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Manufacturing and Characterization of Interpenetrating SiC Lightweight Composites

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Abstract

The current work deals with the gas pressure infiltration of SiC - preforms of selected porosities with an aluminum alloy in order to manufacture an interpenetrating composite with higher ductility in comparison to SiC bulk material and a higher temperature and creep resistance in comparison to aluminum bulk materials. The quality of the manufactured composite is analyzed metallographically which attests a good infiltration of the composite. The residual porosity is also determined and can be attributed to the closed porosity and insufficient infiltration of open porosity. It can be shown that the infiltration of the preform leads to an increase in compressive strength with reasonable ductility in comparison to the unreinforced matrix material.

1. Introduction

Metal Matrix composites with a three dimensional interpenetrating reinforcing network offer enhanced mechanical properties in comparison to particle reinforced composites at both room and elevated temperatures [1]. Topology and volume fraction of the reinforcement phase play deciding roles in controlling the properties of the composites [2].

Based on a preliminary study carried out by [3] a simple approach to fabricate open porous SiC preforms by using polyethylene wax as pore formers is reported in detail in [4-5]. Two wax varieties (Viscowax and Ceridust) with very different particle sizes were used in different quantities to fabricate SiC preforms with different porosities in the range 45–65 vol.% and varying pore morphologies.

In [4-5] the elastic properties of uninfiltrated SiC preforms with different reinforcing amount and a variation in the wax ratio have been determined by using ultrasound phase spectroscopy. The degree of anisotropy depends both on the amount of porosity and the pore structure. For the complete range of porosity studied, preforms fabricated using a mixture of the two wax types show less anisotropy than the preforms fabricated using only one type of pore former. A high volume fraction of smaller pore formers at lower porosities and a high volume fraction of larger pore formers at higher porosities lead to preforms with optimum isotropic elastic properties [5].

Infiltration casting with additional gas pressure is a unique method to force a liquid metal into a preform. In [6] an overview of different techniques for casting composites with their pros and cons is presented.

The current work deals with the manufacturing and infiltration of preforms of selected wax ratios at constant SiC to wax ratio of 50:50. The investigations include gas pressure infiltration [7] of the manufactured preforms resulting in a MMC with an interpenetrating reinforcing network. Beside the determination of the residual porosity also the quasistatic mechanical properties under

compressive load are determined in order to compare different production parameters. Optical analyses confirm different types of fracture evolution in dependence of the chosen wax ratio.

2. Experimental procedure

2.1. Manufacturing of porous SiC

Figure 1 gives an overview of the steps for manufacturing porous preforms with the help of two waxes. The fabrication includes the mixture of the silicon carbide powder (Amperpress) with a particle size of < 38 μm (35 %), > 150 μm (3 %) with two polymer waxes - Viscowax (VW with a particle size of 250 - 630 μm) and Ceridust wax (CD with a particle size of 7.5 - 9.5 μm) - to customise the structure of the preform. Within this work only wax powder mixtures of volume fraction of 50:50 (SiC:wax mixture) have been manufactured. The wax ratio has been varied between VW:CD = 1:2, 1:1 and 2:1.

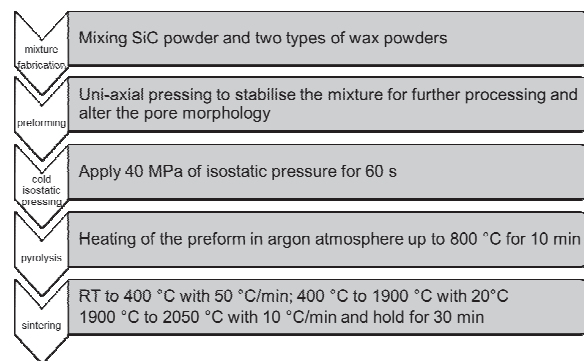|	
mixture fabrication	Mixing SiC powder and two types of wax powders
preforming	Uni-axial pressing to stabilise the mixture for further processing and alter the pore morphology
cold isostatic pressing	Apply 40 MPa of isostatic pressure for 60 s
pyrolysis	Heating of the preform in argon atmosphere up to 800 °C for 10 min
sintering	RT to 400 °C with 50 °C/min; 400 °C to 1900 °C with 20°C 1900 °C to 2050 °C with 10 °C/min and hold for 30 min

Fig. 1. Principle of preform manufacturing

The second step includes the filling of a bottom die and the pressing of the specimens with a uniaxial pressure of 40 MPa. Vacuum packaging was done to prevent contamination of the specimen with oil during cold isostatic pressing in order to gain a relatively cylindrical pore structure. Pyrolysis (in argon atmosphere) leads to melting and extraction of the wax out of the manufactured preforms in order to receive a porous structure. Afterwards the preforms with a porous architecture were sintered in argon atmosphere at a maximum temperature of 2050 °C. Further details about the manufacturing procedure of the preforms can be found in [4-5].

2.2. Gas pressure infiltration

The principle of the gas pressure infiltration process is shown in Figure 2. The preforms (1) have been placed, together with ingots of AlSi12 (2), into an alumina crucible (3). The chemical composition of the AlSi12 matrix material can be found in table 1.

Table 1. Measured chemical composition of AlSi12 in wt%

Si	Fe	Mg	Mn	Ni	Ti	V	Al
12.99	0.127	0.026	0.035	0.004	0.023	0.0101	Bal.

The crucible was placed into the infiltration chamber (4) which was sealed (5). The test started by evacuating the chamber up to a vacuum pressure of about 0.04 mbar (6). Afterwards heating (0.15 K/s) was started using an induction coil (and a mid-frequency generator) (7) and a graphite susceptor (8) for efficient coupling.
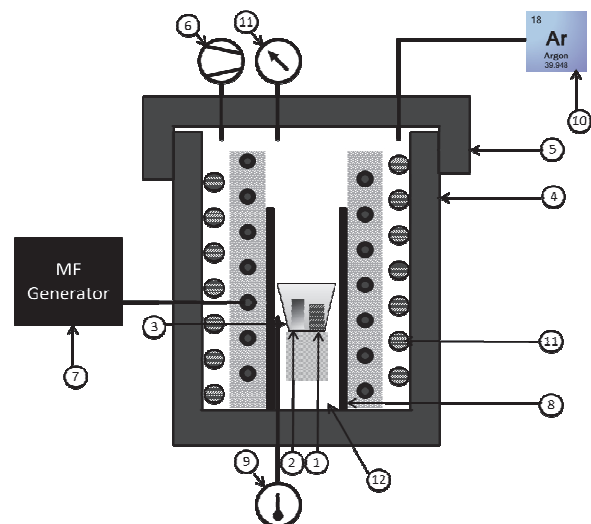

Fig. 2. Scheme of the gas pressure infiltration process

After holding the aimed temperature (9) of 720 °C for 1 h, Argon gas (10) was then applied to pressurize (20 bar) (11) the infiltration chamber. Afterwards pressure was kept constant during cooling the chamber by water circulation (11) and copper heat sink (12).

2.3. Metallographic preparation

Cutting of the infiltrated preforms has been done on a diamond wire saw. The specimens for the qualitative metallographic investigations and for the compressions tests were grinded and polished in several steps with water as coolant. As abrasive paper SiC paper with graining 600-1000-2500-4000 was used.

2.4. Mechanical analysis

The compression tests have been carried out displacement controlled on a Universal testing machine Zwick with a maximum capacity of 500 kN at a constant nominal strain rate of 8.4 10^{-4} 1/s. The experimental setup is shown in Figure 3.
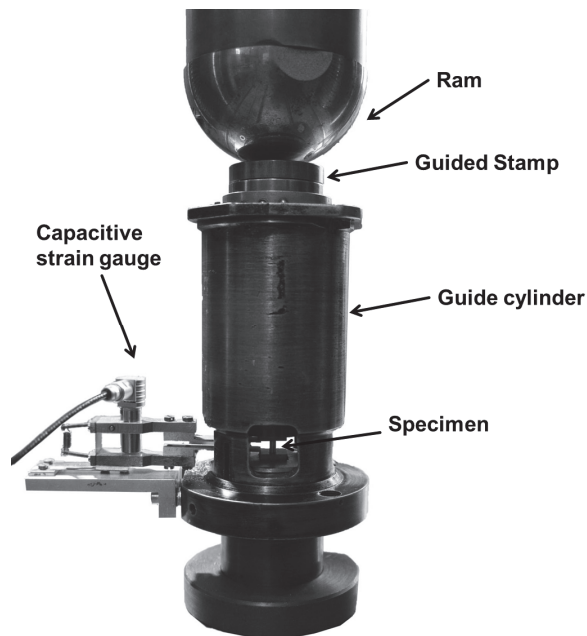

Fig. 3. Experimental setup of the compression test

The strain has been measured using a capacitive measuring gauge. The parallelepiped compression specimens had the dimensions of about 5x5x5mm³, where each specimen has been measured in detail. For each production parameter, three specimens have been tested. The compression direction was perpendicular to the direction of the uni-axial pressing since stiffness is higher compared to the pressing direction [4]. The cylindrical specimen of the reference material AlSi12 had the size hxd = 6 mm x 3 mm.

3. Results

3.1. Metallographic investigations

Figure 4 shows representative micrographs of three different infiltrated preforms. For all specimens the large infiltrated pores can be seen which is observable by the bright aluminum alloy with eutectic structure and constituents of iron and aluminum. Darker (black) areas suggest that these are not well infiltrated showing residual porosity. Specimens with a high content of CD show a good infiltration of the small porous areas which resemble to canals. In contrast, specimens with a high content of VW show a good infiltration of the large pores and a higher amount of residual porosity.
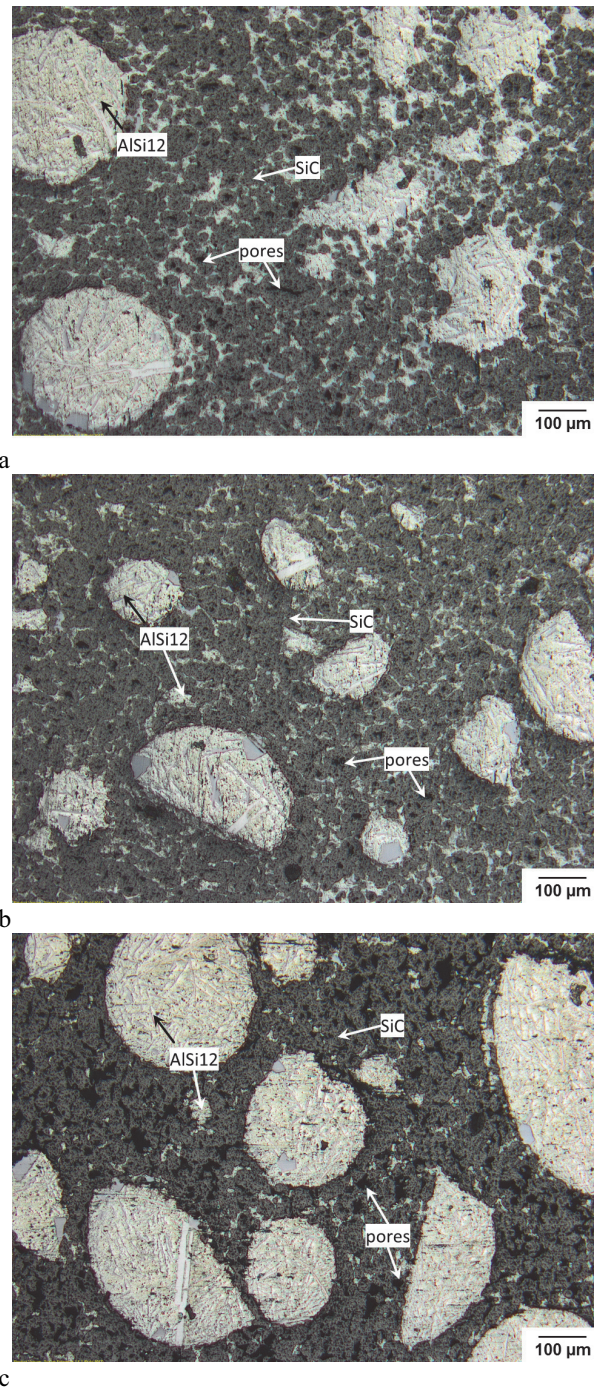

Fig. 4. Representative micrographs of the infiltrated SiC preforms (a) VW:CD = 1:2; (b) VW:CD = 1:1; (c) VW:CD = 2:1

Figure 5 summarises the amount of porosity – the preform density ρ was determined following Archimedes principle, by immersing in distilled water - of the sintered preforms and the infiltrated specimens for all

wax mixtures. The total porosity (average of three specimens per VW:CD ratio) was calculated following the relation:

$$\left(V_P = 1 - \frac{\rho}{\rho_{SiC}} \right) \qquad (1)$$

ρ_{SiC} represents the density of SiC.

The highest closed porosity is existent for preforms with a wax ratio VW:CD of 1:2. For an equal wax ratio and a higher amount of CD the closed porosity is comparable. In all preforms the amount of total porosity is 2.5-5 vol.% less than the total wax content in the initial mixture (50 vol.%). This may be attributed to the sintering effect (shrinkage of the preform).
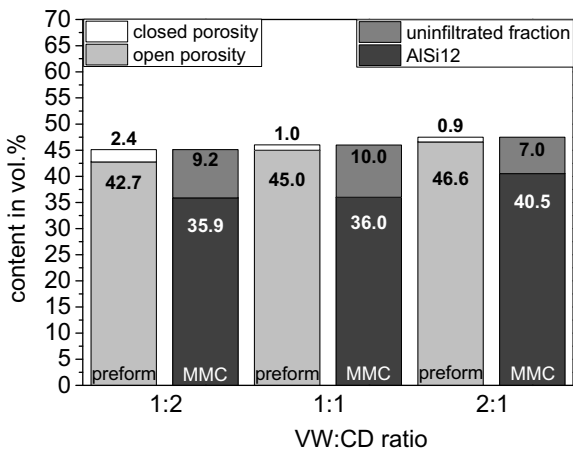

Fig. 5. Measured average porosity and average infiltration content

The MMC denoted beams represent the infiltrated specimens. It can be summarized that none of the open porosity of all three mixtures was completely infiltrated with the parameters used. It is evident that closed porosity is not infiltratable.

By comparing the fractions of uninfiltrated open porosity, VW:CD of 1:1 shows the highest lack of infiltration of about 9 vol.% in comparison to the other mixtures of about 6.8 vol.% (VW:CD of 1:2) and of about 6.1 vol.% (VW:CD of 2:1).

3.2. Mechanical properties

Figure 6 shows representative engineering stress-strain curves of three MMCs in comparison to the matrix material AlSi12. All three curves show an initial progressive increase in stress which can be attributed to the deviation of the sample geometry from perfect parallelism and friction between the specimen and the punches (setting effect). After reaching a maximum

strength, which is higher than that of the matrix material at given strains, a decrease in stress is noticeable due to the partial collapse of the specimens. The maximum strength can be reached at relatively low strains in comparison to AlSi12 which deformation behavior is affected by the buckling of the specimen and therefore a constant increase in stress.
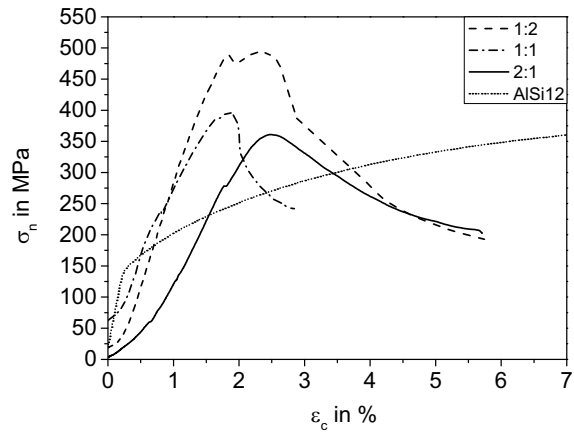

Fig. 6. Representative engineering stress - strain curves of three different specimens in comparison to the matrix material

In order to get an overview of the maximum strength of all investigated specimens, Figure 7 shows the average values and the fluctuations for all three wax compositions.
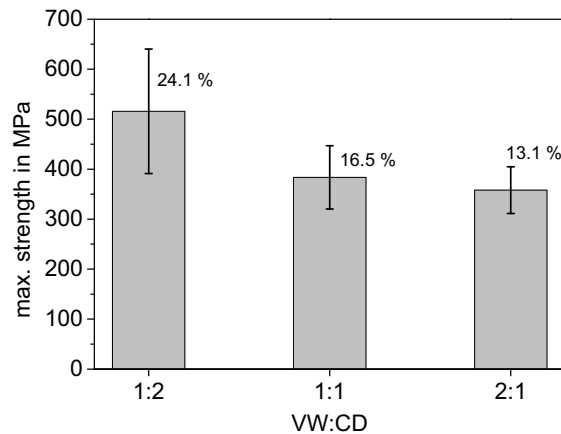

Fig. 7. Average strengths of the three different parameters

It can be concluded that a smaller amount of Viscowax leads to significantly higher strength. In contrast also high fluctuations of about 24 % can be noticed.

The strength of equal ratio and a higher amount of Viscowax are comparable.

3.3. Fractographic investigations

Figure 8 shows representative pictures of three different infiltrated specimens after the compression test.
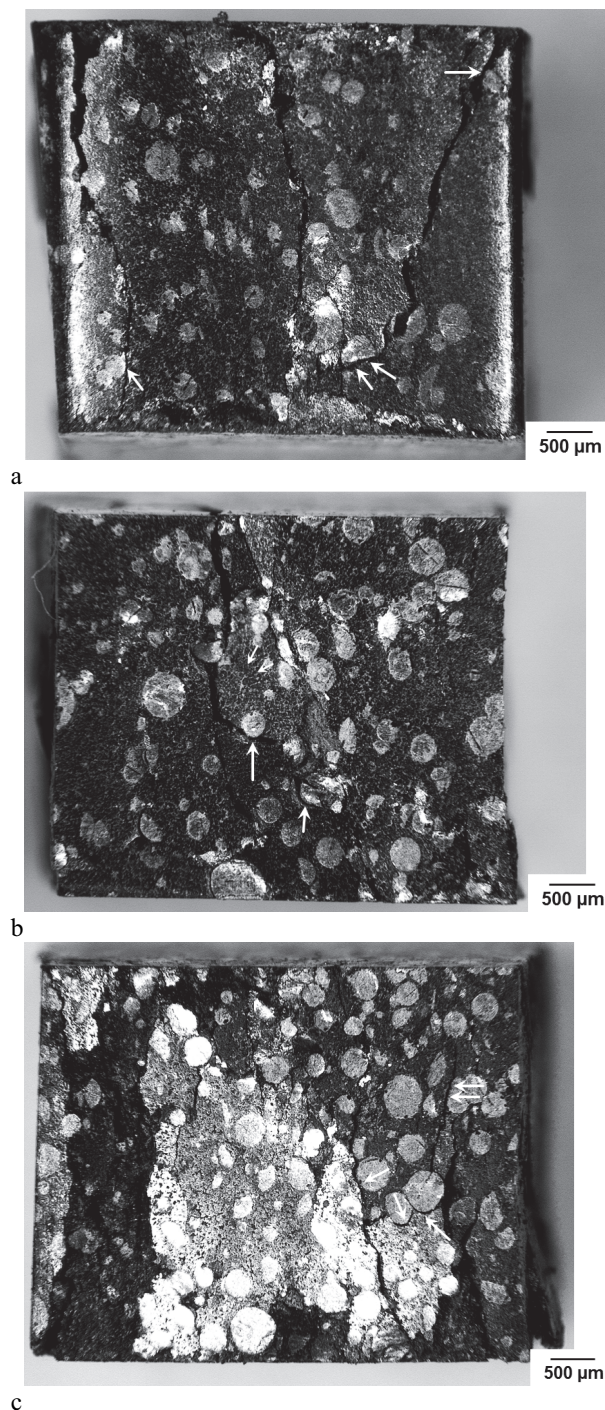

a

b

c

Fig. 8. Representative fractographic pictures after the compression tests (a) VW:CD = 1:2; (b) VW:CD = 1:1; (c) VW:CD = 2:1, loading direction is vertical

In general smaller amounts of larger pores can be seen for specimens with low content of Viscowax.

A high content of Viscowax leads to a more brittle fracture since whole parts of the specimen are fractured. For all specimens crack growth takes mainly place in the SiC than in the metallic matrix material.

Furthermore crack growth can be observed along the interface between ceramic reinforcement and metallic matrix, which is depicted with white arrows.

4. Discussion

The metallographic investigations of the infiltrated preforms show that an increase in amount of Viscowax results in an architecture represented by a few large spherical pores accompanied with a fine network of small canal-like pores. The latter may be explained by preform manufacturing. During heating the wax melts and expands and this gives rise to cracks between the large pores. These cracks act as channels through which the molten wax flows out [4]. The bimodal pore structure has already been observed in [4-5].

A high amount of Ceridust leads to larger pores but reduced residual porosity in comparison to specimens with higher content of Viscowax which could be measured quantitatively.

The measured uninfiltrated (open) porosity of the MMCs can be lead back to the parameters of gas pressure infiltration used which should be improved. In order to increase the amount of infiltrated areas, temperature should be increased resulting in a higher fluidity of the melt. An additional increase in the Argon gas pressure should allow the melt to penetrate even areas with a finer pore structure.

Preparation of the 5x5x5 mm³ cube specimens is relatively complicated in terms of parallelism of the sides. Therefore initial compression of the specimens concentrated on flattening of the anti-parallel sides [8]. A special specimen holder for metallographic preparation will increase the amount of parallelism so that setting effects in the beginning of the compression tests may be reduced. This would furthermore lead to a better determination of the stiffness of the interpenetrating MMCs.

5. Summary and Outlook

It could be shown that porous SiC preforms with different wax ratios can successfully be manufactured. Furthermore the investigated preforms can successfully be infiltrated via the gas pressure infiltration process. Nevertheless the residual porosity should be reduced via an increase in temperature of the melt as well as an increase in Argon gas pressure. In order to increase strength and stiffness, precipitation hardenable alloys

should be used, where an additional heat treatment (solutionizing, quenching, artificial ageing) may lead to improved mechanical properties. Future work will concentrate on the infiltration of preforms with a variation of the amount of reinforcing phase. In order to achieve detailed knowledge about the damage behavior under compressive loading, optical strain measurements should be done. Furthermore, also the mechanical properties under tensile loading should be analyzed in detail.

Acknowledgment

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