MICROSTRUCTURAL STUDY OF SILICA-DOPED ZIRCONIA CERAMICS

L. GREMILLARD*, T. EPICIER, J. CHEVALIER and G. FANTOZZI
GEMPPM, CNRS 5510, Bâtiment 502, INSA-Lyon, F-69621 Villeurbanne Cedex, France

Abstract—The aim of this study was to show the effects of small silica additions on the microstructures and mechanical properties of 3 mol% yttria-stabilised zirconia (3Y-TZP) ceramics. Experiments were conducted on different batches of 3Y-TZP (pure to 2.5 wt% silica-doped). Microstructures were characterised mainly by transmission electron microscopy (TEM), but also by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Silica was found at triple junctions, but neither at grain boundaries nor in the lattice. Undoped zirconia ceramics exhibited faceted grains and significant internal stresses, while doped zirconias showed a much more rounded microstructure and a lower level of internal stresses. Low-temperature degradation (LTD) and slow crack growth (SCG) measurements were conducted on the different batches. The addition of silica strongly increases LTD resistance without affecting the SCG behaviour. The microstructural origins of the different behaviours are discussed. © 2000 Acta Metallurgica Inc. Published by Elsevier Science Ltd. All rights reserved.

Résumé—Cette étude a pour but de montrer l’effet d’un dopage à la silice sur la microstructure et les propriétés mécaniques de différentes zircons stabilisées avec 3% molaire d’oxyde d’yttrium (3Y-TZP) (pures ou dopées à la silice (jusqu’à 2.5% en masse)). Les microstructures ont été observées principalement au microscope électronique en transmission (MET), mais aussi au microscope électronique à balayage (MEB), et en diffraction des rayons X. La silice se retrouve principalement aux joints triples, mais ni aux joints de grains ni dans le réseau. La zirconie non dopée est constituée de grains présentant des angles vifs et présente des contraintes internes significatives, alors dans les zircones dopées les grains sont beaucoup plus ronds et les contraintes internes moins importantes. Des mesures de vieillissement et de propagation sous critique des fissures ont été menées sur les différentes zircons. L’addition de silice accroît la résistance au vieillissement sans détériorer les propriétés mécaniques. L’origine microstructurale de ces différents comportements est analysée. © 2000 Acta Metallurgica Inc. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Transmission electron microscopy (TEM); X-ray diffraction (XRD); Microstructure; Structural ceramics; Zirconia

1. INTRODUCTION

3 mol% Yttria-stabilised zirconia (3Y-TZP) ceramics have been extensively studied [1] and used in many applications because of their unique advantages at room temperature. Indeed, it was shown in the 1970s that zirconia exhibited a phase transformation toughening acting to resist to crack propagation. The stress-induced phase transformation of metastable tetragonal phase towards the monoclinic phase at the crack tip is accompanied by a volumetric expansion that induces compressive stresses, reducing the driving force for crack propagation. 3Y-TZP ceramics can exhibit toughness, $K_{IC}$, and strength, $\sigma$, of more than 6 MPa m$^{1/2}$ and 2 GPa, respectively. However, 3Y-TZP ceramics are susceptible to low-temperature degradation (LTD) and slow crack growth (SCG) when exposed to humid atmospheres.

SCG corresponds to a crack propagation for stress intensity factors $K_I$ below $K_{IC}$, and is experimentally described by plotting the crack velocity versus stress intensity factor ($V$–$K_I$ diagram). Experimental studies [2, 3] have shown that SCG in zirconia ceramics is a consequence of stress-assisted corrosion by water molecules, as for silica glass, which involves a thermally activated reaction between Zr–O and H–O bonds. A threshold value, $K_{Io}$, under which no propagation occurs, defines a safety stress intensity factor. Its value is about 50% of the $K_{IC}$. Recent studies have shown that the whole V–$K_I$ diagram is shifted towards high $K_I$ values when increasing the grain size [3]. Thus, grain size increases SCG resistance by increasing both $K_{IC}$ and $K_{Io}$.

LTD proceeds by a slow, tetragonal-to-monoclinic...