Experimental study and numerical simulation of infiltration of AlSi12 alloys into Si porous preforms with micro-computed tomography inspection characteristics

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Silicon particle preforms with different starch contents (10, 20 and 30 %) and particle sizes (20, 50 and 90 μm) were fabricated by compression mold forming and heat treatment. The pore characteristics of preforms were inspected with a high-resolution (~1 μm) three-dimensional (3D) X-ray micro-computed tomography (μ-CT). The infiltration of AlSi12 alloys into the preforms were carried out under the condition of 800 °C and 400 kPa with different pressure-applied times (3, 8 and 15 s) in a vacuum-assisted pressure infiltration apparatus. A high-resolution (~500 nm) vertical scanning white light interfering profilometer was used to detect the front surfaces of composites. The infiltration was simulated at micro-scale by considering the actual pore geometry from the μ-CT inspection based on the Navier-Stokes equation. The results demonstrated that as the starch content and particle size increased, the front surface area of composite increased. Compared with the starch content, the particle size has more influence on the front surface area. In the simulation, as the infiltration progressed, the pressure of liquid AlSi12 decreased. The residual pores of composites increased with infiltration. According to the experiment and simulation results, a larger pressure drop along the infiltration direction leads to more residual pores of composites.

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Key-words : Pore characteristics, Pressure distribution, Residual pores, Simulation

[Received January 27, 2021; Accepted April 1, 2021]

1. Introduction

Due to their high thermal conductivity, low coefficient of thermal expansion and low density, high volume fraction (55–75 %) particle-reinforced aluminum matrix composites have been proven to be suitable for electronic packaging and aerospace applications.¹–⁴ Compared with other methods, a liquid aluminum infiltration process containing a preparation of porous preforms and an infiltration of liquid aluminum into the preforms can fabricate the composites effectively and economically.⁵–⁷ Recently, researches of infiltration have focused on the effects of particle size,⁸ contact angle,⁹ infiltration pressure,¹⁰ temperature¹¹,¹² and liquid flow.¹³,¹⁴

The liquid aluminum infiltration process is related to flow dynamics in porous materials. Studies of free surfaces and capillary forces have been key factors.¹⁵,¹⁶ Under non-wetting conditions, the free surface is the resistance to infiltration fluid, which has attracted great attention from researchers, in terms of particle-fluid flow,¹⁷ surface tension¹⁸ and oscillation¹⁹ of the free surface. Meanwhile, the pressure is a key parameter for the momentum of any fluid. Since the infiltration of preform is a liquid flow in micropores, the experimental measurement of its flow parameters, such as pressure and velocity, is very challenging. Therefore, simulation has become an effective approach for investigating the infiltration behavior in such cases, mainly on the basis of Darcy flow and non-Darcy flow.²⁰,²¹ The analysis of infiltration behavior considering actual internal characteristics is desirable in the simulation.²²,²³ Currently, the development of three-dimensional (3D) micro-computed tomography (μ-CT) technology has led to the study of mechanics and fluid dynamics at microscale by considering the material internal characteristics.²⁴–²⁸ The pores in preforms have been well characterized in our previous study.²⁹,³⁰ However, the simulation of micro-scale flow based on actual geometric models of preforms has not been reported to date. The numerical simulation of infiltration at micro-scale remains a challenging and complicated task.

In our previous study, the effect of pore characteristics on the infiltration height was discussed, and a modified infiltration equation considering the minimum areal porosity fraction and tortuosity was proposed to calculate the height.³¹ In this paper, the purpose is to study the effect of...
pore characteristics on the infiltration behavior at microscale. The pore characteristics of Si preforms were detected with a high-resolution 3D X-ray μ-CT. The preforms were infiltrated in a vacuum-assisted pressure infiltration apparatus. The front surface areas of the composites were measured with a high-resolution white light interfering profilometer. The effect of pore characteristics on the front surface was calculated and discussed. The simulation of infiltration based on the Navier-Stokes equation was performed by considering the actual pore 3D characteristics. By comparing with the experiment results, the calculated pressure distributions and their effects on the residual pores of composites were discussed.

2. Materials and experiments

2.1 Si preforms and 3D μ-CT inspection

Commercial Si particles with purity exceeding 99.9% and average sizes of 20, 50 and 90 μm were used to prepare the porous preforms. Industrial acid-modified starches (average size 15 μm) and silica sols were selected as pore-forming agents and binders, respectively. The characteristics of Si particles were measured with a Malvern Panalytical Mastersizer 2000 laser particle analyzer. The particle size distributions are plotted in Fig. 1, and the corresponding statistical analysis results are listed in Table 1.

The particles and pore forming agents were mixed in a powder mixing machine for 12 h. Then the binder was added. The mixed materials were put into a mold cavity. Following the pressing of 2 min under the pressure of 100 MPa through a hydraulic press machine. Two contrast preform groups were prepared: 50 μm particles with 10, 20 and 30% starches, moreover, 20 and 90 μm particles with 20% starches. The porous Si preforms of Φ30 (diameter) × 100 (height) mm³ were dried in a box furnace at 120 °C for 120 min. Then, the starches were burned by a slow heating to 340 °C and kept for 180 min. Subsequently, the temperature was increased to 900 °C and maintained 90 min to remove starch as possible. Finally, the preforms were cooled down to room temperature in the furnace. The inspection of particles and pores in the preforms was carried out with a high-resolution 3D X-ray μ-CT (Tianjin Sanying Precision Instruments nanoVoxel-3502E). The samples of 1 × 1 × 10 mm³ were obtained directly from each preform. Details about the inspection process and the reconstruction of preform characteristic are given in Ref. 29. Since the statistical analysis of 3D reconstruction was computationally time consuming, a domain of 600 × 600 × 600 μm³ in the sample central was selected for quantitative analysis.

2.2 Infiltration of preforms and analysis of composites

A pressure infiltration apparatus, whose vacuum level and maximum infiltration pressure were up to 70 Pa and 2.5 MPa, was made to carry out the infiltration of AlSi12 alloys into the preforms. The preforms were infiltrated at a constant temperature of 800 °C and pressure of 400 kPa with different pressure-applied times (3, 8 and 15 s). The procedure of the preform infiltration is illustrated in Ref. 31).

A high-resolution (~500 nm) vertical scanning white light interfering profilometer (Rtec Instruments UP model) was used to inspect the front surfaces of composites with different starch contents and particle sizes. The scanning samples were directly obtained from the composites by removing the non-infiltrated part. The measuring domain of 2.9 × 2.6 mm² in the central area of each sample was scanned and analyzed for processing accuracy and efficiency. In the experiment, the field of each view was 860 × 650 μm². Therefore, 20 raw images were produced for each sample. Data of the front surface of each sample were obtained after levelling and stitching the raw images. The 3D morphologies of the selected domain were reconstructed to analyze the front surface areas of composites with a commercial software Gwyddion (Czech Metrology Institute). The microstructures of samples at heights of 1 mm (away), 10 mm (middle) and front surface (front) from the composites (Fig. 2) with 15 s infiltration were observed with an optical microscope (Leica DM1 5000M) and the area fractions of residual pores of composites were calculated with an image analysis software Image-Pro Plus 6.0.

![Fig. 1. Size distributions of Si particles.](Image)

**Table 1. Statistical analysis results of Si particles**

| Si particle | 20 μm | 50 μm | 90 μm |
|------------|-------|-------|-------|
| D_{10} (μm) | 10.702 | 32.347 | 65.963 |
| D_{50} (μm) | 19.686 | 49.872 | 85.587 |
| D_{90} (μm) | 29.109 | 78.485 | 141.589 |

![Fig. 2. Schematic of different locations of composites.](Image)
2.3 Micro-scale simulation

During infiltration process under vacuum condition, liquid aluminum is usually regarded as an incompressible fluid. The mass conservation of the infiltration flow is given by Eq. (1) and the flow momentum can be calculated with the Navier-Stokes Eq. (2).

\[
\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho \mathbf{V}) = 0 \tag{1}
\]

\[
\frac{\partial \mathbf{V}}{\partial t} = \frac{\rho}{\rho g - \nabla p + \mu \nabla^2 \mathbf{V}} \tag{2}
\]

where, \(\rho\) is the density of fluid, \(\mathbf{V}\) is the velocity vector, \(t\) is the time, \(g\) is the gravity unit, \(\rho\) is the pressure and \(\mu\) is the dynamical viscosity of fluid.

When liquid aluminum flows through a porous preform under the vacuum condition, the surface tension has a great effect on the fluid. Therefore, Eq. (2) can be written as Eq. (3).

\[
\frac{\partial \mathbf{V}}{\partial t} = \frac{\rho}{\rho g - \nabla p + \mu \nabla^2 \mathbf{V}} + 2\sigma \kappa \delta S \mathbf{n} \tag{3}
\]

where, \(2\sigma \kappa \delta S \mathbf{n}\) represents the capillary forces, \(\sigma\) is the surface tension of liquid and particles (surface tension of liquid and gas is neglected due to flow in vacuum condition), \(\delta S\) is the area of surface \(S\), \(\mathbf{n}\) is the unit vector normal to the solid–liquid interface and \(\kappa = (1/r_1 + 1/r_2)/2\) is the mean curvature, which can also be expressed as:

\[
\kappa = \frac{1}{2} \nabla S \cdot \mathbf{n} \tag{4}
\]

where, \(\nabla S\) is the gradient operator restricted to the surface \(S\).

At present, the researches on the flow in porous medium are developed from macro-scale measurement to pore-scale simulation and the calculation domains are limited to micro-scale because of numerous calculation time consuming in pore-scale or micro meshes. The purpose of simulation is to understand the effects of preform starch contents and particle sizes on the pressure distributions along the infiltration direction, which is hard to be measured with experiments, and their influences to the front surfaces of infiltration flow liquid and the residual pores of AlSi12/Si composites.

The simulation of infiltration of preform was carried out based on a FLOW-3D software platform. For the calculation efficiency, the 300 \(\times\) 300 \(\times\) 400 \(\mu\)m\(^3\) domain of the original 3D reconstruction sample was selected as the simulation object. The thermal-physical parameters in the simulation are shown in Table 2.

**Table 2.** Thermal-physical parameters of infiltration simulation with FLOW-3D

| Parameter               | Value                  |
|-------------------------|------------------------|
| AlSi12 Density          | 2388 kg/m\(^3\)       |
| Viscosity               | 0.00111 Pa \(\times\)s |
| Surface tension         | 0.87181 N/m            |
| Infiltration pressure   | 400 kPa (\(4 \times 10^6\) g/(cm\(^2\)s\(^2\))) |

**Fig. 3.** Three-dimensional geometry with the reconstruction technology, enmeshment and infiltration parameters of Si preforms: (a) geometry, and (b) meshes and flow direction.

**Fig. 4.** The average pore radius and throat radius were 4.46 and 2.19 \(\mu\)m, respectively. Hence, pores with radii smaller than 4.46 \(\mu\)m and throats with radii smaller than 2.19 \(\mu\)m were defined as small pores and throats.

3. Results and discussion

3.1 Characteristics of Si preforms

The 3D characteristics of Si preforms with different starch contents and particle sizes are listed in Table 3. Based on the Maximal Ball (MB) algorithm, pores are considered as larger clusters gathering nearby overlapping MBs, and throats were defined as smaller clusters absorbing from other two larger clusters. With an increase in the starch content, the average areal porosity fraction, average pore radius and throat radius increased, whereas the pore and throat numbers decreased. As the particle size increased, the average pore radius and throat radius increased, whereas the average areal porosity fraction, pore and throat numbers decreased. The average throat radius was about 50% of the average pore radius, which was similar to those in our previous papers and those in other porous medium. The reason for the change in pore characteristics was explained in the previous papers.

According to the data from the 3D pore-network models, the number-based frequencies of the effective pore radius and throat radius are plotted in Fig. 4. The average pore radius and throat radius were 4.46 and 2.19 \(\mu\)m, respectively. Hence, pores with radii smaller than 4.46 \(\mu\)m and throats with radii smaller than 2.19 \(\mu\)m were defined as small pores and throats. As the starch content increased from 10 to 30%, the relative frequencies of small pores decreased from 63.59 to 53.48%, and the relative frequencies of small throats went down from 68.12 to 53.18%. The relative frequencies of small pores decreased from 86.67 to 51.04%, and the relative frequencies of small throats went down from 91.26 to 46.71% with increasing particle
size. By comparing the distributions in the figure, it can be seen that the particle size caused more change in the distributions.

3.2 Front surface of infiltration

The composites, which were infiltrated at 800 °C, 400 kPa and 15 s (pressure-applied time), were chosen to observe the front surfaces. The 3D topological morphologies of the front surfaces (“Front” in Fig. 2) detected with the white light interfering profilometer are shown in Fig. 5. The measuring domains of all samples were same. The measured height altitude differences of the samples with the 50 μm particles were 0.4 to 0.43 mm and those of the samples with the 20 % starches were 0.28 to 0.47 mm. This indicated a greater height altitude difference when changing the particle size.

The total front surface areas of the composites are given in Table 4. Under the same conditions (800 °C, 400 kPa and 15 s), the areas of the composites increased from 15.18 to 17.91 mm² with the starch content, and those went up from 14.30 to 18.01 mm² with the particle size.

Since the measured height altitude differences in Fig. 5 were one level larger than the particle sizes, it can be reasonably considered that the differences in the front surface areas were caused by the flow of liquid AlSi12 through pores with different characteristics. To study these differences, a schematic of the capillary tube is illustrated in Fig. 6 for analysis. The Si particles acted as capillary walls, and the pores or throats were capillary tubes. During the preparation of samples for front surface detection, the particles in the surface layer non-wetted with the liquid AlSi12 should be detached from the sample.

The convex surface of AlSi12 in the capillary is assumed to be approximately spherical. Therefore, the height and surface area of AlSi12 convexity in Fig. 6 can be calculated with the following equations:

Table 3. 3D characteristics of Si preforms with different starch contents and particle sizes

|                  | 50 μm (different starch contents) | 20 % (different particle sizes) |
|------------------|----------------------------------|---------------------------------|
|                  | 10 %    | 20 %    | 30 %    | 20 %    | 50 μm   | 90 μm   |
| Average areal porosity fraction | 28.20 % | 33.97 % | 41.46 % | 35.66 % | 33.97 % | 32.02 % |
| Average pore radius (μm)     | 4.05    | 4.46    | 5.26    | 2.70    | 4.46    | 5.99    |
| Average throat radius (μm)   | 1.92    | 2.19    | 2.56    | 1.43    | 2.19    | 3.03    |
| Pore number                 | 8904    | 7963    | 5987    | 32576   | 7963    | 3352    |
| Throat number                | 22485   | 21833   | 19440   | 88176   | 21833   | 7338    |

Fig. 4. Number-based frequencies of effective pore radius and throat radius: (a) effective pore radius of preforms with the 50 μm particles, (b) effective throat radius of preforms with the 50 μm particles, (c) effective pore radius of preforms with the 20 % starches, and (d) effective throat radius of preforms with the 20 % starches.
where, $r$ is the radius of capillary, $R$ is the radius of AlSi12 convexity, $\theta$ is the contact angle (120°), $\beta$ is the supplementary angle of $\theta$, $h$ is the height of AlSi12 convexity, and $S$ is the surface area of AlSi12 convexity.

According to the data in Table 3, the surface areas of AlSi12 convexities in the pores and throats were calculated, and the results are listed in Table 4. For the preforms with different starch contents, the differences among the average pore radii of preforms were small and the average throat radii showed a similar tendency. Hence, the differences among the surface areas of AlSi12 convexities were small. At the same starch content, the average areal porosity fraction decreased slightly with increasing particle size, whereas the average radii of pores and throats increased remarkably (Table 3). According to Eq. (7), the surface areas of AlSi12 convexities showed an exponential correlation of pore and throat radii. Therefore, the surface areas of AlSi12 convexities in the pores and throats of the preforms with larger particles increased obviously, as shown in Table 4.

Since the average throat radii in Table 3 were much smaller than the pores, the effect of calculated surface areas of AlSi12 convexities in the throats can be ignored. Therefore, Fig. 7 compares the average areal porosity fractions of preforms, the calculated surface areas of AlSi12 convexities in the pores and the measured front surface areas of composites. As shown in Fig. 7(a), the average areal porosities of preforms and the surface areas of AlSi12 convexities in the pores increased with the starch content. Therefore, the front surface areas of composites and the surface areas of AlSi12 convexities in the pores increased with the starch content.

### Table 4. Total front surface areas of composites and calculated surface areas of AlSi12 convexities

| 50 µm (different starch contents) | 10% | 20% | 30% | 20% (different particle sizes) | 20 µm | 50 µm | 90 µm |
|----------------------------------|-----|-----|-----|-------------------------------|-------|-------|-------|
| Front surface area (mm²)         | 15.18 | 16.61 | 17.91 | 14.30 | 16.61 | 18.01 |
| Surface area of convexity (pore) (µm²) | 54.68 | 66.98 | 93.16 | 24.55 | 66.98 | 120.81 |
| Surface area of convexity (throat) (µm²) | 12.41 | 16.15 | 22.24 | 6.88 | 16.15 | 30.91 |

![Fig. 5. 3D topological morphologies of front surfaces of composites: (a) 50 µm-10 %, (b) 50 µm-20 %, (c) 50 µm-30 %, (d) 20 µm-20 %, and (e) 90 µm-20 %](image-url)
the areas of AlSi12 convexities in the pores. Therefore, the front surface areas of composites increased obviously although the areal porosity fractions decreased. Based on the above results, it is concluded that the particle size has a greater effect on the front surface areas of composites compared with the starch content.

### 3.3 Pressure distribution

Figure 8 displays the pressure distribution during the infiltration of preform with the 50 μm particles and 20 % starches. It can be seen that the pressures show a decreasing distribution along the flow direction.

To obtain the pressure distribution during the infiltration of preform, the fill fraction of infiltration in the simulation was set to 99 %. Figure 9(a) shows the pressure distributions of liquid AlSi12 during the infiltration of preforms with different fill fractions. It was found that at different fill fractions, the pressures of liquid AlSi12 at free surfaces were almost the same. The pressure distributions of liquid AlSi12 during the infiltrations of preforms with the 50 μm particles are plotted in Fig. 9(b). According to the figure, as the starch content increased, the pressures of liquid AlSi12 at free surfaces increased slightly but the differences among those of the three preforms were small. Figure 9(c) displays the pressure distributions of liquid AlSi12 during the infiltration of preforms with the 20 % starches. With the particle size change, the differences among the pressures of liquid AlSi12 at free surfaces were large. As the particle size increased, the pressure drop...
along the flow direction was smaller and the pressure at free surface was larger. This can support more momentum for the surface tension. Therefore, the largest front surface area was found in the composite with the 90 μm particles and 20% starches, as shown in Table 4.

3.4 Residual pores

The metallographic analysis of the composites was used to investigate the relation of the front surface and the residual pores. Figure 10 shows metallographs of the two composites at a pressure-applied time of 15 s. The gray, white and black features in the figure are the Si particles, AlSi12 alloys and residual pores, respectively. As seen in the figure, the residual pores increased along the flow direction. In the same location, the residual pores decreased as the particle size increased.

Figure 11 displays the area fractions of residual pores of composites. As shown in Fig. 11(a), the differences among the area fractions of residual pores in the same locations were small. This was attributed to the small difference among the pressure distributions of liquid AlSi12 in the cases of different starch contents [Fig. 9(b)], since the pressure was a decisive factor for liquid to fill the pores with small curvature radii. For the composites with the different particles, the difference among the area fractions of residual pores of front surfaces became quite large [Fig. 11(b)]. According to Fig. 9(c), the pressure drop along the infiltration direction in the preform with the 90 μm particles was smaller than that in the preform with the 20 μm particles. The pressure of liquid AlSi12 at free surface of the former preform was approximately twice as high as that of the latter. Therefore, in the same locations, the residual pores of the composite with the 90 μm particles were much less than those with the 20 μm particles.

4. Conclusion

(1) According to the experiment results with the white light interfering profilometer and the calculation results of capillaries, the particle size had more influence on the front surface areas of composites compared with the starch content. With increasing particle size, although the average areal porosities of preforms decreased, the surface areas of AlSi12 convexities in the pores increased significantly. Therefore, the front surface areas of the composites increased.

(2) The simulation results showed that the change in the pressures of liquid AlSi12 at free surfaces during infiltration with different fill fractions was small.

(3) With an increase in the starch content, the change in the pressure distributions of liquid AlSi12 during infiltr-
tion was small, and the differences among the residual pores in the same locations were small. (4) When the particle size increased, the pressure drop of liquid AlSi12 along the infiltration direction decreased. The pressure at free surface of the preform with the 90 \( \mu \)m particles was twice as that with the 20 \( \mu \)m particles, which can provide more momentum for a large free surface area. The decreased pressure drop in the preform with larger particles caused fewer residual pores in the same locations.

Declaration of competing interest  The authors declare that they have no conflict of interest.

Acknowledgements  The research was supported by the Key Area Research and Development Program of Guangdong Province (No. 2019B010942001) and the National Natural Science Foundation of China (No. 51375171). The authors are thankful to the 3D X-ray tomography inspection characteristics among the residual

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