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Sol–gel processing of alumina–zirconia minispheres

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Abstract

Alumina–zirconia minispheres were prepared by the sol–gel method. The starting material used for the preparation of alumina sol was aluminium-tri-isopropoxide. Zirconia sol was prepared from zirconium oxychloride. Required quantity of zirconia sol was added to alumina sol, so that the final composite contains 5, 10, 15, and 20 wt.% zirconia. A suitable binder was added to the mixed sol and aged at room temperature for some gelation to occur. At the appropriate viscosity, spheres were formed in ammonia solution. The spheres were dried at room temperature and then sintered at 1300 °C for 2 h. X-ray diffraction (XRD) analysis shows the presence of α-Al_2O_3 in alumina spheres and α-Al_2O_3 and t-ZrO_2 in spheres containing 5 wt.% zirconia. Monoclinic ZrO_2 is also present in spheres containing higher amount of ZrO_2. Thermogravimetric analysis indicated the removal of most of the volatile up to 600 °C. Differential thermal analysis shows, that the α-Al_2O_3 phase transformation temperature is higher for sample containing zirconia. Fourier transform infrared spectroscopy (FTIR) indicated the phase transition to α-Al_2O_3 in corroboration with X-ray studies. The density of the sintered spheres was found to be highest for spheres containing 10 wt.% zirconia.

Keywords: A. Sol–gel process; A. Sintering; D. Al_2O_3; D. ZrO_2

1. Introduction

Conventionally ceramic-grinding media was produced using powder compaction method. This has certain limitation on the fabrication of high quality, fine grinding media, since slight change in processing parameter leads to variety of size, density and non-uniformity in shape. In order to overcome this difficulty, sol–gel technique has been developed [1]. Sol–gel method is used for the preparation of gels of various shapes, that is, monoliths, fibers, coating films, spheres, etc. It is well known that the composition of the starting alkoxide solution including the kind of catalyst, water content and presence or absence of any additives and the reaction condition affects the rate of hydrolysis, the rate of condensation, the shape of polymer particles produced in the solution and the state of aggregation of particles, and thus the characteristics of the sols and gels produced in the reaction. The flow property of the sol is important in obtaining information on the structure of sol as well as in making shaped gels [2]. Various methods used for preparing spheres, such as drop generation technique, spray pyrolysis, emulsion evaporation technique, emulsion water extraction, emulsion-ion extraction technique have been developed [3].

The discovery of the stress-induced transformation toughening of zirconia [4] has led to the development of new class of tough ceramics. Among these tough ceramics zirconia toughened alumina is widely studied because of its high strength, toughness, and good stability in humid environment [5–9]. Alumina with dispersed zirconia can be expected to combine the high modulus and strength of alumina with toughening by zirconia particle to give good mechanical property [10]. The present work aims at the preparation of alumina–zirconia spheres by the sol–gel technique and the characterization of spheres.

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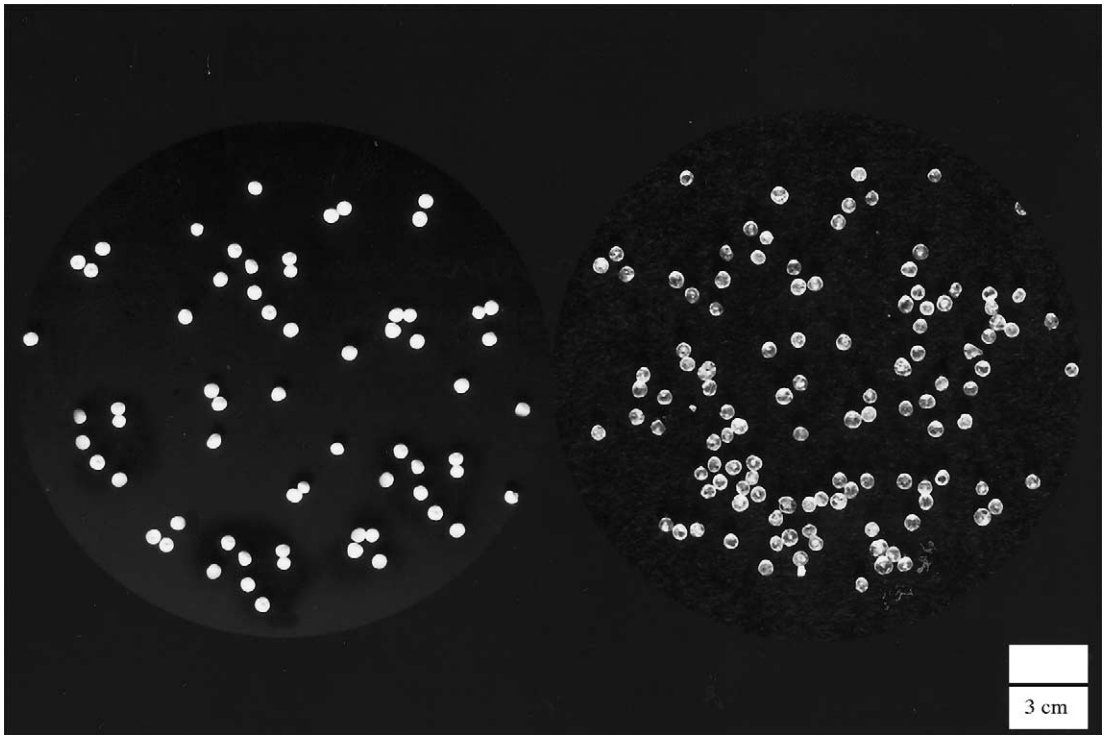

Fig. 1. Photograph of sintered and as-dried alumina–5 wt.% ZrO_2 minispheres.

2. Experimental procedure

Alumina sol was prepared according to the procedure described by Yoldas [11]. Aluminium-tri-isopropoxide (CDH, New Delhi) was dissolved in distilled water to 1M concentration and refluxed at 80 °C for 3 h in the presence of acid catalyst (0.07 mol nitric acid) and then cooled. Zirconium oxychloride (Otto Kemi, Mumbai) was dissolved in distilled water taken in a beaker to 1M concentration.

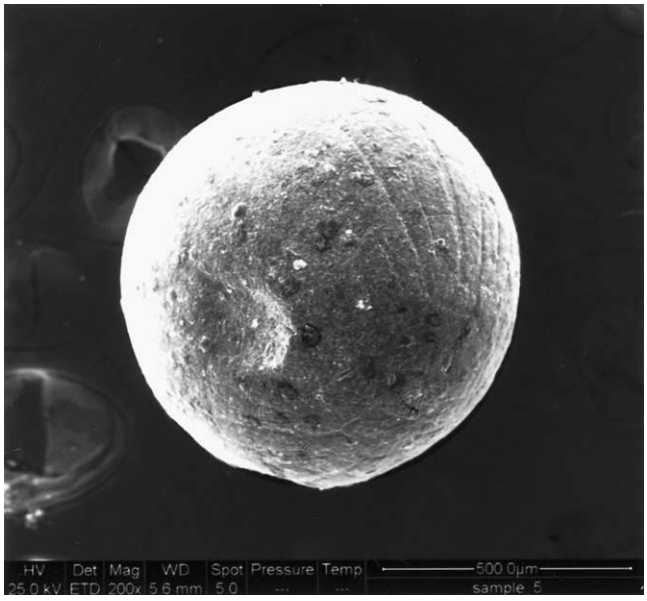

Fig. 2. SEM micrograph of sintered Al_2O_3–5 wt.% ZrO_2.

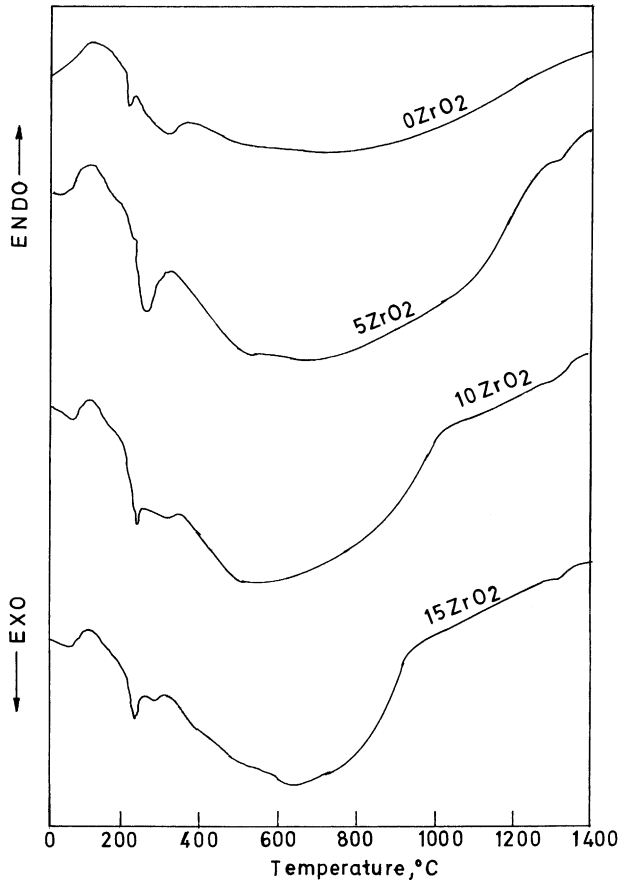

Fig. 3. Differential thermal analysis of alumina and alumina–zirconia minispheres.