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Conceptual design and manufacture of spirally wound alumina tubes with improved thermal shock behavior for refractory applications

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ABSTRACT

Rotationally symmetric refractory nozzles have been fabricated using the spiral winding technique with ceramic alumina green tapes. Different layer designs have been developed in order to obtain a homogeneous fired structure with locally delaminated interfaces, which are expected to improve the thermal shock behavior. In this regard, the microstructure of all fired structures has been investigated. Thermal shock tests have been conducted to evaluate the impact of the layer design on the formation of micro cracks and other damage. Subsequently, the corrosion behavior of these nozzle compositions has been investigated.

1. Introduction

The ceramic multilayer technology is a well-established technology in the field of functional ceramics offering the possibility to create advanced structures by combining layers of different beneficial properties, which has been described by several authors, e.g. Hagymási et al. (2005), Roosen (2006), Partsch et al. (2011) and Gebhardt et al. (2013). Beneath functional applications, there is an increasing interest to use such multilayers for structural ceramics (Gurauskis et al. (2007)) in order to achieve high toughness (Ghosh et al., 2006)) or crack bifurcation (Oechsner et al. (1996)). Until now the refractory industry has not been particularly involved in multilayer activities, but recent research by Götschel et al. (2012) and Hein et al. (2012) demonstrates that multilayered ceramics could be a promising way to design advanced refractories. A roadmap for the refractories industry developed by Geigenmüller et al. (2014) predicts economic interests for such multilayer refractories. The formation of strong interfaces between adjacent layers (Bermejo and Danzer, 2010) offer the possibility to incorporate residual stresses (Green et al. (1999)), which are assumed to improve the thermal shock behavior and thermal cycling capability. Weak interfaces have been implemented in planar ceramic multilayer structures in order to obtain stress-free structures, which offer a higher toughness combined with a so-called "graceful fracture" due to crack deflection and interface delamination (Clegg et al., 1990; Wang et al., 2002).

Regarding the manufacture of rotationally symmetric structures, the principle of spiral winding of ceramic green tapes was adapted from the paper industry. Winding of ceramic green tapes is a less common technique; it was used to generate SiC multilayer tubes (Badini et al., 2002; Pavese et al., 2008), Ceramic Matrix Composite (CMC) components (Shi et al., 2014) or helix-shaped piezo actuators (Wagner et al., 2005). Wound structures can either exhibit weak (Jakobsen et al., 2014) or strong interfaces (Scheithauer et al., 2012). In contrast to parallel and vertical winding, the spiral winding technique offers the possibility of endless winding, which makes it interesting for mass production and industrial applications. Nature has also adapted the principle of weak interfaces in rotationally symmetric structures to improve the mechanical strength of siliceous sponges, as reported by Müller et al. (2007). It could be demonstrated before, that nozzles based on refractory oxides exhibiting partially delaminated interfaces show a promising thermal cycling capability (Jakobsen et al., 2014). Such nozzles can, e.g., be used in steel casting applications.

The present study picks up the observation by Jakobsen et al. (2014) that weak interfaces in multilayered structures contribute

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Table 1
Powder mixtures of the slurries used for tape casting and theoretical density of appropriate dried green tape.

Tape	Fine powder fraction/wt%	Coarse powder fraction/wt%	Theoretical density of dried green tape/g/cm^3
1	100	–	3.99
2	45	55	3.94
3	30	70	3.93
4	15	85	3.92

to good thermal shock behavior. The goal is to generate partially joined interfaces by controlled delamination and local contact points, where the wound tapes are held together. The cohesion at these local contact areas between adjacent layers is required to be able to assure mechanical handling. However, delaminations imply the risk of slag infiltration. It is known that slag infiltrates into pores with pore diameters higher than 20 mm. Corrosion can also be caused by direct and indirect dissolution of refractory components. Additionally, precipitation of new phases can occur, which may slow down the chemical corrosion of the bulk material (Lee and Zhang, 2004; Poirier et al., 2007). Therefore, the gap between delaminated layers should preferably be smaller than 20 μm.

In this study green tapes derived from fine- and coarse-grained Al_2O_3 powders will be used to generate defined defect structures in spirally wound tubes. Due to the different sintering activity of the powders used, the tapes differ in their sintering shrinkage and total porosity. The impact of the achieved structures on thermal shock and corrosion behavior will be investigated and discussed.

2. Experimental procedure

2.1. Tape casting

For all manufactured nozzle compositions, alumina green tapes were used which were based on fine- and coarse-grained alumina powders (CT 1200 SG and T60 -45 my, Almatis GmbH, Ludwigshafen, Germany). The median-diameters d_{50} of these powders were determined by laser diffractometry (Mastersizer 2000, Malvern Instruments Ltd, Malvern, UK). The specific surface area of the powders was measured via BET analysis (ASAP 2000 V2.O2, Micromeritics Instrument Corporation, Norcross, GA, USA) using nitrogen as analysis gas. The densities of the two powders, measured by He-pycnometry (AccuPyc II 1340 V1.07, Micromeritics Instrument Corporation, Norcross, GA, USA), were 3.99 g/cm^3 for the fine-grained and 3.91 g/cm^3 for the coarse-grain powder, respectively. Based on these data, the theoretical densities of the tapes were calculated (see Table 1).

The cast tapes were either composed of one single powder (monomodal tapes) or powder mixtures (bimodal tapes); the slurry compositions are listed in Table 1.

For preparation of the green tapes, the powders were deagglomerated in a solvent mixture (azeotropic mixture of ethanol and toluene) with the dispersant Hypermer (Croda, Edison, NJ, USA; 1.0 wt% of solids) and alumina milling balls in a tumbling mixer (Turbula, WAB AG, Basel, Switzerland) for 24 h. Then, binder (BUTVAR, Solutia, St. Louis, MO, USA) and plasticizer (Santicizer, Ferro, Cleveland, OH, USA) were added and homogenized for another 24 h. For all prepared slurry compositions, the binder to plasticizer ratio was 1:1. Before tape casting, the slurries were screened using a wire cloth with an aperture size of 244 μm to retain the milling balls and other large contaminations. The slurries were degassed for 20 min using underpressure of 230 mbar in a rotary evaporator under slow rotation. The tapes were cast on a tape casting machine of 4 m length with a fixed casting head with a blade gap of 1500 μm in order to achieve dried green tapes with a thickness of around 600 μm. It has been demonstrated before by Jakobsen et al. (2014) that this is an ideal green tape thickness for spiral winding applications. In order to limit thickness variations the hydrostatic pressure during casting was kept constant by means of a double chamber casting head. As a carrier tape, a thin (100 μm) PET tape was used. The casting speed was set to 1.8 m/min, resulting in a shear rate of 20 s^{-1}. After drying in ambient air atmosphere, square samples in the size of 27.5 × 27.5 mm^2 were cut out of the green tape for tape characterization by means of a hot knife (Groz-Beckert KG, Albstadt, Germany), which had a temperature of 60 °C. For spiral winding, the required strips with a length of 200 mm and a width between 10 and 30 mm were cut with the same device. The green density of the samples was determined via geometrical measurement.

The specimens were fired in air at 1700 °C with a dwell time of 5 h (HT 16/17, Nabertherm GmbH, Lilienthal, Germany). Debinding was conducted slowly with a heating rate of 1 K/min up to 500 °C, followed by a heating rate of 3 K/min to the peak temperature. Density and porosity of the fired samples were determined according to EN 623-2 (EN, 1993) via vacuum infiltration as well as Archimedes measurement. The fired samples were characterized concerning the x-, y- and z-shrinkage via geometrical measurement. To gain information about the temperature dependent shrinkage behavior, a small piece (~0.8 × 1.5 mm^2) of green tape was investigated by dilatometry (DIL 402C, Netzsch GmbH, Selb, Germany). The pore size of the bimodal tapes was determined via mercury intrusion porosimetry (Pascal 140, Thermo Electron Corporation, Milan Italy; and PO 2000, Carlo Erba Instruments, Milan, Italy).

3. Manufacture of nozzles

For characterization of the green tapes' bending radius, a simple method patented by Feilhauer (1988) was adapted. The middle of a green tape strip, 200 mm long and 10 mm wide, was inserted between the jaws of a vernier caliper. The jaws of the caliper were carefully closed causing the green tape to bend. The tape strips were bent in such a way that the upper side of the cast tape was put under tension. At a critical point of caliper displacement cracks appeared at the outside of the bent tape strip. This displacement of the caliper was divided by 2 to give the critical bending radius of the tape. This test was performed at room temperature and directly after heating the strip shortly up to 60 and 80 °C, respectively.

For spiral winding a Teflon mandrel with a diameter of 4 mm was used, which was additionally covered with a conventional polymer coated paper sheet of 40 μm thickness (Parafilm M, Bemis Flexible Packaging, Neenah, WI, USA) in order to facilitate the removal of the mandrel after winding. At one end of the mandrel winding of the first layer started using a tape strip of 10 mm width. The strip was wound tightly, so that the single coils were in contact but did not overlap. Due to the binder gradient in the green tapes (Roosen, 1999) the bottom side of the cast tape is more prone to crack formation than the upper side; therefore this side was always facing upwards during winding. After finishing the first layer, it was coated homogeneously with a thin layer of adhesive (copolymerized polyvinyl acetate) to enable joining with the next layer. This so-called Cold Low Pressure Lamination technique has been proved before as a suitable method to join ceramic green tapes by several authors, e.g. Piwonski and Roosen (1999) or Götschel et al. (2013). The next layer was rotated and wound in the opposite direction,