Electron Beam Curing for Space Applications - Where are We?

Chris Saunders

Atomic Energy of Canada Limited

Special Thanks To:

Phillips Laboratory, Kirtland AFB

- Capt. Robert Pittman
- Dr. Bob Acree

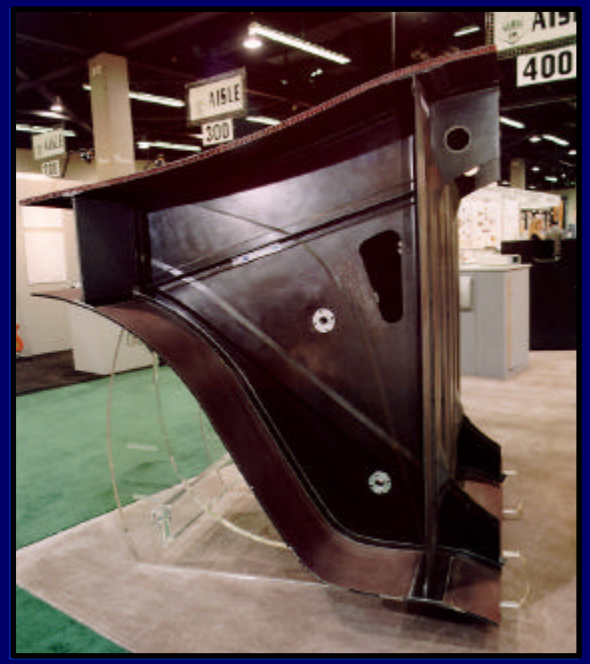
Process Description

- Alternative to heat
- Select curing temperature (-20°C to 200°C)
- Resin Chemistry
- Processing options
- Penetration Issues
- Repair processes/ adhesives

LMSW Tape-Placed Section

Benefits of EB Curing

- Select curing temperature
- Continuous/highly controllable
- Lowers product costs
  - higher production rate
  - cheaper tools
- Improved performance/ unique products

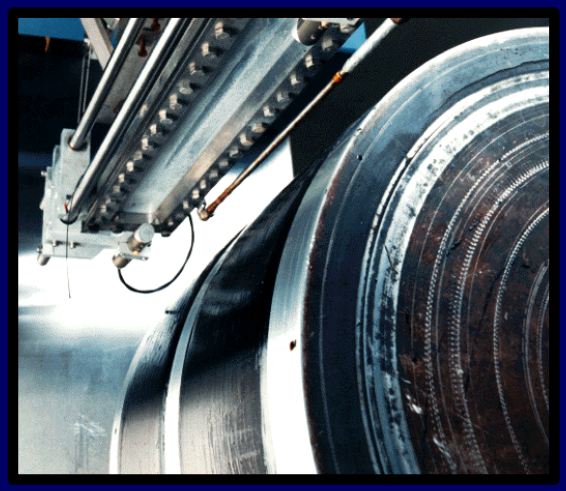


LMSW PD-4

*Are these benefits for space applications?

Typical EB Process

- Material selection
  - * Resins * Fibers
  - * Interface chemistry
  - *Adhesives



- Consolidation
- Fabrication

* Layup * Tape placement

* VARTM * Filament winding

- EB Cure/QA

Objectives of ECSA Program

- Identify/assess current EB technology
- Determine applicability to space
- Value of technical benefits

IMPELA

As of Today:

- Completed technical evaluation
  - benefits
  - materials/processing
  - current R&D
  - economics/size limits
    - Sample fabrication; testing at Phillips
    - Identify Phase 2 prototypes

EB Curing for Space

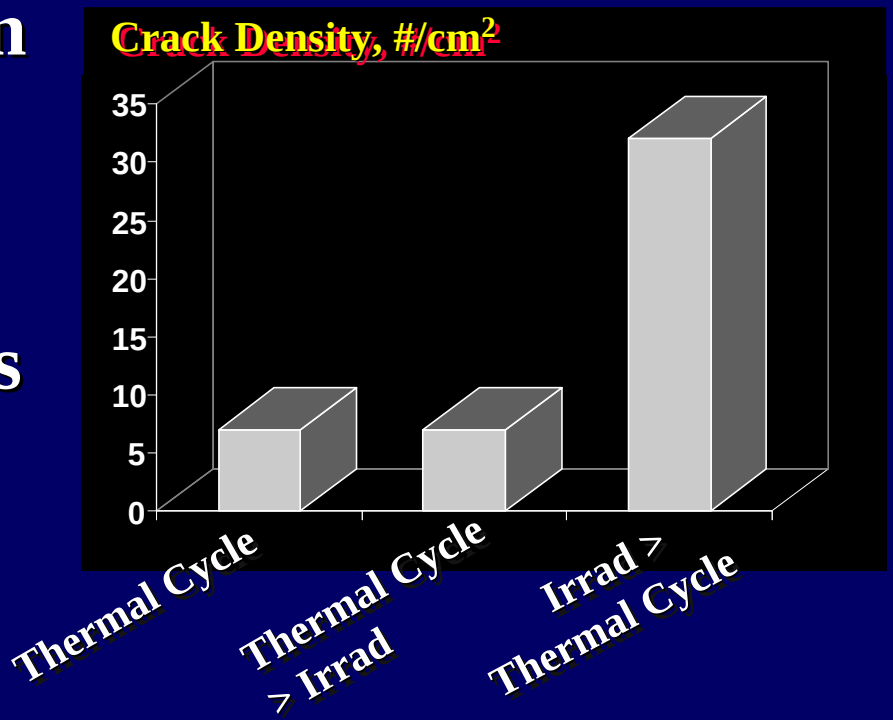
Issues:

- Value of Benefits
  - reduced stresses
  - design flexibility
- Material Performance;

(all aspects related to space)

Space Environment

- LEO & GEO
- AO/Solar UV/Micrometeoroids/Debris
- Ionizing Radiation
- Thermal Cycling
- Vacuum
- Synergistic Effects



Target Properties/Conditions for Space-Grade Polymers

Selected Target Properties	Target Values
Resin Shrinkage	< 5%
Composite Void Content	< 2%
Total Mass Loss (50% RH; composite)	< 1%
Collected Volatile Condensable Materials	< 0.1%
Temperature Range	-100°C to 150°C
Water Uptake	< 3%
Glass Transition Temperature (Tg)	> 200°C (Tan Delta)

Electron Curable Resins/Adhesives

- Limited work to date
- CRADA-produced information
- No long-term testing yet;
- Work continues

Features of EB-Curable Resins

Features	EB-Curable Epoxy	Thermosetting Epoxy
Mechanical Properties	high-performance	high-performance
Manufacturing Costs	moderate	high
Prepreg Storage/Handling	extended life @ 20°C	limited life @ 0°C
Environmental Concerns	low	moderate to high
Shrinkage on Curing (%)	2-3	4-6
Volatile Emissions (%)	<0.1	<1.0
Transition Temp. (°C)	up to 400	up to 300
Residual Stresses	low	moderate to high
Water Absorption (%)	<2	<6
Production Throughput	Fast	Slow

Features of EB-Curable Resins

Features	EB-Curable Epoxy	Thermosetting Epoxy
Thickness Limit	50 mm (EB) 200 mm (X-ray)	20 mm
Tooling Materials	metals, wood, ceramics, plastics, waxes, foams	metals, ceramics, graphite
Tooling Costs	low-moderate	moderate-high
Cure Time (10-mm-thick)	seconds-minutes	hours
Energy Requirements	low to moderate	moderate to high
Capital Cost (facility)	high	high to very high
Materials Availability	Resins/Initiators Available	Resins/Hardeners Available
Material Cost - complete system ($/lb)	2-5 (commercial), 8-20 (high-perf.)	2-4 (commercial), 8-20 (high-perf.)

Selected Neat Resin Properties

Selected Polymer Properties	Target Values	954-2A Cy. Ester	977-3 Epoxy	D-1	D-2
Resin Shrinkage, %	< 5%	<5%	~3%	3.5	3.0
Trans. Temp (^oC)*	> 200^oC	215	190	187	246
Tensile Str, MPa		68.9	--	44.1	56.3
Tensile Mod, GPa		3.03	--	3.47	3.36
Flexural Str, MPa		117	144.7		
Flexural Mod, GPa		0.44	3.79	3.26	3.5
Water Uptake, %	< 3%	1.28	3.0	1.9	2.1
Density, kg/m^3		1240	1200	1210	1220

* Tg based on Tan delta

Selected Neat Resin Properties

Selected Polymer Properties	Target Values	954-2A Cy. Ester	977-3 Epoxy	T-1	T-2
Resin Shrinkage, %	< 5%	<5%	~3%	3.6	2.5
Trans. Temp (^oC)*	> 200^oC	215	190	225	186
Tensile Str, MPa		68.9	--	40.0	35.0
Tensile Mod, GPa		3.03	--	3.70	3.50
Flexural Str, MPa		117	144.7		
Flexural Mod, GPa		0.44	3.79	3.68	3.03
Water Uptake, %	< 3%	1.28	3.0	2.2	2.1
Density, kg/m^3		1240	1200	1202	1198

* Tg based on Tan delta

Selected Resin Blend Properties

Polymer Property	Target Values	EB-1	EB-2	EB-3	EB-4	EB-5
Resin Shrinkage, %	< 5%	3.5	4.0	3.4	3.0	3.3
Trans. Temp (^oC)*	>200^oC	396	393	232	212	212
Water Uptake, %	< 3%	4.90	2.8	2.0	2.90	1.88
Density, kg/m^3		1260	1237	1225	1224	1228

* Tg based on Tan delta

Properties of EB-Cured Unidirectional Laminates

Resin Systems	Fiberite 977-3	EB-1	EB-2
Cure Conditions	3 h; 355°F; 85 psi	250 kGy	150 kGy
Void Volume, %	------	1.77	0.72
Tg, °C (Tan Delta)	190/240	396	392
O° Flex. Str., MPa	1765	1986	2006
O° Flex. Mod., GPa	150	196	163
O° Comp. Str., MPa	1680	1565	
O° Comp., Mod. GPa	154	149	
O° ILSS, MPa	127	77	79
Hot/Wet O° ILSS, MPa	89 (12.9)	61 (8.8)	

* 1 week. in H_2O @ 160°F, tested @ 220°F

Properties of EB-Cured Unidirectional Laminates

Resin Systems	Fiberite 977-3	EB-3	EB-4	EB-5
Cure Conditions	3 h; 355°F; 85 psi	150 kGy	150 kGy	150 kGy
Void Volume, %	------	1.24	0.64	1.18
Tg, °C (Tan Delta)	190/240	232	212	212
O° Flex. Str., MPa	1765	1793	1765	1710
O° Flex. Mod., GPa	150	163	154	150
O° Comp. Str., MPa	1680			
O° Comp., Mod. GPa	154			
O° ILSS, MPa	127	79	89	77
Hot/Wet O° ILSS, MPa	89 (12.9)			

* 1 week. in H_2O @ 160°F, tested @ 220°F

Properties of EB-Cured Unidirectional Laminates

Property	977-3	EB-6	EB-7	EB-8	EB-9
Service Temp.[2], °C	----	197	185	344	304
Transition Temp., °C	190/240	202	137/195	435	232
Resin Visc., poises, @ 70°C	----	108	114	102	102
Flexural Str, MPa	1765	1158	1190	1293	1307
Flexural Mod, GPa	150	143	159	148	144
Flexural Str, MPa (cycled)[3]	-----	1169	1244	1400	1272
Flex Mod, GPa (cycled)[3]	----	144	156	145	139
ILSS, MPa	127	47.3	50.0	47.6	71.4
ILSS, MPa (cycled)[3]	-----	59.3	58.3	54.2	72.4

1. IM7; GP sizing, 12K tow, 0.080”; debulked at 70°C for 1 h.
2. Service temperature: DMA modulus is at 50% .
3. Cycling procedure: -194°C for 30 min; 20 °C for 30 min; 121°C for 30 min; 20 °C for 30 min; 3 cycles.

Properties of EB-Cured Unidirectional Laminates

Property	977-3	EB-10	EB-11	EB-12
Service Temp.[2], °C	----	212	204	365
Transition Temp., °C	190/240	157/420	210	128/435
Resin Visc., poises, @ 70°C	----	74.5	270	223
Flexural Str, MPa	1765	1330	1031	1107
Flexural Mod, GPa	150	146	143	144
Flexural Str, MPa (cycled)[3]	----	1317	1083	1228
Flex Mod, GPa (cycled)[3]	----	147	147	142
ILSS, MPa	127	71.7	52.4	42.7
ILSS, MPa (cycled)[3]	----	75.5	58.6	50.4

1. IM7; GP sizing, 12K tow, 0.080”; debulked at 70°C for 1 h.
2. Service temperature: DMA modulus is at 50% .
3. Cycling procedure: -194°C for 30 min; 20 °C for 30 min; 121°C for 30 min; 20 °C for 30 min; 3 cycles.

Total Mass Loss in EB-Cured Polymers

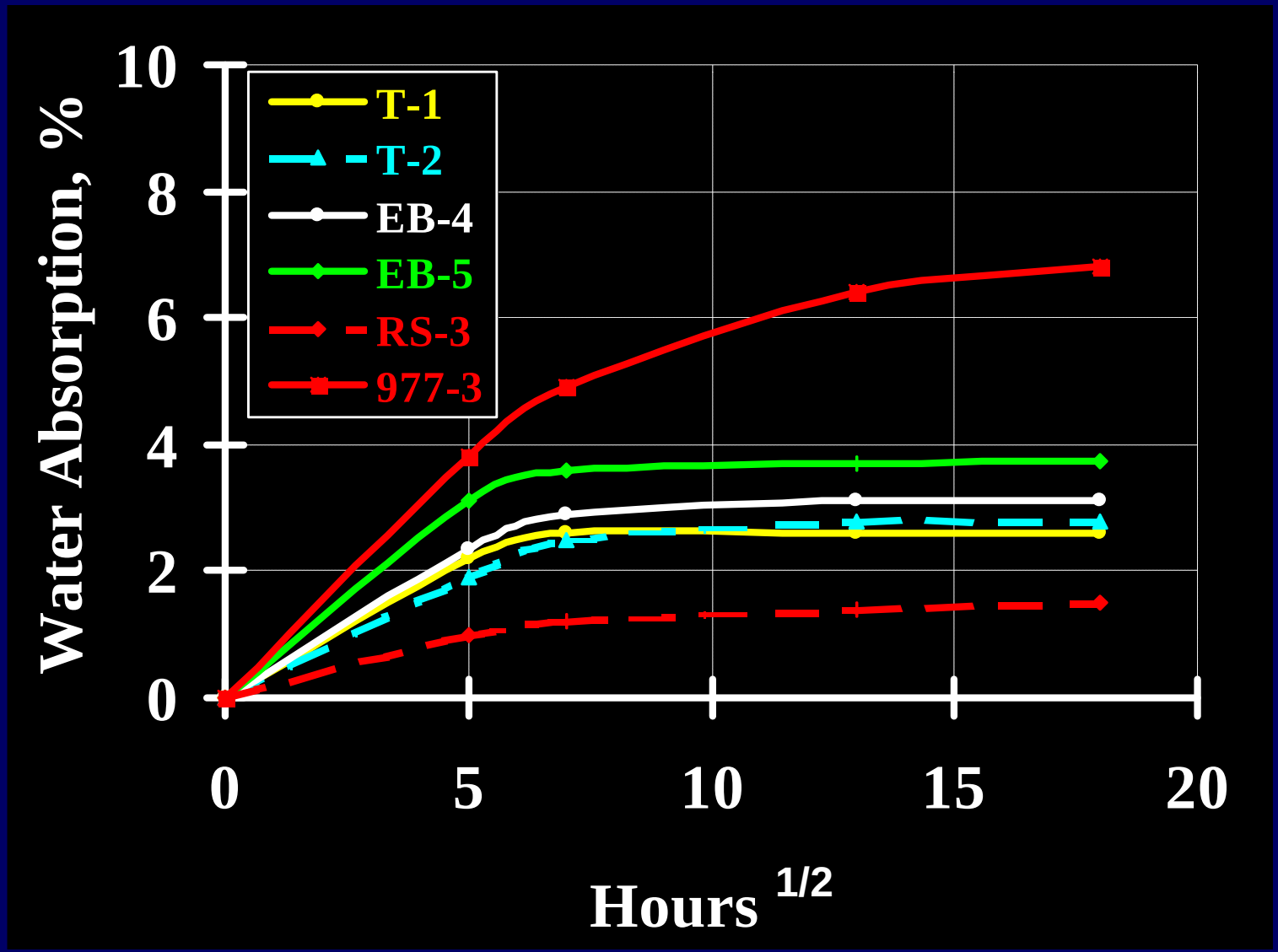
Resin	Total Mass Loss, %
EB-1	0.81
EB-2	0.42
EB-3	TBD
EB-4	0.79
EB-5	0.90
TD-1	0.90
TD-2	0.42
RS-3*	0.40

* Cyanate Ester

Out-gassing of EB-Cured Composites

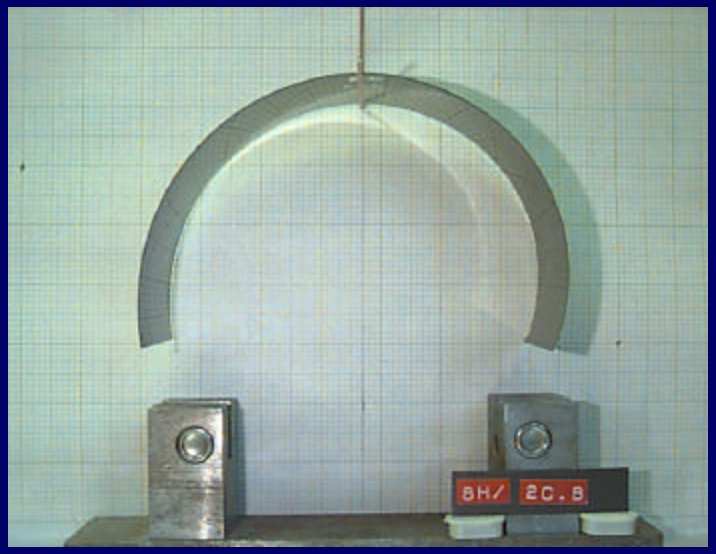
Selected Properties	RS-3 Cy. Ester	EB-1	EB-2	EB-4	EB-5
Total Mass Loss, %	0.12	0.27	0.13	0.23	0.30
CVCM, %	0.06	0.13	0.15	0.14	0.10

Moisture Absorption in Selected Polymers



Effect of Cure Temperature on Internal Stress

25^{0}C;EB-5; IM-7; 2-ply; 50% RH



133^{0}C;EB-5; IM-7; 2-ply; 50% RH

Effects of Thermal Cycling on Internal Stress

Selected Properties	115°C Cure	69°C Cure	25°C Cure	0°C Cure
Initial Radius, mm @ 25°C	107	141	311	**
Radius After One Cycle, mm	102	111	93	206
Radius After Five Cycles, mm	100	88	86	128
Normalized Stress, after 0 cycles, % *	291	221	100	**
Normalized Stress, after 1 cycle, % *	91	84	100	45
Normalized Stress, after 5 cycles, % *	86	98	100	67

* Normalized to 25°C ** Flat Sample

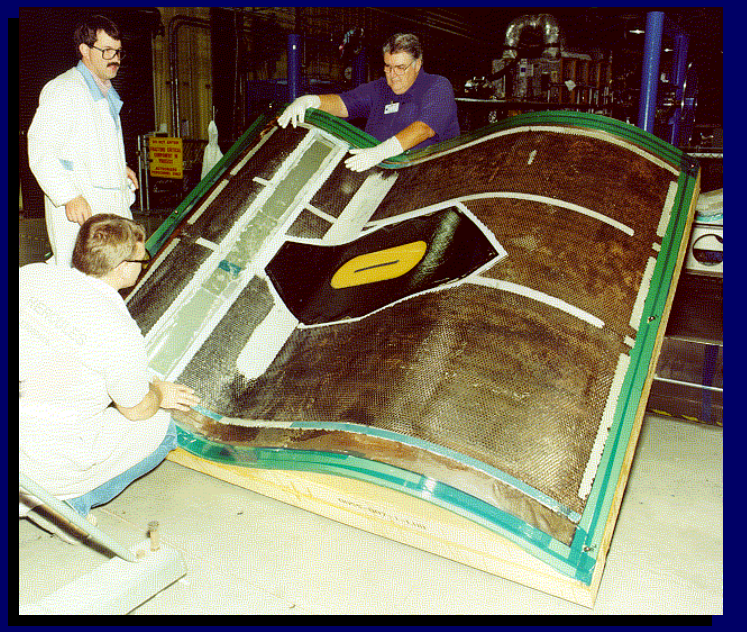
EB-5; IM-7; 2-ply; 50% RH

Products of Interest

-
-
- Launch vehicles
- ???????

Industrial EB Processing

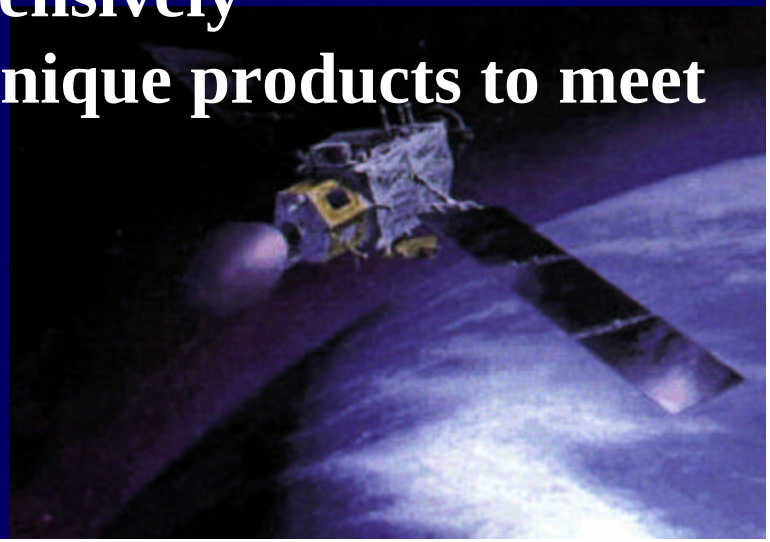
Product Size Limits



- Contract facilities
  - EB Services: 3.7 m x 0.8 m x 0.8 m; 380 kg
  - IOTRON: 2.5 m x 1.1 m x 1.0 m; 250 kg
  - AECL: 2.7 m x 1.2 m x 0.6 m; 100 kg
- Aerospatiale: 10 m x 4 m dia (FW)
-

Value of EB Curing

- Can promised cost savings be realized?
- Requires team of “experts”
- True value not in replacing autoclaves for making existing products less expensively
- True value is in making unique products to meet ever-increasing demands



Future of EB Curing

- Long-term product performance data needed
- Follow the path to production

- Process demonstration /certification
- Industry leadership/ cooperation
- Must put “value” to technology

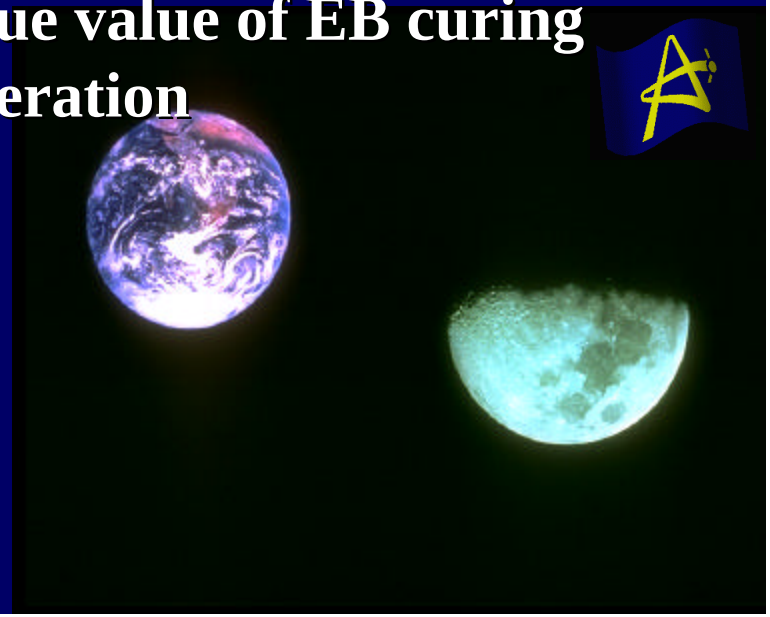
EB Curing Within 3 Years

- Qualified status technically possible
- Resins/processing advances need to continue at current pace
- Full-scale EB facility needed (LMES already interested)
- Full industrial cooperation
- Prototyping needs to start now

Products of Interest

- Cryogenic tanks
- Reflector structures
- Launch vehicles
- ???????

Summary of Electron Curing for Space Applications

- EB curing continues to advance
  - resin development needs to continue
  - interface improvements needed
- Processing/QA needs advancing
- Progress to determining true value of EB curing for space - Industrial cooperation
- Path to Production -

  Take the Next Step