Evaluation and Validation of Organic Materials for Advanced Stirling Convertors (ASCs): Overview

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13th IECEC/AIAA Propulsion & Energy, 27 - 29 July, 2015

Orlando, Florida

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- L. Thieme, J. Schreiber, W. Wong, S. Wilson, NASA-GRC for project guidance and management, T. Williams, NASA-GRC for reviewing various reports and this paper.
- This work was sponsored by the GRC-Radioisotope Power System (RPS) program office with funding from Science Mission Directorate (SMD).

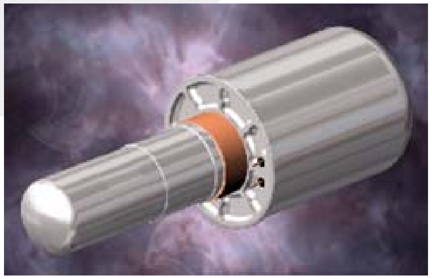
Table of Contents

- Backgrounds and Objectives
- Materials and Processes
- Results and Discussion
  - Aging Behavior of Epoxy Adhesive
  - Aging Behavior of Other Susceptible ASC Organics
  - Evaluation of Thread Lockers
  - Radiation Hardness of ASC Organics
  - Outgassing Behavior and Impact Assessment of ASC Organics
  - Synergistic Durability of ASC Organics
  - Selection of High Temperature Organic Candidates for Future High Temperature ASCs
- Conclusions

Backgrounds

- Organics in ASC for design flexibility or unique properties and functionalities, such as bonding, potting, sealing, thread locking, insulation, and lubrication: a total of ~ 22 gram
- Convertor operating environment
  - Pressurized with dry inert gas and hermetically sealed, but potential outgasses from organics or residual contaminants
  - Elevated operating temperatures and radiation exposures
- Long mission cycles up to 17 years, such as deep space explorations

Can the organics sustain and perform?



Objectives

- Screen, evaluate, and validate organics for the ASC applications, more specifically in terms of
  - Performance, durability, and reliability
  - Material compatibility
- Identify application limits of each organic materials, and develop their performance and lifetime predictions

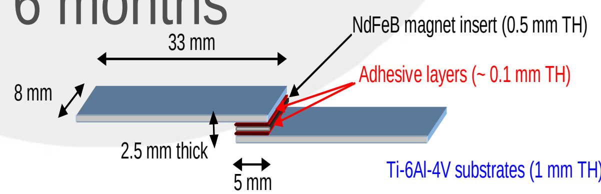
II. Materials and Processes

- Organics in the linear alternator include epoxy potting compounds and adhesives, coil backing paper, liquid locking compounds, o-ring, bearing surface coatings, wire coating or insulations, and shrink tubing.
- Due to their export control and proprietary nature, only identified by their generic material type or functionalities, not by their specific brand name or locations.
- The organic materials tested in this paper were:
  - purchased as the final form, e.g., o-ring, backing paper, wire insulation, or
  - processed to test specimen or coupons from the raw materials purchased
- Fabrication of test specimens followed the standardized conditions and procedures including pre-installation bake-out used for the actual components

III. A. Aging Behavior of Epoxy Adhesive

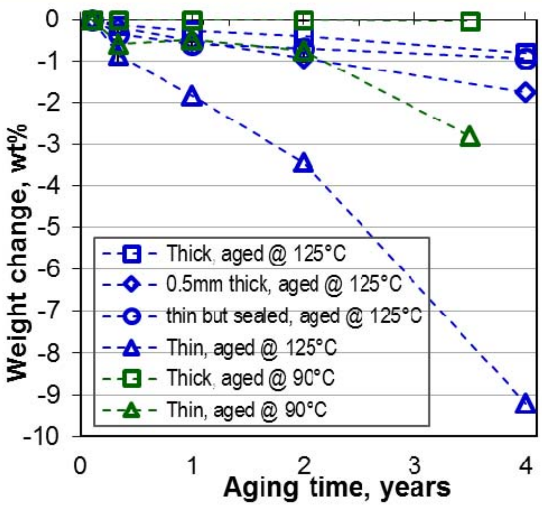
- Most critical organic, single point failure
- Extensive and systematic evaluations via:
  - Mechanical bonding integrity in static and fatigue mode
    - Component level full-scale testing: results met the required specifications
  - Longer-term in-service aging tests at 90 and 125 ºC for 4 years
    - conducted under an inert gas environment to assume the original ASC service conditions

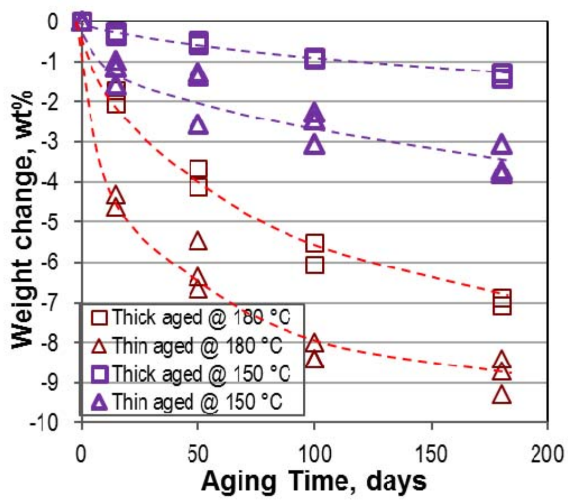
  - Thermally accelerated aging up to 180 ºC for 6 months
    - T_{max} for the accelerated aging determined from 15-day thermal aging tests up to 245 ºC

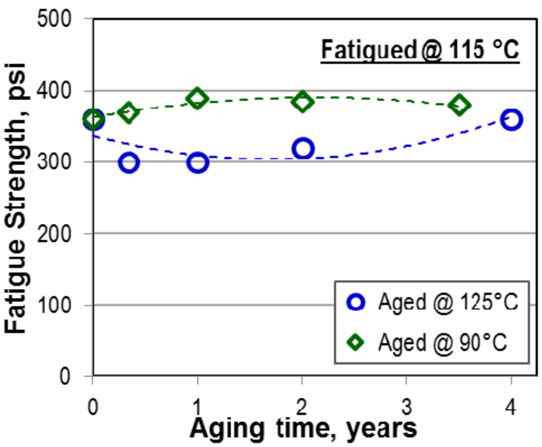


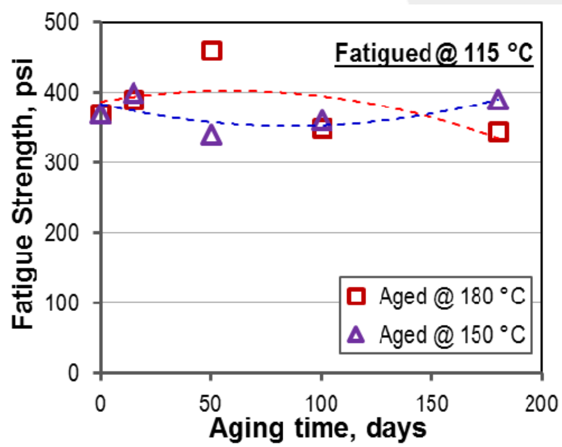
  - Used (i) Epoxy-alone sheet specimens, thin (0.15 mm) and thick (1.5 mm), (ii) Sub-scale sandwich lap shear specimens

III. A. Aging Behavior of Epoxy Adhesive, Cont'd







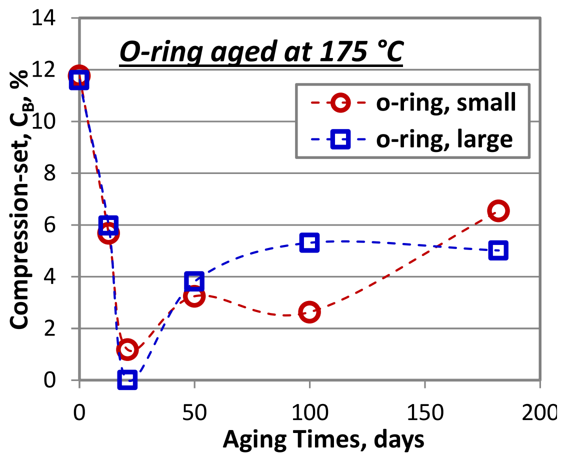


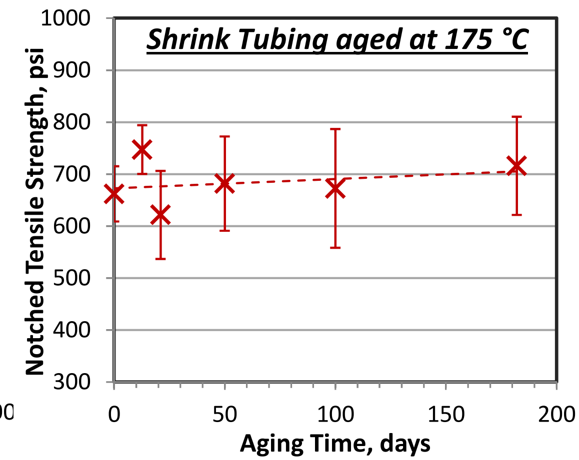
- Significant wt losses, but strongly temp., thickness, or configuration (e.g., sealed) dependent, i.e., a diffusion-controlled process as outgassing
- T_g was only other noticeable property change at early t_a, but no impact on adhesion
- No changes in molecular network structures from either 4-yr life testing or 6-m accelerated aging tests
- No impact on the overall bonding integrity of the epoxy adhesive in terms of both static and fatigue strength

III. B. Aging Behavior of Other Susceptible ASC Organics

- O-ring, Shrink tubing identified from preliminary evaluation
- Thermal stability and reliability assessed via:
  - Thermally accelerated aging tests at 150 and 175 ºC for 6 months
    - T_{max} for the accel. aging determined from 12-day thermal aging tests up to 225 ºC
    - conducted under the inert gas environment to assume the original ASC conditions
  - Used (i) two size commercial o-ring samples, 013 (Nominal ID=7/16", OD=9/16", CS=1/16") and 021 (Nominal ID=15/16", OD=1-1/16", CS=1/16"), baked @ 110 ºC (ii) 3/16" OD commercial shrink tubing section samples shrunk on 3/32" OD aluminum rod at 170 ºC, 30 min, and baked @ 90 ºC

III. B. Aging Behavior of Other Susceptible ASC Organics, cont'd





Aging T. / Material	12-day short-term thermal aging					
	23 °C	90 °C	125 °C	150 °C	175 °C	225 °C
o-ring, small	good	good	good	good	good	poor
o-ring, large	good	good	good	good	good	poor
Shrink Tubing	good	good	good	good	good	poor
Aging T. / Material	**6-month accelerated thermal aging at 150 °C**					
	0 day	15 days	50 days	100 days	180 days	
o-ring, small	good	good	good	good	High wt loss	
o-ring, large	good	good	good	good	good	
Shrink Tubing	good	good	good	good	good	
Aging T. / Material	**6-month accelerated thermal aging at 175 °C**					
	0 day	15 days	50 days	100 days	180 days	
o-ring, small	good	good	good	good	High wt loss	
o-ring, large	good	good	good	good	good	
Shrink Tubing	good	good	good	good	good	

- The aging behavior of the o-ring size-dependent, e.g., small-size suffered significantly higher wt losses and thickness reduction than the large --- Probably due to process conditions but to be further evaluated for the ASC applications
- Overall, no visible property degradations or structural changes from the 6-m accel. aging in both o-ring and shrink tubing
- Thus, good thermal stability up to 175 ºC

III. C. Evaluation of Thread Lockers

- Two commercial liquid locking compounds (LLC) to provide the secondary thread locking mechanism for the 8 ASC joints; but
  - never fully proven for a space flight hardware
  - their performance strongly affected by installation and process conditions
- Experimental validation required in terms of cure state, application method, joint conditions and configurations followed by the NASA specifications, NASA-STD-5017 & 5020, and the final report of NASA Engineering and Safety Center's (NESC) investigations, NESC-RP-04-0929
- A full-scale component level torque testing system capable of testing up to 150 ºC was developed and certified for this investigation.

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III. C. Evaluation of Thread Lockers, Cont'd

Overall Test Plan

Phase	Experiments	LLC Type	Properties / Joint Type	# of Torque Test				Postmortem, #		
				23°C	90°C	130°C	150°C	Δwt%	sOM	FT-IR
Phase I	Baseline Control	LLC 1	Joint 2 - 8	**24**	**21**	**21**	**18**	**7**	**7**	**7**
		LLC 2	Joint 1	**6**	**12**	**12**	**6**	**1**	**1**	**1**
Phase II	Process validation: 7 variations	LLC 1	Joint 2, 6, 7, 8	**9**	**84**	**84**	**9**	**31**	**31**	**31**
	- Cleaning solvent	Loctite242								
	- Quantity & coverage									
	- Alternative LLC*									
	- Other Preloads†	LLC 2	Joint 1		**21**	**15**		**5**	**5**	**5**
	- Application Blind Holes‡	Loctite246								
Phase III	Cure kinetics	LLC 1	Joint 6, 7, 8		**27**	**27**		**9**	**9**	**9**
	- at 3 cure conditions§	LLC 2	Joint 1		**9**	**6**		**3**	**3**	**3**
	10-d aging	LLC 1	Joint 6, 7, 8		**45**	**45**		**15**	**90**	**30**
	- at 5 temperatures¶	LLC 2	Joint 1		**5**	**5**		**5**	**5**	**5**
	150-d Accelerated aging	LLC 1	Joint 6, 7, 8		**81**	**72**		**24**	**144**	**48**
	- at 2 temperatures, 4 times#	LLC 2	Joint 1		**9**			**3**	**9**	**4**
			Total # of test	39	314	287	33	103	304	143

* Loctite 242 for LLC 1 and Loctite 246 for LLC 2

† zero, lower, or higher than standard (1.35x for joint 1, 1.03 - 1.25X for all others)

‡ This applied only on Joint 2

§ 25-hour cure at 80 °C, 70 °C, and 22 °C vs. the standard cure, 25-hour at 60 °C

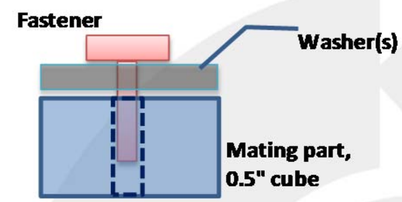
¶ 90 °C, 125 °C, 150 °C, 175 °C, and 200 °C under inert gas (N2)

\# 165 °C and 185 °C for 10, 50, 100, and 150 days under inert gas (N2) determined from 10-d aging tests

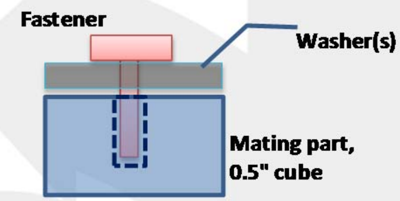
III. C. Evaluation of Thread Lockers, Cont'd

Design of specimen assemblies:

The mating parts and washers made of various metals identical to those of the actual ASC joint components assembled with the fasteners



Thru-hole configuration



Blind-hole configuration

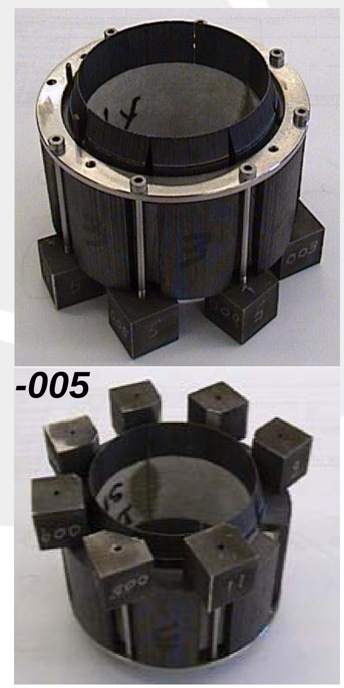
For -001 check valve fasteners, the actual piston parts were used instead of cubes.

-001

-002

-003

-004



-005

-006

-007

-008

III. C. Evaluation of Thread Lockers, Cont'd

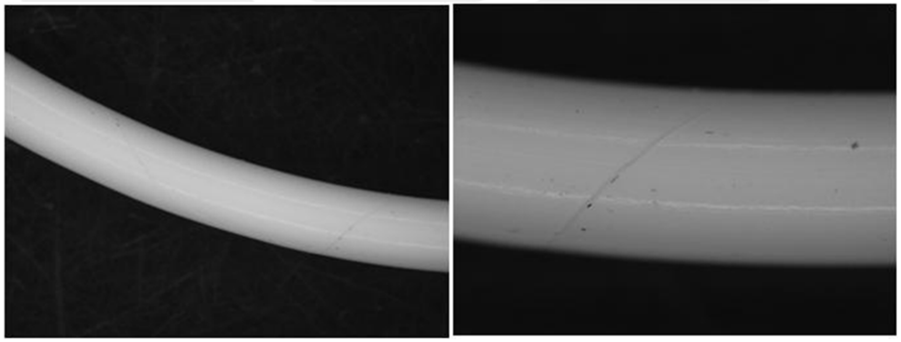
- In general, the secondary locking behavior and performance of the Loctite LLCs were strongly dependent on type of the fastener or mating part in terms of metal ion type, degree of thermal expansion, thread gap distance, and hole configuration, e.g., thru or blind besides preload, etc.
- Effects of application methods and conditions varied, e.g., negatively with cleaning solvent, but less or no changes with application quantity, installation torque, application sequence, or strength of LLCs.
- In conclusion, both LLCs were thermally stable and robust up to 185 ºC, thus were validated for the current ASC applications regardless of joint type.
- It was recommended, however, that more aggressive bake-out processes at higher temperatures for longer time, in addition to the standard cure cycles, should be applied for most ASC joint systems for improved thread locking integrity, whenever possible.

III. D. Radiation Hardness of ASC Organics

- Radiation environment: ASRG-Induced/internal and Natural/external + typical radiation design factor of two
- Organics most susceptible to radiation, strongly affected by the exposure conditions, e.g., temperature, and sample configuration, e.g., thickness
- Validation of radiation hardness of ASC organics via:
  - Coupon-level gamma irradiation testing at High Flux Isotope Reactor (HFIR) spent fuel pool facilities, Oak Ridge National Laboratory (ORNL)
    - 100 krad to 15 Mrad at both 125 and 150 ºC, under inert gas environment
  - Coupon-level neutron irradiation testing at the Texas A&M U TRIGA reactor facilities
    - up to 5×10^{14} n/cm^2 fluency at both 125 and 150 ºC, under inert gas environment
  - ASC alternator subsystem, so-called the Stirling Alternator Radiation Test Article (SARTA), gamma irradiation testing at the gamma irradiation facility (GIF), Sandia National Laboratories (SNL)
    - up to ~ 40 Mrad under the nominal stroke and pressure at both 90 and 125 ºC

III. D. Radiation Hardness of ASC Organics, Cont'd

- Overall, most ASC organics showed a good tolerance to the combined thermal and gamma or neutron radiation exposures based on the ASC specifications, and thus validated for the ASC application
- However, in the case of Teflon wire insulation, the radiation caused embrittlement
  - approximate threshold gamma dose for cracking: 25 Mrad at 125 ºC or 15 Mrad at 150 ºC
  - significant increases in crystallinity and morphological structural changes with irradiation
  - Even though the current Teflon insulation met the specification guidelines, replaced with more radiation resistant material based on the recommendation from this study in order to mitigate the potential risk associated with the uncertainties
- SARTA irradiation testing also showed minimal degradation of all internal components, chemically or physically
  - Cracks on Teflon wire insulation
  - No significant changes on the overall alternator performance up to 40 Mrad

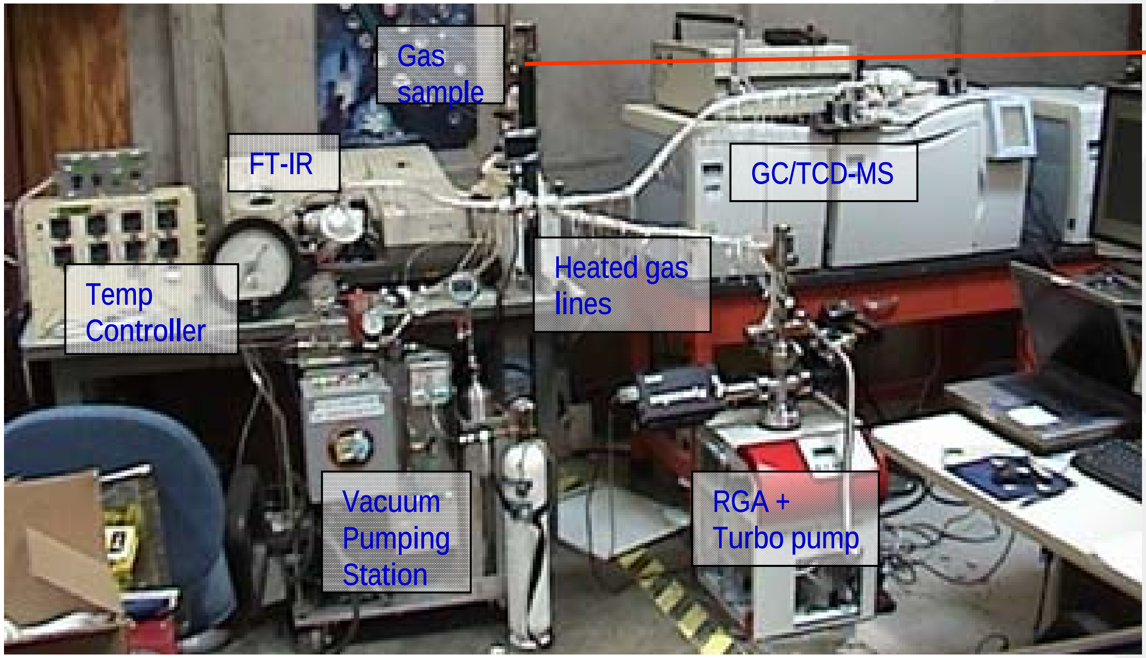


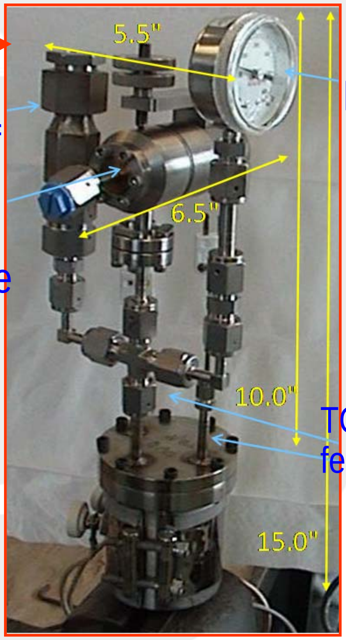
III. E. Outgassing Behavior and Impact Assessment

- ASC environment: pressurized with dry, ultrahigh-purity inert gas and hermetically sealed
  - Potential outgassing from the thermally susceptible organics can contaminate the working fluid in the system and degrade overall convertor performance or even cause a system break-down --- evidenced by considerable wt losses of organics
- Quantitative outgassing assessment of ASC organics via:
  - In-situ outgassing monitoring of individual organics as a function of T and t
    - up to 200 ºC for 28 days, under inert gas simulated environment
  - In-situ outgassing monitoring of all combined ASC organics
    - up to 150 ºC or 200 ºC for accelerated testing for 28 days, under inert gas environment
  - Gamma-induced outgassing test at the gamma irradiation facility (GIF), Sandia National Laboratories (SNL): individual organics vs. all combined organics
    - up to 20 Mrad at both 125 and 150 ºC for a total of 17 hours for the individual test
    - Up to 229 krad at 0.1 rads-Si/sec at 125 ºC under the simulated inert gas environment

III. E. Outgassing Behavior and Impact Assessment, Cont'd

Integrated RGA-GC/TCD-MS-FTIR gas analysis system at GRC





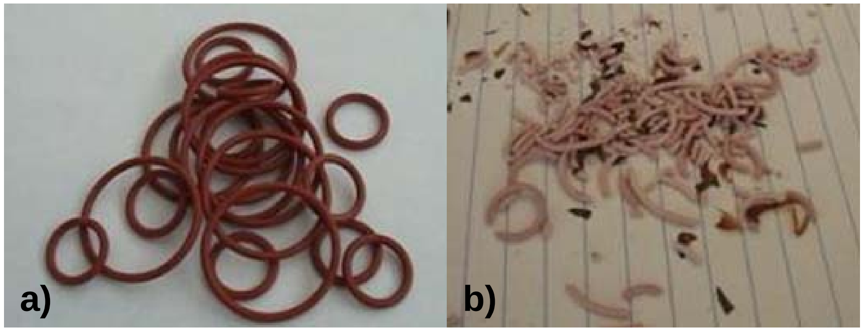
- The outgas-induced degradations or their mechanisms were assessed via the systematic residual property characterizations of the exposed organics after the in-situ outgassing experiments

III. E. Outgassing Behavior and Impact Assessment, Cont'd

- From the individual organics:
  - most common outgas species regardless of the exposure condition, either temp-alone or gamma-induced: H_2, CH_4, H_2O, N_2, O_2, and CO_2
  - N_2 was most dominant but its concentration not affected by exposure conditions
  - only CO_2 increased consistently with increasing temperature
  - note that part of the outgases were residual air contaminant left in the PVs
  - The combined T and γ radiation seemed to accelerate outgassing of several organics, e.g., epoxy adhesive, o-ring, or shrink tube when exposed up to 20 Mrad
  - In the case of the shrink tubing at or above 150 ºC, RGA indicated two additional species, possibly C_4H_{10} (n-Butane) and CF_4 (Tetrafluoromethane) → molecular break-down?
- From the combined organics outgassing tests:
  - outgas species and behavior either under temp-alone or from the low dose gamma-induced test at below 150 ºC almost identical to those from individual organic material
  - At 150 ºC, o-ring, shrink tubing, and the bearing surface coating material showed some-degree of susceptibility to the combined temperature and radiation exposure.
  - However, no major chemical structural changes or degradation of ASC organics was identified from the lower temperature outgassing tests below 150 ºC

III. E. Outgassing Behavior and Impact Assessment, Cont'd

- From the accelerated outgassing test of combined organics at higher T up to 200 ºC;
  - At above 150 ºC, the shrink tube materials suffered outgas-induced thermal degradation and generated potentially harmful outgas containing fluorine.
  - Thus, T_{max} of the shrink tubing for longer-term use < 150 ºC.
  - At 200 ºC, several new outgas species with higher Mw, e.g., 47, 77, 81, 96, as a result of thermal decomposition and their concentrations increased considerably with aging time
    - the suspected sequence of degradation mechanisms: outgasses from the combined ASC organics at above 125 ° C attacked and degraded the shrink tubing → additional fluorine containing outgasses were generated at ~150 ° C or higher → other gas species via inter-gas chemical reactions were formed → o-ring and other organics were attacked, decomposed or de-polymerized at ~200 ° C → additional higher molecular weight outgasses contained Si were generated → other organics, e.g., epoxy adhesive, thread lockers etc., were attacked.



Effect of outgases on o-ring degradation:

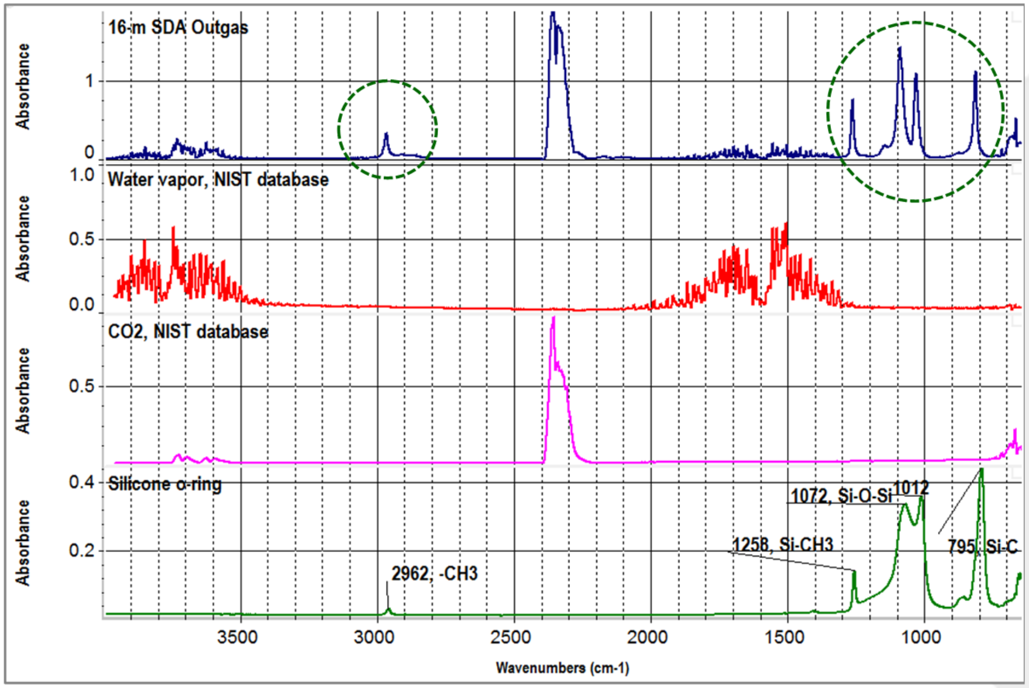
a)no degradation after exposure to 200 ° C but individually, i.e., o-ring alone

b)after 200 ° C exposure with other ASC organics together for 117 days.

III. F. Synergistic Durability of ASC Organics

- The combined effects of irradiation, thermal exposure, and synergistic organic outgassing interactions on stability and durability of ASC organics
  - Irradiated organic samples and coupons as a function of radiation type, γ or neutron, and dose or fluency at both 125 and 150 ºC
    - With objective of determining potential impact of the suspected reactive free radicals in most organic materials generated from the irradiation
  - Thermal aging at 130 ºC, the maximum alternator qualification temperature, for 5 months and 16 months in the pressurized inert gas ASC simulated environment
    - → Various outgas analyses
    - → systematic residual property characterizations of the aged organics samples
    - → determine the synergistic effects on stability and compatibility of the organics

III. F. Synergistic Durability of ASC Organics, Cont'd



- Typical outgas species: H_2, CH_4, H_2O, Ne, N_2, O_2, Ar, CO_2, small amount of acetone, C_3H6_O, and the host gas
- consistent with the actual ASC test units involving trapped air and/or cleaning solvent
- ranged from 10 to 6000 ppm and varied with aging time
- FT-IR analysis also suggested presence of Silicon vapor

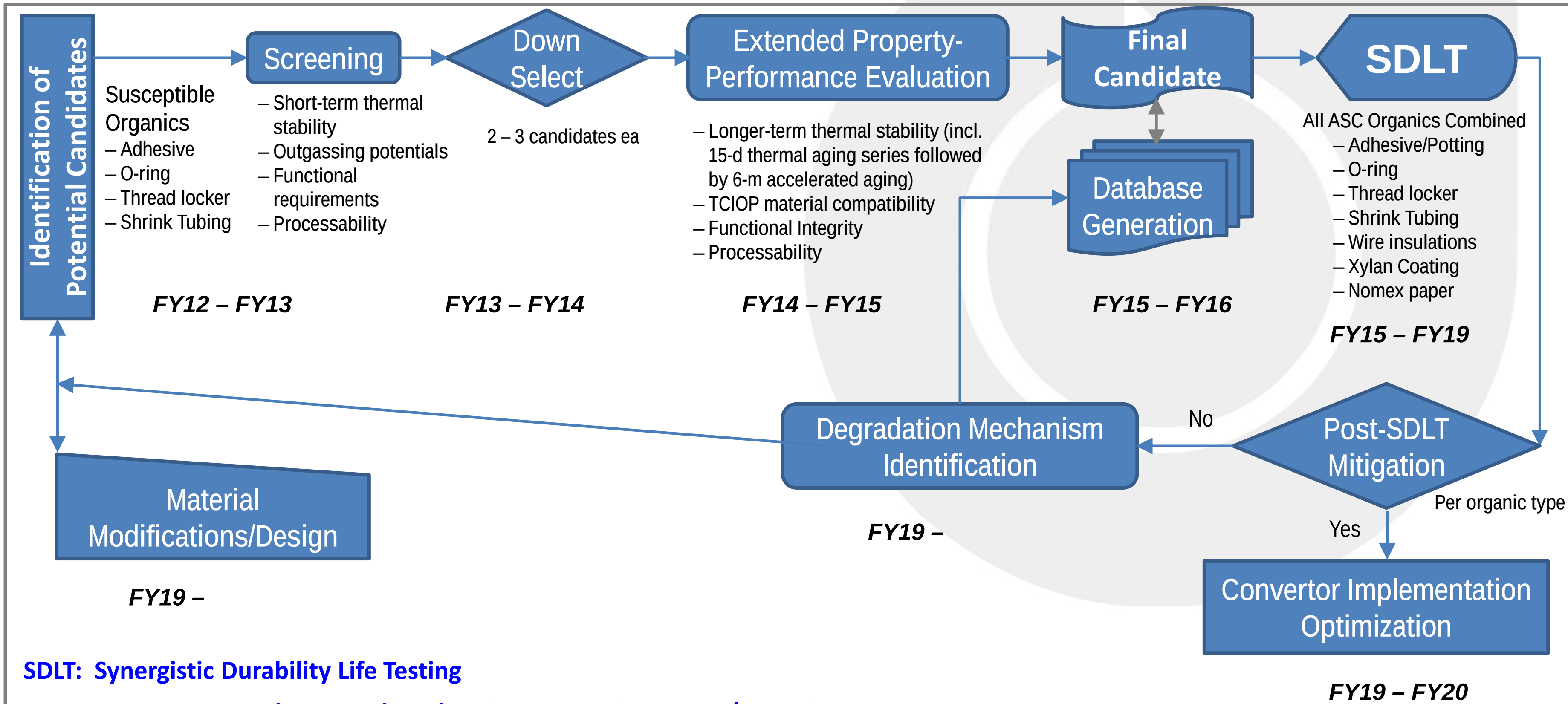
- O-ring and shrink tubing suffered most significant weight losses
- O-ring also showed changes in molecular network structure, decreased thermal properties, higher outgassing potential, but no significant changes in compression-set property
- Most of other organics were stable and compatible with the ASC environment, i.e., validated

III. G. Selection of High Temperature Organic Candidates for Future High Temperature ASCs

- Efforts to develop higher temperature ASCs, potentially 50 to 100 ºC higher operating temperatures than the current ones
  - to allow additional missions, e.g., ones that require a Venus fly by
  - to improve overall efficiency and performance of the convertors
- Requires organics with much higher temperature capability in the first place since organics are more susceptible to temperature changes
  - the higher temperature capable organics can significantly improve reliability margin of the current ASCs
  - most of the aforementioned issues or concerns on the organic materials against the Stirling convertor applications are extremely temperature-sensitive.
  - screening and evaluation of higher temperature organics were initiated in 2012

III. G. Selection of High Temperature Organic Candidates for Future High Temperature ASCs, Cont'd

Overall program plan for best high temperature organics selection

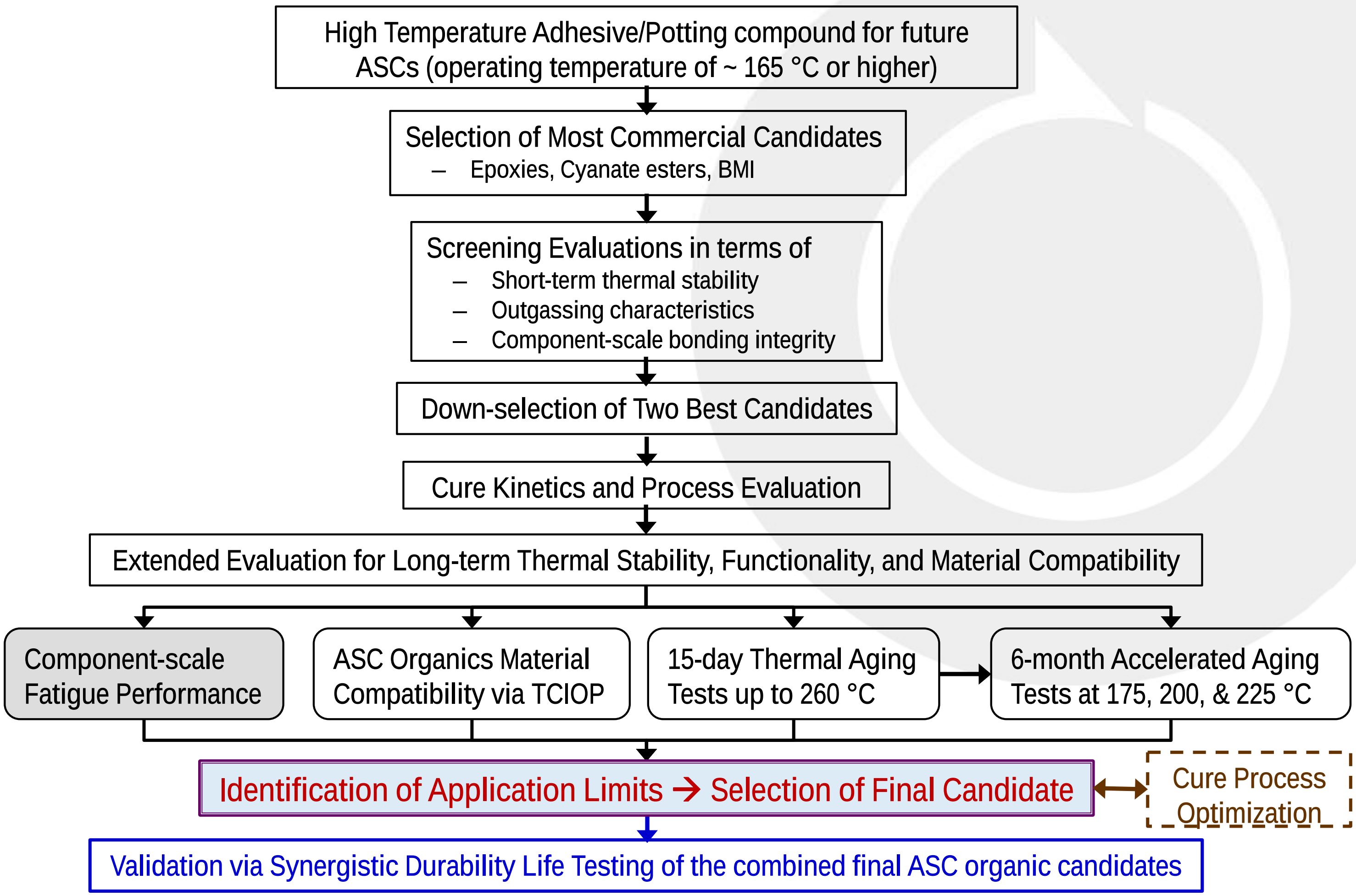


SDLT: Synergistic Durability Life Testing

TCIOP: Temperature-alone Combined In-situ Outgassing test w/ Pre-mix ASC outgas

III. G. Selection of High Temperature Organic Candidates for Future High Temperature ASCs, Cont'd

Selection process of best high temperature adhesive candidate



Conclusions

- The current ASC organic materials including certain backup candidates were extensively and systematically evaluated and successfully validated for various proposed missions after a few mitigation recommendations on a couple of organics such as shrink tubing and o-ring.
- The Teflon insulation material was replaced with more radiation resistant material.
- Practical and specific application limits of each ASC organics were also identified for any future ASC development.
- Based on the findings and experiences from the current ASC organics evaluation and validation, the methodology and procedure of screening and down selecting the final candidates for the future higher temperature ASCs were streamlined and optimized.

Thank You for your attention!

Any Questions?

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