STABILITY OF t-ZrO$_2$ PARTICLES IN ALUMINA-ZIRCONIA COMPOSITES: PART. 1 COMPETITION BETWEEN SIZE AND STRAIN EFFECT

C. Rabache$^{1,2*}$, G. Bouchet$^2$, G. de Calan$^2$, J-M. Kiat$^{1,3}$, N. Guiblin$^1$, F. Porcher$^3$

$^1$ Laboratoire Structure Propriétés et Modélisation des Solides UMR 8580, Ecole Centrale Paris, 92295 Châtenay-Malabry, France
$^2$ Nanoe SARL, 34 route de Longjumeau, 91380 Chilly-Mazarin, France
$^3$ Laboratoire Léon Brillouin, CE Saclay CNRS-UMR12, 91991 Gif-sur-Yvette Cedex, France

* Corresponding author (c.rabache@nanoe.com)

Keywords: Alumina-zirconia composites, size effect, strain effect, microstructural, mechanical properties

1 General Introduction

Alumina-zirconia composites are well-known materials which combine the good mechanical properties of zirconia and the stability properties of alumina. Among them zirconia toughened alumina have lot of potential biomedical applications like orthopedic prosthesis. However most of the fundamental studies which obviously deal with samples synthesized at a laboratory scale shown very different results depending on elaboration, shaping and sintering conditions. In this work, we have studied composites produced at a massive scale by the Nanoe company with a process of mechanical milling in aqueous media leading to composites in a concentration range from 2,5 to 50wt% of zirconia and a relative density above 99% with fine and well dispersed microstructure.

From a fundamental point of view it is a fascinating case in which structural phase transitions and metastability result in enhancement of applicative properties. Indeed the high mechanical properties of these composites are associated to the retention of the high temperature tetragonal structure of zirconia down to room temperature either by adding stabilizing oxides such as yttria or by keeping grain size below a critical threshold. But the stability conditions of the tetragonal particles in the alumina matrix are complex and several parameters play a key role. The residual strains due to the difference of the coefficient of thermal expansion (CTE) between alumina and zirconia, the yttria content, the grain size, the presence of cubic or monoclinic zirconia, the presence of micro-strains, the temperature, interact and alter the stability of tetragonal phase.

All these parameters have been systematically investigated by XRD, SEM and neutron diffraction for composites with increasing content of zirconia and yttria and have been compared with yttria-doped zirconia.

2 Size effect

Several authors [1-5] reported experimental observations of a zirconia critical crystallite size of $\approx$25nm below which the tetragonal phase is stabilized at room temperature. Also the presence of yttria is known to increase this critical crystallite size. In addition the critical crystallite size is different in powders and in ceramics in which the critical grain size is estimated between 200 and 300nm. We have systematically measured zirconia grain size dependence with chemical composition, sintering temperature etc. In particular we have observed a grain size reduction of non-doped zirconia in composites compared to zirconia compounds, i.e a diminishing from some micrometers in zirconia down to 250nm in composite with 10wt% zirconia. This allows the stabilization of the tetragonal phase thanks to the size growth hindering by the alumina matrix. Therefore a retention up to 97wt% of tetragonal phase in this composite compared to 0% of phase in pure zirconia compound.

3 Strain effect

3.1 Hydrostatic strain

Garvie [2] have shown that the critical size of zirconia (CS) increases with hydrostatic pressures. In alumina-zirconia composites the difference of CTE between alumina and zirconia (CTE$_{\text{Al}_2\text{O}_3}$ < CTE$_{\text{ZrO}_2}$) leads to significant residual strains. Thus the alumina is in compression while the zirconia is in tension [6]. We have studied the composition and temperature dependence of strain; in particular we have observed an increasing of the compression on the alumina and a decreasing of the tension on the zirconia with increasing zirconia content. The tension on the zirconia leads to a decrease of the tetragonal phase stability. The hydrostatic strains...
have an opposite effect on tetragonal phase stabilization compared with size effect. Also the anisotropy of zirconia is less pronounced at high content in the alumina (Fig. 1).

3.2 Non-hydrostatic strain

The presence of non-hydrostatic strains, i.e. local strains, has many consequences. The first one is to disperse the lattice parameters without modifying the average value. The second one is to allow phase coexistence in polymorph solids in which a single phase is expected [2]. For the study of non-hydrostatic strain, we have used the Williamson-Hall approach which allows separating size and strain effect at a local scale. We have measured the concentration and temperature evolution of local strain for each phase. The yttria concentration dependence is shown for zirconia in composites in figure 2. Whereas the micro-strains on alumina monotonically increase with the zirconia content, in contrary, the micro-strains on tetragonal and cubic phases increase up to 25-30wt% of zirconia and then decrease at higher concentrations. We also observed that the content of yttria has no influence on the local strains.

4 Conclusions

We have studied alumina-zirconia composites produced at massive scale with various contents of zirconia and yttria by XRD, neutron diffraction and SEM which have allowed us separating and understanding the influence of all parameters: composition of composites, grain size, macro and micro-strain, presence of additional phases (monoclinic or cubic zirconia), temperature etc...

These results have been compared with a study of mechanical properties of these composites (Rabache et al. this conference) which results in predicting the more suitable composition of composite according to the mechanical properties expected by the customers.

References


Fig. 1: Evolution of hydrostatic strain on tetragonal phase function of zirconia content

Fig. 2: Evolution of non-hydrostatic strains with tetragonal phase function of zirconia content