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Evaluation of Cryogenic CCS Seal Integrity using an Incremental Computational Approach

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Overview

Presenter
Jeremy Hemingway, PE
Stress Engineering Services

Co-Author
Chris Folta, M.E.
Janssen, J&J

Computational Modeling is one of many effective tools which can be implemented throughout a product's life cycle. It is commonly used to 'check' designs, however there is a significant opportunity to utilize it to develop designs, project plans, and support risk assessments.

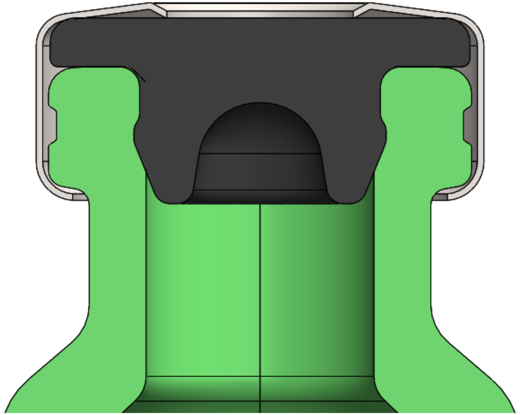
Introduction

- Beneficial to incrementally develop a computational model(s) in parallel with the development cycle, not an *after-thought*.
- The diligence of the model should be appropriate to the objectives of the current development stage, for example:
  - Feasibility → Can it work? *(Subsystems level)*
  - Early Design → Identify sensitivities
  - Detailed design → Establish design margin *(System level)*
  - Sustaining → 'Curve balls' & process support
- Analysis and Experiments should complement, not segregated.
- Especially useful with the introduction of ASME V&V 40

Case Study – ‘Typical CCS’ Cryogenic Application

Can Seal Integrity be maintained at cryogenic storage for a ‘typical’ plastic 2ml Vial and standard assembly lines?

- Feasibility is hypothesized based on:
  - D.H. Weitzel’s 1962 success of highly compressed o-rings
  - An exploratory hand calculation showing 0.5mm compressed stopper resulting 0.4mm compression at -180ºC.
  - Prior literature nominal success with low statistical confidence

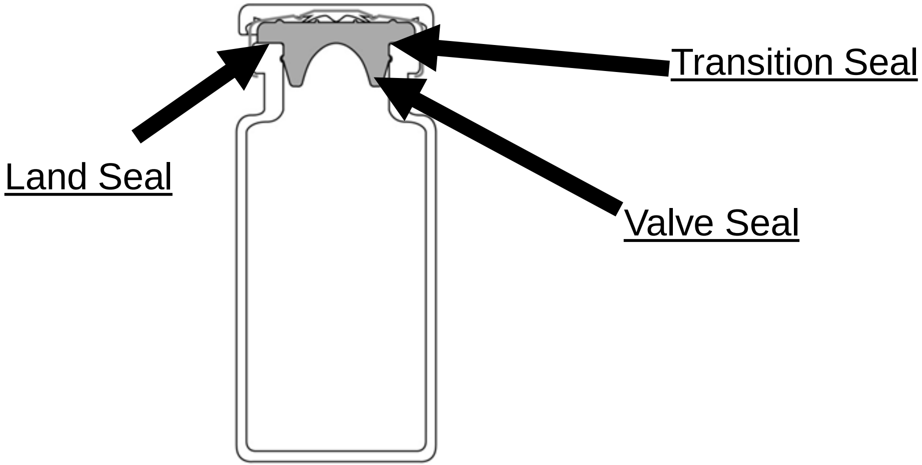


Typical Incremental Computational Approach

1. Identify current design intent
2. Develop a Minimum Viable Computational Model
   – Define Objective
   – Identify/Explore physics-based 1st Principles understanding for functionality
   – Build and execute computational model
   – Verify results
   – Iterate and/or expand conditions
3. Expand computation model for next development phase
4. Maintain model through transfer to manufacturing
   – Digital Twin, IIOT, Design Changes, root cause analyses…

Current Design Intent

- Traditionally, the face seal is considered to be primary seal.



Minimum Viable Computational Model Objective

- Numerous factors through the product life cycle can affect the seal integrity.
- To initially demonstrate feasibility, factors are down-selected to explore success

Component Manufacturing
- Tolerances
- Surface quality
- Material variations
- Washing
- Coatings
- Sterilization
- Post Processing
- Storage
- Local defects
- Etc.

Product Assembly
- Compression force
- Crimp height
- Timing
- Temperature
- Alignment
- Etc.

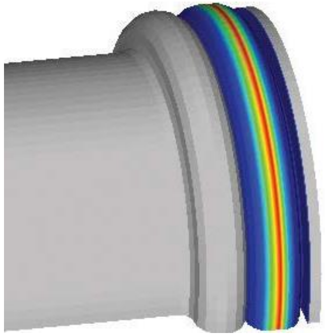
Cryogenic Storage
- Temperature
- Time
- Rate
- Transit
- Environmental
- Etc.

Use
- Environment
- Use cases
- Etc.

Physics of Sealing

- Elastomeric seal = Contact Stresses + Contact Width.
  - Product usage, material stiffness, surface properties, assembly deformations, etc
  - Typically - experimentally derived.
- An analysis of an o-ring with similar hardness used to set targets.
  - Simplified hand calculation ~ RSF value of 27 N (6lbf)
- Sealing stresses between rigid plastics are typically over an order of magnitude higher. Should be developed for temperatures below Tg.

Generic O-Ring



Red > 0.3MPa
>0.3mm

1st Principle Material

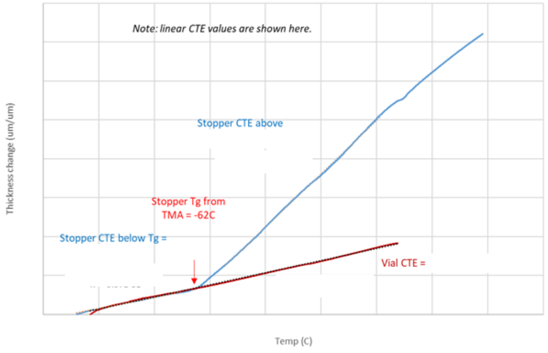
- Elastomeric seal properties are hyperelastic/viscoelastic, and basic material testing strategies are well defined for typical usage.
- Cryogenic storage not recommended by material suppliers for sealing
- Preliminary material testing was performed to develop a basic understanding of:
  - *How do part dimensions change with temperature?*
  - *How does material stiffness change with temperature?*
- This testing is intended as general guidance and is assumed to be the minimum detail necessary for a feasibility model. If feasibility is confirmed, extensive testing would be recommended to explore resin variations, transient properties, failure mechanisms, etc.

1st Principle Material

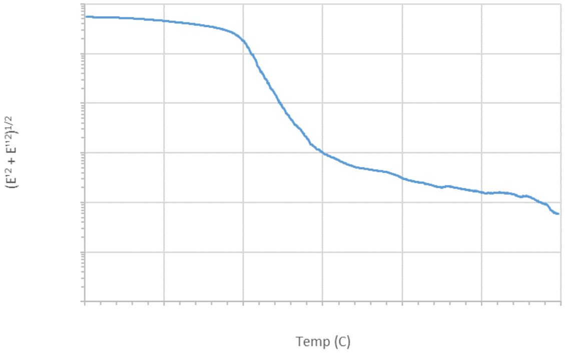
- CTE values were measured using a TMA
- Stopper Modulus vs temperature measured with DMA and uniaxial compression test techniques.
- The Vial and Crimp Modulus were tested at room temperatures

Vial	- COP
Stopper	- Isoprene-based
Crimp	- Aluminum

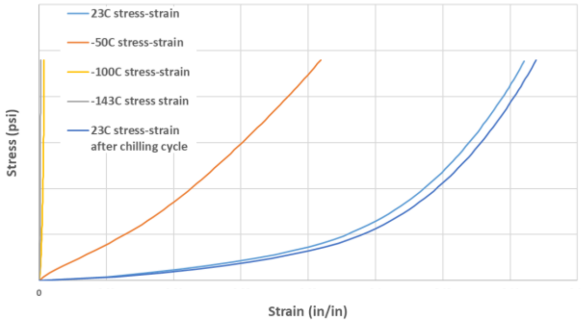
Stopper & Vial CTE



Stopper Modulus

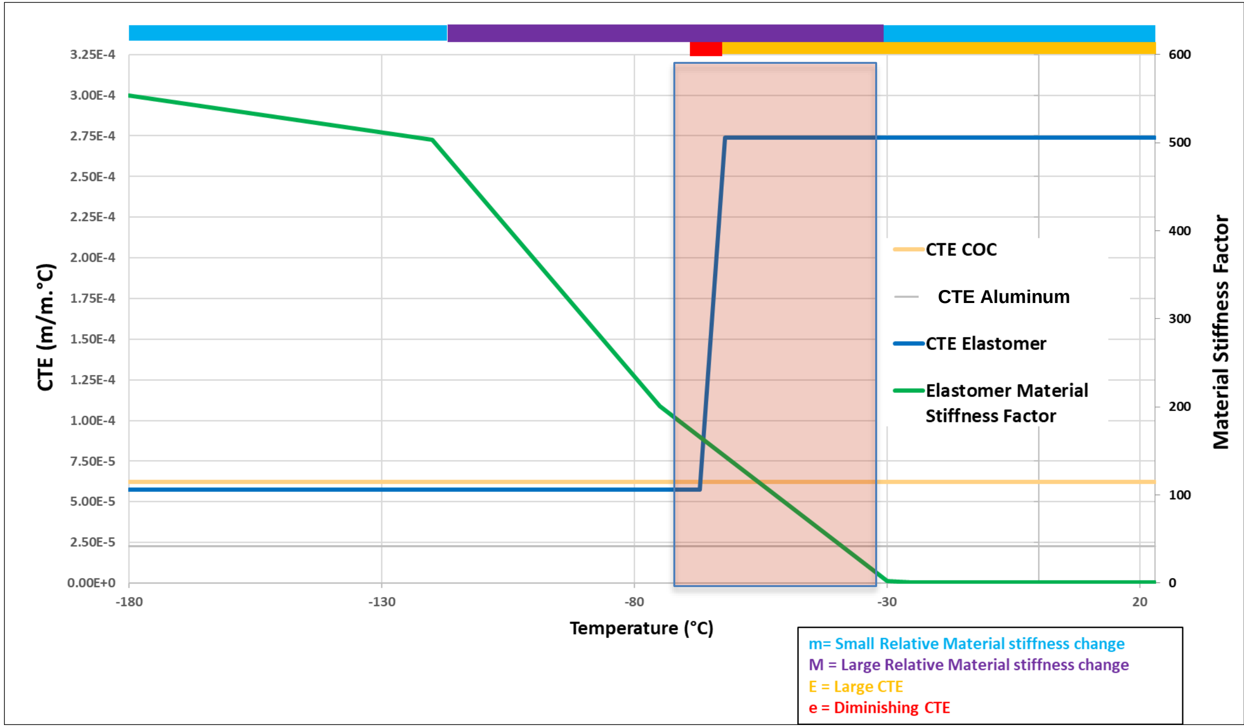


Stopper Compression vs Temp



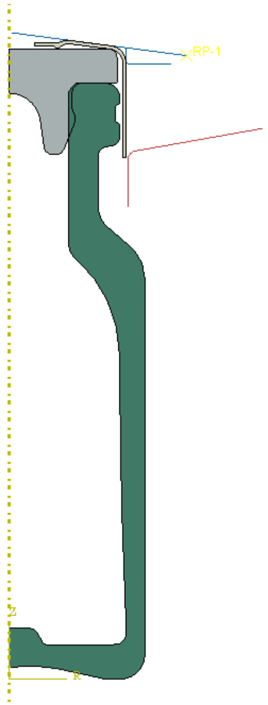
1st Principles Material

- Stopper CTE ~5x higher than other components at >-65ºC. Will continually reduce interference as cooled.
- Stopper stiffness increases below -30ºC. Will increase forces at interference fits
- Transition zone between -30ºC and -65ºC

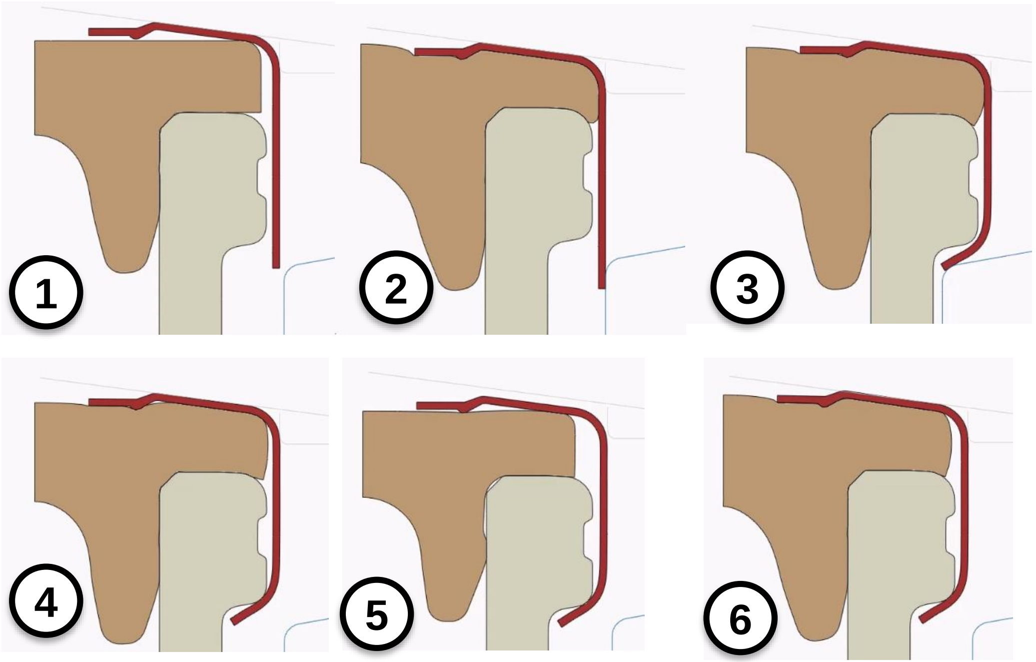


Initial Computational Model

- FEA, 2D axisymmetric model
  - Hyperelastic, temperature dependent
  - Friction = 0.4
  - Time dependent material properties were not included for optimum feasibility
  - Assume rigid press and crimp surfaces



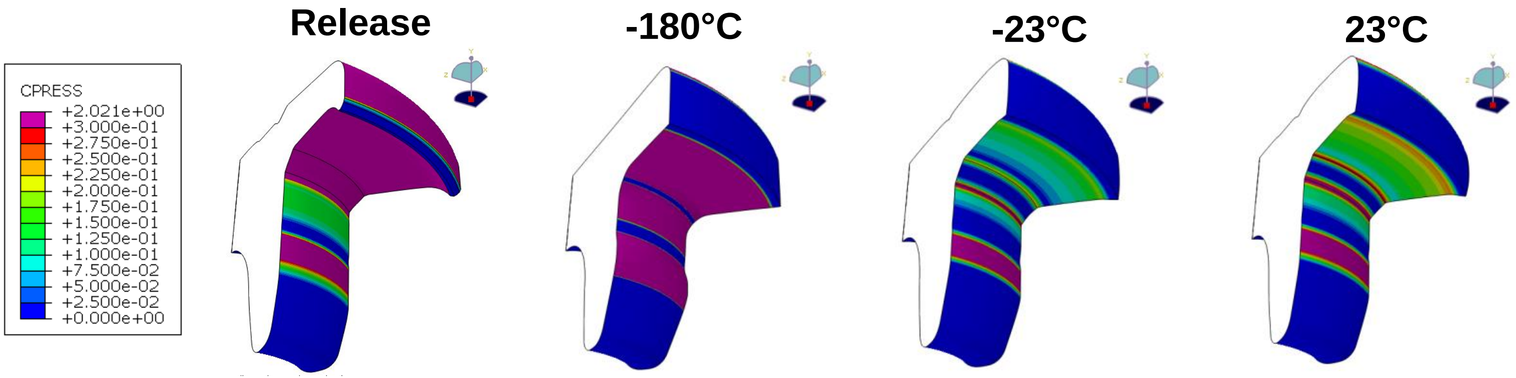
Initial Computational Model



1. Resolve Stopper Interference
2. Load Presser to 140N (32lbf)
3. Crimp
4. Release Crimp and Presser
5. Temperature sweep to -180°C
6. Temperature sweep to +23°C

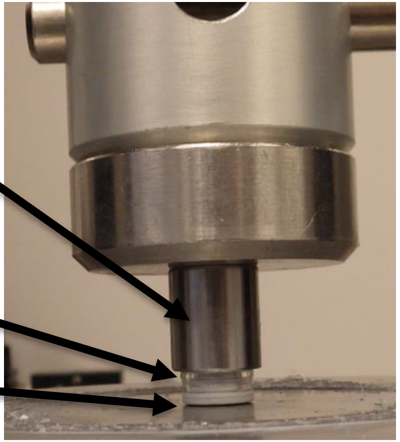
Initial Computational Model

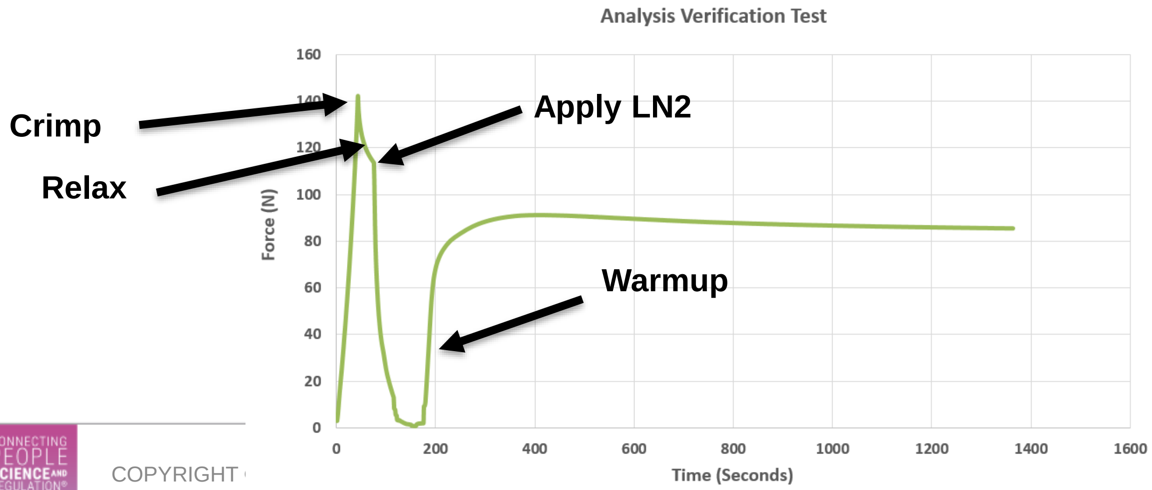
- Pink surfaces = Contact stress > 0.3MPa.
- Primary seal maintains contact but fails to meet target contact stress during the warm up cycle. (Transition Zone)
- Although counterintuitive, the results seems to correlate to prior literature that sealing can be achieved however does not meet the robust requirements.

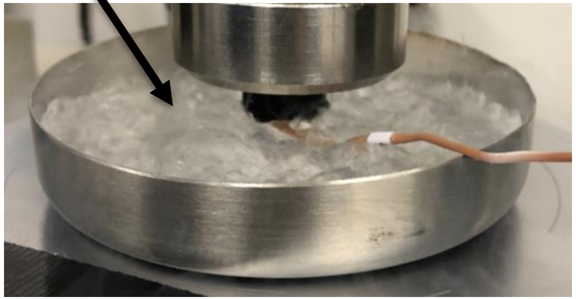


Verify Results

- At -180C, the load is greatly reduced (~140N to 4N)
- Analysis indicates a significantly higher force
- Initial analysis definitions are insufficient to evaluate cryogenic conditions. Must be further developed.



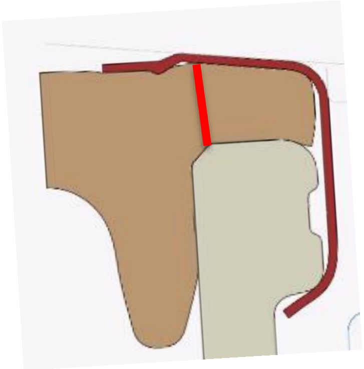




Verify & Interpret Results

- Current analysis verified at room conditions (~2% Error)
- The model utilized to explore the sensitivity of the system.
- The below table summarizes the typical contact pressure at the face seal as **GREEN** if >0.3MPa, **YELLOW** if <0.3MPa, and **RED** if no contact.

	Baseline RSF=140N	Reduced Crimp load, RSF=90N	0.25mm Tighter Crimp	0.25mm Less Crimp	Baseline, LMC
Initial Room Temperature					

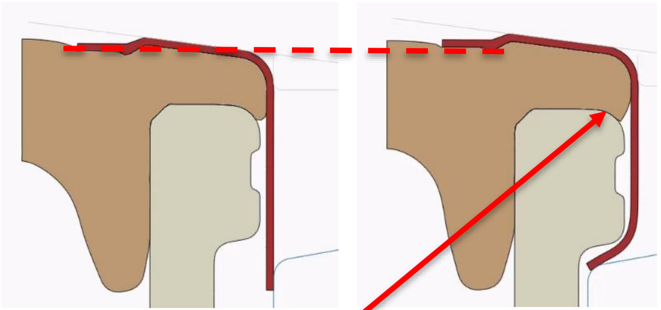


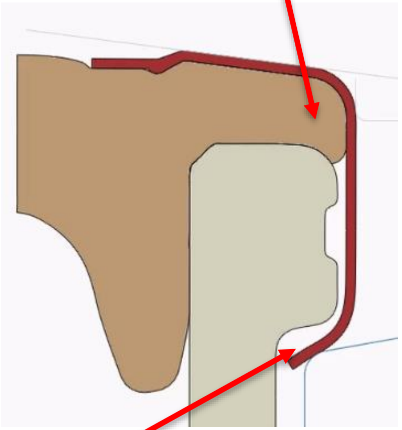
Observations

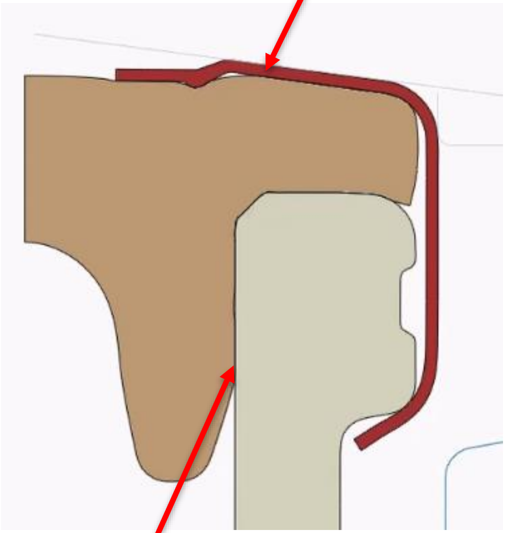
Fixture geometry affects compression

Confined compression

Crimp affects preload







Preload and Crimp Geometry affects overhang

Crimp affects compression

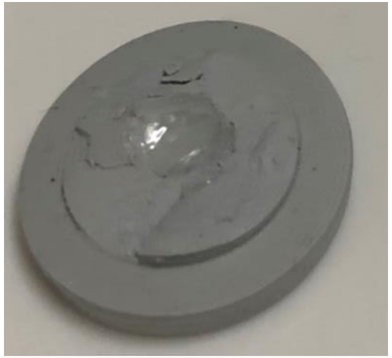
High friction reduces the compression

Material Investigation - Recovery

- Quick ‘binder clip’ experiment at -40^{o}C and 23C to investigate counterintuitive analysis results.
- Current model would predict that the -40C stopper would straighten
- The room temperature stopper quickly recovered its shape,
- The -40C stopper maintained its shape and slowly recovered as it warmed.

Folded Stopper

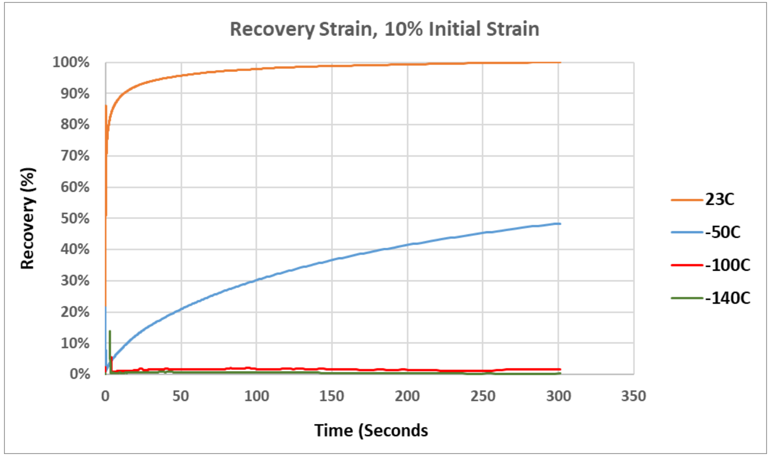
23C

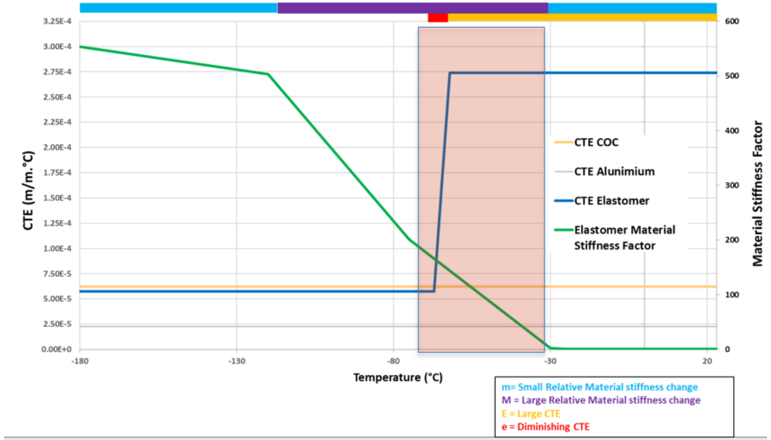


-40C

Material Investigation - Recovery

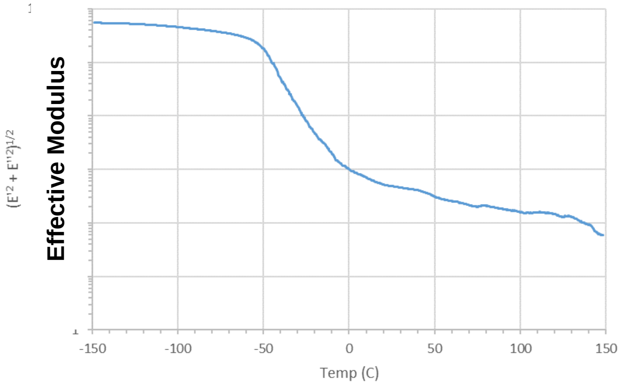
- DMA used to verify the binder clip experiment
- Material's ability to recover is time and temperature dependent.
- The previous material model must be revised to account for 'freezing'
- This phenomenon further complicates the transition region





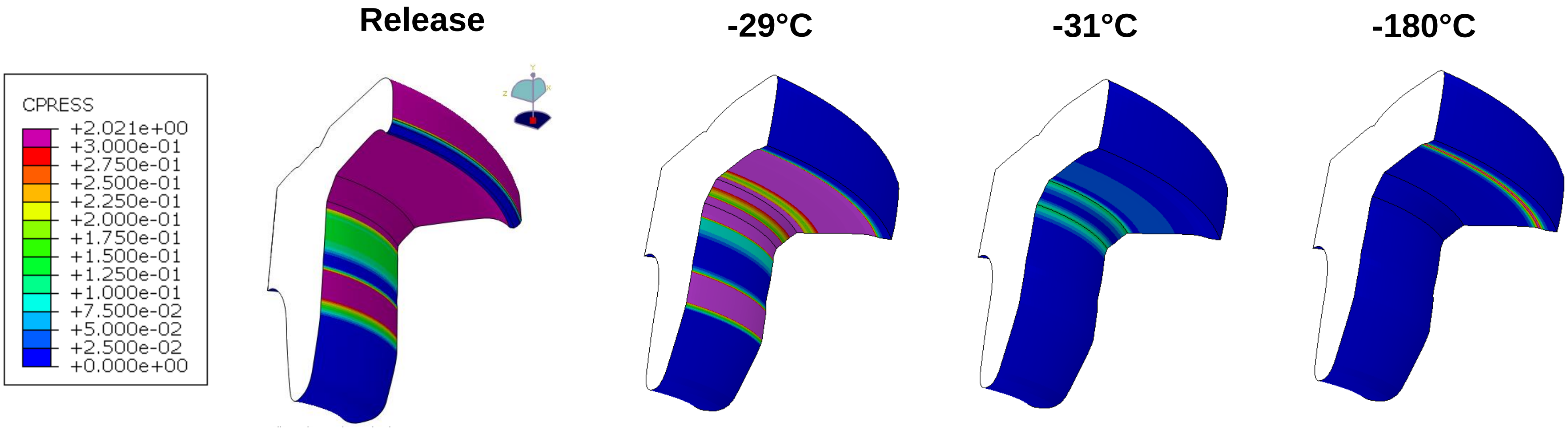
Revised Computational Model

- An abbreviated 'binary' recovery material model rather than fully developing all time dependencies.
- Assume full recovery at temperatures greater than -30C and zero recovery at less than -30C.
- -30C selected because it reflects the temperature where the rate change in effective modulus occurs.
- This method is assumed to be conservative for temperatures lower than -30C.



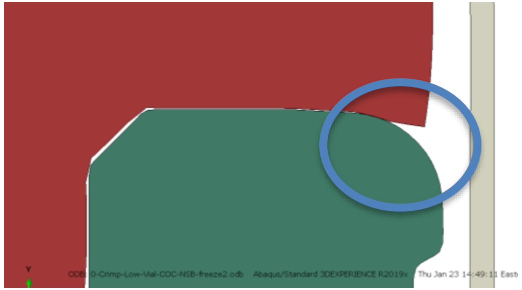
Revised Computational Model

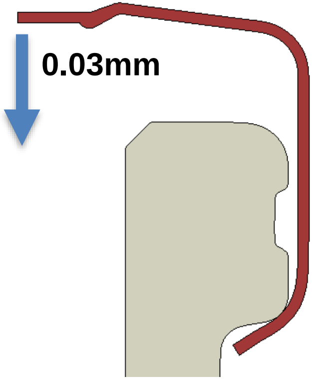
- Primary seal maintained until the ‘freeze point’.
- Contact Maintained, design margin is small
- Contact transitions from the ID to the OD



Discussions

- Below the freeze point, less dependent on initial crimp force and more dependent on:
  - Relative CTE differences
  - Internal stresses of the Crimp and Vial
- Shape and temperature of Stopper ‘freeze’ is critical to sealing
  - If it ‘freezes’ early – the CTE of the Stopper is greater than the Vial and the overhang on the Vial OD creates sealing surface.
  - If it ‘freezes’ later – the CTE differential is less and contact stresses are driven by the ability of internal stresses of the Crimp and Vial to compensate for continual thermal shrinkage.





Case Study Discussions

Why different from CTE hand calculations

– Did not include material effects due to temperature/time

Why different from Weitzel's findings?

– Due to different materials, geometry, or compression

– Gough-Joule effect may influence sealing

Why different from previous test results?

– Conservative target contact stresses

– Rate of cooling may mask instabilities during the transition zone

– May indicate sealing contribution of more exotic properties

  - Polymer entanglement, diffusion of processing material, etc.

Case Study Conclusions

- Primary seal mechanisms transitions from
  - Large Stopper compression, to
  - CTE driven rigid contact
- Understanding the material transition zone and timing is critical
- Low design margins → Higher fidelity model and test fixture recommended
  1. Test method should better compensate fixture shrinkage
  2. Material model should include the temperature, time, and rate dependence for recovery
  3. More complex material behavior should be investigated, (Gough-Joule, CTE vs. initial strains, polymer entanglement, diffusion, etc.)

Incremental Approach Conclusions

Demonstrated an approach to building a minimum viable computation model which can:

- – Develop a physics-based understanding of a system and key elements.
- – Provide a road map for appropriate explorations
- – Predict future challenges
- – Improve overall efficiencies
- – Stimulate novel solutions

Standards and References

1. D. H. Weitzel, R. F. Robbins, P. R. Ludtke, and Y. Ohori, “Elastomeric Seals and Materials at Cryogenic Temperatures,” ASD-TDR 62–31, Part II (1962).

DRAFT

Mr. Chris Folta - Janssen

Stress Engineering Services

- Materials Group
- Analysis Group
- Testing Group