Qualification of new commercial ODS alloys

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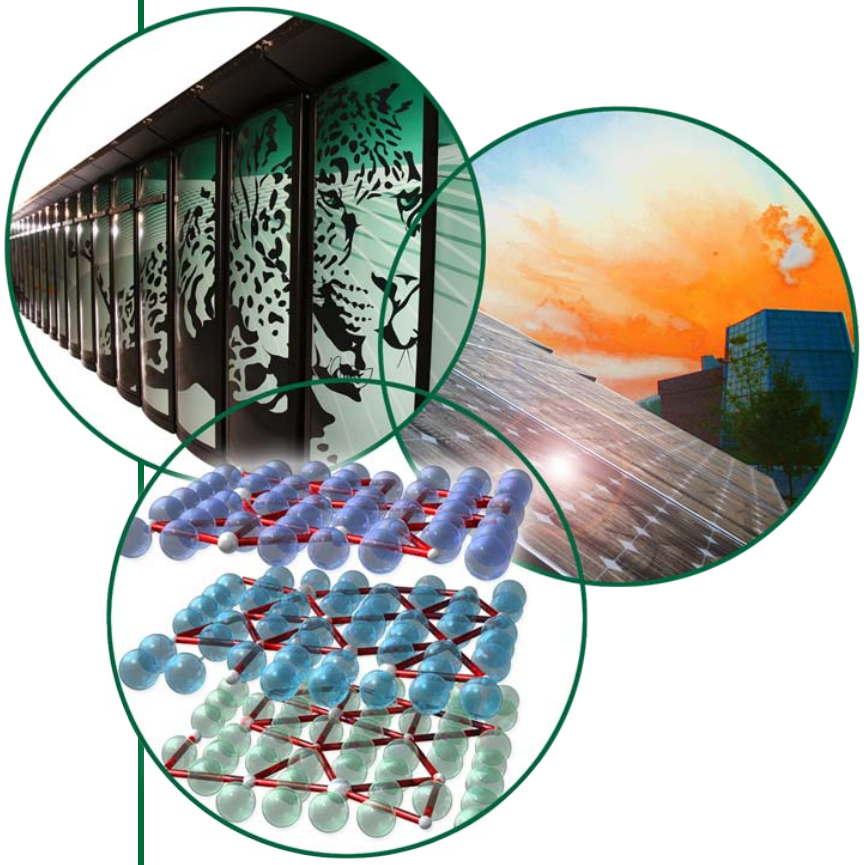


OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE DEPARTMENT OF ENERGY

Presentation Outline

Ames Laboratory

Gas Atomization Reactive Synthetic powder

Industrial Partners

- Dour Metal Sro.
- MBN Nanomaterialia SpA

New commercial ODS alloys

ORNL

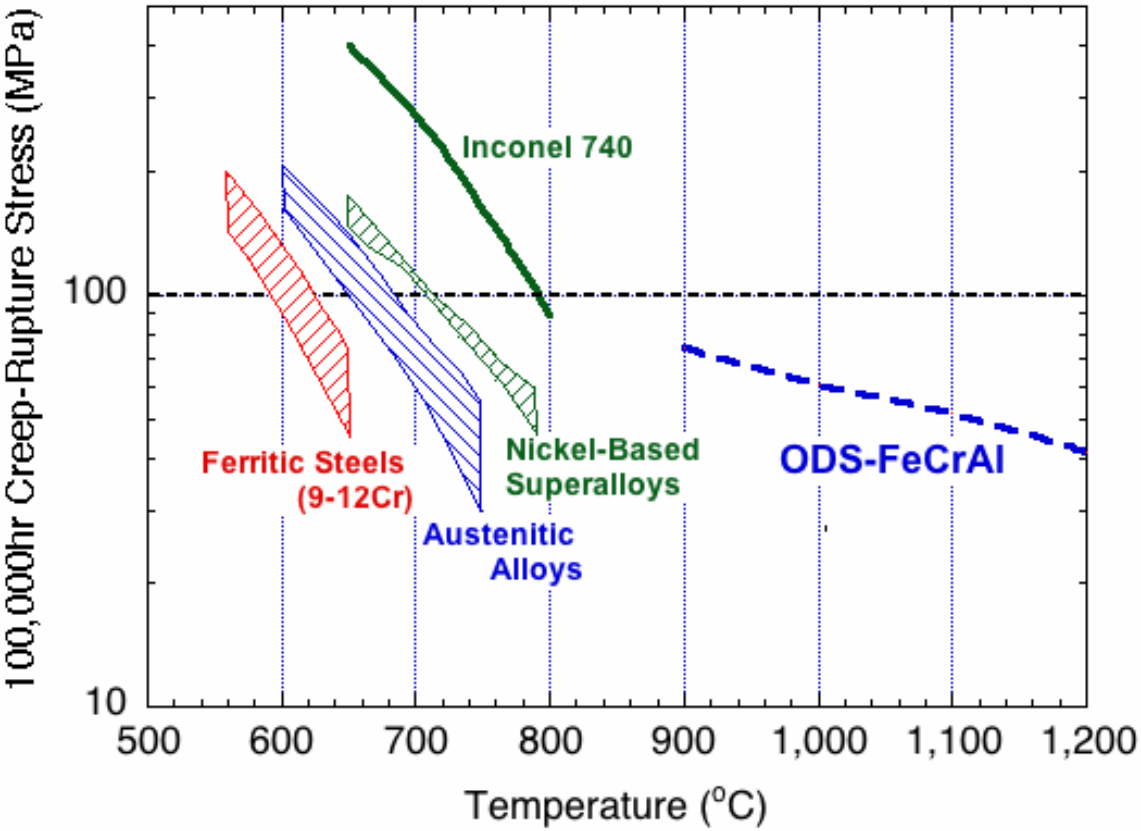
- Mechanical Testing
- Ball Milling
- Oxidation testing in complex environments (H_2O, CO_2)
- Lifetime Modeling

University of Liverpool

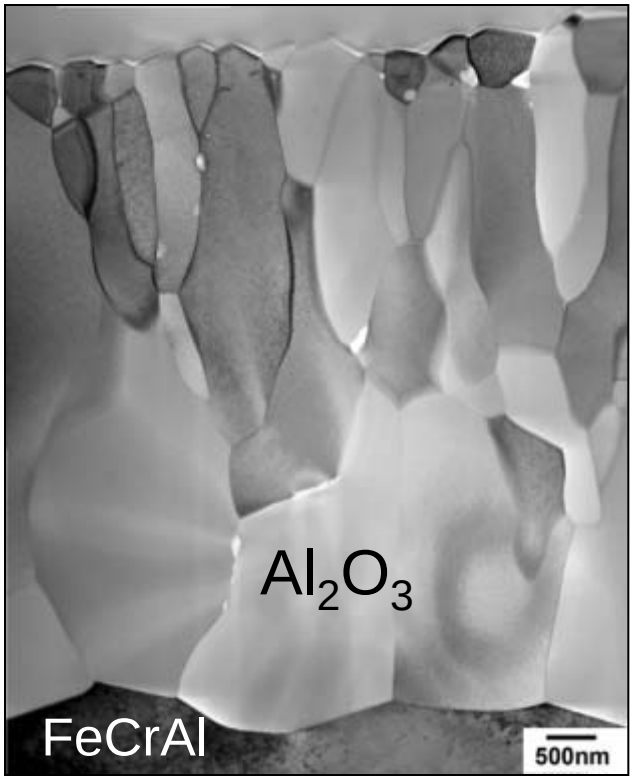
- Selective Laser Melting of ODS alloys
- Friction Stir Welding of ODS alloys
- US/UK partner

Great potential for high efficiency systems using FeCrAl-ODS alloys

- Oxide Dispersion Strengthened FeCrAl alloys exhibit excellent creep and oxidation properties at T>1200ºC.



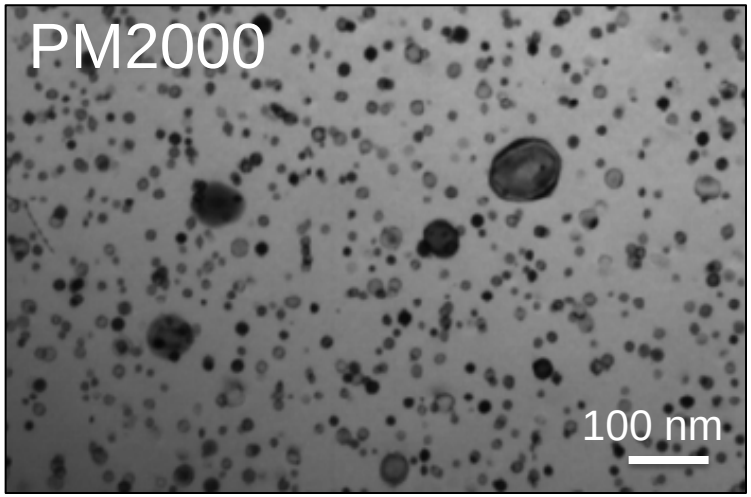
500h, 1100ºC, O_2



courtesy Kinga A. Unocic

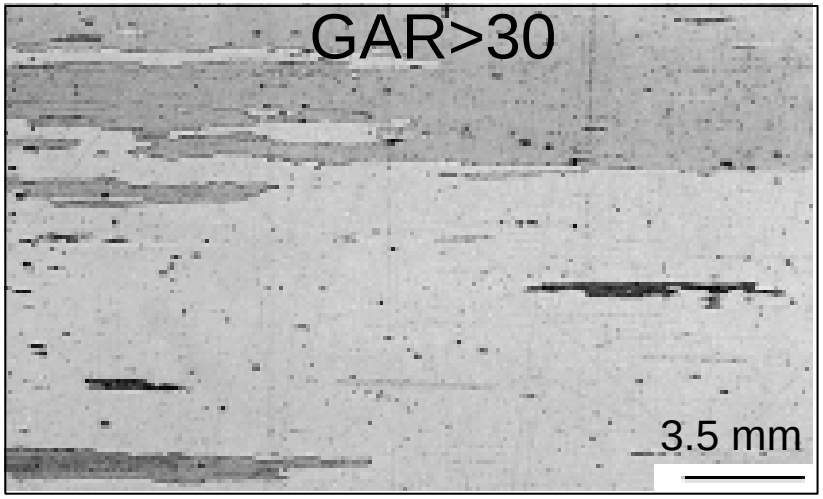
FeCrAl ODS alloy microstructure

PhD Thesis Laurent Marechal



Nano precipitates ~20nm obtained by mechanical alloying

Capdevila & Al. MSE (2008)



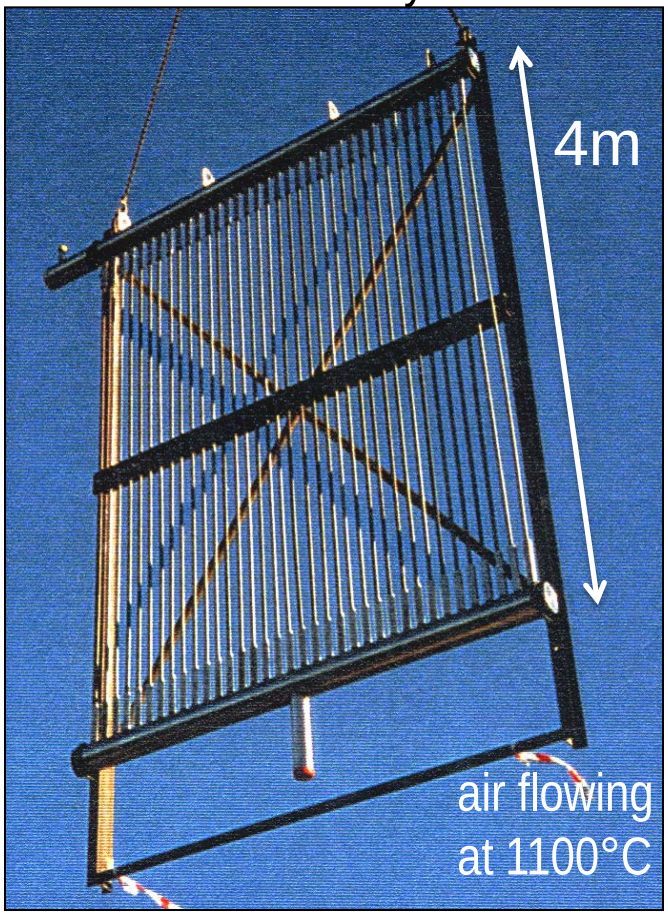
Recrystallisation at HT for large grains (>10mm)

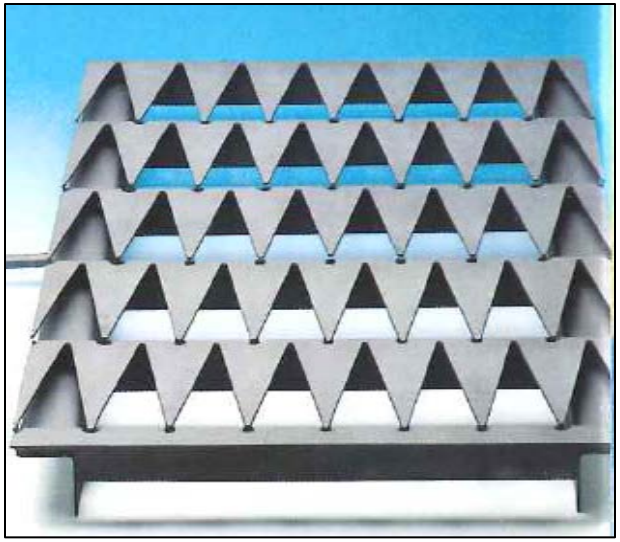
Alloy	Composition (wt.%)					
	Cr	Al	Mo	Ti	Y_2O_3	Fe
PM 2000	20	5.5	<0.02	0.5	0.5	bal
MA 956	20	4.5	-	0.5	0.5	bal
ODM 751	16	4.5	1.5	0.6	0.5	bal

High Temperature Heat Exchanger Furnace components

British Gas demonstrator HTHE

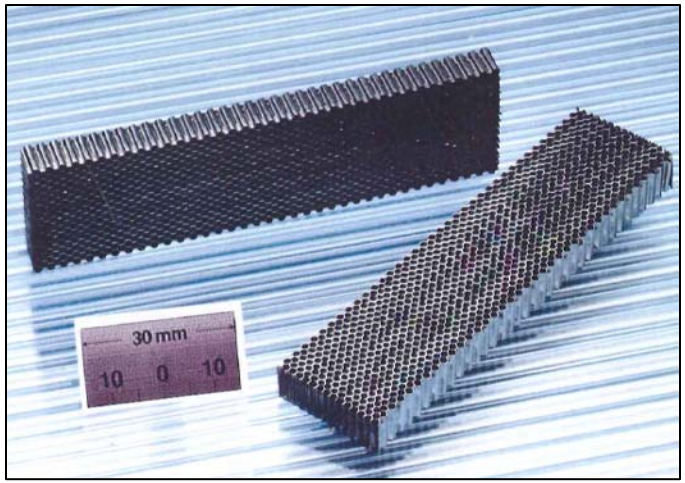
25 mm dia. x 4 m long ODM751 tube fabricated by Dour Metal





PM2000 Furnace ware for high vacuum furnace

Brazed PM2000 honeycomb sealing segment for gas turbine



Collaboration with Dour Metal: Satisfying powder production but need to recommission extrusion press

- Production of ODS powder under controlled atmosphere
- Dour Metal will provide powder for additive manufacturing trials
- 3-step powder consolidation
- Cold and hot compaction
- Billet machining to remove steel from canning
- Hot extrusion

- Complicated extrusion process required for onion shape grain structure
- Dour Metal cannot sub contract extrusion and will recommission its extrusion press

New collaboration with NBM Nanomateriala specialized in high energy ball milling

- Patented high energy ball milling reactors, potentially lower ball milling time (less than 5h)
- 5 production lines, ~10 to 40t/year, making powder for cutting tools, Ti powder for bio applications,…
- Significant experience in ball milling under controlled atmosphere for tight impurity control
- Well-equipped lab for powder quality analysis
- NBM will ball mill ~ 20kg of gas atomized FeCrAl powder from ATI and run 3 to 4 trials to optimize BM parameters

New ODS alloys: Gas Atomization Reactive Synthesis + 5h Ball Milling Collaboration with Ames Lab.

ORNL
Sebastien Dryepondt
David Hoelzer

Ames Lab.
Iver Anderson
Joel Rieken

Atomized Powder

HIPed 700°C

H.T. 1200°C

Hot Rolled 650°C

Annealed 1000°C

Hot tensile tested (RT - 800°C)

Dispersoid formation

Grain size
Dispersoid spatial distribution

Grain Recrystallization

Ball Milled 5 hr.

HIPed 850°C

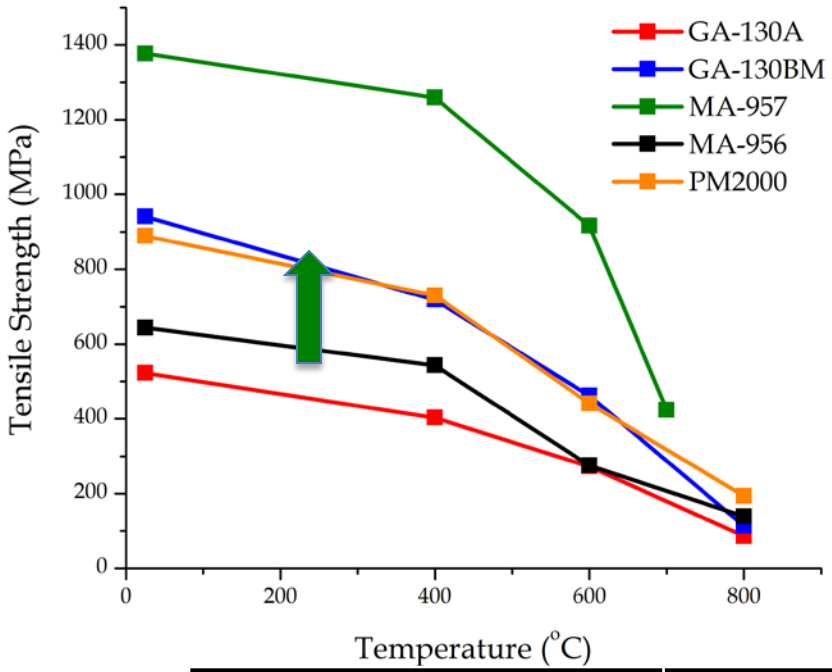
H.T. 1000°C

Hot Rolled 650°C

Annealed 1000°C

Hot tensile tested (RT - 800°C)

Ball milling improved the tensile strength of GA-130 alloy

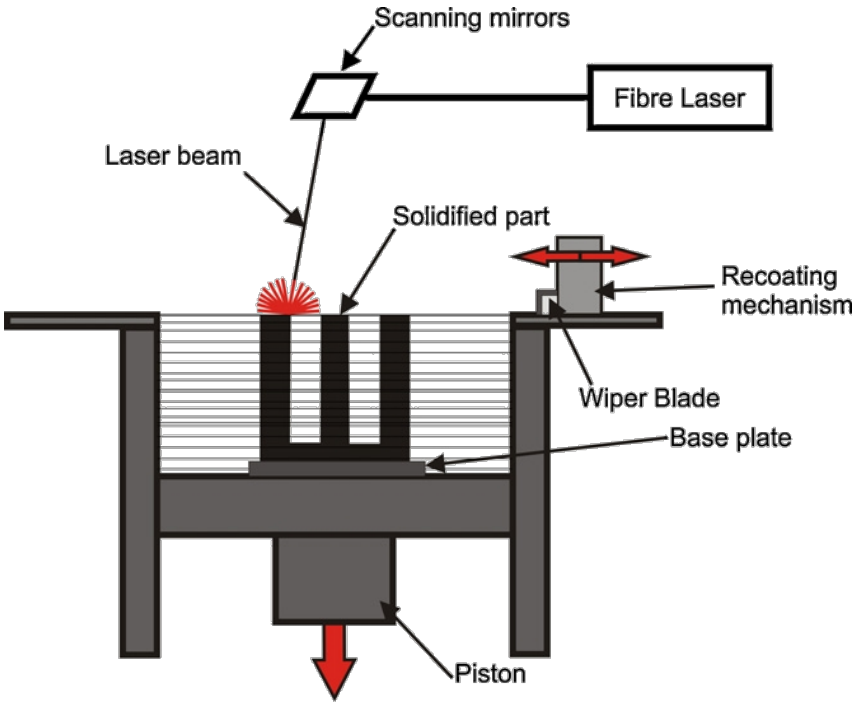


GA-130:Fe~16Cr-1W-0.6Ti+Y and O

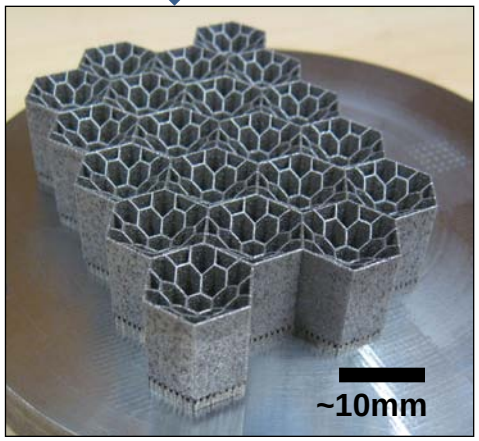
- Significant effect of 5h ball milling
- Increase dispersoid volume fraction and control grain size to reach MA-957 strength
- Similar work with FeCrAl powder

Alloy	Dispersoid (vol.%)	Grain Size (μm)
GA-130A	0.4	10.6 ±3.0 (fully recrystallized)
GA-130BM	0.4	3.8 ±2.2 (partially recrystallized)
MA-957	0.5-1.0	Small (unrecrystallized <1.0)
PM2000	1.0-2.0	Medium (partially recrystallized)
MA-956	1.0-2.0	Large (fully recrystallized)

Selective laser melting (SLM) at Liverpool



(real time)

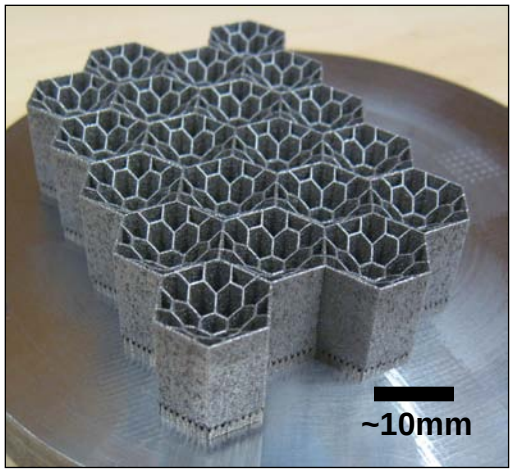


Additive layer by layer rapid prototyping technique

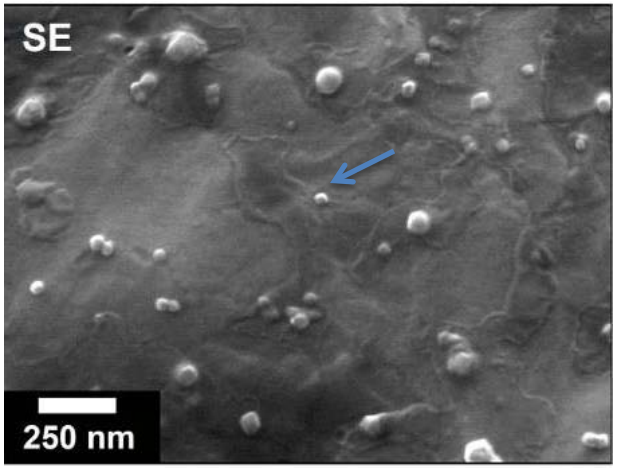
Repeated deposition of thin layers by laser melting

Fully dense solid freeform components from powder beds

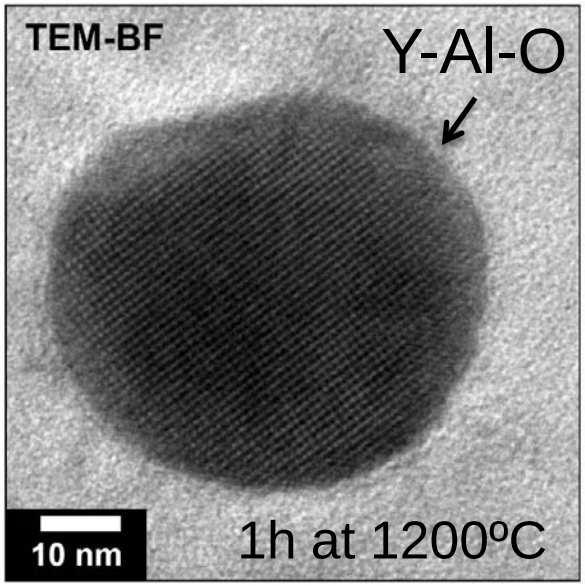
Nano oxide precipitates in SLM builds

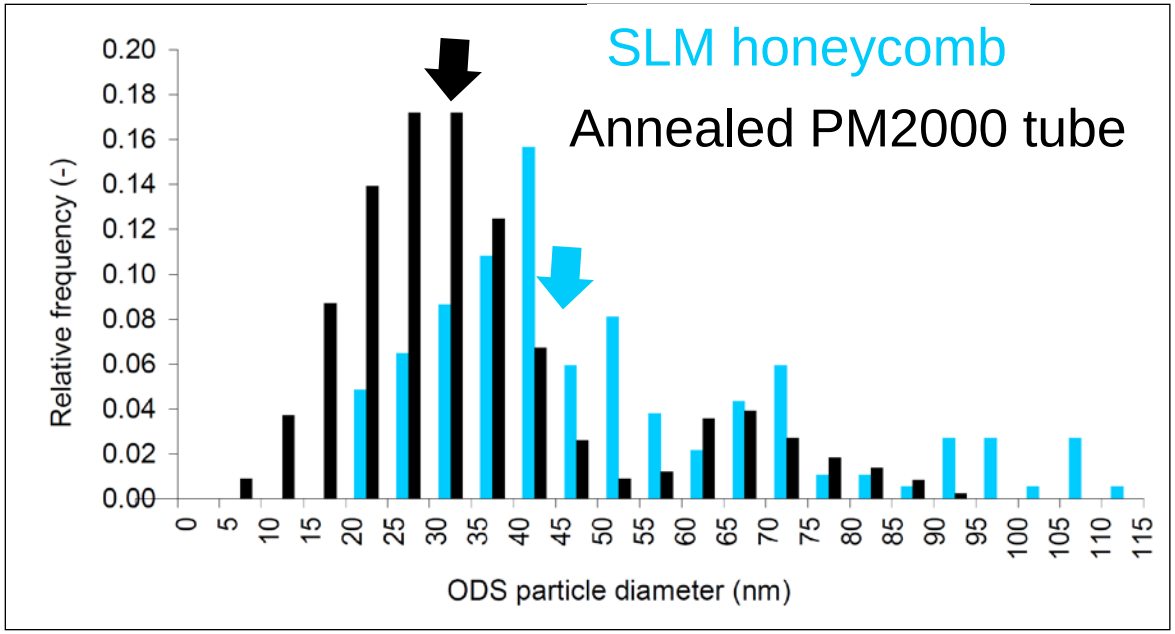


PM2000 honeycomb structure



SEM (etched 10%HCl in methanol)

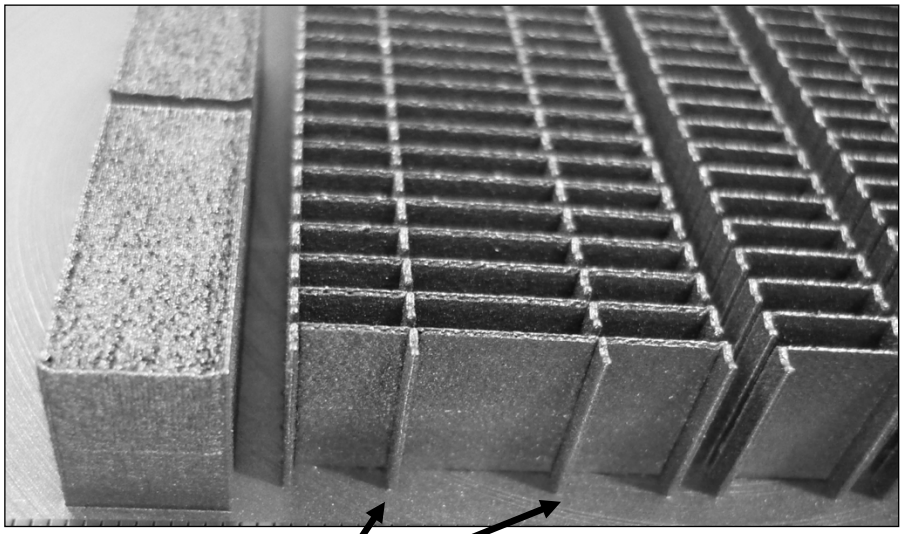




- ODS particles only ~10 nm coarser than conventional PM2000

- Y-Al-O particles after 1h at 1200ºC

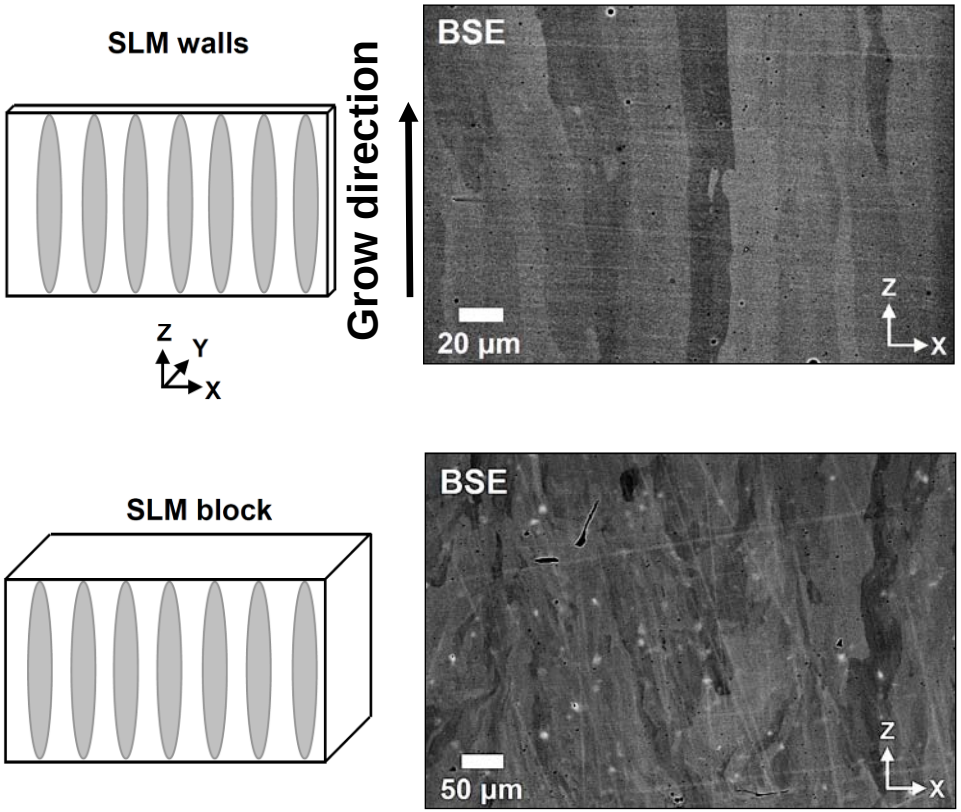
New SML builds for mechanical properties characterization



Vertical ribs for build stabilisation

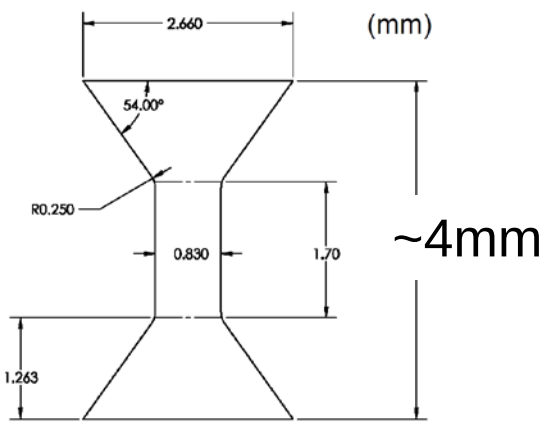
10mm

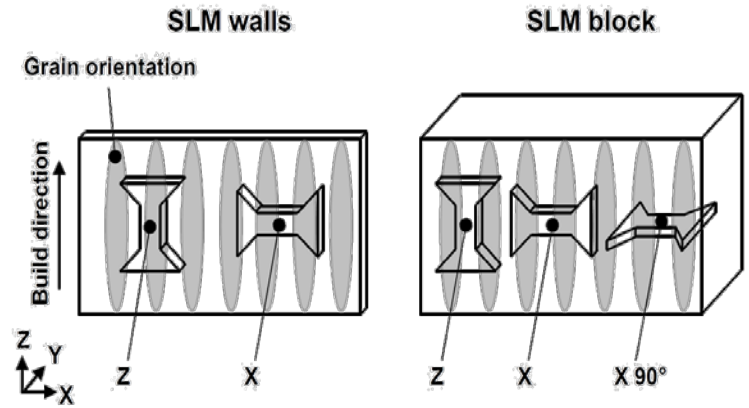
4 different structures:
- 1x scan walls: 180µm
- 2x scan walls: 250µm
- 3x scan walls: 340µm
- Solid blocks



Columnar grain structure related to growth direction

Micro-testing of SML builds at ORNL*





SLM

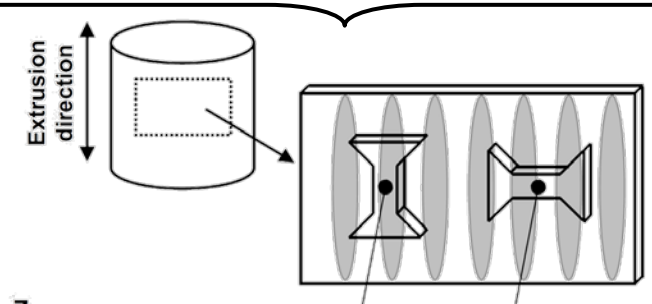
1x scan walls
2x scan walls
3x scan walls
Solid blocks

As grown / **Annealed at 1200°C/1h** / **Along the grow direction** / **Perpendicular to the grow direction (90° block only)**

Standard PM2000

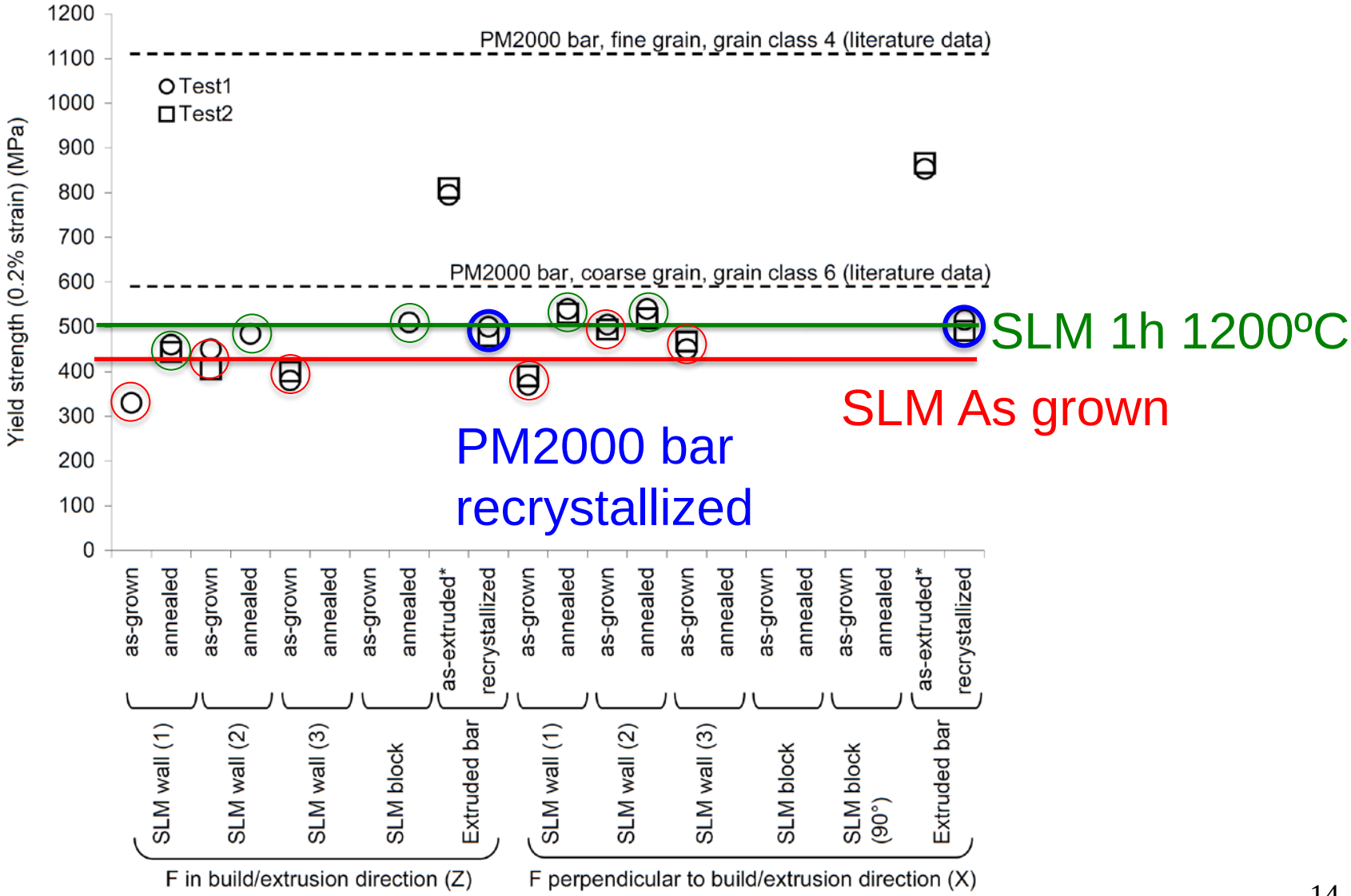
PM2000 extruded bar as-extruded
PM2000 extruded bar recrystallized

Along the extrusion direction / **Perpendicular to the extrusion direction**

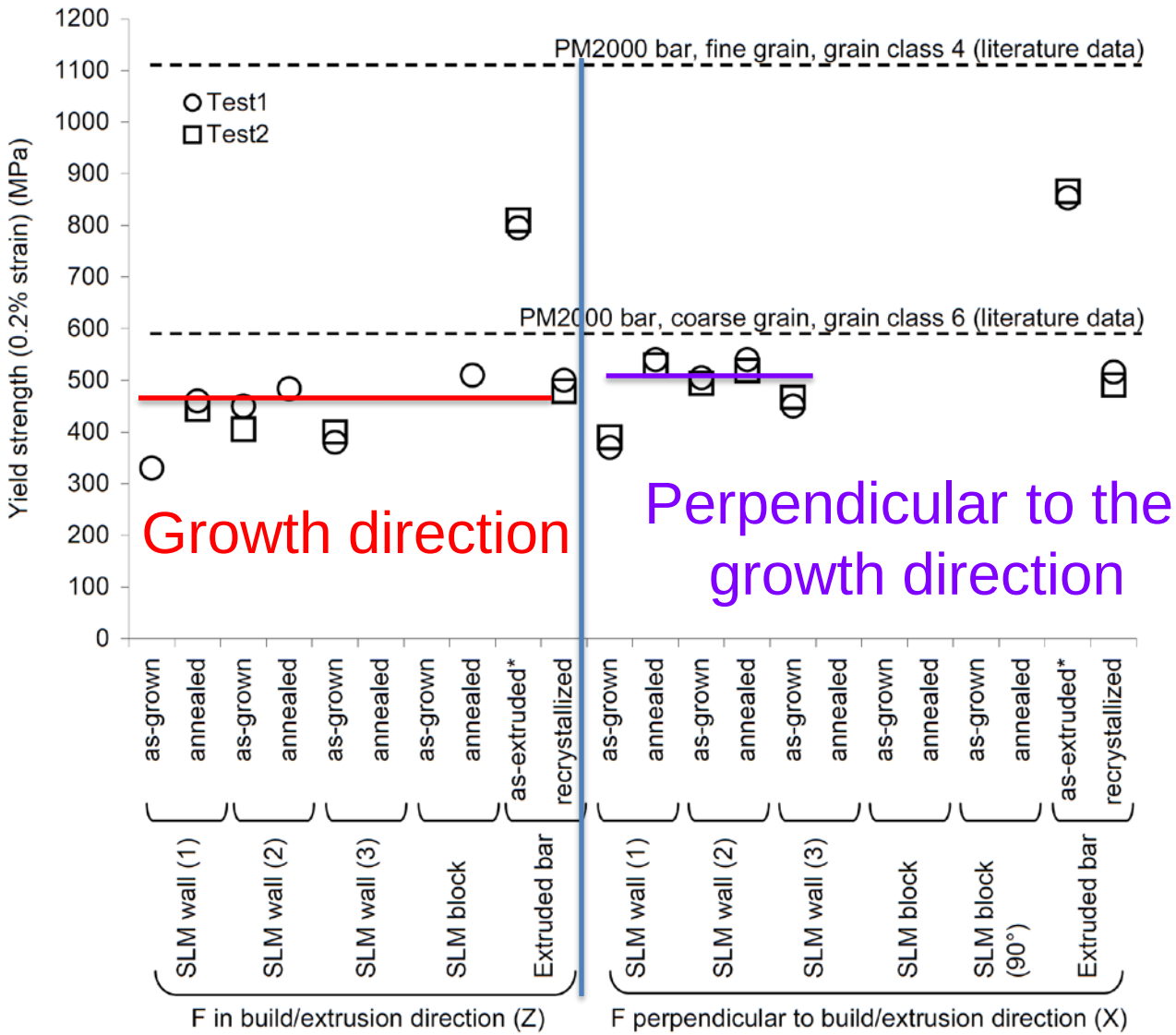


*Collaboration with Amit Pandey

Increase of Yield Strength at room TºC after 1h 1200ºC anneal



Slightly higher YS for specimens perpendicular to the growing direction



Experimental work for predictive model development

Materials

Alloy	Fe	Cr	Al	Si	Ti	Y	C (ppm)	N (ppm)	O (ppm)	S (ppm)
MA 956	69.5	20.1	8.78	0.13	0.4	0.24	640	608	6490	41
PM2000	69.4	18.9	9.82	0.07	0.49	0.22	430	104	8050	34
PM2K	68.7	19.1	10.5	0.04	0.52	0.23	60	318	8028	13

2 different batches of PM2000

1 batch with very low impurities level

Experiments

1h cycle, 1200ºC

specimen mass gain + time to breakaway

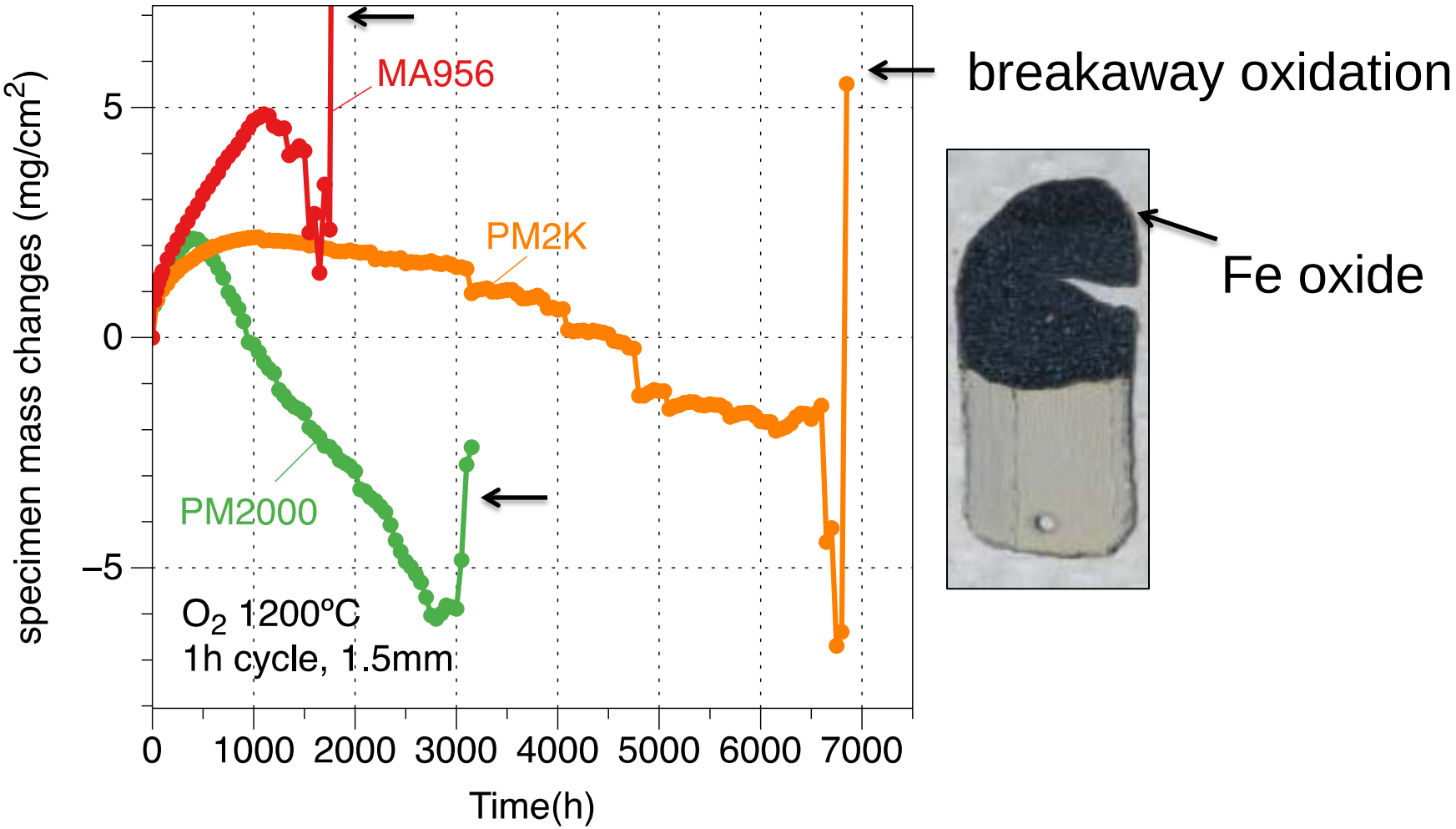
- O_2
- air+10%H_2O

~50%CO_2/50%H_2O + 0.075 O_2

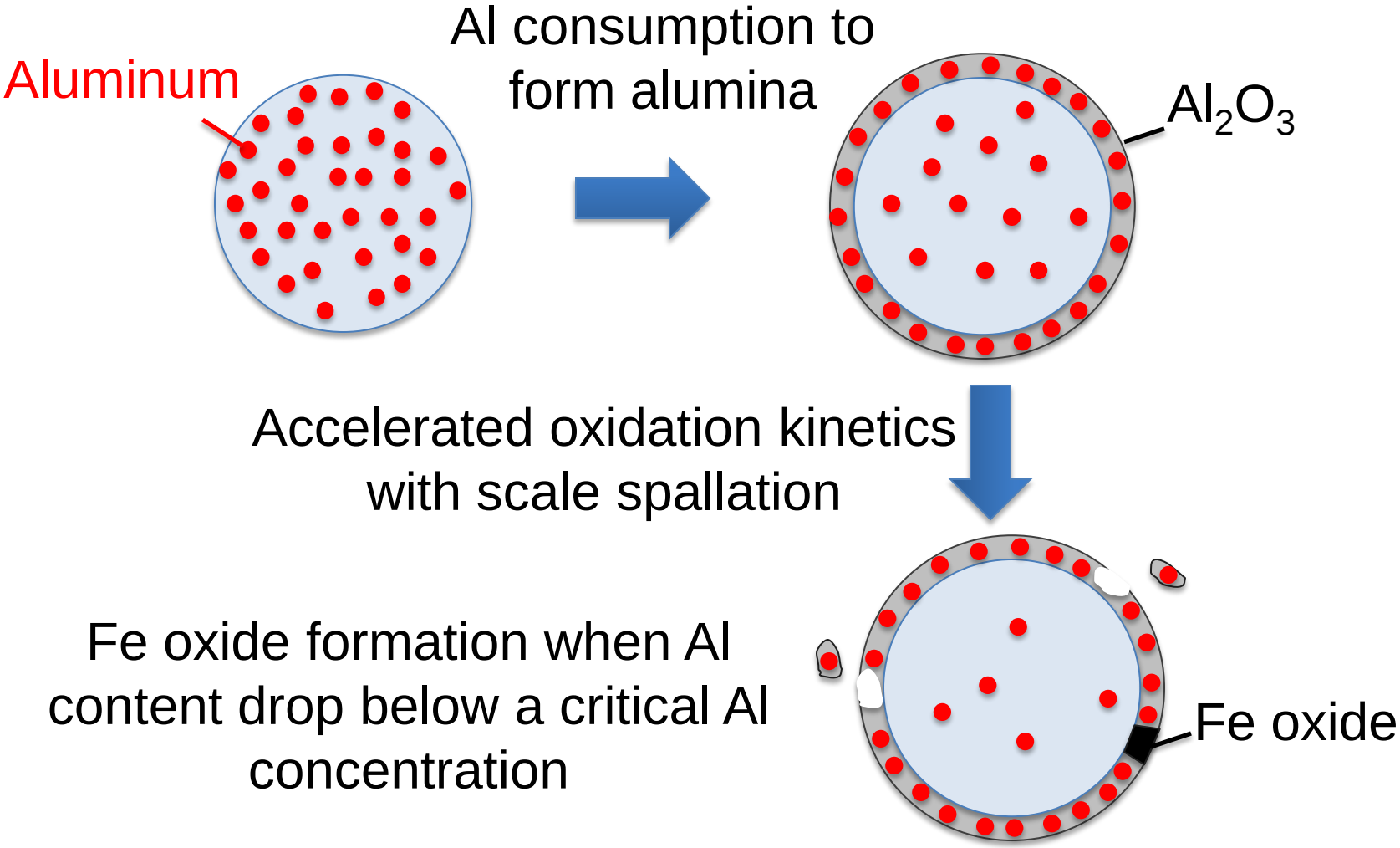
Objective

Predictive lifetime model based on Al consumption to form Al_2O_3 in representative environments

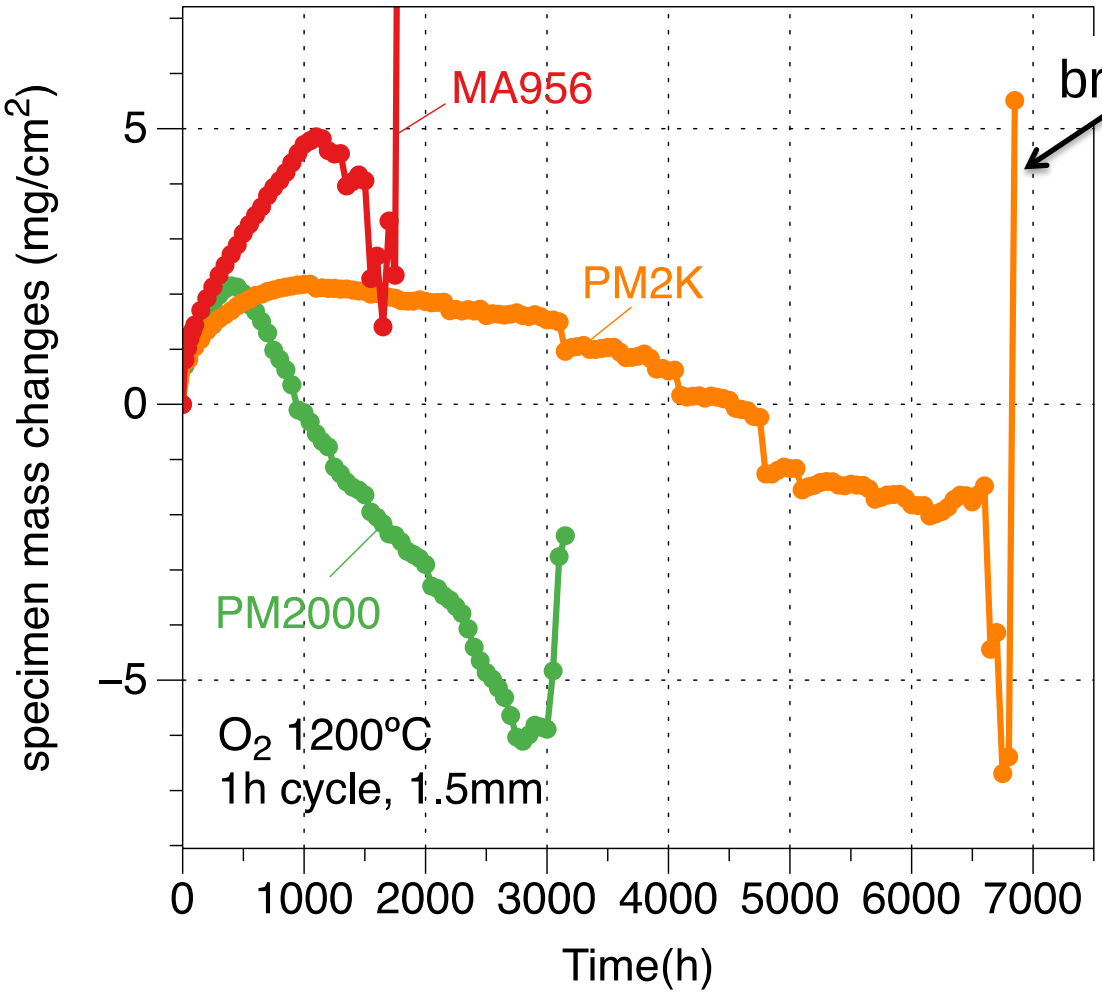
1h cycle at 1200ºC: end of life = Fe oxide formation (breakaway oxidation)



Breakaway oxidation is due to Al consumption to form Al_2O_3



1h cycle lifetime at 1200ºC: low S high Al PM2K> 2X PM2000> 2XMA956

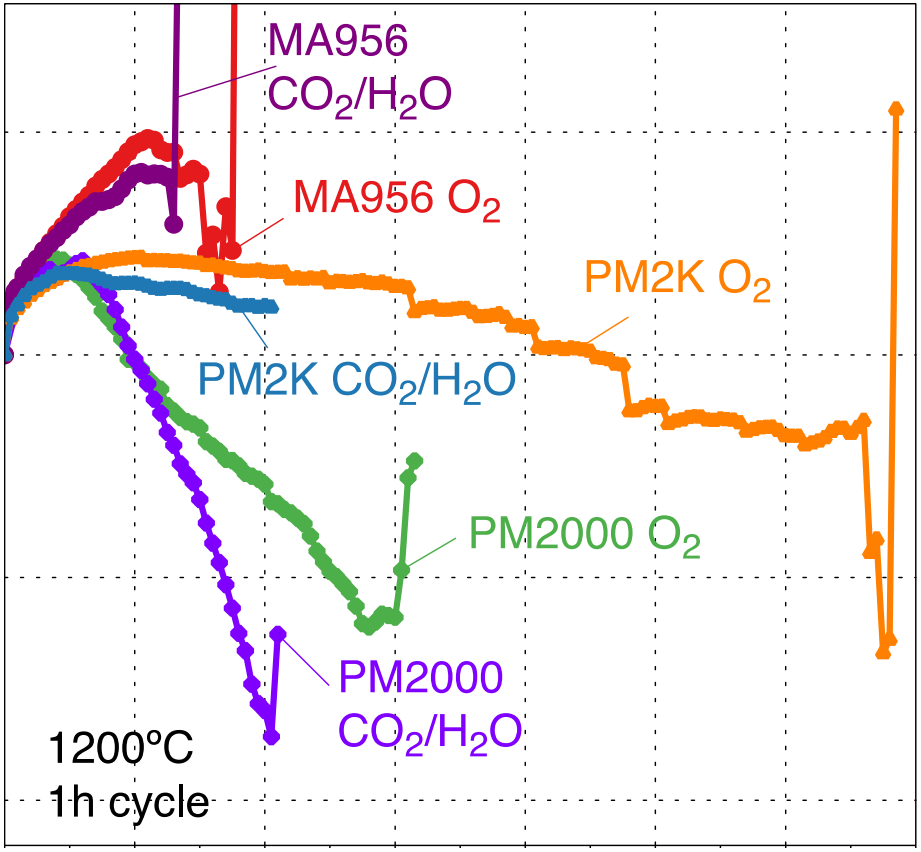


breakaway oxidation = end of life

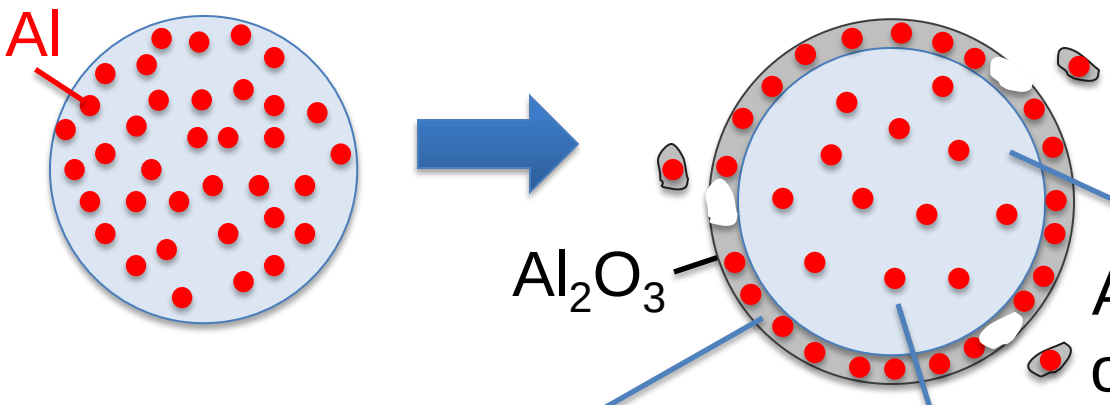
Alloy (at%)	Al	C (ppm)	S (ppm)
S67956	8.77	635	41
1.4768-1	9.82	430	34
1.4768-2	10.48	60	13

Careful control of alloy fabrication is key for high temperature performance

Lower lifetime for specimen exposed to O_2 buffered 50%CO_2/50%H_2O



Model based on the consumption of Al down to a critical Al concentration $C_{b,Al}$



S: Surface
V: Volume

Al consumption depends on S and cyclic oxidation kinetics $F_{oxidation}(t)$

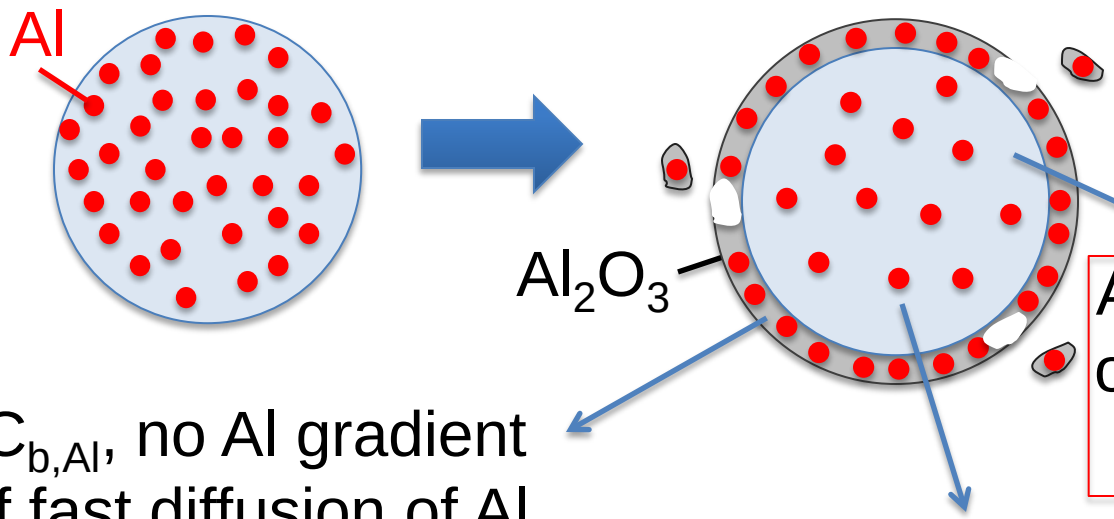
$C_{b,Al}$, no Al gradient if fast diffusion of Al to the surface

Al reservoir depends on V and C_0

Al left at t: $$V \cdot C_{Al} = V \cdot C_{0,Al} - S \cdot F_{oxidation}(t)$$

Al left at rupture: $$V \cdot C_{b,Al} = V \cdot C_{0,Al} - S \cdot F_{oxidation}(t_b)$$

Model based on the consumption of Al down to a critical Al concentration $C_{b,Al}$



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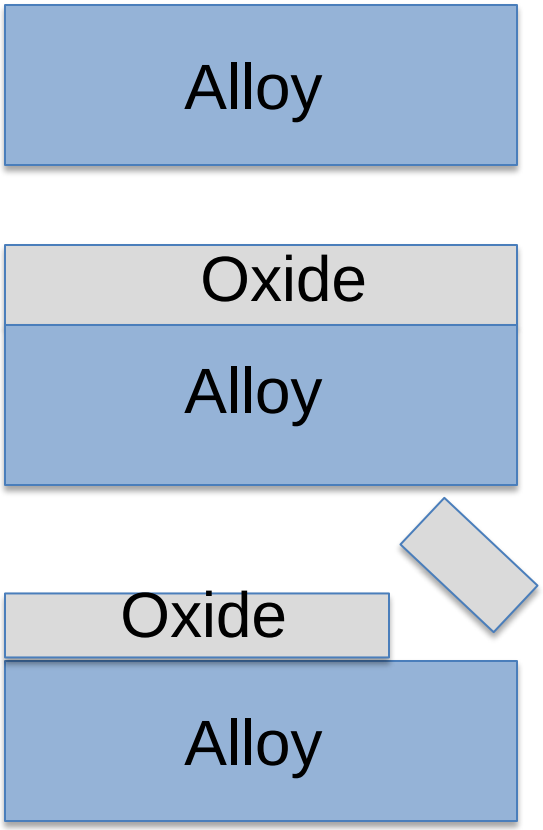
COSP Cyclic Oxidation Model based on 2 parameters Kp and Q



Isothermal

Parabolic oxide scale growth

$$\left(\frac{\Delta W}{S}\right)^2 = k_p t$$

ΔW Weight change
S: Surface

K_p = 0.025 $mg^2cm^{-4}h^{-1}$ from isothermal data

Cooling

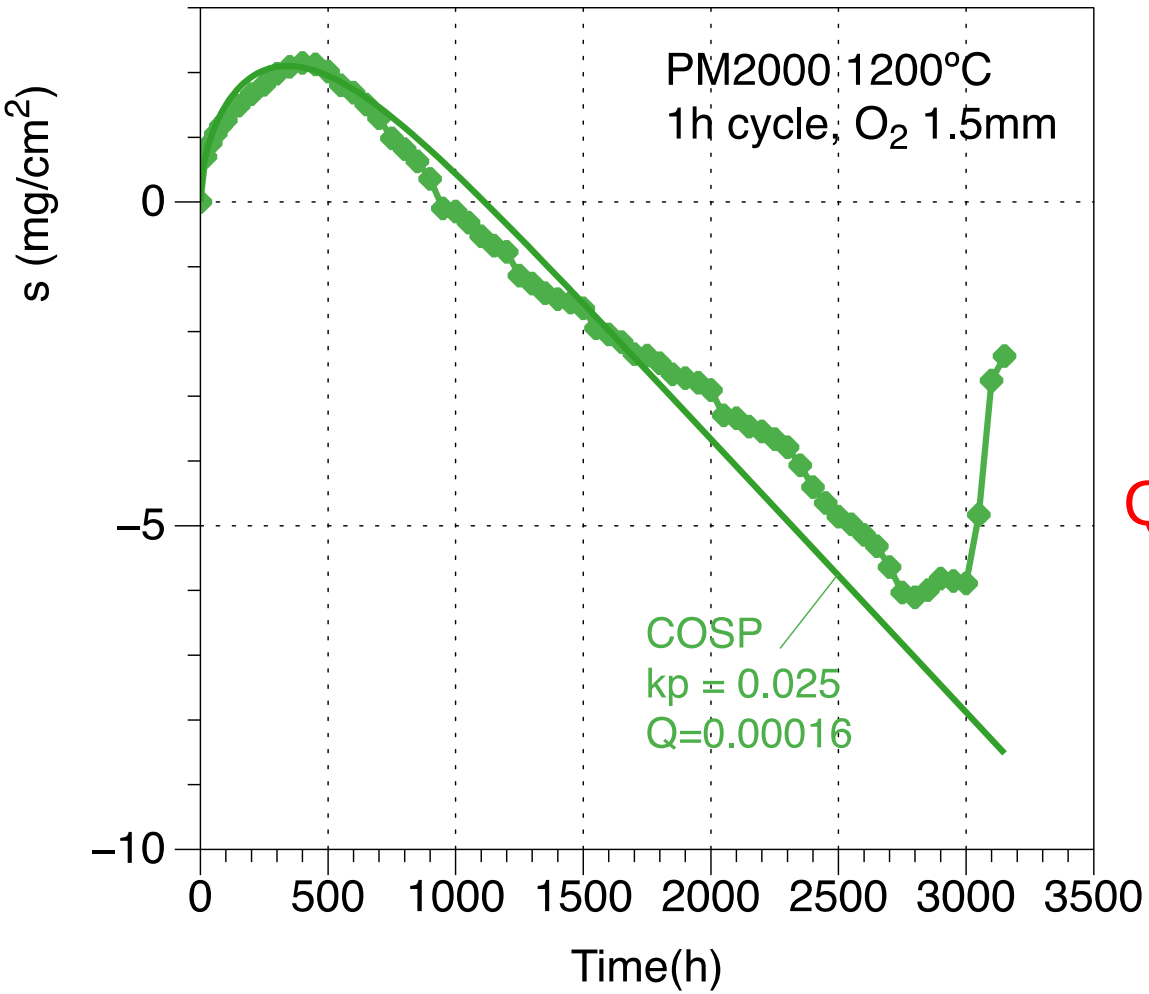
Spallation at each cyclic of the same fraction of the oxide weight

$$F = W_{ri} Q$$

W_{ri} weight after ith cycles

Determination of spallation coefficient Q from cyclic data

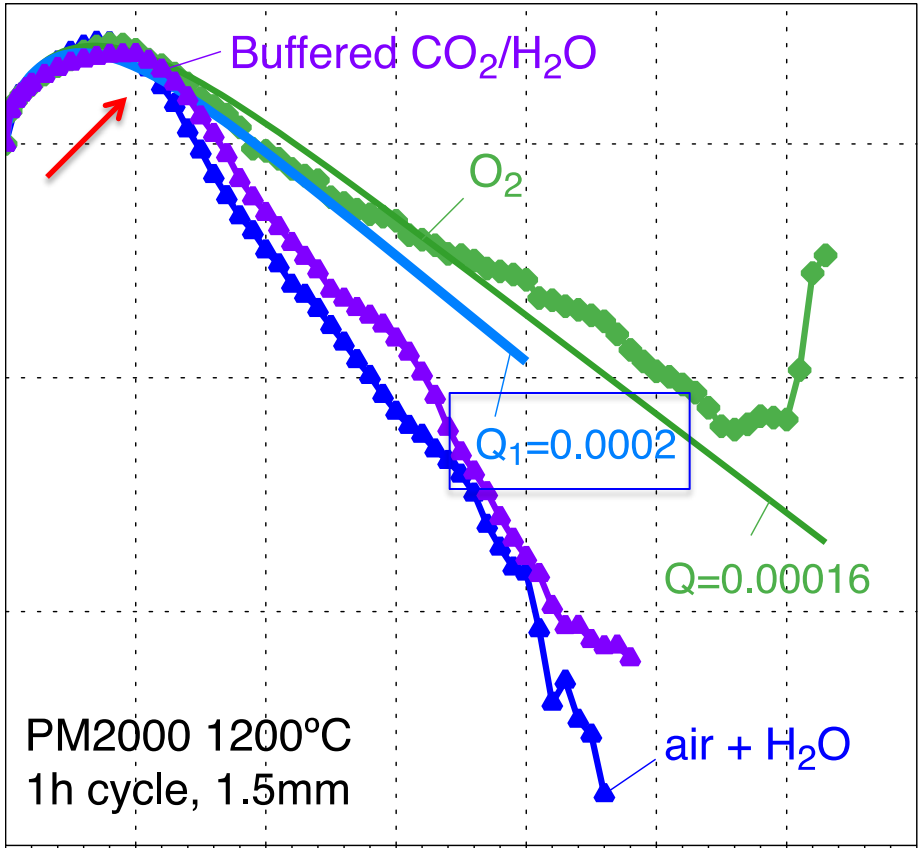
Good fitting of experimental data for alloy PM2000 in O_2



Calculation for thickness>1mm (4 specimens)

Q = 0.00016±0.00002

2 Different spallation coefficients Q to fit the curves in air+H_2O and CO_2/H_2O



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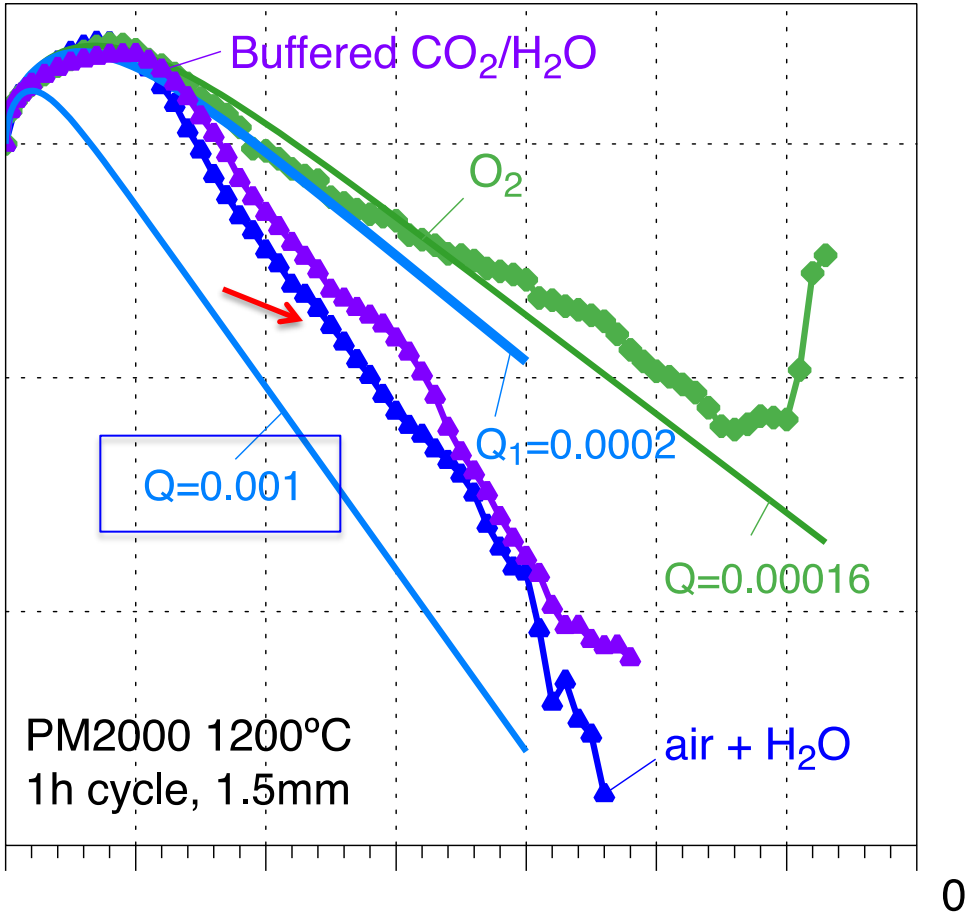
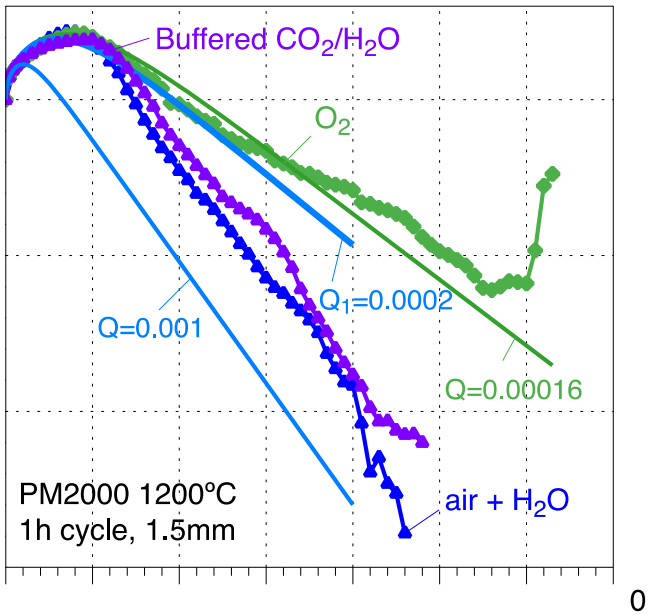
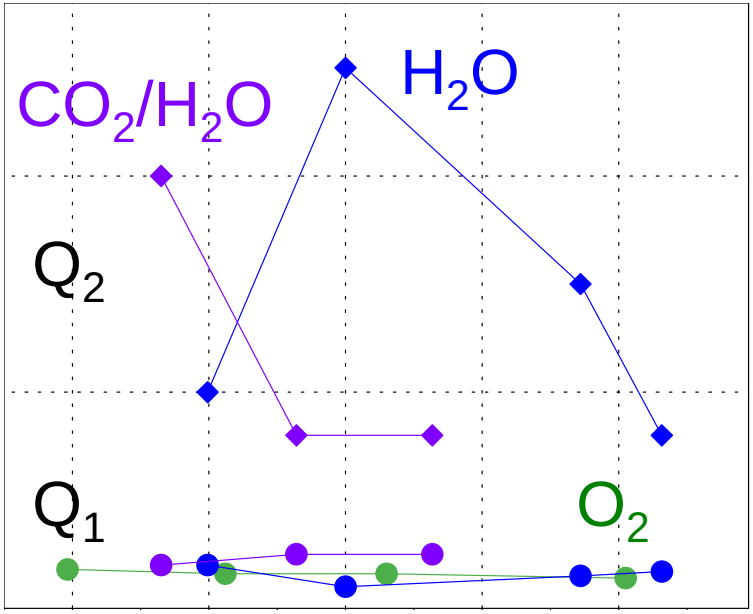


2 Different Q values to fit the curves in air+H_2O and CO_2/H_2O

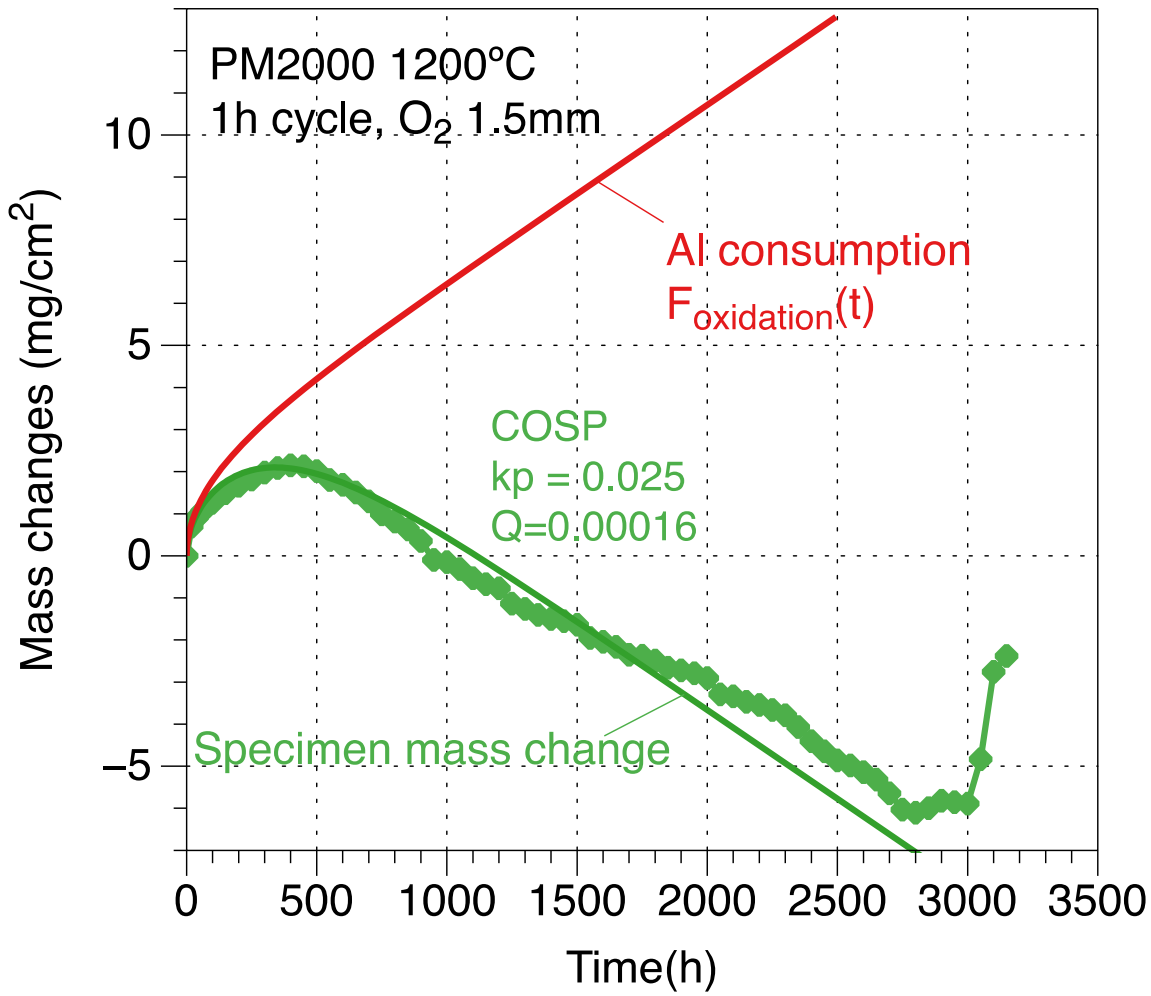




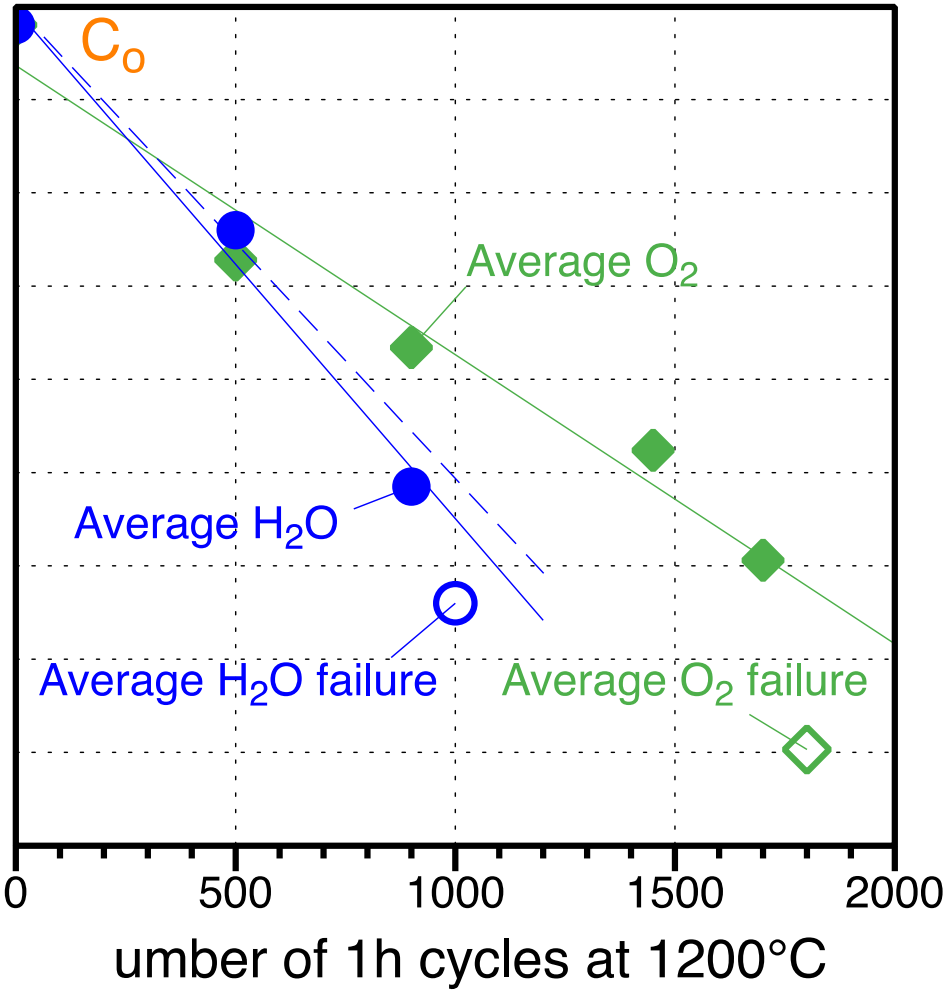
Acceleration of spallation
in H_2O and CO_2

COSP allows the determination of Al consumption to form Al_2O_3

Al left at t: $$V \cdot C_{Al} = V \cdot C_{0,Al} - S \cdot F_{oxidation}(t)$$

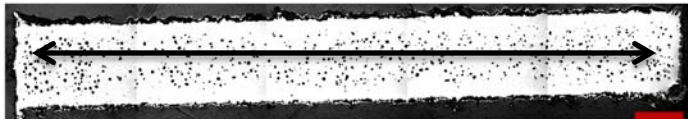


Determination of Q from measurement of Al in exposed specimens



Al remaining content was measured for MA956 specimens after 500, 900 1450 and 1700 cycles

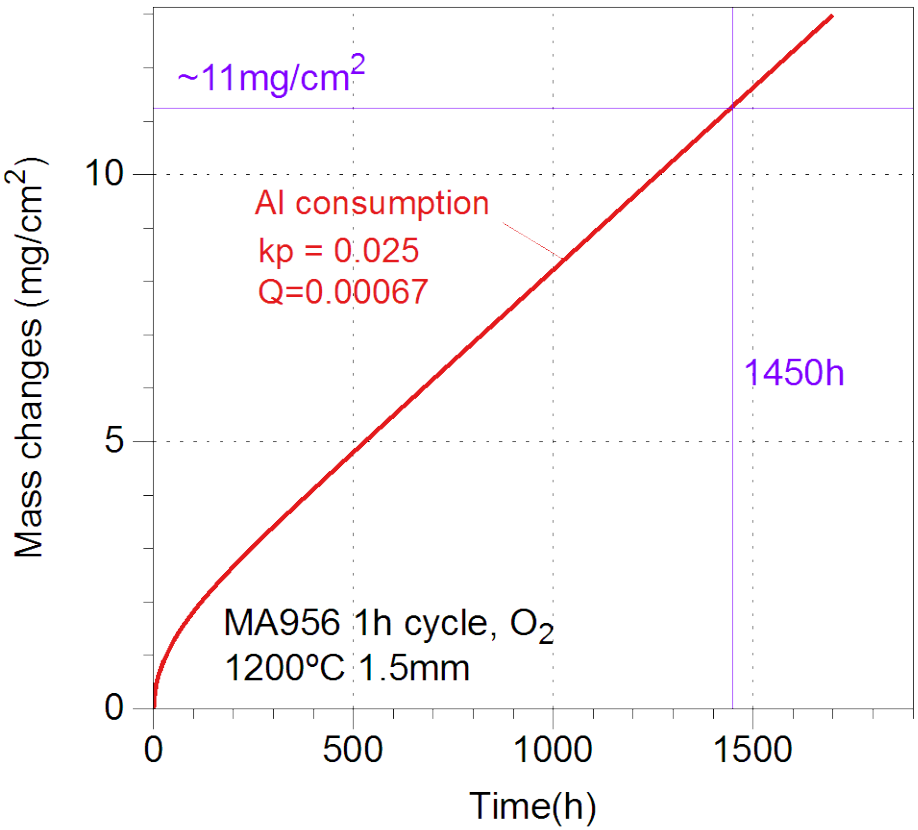
specimen cross-section

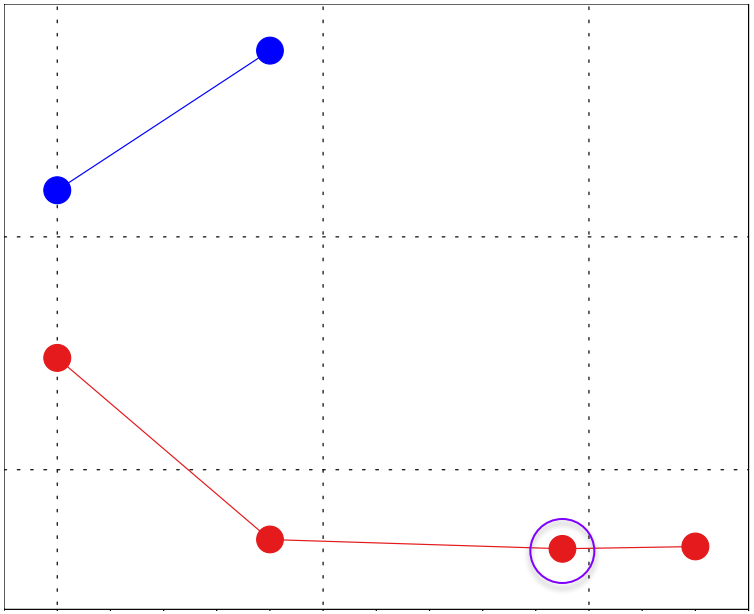


Al measurement by electron microprobe (EPMA)

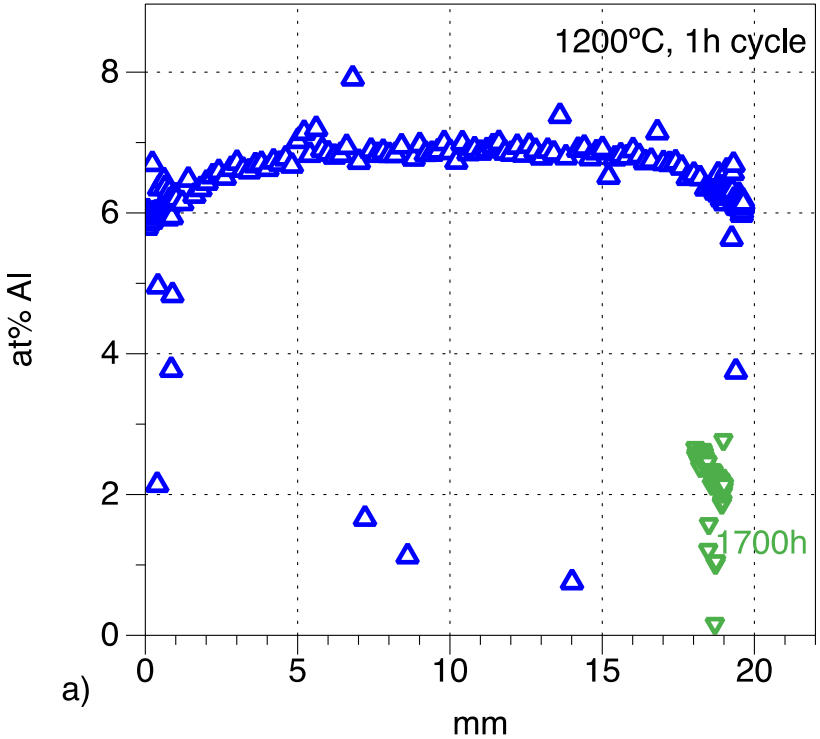
Similar Q values in air for t>500h, Values significantly higher in H_2O

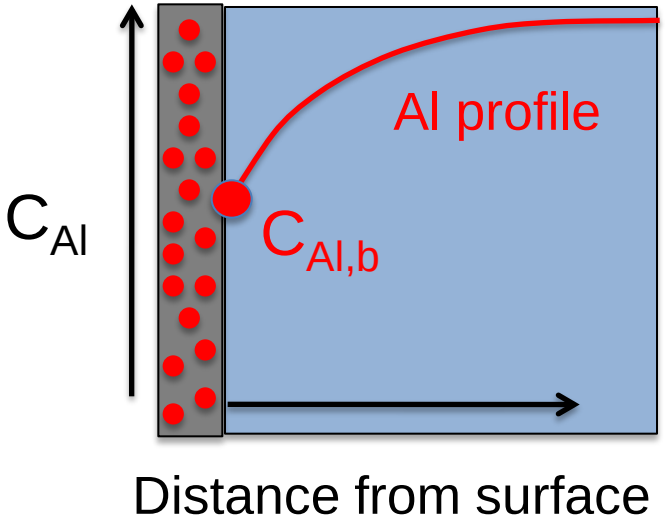
After 1450h, Al dropped from 8.9At% to ~ 4.2At%
This corresponds to a consumption of ~11mg/cm^2
for a 20x10x1.5mm coupon





Better evaluation of Al critical concentration $C_{b,Al}$





- Al gradient in the alloy is related to specimen geometry
- Diffusion model to take into account the gradient
- Initiated exposure of cylindrical specimens

FY13 Milestones

- Properties of joints made by friction stir welding

Successful welding of PM2000 plate. Need FY13 funding for mechanical testing

- Characterization of ball milled GARS alloys

Joel Rieken's presentation at TMS2013

- Lifetime model integrating effect of specimen geometry

Started testing cylindrical specimens + Diffusion model

- Survey of potential industrial partners

On going collaboration with Dour Metal and new partnership with MBN Nanomaterialia SpA

- New ODS component and one new coating by additive manufacturing

New thin walls and new coating fabricated by SLM

Future work

- Characterization of commercial products produced by Dour Metal and MBN.
- High temperature creep and tensile testing of SLM builds.
- Comparison of two additive manufacturing techniques: SLM at Liverpool and electron beam melting at ORNL.
    - Control of defect and grain shape
    - Powder from Dour Metal and NBM
- Continue collaboration with NE on new ODS FeCrAl
- Development of 3D diffusion model to improve lifetime prediction + long term testing at 1100ºC

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