

Heat-resistant Silicon Carbide Refractories for Gasification Reactors

G. Standke, M. Herrmann, S. Kunze, H. Heymer, J. Adler, S. Höhn, G. Himpel

New high-efficiency gasification reactors with higher temperatures and pressures and with different temperature cycles than used previously require new types of refractory linings. Besides the oxide materials currently in use, SiC ceramics show great promise as refractories in the reducing atmospheres found in gasifiers. For this reason, recrystallised silicon carbide (RSiC) materials as well as RSiC materials infiltrated with either carbon or ZrO₂ were prepared and the corrosion resistance in an acidic ash was investigated at 1300 °C for 100 h in a flowing CO/H₂-containing Ar atmosphere. SEM, EDS and XRD analysis of the surfaces and cross sections of the corroded materials revealed the high stability of the modified RSiC materials. Two different mechanisms for corrosion protection were observed. Carbon-infiltrated SiC showed nearly no wettability and very little infiltration of the molten ashes into the RSiC. In contrast, a small amount of slag infiltration was observed for zirconia-infiltrated RSiC materials. At a high degree of pore filling, good protection was obtained through reaction of the ash with zirconia. In all cases no or only minor attack of the SiC skeleton was observed. The results demonstrate the potential of the optimised materials for refractory applications in different environments.

1 Introduction

Modern coal gasification processes are important issues for the production of raw materials for chemical industry and fuel using the fossil raw material coal and renewable biomass. Compared with typical combustion plants, high-efficiency gasification plants have higher operating temperatures, more aggressive gas atmospheres and higher requirements for thermal shock and corrosion resistance of the ceramic materials used for the reactor lining. Typical working conditions are temperatures of 1300 °C with peaks up to 1600 °C, pressures of 25–40 bar and typical gas compositions of 55–60 % CO, 30–35 % H₂, 10–15 % CO₂, 10 % H₂O and 0,4–1,5 % H₂S [1–3]. This requires materials with sophisticated combinations of properties, which can hardly be fulfilled by the traditional refractory ceramic materials because of the complex combination of stresses due to temperature, gas pressure, temperature fluctuations, abrasion by the solid fuel, the oxidising and reducing synthesis gas components and especially chemical reactions with ash and slag, which exhibit a wide range of compositions resulting from the variations in sources used [4–7]. Thus, dam-

age phenomena such as pullout of grains by weakening of the binder phase [1] or spalling [2] or insufficient creep or thermal shock resistance are described in the literature. Furthermore, the gas and ash compositions determine the corrosion process and differ depending on the mixture of coal and biomass used. For this reason, new material concepts for refractories, which meet the thermal and corrosion resistance demands and can be adapted to the varying conditions found in the different types of reactors, e.g. liquid slag gasifiers and fixed bed reactors, with different constructions and working principles are being sought. Silicon carbide materials represent one possibility because of their high stability at high temperatures, thermal shock resistance resulting from their low thermal expansion, high strength and high corrosion resistance [8]. SiC materials bonded by a separate phase, e.g. silicate- or nitride-bonded SiC, are less suitable because of the lower corrosion resistance of the binder phase [9]. However, intrinsically bonded SiC without any additional phases in the microstructure also exists. Pure sintered silicon carbide (SSiC) and recrystallised silicon carbide (RSiC) are two such types, which are of inter-

est for the present application. SSiC is nearly dense and exhibits closed porosity only and RSiC is porous and has a high content of open pores. With a high availability due to its use in kiln furniture and the possibility of forming 3D parts from it at reasonable costs, RSiC is more favourable in terms of manufacturing costs. For this reason, RSiC was chosen for the high-temperature corrosion resistance investigations. Various amounts of ash infiltration reaching full infiltration in the worst case were observed depending on the type of ash that was in contact with the ma-

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Tab. 1 Properties of the commercial RSiC materials

Name	Density [g/cm ³]	Open Porosity [%]	Median Pore Size [μm]	Strength [MPa]
HalSiC ¹⁾	2,69	16	24	90
Crystar 2000 ²⁾	2,71	15	26	89
IKTS-1 ³⁾	2,27	29	43	64
IKTS-2 ³⁾	2,29	28	25	52
IKTS-3 ³⁾	2,14	33	44	51

¹⁾ Haldenwanger; ²⁾ Saint Gobain; ³⁾ IKTS

terial [10]. Under the conditions of the corrosion experiment with basic, acidic or mixed ashes at 1300 °C and a dwell time of 100 h in a flowing CO/H₂-containing Ar atmosphere, no substantial corrosion or degradation of the SiC microstructure was observed. However, ash infiltration into the pores and elements such as Ca, Mg and Fe in the ash will have a deleterious effect on the long-time durability of the RSiC. For this reason a new concept in which two different types of corrosion protection materials (CPM) were infiltrated into the pores of the RSiC was developed. Zirconia and glassy carbon were the materials chosen. The infiltrated materials were tested under identical conditions to those described previously for pure RSiC and were compared with the results obtained previously for pure RSiC.

2 Methods and materials

2.1 Characterisation and preparation of RSiC materials

For the investigation two commercial RSiC materials produced by *Haldenwanger Ltd.* and *Saint Gobain Ltd.*, respectively, were

used. The parts had dimensions of 400 mm × 400 mm × 8 mm and showed a microstructure with an asymmetric pore size distribution with coarse pores in the middle of the plates and finer ones on the surfaces (Fig. 1). As shown in Tab. 1, both materials had nearly the same property profile, irrespective of the manufacturer. Additionally RSiC materials were prepared with tailored pore sizes (IKTS-1–IKTS-3), with a bimodal silicon carbide powder mixture of 2 μm and 105 μm powder being chosen. The powders were mixed with dispersants, various amounts of binders and pressing additives in water-based slurries. The material was then dried, granulated and pressed into green bodies of dimensions 60 mm × 50 mm × 8 mm. Sintering was performed in argon at temperatures of up to 2400 °C. The properties of the materials are summarised in Tab. 1. The microstructure of IKTS-1 is shown in Fig. 2. In comparison with the commercial materials, the new RSiC had coarser pores, a larger amount of open porosity and a more homogeneous pore size distribution (which was necessary for the infiltration). Additionally, the processing conditions could be

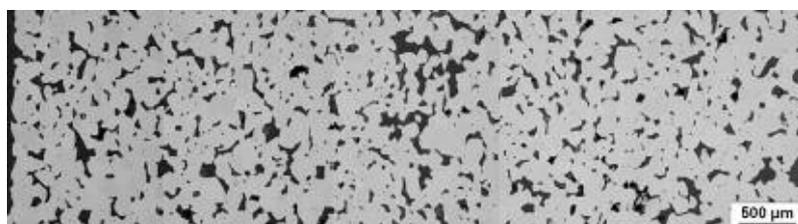


Fig. 1 FESEM micrograph of the cross section of a commercial RSiC Crystar 2000

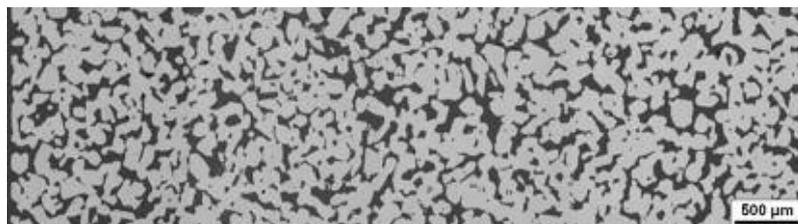


Fig. 2 FESEM micrograph of the cross section of the homogeneous RSiC sample IKTS-1

adapted to yield identical pore sizes with different amounts of porosity. The strength was lower than that of the commercial materials, but at 50–64 MPa was slightly higher than that of typical refractory materials [11].

2.2 Infiltration process

The aim of the work was to improve the corrosion resistance of RSiC by infiltration with corrosion protection materials based on zirconia and carbon. Two different routes were elaborated for the different infiltration materials.

For zirconia, the typical slurry route could be used by the preparation of a water-based pseudoplastic mixture of zirconia powder (*TOSOH TZ-8YS*, median particle size 0,43 μm) and a polyvinyl alcohol binder in a ball mill. At a solids content of 55 mass-%, the slurry was successfully infiltrated into the RSiC under vacuum in 15 min. Samples of dimensions 10 mm × 10 mm × 8 mm and 45 mm × 37 mm × 8 mm were oxidised at 600 °C for 1 h, then dried at 120 °C and sintered in air up to 1300 °C for 1 h. To increase the amount of zirconia and decrease the amount of pores in the samples, the process was carried out a second time. The weight increased by different amounts for the different types of RSiC, as shown in Fig. 3a. The commercial RSiCs with nearly the same porosities and pore sizes exhibited the same amount of infiltrated zirconia, which at 1,5 % was less than half of the amount that infiltrated into the IKTS materials. As expected, more ZrO₂ infiltrated into the IKTS-1–IKTS-3 materials, with the infiltrated amount depending on the pore size and porosity. IKTS-3 with a median pore size of 44 μm and open porosity of 33 % had the highest free volume and could store the highest amount of zirconia. Repeating the process yielded infiltration of only approximately 75 % of the amount infiltrated in the first infiltration, but because the overall amount of infiltrated zirconia was significantly increased through this second step it was carried out for all samples used in the corrosion experiments.

Fig. 3a also shows the volume fraction of the infiltrated pores determined by image analysis of SEM micrographs of the materials. Despite the strong differences in the infiltrated mass, nearly the same fraction of pores was filled in all materials. Fig. 4a shows a detailed view of the infiltrated

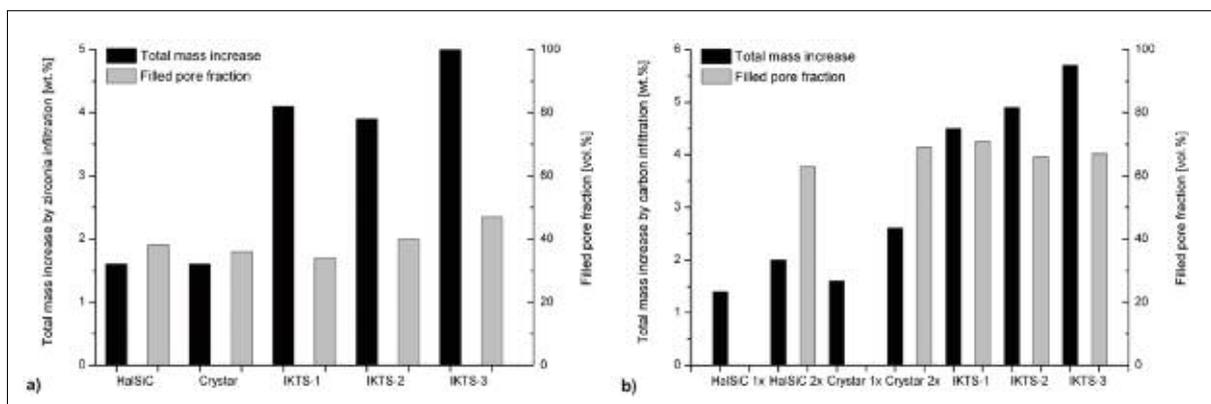


Fig. 3a–b Mass increase and fraction of filled pores for a) zirconia-infiltrated RSiC materials and b) carbon-infiltrated materials

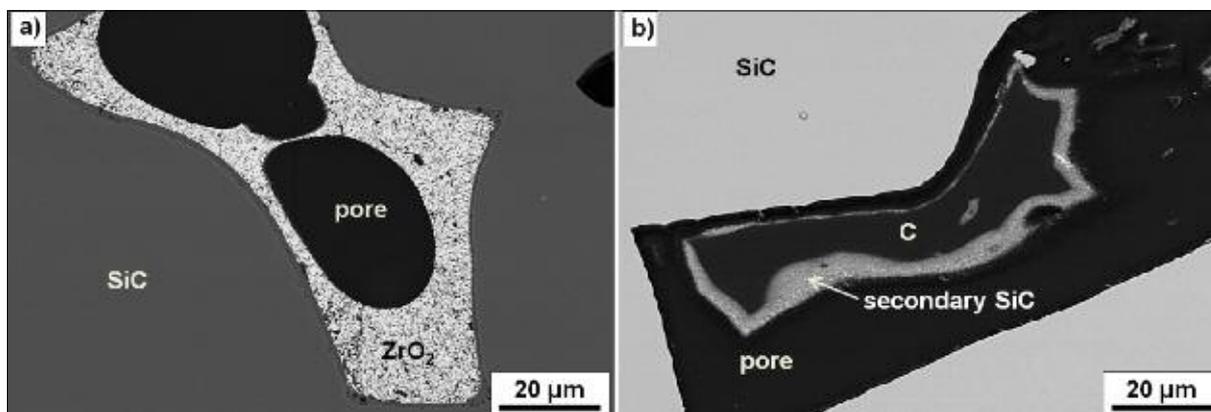


Fig. 4a–b Microstructures of RSiC-materials infiltrated with a) zirconia and b) carbon

pores, Fig. 5 an overview of the entire sample. The zirconia powder used was microporous upon sintering (Fig. 4a) and was mainly found coating the surfaces of the RSiC pores. Typically, not all pores were completely filled; some larger pores remained unfilled. The porosity was caused by shrinkage of zirconia during heat treatment, the relatively low solids content of the slurry, resulting in zirconia with a porous microstructure, and difficult-to-access pore areas with very small open channels which were quickly blocked. This led to inhomogeneously filled areas for all tested RSiCs, as illustrated in Fig. 5 where the pores next to the surfaces (1–2 mm depth) were mainly filled and those in the centre of the sample were partially unfilled.

The results were nearly identical for both tested geometries, with the larger parts showing slightly higher infiltration rates. A phenolic resin (novolac dissolved in furfuryl alcohol) with a viscosity of 300 mPa·s (at 20 °C) and a high carbon yield was used for infiltration of carbon into the pores of the

RSiC. RSiC parts with the same geometries and oxidation treatments as those used for zirconia infiltration were used. However, the resin and ceramic parts were preheated to 60 °C to achieve a high infiltration rate by decreasing the resin viscosity. The parts were infiltrated under vacuum with a processing time of 90 min. The samples were then hardened at 180 °C for 20 h and pyrolysed in an argon atmosphere at 1600 °C for 1 h. In the case of zirconia infiltration, the process was carried out twice to increase the carbon yield (only for commercial materials). In this case, a second pyrolysis step was carried out at a temperature of just 800 °C. The results of

these trials are shown in Fig. 3b. The mass increases of both infiltration steps are given for HalSiC and Crystar 2000. The increase in the second step was comparably low (0,6–1 %). For this reason, all IKTS materials were only infiltrated once. For zirconia infiltration, the total amount of carbon depended on the porosity of the RSiC material. Again, IKTS-3 with the highest porosity and largest pores stored the largest amount of carbon (5,7 mass-%). The volume fraction of filled pores (Fig. 3b) determined by image analysis was approximately 60–70 %, twice as high as for the ZrO₂-infiltrated materials. A detailed view of an infiltrated pore is given

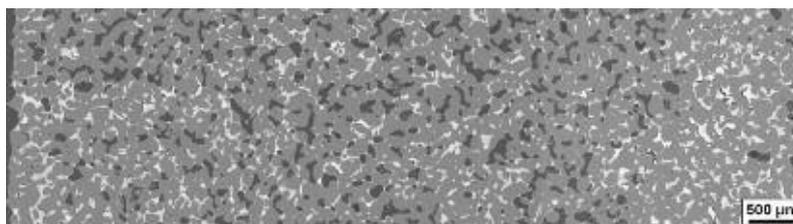


Fig. 5 FESEM micrograph of the cross section of the filled RSiC material IKTS-1 after infiltration with ZrO₂ (cross section over the entire sample height)

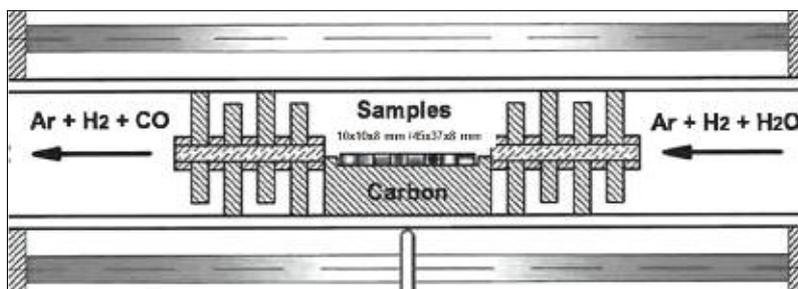


Fig. 6 Schematic illustrating the working principle of the reactor tube furnace

Tab. 2 Composition of the ash [13]

Component	C	Na ₂ O	K ₂ O	MgO	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	SO ₃
Composition [mass-%]	4,6	5,1	3,6	4,3	10,2	19,4	38,2	7,9	1,0	5,0

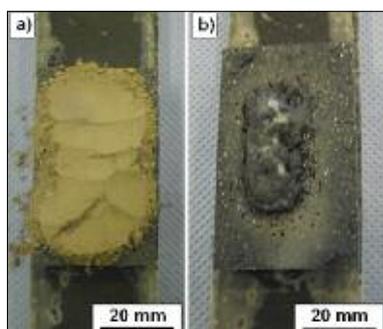


Fig. 7a–b Ash positioning on a carbon-filled RSiC (IKTS-1) a) before and b) after corrosion testing

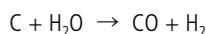
en in Fig. 4b. All the RSiC pores were filled during infiltration. After drying and pyrolysis, the carbon content was lower and the carbon particles were sintered, lying loosely in the centres of the RSiC pores and having nearly the same shapes as the pores (Fig. 4b).

The white zone around the carbon was silicon carbide generated during pyrolysis by reaction with the silicon dioxide always found on the surfaces of the SiC pores.

2.3 Corrosion experiments

The corrosion experiments were carried out in a reactor tube furnace (Fig. 6) at a temperature of 1300 °C with a dwell time of 100 h. The corrosive medium was a coal ash with a slightly acidic character, largely corresponding to the real conditions found in a gasification reactor. The chemical composition of the ash is shown in Tab. 2. Under the stated thermal and chemical conditions, the ash was molten and had a low viscosity.

A gas mixture consisting of 95 vol.-% argon and 5 vol.-% hydrogen with a dew point of 23 °C was guided over graphite moderators inside the reactor tube at a rate of 30 l/h. This was expected to result in the formation of a synthesis gas corresponding to the given reaction temperature according to the equilibrium reaction:



Thermodynamic calculations using *Factsage* 6.3 [12] yielded a composition of the resulting gas atmosphere at 1300 °C of app.

7,4 vol.-% hydrogen, 2,5 vol.-% carbon monoxide and 91,1 vol.-% argon as the main components. The argon content was selected to limit the explosion potential of hydrogen and the poisoning potential of carbon monoxide based on the lengthy period of operation of the laboratory system and to ensure that sufficient carbon would be available for gas synthesis.

The ash was evenly distributed on the tops of the samples, as shown in Fig. 7. Fig. 7a shows the sample before corrosion and Fig. 7b after corrosion.

Following high-temperature corrosion, the samples were investigated using electron microscopy. Cross sections of the SiC samples were prepared and examined using a FESEM (field emission scanning electron microscope) and energy dispersive X-ray spectroscopy. The formation of new phases was detected using X-ray diffraction (diffractometer D8 *Brucker*), CuK α radiation, 40 kV, 30 mA, 2 Θ range 10–90°).

3 Results and discussion

3.1 Stability of uninfiltred RSiC

An unfilled RSiC material was initially tested to provide information about the corrosion behaviour of the original material. As can be seen in the cross section in Fig. 8, the ash nearly completely infiltrated into the sample. The lighter grey in the micrograph represents the ash which filled the pores. Fig. 8b shows a pore at a higher magnification. The silicon carbide grains did not exhibit any corrosion under the given experimental conditions. The infiltrated glassy phase with a higher thermal expansion coefficient than that of SiC cracked during cooling.

3.2 Stability of zirconia-infiltrated RSiC

Zirconia-infiltrated RSiC-materials also showed infiltration of the ash into the remaining pores during the corrosion experiment (Figs. 9, 10). Infiltration was much more pronounced in the commercial materials. The zirconia in contact with the melted ash formed large, nearly round grains throughout the sample. Reaction of the slag with the ZrO₂ and formation of ZrSiO₄ only occurred in isolated areas. For all IKTS variants with higher amounts of zirconia in the micropores, ash infiltration could be reduced significantly (Fig. 10). Selected spectra ob-

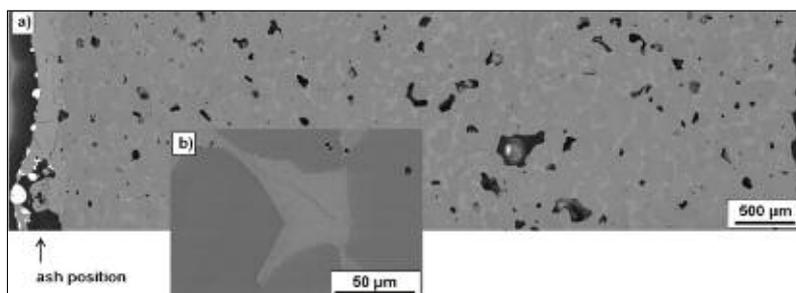


Fig. 8a–b Ash infiltration into RSiC Crystar: a) overview and b) detailed view of a glass-infiltrated pore with cracks in the glass

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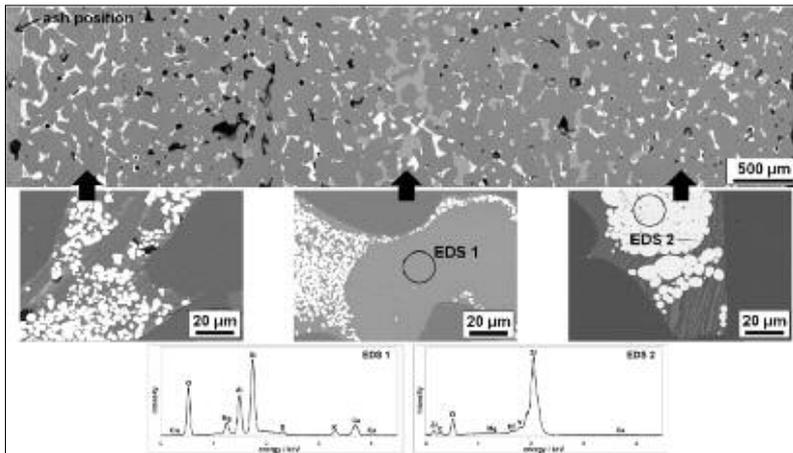


Fig. 9 Ash infiltration into commercial RSiC sample filled with zirconia

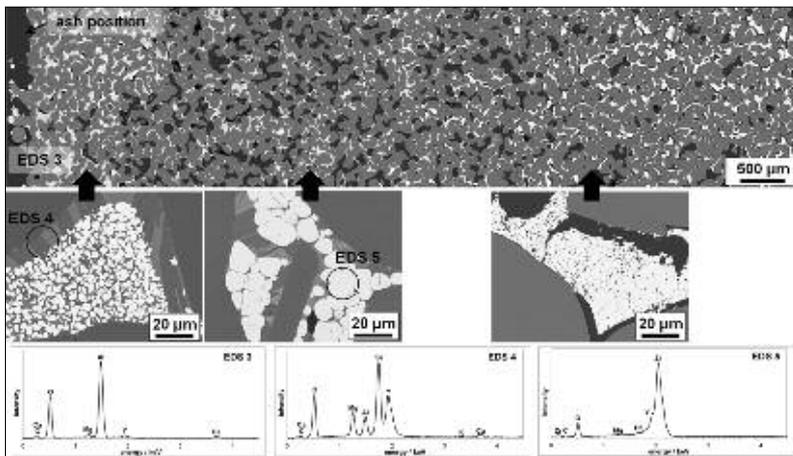


Fig. 10 Ash infiltration into RSiC material IKTS-3 filled with zirconia

tained from different regions are also given in Fig. 10. The corrosion depth was nearly the same for all IKTS-RSiC materials (3–5 mm). The lowest penetration depth was measured for IKTS-3. In all cases, the silicon carbide skeleton was not corroded during the experiments.

The observed ash blocking can be explained through changes in the chemical composition of the ash by reactions with zirconia, formation of new phases and a corresponding shift in the flow behaviour of the molten ash to higher viscosities. Different chemical reactions occurred. ZrO_2 reacted with Ca

and Mg to form fully stabilised ZrO_2 , which resulted in an increase in the viscosity of the molten ash which in turn halted the infiltration process. ZrO_2 also reacted with SiO_2 , as indicated by the EDX results. X-ray diffraction of an area directly beneath the surface yielded silicon carbide and calcium and sodium aluminosilicates. Cubic ZrO_2 was also found, indicating that the ZrO_2 phase was stabilised by cations. The formation of $ZrSiO_4$ observed by EDX was not determined unequivocally by XRD, possibly due to overlapping of peaks. As also observed previously by Herrmann [10], the sulphate in the slag quickly transformed into sulphides. These sulphides were not liquid and therefore were observed in the slag on the surfaces of the materials, but not in the bulk regions.

3.3 Stability of carbon-infiltrated RSiC

Carbon-infiltrated RSiCs exhibited a considerably different corrosion protection mechanism than that of zirconia-infiltrated materials. In conventional refractory materials containing carbon, carbon bonds are known to inhibit infiltration by ash due to their chemical inertness [13]. The carbon-infiltrated RSiC developed in this work exhibited the same behaviour. All infiltrated materials, the commercial and the IKTS-made materials with different pore sizes and fractions, showed identical protection mechanisms with minimal infiltration and mainly low wettability. Cross sections of the surfaces of both material types are presented in Fig. 11. Small amounts of ash covered the surface of Crystar 2000 (Fig. 11a) and infiltrated into the first available pores up to a depth of about 100–200 μm. No ash could be de-

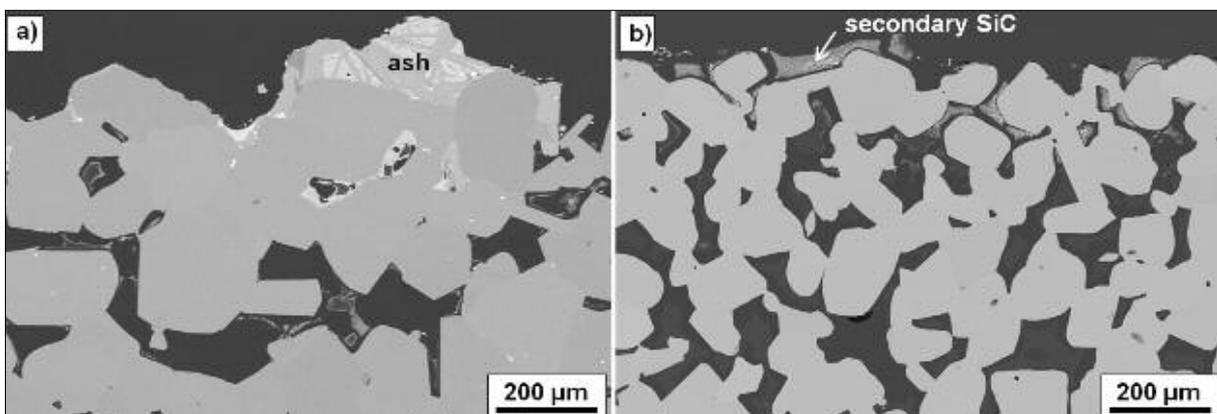


Fig. 11a–b FESEM micrographs of the carbon-infiltrated materials after corrosion, illustrating the low wettability by the molten ash: a) commercial RSiC (Crystar 2000) and b) IKTS-RSiC (IKTS-1)

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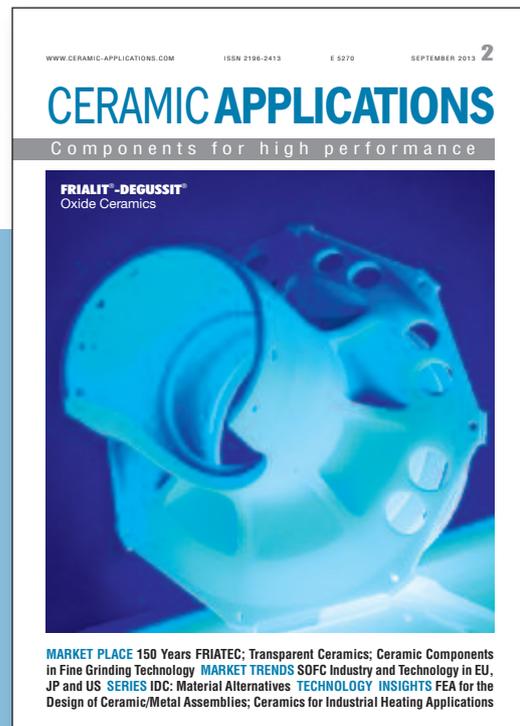
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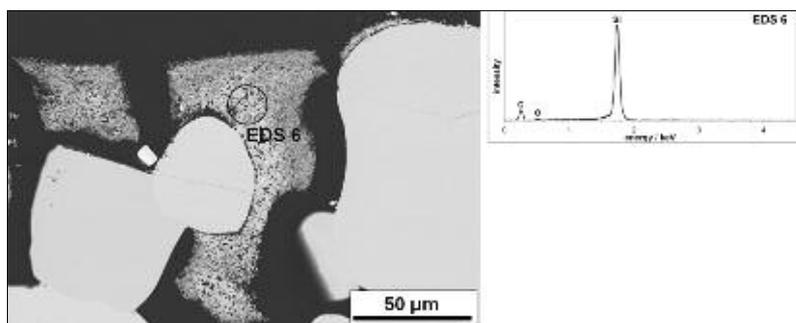


Fig. 12 FESEM micrograph and EDX spectrum of IKTS-1 after corrosion (SiC formed at the interface between the refractory and the molten ash)

tected on the surfaces of the IKTS-RSiC materials; instead it was found lying loosely on the sample surfaces (Fig. 7b). Any carbon situated on the sample surfaces reacted to silicon carbide (Fig. 12); however, no reactions were observed in the bulk regions of the RSiC materials. The SiC grain boundaries showed no changes at all, with the microstructure not appearing to have been influenced by any corrosive attack.

Although both types of RSiC withstood the corrosive conditions very well, the new modification with a higher porosity and thus possibly a higher amount of carbon in the pores exhibited slightly better behaviour. The ash penetration and infiltration could be prevented successfully. Comparison of IKTS-1 to IKTS-3 indicated that the amount of carbon in the pores played a more dominant role than pore size and overall porosity did.

4 Conclusions and outlook

Silicon carbide ceramics with intrinsic bonding are of interest for linings in modern gasifiers to reach higher efficiencies combined with increased long-term durability. A new material concept combining the high stability at high temperatures and thermal shock and chemical resistance of a porous recrystallised silicon carbide with infiltrated corrosion protection materials made from zirconia and carbon was developed and tested. This design allowed the intrinsically high corrosion resistance of the three-dimensional network of SiC to be combined with the possibility of using other components to reduce or avoid the infiltration of slag into the pores of the RSiC during application.

The infiltration process was effective even if only approximately $\frac{1}{3}$ oder $\frac{2}{3}$ of the pores were filled with ZrO_2 or carbon respectively. Infiltration was more homogenous in the

materials with higher porosities and pore sizes. The process can be optimised for commercial application through the use of fine-grained unstabilised ZrO_2 and in situ conditioning of the infiltrated materials during application.

The materials were tested in corrosion experiments in a tube furnace at 1300 °C for 100 h in a reducing atmosphere (95 % argon, 5 % hydrogen) humidified at 23 °C using a slightly acidic coal ash. Two different protection mechanisms were observed. In the case of zirconia, ash infiltration always occurred to an extent depending on the amount of zirconia in the pores. In commercial SiC and at low amounts of zirconia, the samples were infiltrated through their entire thicknesses; in the IKTS materials the infiltration was stopped at a depth of 3–4 mm. Reaction of zirconia with CaO and MgO and formation of fully stabilised cubic zirconia as well as calcium and aluminium silicates were also observed.

In the case of carbon infiltration, the wettability of the ash was drastically reduced. Either no or an extremely small amount of ash infiltration to a depth of only 200 μm was detected. All material varieties with high amounts of carbon (IKTS-1–IKTS-3) showed particularly favourable behaviour which is expected to result in an accordingly higher service life. The only sign of reaction with the liquid ash was the formation of silicon carbide.

A detailed analysis of the wetting behaviour is given elsewhere [14]. The results of the experiments carried out in the present work demonstrate the high potential of the infiltrated RSiC materials for use as refractories.

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