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Hertzian contact damage in silicon nitride ceramics with different porosity contents

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Abstract

This work investigates the Hertzian contact damage in Si_3N_4 ceramics with 3 and 18% porosity processed by spark plasma sintering technique. Low densification was achieved by sintering at pressures lower than needed for a full density. The indentation tests were performed using diamond spheres of various sizes to evaluate the Hertzian contact damage at different scales. Surface damage was observed under electron microscopy while subsurface damage was examined using focused ion beam sectioning technique. Different failure modes were noticed, depending on the size of the volume solicited and the porosity content. At small scale, short cracks initiate from the existing pores then coalesce leading to a quasi-plastic failure mode. At larger scale and for high porosity content, surface ring and radial cracks usually observed for dense brittle materials vanish; the damage mode is mostly related to the fragile rupture of bridges between collapsed pores.

Keywords: Si_3N_4; Porous materials; Hertzian contact; Size effect; Contact damage

1. Introduction

Dense silicon nitride materials are used to fabricate high-speed metal cutting tools, rolling element bearings and many other components for applications at low or high temperatures.[1,2] Most of the brittle materials including Si_3N_4 contain two kinds of pores:(i) well-controlled and voluntarily introduced into the specimens by addition of a pore-forming agent to the starting powder or by decreasing the amount of sintering aid for applications such as catalyst supports, gas filters and biomaterials,[3,4] or (ii) uncontrolled and undesirable with random size distribution and morphology. This second type is mainly due to the processing and can considerably impact the mechanical properties of the component,[5] which in turns significantly reduce its lifetime.[6]

Over the last century, several techniques have been developed to process Si_3N_4 based materials from the starting mixtures containing material powders and small amounts of rare- earth sintering elements. Nowadays, field assisted sintering technology (FAST) better known as spark plasma sintering technique (SPS) is emerging.[7–9] The advantage of this method over the conventional hot pressing and hot isostatic pressing techniques consists in sintering the starting powders at relatively lower temperatures and shorter processing times.[10] It results from the simultaneous application of a pulsed (on–off) direct current of a few thousand amperes and an uniaxial pressure during sintering.

Although the considerable effort made to optimize the processing conditions (time, pressure, temperature) of this technique, final sintered specimens can present some residual porosity. Previous studies in liquid-phase-sintered alumina [11] and silicon nitride materials with 37% of porosity [12] highlighted the key role of the voids on the damage mode transition from tension driven cracks mode usually observed in dense brittle materials with fine microstructure to distributed shear and compression driven subsurface damage mode.[13] On the other hand, the authors reported a significant effect of the porosity on the different mechanical properties (elastic modulus, flexural strength, hardness, etc.). To facilitate the damage observations, Hertzian

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Table 1
List of the materials and corresponding processing conditions.

Material	Yttria (wt%)	Temperature (°C)	Time (min)	Pressure (MPa)
A	5	1700	15	40
B	5	1700	15	20
C	1	1700	15	10

contact tests are made using large spheres of radius a few millimetres. In addition, the state of the art shows a disparity in the size of indenters used to characterise the damage modes. This raises the following question: is there any effect of the indenter size on the Hertzian contact damage mechanisms for porous brittle materials? This question is highly relevant for engineering applications since the contact size may vary by several orders of magnitude depending on the application.

The current study aims to provide some elements to respond to the previous issue. To do that, we discuss the relation between the porosity content ($\leq$ 20%) resulting from an incomplete sintering of the starting silicon nitride powder, the diamond indenter size and the damage phenomena under Hertzian contact conditions. The subsurface damage at large scale was observed using the standard bonded interface sectioning technique after indentation with a sphere of radius 1 mm. While at small scale, tests were made with a sphere of radius 0.2 mm and resulting subsurface damage imaged using focused ion beam sectioning technique.

2. Materials and methods

2.1. Material processing

Polycrystalline Si_3N_4 materials were prepared from mixtures of a commercial high α/β ratio ($\geq$ 95%) Si_3N_4 powder (SN-ESP, Ube Industries, Tokyo, Japan) with addition of 1% and 5% of 99.99% high purity yttria (Wako Pure Chemical industries, Japan). Prepared batches were mixed by ball milling in ethanol with alumina balls and dried in a rotating evaporator. The powder mixtures were sintered using SPS (SPS-1050, SPS Syntex Inc., Japan) technique into disks of diameter 30 mm in nitrogen gas atmosphere. Materials were processed at sintering temperature of 1700 °C for 15 min, heating rates were as follows: 120 °C/min from 0 to 600 °C, 100 °C/min from 600 °C to 1600 °C, then 20 °C/min from 1600 °C to the suitable temperature. To study the influence of the sintering pressure on the contact damage, three different levels were applied: 40, 20 and 10 MPa. All the mixture compositions and experimental conditions are summarized in Table 1. Note that the amount of yttria was decreased to 1% for specimen C in order to increase further the porosity volume fraction.

2.2. Materials characterization

The bulk densities of the materials A, B, and C were measured using Archimedes' method.[14] The porosity volume fractions ϕ were obtained from the experimentally measured densities ρ and true theoretical densities ρ_{th} estimated by the rule of mixtures as follows: $\phi = 1\text{-}\rho/\rho_{th}$.[11] Vickers hardness measurements were carried out on a Buehler micromet 5104 machine with a magnification 40× under a load and dwell time of 9.6 N (1 kgf) and 15 s, respectively. Ten indentations were performed for each material to minimize the experimental error. Ultrasonic measurements (TM506A, Hitachi) were performed to determine the elastic properties (E, ν) of the SPSed disks.

X-ray powder diffraction with CuK_α radiation (Ultima IV, Raigaku) were performed and acquired data analysed by Jade 5.0 software to determine the phase composition of the sintered materials. To reveal the grain structures, polished surfaces were chemically etched with sodium hydroxide (NaOH) at 500 °C, the etching time was optimised for each material, 120, 90 and 60 s for materials A, B and C, respectively. The surfaces were carbon coated before micrograph acquisition under scanning electron microscopy (SEM, Jeol 7500F, Hitachi).

2.3. Indentation tests

Sintered disks were machined into rectangular samples of dimension of 2 mm (thickness) × 3 mm (width) × 30 mm (length). A particular effort was made in polishing the samples using diamond disks and suspensions with grain sizes up to 0.3 μm. Scratch tests with a three-sided pyramid diamond Berkovich tip (Agilent Nano Indenter® G200) were performed to estimate the final surface roughness Ra. Mean values of 0.08, 0.11 and 0.16 μm were obtained for the materials A, B and C, respectively.

Before testing, the specimens were mounted on a flat rigid substrate using a thin layer of commercial crystalbond to avoid radial displacements. The Hertzian contact tests were performed on a standard tensile testing machine (Model 5965, Instron) at cross-head speed of 0.05 mm/min. Two diamond spherical indenters of different radii, 0.2 and 1 mm under loads ranging from 0 to 2000 N were used. To measure the residual indent depths, surface depressions after complete unloading were observed under optical and laser microscopy (VK100, Keyence). The usual procedure to study the subsurface contact damage is the bonded interface sectioning technique. It consists on two polished surfaces clamped face to face using a thin layer of adhesive. Indentation was then symmetrically made on the polished top surface of the specimen. After testing, the adhesive is dissolved, the surface cleaned and the damage observed under optical microscopy. More details about this technique are given in reference.[13]

The method described in the previous paragraph is pertinent in the case of large contacts (few millimetres diameter). However, the use of this technique for very small indents (few micrometers diameter) is complicated and may affect the observed damage. Another approach is then needed. Here, examination of the damage below the indents was performed using focused ion beam cross-sectioning technique (Versa 3D Dual Beam, FEI Company). It consists of three successive steps: firstly, one micrometer carbon layer deposition is made on the top surface of the indent to prevent etching. The second step is digging using a focused beam of gallium ions (Ga+) beneath the contact zone to the desired depth. A first approximation of this depth is given by the Hertz theory.[15] Finally, micrograph