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Determining the temperature dependent fracture toughness of carbon-bonded alumina using chevron-notched specimens

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Abstract: Open cell ceramic foam filters are used for metal melt filtration. The aim is to reduce the number of non-metallic inclusions and therefore to enhance the quality of the cast product. A new generation of multifunctional filters, which is investigated within the collaborative research center CRC920, is made of fine grained carbon-bonded alumina. The filter manufacturing process leads to hollow struts with sharp edge cavities, which could act as a crack tip. To evaluate the integrity of the filter, a fracture mechanical characterization of the bulk material at different temperatures is necessary. In the applied chevron-notched beam method (CNB) the small specimens (5 x 6 x 25 mm³) are loaded with a four-point bending test set-up until failure occurs. The test set-up offers the possibility of testing at temperatures up to 1000°C. With the help of the measured load-displacement curve the fracture toughness is calculated. Additionally, an analysis of the microstructure of the fracture surface of the specimens is presented.

Keywords: chevron-notched beam method; carbon-bonded alumina; fracture toughness

1. Introduction

The presence of non-metallic inclusions is an important question for the quality of cast products. A low number of these inclusions increase the properties of the cast product. This is the reason, why open cell ceramic filters are utilized during metal melt filtration. Furthermore, a reduction of turbulences in the melt flow, which also leads to an enhancement of the quality of the cast product, is achieved. An essential requirement for industrial application is the integrity of the filter with respect to the applied loading by the metal melt during the casting process. The filter has to withstand the mechanical loading at elevated temperatures without any failure to avoid additional impurities of the cast product.

Within the scope of the collaborative research center CRC 920 "Multi-Functional Filters for Metal Melt Filtration – A Contribution towards Zero Defect Materials" at TU Bergakademie Freiberg, Germany, an new generation of filters are made of fine grained carbon-bonded alumina [2]. The filter manufacturing process is based on the Schwartzwalder replica technique [3]. This technique uses polyurethane foams which define the topology and the geometry of the filter. The ceramic slurry is produced and applied to the polyurethane foam as a coating. After a drying process the foam is heat treated. The polyurethane pyrolyzes during the heat treatment. The result of the manufacturing process is a filter structure with hollow struts. The hollow struts have sharp-edged cavities which can act as crack tips. Consequently, a fracture mechanical characterization is required.

A standardized method to evaluate fracture toughness of ceramics [4, 5] is the chevron-notched beam test (CNB), which is the applied testing technique in this study. Experimental bending test set-ups with specimens possessing a chevron notch have been introduced and standardized since the 1960's [6–8]. The advantage of this test set-up is that no sharp pre-crack has to be introduced at the beginning of the test [1] because a sharp crack is formed during loading. Additionally, no crack length measurement is required and a stable crack growth can be reached due to the geometry of the notch [9] because of the increasing crack front with increasing crack length. The challenge in the present work is to qualify the CNB test for high temperature testing at 800°C and to use a non-standard specimen geometry. Therefore, a special test set-up is developed to handle the specimen under high temperature environment. The set-up and the function of each component is explained and described.

2. Materials and Methods

2.1. Carbon-bonded alumina

One of the filter materials is a fine grained carbon-bonded alumina Al₂O₃-C, usually applied to steel filtration. It consists of different raw materials which are 99.8%-pure alumina (Martoxid MR 70, Martinswerk, Germany, d_{90}

$\leq 3.0 \mu\text{m}$) and three different carbon sources. The carbon sources are named modified coal tar pitch powder (Carbores[®] P, Rütgers, Germany, $d_{90} \leq 0.2 \text{ mm}$), fine natural graphite (AF 96/97, Graphit Kopfmühl, Germany, 96.7wt% carbon, 99.8wt% $\leq 40 \mu\text{m}$), and carbon black powder (Luvomaxx N-911, Lehmann & Voss & Co., Germany, $\geq 99.0 \text{ wt.}\%$ carbon, $> 0.01 \text{ wt.}\%$ ash content, primary particle size of 200 – 500 nm). The mass fraction of the residual carbon content is about 30%. The chemical composition of the slurry with a total solid mass fraction of 60% can be found in Table 1.

Table 1. Chemical composition of Al₂O₃-C slurry.

Raw material	Mass fraction in %
Martoxid MR 70	66.0
Carbores [®] P	20.0
AF 96/97	7.7
Luvomaxx N-991 2	6.3
Additives ^a	
Castament VP 95 L	0.3
Contraspum K 1012	0.1
C12C	1.5

^a Related to total solid content

To produce the slurry, the powdery raw materials are mixed together with deionized water. Afterwards, the slurry is used to manufacture the filter or the specimens. The next steps are a mixing and a drying process followed by a heat treatment. This coking regime runs through a stepwise heating up to 800°C and is described in detail by Emmel et al. [10]. Carbores[®] P is acting as the binding phase during the heat treatment. Hence, the alumina is bonded in a carbon matrix. Approximately 80% of the mass fraction of the Carbores[®] P remains inside the material, the other 20% are volatile organic components. The porosity of the material is about 40%.

The slurry of the material is slip cast into rectangular blocks (25 x 25 x 150 mm³), dried and heat treated according to their specific heating regime. Specimens with final dimensions are cut from these blocks after the heat treatment. Finally, the triangularly shaped notch is cut into the specimens made of carbon-bonded alumina using a precision diamond wire saw (Well, Mannheim, Germany) with a wire diameter $d = 0.3 \text{ mm}$ (Well, Type A3-3. The final dimensions of the specimens amount to length $L = 25 \text{ mm}$, width $W = 6 \text{ mm}$, and thickness $B = 5 \text{ mm}$.

2.2. Chevron-notched beam method

In order to determine the fracture toughness K_{Ic} of the brittle filter bulk material, the chevron-notched beam method is applied. Different configurations of chevron-notched specimens are available in literature, for example the short bar [8, 11], short rod [12, 13], and four-point-bending specimens [9]. The triangular shaped notch which is cut into the specimen offers the following advantages: Firstly, no pre-crack is necessary because a sharp crack develops from the tip of the notch during loading. Secondly, no crack length measurement is required because only the maximum load is necessary to calculate the fracture toughness of the specimen after testing. For further investigations, the notch parameters crack length a , chevron tip dimension a_0 and chevron dimension a_1 , see Fig. 1b, are normalized with respect to the specimen width W leading to $\alpha = a/W$, $\alpha_0 = a_0/W$ and $\alpha_1 = a_1/W$, respectively.

In the present study, a four-point-bending set-up is applied to the chevron-notched specimen which differs slightly from the geometry of the standardized method [4, 5]. Figure 1 shows the geometrical parameters and the developed test set-up [14]. One roller of loading and supporting span are replaced by a ball, which is in contrast to DIN-Norm application at which two roller pairs are used. The balls in test set-up are used to fulfill the DIN-requirements of the rotatable support and to offer the possibility of high temperature testing. Both rollers (m) and balls (k) have diameters of $D = 5 \text{ mm}$. A cage structure is assembled to the lower sealing of the testing machine (i). This cage structure was constructed in order to ensure the alignment of the specimen (k) by specimen positioning bolts (g) as well as the loading and bearing components. The cage structure is guided by a housing (d) and consists of three rings, namely lower (h), middle (f) and upper cage ring (c). The upper and lower spacers and guiding rods, which are not visible in Figure 1a, provide the correct distance between the cage rings. The load from the upper punch (a) is transferred to the inner ball-cylinder pair via loading plate (e) and loading ball (b).

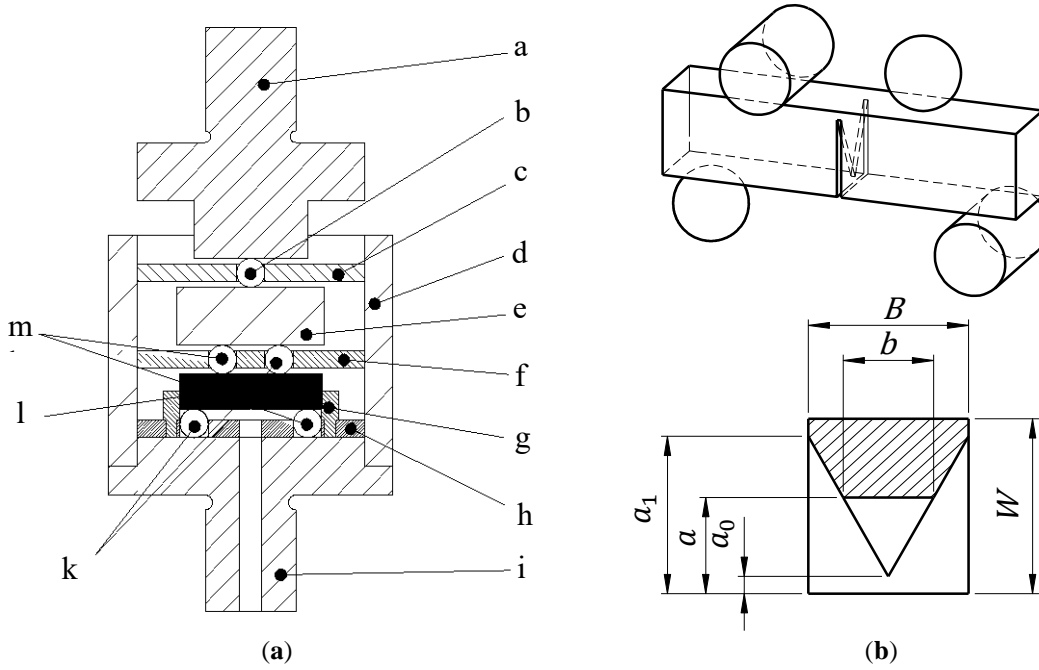


Figure 1. CNB test set-up: (a) Schematic of constructed set-up, (b) Details of specimen and geometrical parameters (S_1 – supporting span, S_2 – loading span, b – length of crack front).

The loading of the chevron-notched specimen is displacement controlled, with a constant displacement rate of $\dot{u} = 0.01$ mm/min until the specimen fails. The test set-up is assembled into a universal testing machine (inspekt table 10, Hegewald & Peschke, Nossen, Germany) that enables the measurement of a load-displacement curve with a 1 kN load cell. A very stiff testing machine is required in order to get valid test results. The experiments are performed at room temperature and 800°C under inert gas atmosphere with argon. For the elevated temperature tests, a three zone furnace (STE-13, Hegewald & Peschke) is used. The crack initiates at the tip of the notch and while propagating the compliance of the specimen increases. Additionally, the crack front b and the necessary energy for crack extension Δa increase continuously during crack growth. This results in a non-linearity at the load-displacement curve. The minimum of the geometry function of the specimen Y_{\min}^* and the maximum of the load-displacement curve coincides with each other. With the help of the maximum load F_{\max} the fracture toughness is calculated. A test is not valid, if the load-displacement curve exhibits only a sudden decrease of the load without a smooth maximum. Hence, no crack arrest is reached and the crack propagates unstably. In that case, no fracture toughness can be calculated. Some other restrictions due to the symmetry condition of the notch can also lead to an invalid test [4].

The fracture toughness is finally calculated with

$$K_{Ic} = \frac{F_{\max}}{B\sqrt{W}} Y_{\min}^*, \quad (1)$$

at which F_{\max} is the maximum measured load and Y_{\min}^* the minimum of the geometry function of the specimen. Munz et al. [1] provides an empirical formula based on compliance measurements and FEM simulations for the minimum of the geometry function:

$$Y_{\min}^* = (3.08 + 5.00\alpha + 8.33\alpha^2) \left(1 + 0.007 \sqrt{\frac{S_1 S_2}{W^2}} \frac{\alpha_1 - \alpha_0}{\alpha - \alpha_0} \frac{S_1 - S_2}{W} \right). \quad (2)$$

The validity area for this formula is given by $0.12 \leq \alpha_0 \leq 0.24$ and $0.9 \leq \alpha_1 \leq 1$.

3. Results and Discussion

The fracture toughness of the filter material carbon-bonded alumina is calculated with Eq. (1) and Eq. (2) and the measured maximum load of the CNB tests. The experiments were performed at room temperature and 800°C. A number of 15 specimens were tested at each temperature, but not every specimen shows a valid load-displacement

curve. Consequently, the number of valid experiments is 14 and 10 at room temperature and elevated temperature, respectively. Examples for measured load-displacement curves of valid tests for each temperature are plotted in Figure 2.

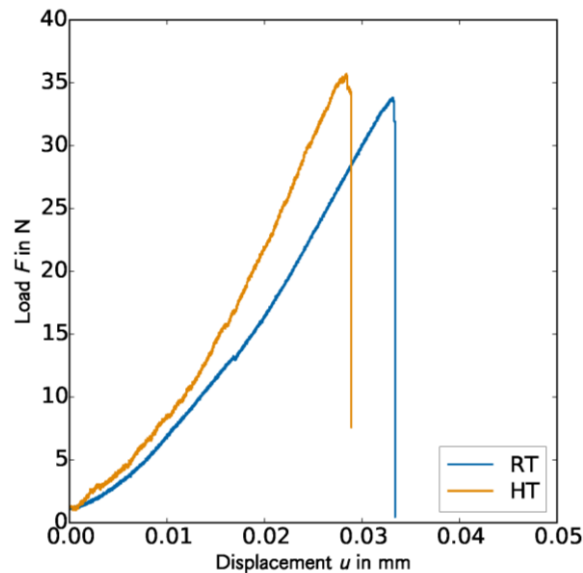


Figure 2. Measured load-displacement of CNB test at room temperature (RT) and 800°C (HT).

The curves start with a running-in characteristic followed by a linear section and a maximum until the specimen fails and a sudden load drop occurs. The load drop starts close to the load maximum because of the low fracture toughness of the material. Nevertheless, a small section of stable crack growth can be obtained. The difference between the curves at both temperatures can be explained by the change of Young’s modules with respect to the temperature [15, 16]. The results of all valid tested specimens and the geometry parameters of the notch are shown Table 2. The analysis of the results points out that the testing temperature shows no significant influence on the fracture toughness of carbon-bonded alumina.

Table 2. Temperature dependent fracture toughness of carbon-bonded alumina.

Testing Temperature in °C	F_{max} in N	W in mm	B in mm	α_0	α_1	K_{Ic} in $MPa\sqrt{m}$
25	31,42	5,93	4,89	0,20	0,90	0,552
	30,76	6,03	4,93	0,22	0,90	0,543
	33,82	6,07	5,01	0,20	0,90	0,561
	35,83	6,04	4,94	0,18	0,89	0,577
	26,76	6,03	4,98	0,29	0,96	0,576
	29,64	5,87	4,91	0,27	0,97	0,660
	32,82	6,07	5,05	0,22	0,93	0,579
	31,76	5,92	4,98	0,21	0,94	0,589
	28,97	5,86	4,88	0,27	0,97	0,648
	29,19	6,05	4,99	0,22	0,93	0,526
	29,92	6,02	4,92	0,25	0,95	0,598
	27,53	6,01	4,83	0,25	0,96	0,565
	27,08	5,96	4,90	0,24	0,94	0,531
	32,98	6,12	4,99	0,21	0,92	0,562
800	38,05	5,99	4,85	0,16	0,86	0,587
	31,65	5,93	4,80	0,18	0,87	0,529

Testing Temperature in °C	F_{max} in N	W in mm	B in mm	α_0	α_1	K_{Ic} in $MPa\sqrt{m}$
41,45	6,07	4,83	0,17	0,83	0,611	
34,77	5,80	4,95	0,16	0,87	0,567	
42,79	5,99	4,89	0,11	0,81	0,562	
33,32	5,94	4,80	0,18	0,83	0,527	
35,72	5,98	4,83	0,20	0,88	0,610	
41,45	6,07	4,94	0,15	0,86	0,602	
42,79	6,01	4,85	0,11	0,82	0,566	
43,31	6,00	4,75	0,13	0,80	0,594	

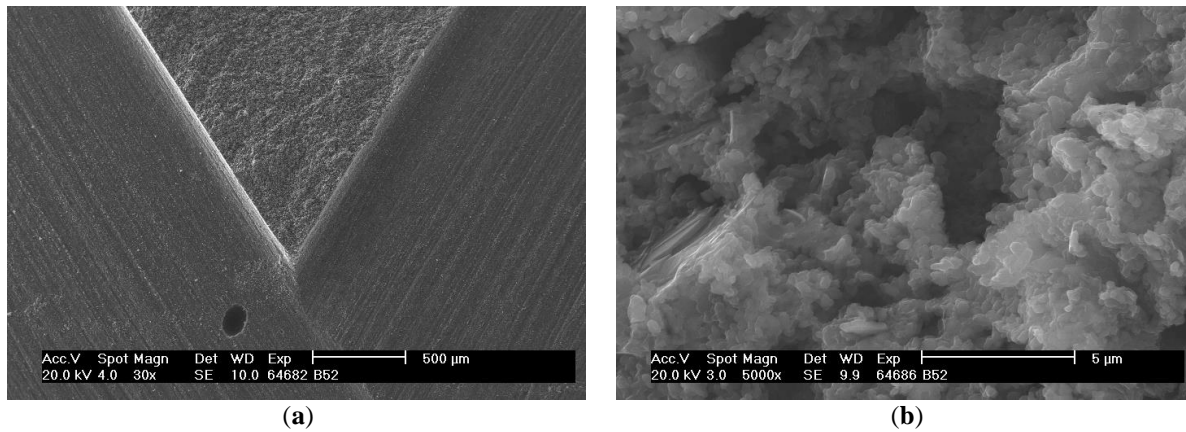


Figure 3. SEM-micrograph of the fracture surface of carbon-bonded alumina: (a) 30-times magnified; (b) 5000-times magnified.

Additionally, the crack surface topologies are analyzed for the carbon-bonded alumina. Figure 3 illustrates the microstructure of the crack surfaces obtained by a scanning electron microscope (SEM), Philips XL 30 (Phillips, Germany) with different magnifications. The fracture surfaces of the material at higher temperatures are equal to those at room temperature and therefore not shown in the present paper. Carbon-bonded alumina exhibits a homogeneous microstructure and a macroscopically flat fracture surface. No plastic deformations and a smooth fracture surface indicate a brittle fracture at both temperatures. Figure 3b shows the alumina particles that are embedded in the carbon matrix. An intergranular fracture can be obtained because of the visible grain boundaries at this magnification.

5. Conclusions

The chevron-notched beam test with a non-standardized specimen geometry was applied to a carbon-bonded alumina at different temperatures. The fracture toughness was determined using the empirical formula proposed by Munz et al. [1]. Carbon-bonded alumina shows a constant value up to the elevated testing temperature of 800°C. A fractographic analysis revealed smooth crack surfaces without plastic deformations and with visible grain boundaries as a result of an intergranular cleavage fracture.

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References

1. Munz, D.; Shannon, J. L.; Bubsey, R. T. Fracture Toughness Calculation From Maximum Load in Four Point Bend Tests of Chevron Notch Specimen. *International Journal of Fracture*, Vol. 16, pp. R137–R141, 1980.
2. Voigt, C.; Zienert, T.; Schubert, P.; Aneziris, C.G.; Hubálková, J. Reticulated Porous Foam Ceramics with Different Surface Chemistries. *Journal of the American Ceramic Society*, Vol. 97 (7), pp. 2046–2053, 2014.
3. Schwartzwalder, K. Method of making porous ceramic articles, Patent US 3,090,094, 1963.
4. DIN EN 14425-3 Advanced technical ceramics - Test methods for determination of fracture toughness of monolithic ceramics - Part 3: Chevron notched beam (CNB) method, 2010.
5. ASTM C-1421-01b Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature, 2001.
6. Nakayama, J. Direct Measurement of Fracture Energies of Brittle Heterogeneous Materials. *Journal of the American Ceramic Society*, Vol. 48 (11), pp. 583–587, 1965.
7. Tattersall H.G.; Tappin, G. The work of fracture and its measurement in metals, ceramics and other materials. *Journal of Materials Science*, Vol. 1 (3), pp. 296–301, Aug 1966.
8. Munz, D.; Bubsey, R.T.; Srawley, J.E. Compliance and stress intensity coefficients for short bar specimens with chevron notches. *International Journal of Fracture*, Vol. 16 (4), pp. 359–374, 1980.
9. Munz, D.; Bubsey, R. T.; Shannon, J. L. Fracture Toughness Determination of Al₂O₃ Using Four-Point-Bend Specimens with Straight-Through and Chevron Notches. *Journal of the American Ceramic Society*, Vol. 63 (5-6), pp. 300–305, 1980.
10. Emmel, M., Aneziris, C.G. Development of novel carbon bonded filter compositions for steel melt filtration. *Ceramics International*, Vol. 38 (6), pp. 5165–5173, 2012.
11. Barker, L.M. Short Bar Specimens for K_{Ic} Measurements. In *Fracture Mechanics Applied to Brittle Materials*. ASTM International, 1979.
12. Barker, L.M. A simplified method for measuring plane strain fracture toughness. *Engineering Fracture Mechanics*, Vol. 9 (2), pp. 361–369, 1977.
13. Munz, D. Determination of fracture toughness of high strength aluminum alloys with chevron notched short rod and short bar specimens. *Engineering Fracture Mechanics*, Vol. 15 (1), pp. 231 – 236, 1981.
14. Zielke, H.; Abendroth M.; Kuna, M. Fracture Toughness Characterization of Carbon Bonded Alumina Using Chevron Notched Specimens. *Key Engineering Materials*, Vol. 754, pp. 71-74, 2017
15. Werner, J.; Aneziris, C.G.; Dudezig, S. Young's Modulus of Elasticity of Carbon-Bonded Alumina Materials up to 1450°C. *Journal of the American Ceramic Society*, Vol. 96 (9), pp.2958-2965, 2013.
16. Werner, J.; Aneziris, C.G; Schafföner, S. Influence of porosity on young's modulus of carbon-bonded alumina from room temperature up to 1450°C. *Ceramics International*, Vol. 40 (9, Part A), pp. 14439-14445, 2014.