Ceramics International xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Carbon-bonded alumina foam filters produced by centrifugation: A route towards improved homogeneity

B. Luchini[a,*], J. Hubálková[a], T. Wetzig[a], J. Grabenhorst[a], J. Fruhstorfer[a], V.C. Pandolfelli[b], C.G. Aneziris[a]

[a] TU Bergakademie Freiberg, Institute of Ceramic, Glass and Construction Materials, Freiberg, Germany
[b] Federal University of São Carlos, Department of Materials Engineering, Materials Microstructure Engineering Group (GEMM), São Carlos, Brazil

ARTICLE INFO

Keywords:
Carbon-bonded alumina
Ceramic filters
Computer tomography
Centrifugation
Refractory

ABSTRACT

An alternative route for producing carbon-bonded alumina filters was analyzed and compared with the state of the art, both based on the replica method. The latter comprises a dip coating and subsequent rolling and spraying steps ($R+S$). The proposed route is based on dip coating and centrifugation (*CF-T1*). Aiming to investigate the influence of an additional thermal treatment on the ceramic coating homogeneity, a modified centrifugation route was analyzed (*CF-T2*). Computer tomography (CT) analyses provided a targeted evaluation of the spatial material distribution after each processing step. It was found that log-normal functions fitted well the wall thickness values attained for all investigated filters. Qualitative surface evaluation was conducted using a digital (optical) microscope. The $R+S$ and *CF-T1* filters exhibited surface macro-cracks and hollow struts, due to the release of the polyurethane foam template during firing. The *CF-T2* presented partly filled struts with a high amount of micro-cracks on the surface, being a potential alternative to increase the filtration efficiency, as the filter surface area was raised. However, in preliminary mechanical tests, the surface micro-cracks seemed to considerably reduce the cold crushing strength of the *CF-T2* filters. Based on the CT data, it was possible to conclude that the centrifuged filters presented a more homogeneous material distribution, as a consequence of higher automation level of the process.

1. Introduction

Ceramic filters are increasingly being used by foundry industries in the last few decades [1,2]. Their main tasks are firstly, filtering endogenous inclusions from the molten metal as due to the inclusions geometry and thermo-mechanical properties mismatch with the metal alloy, they act as stress-concentrators, limiting the materials elongation, ultimate strength and fatigue life [3–6]. Secondly, reducing the turbulence of the melt flow. Under laminar flow, the risks of metal re-oxidation and cavity formation are diminished [7].

Recently, carbon-bonded alumina (Al_2O_3-C) filters are gaining significant importance, mainly motivated by their excellent thermal-shock and creep resistance [7,8]. Moreover, improvements on filtering efficiency were reported for Al_2O_3-C filters associated with special coatings [9–11].

In general, carbon-bonded alumina filters are produced via the replica technique [12]. This method basically uses a polymeric foam as a skeleton for the ceramic impregnation. After coating the foam, the interstage filter is thermally treated to burn out the polymer and sinter the ceramic material, resulting in a ceramic filter with a replicated geometry of the original polymeric foam. The ceramic coating is a crucial step for the final mechanical properties of the filter, as a homogeneously distributed material is required.

The common technology for producing Al_2O_3-C filters comprises two coating steps [7,9,13]. The first one is based on a dip coating of the polyurethane (PU) sponge and subsequent squeezing and rolling stages to empty the void cells, reducing the excess of slurry and homogenizing the material distribution. Afterwards, a drying stage is required before the spray coating can be carried out. Then, the coated sponges are fired up to 800 °C in a reducing atmosphere. More details about this technology can be found at Schmidt et al. [13].

Voigt et al. [14] investigated geometrical features and mechanical properties of plain alumina filters coated by two distinct techniques: spraying and centrifugation. They concluded that the coating procedure has a large influence on the filter homogeneity, directly affecting its compression strength. Low viscous slurries are preferred for centrifugion, whereas slurries with a higher solid content lead to better results for spraying.

* Corresponding author.
E-mail address: bruno.luchini@ikgb.tu-freiberg.de (B. Luchini).

https://doi.org/10.1016/j.ceramint.2018.04.228
Received 4 April 2018; Received in revised form 25 April 2018; Accepted 26 April 2018

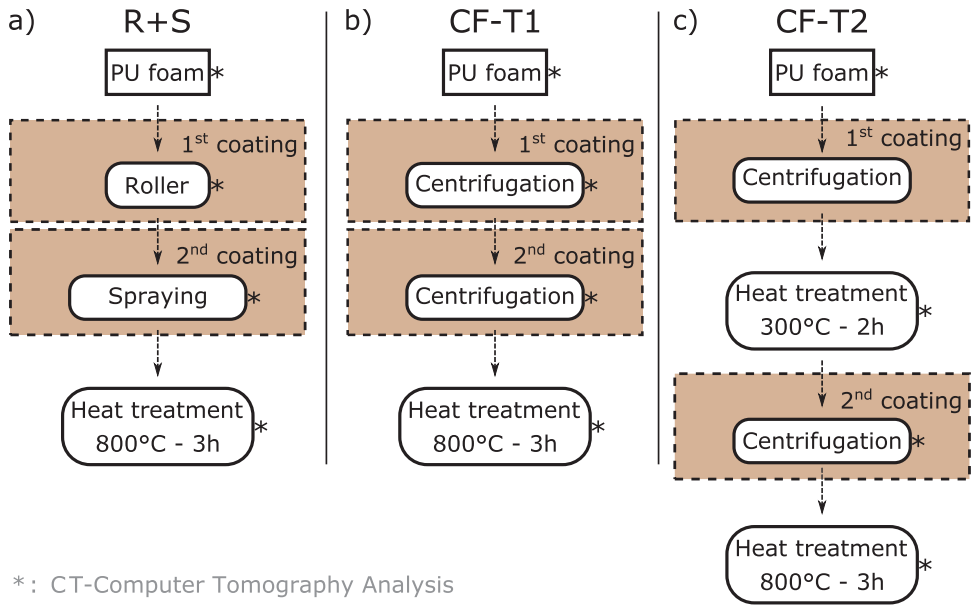

Fig. 1. Three distinct strategies for sample production.

No reports were found in the literature about $Al_2O_3 - C$ filters produced by centrifugation. The present article aims to geometrically characterize the filters produced by this technique. $Al_2O_3 - C$ filters produced by rolling and spraying (as state of the art) were used as reference.

2. Materials and methods

2.1. Sample preparation

As already mentioned in the Introduction, two distinct techniques were used to produce the filters: *(i)* Rolling and Spraying ($R+S$) and *(ii)* Centrifugation (*CF*). Both methods are based on the replica procedure [12]. The $R+S$ technique was widely explored and described in the literature for $Al_2O_3 - C$ filters production [7,9,13], whilst *CF* was applied for plain Al_2O_3 filters [14,15]. In the present study, the filters produced by $R+S$ followed the procedure by Schmidt et al. [13] (Fig. 1a).

Conversely the spraying procedure that acts for the material deposition, the centrifugation and roller steps withdraw it. The first *CF* coating consists of dipping the polymeric foam in the slurry (81.6 wt% solid content) (Table 1), hand squeezing and placing it in the centrifuging cage. The centrifugation step eliminates the excess of slurry from the foam, emptying the void cells. The second *CF* coating is similar to the first one, except that it was used a thinner slurry (65 wt% solid content) and no squeeze step was applied. Because the sponge was already coated, squeezing would damage the ceramic structure.

Table 1
Composition of the investigated samples.

Raw materials	wt. [%]
Al_2O_3 Martoxid MR 70	66
Carbores® P	20
Graphite AF 96/97	8
Carbon black MT N-991	6
Additives[a]	
Lignin sulfonate T11B	1.5
Castament VP 95 L	0.3
Constraspum K 1012	0.1
Total Solid content[b]	
1st coating (Rolling)	81.6
2nd coating (Spraying)	70.0
1st coating (Centrifugation)	81.6
2nd coating (Centrifugation)	65.0

[a] related to the sum of raw materials.
[b] without additives.

For centrifuging the specimens, the equipment RZR2102 Control (Heidolph, Germany) was used. For each coating, the filters were centrifuged for 20 s at a rotational speed of 850 rpm. A 24-h interval was taken in between the first and the second coating for drying the slurry at room temperature (Figs. 1b and 1c).

The raw materials for the filters production were chosen based on Emmel and Aneziris [7]. As alumina source, Martoxid MR 70, 99.8% Al_2O_3 (Martinswerk, Germany) with $d_{90} < 3.0\,\mu m$ was selected. The carbon sources were composed by Carbores P (Ruetgers, Germany) with $d_{90} < 0.2$ mm; graphite (Graphit Kropfmuehl, Germany) with $d_{90} < 30\,\mu m$ and carbon black (Lehmann & Voss, Germany) with a primary particle size of 200–500 nm. The additives were ligninsulfonate (Otto-Dille, Germany) as wetting agent and temporary binder, Castament VP 95 L (BASF, Germany) as dispersing agent and Contraspum K1012 (Zschimmer & Schwarz, Germany) as antifoam (Table 1).

One of the drawbacks associated with using the replica method for producing ceramic filters is the inherent presence of hollow struts due to the polymer burn out [7,16]. This effect directly affects the final mechanical properties of the filters, such as compression strength and friability [17,18]. Intending to reduce this disadvantage, an enhanced *CF* procedure was introduced (Samples *CF-T2*: Fig. 1c). In this case the samples were twice thermally treated. The first one was carried out right after the first coating. *CF-T2* samples were heated up to 300 ° C in air with 2 h dwell time at this temperature. The heating rate was 1 K/min. The purpose of this thermal treatment was to partially burn out the PU foam. As the Carbores® P possess the softening point at 235 ° C [19] it was expected that the cracks generated during the PU release could be healed. Therefore, the second coating could partly fill the hollow struts, increasing the filter rigidity.

After the second coating, the samples were thermally treated inside retorts filled with calcined petrol coke up to 800 ° C to avoid oxidation. The heating rate was 1 K/ min with 30 min dwell time every 100 K and a final 3 h dwell time at 800 ° C. The same thermal treatment was applied for the $R+S$ and *CF-T1* samples (Figs. 1a and 1b, respectively).