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Shrinkage characteristics studies on conventional sintered zirconia toughened alumina using computed tomography imaging technique

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ABSTRACT

Shrinkage is an important issue during sintering of ceramics as it affects the structural and mechanical properties. In the current work, the shrinkage characteristics of sintered zirconia toughened alumina (ZTA) compact produced by conventional powder forming process was investigated by using computed tomography imaging technique. The effect of process parameters such as weight percentage of zirconia added to toughened alumina, compaction pressure, and sintering temperature on ZTA was studied. The Box–Behnken technique in response surface methodology was used to develop the experimental design to analyze the shrinkage phenomena. The sintered samples were subjected to computed tomography scan and analyzed by using MIMICS Software wherein the three dimensional shrinkage of the ZTA composites was done. The mathematical regression model relating powder forming process parameters to the shrinkage was developed. It was observed that due to the effect of gravity, shrinkage is found to be 20% in the top for 1600 °C sintered sample. From the study shrinkage of the sintered ZTA was influenced by the sintering temperature. Being a non-contact type digital measurement method the odds of error created by instrument or human was avoided. Shrinkage of three directional complex shapes can also be identified effectively with the help of solid modeling packages.

1. Introduction

Zirconia toughened alumina composites are used for a variety of application such as implants [1], machining inserts [2,3], creep resistant materials [4], armor material in defense application. Ceramic composite fabricated by conventional sintering technique is an economical and simple method to form dense material. The powders are packed by compaction are bonded together during sintering. Fairly, a large amount of shrinkage is observed due to the removal of pores among powder particles in ceramic compacts. The reduction of the inter particle surfaces due to surface tension is the driving force of shrinkage [5]. It is caused due to the decrease in pore volume, porosity, and with increase in temperature [6]. It is essential to assess the shrinkage mechanism to predict the performance of sintering techniques. Prediction of the shrinkage characteristics of ceramic composites is important for the production of defect free near net shape final sintered product. The thermo-mechanical behavior of ceramics was found to be altered with the change in porosity as the interfaces between particles are consumed. Shrinkage in the compacts occurs anisotropically during sintering of the elongated shaped particles, while difference is observed for spherical shaped particles made with similar processing condition [7]. Shrinkage anisotropy is found to have a linear relationship with particle orientation for spherical and elongated particle of ceramic compact [8]. The α-alumina of particle sizes 0.25, 0.31 and 0.61 μm was subjected to sintering studies. By varying the temperature from 1050 °C to 1400 °C it exhibits a low linear shrinkage at 1050 °C as reported by Bisset et al. [9]. The shrinkage is also varied during sintering by the addition of additives in the composites. Particle size of the additive also affects the shrinkage behavior of composites [10]. Elevated sintering temperatures and prolonged soaking time contribute to higher shrinkage rate in ceramics [11].

2. Literature survey

Based on the complexity of profiles and required accuracy different measurement techniques can be employed to assess the shrinkage characteristics of composites. The influences of powder injection molding process parameters on shrinkage for Fe–Ni samples were found using a Vernier caliper and micrometer [12]. The shrinkage for alumina ceramics used for MEMS application was measured with a resolution of 1 μm using a charge coupled device microscope [13]. The linear shrinkage of ceramic composites found using dilatometer at different temperature was used to estimate density and activation energy [14–15]. Watanabe et al. studied the two-dimensional shrinkage of complex shaped structure formed by metal injection molding. This was done using a multi reference digital image in-situ and non-contact type shrinkage studies for up to 1370 °C using this method [16].A novel technique in dilatometer, which exploits white-light non-contact surface profilometry, is applied for measuring shrinkage rate in small cylindrical powder [17] with an z-resolution of less than

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10 nm. Maca et al. [18] used the data from dilatometric shrinkage to develop a plot for densification curves with an automatic procedure. The shrinkage studies were performed with a CCD camera and video frame grabber on WC–6% Co cylindrical specimen during sintering by Gasik and Zhang using the data obtained from optical dilatometer [19]. Cristofolini et al. used coordinate measuring machine (CMM) to evaluate the dimensional deviation and geometrical variation of steel part formed by powder metallurgy route [20]. The shrinkage analyses to examine the 3 dimensional shrinkage behavior of alumina during the sintering were made by digitizing them using a gray-code fringe projection and detection by a CCD camera [21]. A wide-range of work on axial and radial shrinkage behavior for ceramics and glass powder compacts reinforced with alumina-platelets during the sintering were carried out by Boccaccini et al. using hot stage microscopy (HSM) [22–24]. The shrinkage behavior of porcelain stoneware was obtained using HSM photos showed that the sample expanded at 1000 °C exhibits no significant shrinkage up to 1120 °C. The decrease in sample height started from 1120 °C and increased exponentially up to 1220 °C, at which the maximum shrinkage was observed [25]. Designs of experiments (DOE) methods were used to model the shrinkage of powder compacts by varying the parameters. Factorial design was used to study the shrinkage of powder injection molding with four process parameters such as the debinding method, sintering heating rate, hold temperature, and time [12]. Taguchi technique was chosen to study and optimize the shrinkage behavior of selective laser sintering (SLS). The empirical relationships between different SLS process parameters such as scan length, laser beam power, hatch spacing, laser beam speed, and part bed temperature to predict shrinkage was developed [26]. RSM was used to identify the influencing process parameters of the shrinkage for the ZTA ceramic composites. Although a lot of techniques were used to model the shrinkage behavior of ceramics, literature is still limited on computed tomography (CT) based methods for studying the three dimensional shrinkage. Hence, the present work has been envisaged to investigate the shrinkage of ZTA using CT imaging technique, which is a new approach.

3. Materials and method

3.1. Experimental design by response surface methodology (RSM)

RSM is a combination of mathematical and statistical procedures used to analyze the influence of multiple variables on a single or multiple responses [27]. Response surface methodology (RSM), is a design of experiment method used to model a phenomenon, evaluate the performance, analyze the behavior and optimize the influencing factors [28]. Design of experiment technique called Box–Behnken design (BBD) is selected for shrinkage study. BBD for three parameters such as composition, compaction pressure and sintering temperature, each of three levels with shrinkage as the response was studied. The BBD allows the usage of relatively least combinations of process parameters to determine the response function. In various experimental conditions, it is possible to express the independent process parameter involved into a quantitative form given in Eq. (1).

$$Y = \emptyset\,(x_1, x_2, \ldots, x_k)\, e_r \tag{1}$$

where, the response is Y and $x_1, x_2,\ldots, x_k$ of k quantitative factors and e_r measures the experimental errors, this function is called response surface function. The mathematical form of Ø is unknown; it can be approximately satisfactory within the experimental region by polynomial. The regression equation of second order polynomial was used to represent the response surface Y as given by Eq. (2).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_i x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ij} x_i x_j + \epsilon \tag{2}$$

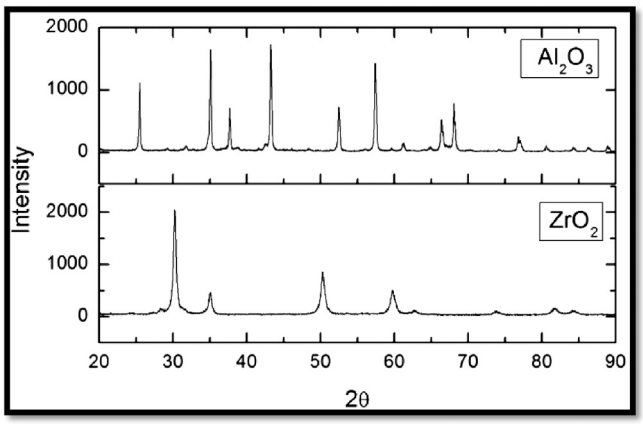

Fig. 1. XRD pattern of starting powders alumina and zirconia.

3.2. Experimental details

The commercial grade of alpha phase alumina (α-Al_2O_3) and cubic phase zirconia (ZrO_2) were used in the present work. The mean crystallite size and phases for alumina and zirconia identified from XRD results were 40 nm and 56 nm, respectively (Fig. 1). Powders of alumina with different weight ratio (5, 10 and 15%) of zirconia are blended together using Ball mill for 2 h with 10 min milling and 10 min stoppage with charge to powder ratio of 1:1. The blended powder is then poured into the 10 mm diameter circular cavity die and compacted. Cylindrical ZTA green compacts with diameter 10 mm and length 40 mm for 6 g of powder were obtained for various loads of 120, 140 and 160 MPa as shown in Fig. 2. The temperature at which the shrinkage initiates for alpha-alumina is at 1100 °C to 1150 °C [29], which are independent of the particle size and atmosphere. At 1600 °C, a maximum density is reported for ZTA composite [30]. Therefore, 1200 °C is selected as the initial temperature level and 1600 °C is selected as the final temperature level. The compacts are sintered at temperatures of (1200 °C, 1400 °C, and 1600 °C) at 5 °C raise in temperature per minute with a soaking period of 5 h in the box furnace. All the specimens were prepared according to the experimental runs developed by the DESIGN EXPERT 8. The controlling parameter set for running the design matrix is given in Table 1. Physical and mechanical characteristic of the ZTA composite fabricated by the same process is published in [31,28] respectively.

Fig. 2. Green compact of the specimen.