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Threshold pressure and infiltration behavior of liquid metal into fibrous preform

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Abstract: A dynamic measuring apparatus was developed to investigate the infiltration process of liquid metal into the fibrous preform. 10% (volume fraction) chopped carbon fiber preforms were infiltrated with magnesium alloy under different infiltration pressures. The threshold pressure and flow behavior of liquid metal infiltrating into the preforms were calculated and measured. The microstructure of obtained C_f/Mg composites was observed. The results indicate that the measured threshold pressure for infiltration was 0.048 MPa, which was larger than the calculated value. The infiltration rate increased with the increase of infiltration pressure, but the increase amplitude decreased gradually. The tiny pores in the composites could be eliminated by increasing the infiltration pressure. When the infiltration pressure rose to 0.6 MPa, high quality C_f/Mg composite was obtained.

Key words: metal matrix composites; carbon fiber; Mg; perform; infiltration; threshold pressure; infiltration rate

1 Introduction

Recently, carbon fiber reinforced magnesium matrix composites (C_f/Mg composites) have received considerable attention in aviation and aerospace industries due to their light mass, excellent mechanical properties and low thermal expansion coefficient [1−3]. Among all the fabrication techniques for C_f/Mg composites, liquid infiltration method is one of the most economical and widely used methods, such as pressureless infiltration [4], vacuum-assisted pressure infiltration [5], squeeze casting [6] and extrusion directly following infiltration [7]. In the process of C_f/Mg composite fabrication, high enough external pressure has to be applied to obtain good infiltration quality due to the poor wettability between carbon fibers and magnesium alloy. A quantitative understanding of the infiltration behavior will be helpful to selecting the fiber coatings and process parameters so as to optimize the process.

Many research works have been conducted on the infiltration behavior [8,9]. Theoretical research on infiltration can be classified into two kinds: 1) the threshold infiltration pressure is calculated according to the Laplace' equation, which ignores the effect of infiltration resistance during the process [10]; 2) the kinetics characteristic of the infiltration process is revealed on the basis of neglecting the tiny non-infiltration phenomenon in the composites [11]. In addition, a lot of simulation researches have also been carried out [12,13]. However, the infiltration of liquid metal into a porous preform is a rather complex physical process. These theoretical results do not coincide with the experimental results quantitatively, because too many assumptions are made during establishing the mathematical model. Thus, many scholars use experimental research methods to visualize the infiltration process. One method is that the relationship between infiltration depth and infiltration parameters is established through a series of experiments. WANNASIN and FLEMINGS [14] measured the threshold pressure of Sn−Pb alloy infiltrating into different volume fractions of compact ceramic powders and established an equation to predict the threshold infiltration pressure. RODRIGUEZ-GUERRERO et al [15] investigated the effect of alloy composition and

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infiltration temperature on the infiltration of aluminum alloy into compact graphite particles. Another method is that the flow behavior of liquid metal in the fibrous preform is monitored by some thermocouples pre-embedded in the preform. QI et al [16] measured the infiltration process of liquid magnesium alloy into Al_2O_3 short fiber preform and discussed the formation mechanism of infiltration front. LONG et al [17] visualized the flow behavior of an AlSi10Mg alloy melt in chopped Saffil fiber preform and established a model to predict the hydrodynamic features of the infiltration process. Infiltration pressure is an important parameter in composite fabrication. DEMIR and ALTINKOK [18] investigated the effect of gas pressure on the microstructure and bending strength of Al_2O_3/SiC reinforced aluminum matrix composites fabricated by gas pressure infiltration method, and reported that the density and bending strength increased with the increase of infiltration pressure.

In this work, a dynamic measuring apparatus for infiltration process was developed, by which the whole infiltration process can be carried out in a sealed container. On this basis, the threshold pressure of liquid magnesium alloy infiltrating into 10% (volume fraction) chopped carbon fiber preform was measured, and the effect of infiltration pressure on the infiltration rate and microstructure of the obtained C_f/Mg composites were also investigated.

2 Experimental

2.1 Materials

A commercial AZ91D magnesium alloy with composition (mass fraction) of 8.30%−9.70% Al, 0.35%−1.00% Zn, 0.15%−0.50% Mn, ≤0.10% Si, ≤0.03% Cu, balance Mg, was selected as matrix alloy. The reinforcement used was T300 chopped carbon fiber with an average diameter of 7 μm. The fiber volume fraction of preform was 10%. The preform was fabricated using a wet forming method, and chemical vapor deposition process was carried out subsequently to deposit pyrolytic carbon coating on the carbon fibers which acted as a barrier for reactions.

2.2 Measurement of infiltration process

The infiltration process for composite fabrication consisted of three steps. Firstly, liquid metal began to infiltrate into the preform either spontaneously by capillarity or with the assistance of external pressure. Secondly, liquid metal flowed in the preform and filled the voids. Finally, the liquid metal solidified under pressure while the whole structure was cooled. In this work, Steps 1 and 2 and microstructure of the obtained composites were investigated.

Figure 1 shows the developed dynamics measuring apparatus for liquid metal infiltration process. It mainly consists of smelting crucible, resistance furnace, gas pressure control system and data collection system. The gas pressure control system can provide argon gas with continuous variable pressure in the range of 0−1 MPa through an electric proportional valve. The data collection system collects gas pressure and temperature information through the pressure sensor and thermocouples during the infiltration process, respectively. AZ91D magnesium alloy is put into the stainless steel crucible which has higher strength at elevated temperatures. The fibrous preform is placed in the infiltration chamber and fixed with a graphite plate. There are some small holes in the graphite plate for inserting the thermocouples and exhausting the air in the

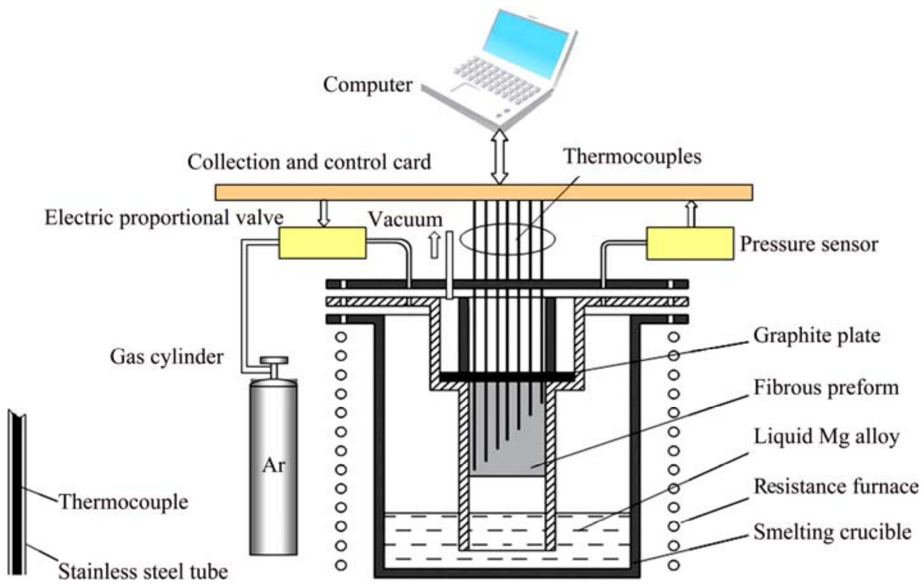

Fig. 1 Schematic diagram of dynamic measuring apparatus for liquid metal infiltration process

preform. Seven high precision thermocouples with 1 mm diameter are inserted into the preform. In order to avoid bending deformation of the thermocouples, they are protected by stainless tubes with thin-wall. The horizontal and vertical spacing between each thermocouple are 6 mm and 10 mm, respectively. Compared with the previous work [15], thermocouples contact with the preform directly, so as to improve the measuring accuracy.

The specific experimental process is described as follows. The experimental facilities were installed and sealed as shown in Fig. 1. In order to avoid the air in the preform affecting the infiltration process, magnesium alloy ingot was heated after the crucible was vacuumed. The magnesium alloy was preheated to 800 °C and held for 1 h. A gradually increasing gas pressure was applied to force the liquid magnesium alloy infiltrate into the preform. Since the temperature of magnesium alloy was higher than that of the carbon fiber preform, there would be a temperature rise when liquid magnesium alloy arrived at the thermocouples' position. The data collection system collected the real-time temperature information during the infiltration process. On the basis of the similarity between seepage field and temperature field, the infiltration height—time curve was obtained. In addition, combining the gas pressure—time curve, the threshold pressure for infiltration of magnesium alloy into a chopped carbon fiber preform was obtained. Moreover, constant gas pressures were applied into the crucible, and the flow behavior of liquid magnesium alloy into chopped carbon fiber preforms under different infiltration pressures was also obtained. After infiltration, the density of the C_f/Mg composites was measured, and the microstructure was observed by JSM-6390A scanning electron microscope (SEM).

3 Results and discussion

3.1 Theoretical calculation of infiltration process

3.1.1 Threshold pressure for infiltration

In general, threshold pressure for infiltration is defined as the critical pressure under which liquid metal begins to infiltrate into the preform, and the main form is capillary pressure. According to the classical Young-Laplace equation, the capillary pressure P_c can be expressed as [19]

$$P_c = \frac{2\gamma_{lv}\cos\theta}{R_{eq}} = \frac{2\gamma_{lv}V_f\cos\theta}{R_f(1-V_f)} \tag{1}$$

where γ_{lv} is the surface tension of the liquid metal; θ is the contact angle; R_{eq} is the equivalent capillary radius of the perform; V_f is the volume fraction; R_f is the radius of carbon fiber.

The wettability between liquid magnesium alloy and carbon fiber is poor. Thus, liquid magnesium alloy can not infiltrate into the carbon fiber preform spontaneously. Considering the actual liquid metal infiltration process, as shown in Fig. 2, the threshold pressure P_{th} for infiltration can be expressed as

$$P_{th} = \frac{-2\gamma_{lv}V_f\cos\theta}{R_f(1-V_f)} + \rho g h_0 \tag{2}$$

where ρ is the density of the liquid metal; h_0 is the initial height of the liquid magnesium alloy before infiltration. In general, the value of $\rho g h_0$ is very small, which can be ignored. The average radius R_f of carbon fiber is 3.5 μm. The surface tension γ_{lv} of liquid magnesium alloy is about 0.559 N/m, and the contact angle is 120°, which was measured in Ref. [20]. The calculated threshold pressure for liquid magnesium alloy into 10% chopped carbon fiber preform is 0.02 MPa.

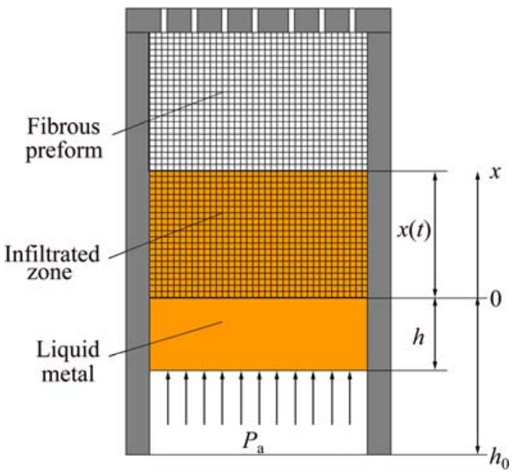

Fig. 2 Schematic diagram of infiltration process of liquid metal into fibrous perform

3.1.2 Infiltration kinetics

During the infiltration process, suffered resistance increases with the increase of infiltration height. In order to simplify the problem, a series of assumptions are made as follows: 1) Because the preform is preheated before infiltration, and the infiltration process is usually finished in a very short time, the heat transfer can be ignored, and the temperature of molten alloy can be assumed as a constant during the infiltration process; 2) Liquid metal flow in the pores is in the form of steady-state flow, so the tip resistance can be ignored; 3) There is no residual air in the preform, so the gas anti pressure can be ignored.

Considering the liquid metal as an incompressible homogeneous fluid and assuming the metal flow in the preform is a laminar flow, according to Fig. 2, an equation for the infiltration process can be expressed as [21]

$$\begin{cases} Ax\dfrac{\mathrm{d}x}{\mathrm{d}t}=P_\mathrm{a}+B \\ A=\dfrac{24\mu}{nR_\mathrm{eq}^{\ 2}}(1-V_\mathrm{f})+\dfrac{8\mu}{R_\mathrm{eq}^{\ 2}} \\ B=-\rho g h_0+\dfrac{2\gamma_\mathrm{lv}\cos\theta}{R_\mathrm{eq}} \end{cases} \tag{3}$$

where P_a is the external pressure applied to the liquid metal; n is the porosity of the fibrous perform; μ is the viscosity of the liquid metal.

It should be noted that infiltration will only occur when $P_\mathrm{a}+B>0$, which is the threshold pressure for infiltration as discussed in section 3.1.1. Considering pressure-assisted infiltration process is often carried out under constant external pressure, when P_a is constant, the solution to Eq. (3) is

$$x=\zeta t^{\frac{1}{2}} \tag{4}$$

where the infiltration rate ζ is defined as

$$\zeta=\left[\frac{2(P_\mathrm{a}+B)}{A}\right]^{\frac{1}{2}} \tag{5}$$

The viscosity of the liquid magnesium alloy is 0.003 Pa·s [22], the fiber volume fraction is 10%, and the threshold pressure is 0.02 MPa. Figure 3 shows the infiltration height — time curves under different infiltration pressures.

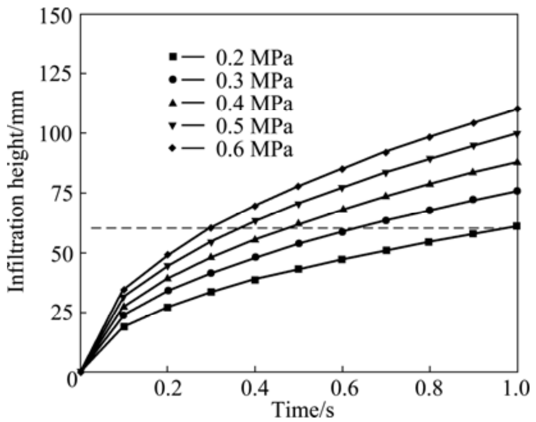

Fig. 3 Infiltration height — time curves under different infiltration pressures

3.2 Measurement of threshold pressure

In section 3.1.1, the threshold pressure for liquid magnesium alloy infiltrating into a 10% chopped carbon fiber preform was calculated as 0.02 MPa. However, in actual infiltration process, liquid metal will begin to solidify with the increase of infiltration height, because the interspaces between adjacent carbon fibers will become narrow, which leads to a higher infiltration resistance. Thus, in order to obtain a good infiltration result, the infiltration pressure should be larger than the calculated value.

According to the kinetic analysis results in Fig. 3, liquid magnesium alloy can infiltrate 60 mm in 1 s under a constant gas pressure of 0.2 MPa. Therefore, a continuous increasing gas pressure from 0 to 0.2 MPa in 2 s is applied on the surface of liquid metal to measure the threshold infiltration pressure. Figure 4 shows the variation of gas pressure during the infiltration process. In the gas pressure loading stage, the measured value is in good agreement with the set value. But, in the uploading stage, there is a delay between the set value and the measured value.

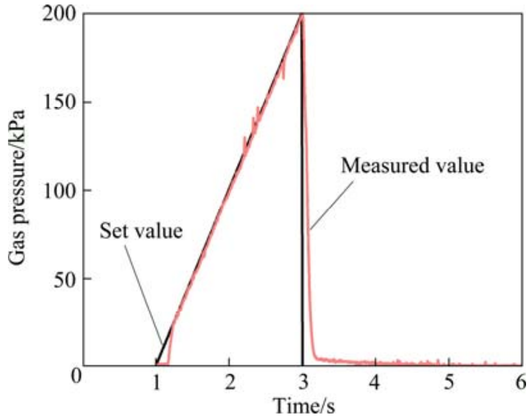

Fig. 4 Variation of gas pressure during infiltration process

Figure 5 shows the temperature—time and gas pressure—time curves during the infiltration process. When the gas pressure increases to a certain value, the temperatures begin to rise suddenly. The sudden rise of temperatures is not simultaneous, and there exists time interval between two adjacent temperature curves. The sudden rise of time vs temperature curve is just the time when liquid magnesium begins to infiltrate into the preform. The corresponding gas pressure is 0.048 MPa which is larger than the calculated value of 0.02 MPa. The main reason is that the temperature field of the preform is not uniform along the height direction. Temperatures of thermocouple 1 and 7 are 505 °C and 680 °C, respectively. With the increase of infiltration height, temperature of the preform decreases gradually. Effect of temperature on the surface tension of liquid alloy can be expressed as [23]

$$\sigma\left(\frac{M}{\rho}\right)^{-2/3}=K\left(T_\mathrm{c}-T\right) \tag{6}$$

where σ and ρ are the surface tension and density of the liquid metal, respectively; M is the relative molecular mass of the melt; K is a constant; T_c is the threshold temperature; T is the temperature of the melt. It can be seen that the surface tension increases with the decrease of temperature. According to Eq. (2), the threshold pressure will increase accordingly. In addition, the decrease of temperature may also lead to premature solidification of liquid metal and higher infiltration resistance.

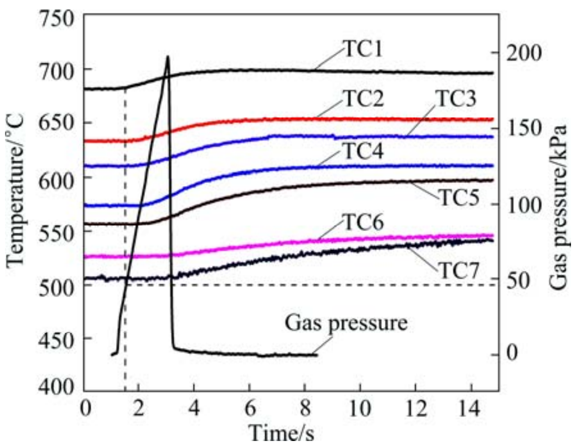

Fig. 5 Temperature—time and gas pressure—time curves during infiltration process

3.3 Effect of infiltration pressure on infiltration rate

Infiltration experiments were conducted under the threshold pressure, but the infiltration quality was poor. Thus, higher gas pressure should be applied to improve the infiltration quality. However, too large infiltration pressure will result in entrapped gas bubbles in the composite. It will also cause the fibrous preform deformed, and even collapsed, which affects the final mechanical properties of the composite. In this work, the infiltration pressure was selected in the range of 0.4−0.6 MPa.

Experiments of liquid AZ91D alloy infiltrating into 10% chopped carbon fiber preforms were carried out under different infiltration pressures (0.4, 0.5 and 0.6 MPa). During the infiltration process, temperatures at different height of the preform were collected through the data collection system, as shown in Fig. 6. According to the measurement results, the infiltration height—time curves were obtained using data extracting and fitting method. The fitting equation of infiltration height z−time (t) is expressed as

$$\begin{cases} z(t)=71.61\sqrt{t}-6.345 \ (P_a=0.4\,\text{MPa}) \\ z(t)=86.97\sqrt{t}-3.331 \ (P_a=0.5\,\text{MPa}) \\ z(t)=91.28\sqrt{t}--3.817 \ (P_a=0.6\,\text{MPa}) \end{cases} \tag{6}$$

Figure 7 shows the infiltration rate under different infiltration pressure. The experimental results are slightly less than the calculated values. The main reason is that the effect of liquid metal solidification was ignored during the kinetic analysis. Due to the temperature gradient, liquid metal solidified gradually and hindered the flow. With the increase of infiltration pressure, the

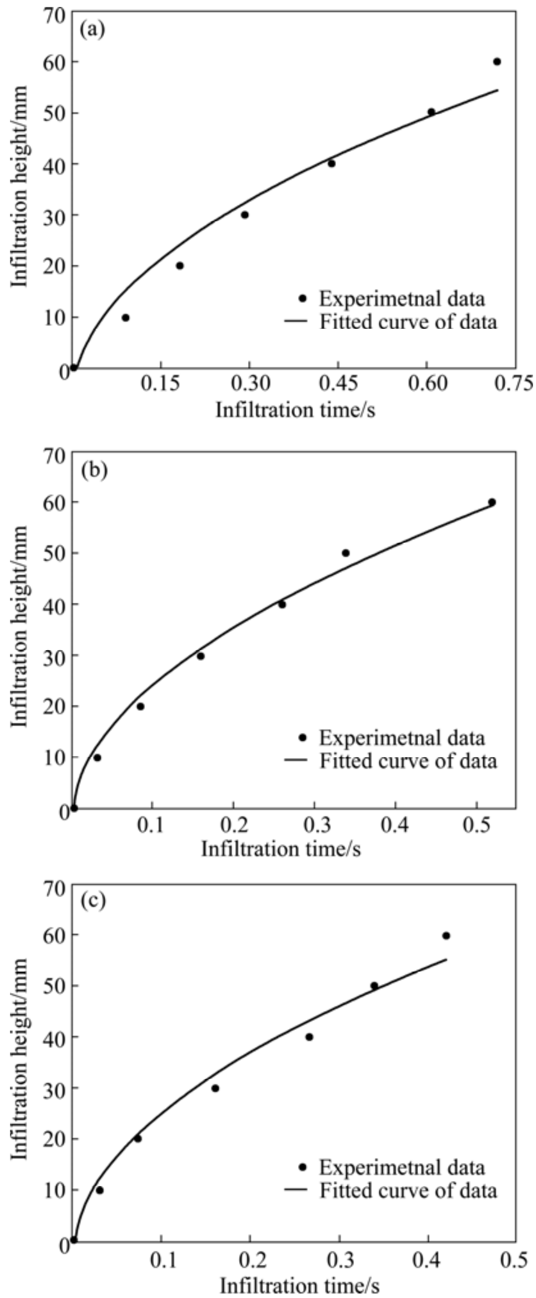

Fig. 6 Infiltration height—time curves under different infiltration pressures: (a) 0.4 MPa; (b) 0.5 MPa; (c) 0.6 MPa

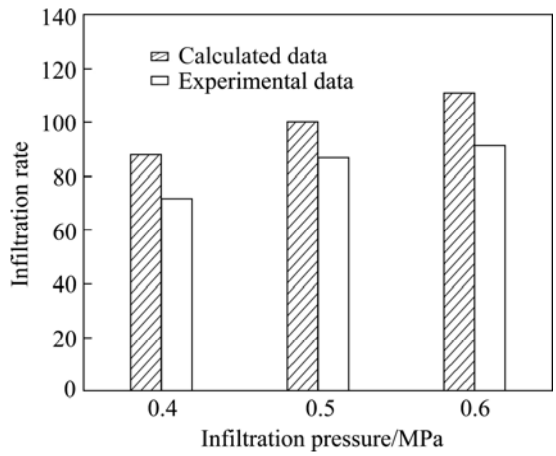

Fig. 7 Infiltration rate under different infiltration pressure

average infiltration rate increases, but the increase amplitude decreases gradually. When the infiltration pressures are 0.4, 0.5, 0.6 MPa, the average infiltration rates are 71.61, 86.97 and 91.28, respectively.

3. 4 Effect of infiltration pressure on microstructure

During the infiltration process, liquid metal will preferentially penetrate into the large interspaces between adjacent carbon fibers, and then fill the small pores under larger pressure. Therefore, increasing of the infiltration pressure not only accelerates the infiltration process but will also improves the infiltration quality.

The density of the composites was measured using drainage method. Three samples were cut from the top, middle and bottom of the composites. When the infiltration pressures were 0.4, 0.5 and 0.6 MPa, the average densities were 1.627, 1.667 and 1.697 g/cm^3, respectively. Figure 8 shows the SEM images of C_f/Mg composites fabricated under different infiltration pressures. It can be seen that there exist lots of micro pores with diameter of 50−100 μm, when the infiltration pressure is 0.4 MPa. When the infiltration pressure is 0.5 MPa, the amount and dimension of pores reduce obviously. When the infiltration pressure rises to 0.6 MPa, there is no pore in the composite. With the increase of infiltration pressure, the infiltration degree is improved. The main reasons are as follows: 1) increasing infiltration pressure accelerates the infiltration process, reduces the heat loss, and inhibits the solidification of liquid metal; 2) it improves the filling capability of liquid alloy into the finer pores.

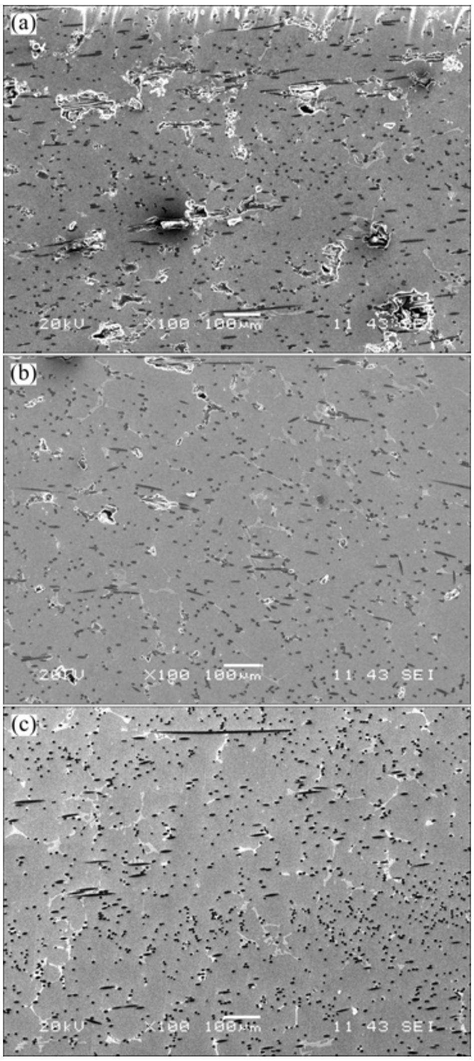

Fig. 8 SEM images of C_f/Mg composites under different infiltration pressure: (a) 0.4 MPa; (b) 0.5 MPa; (c) 0.6 MPa

4 Conclusions

1) A dynamic measuring apparatus for infiltration process was developed, and the whole infiltration process could be carried out in a sealed container. The threshold pressure and infiltration behavior were measured based on the similarity between seepage field and temperature field.

2) For 10% chopped carbon fiber preform, the threshold infiltration pressure for liquid magnesium alloy at 800 °C was 0.048 MPa. It was larger than the calculated value of 0.02 MPa, which was due to the uneven temperature gradient of the preform.

3) The infiltration height—time relationships under different infiltration pressures were obtained from the experimental results. Increasing infiltration pressure could improve the infiltration rates effectively, but the effect decreased gradually.

4) With the increase of infiltration pressure, pore defects were eliminated effectively. When the infiltration pressure rose to 0.6 MPa, high quality C_f/Mg composites were obtained.

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液态金属浸渗纤维预制体的临界压力与浸渗行为

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摘　要：开发液态金属浸渗过程动态测量装置，以其深入研究液态金属在纤维预制体内部的渗流行为。利用该装置进行液态镁合金浸渗 10%体积分数短碳纤维预制体的研究，测量浸渗过程中的临界浸渗压力，研究浸渗压力对浸渗速率的影响，观察不同浸渗压力下复合材料的微观形貌。结果表明：浸渗所需的临界压力为 0.048 MPa，大于理论计算结果；随着浸渗压力的增大，液态金属的浸渗速率提高，但提高的幅度逐步降低；增大浸渗压力能够有效消除复合材料中的未浸渗缺陷；浸渗压力为 0.6 MPa 时获得组织致密的 C_f/Mg 复合材料。

关键词：金属基复合材料；碳纤维；镁；预制体；浸渗；临界压力；浸渗速率

(Edited by Chao WANG)