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### Synthesis and mechanical and tribological characterization of alumina-yttria stabilized zirconia (YSZ) nanocomposites with YSZ synthesized by means of an aqueous solution-gel method or a hydrothermal route

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#### Abstract

In the present study, yttria stabilized zirconia (YSZ) nanoparticles, prepared by means of an aqueous solution–gel method or a hydrothermal route, are incorporated in a matrix of submicron alumina particles by wet mechanical milling. The microstructural characteristics and the mechanical and tribological properties of the obtained alumina–YSZ nanocomposites are evaluated as a function of different processing conditions like milling time, YSZ amount, sintering procedure and synthesis method of YSZ.

It is noticed that the synthesis procedure and the agglomeration degree of the YSZ nanoparticles seriously affect the densification process of the alumina–YSZ nanocomposites and also their mechanical and tribological properties.

The most probable cause for the difference is that the hydrothermally prepared YSZ nanoparticles are not as homogeneously distributed in the alumina matrix as the solution–gel prepared nanoparticles. Moreover the former nanoparticles have surface groups which release undesired gases during sintering. Thus to obtain a dense sample, nanocomposites with these nanoparticles require a higher sintering temperature and this has a negative effect on the mechanical and tribological properties of these materials. © 2007 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

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### 1. Introduction

Zirconia or yttria stabilized zirconia (YSZ) particles are frequently employed as a toughening agent for alumina [1–4]. Although the toughness of alumina is indeed enhanced by introducing zirconia particles, the improvement of toughness may, depending on crack control or transformation control, be accompanied by a decrease in strength [1]. Thus, the optimization of all the mechanical properties is widely studied for alumina–zirconia composites [1,2,4,5].

As discussed in literature, the transformation toughening ability of zirconia is based on fracture energy absorption by the tetragonal to monoclinic zirconia phase transformation [2,3,6]. To achieve the maximum benefit of this effect, the distribution of the  $ZrO_2$  or YSZ particles in the alumina matrix must be uniform and the size should be as small as possible [2]. All mechanical properties of such alumina–zirconia composites are affected by, e.g. the powder characteristics, the processing methods and the variation of the  $ZrO_2$  or YSZ content.

Another driving force for the development of composites is the fact that they can possess good wear resistance and are

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hence potential candidate materials for a number of tribological applications. The friction and wear properties of composites depend on material parameters (matrix, reinforcement chemistry and volume, hardness, toughness), counterbody material, operating parameters (sliding speed, load, humidity, etc.) and are system dependent [7,8].

In this study, a commercial alumina powder, used as the matrix material, is mixed with a second phase of freshly synthesized YSZ nanoparticles, either prepared via an aqueous solution-gel method or a hydrothermal route. By freshly synthesizing YSZ nanoparticles in different ways parameters like particle size, particle size distribution and surface characteristics can be varied. It is believed that the characteristics of the nanopowders, which are influenced by, e.g. the synthesis mechanism and the starting materials, can affect the sintering and densification mechanisms of the nanocomposites. The purpose of this work is not only to detect and explain possible differences in the microstructure or in the densification mechanism of alumina-YSZ nanocomposites, but also to evaluate the possible influence of the YSZ preparation method, the sintering conditions, the YSZ amount, etc... on the mechanical and tribological properties of these alumina-YSZ nanocomposites.

#### 2. Experimental procedure

## 2.1. Synthesis of YSZ nanoparticles by means of an aqueous solution–gel method

The following starting materials are used for the synthesis of YSZ nanoparticles via an aqueous solution–gel route: zirconium(IV) propoxide solution ( $C_{12}H_{28}O_4Zr$ , ~70% in propanol, Fluka), yttrium(III) oxide ( $Y_2O_3$ ; 99.99%, Acros), acetic acid ( $C_2H_4O_2$ , glacial, p.a., Acros), citric acid ( $C_6H_8O_7$ , 99%, Aldrich), hydrogen peroxide ( $H_2O_2$ , 35wt% in  $H_2O$ , p.a., stabilized, Acros) and ammonia (ammonium hydroxide volumetric standard; 4.98N solution in water, Aldrich). The synthesis is based on earlier work [9], but, in this study, zirconia is stabilized with yttria.

At first, precursor solutions of the separate metal ions are synthesized as shown in Fig. 1. The exact concentrations of the monometallic precursor solutions are determined by ICP-AES (Perkin-Elmer, Optima 3000). The monometallic solutions are mixed in the desired proportion, according to the ratio 3 mol%  $Y_2O_3$  and 97 mol% ZrO<sub>2</sub>, and stirred for 1 h.

The thus obtained combined precursor solution is then poured out in a petri-vessel and the solvent (water) is evaporated in an air-flushed oven at 60 °C in order to allow gelation. The obtained gel is calcined in a tube furnace (heating rate 20 °C/min; dry air) at 700 °C for 30 min to form yttria stabilized ZrO<sub>2</sub>, further on called YSZ<sub>SG</sub>.

### 2.2. Synthesis of YSZ nanoparticles via a hydrothermal route

The hydrothermal synthesis of YSZ nanoparticles is already described in previous work [10]. First, an aqueous solution (0.25 M) of ZrO(NO<sub>3</sub>)<sub>2</sub>·xH<sub>2</sub>O (zirconyl nitrate hydrate, 99.9%, Aldrich) is prepared. A suitable amount of Y(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O (yttrium(III) nitrate hexahydrate, +99%, Merck) is added, according to the ratio 3 mol% Y<sub>2</sub>O<sub>3</sub> and 97 mol% ZrO<sub>2</sub>. After stirring for 30 min, a diluted solution of ammonia (ammonium hydroxide volumetric standard; 4.98N solution in water, Aldrich) is slowly added until a pH of about 7.5 is reached. Under the present conditions, both metal ions are present as hydroxides, forming a gelly precipitate. This precipitate is filtered and repeatedly washed with water to remove remaining nitrates. The thus obtained washed precipitate is put into a Teflon-lined Parr<sup>®</sup> (120 ml) pressure-vessel together with water (70 ml), and hydrothermally treated in an oven at 200 °C for 2 h. After the hydrothermal treatment a suspension of YSZ particles (or YSZ<sub>HT</sub>) is obtained.

### 2.3. Preparation of alumina-YSZ nanocomposites

YSZ nanoparticles, either synthesized by means of an aqueous solution–gel technique (YSZ<sub>SG</sub>) or via a hydrothermal method (YSZ<sub>HT</sub>), are incorporated in a matrix of commercial



Fig. 1. Overview of the synthesis of a clear precursor solution consisting of a  $Zr^{4+}$  solution and a  $Y^{3+}$  solution.

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