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The influence of process parameters on *in situ* inorganic foaming of alkali-bonded SiC based foams

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Abstract

Silicon carbide (SiC) foams were developed with a low temperature process by using an inorganic alkali aluminosilicates binder, also known as geopolymer. The foaming agent was the metallic silicon present as impurity in the SiC powder. Si^0 in the alkaline solution led to gas evolution that induced the foaming of the slurries. The binder was a geopolymeric resin with atomic ratio Si/Al = 2 and potassium as alkaline cation, classified as (K)poly(silalate-siloxo). The geopolymeric resin was prepared using metakaolin as aluminosilicatic raw powder, while the alkali aqueous solution was KOH/K_2SiO_3. Metakaolin in alkaline conditions dissolved and re-precipitated to form geopolymeric nano-particulates that acted as a glue to stick together SiC particles (90 wt.%). Process parameters such as water addition, mixing time and curing temperature were correlated to the foam structure. The formation of prolate pores induced anisotropy in the compressive strength. The foams were studied by dilatometric analysis in inert and oxidative atmospheres up to 1200 °C.

Keywords: B. Porosity; C. Thermal properties; D. SiC; Geopolymer

1. Introduction

Reticulated porous ceramics characterized by high porosity (70–95%) are expected to be used in many different technological applications because of their high gas permeability and large surface area as well as for the high temperature stability and thermal shock resistance [1–3]. Industrial uses include structural lightened parts, insulator panels, filters and membranes (for micro- and ultra-filtration, separation, particulate environmental clean-up and reuse, molten metal filters), radiant burners, gas or chemical sensors, support materials for catalysis or adsorbents. For all the mentioned applications, it is mandatory to control the pore structure (shape, morphology, orientation, surface properties) as well as the texture, porosity and pore size distribution.

Several ways to produce macroporous ceramics have been reported [4,5], such as sacrificial template [3,6–10], replica [11] or direct foaming methods [12,13]. Regardless of the technique used, ceramics foams are usually treated at high temperature for the burnout of fugitive additives or templates and for consolidation (sintering) in order to reach specific mechanical and functional properties.

Consolidation by chemically activated inorganic binders is suitable to replace the sintering step. Among inorganic binders, alkali alumino silicate binders behave as inorganic polymers, the so called geopolymers, and they were developed as an alternative to organic matrices for composites [14,15].

Geopolymers are alkali bonded ceramics (ABC) [16], i.e. they belong to the class of the chemically bonded ceramics (CBCs) meaning that ceramics can be produced at low temperatures using chemical reactions. Actually, the reaction of an aluminosilicate powder with a highly concentrated aqueous alkali hydroxide and/or silicate solution produces a synthetic amorphous to semi-crystalline alkali aluminosilicate material which is amorphous to X-ray analogously to zeolites. Among the aluminosilicatic raw powders, metakaolin is the most reactive in alkaline conditions [17]. During geopolymerization a gel network is formed, consisting of SiO_4 and AlO^{4-} tetrahedra linked in an alternating sequence. The geopolymeric resin consists of nanoprecipitates [18,19] that act like a glue sticking together the fillers (powders or fibres, glass, ceramic,

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metal or organics) forming the geopolymeric composite materials. Different fillers are used to tailor specific physical and mechanical properties of the geopolymers in dependence of the applications [20–24].

Hydrogen peroxide is a well-known blowing agent [25], while the redox reaction of Al in alkaline solution induces porosity by H_2 evolution [26]. Also redox reaction of metallic Si impurities in silica fume could be used to prepare geopolymeric foams [27,28]. Actually, the Pourbaix diagram of silicon indicates that in alkaline conditions hydrogen evolution is always favoured [29]. The reaction responsible for gas evolution is [28]:

$$4H_2O + Si^0 \rightarrow 2H_2\uparrow + Si(OH)_4, \quad \Delta H = -314\,\text{kJ} \tag{1}$$

In a previous work [30] it was demonstrated the possibility to develop silicon carbide foams with a low temperature process, i.e. *in situ* inorganic foaming with a contemporary chemically bonding. The foaming agent was metallic silicon that is always present as impurity in SiC powders because of the synthesis process. The binder was a geopolymeric resin with atomic ratio Si/Al = 2 and potassium as alkaline cation, classified as (K)poly(silalate-siloxo) [14,15]. The geopolymeric resin was prepared using metakaolin as aluminosilicatic raw powder, while the alkali aqueous solution was KOH/K_2SiO_3.

In the present paper, process parameters such as water addition, mixing time and curing temperature were correlated to the foam structure. The foams were also studied by compressive tests and dilatometry in inert and oxidative atmospheres up to 1200 °C.

2. Experimental

2.1. SiC foam preparation

A calcined kaolin (metakaolin) with high specific surface (30 m^2/g) was used to prepare the alkali aluminosilicate binder. The powder was prepared from a commercial kaolin (grade BS4, AGS Mineraux, Clèrac, France) by calcination in electric kiln (750 °C for 15 h). Details about this raw powder are reported elsewhere [31].

Potassium silicate solutions with molar ratios of $SiO_2:K_2O = 2$ and $H_2O:K_2O = 23$ were prepared by dissolving KOH pellets (purity > 99%, Merck, Darmstadt, Germany) into potassium silicate aqueous solution with molar ratio $SiO_2:K_2O = 3.57$ (KSil 35Bè R3.5, Ingessil srl, Montorio, Italy) by magnetic stirring.

SiC grade 100F (SIKA TECH, Saint-Gobain Ceramic Materials GmbH, Germany; α-SiC polytypes with metallic Si (0.7 wt.%) and free C (1 wt.%); specific surface area: 0.58 m^2/g; bi-modal distribution centred at 150 μm (20%) and 45 μm (80%)) was used as the main component of the foams.

Foams were prepared by mechanical mixing (Yellow line OST 20 basic, IKA-WERKE GmbH & Co., Staufen, Germany) at 100 rpm from 1 to 10 min the SiC powder (75.00 wt.%) with metakaolin (8.33 wt.%) and with the KOH/K_2SiO_3 solution (16.67 wt.%). From 6.25 to 25.00 wt.% of distilled water was added to the slurries. After setting and water removal, the weight fraction of SiC corresponded to 90% of the foam.

The slurries (20 ml) were cast in plastic cylindrical open moulds with a diameter of 40 mm and maturated at room temperature or cured at 40 °C, 60 °C or 80 °C for 24 h. The foam expansion mainly occurred in the axial direction of the cylindrical mould. After curing, the foams were let at 80 °C for other 24 h to complete the setting process.

2.2. Micro- and macro-structural characterizations

The bulk density and porosity of the sponges were determined by weight-to-volume ratio. The morphological and microstructural features were observed by SEM-EDS (SEM, Cambridge S360, Cambridge, UK; EDS, INCA Energy 300, Oxford Instruments, Oxford, UK). Ultra-macro-porosity was investigated by image analysis (Image Pro Plus 6.0, Media Cybernetics, Inc., Bethesda, MD, USA) of high resolution photos (scanner Sharp JX330, Japan) and of scanning electron micrographs of the cross-sections.

2.3. Compressive strength

Compressive strength tests on SiC foams were carried out on 20 mm cubic samples using a Zwick Z050 testing machine (Zwick GmbH, Ulm, Germany). The compressive load was applied parallelly and perpendicularly to the foam expansion direction. The crosshead speed was set at 1 mm/min.

2.4. Thermal characterization

Dilatometric analyses were performed with a DIL402 dilatometer (NETSCH, Geraetebau, Germany) on 25 mm-long bars up to 1200 °C in flowing Ar or air (heating rate 5 °C/min). Bruker D8 Advance diffractometer with CuKα radiation (Karlsruhe, Germany) was used to determine the crystalline phases.

3. Results and discussion

3.1. Microstructure and chemical bonding

Fig. 1 shows some microstructural features of the developed alkali bonded SiC-based foams. As already observed [30], pore walls and struts are not compact but formed by large and elongated SiC grains where the alkali aluminosilicate binder is not homogenously distributed (Fig. 1a). The surface of the SiC particles is covered by geopolymeric nano-precipitate of 60 nm and almost fully geopolymerized metakaolin particles (Fig. 1b). These features are observed at any setting condition.

The geopolymerization mechanism is quite complex [32], but may be simplified into a few steps [33]: dissolution (consuming water); speciation equilibrium; gelation; reorganization; polymerization (releasing water); and hardening.

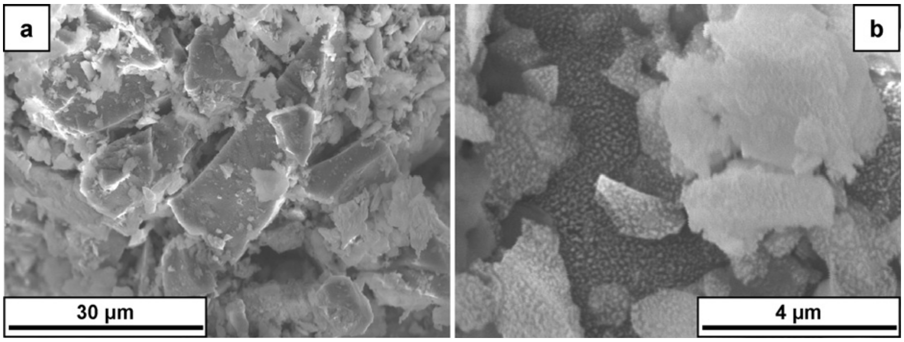

Fig. 1. (a) Scanning electron micrographs showing the microstructure of a SiC foam. (b) A particular of a SiC particle surface covered by geopolymeric nano-precipitates.

During the first step of geopolymerization, the metakaolin (i.e. the aluminosilicate source) is dissolved by alkaline hydrolysis, giving aluminate and silicate species. As the surface of silicon carbide grains is always enriched in oxygen, it is chemically similar to the surface of silica [34]. The hydroxyl anion (OH^-) is considered to be a catalyst for the dissolution reaction of silica [35]. As such, silica readily dissolves in highly alkaline media ($pH > 11$) in the form of monomeric $Si(OH)_4$ until the equilibrium concentration is reached [36]. Moreover, silica solubility increases with temperature [37,38]. It follows that due to the presence of silica, the surface of SiC particles participates to the geopolymeric process and geopolymeric nano-precipitates easily forms on SiC surface [30,31].

This behaviour was previously confirmed by TG–DTA analyses [30,31] as the stating oxidation temperature was 80 °C lower than in pure SiC powder. The oxidation started at lower temperature because the protective silica layer on the SiC surface was leached and replaced by a nano-porous geopolymeric layer.

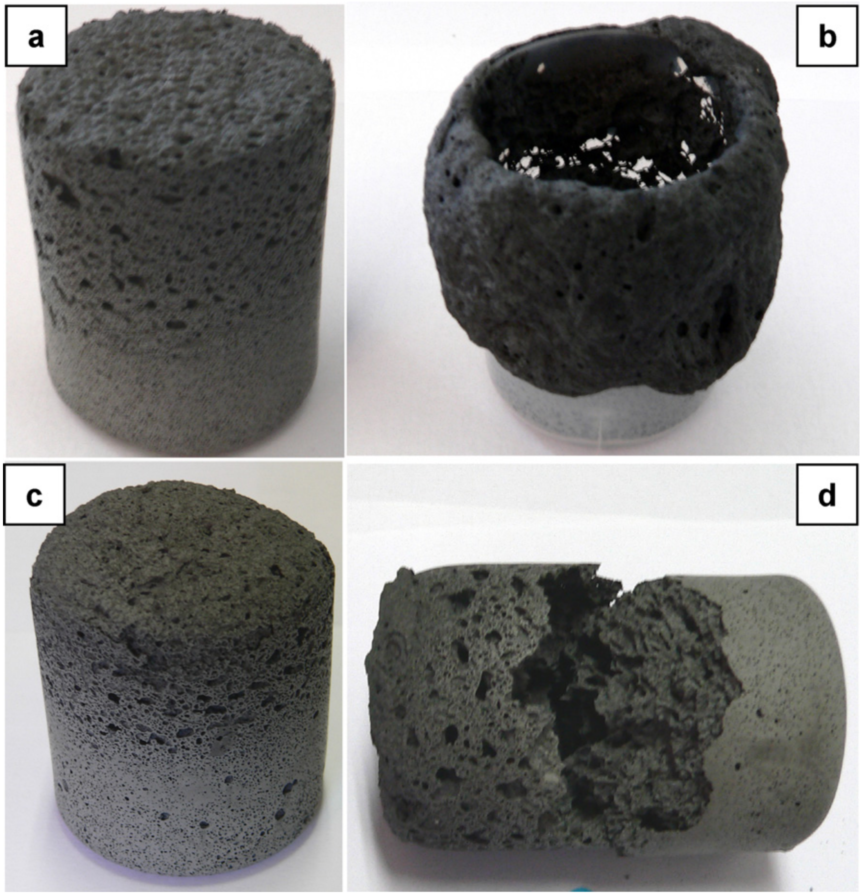

Fig. 2. Examples of foaming behaviour under different foaming condition. SiC foams prepared at 40 °C (a and b) and 80 °C (c and d) with 6.25 wt.% (a and c) and 14.3 wt.% (b and d) water addition.

3.2. Macrostructure and foaming mechanism: influence of water addition, mixing time and process temperature

During maturation at room temperature or curing up to 80 °C of the slurries, metallic Si impurities in the SiC powders induce H_2 evolution as a consequence of the redox reaction (1) occurring in alkaline aqueous solution.

As reported by Prud'homme et al. [27], gas evolution leads to foamed macro-structures, i.e. inorganic *in situ* foam formation, when the gas pressure is lower strength to collapse the pore wall. In this case, the viscosity of the composite slurries increases and the material consequently consolidates. On the contrary, when the gas pressure exceeds the strength of the pore walls the structure collapses (Fig. 2b and d). Examples of foaming behaviour under different foaming condition are shown in Fig. 2.

Fig. 3 reports the plots of the final density and volume expansion of the slurries maturated at room temperature in function of the water addition and mixing time. By increasing the water amount or the mixing time, the expansion decreases while the density increases.

The increase of mixing time reduces the foam expansion as mixing promotes and speeds up the geopolymeric setting by homogenizing the slips and renewing the reactive surface of the raw powders [21,39]. When fast setting (hardening) takes place, reaction (1) does not occur because the alkaline aqueous solution is the medium for the silicon anodic corrosion [30]. This is the case of previously developed SiC-based paints prepared with alkali aluminosilicate binders, when 200 μm-thick coatings deposited by brushing quickly set and hardened [31].

Since water addition decreases the viscosity of the slurries, gas easily evolves through the slurries with very low volume expansion as the strength of the pore walls is not enough to counterbalance the gas pressure. Moreover, water addition affects also the setting behaviour. Geopolymerization process can be approximately divided into two periods, i.e. dissolution–hydrolysis and hydrolysis–polycondensation, that overlap once the solid material mixes with liquid activator [40]. High alkalinity is necessary to leach raw material as water dilution reduces the geopolymerization rate at dissolution–hydrolysis stage [41]. However, water is necessary for the leaching of solid particles and the hydrolysis of dissolved Al^{3+} and Si^{4+} ions. Water is a vehicle for the geopolymerization reaction to take place and does not remain in the final structure [32,33]. During polycondensation water behaves as a product [33,42] and, if too much, hinders the polycondensation kinetically.

On the basis of the above reported results, mixing time of 1 min and 6.25 wt.% water addition were used to prepare samples for checking the curing temperature effects on the foaming process. The enhancement of the curing temperature favoured the foaming process with a clear effect on the volume expansion of the slurries (Table 1). Actually, reaction (1) is favoured by temperature increase (from $\Delta G = -359.4$ kJ at 25 °C to $\Delta G = -366.6$ kJ at 80 °C [43]).

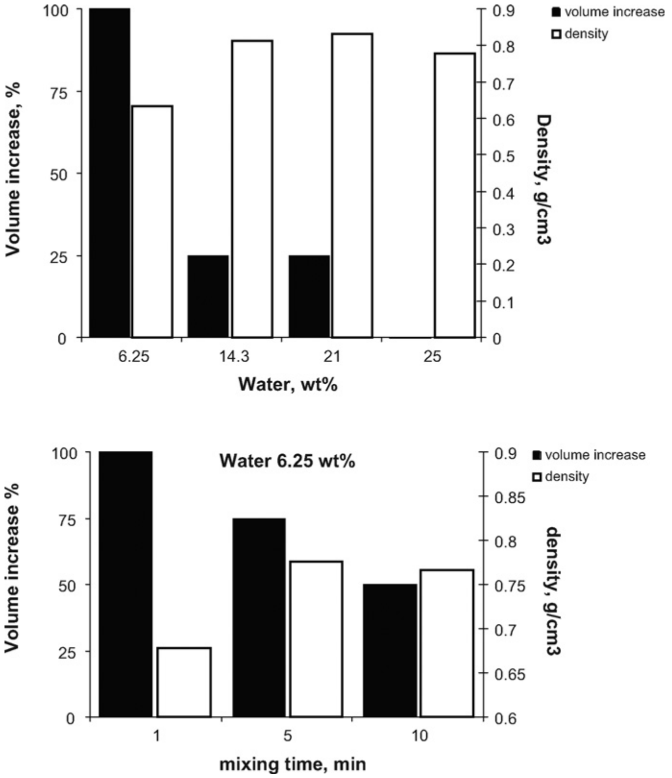

Fig. 3. Plots of the final density and volume expansion of the slurries maturated at room temperature as a function of the water addition and mixing time.

The temperature dependence of the foaming is well evidenced in Fig. 4. Volume expansion, macropores dimension and prolate shape belong to the balance of the thermodynamic of reaction (1), viscosity and geopolymerization (hardening). Moreover, SiC starting powder has bimodal size distribution, centred at 150 μm (20%) and 45 μm (80%), and the grains have an elongated shape [31]. It follows that the pore size of the foams is very broadened, ranging from 50 μm to about 500–600 μm, with thin (micrometric) porous walls [30].

Generally, the viscosity of a slurry decreases with the rise of the process temperature, while H_2 evolution (and consequently gas pressure) is enhanced.

The foam expansion occurred mainly in the axial direction of the open cylindrical mould due to gas escaping from the open

Table 1
Characteristics of the alkali-bonded SiC based foams with 6.25 wt.% water addition, prepared at different curing temperatures.

	Curing temperature			
	25 °C (RT)	40 °C	60 °C	80 °C
Slurry volume expansion, %	100	150	150	150
Final density, g/cm^3	0.63 ± 0.02	0.55 ± 0.03	0.50 ± 0.02	0.50 ± 0.01
Total porosity, %	78	80	83	83

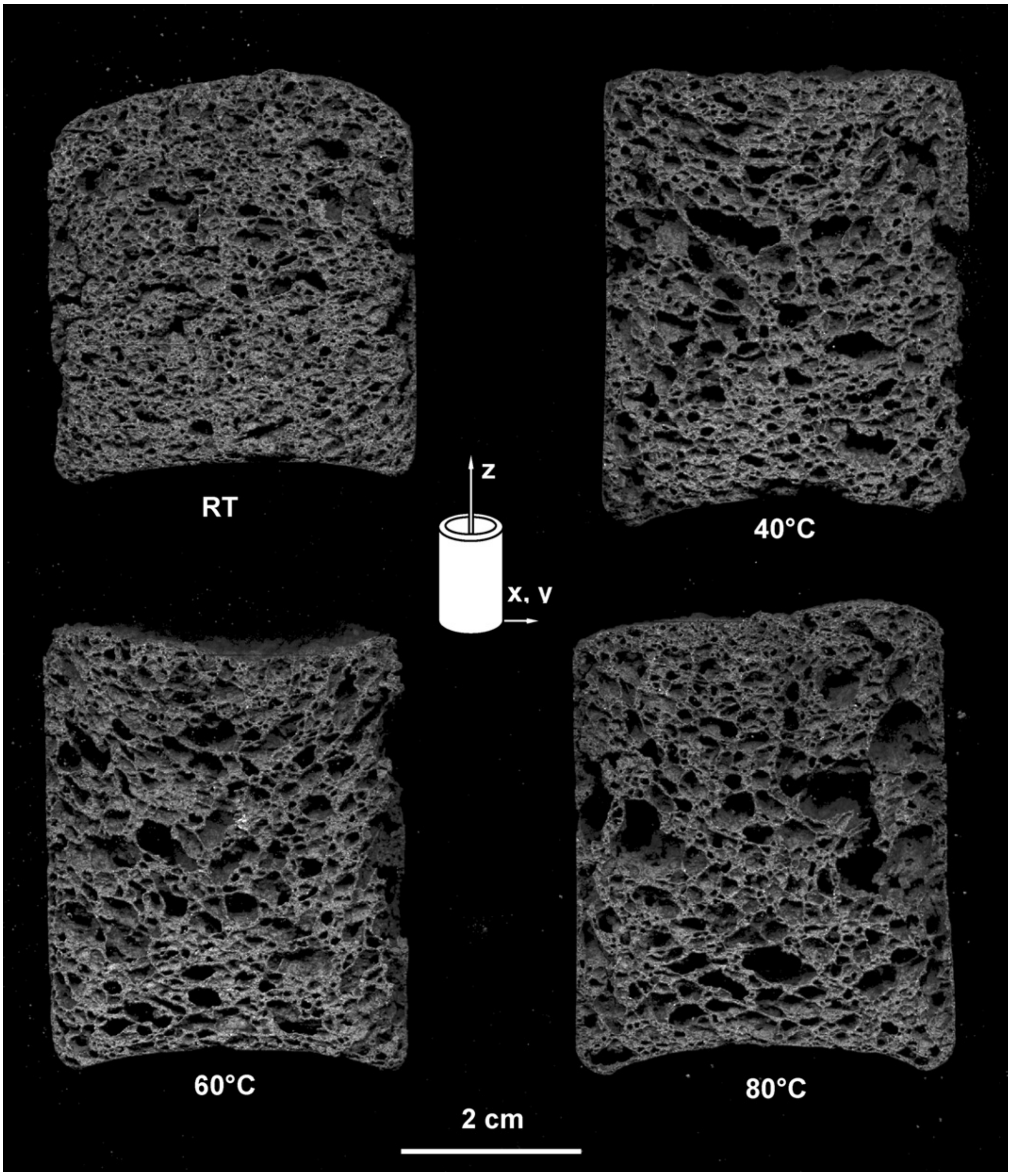

Fig. 4. High resolution photos of the cross-sections of the SiC foams prepared with 6.25 wt.% water addition and cured at RT, 40 °C, 60 °C and 80 °C. In the sketch of the open top mould, *z* axis represents the main expansion direction of the foams.

top and the constrain of mould in the other directions. Image analysis of the macropores shape, aspect ratio and alignment in the upper part of the foams cross-sections indicates that (Fig. 4):

- at RT, the top surface is convex and the few prolate pores are aligned perpendicularly to the expansion direction. A moderate volume expansion occurs because of the low gas pressure.
- In the range 40–60 °C, the top surface is flat (40 °C) or slightly concave (60 °C) and the prolate pores are distributed along the concave curve as well. A moderate structure collapse takes place because of the high H_2 pressure and the low viscosity.
- At 80 °C, the top surface is slightly rounded. The hardening due to the setting of the alkali aluminosilicate binder takes place faster than at the other temperatures. The hardening is also favoured by the viscosity increase due to partial water evaporation occurring at 80 °C. Tight pore walls are formed, thus limiting the structural collapse under high H_2 pressure and promoting foaming expansion.

3.3. Compressive strength

SiC-based foams maturated at room temperature were mechanically tested in compression tests both parallelly and perpendicularly to the expansion direction of the foams. In all

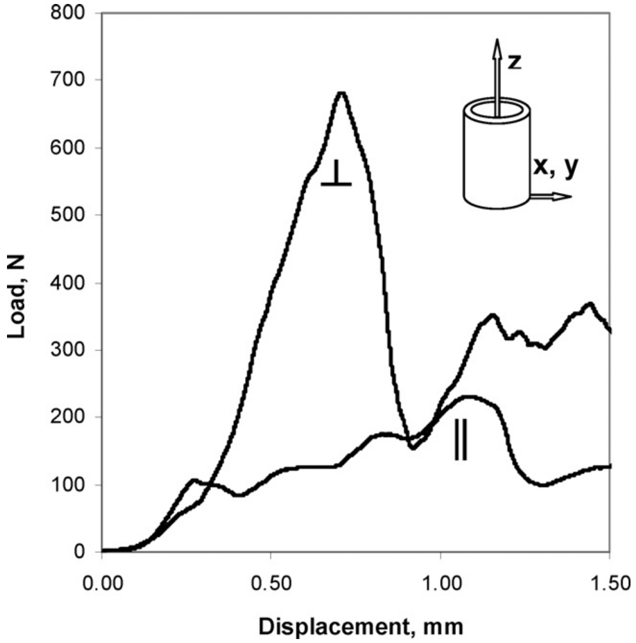

Fig. 5. Examples of load-displacement curves of SiC foams tested under a compressive load applied parallelly (||) and perpendicularly (⊥) to the main expansion direction of the foams (*z* axis in the sketch).

the tests, the specimens fail showing a jagged load-displacement curve (Fig. 5), which is a typical feature of porous ceramics in compression [44]. The compressive strength in the parallel direction is 0.9 ± 0.2 MPa [30], while in the perpendicular direction is 1.7 ± 1.1 MPa. The spread in the pore size distribution contributes to the data dispersion. Moreover, structural anisotropy due the prolate shape of the macropores with an aspect ratio ranging from 2 to 5 (Fig. 4) is responsible for the difference in the mechanical strength along the parallel and perpendicular direction to the main foaming expansion [45].

3.4. High temperature behaviour

The thermal behaviour of the SiC-based foams maturated at room temperature was monitored by dilatometric analysis up to 1200 °C both in air and in Ar (Fig. 6).

The derivatives of the dilatometric plots (dashed lines in Fig. 6) evidence the changes in axial shrinkages. The first contraction occurring in the temperature range 100–200 °C is related to the overall desorption of water from the micro- and macropores as already observed in geopolymers by Bell et al. [46]. The maximum rate of contraction is observed at about

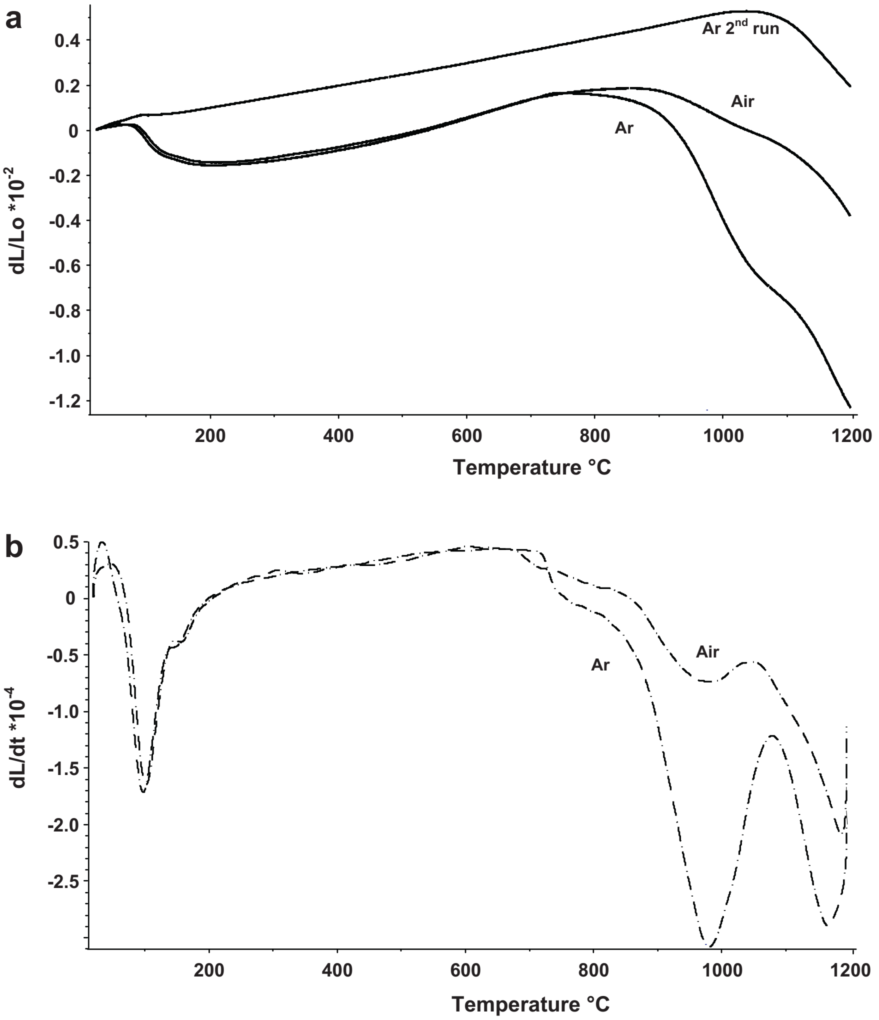

Fig. 6. Dilatometric analyses in air and argon (two heating runs) of SiC foam prepared at RT (a) and derivatives of the dilatometric plots (b).

100 °C both in air and argon. Above this temperature, the plots exhibit an almost linear trend up to about 700 °C. Up to 750 °C, the dilatometric plots in air and Ar can be nearly superimposed. At 750 °C, the shrinkages due to viscous flow and particles rearrangements starts. In the case of the dilatometric analysis carried out in air, the shrinkage is counterbalanced by volume expansion due SiO_2 formation during oxidation. In a previous work [30], non-isothermal simultaneous thermal analyses in air of SiC-based foams were performed. The apparent weight gain due to SiC oxidation to form silica and gaseous carbon oxides starts at 760 °C, following reaction (2) [47]:

$$SiC + 3/2O_2(g) \rightarrow SiO_2 + CO(g) \tag{2}$$

Consequently, after the dilatometric tests the measured shrinkages are 1.35% in Ar and 0.66% in air.

During a second heating run in argon, the dilatometric plot is linear up to about 1000 °C. In the temperature range 25–1000 °C, the thermal expansion coefficients (CTE) is $5.27 \times 10^{-6}\,^{\circ}C^{-1}$. This value is close to that of α-SiC value ($(5.0–5.5) \times 10^{-6}\,^{\circ}C^{-1}$) [48], as a consequence of the rule of the mixtures, and confirms previous results [31]. At about 1040 °C, the shrinkage starts. In both the first run in air and argon a second stage of viscous flow occurred around this temperature (Fig. 6) and it could be related to the melting of leucite plus liquid [49]. Geopolymeric resin with atomic ratio Si/Al = 2 and potassium as alkaline cation has been shown to crystallize into leucite, $K_2O{\cdot}Al_2O_3{\cdot}4SiO_2$, by heating [46]. Moreover, compositional heterogeneities, together with the presence of free alkali ions in the geopolymer structure, favour the formation of a glassy phase [46]. After the second heating run in argon, the XRD spectrum (Fig. 7) reveals α-SiC polytipes as the main phases and the presence of traces of leucite and cristobalite.

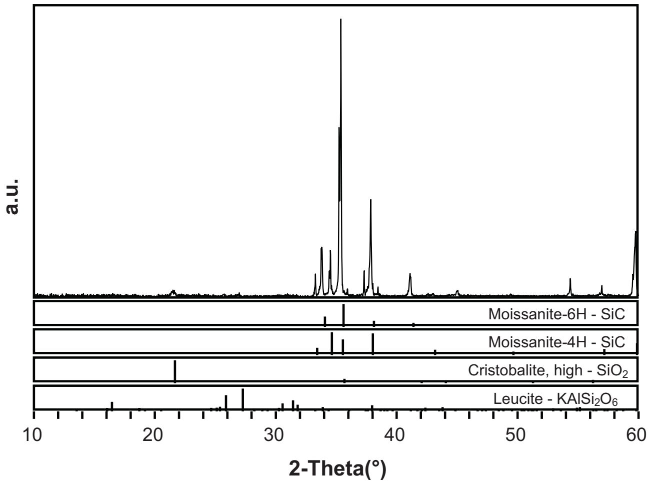

Fig. 7. XRD spectrum SiC foam after two heating runs up to 1200 °C in argon.

After the dilatometric tests up to 1200 °C, the macrostructures of the SiC foams remain almost the same with very low dimensional changes (Fig. 8). On the contrary, the microstructures showed significative changes as micro- and nano-porosity disappear because of the glass formation due to SiC oxidation in air and melting of the geopolymeric binder both in air and argon atmosphere.

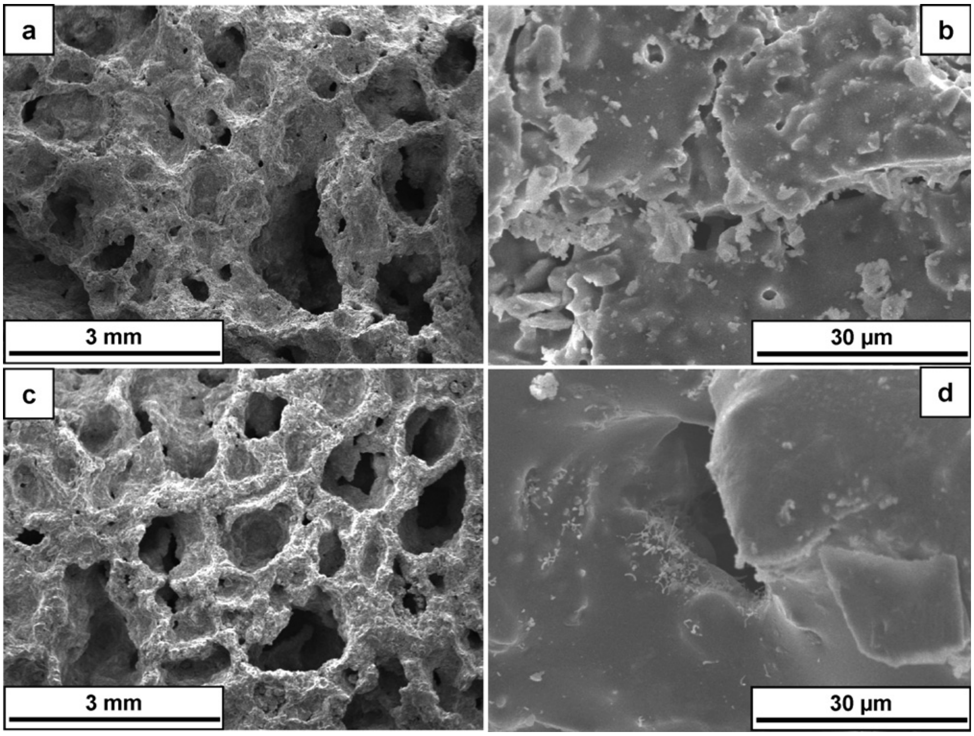

Fig. 8. Scanning electron micrographs of the SiC foams after dilatometric tests up to 1200 °C in air (a and b) and argon (c and d).

4. Conclusion

In order to replace the sintering step, SiC-based foams were prepared using alkali activated inorganic binders to contemporarily promote i*n situ* foam formation and consolidation.

The foaming agent is Si^0 that is present in SiC powder as impurity of the synthesis process. Process parameters such as water addition, mixing time and curing temperature were correlated to the foam structures. By increasing the water amount or the mixing time, the density increases and the expansion decreases. The increase of the foaming temperature enhances the volume expansion. The final foam structure belongs to the balance of the thermodynamic of the foaming reaction, viscosity and geopolymerization process.

The structural anisotropy due the prolate shape of the macropores gives higher compressive strength in the perpendicular direction to the foam expansion than in parallel direction.

Dilatometric analyses indicate different behaviours in air or in argon atmosphere due to the SiC oxidation occurring in air. Both in air and argon, glass formation occurs and viscous flow sintering induces a slight shrinkages starting at 750 °C.

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