ROLE OF MATERIALS RESEARCH IN LABORATORY TIRE AGING AND DURABILITY TEST DEVELOPMENT.

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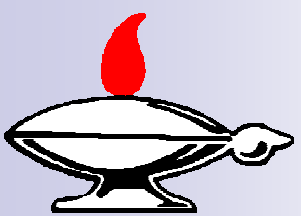


ACS Rubber Division 2006

History

- Tire Involvement
  - Forensic Investigations of Tire Failures
  - Employees from the Tire Industry
  - Expanding Engineering Capability, $$
- The Materials Science “Gap”

ARDL Corporate Goal

- Understand Tire Aging Mechanisms
- Marry Chemistry & Physics Mechanisms with Engineering & Dynamic Testing
- Identify/Develop Tests that Correlate with Field Benchmarks
- Become Recognized for Tire Materials R&D
- Assist with Robustness Assessments on New Tires

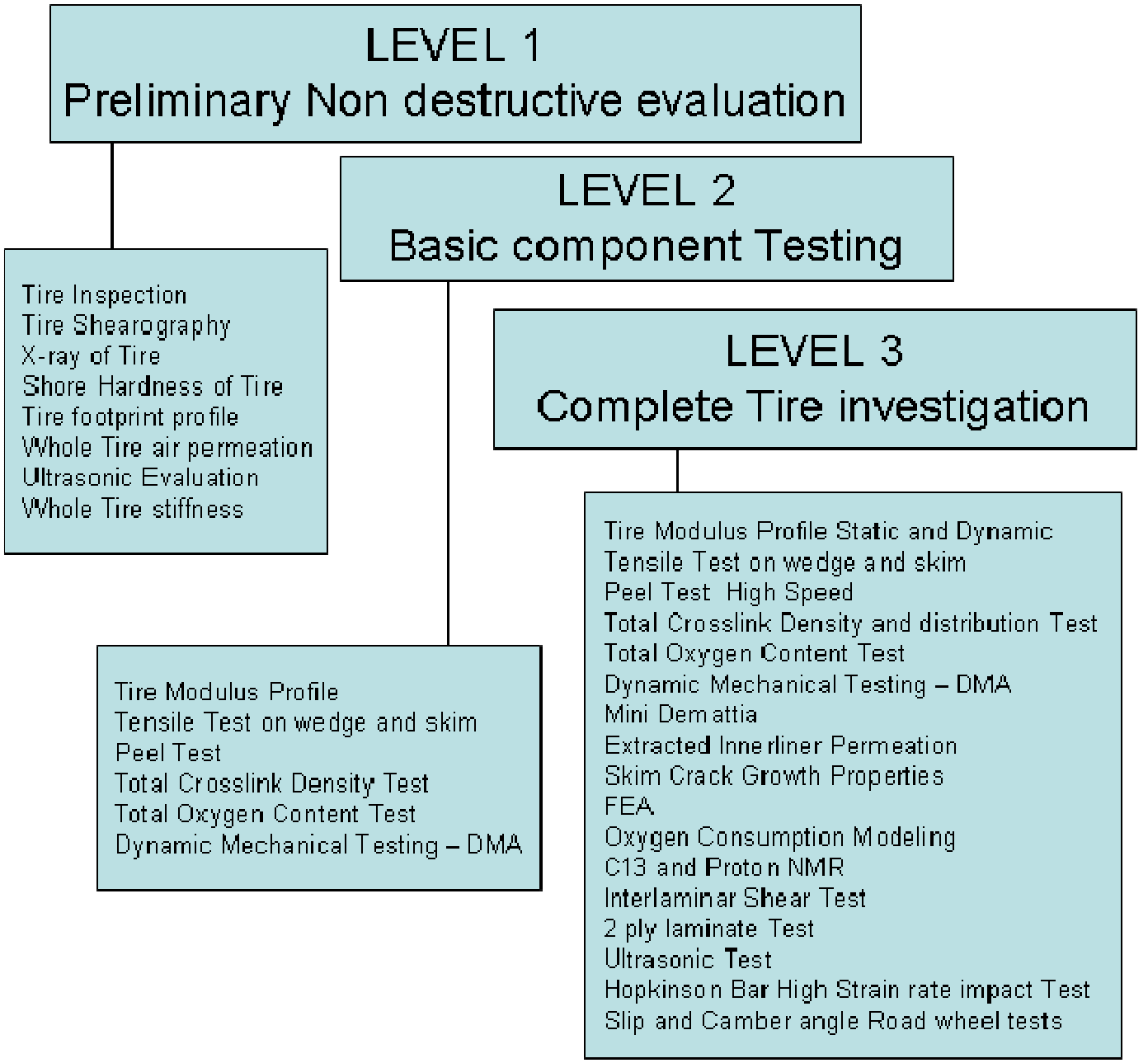
Methodology

- Problem Definition
  - Industry-Wide, Political, Costly, Expediency
  - Geographic, Materials, Time, Mechanisms
- Assessing Robustness of New Radial Tires
  - Learning & Researching
  - Gathering Data
  - Designing Informative Tests
  - Establishing Field Benchmarks

What We Have Learned

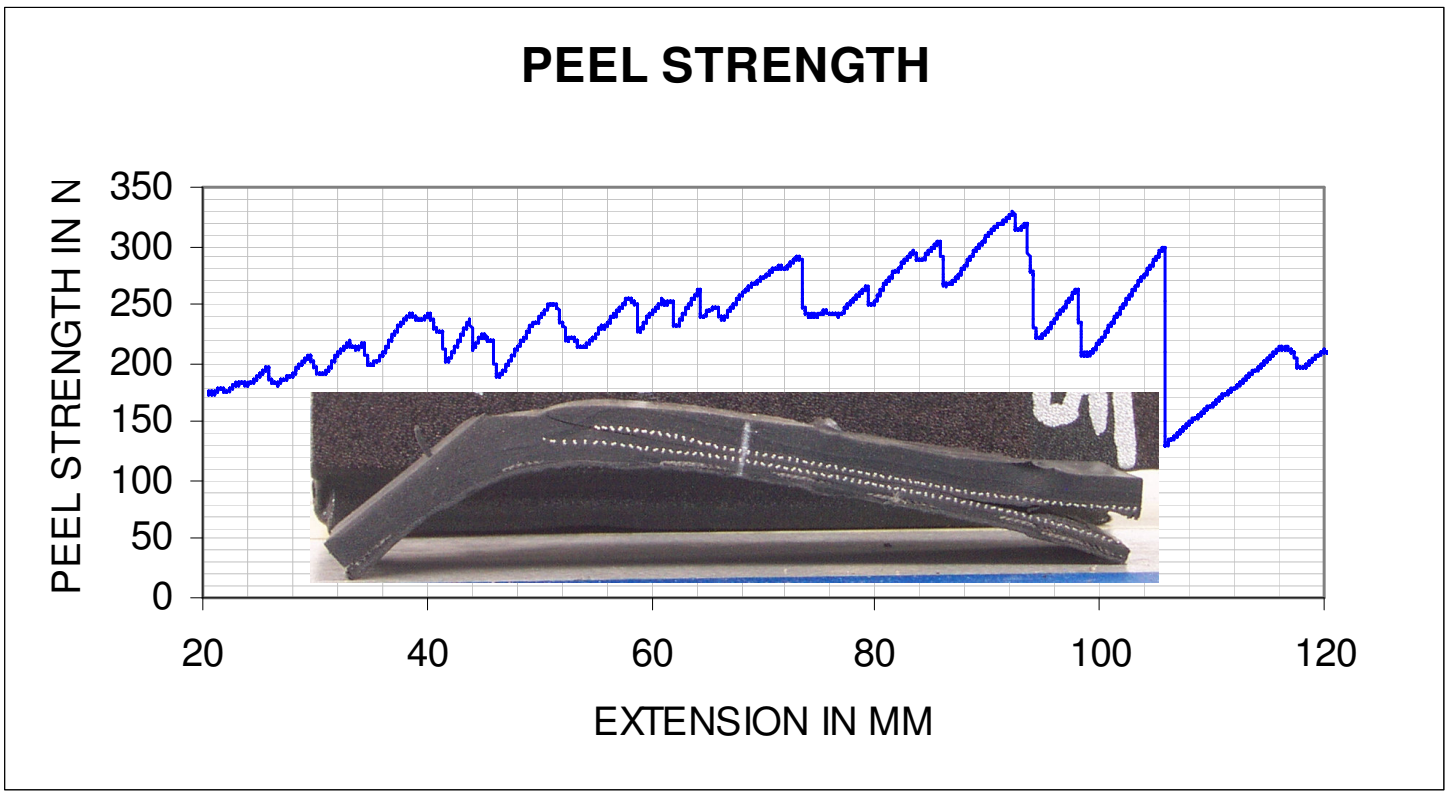
- How to Dissect Tires & Test Pieces
- How Not to Test Tires
- The Effects of Aging on Tires is Generally Understood, but the Chemistry and Physics of Tire Aging is Poorly Understood
- Accelerated Aging Methods on New Tires Must Include Both Thermo-Oxidative & Dynamic Aging Mechanisms

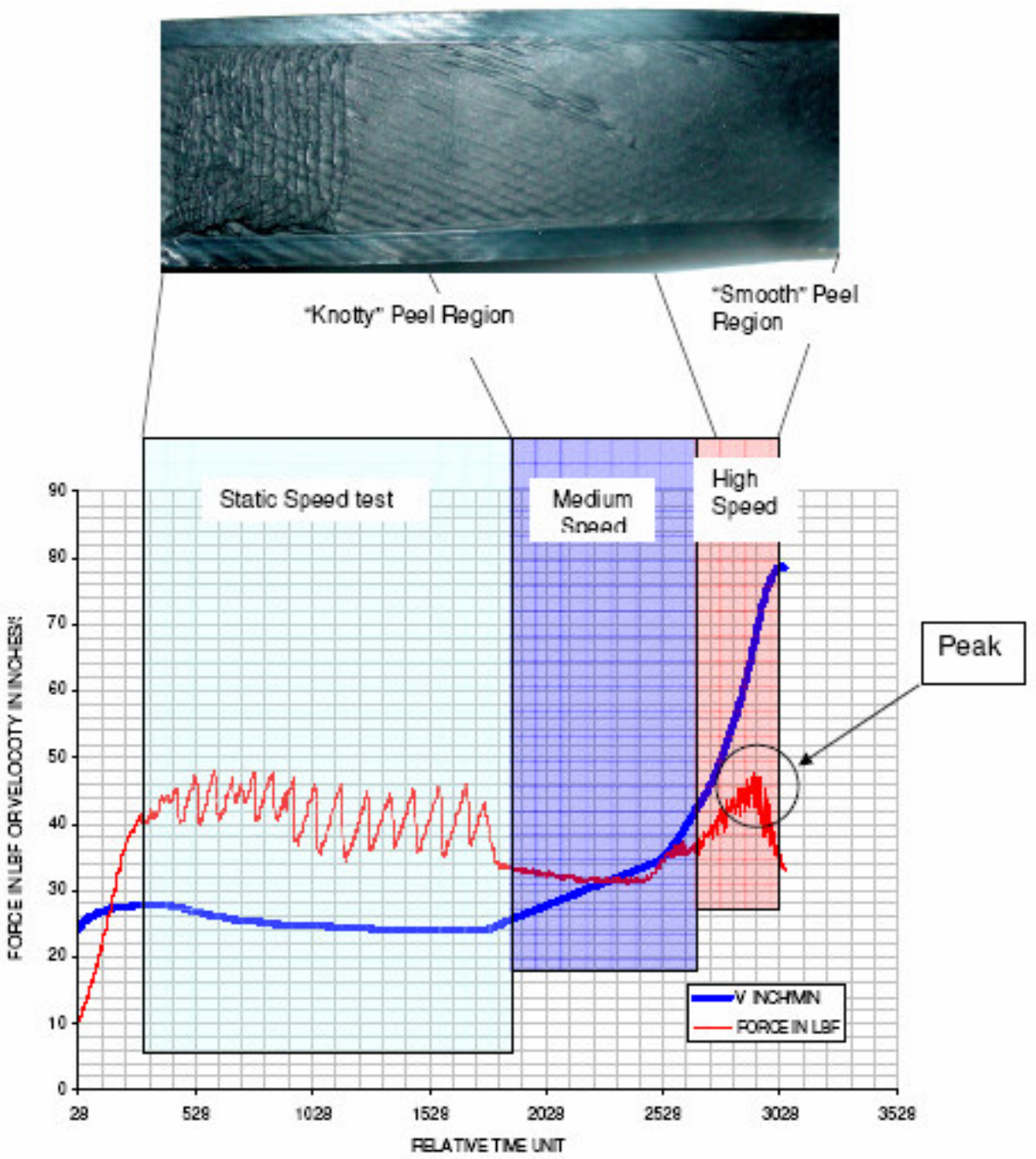
State of the Art


LEVEL 1
Preliminary Non destructive evaluation
Tire Inspection
Tire Shearography
X-ray of Tire
Shore Hardness of Tire
Tire footprint profile
Whole Tire air permeation
Ultrasonic Evaluation
Whole Tire stiffness
LEVEL 2
Basic component Testing
Tire Modulus Profile
Tensile Test on wedge and skim
Peel Test
Total Crosslink Density Test
Total Oxygen Content Test
Dynamic Mechanical Testing – DMA
LEVEL 3
Complete Tire investigation
Tire Modulus Profile Static and Dynamic
Tensile Test on wedge and skim
Peel Test High Speed
Total Crosslink Density and distribution Test
Total Oxygen Content Test
Dynamic Mechanical Testing – DMA
Mini Demattia
Extracted Innerliner Permeation
Skim Crack Growth Properties
FEA
Oxygen Consumption Modeling
C13 and Proton NMR
Interlaminar Shear Test
2 ply laminate Test
Ultrasonic Test
Hopkinson Bar High Strain rate impact Test
Slip and Camber angle Road wheel tests

Peel Strength

- Good indicator of the extent of oxidative aging of the skim and wedge

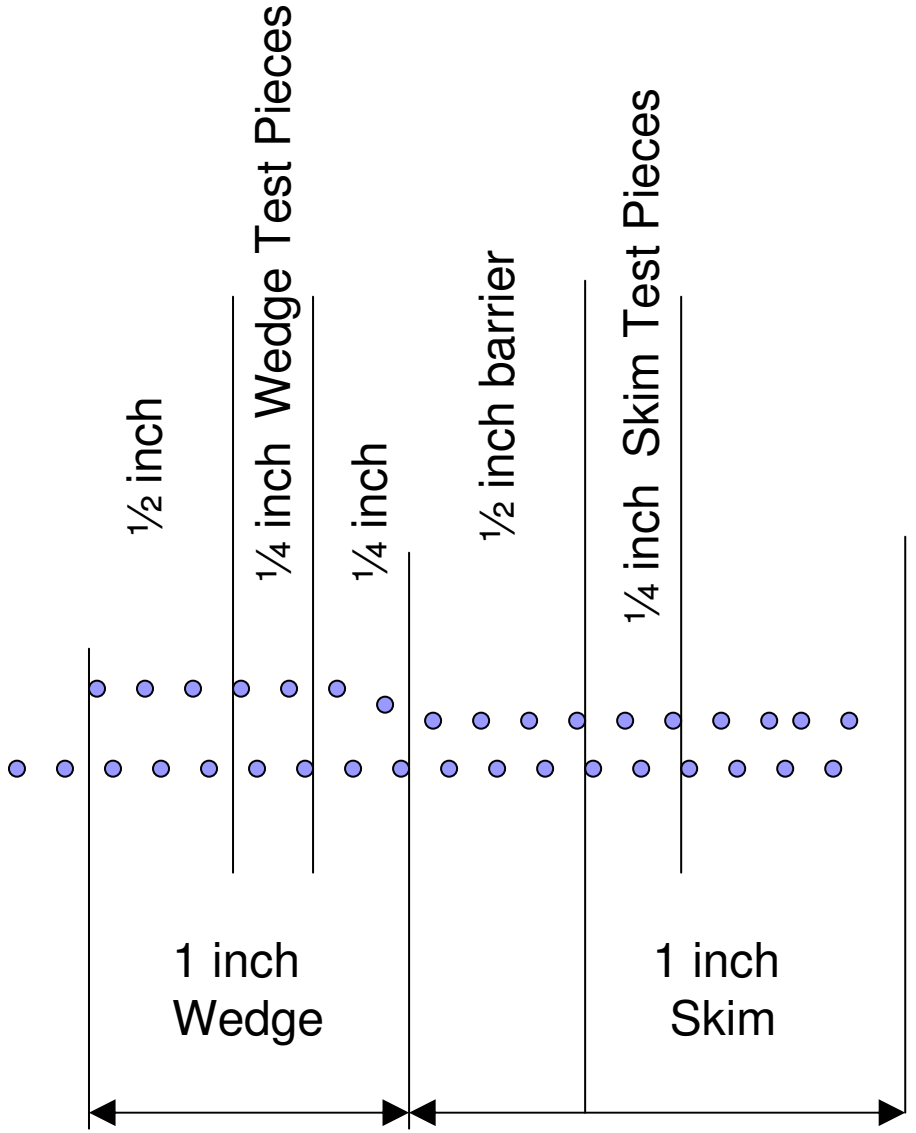




VARIABLE RATE CIRCUMFERENTIAL PEEL TEST

High Speed Peel Test

Total Crosslink Density



- Chemical Tests conducted to validate that the mechanism of aging is oxidative
- Very good correlation with Peel strength

Illustrative study

Study Goals

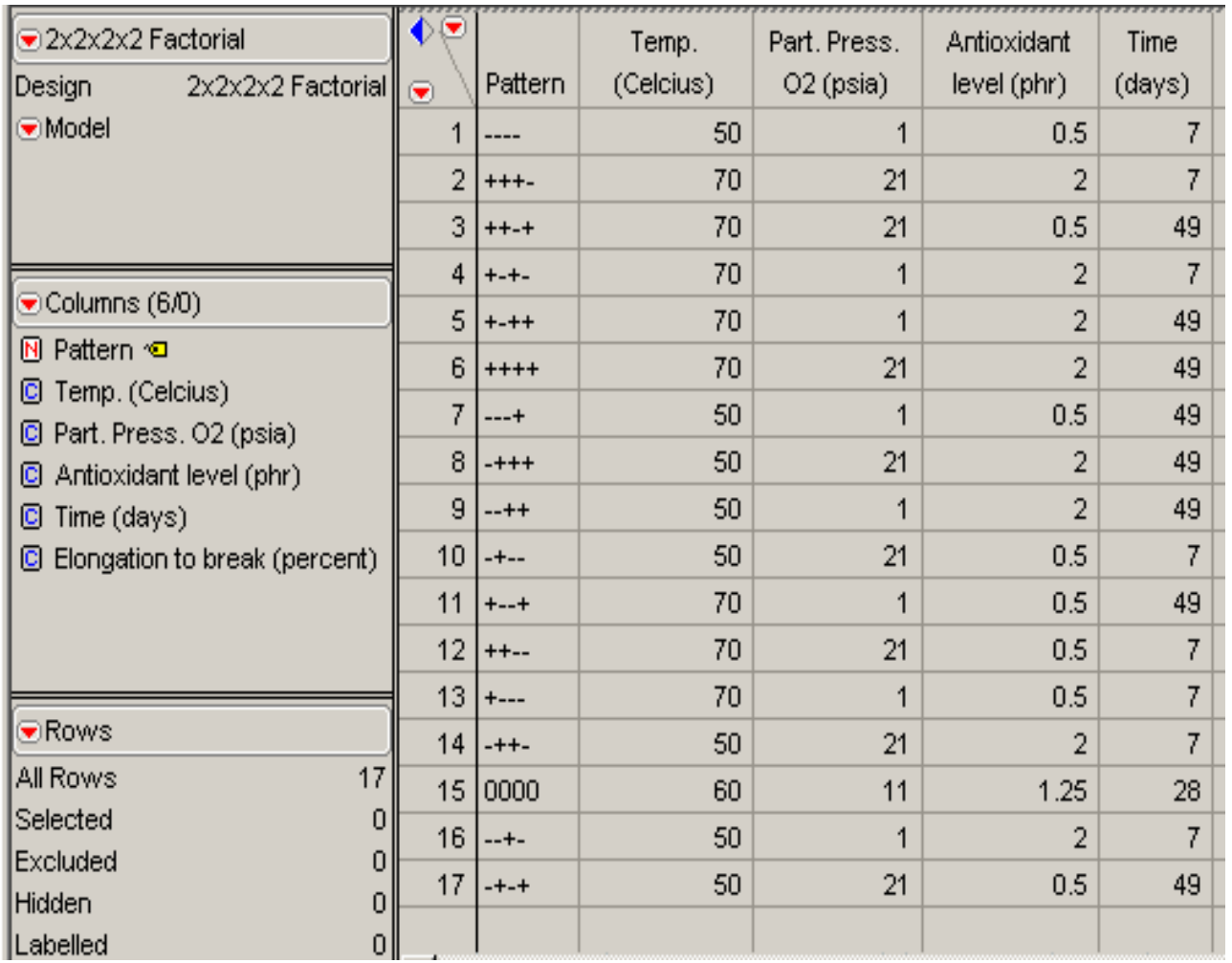
- Quantify the effect of partial pressure of oxygen, temperature, time on tire durability
- Generate data to understand the effect of aging on tire belt edge crack growth initiation and propagation.

Design of experiments

- Optimal usage of time and materials
- 2 level superimposed design
- Added benefit of studying model compounds aged in the tire cavity
- The **performance test** is FMVSS 139 Stepped up load test to failure
- The **performance parameter** is internal belt edge defects in the tires as they progress along the performance test to failure.
- The performance parameter is measured using nondestructive technique of **shearography and circumferential cuts**

Tire reactor ID	Temperature in C	Actual Aging time in days	Partial Pressure of O2 in psia	Tire Id pressur psig
3	50	49	21	80
4	70	49	1	35
2	70	49	21	74
1	50	49	1	35
5	60	28	10	66
6	70	7	1	35
7	50	7	1	35
8	70	7	21	74
9	50	7	21	80

Design of Experiment



2x2x2x2 Factorial

Design 2x2x2x2 Factorial

Model

Columns (6/0)

- Pattern
- Temp. (Celcius)
- Part. Press. O2 (psia)
- Antioxidant level (phr)
- Time (days)
- Elongation to break (percent)

Rows

All Rows	17
Selected	0
Excluded	0
Hidden	0
Labelled	0

	Pattern	Temp. (Celcius)	Part. Press. O2 (psia)	Antioxidant level (phr)	Time (days)
1	----	50	1	0.5	7
2	+++-	70	21	2	7
3	++-+	70	21	0.5	49
4	+-+-	70	1	2	7
5	+-++	70	1	2	49
6	++++	70	21	2	49
7	---+	50	1	0.5	49
8	-+++	50	21	2	49
9	--++	50	1	2	49
10	-+--	50	21	0.5	7
11	+--+	70	1	0.5	49
12	++--	70	21	0.5	7
13	+---	70	1	0.5	7
14	-++-	50	21	2	7
15	0000	60	11	1.25	28
16	--+-	50	1	2	7
17	-+-+	50	21	0.5	49

Results

Response D-Elongation to break (percent)

Parameter Estimates

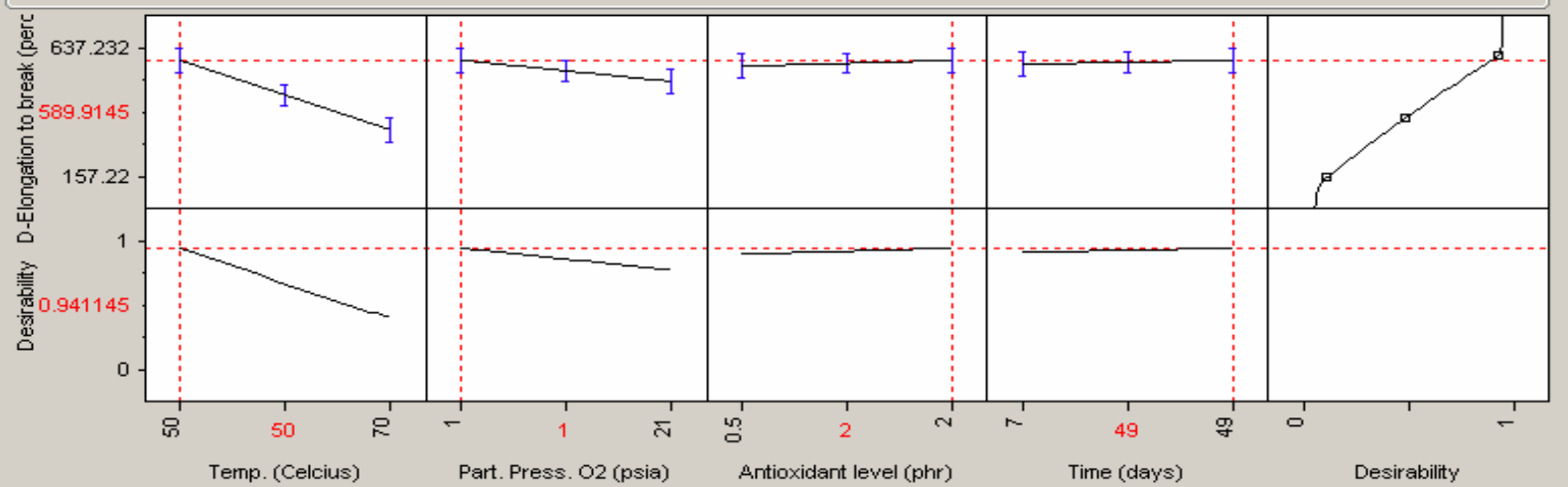
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Temp. (Celcius)(50,70)	1	1	131952.38	241.2955	<.0001
Part. Press. O2 (psia)(1,21)	1	1	16981.35	31.0530	0.0014
Antioxidant level (phr)(0.5,2)	1	1	2895.25	5.2944	0.0610
Time (days)(7,49)	1	1	67242.97	122.9643	<.0001
Temp. (Celcius)(50,70)*Part. Press. O2 (psia)(1,21)	1	1	3386.95	6.1936	0.0472
Temp. (Celcius)(50,70)*Antioxidant level (phr)(0.5,2)	1	1	0.11	0.0002	0.9893
Part. Press. O2 (psia)(1,21)*Antioxidant level (phr)(0.5,2)	1	1	0.21	0.0004	0.9850
Temp. (Celcius)(50,70)*Time (days)(7,49)	1	1	45670.90	83.5164	<.0001
Part. Press. O2 (psia)(1,21)*Time (days)(7,49)	1	1	7378.38	13.4925	0.0104
Antioxidant level (phr)(0.5,2)*Time (days)(7,49)	1	1	117.23	0.2144	0.6597

Effect Details

Scaled Estimates

Prediction Profiler

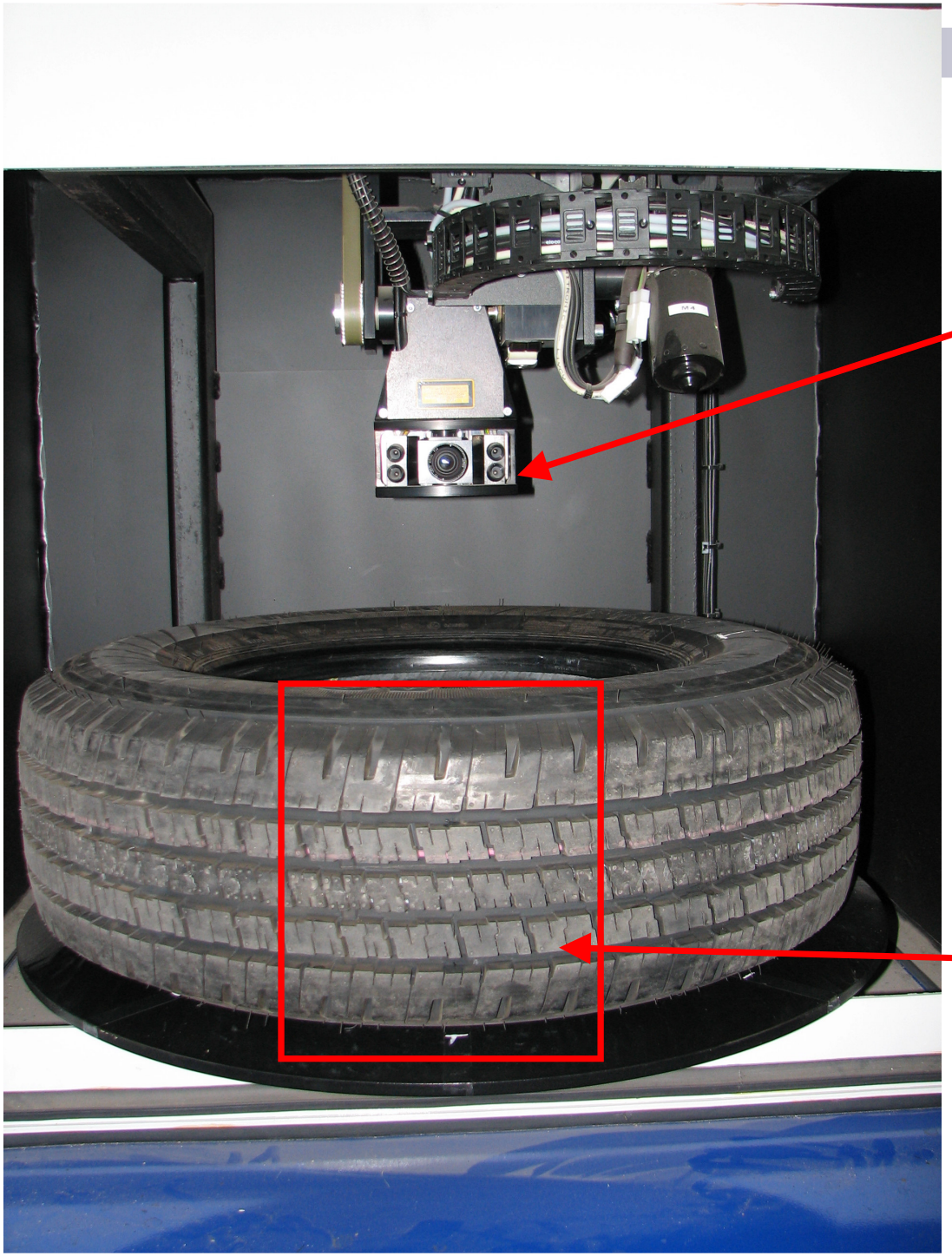


ARDL Shearography Principles

- Tire testing is done by applying a small external vacuum to the tire.
- The testing done at an angle to focus on the belt edge
- Through this way invisible separations inside the tire show up as bulges.
- These structure defects are detected by an interferometric measurement process.
- This test procedure is non destructive

Procedure

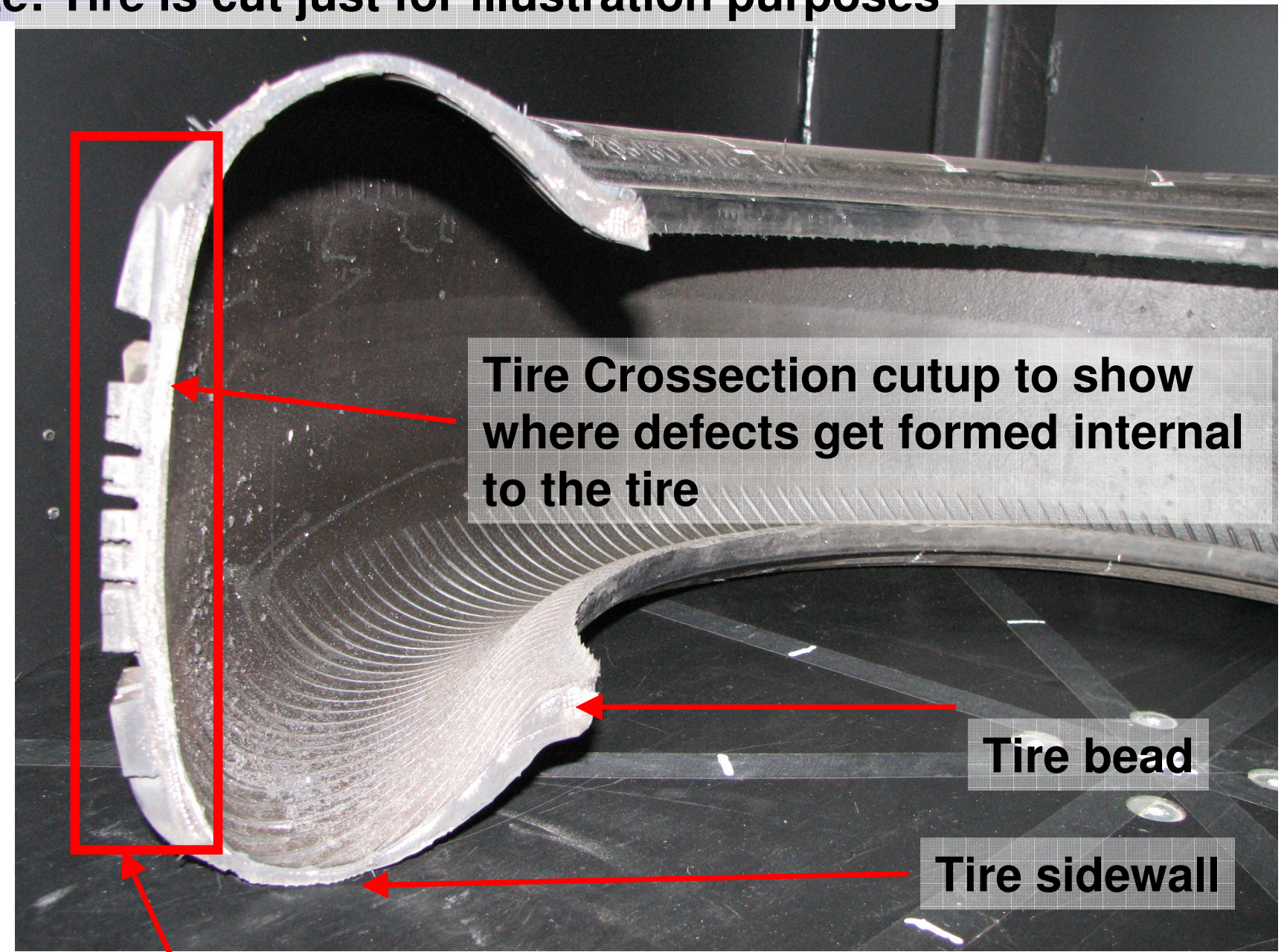
- A full tire is placed inside the vacuum chamber of the machine.
- The machine is setup to view the critical areas of the tire “Belt Edge Defects”
- The machine software runs a program that measures the surface deformations due to internal defects before and after applying the vacuum and give a 360 degree view of the internal defects in the tire.

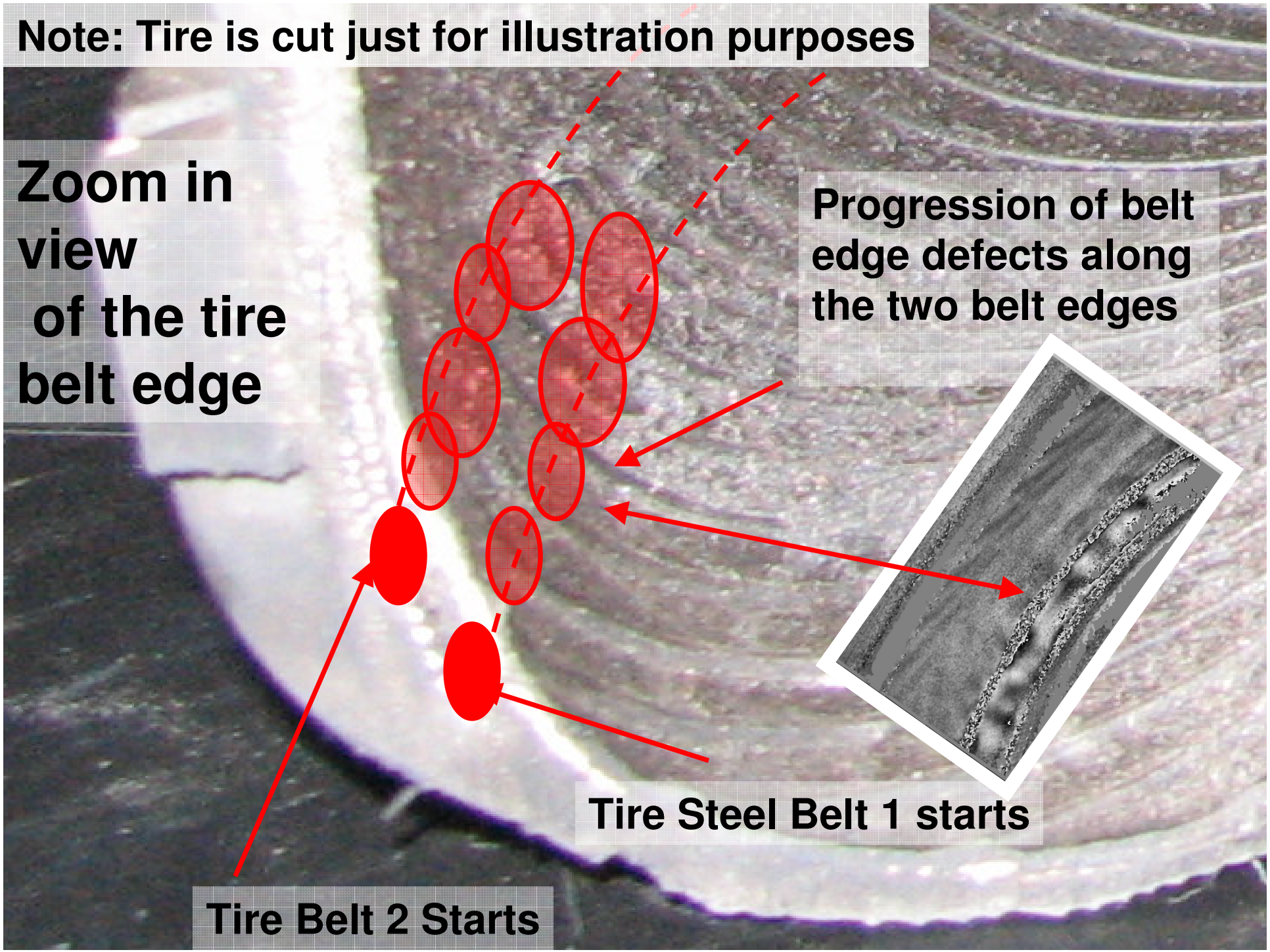


ARDL laser head configured to identify and follow progression of belt edge defects inside tires

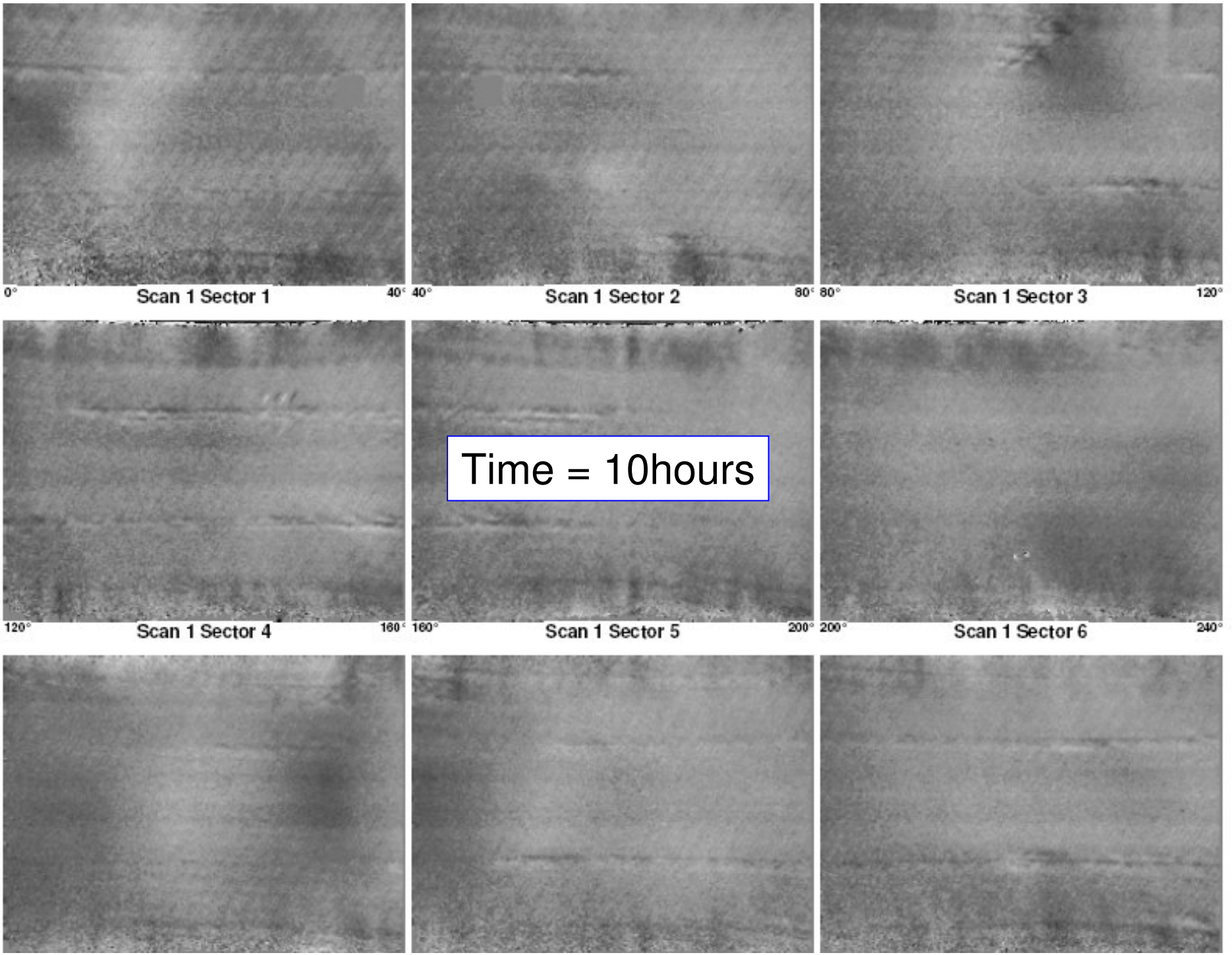
Sector of the tire under inspection by laser

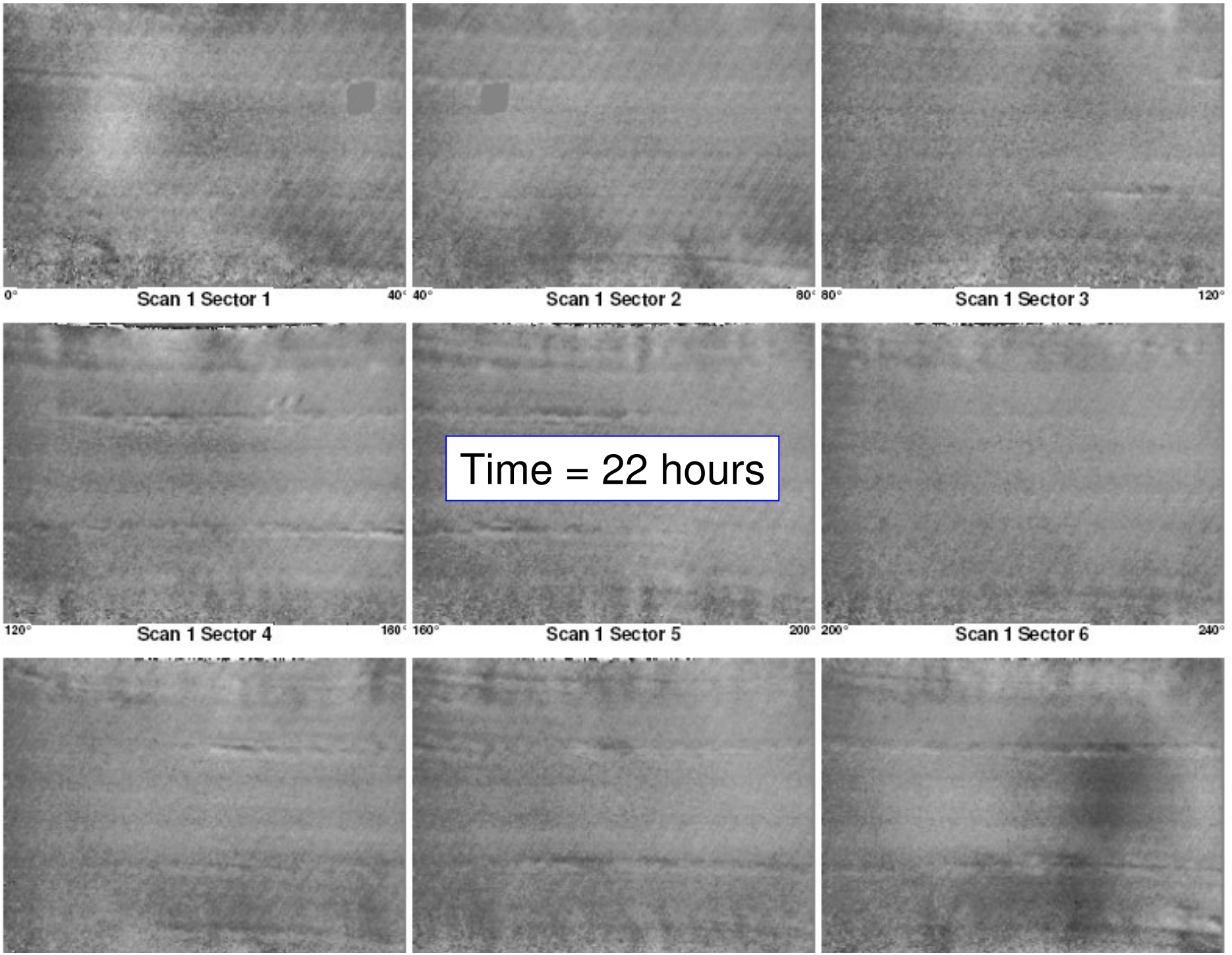
Note: Tire is cut just for illustration purposes

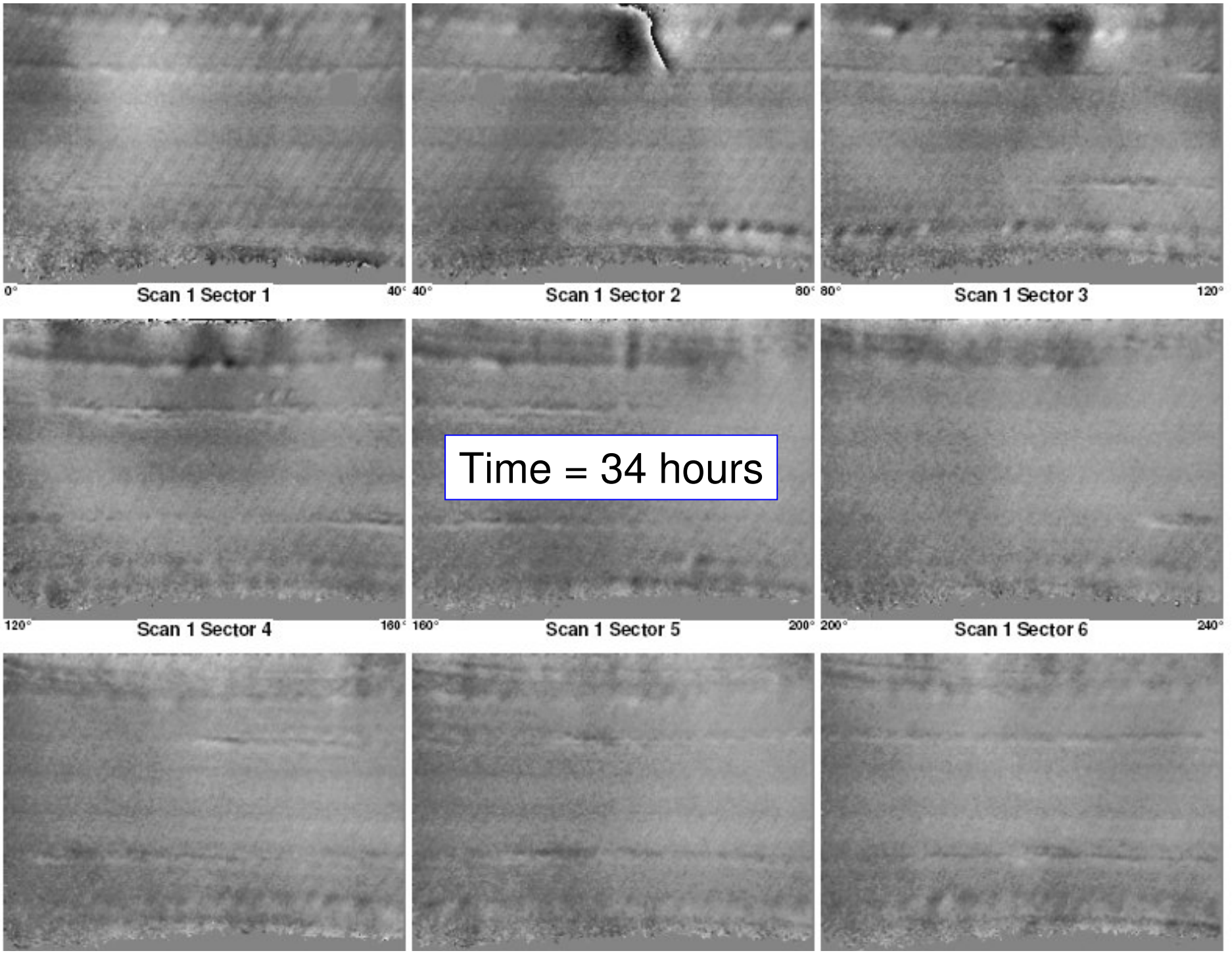


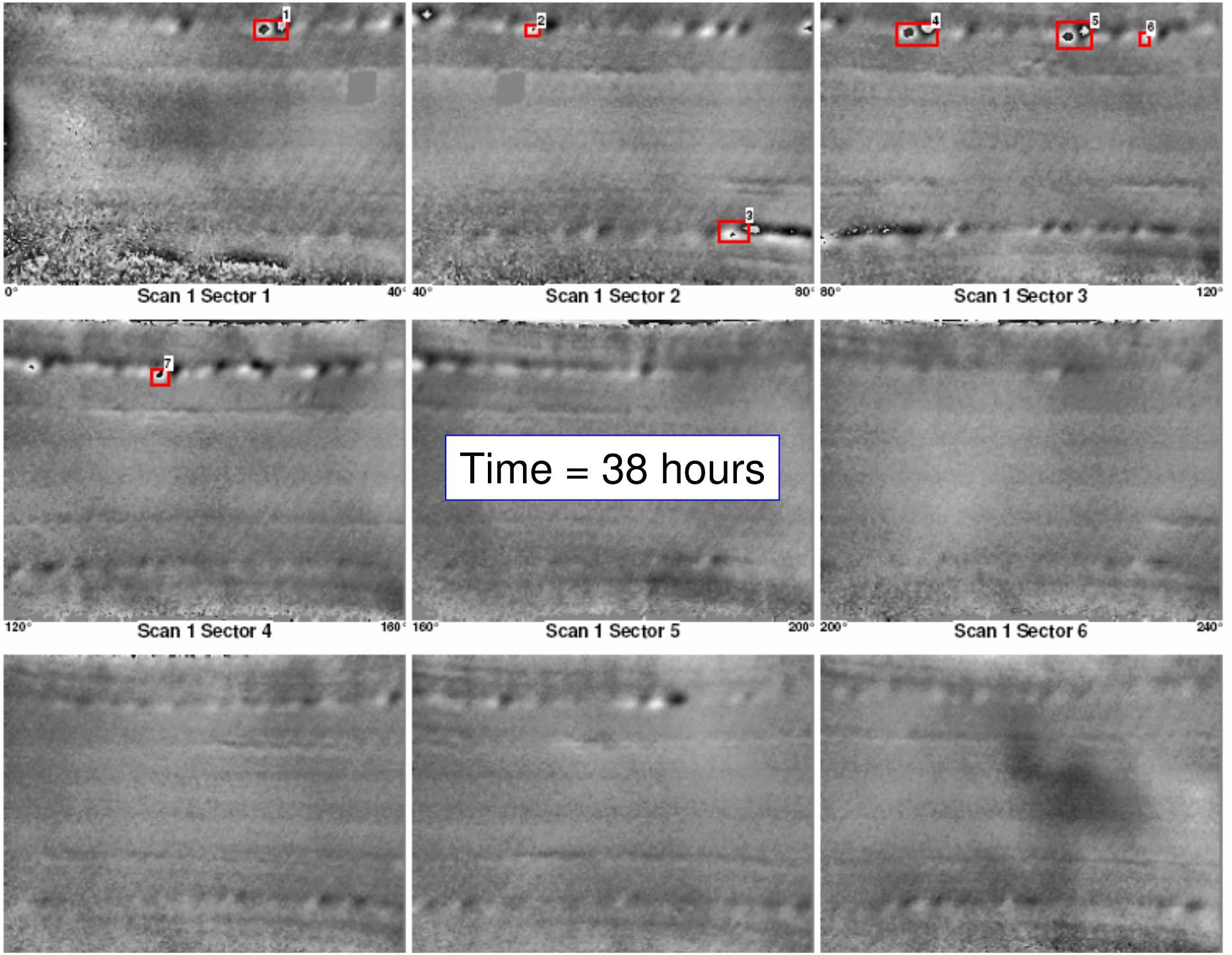

Note: Tire is cut just for illustration purposes
Zoom in view of the tire belt edge
Progression of belt edge defects along the two belt edges
Tire Steel Belt 1 starts
Tire Belt 2 Starts

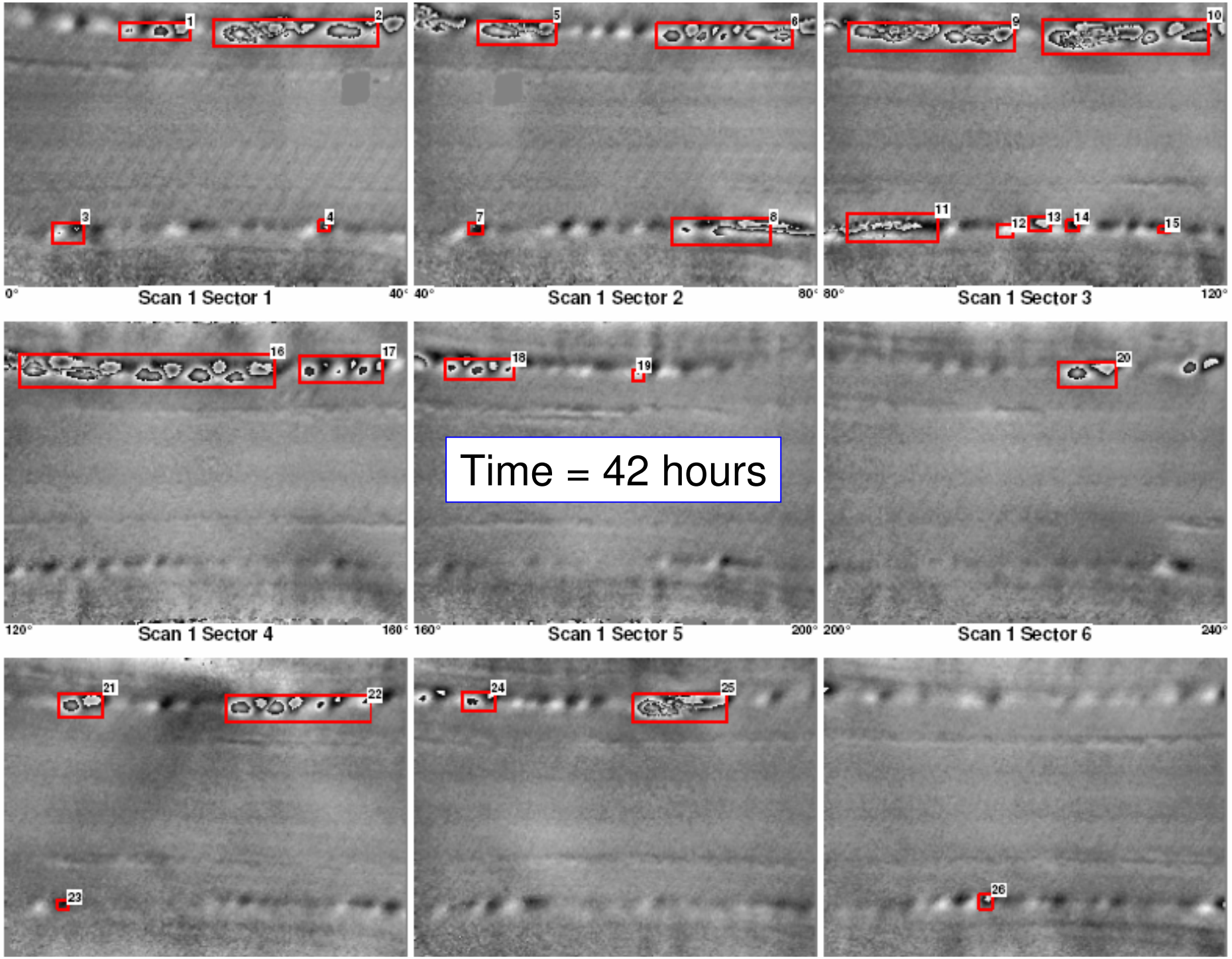
EFFECT OF TEST TIME


0°
Scan 1 Sector 1
40°
40°
Scan 1 Sector 2
80°
80°
Scan 1 Sector 3
120°
120°
Scan 1 Sector 4
160°
Time = 10hours
160°
Scan 1 Sector 5
200°
200°
Scan 1 Sector 6
240°

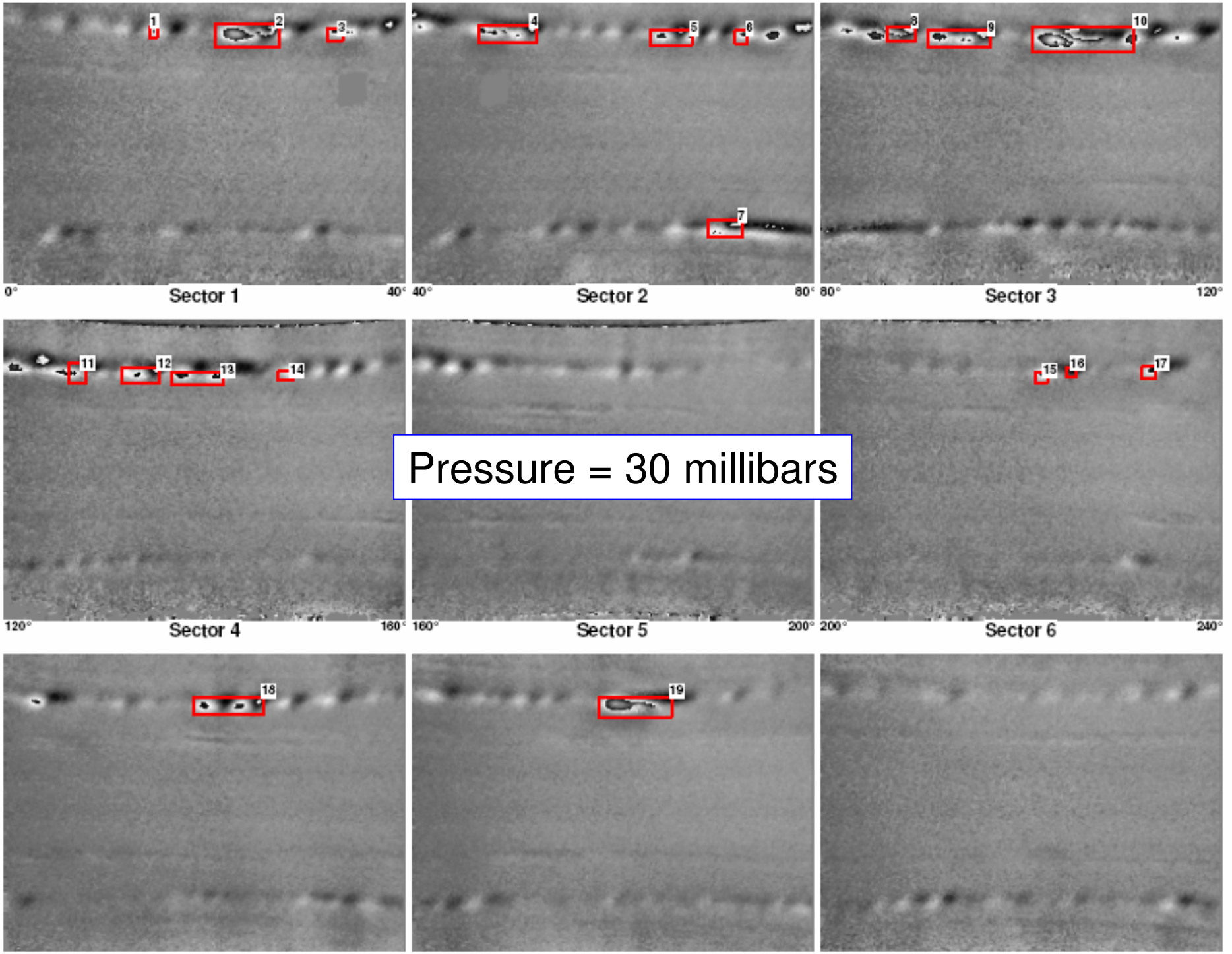

0°
Scan 1 Sector 1
40°
40°
Scan 1 Sector 2
80°
80°
Scan 1 Sector 3
120°
120°
Scan 1 Sector 4
160°
Time = 22 hours
160°
Scan 1 Sector 5
200°
200°
Scan 1 Sector 6
240°

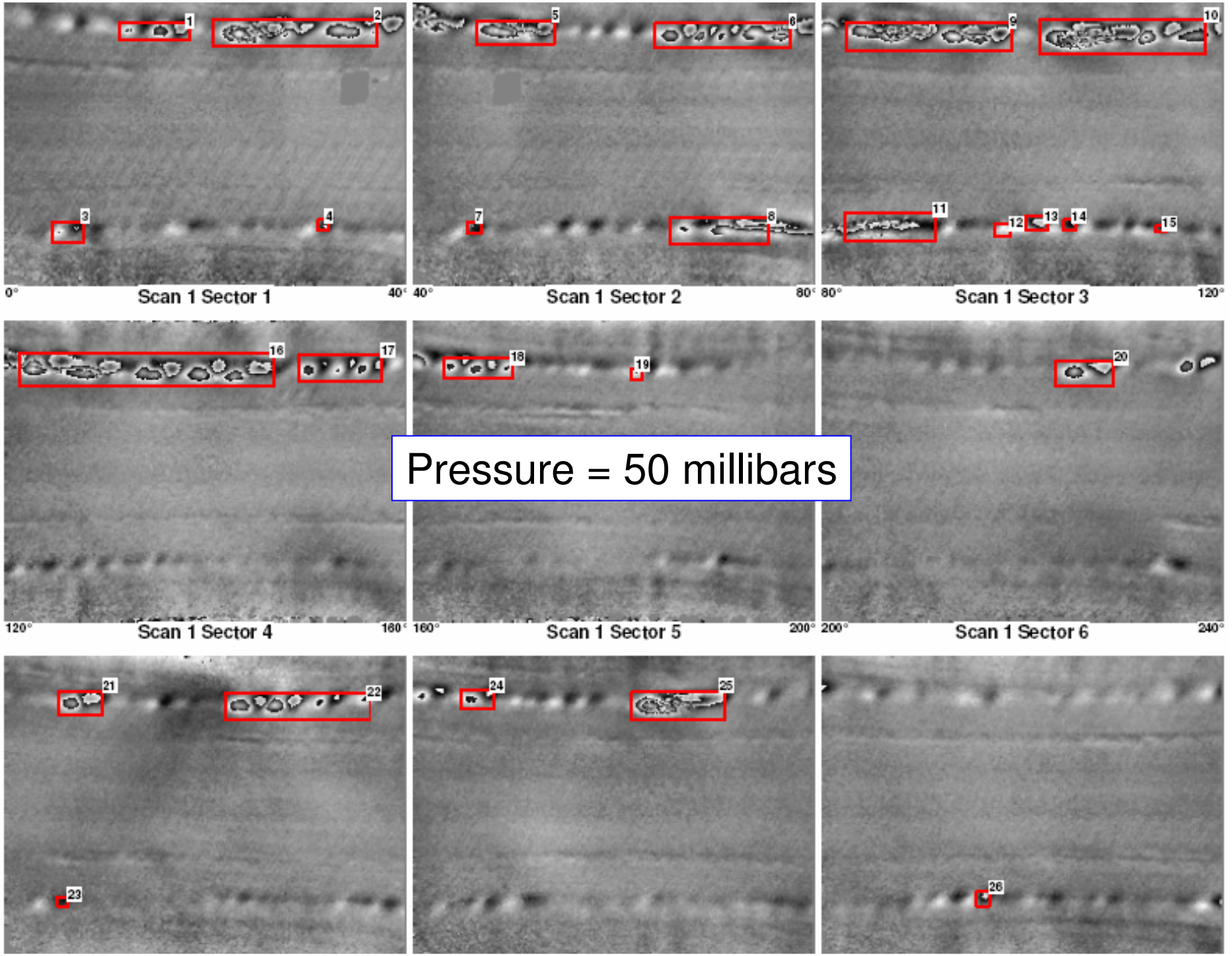

0°
Scan 1 Sector 1
40°
40°
Scan 1 Sector 2
80°
80°
Scan 1 Sector 3
120°
120°
Scan 1 Sector 4
160°
Time = 34 hours
160°
Scan 1 Sector 5
200°
200°
Scan 1 Sector 6
240°

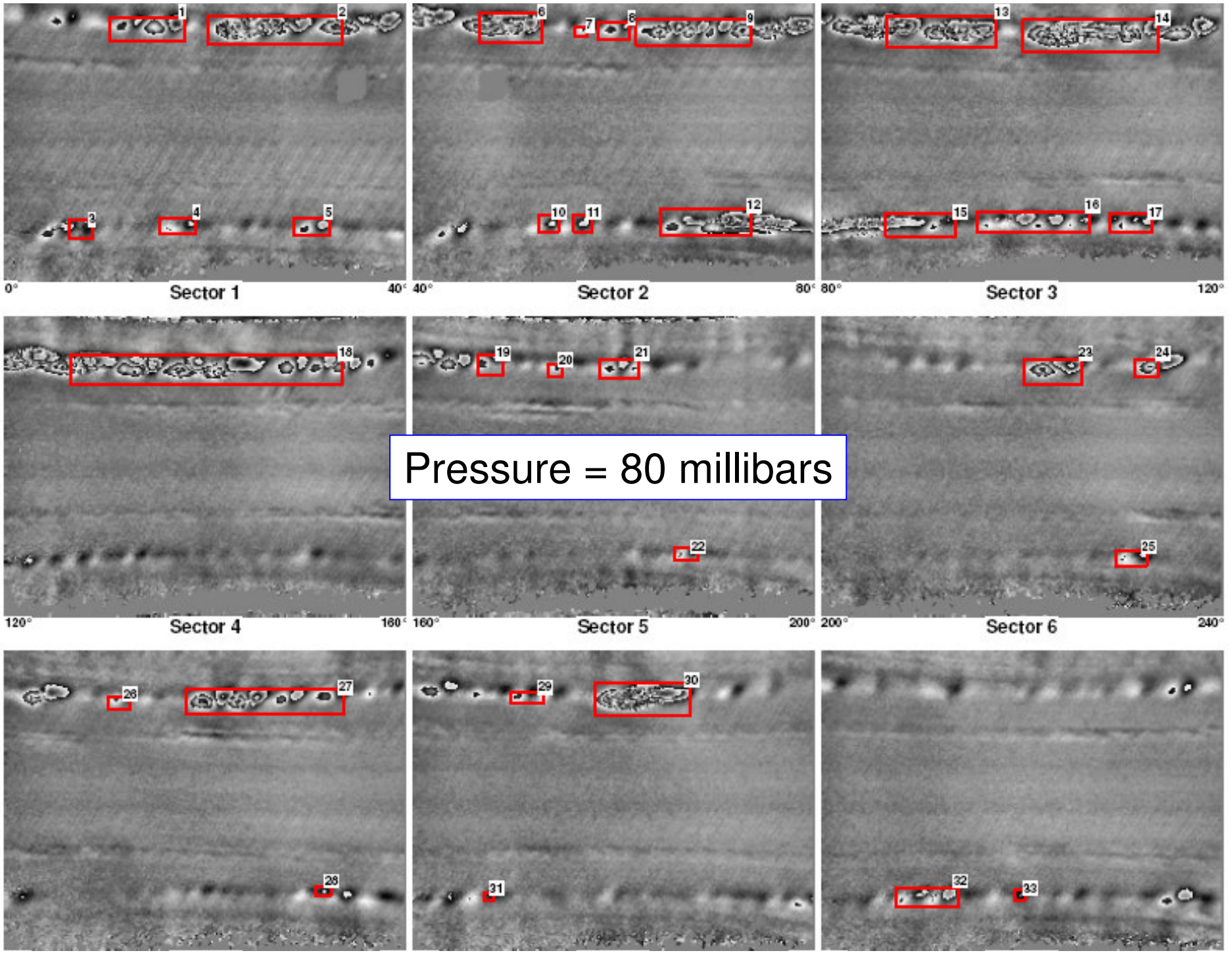

1
2
3
4
5
6
0°
Scan 1 Sector 1
40°
40°
Scan 1 Sector 2
80°
80°
Scan 1 Sector 3
120°
7
120°
Scan 1 Sector 4
160°
Time = 38 hours
160°
Scan 1 Sector 5
200°
200°
Scan 1 Sector 6
240°


1
2
3
4
0°
Scan 1 Sector 1
40°
5
6
7
8
40°
Scan 1 Sector 2
80°
9
10
11
12
13
14
15
80°
Scan 1 Sector 3
120°
16
17
120°
Scan 1 Sector 4
160°
18
19
Time = 42 hours
160°
Scan 1 Sector 5
200°
20
200°
Scan 1 Sector 6
240°
21
22
23
24
25
26

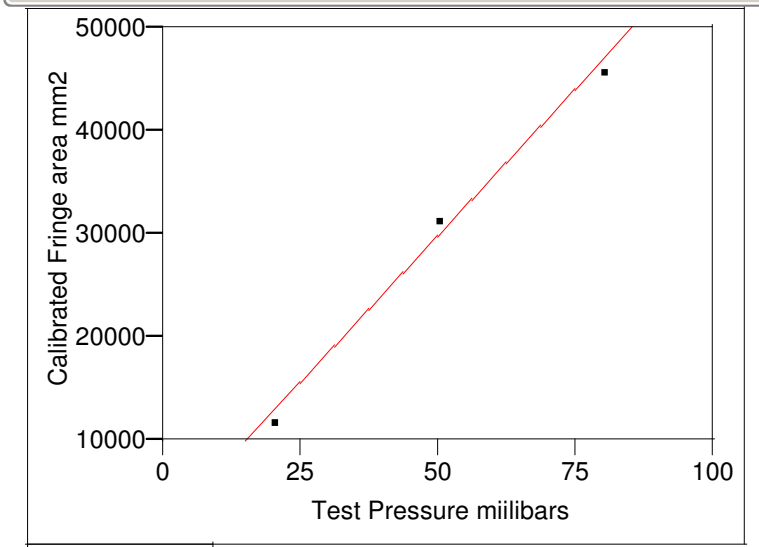
EFFECT OF TEST PRESSURE ON THE FRINGE PATTERNS AREA


1
2
3
4
5
6
7
8
9
10
0°
Sector 1
40°
40°
Sector 2
80°
80°
Sector 3
120°
11
12
13
14
15
16
17
120°
Sector 4
160°
160°
Sector 5
200°
200°
Sector 6
240°
18
19
Pressure = 30 millibars


1
2
3
4
0°
Scan 1 Sector 1
40°
5
6
7
8
40°
Scan 1 Sector 2
80°
9
10
11
12
13
14
15
80°
Scan 1 Sector 3
120°
16
17
120°
Scan 1 Sector 4
160°
18
19
160°
Scan 1 Sector 5
200°
20
200°
Scan 1 Sector 6
240°
21
22
23
24
25
26
Pressure = 50 millibars


Sector 1
Sector 2
Sector 3
Sector 4
Sector 5
Sector 6
Pressure = 80 millibars

Bivariate Fit of Calibrated Fringe area mm2 By Test Pressure miilibars



Linear Fit

Calibrated Fringe area mm2 = 1350.5 + 567.38333 Test Pressure miilibars

Summary of Fit

RSquare	0.992537
RSquare Adj	0.985074
Root Mean Square Error	2087.374
Mean of Response	29719.67
Observations (or Sum Wgts)	3

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	579462924	579462924	132.9919
Error	1	4357128	4357128.2	Prob > F
C. Total	2	583820053		0.0551

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1350.5	2739.333	0.49	0.7084
Test Pressure miilibars	567.38333	49.19987	11.53	0.0551

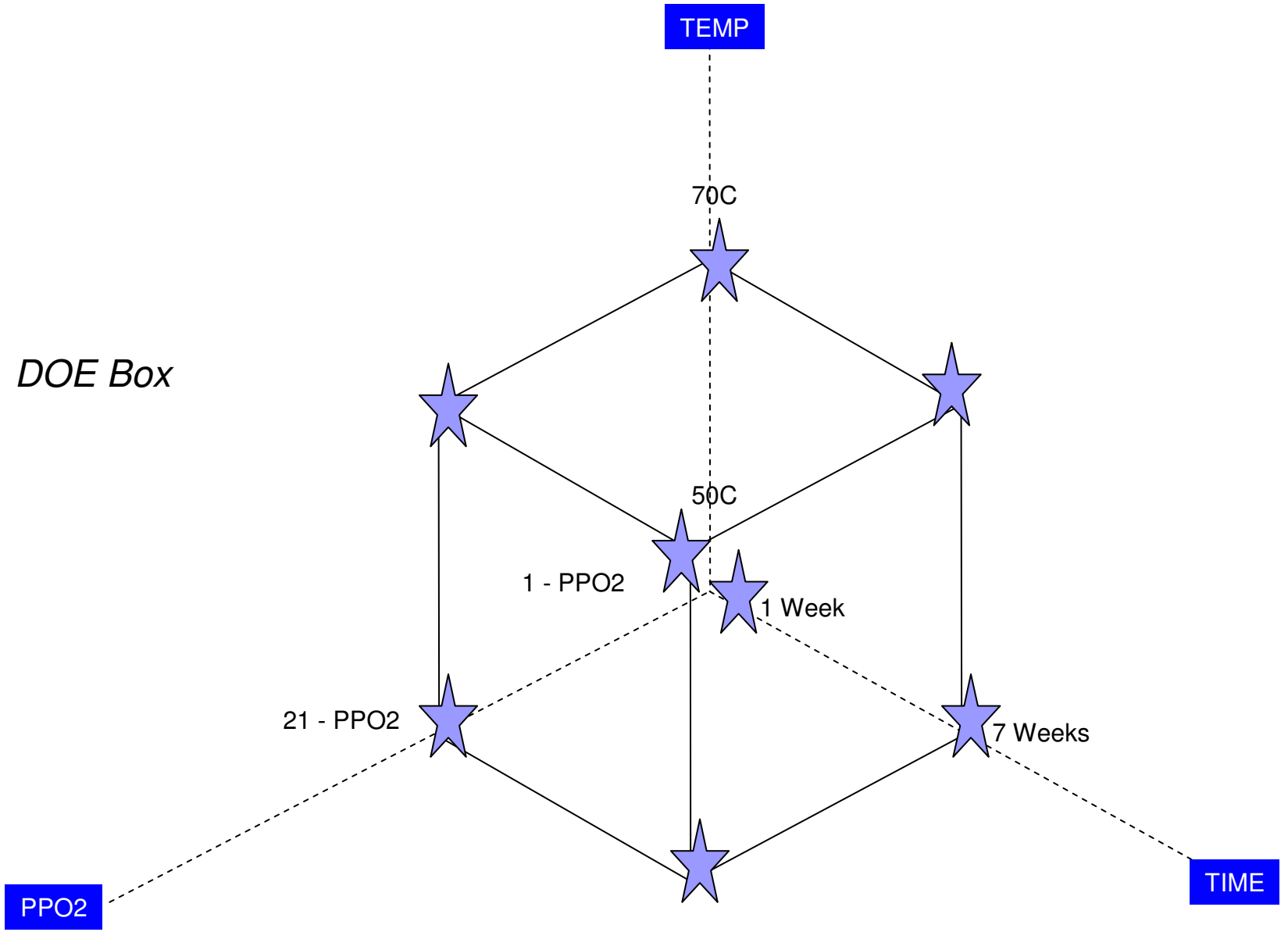
Preliminary Conclusion

- Test pressure affects fringe pattern area
- Test duration affects fringe pattern area
- Need to correlate the results to actual belt edge defects

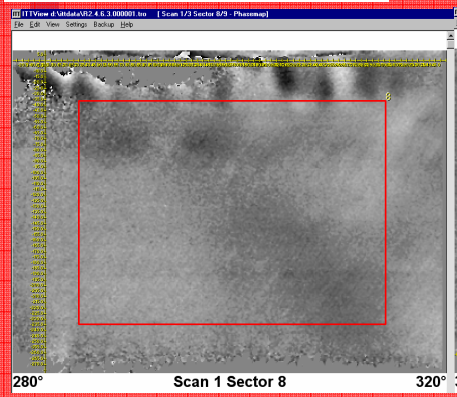
Results

- Study the effect of the 3 inputs of time temperature and partial pressure of oxygen on the belt edge fringe pattern area growth

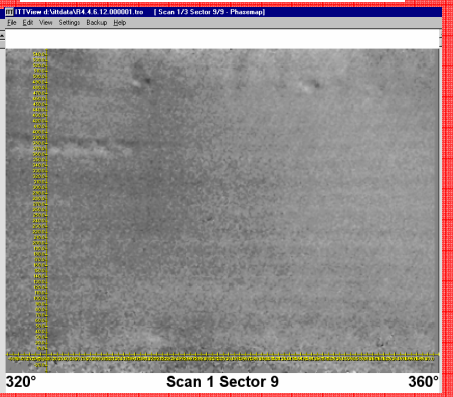
DOE Box



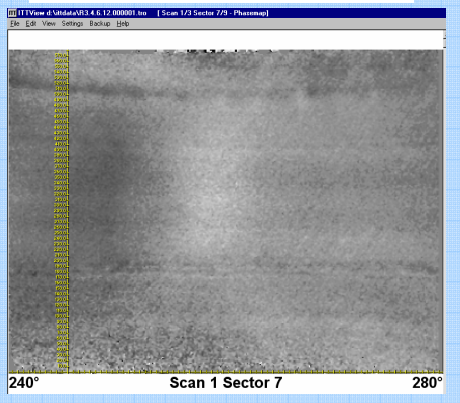
70C – 7WK – 21 PPO2



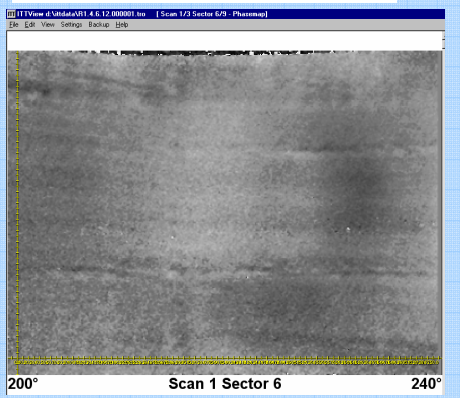
70C – 7 WK – 1 PPO2



50C – 7 WK – 21 PPO2



50C – 7 WK – 1 PPO2



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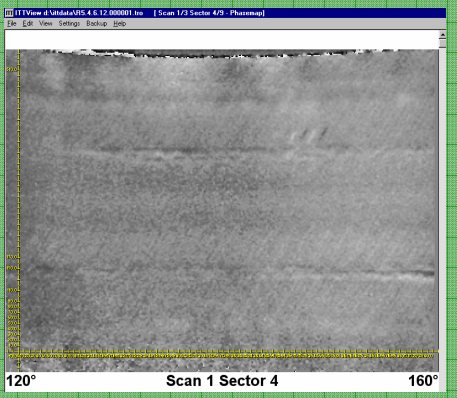
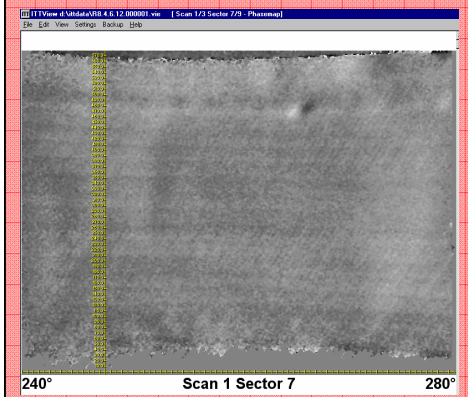
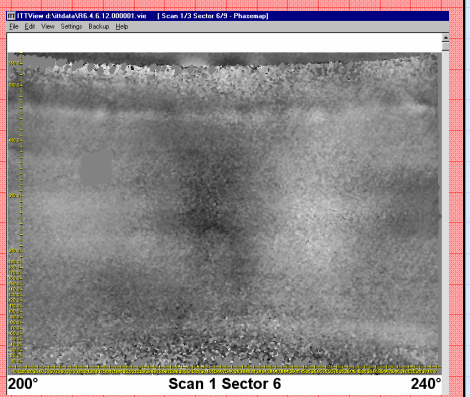


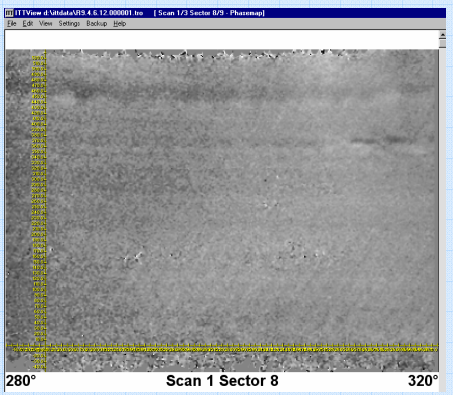
60C – 4WK – 11PPO2



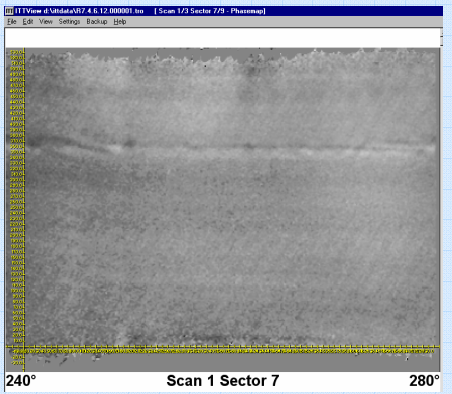
70C – 1 WK – 21 PPO2



70C – 1 WK – 1 PPO2

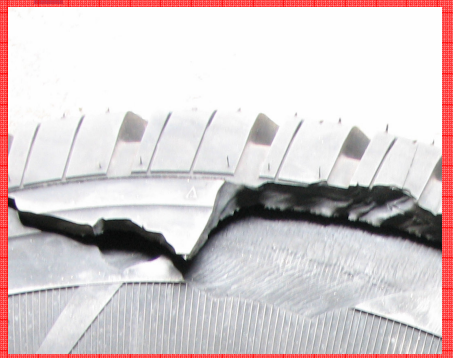


50C – 1 WK – 21 PPO2

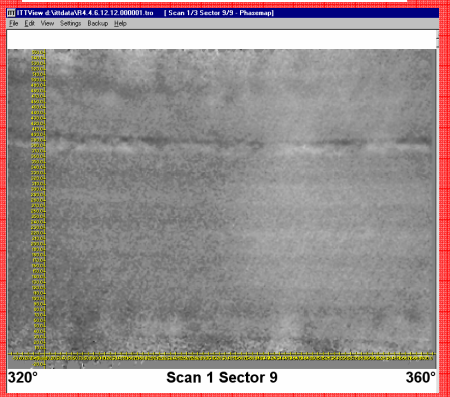


50C – 1 WK – 1 PPO2

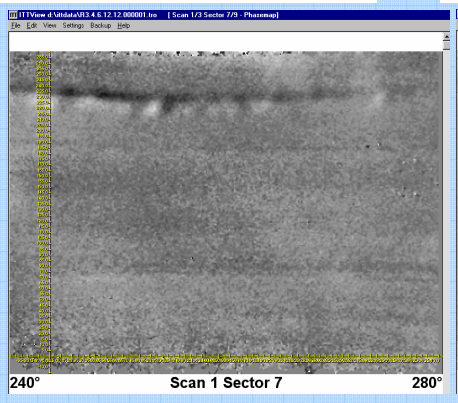
70C – 7WK – 21 PPO2



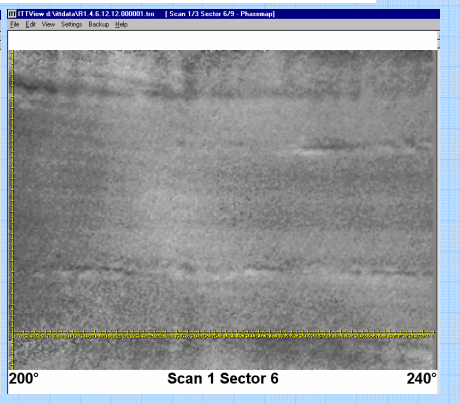
70C – 7 WK – 1 PPO2



50C – 7 WK – 21 PPO2



50C – 7 WK – 1 PPO2



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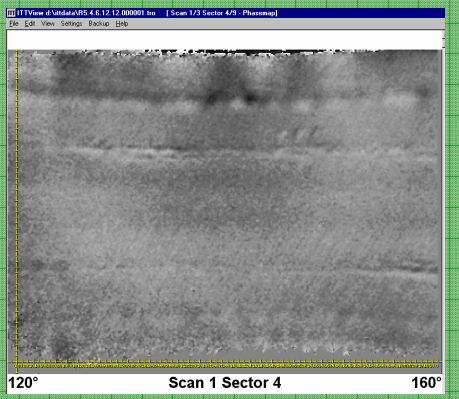
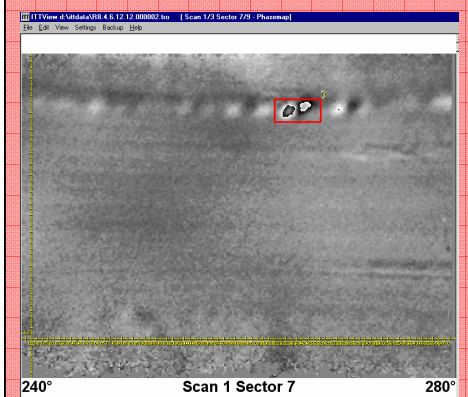
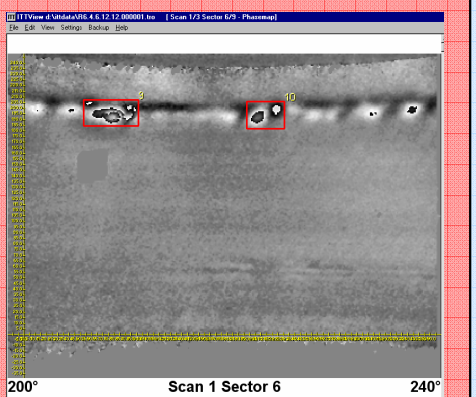


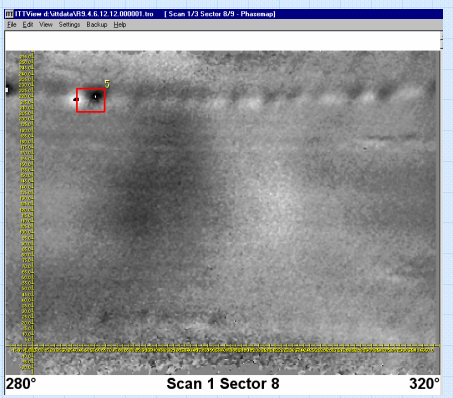
60C – 4WK – 11PPO2

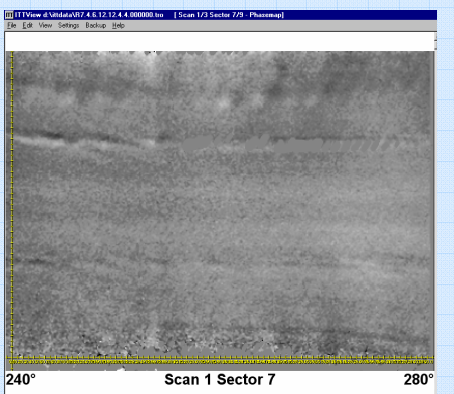




70C – 1 WK – 21 PPO2

70C – 1 WK – 1 PPO2

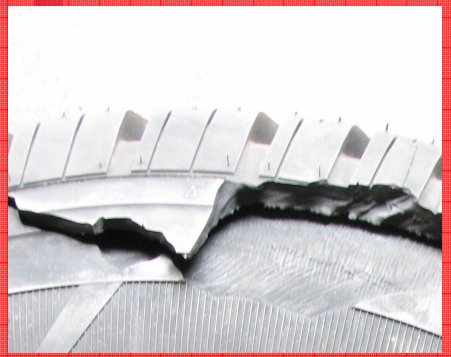




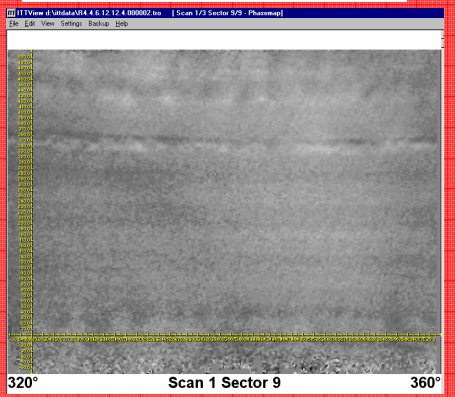
50C – 1 WK – 21 PPO2

50C – 1 WK – 1 PPO2

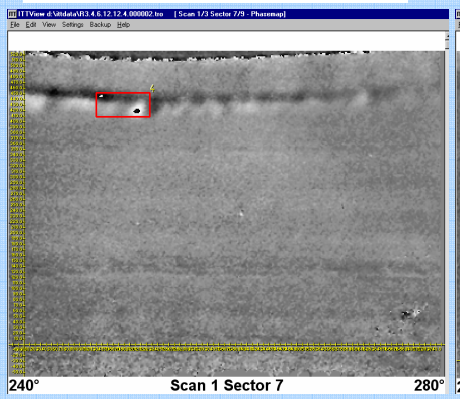
70C – 7WK – 21 PPO2



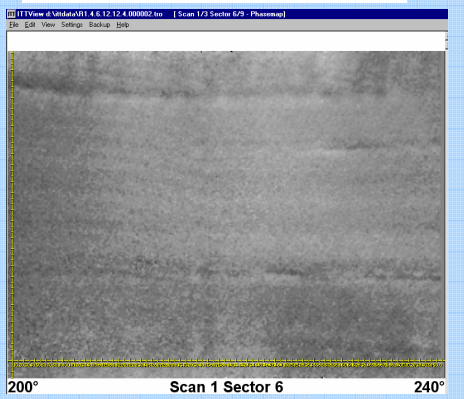
70C – 7 WK – 1 PPO2



50C – 7 WK – 21 PPO2



50C – 7 WK – 1 PPO2



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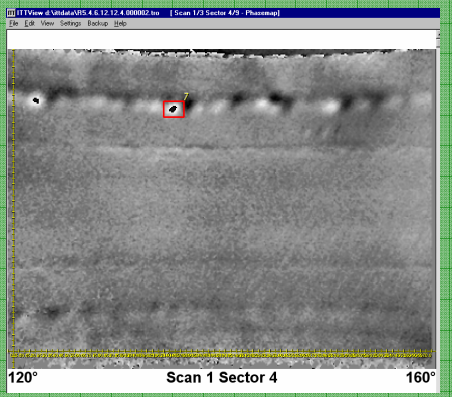
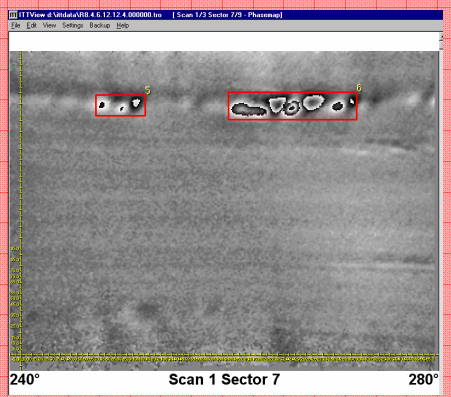
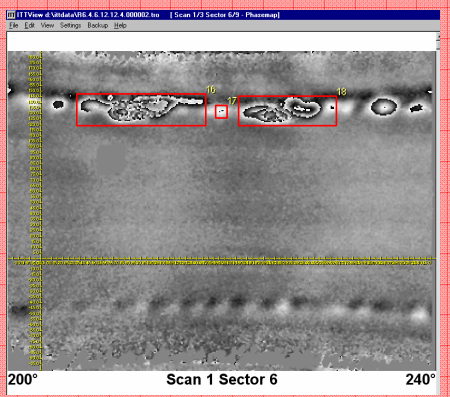


60C – 4WK – 11PPO2



70C – 1 WK – 21 PPO2



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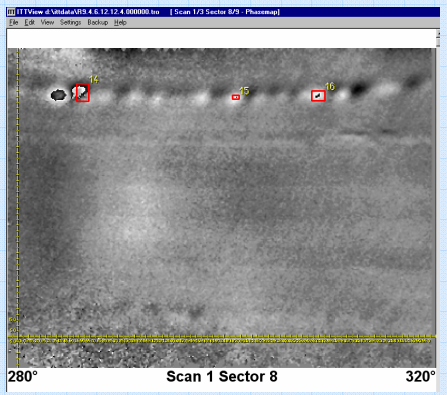

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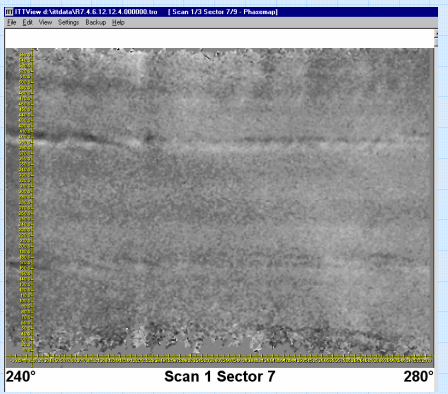

50C – 1 WK – 1 PPO2