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M. Filal, D. Monceau, G. Petot-Ervas, C. Petot, J. Zao. Processing of submicron α -alumina powders doped with Mg-physical and chemical characterization. *Journal de Physique IV Proceedings*, 1993, 03 (C7), pp.C7-1459-C7-1462. 10.1051/jp4:19937227 . jpa-00251863

HAL Id: jpa-00251863

<https://hal.science/jpa-00251863>

Submitted on 4 Feb 2008

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Processing of submicron α -alumina powders doped with Mg-physical and chemical characterization

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ABSTRACT

This work examines the influence of the thermal treatment conditions at $T > 1150^\circ\text{C}$, on the microstructure and microchemistry of submicron α -alumina powders doped with 500 and 1650 ppm by weight of MgO. The powders have been characterized by XPS, TEM, SEM, BET surface area measurements and chemical analysis. Quantitative XPS analysis show a strong segregation of Mg at the near surface of the grains. The determining factor is the size of the grains and it has been possible to relate the amount of Mg at the periphery of the grains to the BET surface area measurements.

I-INTRODUCTION

The Mg-doped α -alumina powders studied in the present work have been prepared from the alum. This paper details the effect of the last thermal treatment at $T > 1150^\circ\text{C}$ on the physical and chemical characteristics of the material. The powders have been characterized by BET surface area measurements, XPS, SEM, MET and chemical analysis.

II-MATERIALS

In the alum route the dopants are added before the decomposition of the hydroxides which occurs at approximately 300°C . This gives a uniform distribution of the dopants in the amorphous alumina powder. The next calcination occurs at temperature higher than 1150°C . It allows to obtain the α -alumina submicron powder^(1,2). This thermal treatment has been performed in large silica crucibles (20cm diameter and 15 cm high) or in smaller alumina crucibles (3 cm diameter and 10 cm high). Pt / Pt-10%Rh thermocouples are placed in the powder to monitor its thermal history at different positions^(3,4). As an example, Fig.1 shows variation in the calcining schedule for the powder in the silica crucible. Powders from position 6 and 7 have the largest difference in thermal treatment conditions and have been characterized by XPS.

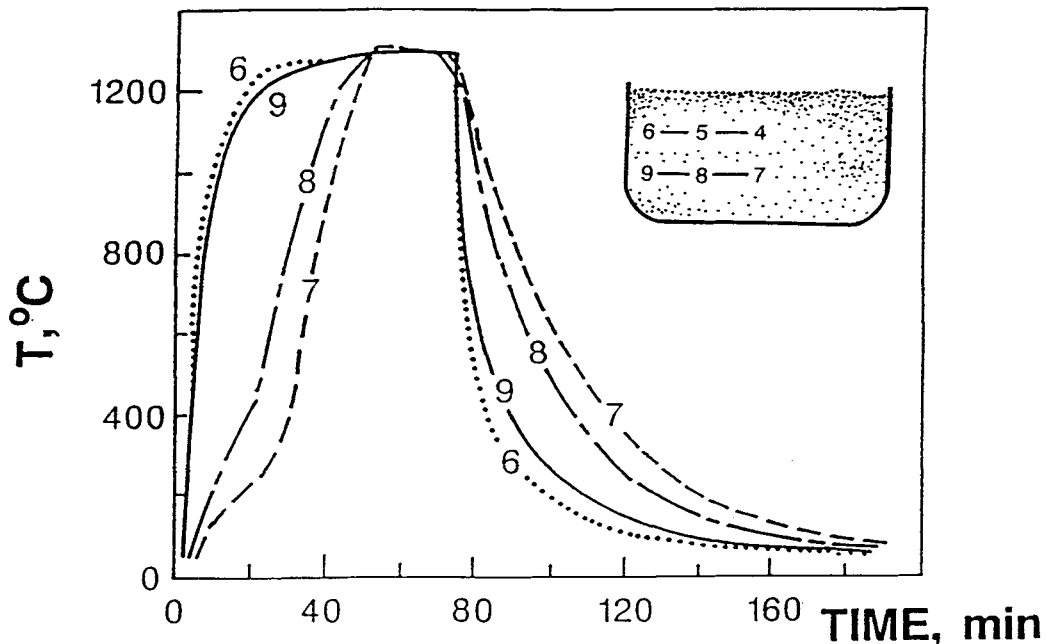


Figure 1- Thermal treatment of powders at different positions in the silica crucible.

III- RESULTS AND DISCUSSION

Chemical analysis, BET surface area measurements, SEM and TEM observations.

The main impurities found in the powder are K(13 ppm by wt), Ca (<10 ppm), Fe (<10 ppm), Na and Si. The amount of Si (19 ppm to 63 ppm) and Na (5 ppm to 22 ppm) depends on the thermal treatment conditions and of the position of the powder in the silica crucible.

The BET analysis and SEM observations show that the size of the α -alumina particles increases both with the annealing time and cooling rates. Furthermore, for the same thermal treatment conditions the BET specific surface area of the powder increases with the amount of Mg and Si. The grains have generally elongated forms. Their size is included between 50 and 400nm. Whatever the thermal treatment conditions of the powder, the TEM observations show that the microstructure of the powder is very similar. The calcination is generally accompanied by a coarsening and sintering of the particles which leads to complex grain shapes and in some grains nanometer scale porosity. A texture has been observed at the surface of some grains which may be due to spinel precipitates.

Nano-chemical analysis

XPS analysis show a strong surface segregation of Mg at the periphery of the grains. (Table 1). It has been calculated from the Mg_{1s} , Mg_{2s} and Mg_{2p} peaks⁽²⁻⁴⁾. XPS also detected the presence of Na in the surface region of the grains. The level of Na at the periphery of the particles is higher (1.4at %) in powder from position 7 than in powder from position 6 (0.6 at.%), in agreement with the chemical analysis .

Sample and position in the crucible	Mg 1s	Mg 2s	Mg 2p
Al ₂ O ₃ 1650 / 6	4,7	1,0	0,9
Al ₂ O ₃ 1650 / 7	4,4	1,0	1,0
Al ₂ O ₃ 550 / 6	3,4	1,0	0,7
Al ₂ O ₃ 550 / 7	2,4	0,6	0,5

Table 1- Amount of magnesium (at%) determined by XPS at the near surface of the alumina powder treated in a silica crucible (position 6 and 7).

In Fig.2 we report the main concentration of Mg in a depth of 2.0nm (Mg_{2s}) as a function of the mean concentration in a depth of 0.5nm (Mg_{1s}). The data exhibit a straight line whose slope is equal to the ratio of the analysis depth (1/4). This result suggests that Mg segregated mainly on a depth lower or equal to 0.5nm. This result is in agreement with the estimate of the repartition of Mg in the grains (Fig.3) taking into account the amount of Mg in the powder and the XPS results.

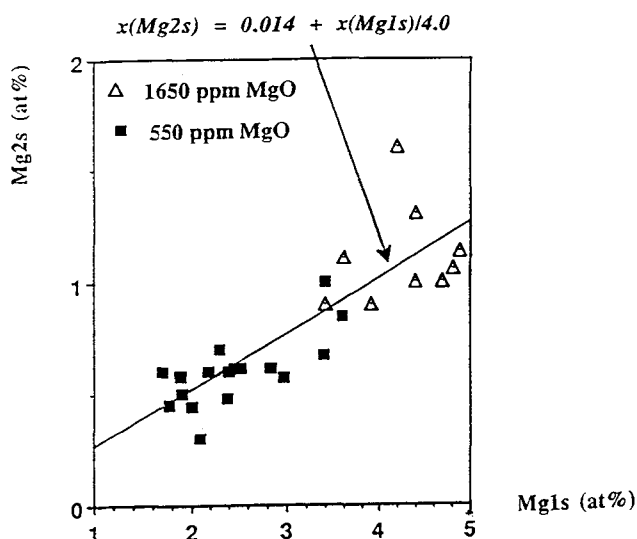


Figure 2- Concentration of magnesium in a depth of 2.0 nm (Mg_{2s}) as a function of the concentration in a depth of 0.5nm (Mg_{1s}).

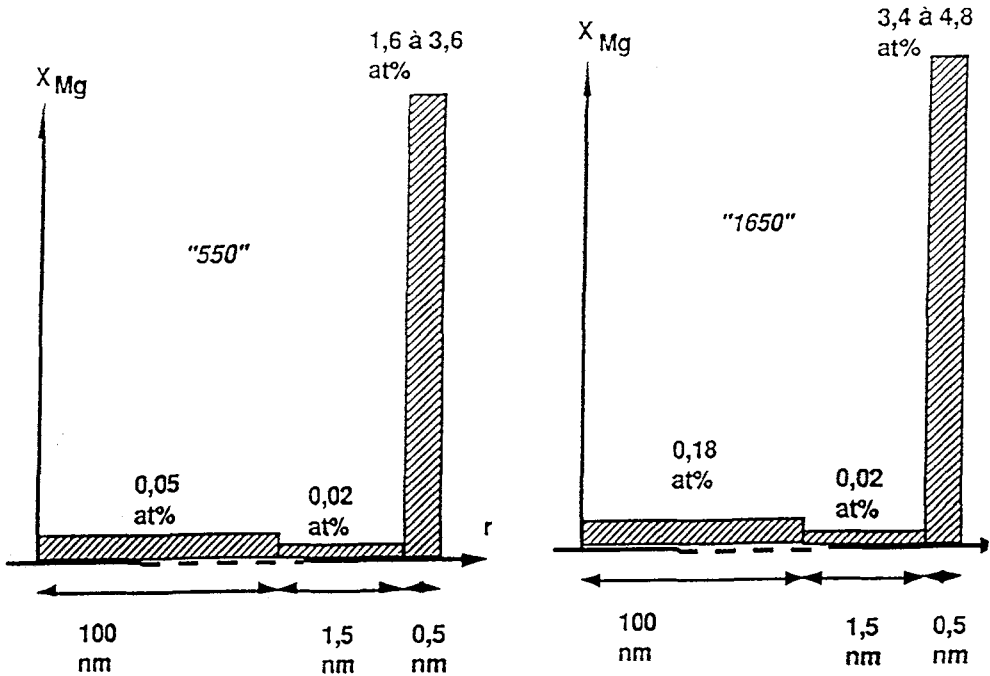


Figure 3- Distribution profile of magnesium through an alumina grain treated at 1300°C, for both 550 ppm and 1650 ppm MgO-doped powders.

ACKNOWLEDGEMENTS : This work has been sponsored by the "Ministère de la Recherche et de la Technologie-département Matière et Matériaux", contract N°90 A 0220. The authors are indebted to J.C.Droguet and L.Du Repaire (Baikowski-Chimie, Annecy), I.Sproule and M.Graham (CNRC, Ottawa) for their collaboration and fruitful discussions.

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