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PURE AND FINE YTTRIA-DOPED α-ALUMINA SAMPLE ELABORATION AND DIFFUSION STUDIES

OBJECTIVES

Yttrium is a key-element to improve the properties of alumina in various fields (aeronautic, energy, automotive.......). To ensure reproducible properties it is necessary to use materials with an homogeneous microstructure.

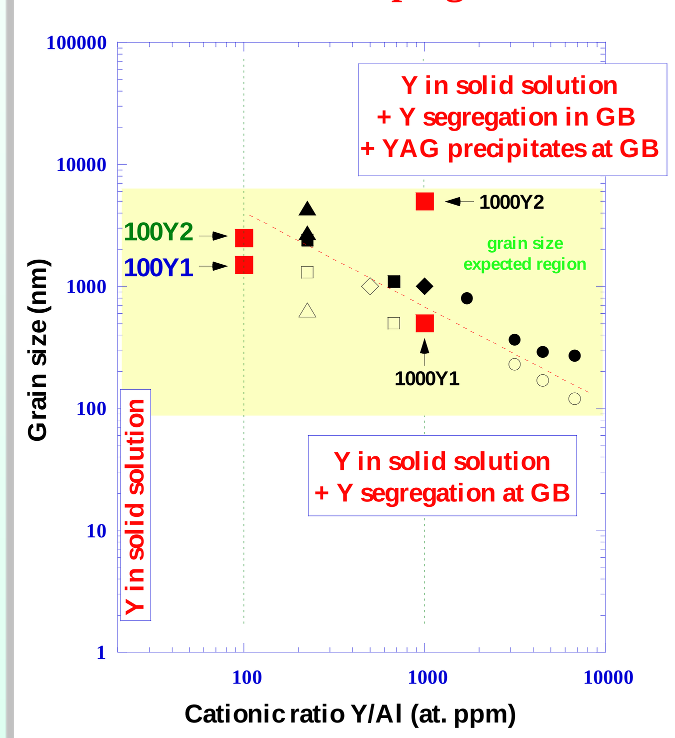
Yttrium usually has a very low solubility in the alumina lattice (~ 10ppm). So it can exist in three forms : first, a lattice solid solution is formed ; beyond saturation of the lattice, the grain boundaries (GB) are enriched in Y ; finally, after saturation of both lattice solid solution and GB sites, a second phase precipitates inside the grain boundaries ($Y_3Al_5O_{12}$, YAG).

Earlier studies on ionic transport properties in Y-doped single crystals and poorly-defined polycrystalline α-alumina have indicated that yttrium may increase the oxygen mobility in the bulk but decreases it in the grain boundaries.

The present study concerns the ^{18}O transport properties of well-defined Y-doped α-alumina, which will provide important informations relevant to understand different processes (sintering, mechanical creep of α-alumina, growth kinetics of alumina scales on aluminium containing alloys at high temperatures).

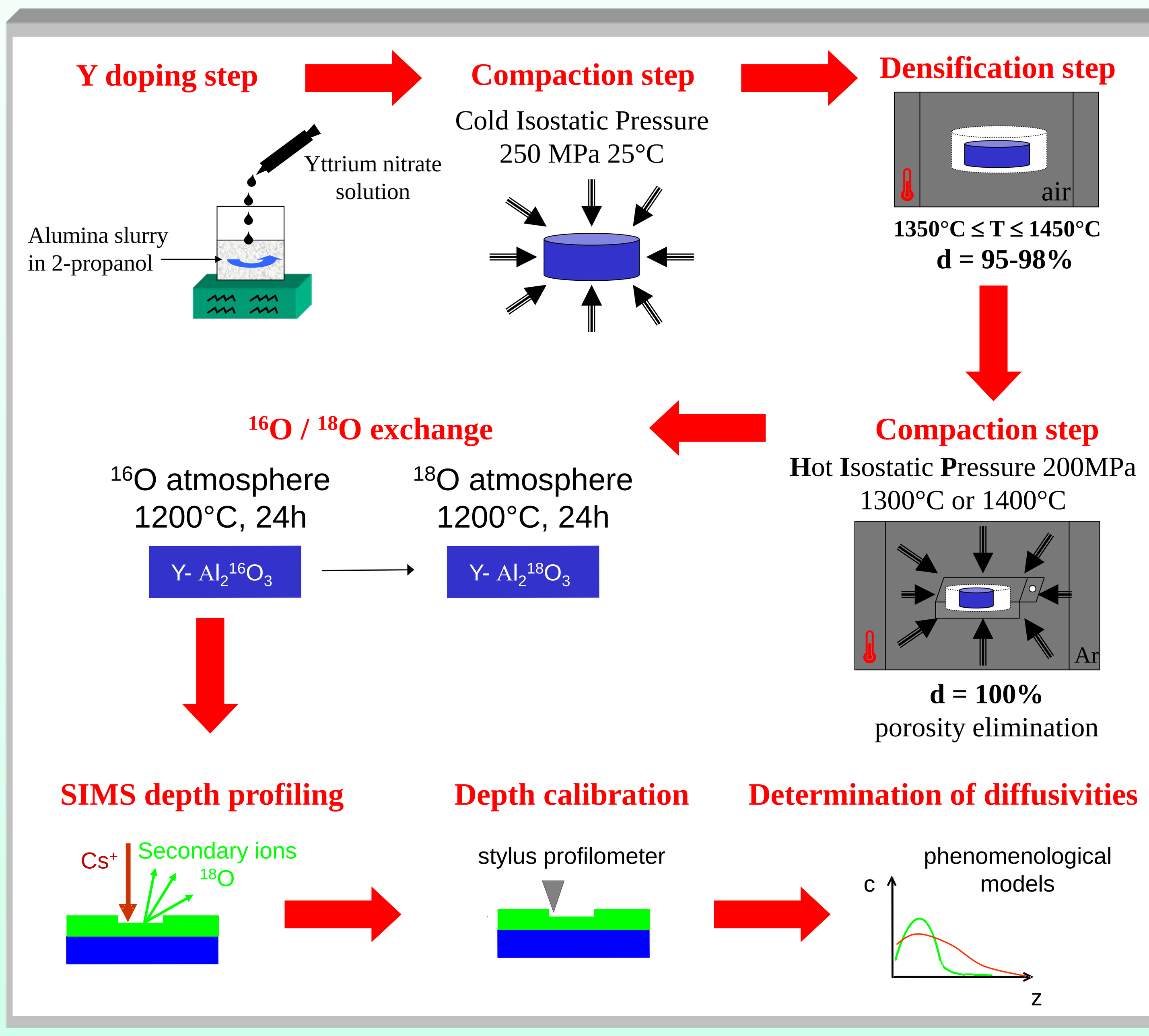
EXPERIMENTAL

Choice of doping level



Characterization methods

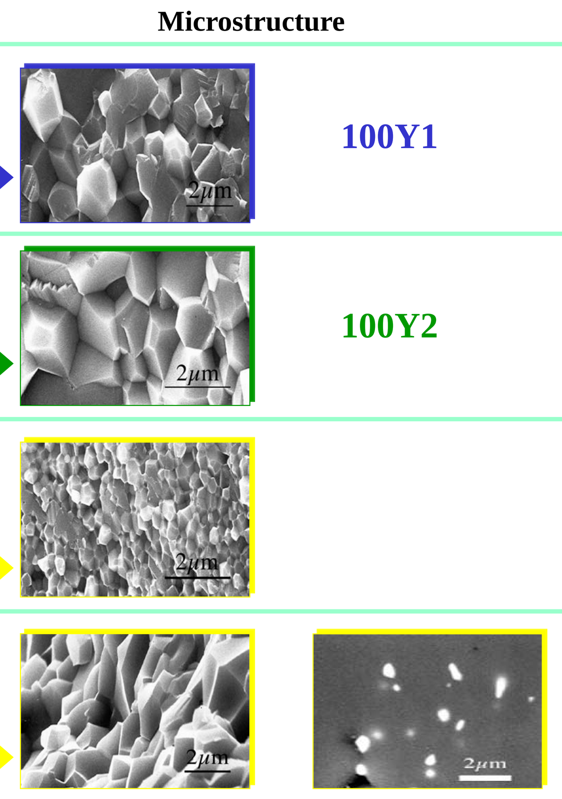
- PIXE semi-quantitative analysis
- Archimedes' method for density
- Scanning electron microscopy (SEM)



RESULTS

SAMPLES

Sample	Pressing	Sintering	Y analysis (ppm)	Grain size (µm)	Density (% d_{th})	Microstructure
100Y1	CIP	1350°C + 1h	-	< 1	98	
	HIP	1300°C + 2h	82	1.5	100	100Y1
100Y2	CIP	1400°C + 2h	-	2	98	
	HIP	1400°C + 2h	76	2.5	100	100Y2
1000Y1	CIP	1375°C + 1h	-	< 1	96	
	HIP	1300°C + 2h	910	0.5	100	
1000Y2	CIP	1450°C + 2h	-	3-4	96	
	HIP	1400°C + 2h	1000	5	100	

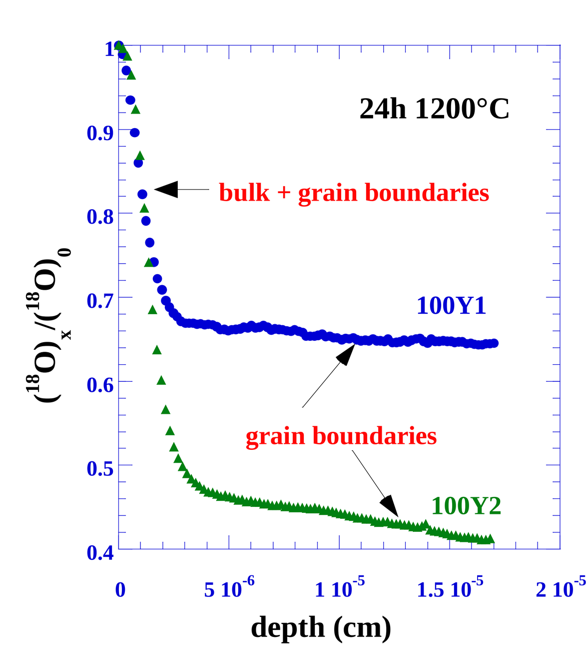


Backscattered electron image on a polished surface

- Elaboration of full dense samples.
- Samples have approximately the amount of yttrium strived for. The scale factor between 100Y and 1000Y doping levels are conserved.
- Microstructures and grain sizes correspond well to the expected data from grain size vs (Y/Al) graph. For 1000Y2, the backscattered electron image of a polished surface shows clearly precipitates of $Y_3Al_5O_{12}$ (YAG) probably at GB.

DIFFUSION STUDIES

♦ Normalised ^{18}O penetration profile



Two parts

① a strong decrease of ^{18}O concentration interpreted as bulk diffusion

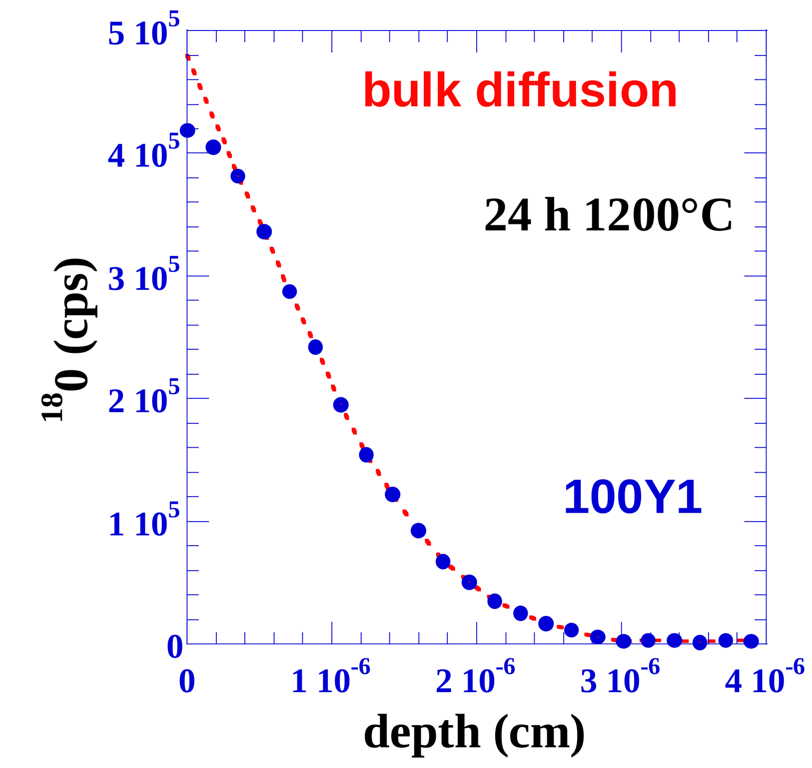
Bulk diffusion is quite the same in 100Y1 and 100Y2 because the bulk is in both cases saturated with yttrium

② a long range diffusion related to diffusion in grain boundaries

GB diffusion is faster in 100Y1 than in 100Y2 because the density of Y atoms segregated in grain boundaries is smaller in the fine-grained 100Y1

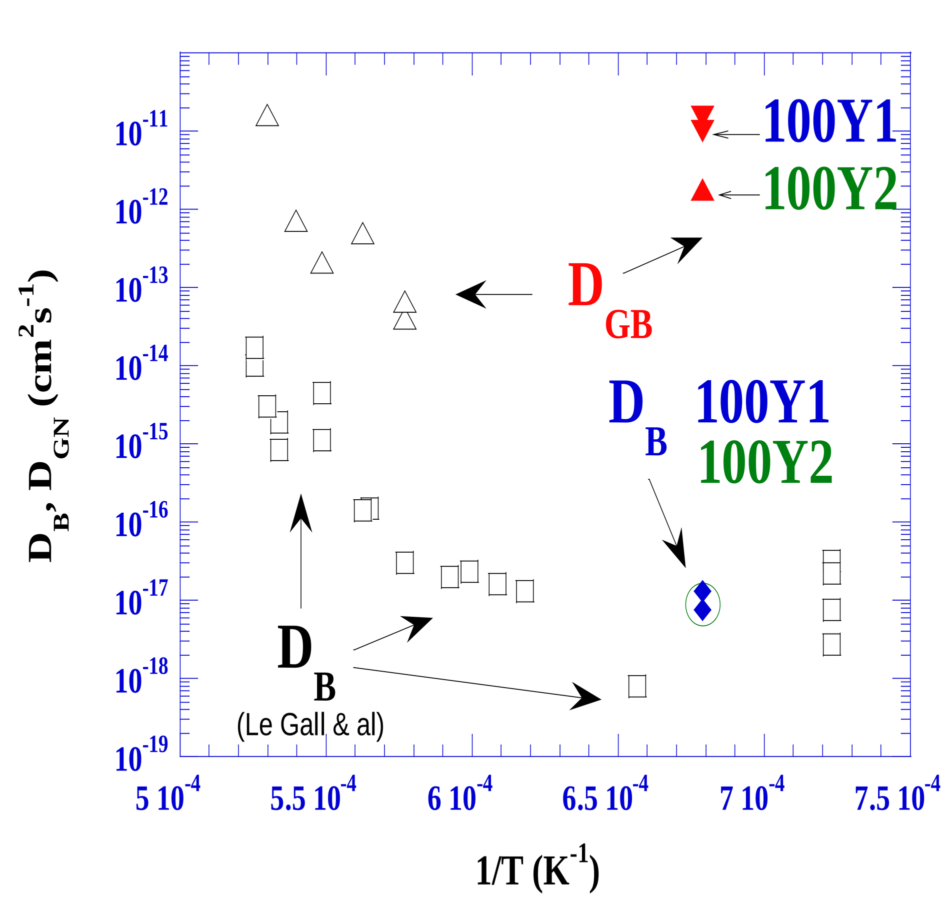
♦ ^{18}O penetration profile for bulk diffusion

Corrected profile obtained by substraction of the grain boundary diffusion



100Y1 ⇨	$D_B = 7.5\ 10^{-18}\ cm^2s^{-1}$
100Y2 ⇨	$D_B = 8.8\ 10^{-18}\ cm^2s^{-1}$

♦ ^{18}O diffusion in Y-doped α-alumina



D_{GB} was calculated using the Wipple-Le Claire equation

$$D_{GB} * \delta = 1.322 \sqrt{\frac{D_B}{t}} \left(\frac{-d \ln C}{dx^{6/5}} \right)^{-5/3}$$

100Y1 ⇨	$D_{GB} \approx 10^{-11}\ cm^2s^{-1}$
100Y2 ⇨	$D_{GB} \approx 2.10^{-12}\ cm^2s^{-1}$

- D_{GB} decreases with grain size due to a lower segregant atom density in smaller grains
- For bulk diffusion, results are in agreement with earlier results
- For GB diffusion, D_{GB} values are much greater than those deduced from the extrapolation of D_{GB} values at higher temperatures

CONCLUSION

- α-alumina was doped with yttrium (100 and 1000 ppm cationic Y/Al) from a slurry of high purity α-alumina powder and an aqueous yttrium nitrate solution.
- ^{18}O diffusion tests were performed on 100 ppm Y/Al full dense and homogeneous α-alumina polycrystals with two different grain sizes.
- Oxygen diffusion coefficients in the bulk are independent of the grain size because the solubility in bulk α-alumina doesn't change (~ 10 ppm). These results are in good agreement with observations of Le Gall & al. on Y doped α-alumina single crystals.
  (*M. Le Gall, A.M. Huntz, B. Lesage, C. Monty, J. Bernardini, J. Mater. Sci. 30, 201 (1995)*)
- Oxygen diffusion coefficients in GB depends on the Y concentration in GB :
  ⇨for large grain sizes, the oxygen mobility is slow because the yttrium saturation level in GB is reached and induces $Y_3Al_5O_{12}$ (YAG) precipitation.
  ⇨for small grains sizes, the oxygen diffusion is enhanced, because of the low density of Y in GB (below the saturation limit) increasing the number of free defects.