Fatigue Testing of a New Generation of Commercial Scale Ultra-low Inclusion NiTi Alloy

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saes group

memry a SAES Group company™

nitinol +™

Enduro

Objectives

1. Characterize Nitinol wire material produced from SAES Smart Material (SSM) ultra-low inclusion Enduro ingot and benchmark with SSM's standard ingot
2. Demonstrate improved fatigue performance of the new Enduro Nitinol alloy

Agenda

- Background on Gen III Nitinol and Fatigue
- Experimental Sample Prep.
- Material Characterization
- RBT Fatigue Testing and Results
- Z-Specimen Testing and Results
- Summary

Enduro

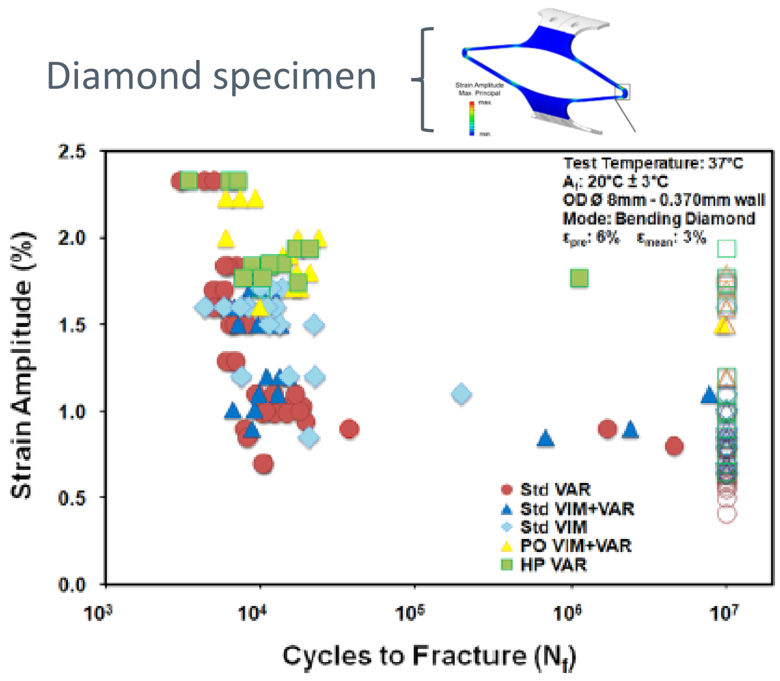
A new nitinol material, engineered to go the distance in ultra-demanding applications.

Background

Why low-inclusion Nitinol?

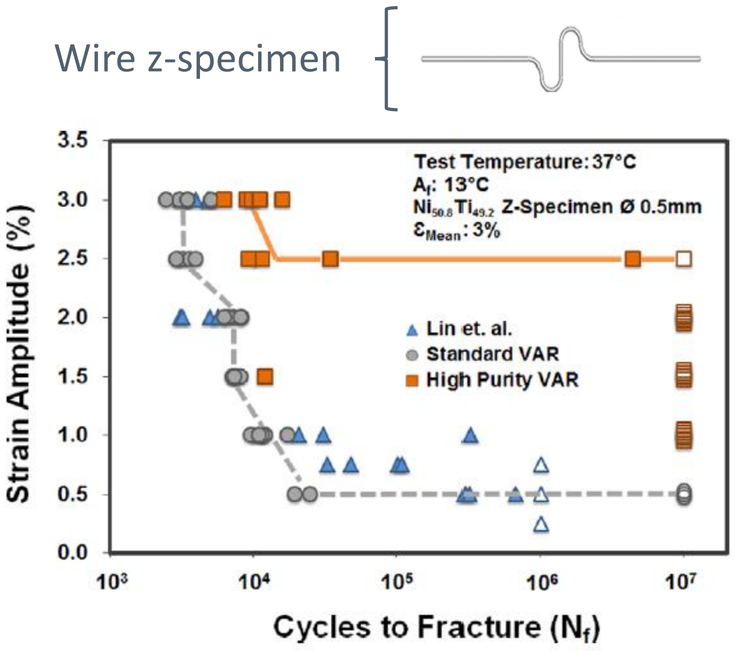
- Extensive studies conducted on the significant impact of inclusion size and density on fatigue
- Ultra-clean Nitinol materials becoming a requirement for cardiovascular and neurovascular implant applications that demand extreme durability

Diamond specimen



[S.W. Robertson et al., JMBBM, 51 (2015), p 119-131]

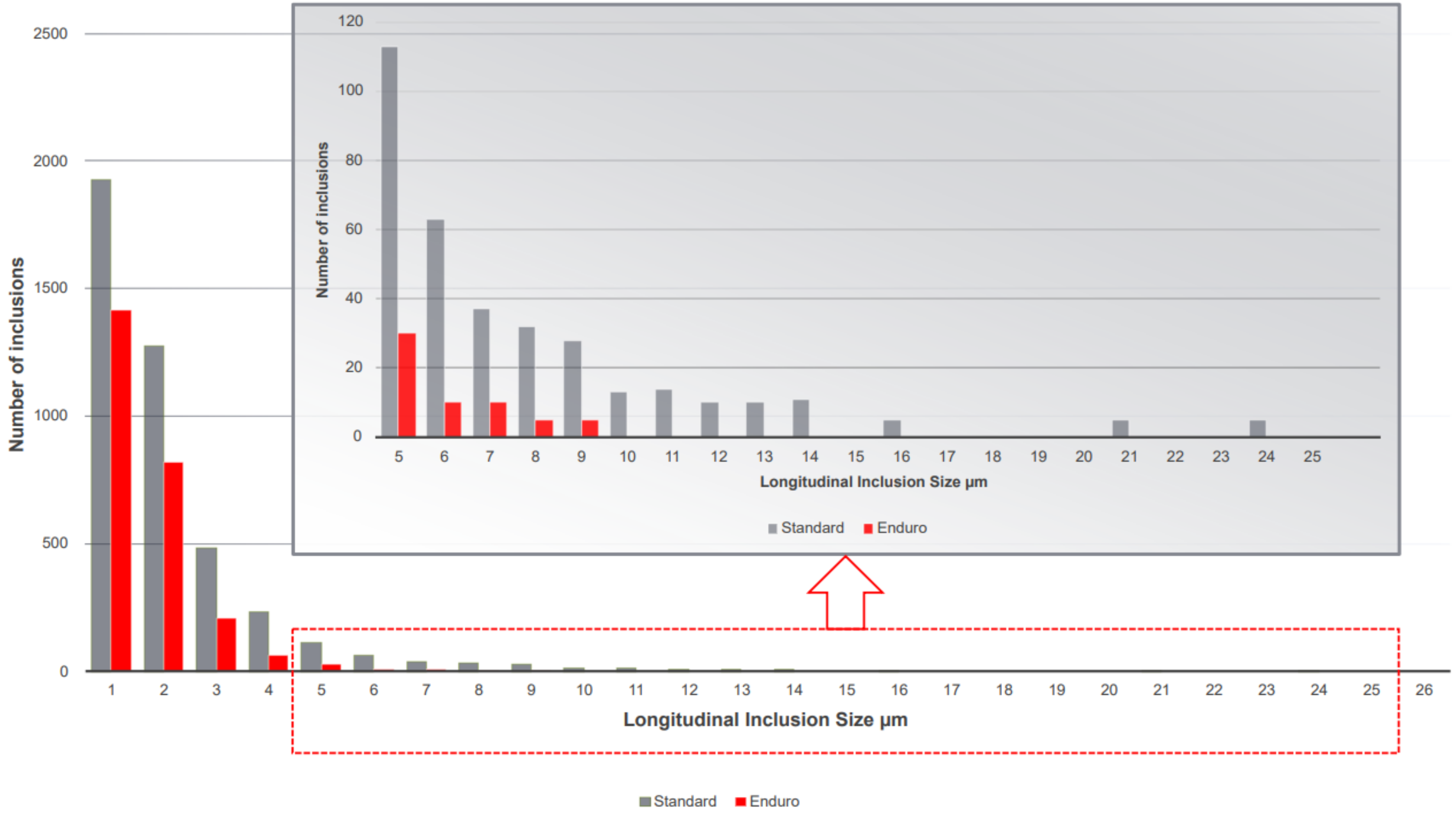
Wire z-specimen



[M. Launey et al., JMBBM, 34 (2014), p 181-186]

The Enduro Nitinol Alloy

- Engineered to reduce inclusion size and density through newly developed proprietary vacuum melting and optimized conversion processes
- ASTM F2063-18 compliant Nitinol ingot
- Available in all Nitinol product forms



The Enduro Nitinol Alloy

Robustness Testing / Specification Development

- Comprehensive robustness campaign executed to validate processes & develop inclusion specs.
  - 18 ingots produced in three separate melting campaigns of six ingots each
  - Total of 486 fields of view analyzed at 500x magnification from 6 mm coil and 25 mm bar
- Effectively eliminated non-metallic inclusions with length > 12 µm and greatly reduced inclusion density of particles over 1 µm
  - Density of >5 µm inclusions reduced 10x from standard VIM+VAR alloy!

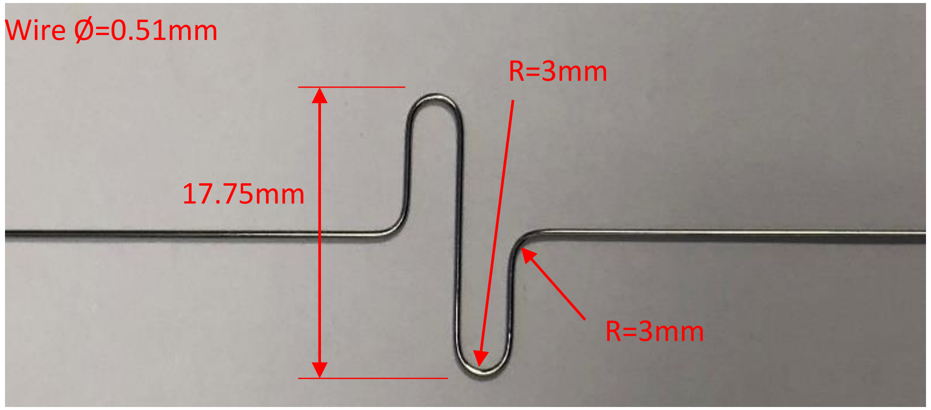
SAES Smart Materials (SSM) Alloy Inclusion Specifications

	Max Inclusion Size [µm]	Max Inclusion Area [%]
ASTM F2063-18 Requirement	39.0	2.8
SSM Standard	26.0	2.0
SSM Enduro	**12.0**	**0.5**

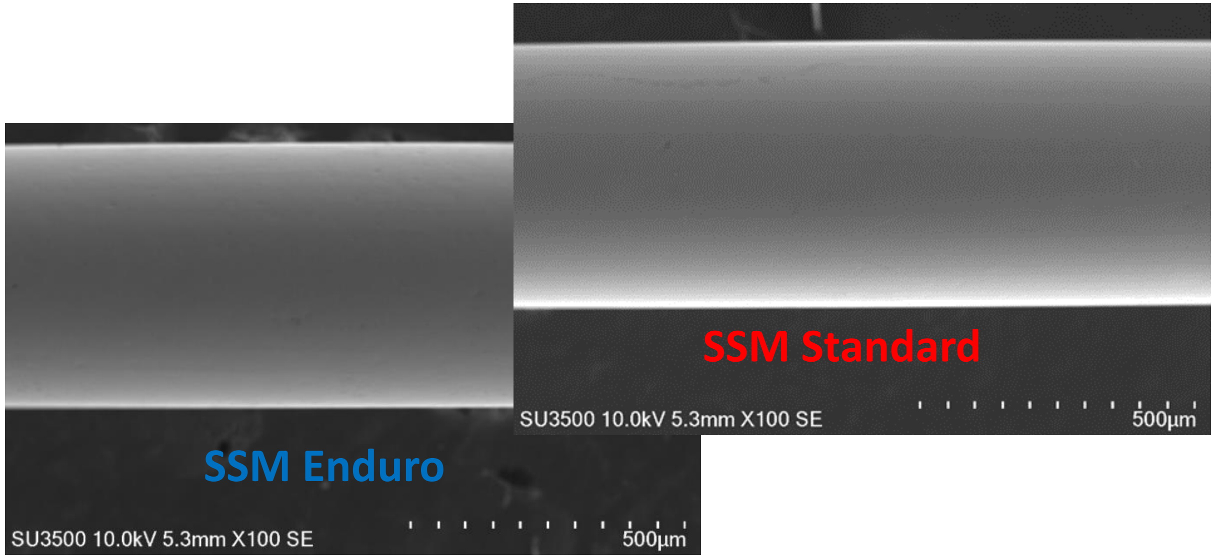
Experimental

Fatigue Sample Preparation (RBT & Z-Specimen)

- SSM standard and Enduro, A_s -15±10°C, ingots used in study
- 0.53mm (0.021”) wire drawn at Memry *using identical draw schedules*
- Shape set heat treatment → 2.5 min. @ 525°C in salt pot
- Surface Finish → Electropolished to 0.51mm (0.020”)



Z-specimen Geometry [K. Pike, et al. 2010]



Electropolished surface

Material Characterization

Inclusion & Composition Analysis

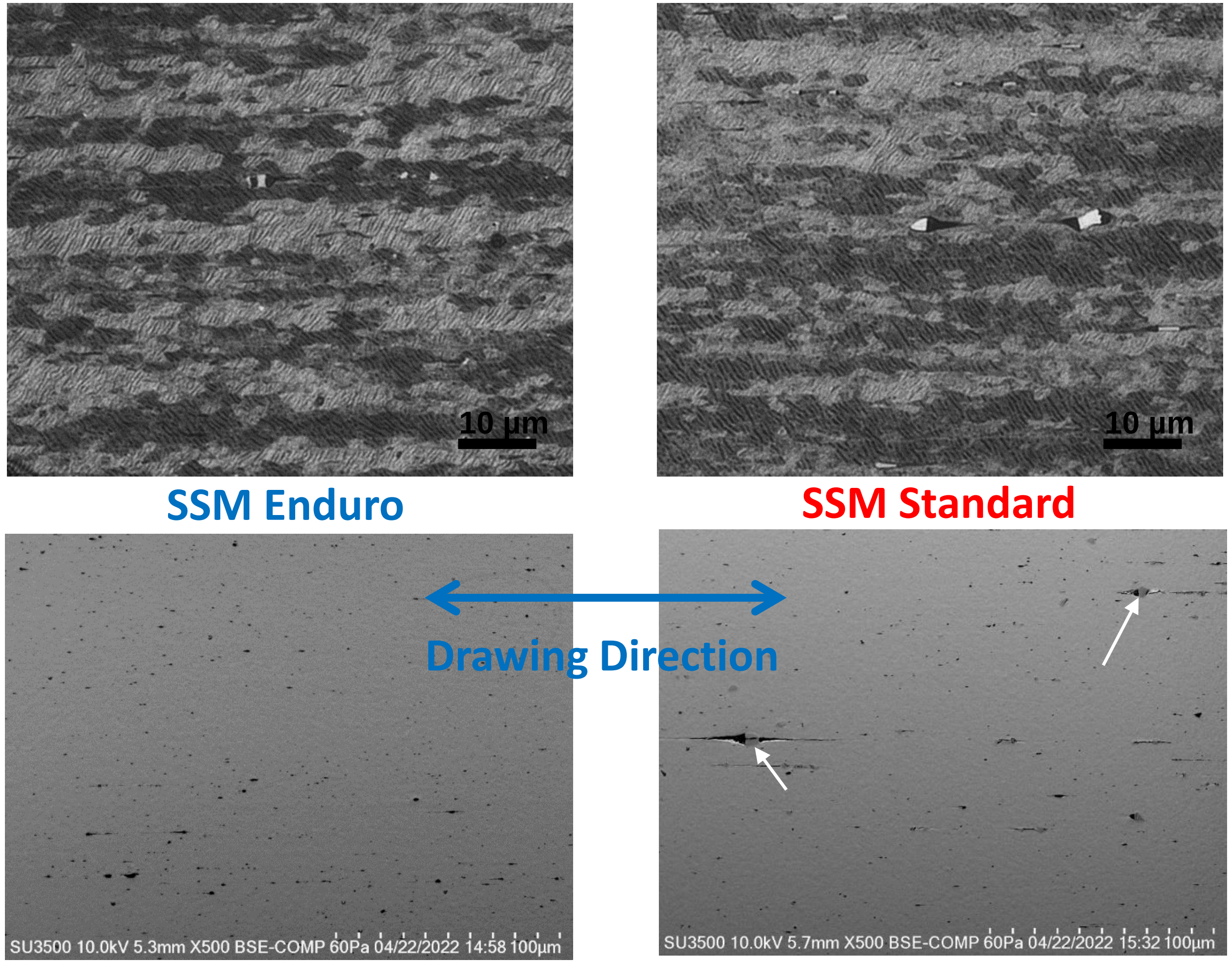
- Ingot inclusion analysis performed on 6 mm dia. coils with 27 fields of view at 500x magnification
- Enduro Oxygen and Carbon composition in line with average identified in robustness campaign (i.e. C → 258±18 ppm, O → 208±26ppm)

	Ingot Inclusion Analysis	
	MAX. Length (µm)	MAX. Area (%)
SSM Standard	16.0 – 17.53	0.56-0.94
SSM Enduro	8.05	0.27

Element	SSM Standard Value [wt.%]	SSM Enduro Value [wt.%]	ASTM F2063-18 Requirement [wt.%]
Ni	55.86 - 56.05	56.02	54.5 to 57.0
C	0.0313	0.0258	0.040 MAX.
Co	0.0001	0.0001	0.050 MAX.
Cu	0.0007	0.0001	0.010 MAX.
Cr	0.0031	0.0017	0.010 MAX.
H	< 0.0050	<0.0050	0.005 MAX.
Fe	0.013	0.009	0.050 MAX.
Nb	0.0001	0.0001	0.025 MAX.
N	0.0014	0.0012	0.005 MAX.
O	0.028	0.022	0.040 MAX.
Ti	Balance	Balance	Balance

Material Characterization

Wire Microstructure



Average Grain Size

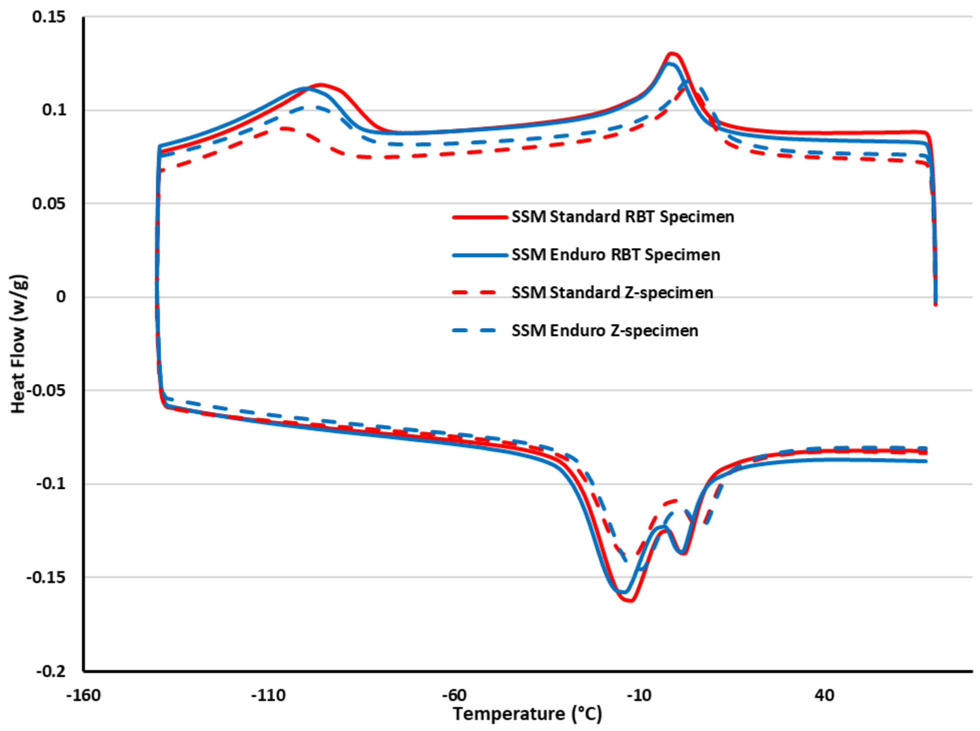
SSM Std.		SSM Enduro	
Longitudinal [µm]	Transverse [µm]	Longitudinal [µm]	Transverse [µm]
3.32 ± 0.70	1.46±0.26	3.52±0.71	1.55±0.42

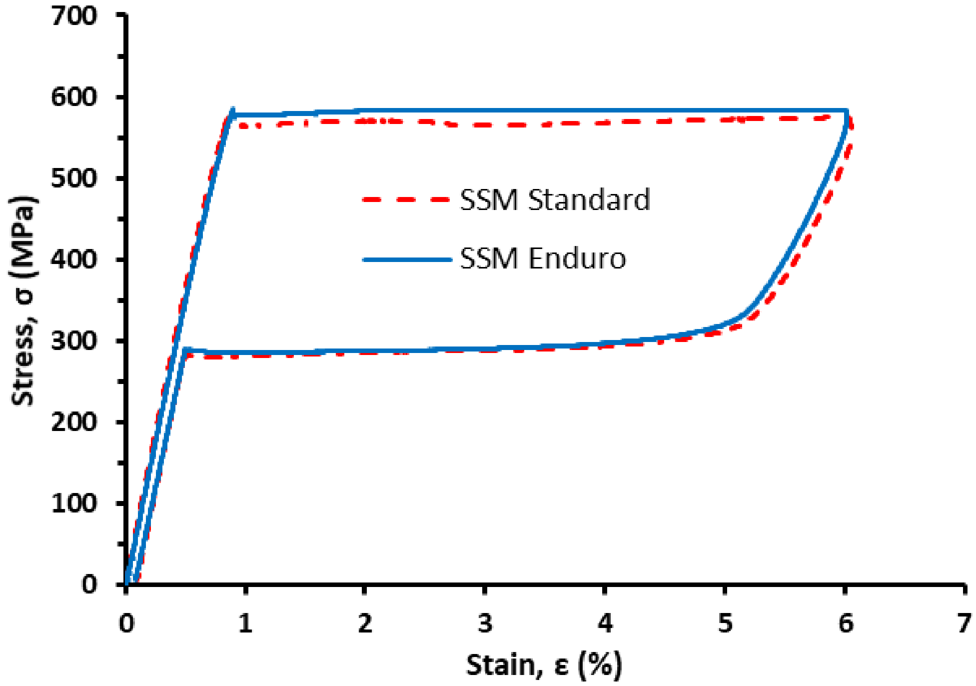
- Grain size verified to be similar between materials in both longitudinal and transverse directions
- Larger inclusions found in standard material lead to more voids and longer stringers in the drawn wire microstructure

Material Characterization

Thermomechanical Properties

- DSC performed per ASTM F2004-17 on non-annealed samples (i.e. final specimen conditions)
- Tensile Testing per ASTM F2516-18 performed at 37°C





Material Characterization

Material Property Summary

- Included Active Af results obtained from BFR for RBT specimen here (ASTM F2082-16)
- Extremely consistent thermomechanical properties realized when comparing samples produced from standard and Enduro ingots

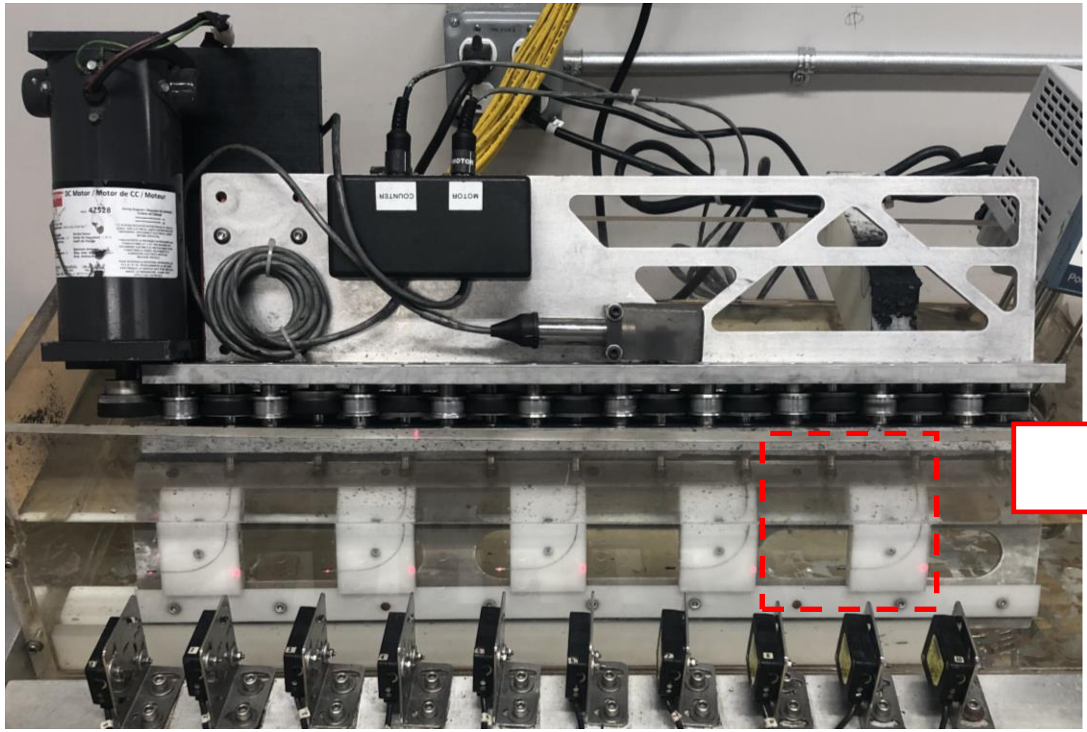
	Specimen Type	DSC, Af (°C)	BFR, Active Af (°C)	UPS (MPa)	LPS (MPa)	UTS (MPa)
SSM Standard	RBT Specimen	8.81	3	565	288	1300
	Z-Specimen	15.3	-	-	-	-
SSM Enduro	RBT Specimen	10.01	4	583	288	1350
	Z-Specimen	14.97	-	-	-	-

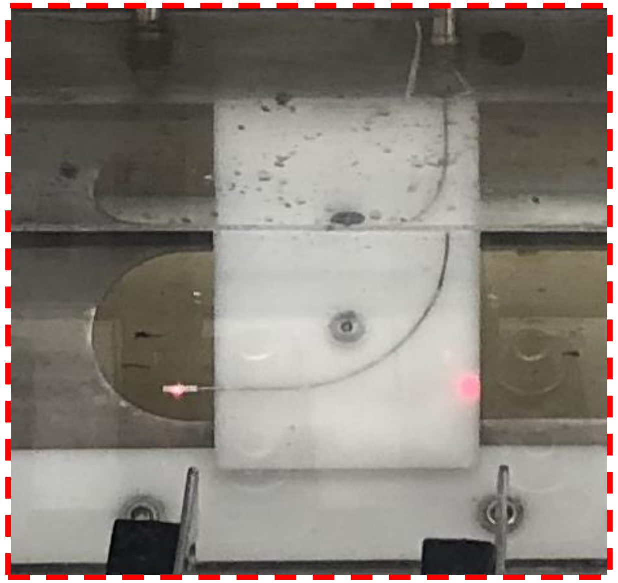
Rotating Beam Testing (RBT)

SMST2022
Shape Memory and Superelastic Technologies Conference and Exposition

In-house Testing per ASTM E2948-16a

- **Test Conditions** → Mean strain = 0%, 1000 RPM, Strains = 2.4, 1.8, 1.0, 0.8 & 0.65 %, Temp. = 37°C
- Sample size, n=15
- Guided style rotating beam test setup with laser counter break detection





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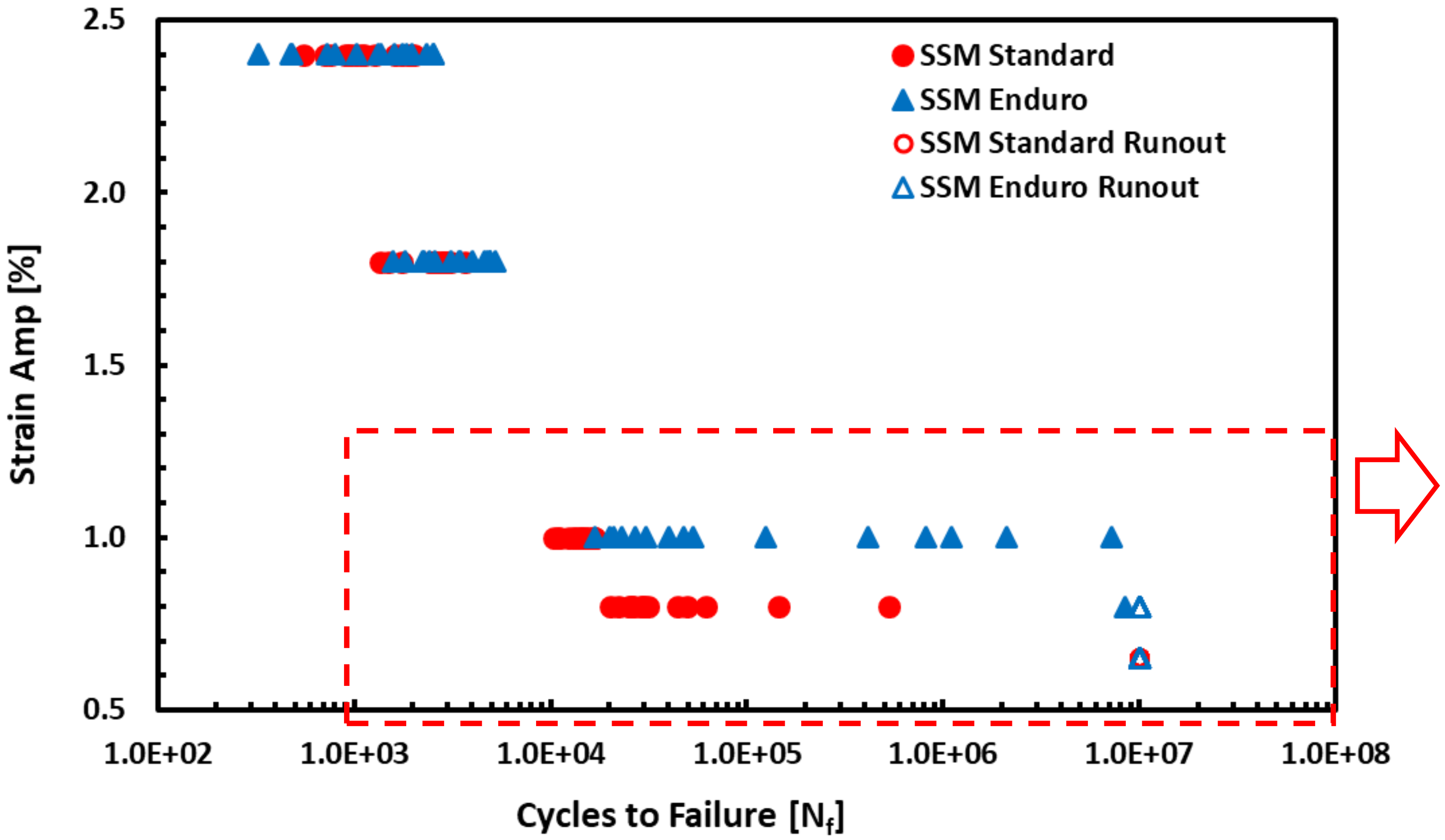
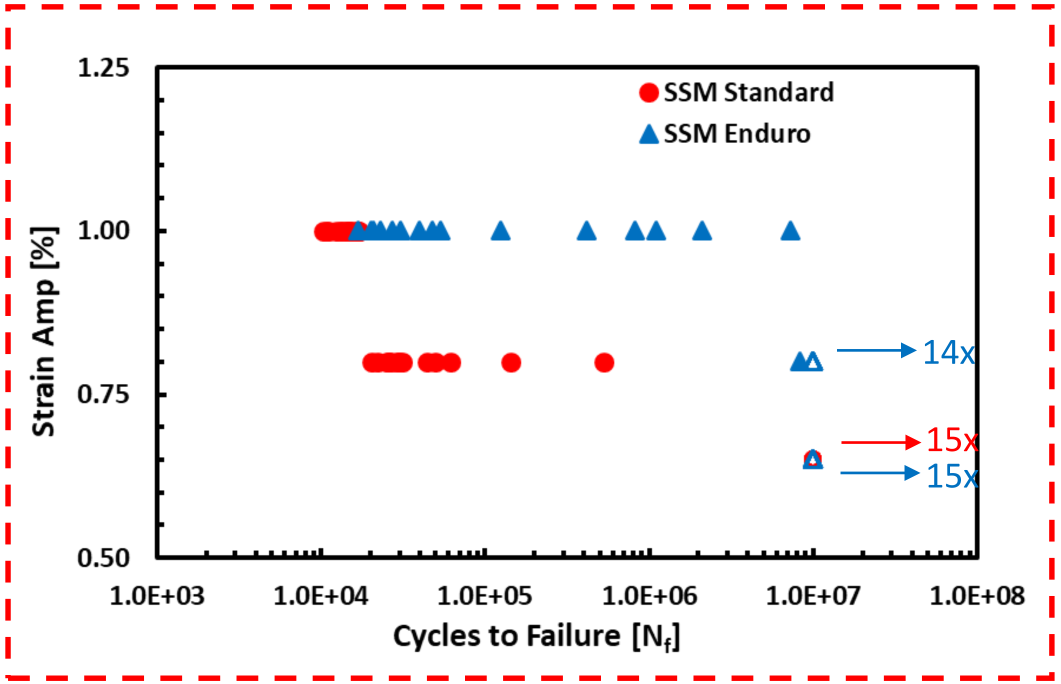
Rotating Beam Testing (RBT)

Results

- Enduro showing a significant improvement in high cycle fatigue





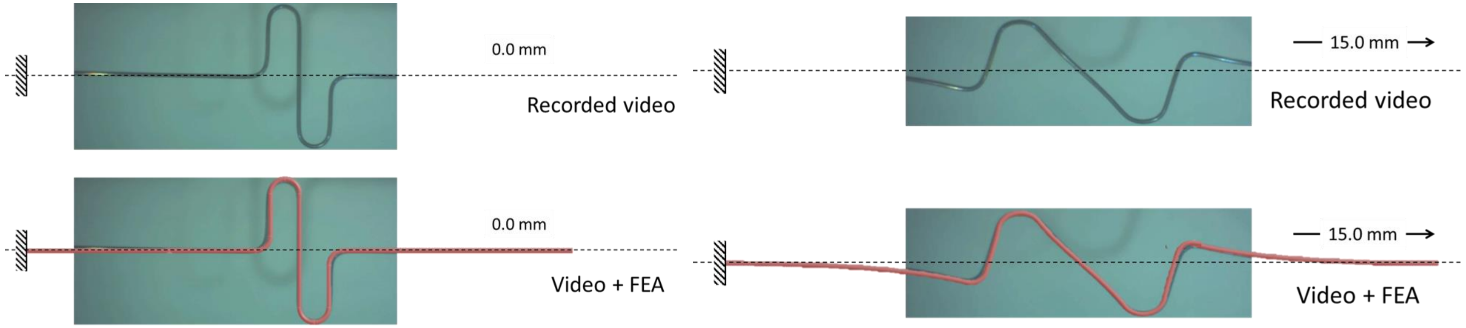
Z-Specimen Testing

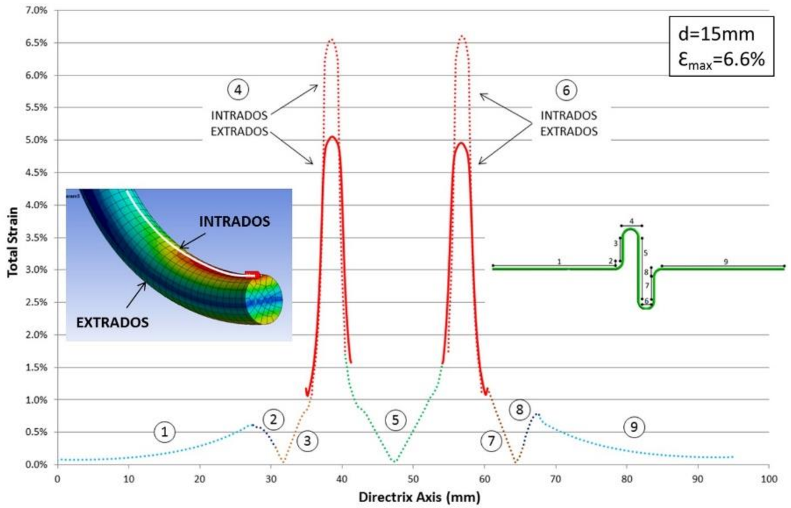
Z-specimen test designed to simulate z-stent application

- **Background** → Specimen developed by K. Pike et al. 2010 and a similar study performed by M. Launey et al. using the z-specimen in 2014

FEA Analysis

- Non-linear FEA analysis used to identify maximum strains
- Model created using material properties as inputs and validated using load displacement curves obtained from pull testing of z-specimen

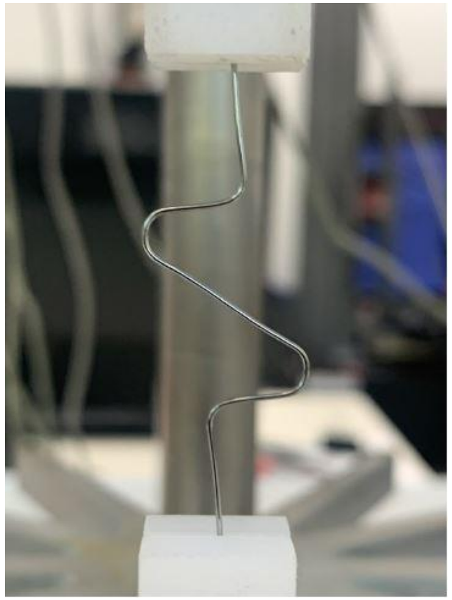


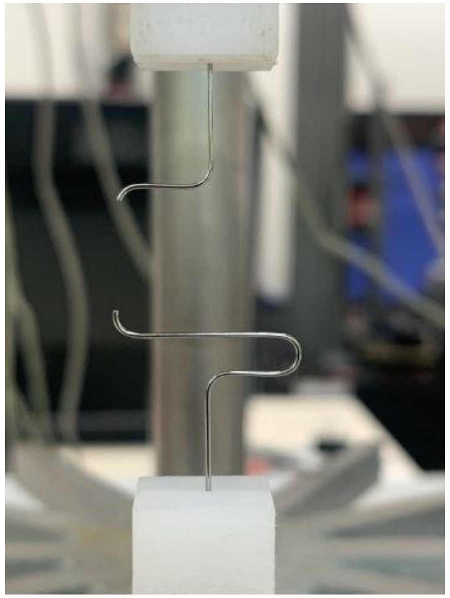


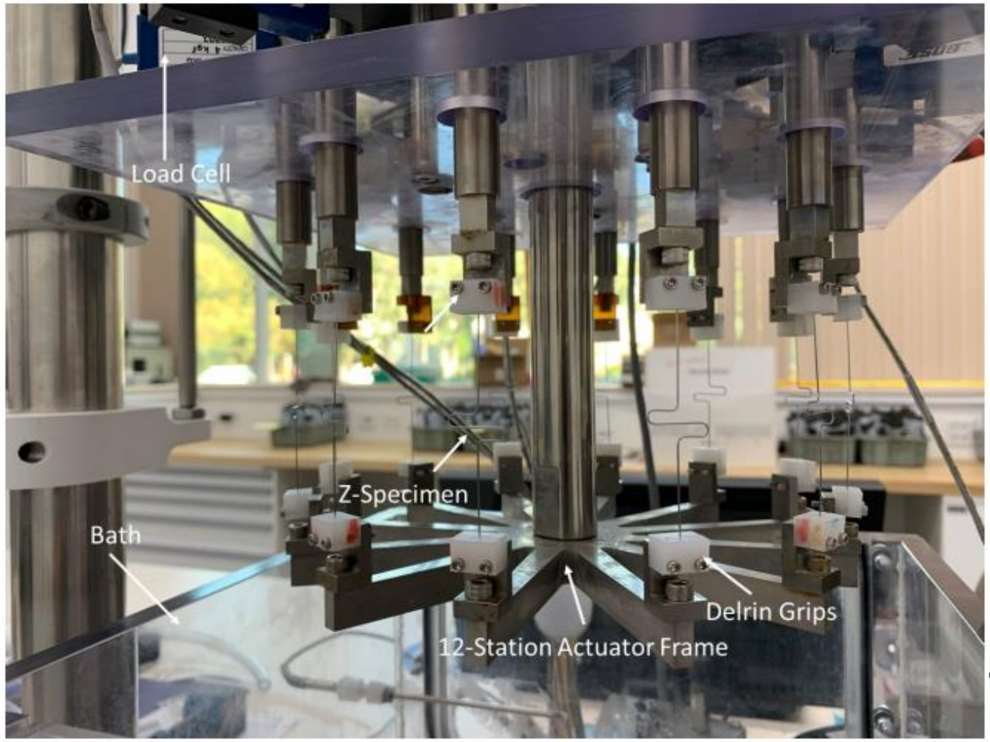
Z-Specimen Testing

Experimental - Outsourced Testing

- **Equipment** → 12-station BOSE ElectroForce Model 3330 multi-specimen fatigue tester
- **Bath conditions** → 37°C, PBS solution
- **Frequency** → 20 – 30 Hz for high to low strain amplitude conditions respectively
- **Strains** → Mean = 3.5%, Amplitudes = 2.5, 2.0, 1.5, 1.3, 1.0, and 0.5 %
- n=12 samples



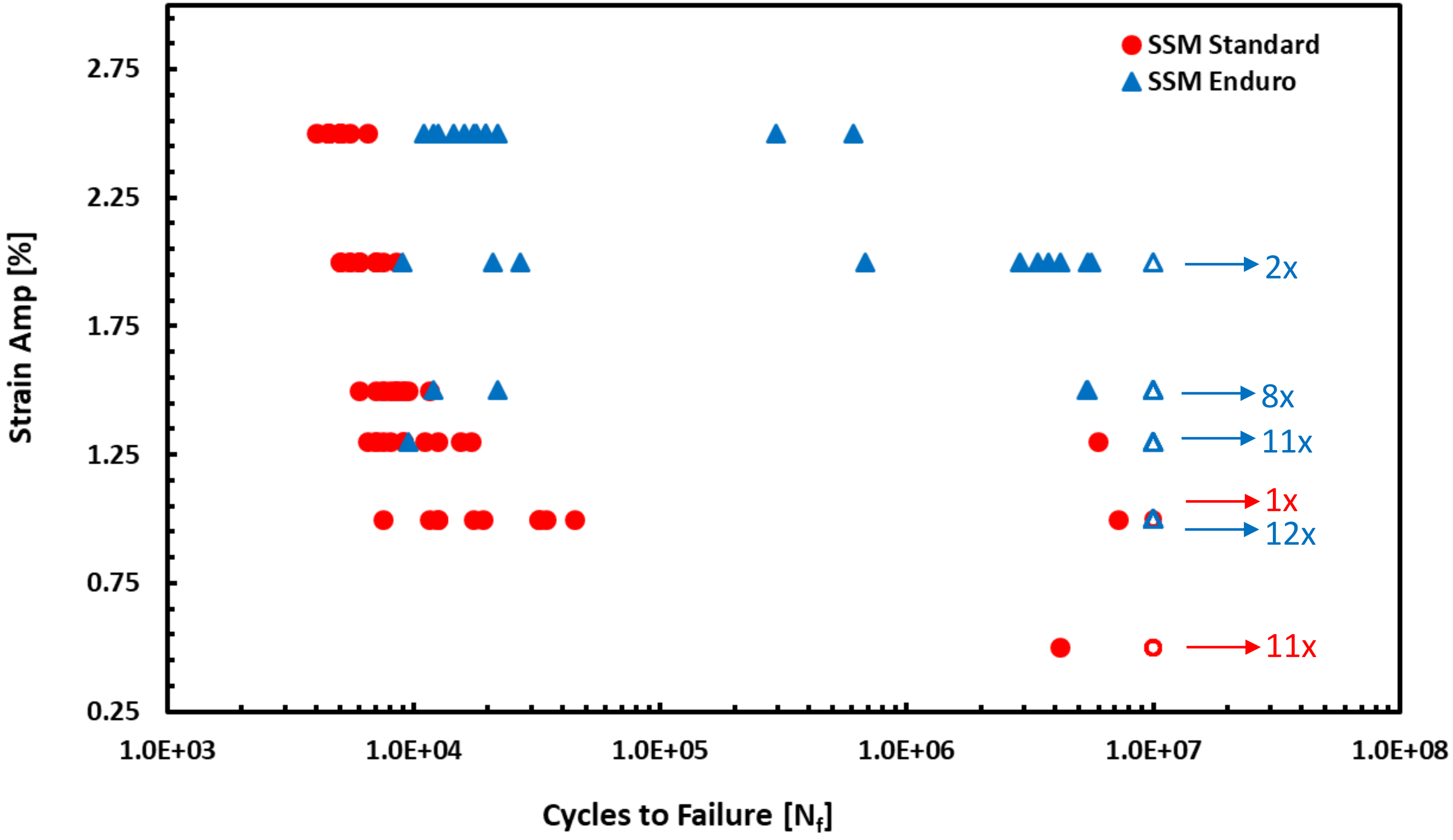


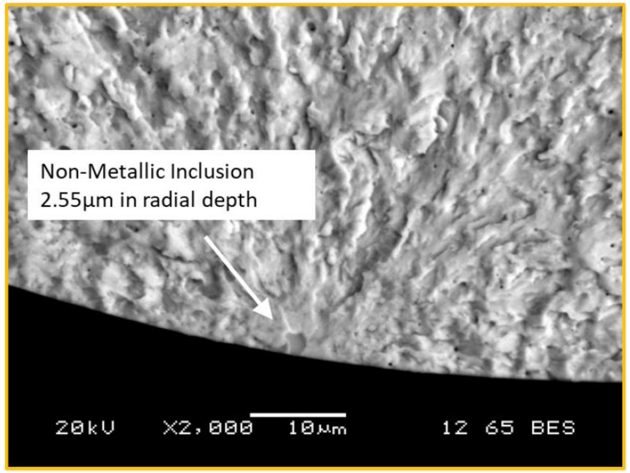


Z-Specimen Testing

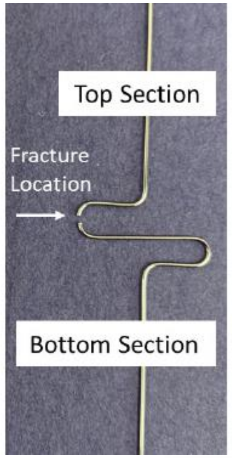
Results

- Enduro out performed the standard material at all strain conditions in this 3.5% mean strain fatigue test





- All samples failed at apex. as expected

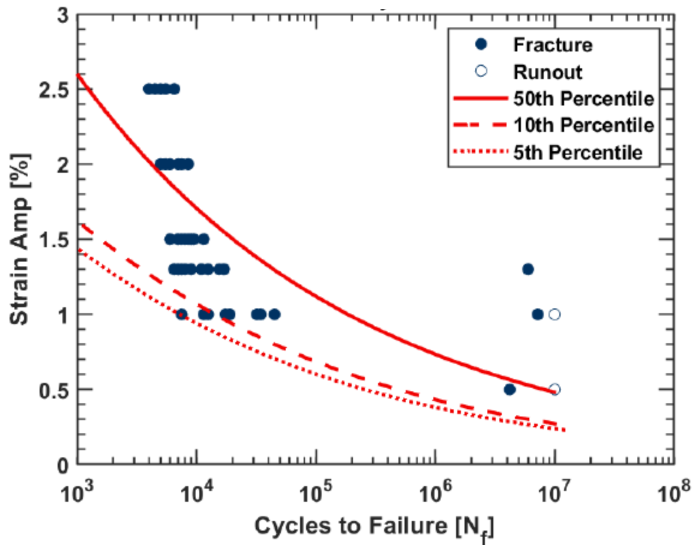


Z-Specimen Testing

Fatigue Limit at 10^7 Cycles

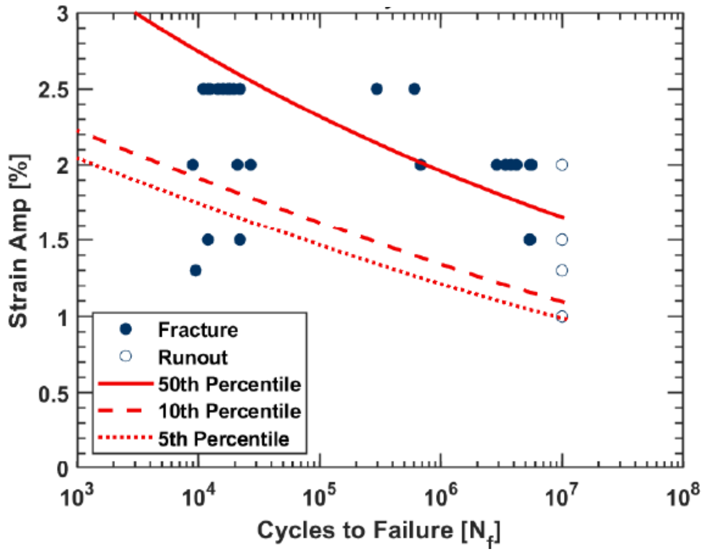
- **4.2x** improvement in fatigue limit at 10^7 cycles calculated using Lognormal regression fit with 95% confidence

SSM Standard



Percentile	10^7 Cycle Fatigue Limit
50th	0.48%
10th, 90% Confidence	0.27%
5th, 95% Confidence	**0.23%**

SSM Enduro



Percentile	10^7 Cycle Fatigue Limit
50th	1.65%
10th, 90% Confidence	1.09%
5th, 95% Confidence	**0.98%**

Summary

- Consistent Nitinol thermomechanical properties are achieved using both SSM standard and Enduro wrought materials without any changes required to downstream processes

- Greatly improved fatigue performance was demonstrated in both RBT and Z-specimen fatigue testing when using the Enduro Nitinol material due to smaller non-metallic inclusion size and lower inclusion density