A WEBINAR ON:

RESIN BEHAVIOUR DURING PROCESSING:

Key Resin Properties to Consider When Developing a Manufacturing Process

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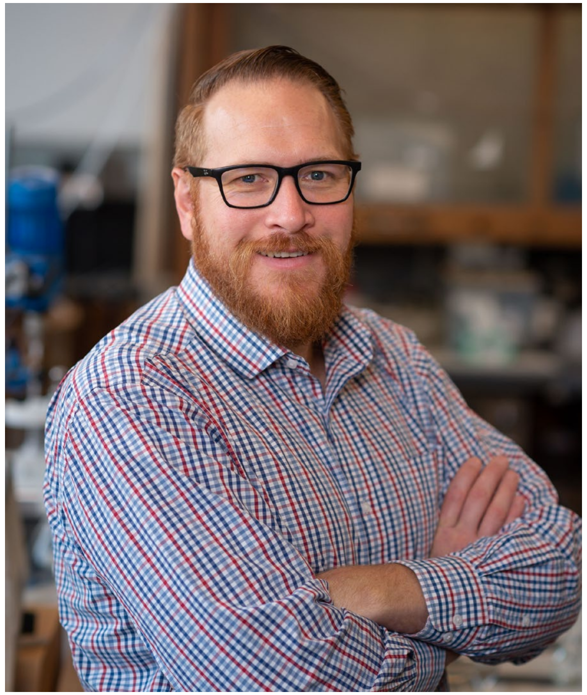

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Co-Director of Advanced Materials Manufacturing MEL Program, UBC
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- Ph.D. and M.A.Sc. in Composite Materials Engineering
- Over 15 years experience in industry and academia working on polymer matrix composites in aerospace, automotive, marine, energy, recreation and others
- Experience working with over 150 companies from SME to major international corporations
- Expertise in liquid composite moulding and thermal management

YOUR HOSTS

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- Ph.D. and M.A.Sc. in Chemical Engineering
- Awarded materials scientist – Rio Tinto Alcan award for academic research excellences
- Over 30 publications, conference presentations and patents in the field of polymer matrix composites
- Experience working with over 80 SMEs and international companies to develop new manufacturing processes and products

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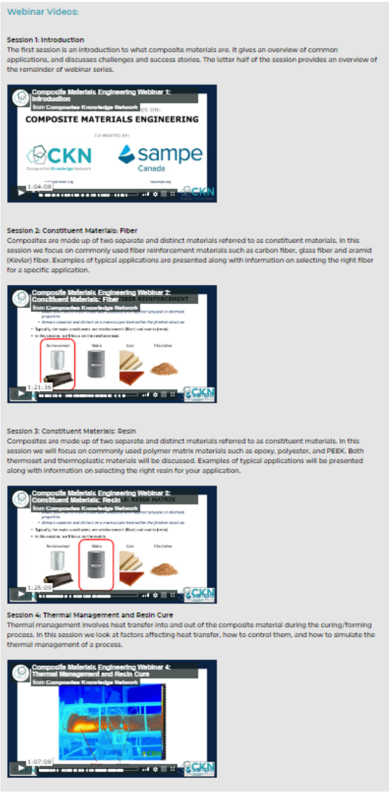

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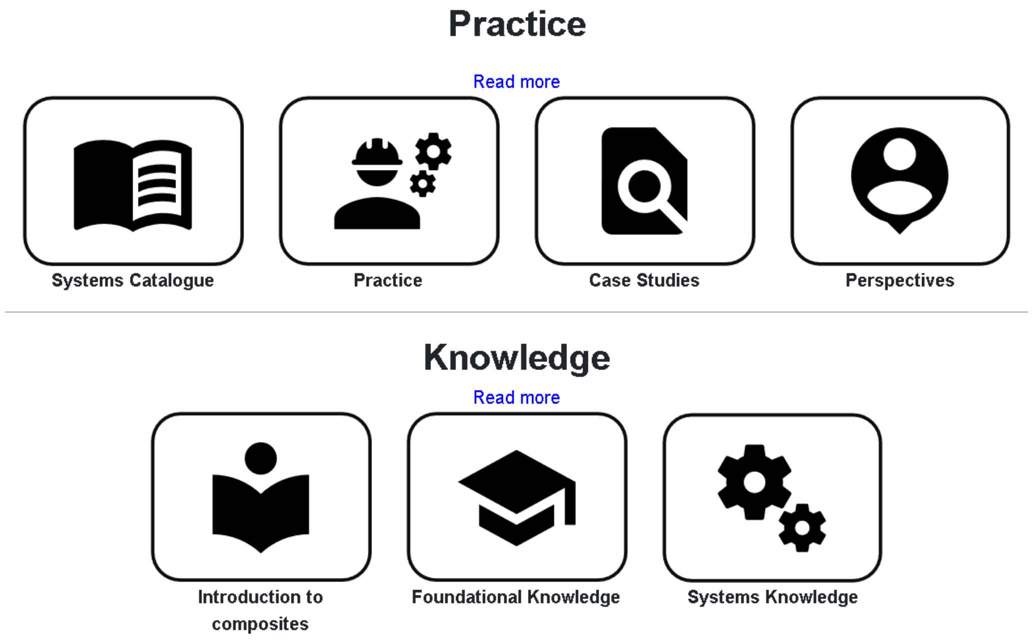
KNOWLEDGE IN PRACTICE CENTRE (KPC)

- A freely available online resource for composites engineers: https://compositeskn.org/knowledge-in-practice-centre/
- Focus on practice, guided by foundational knowledge and a systems-based approach to composites manufacturing



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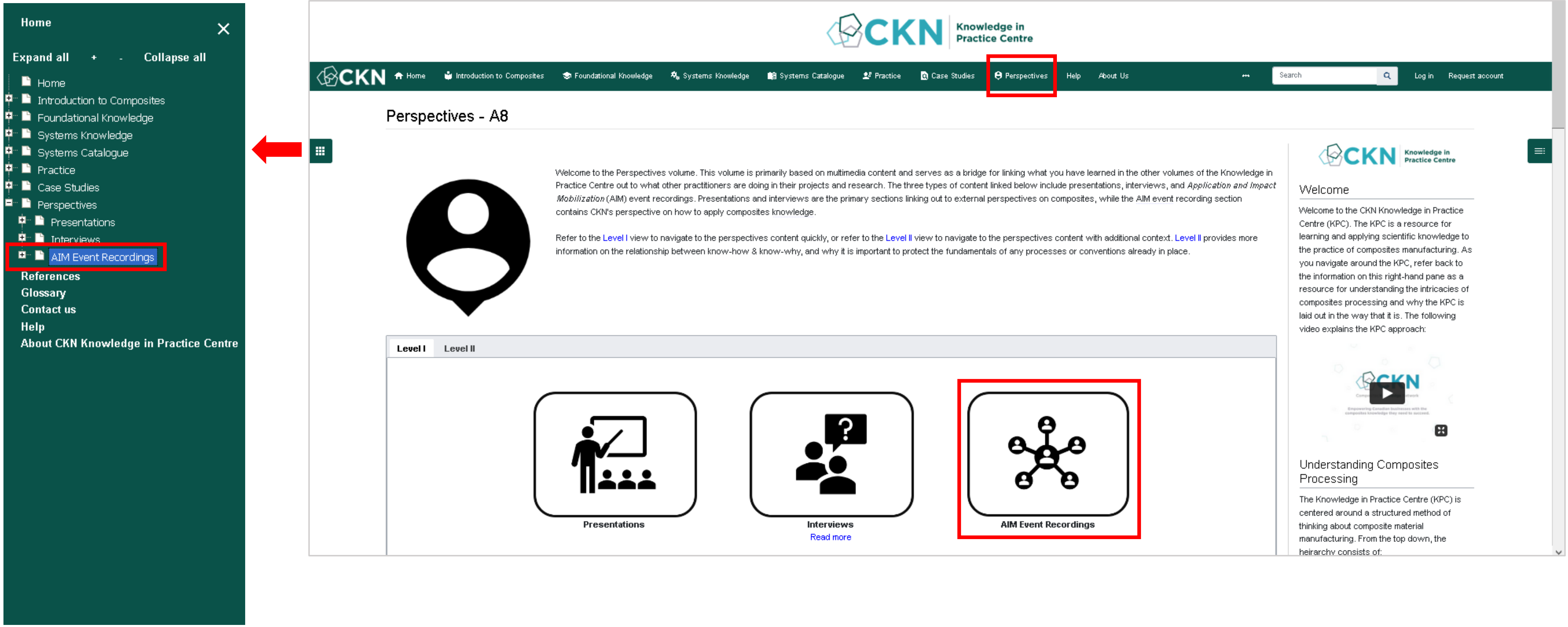

TODAY'S TOPIC:

RESIN BEHAVIOUR DURING PROCESSING:

Key Resin Properties to Consider When Developing a Manufacturing Process

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INTRODUCTION

- Learning objectives:
  - Understand how the resin transforms during processing
  - Identify and understand the key properties to consider when developing, optimizing or troubleshooting a manufacturing process
  - Understand the information commonly available in technical data sheet
  - Understand instruments used to characterize manufacturing properties

HOW DOES THE RESIN TRANSFORM DURING MANUFACTURING?

The resin liquid-to solid-transition

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KPC A162: CURE OF THERMOSETTING POLYMERS

- During polymerization the resin goes through different phases
- At gelation it changes from a viscous liquid to a rubbery gel
- As the curing advances, the rubbery gel transforms into a glassy solid at vitrification

Gelation

Vitrification

Viscous Liquid

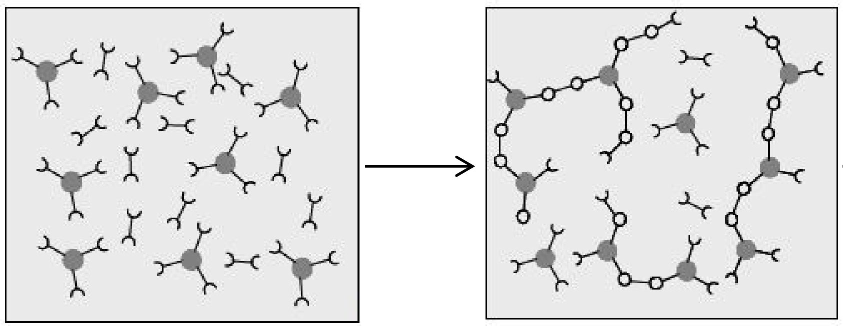
Unreacted monomers and cross linking agents form small branched molecules

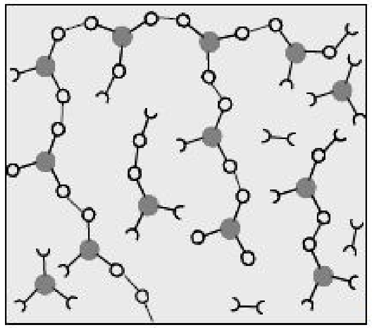
Rubbery Gel

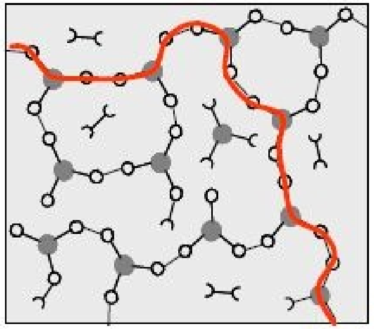
A giant macromolecule (gel) appears that percolate through the sample

Glassy Solid

Cured, cross linked polymer (one giant molecule) with some unreacted groups







To learn more about thermosetting resins watch:
https://compositeskn.org/KPC/A122 Webinar Session 3

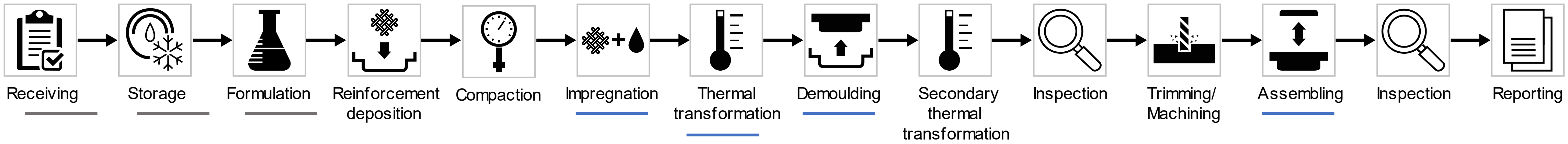
WHAT ARE THE KEY PROPERTIES TO CONSIDER?

Viscosity, gel time, heat of reaction, degree of cure, etc.

KPC A215: MANUFACTURING WORKFLOW

- Let's look at a Resin Transfer Moulding (RTM) workflow
- Underlined in blue are the steps we will discuss

Key Manufacturing Steps:



IMPREGNATION STEP

INPUT

Liquid resin →

STEP

Impregnation

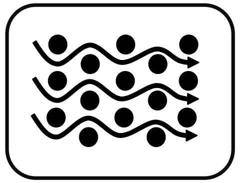
→

OUTCOMES

Impregnated reinforcement

ANALYSIS

Flow and consolidation management



KPC A158

Material properties:

- **Resin viscosity**
- **Resin gel time**
- *Reinforcement's permeability*
- *Resin/reinforcement surface tension and contact angle*
- *etc.*

→ Filling time, dry spots, micro/macro-voids formation

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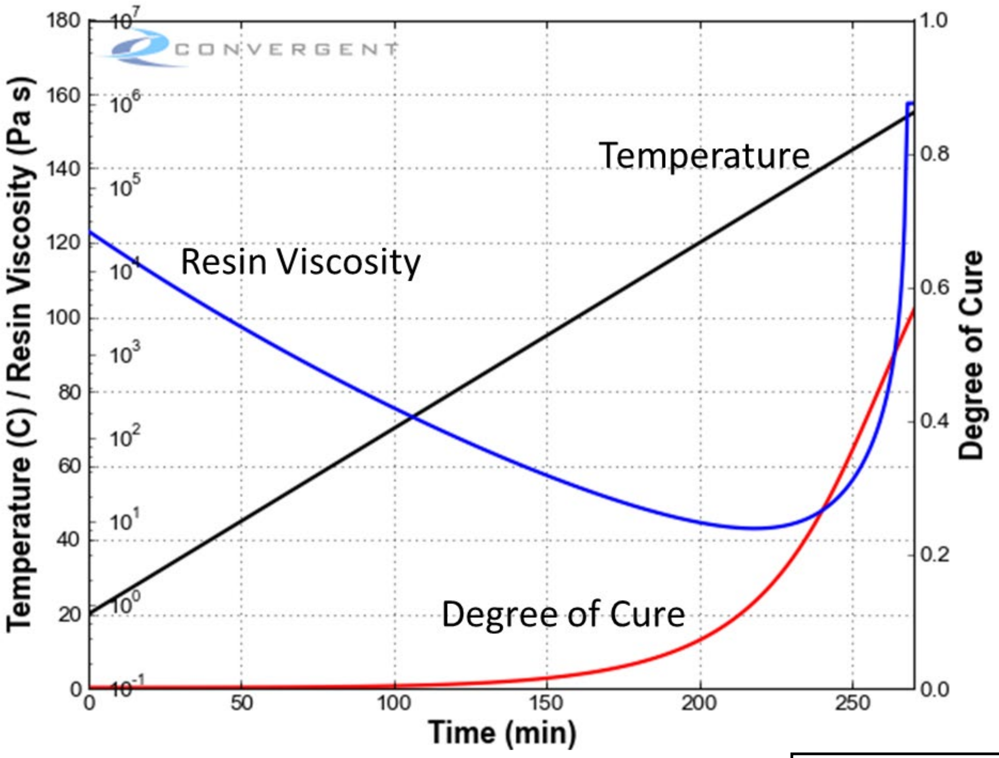
KPC A203: VISCOSITY

- Viscosity is an indicator of how easily the resin will impregnate the reinforcement
- Viscosity is a function of both temperature and degree of cure and can be described using empirical models such as an Arrhenius-type equation:

$$\mu = Ae^{\frac{\Delta E}{RT}f(\alpha)}$$

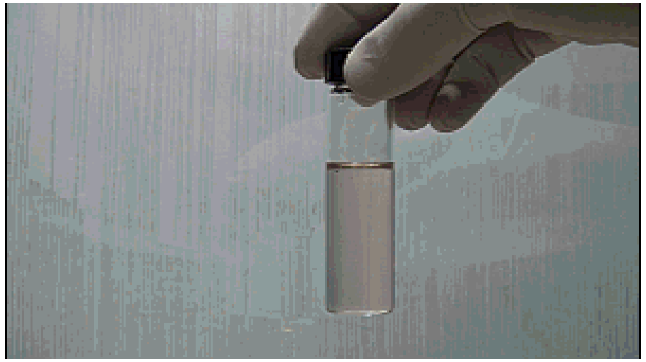
Where A is a constant, ΔE is the activation energy, $f(\alpha)$ is the dependency on the degree of cure, α and RT describes the kinetic energy as a function of the temperature, T

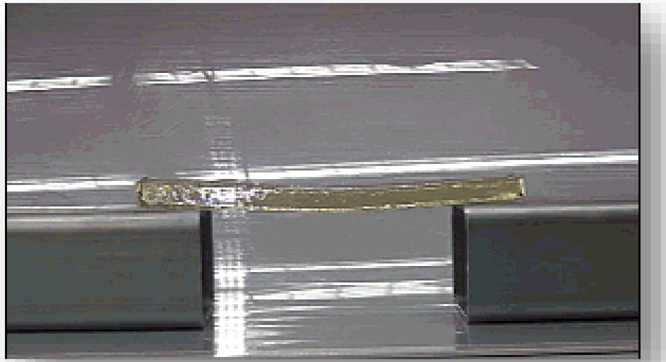


KPC A162: GEL TIME

- Gel time indicates when gelation occurs, i.e. when the resin transforms from a liquid to a soft gel and stops flowing

GELATION
Liquid → Gel





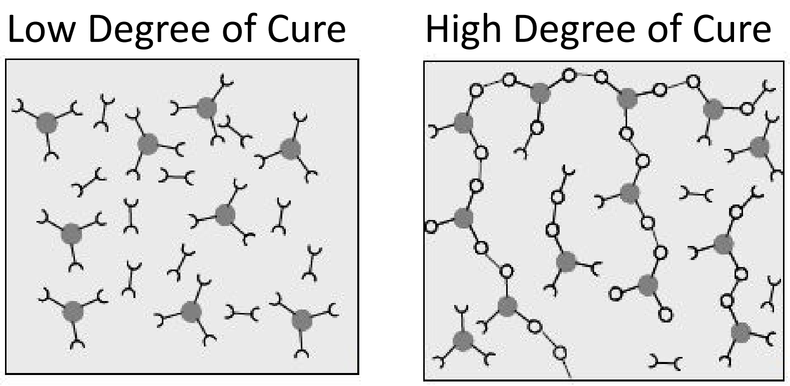
- Gel time depends on degree of cure. Epoxy resins typically gel at a degree of cure of 0.5 while polyester resins gel before a degree of cure of 0.15

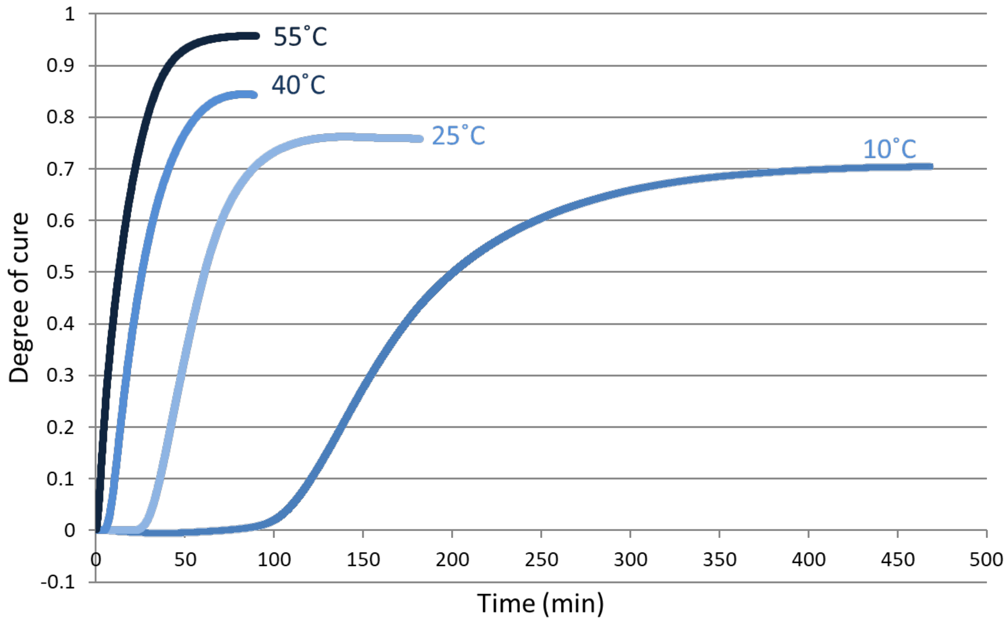
KPC A172: THERMAL TRANSFORMATION STEP

INPUT		STEP		OUTCOMES
		Thermal transformation		
Uncured resin	→		→	Cured resin
		ANALYSIS		
Material properties: • **Resin's cure kinetics** • **Resin's heat of reaction** • *Resin's thermal conductivity* • *Resin's heat capacity* • *Resin's density* • *Reinforcement's thermal conductivity* • *etc.*	→	Thermal management KPC A107	→	Degree of cure, exotherm, etc.

KPC A104: DEGREE OF CURE

- Degree of cure (DOC) is an indication of how far the crosslinking is advanced in a thermoset resin
- DOC is defined with a number between 0 and 1 (or 0% and 100%) where 100% is a fully cured resin

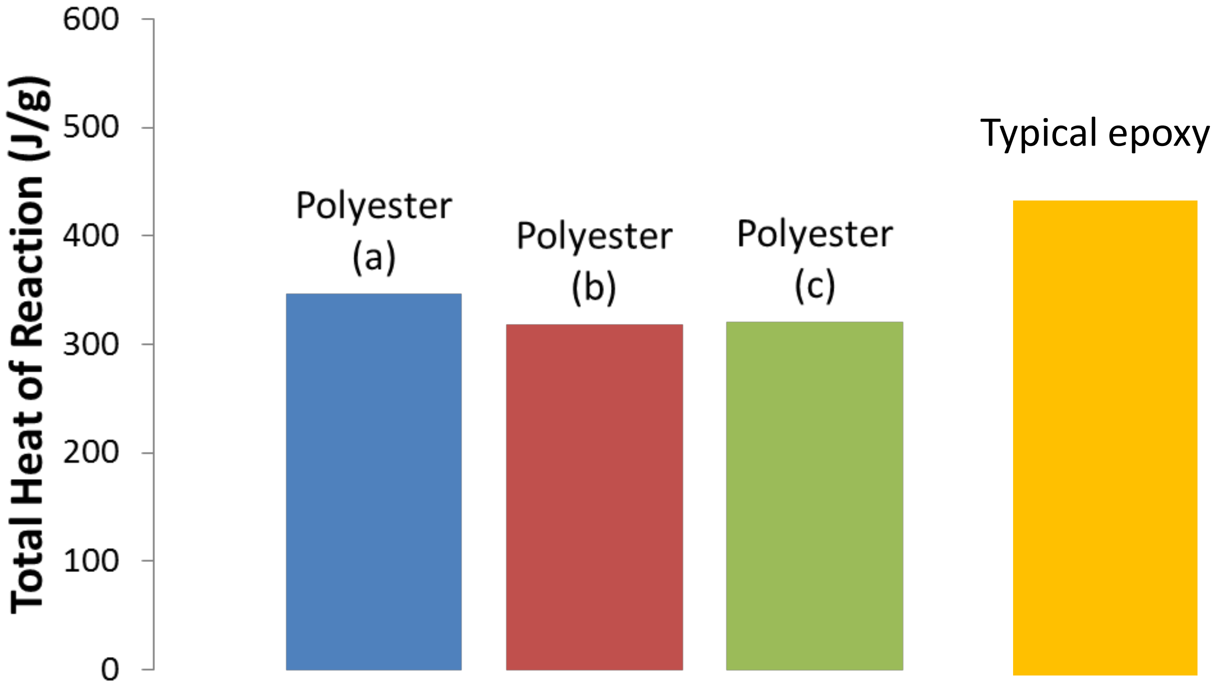




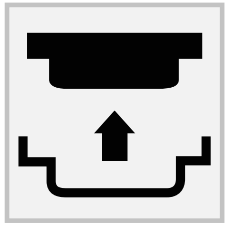
- Degree of cure is dependent on temperature and time (thermal history)

KPC A114: HEAT OF REACTION

- Cure of thermoset resins is an exothermic reaction and heat is generated during the curing process
- A thermoset resin has the potential to release a certain amount of energy while curing, referred to as the total heat of reaction, H_R (unit of J/g)



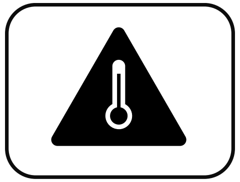
DEMOLDING STEP

INPUT	STEP	OUTCOMES
Part on tool →	Demolding 	→ Free-standing part

ANALYSIS

Material properties:
- Resin's cure kinetics
- **Resin's glass transition temperature**
- *Resin's thermal conductivity*
- *Resin's heat capacity*
- *Resin's density*
- *Reinforcement's thermal conductivity*
- *etc.*

→ Thermal management



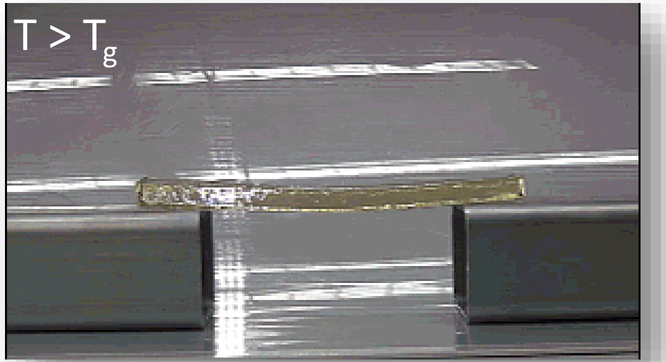
KPC A107

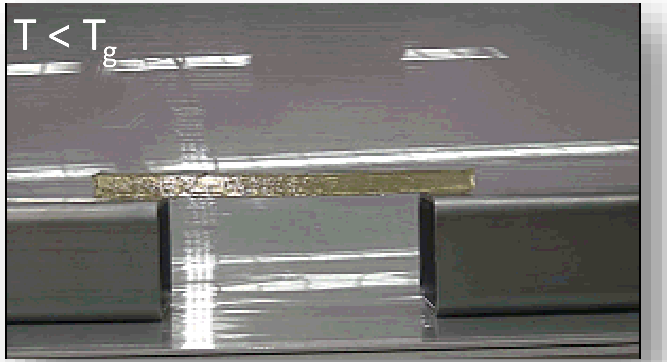
→ Resin's glass transition temperature and part's temperature

KPC A102: GLASS TRANSITION TEMPERATURE (Tg)

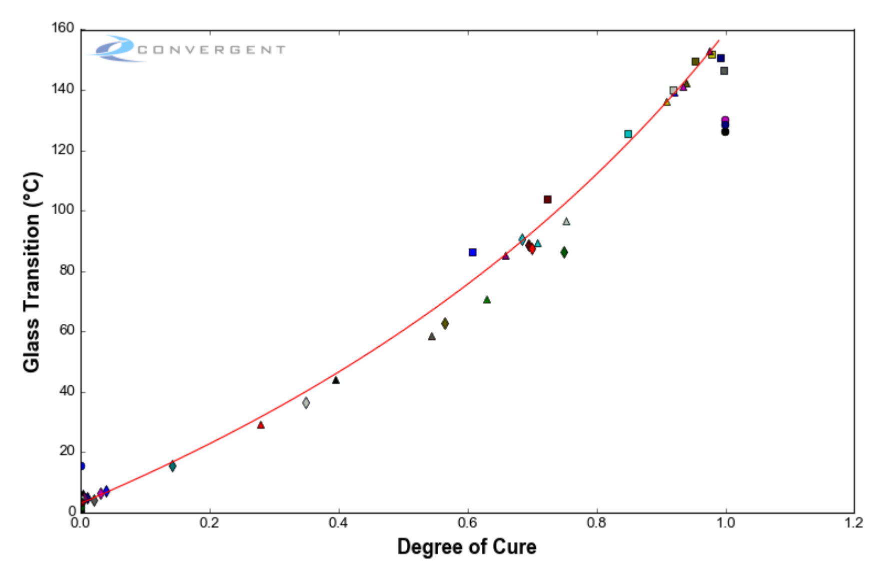
- When heated above their Tg, thermosets soften and transition from hard, glassy materials to soft, rubbery materials

GLASS TRANSITION
Gel → Glass

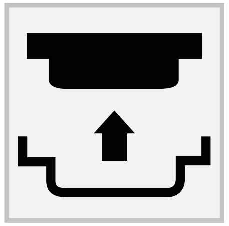




- The glass transition temperature is a function of degree of cure
- Typically want to demould below Tg

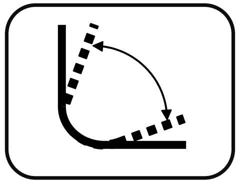


ASSEMBLY STEP

INPUT	STEP	OUTCOMES
Multiple components →	Assembly 	→ Assembly

ANALYSIS

Residual stress and dimensional control management



KPC A165

Material properties:

- **Resin's cure shrinkage**
- **Resin's coefficient of thermal expansion**
- *Reinforcement coefficient of thermal expansion*
- *etc.*

→ Spring in, warpage, residual stresses

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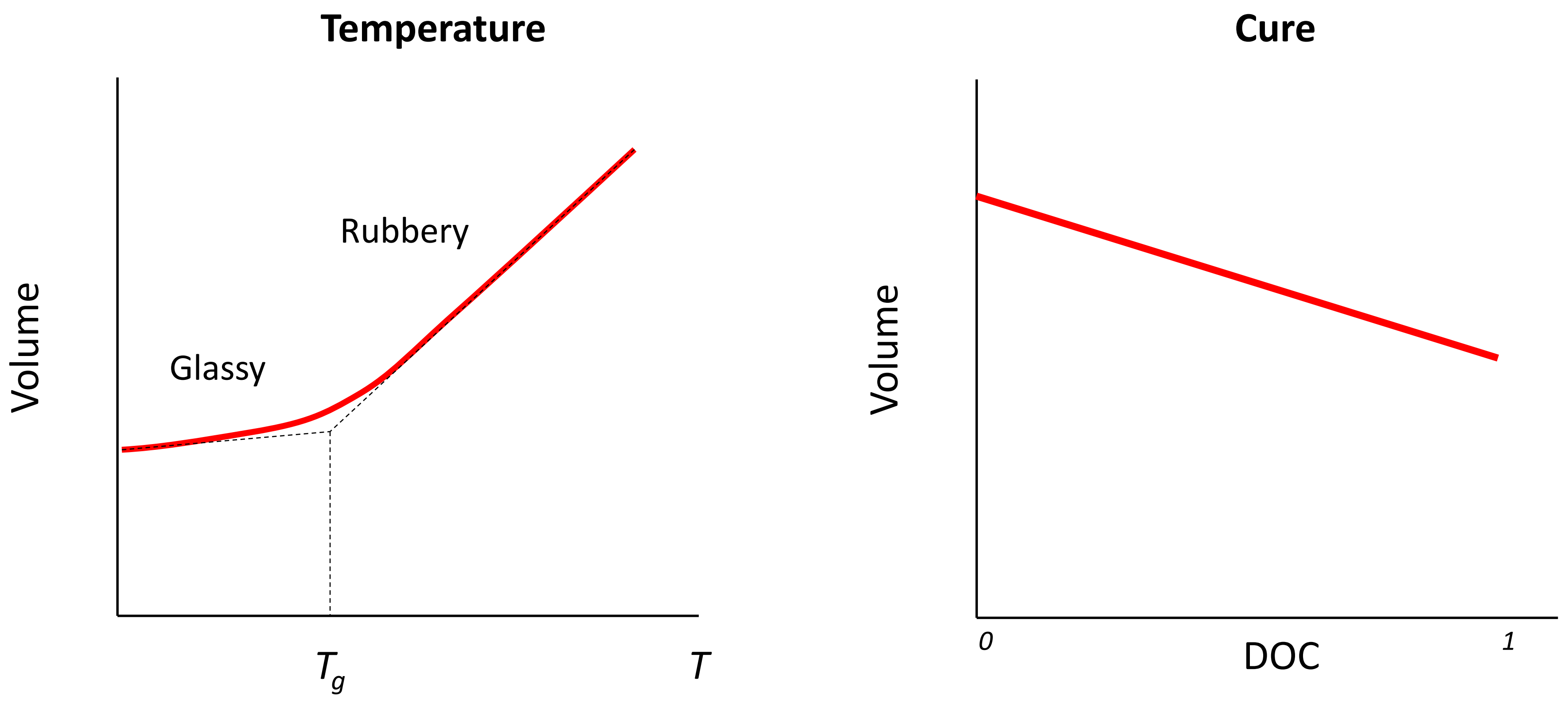
KPC A102: THERMO-VOLUMETRIC BEHAVIOUR

- Volume of thermoset resins changes with temperature and cure



Coefficient of thermal expansion (CTE)

Coefficient of cure shrinkage (CS)

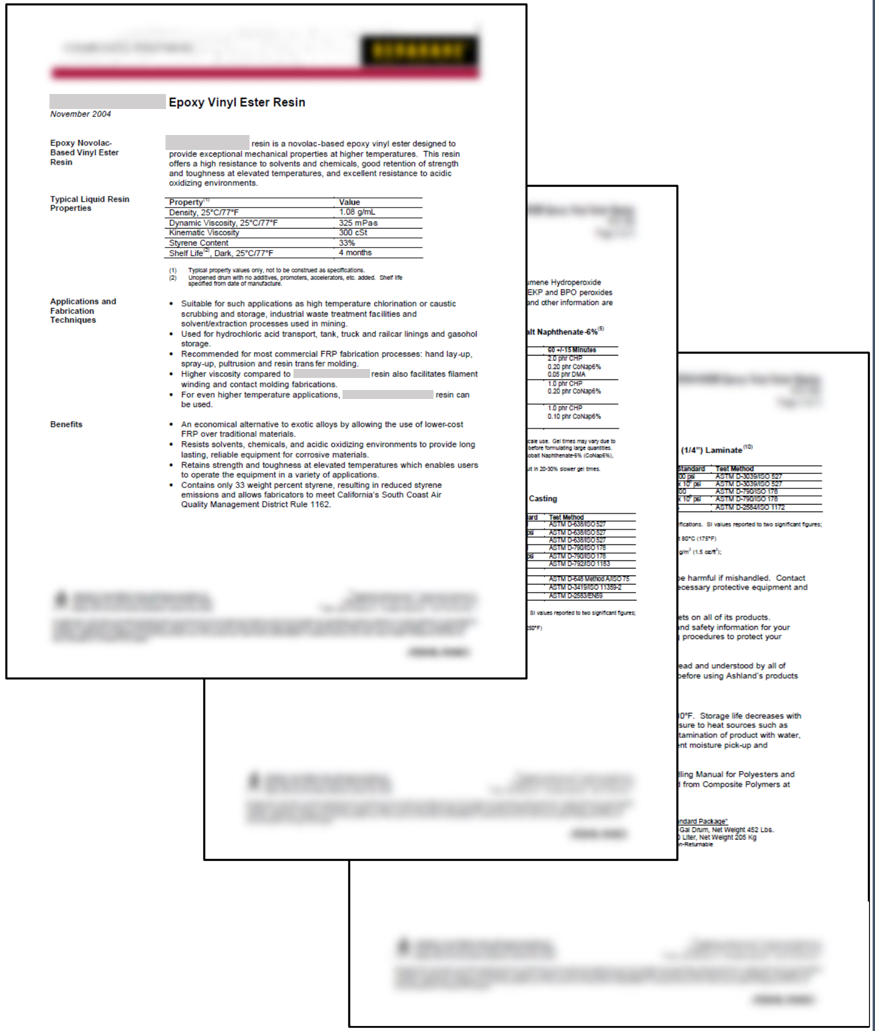
WHAT INFORMATION IS COMMONLY AVAILABLE IN TECHNICAL DATA SHEET?

Let’s look at neat resin and prepreg material systems

Example Technical Data Sheets

- Technical Data Sheets (TDS) usually focus on design properties and include limited manufacturing properties*
- Viscosity, gel time and a baseline cure cycle are commonly given
- With polyester and vinyl ester, end users can define their own formulations, which further limits the information in a TDS
- The TDS is for the resin, what about the fibre, what about the composite?

- *Let's review a vinyl ester resin and epoxy prepreg TDS*


Epoxy Vinyl Ester Resin

November 2004

Epoxy Novolac-Based Vinyl Ester Resin

resin is a novolac-based epoxy vinyl ester designed to provide exceptional mechanical properties at higher temperatures. This resin offers a high resistance to solvents and chemicals, good retention of strength and toughness at elevated temperatures, and excellent resistance to acidic oxidizing environments.

Typical Liquid Resin Properties

Property(1)	Value
Density, 25°C/77°F	1.08 g/mL
Dynamic Viscosity, 25°C/77°F	325 mPa·s
Kinematic Viscosity	300 cSt
Styrene Content	33%
Shelf Life(2), Dark, 25°C/77°F	4 months

(1) Typical property values only, not to be construed as specifications.
(2) Unopened drum with no additives, promoters, accelerators, etc. added. Shelf life specified from date of manufacture.

Applications and Fabrication Techniques

- Suitable for such applications as high temperature chlorination or caustic scrubbing and storage, industrial waste treatment facilities and solvent/extraction processes used in mining.
- Used for hydrochloric acid transport, tank, truck and railcar linings and gasohol storage.
- Recommended for most commercial FRP fabrication processes: hand lay-up, spray-up, pultrusion and resin transfer molding.
- Higher viscosity compared to resin also facilitates filament winding and contact molding fabrications.
- For even higher temperature applications, resin can be used.

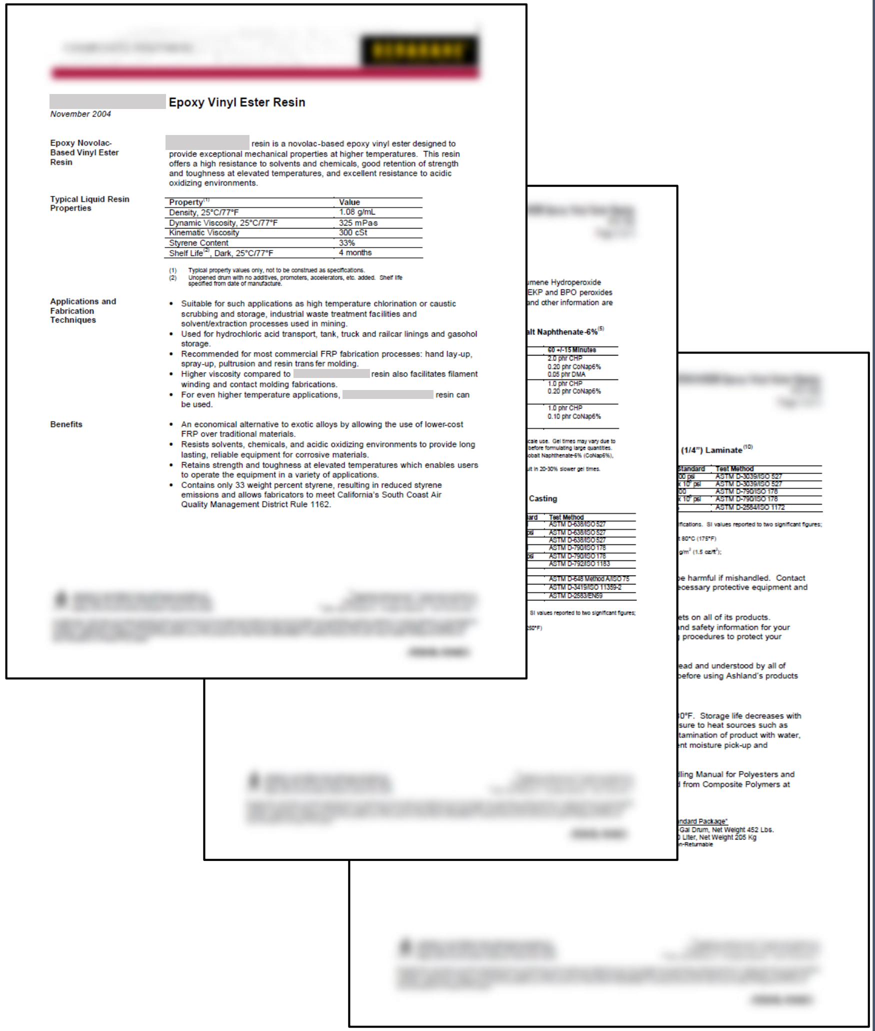
Benefits

- An economical alternative to exotic alloys by allowing the use of lower-cost FRP over traditional materials.
- Resists solvents, chemicals, and acidic oxidizing environments to provide long lasting, reliable equipment for corrosive materials.
- Retains strength and toughness at elevated temperatures which enables users to operate the equipment in a variety of applications.
- Contains only 33 weight percent styrene, resulting in reduced styrene emissions and allows fabricators to meet California's South Coast Air Quality Management District Rule 1162.

*Based on the review of over forty TDSs. Soon available on the KPC

Example TDS – Vinyl Ester Resin

- What can we expect from a resin TDS, specifically a vinyl ester or polyester resin?
- This is an ‘industrial grade’ resin vs an aerospace grade that we’ll see next



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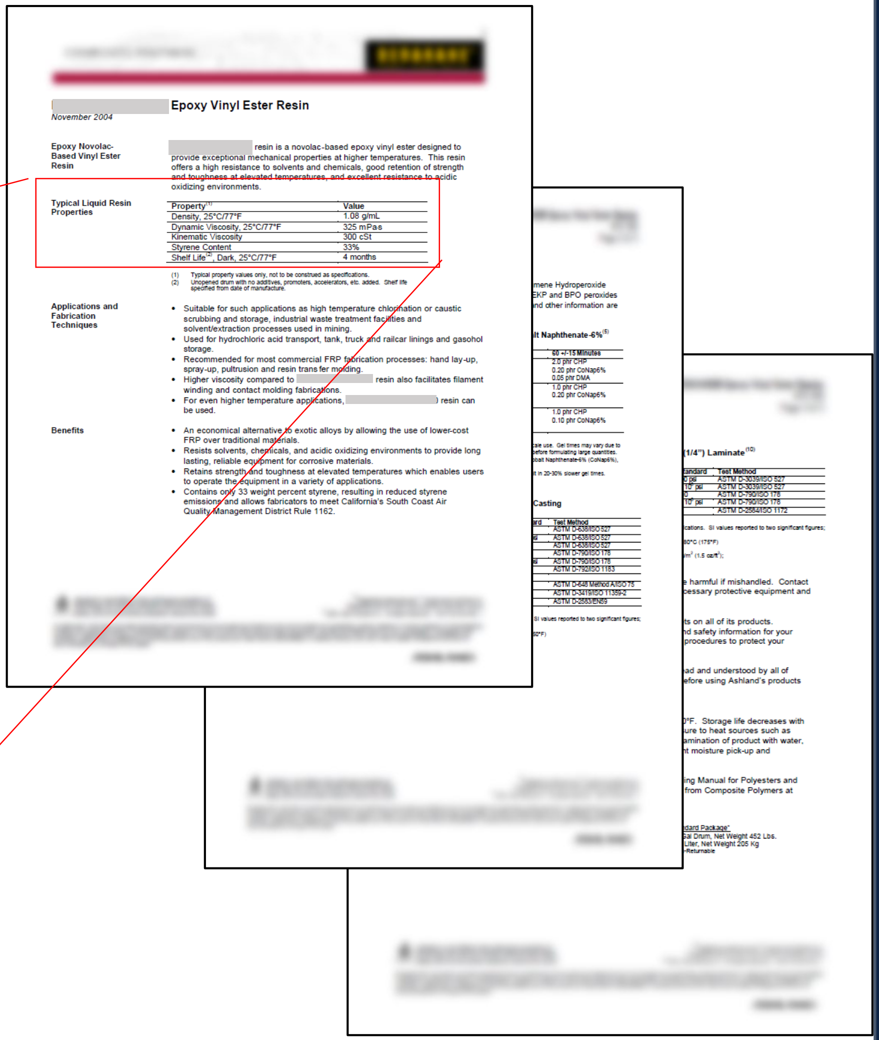
Example TDS – Vinyl Ester Resin

- Example of a common vinyl ester resin



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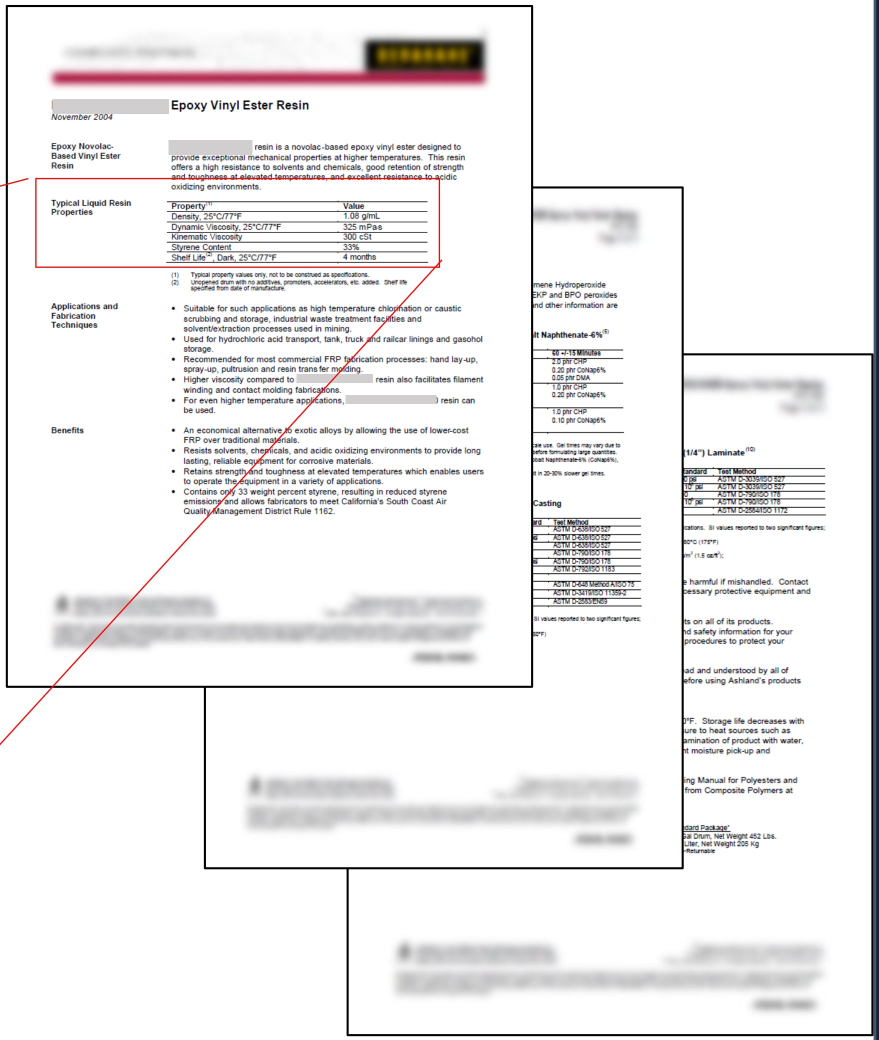

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Kinematic viscosity, ν is the ratio of dynamic viscosity, μ to density, ρ:

$$\upsilon = \frac{\mu}{\rho}$$

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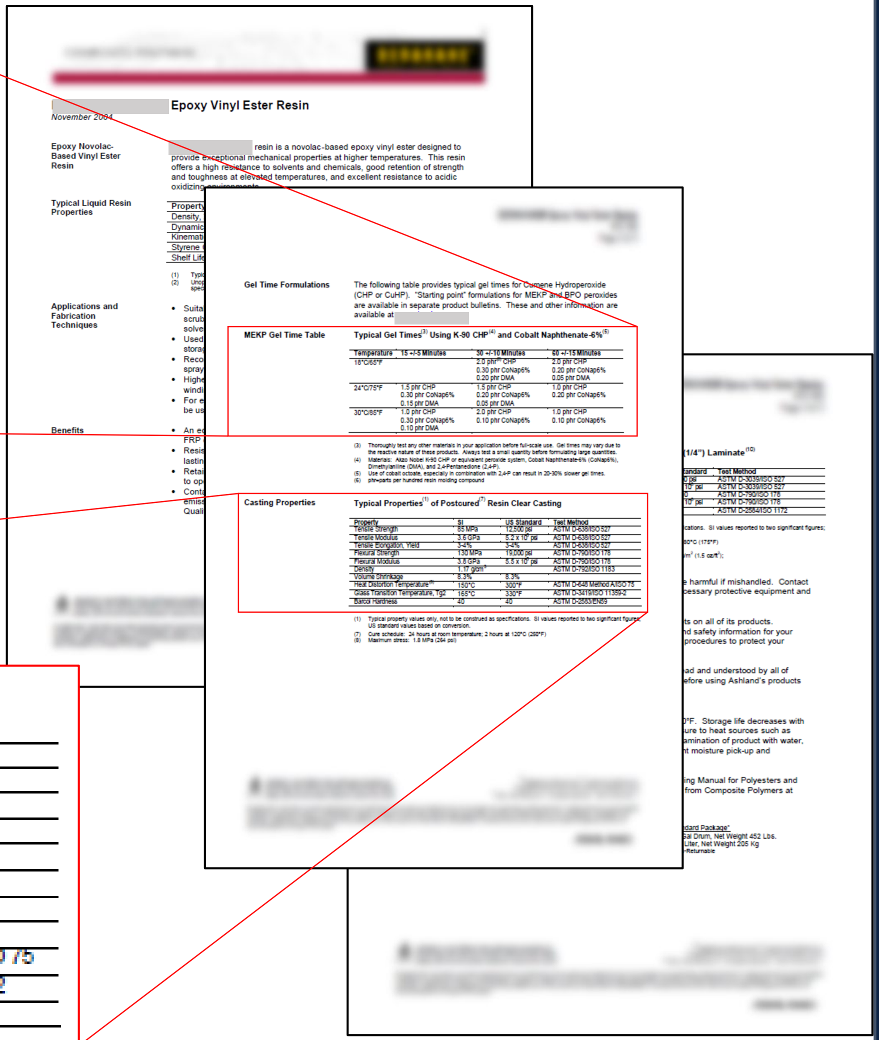
Example TDS – Vinyl Ester Resin

MEKP Gel Time Table

Typical Gel Times[3] Using K-90 CHP[4] and Cobalt Naphthenate-6%[5]

Temperature	15 +/-5 Minutes	30 +/-10 Minutes	60 +/-15 Minutes
18°C/65°F		2.0 phr[6] CHP 0.30 phr CoNap6% 0.20 phr DMA	2.0 phr CHP 0.20 phr CoNap6% 0.05 phr DMA
24°C/75°F	1.5 phr CHP 0.30 phr CoNap6% 0.15 phr DMA	1.5 phr CHP 0.20 phr CoNap6% 0.05 phr DMA	1.0 phr CHP 0.20 phr CoNap6%
30°C/85°F	1.0 phr CHP 0.30 phr CoNap6% 0.10 phr DMA	2.0 phr CHP 0.10 phr CoNap6%	1.0 phr CHP 0.10 phr CoNap6%



Casting Properties

Typical Properties[1] of Postcured[7] Resin Clear Casting

Property	SI	US Standard	Test Method
Tensile Strength	85 MPa	12,500 psi	ASTM D-638/ISO 527
Tensile Modulus	3.6 GPa	5.2×10^5 psi	ASTM D-638/ISO 527
Tensile Elongation, Yield	3-4%	3-4%	ASTM D-638/ISO 527
Flexural Strength	130 MPa	19,000 psi	ASTM D-790/ISO 178
Flexural Modulus	3.8 GPa	5.5×10^5 psi	ASTM D-790/ISO 178
Density	1.17 g/cm^3		ASTM D-792/ISO 1183
Volume Shrinkage	8.3%	8.3%	
Heat Distortion Temperature[8]	150°C	300°F	ASTM D-648 Method A/ISO 75
Glass Transition Temperature, Tg2	165°C	330°F	ASTM D-3419/ISO 11359-2
Barcol Hardness	40	40	ASTM D-2583/EN59

Example TDS – Vinyl Ester Resin

Laminate Properties

Typical Properties(1) of Postcured(9) 6 mm (1/4") Laminate(10)

Property	SI	US Standard	Test Method
Tensile Strength	130 MPa	19,000 psi	ASTM D-3039/ISO 527
Tensile Modulus	12 GPa	1.7 x 10^6 psi	ASTM D-3039/ISO 527
Flexural Strength	210 MPa	30,000	ASTM D-790/ISO 178
Flexural Modulus	8.5 GPa	1.2 x 10^6 psi	ASTM D-790/ISO 178
Glass Content	40%	40%	ASTM D-2584/ISO 1172

(1) Typical property values only, not to be construed as specifications. SI values reported to two significant figures; US standard values based on conversion.
(9) Cure schedule: 24 hours at room temperature; 6 hours at 80°C (175°F)
(10) 6 mm (1/4") Construction – V/M/M/Wr/M/Wr/M
V = Continuous veil glass; M = Chopped strand mat, 450 g/m² (1.5 oz/ft²);
Wr = Woven roving, 800 g/m² (24 oz/yd²)

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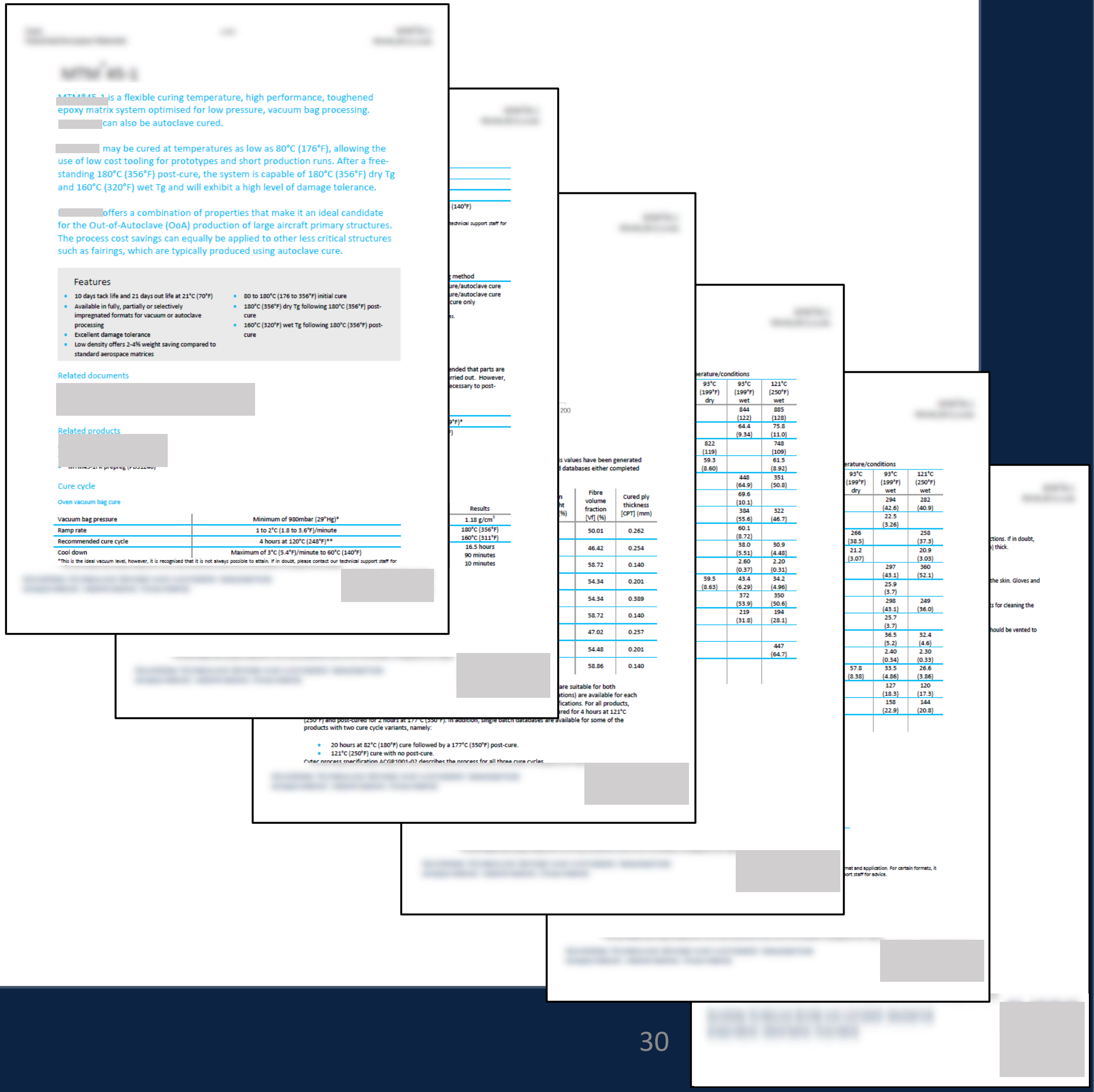
Example TDS – Prepreg Material

- This is an aerospace grade material
- This TDS focuses on the resin but some information is specific to a certain prepreg model
- What can we expect from a prepreg TDS?



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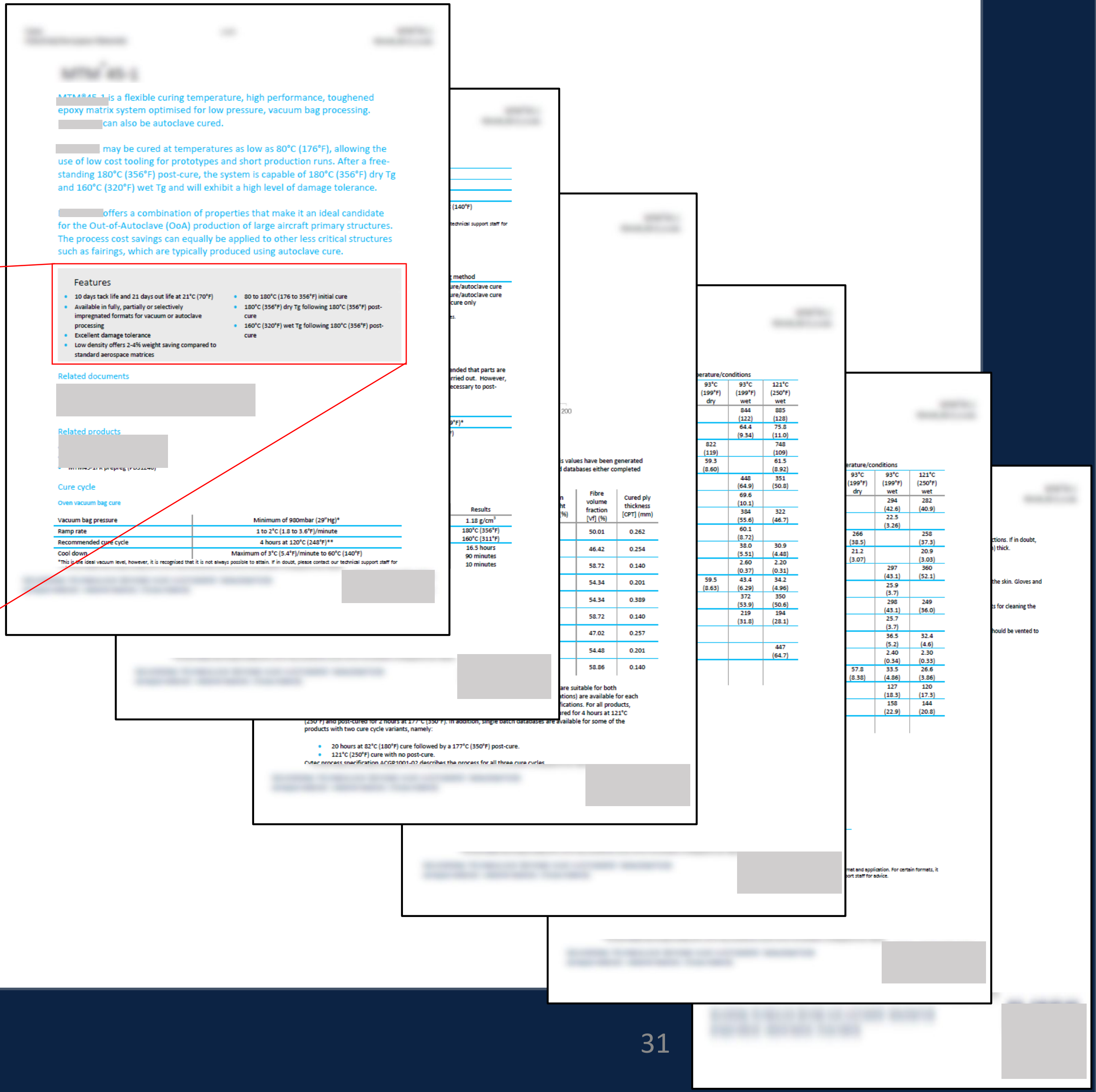
Example TDS – Prepreg Material

Features

- 10 days tack life and 21 days out life at 21°C (70°F)
- Available in fully, partially or selectively impregnated formats for vacuum or autoclave processing
- Excellent damage tolerance
- Low density offers 2-4% weight saving compared to standard aerospace matrices
- 80 to 180°C (176 to 356°F) initial cure
- 180°C (356°F) dry Tg following 180°C (356°F) post-cure
- 160°C (320°F) wet Tg following 180°C (356°F) post-cure



is a flexible curing temperature, high performance, toughened epoxy matrix system optimised for low pressure, vacuum bag processing. can also be autoclave cured.

may be cured at temperatures as low as 80°C (176°F), allowing the use of low cost tooling for prototypes and short production runs. After a free-standing 180°C (356°F) post-cure, the system is capable of 180°C (356°F) dry Tg and 160°C (320°F) wet Tg and will exhibit a high level of damage tolerance.

offers a combination of properties that make it an ideal candidate for the Out-of-Autoclave (OoA) production of large aircraft primary structures. The process cost savings can equally be applied to other less critical structures such as fairings, which are typically produced using autoclave cure.

Features

- 10 days tack life and 21 days out life at 21°C (70°F)
- Available in fully, partially or selectively impregnated formats for vacuum or autoclave processing
- Excellent damage tolerance
- Low density offers 2-4% weight saving compared to standard aerospace matrices
- 80 to 180°C (176 to 356°F) initial cure
- 180°C (356°F) dry Tg following 180°C (356°F) post-cure
- 160°C (320°F) wet Tg following 180°C (356°F) post-cure

Related documents

Related products

Cure cycle

Oven vacuum bag cure

Vacuum bag pressure	Minimum of 980mbar (29"Hg)*
Ramp rate	1 to 2°C (1.8 to 3.6°F)/minute
Recommended cure cycle	4 hours at 120°C (248°F)**
Cool down	Maximum of 3°C (5.4°F)/minute to 60°C (140°F)

*This is the ideal vacuum level, however, it is recognised that it is not always possible to attain. If in doubt, please contact our technical support staff for

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Example TDS – Prepreg Material

Autoclave cure

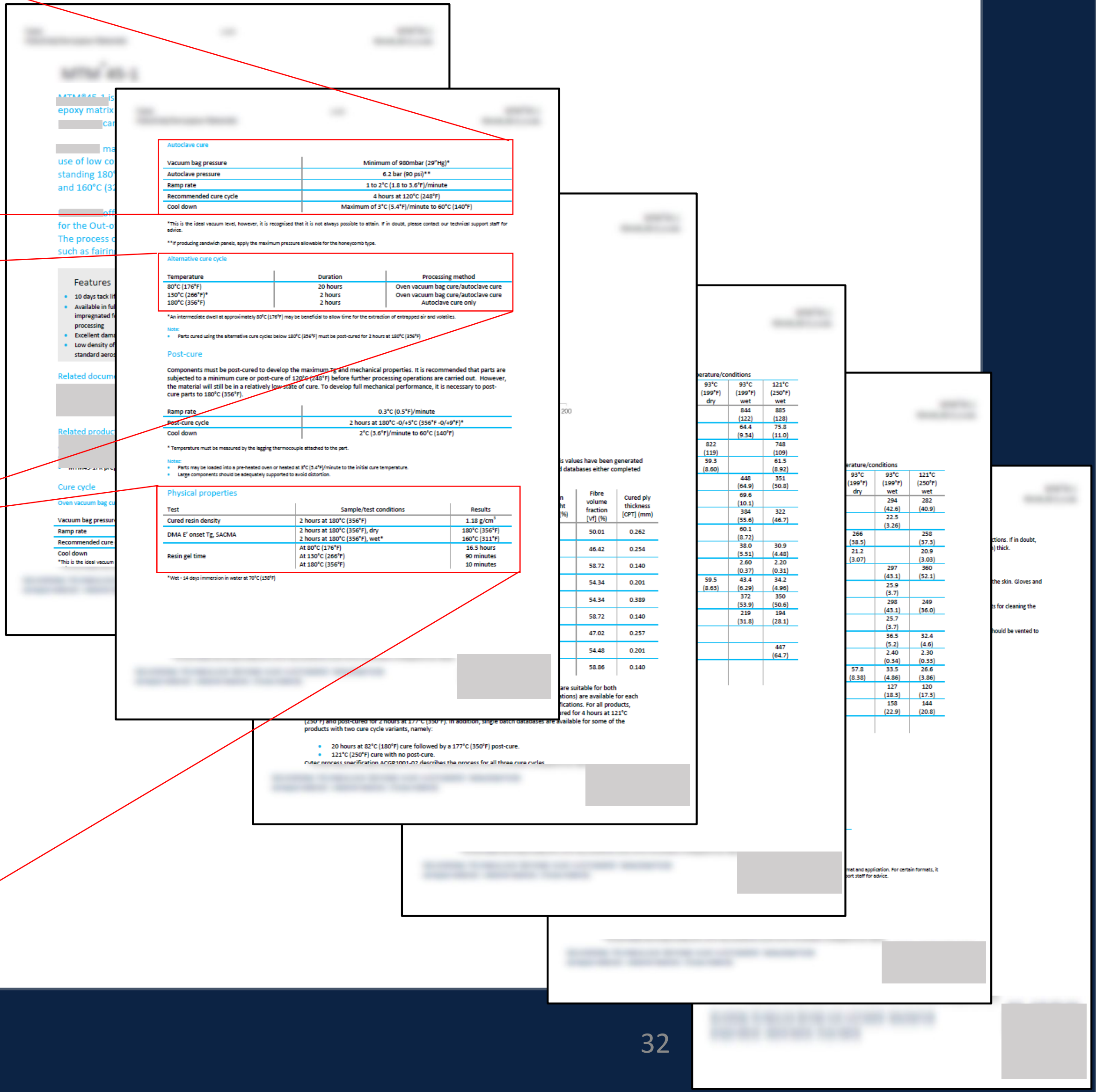
Vacuum bag pressure	Minimum of 980mbar (29"Hg)*
Autoclave pressure	6.2 bar (90 psi)**
Ramp rate	1 to 2°C (1.8 to 3.6°F)/minute
Recommended cure cycle	4 hours at 120°C (248°F)
Cool down	Maximum of 3°C (5.4°F)/minute to 60°C (140°F)

Alternative cure cycle

Temperature	Duration	Processing method
80°C (176°F)	20 hours	Oven vacuum bag cure/autoclave cure
130°C (266°F)*	2 hours	Oven vacuum bag cure/autoclave cure
180°C (356°F)	2 hours	Autoclave cure only

Physical properties

Test	Sample/test conditions	Results
Cured resin density	2 hours at 180°C (356°F)	1.18 g/cm^3
DMA E' onset Tg, SACMA	2 hours at 180°C (356°F), dry 2 hours at 180°C (356°F), wet*	180°C (356°F) 160°C (311°F)
Resin gel time	At 80°C (176°F) At 130°C (266°F) At 180°C (356°F)	16.5 hours 90 minutes 10 minutes



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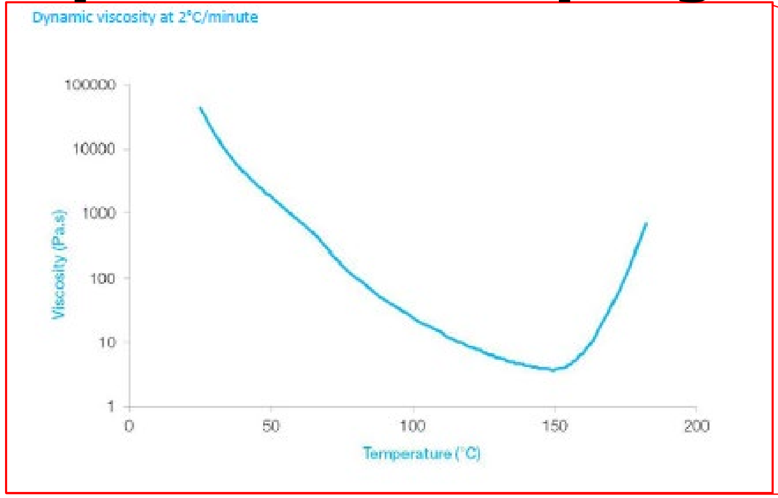
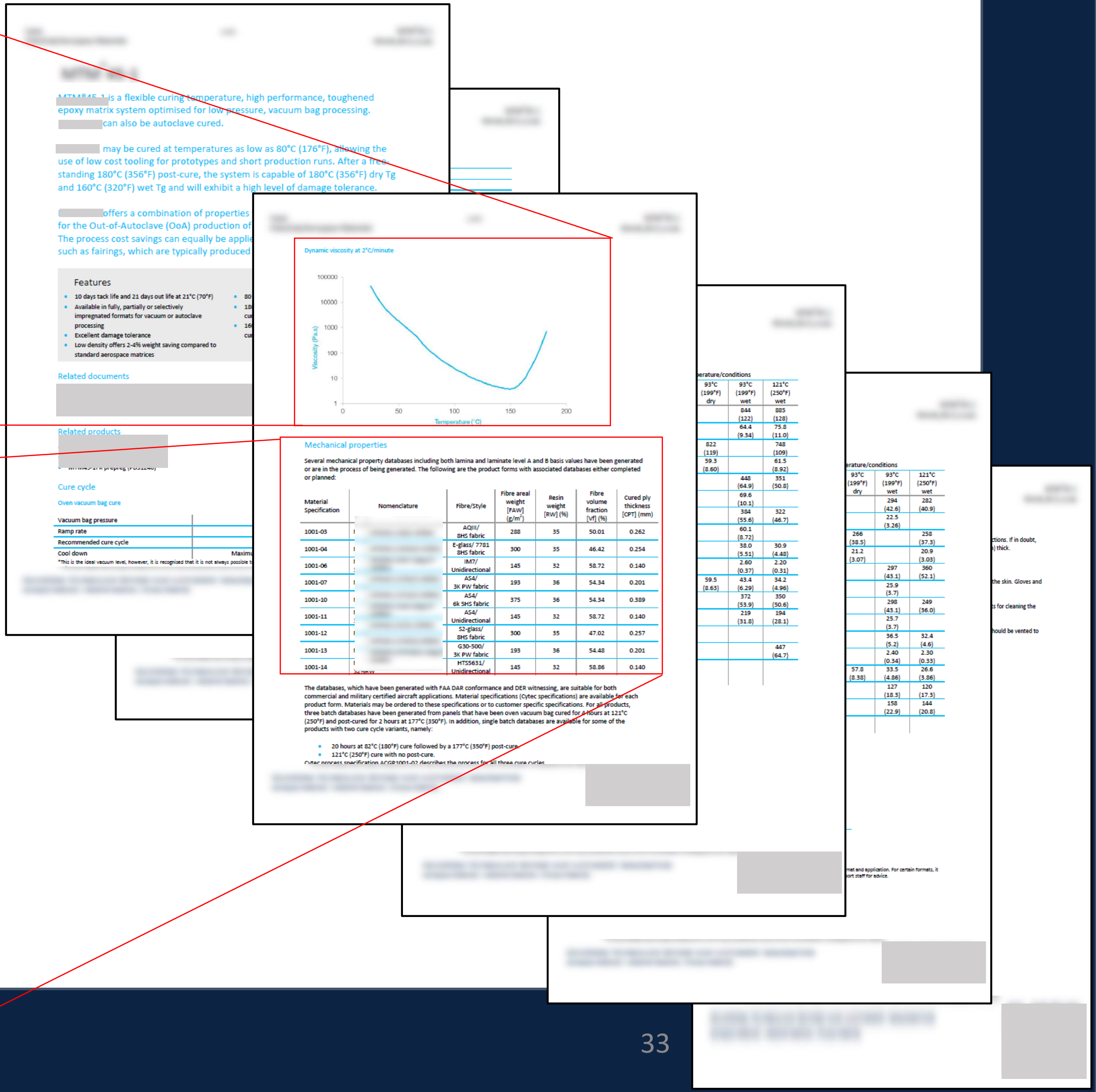
Example TDS – Prepreg Material



Mechanical properties

Several mechanical property databases including both lamina and laminate level A and B basis values have been generated or are in the process of being generated. The following are the product forms with associated databases either completed or planned:

Material Specification	Nomenclature	Fibre/Style	Fibre areal weight [FAW] (g/m²)	Resin weight [RW] (%)	Fibre volume fraction [Vf] (%)	Cured ply thickness [CPT] (mm)
1001-03	[illegible]	AQIII/ 8HS fabric	288	35	50.01	0.262
1001-04	[illegible]	E-glass/ 7781 8HS fabric	300	35	46.42	0.254
1001-06	[illegible]	IM7/ Unidirectional	145	32	58.72	0.140
1001-07	[illegible]	AS4/ 3K PW fabric	193	36	54.34	0.201
1001-10	[illegible]	AS4/ 6k 5HS fabric	375	36	54.34	0.389
1001-11	[illegible]	AS4/ Unidirectional	145	32	58.72	0.140
1001-12	[illegible]	S2-glass/ 8HS fabric	300	35	47.02	0.257
1001-13	[illegible]	G30-500/ 3K PW fabric	193	36	54.48	0.201
1001-14	[illegible]	HTS5631/ Unidirectional	145	32	58.86	0.140



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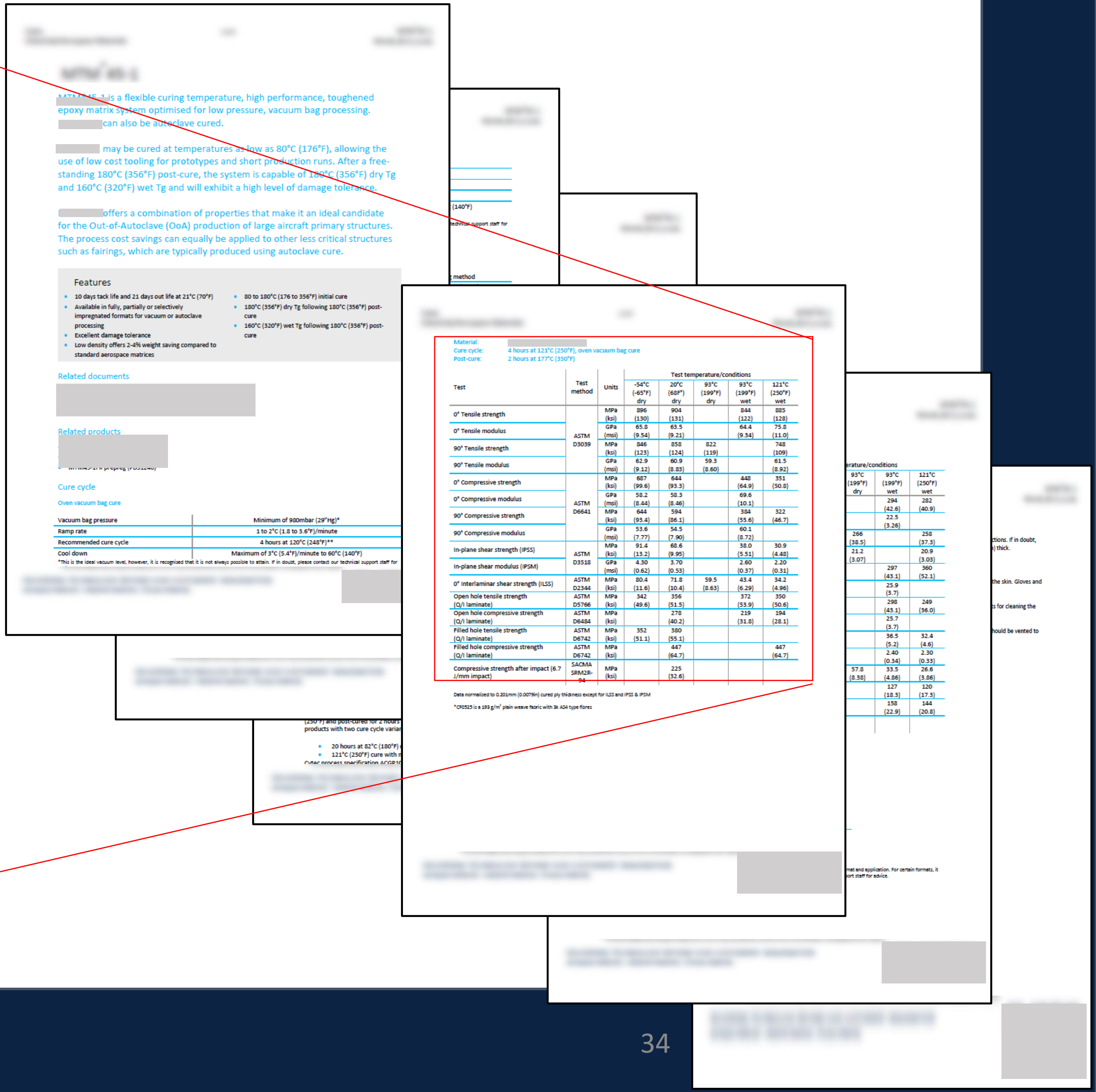
Example TDS – Prepreg Material

Material:
Cure cycle: 4 hours at 121°C (250°F), oven vacuum bag cure
Post-cure: 2 hours at 177°C (350°F)

Test	Test method	Units	Test temperature/conditions -54°C (-65°F) dry	20°C (68°F) dry	93°C (199°F) dry	93°C (199°F) wet	121°C (250°F) wet
0° Tensile strength	ASTM D3039	MPa (ksi)	896 (130)	904 (131)		844 (122)	885 (128)
0° Tensile modulus	ASTM D3039	GPa (msi)	65.8 (9.54)	63.5 (9.21)		64.4 (9.34)	75.8 (11.0)
90° Tensile strength	ASTM D3039	MPa (ksi)	846 (123)	858 (124)	822 (119)		748 (109)
90° Tensile modulus	ASTM D3039	GPa (msi)	62.9 (9.12)	60.9 (8.83)	59.3 (8.60)		61.5 (8.92)
0° Compressive strength	ASTM D6641	MPa (ksi)	687 (99.6)	644 (93.3)		448 (64.9)	351 (50.8)
0° Compressive modulus	ASTM D6641	GPa (msi)	58.2 (8.44)	58.3 (8.46)		69.6 (10.1)	
90° Compressive strength	ASTM D6641	MPa (ksi)	644 (93.4)	594 (86.1)		384 (55.6)	322 (46.7)
90° Compressive modulus	ASTM D6641	GPa (msi)	53.6 (7.77)	54.5 (7.90)		60.1 (8.72)	
In-plane shear strength (IPSS)	ASTM D3518	MPa (ksi)	91.4 (13.2)	68.6 (9.95)		38.0 (5.51)	30.9 (4.48)
In-plane shear modulus (IPSM)	ASTM D3518	GPa (msi)	4.30 (0.62)	3.70 (0.53)		2.60 (0.37)	2.20 (0.31)
0° Interlaminar shear strength (ILSS)	ASTM D2344	MPa (ksi)	80.4 (11.6)	71.8 (10.4)	59.5 (8.63)	43.4 (6.29)	34.2 (4.96)
Open hole tensile strength (Q/I laminate)	ASTM D5766	MPa (ksi)	342 (49.6)	356 (51.5)		372 (53.9)	350 (50.6)
Open hole compressive strength (Q/I laminate)	ASTM D6484	MPa (ksi)		278 (40.2)		219 (31.8)	194 (28.1)
Filled hole tensile strength (Q/I laminate)	ASTM D6742	MPa (ksi)	352 (51.1)	380 (55.1)			
Filled hole compressive strength (Q/I laminate)	ASTM D6742	MPa (ksi)		447 (64.7)			447 (64.7)
Compressive strength after impact (6.7 J/mm impact)	SACMA SRM2R-94	MPa (ksi)		225 (32.6)			



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Example TDS – Prepreg Material

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Cure cycle: 4 hours at 121°C (250°F), oven vacuum bag cure
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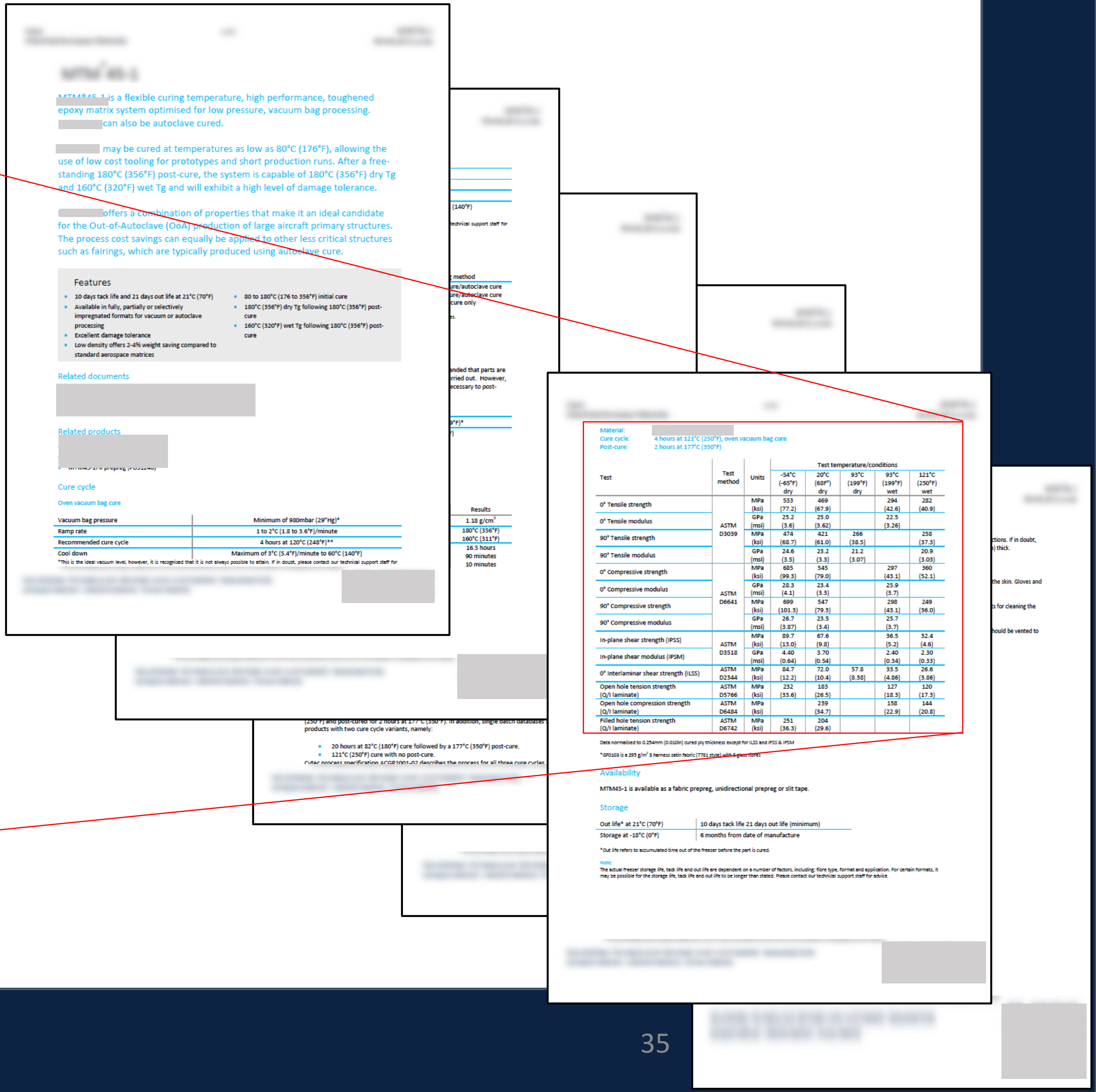
Test	Test method	Units	Test temperature/conditions -54°C (-65°F) dry	20°C (68°F) dry	93°C (199°F) dry	93°C (199°F) wet	121°C (250°F) wet
0° Tensile strength	ASTM D3039	MPa (ksi)	533 (77.2)	469 (67.9)		294 (42.6)	282 (40.9)
0° Tensile modulus		GPa (msi)	25.2 (3.6)	25.0 (3.62)		22.5 (3.26)	
90° Tensile strength		MPa (ksi)	474 (68.7)	421 (61.0)	266 (38.5)		258 (37.3)
90° Tensile modulus		GPa (msi)	24.6 (3.5)	23.2 (3.3)	21.2 (3.07)		20.9 (3.03)
0° Compressive strength	ASTM D6641	MPa (ksi)	685 (99.3)	545 (79.0)		297 (43.1)	360 (52.1)
0° Compressive modulus		GPa (msi)	28.3 (4.1)	23.4 (3.3)		25.9 (3.7)	
90° Compressive strength		MPa (ksi)	699 (101.3)	547 (79.3)		298 (43.1)	249 (36.0)
90° Compressive modulus		GPa (msi)	26.7 (3.87)	23.5 (3.4)		25.7 (3.7)	
In-plane shear strength (IPSS)	ASTM D3518	MPa (ksi)	89.7 (13.0)	67.6 (9.8)		36.5 (5.2)	32.4 (4.6)
In-plane shear modulus (IPSM)		GPa (msi)	4.40 (0.64)	3.70 (0.54)		2.40 (0.34)	2.30 (0.33)
0° Interlaminar shear strength (ILSS)	ASTM D2344	MPa (ksi)	84.7 (12.2)	72.0 (10.4)	57.8 (8.38)	33.5 (4.86)	26.6 (3.86)
Open hole tension strength (Q/I laminate)	ASTM D5766	MPa (ksi)	232 (33.6)	183 (26.5)		127 (18.3)	120 (17.3)
Open hole compression strength (Q/I laminate)	ASTM D6484	MPa (ksi)		239 (34.7)		158 (22.9)	144 (20.8)
Filled hole tension strength (Q/I laminate)	ASTM D6742	MPa (ksi)	251 (36.3)	204 (29.6)			



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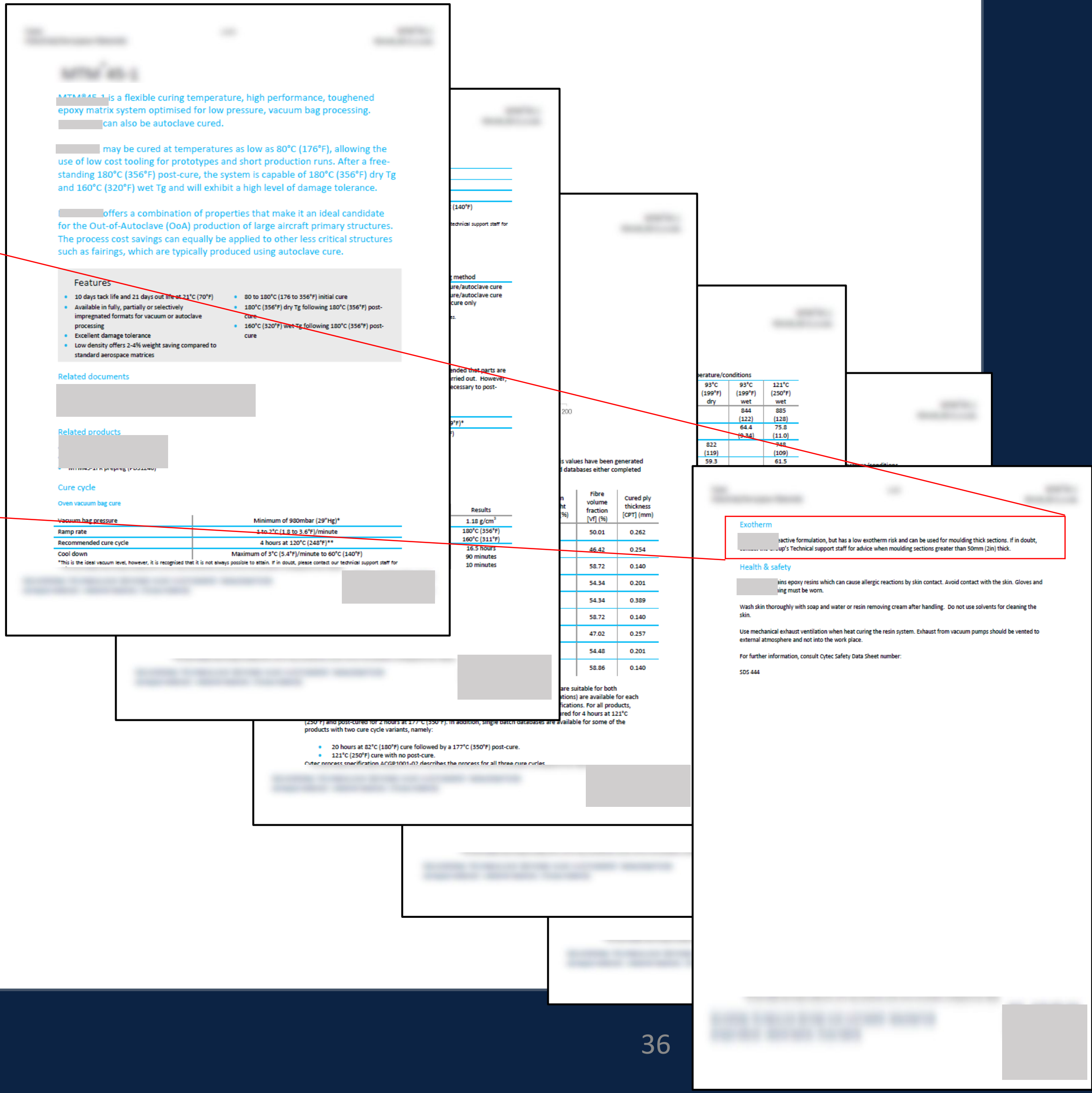
Example TDS – Prepreg Material

Exotherm

[redacted] is a reactive formulation, but has a low exotherm risk and can be used for moulding thick sections. If in doubt, contact the Group's Technical support staff for advice when moulding sections greater than 50mm (2in) thick.


[redacted] is a flexible curing temperature, high performance, toughened epoxy matrix system optimised for low pressure, vacuum bag processing. [redacted] can also be autoclave cured.

[redacted] may be cured at temperatures as low as 80°C (176°F), allowing the use of low cost tooling for prototypes and short production runs. After a free-standing 180°C (356°F) post-cure, the system is capable of 180°C (356°F) dry Tg and 160°C (320°F) wet Tg and will exhibit a high level of damage tolerance.

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Related documents

[redacted]

Related products

[redacted]

Cure cycle

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HOW ARE THE KEY MANUFACTURING PROPERTIES CHARACTERIZED?

Gel timer, rheometer, differential scanning calorimeter

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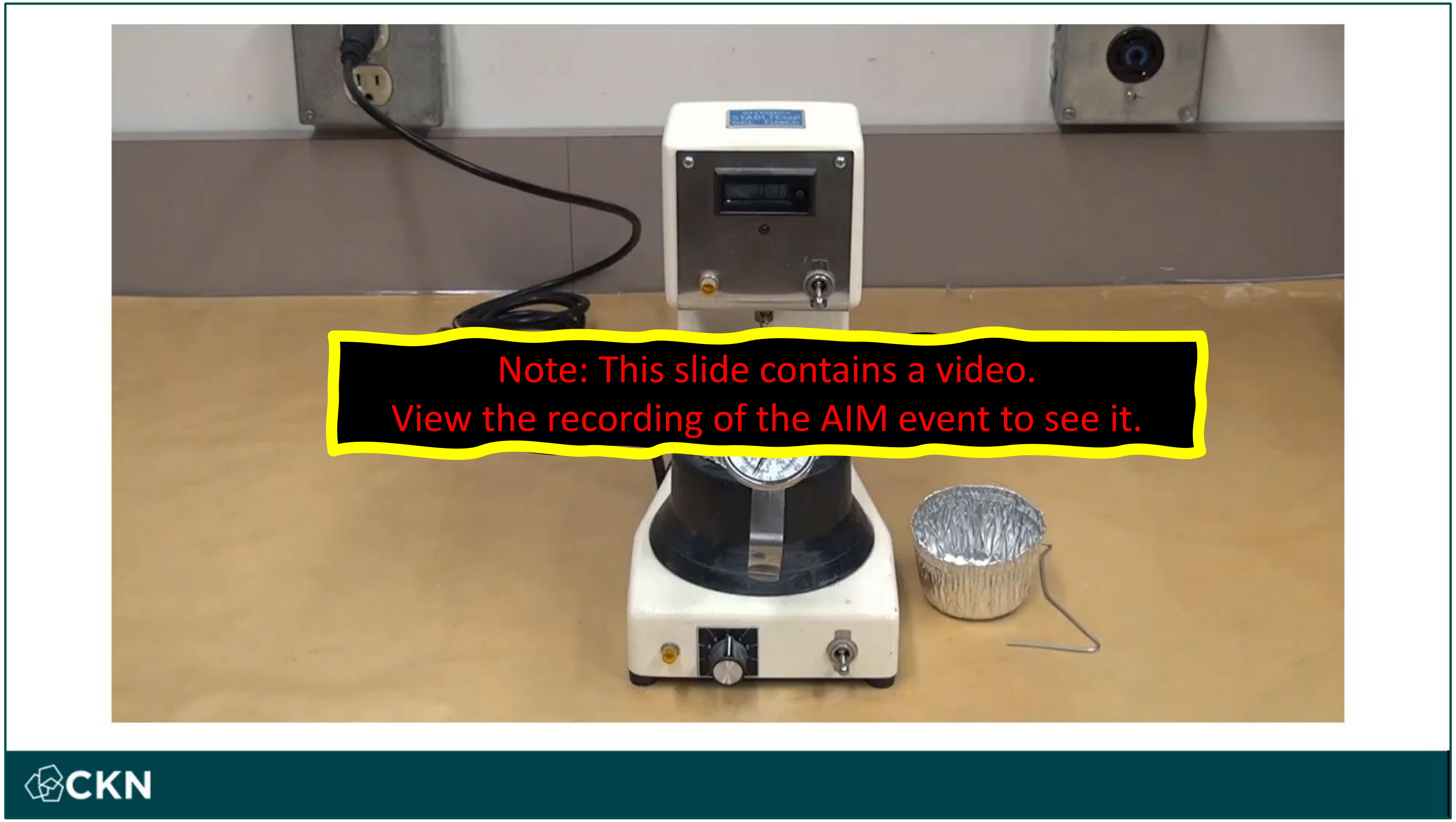
KPC M101: GEL TIME



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https://compositeskn.org/KPC/M101

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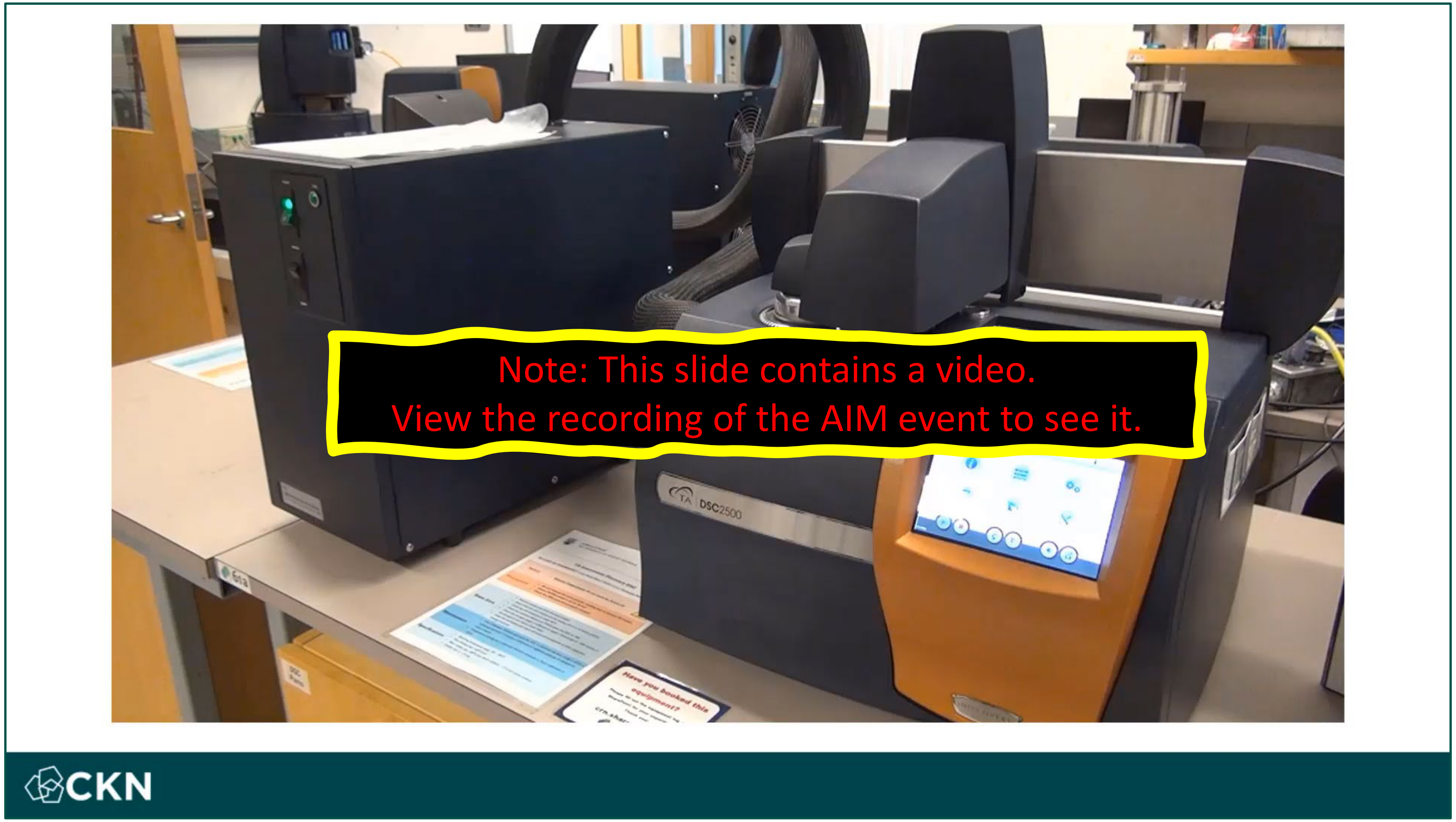
KPC M100: DEGREE OF CURE



Note: This slide contains a video.
View the recording of the AIM event to see it.

KPC A203: RHEOMETER

Summary

- There are a number of important resin properties and concepts one needs to understand when developing, optimizing, or troubleshooting a manufacturing process

Manufacturing Step	Analysis Type	Resin Property
Storage, formulation, impregnation, thermal transformation, demolding, secondary thermal transformation	Thermo-Chemical Analysis	Degree-of-cure
		Glass Transition (vitrification)
		Heat of Reaction
		Density
		Specific Heat Capacity
		Thermal Conductivity
	Flow-Compaction Analysis	Viscosity
		Gelation
Assembly	Stress-Deformation Analysis	(Visco) Elastic Constants
		Thermal Expansion
		Cure Shrinkage

Thank you for joining us!

Keep an eye out for announcements on the next AIM events
And don't forget to visit the KPC for more information:

https://compositeskn.org/knowledge-in-practice-centre/

Questions?

For more information on future dates and times visit:

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