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Conference and Exposition

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The Assessment of Physical and Mechanical Property Variability in a New Generation of Ultra-low Inclusion NiTi Alloy

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SAES Smart Materials, Inc

saes group

making innovation happen, together

Background and Motivation

Medical device designers continually innovate, often challenging Nitinol durability boundaries

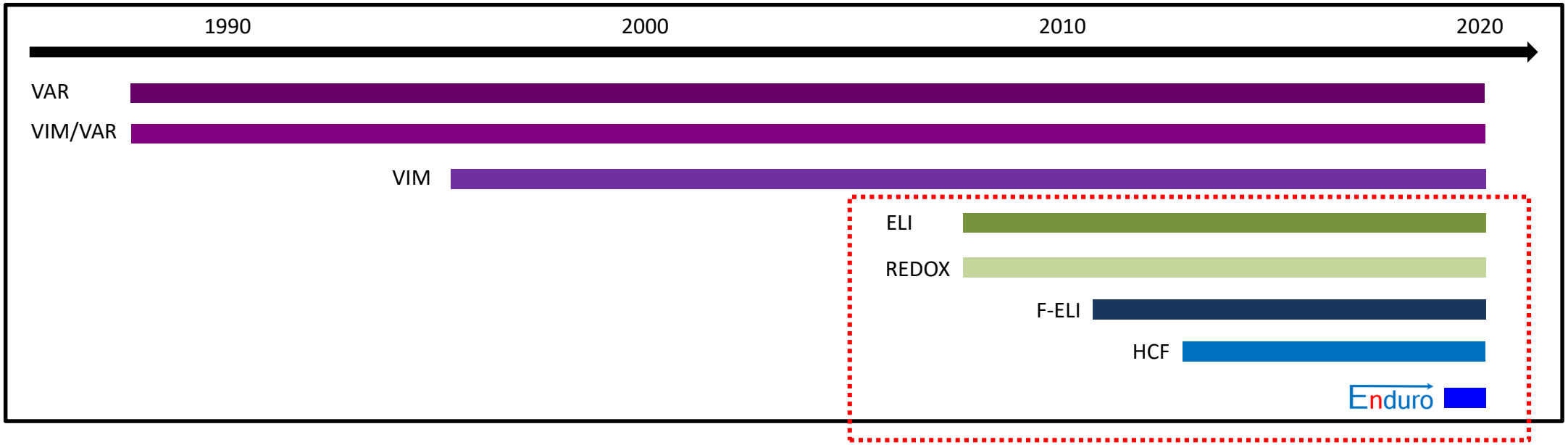
- Smaller features
- More complex applications
- Higher cycles to failure

Device durability is primarily affected by:

- Application
- Design
- Component processing and surface finish
- Raw material micro-cleanliness

Background and Motivation

Major Commercially Available Nitinol Alloys



Nitinol Suppliers have responded in recent years by improving their Raw Material offer

Background and Motivation

This newest, cleanest offering from SAES Smart Materials, Inc. was engineered for ultra-demanding applications by minimizing the size of non-metallic inclusions

Enduro Highlights

- Development started 2017 and was commercially released 2021
- Proprietary commercial scale melting process
- Optimized conversion process
- Available in all Nitinol product forms, ASTM F2063 compliant

Process robustness and consistency was deemed critical and was extensively challenged prior to market release in 2021

This important work will be summarized in the presentation

Experimental Procedures and Planned Studies

Enduro Process Robustness Study

- 18 commercial scale heats were melted in 3 discrete campaigns of 6 heats each
- Each campaign was separated by at least 4 weeks to capture variability in raw materials, melting and downstream processing
- Evaluation was performed on hot rolled products:
  6 mm diameter coils and 25 mm diameter bars

Characterization per ASTM F2063-18

- Chemistry
- Mechanical properties
- Microstructure

Micro-cleanliness Characterization and Analysis

Sampling Plan for Inclusion Characterization

- 3 locations along ingot length, each checked at 3 radius positions
- 3 images taken at all 9 sites, totaling 27 micrographs per ingot (~ 1 mm^2 area analyzed)
- Two cross-sections at each site (longitudinal per ASTM F2063 and transverse)

Consistent protocol as used for standard production

- Olympus AX70 optical microscope with a Teledyne Retiga 6 camera
- Image processing through Image Pro 10 software

Micro-cleanliness Analysis

- Inclusion maxima, Inclusion density, Gumbel distribution

Consistency of Main Gas Impurities

Carbon

- 258 ppm average
- 18 ppm standard deviation

Oxygen

- 208 ppm average
- 26 ppm standard deviation

Trace impurities not reported in table but consistent with standard nitinol

Campaign	Ingot	C, ppm	O, ppm	N, ppm
E1	E1-1	280	210	7
	E1-2	237	210	<5
	E1-3	226	250	<5
	E1-4	244	240	<5
	E1-5	229	230	<5
	E1-6	247	240	<5
E2	E2-1	299	210	23
	E2-2	255	230	17
	E2-3	271	220	8
	E2-4	258	220	12
	E2-5	261	220	15
	E2-6	277	200	10
E3	E3-1	255	160	<5
	E3-2	259	170	<5
	E3-3	244	170	<5
	E3-4	263	190	5
	E3-5	260	200	<5
	E3-6	275	180	<5

Transformation Temperature Consistency (As)

DSC per ASTM F2004

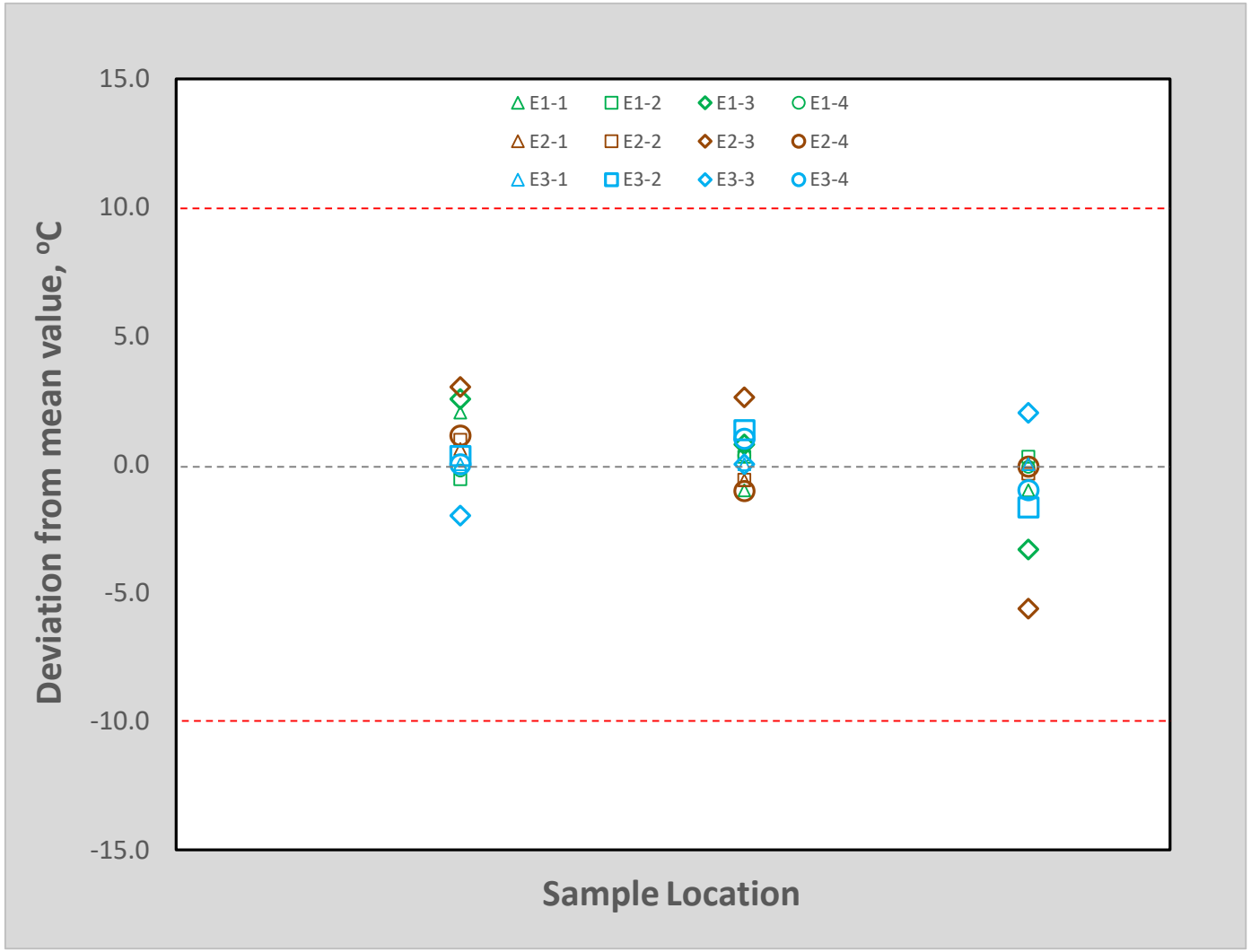
- 6 mm diameter coils
- Annealed at 850°C for 30 min.

Examine each ingot at 3 locations

- Variation ~ 5°C As in each ingot

ASTM F2063-18

- Stated tolerance of +/- 10°C



Properties of Mill Products

Tensile Tests per ASTM E8/E8M

- Specimens from bars and coils
- Annealed at 850°C for 30 min

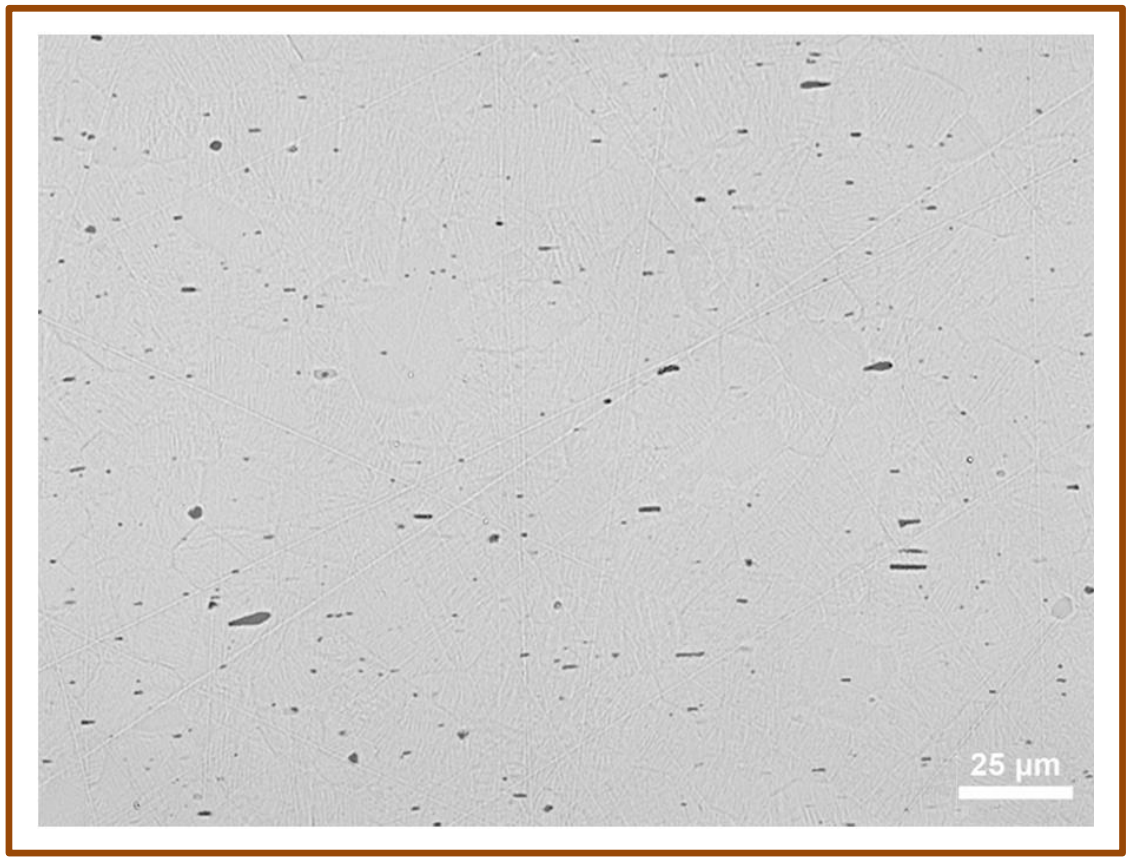
Grain size per ASTM E112

- All coils: ASTM G = 8
- All bars: ASTM G = 6

Campaign	Ingot	Product Type	Product Size, mm	Grain Size, ASTM G	UTS, MPa	Elongation, %
E1	E1-1	Coil	6	8	641	15
	E1-2				752	31
	E1-3				724	24
	E1-4				738	30
	E1-5	Bar	25	6	666	16
	E1-6				678	23
E2	E2-1	Coil	6	8	634	19
	E2-2				655	22
	E2-3				648	18
	E2-4				641	21
	E2-5	Bar	25	6	646	17
	E2-6				667	17
E3	E3-1	Coil	6	8	621	21
	E3-2				607	20
	E3-3				683	23
	E3-4				648	23
	E3-5	Bar	25	6	712	19
	E3-6				689	18
ASTM F2063-18			5.50-94.0	≥ 4	≥ 551	≥ 15

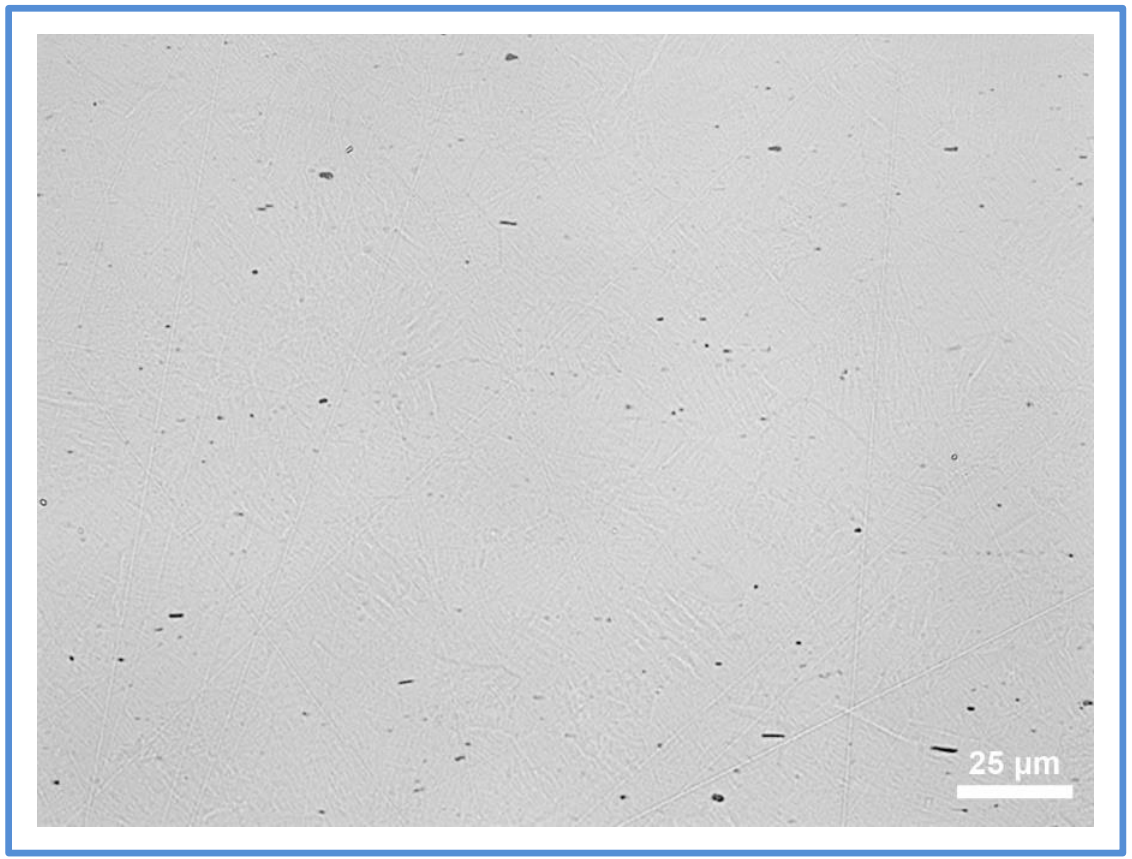
Microcleanliness – Longitudinal Section

Representative microstructure of Enduro and standard materials.



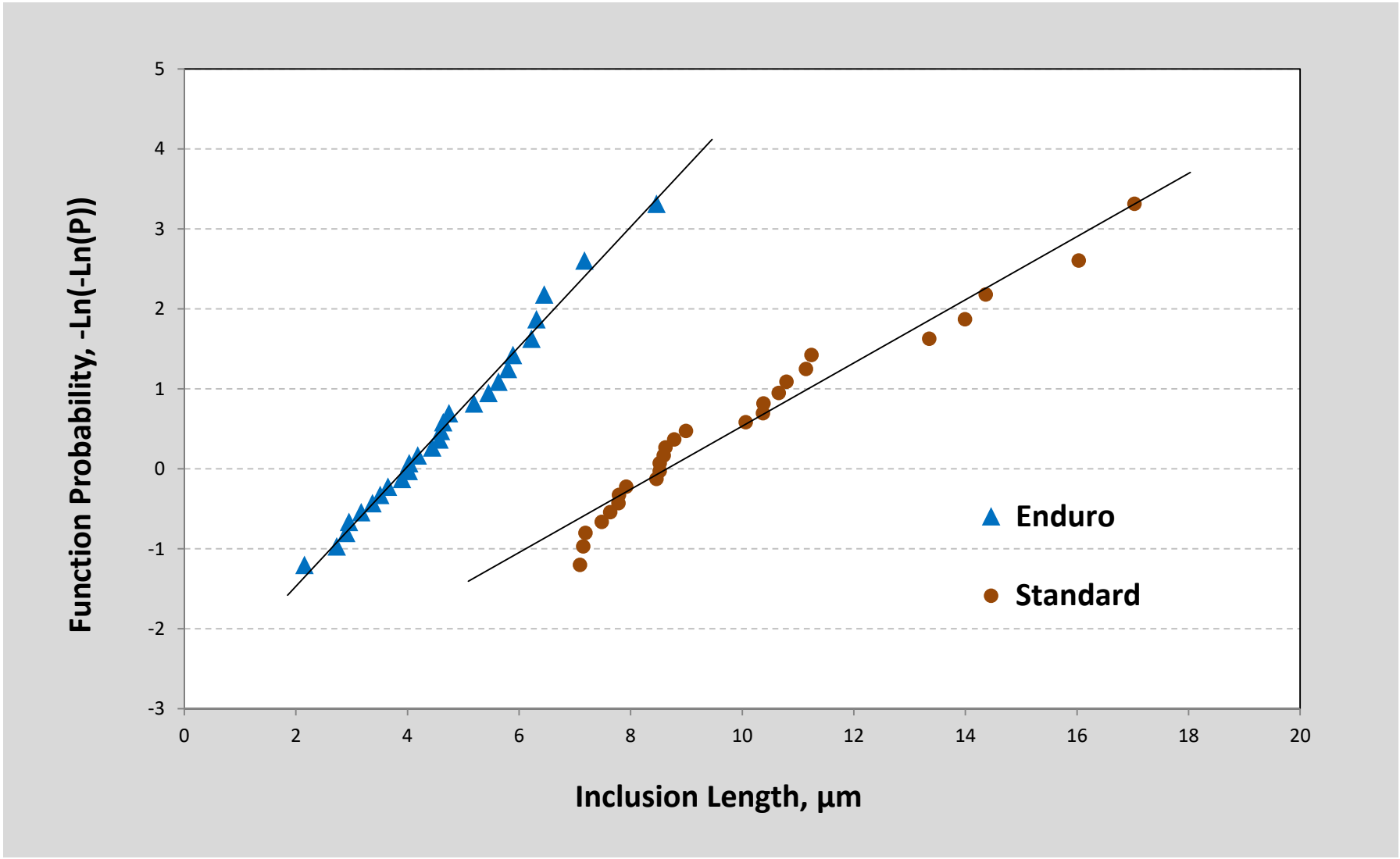
Standard

Rolling Direction



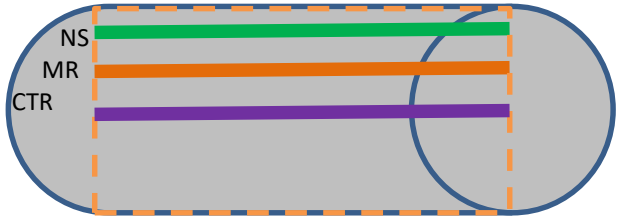
Enduro

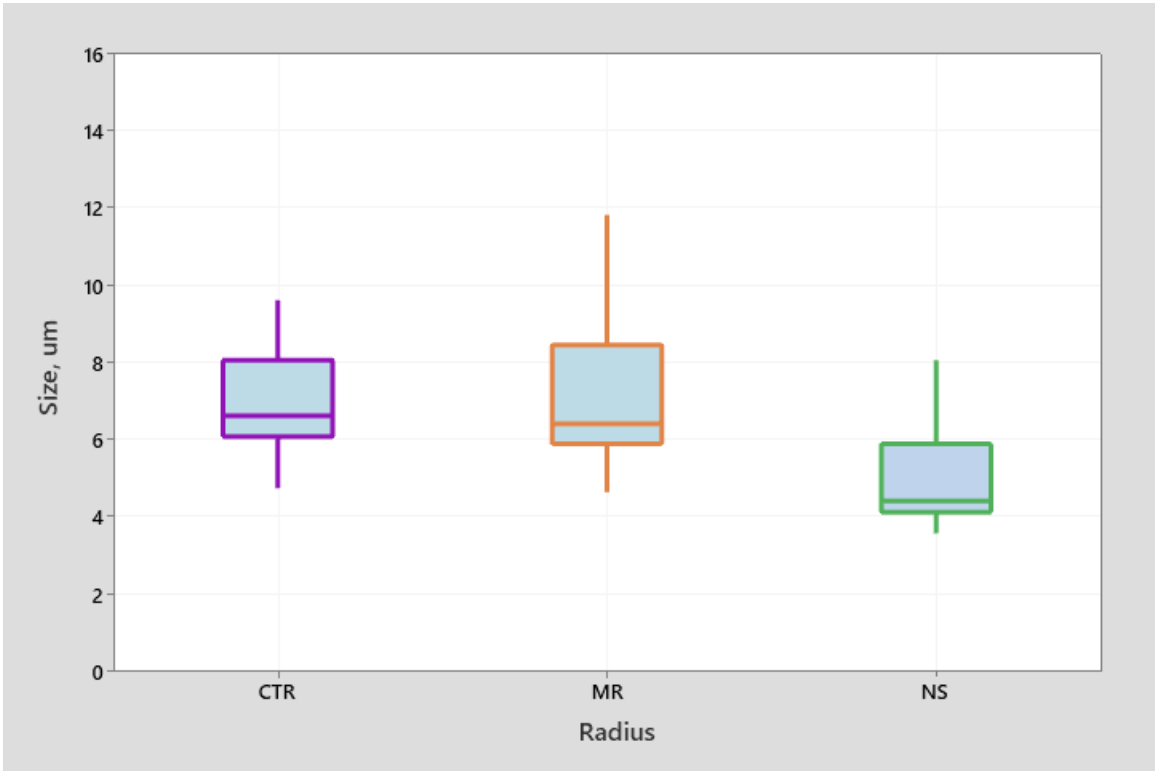
Longitudinal Inclusions – Gumbel Distribution

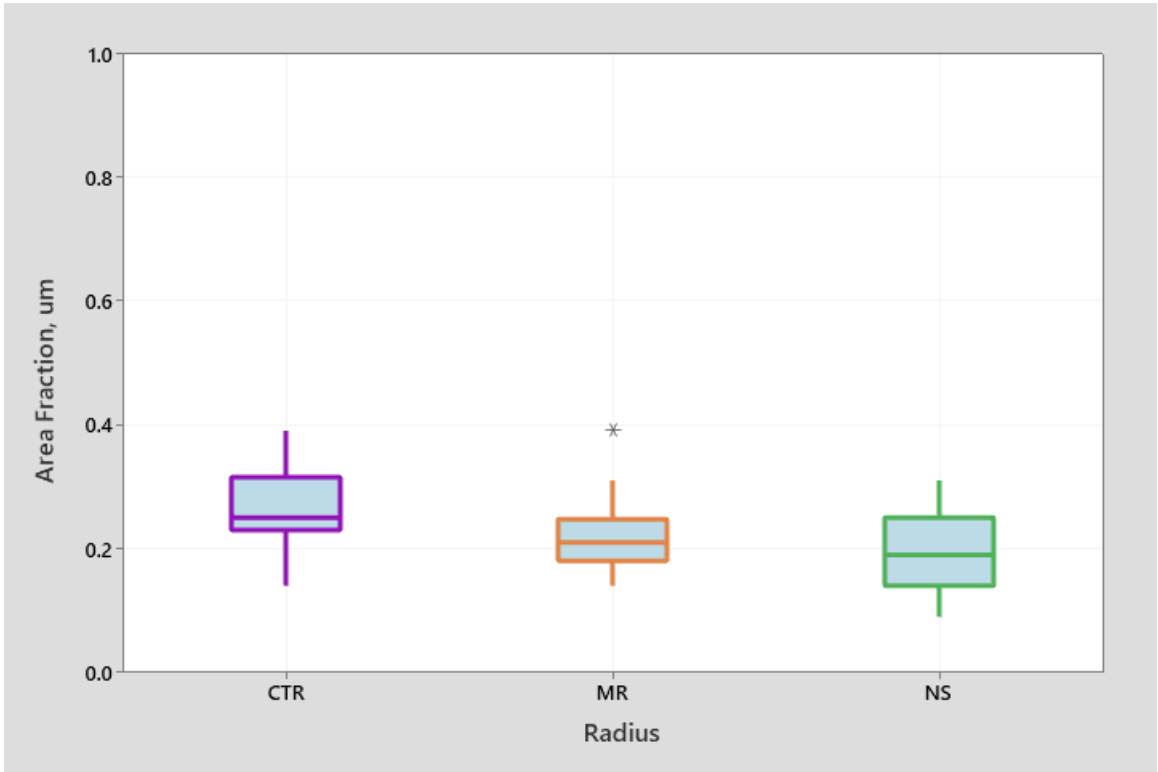


Radius Distribution of Inclusion Maxima

Longitudinal Inclusions in Enduro material
at center (CTR), mid-radius (MR) and near surface (NS)







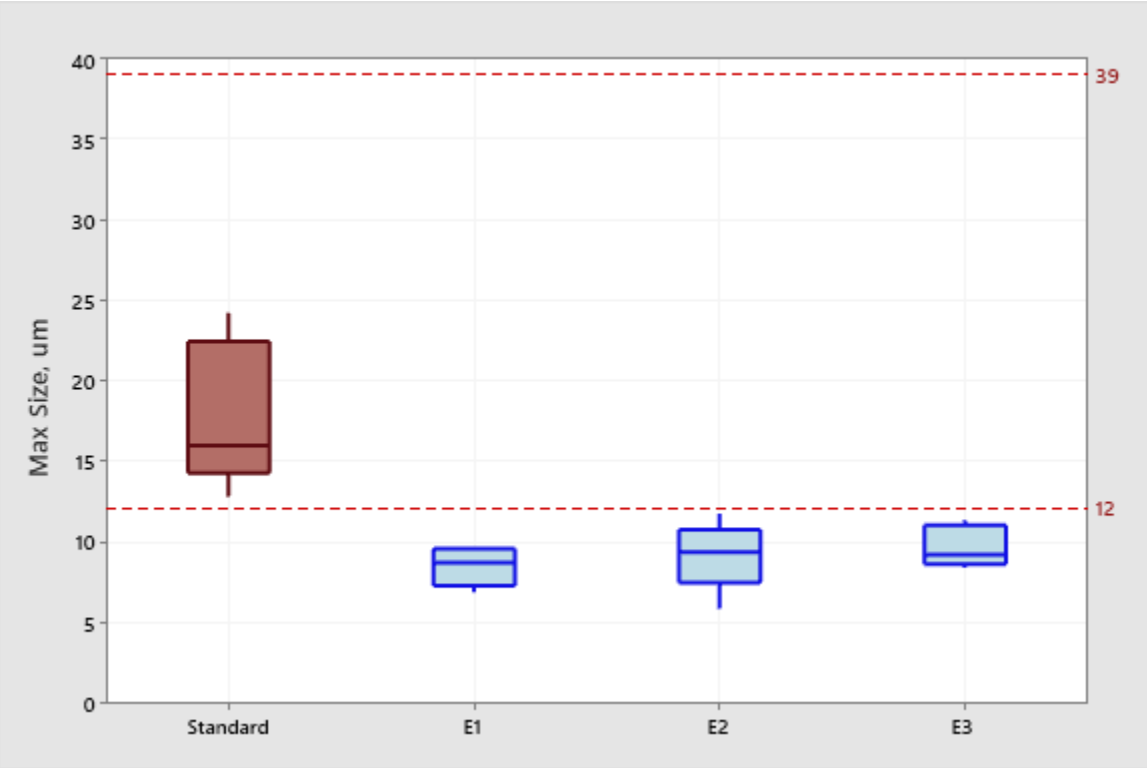
Longitudinal Inclusions Characteristics

Campaign	Ingot	Product		Longitudinal Inclusions		Inclusions Density, count/mm^2			
		Type	Size, mm	Max. Size, µm	Max. Area, %	> 1 µm	> 2 µm	> 5 µm	> 10 µm
E1	E1-1	Coil	6	8.3	0.36	1424	403	15	0
	E1-2			9.6	0.28				
	E1-3			9.7	0.37				
	E1-4			9.2	0.25				
	E1-5	Bar	25	6.9	0.33				
	E1-6			7.5	0.27				
E2	E2-1	Coil	6	9.7	0.46	1725	517	20	1.0
	E2-2			11.8	0.39				
	E2-3			10.5	0.44				
	E2-4			8.1	0.27				
	E2-5	Bar	25	9.1	0.42				
	E2-6			5.9	0.37				
E3	E3-1	Coil	6	8.9	0.22	1047	310	18	0.6
	E3-2			9.6	0.25				
	E3-3			8.5	0.23				
	E3-4			11.4	0.22				
	E3-5	Bar	25	11.0	0.30				
	E3-6			8.7	0.37				

Longitudinal Inclusions Maxima

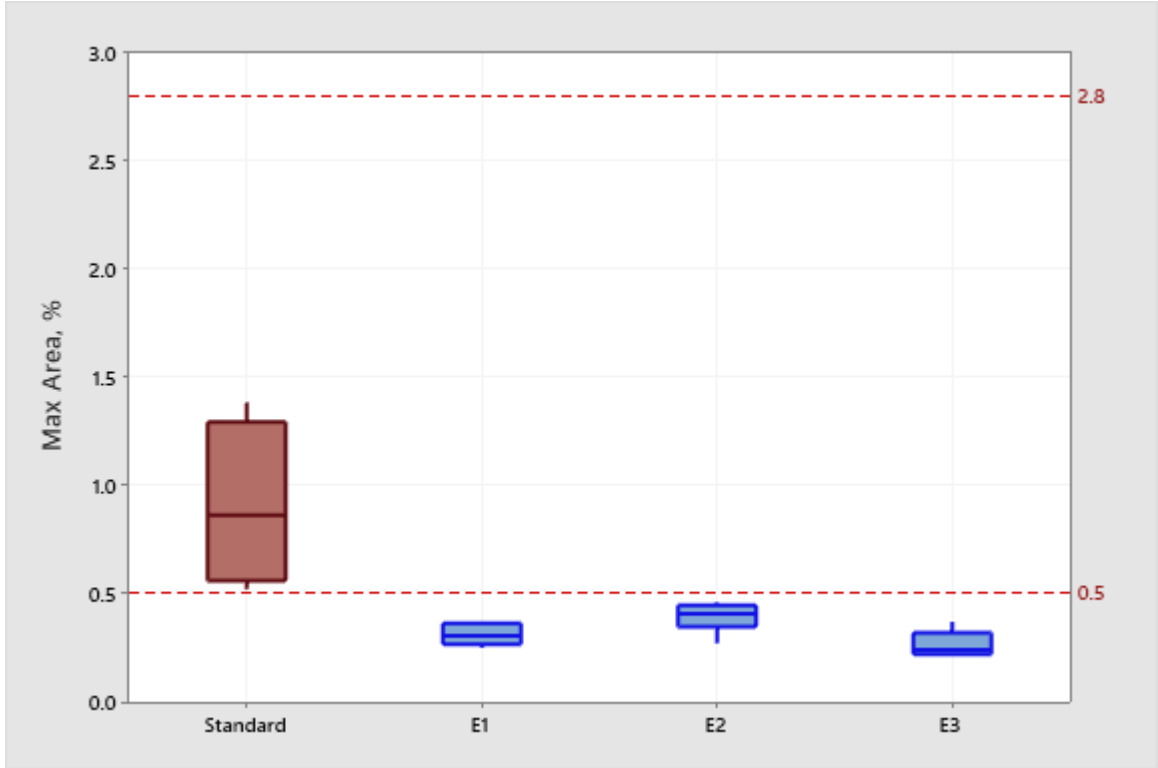
Inclusions Size

Eliminated large inclusion particles > 12 µm in Enduro



Inclusions Area Fraction

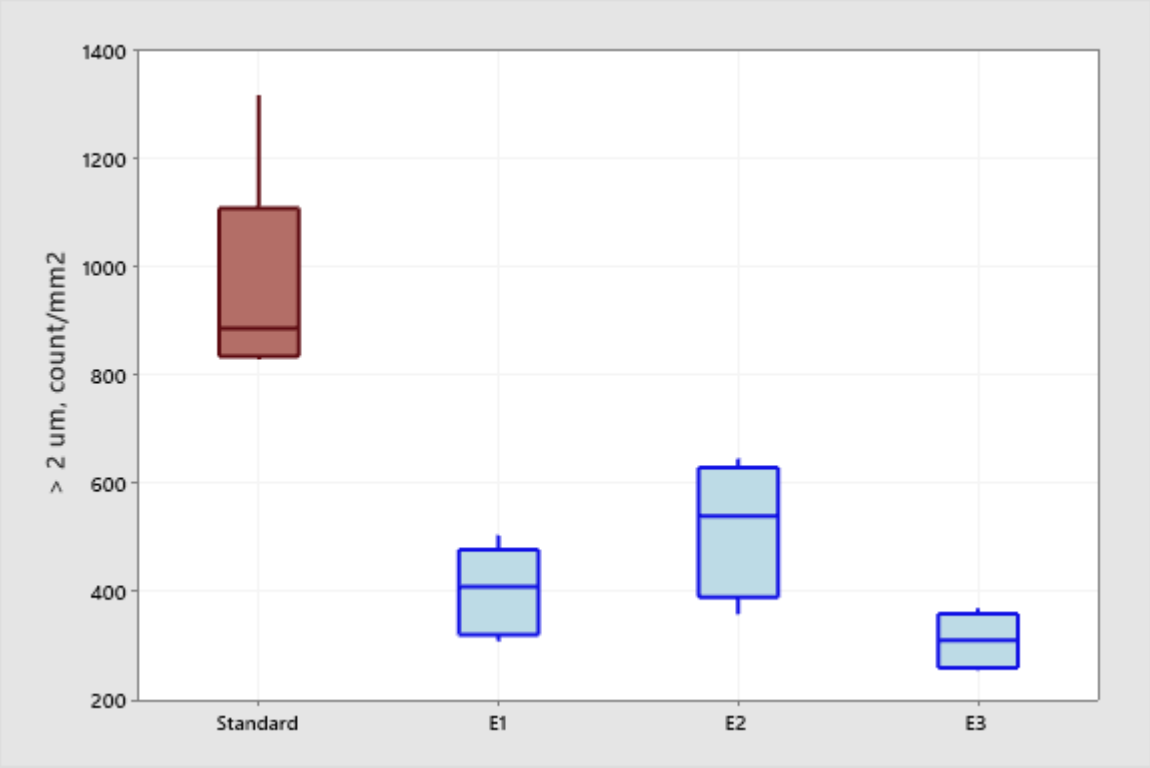
Reduced inclusion area fraction below 0.5 % in Enduro



Longitudinal Inclusions Density

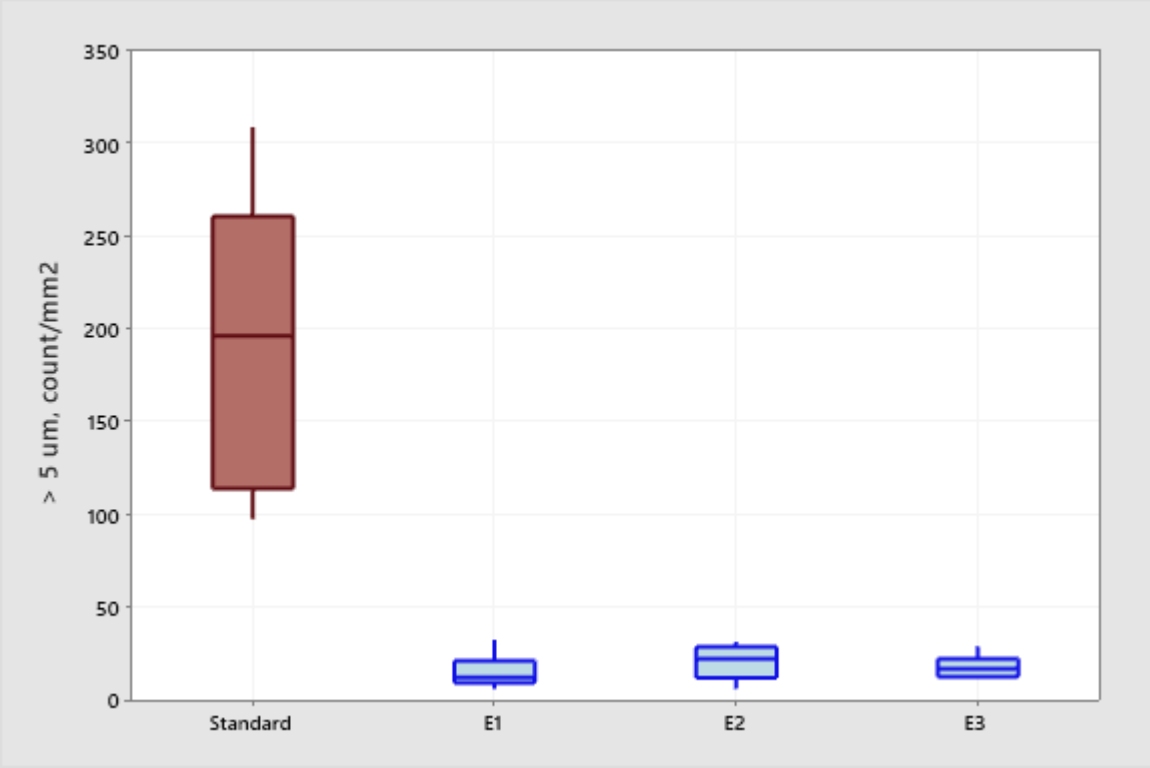
Inclusions > 2 µm

Inclusion density in Enduro has been reduced by ~ 2X



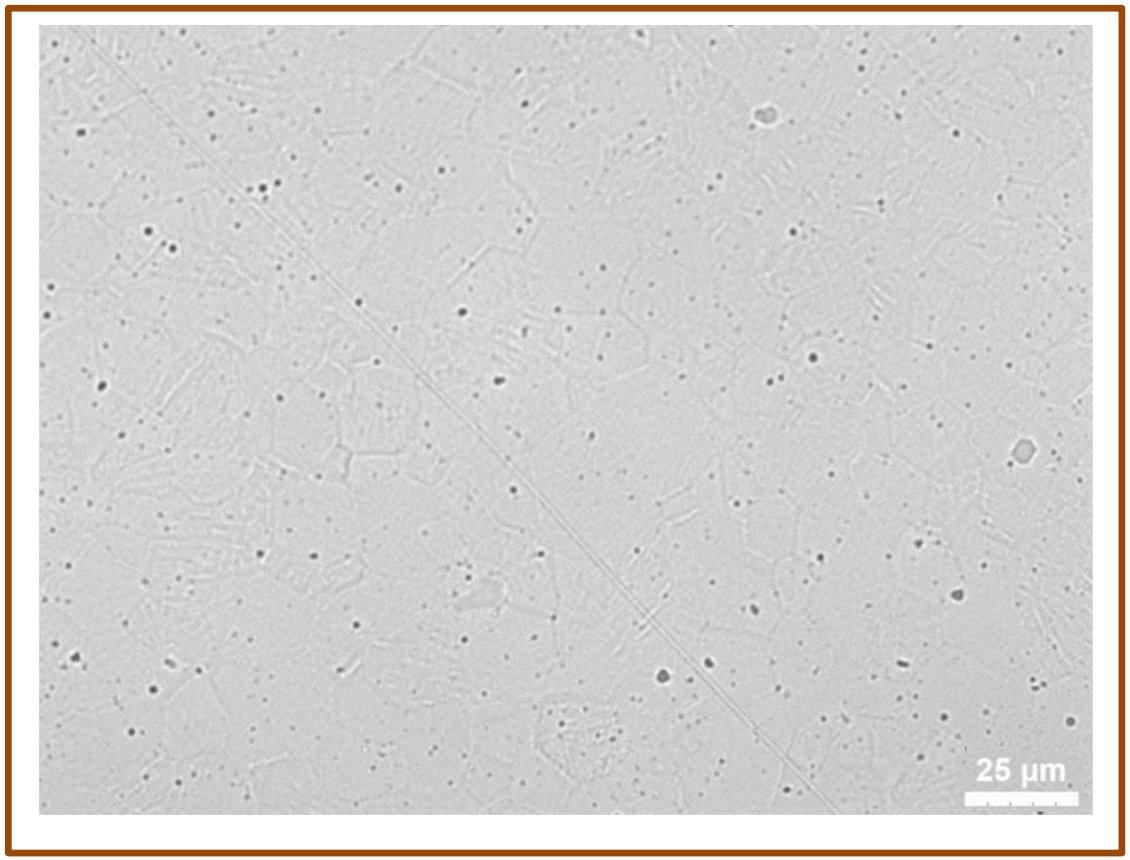
Inclusions > 5 µm

Inclusion density in Enduro has been reduced by ~ 10X

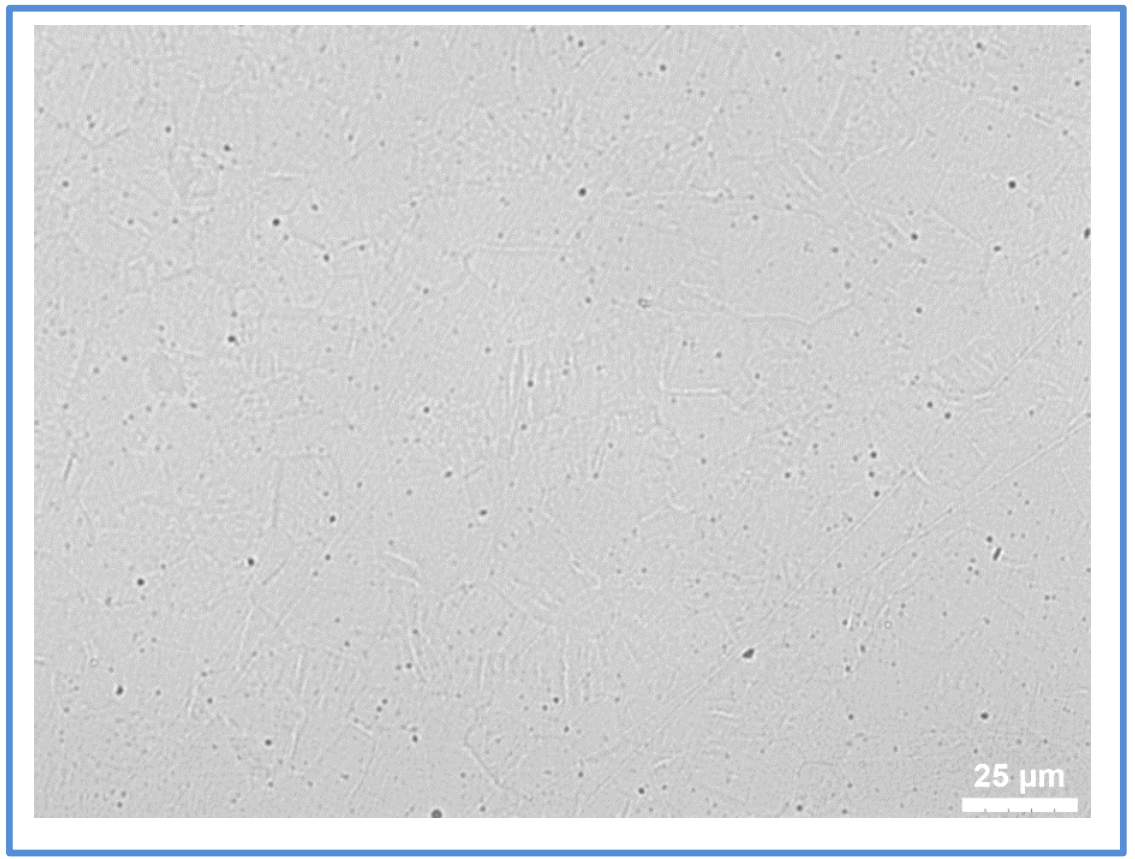


Microcleanliness – Transverse Section

Representative microstructure of Enduro and standard materials.

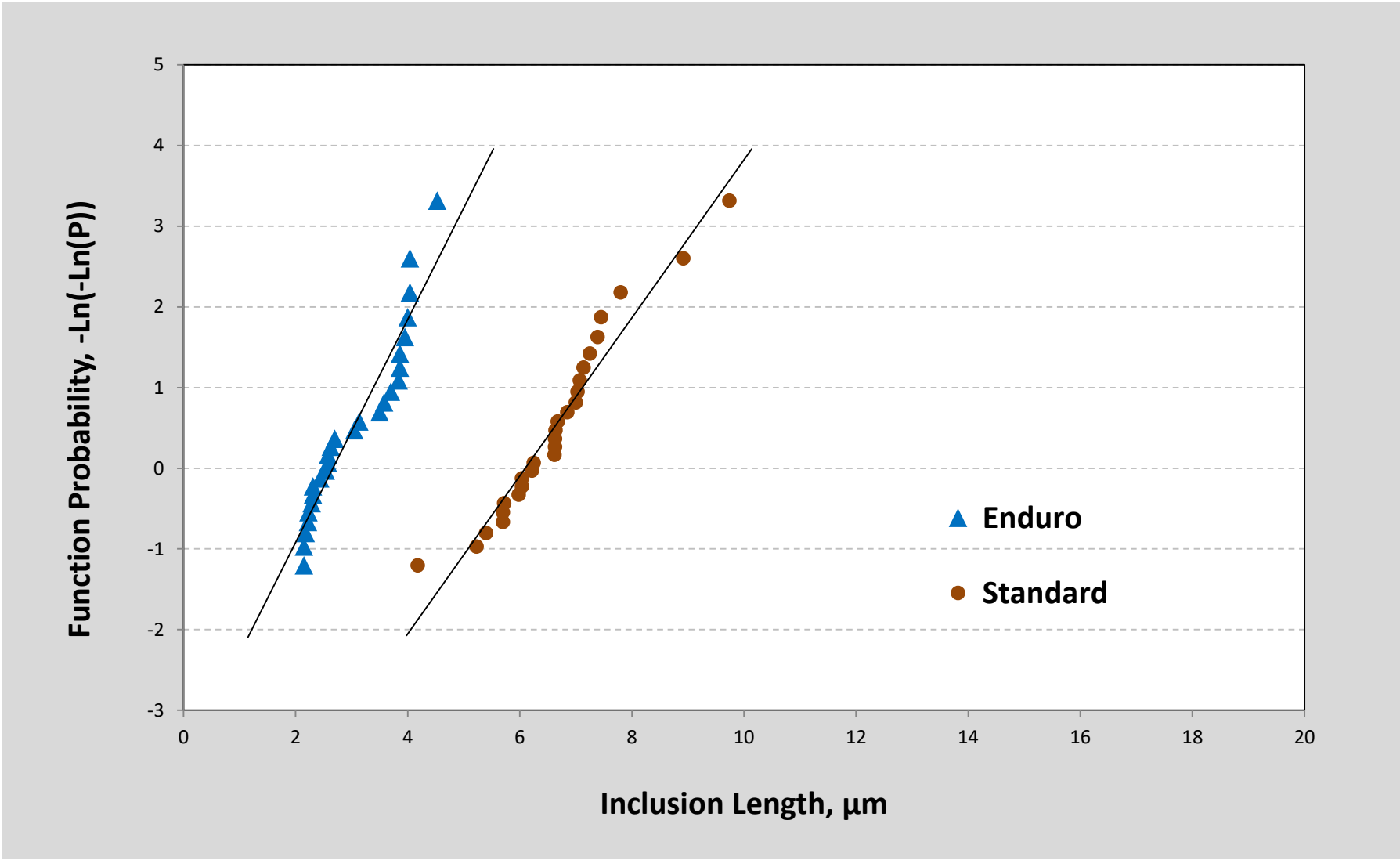


Standard



Enduro

Transverse Inclusions – Gumbel Distribution



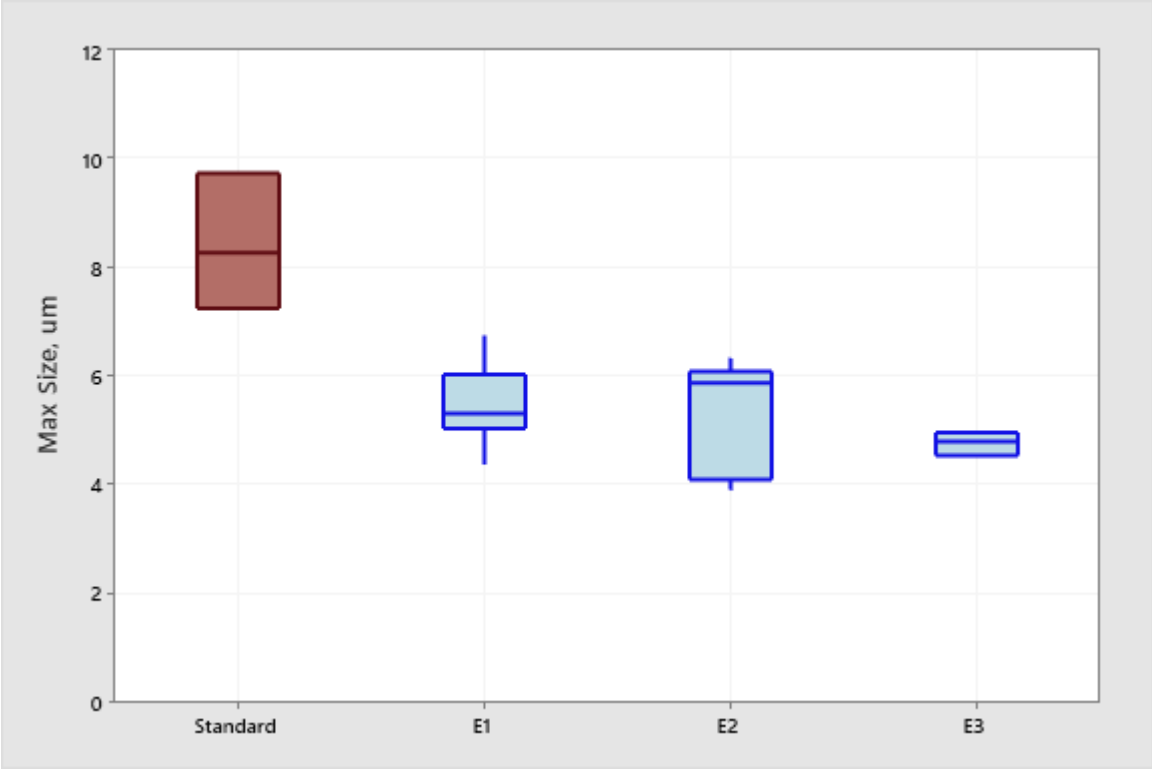
Transverse Inclusions Characteristics

Campaign	Ingot	Product		Transverse Inclusions		Inclusions Density, count/mm^2			
		Type	Size, mm	Max. Size, µm	Max. Area, %	> 1 µm	> 2 µm	> 5 µm	> 10 µm
E1	E1-1	Coil	6	5.3	0.34	1112	139	2.9	0
	E1-2			4.4	0.31				
	E1-3			6.8	0.36				
	E1-4			5.3	0.30				
	E1-5	Bar	25	5.3	0.39				
	E1-6			5.8	0.29				
E2	E2-1	Coil	6	4.1	0.35	1147	134	1.1	0
	E2-2			5.9	0.37				
	E2-3			5.1	0.40				
	E2-4			6.1	0.34				
	E2-5	Bar	25	6.3	0.43				
	E2-6			6.0	0.37				
E3	E3-1	Coil	6	3.9	0.27	1167	132	0.2	0
	E3-2			5.0	0.33				
	E3-3			4.5	0.32				
	E3-4			4.6	0.32				
	E3-5	Bar	25	4.8	0.38				
	E3-6			4.9	0.35				

Transverse Inclusions Maxima

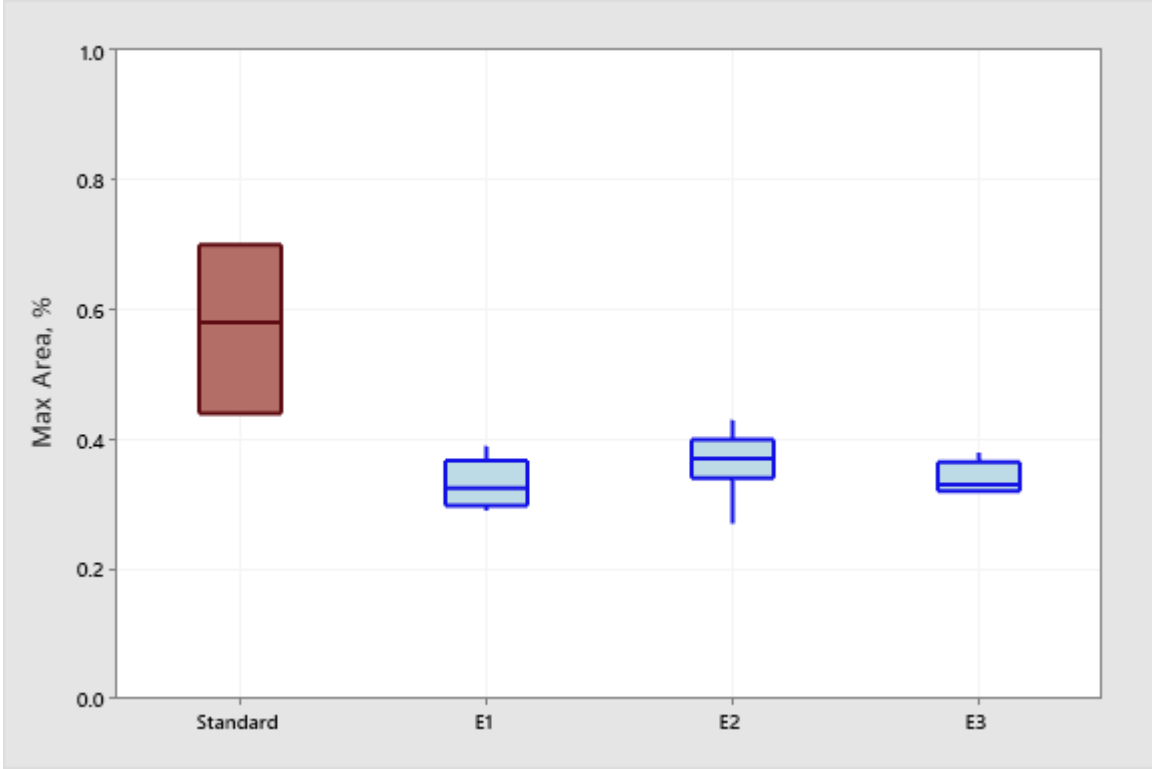
Inclusions Size

Reduced inclusion size about 100% in Enduro



Inclusions Area Fraction

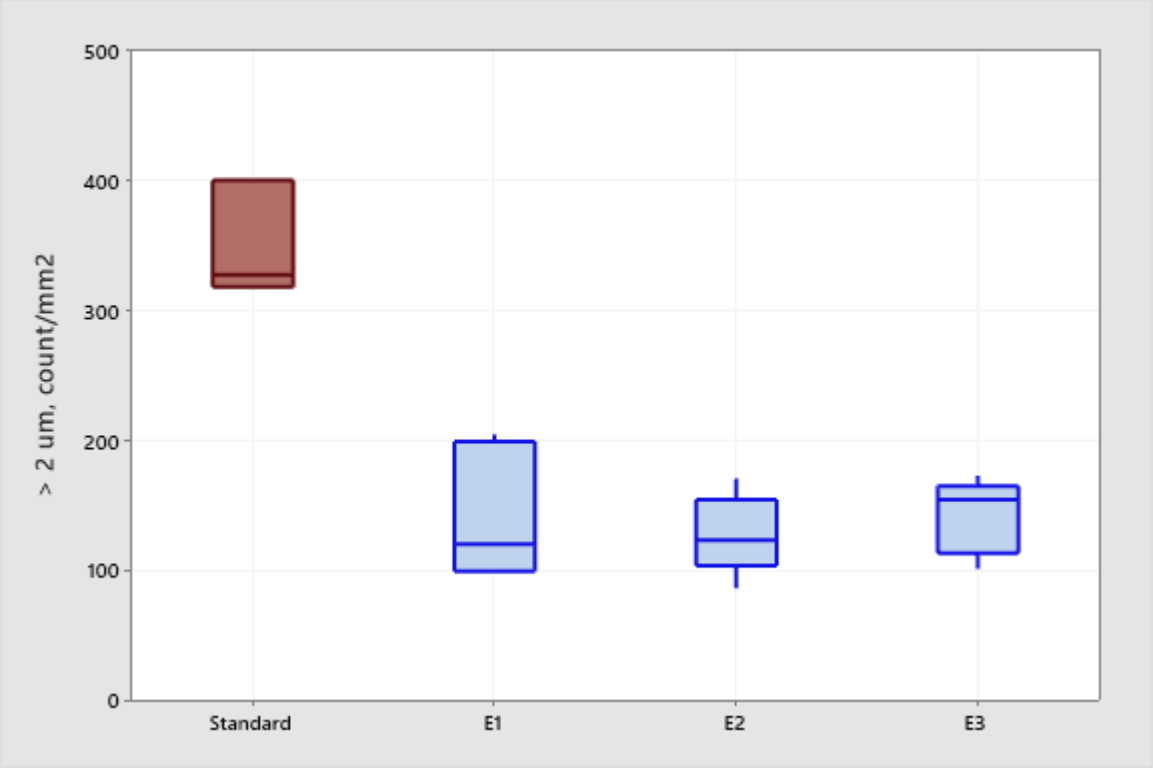
Lowered inclusion area fraction about 100% in Enduro



Transverse Inclusions Density

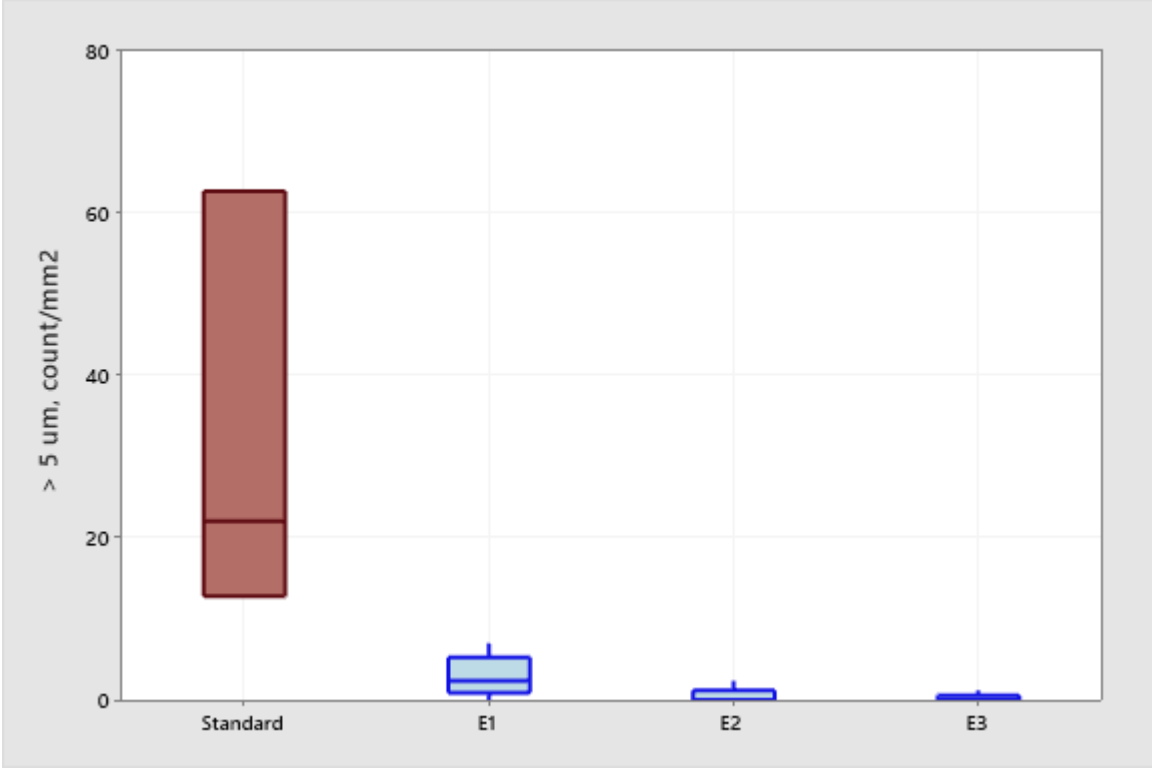
Inclusions > 2 µm

Inclusion density in Enduro has been reduced by ~ 3X



Inclusions > 5 µm

Inclusion density in Enduro has been reduced by ~ 20X



Enduro Characterization Summary

The micro-cleanliness in Enduro material is significantly improved over standard material with good consistency

- Reduced maximum inclusion size and area fraction
- Lowered density of large inclusion particles (>5 µm) by a factor of 10
- Improved micro-cleanliness across entire sections of mill products
- Consistent results within campaigns and across campaigns
- Thermal and mechanical properties are comparable to standard material
- ASTM F2063-18 compliant