared energy absorbed in a sample. The sample is sealed in a chamber containing a microphone. Infrared radiation shines on the sample. If the sample absorbs the radiation, its temperature and pressure oscillate, which the microphone detects as sound waves.

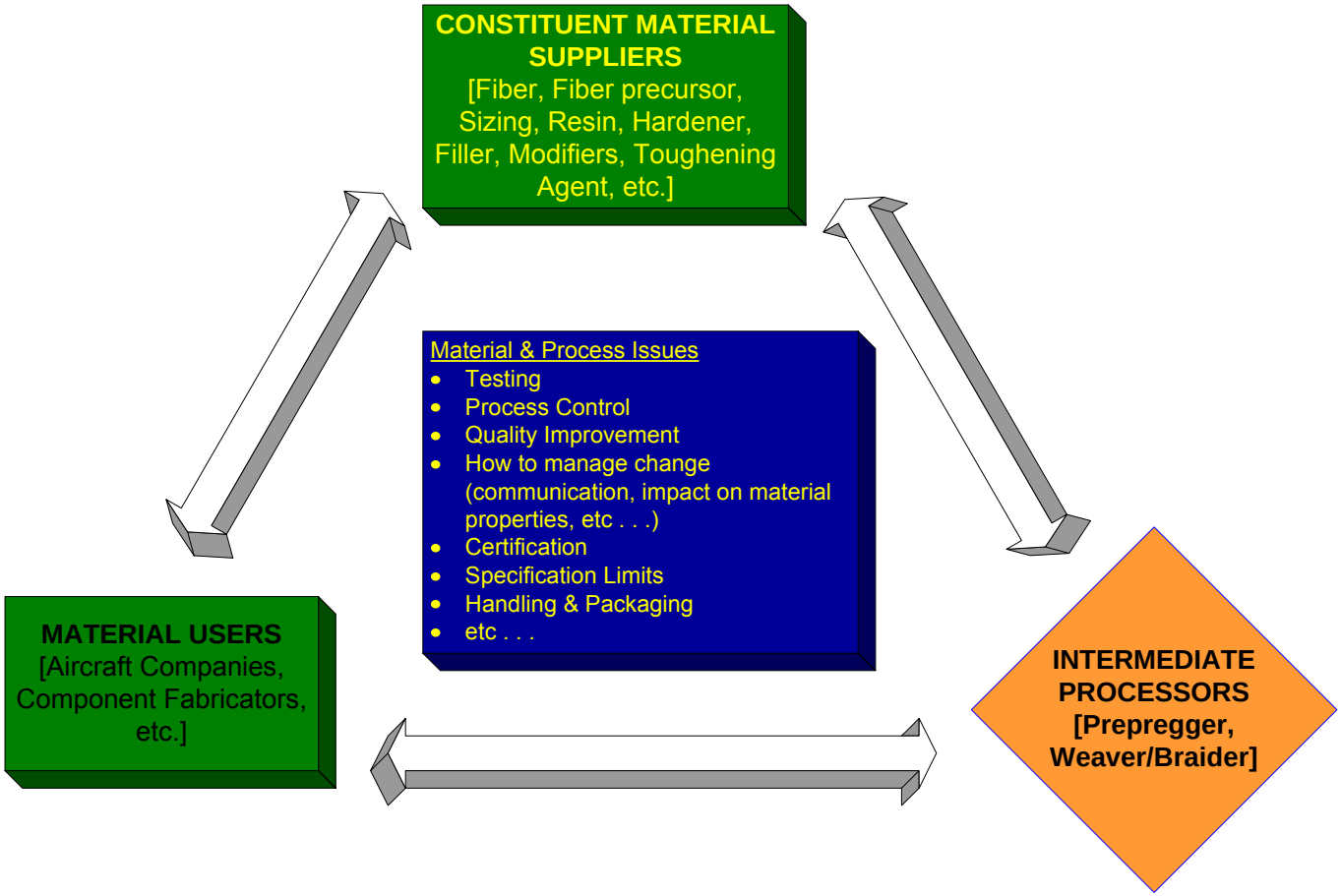
Prepreg Aging Study Conclusion

- PAS is the most sensitive technique for measuring prepreg out-time
- Other techniques are not very reliable; so aging of prepreg cannot (or need not) be incorporated into process modeling
- PAS is easy to perform and conducive to shop environment
- More research is needed to validate PAS; need to take into account the effects of humidity

Documentation of practices and lesson's learned in NCAMP

- NCAMP offers a practical venue to try things out
- Document NCAMP experiences in the FAA report
  - What works and what doesn't
  - What's required
- The roles and functions of industry organizations
  - CMH-17, SAE PRI, SAE Nadcap, SAE P-17, ASTM D30, NCAMP, etc.
- The roles and functions of material users and various levels of material suppliers

Understanding Each Others' Roles, Requirements, and Responsibilities



Reduce duplicate efforts while maintaining sufficient checks and balances

How all of these fit together?

Industry Organizations (CMH-17, SAE, Nadcap, PRI, NCAMP, ASTM D30)

Material Specifications

Audit

Process Specifications

Quality Control

Material Change Management

Accreditations

Regulatory Agencies

Material Qualifications

Processing Tolerance Limits

Process Control Documents

Material Compatibility

Qualified Products Listing


AIRBUS
Piper
Freedom of flight
Cessna
A Textron Company
BOMBARDIER
MAGELLAN
AEROSPACE CORPORATION
BRISTOL AEROSPACE LIMITED
TohoTenax
Beechcraft
Jawker
COMTEK
Advanced Structures
THE NORDAM GROUP
HEXCEL
TOYOTA
J.D. Lincoln Inc.
The Leader In Composite Material Technology
LOCKHEED MARTIN
We never forget who we're working for™
ARL
Gulfstream
ATK
ATK SPACE SYSTEMS
Charting the Next Century of Flight
FEDERAL AVIATION ADMINISTRATION
Pratt & Whitney
A United Technologies Company
GKN AEROSPACE
CMH-17
COMPOSITE MATERIALS HANDBOOK
EXCELNET
CTRM Excelnet
Sdn Bhd
SPIRIT
AEROSYSTEMS
nelcote™
CYTEC
Technology ahead of its time™
COLUMBIA
BOEING
SUBARU
Fuji Heavy Industries
Advanced Composites Group
'TORAY'
AAR
aarcorp.com
NCAMP
AIR FORCE RESEARCH LABORATORY
CIRRUS
Bell Helicopter
A Textron Company
Henkel
Brand like a Friend
IAI
ISRAEL AIRCRAFT INDUSTRIES
GOODRICH
SAE International
NORTHROP GRUMMAN
NEWPORT
ADHESIVES and COMPOSITES, Inc.
Nadcap
MITSUBISHI HEAVY INDUSTRIES, LTD.
SCALED
COMPOSITES
TENCATE
materials that make a difference
GENERAL ATOMICS
AND AFFILIATED COMPANIES
Sikorsky
A United Technologies Company
ASTM
INTERNATIONAL
Standards Worldwide
PRI
kaneka
Vought
Aircraft Industries, Inc.
northwest
composites
centre
Liberty
AEROSPACE
Kawasaki
Kawasaki Heavy Industries
NAVAIR
ADAM
AIRCRAFT
Lewcott
JAXA
Japan Aerospace Exploration Agency
Rocky Mountain Composites

A Look Forward

- Benefits to Aviation
  - Provides solution and guidance to the industry
  - Documents lessons learned
  - Ensures a supply of composite materials with stable properties
- Future needs
  - Continue to work with the industry on material & process issues
    - Develop other essential guidance documents such as Carbon Fiber PCD Preparation Guide