Fabrication of dense SiSiC ceramics by a hybrid additive manufacturing process

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Abstract

In this work, we report the fabrication of Silicon infiltrated Silicon Carbide (SiSiC) components by a hybrid additive manufacturing process. Selective laser sintering of polyamide powders was used to 3D print a polymeric preform with controlled relative density, which allows manufacturing geometrically complex parts with small features. Preceramic polymer infiltration with a silicon carbide precursor followed by pyrolysis (PIP) was used to convert the preform into an amorphous SiC ceramic, and five PIP cycles were performed to increase the relative density of the part. The final densification was achieved via liquid silicon infiltration (LSI) at 1500°C, obtaining a SiSiC ceramic component without change of size and shape distortion. The crystallization of the previously generated SiC phase, with associated volume change, allowed to fully infiltrate the part leading to an almost fully dense material consisting of β-SiC and Si in the volume fraction of 45% and 55% respectively. The advantage of this approach is the possibility of manufacturing SiSiC ceramics directly from the preceramic precursor, without the need of adding ceramic powder to the infiltrating solution. This can be seen as an alternative AM approach to Binder Jetting and Direct Ink Writing for the production of templates to be further processed by silicon infiltration.

graphical abstract

Keywords

Additive manufacturing, SiSiC, SLS, PIP, LSI, infiltration, preceramic polymer.

INTRODUCTION

Ceramic materials are widely used in applications where the components operate at high temperature (above 1000°C), such as industrial burners, solar absorbers, heat exchangers, heat storage systems and energy plants[1-4]. Such materials suffer high thermal and oxidative stresses during their operation, and therefore they must meet several requirements such as good strength, high temperature resistance, high thermal shock resistance and oxidation resistance[5-8]. Reaction

bonded Silicon Carbide (RB-SiC or SiSiC), also known as siliconized Silicon Carbide or silicon infiltrated Silicon Carbide, is widely used in several engineering applications where endurance and thermal stability is required[9-11]. In recent years, the new challenge is to build SiSiC parts through additive manufacturing (AM)[12-16], in order to exploit the excellent properties of this material combined with the possibility to obtain complex architectures[17-20], not achievable with the traditional manufacturing approaches (reaction sintering, hot pressing sintering, pressureless sintering and other techniques). Several components have been successfully manufactured by combining 3D printing with liquid silicon infiltration, which involves the infiltration of a carbon (C) porous preform with molten silicon (Si) at a temperature exceeding its melting point (1414°C)[21,22]. Several types of preforms can be manufactured via different AM approaches: binder jetting[22], direct or indirect selective laser sintering[24,25], laminated object manufacturing[26], robocasting[14], extrusion free forming[22], direct ink writing and stereolithography[27]. The drawbacks of these approaches are the low relative density, low mechanical strength and coarse resolution of the parts with respect to the traditional approaches. Several studies have been performed in order to investigate the nature of the residual porosity after the LSI, and it was found that the infiltration process is not controlled by viscous flow but by the reaction at the infiltration front. The reaction between C and Si causes the formation of a dense surface which might limit subsequent infiltration from the liquid, leading to the presence of residual porosity in the part[28,29].

In this work, we aimed at introducing a hybrid fabrication approach based on AM for the manufacturing of nearly fully dense, net-shape SiSiC components. This study is focused on the development of the bulk material, and we also assessed the microstructure and mechanical properties of the produced component. This method exploits the selective laser sintering (SLS) of a polymeric preform combined with few cycles of polymer infiltration and pyrolysis[30] (the combination of these two methods was investigated in the authors' previous work[31]), followed by liquid silicon infiltration. Thanks to SLS 3D printing, it is possible to produce geometrically complex shapes and to maintain their micro details after the polymer-to-ceramic transformation. This occurs through the choice of the printing parameters, which allows controlling the relative density of the printed part and therefore the infiltration rate and the shrinkage after the first pyrolysis cycle. The final densification is achieved via LSI of the solid amorphous SiC network derived from the pyrolysis of the preceramic precursor. During LSI at high temperature, the SiC phase crystallizes and shrinks allowing for a full infiltration by molten Si.

The notable result is the possibility to obtain almost fully dense ceramic parts with the crystalline SiC phase deriving directly from the preceramic precursor, without the addition of any ceramic powder. This can overcome several processing problems that have been found in other AM approaches, such as binder jetting.

The author's future work will focus on investigating the precise role of molten silicon in the crystallization of the SiC phase, which could be driven by several phenomena, with the aim of minimizing the amount of residual Si.

EXPERIMENTAL PROCEDURE

Commercially available polyamide (Nylon12) powders (PA12, Sintratec AG, Brugg, Switzerland) were used to manufacture highly micro porous polymeric preforms (Sintratec KIT, Sintratec AG, Brugg, Switzerland). The selection of the proper printing parameters (laser speed, layer thickness and powder surface temperature) allowed to control the micro porosity of the bulk material because they affect the sintering rate of the powders[31]. The purpose of this phase was to manufacture a polymeric preform with low relative density (with respect to the particle density of 1.0 g/cm^3) in order to subsequently infiltrate the residual porosity with a preceramic polymer. The printed preforms had a disc shape, with a diameter of 30 mm and a height of 6 mm.

The polymeric preforms were then infiltrated with allylhydridopolycarbosilane (AHPCS) preceramic polymer (StarPCS™ SMP-10, Starfire Systems Inc, Glenville, NY, USA). AHPCS forms amorphous SiC when pyrolyzed at 850-1200°C in inert atmosphere, with a ceramic yield of 72-78 wt%. Nano-crystalline β-SiC forms upon firing at temperature higher than ~1250°C. The infiltration process was performed in light vacuum at room temperature. After infiltration, the samples underwent pyrolysis at 960°C in flowing Argon (99.99%, 30 l/h) in order to obtain the polymer-to-ceramic transformation. PIP were repeated six times to obtain a high degree of densification of the samples and a high amount of SiC phase.

Silicon grains (HQ1, Sicerma, Erkelenz, Germany) with a grit size of 0.2-2.0 mm were then used to perform LSI of the ceramic discs in vacuum atmosphere (10^{-2} mbar) at 1500°C for 1/2 h, with a heating rate of 20°C min^{-1}. LSI was used to fill the residual porosity and to increase the mechanical strength of the material. Figure 1 shows the schematic of the overall hybrid AM process to fabricate SiSiC components. For comparison, a preform infiltrated only with allylhydridopolycarbosilane was also heated at 1500°C in the same conditions as for the Si infiltration.

The density of the samples was measured by means of a gas pycnometer, using Helium (Ultrapyc3000, Anton Paar QuantaTec Inc., Florida, USA). A bulk sample piece and sample powder were used for obtaining the apparent and true density values, respectively.

Mercury intrusion porosimetry (MIP) tests (PoreMaster 60, Anton Paar Switzerland AG, Buchs, Switzerland) were performed in order to evaluate the bulk porosity of the samples after SLS, each PIP cycle and LSI. Analyses were performed by using a pressure range from 0.0014 MPa to 414 MPa.

Scanning electron microscopy (SEM) analyses (JSM- 6010PLUS/LA, Jeol Ltd., Japan) were carried out to investigate the microstructure of the samples obtained from the fractured ceramic discs after B3B tests (see below).

The phase assemblage of the parts was investigated on sample powder, using an X-ray diffractometer (D8 Advance, Bruker Italia Srl, Milano, IT) with Cu(kα) radiation, from 10° to 80°, 0.05°/step, 2 s/step. The Match! software package (Crystal Impact GbR, Bonn, Germany) was used for a semi-automatic phase identification, supported by data from the PDF-2 database (ICDD-International Centre for Diffraction Data, Newtown Square, PA, USA).

The mechanical strength of the ceramic samples was evaluated through biaxial flexural tests (Zwick Z050, Zwick GmbH & Co.KG, Ulm, Germany) employing the ball-on-three-balls (B3B) setup,

using stainless steels balls of 13 mm diameter. Tests were performed at a strain rate of 10^{-3} s^{-1} and a cell load of 5kN (KAP- S, AST, Dresden, Germany) was used to record the reaction force. A Poisson's ratio of 0.2 was assumed. Five samples were tested for each PIP cycle and after LSI.

RESULTS AND DISCUSSION

The different combination of the printing parameters allowed to achieve very different relative density of the polymeric preforms. In general, the relative density decreased with increasing laser speed, decreasing temperature and increasing layer thickness. However, the decreasing in the relative density led to a significant decrease in the mechanical strength and handleability of the parts. The SLS process was already extensively optimized as published in our previous work[31]. The optimal combination to manufacture the preforms was: laser speed of 848 mm/s, layer thickness of 100 μm and powder surface temperature of 166°C.

The PA12 disc preforms, with a relative density of 0.53, were subsequently infiltrated with the preceramic polymer and pyrolyzed resulting in porous SiC ceramic discs, with 23 mm diameter, 5 mm height and relative density of 0.30. The samples underwent a linear shrinkage of 24% without shape distortion and macroscopic cracks formation. This is due to the PA12 degradation upon heating, that formed pores inside the part. Furthermore, it was found that the relative density of the polymeric preform had an effect on the shrinkage after the first pyrolysis: decreasing the relative density increased the infiltrated volume of AHPCS in the preform, and consequently the shrinkage of the ceramic part decreased[30]. For example, a shrinkage of 5% was obtained by using a polymeric preform with relative density of 0.40. This means that by regulating the printing parameters, it is possible to control the size of the resulting ceramic part. The densification of the ceramic discs was performed by re-infiltrating five times with AHPCS and then pyrolyzing at 960°C after each infiltration. The ceramic part, after six infiltration and pyrolysis cycles, had an apparent density of 2.043 ± 0.001 g/cm^3, a true density of 2.403 ± 0.014 g/cm^3 and a relative density of 0.85 ± 0.005 (with respect to the true density), meaning that the material was not fully dense and therefore micro-sized pores and cracks were still present.

In order to further increase the relative density and to increase the mechanical strength of the material, the samples after six PIP cycles then underwent liquid silicon infiltration. The LSI produced a ceramic part without change of size and shape distortion. The final SiSiC discs had an apparent density of 2.672 ± 0.015 g/cm^3, a true density of 2.718 ± 0.015 g/cm^3 and a relative density of 0.983 ± 0.011. The residual porosity was, therefore, only ~1.7 vol%.

Figure 1A shows the SEM images of the fracture surface of the PA12 sample, the SiC samples after the first and the last PIP cycle and the SiSiC sample after LSI. The microstructure of the 3D printed sample (Fig. 1A) showed that the PA12 particles formed small agglomerates and therefore, in the selected experimental conditions, they did not melt but were partially sintered just enough to hold together. This allowed for the formation of large number of interconnected pores inside the polymeric preform, which could be subsequently infiltrated by the preceramic polymer. After the first pyrolysis, the AHPCS was converted into SiC (light grey area in Fig. 1B), while the PA12 particles completely decomposed at ~520°C[31], generating additional pores (dark areas in Fig. 1B - pores were

filled with a resin to enable polishing). The newly formed porosity as well as the porosity deriving from the shrinkage occurring in the AHPCS phase due to the polymer-to-ceramic conversion, were then infiltrated again 5 times using the same preceramic polymer, leading to a reduction of the volume fraction of pores as well as an increase of the amount of amorphous SiC phase. Fig. 1C shows that after six PIP cycles there was still some residual porosity (about 15 vol%, dark grey areas) in the material, due to incomplete infiltration and the fact that the infiltrated preceramic polymer shrinks upon pyrolysis. After LSI at 1500°C (Fig. 1D), the SiC phase (dark grey areas) appeared to have shrunk by about of 53 vol% with respect to Fig. 1C, as assessed by image analysis. This phenomenon actually occurred during the LSI process, enabling for a complete infiltration of the part by the molten silicon, resulting in an almost fully dense ceramic part. Image analysis carried out in order to distinguish and quantify the two phases observable in Fig. 1D (porosity was not identifiable), indicate that SiC and Si were present in a volume fraction of 45% and 55%, respectively. SEM micrographs with higher magnification are provided in the supplementary material.

Figure 2 shows the comparison between the SEM micrographs of: (A) a SiSiC sample after LSI at 1500 °C and (B) a SiC sample heated at 1500°C without Si infiltration. In Fig. 2A, the full crystallization of the amorphous matrix into the β-SiC phase is visible due to the faceting of the crystals' boundary (dark grey areas), and the full infiltration by the Si phase can also be observed (light grey areas). Fig. 2B shows a different microstructure, due to the absence of Si in the material pores and the non complete crystallization of the SiC phase. This means that the transformation of amorphous SiC into β-SiC occurred in contact with the molten Si, which while infiltrating the pores raises the part temperature very quickly during the reaction (few seconds), favouring crystallization (see the process schematic in Figure 3). This aspect will be further investigated in the authors' future work. The non-infiltrated sample, after heating at 1500°C, had an apparent density of 2.993 ± 0.036 g/cm^3 and a true density of 3.206 ± 0.099 g/cm^3, in very good agreement with the theoretical density of SiC (3.210 g/cm^3) and indicating the very limited presence of residual carbon in the material. The relative density was 0.934 ± 0.040 g/cm^3 and the residual porosity was, therefore, only ~6.6 vol%. After the heating cycle, the sample underwent a linear shrinkage of ~7.4%, which did not occur in the Si infiltrated sample. SEM micrographs with higher magnifications and EDS mapping are provided in the supplementary material.

Figure 3 shows the schematic of the LSI process and SiC crystallization. The porous and amorphous SiC derives from the PIP cycles (A), and it is then infiltrated with molten Si at 1500°C (B). During this step, SiC starts crystallizing and shrinks, opening the way to further infiltration by molten silicon (C). The infiltration proceeds inside increasingly smaller pores, while the amorphous SiC phase concurrently fully crystallizes into β-SiC. The Si does not react with any other elements (there is only a very negligible amount of free carbon inside the preform), which means that the process does not produce reaction bonded SiC, but rather Si infiltrated SiC.

XRD analysis of the samples (Fig. 4A) demonstrated that amorphous SiC crystallized into β-SiC during LSI, and this is the reason for the observed volume shrinkage of that phase. The phase assemblage of the sample after pyrolysis (black line, 6 PIP cycles) was comprised by amorphous SiC and a very limited amount of graphitic carbon, while that for the silicon infiltrated sample (red line, 6 PIP + LSI) showed also the presence of Si, besides well crystallized β-SiC and some graphitic carbon. As expected, the XRD analysis of the preform sample infiltrated only with allylhydridopolycarbosilane (no Si infiltration) heat treated at 1500°C showed only the SiC phase,

which appears to comprised by smaller crystallites (broader peaks) with respect to that formed during the Si infiltration and containing also some stacking faults (shoulder at ~33.9°), as well as probably some residual amorphous phase (blue line, 6 PIP heated at 1500°C).

The amorphous SiC phase therefore appears to have been completely eliminated from the sample infiltrated with Si (see inset). Indeed, considering that crystalline SiC and Si have a density of 3.210 g/cm^3 and 2.329 g/cm^3 respectively, it was possible to calculate the density of the SiSiC part by using the respectively volumetric fractions, as determined by image analysis. The calculated density was 2.705 g/cm^3, in very good agreement with the measured true density value of that sample (2.718 ± 0.015 g/cm^3).

Figure 4B shows the biaxial flexural strength of the samples as a function of their relative density. As expected, the average strength and the relative density increased with increasing of the number of PIP cycles. After the first pyrolysis the strength was very low (7±0.48 MPa), increasing to a more than three times higher value (23±3.66 MPa) after six PIP cycles. The samples after LSI showed a significant increase both in strength (127 MPa) and relative density (98.3%). Table 1 shows the comparison between SiSiC ceramics fabricated by AM technologies combined with LSI. By taking into account the effective volume and the defects population, the bending strength results coming from different testing methods can be compared. In general, B3B tests show the smallest effective volumes and therefore highest strength values compared to 3- and 4-point bending[32,33]. This means that the SiSiC material developed in this work should possess a lower strength than the SiSiC ceramics reported in literature, when tested with 3- or 4-point bending. The difference is certainly attributable to the silicon content in the samples, which is higher (2 to 8 times) than in the other ceramic materials.

The strength of the material developed in this work could be further increased through the optimization of the process, e.g. by increasing the volume fraction of SiC with respect to the Si phase. This could be achieved, for instance, either by infiltrating the preform with AHPCS containing also SiC particles (especially for the first PIP cycle), or by infiltrating the preform with phenolic resin (for the last PIP cycle) in order to generate crystalline SiC through the reaction between residual C (derived from the pyrolysis of the phenolic resin) and molten Si provided by LSI, or by printing a bed comprised of both PA12 and SiC particles thereby generating a preform already containing a high amount of crystalline silicon carbide phase.

CONCLUSIONS

The fabrication of SiSiC components was successfully achieved through a hybrid additive manufacturing technique. The notable result is the possibility to obtain almost fully dense ceramic parts with the crystalline SiC phase produced directly from the preceramic precursor, without the need of adding ceramic powders to the infiltrating solution which would hinder its diffusion into the preform. The process involved the SLS 3D printing of a polymeric preform with the desired shape and relative density. The preform was infiltrated with a preceramic precursor and pyrolyzed (up to six times) in order to obtain amorphous SiC through the polymer-to-ceramic transformation, and to increase its relative density. The obtained porous part was then infiltrated with molten silicon at

1500°C to produce the final SiSiC ceramic component without change of size and shape distortion. Crystalline SiC and Si composed the final nearly fully dense (98.3%) ceramic part with a volume fraction of 45% and 55%, respectively. However, the limited biaxial strength (127 MPa) can be further increased though the optimization of the process. Furthermore, this novel technique allows for the manufacturing of geometrically complex components with very small features (such as cellular architectures), which will be produced in a future work.

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Supporting Information

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AM method	Density	Rel. density	Si content	Strength	Test method	Ref.
-	g cm^{-3}	%	%	MPa	-	-
Binder Jetting + LSI	3.05	~100	15-25	358-380	B3B	13
Robocasting + LSI	2.94	98	23	224	4-point	14
Extrusion FreeForming + LSI	-	-	-	300	3-point	22
SLS + Cold Isostatic Pressing + LSI	2.96	-	7	292-348	3-point	24
SLS + LSI	2.69	-	-	200	3-point	25
Laminated Object Manufacturing + LSI	2.60	-	-	315	4-point	26
Binder Jetting + LSI	2.49	91	41	245	4-point	34
SLS + PIP + LSI	2.718	98.3	55	127	B3B	This work

Table 1: Comparison between SiSiC ceramics fabricated by different AM technologies combined with LSI.

Selective Laser Sintering 3D printing → Preceramic precursor infiltration and pyrolysis → Additional infiltration and pyrolysis (5 cycles) → Liquid silicon infiltration

A B C D

A B

Silicon intruding large pores

Silicon intruding new pores

A

B

C

D

Amorphous SiC

Partially crystallized SiC

β-SiC

A

Intensity (a.u.)

◆ Si
● β-SiC
✳ C

Intensity (a.u.)

2θ (degrees)

2θ (degrees)

B

Strength [MPa]

■ 1 PIP
▲ 2 PIP
● 3 PIP
◆ 4 PIP
□ 5 PIP
△ 6 PIP
■ 6 PIP + LSI

Relative Density [-]

Selective Laser
Sintering 3D printing → Preceramic precursor
infiltration and
pyrolysis → Additional infiltration
and pyrolysis
(5 cycles) → Liquid silicon
infiltration

200μm

200μm

200μm

200μm