



Behavior of alkali bonded silicon carbide foams in modified synthetic body fluid



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ABSTRACT

Alkali bonded SiC based (90 wt%) foams were prepared exploiting the ability of impurities of metallic silicon present in the SiC powder to develop hydrogen gas during the synthesis, thus inducing the formation of interconnected ultra-macropores, needed for bone in-growth. A treatment into a simulated body fluid enriched with Sr²⁺ was exploited to nucleate substituted biomimetic calcium phosphates onto the material in order to preliminarily study the material potentialities for bone substitution.

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1. Introduction

The porous structure and chemical nature of ceramic materials play a critical role in improving the efficiency of bioceramics in tissue engineering. SiC has already been demonstrated to have biomaterial properties [1–4]; bone substitutes were obtained by wood ceramization [3,4]. Sponge replica or pore sacrificial templates are also widely used wet forming techniques, that need sintering treatments to obtain synthetic bone substitutes.

Silicon carbide foams characterized by high porosity (75–85%) and a compressive strength of ~1 MPa were developed with a low temperature process [5,6], i.e. *in situ* inorganic foaming with a contemporary chemically bonding based on geopolymerization [7,8].

Geopolymers have been already proposed as biomaterials [9–11]. The health risks related to excessive alkalinity, that can induce cell death, were overcome by thermal treatments at 500 °C [10], while the presence of Al in the structure, that can lead to brain disease [12], was reduced by increasing Si/Al ratio or realizing composite materials, since low Al concentration seemed to favor new bone formation [13].

The aim of this study is to produce an alkali bonded 90 wt% SiC based foam in which the pH of the material in wet condition is controlled by washing treatment after setting. A treatment into a Sr²⁺ enriched SBF is exploited to assess the ability of Sr substituted biomimetic apatites to nucleate in the foam, in view of potential

use as bone substitute in osteoporotic patients. Sr is reported to improve bone cell growth and bone quality and quantity [14–17].

2. Materials and methods

Foam were prepared by mechanical mixing of the SiC powder (85.75 wt%, grade 100 F SIKA, Saint-Gobain, metallic Si=2 vol%; no free C; bi-modal distribution: 150 μm=20% and 45 μm=80%) with metakaolin (3.85 wt%, M1200S, AGS Mineraux, see [18]) and with a NaOH/Na₂SiO₃ solution (10.40 wt%). The NaOH/Na₂SiO₃ aqueous solution with molar ratios of SiO₂:Na₂O=2 and H₂O:Na₂O=23 was prepared by dissolving NaOH pellets (purity 98%, Aldrich) into sodium silicate solution (NACOREN 330B, Ingessil srl). After setting (24 h at room temperature and 24 h at 80 °C), SiC corresponded to 90 wt%. A washing treatment was realized by using 10 ml of deionized water for 0.2 g of material. Foams were then dried at 100 °C.

SBF solution was prepared following the procedure in [19], but including Sr²⁺ ions. The pH of SBF was 7.4. The nominal concentrations of HCO₃²⁻, HPO₄²⁻, Ca²⁺, Mg²⁺, Na⁺ and K⁺ ions were the same as those of the human plasma: 27.0, 1.0, 2.5, 1.5, 142.0 and 5.0 mM, respectively. The concentration of Sr²⁺ was 0.20 mM. The ion concentration was checked by ICP-OES (Liberty 200, Varian). The foams were immersed in the SBF solution (0.2 g of foam in 10 ml of SBF), at 37 °C in closed vessels that were kept rotating for 21 days.

The bulk density and porosity of the foams were determined by weight-to-volume ratio. The morphological, microstructural, pore sizing and chemical features were studied by SEM-EDS (Cambridge S360 and INCA Energy 300) and by high resolution

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photos (Scanner Sharp JX330). Crystalline phases were detected by XRD (Bruker D8 Advance).

3. Results and discussion

Besides an open nano-micro-pore's network allowing a fast permeation with biological fluids and the vascularization, the bone substitute should possess interconnected ultra-macropores to allow the new bone ingrowth. SiC powder auto-induced gas blowing in the synthesis conditions, causing interconnected ultra-macropores formation, because of H₂ evolution due to the reduction reaction of water caused by metallic silicon.

Fig. 1 shows the macro- and microstructure of the as-consolidated foams. The density of the foam was 0.92 g/cm³ while the total porosity was 70%. Rounded ultra-macropores ranged from 100 μm to 2 mm (Fig. 1a). SiC surface participated in the geopolymerization [20] and the poly-sialates nanoprecipitates acted as a binder for the structural powder (Fig. 1b and c).

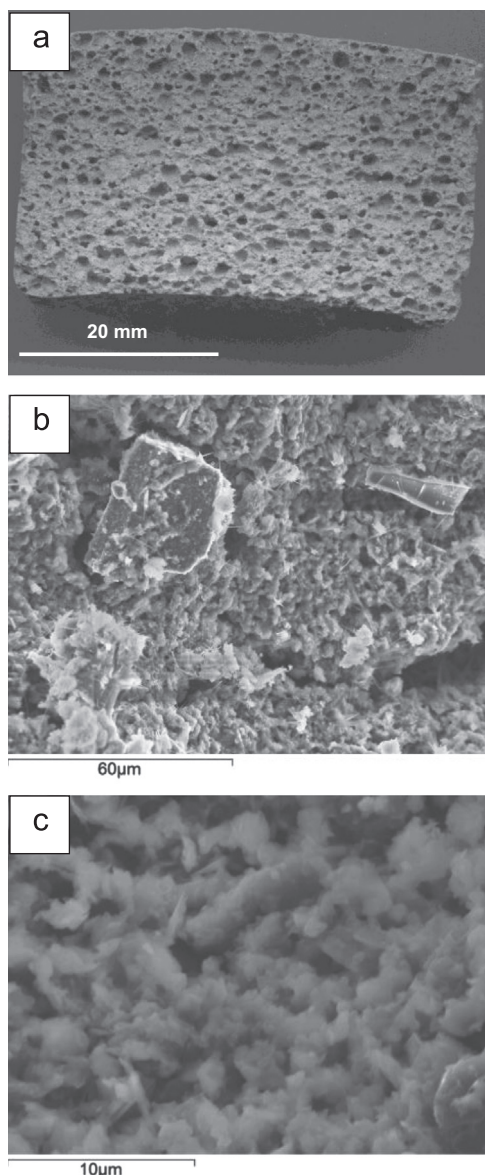


Fig. 1. Cross section (a) and fracture surfaces (b, c) showing macro- and micro-structures of the as-consolidated foam.

After washing, the material pH in wet condition decreased from 9 to 6. After SBF treatment the microstructure changed (Fig. 2). Water soluble needle-like carbonates observed in the as-consolidated foams (Fig. 1c), due to an excess of alkaline solution to promote foaming, disappeared and softer nanoparticles aggregates were observed (Fig. 2c). Dendritic phases formed on ultra-macro-pores surface (Fig. 2d) and different atomic compositions in bulk (fracture) material, pores' wall surface and dendrites were detected (Table 1).

The dendrites were enriched in Ca, Mg, P and Cl and also slightly in C compared to the macro-pores' wall surface, which in turn was strongly lower in C content compared to the bulk (mainly SiC) material. The formation of dendrites of carbonate substituted hydroxyapatite has been already reported in literature for glass ceramics systems containing Mg [21]. The Sr amount was undetectable on dendrites probably due to low availability. Besides apatite precipitation, ion exchange of geopolymeric Na⁺ possibly occurred with SBF ions (Sr, Ca), since the alkali cations balancing the negative charge of AlO₄ tetrahedra in the geopolymer structure can be replaced by monovalent or divalent cations [22]. The low content of Na and P detected on the pore surface respectively compared to the fracture and the dendrite, besides the detected localizations of Sr and Ca, support such hypothesis. The Na-based geopolymer was chosen instead of the K-based [5,6], since SBF contains high amount of Na⁺ and a preferential exchange with K⁺ would occur. XRD (Fig. 3a) revealed, beside moissanite and quartz belonging to SiC and the geopolymer amorphous hump, signals imputable to low crystalline calcium phosphates, hydroxylapatite and traces of sodium chloride. The detection of signals of very low amount of quasi-amorphous calcium phosphates is quite complex since the main crystallographic peaks fall in the range of the amorphous hump. As for comparison Fig. 3b and c shows the very broadened XRD spectra of as synthesized nanoapatites respectively nucleated in SBF solution [19], and partially substituted with silicate and carbonate [23].

The biomimetic calcium phosphates nucleated on the biomaterial through a SBF treatment before implantation, could be *per se* useful, thanks to the *in situ* release of its constituting ions, which have specific biological functions improving bone metabolism, quantity and quality of new forming bone and collagen synthesis; Sr in particular has anti-osteoporotic properties [14–17]. Thus we used concentration of Sr²⁺ (0.20 mM) higher than in human blood serum (0.22 μM [24]). In our strategy, the selected Sr²⁺ amount in the SBF, in term of Sr/Ca molar ratio (0.20) aims to approach the Sr/Ca value (0.10 [25]) that hypothetically characterizes the physiological fluid at the interface with bone in patients under anti-osteoporotic treatment. In fact a Sr/Ca=0.20 was able to nucleate poorly crystalline apatites with a Sr/Ca=0.10 [15,16]. However, to assure the availability of Sr to nucleate Sr substituted biomimetic apatite, the Sr amount possibly involved in exchange with Na ions should be compensated. Thus, an optimized pre-conditioning treatment of the foam would allow the completion of the Sr exchange for Na in the alkali bonded SiC before implantation and avoid any depletion *in situ* of Sr supply to new forming bone.

4. Conclusions

SiC foams are realized by alkali bonding of powders. SiC is able to auto-induce gas blowing in the synthesis conditions avoiding the need of burning out of organic foaming additives or sacrificial templates. Moreover, no heat treatments are needed to decrease the alkalinity related to geopolymers, but simple water washing. The nucleation of multi-substituted biomimetic apatite on the alkali bonded foams, from Sr-enriched SBF, was demonstrated as

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