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Microstructure and mechanical properties of fine grained carbon-bonded Al_2O_3–C materials

Y. Klemm[a,*], H. Biermann[a], C.G. Aneziris[b]

[a]Technische Universität Bergakademie Freiberg, Institute of Materials Engineering, Gustav-Zeuner-Straße 5, 09599 Freiberg, Germany
[b]Technische Universität Bergakademie Freiberg, Institute of Ceramic, Glass and Construction Materials, Agricolastraße 17, 09599 Freiberg, Germany

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Abstract

Fine grained carbon-bonded Al_2O_3–C materials as used in ceramic filters have been manufactured by uniaxial and isostatic pressing, respectively. The variation in the microstructure over the cross section of the samples which in particular depends on the shaping technique plays an important role in the wetting of the material by liquid steel. Moreover, the amount and grain size of the binder has a decisive influence on the porosity and bulk density and therefore on the mechanical properties. For this, two different grain size distributions of Carbores® P binder were used, and in addition the fraction of binder was varied from 5–30 wt%. Tests of the cold crushing strength and of the cold modulus of rupture were performed at room temperature. The adjusted bulk density, open porosity and shrinkage of the samples were determined and the microstructure was analyzed by means of scanning electron microscopy. For control of a homogeneous distribution of carbon in the samples, the residual carbon content was measured also within individual samples at different positions.
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1. Introduction

For the production of clean high quality cast steel components ceramic filters are applied in the last decade in order to reduce non-metallic inclusions [1]. The filter materials have to fulfill different thermo mechanical requirements and especially to withstand the thermal shock attack in the beginning of the steel melt casting [2,4]. Carbon-bonded alumina materials provide an excellent thermo mechanical performance and are since several years used in functional components such as submerged entry nozzles, stoppers etc., whereby coarse grain size distributions up to 1 mm are used [3]. In carbon bonded filter materials due to the small strut thickness in the area of 300–400 μm only fine grains up to 30–40 μm are acceptable [6]. Therefore the main approach of this contribution is the manufacturing of bulk samples due to pressing with approximately the same pore size and grain size distribution in order to be able to characterize the mechanical properties of the fine grained Al_2O_3–C materials as used in filter applications. These mechanical properties will be used for the simulation of the filter behavior and the development of constitutive laws.

As binder as well as a carbon source Carbonaceous Resin (Rütgers Chemicals AG, Germany) with a lower amount of carcinogenic polycyclic aromatic hydrocarbons (300 ppm) than standard pitch (10.000 ppm) is used. The high residual graphitic like carbon amount derived from the Carbores® P binder has compared to phenolic resins the advantage of higher flexibility and high thermal conductivity combined with low Coefficient of Thermal Expansion (CTE) which result in an excellent thermal shock and oxidation resistance [5,7].

2. Materials and methods

2.1. Raw materials and processing

In Table 1 the compositions of the specimens used for the production of bulk carbon-bonded Al_2O_3–C are listed.

*Corresponding author. Tel.: +49 37 3139 3727.
E-mail address: Yvonne.Klemm@iwt.tu-freiberg.de (Y. Klemm).

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Table 1
Composition of the investigated samples [wt%].

	AC 5	AC 10	AC 15	AC 20	AC 30
Al_2O_3 martoxid® MR70	66	66	66	66	66
Carbores® P	5	10	15	20	30
Carbon black MT N–991	13	11	9	6	2
Grafite AF 96/97	16	13	10	8	2

Table 2
Particle size distribution of the raw materials [μm].

	d10	d50	d90
Al_2O_3 martoxid® MR70	0.2–0.4	0.5–0.8	1.5–3
Carbores® P coarse‘	–	80	400
Carbores® P fine‘	1.8	4.9	11.6
Carbon black MT N–991	0.2	1.1	9.3
Grafite AF 96/97	≤4	8.5–11	≤25

The materials have a content of 66 wt% fine alumina Martoxid® MR70 from Albemarle (Bergheim, Germany). As binder Carbores® P (CARBO naceous RESin) from Rütgers Chemicals (Castrop-Rauxel, Germany), which is a high melting coal-tar resin, with two different particle size distributions (cf. Table 2) was applied with contents of 5 to 30 wt%. Carbores® P softens at temperatures higher than 200 °C and has a highly oriented graphite-like carbon structure after coking [2]. The coke residue of Carbores® P is up to 85%.

Furthermore carbon black MT-Thermax N991 from Lehmann & Voss (Hamburg, Germany) and graphite AF 96/97 of Graphit Kropfmühl (Kropfmühl, Germany) were used with different fractions.

The raw materials were dry mixed in an Eirich mixer for about 5 min. Afterwards, granules were produced with the addition of water and 1% Glycerol. The formation of the granules depended on the fraction of the binder in the mixture. As the moisture content was too high for compression, the granules were afterwards dried in a drying oven at 60 °C down to a moisture content below 3%. Afterwards, shaping by uniaxial and isostatic pressing has been performed, respectively. Cylinders (diameter: 50 mm, height: 50 mm) and bending bars (7 mm × 7 mm × 70 mm) were pressed at 150 MPa at a lab press ES 270.00 of Rucks Maschinenbau GmbH, Germany.

The isostatic pressing was also carried out at a pressure of 150 MPa at a SO 5-8359-0 of EPSI NV., Belgium, with a holding time of 1 min at the maximal pressure. In order to avoid skin effects samples of 50 mm × 50 mm and 7 mm × 7 mm × 70 mm were cut out.

After shaping, the green samples were coked up to 800 °C at a heating rate of 1 K/min and a holding time of 30 min after each 100 °C step and of 3 h at 800 °C according to conventional filter materials, Fig. 1. The samples are embedded in a retort filled with calcined petcoke (Müco, Germany) and heated in a Nabertherm (Germany) LH 15/14 furnace. This leads to the formation of a coke network, the binding matrix, into which the alumina particles are integrated.

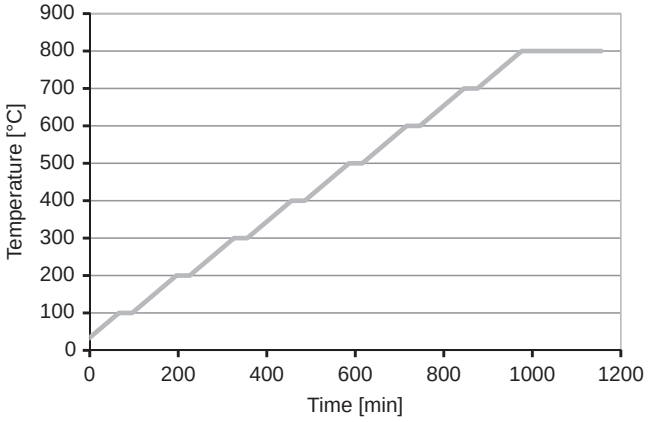

Fig. 1. Heating curve for thermal treatment.

2.2. Experimental techniques

The bulk density, the open porosity and pore size distribution were measured using a Pascal mercury porosimeter (Pascal 440, Thermo Fisher Scientific, Germany). To obtain the shrinkage, the width and length of the sample were measured before and after pyrolysis.

Due to changes in raw material contents, this may influence the quantitative amount of carbon in total and its distribution in the sample. As this will influence the mechanical properties, it is important to determine the residual carbon content. About 20 mg of material was fired under an oxidizing atmosphere in a carbon analyzer (RC 412, Leco, USA) under formation of CO_2. The firing curve from 20 °C to 1000 °C was measured with a heating rate of 62 K/min. The carbon measurement results from changes in mass of the sample. For an investigation of the distribution of carbon in the sample, material from the top surface and from the center area, respectively, was subject to the oxidation test, Fig. 2.

The microstructure of the materials was studied with a field emission scanning electron microscope (MIRA3, TESCAN, Brno, Czech Republic). Specimens were prepared by cutting and mechanical polishing with a final diamond grade of 1 μm.

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