
Standardisation in High Temperature Corrosion Testing

Review of Task 4 of the UK-US Collaboration

23rd Annual Conference on Fossil Energy Materials
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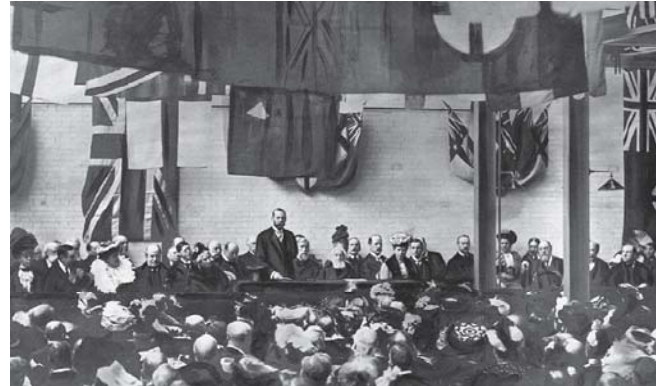
Overview

- Introduction to Task 4
- Data collection and storage
- Standardisation
- Inter-comparison



What was task 4 concerned with?

- To identify critical differences between standards for measurement of high temperature materials properties
- To identify where further standardisation for measurement of high temperature materials properties is required
- To develop a common format for data exchange
- To investigate the use of commercial database software for collecting and maintaining materials properties data and micrographs



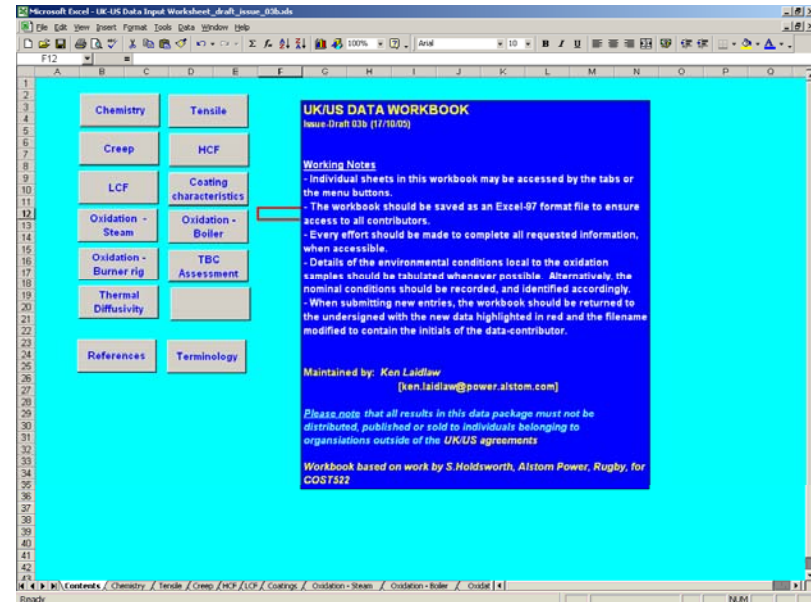
1902
NPL Opens



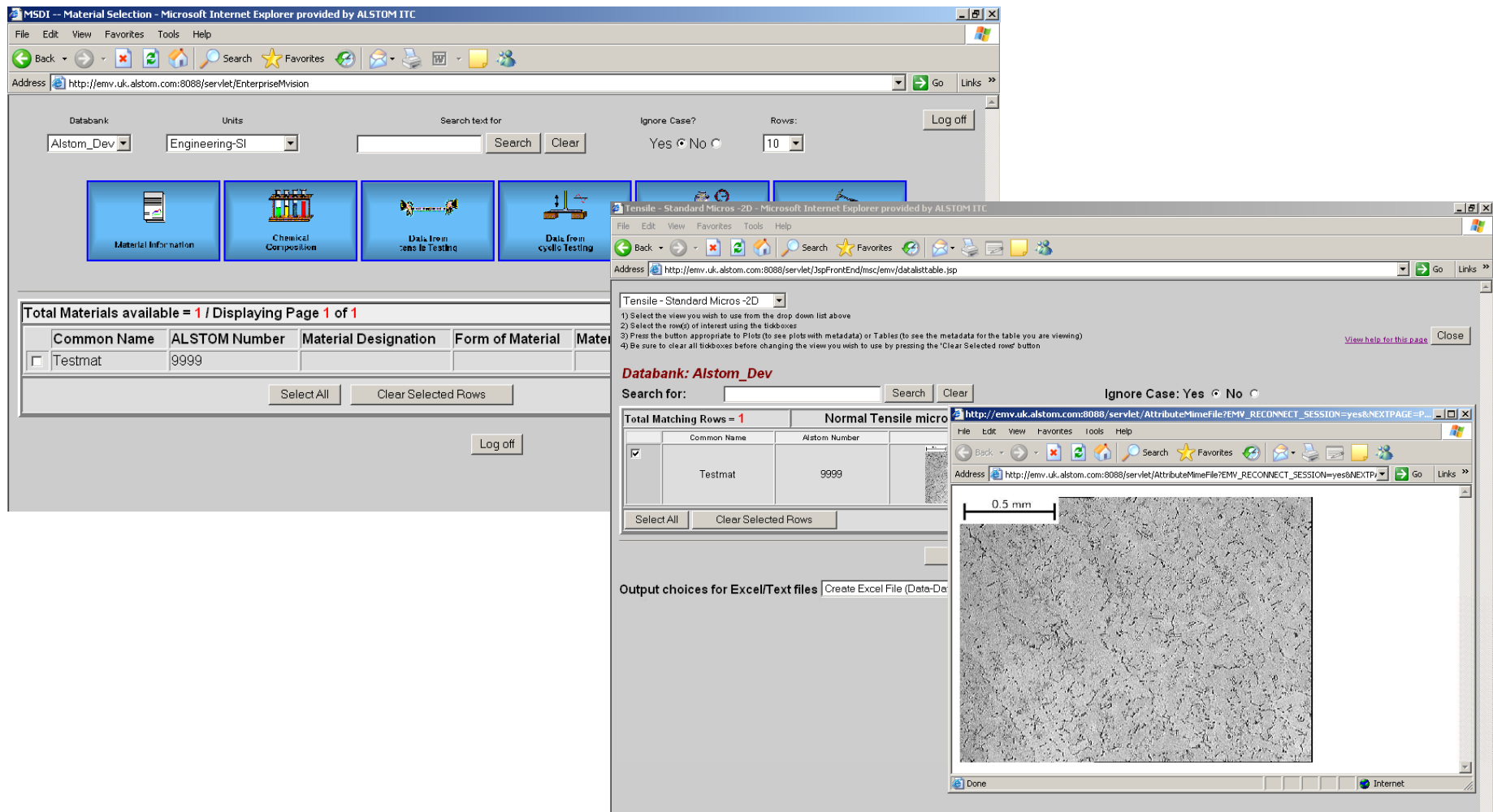
1906
Metallurgical
laboratory

The collaboration will generate a lot of data – how do we manage and collect it?

- What data is being recorded?
- Can we agree on what should be reported?
- What is the best method of capturing this data?
- Can everyone use the selected method?



Data collation in a database, which includes metadata



The screenshot displays the MSDI Material Selection web application interface. The main window shows search results for 'Testmat' with an ALSTOM Number of 9999. The interface includes a search bar, a table of results, and a 'Log off' button. A secondary window shows a detailed view of the material, including a micrograph of the material's surface texture with a 0.5 mm scale bar.

MSDI -- Material Selection - Microsoft Internet Explorer provided by ALSTOM ITC

Address: <http://emv.uk.alstom.com:8088/servlet/EnterpriseMvision>

Databank: **Alstom_Dev** Units: **Engineering-SI** Search text for: Ignore Case? ☒ Yes ☐ No Rows: **10** **Log off**

Total Materials available = 1 / Displaying Page 1 of 1

Common Name	ALSTOM Number	Material Designation	Form of Material	Material
<input type="checkbox"/> Testmat	9999			

Select All **Clear Selected Rows** **Log off**

Tensile - Standard Micros - 2D - Microsoft Internet Explorer provided by ALSTOM ITC

Address: <http://emv.uk.alstom.com:8088/servlet/JspFrontEnd/msc/emv/datalsttable.jsp>

Tensile - Standard Micros - 2D

1) Select the view you wish to use from the drop down list above
2) Select the row(s) of interest using the tickboxes
3) Press the button appropriate to Plots (to see plots with metadata) or Tables (to see the metadata for the table you are viewing)
4) Be sure to clear all tickboxes before changing the view you wish to use by pressing the 'Clear Selected rows' button

Databank: Alstom_Dev

Search for: **Search** **Clear** Ignore Case: ☒ Yes ☐ No

Total Matching Rows = 1 **Normal Tensile micro**

Common Name	Alstom Number
<input checked="" type="checkbox"/> Testmat	9999

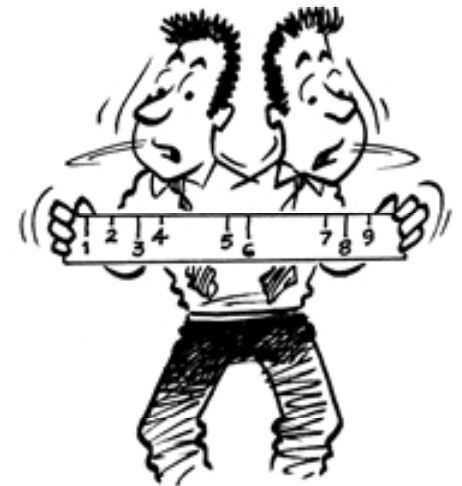
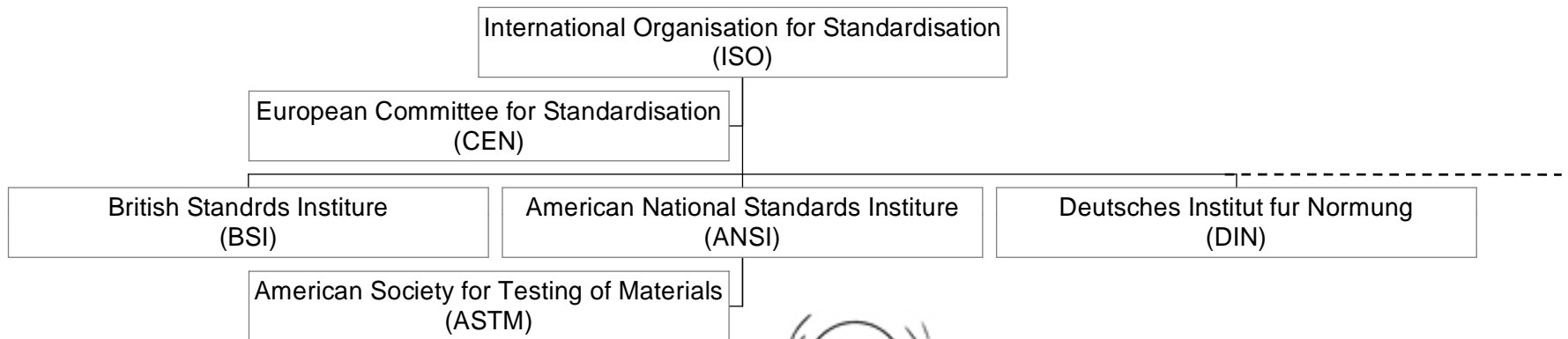
Select All **Clear Selected Rows**

Output choices for Excel/Text files **Create Excel File (Data-D)**

0.5 mm

Done **Internet**

How can we be sure the data is comparable?



How do the UK and US standards compare?

Mechanical Tests

- Hardness
- Tensile
- Creep
- Low Cycle Fatigue (LCF)
- High Cycle Fatigue (HCF)

Physical Testing

- Thermal Diffusivity
- Dilatometry
- Surface Area Measurement

Corrosion Testing

- Laboratory exposures in steam and mixed gases (including deposits)
- Post-exposure evaluation of environmental attack
- Steam Loop exposures
- Coating Thickness
- Burner rig testing
- Thermal Cycling/Cyclic Oxidation

Findings of the review – mechanical testing

Test	Addressed by	Standards	Further work needed
Hardness	ISO - TC164	Knoop, Brinell, Vickers, Rockwell	No
Tensile	ISO - TC164	Ambient Temp. High Temp.	No
Creep	ISO	Uniaxial	No
LCF	ASTM, CEN, BSI	Standards exist for strain controlled, TMF.	Not at this point
HCF	Yes	Ambient Temp.	Yes, HT HCF

Findings of the review – physical testing

Test	Addressed by	Standards	Further work needed
Thermal Diffusivity	ASTM	Laser flash	No
Dilatometry			Yes
Surface Area Measurement	ISO	Gas adsorption or permeability	No

Findings of the review – corrosion testing

Corrosion Testing

- Laboratory exposures in steam and mixed gases (including deposits)
- Post-exposure evaluation of environmental attack
- Steam Loop exposures
- Coating Thickness
- Burner rig testing
- Thermal Cycling/Cyclic Oxidation

At the time of the review there were no international, US or European standards existing for high temperature corrosion of metallic materials.

this area is being actively pursued by ISO TC156 WG13 and standards for:

- Test Method for Isothermal Exposure Testing under High Temperature Corrosion Conditions
- Method for Metallographic Examination of Samples after Exposure to High Temperature Corrosive Environments
- Thermal Cycling Exposure Testing Under High Temperature Corrosion Conditions

are in preparation.



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No standards, so what... Inter-comparison



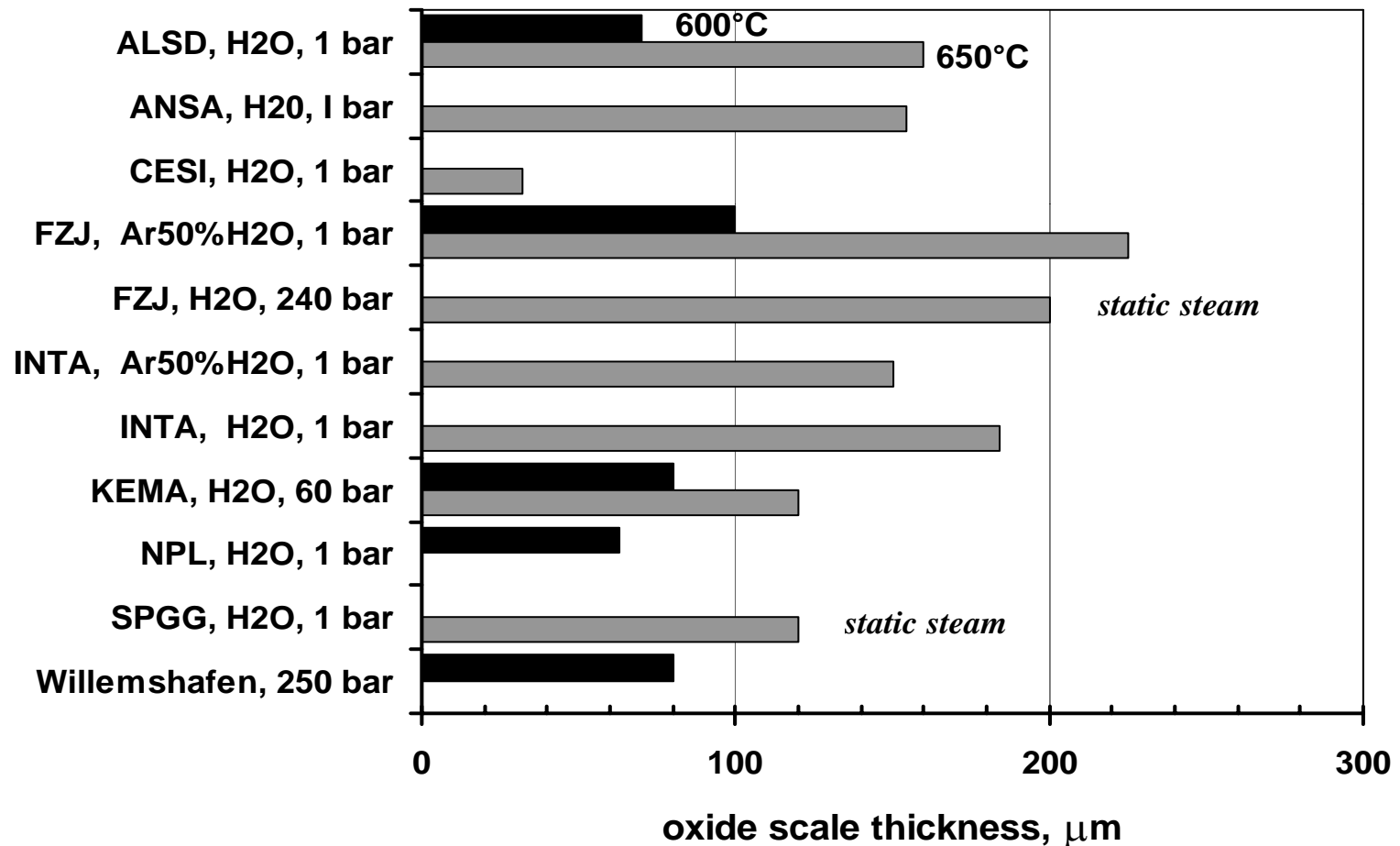
Seam Oxidation

- 3 materials (T92, 347HFG, IN740)
- Same material stock
- Same temperatures
- Lab could prepare samples in their standard manner
- Tests conducted using their own preferred method
- Data analysed in their own preferred technique

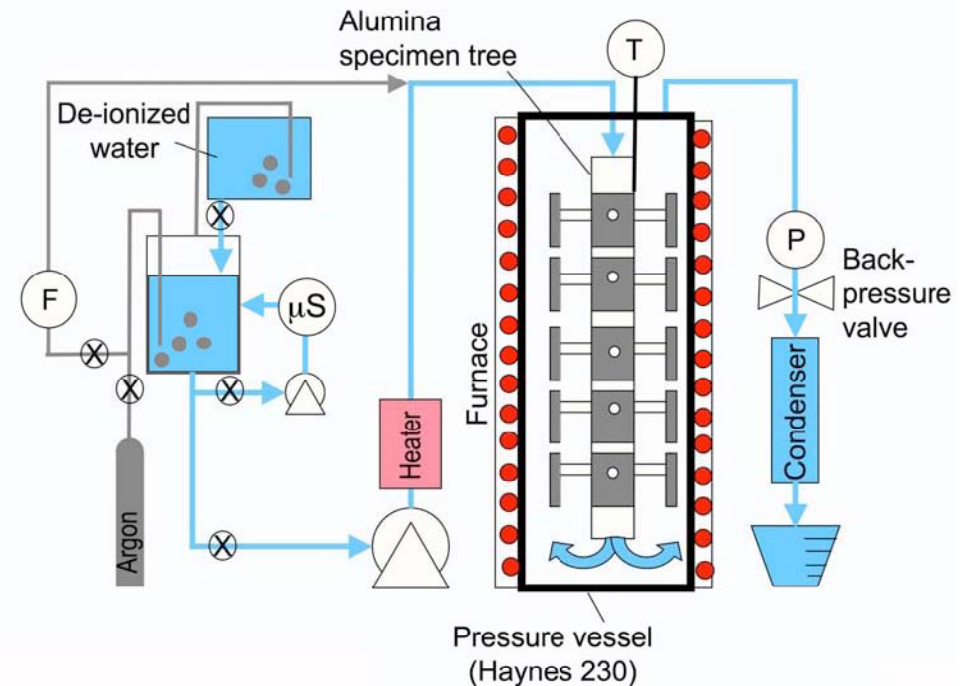
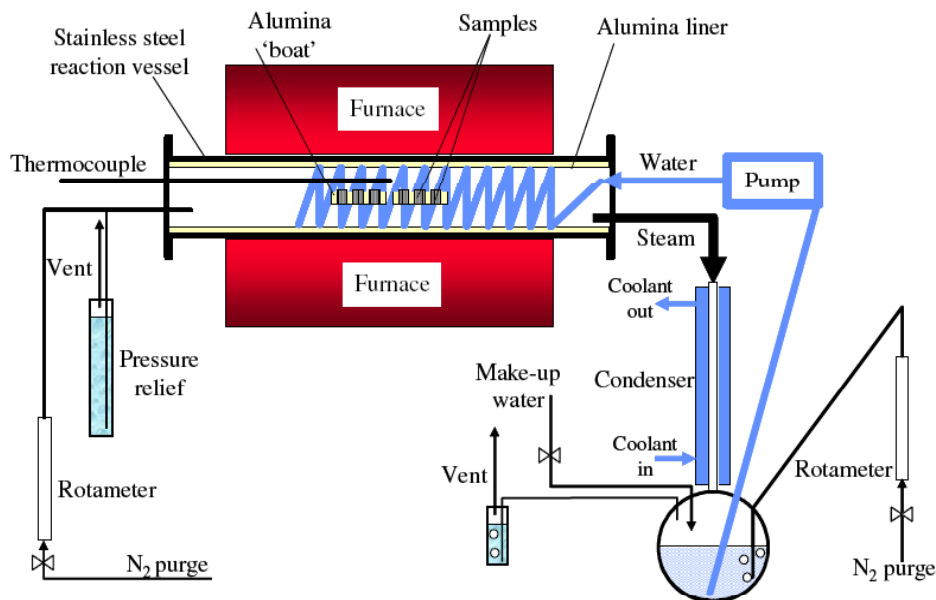
Boiler Corrosion

- 2 materials (T22 and P92)
- Same material stock
- Gas composition set
 - 0.3% SO₂, 6.0% O₂, 14.6% CO₂, 74.2% H₂
- Ash composition set
 - Na₂SO₄/K₂SO₄/Fe₂O₃ (1.5/1.5/1 on a molar basis)
- Temperatures set
- Lab left to prepare samples and expose using their preferred method

P92, 1000 h exposure



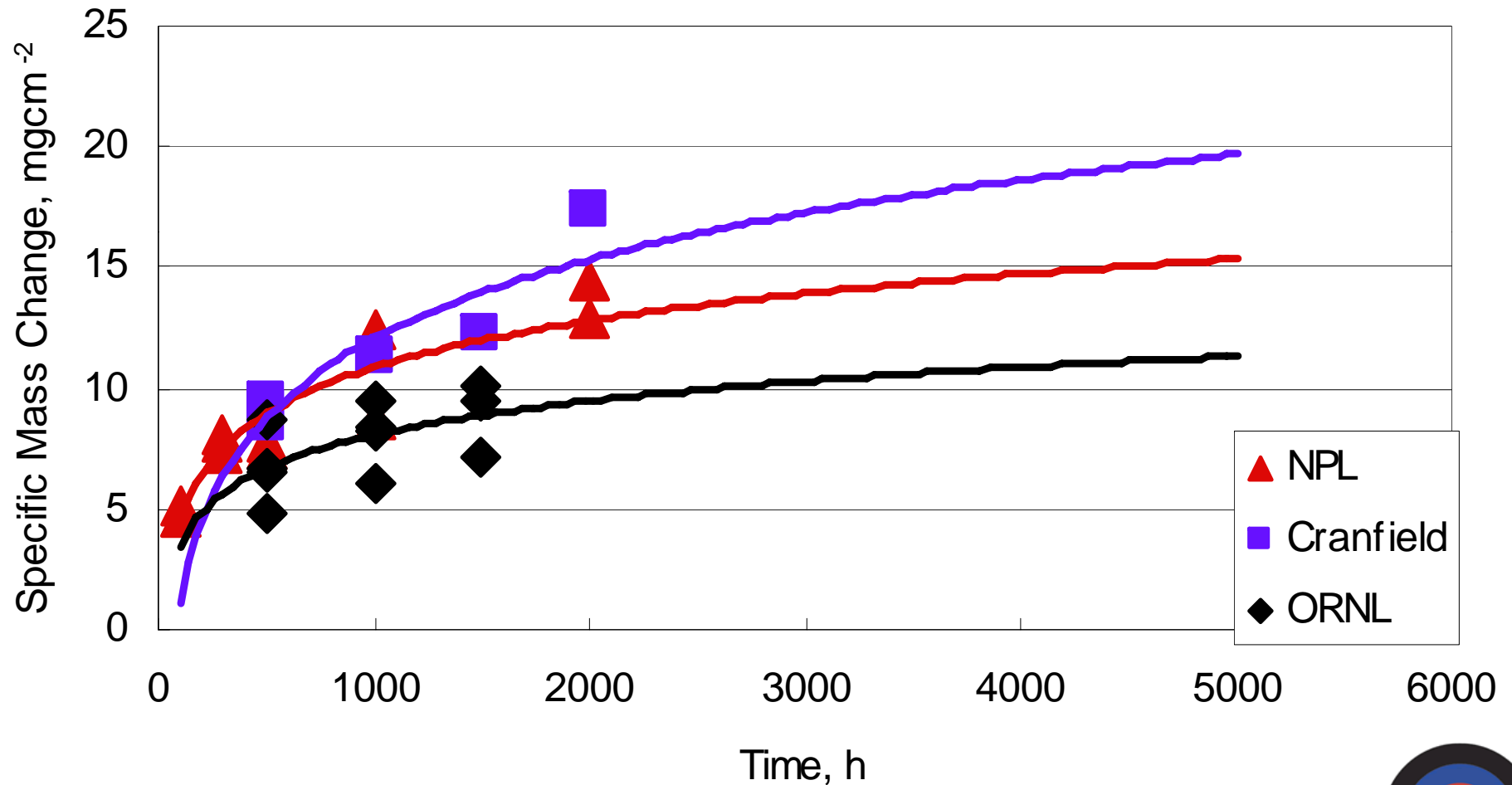
Steam Oxidation experimental setup



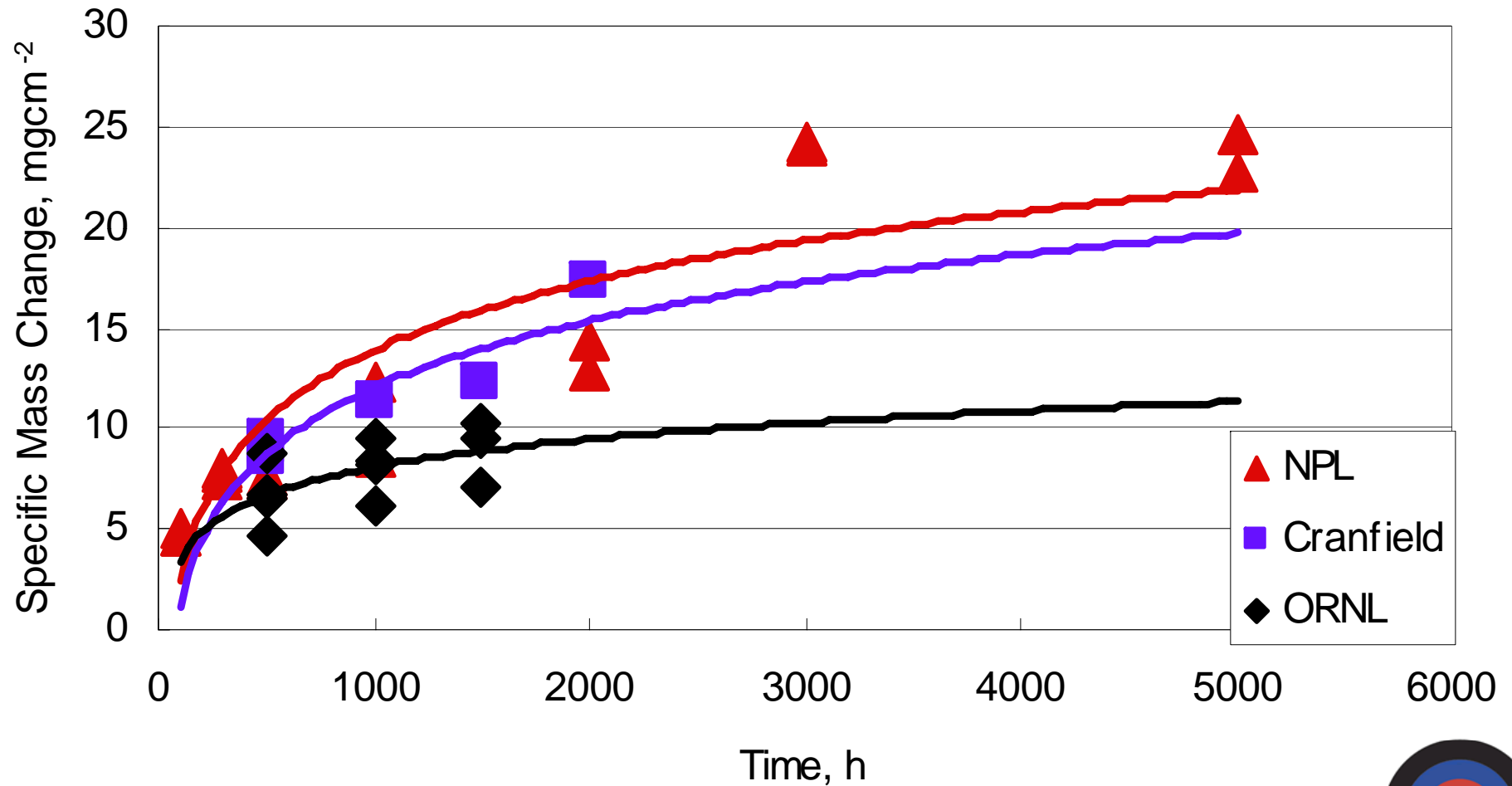
Steam oxidation experimental

- Sample geometry
 - 10 x 10 x 3mm
 - 20 x 10 x 2mm
 - Semicircular section
- Surface Preparation
 - Samples from bulk
 - Samples retained original surface
 - Surfaces prepared 600-grit SiC
- Exposure procedure
 - Duplicate samples exposed for a set time duration (no cycling)
 - Sample all exposed at the same time, thermal cycles introduced to remove samples
 - Ambient pressure & 17 bar

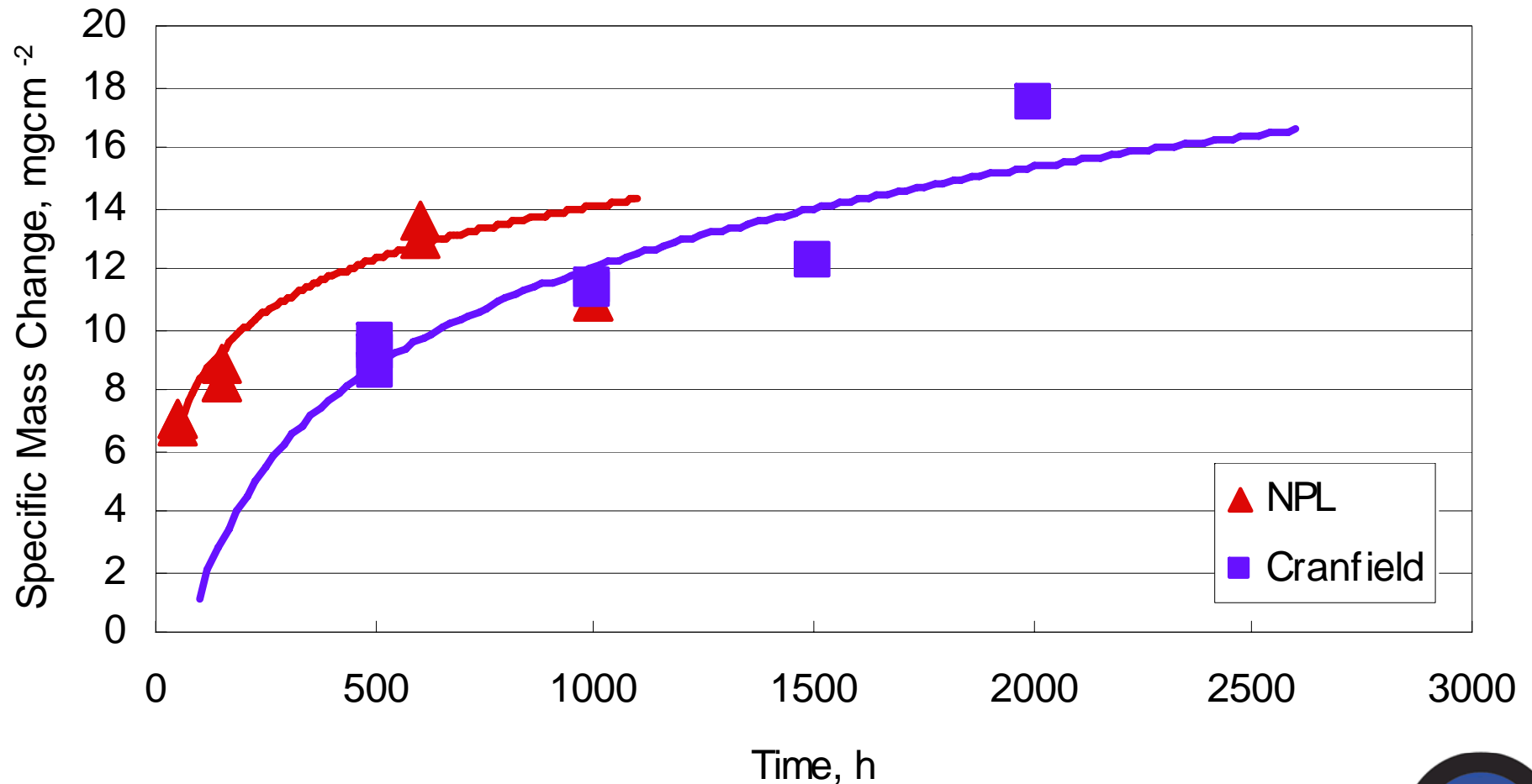
Steam oxidation results T92 at 600 °C



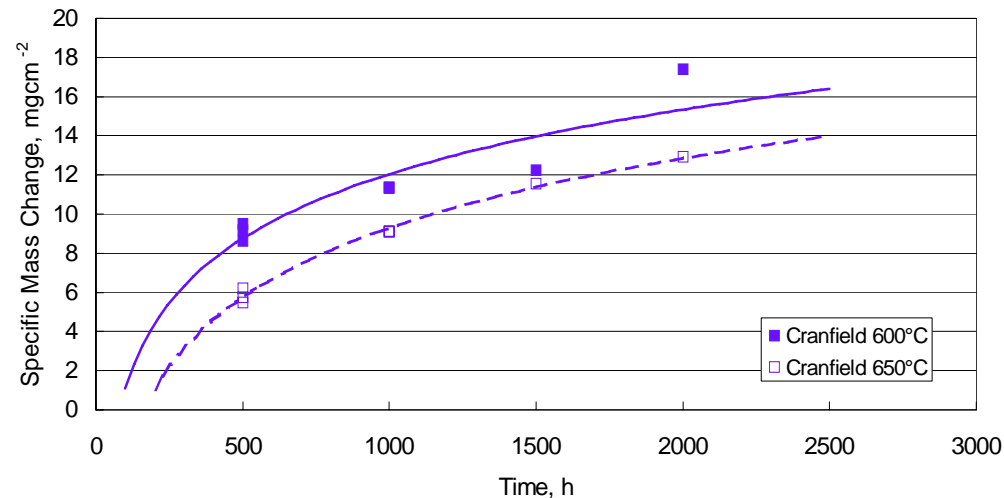
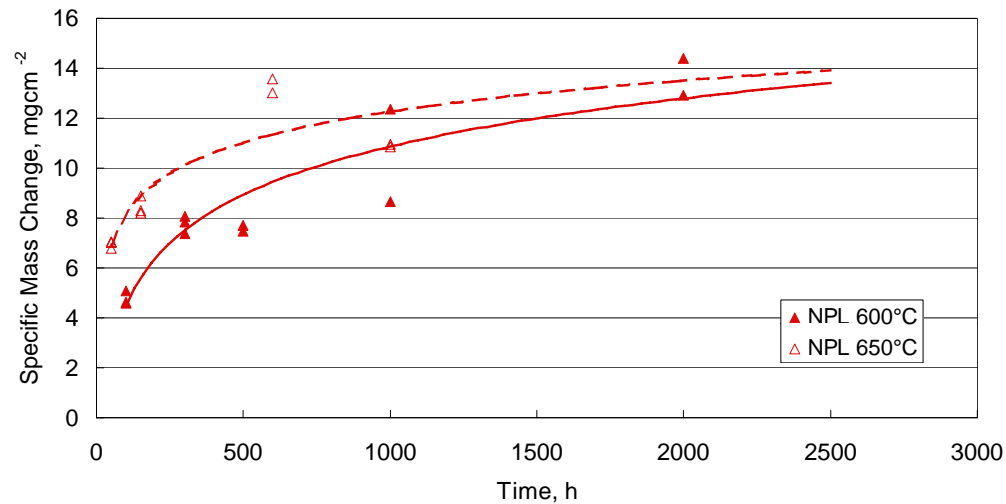
Steam oxidation results T92 at 600 °C



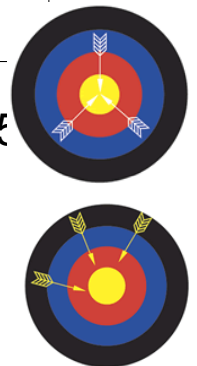
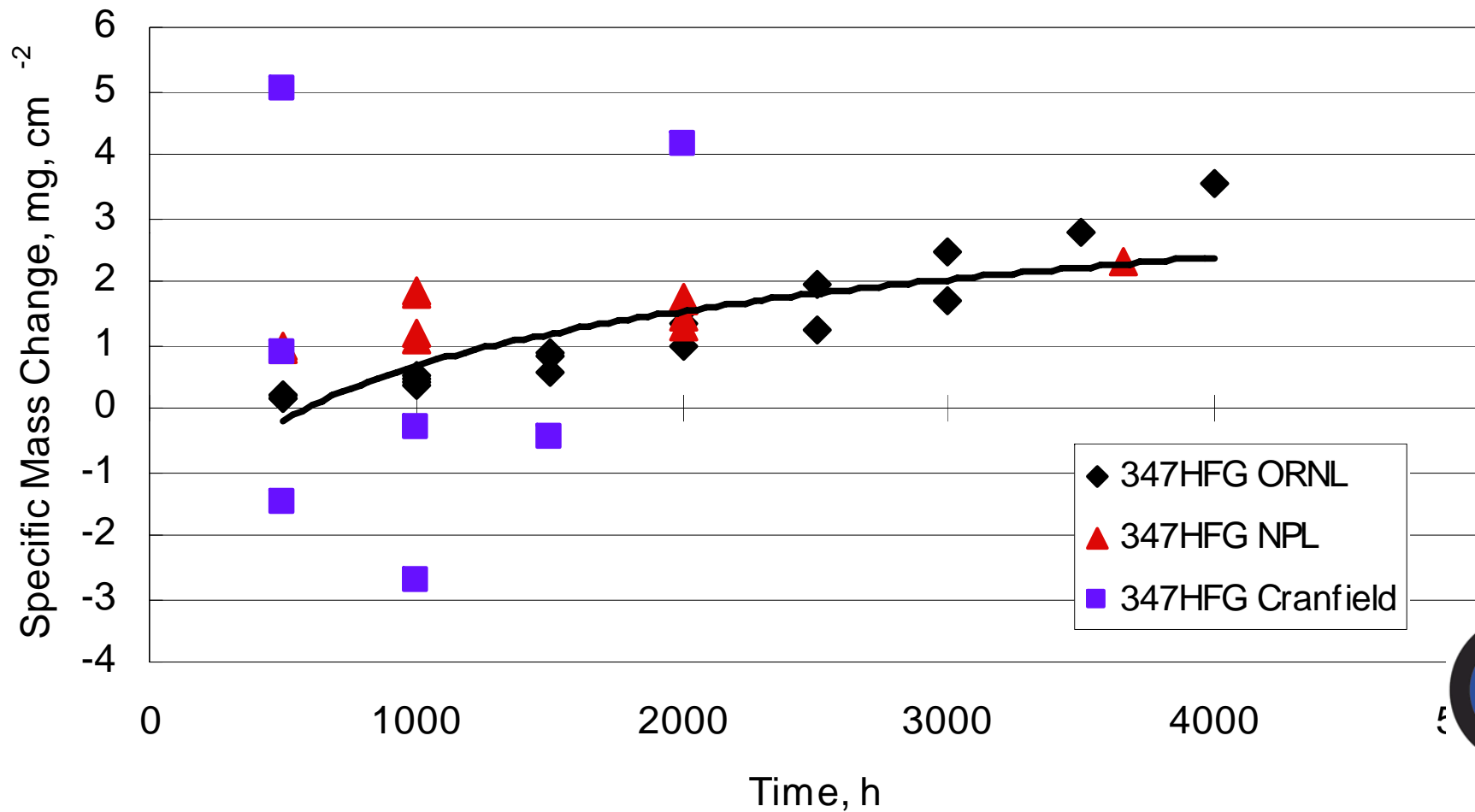
Steam oxidation results T92 at 650 °C



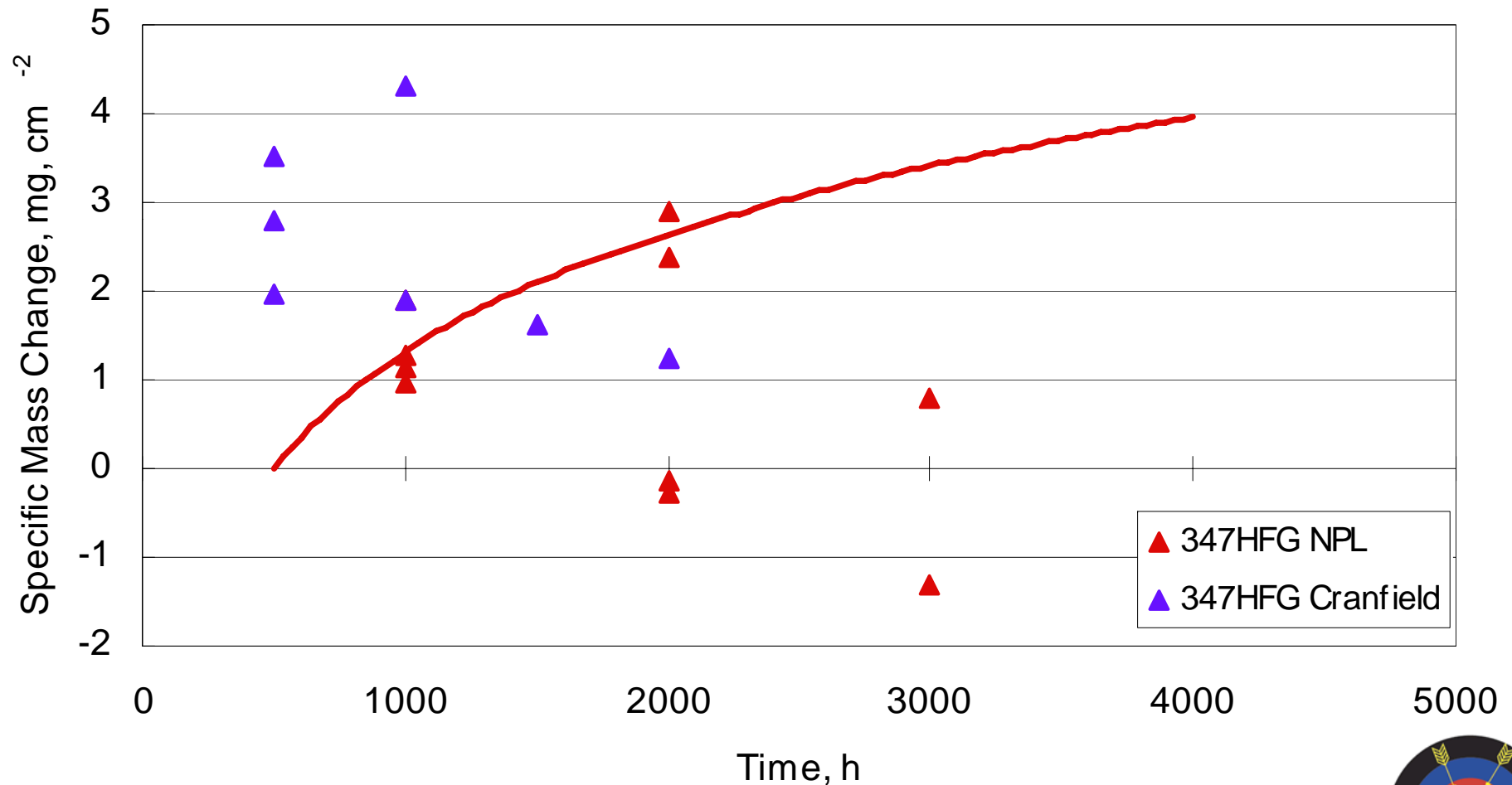
Steam oxidation T92 temperature dependency



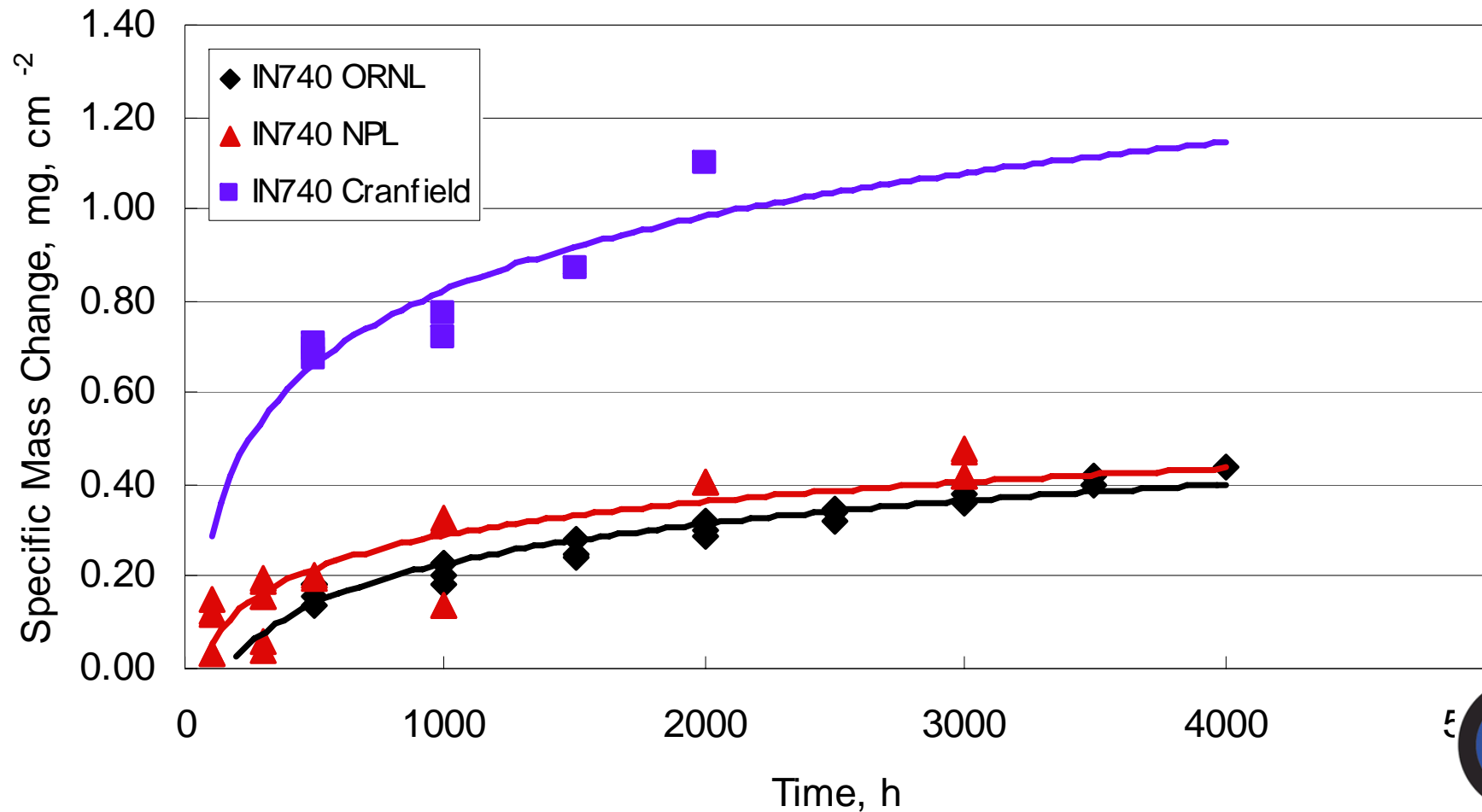
Steam oxidation results 347HFG at 650 °C



Steam oxidation results 347HFG at 700 °C

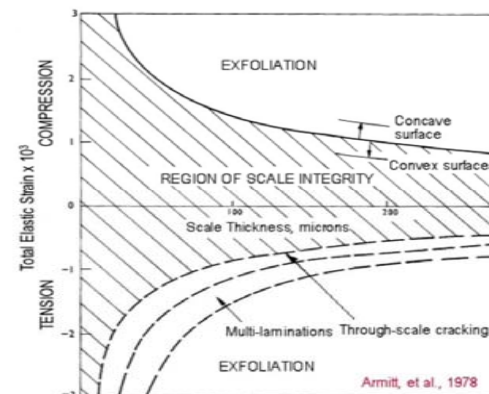
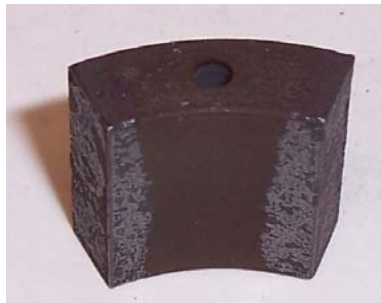
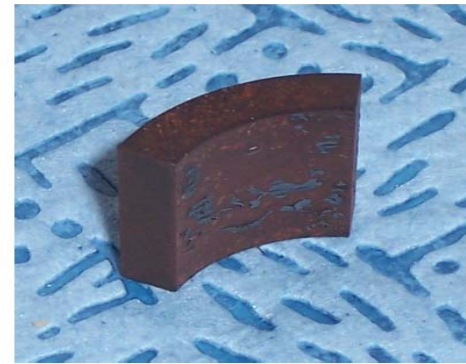


Steam oxidation results IN740 at 750 °C



What could be causing the differences?

- Specimen Geometry
- Thermal Cycling
- Orientation of grains
- Spallation



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- Lab left to prepare samples and expose using their preferred method

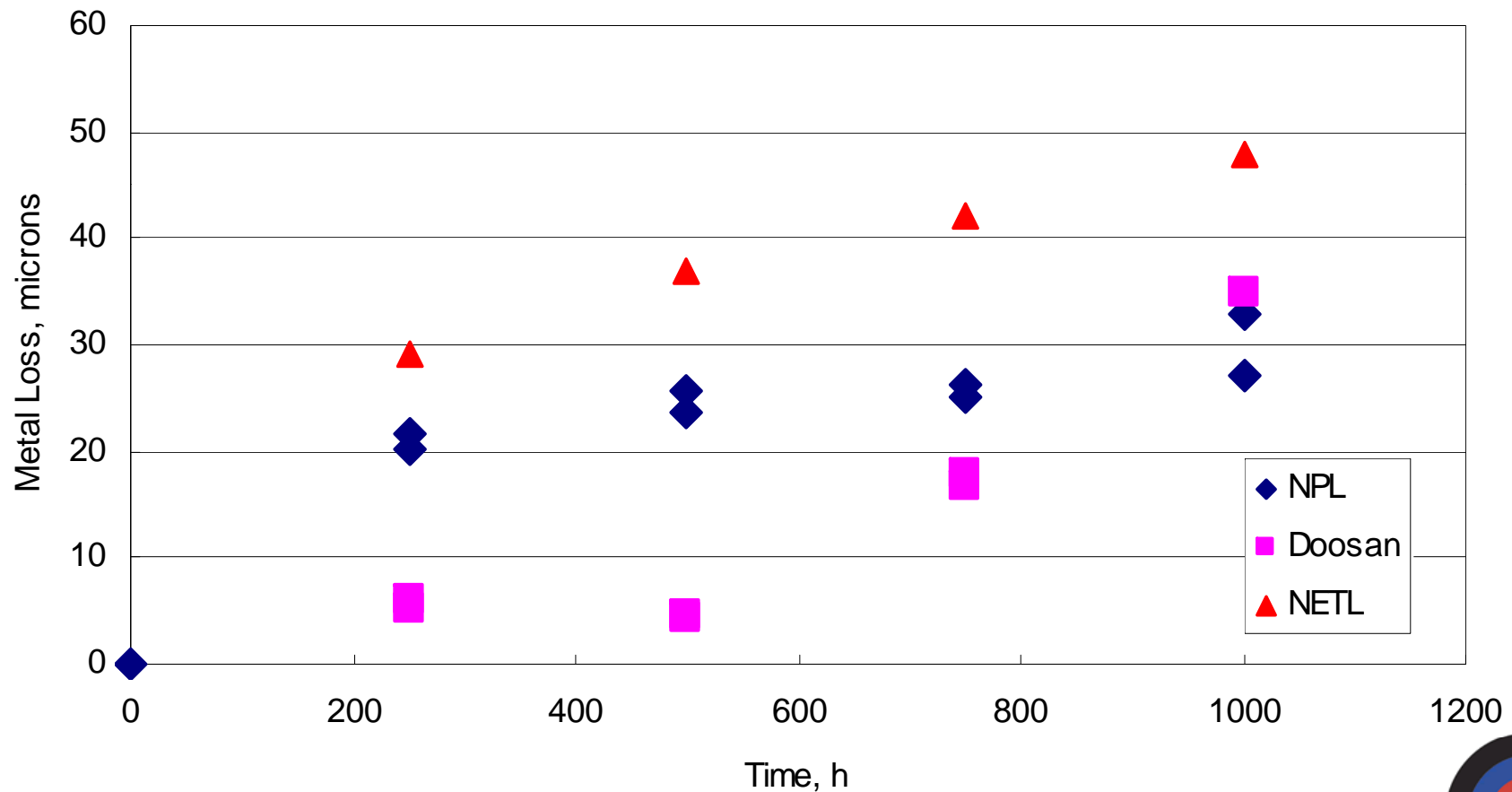


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What about boiler corrosion, that's OK...isn't?



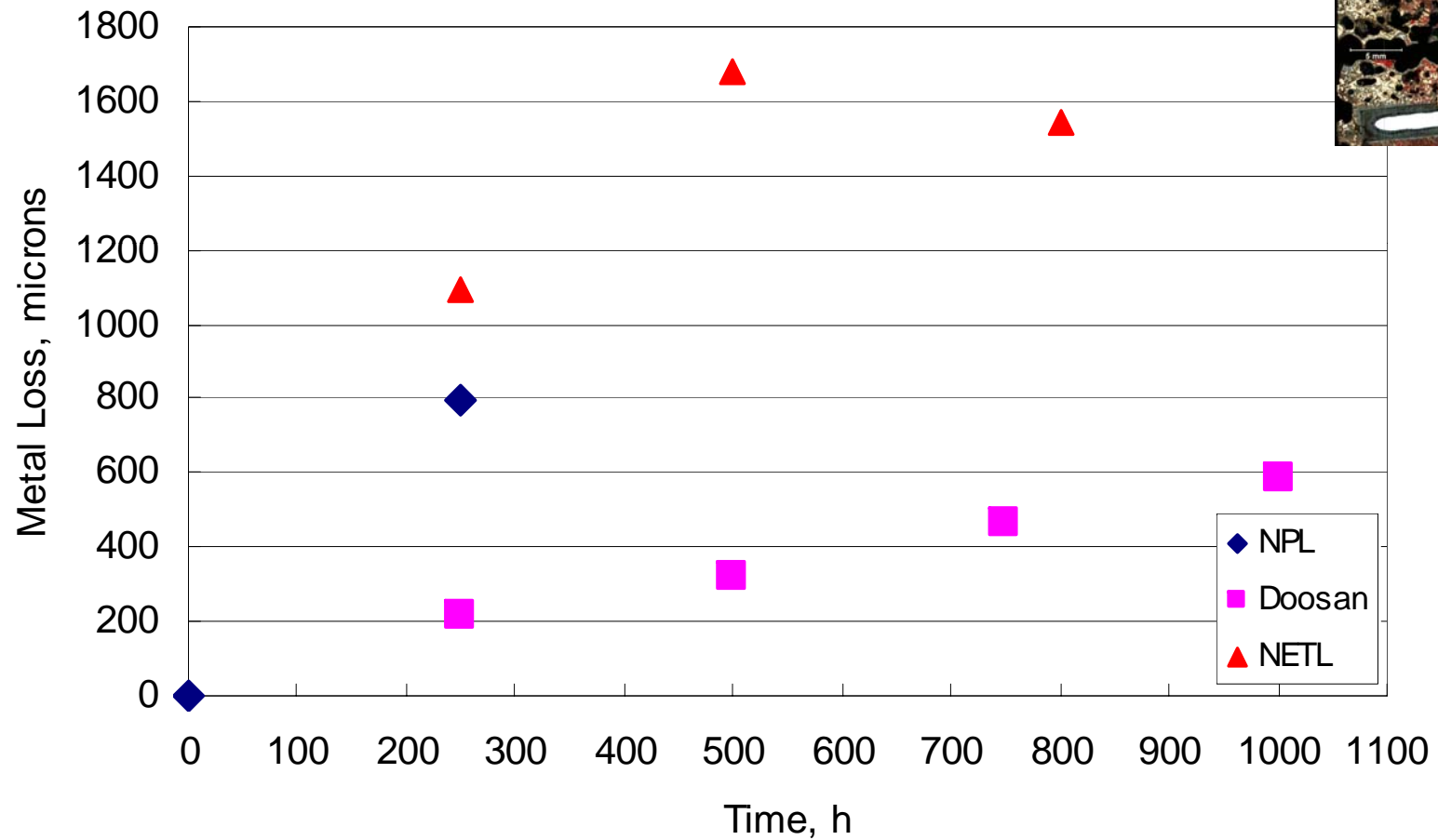
T22 at 425 °C



Pittsburgh – 14th May 2009

UK-US Collaboration on Fossil Energy R&D - Advanced Materials

P92 at 675 °C



Why the differences?

- Specimen manufacture
- Measurement accuracy
- Fundamental differences in the apparatus
- ?



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What does all this mean?



No one is wrong, the results are just different

- Results are self consistent with a laboratory
- Measurements are precise but there is scatter due to material effects (i.e. spalling)
- Ideally we would like high precision and good repeatability



Where do we go from here?

- Standard test methods for corrosion testing which address specimen manufacture and preparation as well as the actual test procedure and analysis.
- Need to address
 - Specimen geometry
 - Surface preparation
 - Testing procedures
 - Measurement accuracy & uncertainty

Phase 2 will be addressing some of these issues

Thank you for your attention

Any questions?