Effects of preform aperture/hole wall ratio on the microstructure and properties of ZTAp/HCCI matrix honeycomb structure composites

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Abstract

Zirconia toughened alumina particles (ZTAp)/high chromium cast iron (HCCI) matrix honeycomb structure composites were successfully prepared by the non-pressure infiltration casting process. The transformations of ZTA ceramic particles and high chromium cast iron during the compound process were analyzed by using x-ray diffraction (XRD) and Electron probe microanalysis (EPMA). The maximum stress and average value of the maximum stress simulated by COMSOL Multiphysics software shows a trend of decreasing first and then increasing with decreasing preform aperture/hole wall ratios. The maximum stress and average value of the maximum stress are minimal among the six comparison groups and the stress range is 0.8 ~ 1.8 GPa with preform aperture/hole ratio of 1.25. Owing to the change in the main impregnation resistances influenced by the hole wall value, the dispersion uniformity of ZTA ceramic particles in the composites becomes better with preform aperture/wall ratio of 1.43, 1.25 or 1.11. When the preform aperture/hole ratio is 1.25, the compressive strength of the samples reaches a maximum value of 1240 MPa. And the trend of the compressive mechanical properties is consistent with the results of the stress simulation. The wear volume loss of the composites reaches the smallest with preform aperture/hole wall ratio of 1.25, because the protective effect of ZTA ceramic particles and the supporting effect of high chromium cast iron achieve the best synergistic cooperation at this ratio.

1. Introduction

The ceramic particles reinforced metal matrix honeycomb structure composites are a good choice for improving the wear resistance to replace traditional steel wear-resistant materials, mainly because of their high hardness, high specific modulus, low cost and honeycomb structure with pinning effect [1–4]. Nowadays, many engineering components made of the composites are widely used in metallurgy, mining, coal and other industries [5, 6]. Meanwhile, these components have also shown great application prospects in the fields of aerospace, automotive and mechanical processing [7, 8].

At present, relevant research has developed rapidly. Tao et al [9] suggested that a gradient distribution of Si3N4 nanowires in the carbon fiber preform achieved by optimizing the preform structure can improve the compactness of the composites and the strength and toughness of the SiC matrix. Abdel-Fattah et al [10] reported that a proper stacking sequence and the proper combination of number of layers can heighten the tensile properties of the multilayer 3D fibrous composites. Mei et al [11] reported that the porous oblique honeycomb structures with well-designed angle (30°, 45°, 60° and 75°) can optimize wave absorbing performance of Al2O3/SiCw honeycomb composites. Li et al [12] reported that the fiber preform structures prepared by different stacking and stitching processes can significantly affect the fiber retention ratio and the fiber array direction of C/SiC nuts and bolts. Lu et al [13] suggested that two-layer and three-layer carbon fiber needle-punching preform structures can change the pore size distribution on the preform, and then lead to the...
faster densification rate and the higher compressive strength of carbon/carbon composites. Relatively speaking, the research on the structure design of ceramic particles reinforced metal matrix honeycomb structure composites is relatively rare.

In this study, ZTA (zirconia toughened alumina) ceramic particles were selected as the reinforcement material, which were made into the honeycomb structure preforms. And, high chromium cast iron (HCCI) was selected for the matrix material. ZTAp/HCCI matrix honeycomb structure composites were prepared by the non-pressure infiltration casting process. The effects of preform aperture/hole wall ratio on the microstructure and properties was studied by means of the stress simulation and corresponding experiments.

2. Experimental procedures

2.1. Sample preparation

The ZTA ceramic particles (56%Al₂O₃, 40%ZrO₂, 3%TiO₂ and 1%Fe₂O₃) with irregular shape of about 120 μm in equivalent diameter and high chromium cast iron (3.0%C, 26%Cr, 0.8%Mn, 0.4%Si, 0.3%Ni, 0.4%Mo) were selected for reinforcement and matrix material in this work, respectively (in wt%). The unit 1, 2 were selected from the cuboid-shaped preform (figure 1) commonly used in industrial production [14]. In this study, the preforms placed in the sand cavity were closely related to the unit 1, 2. And, the sum of the aperture value and the hole wall value was constant. The specific dimensions of the preforms with different aperture/hole wall ratios are given in table 1. The high chromium cast iron was melted at 1550 °C in a medium frequency induction furnace. Molten high chromium cast iron metal was poured at 1500 °C into the sand cavity, which was preheated to 300 °C. The size of the sand cavity was 100 mm × 80 mm × 50 mm.

2.2. Characterization method

X-ray diffraction (XRD, D/max-2500) was used to identify the phase existing in the high chromium cast iron, ZTA ceramic particles and composites. The specimens were scanned in the 2θ range of 20°–80° at a scanning rate of 2° min⁻¹. Electron probe microanalysis (EPMA, JXA-8230F) was employed to detect the element distribution in the samples. Optical microscope (OM, NIKON M300) was used to observe the microstructure of the composites. The metallographic photographs were analyzed by using image-pro plus (IPP) software to obtain the percentage of ZTA ceramic particles on the sample cross-section. The diffraction patterns with different incidence angles (0°, 15°, 30° and 45°) at the (011) crystal face were obtained by x-ray diffraction (XRD, D/max-2500) with preform aperture/hole wall ratios of 2.5, 1.67, and 1.25. The diffraction patterns were analyzed by using calculate stress module in Jade software to obtain corresponding residual thermal stress values. The compressive mechanical properties of the composites were tested by using a compression tester (Shimadzu AG-
The compression samples were prepared according to the GB/T232-2010 Chinese standard with the size of 15 mm × 15 mm × 25 mm (figure 2(a)). In order to study the wear behavior of ZTAp/HCCI matrix honeycomb structure composites, three-body abrasive wear tests were carried out in a block-on-ring geometry (figure 2(b)). The wear samples (figure 2(a)) were rotated at a speed of 30 r/min against the ring-shaped grinding surface under 3.0 Kg horizontal load (figure 2(c)). At the beginning, pre-wear test was conducted for 30 min. Then, wear tests were conducted through 6 wear cycles, each of which lasted for 1 h. After each cycle, the samples were cleaned by using an ultrasonic cleaner and the mass losses were measured on an electronic balance having an accuracy of 0.0001 g (average value of 5 test results). The wear resistance of the composites is generally characterized by the volume loss:

\[ V_{\text{loss}} = \frac{G_{\text{loss}}}{\rho} \]

where \( V_{\text{loss}} \) and \( G_{\text{loss}} \) represent the volume loss and mass loss of the samples at each wear cycle. The calculation formula of the average density \( \rho \) in the composite layer [16] is expressed as follows:

\[ \rho = \rho_p \alpha + \rho_m (1 - \alpha) \]

where \( \rho_p \) and \( \rho_m \) represent the density of ZTA ceramic particles and high chromium cast iron, respectively, and \( \alpha \) represents the volume fraction of ZTA ceramic particles in the composite layer with thickness of about 10 mm.

The compression fracture morphology and wear morphology of the samples were observed by scanning electron microscopy (SEM, ZEISS EVO).

### 2.3 Stress simulation

3D CAD files with different preform aperture/hole wall ratios were imported into COMSOL Multiphysics software (figure 3(a)). Set the relevant parameters in material module according to the physical properties and chemical composition of the selected ZTA ceramic particles and high chromium cast iron. The corresponding boundary conditions were set by solid mechanics and solid heat transfer multi-physics coupling [17].

In solid mechanics module, the transient form of the assumed equation is as follows [18, 19]:

\[ \nabla \cdot S + F_v = 0 \]

where \( \nabla \) is the vector differential operator, \( F \) is the force per unit volume vector, \( v \) is the speed corresponding to \( F \), and \( S \) represents the stress, which is defined by the following:

\[ S = S_0 + S_{\text{ext}} + S_q + C: (\varepsilon - \varepsilon_0 - \varepsilon_{\text{ext}} - \varepsilon_{\text{th}} + \varepsilon_{\text{pl}} + \varepsilon_{\text{cr}} + \varepsilon_{\text{vp}}) \]

where \( S_0 \) is the pre-stress, \( S_{\text{ext}} \) is the external stress, \( S_q \) is the viscous stress, \( C \) represents an elastic matrix related to Young’s modulus and shear modulus, \( \varepsilon \) is the elastic strain, \( \varepsilon_0 \) is the pre-strain, \( \varepsilon_{\text{ext}} \) is the external strain, \( \varepsilon_{\text{th}} \) is the thermal strain, \( \varepsilon_{\text{pl}} \) is the wetting expansion, \( \varepsilon_{\text{cr}} \) and \( \varepsilon_{\text{vp}} \) represent the plastic strain and creep in the complementary nonlinear structural material, respectively, and \( \varepsilon_{\text{vp}} \) is the time-dependent viscoplastic strain.

In solid heat transfer module, the transient form of the assumed equation is as follows [20, 21]:

\[ \rho C_p \frac{\partial T}{\partial t} + \rho C_p u \cdot \nabla T + \nabla \cdot (-K \nabla T) = Q \]

where \( \rho \) is the density, \( C_p \) is the constant pressure heat capacity, \( T \) is temperature, \( t \) is time, \( u \) represents the displacement field, \( \nabla \) is the vector differential operator, \( K \) is the thermal conductivity, and \( Q \) is the heating power per unit volume.
As molten high chromium cast iron metal came into contact with the preforms and gradually cooled, a large amount of latent heat generated by the phase transformation was released and further led to the change in pressure in the sand cavity. Equation (6) is a supplement to equation (5) in the phase transition process:

\[
\rho = \theta_1 \rho_1 + \theta_2 \rho_2 \\
C_p = \frac{1}{\rho} (\theta_1 \rho_1 C_{p,1} + \theta_2 \rho_2 C_{p,2}) + L_{1 \rightarrow 2} \frac{\partial}{\partial T} \left( \frac{1}{\theta_1 \rho_1 + \theta_2 \rho_2} \right) \\
K = \theta_1 K_1 + \theta_2 K_2 \\
\theta_1 + \theta_2 = 1
\]

where \( l, 2 \) represent high chromium cast iron liquid phase and high chromium cast iron solid phase, respectively, \( \theta_1, \theta_2 \) represent the percentage of phase 1 and phase 2 in the phase transition process, and \( L_{1 \rightarrow 2} \) is the latent heat from phase 1 to phase 2.

### 3. Results and discussion

#### 3.1. Analysis of the stress simulation results

The results of the stress simulation in COMSOL Multiphysics software are shown in figure 4. Figures 4(a)–(f) are the stress-time curves from point 1 to point 7 with preform aperture/hole wall ratios of 2.5, 2.0, 1.67, 1.43, 1.25 and 1.11, respectively. It can be seen, from table 2, that the maximum stress and average value of the maximum stress show a trend of decreasing first and then increasing with decreasing preform aperture/hole wall ratios. As shown in figure 4(e), the maximum stress and average value of the maximum stress are minimal among the six comparison groups and the stress range is \( 0.8 \sim 1.8 \) GPa with preform aperture/wall ratio of 1.25. The increases in the maximum stress and average value of the maximum stress might lead to a more pronounced shrinkage mismatch between the various parts of the samples during the solidification process [22, 23]. Furthermore, it might cause an increase in the residual thermal stress, and the deformation in the composites influenced by the stress concentration might occur more easily [24, 25]. When the tendency of crack propagation caused by the deformation increased, the reduction of the composite properties was very possible.

#### 3.2. Microstructure

Figure 5 shows the phase existing examined by XRD in the high chromium cast iron, ZTA ceramic particles and composites. It can be seen, from figure 5(a), that the high chromium cast iron is composed of \( \gamma \)-Fe and M7C3 type carbide (\( (\text{Cr}, \text{Fe})_7\text{C}_3 \)) phases. Figure 5(b) presents the ZTA ceramic particles that consist of \( \alpha \)-Al2O3 and t-ZrO2 phases. After adding high chromium cast iron to ZTA ceramic particles, a new phase of m-ZrO2 was detected in the composites. The production of m-ZrO2 indicated that a phase transformation toughening mechanism occurred in the ZTA ceramic particles [4, 26] during the compound process. The occurrence of the phase transformation (t-ZrO2 \( \rightarrow \) m-ZrO2) depended on absorbing thermal stress generated by a large difference of the thermal expansion coefficient (ZTA ceramic particles: \( 8.77 \times 10^{-6}/\text{K}^{-1} \); high chromium cast iron: \( 15.30 \times 10^{-6}/\text{K}^{-1} \), at 1573 K) between high chromium cast iron and ZTA ceramic particles [17, 27–29].
Figure 6 shows a ZTA particle in contact with high chromium cast iron on one side. The distribution maps of Cr, Fe, Al, and Zr elements obtained by EPMA are shown in figures 6(b)–(e). Combined with the XRD results, the formation of M7C3 type carbide ((Cr, Fe)7C3) phase in high chromium cast iron was closely related to the distribution and diffusion behavior of Cr and Fe elements. The M7C3 type carbide ((Cr, Fe)7C3) phase can significantly improve the mechanical properties and wear resistance of the high chromium cast iron because of the internal stacking dislocation [30, 31]. The distribution maps of Zr and Al elements shows m-ZrO2 and t-ZrO2 existed between α-Al2O3 in the form of network and toughened α-Al2O3.

Figures 7(a)–(f) show the metallographic photographs of the composites with preform aperture/hole wall ratios of 2.5, 2.0, 1.67, 1.43, 1.25, and 1.11 at 50 times, respectively. At least 30 pictures were used to measure the percentage of ZTA ceramic particles on the sample cross-section. The results obtained by Image-Pro plus (IPP) software were 35.1%, 48.8%, 56.1%, 58.6%, 60.1% and 70.2%. Thus, the degree of dispersion of ZTA ceramic particles in the composites was continuously reduced. It is obvious that the hole wall values would be increased.
with decreasing aperture/hole wall ratios, because the sum of the aperture value and the hole wall value was constant. Therefore, when the molten metal was impregnated into the preforms, the effect of the main impregnation resistances such as capillary force, solidification resistance, and viscous resistance became more significant. The flow rate of the molten metal would be decreasing. And the degree of impact of the molten metal on the ceramic particles would correspondingly reduce. There must be a critical flow rate, in which the degree of impact of the molten metal on the ceramic particles motivated a uniform distribution of ZTA ceramic particles in the composites. The effect of this trend was reflected in the metallographic photographs. It can be seen that the dispersion uniformity of ZTA ceramic particles in the composites shows a tendency to become uniform first and then become scraggly. When the preform aperture/hole wall ratio is 1.43, 1.25 or 1.11, the dispersion uniformity is pretty good.

3.3. Mechanical properties
Figure 8(a) shows the stress-strain curves of the compression samples with preform aperture/hole wall ratios of 2.5, 2.0, 1.67, 1.43, 1.25 and 1.11. The compressive strength was obtained by taking the stress values at the highest point of each curve. It can be seen, from figure 8(b), that the compressive strength of the samples increases first and then decreases with decreasing aperture/hole wall ratios. And, the compressive strength reaches a maximum value of 1240 MPa with aperture/wall ratio of 1.25. The change of the compressive strength was closely related to the trend of the dispersion uniformity of ZTA ceramic particles in the composites. The
better the dispersion uniformity was, the smaller the stress value in each region of the composites and the stress difference between the regions were. And the residual thermal stress of the samples (preform aperture/hole wall ratio 2.5: 447.89 ± 34.97 MPa; preform aperture/hole wall ratio 1.67: 429.47 ± 33.18 MPa; preform aperture/hole wall ratio 1.25: 382.5 ± 29.77 MPa) was correspondingly reduced. Furthermore, ZTA ceramic particles could achieve more uniform enhancement distribution on the high chromium cast iron. The mechanical properties of the composites such as the compressive strength would improve accordingly [33, 34]. And the trend of the compressive mechanical properties is consistent with the stress simulation results.

In order to better understand the results of the compressive mechanical properties tests, the compression fracture morphology of the samples with preform aperture/hole wall ratios of 2.5, 1.25 and 1.11 were observed by scanning electron microscopy (SEM). Figure 9(a) shows the cracks initiated at the interface between ZTA ceramic particles and high chromium cast iron expanded on the metallic matrix. It can be seen, from figure 9(b), that ZTA ceramic particles could sometimes hinder crack propagation. Figure 9(c) presents a network of cracks formed by crack propagation would lead to the pit defects. The proportion of each form shown in figure 9 was different in different samples. Combined with the metallographic photographs, the degree of dispersion of ZTA ceramic particles in the composites was appropriate with preform aperture/pore wall ratio of 1.25. Therefore, ZTA ceramic particles had a stronger inhibitory effect on crack propagation in the sample. The cracks on the sample were shorter and the crack networks were less. The excellent microscopic performance brought about an increase of the compressive strength at the macroscopic level.
3.4 Wear performance of the composites

The results of the three-body abrasive wear tests are shown in Figure 10. It can be seen from Figure 10(a) that the wear volume loss of the composites decreases firstly and reaches the minimum at the fourth or fifth cycle under the same wear conditions. Then, with the increase of wear time, the wear volume loss increases slightly. Referring to Figures 10(a) and (b), it can be seen that the wear volume loss of the sample with aperture/hole wall ratio of 1.11 is minimal among the six comparison groups at the first four wear cycles. However, the relative increases of the wear volume loss at the fifth and sixth periods results in the smallest overall volume loss of the sample with preform aperture/hole wall ratio of 1.25.

The wear mechanism of the composites is shown in Figure 11. After the pre-grinding, ZTA ceramic particles and high-chromium cast iron were first in the same plane (Figure 11(a)). The wear volume loss was large at the first and second periods, because the abrasive particles would first cut the soft matrix (Figure 11(b)). With the increase of wear time, the ceramic particles gradually protruded from the metallic matrix and sustained the main wear effect (Figure 11(c)). At this time, the good coordination between the protective effect of ceramic particles on the metallic matrix and the supporting effect of the metallic matrix on the ceramic particles [15, 16, 35] made the wear volume loss gradually reduce. However, as the wear continued, the metallic matrix of the composites was worn to concave so severely that the supporting effect of the metallic matrix on the ceramic particles became deficient. Thus, it was found some reinforcing particles were broken (Figure 11(d)) and even pulled off (Figure 11(e)). Finally, ZTA ceramic particles and high chromium cast iron would once again be in the same plane (Figure 11(f)).

In order to better explain the results of the three-body abrasive wear tests, the wear morphology of the samples with preform aperture/hole wall ratios of 2.5, 1.25 and 1.11 were observed by scanning electron microscopy (SEM). Figure 12 shows furrows and concaves in the metallic matrix became less and lighter with decreasing preform aperture/hole wall ratios. Meanwhile, broken and even pulled off of ZTA ceramic particles were more significant. For the sample with preform aperture/pore wall ratio of 1.11, the increasing of the pore wall value expanded the protection area of ZTA ceramic particles to the metallic matrix. Meanwhile, the decreasing of the metallic matrix between ZTA ceramic particles improved the protection ability of ZTA ceramic particles on the metallic matrix. Therefore, the wear volume loss of the sample was minimal among the six comparison groups at the first four wear cycles. However, this trend also brought about a decrease in the degree...
of dispersion of the ceramic particles and a decline in the supporting effect of the metallic matrix on the ceramic particles, which motivated the occurrence of significant broken and even pulled off of ZTA ceramic particles shown in figure 12(c). Therefore, in figure 10(a), the wear volume loss of the sample was relatively increased at the fifth and sixth periods. According to figures 10, 11 and 12, there must be a critical point of the preform aperture/pore wall ratio, in which the protective effect of ZTA ceramic particles and the supporting effect of the metallic matrix achieve the best synergistic cooperation [36, 37]. In this work, the critical point is found to be the preform aperture/hole wall ratio of 1.25, at which point the wear volume loss of the composites reaches the smallest.

4. Conclusions

The effects of preform aperture/hole wall ratio on the microstructure and properties of ZTAp/HCCI matrix honeycomb composites were investigated. The following conclusions can be drawn:

(1) The maximum stress and average value of the maximum stress simulated by COMSOL Multiphysics software shows a trend of decreasing first and then increasing with decreasing preform aperture/hole wall ratios. The maximum stress and average value of the maximum stress are minimal among the six comparison groups and the stress range is 0.8 ~ 1.8 GPa with preform aperture/wall ratio of 1.25.

(2) The surface stress simulated by COMSOL Multiphysics software shows a trend of decreasing first and then increasing with decreasing preform aperture/hole wall ratios. The overall stress value and stress difference are minimal among the six comparison groups and the stress range is 1.18 ~ 1.8 (×10^9 N m^-2) with preform aperture/wall ratio of 1.25.

(3) The percentage of ZTA ceramic particles on the sample cross-section increases from 35.1% to 70.2% when the preform aperture/hole ratio reduces from 2.5 to 1.11. The dispersion uniformity of ZTA ceramic particles in the composites becomes better with preform aperture/wall ratio 1.43, 1.25 or 1.11.
(4) The compressive strength of the samples reaches a maximum value of 1240 MPa with preform aperture/hole wall ratio of 1.25. And the trend of the compressive mechanical properties is consistent with the stress simulation results.

(5) The wear volume loss of the composites reaches the smallest with preform aperture/hole wall ratio of 1.25, because the protective effect of ZTA ceramic particles and the supporting effect of the metallic matrix achieve the best synergistic cooperation at this ratio.

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