jmr&t
Journal of Materials Research and Technology
www.jmrt.com.br

Original article

Powder injection molding of a mullite–zirconia composite

Renee Martin[a], *Michael Vick*[b], *Matthew Kelly*[b], *Jupiter Palagi de Souza*[c], *Ravi Kumar Enneti*[d,*], *Sundar V. Atre*[a]

[a] *Oregon State University, Corvallis, OR, USA*
[b] *Naval Research Laboratory, Washington, DC, USA*
[c] *Universidade Federal Rio Grand do Sul, Porto Alegre, RS, Brazil*
[d] *Global Tungsten and Powders Corp, Towanda, PA, USA*

ARTICLE INFO

Article history:
Received 15 January 2013
Accepted 7 March 2013
Available online 29 July 2013

Keywords:
Mullite
Powder injection molding
Miniature gas turbine

ABSTRACT

Mullite–zirconia composites have been identified as a potential material for high-temperature structural applications due to high-temperature strength, thermal shock resistance, and low coefficient of thermal expansion. In the present study, the feasibility of using powder injection molding to fabricate parts from a mullite–zirconia composite was investigated. The properties of the developed feedstock were used to simulate and identify suitable molding conditions for fabricating parts like miniature turbine stators. A test coupon was successfully injection molded from the developed feedstock. The results from the study confirm the ability of powder injection molding to fabricate complex shapes made from mullite–zirconia composites.

1. Introduction

Mullite is a candidate for high-temperature structural applications due to high melting point, low thermal expansion coefficient, thermal shock resistance, and high-temperature strength and creep resistance [1–3]. However, the fracture toughness and low temperature properties of the material are poor [4]. Conversely, partially stabilized tetragonal ZrO_2 polycrystals exhibit high room temperature strength and fracture toughness but have inferior mechanical properties at elevated temperatures [4–6]. Thus, mullite–zirconia composites have the potential to address either of the shortcomings of the monolithic materials, depending on the selected composition [7].

Improved properties have been documented for mullite–zirconia composites over a range of compositions [4,7–12]. Even so, the feasibility of using the materials is nevertheless hindered by processability. Machining is difficult due to the high hardness of the composites and so a method for fabrication of near-net shapes is needed [12]. Powder injection molding (PIM) is one such a method that has shown success with a variety of powder systems and geometries [13]. However, the PIM of mullite–zirconia composites has not been previously reported.

* *Corresponding author.*
E-mail addresses: ravi.enneti@globaltungsten.com, ravienneti@gmail.com (R.K. Enneti).
2238-7854 http://dx.doi.org/10.1016/j.jmrt.2013.03.014

The present paper investigates the feasibility of using the PIM process to fabricate components from a mullite–zirconia composite in the zirconia-rich region. Simulations are also carried out based on feedstock properties to identify suitable processing conditions for injection molding a stator for a miniature gas turbine.

2. Methods

Commercially available high-purity mullite and ceria-stabilized zirconia powders were used as starting materials. Both powders were used in as-received condition at a zirconia rich composition of ~57 vol.% zirconia and ~43 vol.% mullite. A multi-component binder system composed of polypropylene, paraffin wax, linear low-density polyethylene, and stearic acid was used in the present study. This binder blend was developed based on past binder systems that showed feasibility for PIM processing [14]. The feedstock was mixed using an Entek co-rotating 27 mm twin-screw extruder with an L/D ratio of 40. A powder loading of approximately 84 wt.% was used.

Thermogravimetric analysis (TGA) of the mixed feedstock was carried out using a Q500 (TA Instruments). TGA experiments were carried out in nitrogen atmosphere at a heating rate of 20 °C/min for 50–600 °C temperature range. Analysis was also performed in air for the same temperature range and heating rate. Differential scanning calorimetry (DSC) measurements were carried out in nitrogen atmosphere using a Q2000 (TA Instruments) calorimeter over a temperature range of 20–200 °C and at a rate of 20 °C/min. Viscosity measurements were measured in accordance with ASTM D3835 (2008) on a Goettfert Rheograph 2003 capillary rheometer. Specific heat was determined on a Perkin Elmer DSC7 calorimeter. Testing was based on ASTM E1269 (2005) and was carried out in a nitrogen atmosphere at 20 °C/min. Thermal conductivity was measured on a K-System II system in accordance with ASTM D5930 (2001). Pressure-specific volume-temperature (PVT) measurements were made using high-pressure dilatometry on a Gnomix PVT dilatometer.

Test coupons were molded on an Arburg 221M molding machine. The green parts were debound and sintered in air at 1500 °C for 4 h. Hardness measurements were made in accordance with ASTM C1327 and fracture toughness was measured using Vickers indentation [15,16]. Mold filling simulations were performed using Autodesk Moldflow 2010 software.

3. Results and discussion

TGA was used to confirm the powder-to-binder-ratio after mixing. Fig. 1 shows the results of the TGA in nitrogen (top) and air (bottom). Both results show that the final composition of the mixture is close to 84 wt.% solids content. The results also confirmed the feasibility of a multi-step debinding process with lower molecular weight components burning off first followed by the higher molecular components [16]. Furthermore, TGA experiments helped identify the temperatures at which degradation of polymers start and finish. This information will be used in selecting limits for injection molding process

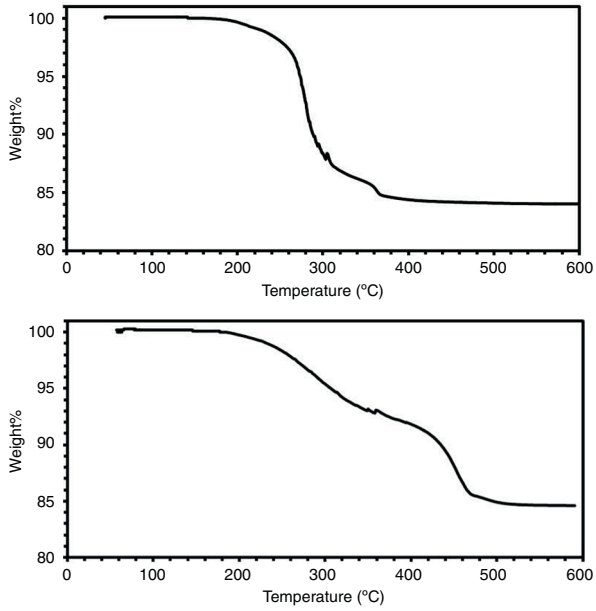

Fig. 1 – Thermogravimetric analysis of mullite–zirconia feedstock in nitrogen (top) and in air (bottom).

and also in designing debinding cycles for removing the polymers. In air, the majority of the binder components primarily degrade below 300 °C, with a peak rate at ~280 °C. However, in nitrogen, two distinct steps are seen at around ~280 °C and ~450 °C, that are attributed to the low and high molecular weight constituents in the binder mixture [16]. The differences in the results between nitrogen and air carry important implications for the debinding process, as degradation of the organics is markedly slower in nitrogen than it is in air.

The differential scanning calorimetery (DSC) results for the feedstock is shown in Fig. 2. The melting temperature of the feedstock can be identified from the plot. The plot shows a melting of lower molecular weight components at ~60 °C and the higher molecular weight components at ~140 °C. The melting point information was used in selecting temperature during injection molding process.

Fig. 3 shows the rheological results for the mullite–zirconia feedstock. Viscosity was not strongly affected by temperature presumably due to the high solids content. However, a strong correlation between viscosity and shear-rate was

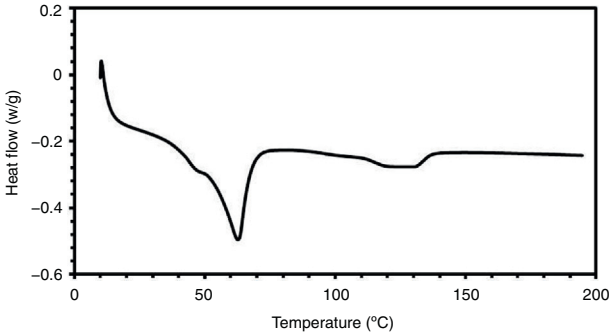

Fig. 2 – Differential scanning calorimetry of mullite–zirconia feedstock.