

# Safety Technology Verification for Materials and Corrosions on the U.S. Outer Continental Shelf

## Project Closeout Meeting

May 23, 2017

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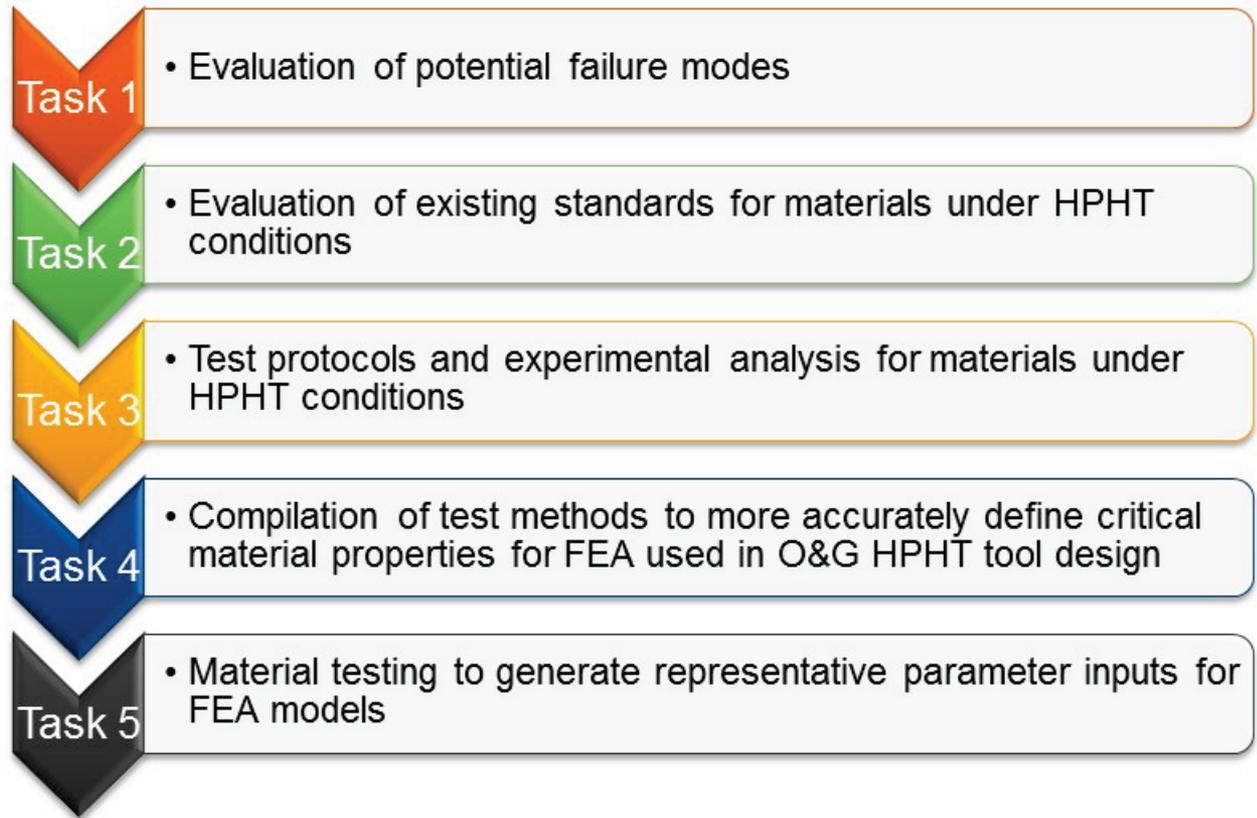
Battelle Memorial Institute  
505 King Avenue  
Columbus, OH 43201

The logo for Battelle, featuring the word "BATTELLE" in a bold, italicized, blue sans-serif font.The logo for PetroMar Technologies Inc, featuring the word "PetroMar" in a stylized font with "Petro" in blue and "Mar" in orange, with "TECHNOLOGIES·INC" in blue below it, and a small blue globe icon to the right.The logo for Endurica, featuring the word "Endurica" in a blue sans-serif font with a red swoosh underneath, and the tagline "Get Durability Right" in a smaller blue font below it.

# Project Overview

- Project Objectives:

- Evaluate current test protocols, procedures, and material property analyses which operators use to demonstrate equipment is fit for service.
- Develop a first generation FEA model of an O-Ring seal, validated through HPHT testing.
- Demonstrate feasibility to predict onset of failure of elastomers in HPHT settings.



# Testing & Modeling Approach

## 1. Materials and testing conditions:

- a. Each elastomer was tested in a customized HPHT seal test fixture at a combination of temperatures up to their maximum recommended service temperature.
- b. Tests were conducted at 100°C and 175°C for FKM, FEPM, and FFKM; at 100°C and 150°C for HNBR (160°C max.); and at 100°C for NBR (120°C max.).
- c. The experimental critical tear pressures (E-CTP) for each elastomer were determined for combinations of temperature, pressure, and five clearance gaps: 2 mil, 4 mil, 8 mil, 12 mil and 15 mil.

## 2. The E-CTPs were used as applied pressure inputs for the FEA model where corresponding tresca stress and modeling predicted (M-CTP) values were calculated.

## 3. An experimentally validated FEA-based computer model was developed (executed in ABAQUS™ and MATLAB®) to predict the onset of tearing and failure of elastomer O-rings under HPHT.

# Background

70 years ago...

- David R. Pearl (Engineer at Hamilton Standard Propellers (HSP), United Aircraft Corp.(UAC)) conducted the first research program to investigate the sealing and wearing characteristics of synthetic-rubber O-ring seals.
- They were the first to systematically test and describe O-ring seal failure for hard and soft O-rings. They observed that when the seal begins to fail it rotates slightly to cover the gap with new material, and the extrusion continues until finally the seal unrolls into the clearance gap.
- They were the first to collect extrusion data and publish an Extrusion Pressure vs. Clearance Guideline Plot which is similar to many seen today.

Pearl, D.R., "O-Ring Seals in the Design of Hydraulic Mechanisms" SAE Quarterly Transactions, Volume 1, No. 4, October, 1947. pp-602-611



*The Familiar Lesson of the Super Highway*

Wright Aeronautical Corporation, Wood-Ridge N.J. SAEJ, Jan. 1947, page 145

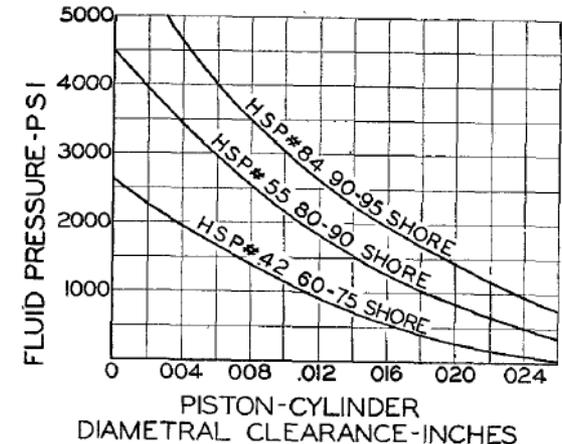


Fig. 10 - Maximum piston-cylinder clearances for O-ring seals of various compositions at various fluid pressures

# Background

- Today: Parker O-Ring Handbook Pressure Limits for O-ring Extrusion are also based on Shore A Hardness
- The maximum temperature for the test data is = 71°C (160°F).
- The highest pressure for the published “Limits for Extrusion” is 10,000 psi, which occurs for 90 Shore Hardness materials at zero clearance gap.
- These values are significantly below the HPHT region of 15,000 psi and 175°C (350°F).

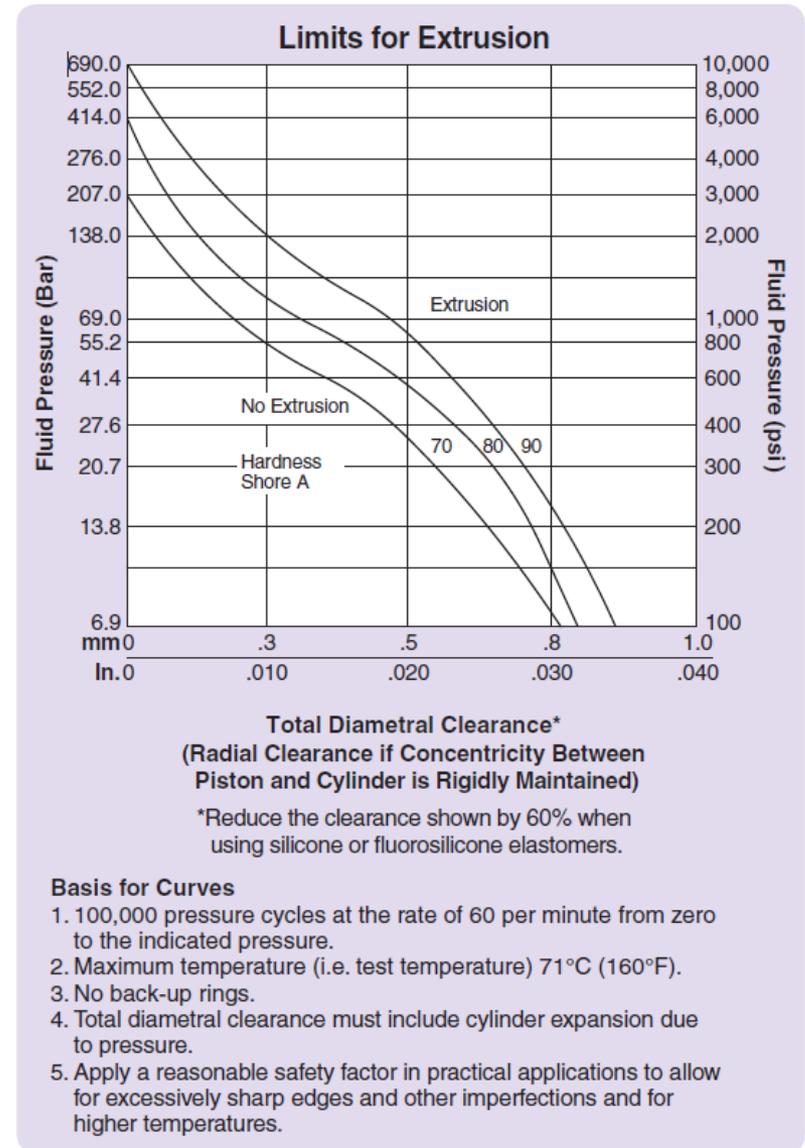
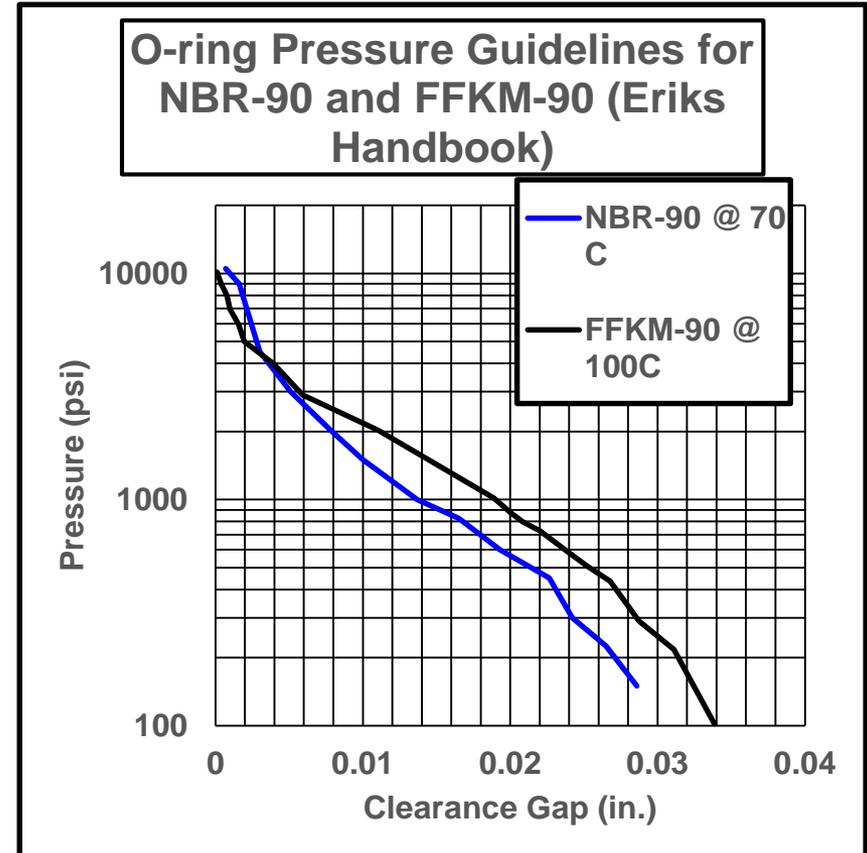
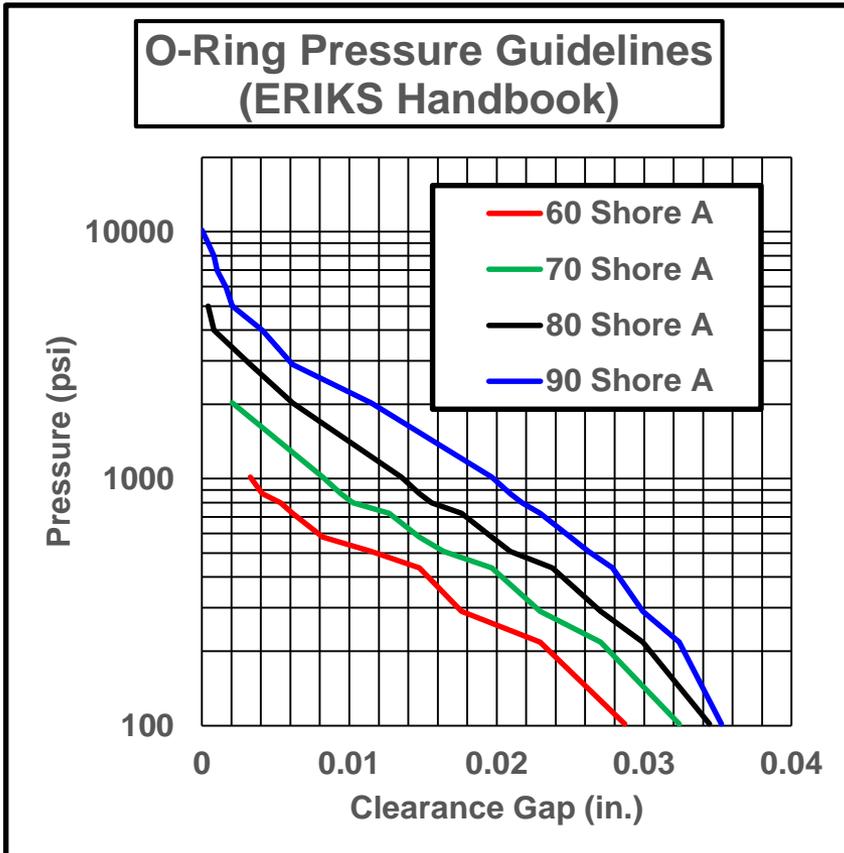


Figure 3-2: Limits for extrusion

# Background

O-ring pressure ratings are based on Shore A hardness and are usually not material or temperature specific.

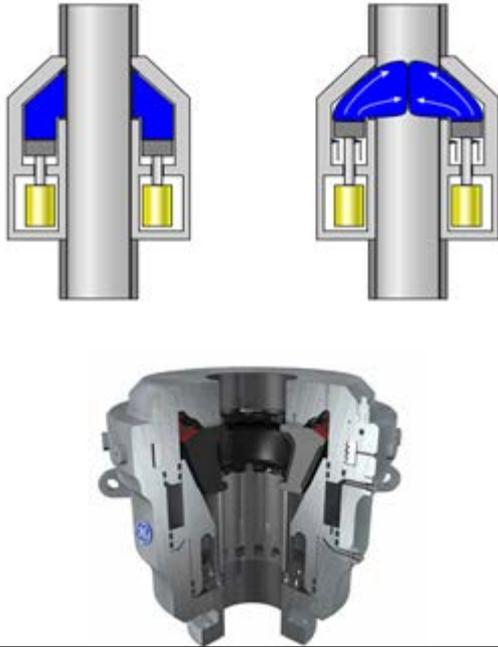


# Executive Summary: Key Findings

1. New or revised guidance, followed by formal industry standards, are needed to ensure safe operation in HPHT environments (over 15,000 psi and 175°C) since current industry standards only provide guidance for elastomer use at pressures up to 5,000 psi.
2. The dominant failure mechanism in HPHT elastomer testing and FEA model development was crack tear propagation via extrusion-initiated spiral failure, which could guide development of new crack resistant materials.
3. A FEA-based computer model was developed to predict the onset of tearing and failure of elastomer O-rings under HPHT. The FEA (M-CTP) model was parameterized with a comprehensive set of elastomer mechanical property data at multiple temperatures, including multi-axial, compression, hyper-elastic, crack-initiation and creep-crack-growth tests. The model was successfully validated by comparing the predicted results with the experimental (E-CTP) from a HPHT test cell.

## Executive Summary: Key Findings – cont.

- The FEA model can be used in the design and evaluation of elastomer seals in downhole tools and well control applications such as Blow-out Preventers (BOP).



Annular Blow-out Preventer Uses Flow Extrusion of Elastomer for Well-Control (7" GK\* BOP for 15,000-20,000 psi shown)

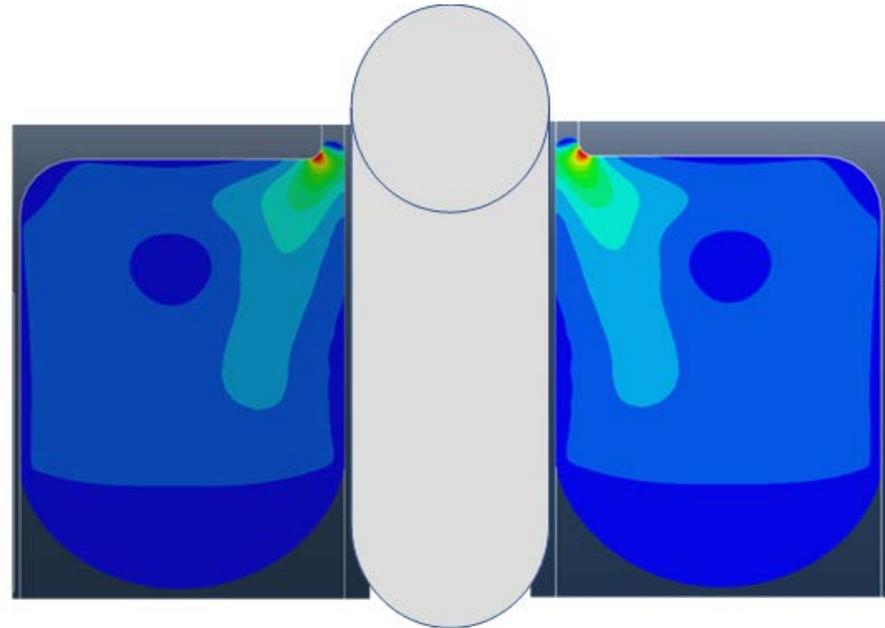


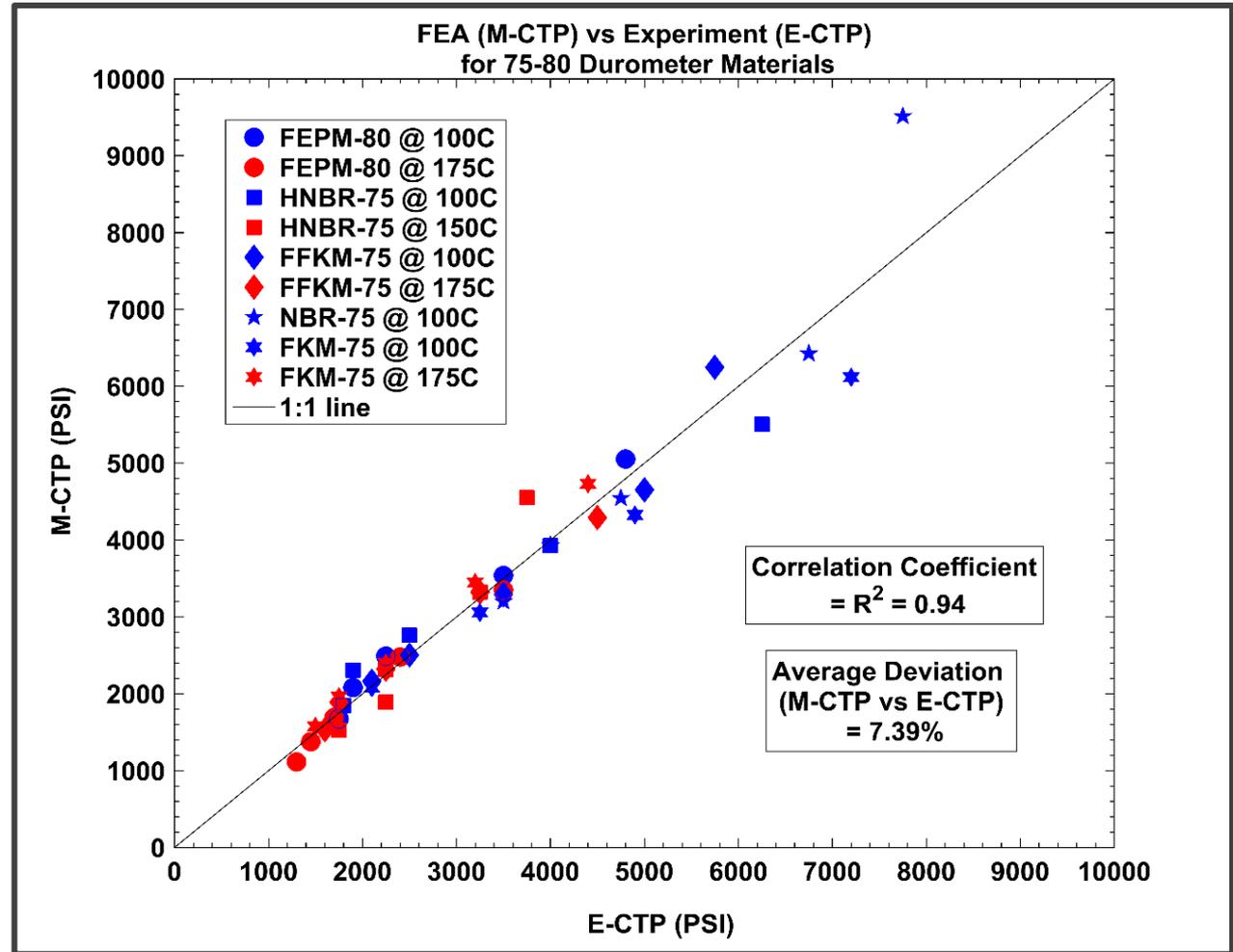
Illustration of Scaled-up FEA analysis of BOP Elastomer Seal using M-CTP model Approach (FKM-90 @ 175°C shown)

Battelle emphasizes that any large-scale extension of the FEA model that was developed and validated on AS568-210 size O-ring seal fixtures should be revalidated on larger scale devices before implementation.

# Executive Summary: Key Findings – cont.

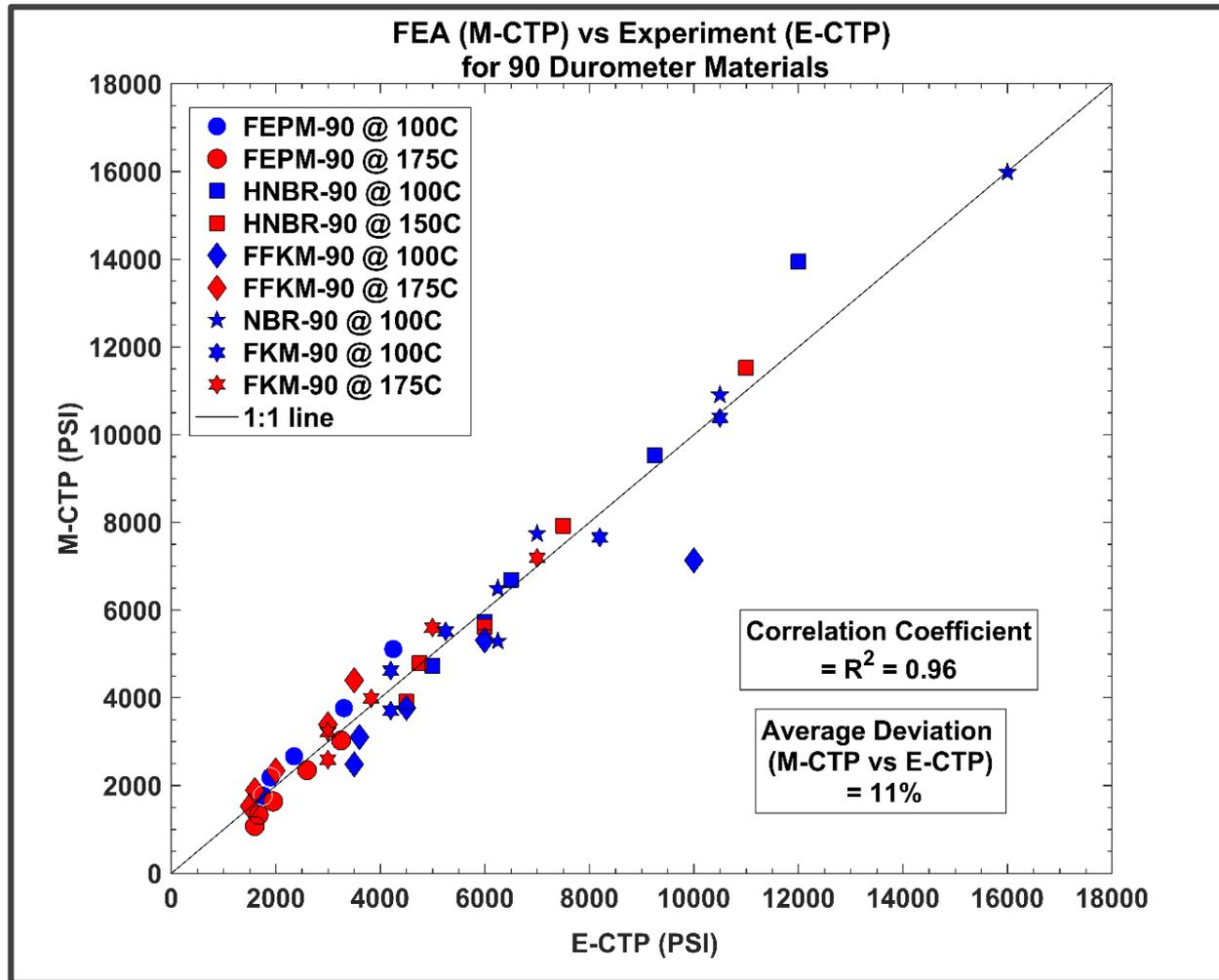
- The high correlation between model and experiment for all O-ring materials, clearance gaps, and temperatures provides users with high confidence in the model accuracy.

The FEA (M-CTP) model was parameterized with elastomer property data at multiple temperatures and confirmed through experimental data.



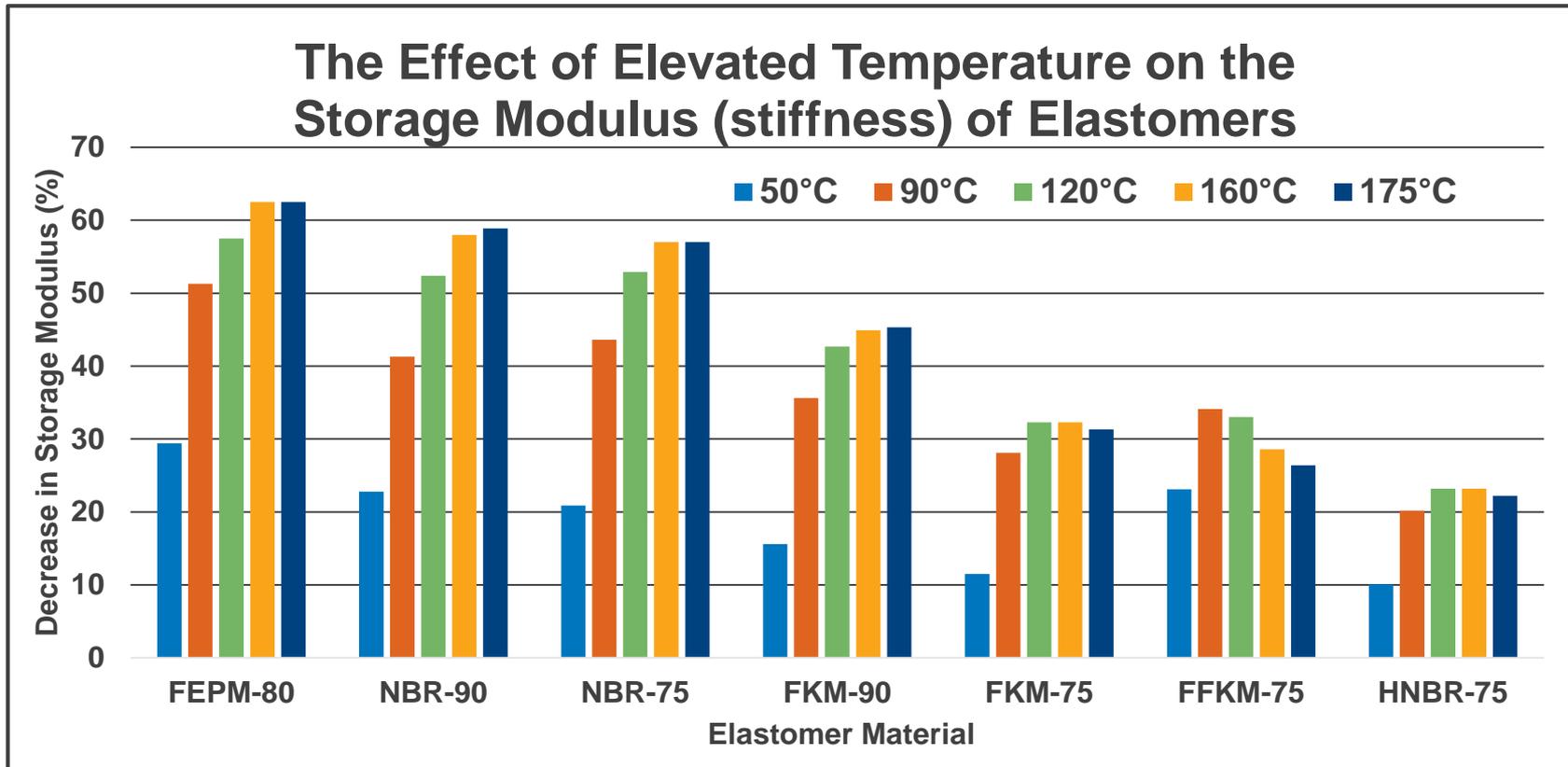
# Executive Summary: Key Findings – cont.

6. High correlation between FEA model (M-CTP) and experimental (E-CTP) was also achieved for harder 90 Shore A durometer materials.



## Executive Summary: Key Findings – cont.

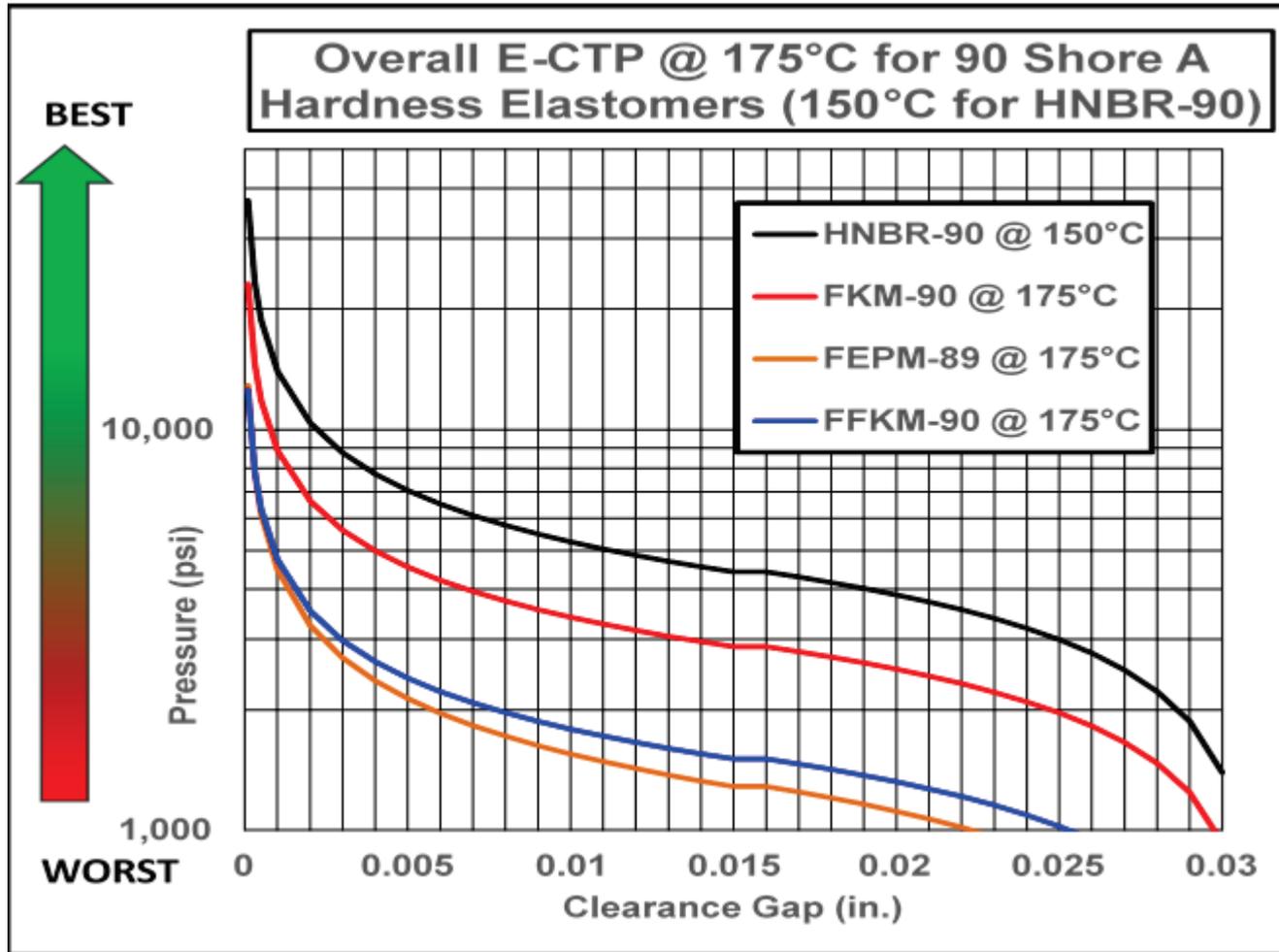
- Most elastomers have much lower mechanical properties (e.g. tensile modulus, tensile strength and elongation at break) at temperatures above 90°C.



Industry should validate the test data on larger scale devices before implementation.

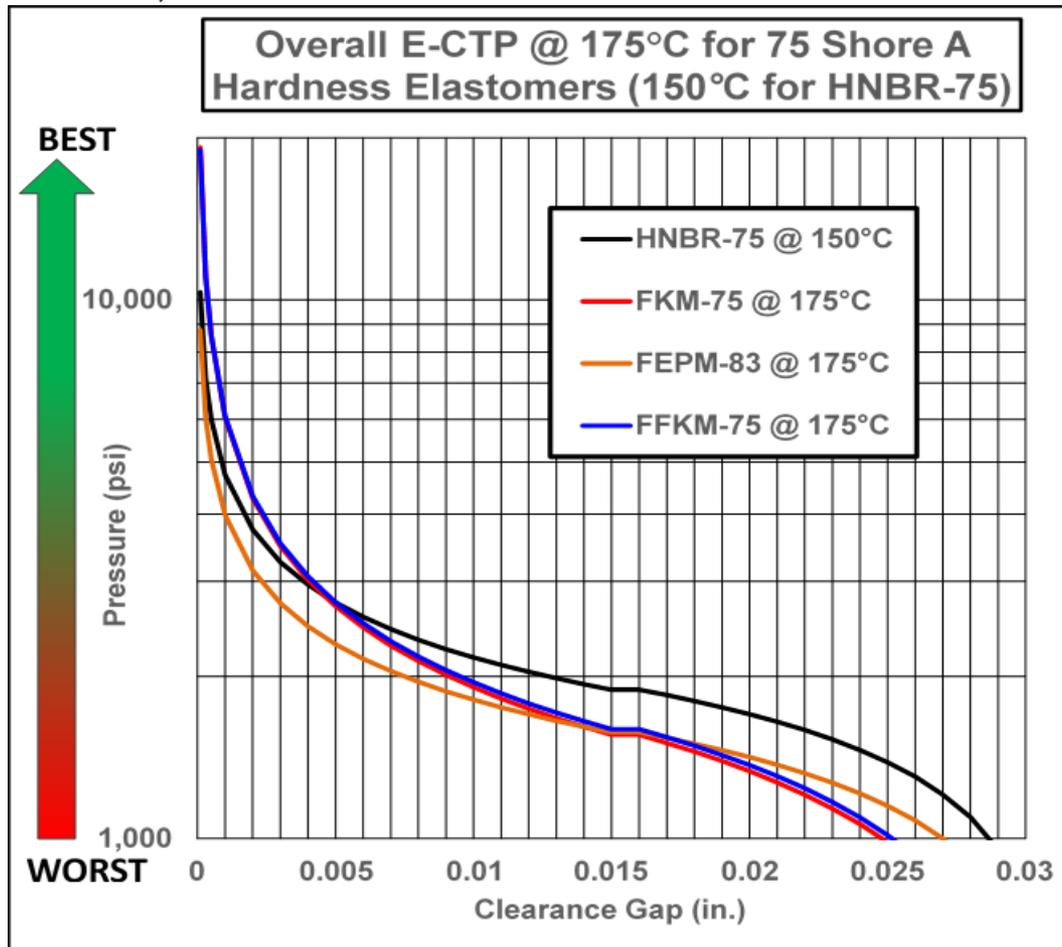
## Executive Summary: Key Findings – cont.

- For 90 Shore A Hardness Elastomers: HNBR-90 and FKM-90 are superior to FFKM-90 and FEPM-90 at 150°C to 175°C across all clearance gaps.



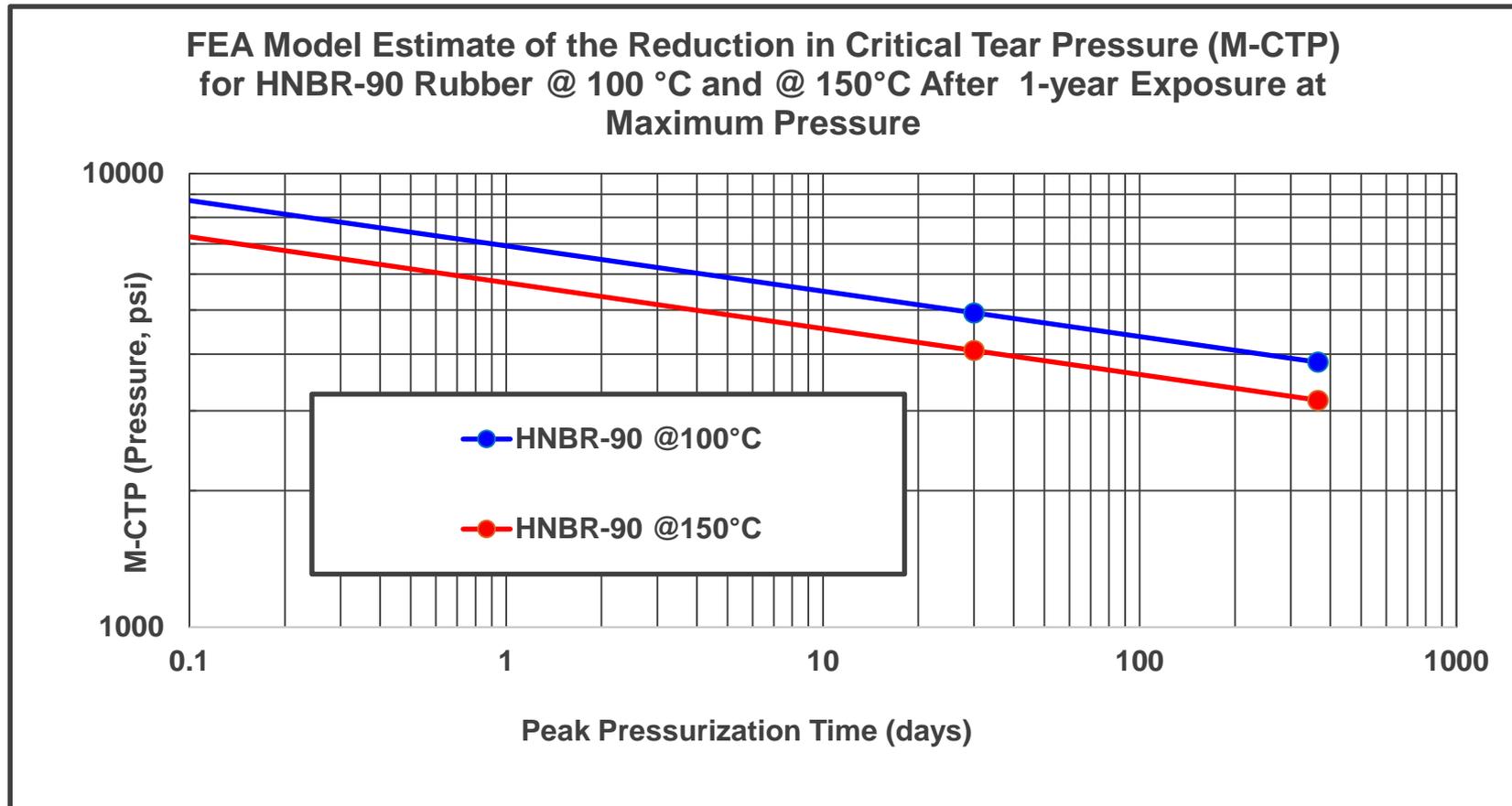
## Executive Summary: Key Findings – cont.

9. Lower Hardness Elastomers (~75-83 Shore A) have similar E-CTP at 150°C to 175°C across all clearance gaps, much lower than HNBR-90 and FKM-90, but similar to FFKM-90, FEPM-90.



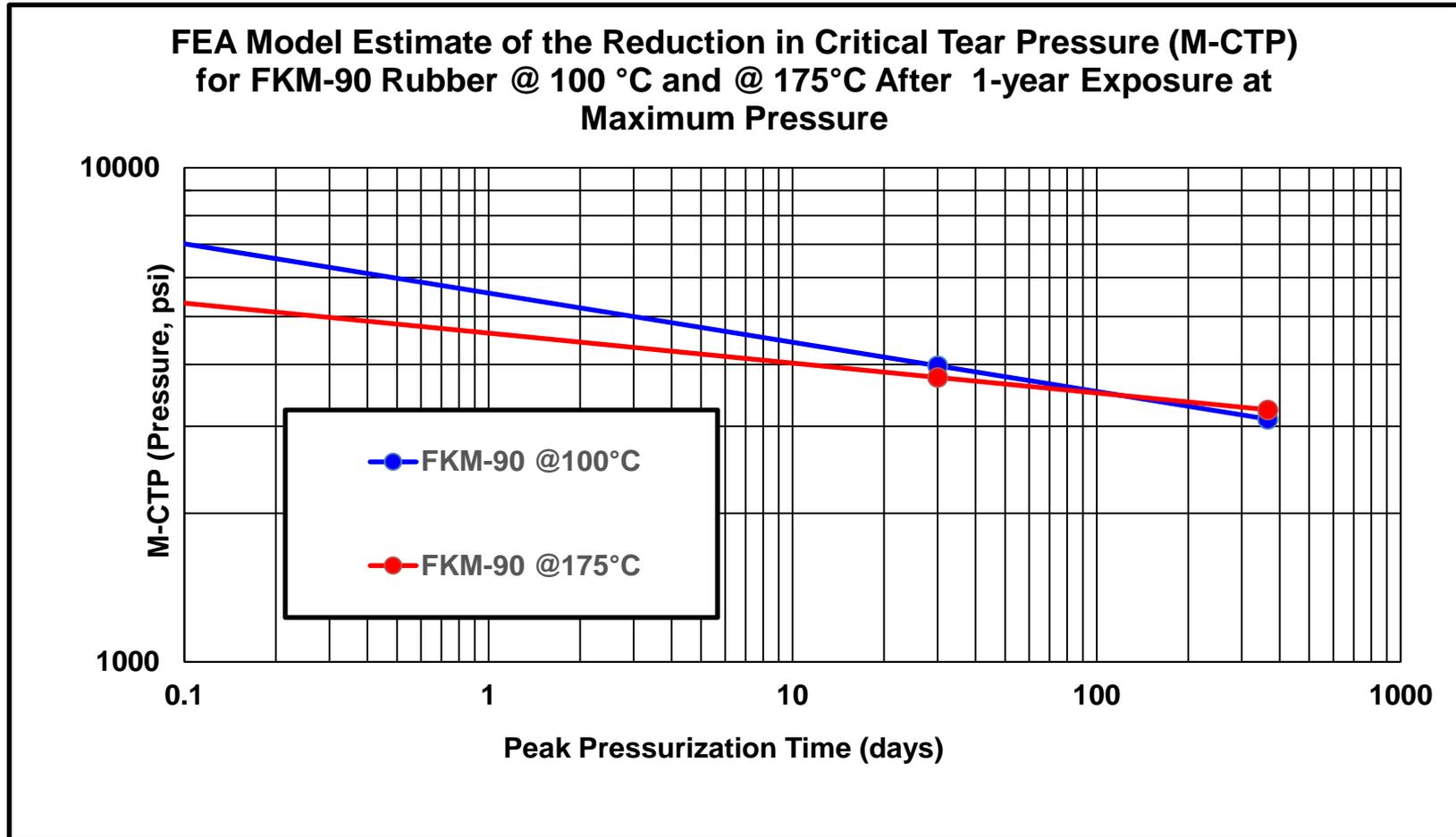
## Executive Summary: Key Findings – cont.

10. Model accurately predicts the M-CTP and Power-law predictions of the change in M-CTP over time indicate approximately 50% reduction after 1 year.



# Executive Summary: Key Findings – cont.

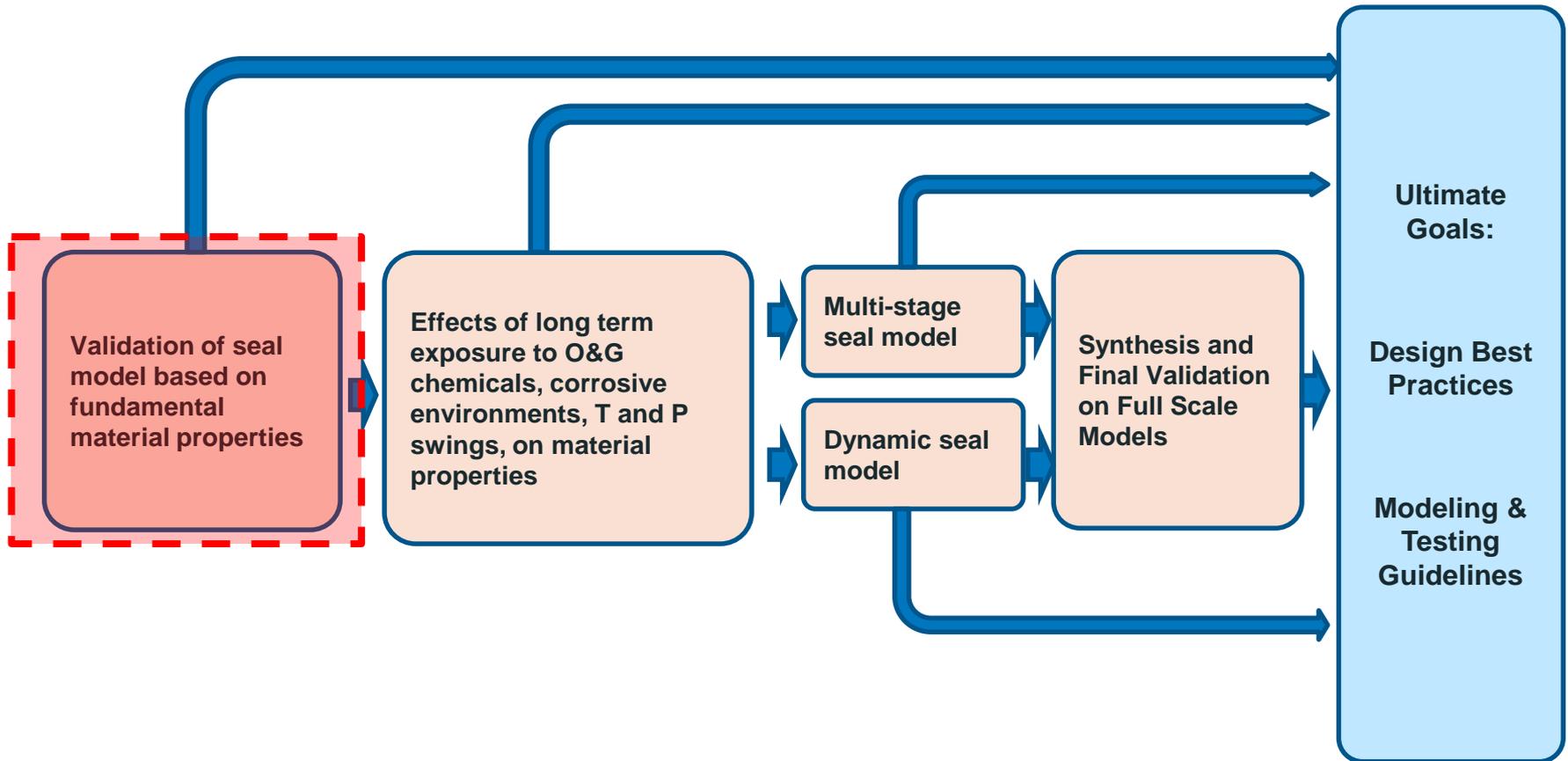
11. FEA model estimate of aging behavior can be improved by validation of creep crack growth at several time points after tearing begins.



# Executive Summary: Recommendations

1. O-ring seal failures can be caused by other stresses, encountered under HPHT conditions, including chemical environments ( $H_2S$ ,  $CO_2$ , hydrocarbon liquid and vapor). Additional testing and model development are recommended for these conditions.
2. The testing in this program was done at the O-ring level and should be expanded to component and device level, i.e. BOPs, SSVs, packers, etc.
3. Future studies can include extending the FEA model and HPHT testing to include aging effects in corrosive and non-corrosive environments as well as extended lifecycle testing (cyclic pressurization and associated crack growth) under these conditions.
4. Future development efforts should expand the FEA model to include longer term (minimum several weeks) creep crack growth of elastomers in combination with experimental validation.

# Executive Summary: Recommendation Roadmap



# Task 1 – Review of Material Properties and Failure Modes.

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- Background research was conducted on each of five (5) elastomers selected for this study.
- Material property information, including temperature, pressure, and exposure limits, was identified.
- End-use O&G tools identified which commonly use elastomers for critical service applications.
- Failure modes of elastomers researched:
  - Similar failure mechanism observed in laboratory testing – spiral failure, and crack tear propagation
- Historical failure data evaluated indicate that components which rely on elastomers to function properly are present in wells which have suffered loss of well control.
  - A mechanism to predict performance of elastomers is required to safely design components.

## The five most used O&G industry elastomers were evaluated as part of this study.

Elastomer	Abbreviation	Common Industry Trade Names
Acrylonitrile-butadiene rubber	NBR	KRYNAC <sup>®</sup> , Nipol <sup>®</sup>
Hydrogenated acrylonitrile-butadiene rubber	HNBR	Elasto-Lion <sup>®</sup>
Fluoroelastomers	FKM	Viton <sup>®</sup> , Fluorel <sup>®</sup> , Daiel <sup>®</sup> , Tecnoflon <sup>®</sup>
Perfluorinated elastomers	FFKM	Kalrez <sup>®</sup> , Tecnoflon <sup>®</sup> , Chemraz <sup>®</sup>
Propylene tetrafluoroethylene copolymers	FEPM	Alfas <sup>®</sup> , Viton <sup>®</sup> Extreme <sup>™</sup>

# Temperature limits of elastomers were used to guide testing in Task 3 and 4.

	FKM	FEPM	FFKM	NBR	HNBR
<b>High Temperature Limit</b>	204°C <sup>1</sup> (400°F) (continuous) 250°C <sup>7</sup> (482°F) (intermittent)	230°C <sup>2</sup> (446°F) 260°C <sup>7</sup> (500°F) (in steam)	327°C <sup>3</sup> (621°F) 220°C to 316°C <sup>4</sup> (428°F to 601°F)	100°C <sup>7</sup> (248°F) (continuous) 130°C <sup>7</sup> (266°F) (intermittent)	150°C <sup>7</sup> (302°F) (continuous) 180°C <sup>7</sup> (356°F) (intermittent)
<b>Low Temperature Limit</b>	-30 to -8°C <sup>1</sup> (-22 to 18°F)	-12 <sup>1</sup> to -3°C <sup>6</sup> (10 to 27°F)	-5°C <sup>5</sup> (23°F)	-50 to -5°C <sup>7</sup> (-58 to 23°F)	-30°C <sup>7</sup> (-22°F)
<b>Chemicals Suitable for Sealing with the Material</b>	Hydrocarbon fuels, oil, aliphatic and aromatic chemicals. <sup>1</sup>	Strong acids and bases, steam, light oxygenates (MeOH) and amines. <sup>2</sup>	Fuels, oils, solvents, alcohols, ketones, mineral acids and bases. <sup>3</sup>	Aliphatic oils and fuels, lower alcohols. <sup>7</sup>	Aliphatic oils and fuels, lower alcohols. <sup>7</sup>
<b>Chemicals Incompatible with the Sealing Material Applications</b>	High pH caustic and amines, low molecular weight carbonyls. Some concerns with light oxygenates (MeOH), steam and mineral acids. <sup>1</sup>	Esters and ketones, light oils, gasoline, chlorinated and hydrocarbon solvents. <sup>2</sup>	Some concern with hot water and amines. <sup>4</sup>	Aromatic hydrocarbons, ketones, acids and bases, ketones. <sup>3</sup> UV, weathering, ethers, aldehydes, chlorinated solvents, phosphate esters. <sup>7</sup>	Aromatic hydrocarbons, ethers, ketones, phosphate esters. <sup>7</sup>
<b>Compression Set</b>	12 to 40% <sup>1</sup>	35 <sup>6</sup> to 40% <sup>7</sup>	14 to 29% <sup>4</sup>	2 to 20% <sup>8</sup>	20% <sup>9</sup>

Note that due to the different temperature range of applications for the elastomers, the compression set values are at different times and temperatures; see the references for specific test conditions.

<sup>1</sup> DuPont FKM Selection Guide. Compression Set is a 70 hr. /200°C (392°F) test. Low temperature limit is the temperature of retraction 10% result. High temperature limit is for continued exposure.

<sup>2</sup> FEPM Fluoroelastomers Guide.

<sup>3</sup> Dupont FFKM Parts Chemical Resistance

<sup>4</sup> 3M Dyneon Fluoroelastomers Product Comparison Guide. Compression Set is a 70 hr/200°C (392°F) test.

<sup>5</sup> Dupont FFKM Spectrum 7090 Technical Information

<sup>6</sup> FEPM 100-150 Series Standard Grade, Commercial Polymer Types and Physical Properties. Compression Set is a 70 hr/200°C (392°F) test. Low temperature service is the glass transition point.

<sup>7</sup> James Walker Elastomer Engineering Guide.

<sup>8</sup> Parco Nitrile Selection Guide. Compression set is at 22 hrs. and 100°C (212°F).

<sup>9</sup> Lanxess Therban Technical Information. Compression set is at 70hr and 150°C (302°F).

# Failure Mechanisms

- Presence of chemicals downhole leads to different elastomer selections.
- Failure modes include:
  - Extrusion and Nibbling
  - Compression Set
  - Rapid Gas Decompression
  - Wear
  - Chemical Degradation
  - Spiral Failure
  - Crack Tear Propagation

Condition	More Suitable Elastomers	Less Suitable Elastomers
Temperature above 280°C (536°F)	FFKM triazine cure	FKM, FEPM FFKM bisphenol and peroxide cures
Temperature above 200°C (392°F)	FFKM	FKM, FEPM
Temperature above 150°C (302°F)	FKM, FEPM, FFKM	HNBR
Temperature above 100°C (212°F)	HNBR, FKM, FEPM, FFKM	NBR
High pH Corrosion Inhibitors	FEPM FFKM peroxide cure	FKM, NBR
Acid Treatments	FEPM FKM peroxide cure FFKM triazine and peroxide cures HNBR	NBR FKM bisphenol cure
Sour Gas Conditions	FEPM, FFKM	NBR, HNBR FKM bisphenol cure
Aromatic Solvents	FKM, FEPM, FFKM	NBR, HNBR
Light Carbonyl Solvents (Acetone, MEK, etc.)	FKM terpolymers with higher fluorine content FEPM TFE/E/PMVE terpolymers FFKM	NBR HNBR FKM VDF/HFP copolymers, lower fluorine contents FEPM
Sub-Freezing Temperatures	NBR, HNBR FKM (PMVE co- and terpolymers)	FFKM, FEPM

# Task 2 – Industry Standards Review and Gap Analysis.

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- Standards evaluated include:
  - American National Standards Institute (ANSI)
  - American Petroleum Institute (API)
  - American Society for Testing Materials (ASTM) International
  - International Organization for Standardization (ISO)
  - National Association of Corrosion Engineers (NACE)
  - Aerospace Material Specifications (AMS)
  - United States Military Standard (MIL-SPEC)
  - Norsk Søkkelkonkurranseposisjon (NORSOK)

# Task 2 – Industry Standards Review and Gap Analysis.

- API standards evaluated provided mixed coverage of HPHT service conditions.
- Newer standards (API 11D, 14A) covered HPHT design criteria.
- None specified that laboratory qualification testing be conducted under HPHT conditions.

	HPHT Conditions	API 6A (12 <sup>th</sup> Edition)	API 11D (3 <sup>rd</sup> Edition)	API 14A (12 <sup>th</sup> Edition)	API 14B 6 <sup>th</sup> Edition	API 16A (3 <sup>rd</sup> Edition)	API 17D** (2 <sup>nd</sup> Edition)
Temperature (°F)	>350	Varies	>350	>350	N/A	<350	<140
Pressure (psi)	>15k	1k	>15k	>15k	N/A	<20k	<15k
Chemical Compatibility Issues	Yes	Yes	Yes	Yes	N/A	No	No
Rapid Gas Decompression Issues	Yes	No	Yes	Yes	N/A	No	No

# Task 2 – Industry Standards Review and Gap Analysis.

- Laboratory testing covered by NACE and ASTM standards are typically conducted at pressures <15ksi.

Standard	Test Type	Test Temperature	Test Pressure
NACE TM0296	Elastomer resistance to sour liquids	212, 250, 302, 347°F	1,000 ± 100 psig
NACE TM0187	Elastomer resistance to sour gas	212, 302, 347°F	1,000 ±100 psig
ASTM D471	Elastomer resistance to petroleum-based oils	-103 ± 4°F to 482 ± 4°F	Atmospheric (14.7 psi)
NACE TM0297	Elastomer resistance to elevated temperature and pressure gaseous CO <sub>2</sub>	122-446°F	1,000-5,500 psig
NACE TM0192	Effect of rapid depressurization from elevated pressure in dry CO <sub>2</sub>	77 ± 9°F	750 ± 50 psig
ASTM D575	Compression-deflection of rubber compounds	73.4 ± 3.6°F	Atmospheric (14.7 psi)
ASTM D945	Mechanical and deformation properties	N/A	N/A
ASTM D6147	Force decay in air or liquid	-103 ± 3.6°F to 572 ± 5.4°F	N/A

# Task 2 – Industry Standards Review and Gap Analysis.

- Few standards evaluated adequately described storage conditions for elastomers during long term storage, transit, and installation.

Standard	Storage Conditions	Storage Temperature	Packaging
SAE ARP 5316C	Humidity <75%	<100°F	Individually packaged, protection against ultraviolet light
ISO 27996	Humidity < 65%	41°F-86°F	Individually packaged to prevent damage
ISO 10417	N/A	N/A	Protection against ultraviolet light

# Task 2 – Industry Standards Review and Gap Analysis.

- A gap analysis shows that few standard organizations cover HPHT laboratory testing of elastomers
- Thorough shipping/storage and Field Re-qualification testing is not covered by many standard organizations.

Process	Notes	Evaluated Standard					
		API	ISO	NORSOK	NACE	ASTM	MIL-SPEC
System Design Guidance	System/tool performance criteria	X	X				
Material Selection Guidance	Selection of appropriate elastomers			X			X
Laboratory Material Qual	Lab testing of material properties				X	X	X
HPHT Laboratory Qualification	HPHT laboratory testing of properties						
Chemical Compatibility Qual	Lab chemical compatibility testing			X	X	X	X
Installed System Qualification	Performance testing of system/tool	X	X				
HPHT System Qualification	HPHT testing of system/tool						
Storage/Shipping Guidance	Packaging and storage considerations	X	X				X
Field Requalification	Field evaluation of system components						X

# Task 3 – Material Property Testing

## Endurica

# Task 3 – Material Property Testing

- Material property testing conducted to gather inputs for FEA model.
- Primary material characterization testing conducted at Axel Labs, led by Endurica.
- Confirmatory testing (including membrane inflation testing and Dynamic Mechanical Analysis (DMA) testing) conducted at Battelle to validate testing conducted at Axel Labs.

# Material Characterization for FEA Model

- Critical Tearing Energy ( $T_c$ )
- Quasi-static Cyclic Simple Tension
- Quasi-static Cyclic Planar Tension
- Quasi-static Cyclic Equi-biaxial Tension
- Volumetric Compression
- Thermal Expansion Coefficient (CTE)
- Creep Crack Growth

# Determining Critical Tearing Energy : $T_c$

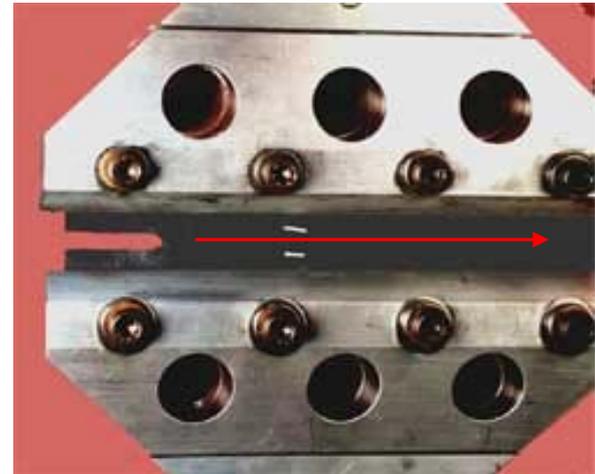
- Purpose

- Quantify the ultimate capacity of the material to resist spontaneous rupture

- Method

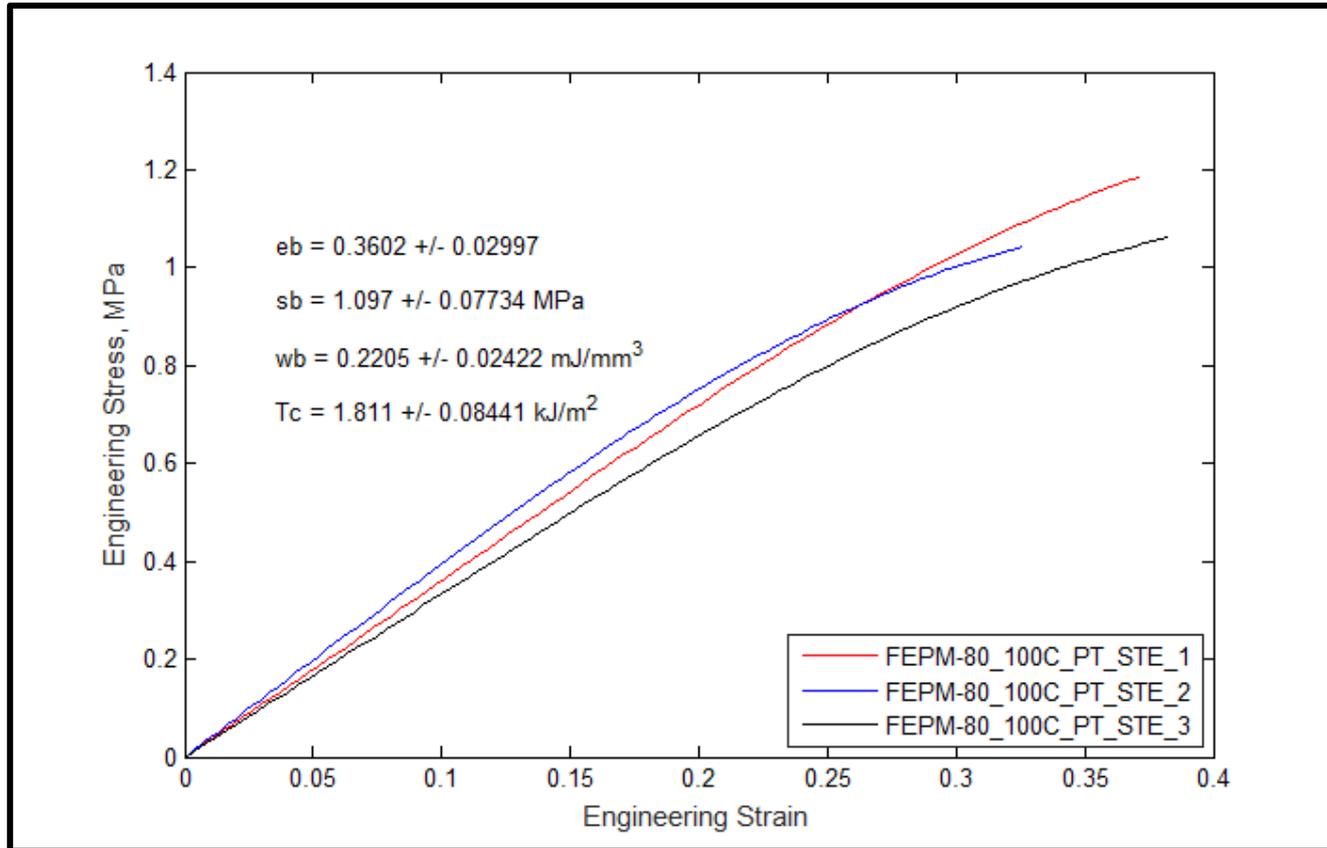
- Edge-cracked planar tension
- 150 mm x 10 mm x 1 mm
- Strain rate = 1 % / sec

$$T_c = - \frac{d(U - V)}{dA} = W_c h$$



Courtesy Axel Products

# Tc Raw Calculation Results: FEPM-80 at 100°C



$$W_c = \int_0^{\varepsilon} \sigma d\varepsilon$$

# Critical Tearing Energy (Tc) Results for Elastomers

Material	Test Temp. (°C)	Engineering Strain at break (1", notched)	Engineering Stress at break, MPa	Strain Energy Density at break, mJ / mm <sup>3</sup>	Tc, Critical Tearing Energy, kJ/m <sup>2</sup>	Relative Change (100-175°C) (% decrease)
FEPM-80	100	0.3602	1.097	0.2205	1.811	
FEPM-80	175	0.2015	0.6455	0.06655	0.5244	71.0
FEPM-90	100	0.3825	1.495	0.3168	2.625	
FEPM-90	175	0.2506	0.814	0.1011	0.7422	71.7
HNBR-75	100	0.2727	1.610	0.2491	2.314	
HNBR-90	100	0.2580	1.988	0.2824	2.665	
HNBR-90	150	0.2204	1.450	0.1719	1.399	47.5 (150°C)
FFKM-75	100	0.1558	0.7264	0.05881	0.4483	
FFKM-75	175	0.07006	0.3742	0.01362	0.09397	79.0
FFKM-90	100	0.2349	1.068	0.1325	1.021	
FFKM-90	175	0.09617	0.5445	0.02714	0.2286	77.6
NBR-75	100	0.3419	1.402	0.2498	2.056	
NBR-90	100	0.1658	2.241	0.1903	1.853	
FKM-75	100	0.1935	0.969	0.09991	0.7701	
FKM-75	175	0.1012	0.6458	0.03421	0.350	54.5
FKM-90	100	0.1225	1.638	0.1112	1.091	
FKM-90	175	0.06701	1.001	0.03551	0.4034	63.0

Note: Most had ~ 75% lower Tc energy @ 175°C vs 100°C, FKM and HNBR were exceptions



# Quasi-static and Cyclic Stress-Strain Behavior

- Purpose

- Obtain parameters for modeling stress-strain behavior in a finite element model
- Validate model parameters across a range of deformation modes
- Ability of the material to resist shape changes

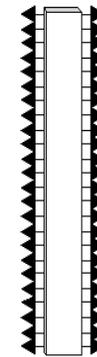
- Method

- Simple tension
- Planar tension
- Biaxial tension
- 1% / sec strain rate

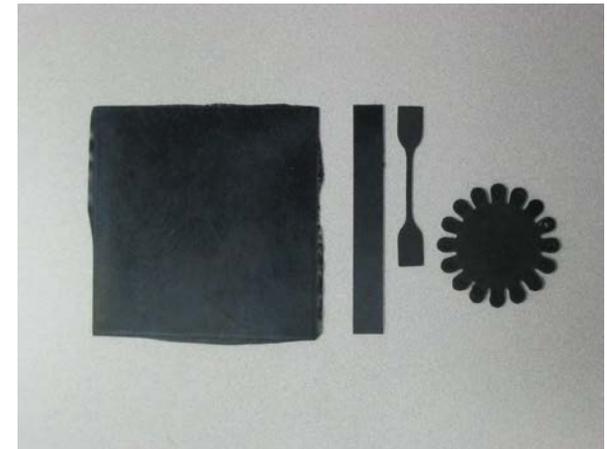
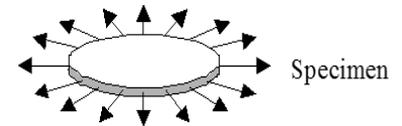
Simple  
Tension



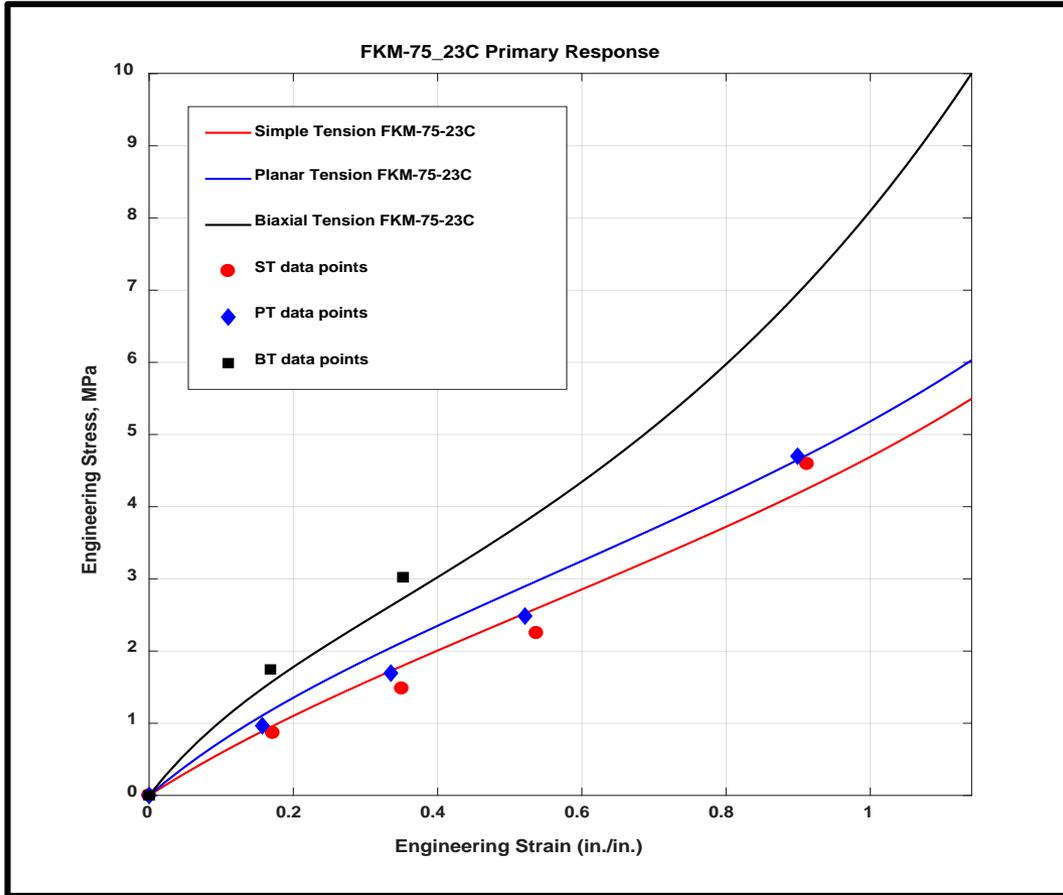
Planar  
Tension



Equibiaxial  
Tension

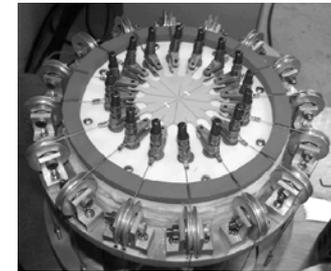
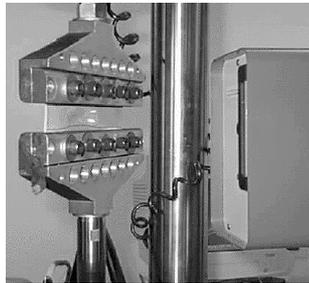
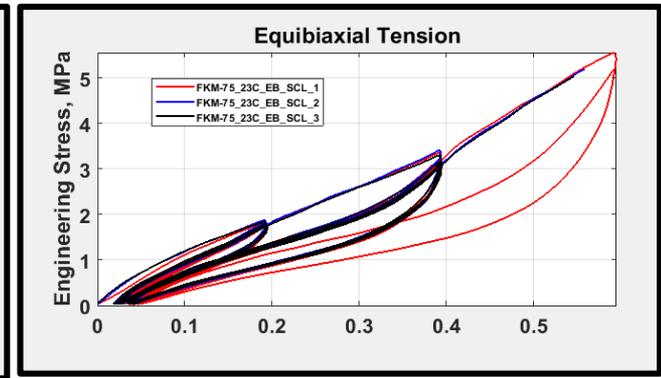
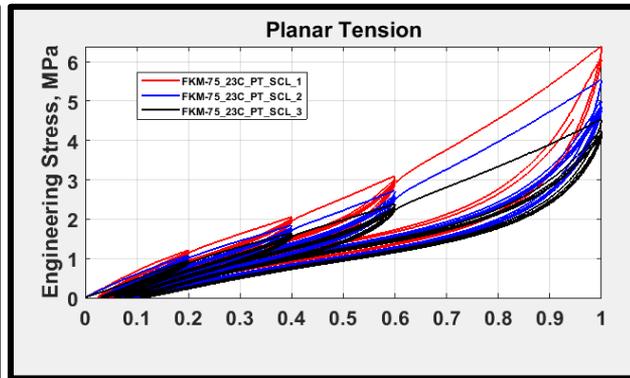
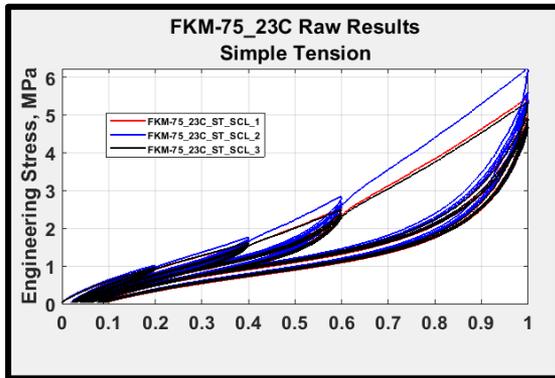


# Example of the three components of Primary Stress-Strain: Shown for FKM-75 at 23°C



$$W = \sum_{i=1}^N \frac{2\mu_i}{\alpha_i^2} (\bar{\lambda}_1^{\alpha_i} + \bar{\lambda}_2^{\alpha_i} + \bar{\lambda}_3^{\alpha_i} - 3)$$

# Cyclic tests at increasing strain levels illustrate the “Mullin’s Effect” (FKM-75 at 23°C)



# Ogden Model Parameters for Elastomers

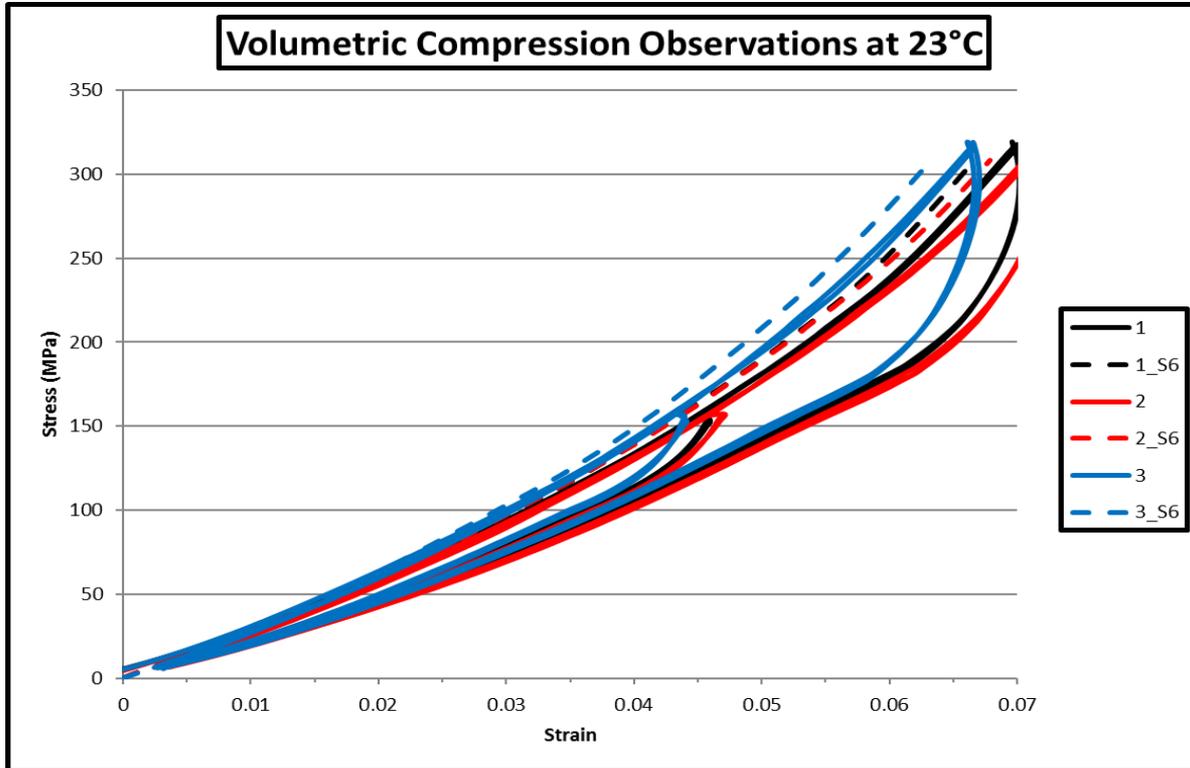
Material	Test Temp, °C	$\mu_1$ , MPa	$\mu_2$ , MPa	$\mu_3$ , MPa	$\alpha_1$	$\alpha_2$	$\alpha_3$
FEPM-80	100	1.354	0.109	0.005495	3.151	-3.151	8.559
FEPM-80	175	1.31	0.1055	0.005317	3.151	-3.151	8.559
FEPM-90	100	1.773	0.1972	0.01231	1.184	-1.184	11.2
FEPM-90	175	1.49	0.1657	0.01034	1.184	-1.184	11.2
HNBR-75	100	2.007	0.1698	0.007936	2.339	-2.339	8.023
HNBR-90	100	2.514	0.3280	0.01320	2.756	-2.756	9.097
HNBR-90	150	2.531	0.3302	0.01329	2.756	-2.756	9.097
FFKM-75	100	1.79	0.06131	0.008854	1.431	-1.431	13.66
FFKM-75	175	2.167	0.07421	0.01072	1.431	-1.431	13.66
FFKM-90	100	1.329	0.6496	0.003826	3.469	-3.469	13.32
FFKM-90	175	1.305	0.6375	0.003755	3.469	-3.469	13.32
NBR-75	100	1.743	0.1984	0.000745	2.648	-2.648	8.738
NBR-90	100	4.724	5.724e-9	0.008121	2.736	-2.736	4.154
FKM-75	23	1.917	0.2228	0.001367	2.661	-2.661	10.79
FKM-75	175	1.724	0.2003	0.001229	2.661	-2.661	10.79
FKM-90	23	3.865	1.055	0.009981	2.38	-2.38	10.31
FKM-90	175	3.41	0.9303	0.008805	2.38	-2.38	10.31

# Volumetric Compression Test

- Purpose
  - Measure the bulk modulus for input to FEA
  - Ability of the material to resist volume changes
- Method
  - Confined compression



# Compression Test Results for FKM-75 at 23°C



$$W = \sum_{i=1}^N \frac{1}{D_i} (J_{el} - 1)^{2i}$$

# Bulk Modulus Test Results for Elastomers

Material	Test Temperature, °C	Bulk Modulus, MPa	D1, 1/MPa	D2, 1/MPa
FEPM-80	100	2363	0.002682	0.001122
FEPM-80	175	3141	0.002922	0.001697
FEPM-90	100	2503	0.002386	0.0007123
FEPM-90	175	3087	0.002836	0.001628
HNBR-75	100	2605	0.002215	0.001082
HNBR-90	100	2525	0.002032	0.0008483
HNBR-90	150	2224	0.002388	0.00101
FFKM-75*	100	1180	0.004156	0.002139
FFKM-75*	175	1251	0.005677	0.003555
FFKM-90*	100	1206	0.004404	0.002078
FFKM-90*	175	1302	0.005466	0.003177
NBR-75	100	2325	0.002391	0.001183
NBR-90	100	2764	0.001978	0.0006899
FKM-75	23	2800	0.002033	0.000392
FKM-75	175	1582	0.003336	0.001915
FKM-90	23	3145	0.001825	0.0002386
FKM-90	175	1702	0.003016	0.001349

\* Note: FFKM perfluoropolymer had lowest Bulk Modulus (MPa)

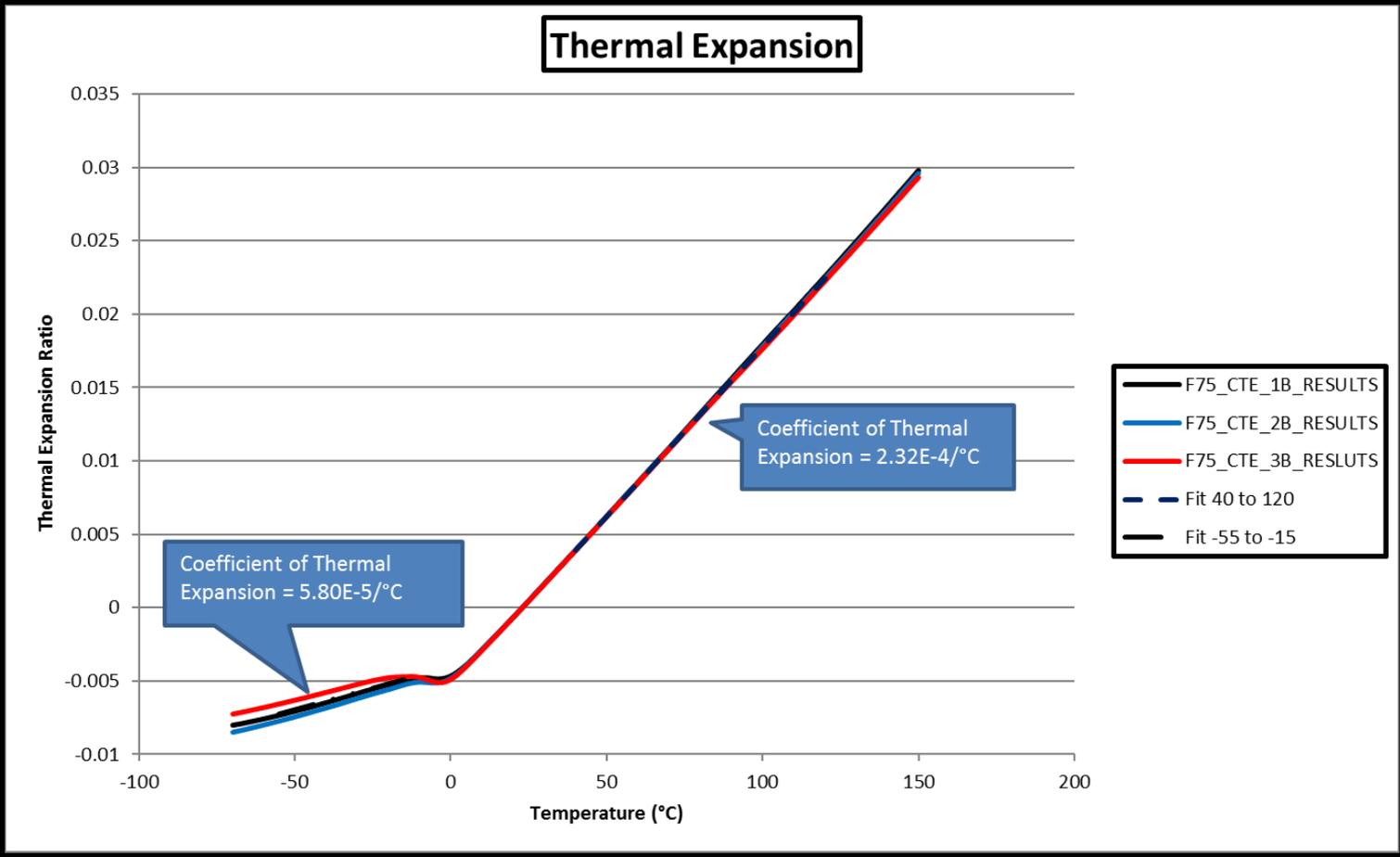
# Thermal Expansion Test

- Purpose
  - Measure thermal expansion (CTE) for input to FEA
  - Tendency of stress-free material to elongate as temperature increases
- Method
  - TMA
  - Temperature Sweep:  $-75^{\circ}\text{C}$  to  $150^{\circ}\text{C}$
  - $0.5^{\circ}\text{C} / \text{min}$



Courtesy Axel Products

# Thermal Expansion Test Results for FKM-75



# Thermal Expansion Coefficients of Elastomers

Material	Coefficient of Thermal Expansion -55°C to -15°C (10 <sup>-6</sup> / °C) (ppm)	Coefficient of Thermal Expansion 40°C to 120°C (10 <sup>-6</sup> / °C) (ppm)
FEPM-80	70	244
FEPM-90	52	203
HNBR-75	78	201
HNBR-90	60	147
FFKM-75*	93	298
FFKM-90*	105	430
NBR-75	52	176
NBR-90	31	89
FKM-75	62	232
FKM-90	54	185

\* Note: FFKM perfluoropolymer had largest CTE (ppm)

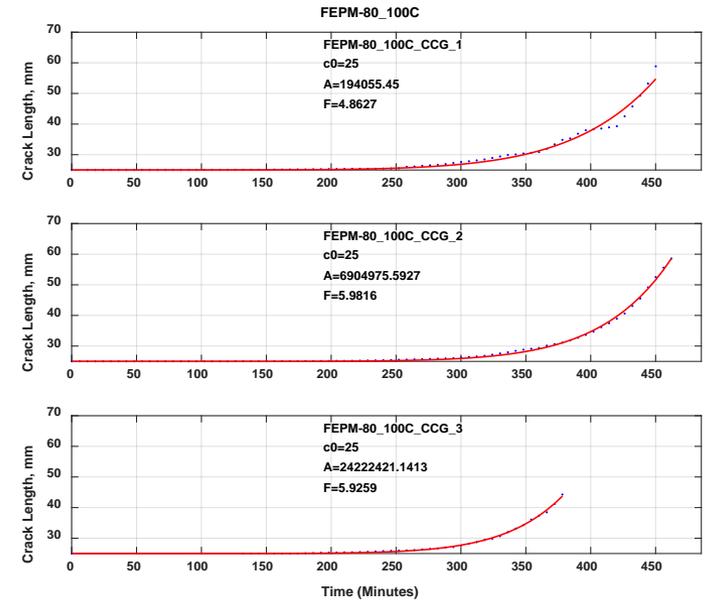
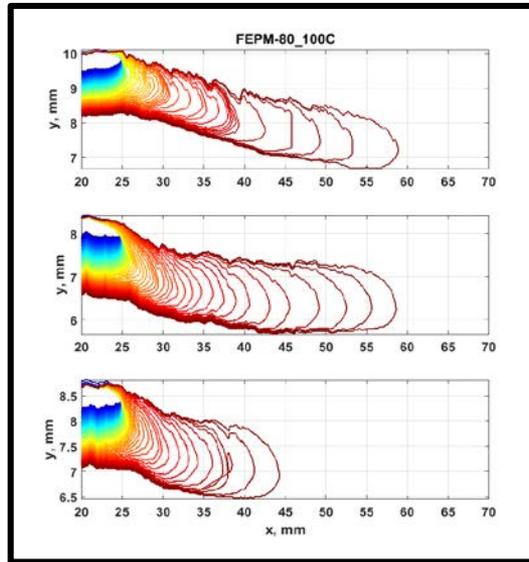
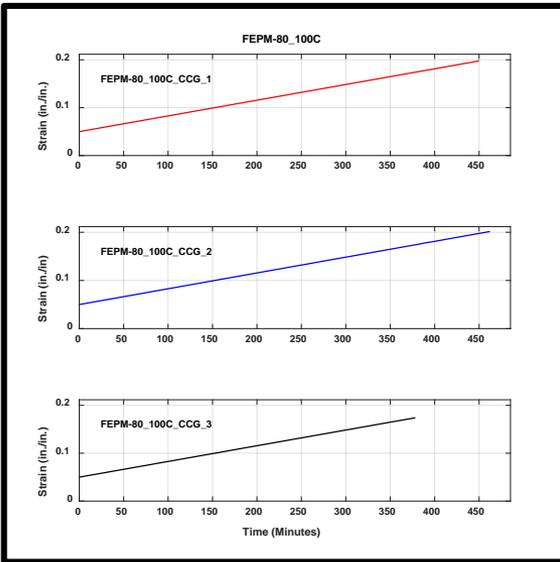
# Determining Creep Crack Growth (CCG) Rate

- Purpose
  - Quantify time-dependence of the crack growth rate
- Method
  - Edge-cracked planar tension
  - Ramped static strain
  - Camera imaging of crack growth

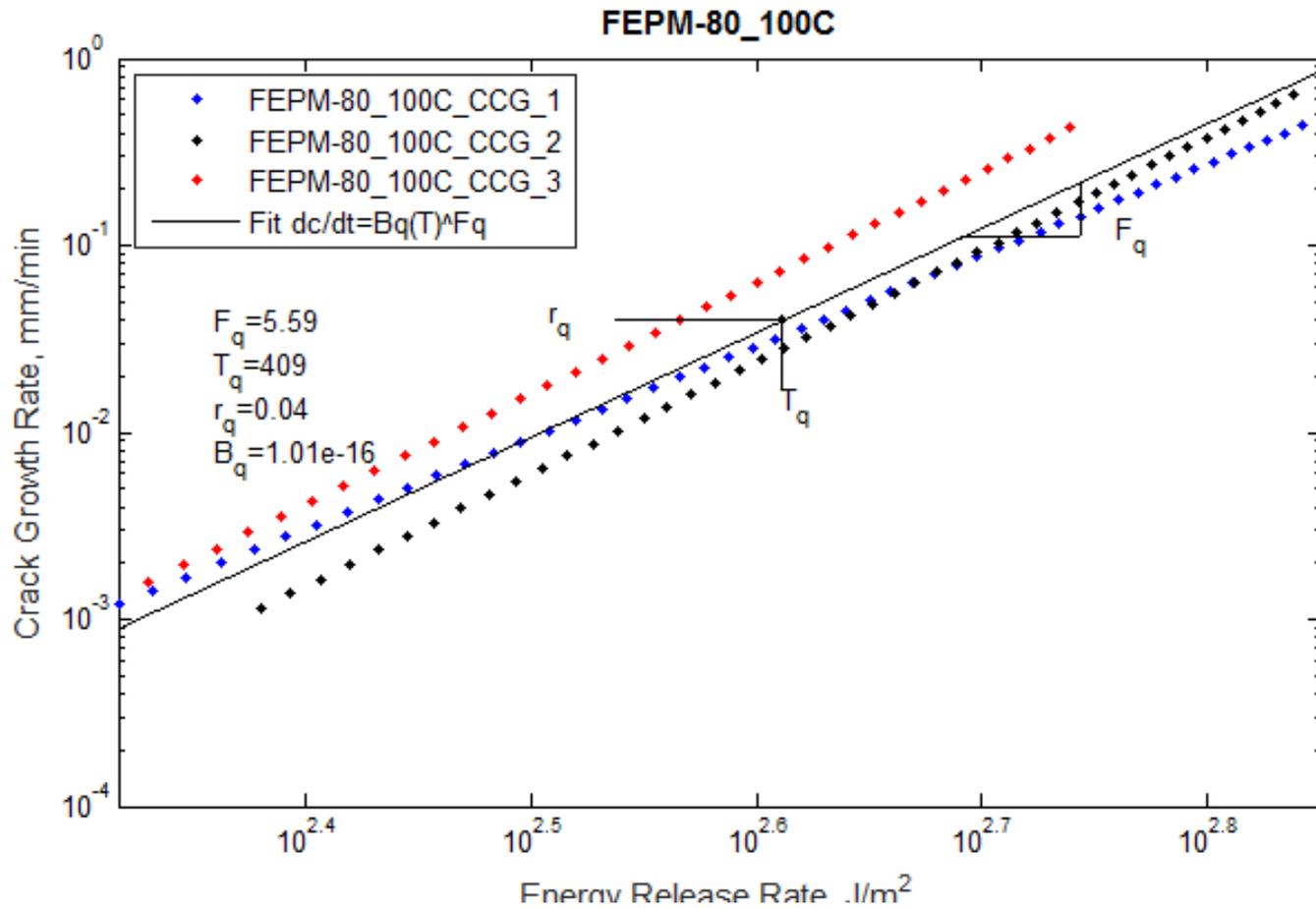


Courtesy Axel Products

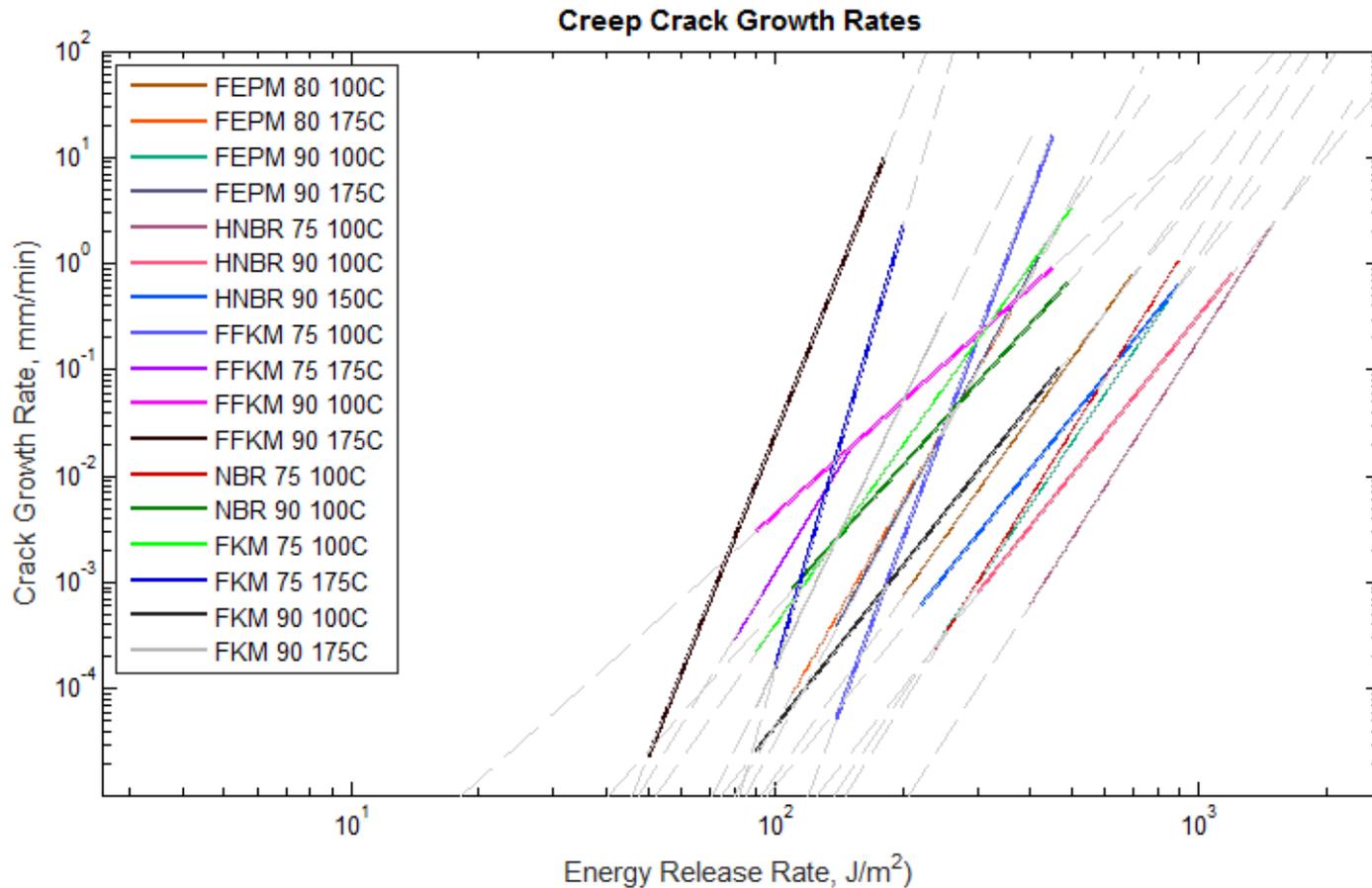
# CCG Test Results for FEPM-80 at 100°C



# Fitting of CCG Data for FEPM-80 at 100°C



# Creep Crack Growth Rate Power Law Results for Elastomers at 100°C and 175°C



# Task 4 – HPHT Testing

## PetroMar

# Task 4 – HPHT Testing

- HPHT testing conducted to validate FEA model developed as part of Task 5.
- Testing conducted by PetroMar at their facility in Exton, PA.
- Each elastomer was tested at a total of 2 temperatures (with exception of NBR).
  - 100°C
  - 175°C (150°C for HNBR)
- AS568-210 O-Rings were used for testing.
- Stepped scan and dwell tests were performed.
- Experimental Critical Tear Pressure (E-CTP) was identified for each material.

- To conduct a series of HPHT tests of O-ring seals sufficient for calibration and validation of the FEA model of elastomers exposed to high pressure/temperature.
- To determine critical pressures at which O-rings' tear starts to occur under the given test conditions, e.g. O-ring material & durometer, temperature profile, extrusion gap, time of exposure to loads, etc.



Material	Compound Reference	Nominal Hardness Shore A	Actual Hardness mean	Service Temperature, [°C]	Test Temp#1, [°C]	Test Temp#2, [°C]
<b>FKM-75</b>	F-13664 (F75)	75	77	-20 to +200	100	175
<b>FKM-90</b>	F-13681 (F90)	90	91	-20 to +200	100	175
<b>NBR-75</b>	B1016	75	76	-30 to +120	100	n/a
<b>NBR-90</b>	B1001	90	94	-30 to +120	100	n/a
<b>HNBR-75</b>	R1006	75	76	-35 to +160	100	150
<b>HNBR-90</b>	R1003	90	92	-35 to +160	100	150
<b>FEPM-80</b>	L1000	80	89	-20 to +230	100	175
<b>FEPM-83</b>	210-A-83	83	83	-20 to +230	100	175
<b>FFKM-75</b>	K4079	75	76	-2 to +316	100	175
<b>FFKM-90</b>	K3018	90	94	-40 to +270	100	175

- 5 compounds X 2 durometers X 2 test temperatures
- 5 clearance gaps
- 4-7 specimens

# Procedure to Determine Critical Pressures

Select

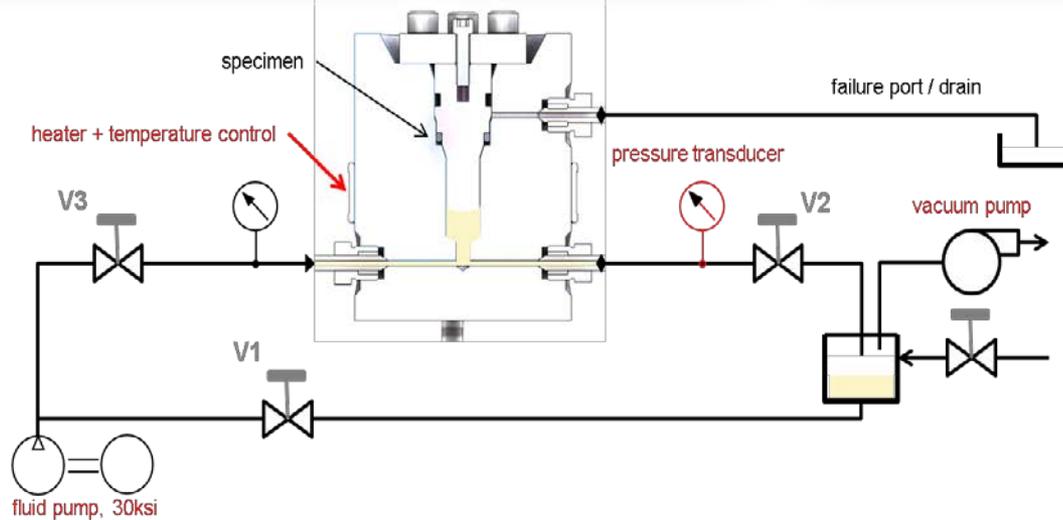
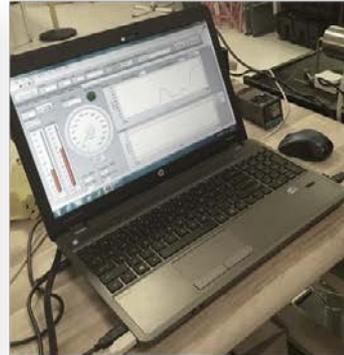
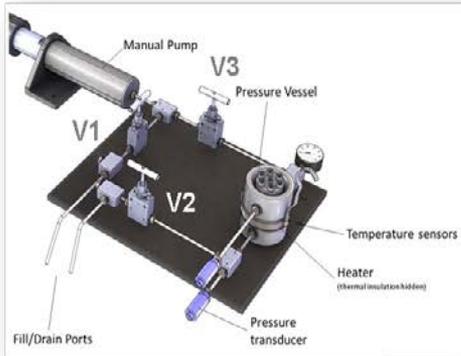
- MATERIAL, M = [ Viton, **Aflas**, Kalrez, Nitrile, HNBR ]
- DUROMETER, D = [ 75A (**80A**), 90A ]
- TEMPERATURE, T = [ **100°**, 175°(150°) ]
- CLEARANCE, C = [ **.002"**, .004", .008", .012", .015" ]

Install SPECIMEN, S = [ 1,2,...,n ]  
Apply Temperature, T  
Apply Pressure, P  
Review results



Aflas80 T=100°C	PistonSize / Clearance				
	#50/.015"	#40/.012"	#30/.008"	#20/.004"	#10/.002"
Stepped-Scan-1	<u>001</u>	<u>002</u>	<u>013</u>	<u>019</u>	<u>025</u>
	a001_Aflas80_T=100°C_P50.csv Pfle=2500 psi	a002_Aflas80_T=100°C_P40.csv Pfle=0 psi	a013_Aflas80_T=100°C_P30.csv Pfle=3500 psi	a019_Aflas80_T=100°C_P20.csv Pfle=6500 psi	a025_Aflas80_T=100°C_P10.csv Pfle=11000 psi
[Stepped-Scan-2]	<u>007</u>	<u>008</u>	<u>014</u>	<u>020</u>	<u>026</u>
	a007_Aflas80_T=100°C_P50.csv Pfle=2500 psi	a008_Aflas80_T=100°C_P40.csv Pfle=3000 psi	a014_Aflas80_T=100°C_P30.csv Pfle=0 psi	a020_Aflas80_T=100°C_P20.csv Pfle=0 psi	a026_Aflas80_T=100°C_P10.csv Pfle=0 psi
Specimen-1 at Pressure=X1*Pfle	<u>003</u>	<u>009</u>	<u>015</u>	<u>021</u>	<u>027</u>
	a003_Aflas80_T=100°C_P50.csv 2000	a009_Aflas80_T=100°C_P40.csv 2200	a015_Aflas80_T=100°C_P30.csv 2500	a021_Aflas80_T=100°C_P20.csv 3500	a027_Aflas80_T=100°C_P10.csv 5750
Specimen-2 at Pressure=X2*Pfle	<u>004</u>	<u>010</u>	<u>016</u>	<u>022</u>	<u>028</u>
	a004_Aflas80_T=100°C_P50.csv 1850	a010_Aflas80_T=100°C_P40.csv 2000	a016_Aflas80_T=100°C_P30.csv 2250	a022_Aflas80_T=100°C_P20.csv 3750	a028_Aflas80_T=100°C_P10.csv 5500
Specimen-3 at Pressure=X3*Pfle	<u>005</u>	<u>011</u>	<u>017</u>	<u>023</u>	<u>029</u>
	a005_Aflas80_T=100°C_P50.csv 1750	a011_Aflas80_T=100°C_P40.csv 1900	a017_Aflas80_T=100°C_P30.csv	a023_Aflas80_T=100°C_P20.csv	a029_Aflas80_T=100°C_P10.csv 5000
[Specimen-4 at Pressure=X4*Pfle]	<u>006</u>	<u>012</u>	<u>018</u>	<u>024</u>	<u>030</u>
	a006_Aflas80_T=100°C_P50.csv	a012_Aflas80_T=100°C_P40.csv	a018_Aflas80_T=100°C_P30.csv	a024_Aflas80_T=100°C_P20.csv	a030_Aflas80_T=100°C_P10.csv 4800

# HPHT O-ring Test Setup Layout



- Piston-type static seal
- Only new -210-size O-rings utilized
- Other than the clearance gap, the gland dimensions are based on the [Parker O-ring Handbook recommendations](#) for static seals
- The initial squeeze is set to be 18%
- No stretching to install O-rings
- All tests are single-cycle tests
- Silicone oil, [Rhodorsil 47V100](#)
- Oil de-airing before loads are applied
- Single-cycle HPHT tests
- For a more accurate estimation of the extrusion gap, the actual pressure and temperature were accounted for (a 0.4mil radial bore increase@25ksi,  $CTE_{17-4}=11\text{ppm}/^{\circ}\text{C}$ )



**NOTES:**

1.) MATERIAL: 17-4 PH, S/S, COND.: H1025, ASTM A564M (UNS S17400), 150,000 PSI MIN YIELD, 12% MIN ELONGATION.

2.) MATERIAL CERTIFICATION REQUIRED WITH PART.

3. INDIVIDUALLY BAG PARTS WITH LABEL INDICATING DRAWING NO., REVISION LEVEL AND PO NUMBER.

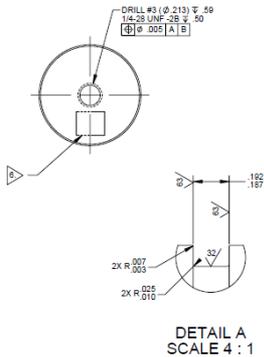
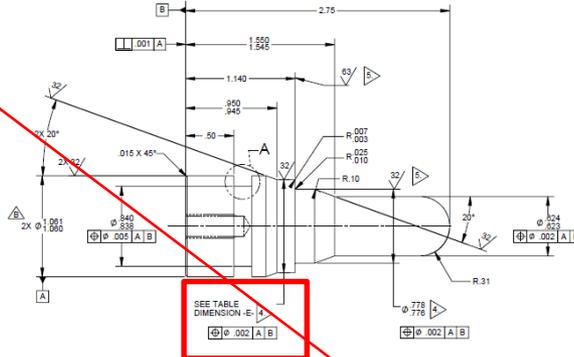
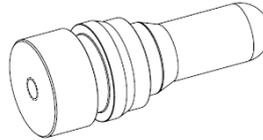
4. ACCURATELY MEASURE AND RECORD FINISHED DIAMETER.

5. ACCURATELY MEASURE AND RECORD SURFACE FINISH ROUGHNESS.

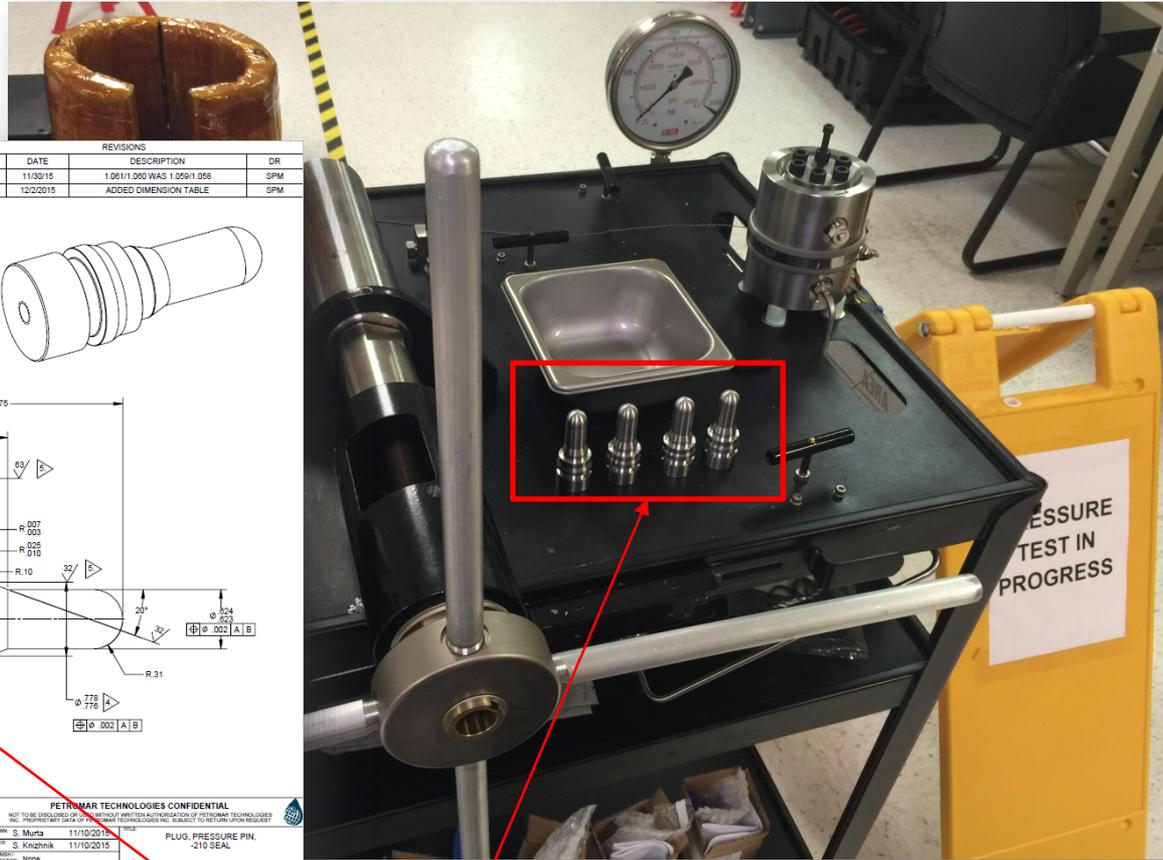
6. CHEMICAL OR LAZER ETCH DASH NUMBER INDICATING SIZE OF DIMENSION -E-, WITH 1/8" HIGH CHARACTERS.

PMT104919	Dimension -E-
-1	.997/.996
-2	.993/.992
-3	.985/.984
-4	.977/.976
-5	.971/.970

REVISIONS				
REV	ZONE	DATE	DESCRIPTION	DR
B		11/03/15	1.06111.060 WAS 1.05911.058	SPM
C		12/2/2015	ADDED DIMENSION TABLE	SPM

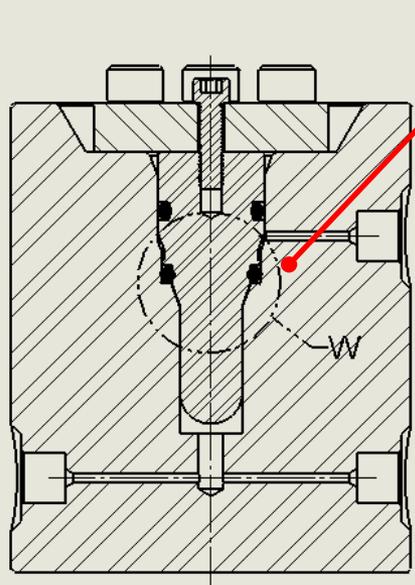


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NOT TO BE DISCLOSED OR USED WITHOUT WRITTEN AUTHORIZATION OF PETROMAR TECHNOLOGIES INC. PROPER PARTS OF PETROMAR TECHNOLOGIES INC. SUBJECT TO RETURN FROM PRODUCT			
TOLERANCES	DRN: S. Murta	11/10/2015	PLUG, PRESSURE PIN, -210 SEAL
DESIGNED BY	S. Kizhnik	11/10/2015	
DATE			
PROJ:			
COATING:	None		
REV			
C			PMT104919
MATERIAL:	See Notes		
SCALE:	1 OF 1		

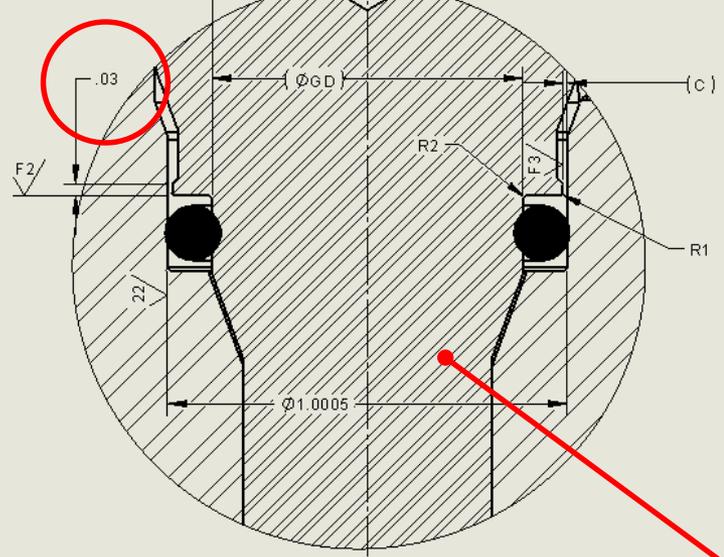


5 different pistons were utilized to create 5 different-size clearance gaps: .002", .004", .008", .012", .015" (nominal sizes)

# Piston Array with Short Extrusion Gaps of .03"



VESSEL	PMT104920	BD	F0
		1.0005	22

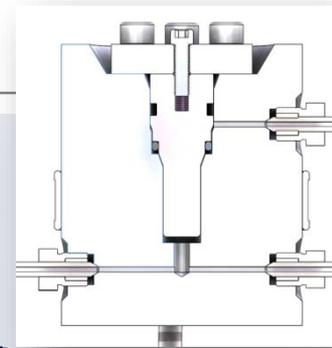


## PMT10530

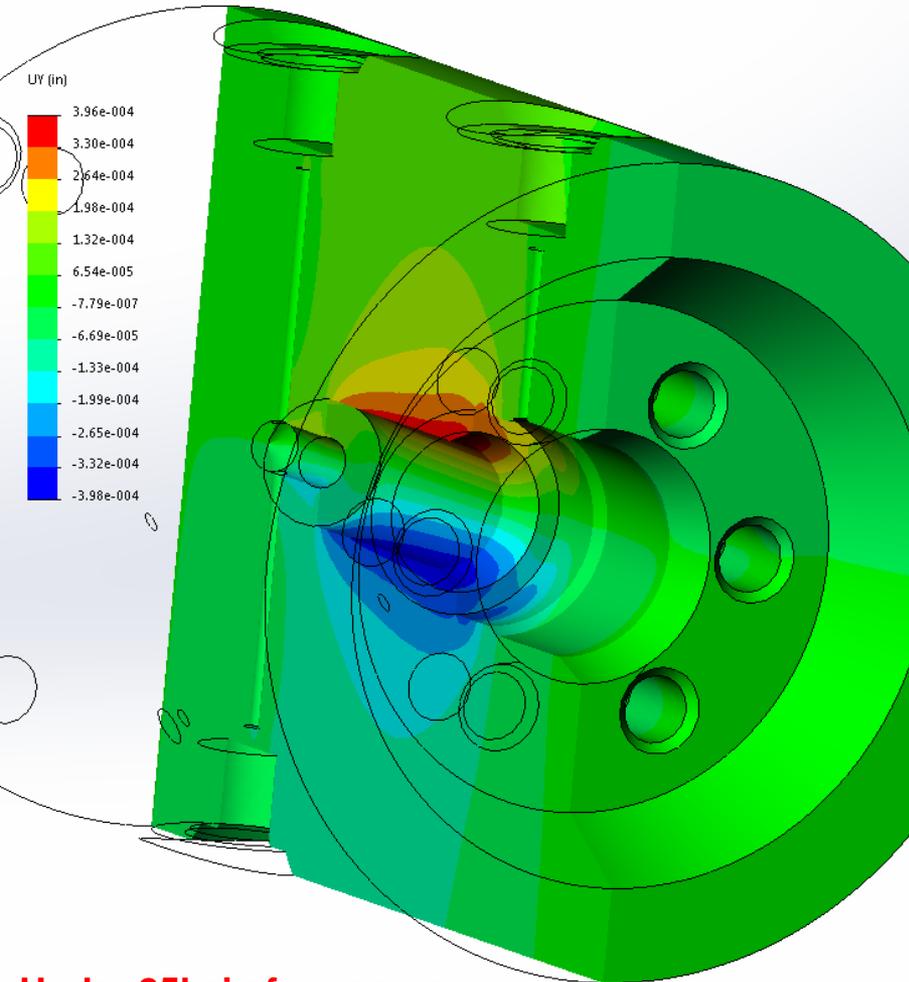
2	C	GD	R1	R2	F1	F2	F3	PD
-10	<b>0.0017</b>	0.7779	0.0050	0.0150	9	9	9	0.9970
-20	<b>0.0037</b>	0.7778	0.0050	0.0197	10	10	32	0.9930
-30	<b>0.0077</b>	0.7778	0.0050	0.0197	12	12	32	0.9850
-40	<b>0.0120</b>	0.7772	0.0060	0.0196	32	63	32	0.9765
-50	<b>0.0148</b>	0.7780	0.0050	0.0197	16	16	16	0.9710



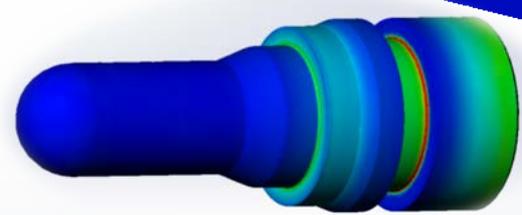
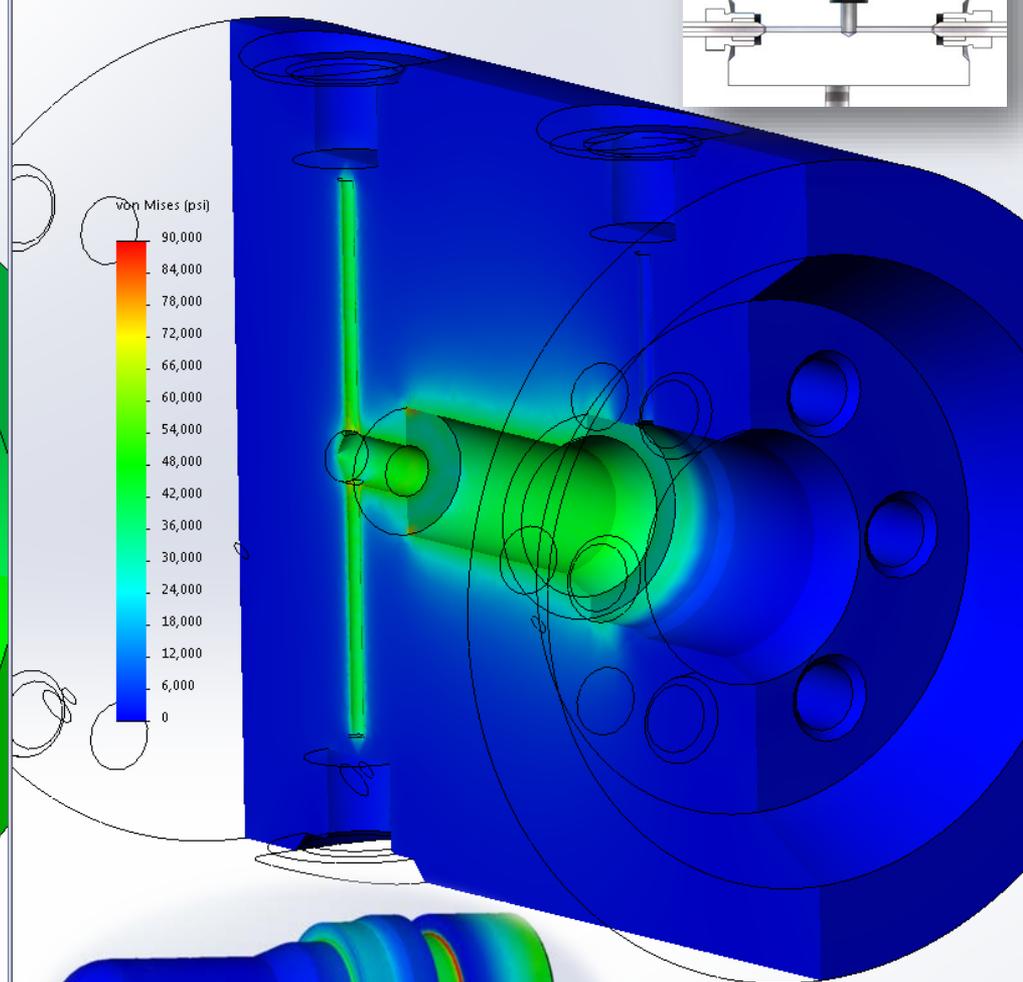
Actual dimensions of parts have been used in the report to correct values of the nominal clearances



Model name:OringTestBlock\_v4  
Study name:25ksi(-fea-)  
Plot type: Static displacement Displacement1  
Deformation scale: 1

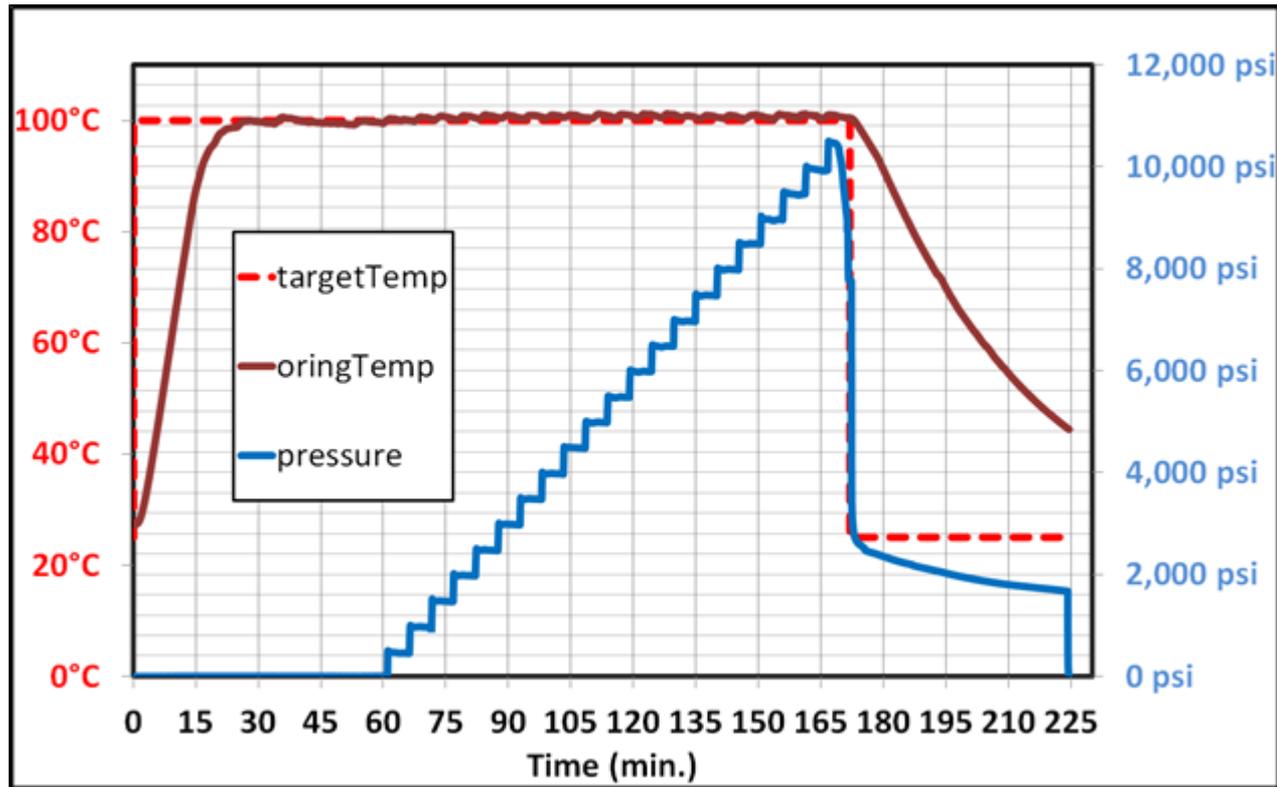


Model name:OringTestBlock\_v4  
Study name:25ksi(-fea-)  
Plot type: Static nodal stress Stress1  
Deformation scale: 1  
Volume (Element/Geometric) = 0.66 % / 0.18 %



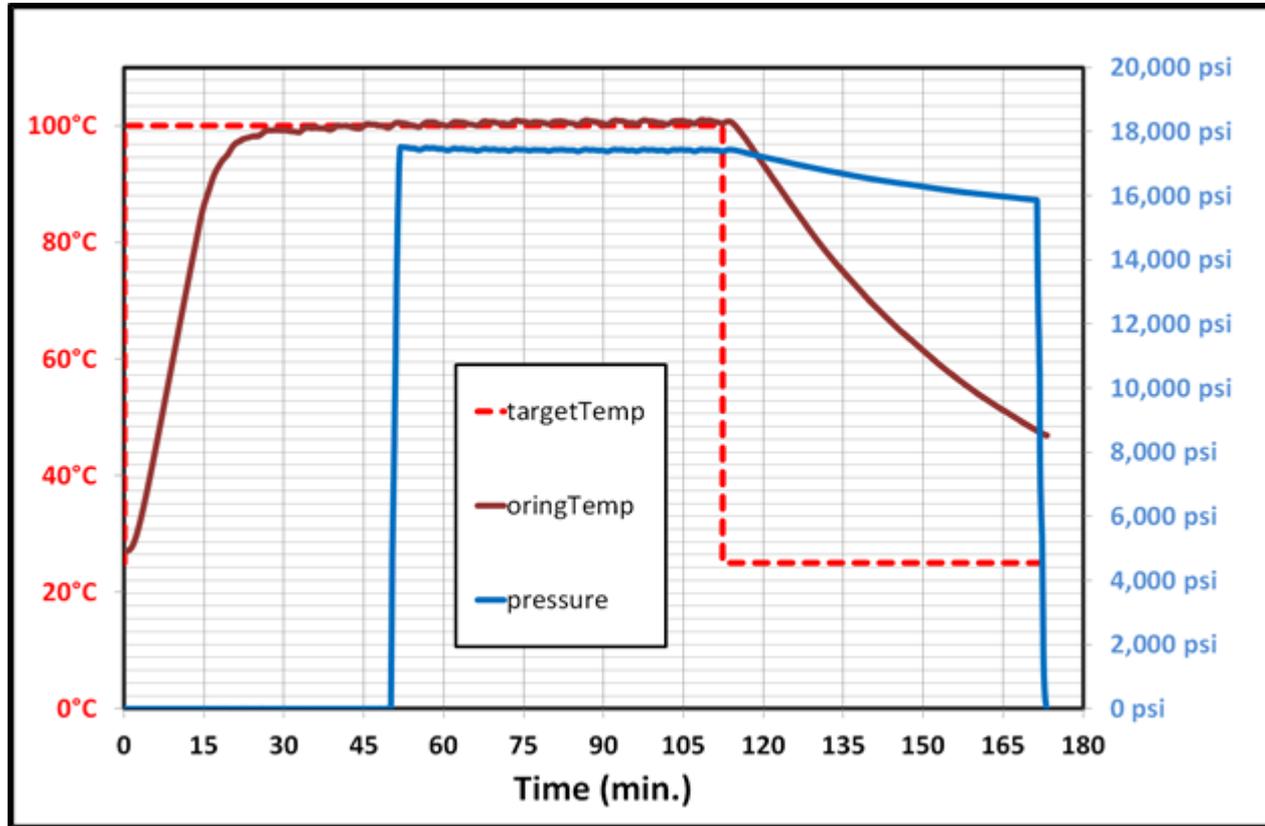
- Under 25ksi of pressure:**
- Radial bore increase is less than .4mil
  - Vessel's stresses contained within 70ksi

# Stepped Scan's Profile vs Time



HPHT step test profile per the D100511 procedure. The main steps are:

- (1) Heat to the target temperature,
- (2) when target Temp is reached, wait for at least 10 minutes before starting applying pressure,
- (3) increase pressure in increments of 500psi, keeping it at each pressure setting for 5 minutes until reaching the level at which the specimen fails to hold pressure,
- (4) turn the heater off and wait until temperature decreases below 50°C,
- (5) bleed pressure and stop logging

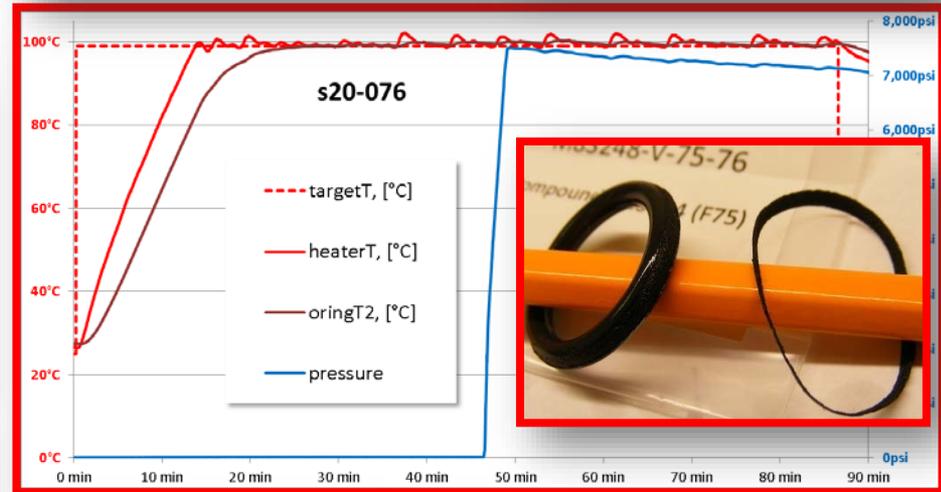
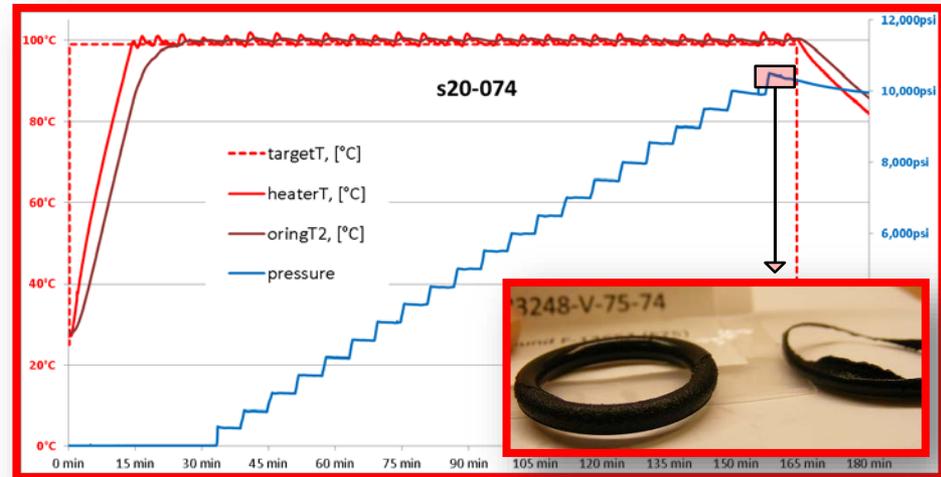


HPHT dwell test profile per the D100511 procedure: The main steps are:

- (1) Heat to the target temperature
- (2) When target Temp is reached, wait for at least 10 minutes before starting application of pressure
- (3) Increase pressure slowly to the target pressure,
- (4) Dwell for 1 hour and then turn the heater off,
- (5) Wait until temperature decreases below 50°C before releasing pressure,
- (6) Bleed pressure and stop logging.

# Extrusion Grouping / Color Coding

- **Red:**
  - First Large Extrusion event (stepped scans)
  - Large extrusion / deep circumferential cuts
- **Yellow:**
  - Thin-band cut-off
  - Small-size extrusion with visible damage
  - Localized cuts/tears
  - Nibbled surface
- **Green:**
  - No visible damage
  - Seating



# Green Group Examples

Aflas83: Dwell, 15mil,  
100°C, 1750psi



Nitrile75: Dwell, 8mil,  
100°C, 4250psi



Kalrez75: Dwell,  
12mil, 100°C, 2500psi

210-H-75-65  
Compound R100

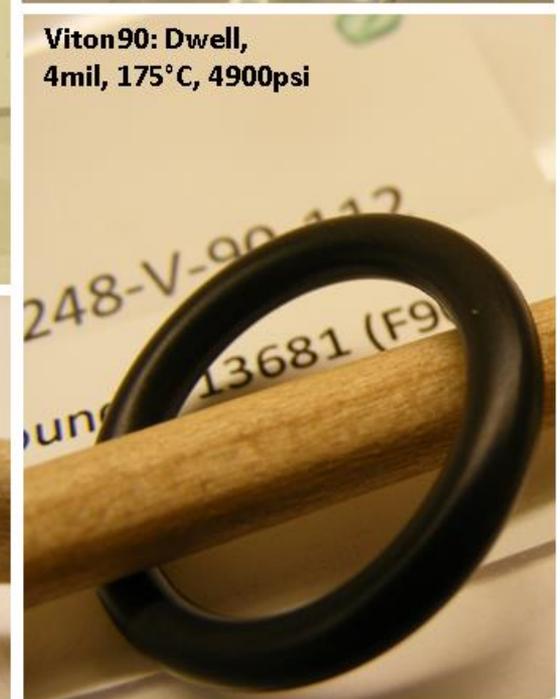


210-K-90-52  
Compound K3018



Kalrez90: Dwell,  
15mil, 175°C, 1500psi

Viton90: Dwell,  
4mil, 175°C, 4900psi



HNBR75: Dwell,  
8mil, 150°C, 2250psi



Kalrez90: Dwell, 15mil,  
100°C, 3500psi



Viton75: Dwell,  
4mil, 100°C, 4900psi

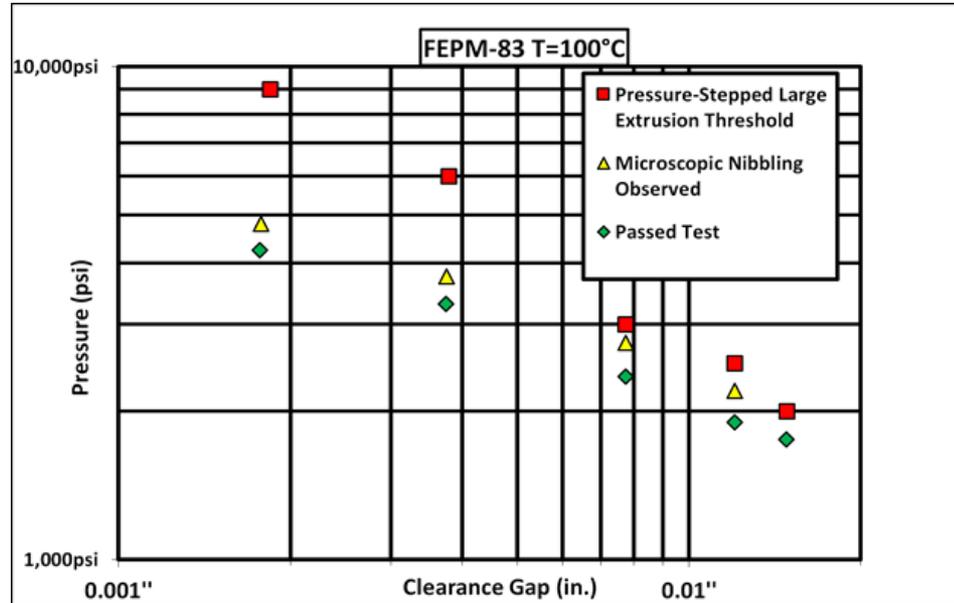
# Yellow Group Examples



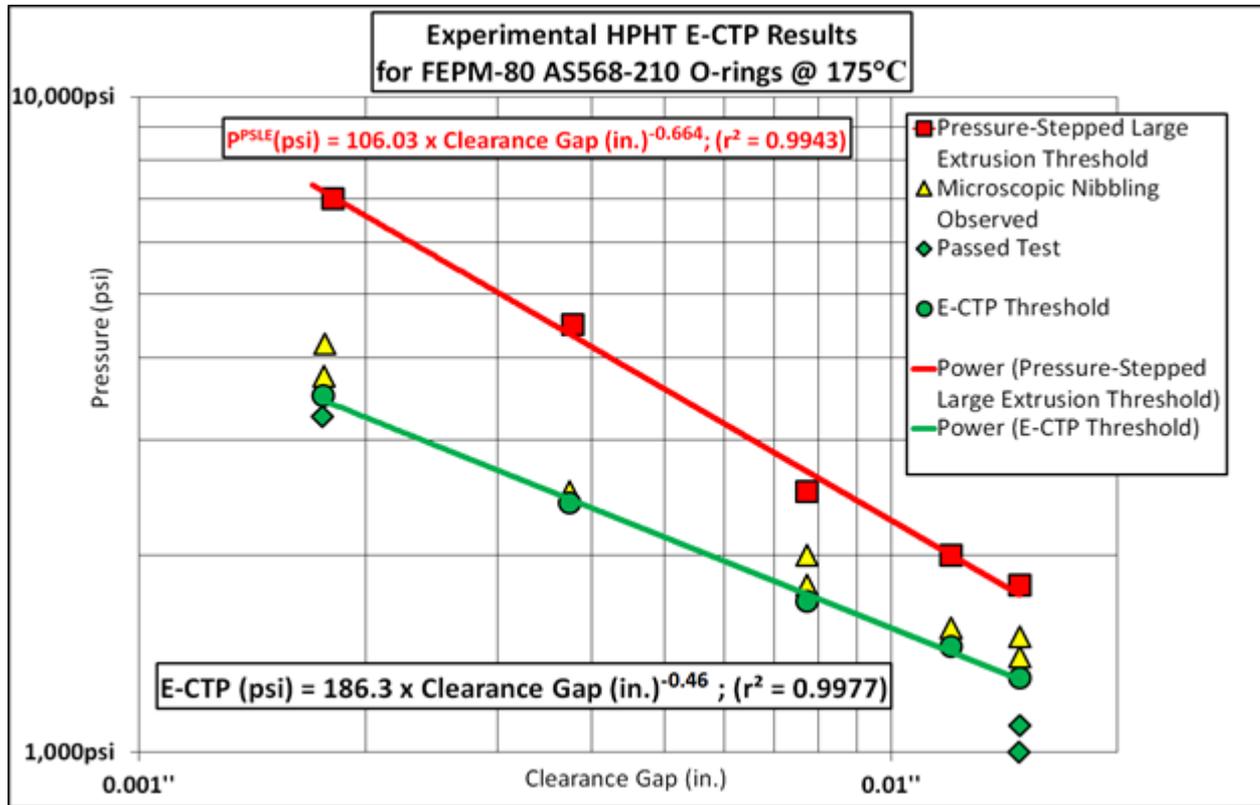
# Red Group Examples



# Test Matrix & Graphical Representation



	#10/.002"	#20/.004"	#30/.008"	#40/.012"	#50/.015"
O-Ring #	25	19	13	7	1
Pressure (psi)	9,000	6000	3,000	2,500	2,000
O-Ring #	26	20	14	8	2
Pressure (psi)	nt	nt	nt	nt	nt
O-Ring #	27	21	15	9	3
Pressure (psi)	4,800	3,300	2,350	1,900	1,750
O-Ring #	28	22	16	10	4
Pressure (psi)	4,250	3,750	2,750	2,200	2,000
O-Ring #	29	23	17	11	5
Pressure (psi)	nt	nt	nt	nt	nt
O-Ring #	30	24	18	12	6
Pressure (psi)	nt	nt	nt	nt	nt



To an acceptable degree of accuracy, the upper pressure level of the Green group will define the E-CTP for each set of test parameters. It was found that the critical pressures for the five clearances tested could be interpolated using power regression

$$E-CTP = A * C^B,$$

where

E-CTP is a critical tear pressure in [psi],  
C is clearance gap in [inch],  
A and B are coefficients.

# Coefficients of E-CTP power regression for extrusion tests at 100°C and 175°C

Power Regression Coefficients				P=A*C^(B), where P [psi] & C [inch]			
				E-CTP		SS-FLE	
Material	Compound Reference	Durometer	Temp (°C)	A	B	A	B
FKM-75	F-13664 (F75)	75	100	184.572	-0.584	73.010	-0.904
FKM-90	F-13681 (F90)	90	100	533.899	-0.479	135.482	-0.905
NBR-75	B1016	75	100	720.374	-0.386	181.667	-0.814
NBR-90	B1001	90	100	760.005	-0.479	126.031	-1.002
HNBR-75	R1006	75	100	133.608	-0.608	127.474	-0.701
HNBR-90	R1003	90	100	918.098	-0.411	191.328	-0.850
FEPM-80	L1000	89	100	217.904	-0.490	120.629	-0.715
FEPM-83	A-210	83	100	292.565	-0.427	137.227	-0.664
FFKM-75	K4079	75	100	304.152	-0.481	5.802	-1.445
FFKM-90	K3018	90	100	399.140	-0.502	58.838	-1.092

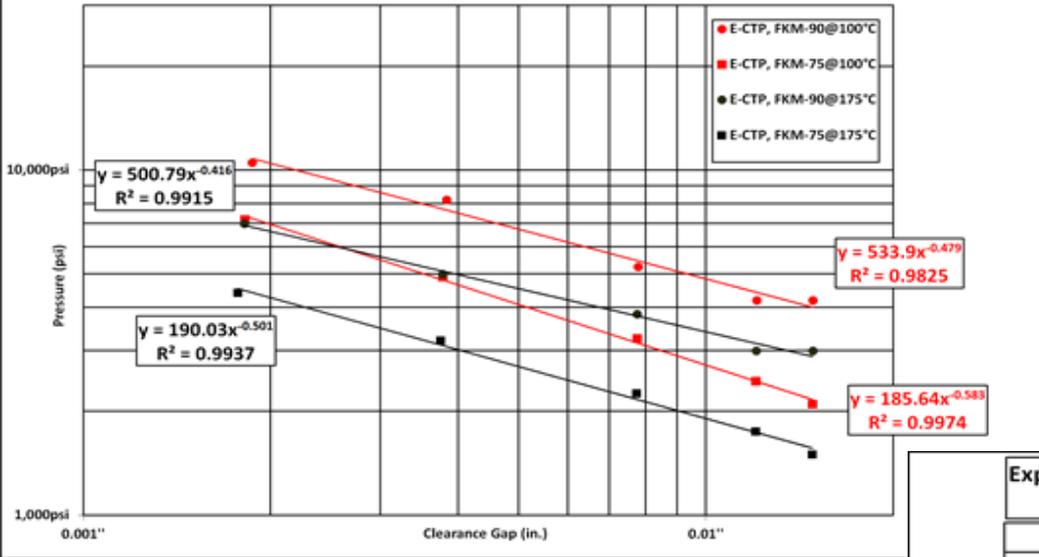
Power Regression Coefficients				P=A*C^(B), where P [psi] & C [inch]			
				E-CTP		SS-FLE	
Material	Compound Reference	Durometer	Temp (°C)	A	B	A	B
FKM-75	F-13664 (F75)	75	175	190.034	-0.501	90.945	-0.760
FKM-90	F-13681 (F90)	90	175	500.792	-0.416	148.567	-0.779
NBR-75	B1016	75	--				
NBR-90	B1001	90	--				
HNBR-75	R1006	75	150	455.224	-0.339	375.276	-0.500
HNBR-90	R1003	90	150	737.688	-0.426	268.642	-0.759
FEPM-80	L1000	89	175	186.302	-0.460	106.028	-0.664
FEPM-83	A-210	83	175	373.009	-0.343	129.339	-0.647
FFKM-75	K4079	75	175	200.431	-0.494	36.051	-1.008
FFKM-90	K3018	90	175	254.974	-0.423	110.993	-0.758

## (Tabular Data)

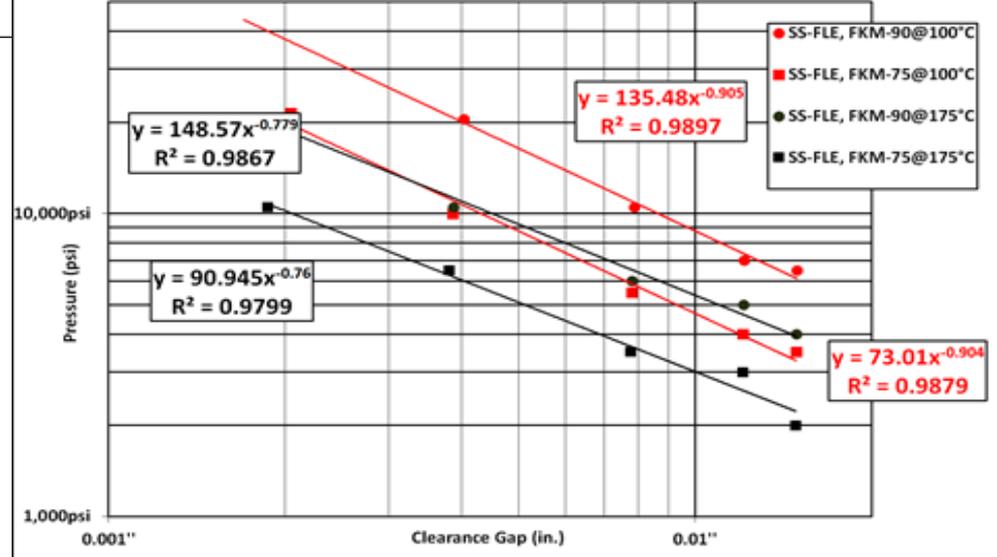
<b>E-CTP FKM-75, FKM-90</b>								
Nominal Clearance Gap (@ 25°C)	Clearance Gap (mils)@ 100°C	E-CTP, FKM-75 @ 100°C (psi)	Clearance Gap @ 100°C (mils)	E-CTP, FKM-90 (psi)	Clearance Gap (mils) @175°C	E-CTP, FKM-75@175°C (psi)	Clearance Gap @175°C (mils)	E-CTP, FKM-90 @ 175°C (psi)
0.0148"	14.8336	2,100	14.8672	4,200	14.824	1,500	14.848	3,000
0.012"	12.0392	2,450	12.0672	4,200	12.028	1,750	12.0248	3,000
0.0077"	7.752	3,250	7.784	5,250	7.736	2,250	7.7612	3,825
0.0037"	3.774	4,900	3.8312	8,200	3.7512	3,200	3.78	5,000
0.0017"	1.8152	7,200	1.868	10,500	1.7704	4,400	1.812	7,000
<b>SS-FLE FKM-75, FKM-90</b>								
Nominal Clearance Gap (@ 25°C)	Clearance Gap (mils) @ 100°C	SS-FLE, FKM-75 @100°C (psi)	Clearance Gap @ 100°C (mils)	SS-FLE, FKM-90 @100°C (psi)	Clearance Gap (mils) @175°C	SS-FLE, FKM-75 @175°C (psi)	Clearance Gap @175°C (mils)	SS-FLE, FKM-90 @175°C (psi)
0.0148"	14.856	3,500	14.904	6,500	14.832	2,000	14.864	4,000
0.012"	12.064	4,000	12.112	7,000	12.048	3,000	12.08	5,000
0.0077"	7.788	5,500	7.868	10,500	7.756	3500	7.796	6,000
0.0037"	3.86	10,000	4.028	20,500	3.804	6,500	3.868	10,500
0.0017"	2.044	21500	1.7	Use Power law	1.868	10,500	2.02	20,000

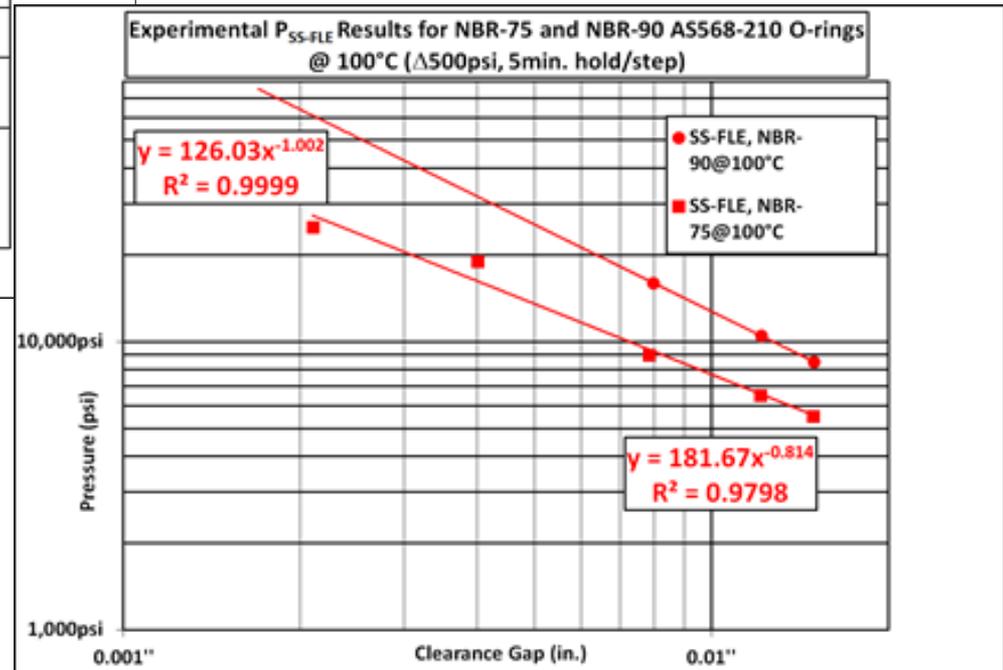
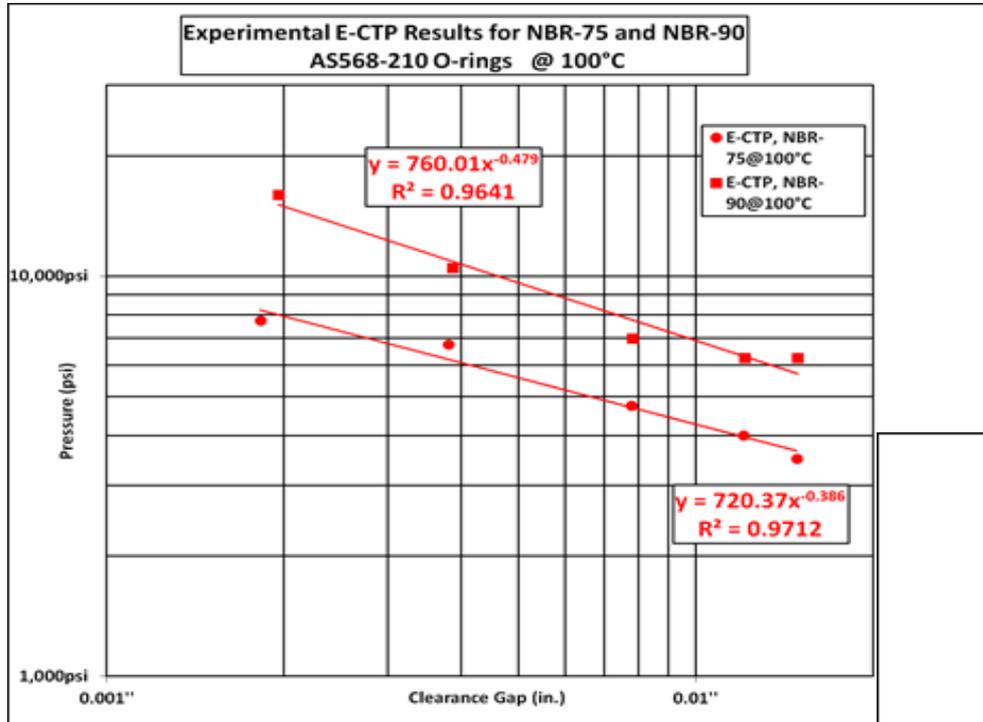
Clearance Gap is adjusted for Thermal Expansion of metal gland and actual pressures applied

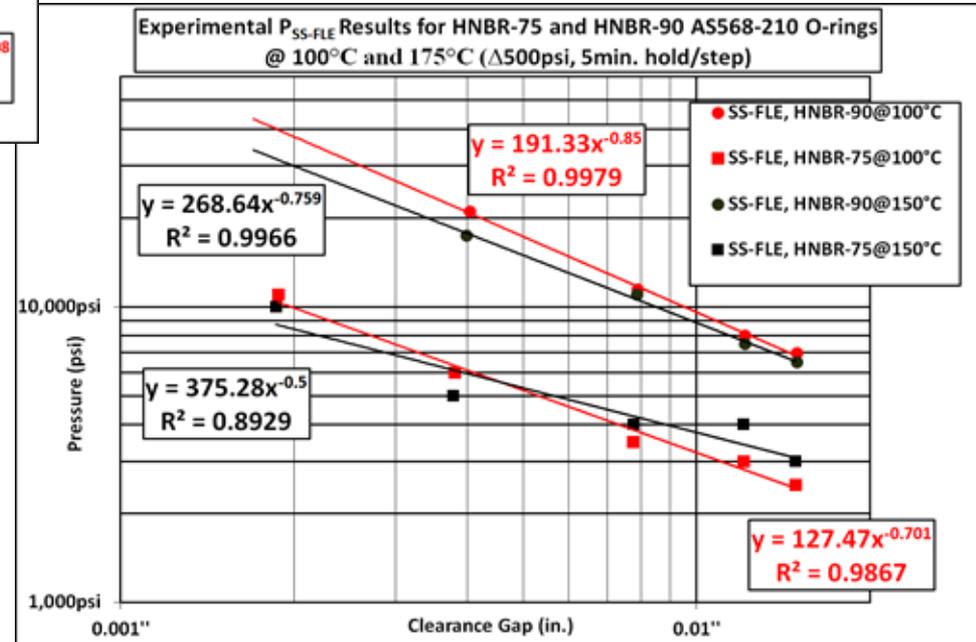
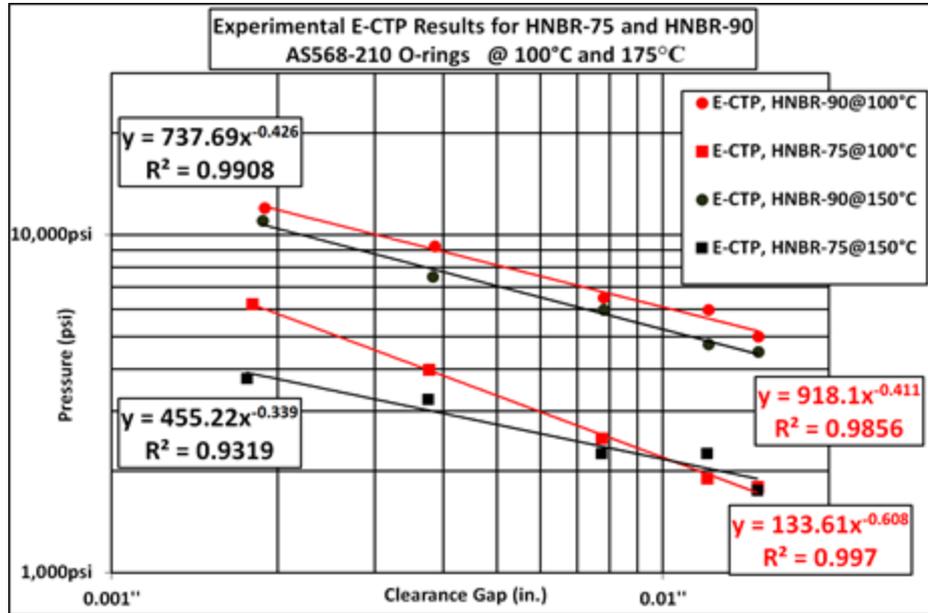
Experimental E-CTP Results for FKM-75 and FKM-90 AS568-210 O-rings  
@ 100°C and 175°C

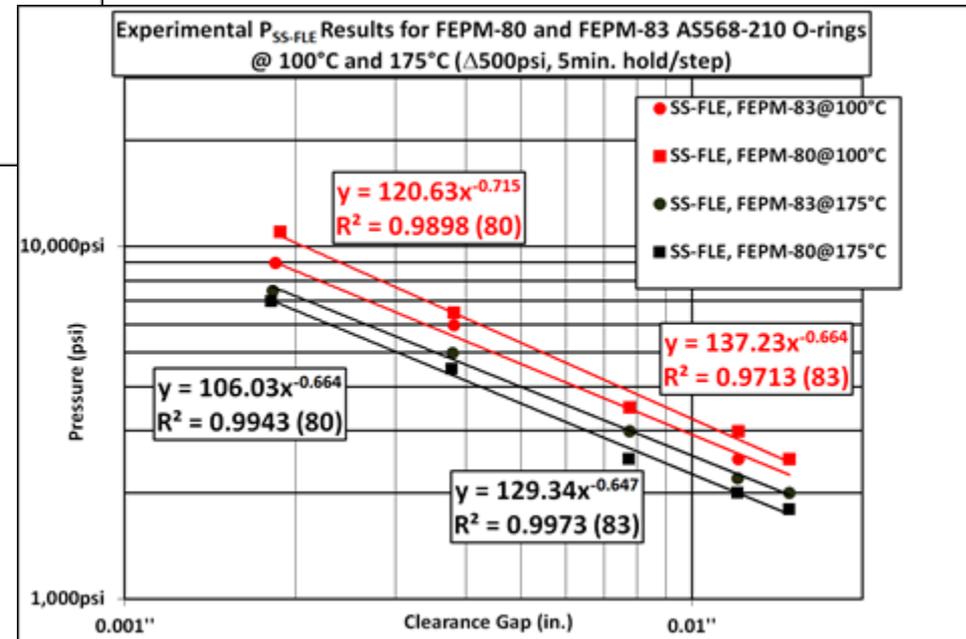
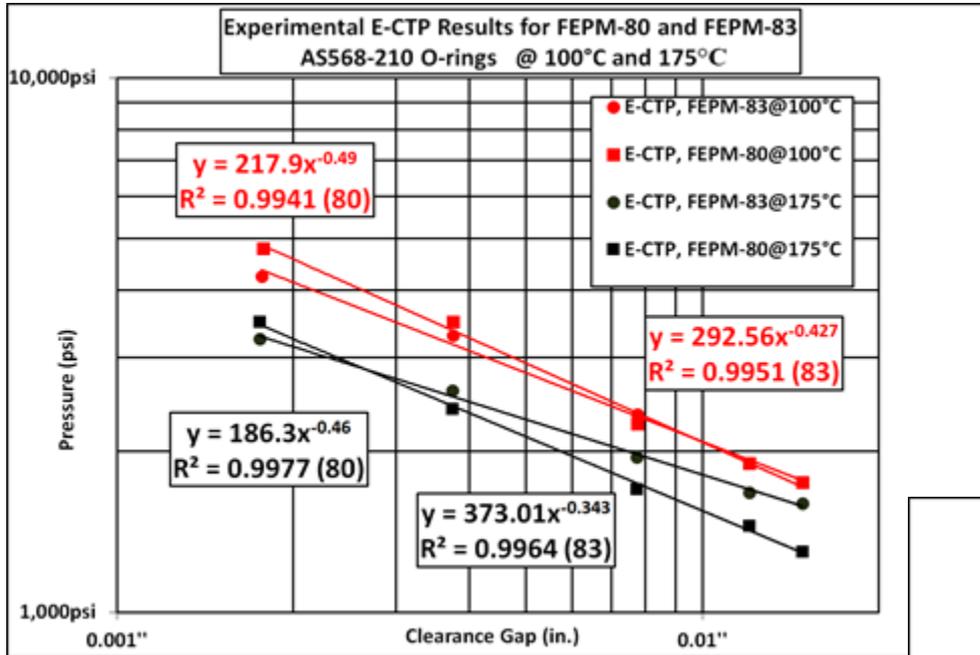


Experimental P<sub>SS-FLE</sub> Results for FKM-75 and FKM-90 AS568-210 O-rings  
@ 100°C and 175°C (Δ500psi, 5min. hold/step)

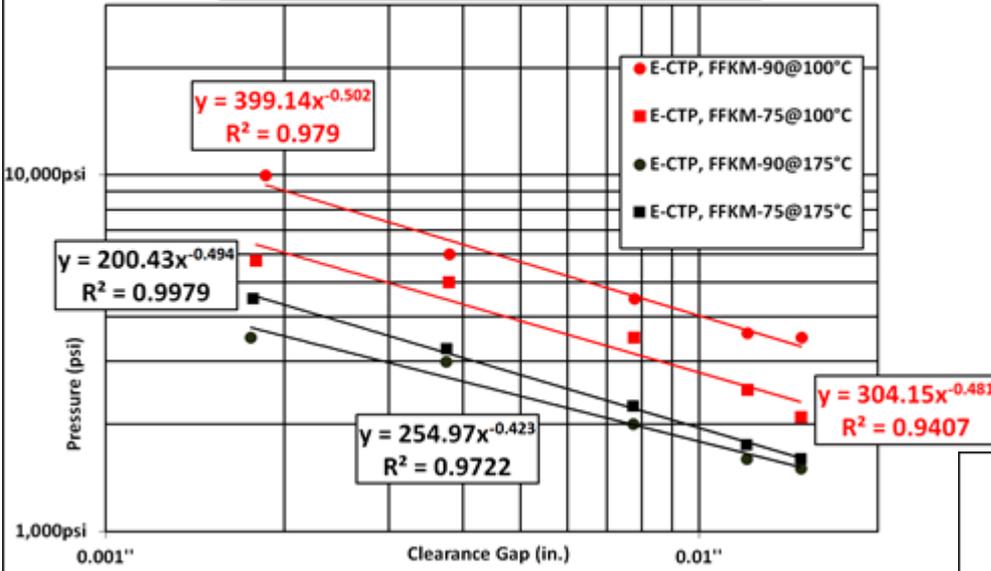




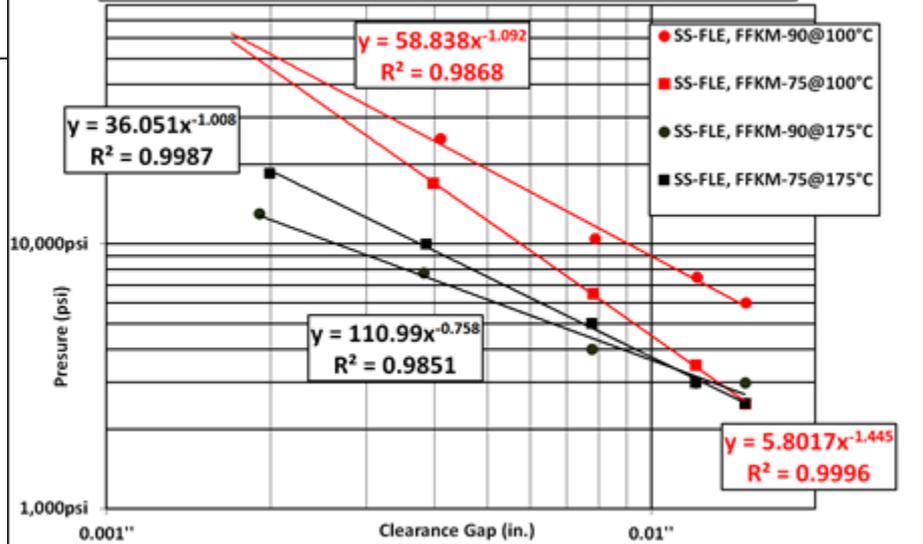


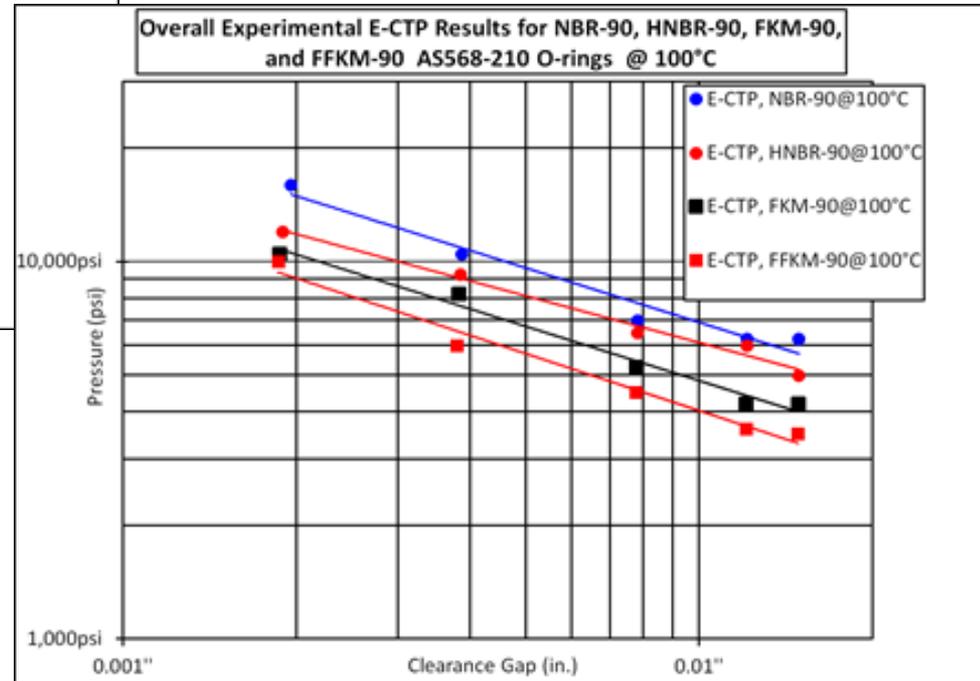
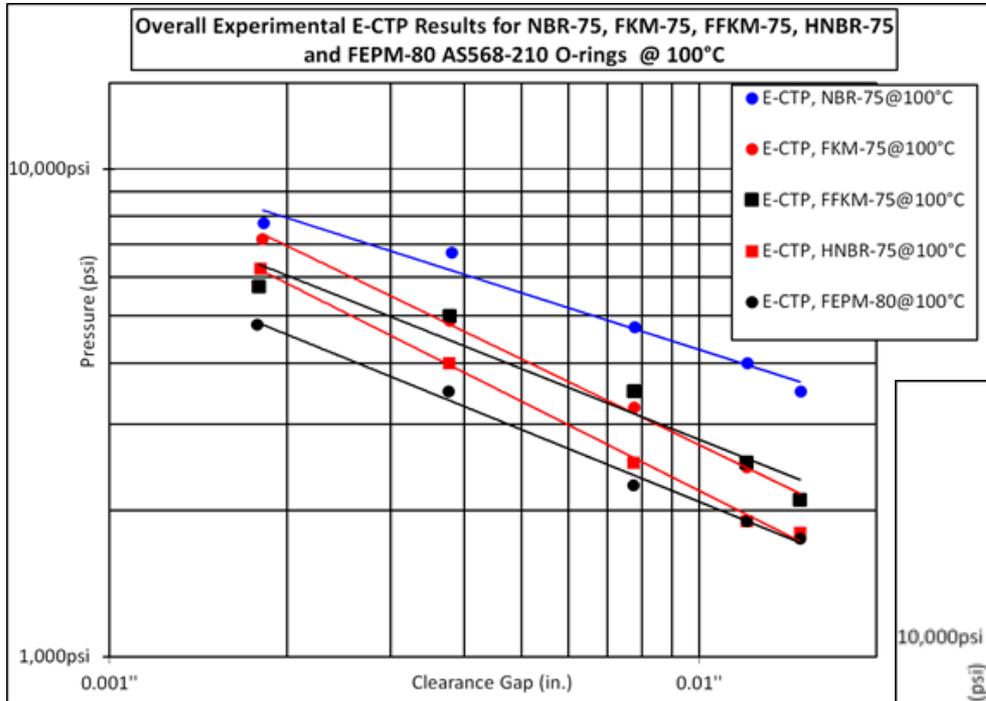


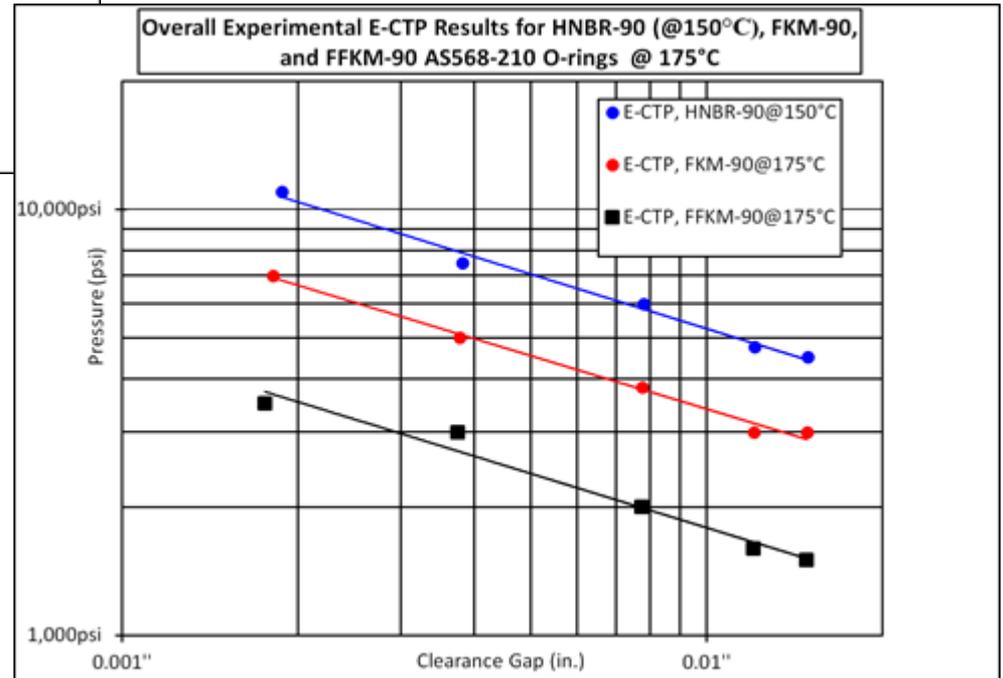
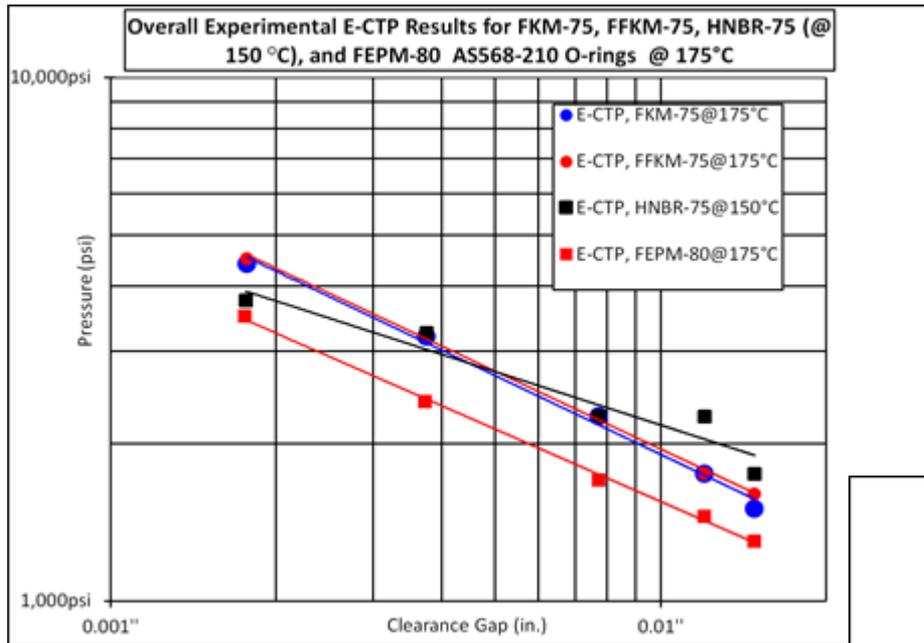
Experimental E-CTP Results for FFKM-75 and FFKM-90 AS568-210 O-rings @ 100°C and 175°C

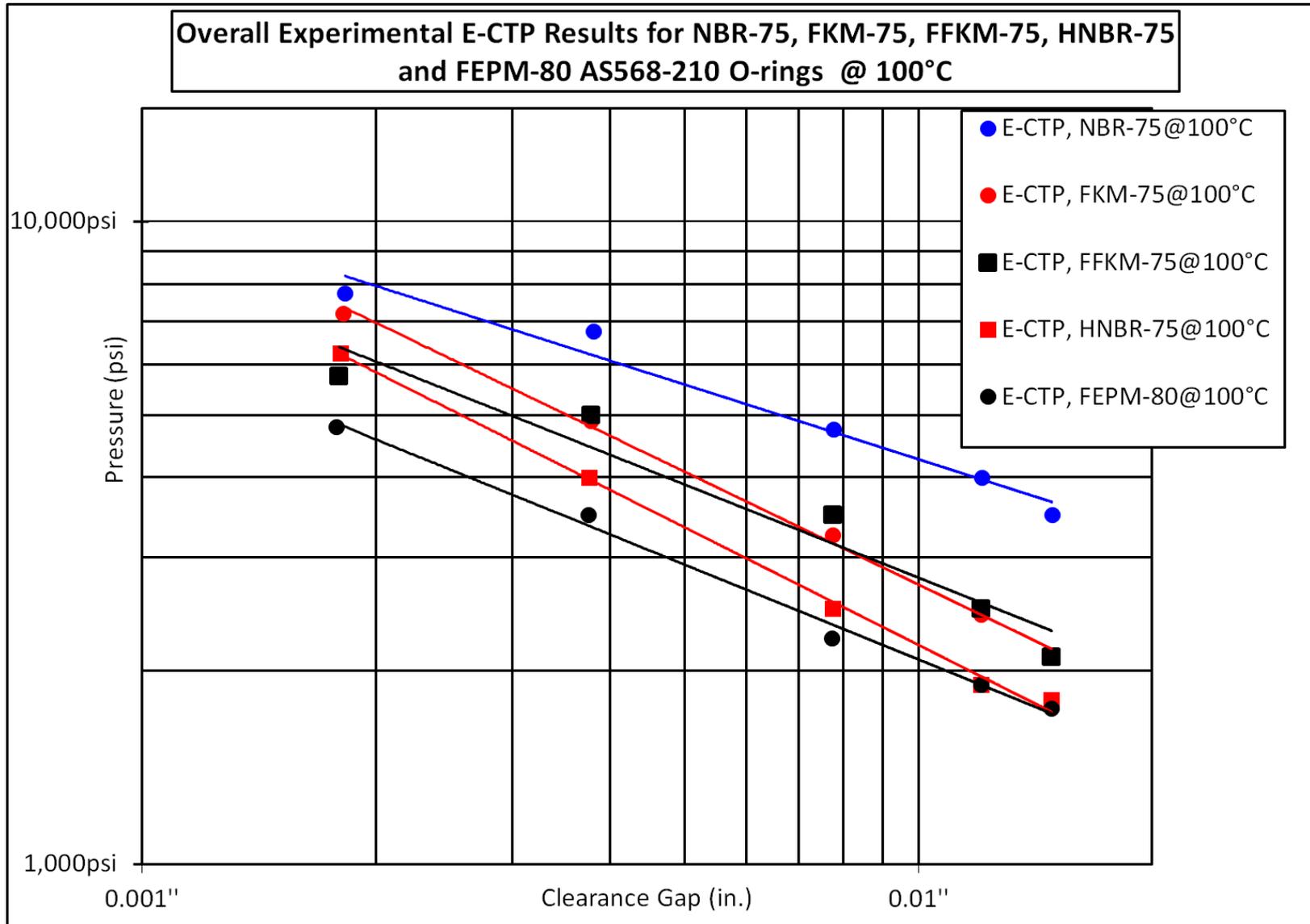


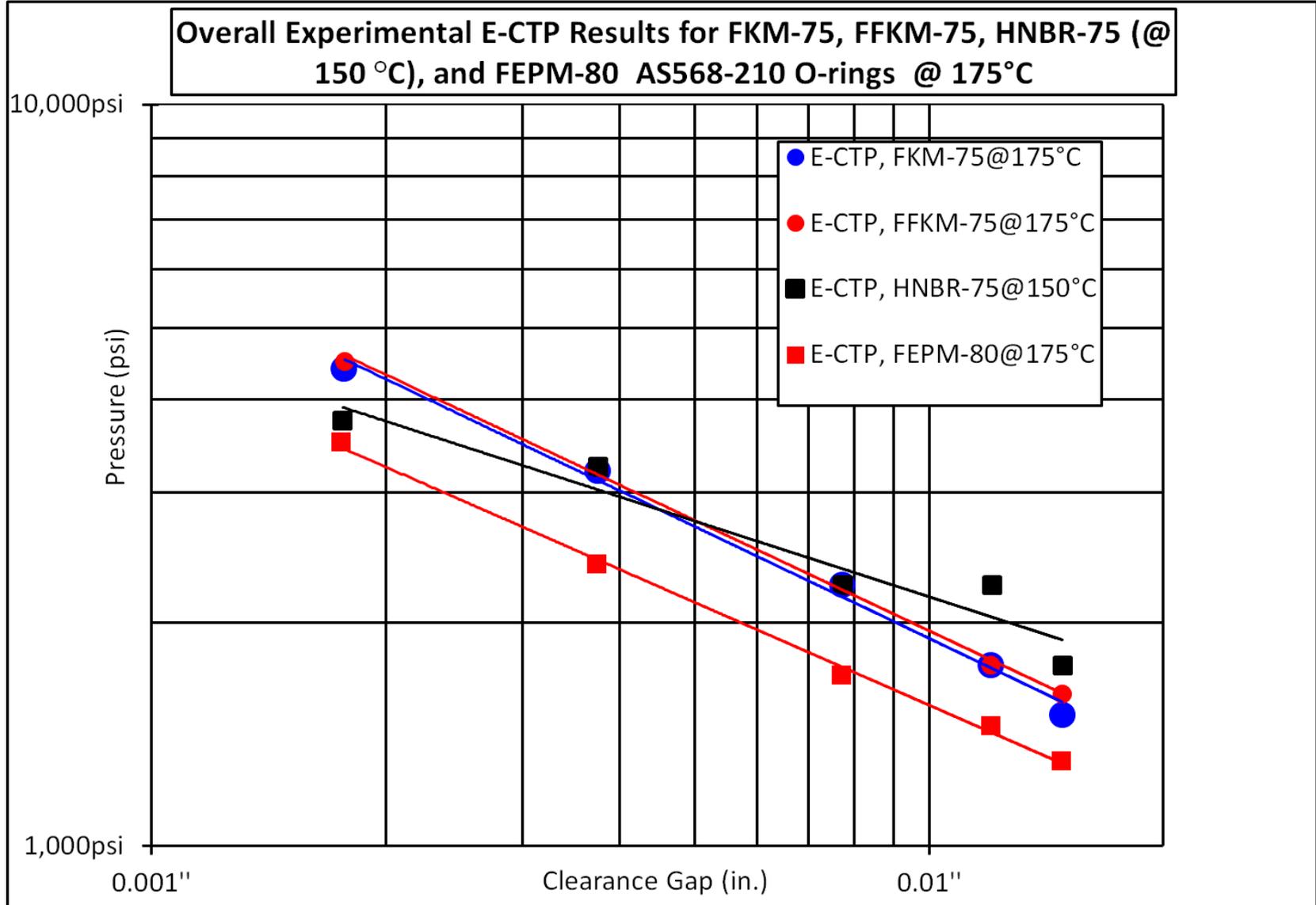
Experimental P<sub>SS-FLE</sub> Results for FFKM-75 and FFKM-90 AS568-210 O-rings @ 100°C and 175°C (Δ500psi, 5min. hold/step)

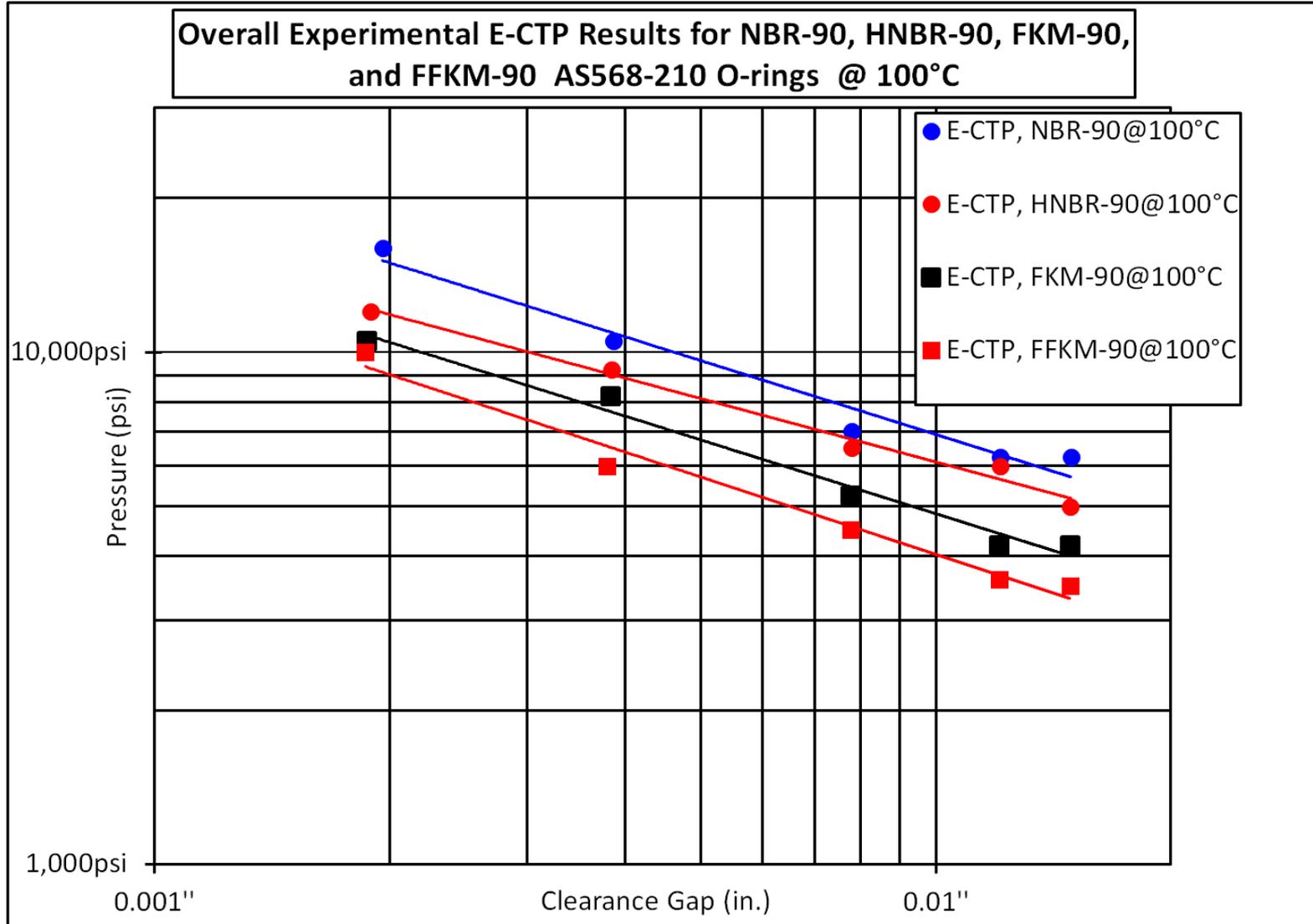


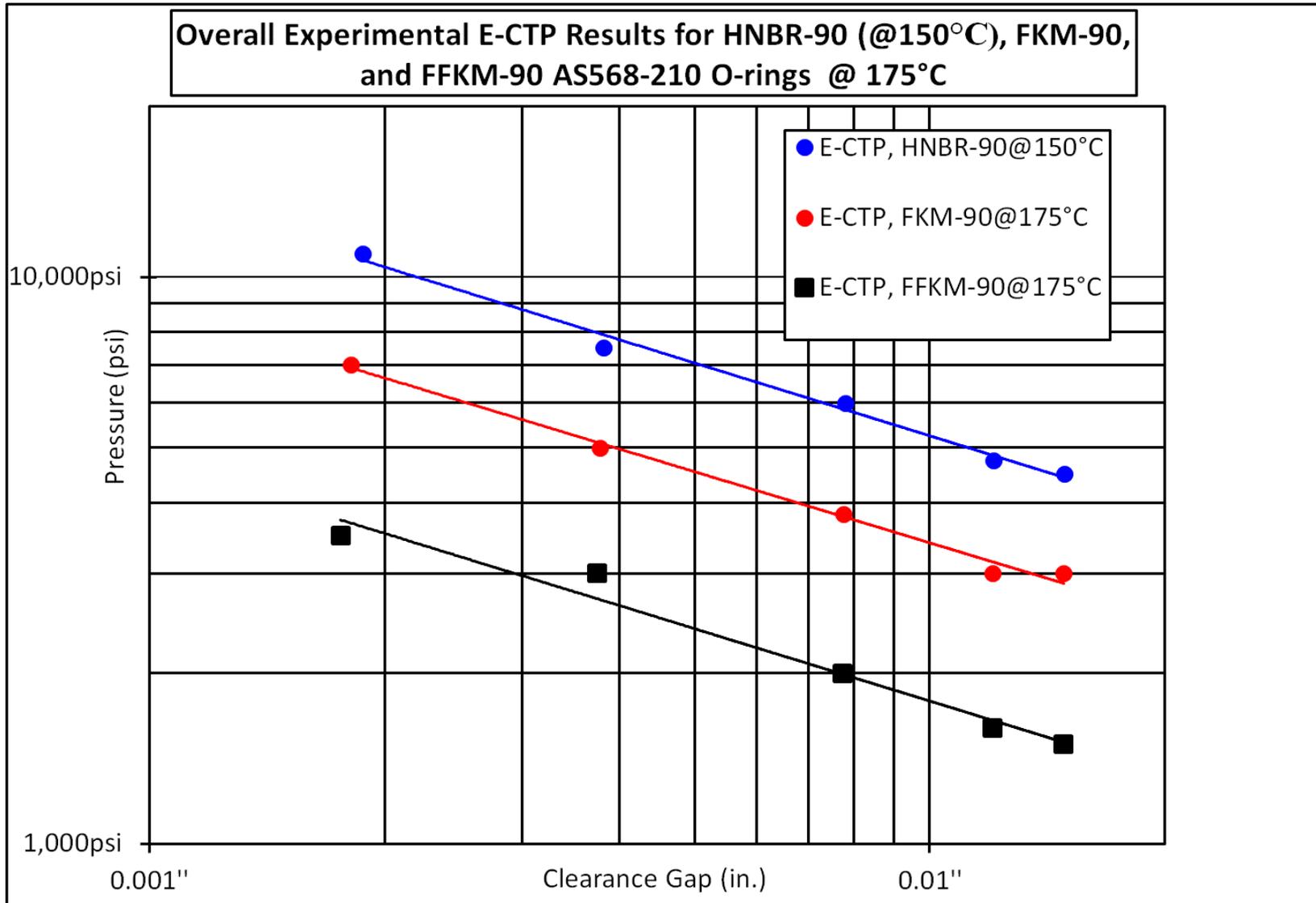


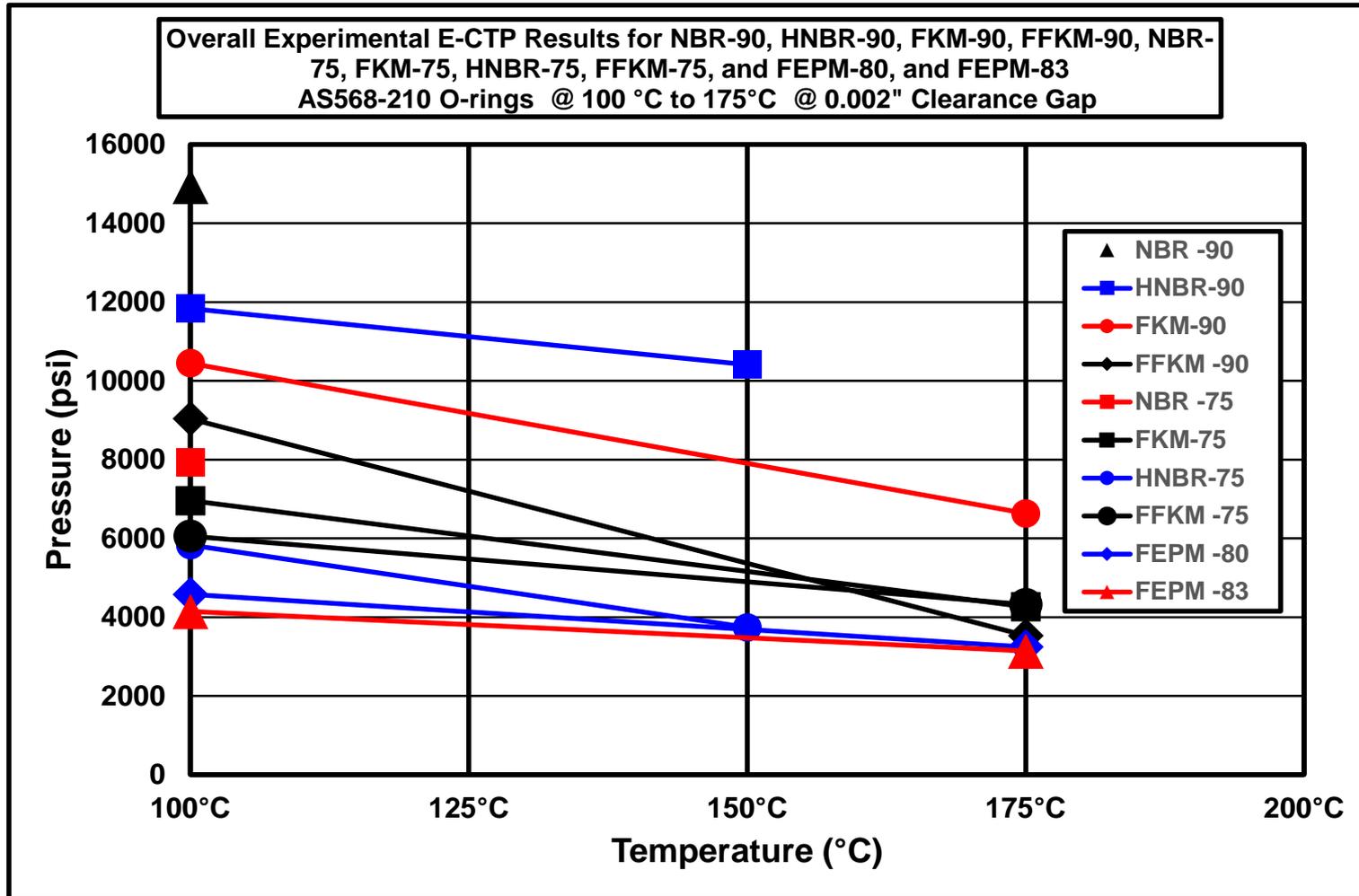


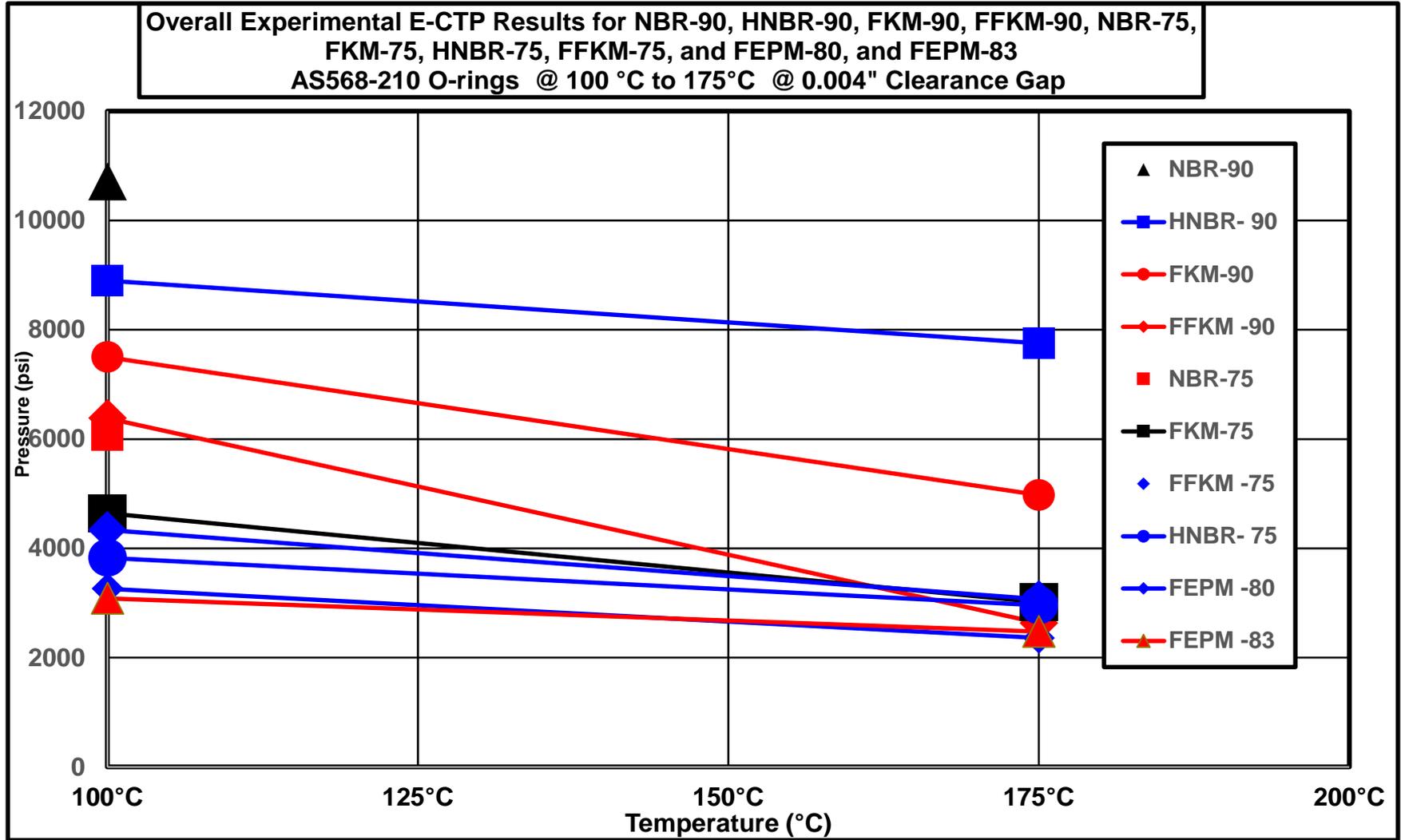












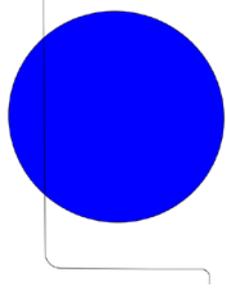
# Task 5 – FEA Model Development

## Endurica

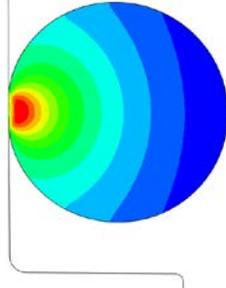
# Task 5 – FEA Model Development

- Data collected in Task 3 and Task 4 were used to create a FEA model capable of predicting the onset of failure.
- Model Critical Tear Pressure (M-CTP) was determined for each material.
  - Pressure at which tearing and failure of material is expected to occur.
- E-CTP and M-CTP results compared for 75 and 90 Shore A hardness materials.
- Effect of test fixture radius and friction factors explored.

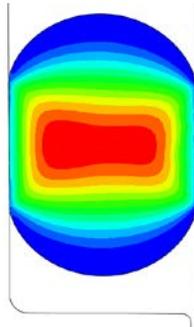
# FEA Model Execution Steps



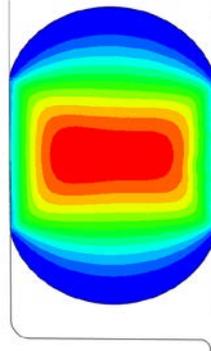
1 - Initial



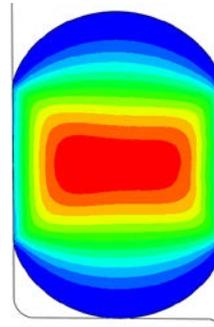
2 – Stretch  
around  
piston



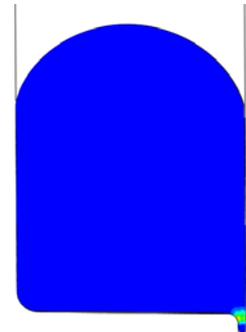
3 – Squeeze  
inside  
cylinder



4 – Thermal  
Expansion

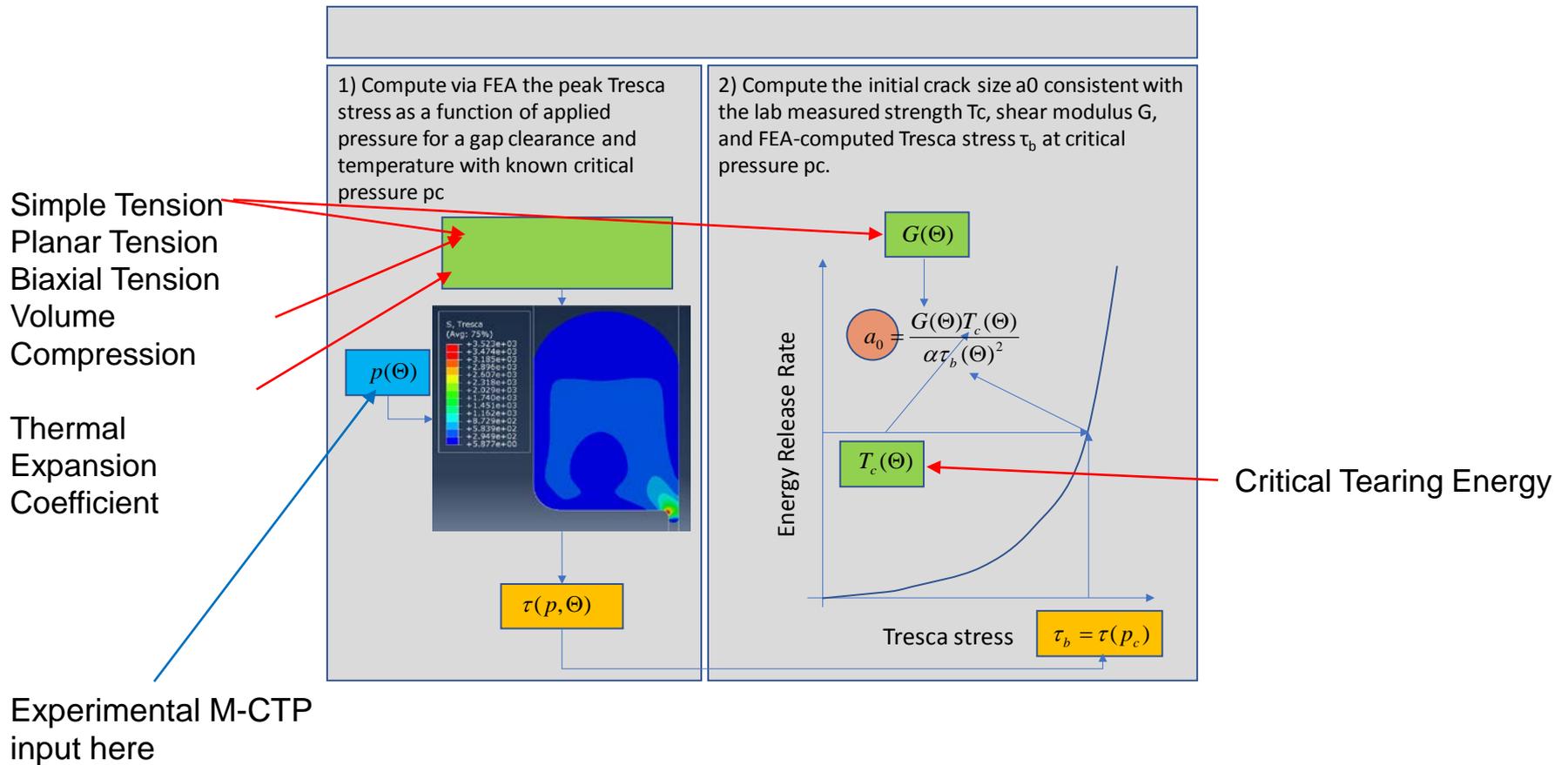


5 – Initiate  
contact with  
gland face

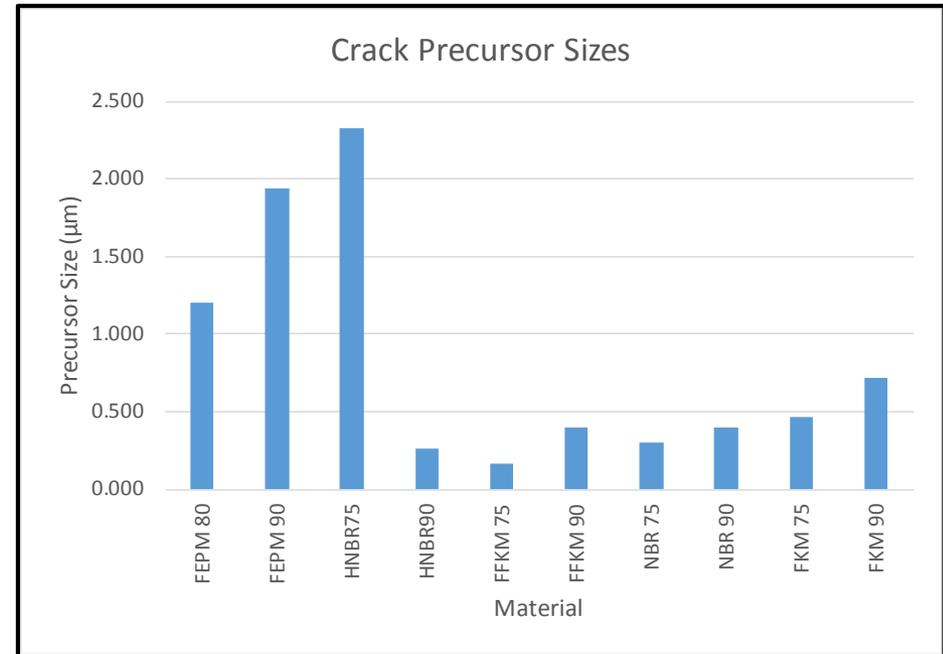
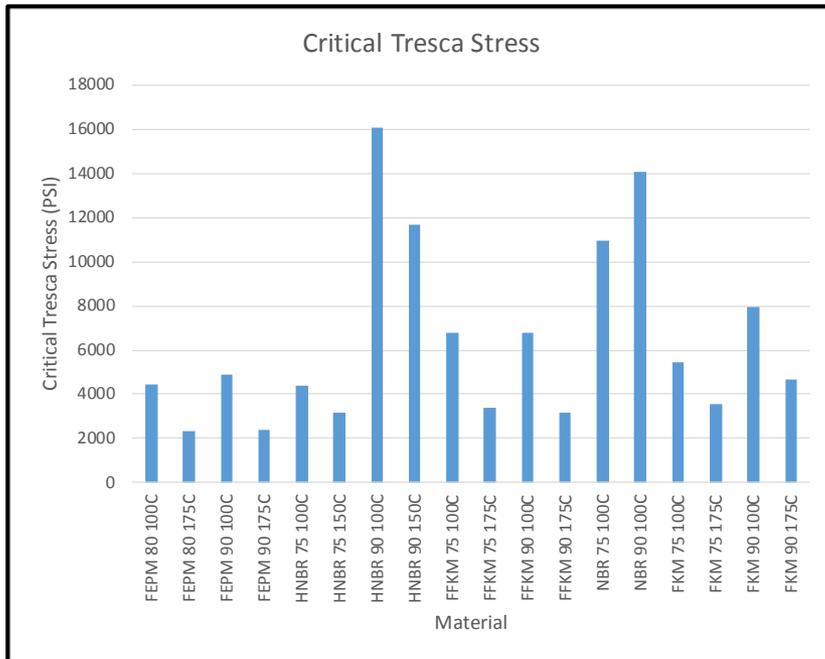


6 – Apply  
Pressure

# Calibrating Crack Precursor Size

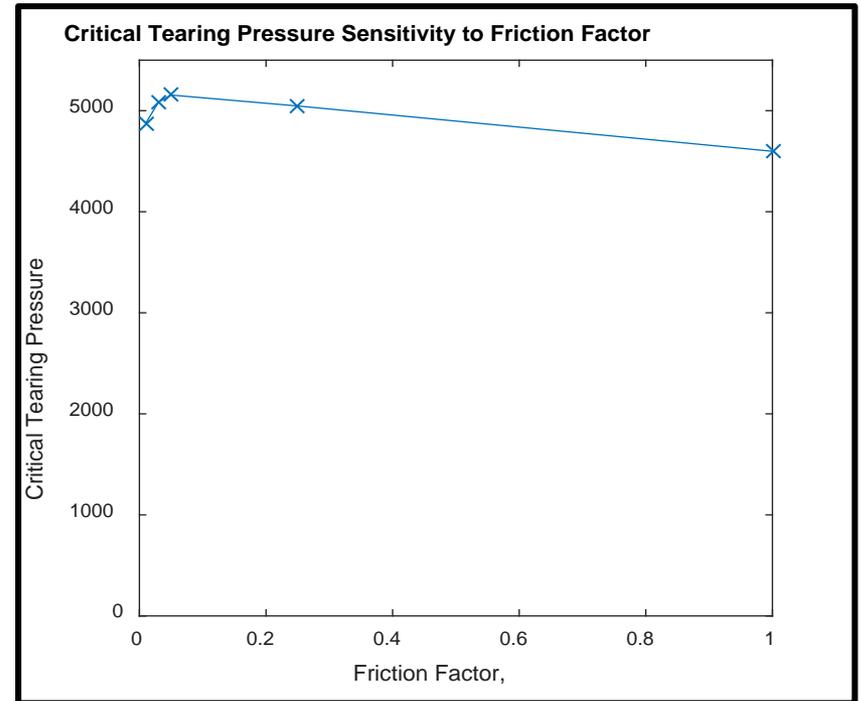
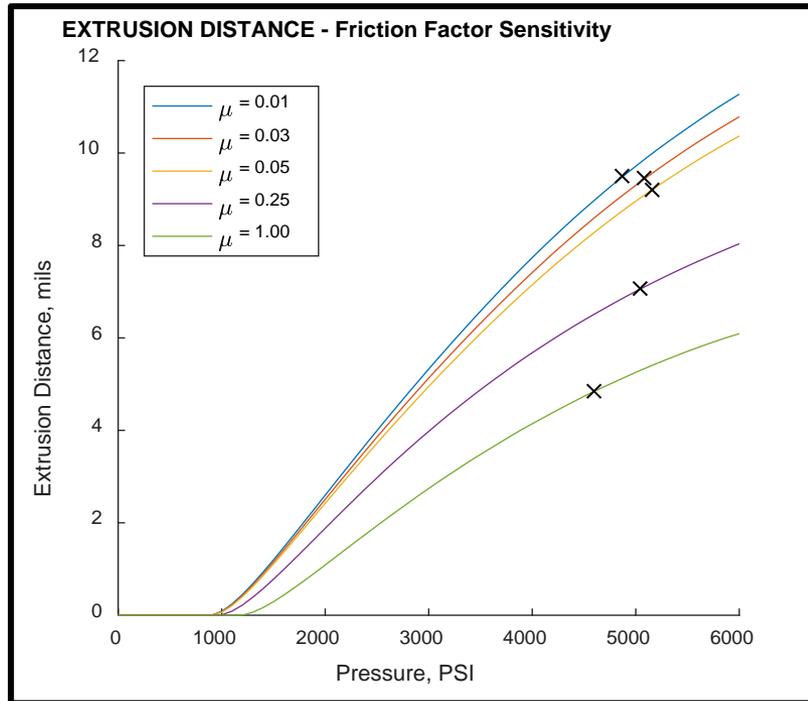


# Model Calibration Results for Each Elastomer

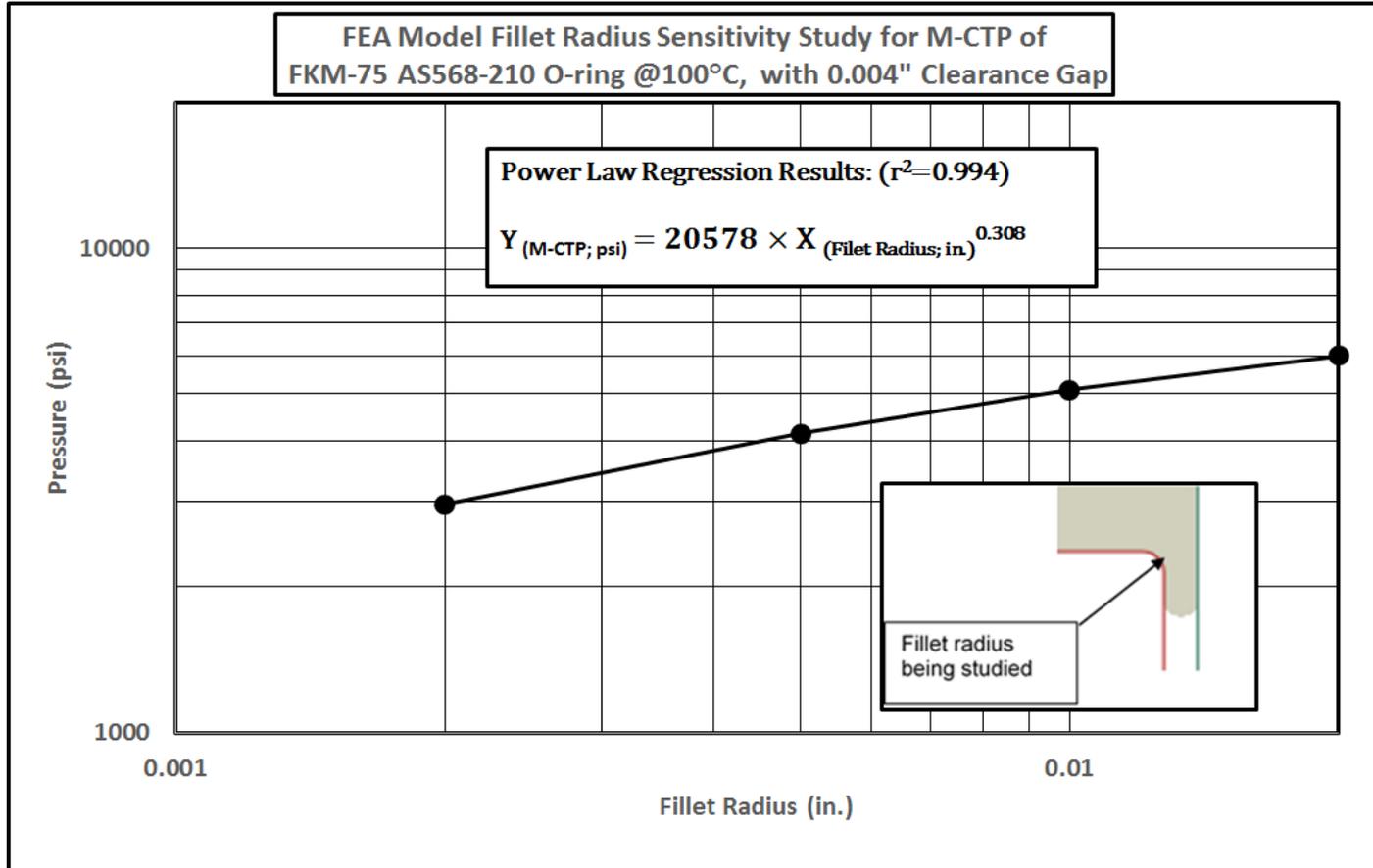


- NBR and HNBR-90 had highest Critical Tresca Stress Values and small Crack Precursor sizes
- FKM and FFKM had low Critical Tresca Stress and small Crack Precursor sizes
- FEPM had low Critical Tresca Stress and larger Crack Precursor sizes

# Effect of friction coefficient between Elastomer and Steel Gland was minimized

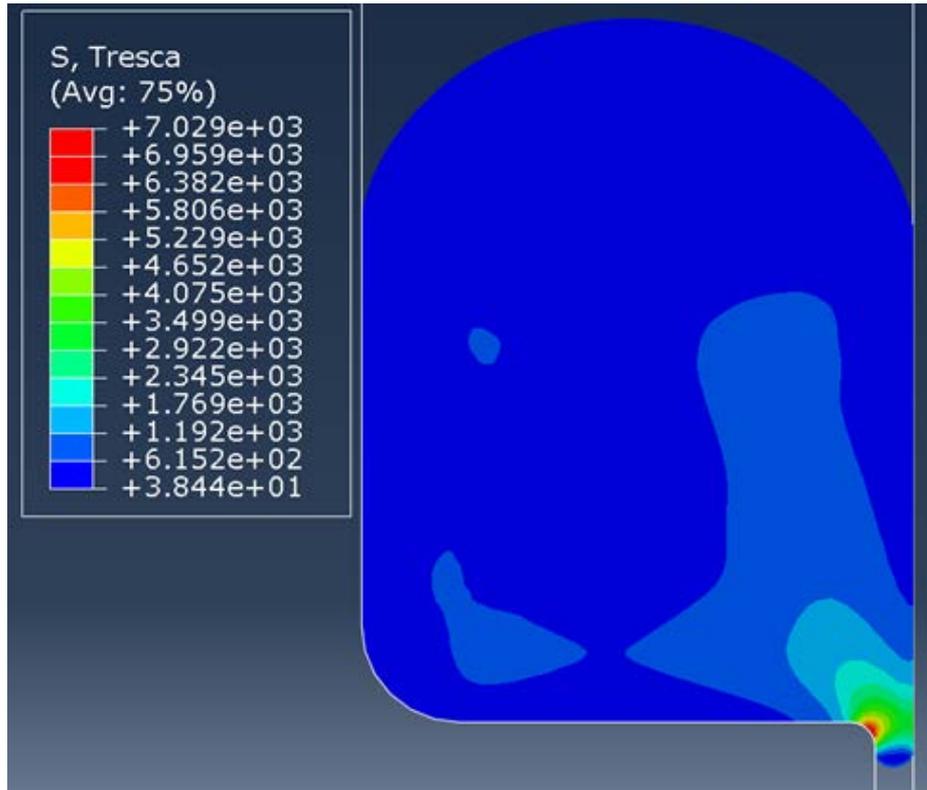


# Effect of radius on M-CTP for Elastomers has been determined

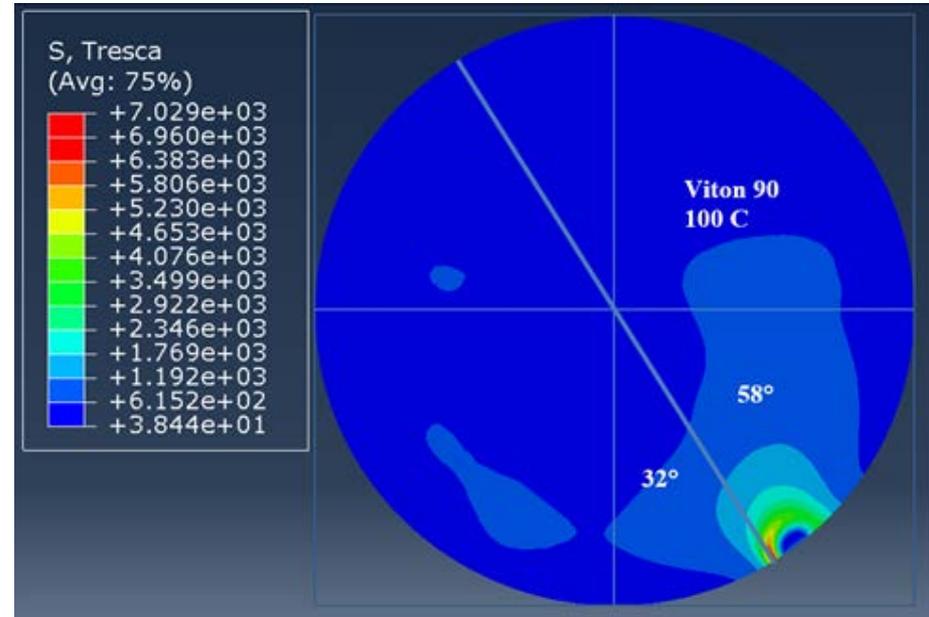




# Tresca Stress Plotted on O-Ring: FKM-90 @ 100°C

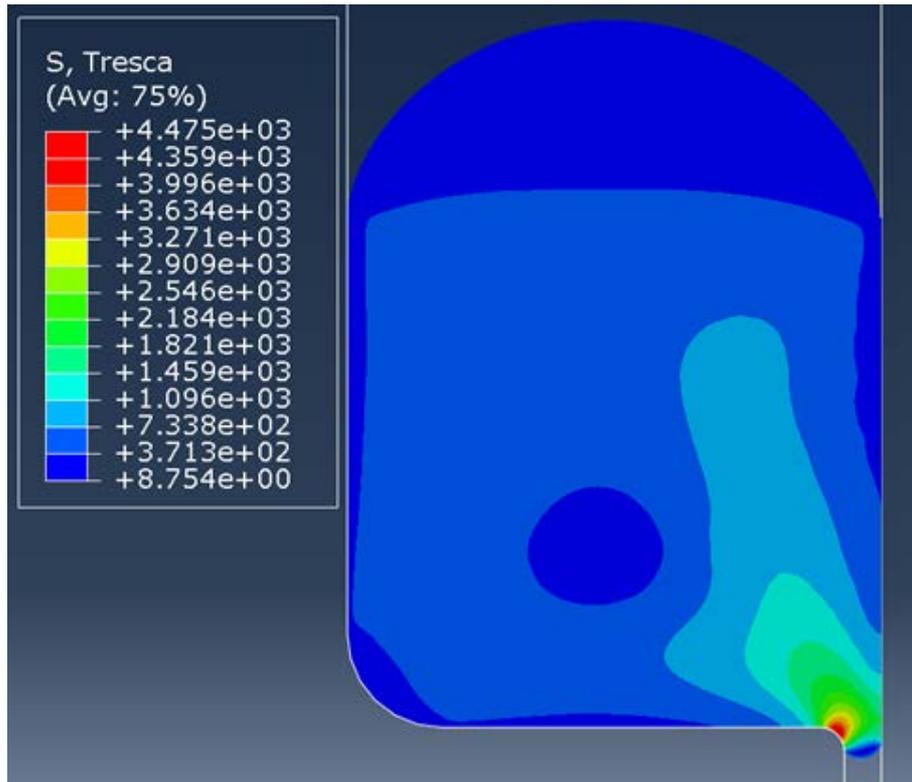


Deformed State @ M-CTP

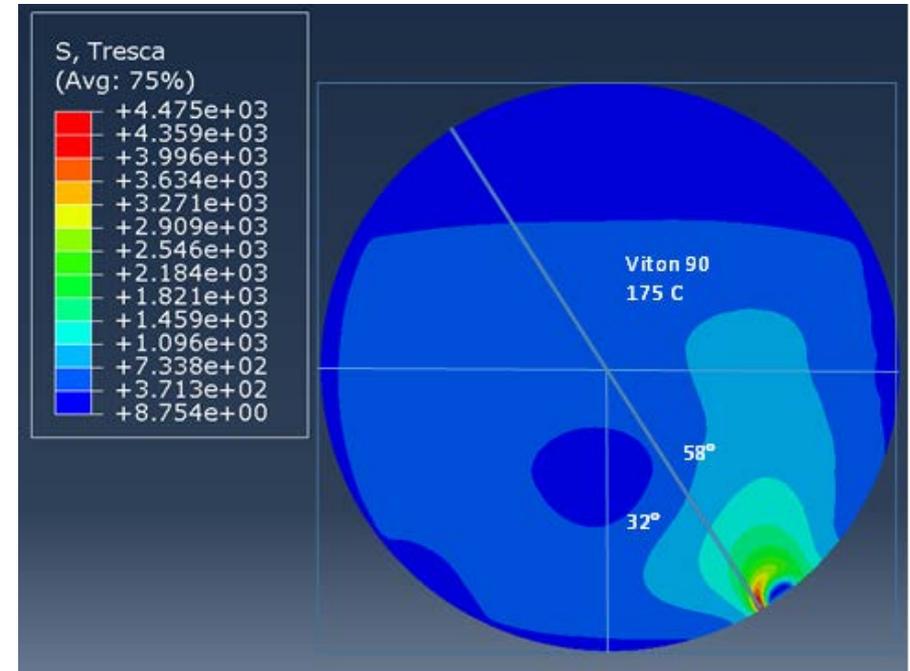


Relaxed back to Un-Deformed State

# Tresca Stress Plotted on O-Ring: FKM-90 @ 175°C

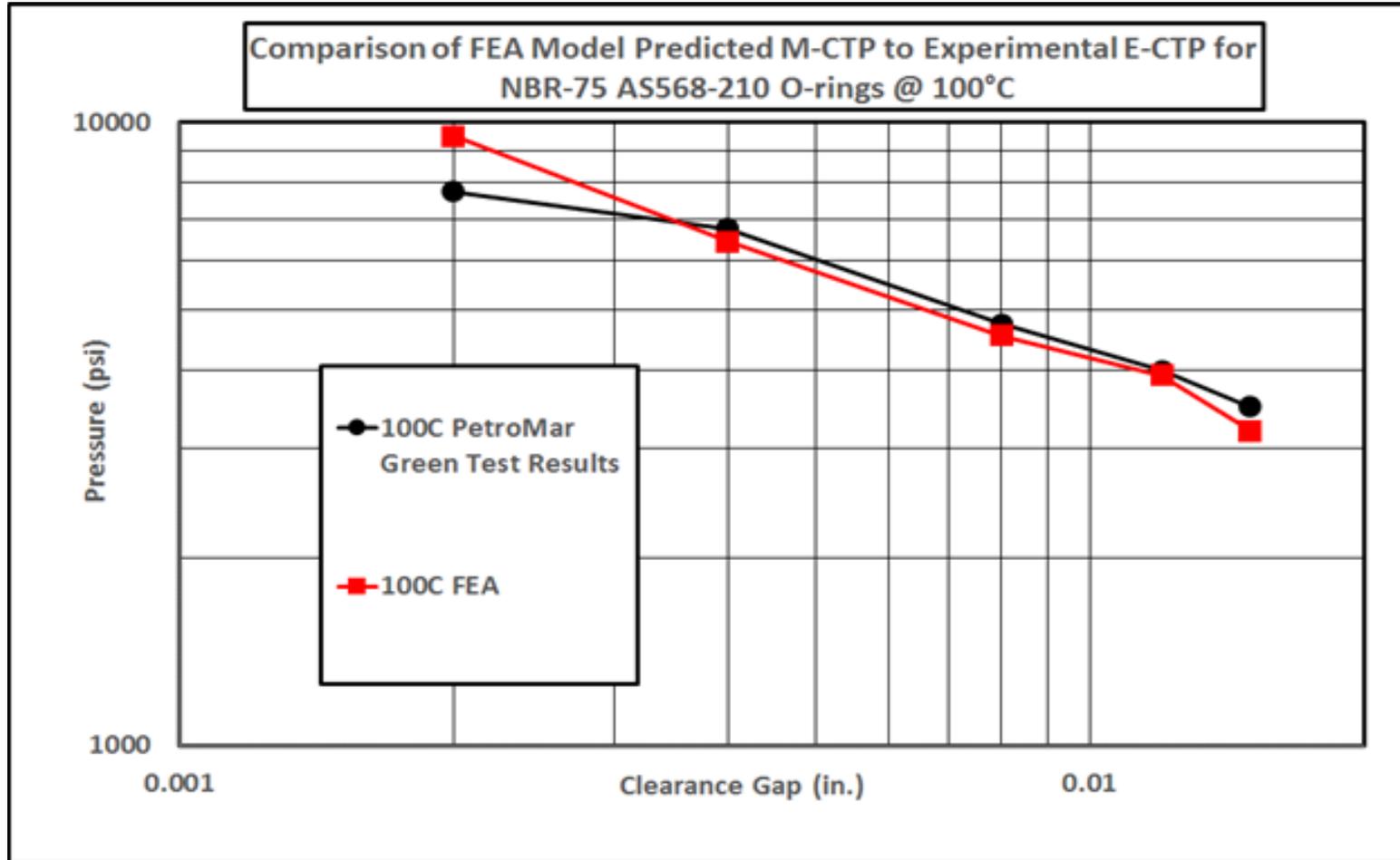


Deformed State @ M-CTP

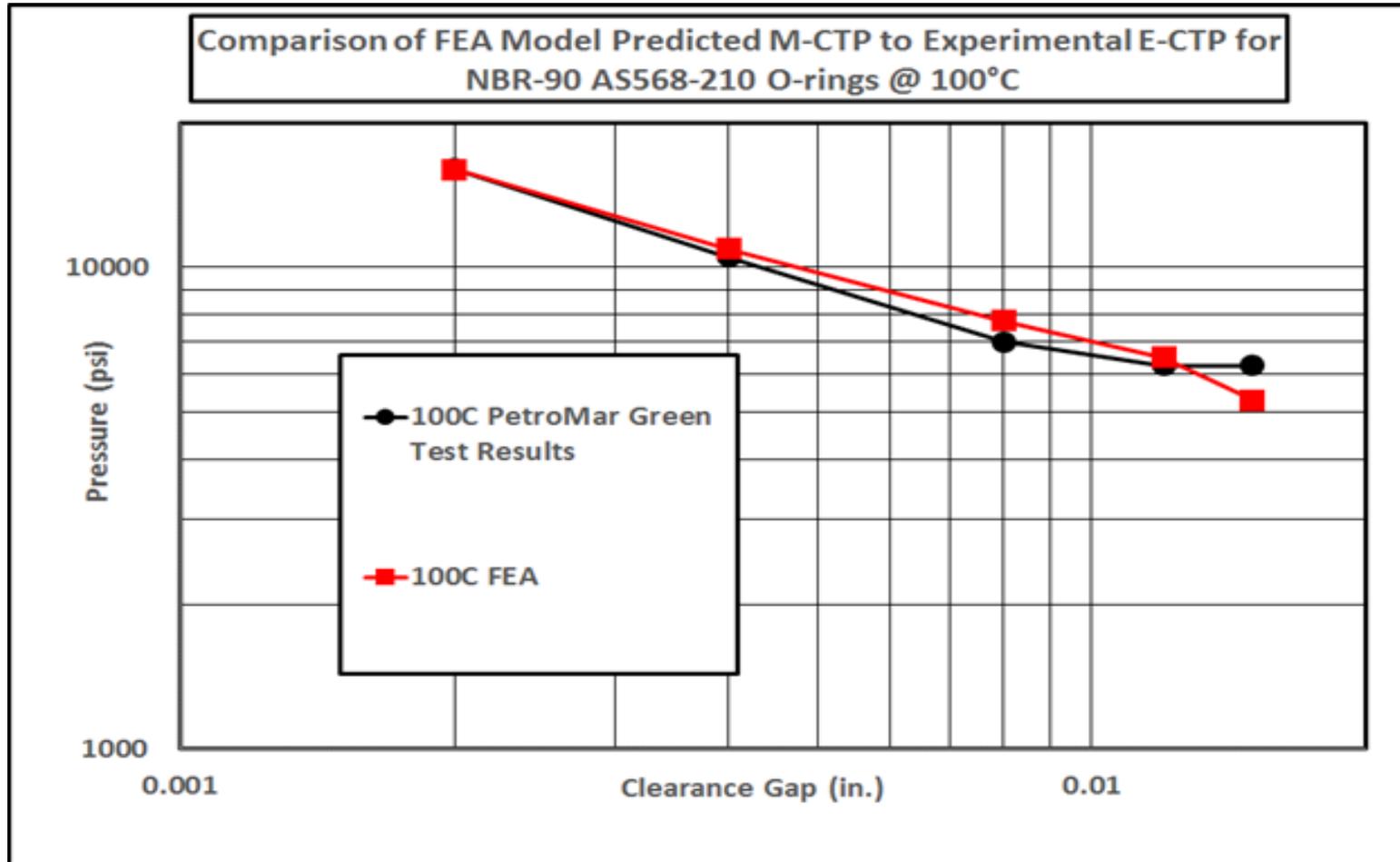


Relaxed back to Un-Deformed State

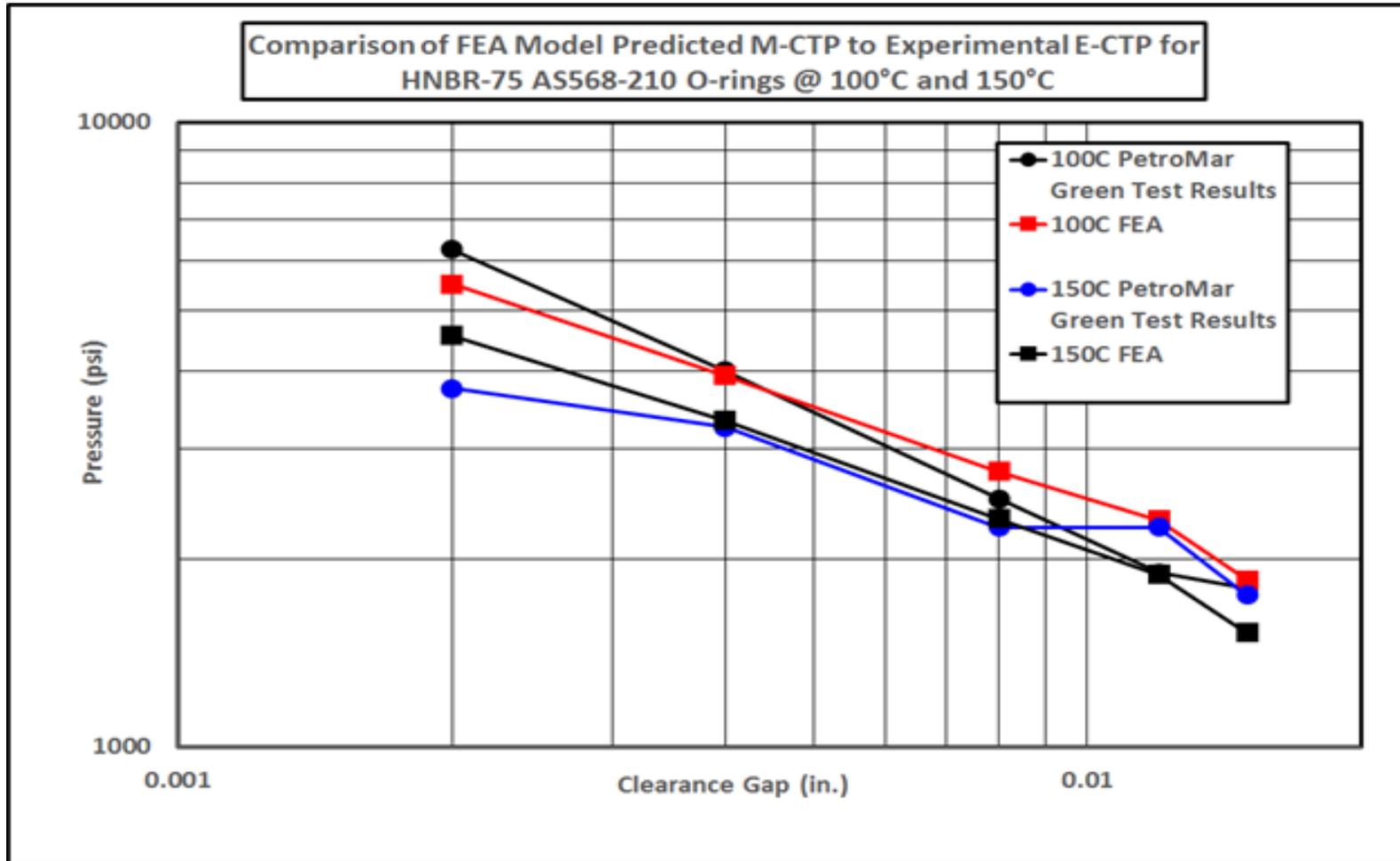
# Comparison of Experimental E-CTP vs. FEA M-CTP for NBR-75 @ 100°C (Max. use Temp. ~125°C)



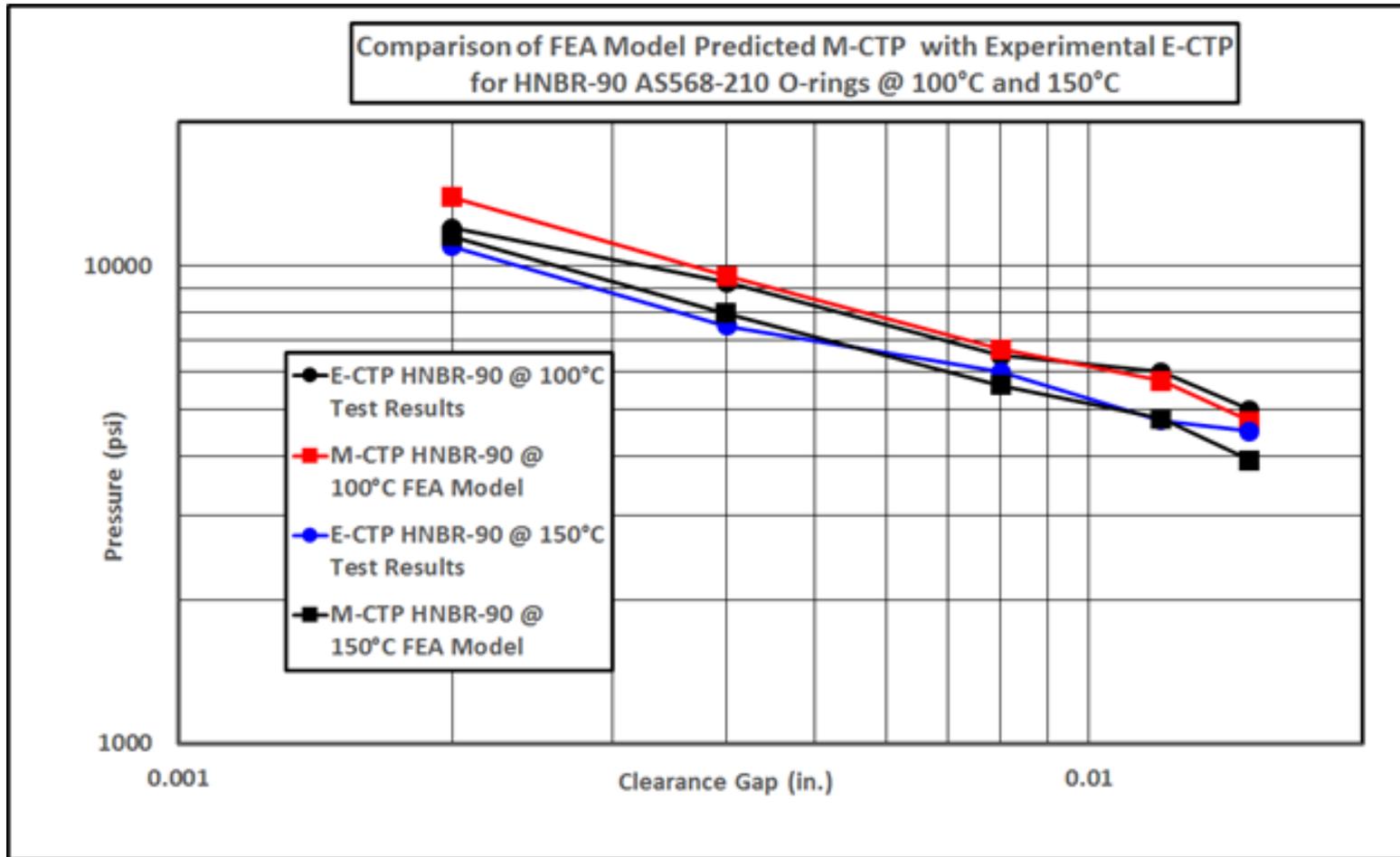
# Comparison of Experimental E-CTP vs. FEA M-CTP for NBR-90 @ 100°C (Max. use Temp. ~125°C)



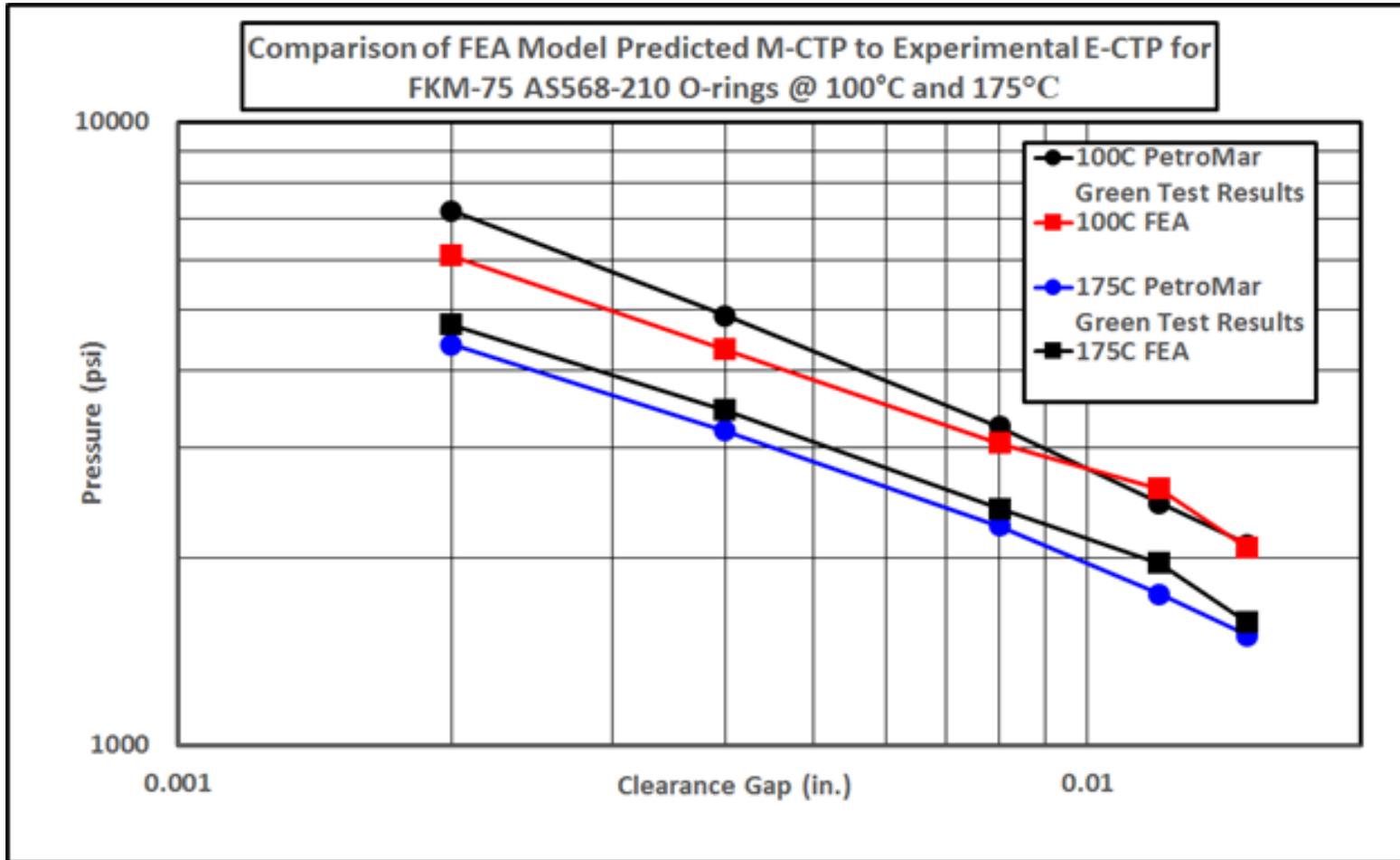
# Comparison of Experimental E-CTP vs. FEA M-CTP for HNBR-75 @ 100°C and 150°C



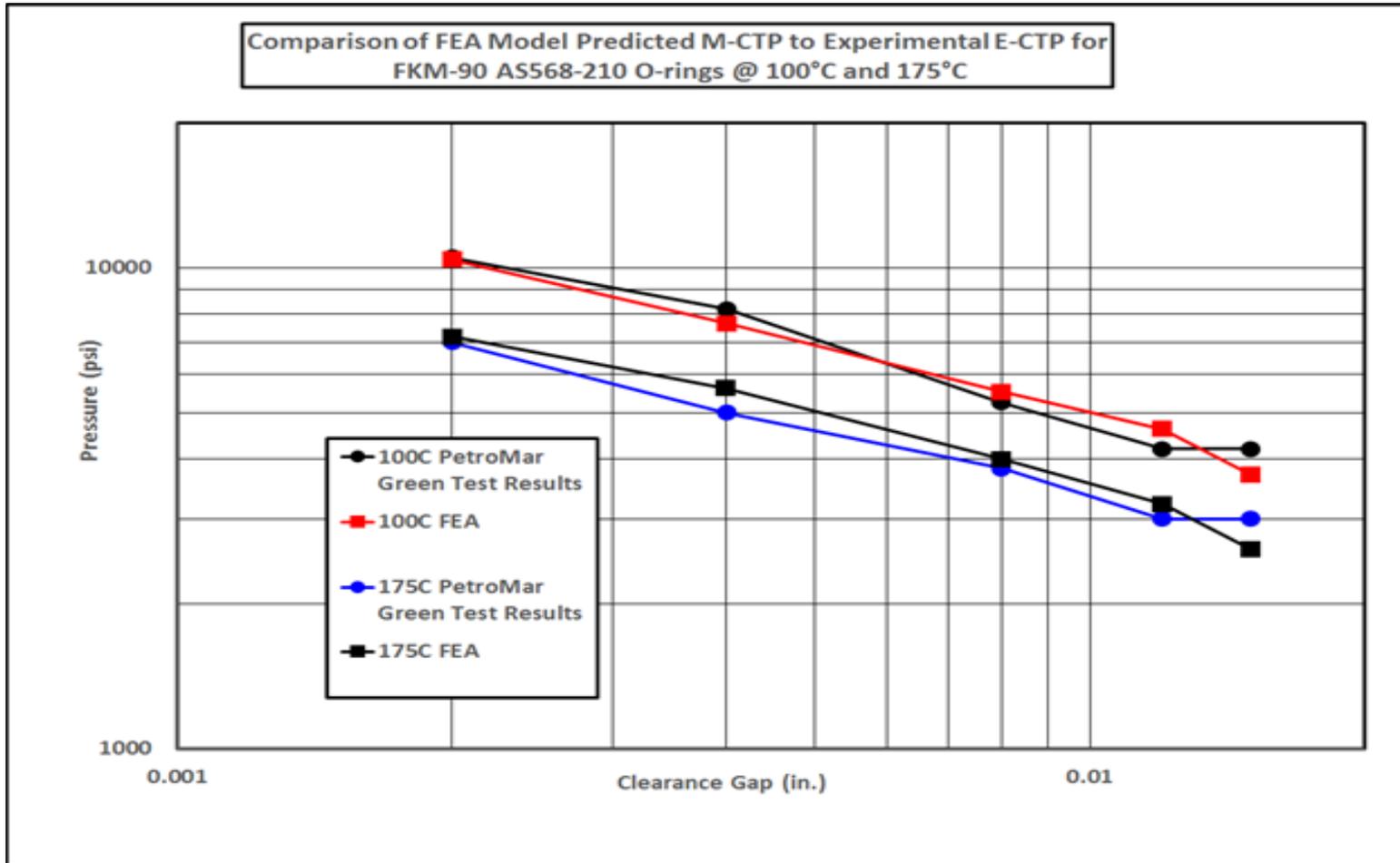
# Comparison of Experimental E-CTP vs. FEA M-CTP for HNBR-90 @ 100°C and 150°C



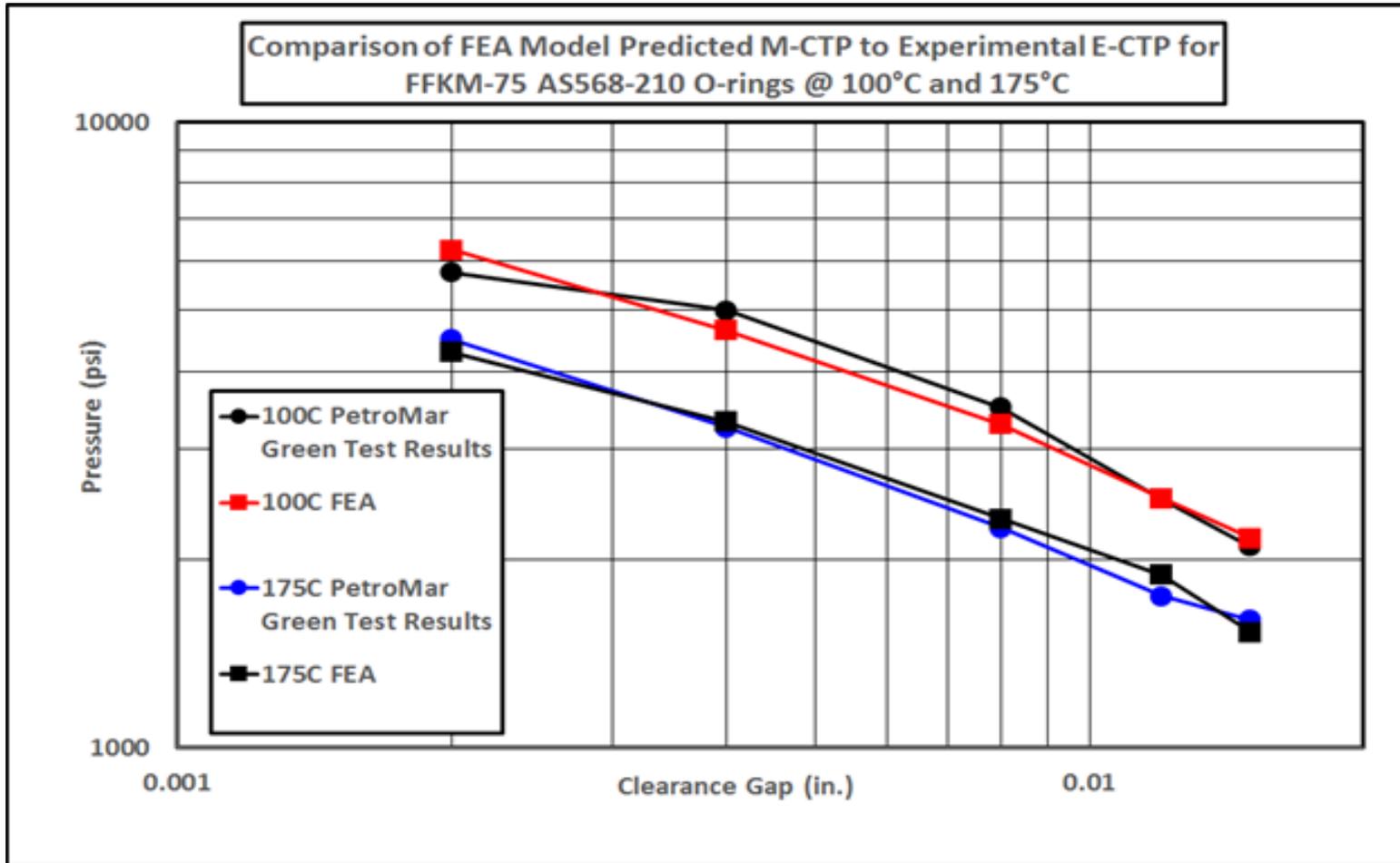
# Comparison of Experimental E-CTP vs. FEA M-CTP for FKM-75 @ 100°C and 175°C



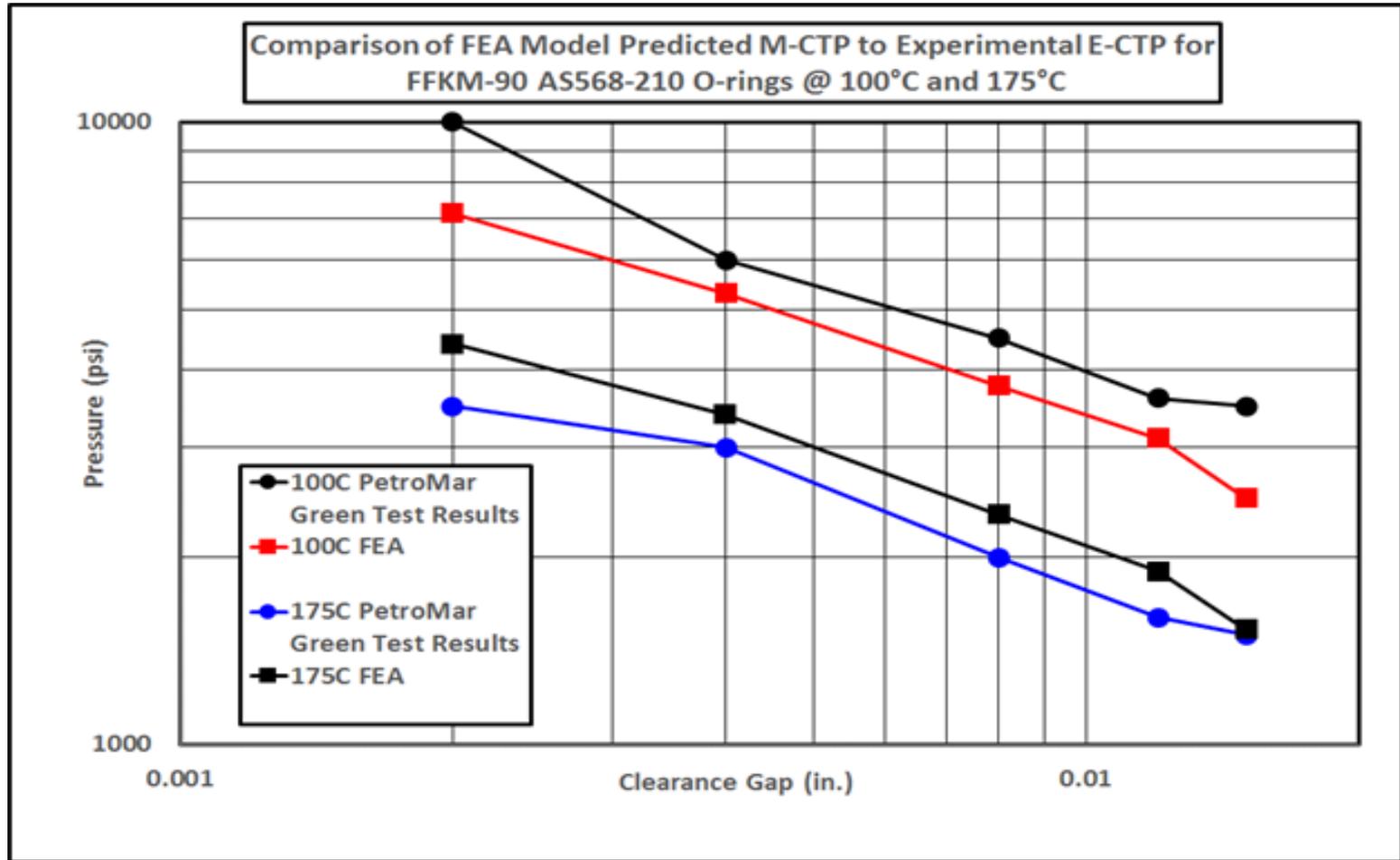
# Comparison of Experimental E-CTP vs. FEA M-CTP for FKM-90 @ 100°C and 175°C



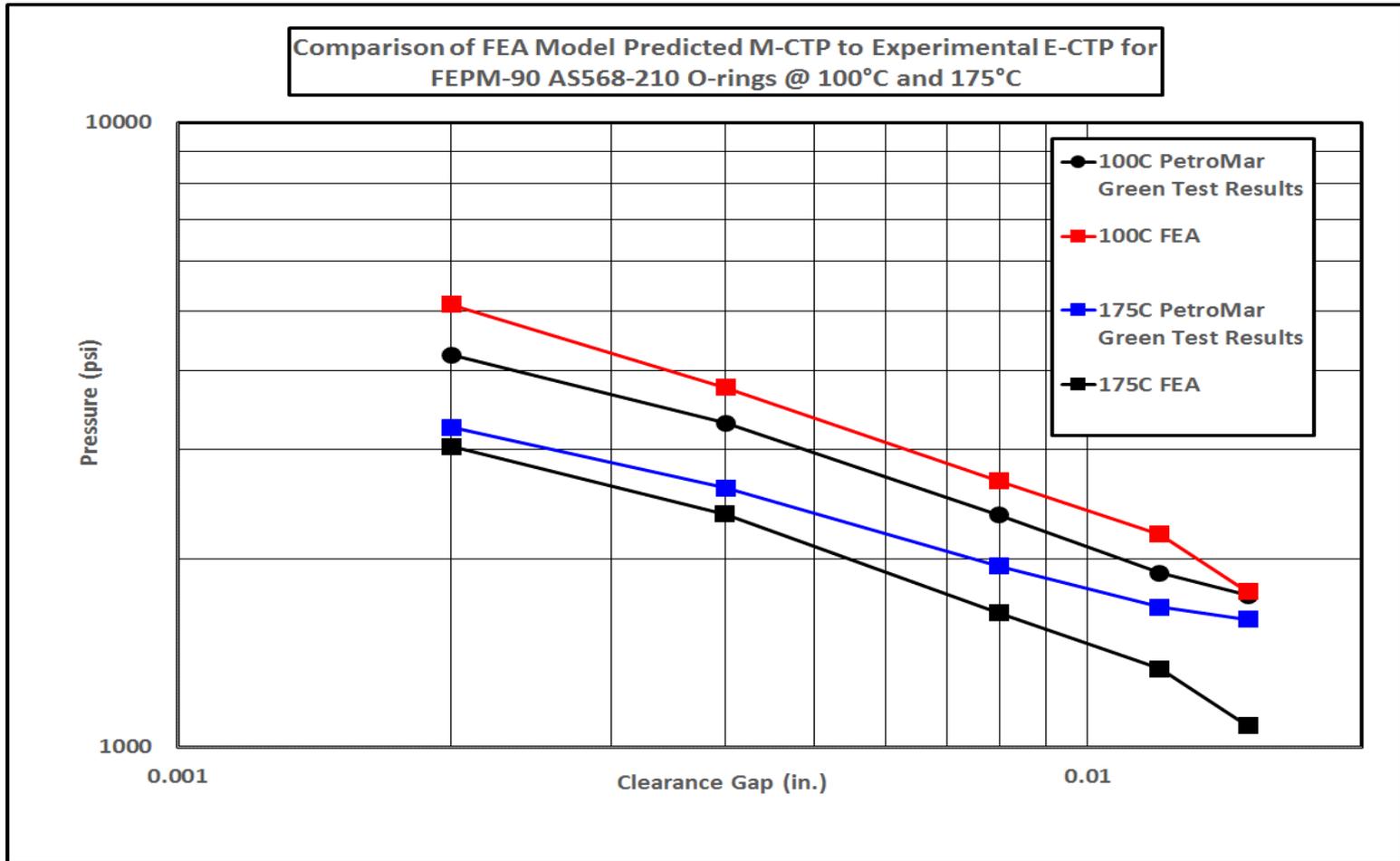
# Comparison of Experimental E-CTP vs. FEA M-CTP for FFKM-75 @ 100°C and 175°C



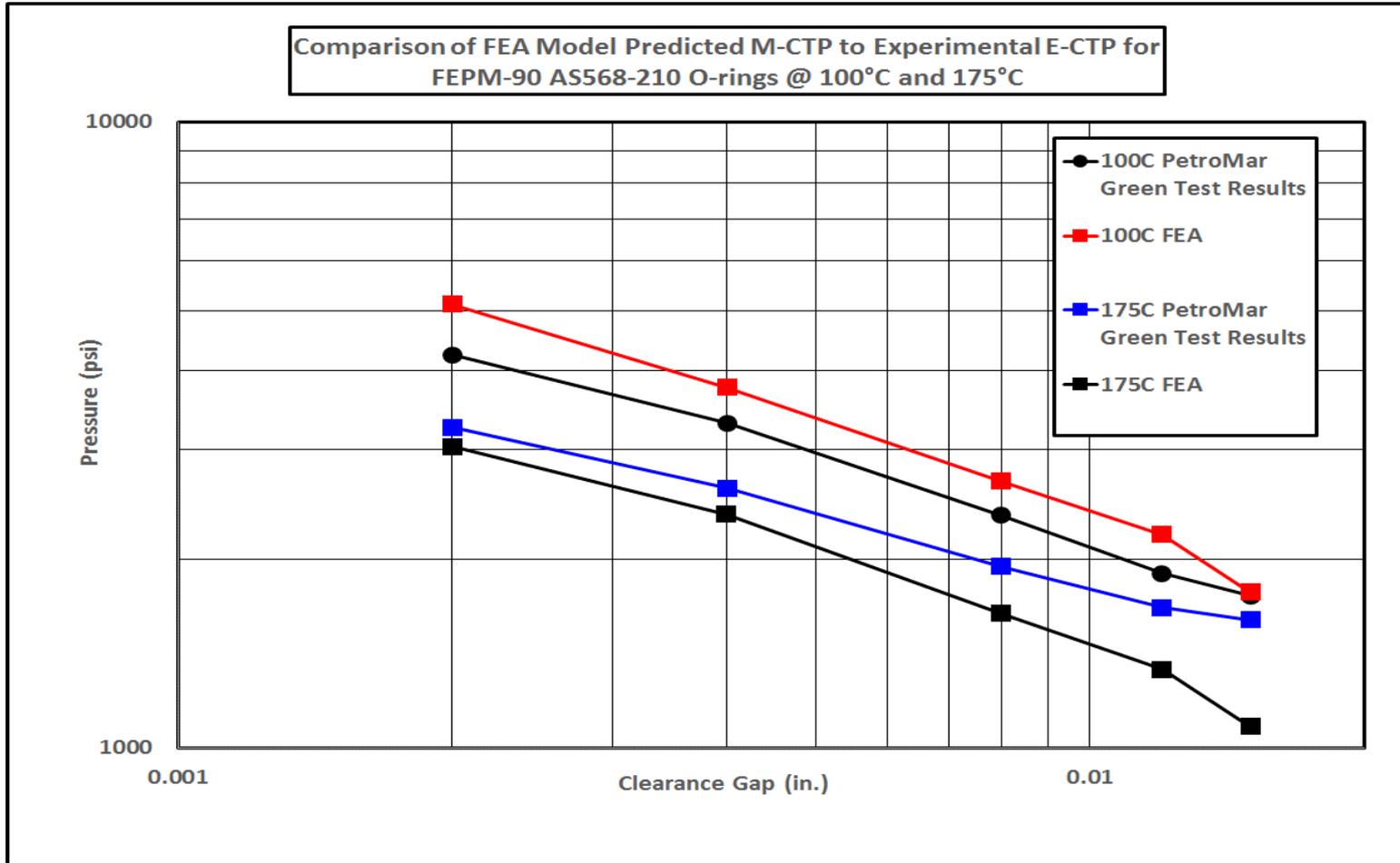
# Comparison of Experimental E-CTP vs. FEA M-CTP for FFKM-90 @ 100°C and 175°C



# Comparison of Experimental E-CTP vs. FEA M-CTP for FEPM-90 @ 100°C and 175°C



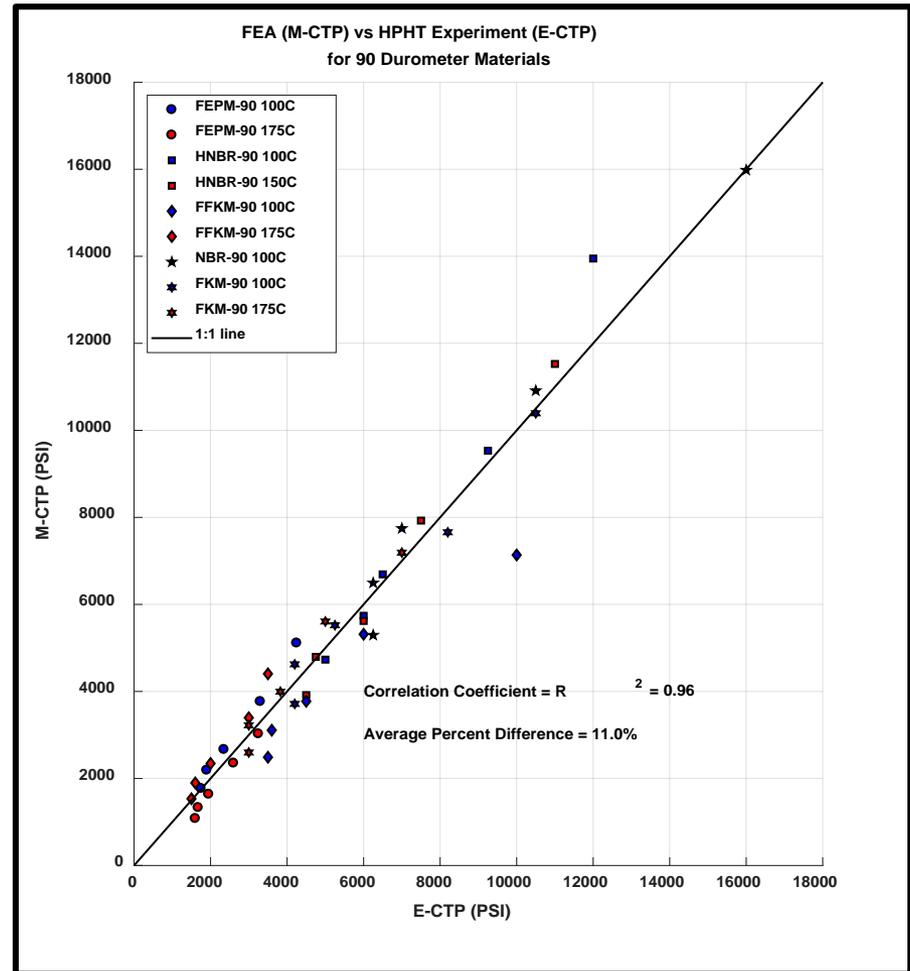
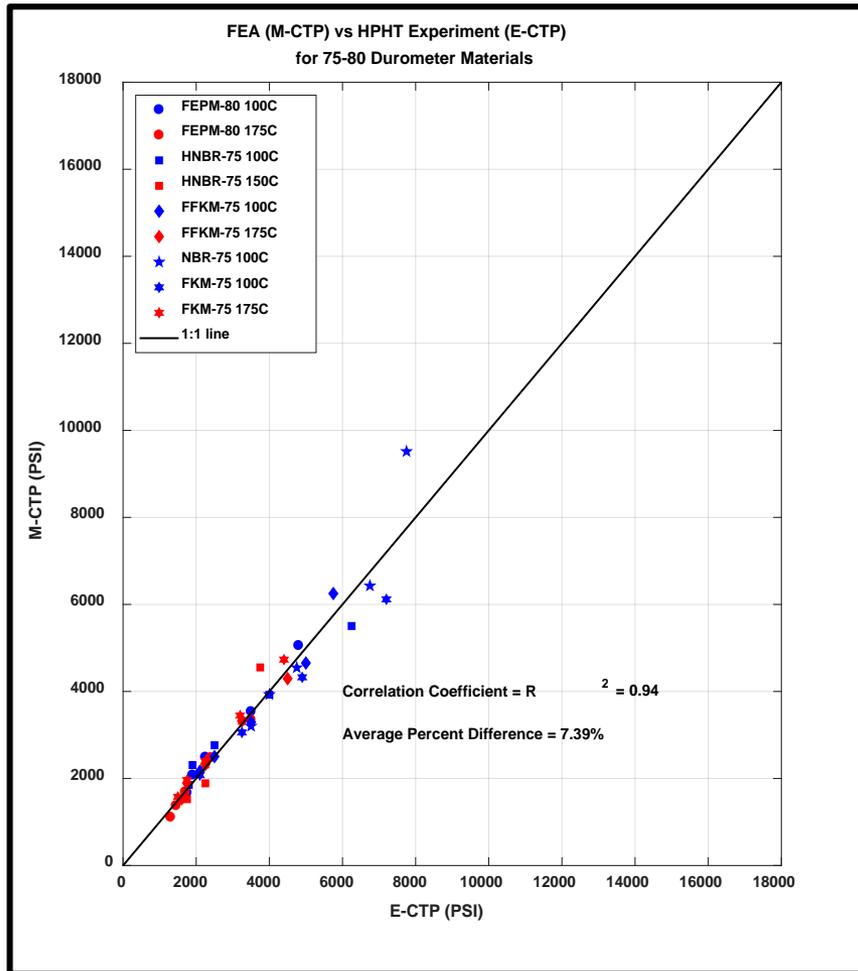
# Comparison of Experimental E-CTP vs. FEA M-CTP for FEPM-90 @ 100°C and 175°C



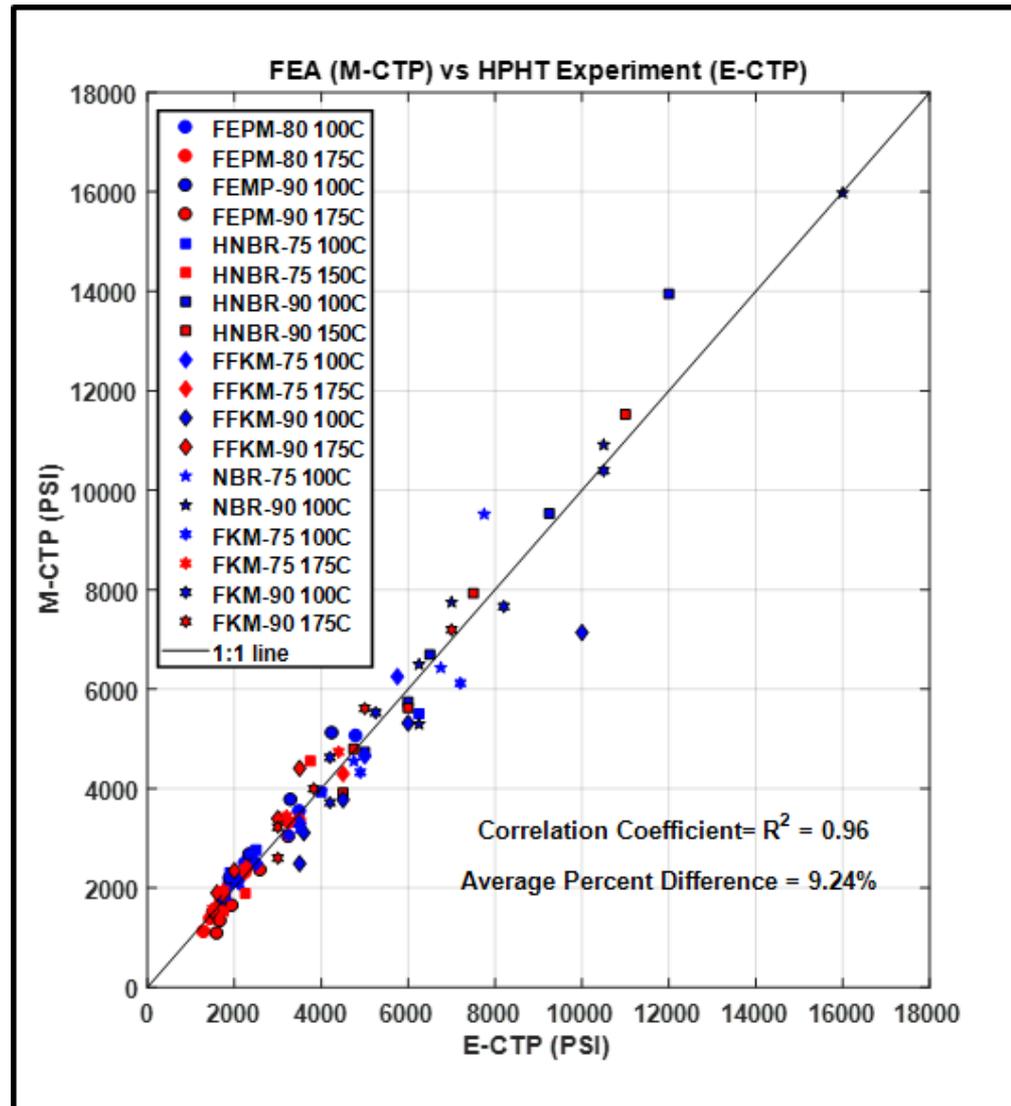
# Summary Table: Comparison of FEA model M-CTP with Experimental E-CTP for All Elastomers

Material	M-CTP at 100°C 0.004" clearance gap	E-CTP at 100°C 0.004" clearance gap	Material	M-CTP at 175°C 0.004" clearance gap	E-CTP at 175°C 0.004" clearance gap
<b>NBR 90</b>	10912	10500	<b>HNBR-90 at 150°C</b>	7926	7500
<b>HNBR-90</b>	9521	9250			
<b>FKM-90</b>	7658	8200	<b>FKM-90</b>	5610	5000
<b>NBR-75</b>	6428	6750	<b>FKM-75</b>	3447	3200
<b>FFKM-90</b>	5315	6000	<b>FFKM-90</b>	3394	3000
<b>FFKM-75</b>	4653	5000	<b>FFKM-75</b>	3325	3250
<b>FKM-75</b>	4325	4900	<b>HNBR-75 at 150°C</b>	3323	3250
<b>HNBR-75</b>	3927	4000			
<b>FEPM-90</b>	3769	3500	<b>FEPM-80</b>	2481	2400
<b>FEPM-80</b>	3540	3300	<b>FEPM-90</b>	2356	2600

# Critical Tearing Pressure: Correlation between FEA (M-CTP) vs. Experiment (E-CTP)



# Critical Tearing Pressure: Correlation between FEA (M-CTP) vs. Experiment (E-CTP)



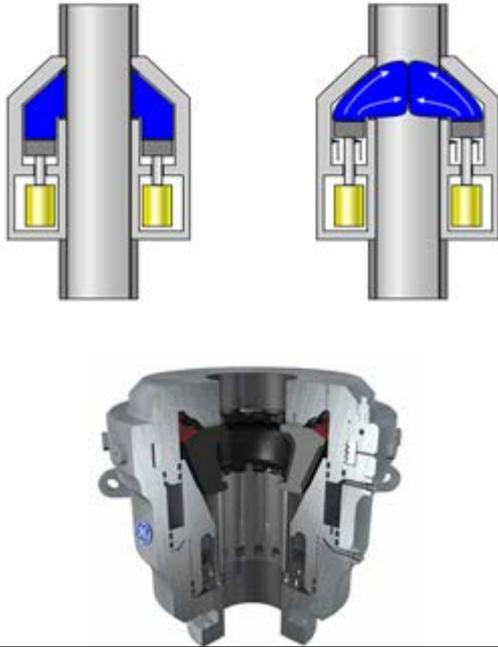
# FEA Demo

## Conclusion: Key Findings

1. New or revised guidance, followed by formal industry standards, are needed to ensure safe operation in HPHT environments (over 15,000 psi and 175°C) since current industry standards only provide guidance for elastomer use at pressures up to 5,000 psi.
2. The dominant failure mechanism in HPHT elastomer testing and FEA model development was crack tear propagation via extrusion-initiated spiral failure, which could guide development of new crack resistant materials.
3. A FEA-based computer model was developed to predict the onset of tearing and failure of elastomer O-rings under HPHT. The FEA (M-CTP) model was parameterized with a comprehensive set of elastomer mechanical property data at multiple temperatures, including multi-axial, compression, hyper-elastic, crack-initiation and creep-crack-growth tests. The model was successfully validated by comparing the predicted results with the experimental (E-CTP) from a HPHT test cell.

## Conclusion: Key Findings – cont.

- The FEA model can be used in the design and evaluation of elastomer seals in downhole tools and well control applications such as Blow-out Preventers (BOP).



Annular Blow-out Preventer Uses Flow Extrusion of Elastomer for Well-Control (7" GK\* BOP for 15,000-20,000 psi shown)

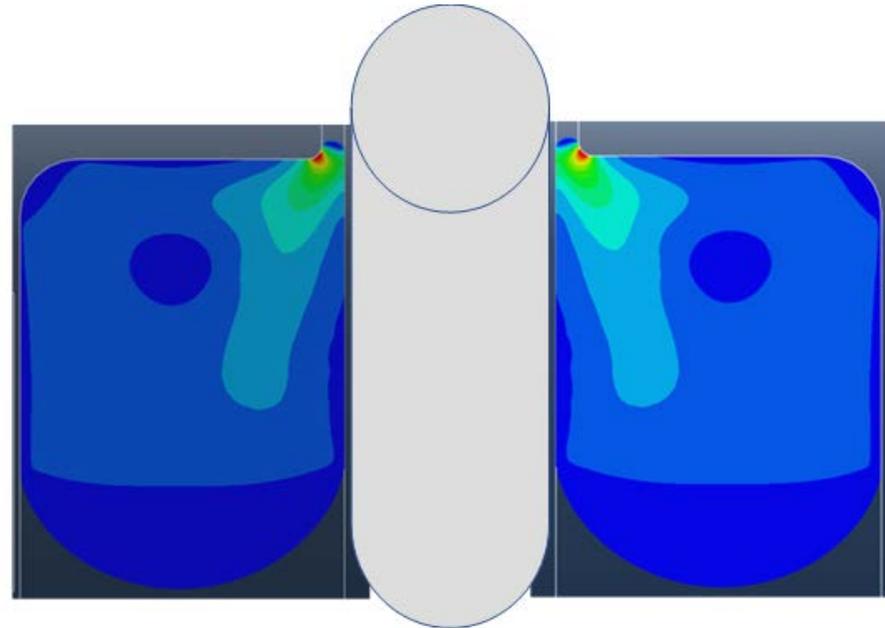


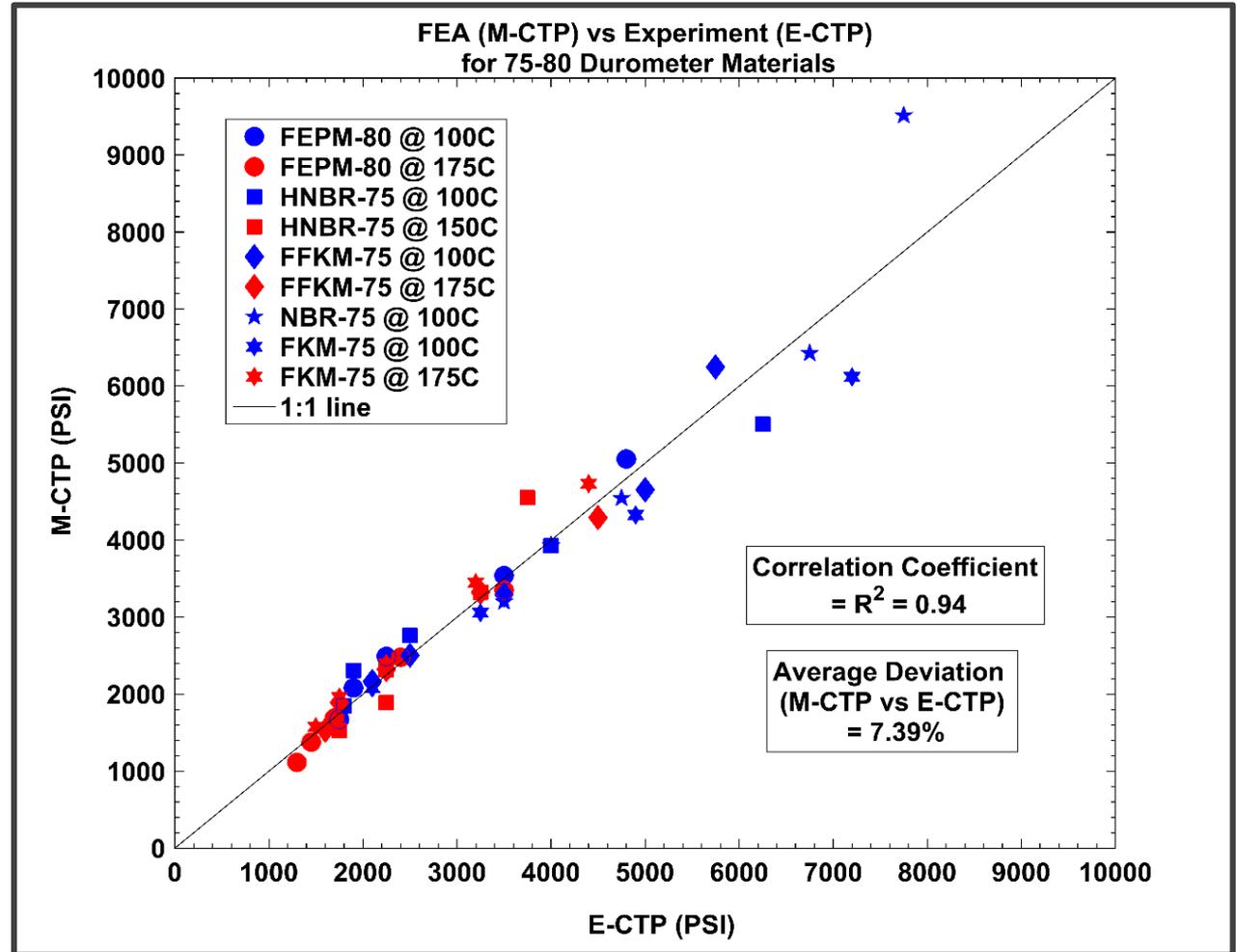
Illustration of Scaled-up FEA analysis of BOP Elastomer Seal using M-CTP model Approach (FKM-90 @ 175°C shown)

Battelle emphasizes that any large-scale extension of the FEA model that was developed and validated on AS568-210 size O-ring seal fixtures should be revalidated on larger scale devices before implementation.

# Conclusion: Key Findings – cont.

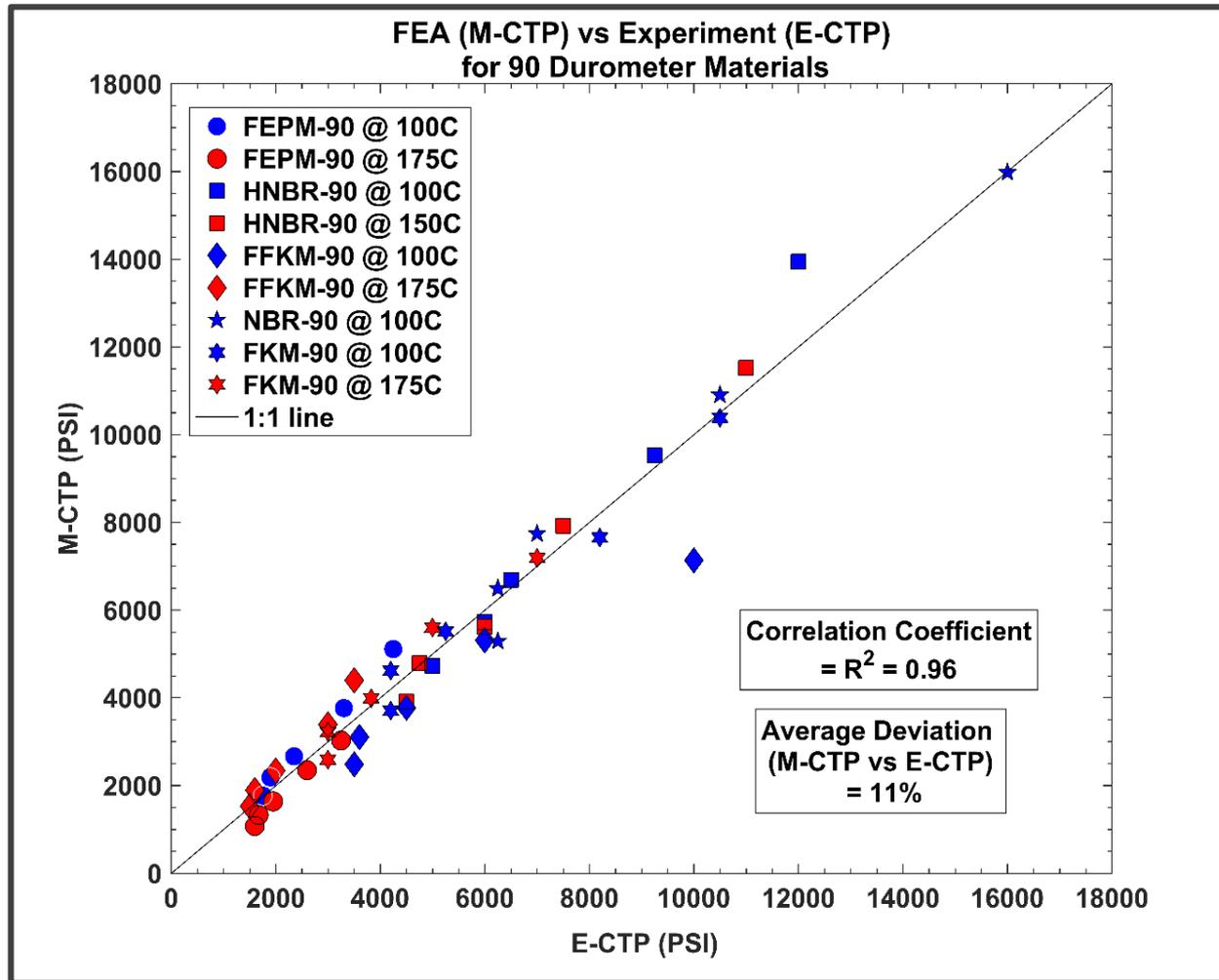
- The high correlation between model and experiment for all O-ring materials, clearance gaps, and temperatures provides users with high confidence in the model accuracy.

The FEA (M-CTP) model was parameterized with elastomer property data at multiple temperatures and confirmed through experimental data.



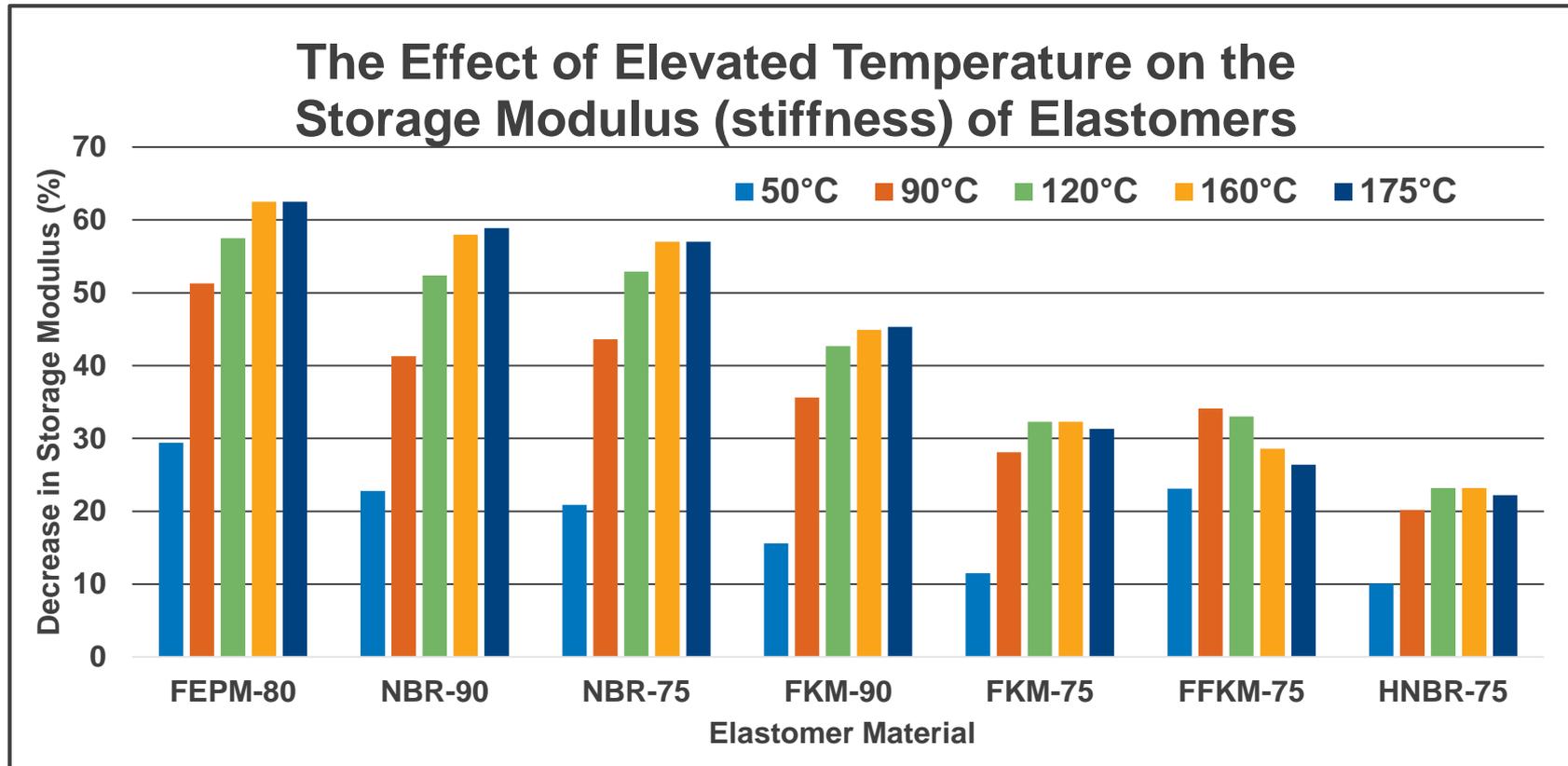
## Conclusion: Key Findings – cont.

- High correlation between FEA model (M-CTP) and experimental (E-CTP) was also achieved for harder 90 Shore A durometer materials.



## Conclusion: Key Findings – cont.

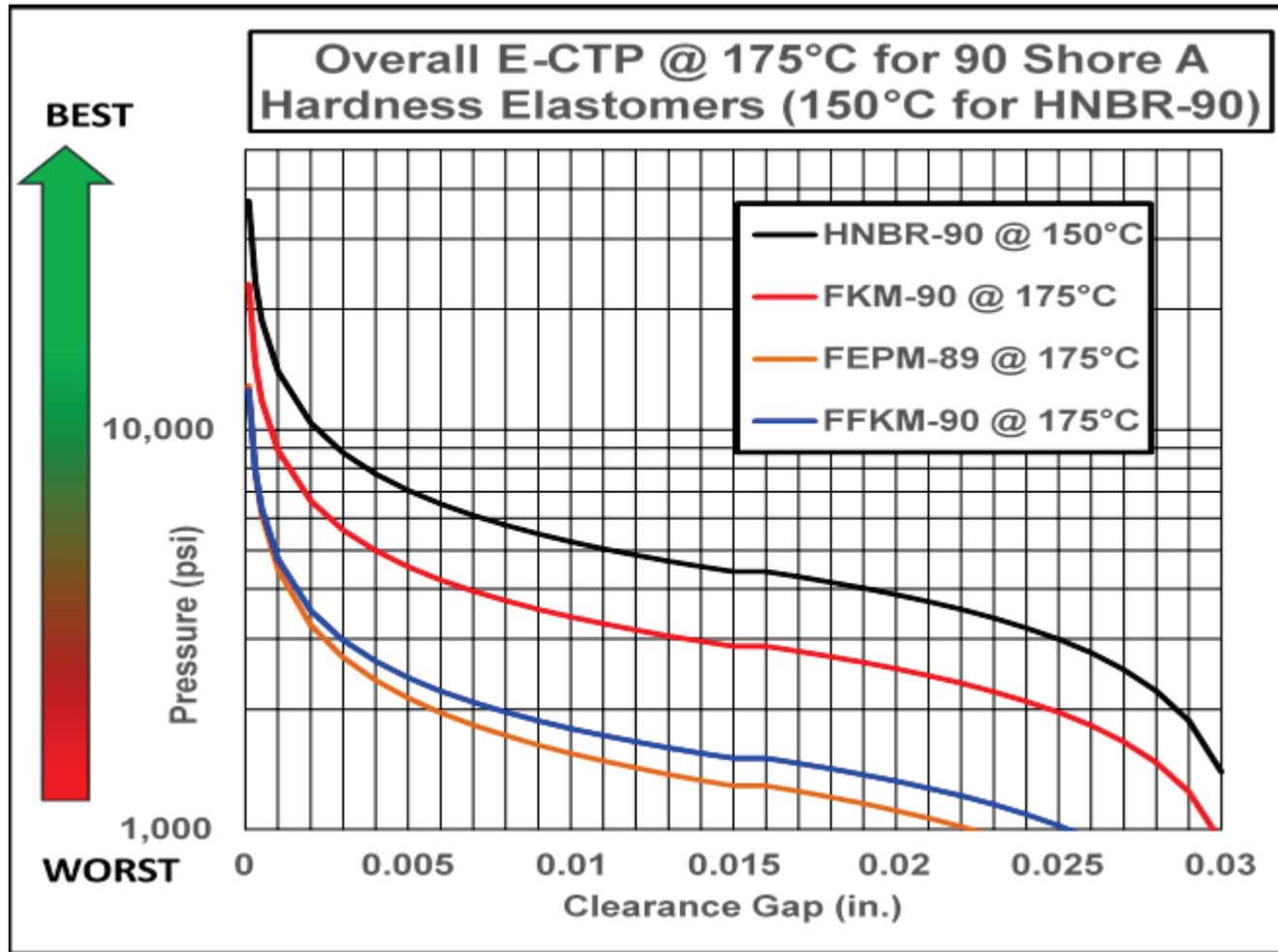
- Most elastomers have much lower mechanical properties (e.g. tensile modulus, tensile strength and elongation at break) at temperatures above 90°C.



Industry should validate the test data on larger scale devices before implementation.

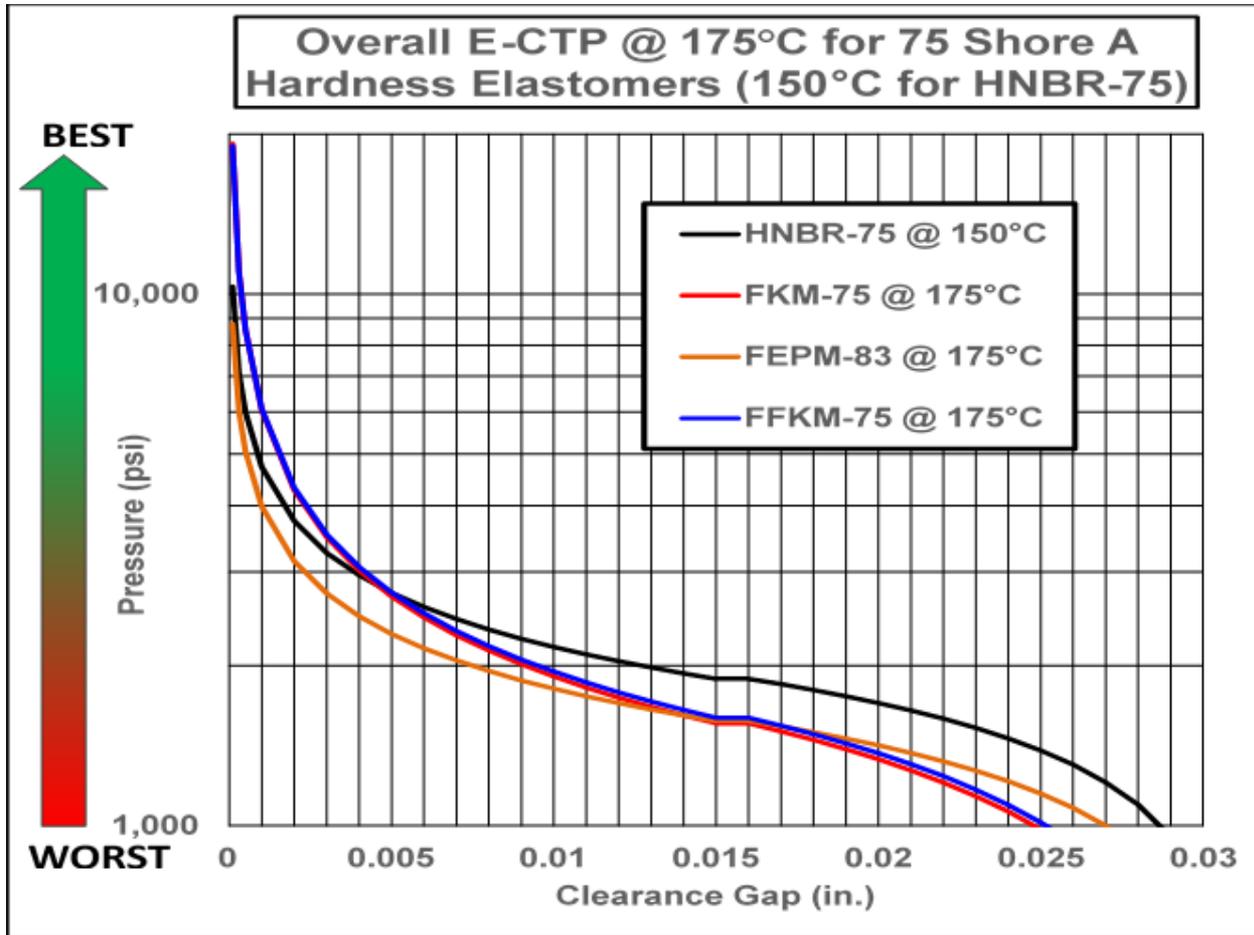
## Conclusion: Key Findings – cont.

- For 90 Shore A Hardness Elastomers: HNBR-90 and FKM-90 are superior to FFKM-90 and FEPM-90 at 150°C to 175°C across all clearance gaps.



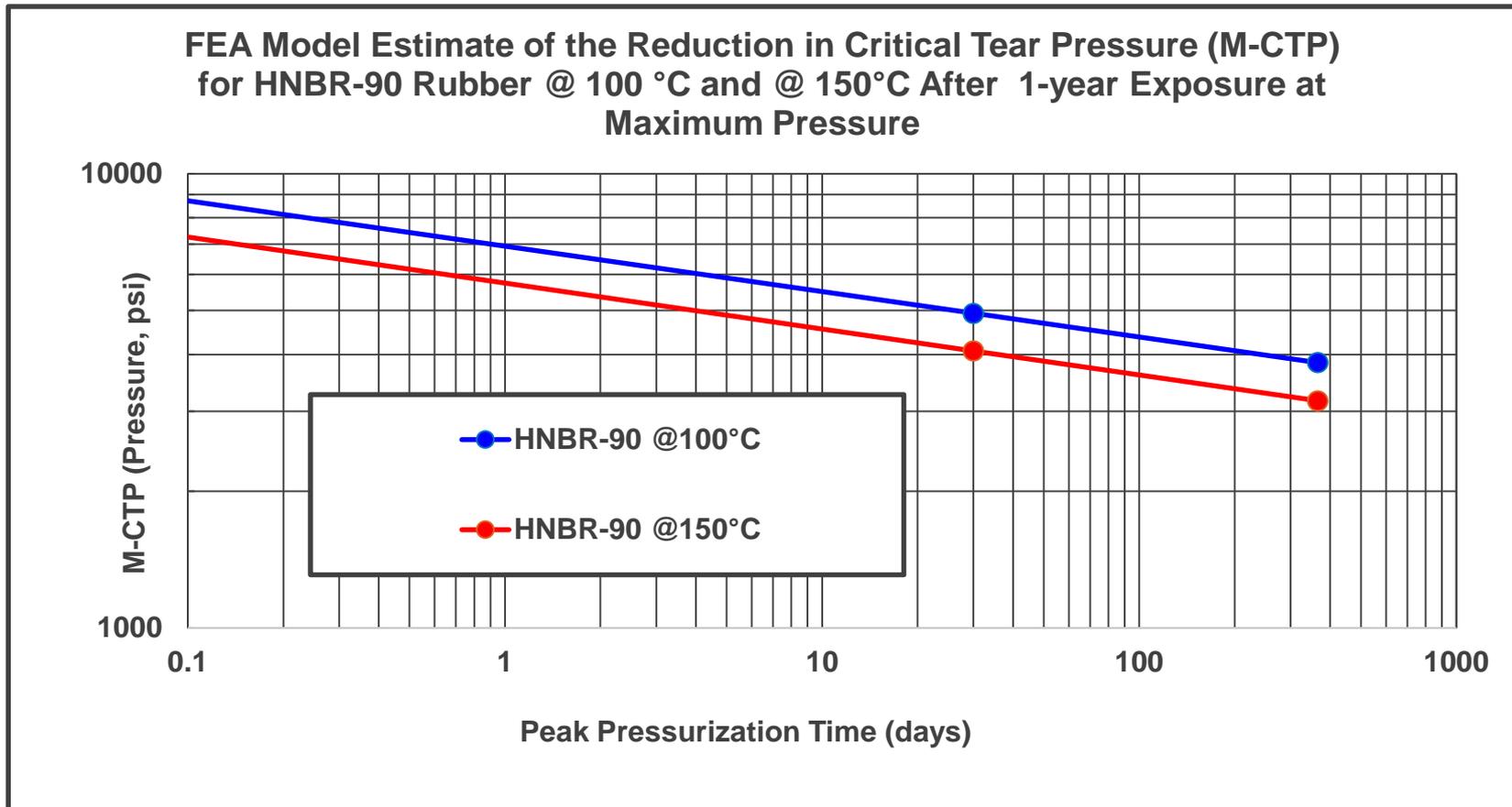
## Conclusion: Key Findings – cont.

9. Lower Hardness Elastomers (~75-83 Shore A) have similar E-CTP at 150°C to 175°C across all clearance gaps, much lower than HNBR-90 and FKM-90, but similar to FFKM-90, FEPM-90.



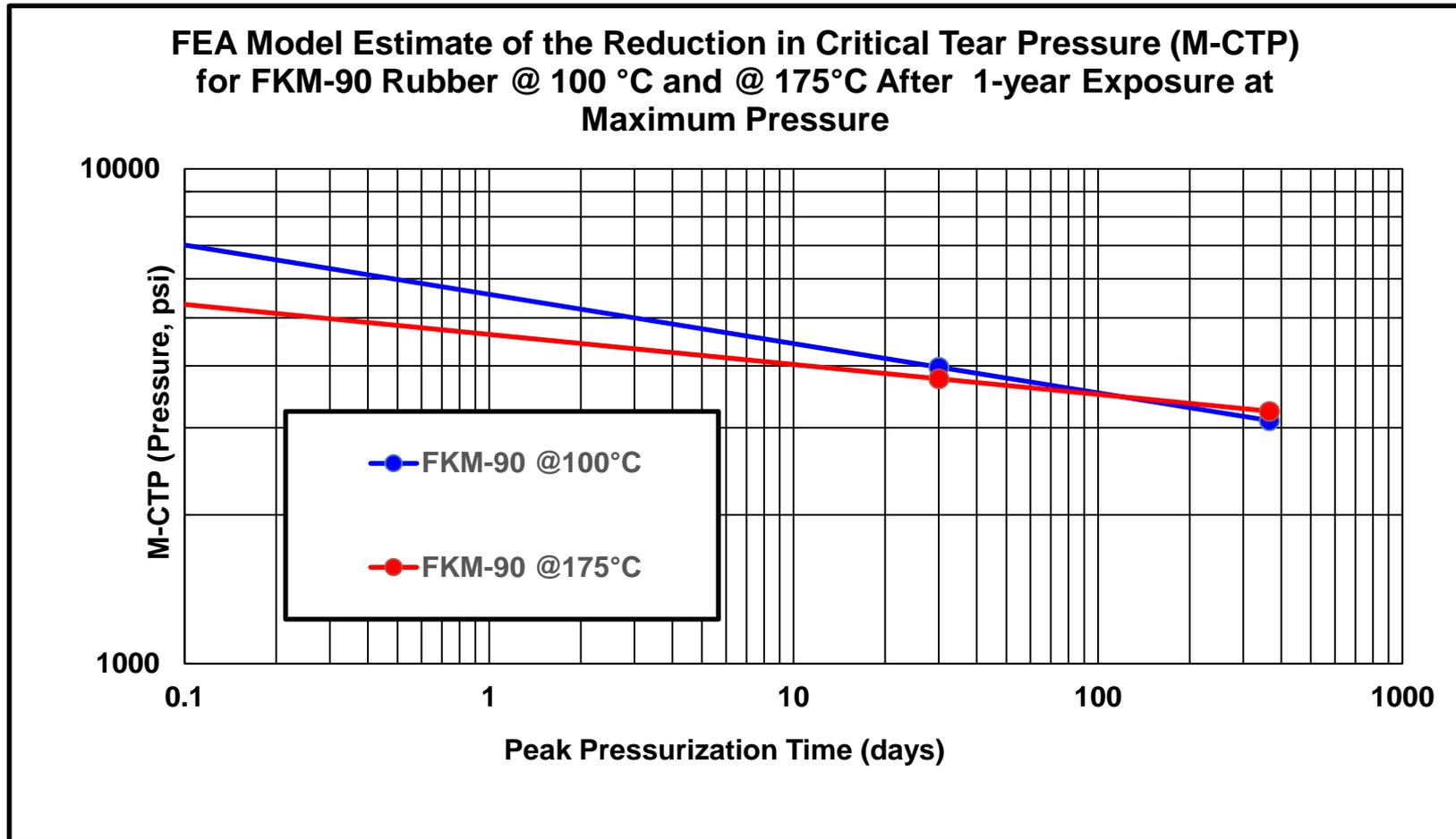
## Conclusion: Key Findings – cont.

10. Model accurately predicts the M-CTP and Power-law predictions of the change in M-CTP over time indicate approximately 50% reduction after 1 year.



## Conclusion: Key Findings – cont.

11. FEA model estimate of aging behavior can be improved by validation of creep crack growth at several time points after tearing begins.



## Conclusion: Recommendations

1. O-ring seal failures can be caused by other stresses, encountered under HPHT conditions, including chemical environments ( $H_2S$ ,  $CO_2$ , hydrocarbon liquid and vapor). Additional testing and model development are recommended for these conditions.
2. The testing in this program was done at the O-ring level and should be expanded to component and device level, i.e. BOPs, SSVs, packers, etc.
3. Future studies can include extending the FEA model and HPHT testing to include aging effects in corrosive and non-corrosive environments as well as extended lifecycle testing (cyclic pressurization and associated crack growth) under these conditions.
4. Future development efforts should expand the FEA model to include longer term (minimum several weeks) creep crack growth of elastomers in combination with experimental validation.

***BATTELLE***

**It can be done**