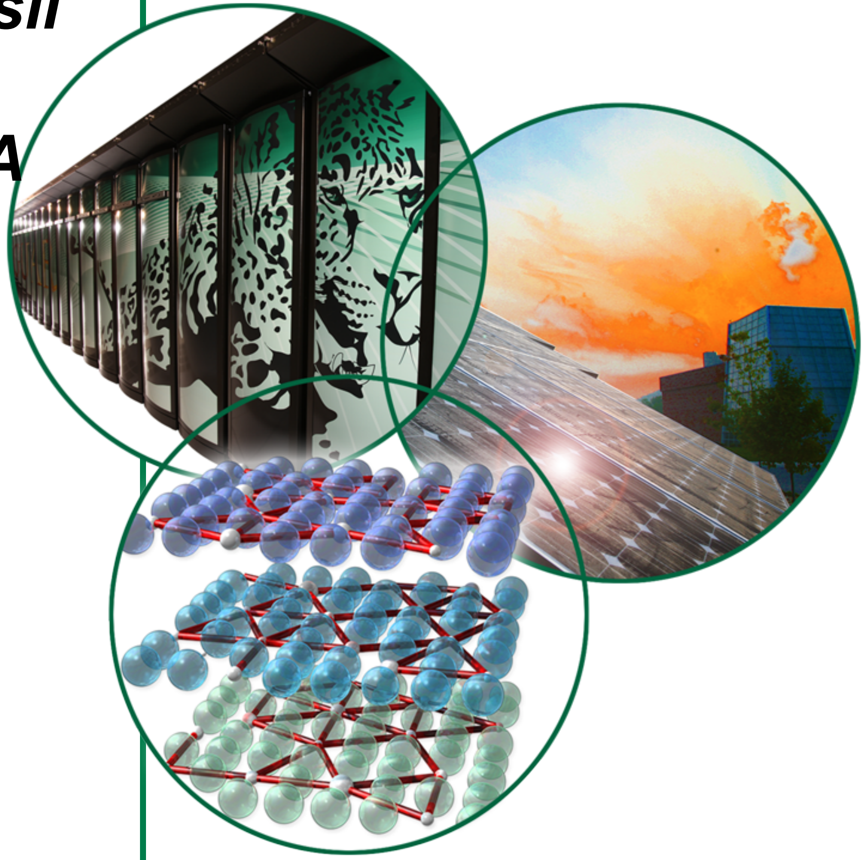
Qualification of new commercial ODS alloys

26th Annual Conference on Fossil Energy Materials
April 17-19, 2012, Pittsburgh PA



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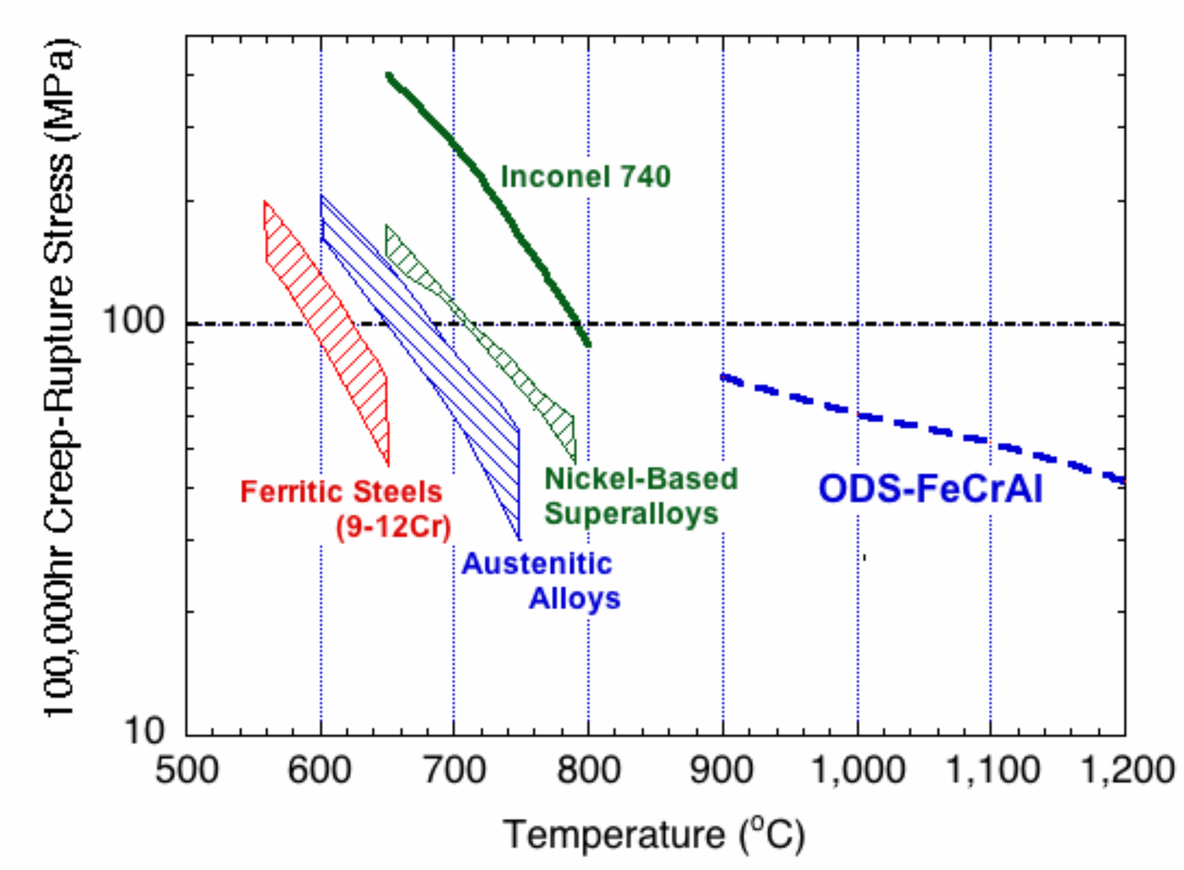
OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE DEPARTMENT OF ENERGY

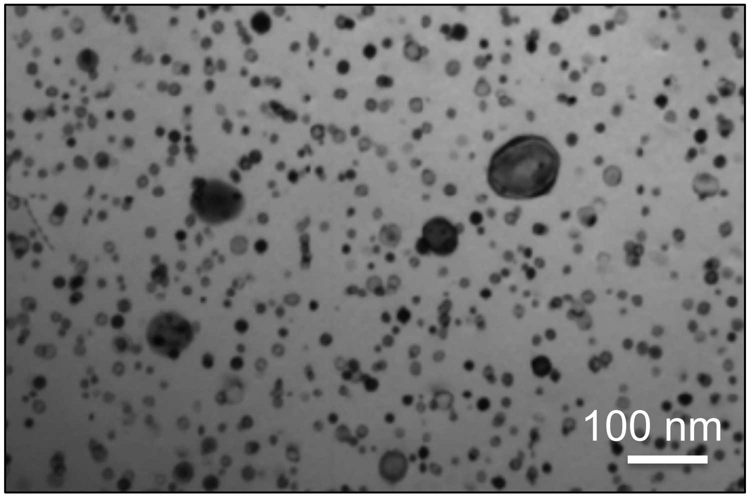
Great potential for high efficiency systems using FeCrAl-ODS alloys

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



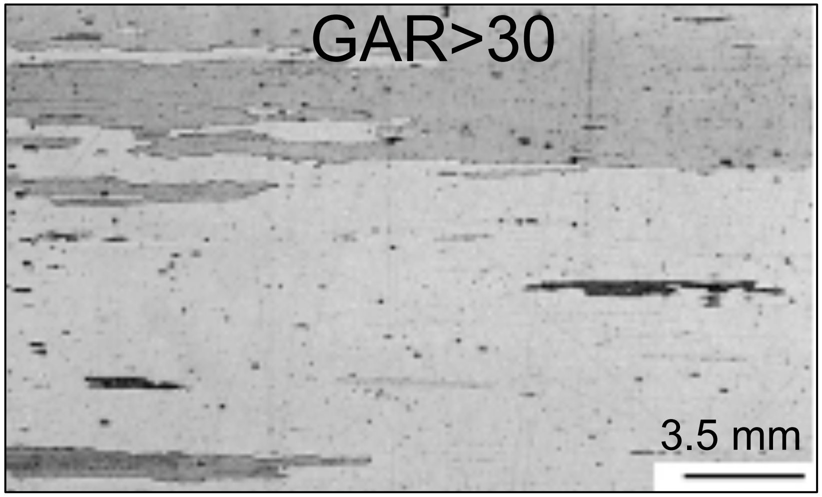
FeCrAl ODS alloys microstructure

PhD Thesis Laurent Marechal



Nano precipitates 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

Presentation Outline

Development of new supply of ODS alloys

- New commercial ODM751
- Ball milling of Gas Atomization Reactive Synthetic powder
- Synergies with nuclear industry

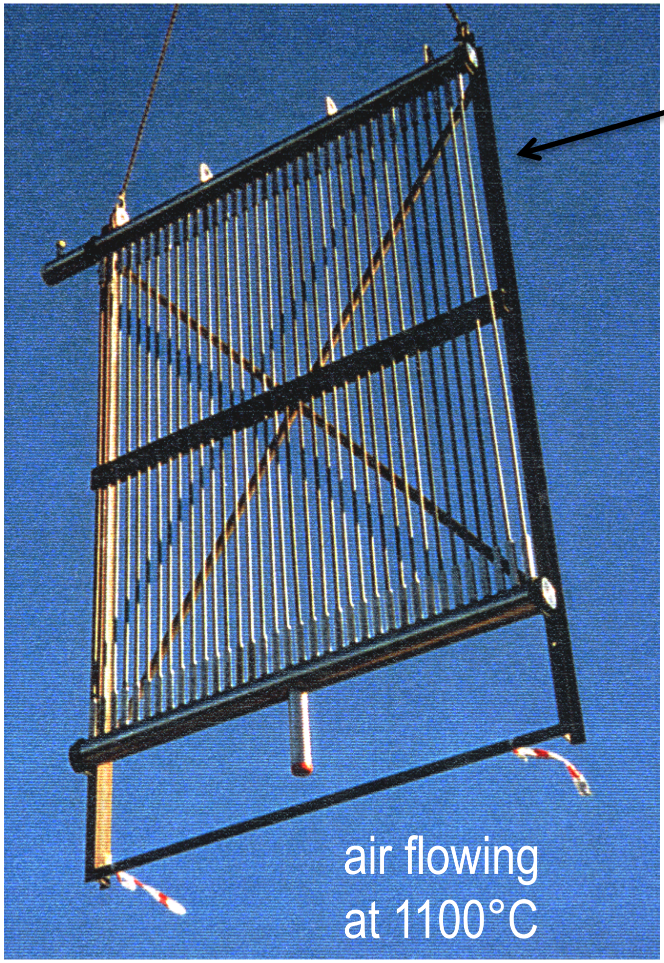
Selective laser melting of ODS alloys

- Complex near net shape components
- Coatings

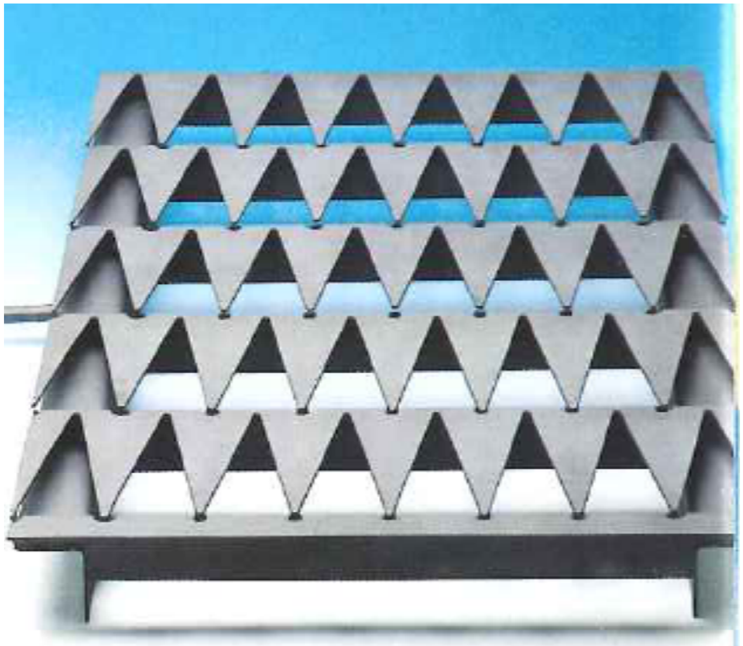
Life prediction for ODS-FeCrAl alloys in H_2O and CO_2 rich environments.

High Temperature Heat Exchanger Furnace components

British Gas demonstrator HTHE

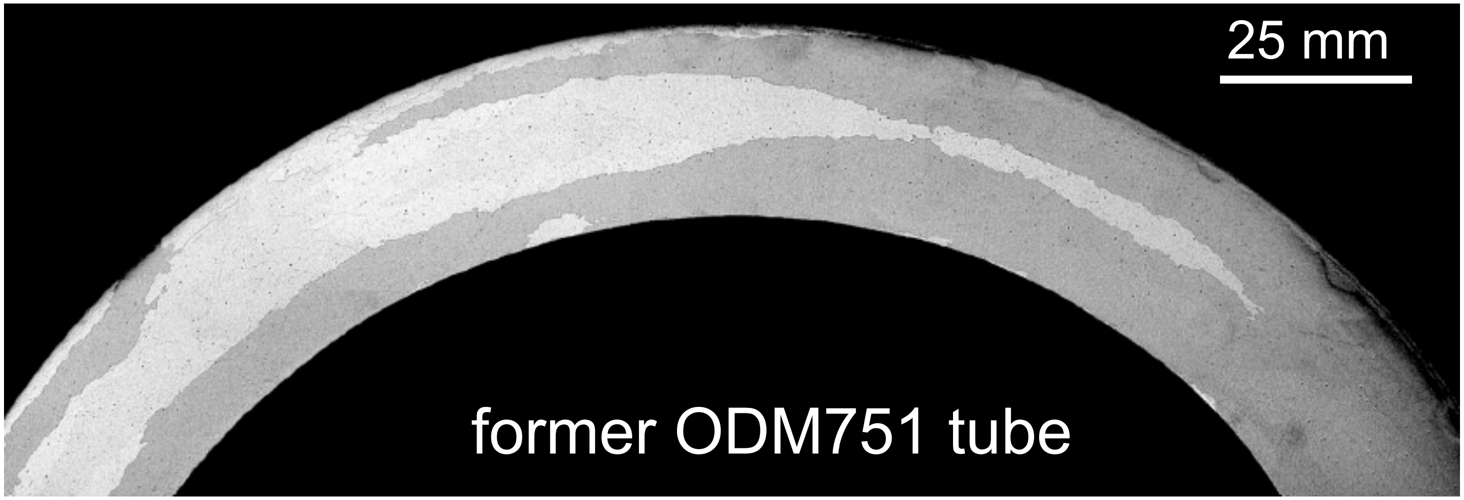


25 mm dia. x 4 m long ODM751 tube fabricated by Dour Metal



PM2000 Charge carrier for high vacuum furnace

New ODS alloys: Qualification of a new commercial ODM751 from Dour Metal



-Collaboration with Dour Metal Sro. to develop a new commercial ODM751 alloy and fabricate 200kg of rods and tubes with an “onion skin” grain structure with high hoop creep strength

Kaz4: New batch of ODS alloy ball milled under low vacuum

- Improvement of the ball milling facility to control the environment.

No evidence of coarse oxides

- supply chain lined up
- One rod and one tube

were extruded

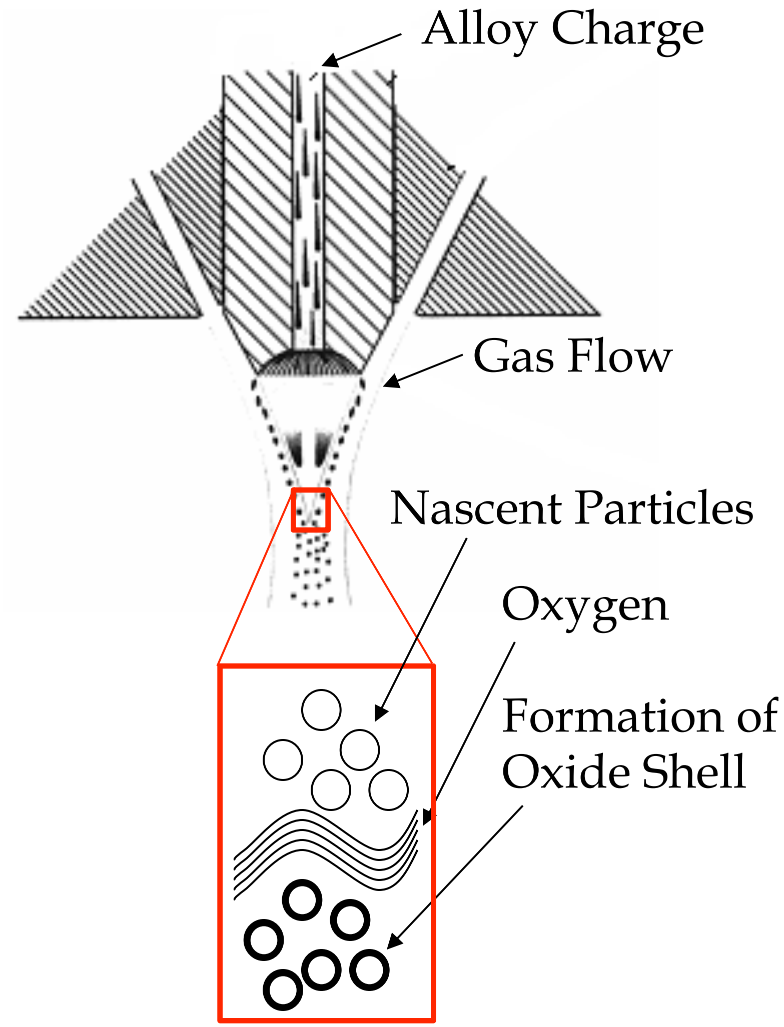
Good room T°C tensile properties:

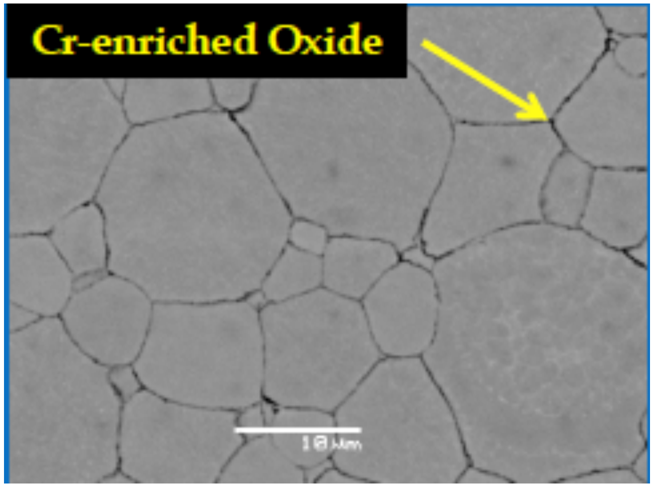
YS: 670MPa, UTS: 970MPa, A%:12%

- Composition out of range

but new batch under fabrication

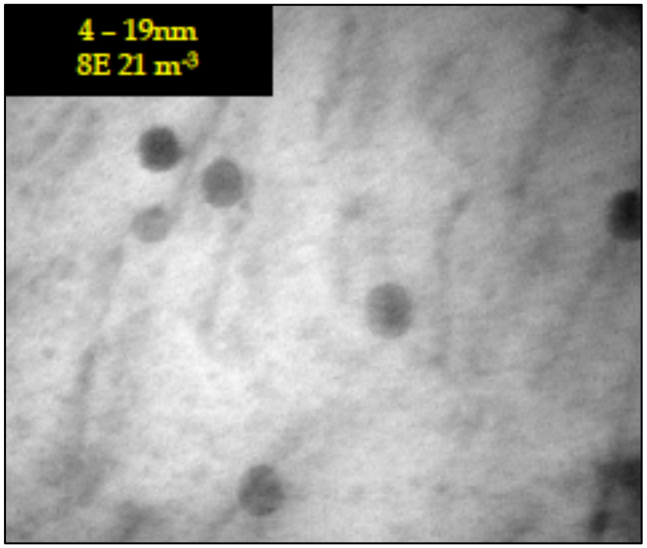
New ODS alloys: Gas Atomization Reactive Synthesis + Ball Milling





HIP 700ºC

O diffuses and reacts with Y



HT 1200ºC

Ames Lab, I. Anderson, JR Rieken

New ODS alloys: Gas Atomization Reactive Synthesis + Ball Milling

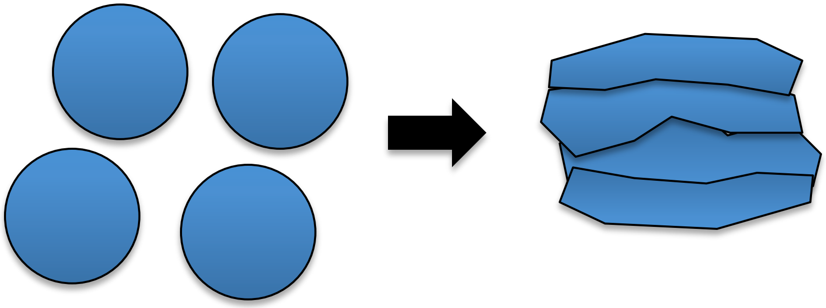
Collaboration with Dave Hoelzer at ORNL:

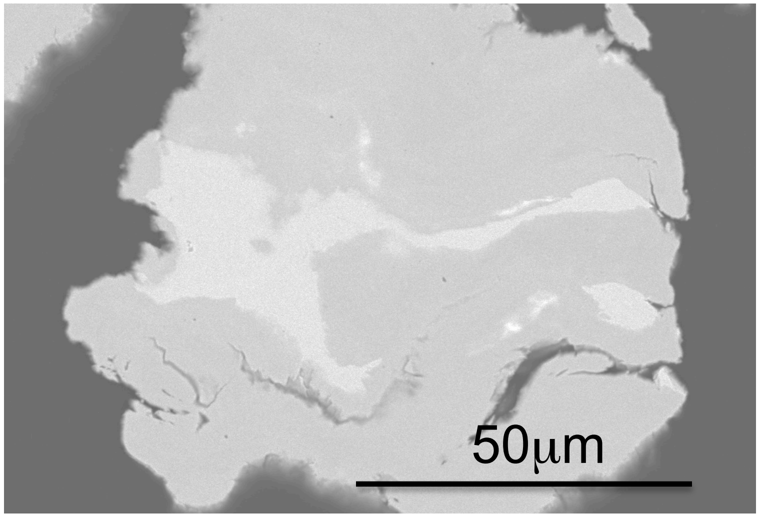
GARS Fe-15Cr-3W-0.5Ti-0.12Y + 5h ball milling

- To reduce O diffusion distance to react with Y
- Potential route to add Al

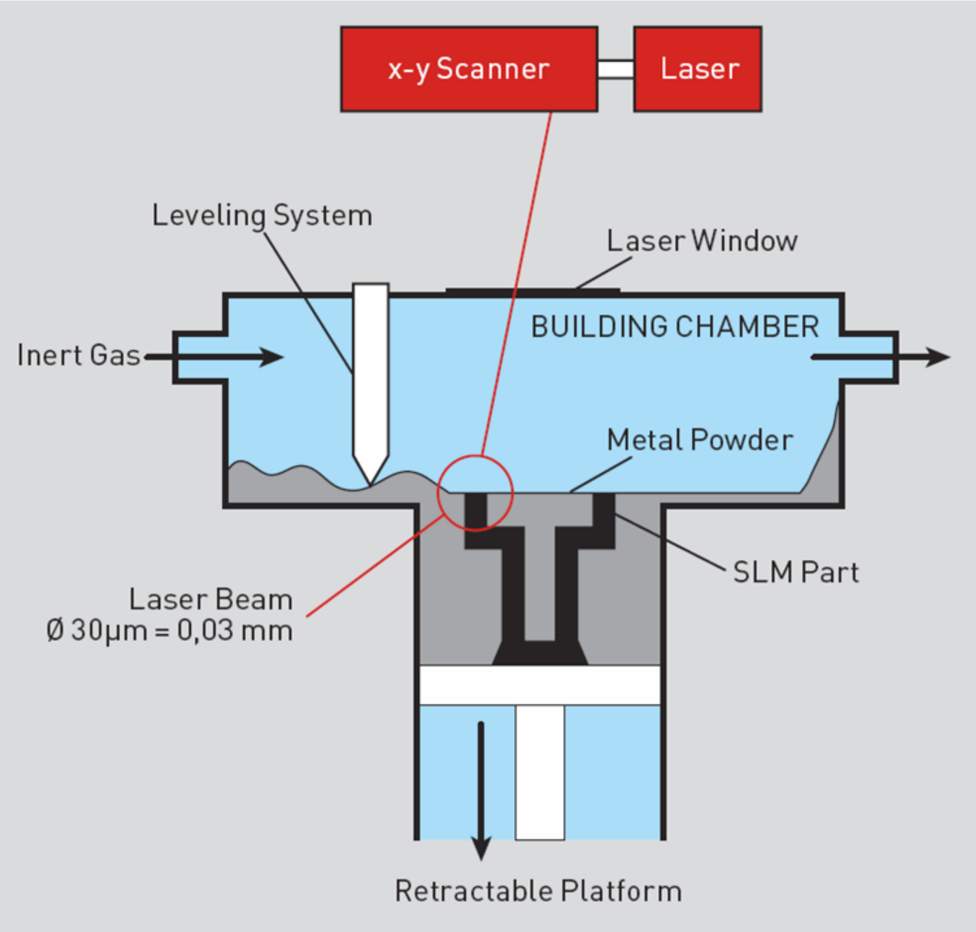
Decrease of ball milling time compare to conventional Gas Atomized powder:

- - decrease of cost
- - impurities control



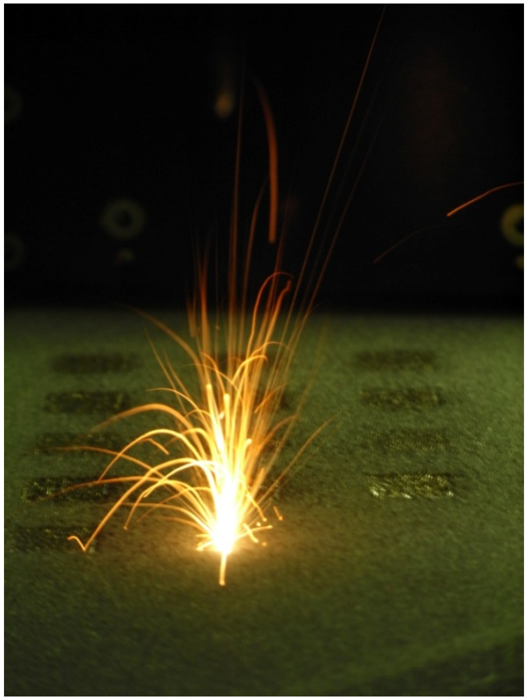


Selective Laser Melting (SLM) of PM2000 alloy powder



SLM process

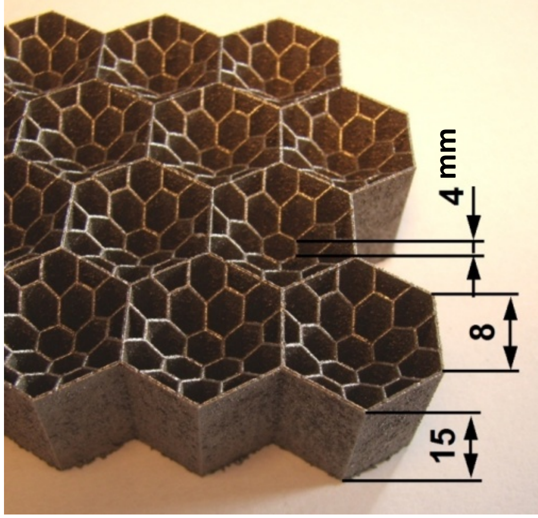
50W CW Nd:YAG laser
80µm spot size and
scan speed of 0.15m/s



SLM build in progress

Advantages of Selective Laser Melting in powder based additive manufacture

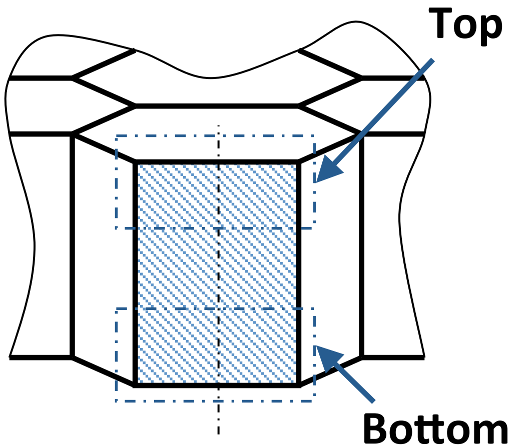
- Production of solid freeform component
- Production of thin layers from metal powder
- Processing under inert atmosphere or vacuum
- Use of CAD files for component shapes

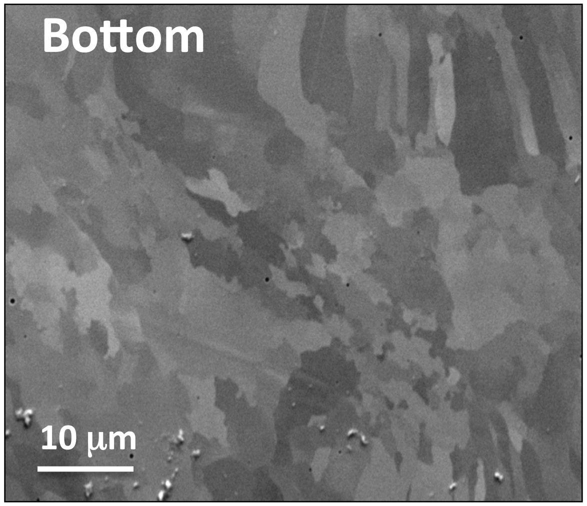


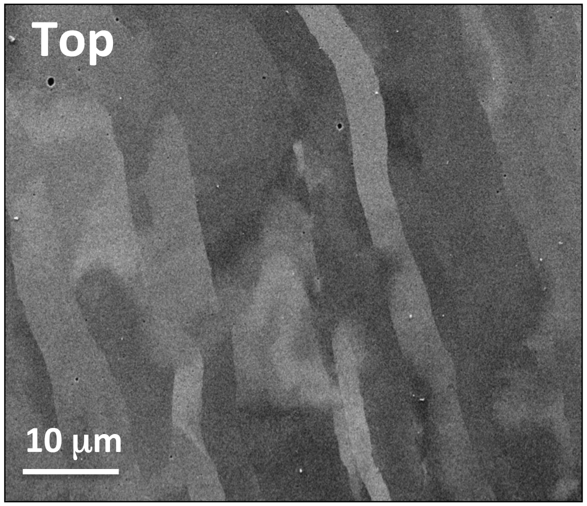
More complex hexagonal honeycomb structure as a near net shape component

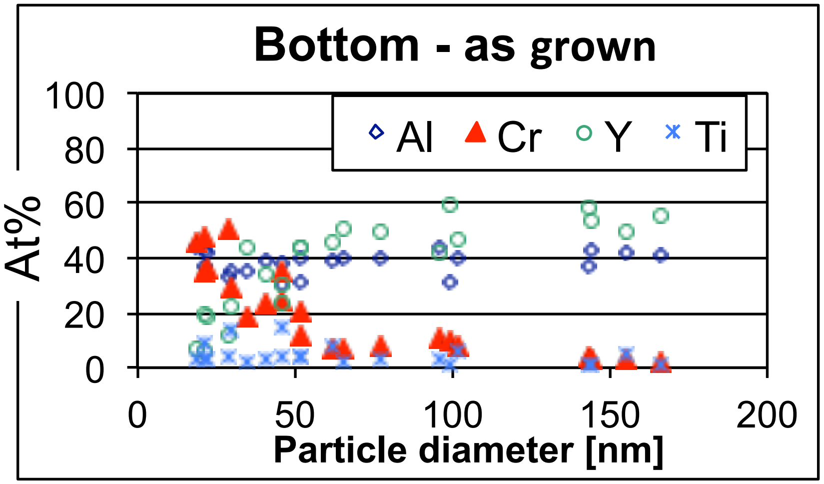
Use of SLM to apply ODS coating

SLM of PM2000 alloy powder to form a honeycomb structure





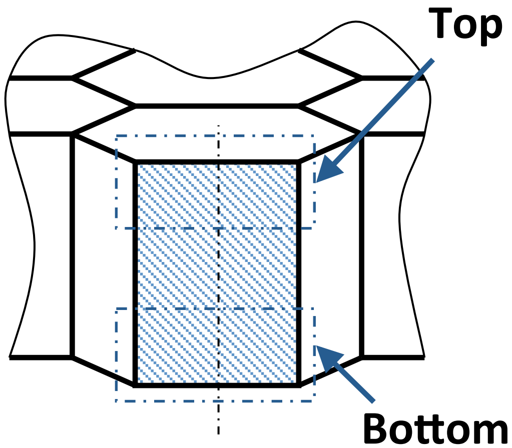


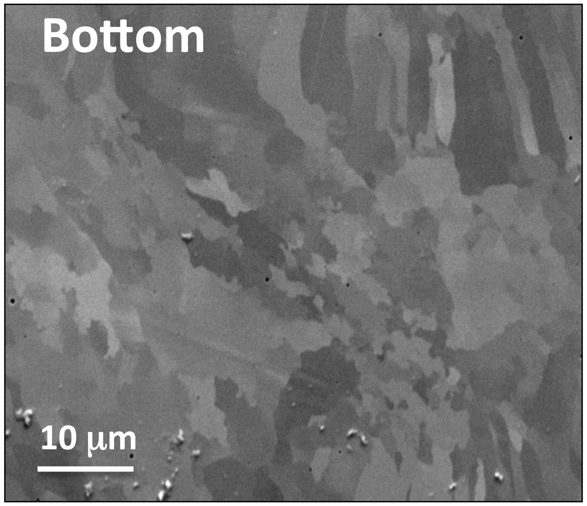


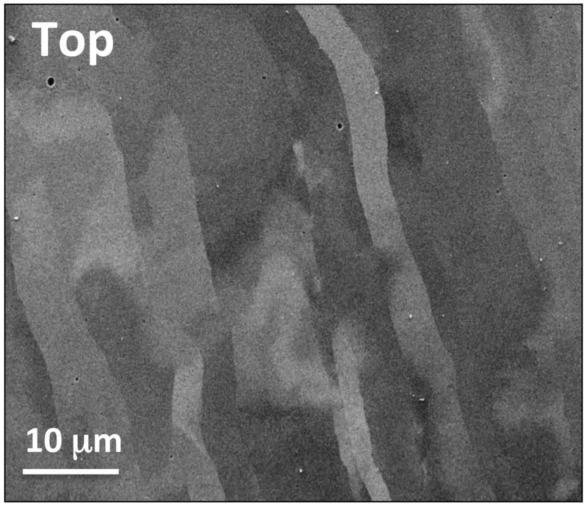
Al+Y+Cr+Ti=100at%

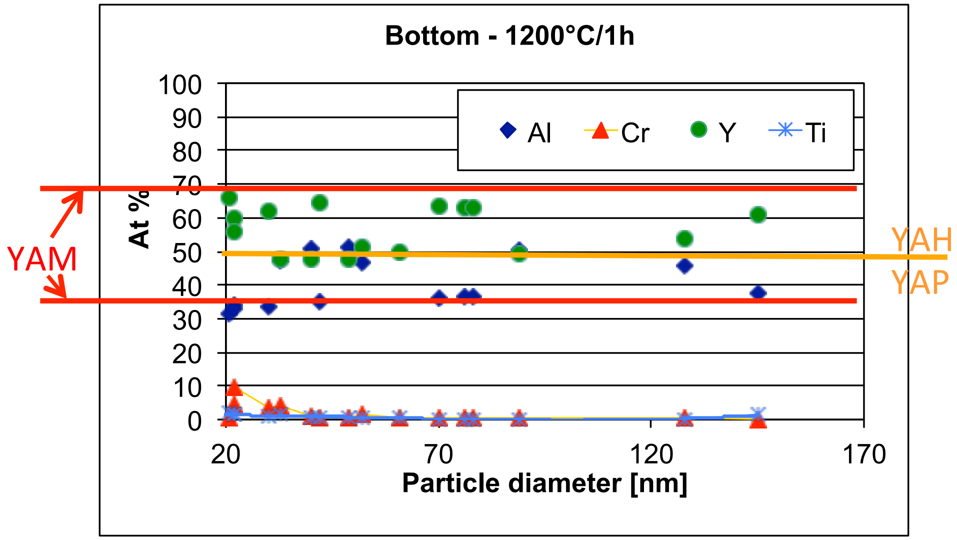
- Different grain sizes
- Higher Cr level for smaller particles = Core shell structure?

SLM of PM2000 alloy powder to form a honeycomb structure









Al+Y+Cr+Ti=100at%

"typical" Y, Al, O precipitates after 1h at 1200°C

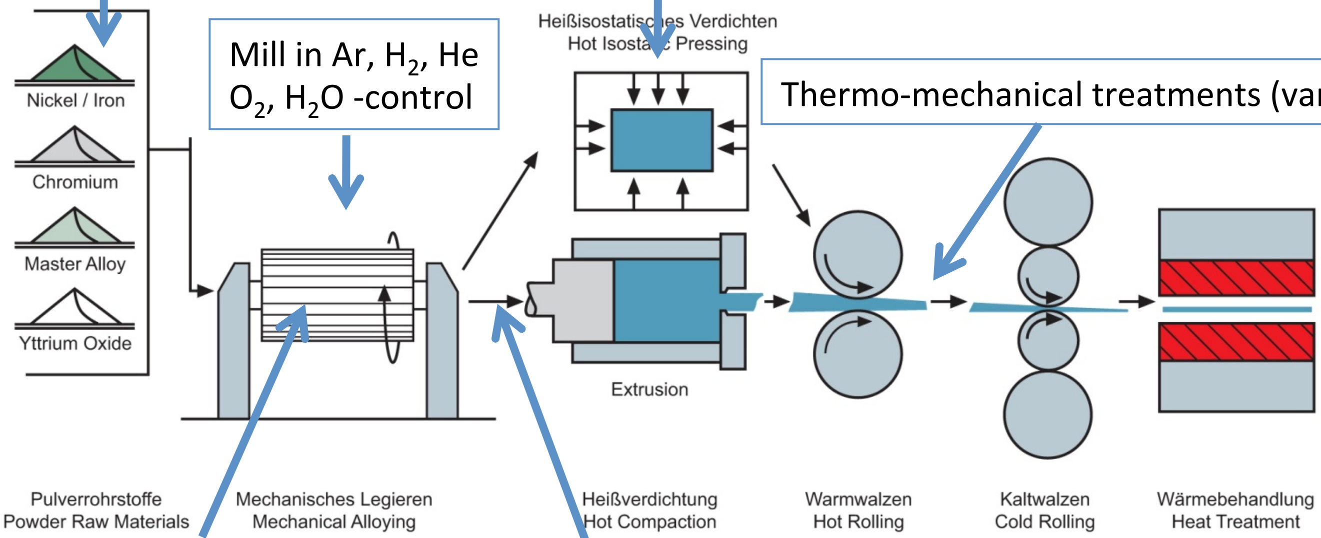
Implications for fossil energy of nuclear related studies on ODS alloys

All powder handling/processing stages (before, during, after MA) can influence alloy microstructure and end properties:

Elemental/Master alloy
Y_2O_3 , YFe_3 , Y
Blend powders pre-MA
Ar atmosphere for powder handling steps
Particle size range - constituent powder

HIP T^0C, time, p
Post-HIP heat treatment
HIP + roll etc
Vacuum hot pressing
High speed hydrostatic extrusion

Reasons for focus on process control for nuclear applications:
- compositional control (RAF)
- impact toughness
- achieve isotropy



Mill in Ar, H_2, He
O_2, H_2O -control

Thermo-mechanical treatments (various, multiple)

Mill type. Milling speed, energy, duration
Contamination from mill materials (e.g. C, Al)
Ball sizes, size mixtures, composition
ball to powder ratio

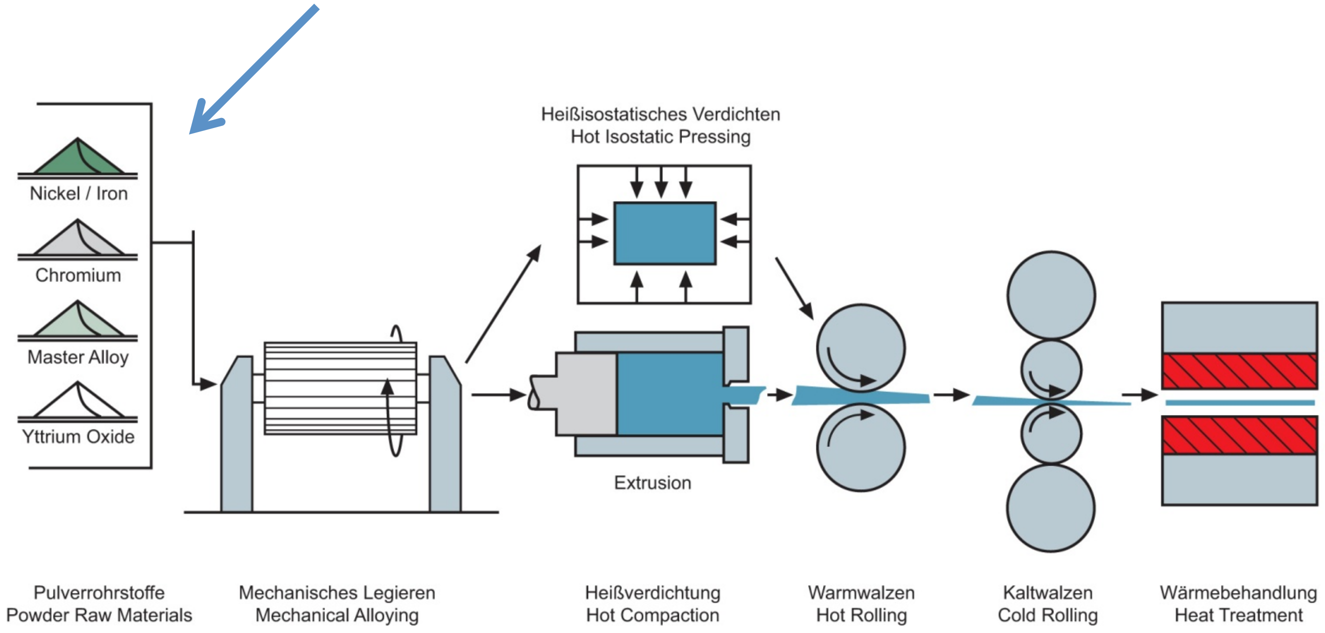
Vacuum degass, T^0C, p, time
Gas shielded handling/storage

Implications for fossil energy of nuclear related studies on ODS alloys

- Use of GA master alloy rather than elemental powder:

  low levels -O (and C, N) as-MA
- Glove box powder handling under dry shield gas (avoid N_2)

  reduced O,N contamination



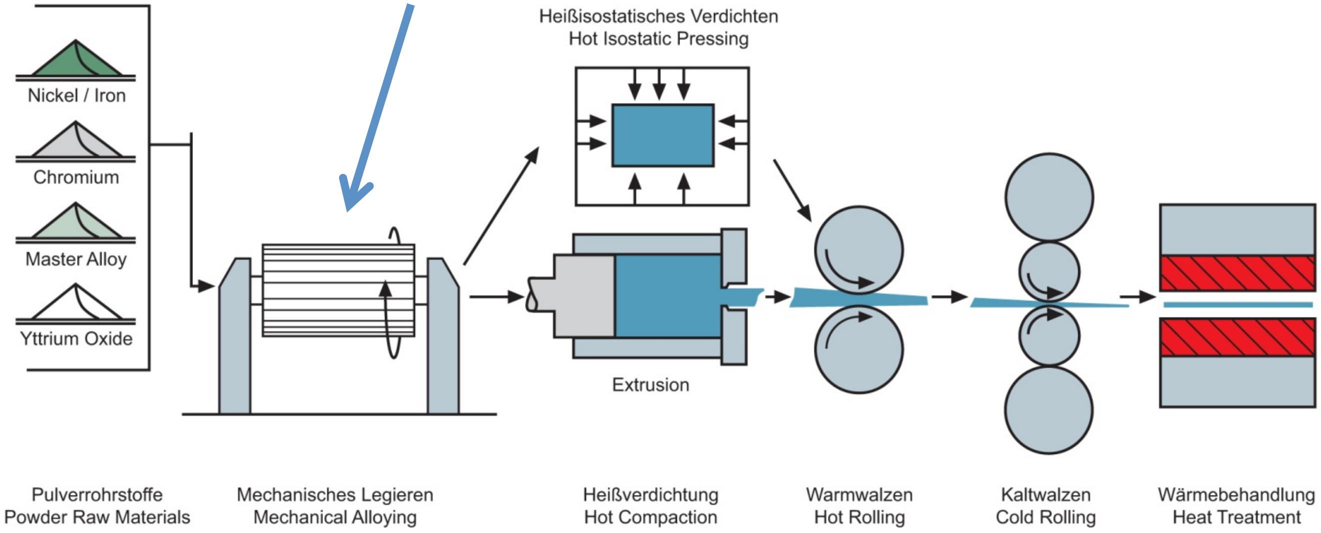
Implications for fossil energy of nuclear related studies on ODS alloys

MA under H_2 rather than Ar shield gas

O,N and micro-porosity reduced

HIP density increased

ductility, and toughness improved

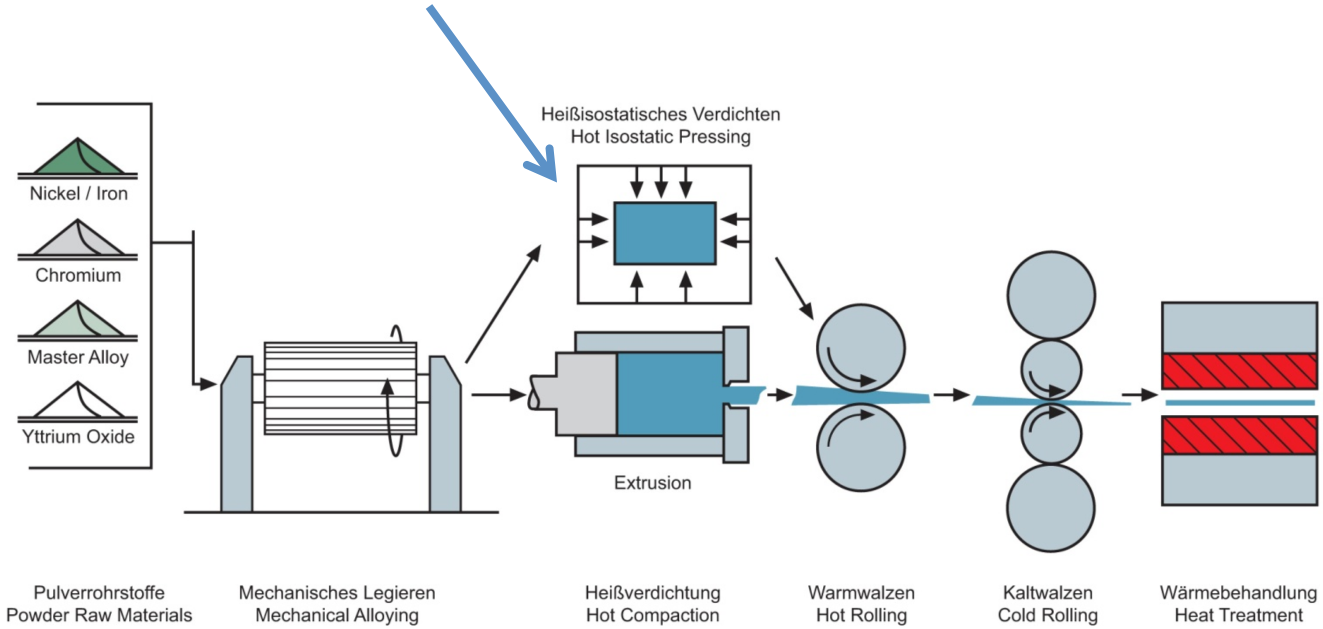


Implications for fossil energy of nuclear related studies on ODS alloys

- HIP consolidation without additional thermo-mechanical treatment

  residual porosity, PPBs

  reduced impact properties, ductility



NE studies on ODS alloys: Potential compositional refinements

Legacy MA ODS FeCrAl alloys

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


PM2000 burner tubes

- Addition of **~2 wt.% W**, introduce via master alloy

  additional solid solution strengthening

- Restrict **Ti ≤ 0.25 wt.%** to reduce incidence of large Ti(C,N)

  affect alloy toughness, integrity during thermo-mechanical processing and subsequent creep strength

- Addition of **~0.5 wt.% Hf (Zr)** potentially to form $Y_2Hf_2O_7$ (pyrochlore) in preference to YAM ($Y_4Al_2O_4$), YAP ($YAlO_3$) or YAG ($Y_3Al_5O_{12}$)

  improved particle Nv, reduced mean size, additional Al in solid solution

- much closer **control of excess-O** during MA processing

  reduce incidence of Al_2O_3 stringers

➡ See review by Andy Jones

Development of models to predict and improve ODS alloys durability

- High temperature creep and oxidation are expected to be the main mode of degradation

- Existence of a stress threshold at a given temperature below which deformation is minimum

- For a mechanically sound component, oxidation will determine the components durability

- Need lifetime models for relevant environments ie containing species such as H_2O and CO_2

Composition and experimental work

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

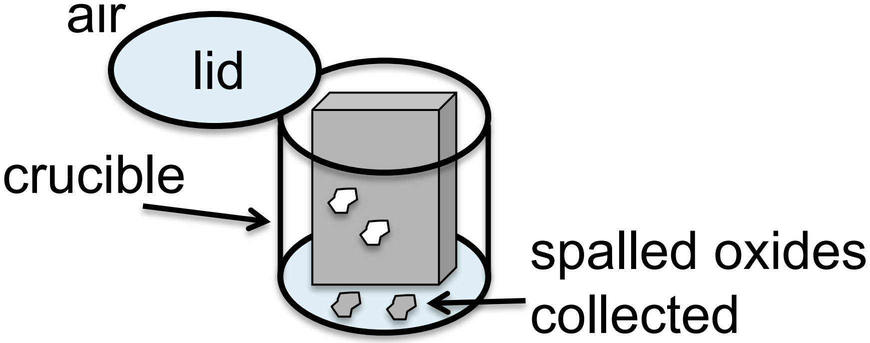
2 different batches of PM2000

Long term testing

100h cycle, 1100ºC

specimen and total mass gain

air

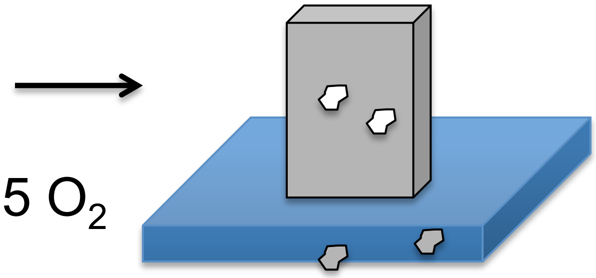


500h cycle, 1100ºC

specimen mass gain

$50\%CO_2/50\%H_2O$

$\sim 50\%CO_2/50\%H_2O + 0.075\ O_2$



Accelerated testing

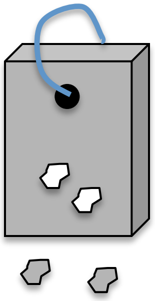
1h cycle, 1200ºC

specimen mass gain + time to breakaway

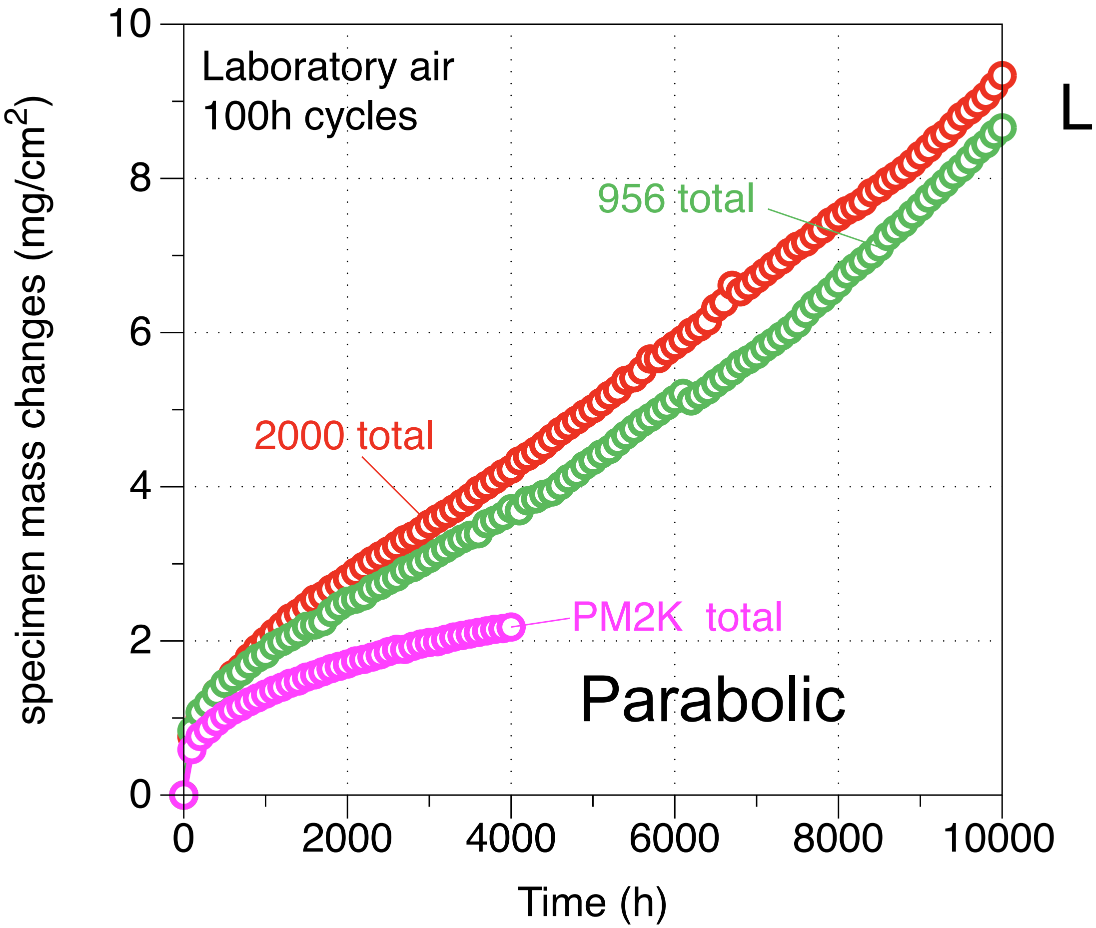
O_2

$50\%CO_2/50\%H_2O$

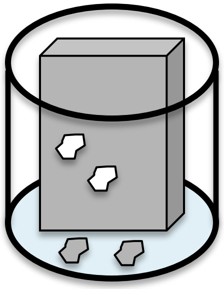
$\sim 50\%CO_2/50\%H_2O + 0.075\ O_2$



100h cycle 1100°C: Similar total oxidation rates for MA956 and PM2000 PM2K significantly better

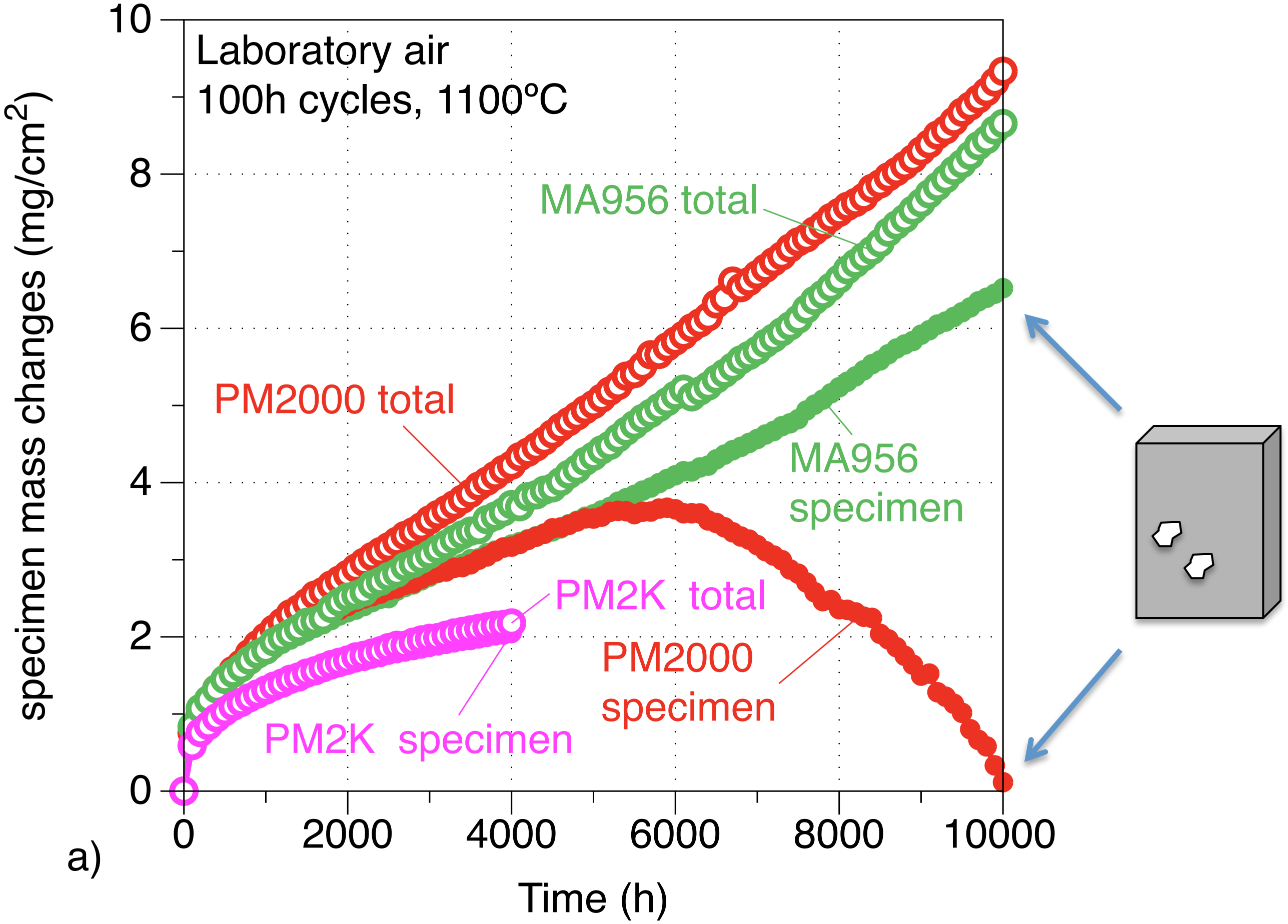


Linear

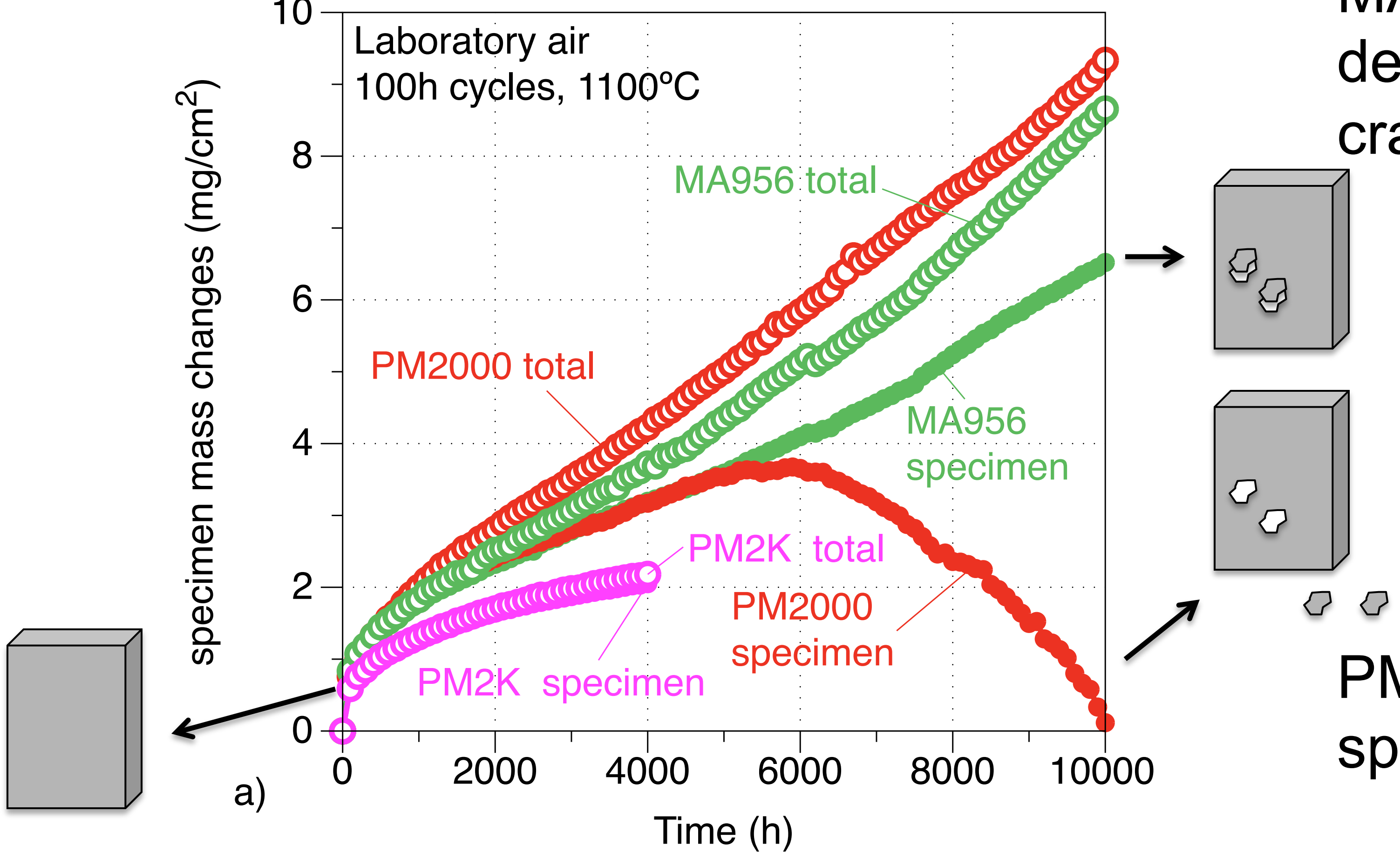


Total

100h cycle 1100°C: mass gain for MA956, mass loss for PM2000 No spallation for PM2K



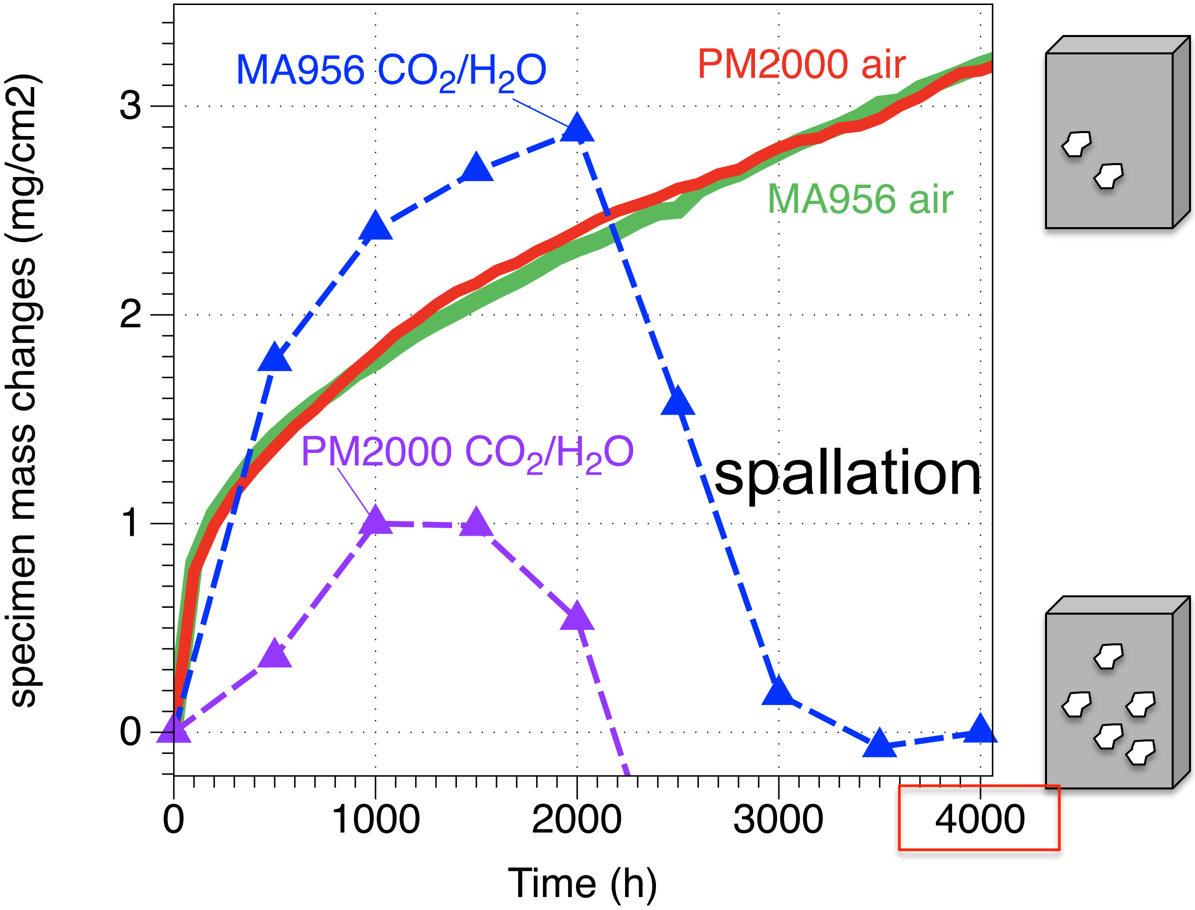
100h cycle 1100°C: mass gain for MA956, mass loss for PM2000 No spallation for PM2K



MA956
debonding
cracking

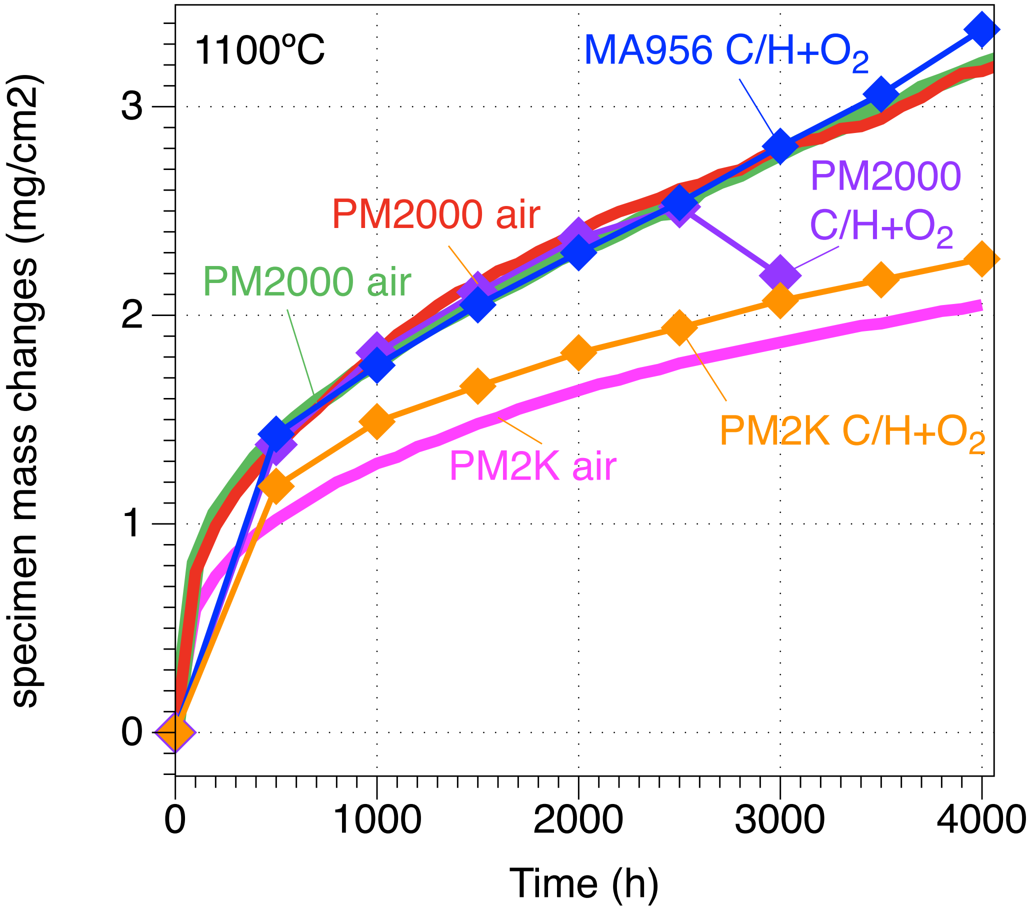
PM2000
spallation

Significant spallation at 1100ºC in 50%CO_2/50%H_2O atmosphere



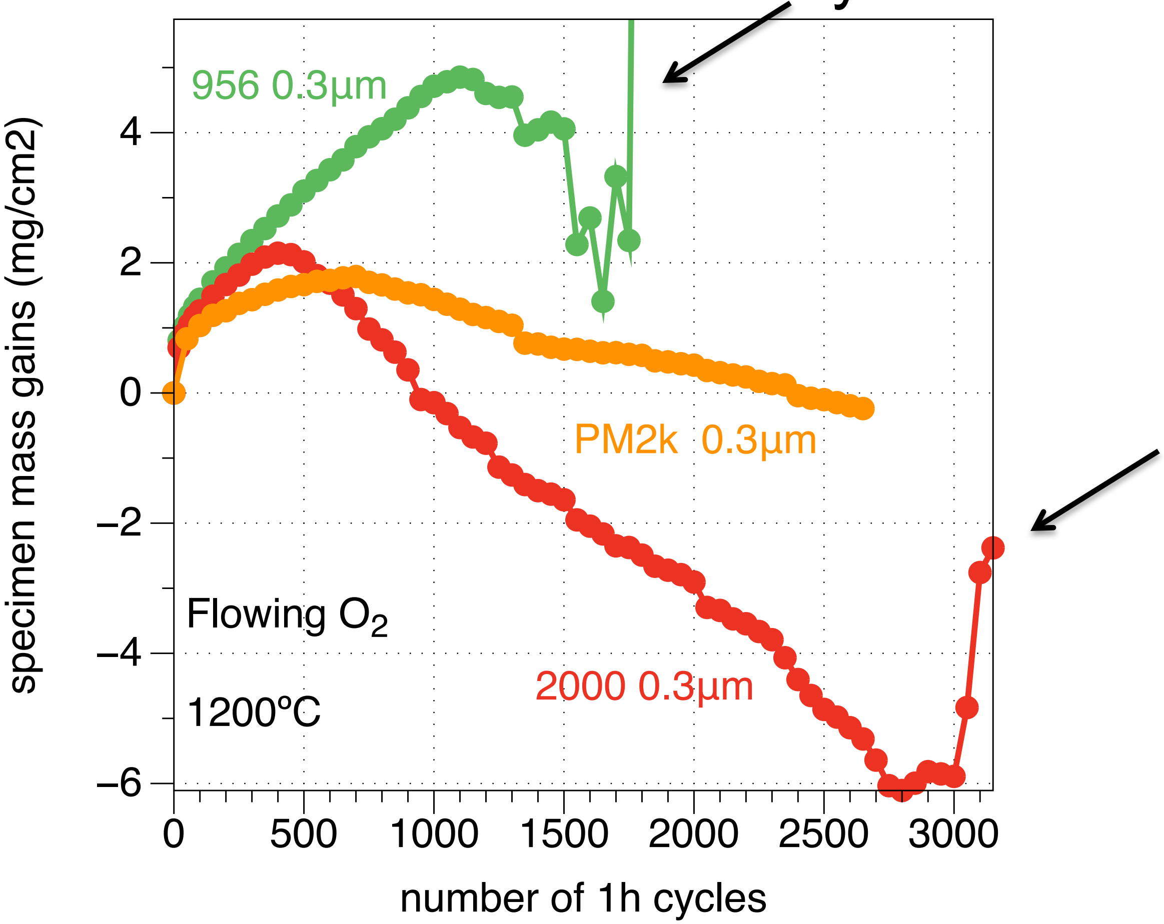
Slight effect at 1100ºC of 50%CO_2/50%H_2O+O_2 only for PM2000

Significant effect of O_2 buffer



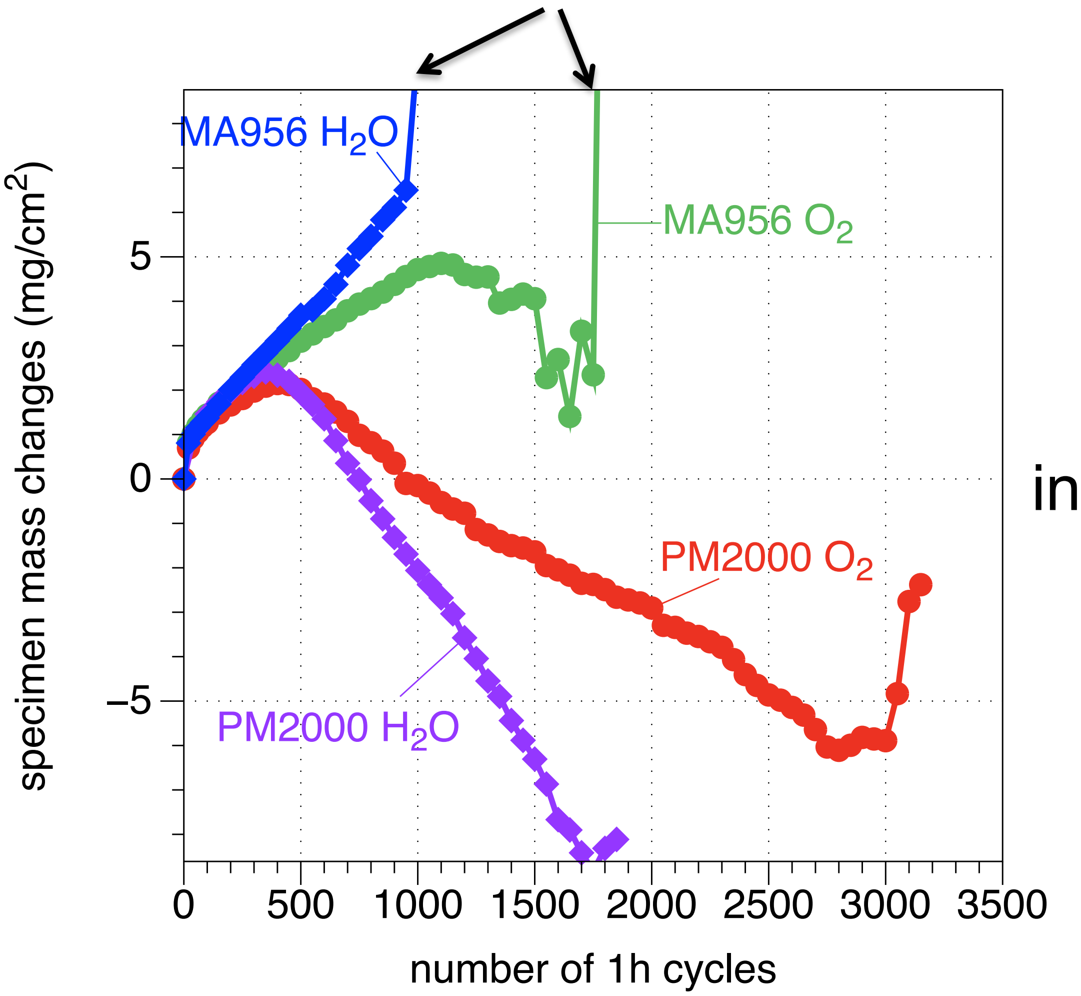
1h cycles 1200°C: lower oxidation rate for PM2K. Pm2K lifetime> X3 PM2000 lifetime> X2 MA956 lifetime

breakaway oxidation = end of life



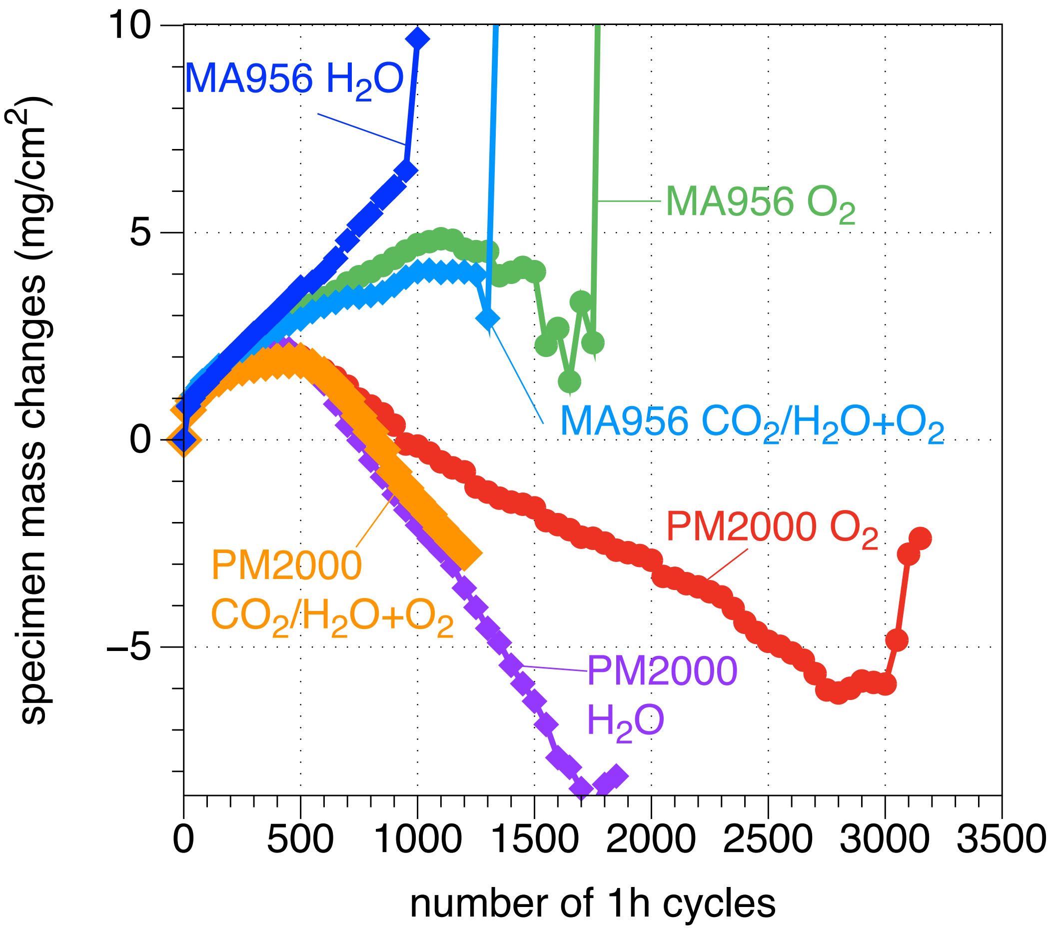
1h cycle 1200°C: Significant effect of air+10%H_2O on oxidation and lifetime

breakaway oxidation = end of life



likely due to
increase spallation
in H_2O

1h cycle 1200°C: lower effect of $50\%CO_2/50\%H_2O+0.075\%O_2$



Breakaway oxidation is due to Al consumption to form Al_2O_3

-$C_{b:}$ critical Al content below which Al_2O_3 cannot form

-C_o initial Al concentration

-FeCrAl models : lifetime = time to drop from C_o to C_b

Al to form Al_2O_3 = Al consumed in the alloy

Al_2O_3

$$S \cdot 1.125 \cdot k \cdot t^n$$

k, n: oxidation parameters

S: surface

FeCrAl

$$V \cdot \Delta C$$

$\Delta C = C_b - C_0$

V: volume

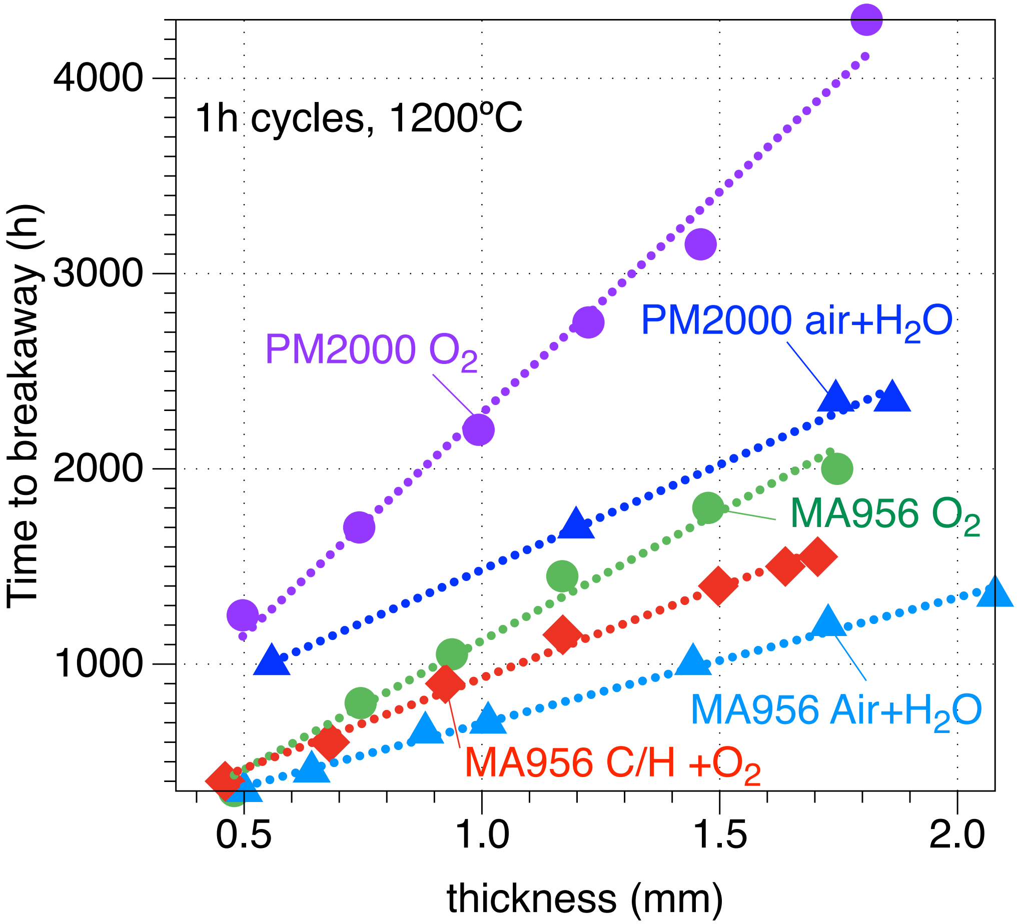
Quaddakers et al. (1994)

$$\boxed{1.125 \cdot k \cdot t_b^n = \frac{\Delta C}{100} \cdot \rho \cdot \frac{d}{2}}$$

d thickness

ρ density

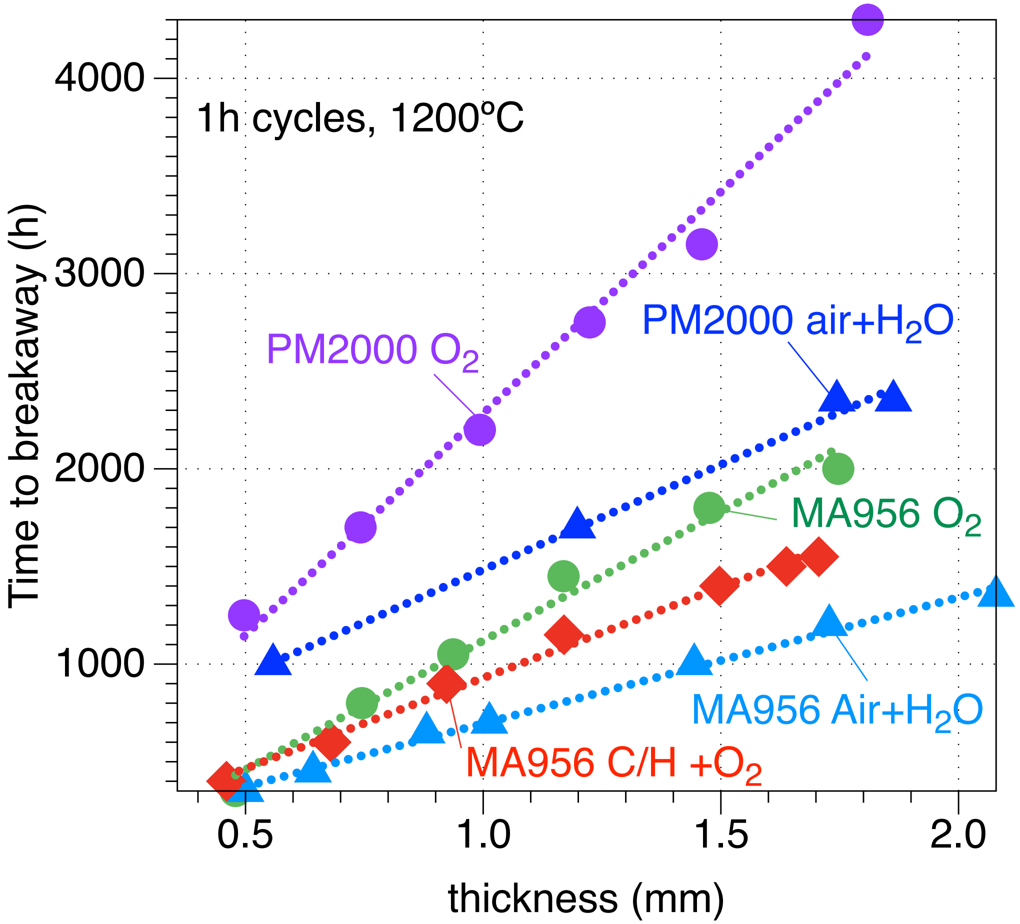
Decrease of lifetime: high in air+H_2O lower in 50%CO_2/50%H_2O+O_2 Linear trend in all environments



$$1.125 \cdot k \cdot t_b^n = \frac{\Delta C}{100} \cdot \rho \cdot \frac{d}{2}$$

n = 1

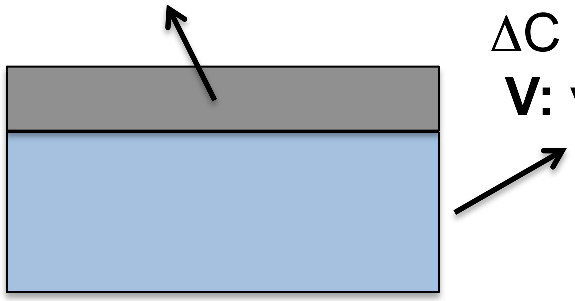
Extrapolation for large components? How to obtain failure at lower temperature and/or long cyclic testing?



New method to estimate component lifetime = Al profile in interrupted tests

k, n: oxidation parameters

S: surface



Al_2O_3

$$S\cdot 1.125\cdot k\cdot t^{n}$$

FeCrAl

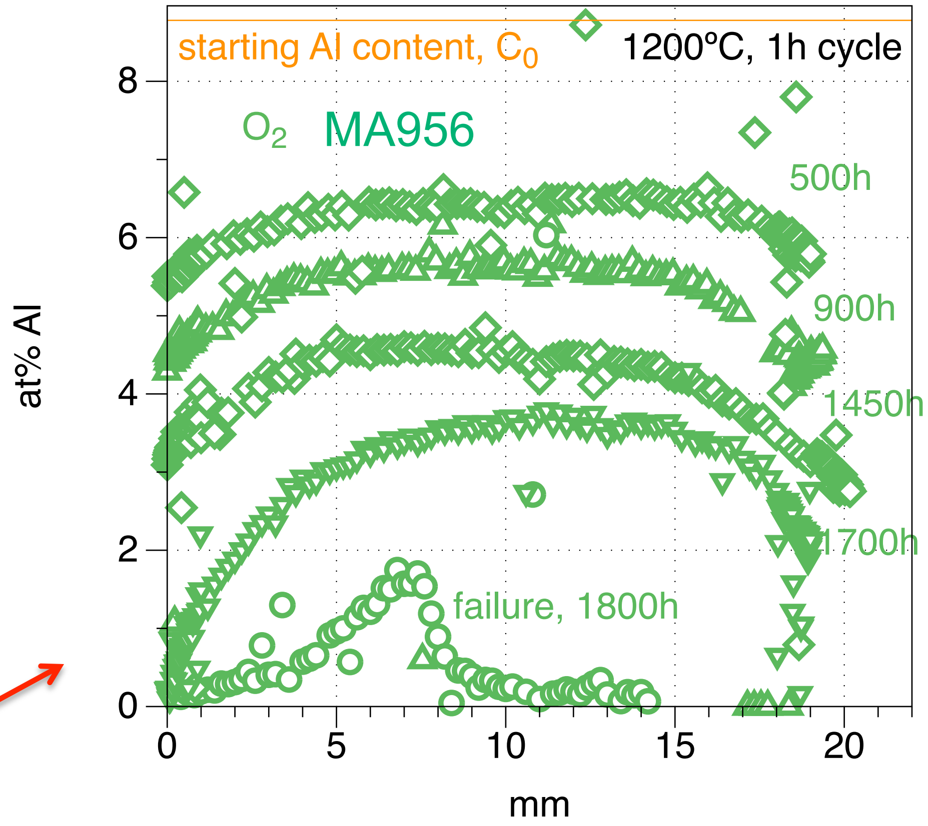
$$V\cdot \Delta C$$

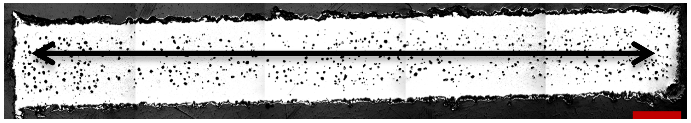
$\Delta C = C_b - C_0$

V: volume

at t = 500h

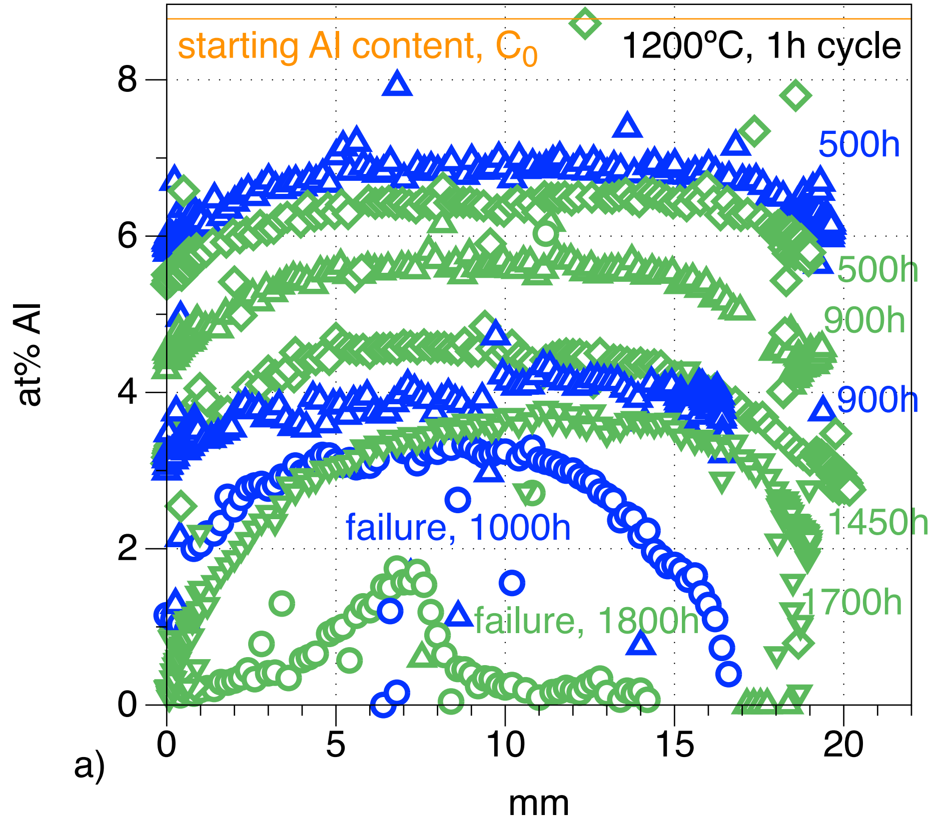
$$1.125\cdot k\cdot t_{500}^{n} = \frac{C_0 - \boxed{\int C_{500}}}{100}\cdot V$$

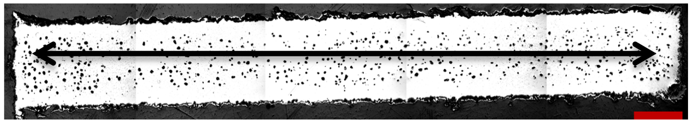




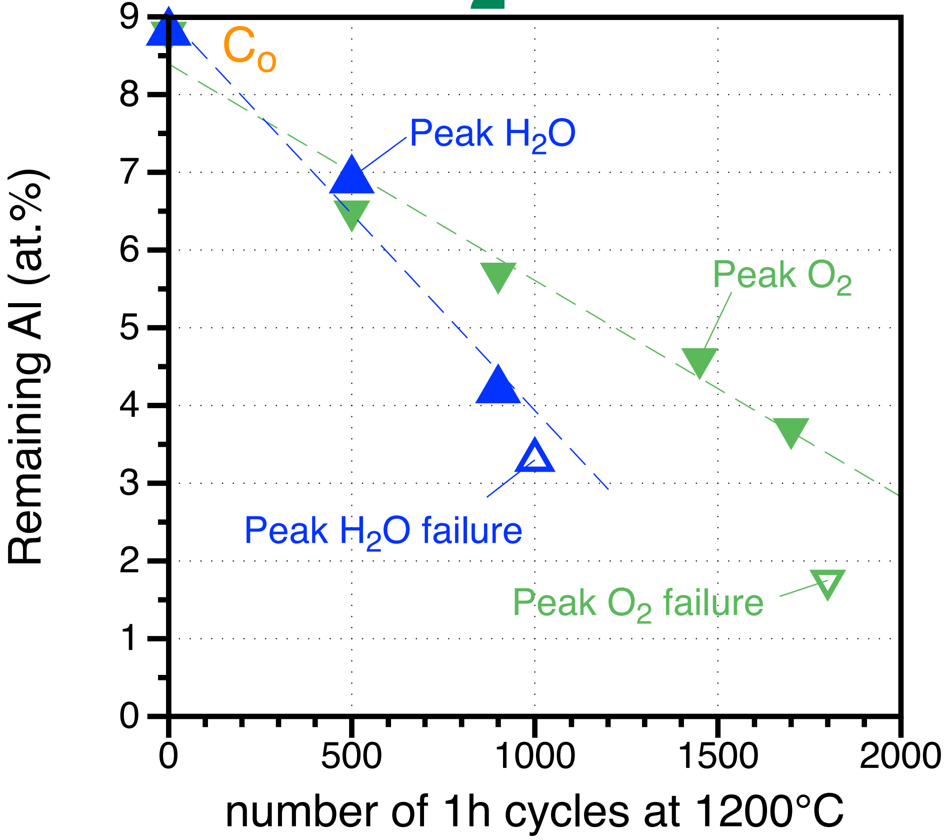
Al

Linear decrease of the Al content with number of cycles in O2 and H_2O Faster decrease in H_2O

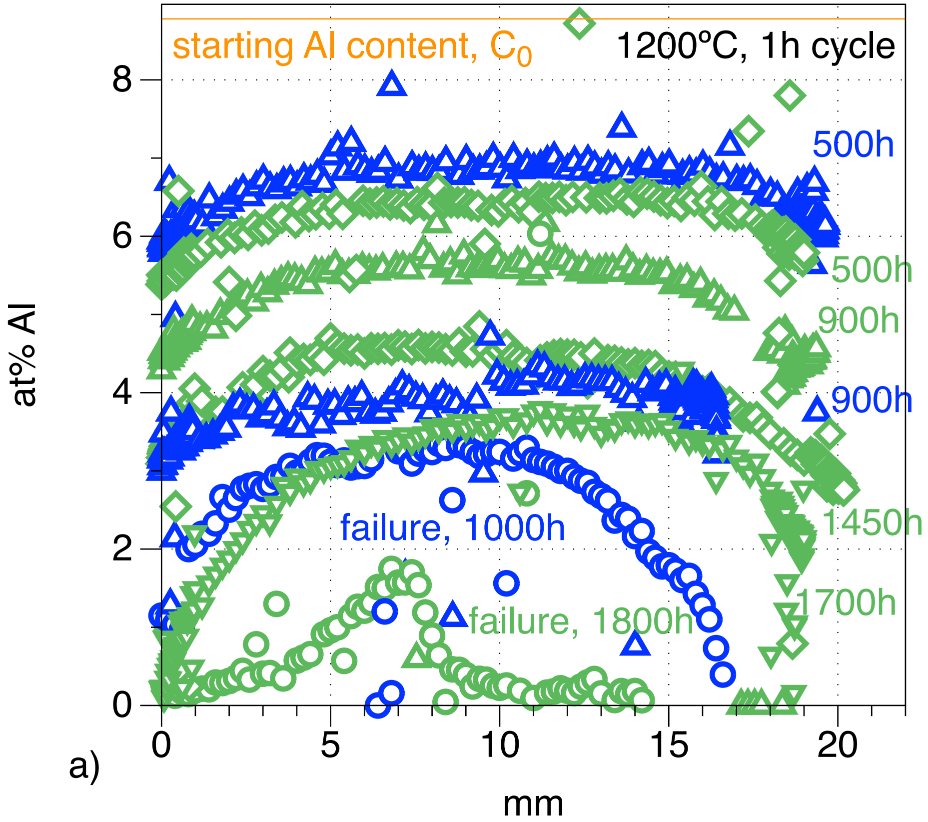


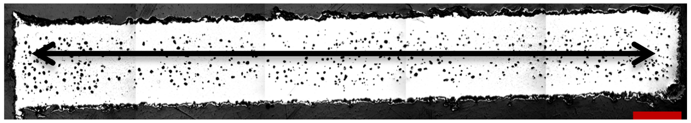


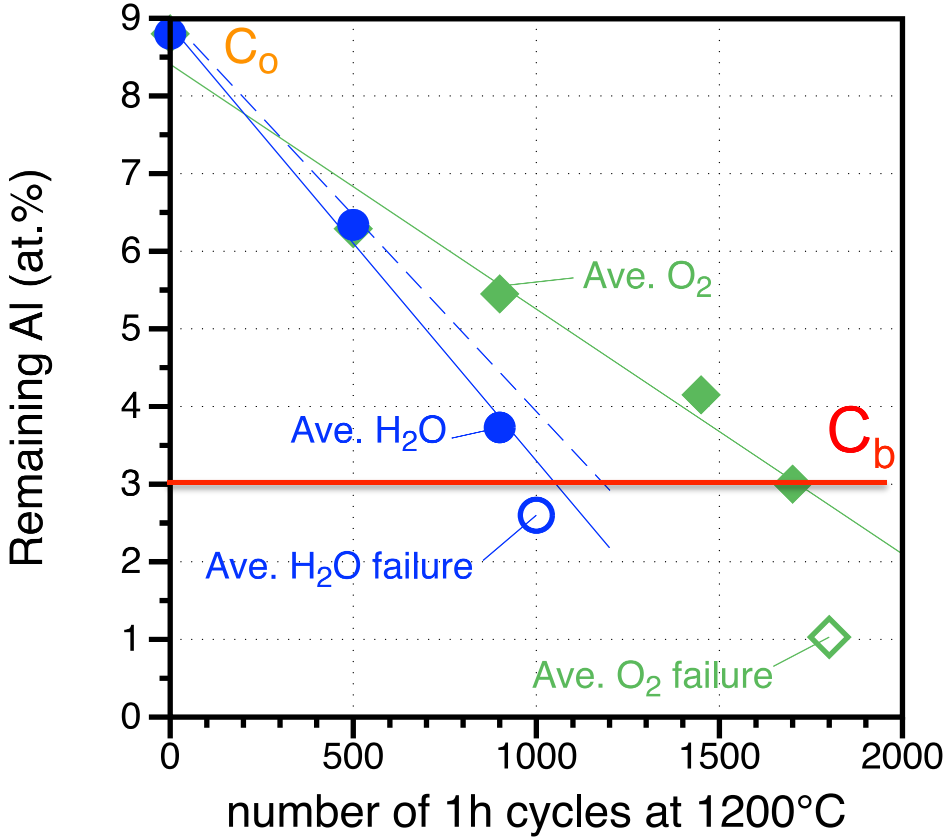
Al



C_b difficult to obtain from profile after failure. C_b~3% for MA956 Method will be used at lower T°C

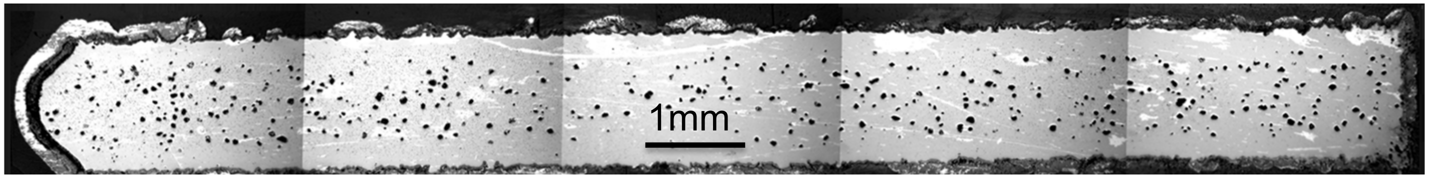




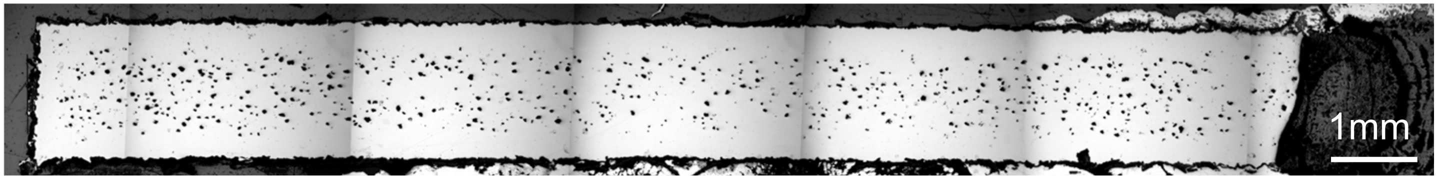


Porosity formation at 1200°C higher in CO_2/H_2O for MA956

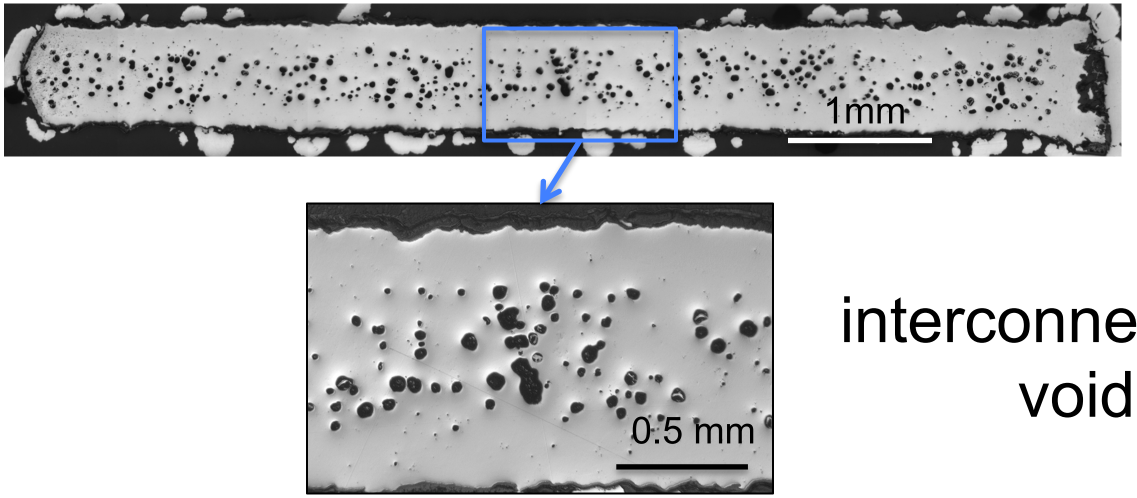
porosity fraction: O_2~1 to 4%



porosity fraction: air+10%H_2O, ~1 to 5%



porosity fraction: 50%CO_2/50%H_2O ~ 7 to 11%

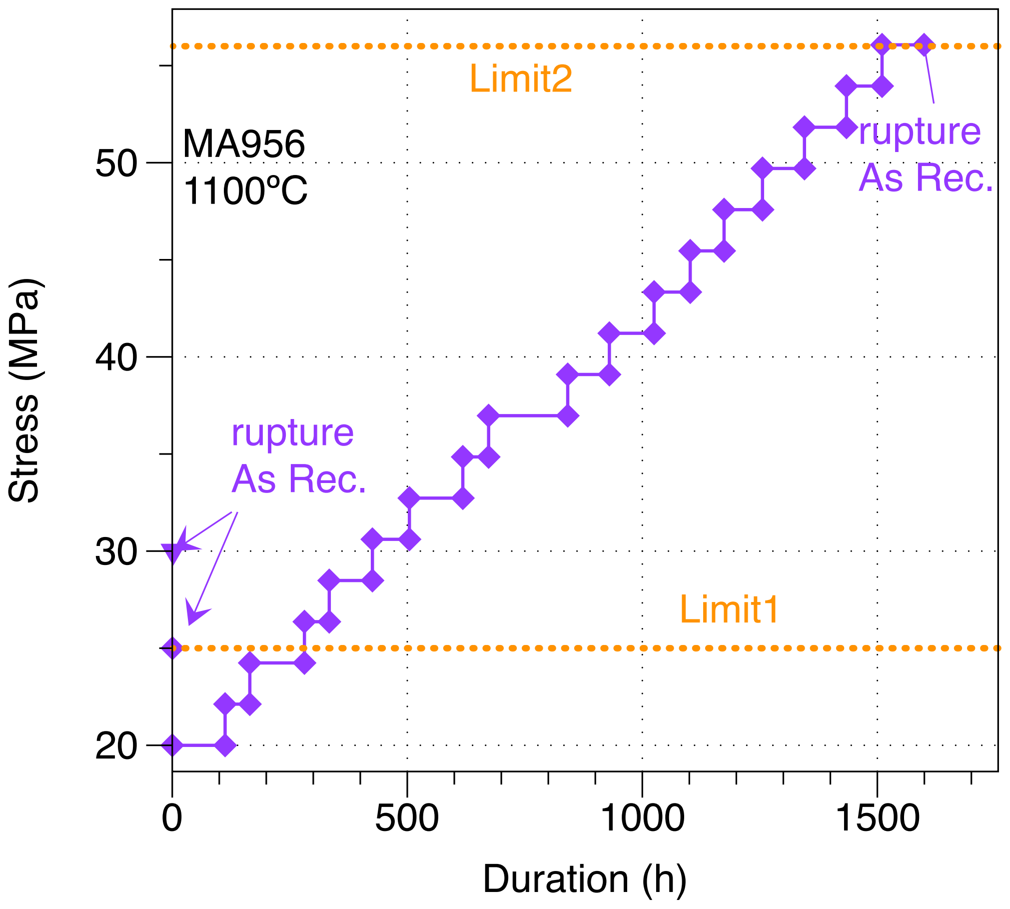


interconnection of voids

Two different creep thresholds for alloy MA956 at 1100ºC?

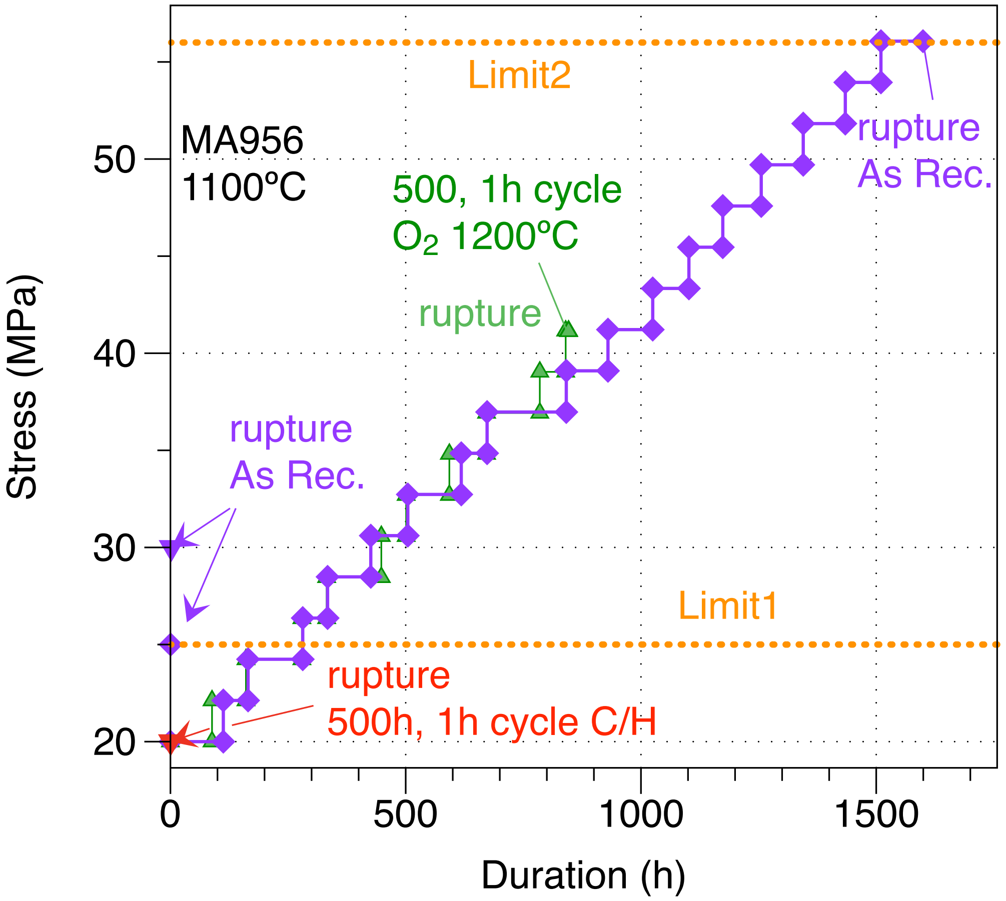
-Incremental creep testing (~2 MPa every 3 days)

- Existence of 2 different thresholds?

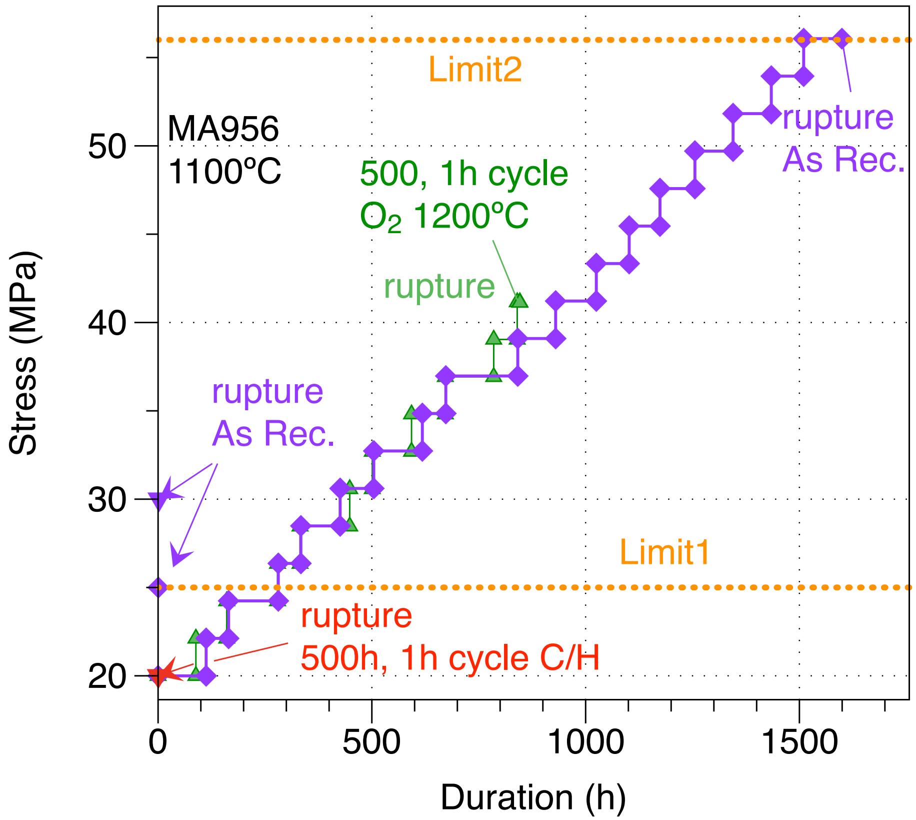


Specimens pre exposed in O_2 and H_2O/CO_2 exhibit shorter lifetimes

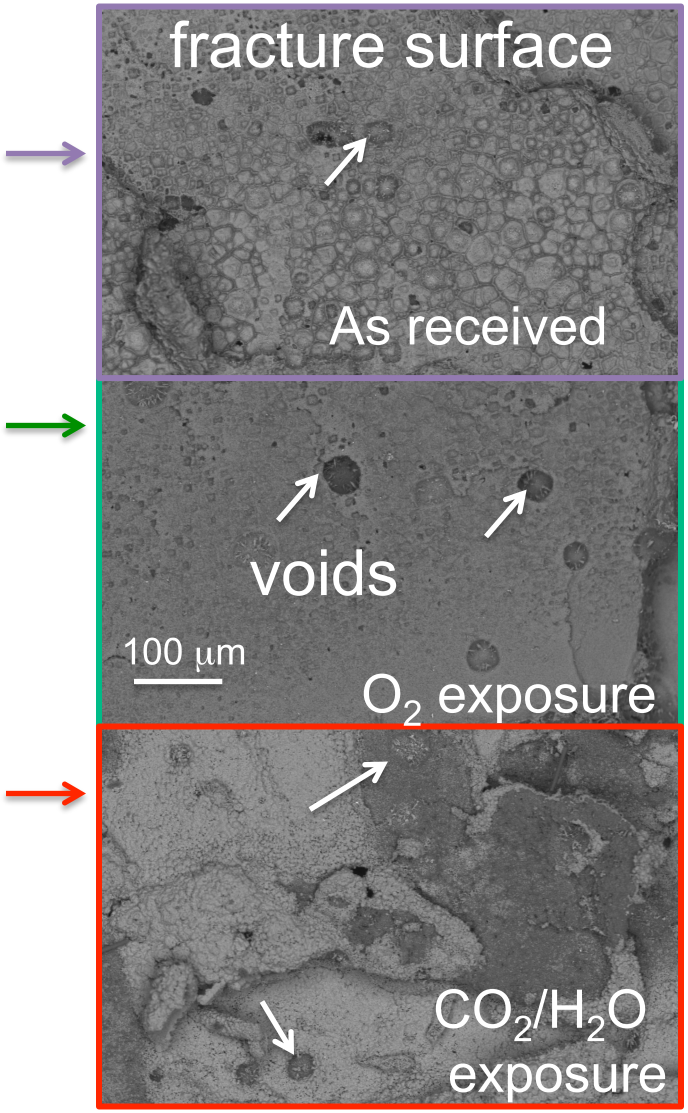
Specimen pre-exposed for 500, 1h cycles in O_2 and CO_2/H_2O at 1200ºC



Voids responsible for earlier ruptures after pre exposure in O_2 and CO_2/H_2O



Result dispersion due to material inhomogeneity?



Conclusions

- ODM751 alloy shows good room temperature properties but high level of impurities

- “New” PM2000 exhibits excellent oxidation performance

- Optimization of fabrication process and composition could result in outstanding high temperature properties

- Various paths for the next generations of ODS components: ODM751, GARS powder, additive manufacturing, new NE ODS alloys

- Work still going on to improve the understanding of ODS alloys oxidation in complex environments (H_2O, CO_2…) and improve lifetime models

Acknowledgements

- G. Garner, T. Lowe, M. Howell, M. Stephens, L. Hu, J. Moser, L. Walker and D Leonard for assistance with the experimental work
- B. Pint, P. Tortorelli and I. Wright for exciting scientific discussions
- This research was sponsored by the U.S. Department of Energy, Office of Fossil Energy under the supervision of Vito Cedro III