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Determining the fracture toughness of ceramic filter materials using the miniaturized chevron-notched beam method at high temperature

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ABSTRACT

The aim of the application of open cell ceramic foam filters during casting of metals is the reduction of non-metallic inclusions and turbulences in the melt flow. Hence, an improvement of the quality of the cast products is achieved. The integrity of the filter at mechanical loading under elevated temperatures requires a mechanical characterization of the bulk material of the filter. In particular, fracture toughnesses have to be determined for a new generation of filter materials. The presented work describes an experimental method to measure fracture toughnesses of the filter materials.

The mechanical testing is performed with the help of 4-point-bending tests using miniaturized chevron-notched specimens at different temperatures. Additionally, the geometry function of the test set-up is calculated and compared with an empirical formula by Munz [1]. At the end, the fracture toughness is determined at room temperature and 800°C. Further results characterize the influence of different geometrical parameters of the test set-ups on the maximum tensile stresses in the specimen and the load-displacement curves.

1. Introduction

The presence of non-metallic inclusions influences the quality of cast products. Reducing the number of these inclusions leads to an enhancement of the cast product properties. Therefore, open cell ceramic filters are utilized during metal melt filtration. Additionally, the ceramic filter reduces turbulences in the melt flow, which also increases the quality of the cast product. 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 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, filters are made of fine grained carbon-bonded alumina [2] and alumina [3]. The filter manufacturing process is based on the Schwartzwalder replica technique [4]. This technique uses polyurethane foams which define the topology and the geometry of the filter. A ceramic slurry is produced and applied to the polyurethane foam as a coating. After a drying process the foam is heat treated. During the heat treatment the polyurethanepyrolizes and the ceramic foam filter remains. 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. Therefore, a fracture mechanical characterization is required, preferably with small-sized specimens manufactured in the same way as the filters.

The applied testing technique is the chevron-notched beam test (CNB), which is a standardized method to evaluate fracture toughness of ceramics [5,6], also used for brittle metals like bearing steel [7,8]. Experimental bending test set-ups with specimens possessing a chevron notch have been introduced and standardized since the 1960's [9–11]. The advantage of this test set-up is that no sharp pre-crack has to be introduced because a sharp crack is formed during loading at the beginning of the test [1]. Furthermore, no crack length measurement is required and a stable crack growth can be reached due to the geometry of the notch [12]. The new 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. Since the assembly of the loading apparatus and the fixture of the specimen are difficult to handle under the high temperature environment of the furnace, a special set-up is developed. The influence of certain positioning errors and misalignments of bearings on the results is analyzed.

In addition to the experimental work numerical investigations are performed to simulate the CNB with the help of the finite element method. First, the geometry function for the stress intensity factors of the specimen is analyzed and its minimum is calculated with different approximations and compared with an empirical formula by Munz [1].

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Nomenclature

a	crack length
a_0	chevron tip dimension
a_1	chevron dimension
Δa	crack extension
b	length of crack front
B	thickness of specimen
c_0	clearance with zero contact pressure
$C(\alpha)$, $C(\xi)$	slice compliance
C_{Ch}	compliance of chevron-notched specimen
d	wire diameter
d_S	diameter of bearing and loading bodies
d_{50}, d_{90}	particle size distribution parameter
E	Young's modulus
F	load
F_{max}	maximum load
G_I	energy release rate
G_{Ic}	critical energy release rate
k	interlaminar shear factor for Bluhm's model
K_I	stress intensity factor
K_{Ic}	fracture toughness
L	length of specimen
n	number of slices for Bluhm's model
p_0	contact pressure at zero distance
ΔP	available energy for extension of crack by Δa
S_1	outer span
S_2	inner span
u	displacement
$\dot{u}$	displacement rate
W	width of specimen
ΔW	necessary energy for extension of crack by Δa
Y^*	geometry function
Y^*_{min}	minimum of geometry function
α	crack length normalized with W
α_0	chevron tip dimension normalized with W
α_1	chevron dimension normalized with W
γ	specific surface energy
θ	chevron notch angle
ν	Poisson ratio
σ_{max}	maximum tensile stress at notch tip
φ	rotation angle of loading bodies
CNB	chevron-notched beam method
CRC	collaborative research center
FEM	finite element method
FEM-C	finite element method approach using compliance calculation
SEM	scanning electron microscope
STCA	straight through crack assumption

Subsequently, a parameter study of the geometric influence factors of the experimental set-up on the maximum stress value and force-displacement curve is performed.

2. Materials

2.1. Carbon bonded alumina

One of the filter materials is a fine grained carbon-bonded alumina Al_2O_3-C, usually applied to steel filtration. It consists of different raw materials which are 99.8%-pure alumina (Martoxid MR 70, Martinswerk, Germany, $d_{90} \leq 3.0\,\mu m$) and three different carbon sources. The carbon sources are named modified coal tar pitch powder (Carbores® P, Rütgers, Germany, $d_{90} \leq 0.2$ mm), fine natural graphite (AF 96/97,Graphit Kopfmühl, Germany, 96.7 wt% carbon, 99.8 wt% $\leq 40\,\mu m$), and carbon black powder (Luvomaxx N-911, Lehmann & Voss & Co., Germany, $\geq$ 99.0 wt% carbon, >0.01 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.

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. After the mixing process follows a drying process step and a heat treatment. This coking regime runs through a stepwise heating up to 800°C and is described in detail by Emmel et al. [2]. 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 open porosity of the materials is about 40%.

2.2. Alumina

Another filter material is alumina which is used for the filtration of molten aluminium. The slurry consists of three different alumina powders provided by Almatis: tabular alumina T60/64 (0 – 0.045 mm), calcined alumina CT 9 FG ($d_{50} = 5.0\,\mu m$), and reactive alumina CT 3000 SG ($d_{50} = 0.5\,\mu m$). Additionally deionised water and processing additives are mixed into the slurry. Details of the mixing process are described by Voigt et al. [3]. The chemical composition of the slurry with a total solid mass fraction of 85.5% can be found in Table 2. A sintering process leading to the bonding of the particles is applied to the slurry up to 1600°C with a heating rate of 2 K/min and a holding time of one hour [3]. The open porosity of the materials is about 10%.

2.3. Specimen preparation

The slurry of both materials is slip cast into rectangular blocks

Table 1
Chemical composition of Al_2O_3-C slurry.

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

[a] Related to total solid content.

Table 2
Chemical composition of Al_2O_3 slurry.

Raw material	Mass fraction in %
Al_2O_3-CT 9 FG	33.3
Al_2O_3-CT 3000 SG	33.3
Al_2O_3-T60/T64	33.3
Additives[a]	
Axilat RH 50 MD	0.0
Dolapix CE 64	0.6
Optapix AC 170	1.0
PPG400	0.0

[a] Related to total solid content.