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In-Service Testing:
Hot Surface Measurement with XRF

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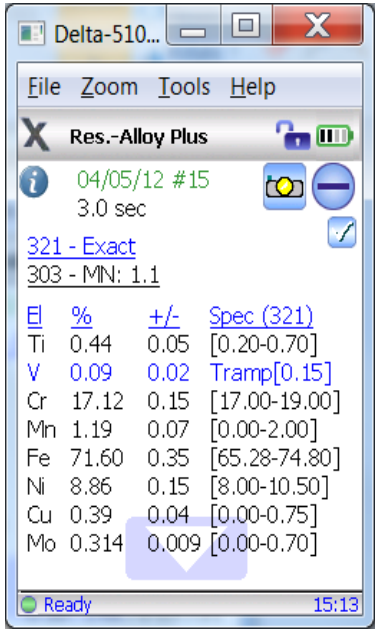
In-service testing: hot surface measurement with XRF

Agenda

- Positive Material Identification and in-service testing
- In-service testing challenges
- Handheld XRF innovations
- Application examples



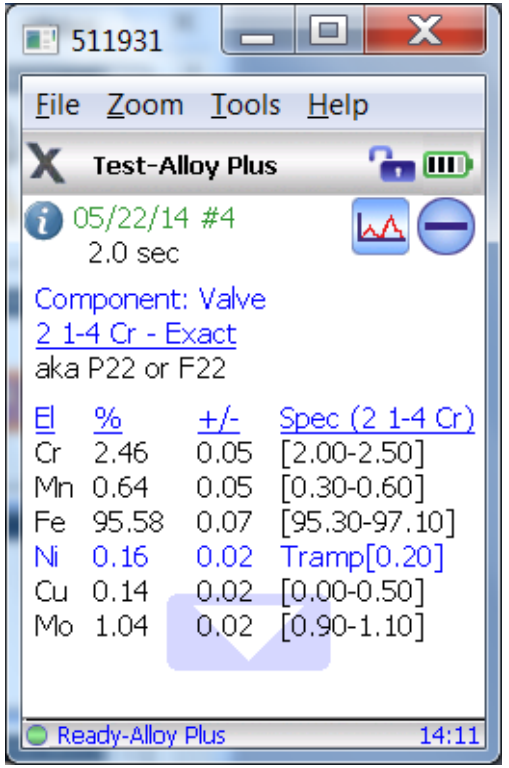
Handheld XRF for plant PMI

Positive Material Identification – Alloy grade & chemistry

- Test: pipes, flanges, valve components, welds
- Suppliers: Quality Control at receiving
- Repair: material verification for existing components

Good PMI protocol can prevent

- Mix-up
- Components failure
- Costly downtime
- Repair and replacement
- Environment and safety hazards



Handheld XRF for plant PMI

Handheld XRF benefits

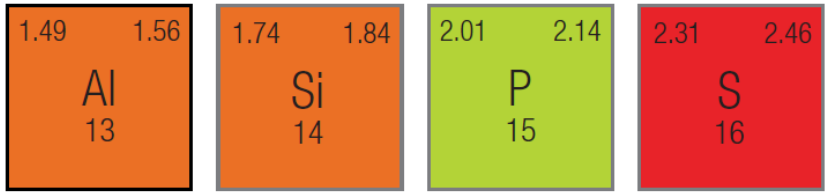
- Ease of use
  - Small size
  - Simple, point and shoot
- Fast analysis
  - Depends on model and sample type
  - 2 to 5 seconds is an ordinary range for test duration
- Proven - in the last 8 years alone
  - >50,000 Handheld XRFs have been delivered into field elemental analysis applications
  - More than half of those are alloy analysis applications
- Time saving and aid planning

In-service testing: oil refineries

API RP 939-C - Guidelines for Avoiding Sulfidation (Sulfidic) Corrosion Failures in Oil Refineries:

- When exposed to sulfidic corrosion conditions, individual carbon-steel piping components with low silicon content (less than 0.10 wt% Si) can corrode at an accelerated rate.
- 1/3 of high-temperature sulfidic corrosion failures are the result of low silicon content

In- service testing: oil refineries



Challenges

- Silicon – an unfriendly analyte!
  - A weak (slow) responder for XRF
  - Everywhere: comprises 29% of the Earth's crust
  - Low target measurement level: 1000 PPM - (0.1%)
- Temperature
  - Target processes run as hot as 650˚F / 343˚C
- Vibration
- Contamination

Innovation meets in-service testing

Hardware Advances

- Tube Improvements
  - More reliable
  - Higher current
- Detector
  - More sensitive: 2 orders of magnitude!
- Speed & Analytical performance
- Cost has dropped in the last 4 years

Innovation meets in-service testing

Rh X-Ray tube + SDD = greater range & speed

Timeline	2000s	2010s	Delta
Detector	SiPin	Silicon Drift (SDD)	
Resolution/CPS	250 eV/4k cps	160 eV/100k cps	40% - 25x
Source	X-ray tube	X-ray tube	2x+
Al - Aluminum	marginal w purge	0.200%	Equal to Ti
Si - Silicon	marginal w purge	0.050%	Hot Test
S - Sulfur	marginal w purge	0.010%	416 & 303
Ti - Titanium	0.20%	0.080%	2 seconds
Cr - Chromium	0.05%	0.008%	Residuals
Ni - Nickel	0.20%	0.009%	Residuals
Cu - Copper	0.20%	0.006%	Residuals

Innovation meets in-service testing

Silicon Drift Detectors change everything

- Handle up to 100k counts per second (vs. 4k in SiPin Detectors they replaced)
- Net count rate (the square root of)
  - Drives speed, precision, & detection Limit
  - Allows direct analysis lighter elements such as Magnesium (Mg), Aluminum (Al), Silicon (Si), Phosphorous (P), and Sulfur (S)

Faster, more precise, more complete testing period

In-service testing challenges: temperature

Dedicated heat sink – Design benefits

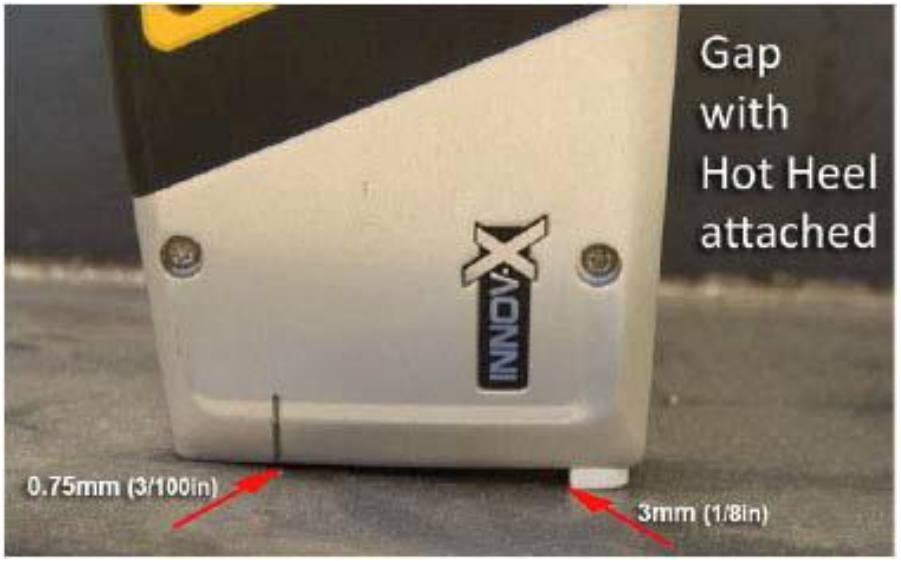
- Durability:
  - Critical to a sealed, protected housing: rubber over-molding, protective but insulative
- Longer component life:
  - Improved heat dissipation means longer life time for electronic components
- Better temperature stability:
  - Longer testing times and longer shifts
- Superior performance on hot, in-service samples

Heat dissipation

In-service testing challenges: temperature

Superior performance on hot, in-service samples

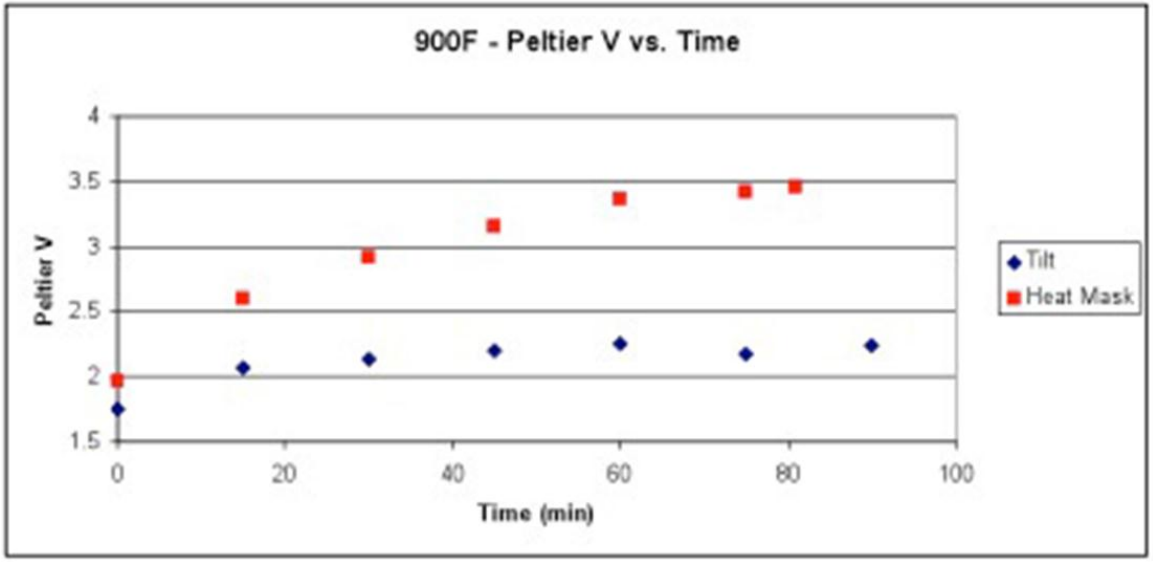
- Short test (2-6 sec) - Max Temp. 900 °F / 480 °C
  - >600 ˚F / 316 ˚C: hot heel recommended
  - >800 ˚F / 425 ˚C: hot heel required
- Long test (20-30 sec) - Max Temp. 650 ˚F / 340 ˚C
  - hot heel required always



In-service testing challenges: temperature

Flush, Tilt and Heat mask techniques

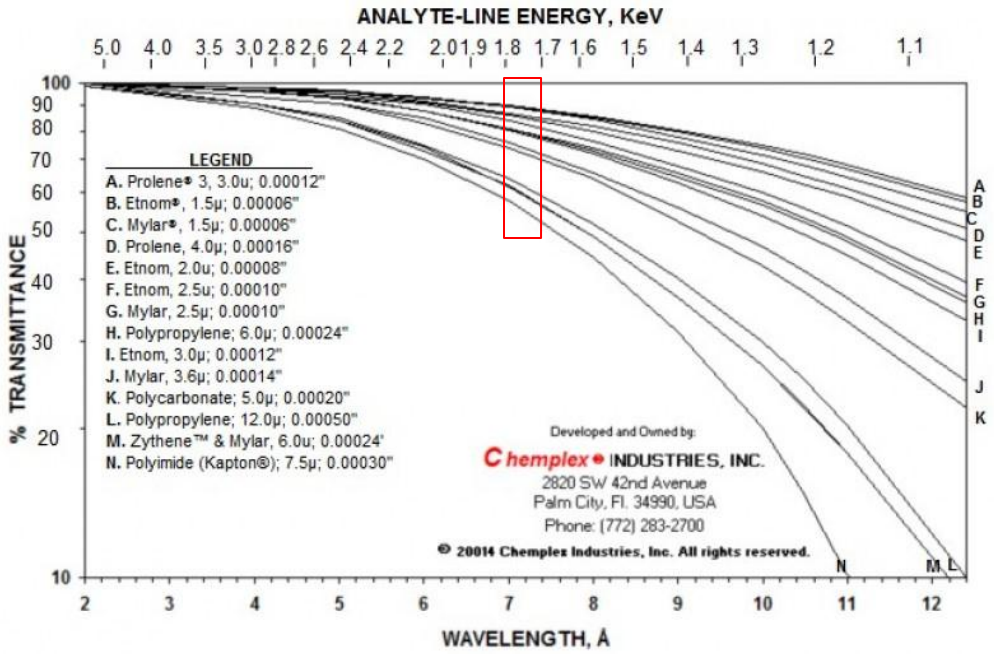
- Tilt technique works best
- Hot heel for ease of use
  - Constant position



In-service testing challenges: temperature

Analyzer window

- Prolene® melting temperature: 350˚F / 178˚C
- High Temperature window (Kapton®) to be used
  - Special high temperature calibration



http://www.chemplex.com/thin-film/thin-film-selection-guide/thin-film-selection-guide

In-service testing challenges: temperature

Larger window geometry for:

- Size, Shape, & Distance corrections
- Allows 2cm gap for testing vibrating samples
- Higher count rates for light elements such as Silicon (Si):
  - Test speed
  - Detection limits

In-service testing challenges: contamination

Handheld XRF is surface measurement technique – best practice:

- Do not touch the Kapton® window with your finger or the testing surface
- Do not bring the Kapton® window into contact with a dirty surface
  - the hot heel creates a space that normally prevents contact between the test surface and the Kapton®
- Regularly test the analyzer against a reference sample

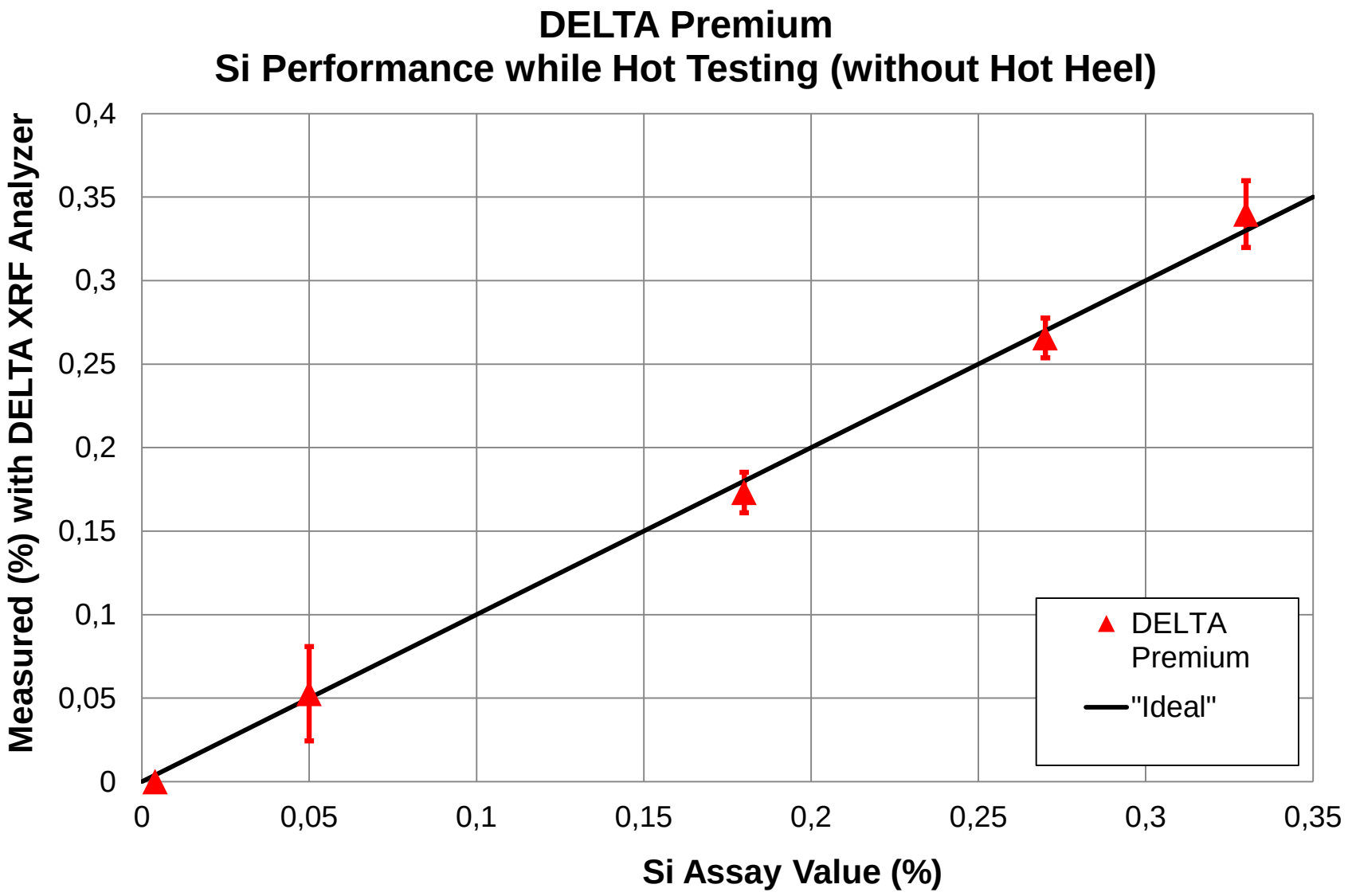
Corrosion, scale, oxide layer

Trace Level Testing (Low alloy steels)

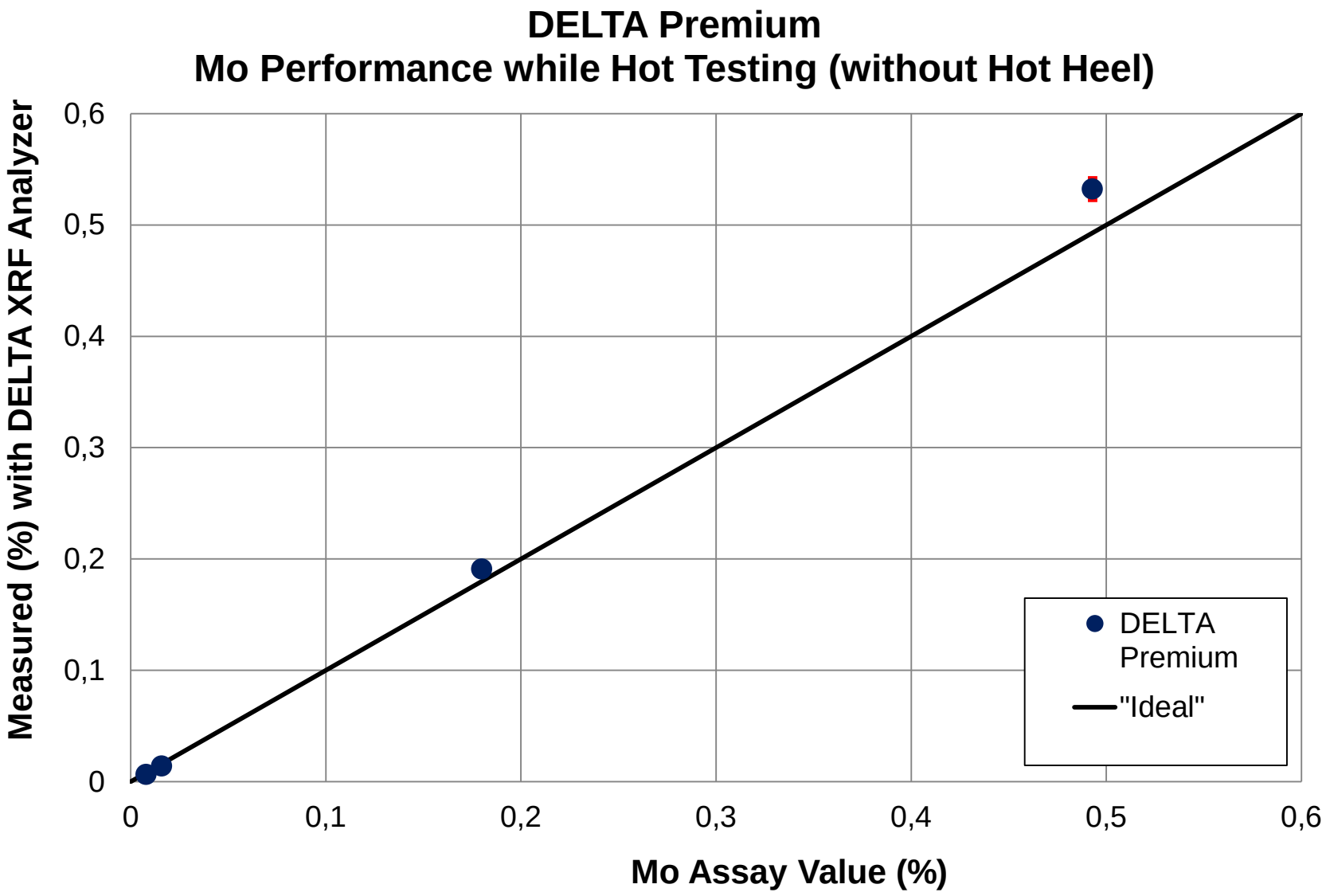
High temperature calibration validation

- Alloys with Si content between 0 and 0,33%:
  - 12L14
  - CS 1117
  - CS 1144
  - 4140
  - Carbon 1/2Mo
- Kapton® Window
- Measurement time: 30sec

High temperature calibration validation



High temperature calibration validation



Site testing

RP 939C field data: tests near top of distillation tower

- Sample Temp 650˚F / 340˚C
- Zirconium Oxide grinding wheel
- Combustible gas meter required to use grinder
- Certified standards to verify window is free of Si contamination & unit is functioning correctly.

Site testing

Comment	Seconds	Al	Al +/-	Si	Si +/-
Hot Sample #1A - no prep	30.59	6.17	0.18	2.117	0.028
Hot Sample #1B - full prep	30.67	<LOD	0.053	0.137	0.012
Hot Sample #2A - no prep	16.03	3.8	0.23	6.93	0.07
Hot Sample #2B - full prep	14.2	<LOD	0.087	0.246	0.02
Hot Sample #3A - no prep	11.36	4.58	0.31	2.91	0.06
Hot Sample #3B - full prep	26.88	<LOD	0.06	0.086	0.012
Check Standard - 0.27% Si	13.56	<LOD	0.076	0.263	0.02
Check Standard - 0.0% Si	30.49	<LOD	0.049	<LOD	0.005

Conclusion

Innovation

- High sensitive SDD detector
- Rh Anode X-ray tube
- Large window aperture
- Efficient temperature control (heat sink)
- Dedicated High Temperature calibration
- Hot heel solution

To meet demanding environment (In-Service Testing)

- High temperature
- Vibration
- Contaminated
- Light elements