HIGH TEMPERATURE BEHAVIOR OF CARBON-BONDED FILTER STRUCTURES

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ABSTRACT

Filter structures of fine-grained, carbon-bonded alumina (Al_2O_3-C) are used to reduce non-metallic inclusions in metals. To improve understanding of their behavior, these filters were investigated regarding their mechanical properties at temperatures up to 1500°C. Therefore, quasi-static tests were carried out in compression. The tests were complemented by investigations of microstructure by scanning electron microscopy. The experiments showed brittle behavior of the material at low and intermediate temperatures. The filter specimens showed fracture of single struts, leading to drops of stress in stress-strain curves. At temperatures above 1400 °C, plastic deformation of the filter occurred, leading to smooth stress-strain curves. The filters showed a maximum of strength at approximately 1400°C.

INTRODUCTION

The application of ceramic foam filters for filtration of metal melts in casting processes is common technique in today steel casting to ensure clean steels with improved strength[1]. This is necessary since small inclusions act as crack initiators during mechanical loading, especially at cyclic loads[2]. Therefore, extensive efforts were made to improve properties of filters.

In Collaborative Research Centre (CRC) 920 filter materials are developed and investigated regarding their mechanical, chemical and physical properties[3]. In these investigations, carbon-bonded materials have shown promising results at both compact specimens[4,5] and foam specimens[3]. Nevertheless, investigations of high-temperature behaviour of carbon-bonded foams are not reported yet. However, Bruneton et al.[6] reported results of compression tests on pure carbon foams from compression tests at temperatures up to 3100 °C. The results show a transition from brittle to ductile behaviour at temperatures above 2000 °C due to thermally activated visco-plastic deformation (creep). This change in mechanical behaviour led to a maximum of strength at about 2200 °C.

In the present study, the mechanical properties and the behaviour of fine-grained carbon-bonded alumina foams was investigated at high temperatures. The open-cell foams produced with the Schwartzwalder technique had a macro porosity of 10 ppi and a residual carbon content of about 30 %. The foams were tested in an electro-mechanical testing machine under inert atmosphere at temperatures up to 1500 °C in quasi-static compression tests. Additionally, microstructural investigations with scanning electron microscopy were carried out. The present results were already submitted to Carbon with a more detailed discussion[7].

EXPERIMENTAL DETAILS

Material

Open-cell foam specimens of fine-grained, carbon-bonded alumina were produced with the Schwartzwalder technique. Firstly, all solid and liquids parts (see Table 1) were mixed to a fine viscous impregnating slurry. Afterwards, preshaped polyurethane foams were dipped into the slurry and dried for 24 hours at 100 °C, followed by an additional coating with a less viscous spray slurry. After a further drying for 24 hours at 100 °C, the specimens were coked under reducing atmosphere in a coke bed at 1400 °C. Finally, all foams were analysed regarding their mass, height and diameter. Foams with abnormal values, foams with high amounts of closed cells and foams with too high or low strut thickness were sorted out. More detailed information about processing of foams were reported by Emmel et al.[3]

Table 1: Foams' raw materials: Solid components yield 100 %.

Part	Material	Product	Producer
66	Alumina	Martoxid® MR70	Albemarle
20	Bonding agent	Carbores P®	Rütgers Chemicals
6	Carbon black	Luvomaxx N991	Lehmann & Voss
8	Graphite	AF 96/97	Graphit Kropfmühl
0.3	Dispersant	Castament VP 95 L	BASF
0.8	Wetting agent	PPG P400	Sigma-Aldrich
0.1	Defoamer	Contraspum K 1012	Zschimmer & Schwarz
1.5	Wetting agent	Ammonium lingo-sulphonate T11B	Otto-Dille

Methods

An electromechanical testing machine (Z020, Zwick Roell, Neu-Ulm) equipped with an inert gas chamber (Maytec, Singen) was used for testing of the foams in compression at temperatures up to 1500 °C. Heating rates were up to 20 K/sec. The setup of the tests is shown in Fig. 1. The foam specimens (1) were placed within a susceptors cage (2) between two susceptors (3). Both susceptors and the cage were heated with an inductive heating device using a water-cooled copper coil (4) connected to a middle-frequency generator (HF 5010, TRUMPF Hüttinger, Freiburg). For testing, the lower Si_3N_4-piston (5) was driven upwards. The strain measurement was carried out with a high-temperature strain gage with alumina rods (6). Measurement of temperature was carried out contact-less with a pyrometer (Metis MS09, Sensortherm, Sulzbach). Therefore, the susceptor cage had an elongated hole. The emission coefficient for the material was 0.92. Prior to testing, the chamber was evacuated and filled with Argon twice. Strain rate during the tests was 10^{-4} s^{-1}. To ensure plane parallelism, both bases of the foams were polished.

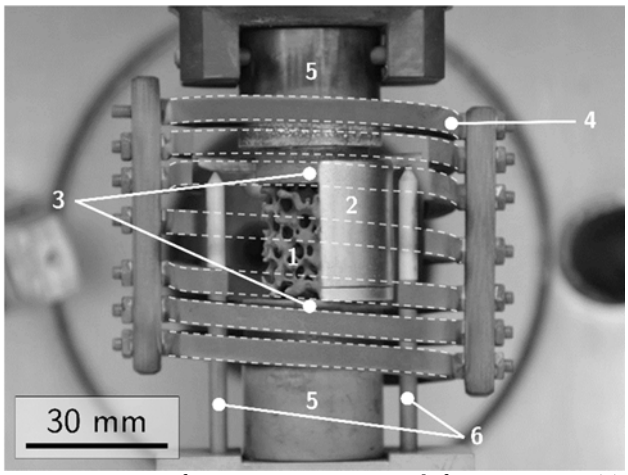

Fig. 1: Test setup for compression tests with foam specimen (1), susceptors cage (2), susceptors (3), copper coil (4), Si_3N_4-pistons (5) and alumina rods for strain measurement (6).

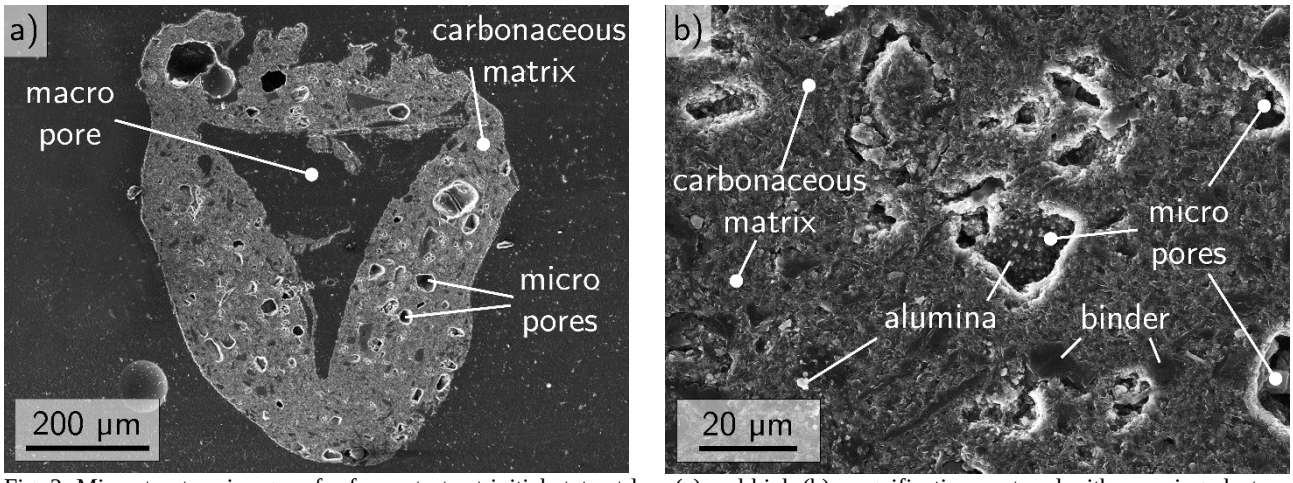

Fig. 2: Microstructure images of a foam struts at initial state at low (a) and high (b) magnification captured with scanning electron microscopy in secondary electron mode.

In addition to the mechanical tests, the material was characterised regarding its microstructure with scanning electron microscopy (Mira 3 XMU, Tescan, Brno).

RESULTS

Microstructure

Fig. 2 shows the microstructure of a foam struts' cross section observed with SEM. In a) a complete strut with its macro pore in the centre is shown. This pore was formed during coking due to volatilisation of the initial polyurethane foam. In the picture the pore is filled with the embedding compound. Additionally high amounts of micro pores are distributed within the struts. At higher magnification (b) residual amounts of unsolved binder and the fine-grained alumina are visible.

Mechanical Tests

Fig. 3 shows the force-displacement curves of specimens tested at temperatures at and below 1200 °C (a) and at and above 1400 °C (b). At lower temperatures the curves showed numerous drops in force (arrows). At higher temperatures, fewer drops of force occurred and the course of the curves were steadier.

This change in course of the force-displacement curves of the foams can be explained with a change in mechanical behavior of the material. High-temperature investigations on a comparable material showed thermally activated deformation (creep) at temperatures above 1050 °C[5]. However, at 1050 °C creep rates were low and thus, did not affect the behavior in the present case.

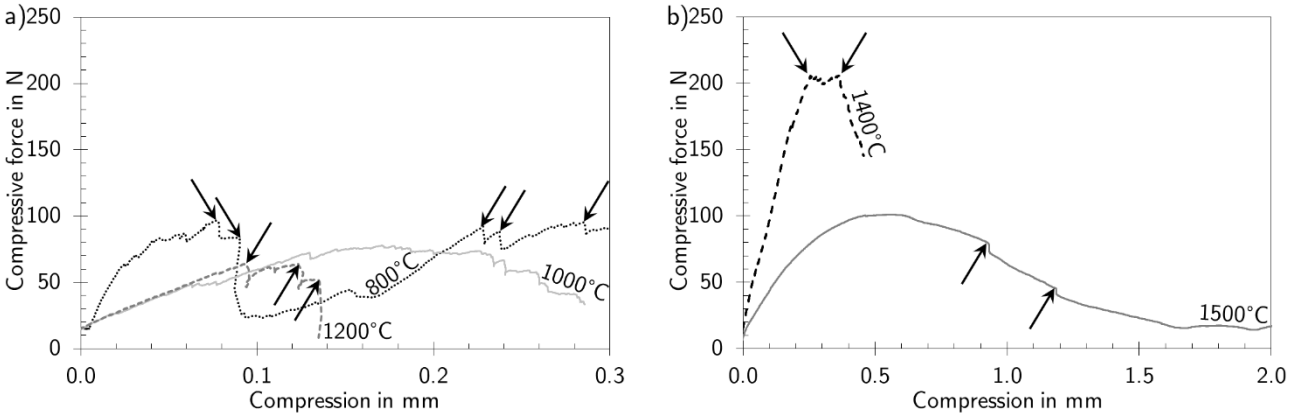

Fig. 3: Force-displacement curves of foams at temperatures below (a) and above 1300 °C (b). Arrows indicate drops in force at which single struts fail[7].

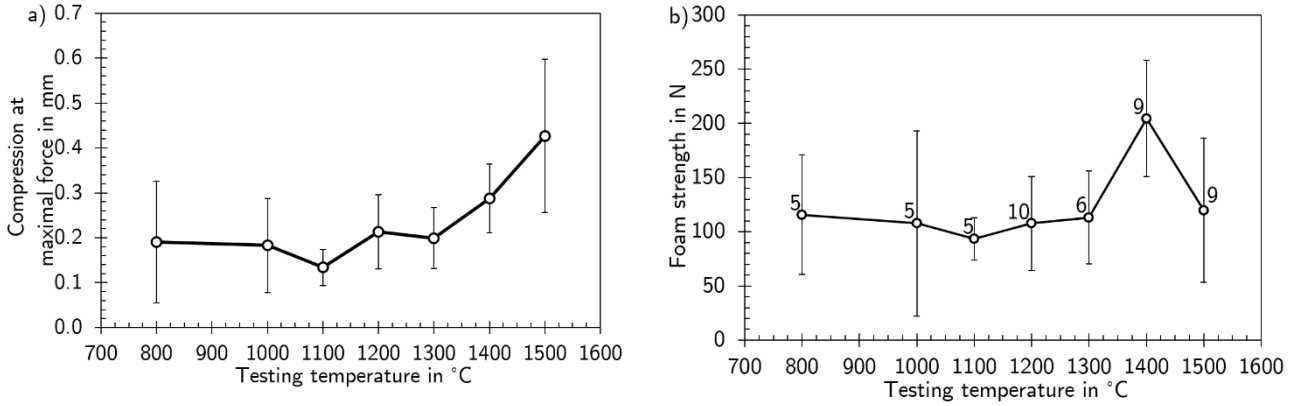

Fig. 4: Evolution of strain at maximal force (a) and foam strength (b) versus temperature. The numbers indicate the amount of tested specimens for each temperature[7].

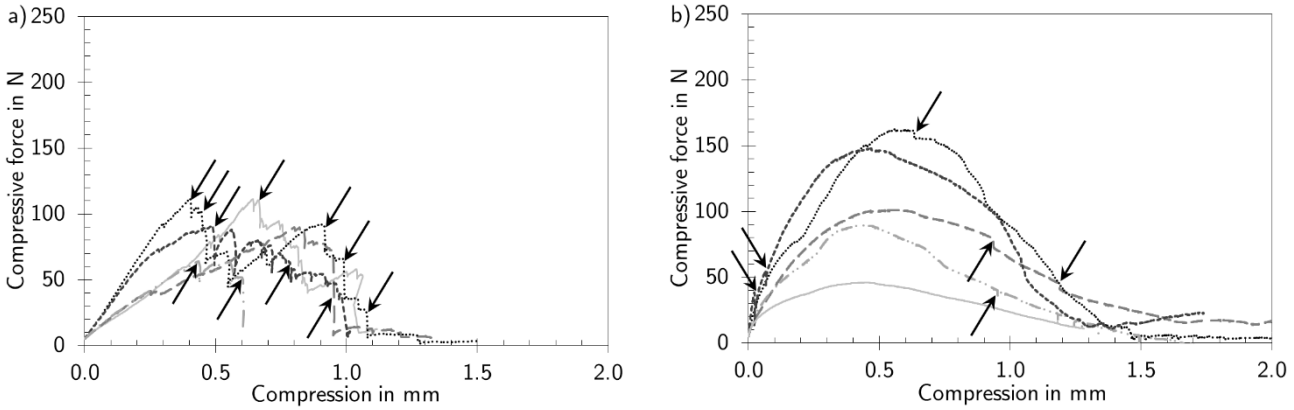

Fig. 5: Force-displacement curves of foams at 1100 °C (a) and 1500 °C (b). Arrows indicate drops in force at which single struts within the foam fail[7].

With increasing temperature, creep rate and amount of creep deformation increase[5]. At approximately 1400 °C, creep rates of the material are high enough to affect compression tests with the given strain rate of 10^{-4} sec^{-1}. This led to an increase in foam deformation, see Fig. 4a. In the figure, the average of the deformation at the maximal force is plotted versus test temperature. Whereas no change between 800 °C and 1300 °C occurred, compression increased above 1300 °C.

This change in mechanical behavior from brittle to more ductile led to an increase of foam strength from 1300 °C to 1400 °C. At low temperatures the foams behave brittle and show no plastic deformation. Compressive loading of the cylindrical foams lead to torques within the intersection nodes and bending stresses within the struts. When the strength of a single strut is reached it fails leading to a release of stress and loading of neighbor struts. Due to that, stresses within the struts are highly inhomogeneous. At higher temperatures, a transition to more ductile behavior occurs and struts show deformation under mechanical loading. This leads to loading of neighbor struts and a more homogeneous distribution of the stress within the foam and thus, an increase foam strength. With further increasing temperature, strength reduces significantly at 1500 °C due to enhanced softening of the foams. Thus, a maximum of strength is recorded at 1400 °C.

Comparable observations with creep deformation of carbon-bonded materials at high temperatures were reported by other authors before[8]. Additionally, increasing strength at high temperatures were reported for compact specimens of carbon-bonded materials[9] and graphitic materials[10] as well. Bruneton et al.[6] have shown, that such a thermally activated deformation can lead to an increase in foam strength with a maximum before softening reduces strength. It was not reported, whether this maximum in strength was related to material properties or if it took place due to the foam geometric structure of the specimens.

Scatter of the tests

For ceramic materials, scatter always has to be addressed. Therefore, several tests have been carried out at each temperature. The curves of five tests are illustrated in Fig. 5 at 1100 °C (a) and 1500 °C (b). The curves show high scatter. Nevertheless, the general courses of the curves are comparable in both cases: jerked at lower temperatures and steady at higher temperatures. The high scatter of the tests can be explained by the small specimens' size.

Ex situ characterisation

After testing in compression at 1500 °C, selected specimens were tested again at room temperature to investigate whether the change of mechanical behavior is thermally activated or caused by microstructural changes. The tests on pre-deformed foams did not show any differences compared to tests on initial specimens. Thus, ductile behavior of the foams was a result of thermally activated deformation and did not occur due to microstructural changes within the materials.

Selected foams were investigated with scanning electron microscopy after testing. Typical results are shown in Fig. 6 for foams tested at RT (a) and 1500 °C (b, c). At temperatures below 1300 °C, numerous broken struts were found. Their fracture surfaces showed no plastic deformation, which underlines the brittle character of the foams at these temperatures. In contrast, stopped cracks were found at foams tested at 1500 °C. Fig. 6b shows a foam specimen with a crack on one strut in the lower part. At higher magnification (Fig. 6c), a high degree of deformation was observed at the crack. In contrast to the fracture surface of the foam tested at room temperature, where instable crack propagation took place, this is a clear proof for stable crack propagation, taking place at high temperatures in carbon-bonded refractories.

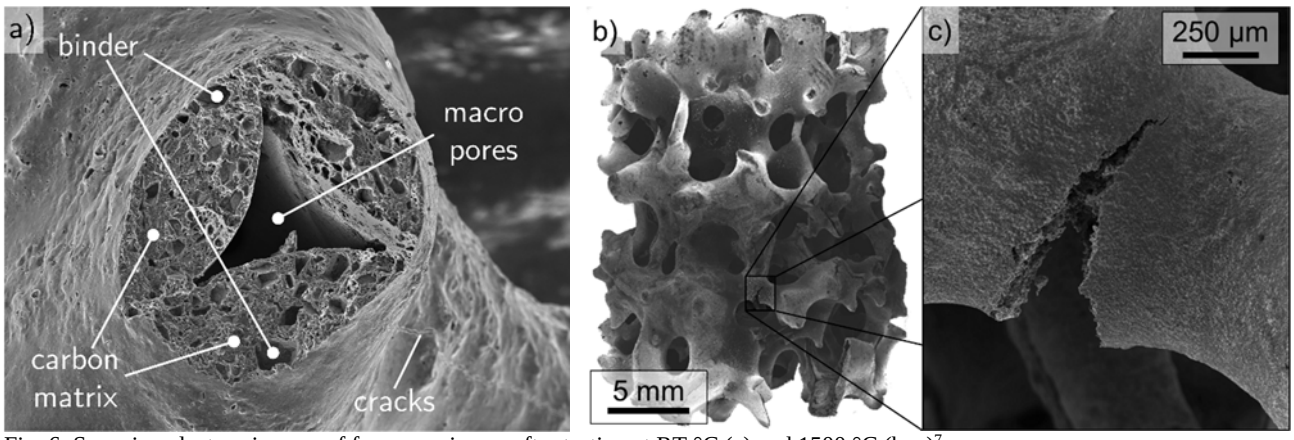

Fig. 6: Scanning electron images of foam specimens after testing at RT °C (a) and 1500 °C (b, c)[7].

CONCLUSIONS

The mechanical behavior of fine-grained, carbon-bonded alumina foams was investigated at temperatures up to 1500 °C under protective atmosphere. The material showed a change in mechanical behavior from brittle to more ductile above 1300 °C. Due to the thermal activation of visco-plastic deformation, a maximum of strength was recorded at 1400 °C. At 1500 °C softening of the material led to decrease in strength. These conclusions drawn from the change in force-displacement curves were supported by microstructural investigations. At low temperature, fracture surfaces showed no plastic deformation, whereas stable crack propagation and high deformations around cracks were observed at specimens tested at 1500 °C.

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