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Experimental study and simulation of plastic deformation of zirconia-based ceramics in a pulsed electric current apparatus

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Abstract

Plastic deformation by compression of cylindrically shaped zirconia (ZrO_2)-based ceramics in a pulsed electrical current apparatus was studied using a combined experimental and theoretical approach. Both fully dense electrically insulating 3Y–ZrO_2 and electrically conductive 3Y–ZrO_2–TiCN 60/40 (vol.%) ceramics were subjected to a compressive load at temperatures above 1200 °C. Deformed non-conductive 3Y–ZrO_2 samples were concave shaped, whereas the composite samples exhibited a different behaviour depending on the electrical current path within the set-up. A convex shape was obtained when the current was freely flowing through them, while they started to become concave shaped when the samples were separated from the graphite pressing punches by relatively low conductive silicon carbide disks. The secondary titanium carbonitride (TiCN) phase in the composite materials exhibited a grain boundary pinning effect, which limited coarsening of their microstructure. The influence of current flow on the shape of the deformed ceramic samples was interpreted in terms of the temperature distribution generated during hot deformation. Finite-element simulations, coupling thermal, electrical and mechanical fields, were used to explain the deformation behaviour of the different samples. A subsequently coupled thermal–electrical and mechanical analysis procedure was developed for this aim. Special attention was paid to the materials and interactions properties used during modelling. The modelling results are in good agreement with the experimental data, so that the developed finite-element approach and code can be used for the analysis of near net shaping of ceramic parts assisted by an electrical field.

Keywords: Plastic deformation; Ceramics; Composites; Finite-element modelling; Pulsed electric current sintering

1. Introduction

Superplastic deformation of fully dense polycrystalline ceramics is an exciting phenomenon discovered in 1986 by Wakai et al. [1] and extensively investigated since then [2]. Potential practical applications for net shape forming of ceramics were shown in experiments on punch and gas pressure forming of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) sheets and tubes [3,4], superplastically compression of Si_3N_4-based nanocomposites, closed-die isothermal forging of Y-TZP preforms [4,5], sinter-forging of Si_3N_4–Si_2N_2O gears [6] and joining of Y-TZP layers with different compositions [7]. Despite these promising results, the industrial application of net shape forming is still very limited because enhanced plasticity in most ceramics occurs at strain rates of approximately $10^{-4}\ s^{-1}$ or less even at high temperatures in the range of 1400–1650 °C [8]. At such strain rates, a forming process takes too long to be economically attractive for any industrial application. To overcome this limitation, the strain rate should be increased to at least $10^{-2}\ s^{-1}$ [8].

Enhanced plasticity of ceramics is usually observed when the grain size is <1 μm [9]. In order to successfully realize high-speed plastic deformation of ceramics, dynamic grain growth must be suppressed [8]. This can

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be achieved by using multiphase ceramic composites and by lowering the deformation temperatures and time. Due to the very direct way of heating and the presence of an electrical current/field, we believe that pulsed electric current sintering (PECS) technology, also known in the literature as spark plasma sintering or the field-assisted sintering technique, when combined with adequate material processing, can be used to drastically enhance the superplastic deformation rates. PECS technology is traditionally used to consolidate powders in a graphite tool and allows the application of very high heating rates due to direct Joule heating, originating from a pulsed DC current. If the powder compact is electrically conductive, the current passes through it and results in an additional internal heating. During PECS densification, the powder is simultaneously uniaxially compressed by a force that can be constant or time dependent [10]. A non-conductive powder heats up by thermal conduction from the resistively heated mould. In both cases, the obtained heating rates are much higher and the total processing time needed to realize full densification can be significantly reduced as compared to traditional hot pressing where heat transfer takes place by radiation from an externally positioned heating element. Even more important than the direct Joule heating effect may be the intrinsic influence of the electrical field on the superplastic behaviour of ceramics during PECS. A decrease in the flow stress, a retardation of the grain growth and a substantial rise in the ductility of ceramics deformed in an electric field has been reported [11,12]. This effect becomes more pronounced when a pulsed field is used [13].

A few literature reports mention the successful application of PECS for the shaping of ceramics. Shen et al. deformed fully dense Si_3N_4-based ceramics containing a very small amount of glassy phase at compressive strain rates of $10^{-2}\ s^{-1}$ at temperatures higher than 1500 °C. Accordingly, a component with complicated shape, sharp edges and excellent surface finish was formed [14]. Jiang and co-workers forged fully dense Al_2O_3–ZrO_2–$MgAl_2O_4$ composites into a complex shaped part by PECS with a strain rate of approximately $10^{-2}\ s^{-1}$ and at temperatures as low as 1150 °C. The obtained product was free of surface cracks and the starting nanocrystalline structure remained unaltered [15].

Despite these achievements, some problems may occur when PECS technology is used for shaping purposes, especially when the processing of large and complex parts is envisaged. Major problems are the generation of inhomogeneous microstructures and properties and the concomitant risk of crack generation due to stresses resulting from the temperature gradients arising during PECS. The main reasons for the development of the temperature gradients are the high applied heating rates, reaching 1000 °C min^{-1}, in combination with the low thermal conductivity of most ceramic materials as well as the interplay between tool geometry and direct Joule heating of the same pressing tool. The temperature distribution inside a PECS set-up containing a fully dense powder sample has been studied by the finite-element method (FEM) [16–18]. The influence of the tool geometry, the presence of thermal insulation surrounding the die and the sample conductivity on the temperature inhomogeneity was highlighted. Significantly larger temperature gradients were observed in electrically conductive samples as compared to nonconductive samples when identical PECS conditions were applied [17]. Similar or even larger thermal gradients can be expected during plastic forming of near net shape components by PECS. An additional reason to assume the existence of even larger thermal gradients during PECS forming is the absence of a contact zone between certain parts of a partially deformed preform and its surrounding counterpart, which simultaneously acts as a die during PECS forming, at least in the early stages of the forming process. In the latter case a nonconductive part will only be heated by conductive heat transfer from the punch, thereby increasing the temperature gradient in the preform and leading to an additional shape distortion. Therefore, the goal of the present paper is to understand the influence of the electrical properties of the ceramic preforms on their deformation behaviour during plastic forming by PECS and to find a way to predict and influence the deformation mode by changing the current paths. This work combines experimental PECS compression tests on cylindrical preforms and FEM simulations that predict temperature distributions and their high-temperature deformation behaviour.

2. Experimental procedures

The starting materials were a commercial zirconia powder (TZ-3Y grade, Tosoh, Tokyo, Japan, $d_{50} = 20$–50 nm) and TiCN powder with a C:N ratio of 1 (HTNMC grade, Hebei Sinochem, PR China, $d_{50} = 50$ nm). For the production of ZrO_2–TiCN (60/40 vol.%) composite materials, the ZrO_2 and TiCN starting powders were bead milled (Dispermat SL, VMA Getzmann GmbH, Reichshof, Germany) in ethanol at 6000 rpm for 2 h using 3Y–ZrO_2 beads with an average size of 0.4–0.7 mm. The ethanol was removed after mixing in a rotating evaporator. The dry powder mixture was sieved through a 325 mesh sieve, loaded in a graphite die with a diameter of 45 mm and densified at 1450 °C in a hot press (W100/150-2200-50 LAX, FCT Systeme, Rauenstein, Germany) under vacuum. The 45 mm diameter 3Y–TZP cylinder was hot pressed under the same conditions. Fully dense cylindrical parts were obtained and then used for further sample manufacturing. The cylinders were ground plan parallel down to a height of 24 mm and smaller 12 mm diameter cylinders were machined from them by electrical discharge machining for the composite material [19] and with a hollow diamond drill for the 3Y–ZrO_2 material.

Plastic deformation by compression of the 12 mm diameter cylinders has been performed in a FCT PECS machine (Type HP D 25/1, FCT Systeme). Technical details of the PECS device used can be found elsewhere [17]. Throughout