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ARTICLE

Effect of Pouring Temperature on Microstructure and Mechanical Properties of Continuous CF-Reinforced Al-10Mg Matrix Composites

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Abstract: The Al-10Mg composites containing 10wt% Mg reinforced by 6.8vol% continuous carbon fibers (CFs) were prepared by twin-roll casting method at different pouring temperatures (943, 963, 983, and 1003 K). The Ni coating on the CF surface was used to inhibit the formation of Al₄C₃ brittle phase. Results show that CFs and Al-10Mg matrix are well bonded without the formation of Al₄C₃ brittle phase. The ultimate tensile strength (UTS) of the composites is firstly increased and then decreased with increasing the pouring temperature. When the pouring temperature is 963 K, UTS of CFs/Al-10Mg composites reaches 185 MPa, which is increased by 41.2% compared with that (131 MPa) of the substrate. Meanwhile, the fracture surface of CFs/Al-10Mg composites was also investigated. The good interface bonding between CFs and Al matrix can be observed. Thus, the twin-roll casting is an effective method to prepare CFs/Al-10Mg composites with a promising prospect.

Key words: Al-10Mg composites; continuous carbon fibers; twin-roll casting; fracture surface; tensile strength

The continuous carbon fibers (CFs) possess unique characteristics, including the high tensile strength (more than 3.5 GPa^[1]), high Young's modulus (higher than 200 GPa^[2]), and low density (1.75~2.20 g/cm³^[3]), and they are lighter than other alloying elements (Mn, Zn, Zr) for Al-based alloys^[4]. Thus, CFs are usually considered as an reinforcement in metal matrix composites^[5]. Furthermore, CFs-reinforced metal matrix composites attract extensive attention from the aerospace, automobile, and electronics industries^[6,7].

However, the poor wettability between CFs and Al matrix restricts their application. The researches of CFs/Al composites mainly focus on the optimization of preparation methods, such as metallurgy processing, stirring casting processing, and pressure infiltration processing^[8-12]. For instance, Pippel et al^[13] revealed that the formation of aluminum carbide (Al₄C₃) is detrimental to the mechanical properties of CFs/Al composites. However, Tham et al^[14] found that the formation of Al₄C₃ is beneficial to the chemical bonding between the matrix and the reinforced phase. Etter et

al^[15] observed that the addition of alloying elements, especially Si element, can inhibit the formation of Al₄C₃. Li et al^[16] also found that the addition of Mg element can effectively inhibit the appearance of brittle phase. Singh et al^[17] prepared the short CFs-reinforced Al matrix composites by stirring casting technique. The Cu coating is deposited on CFs surface to avoid interface reactions. Zhang et al^[18] fabricated woven CFs-reinforced Al5083 matrix composites via semisolid-rolling process. The bending strength and tensile strength of the composites are enhanced by 25% and 52%, respectively. Kusakabe et al^[19] considered the silicon carbide (SiC) as a promising coating material for CFs/Al composites.

The solid and liquid processes are commonly used for preparation of CFs/Al composites^[1]. Although the twin-roll casting method is widely used to prepare the metal matrix composites, its application on CFs/Al composites is rarely reported^[20]. Therefore, the rolling pressure is introduced to fabricate CFs/Al composites, which can provide a significant enhancement for continuous production of CFs/Al

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composites. In addition, Zhang et al.^[18] also investigated the effects of rolling speed and covering matrix on the composite properties. However, the results cannot be applied for continuous production, and the tensile property of composites is only improved by 52%. In addition, the pouring temperature is an important factor on the mechanical properties of composites. According to Ref. [21], the semisolid state material has better infiltration behavior and can protect CFs from damage. Besides, the coating is vital for composite fabrication^[22,23]. Thus, the Ni coating was deposited on CF surface.

In this research, the continuous CFs-reinforced Al-10Mg composites were prepared through twin-roll casting method. The Ni coating on CFs surface was used to inhibit the formation of Al_4C_3 and to improve the wettability between CFs and the molten Al matrix. The effect of the pouring temperature on the microstructure and mechanical properties of CFs/Al-10Mg composites was discussed. The strengthening mechanism of CFs/Al-10Mg composites was also investigated.

1 Experiment

The experiment materials were continuous CFs (approximately 12 000 fibers per bundle with mean diameter of 7 μm) and Al-10Mg alloy (88.3wt% Al, 10wt% Mg, and other alloying elements), as shown in Fig. 1. The continuous CFs were uniformly distributed in the composites with the volume fraction of 6.8vol%. The continuous CFs were firstly electroplated through the self-made electroplating device for 6 min under the condition of voltage at 2.0 V and current at 0.35 A. The obtained coatings had a thickness of 0.6 μm . Fig. 2a shows the schematic diagram of the twin-roll casting process. The Al-10Mg alloy was put in a crucible and then heated to different temperatures (943, 963, 983, and 1003 K). Afterwards, the continuous CFs were fixed at both ends of the

twin-roll casting machine and the pre-tensioning device started. The molten Al-10Mg alloy was poured into the front box and then the machine started. CFs/Al-10Mg composites were obtained after solidification in the air. For comparison, the Al-10Mg substrate specimens were also fabricated under the same conditions.

The microstructures of CFs/Al-10Mg composites were observed via FEI Sirion 200 scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS). The main phase composition of CFs/Al-10Mg composites was determined through X-ray diffraction (XRD, Cu $K\alpha$ radiation) with $2\theta=10^\circ\sim 90^\circ$ and a scanning speed of $2^\circ/\text{min}$ at 40 kV and 30 mA. The tensile tests were conducted using a testing machine at a crosshead speed of 1 mm/min. The thickness of specimens was 2 mm, and the dimension of the tensile specimens is shown in Fig.2b. After the tensile tests, SEM was used to observe the fracture surface of the specimens.

2 Results and Discussion

2.1 Microstructure

The microstructures of CFs/Al-10Mg composites are shown in Fig. 3. When the pouring temperature is 943 K, many un-infiltrated defects can be obviously found on the composite interface, as shown in Fig. 3a and 3b. Moreover, CFs are seriously damaged, which is attributed to the early solidification process. Thus, CFs are broken by the rolling pressure, which significantly weakens the wettability. There are some pores in the interfaces, which is mainly due to the mismatch in coefficient of thermal expansion between CFs and Al-10Mg matrix^[24]. The presence of pores is harmful to the mechanical properties of composites. With increasing the pouring temperature to 963 K, CFs are uniformly distributed in the Al-10Mg alloy matrix, as shown in Fig.3c and 3d. The surface of CFs and Al-10Mg alloy matrix is smooth and

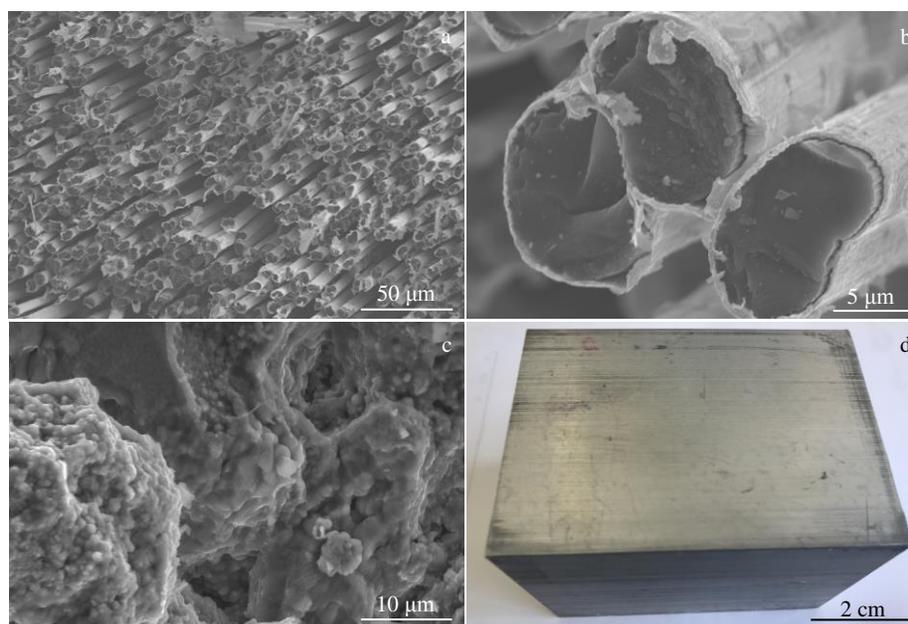


Fig.1 SEM morphologies of continuous CFs (a, b) and Al-10Mg matrix (c); appearance of Al-10Mg alloy ingot (d)

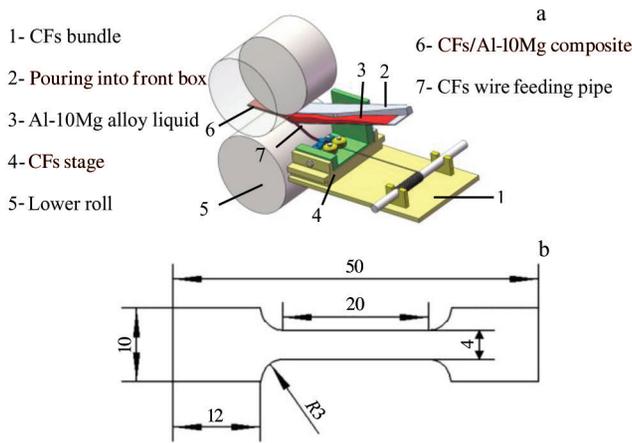


Fig.2 Schematic diagrams of twin-roll casting process (a) and tensile specimen (b)

uniform without any defects, and no pores can be observed. In addition, no defects caused by broken CFs appear in the composites, because the molten Al-10Mg alloy matrix is transformed into semisolid state during cooling process. Zhang et al^[25] found that the semisolid rolling is optimal for CFs/Al-10Mg composite preparation by twin-roll casting. It can be concluded that the suitable pouring temperature to prepare CFs/Al-10Mgss composites by twin-roll casting method is 963 K.

When the pouring temperature is 983 K, the microstructures of the obtained composites are shown in Fig.3e and 3f. It can be seen that CFs are well infiltrated into the Al-10Mg alloy matrix with a uniform distribution. However, in the interface between CFs and Al-10Mg alloy matrix, some casting defects can be observed after the forming process. Moreover, a small burning area can be observed at the outermost layer of CFs bundle, indicating that the high temperature prevents the solidification process. Fig.3g and 3h show the microstructures of composites at the pouring temperature of 1003 K. A large burning region is formed at CFs bundle, which seriously

affects the mechanical properties of the composites. Therefore, the pouring temperature of 963 K is optimal.

Fig.4 shows the microstructure and element distributions of the composites at the pouring temperature of 963 K. A good bonding at CFs/Al-10Mg composite interface is achieved without any defects. The Ni coating retains its original morphology and prevents CFs from damage. Fig. 5 shows XRD pattern of CFs/Al-10Mg composites at the pouring temperature of 963 K. The distinct peaks at 2θ of 38.56° , 44.78° , 65.12° , 78.22° , and 82.42° correspond to the Al peaks, which are $2\theta=38.47^\circ$, 44.71° , 65.10° , 78.23° , and 82.44° according to PDF#04-006-2586, respectively. It must be noted that XRD pattern of CFs/Al-10Mg composites has a deviation of 0.1° mainly due to the experiment error. Moreover, the distinct peak of CFs/Al-10Mg composites at $2\theta=26.44^\circ$ corresponds to the C peak, which is $2\theta=26.5^\circ$ according to PDF#00-026-017. Lalet et al^[26] found that the peaks of Al_4C_3 ($2\theta=31.13^\circ$, 31.77° , 35.89° , 40.12° , and 55.05° according to PDF#04-008-7186) can be detected in CFs/Al composites. However, no peaks of Al_4C_3 can be found in Fig. 5. Furthermore, Tham et al^[14] found that the addition of alloying elements has a certain effect on the appearance of Al_4C_3 phase. In this research, the existence of Mg plays a very important role.

Fig. 6 shows SEM microstructure of CFs/Al-10Mg composites and EDS line scanning from point A to point B in Fig.6a. The gray areas are the Al-10Mg alloy matrix while the dark heart-shaped areas are CFs. The content of Al element is relatively low in CFs bundle, and the content of Ni element is relatively high around CFs. Ni coating may react with the Al matrix to form Al_3Ni , but the Al-Ni product cannot be seen from Fig.6a. Moreover, the oxygen content is high between the Al matrix and CFs, which may be due to the formation of intermetallic oxides at the interface, such as Al_2O_3 and MgO. Zhang et al^[27] found that the formation of Al_2O_3 is beneficial to enhance the interface bonding of CFs/Al-10Mg composites.

The wettability between Al matrix and CFs is one of the important factors influencing the properties of continuous CFs-

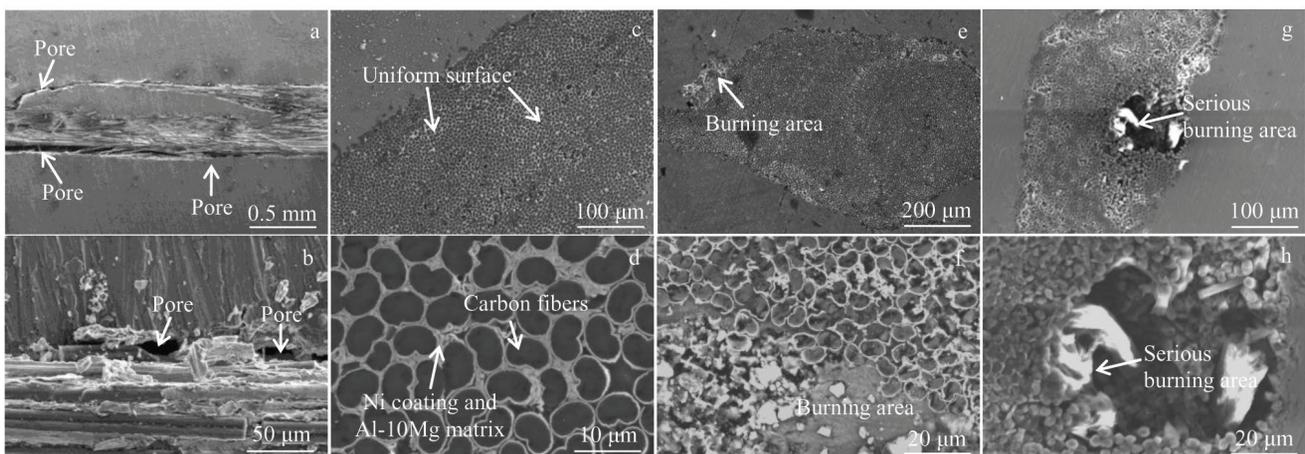


Fig.3 SEM microstructures of CFs/Al-10Mg composites at different pouring temperatures: (a, b) 943 K, (c, d) 963 K, (e, f) 983 K, and (g, h) 1003 K

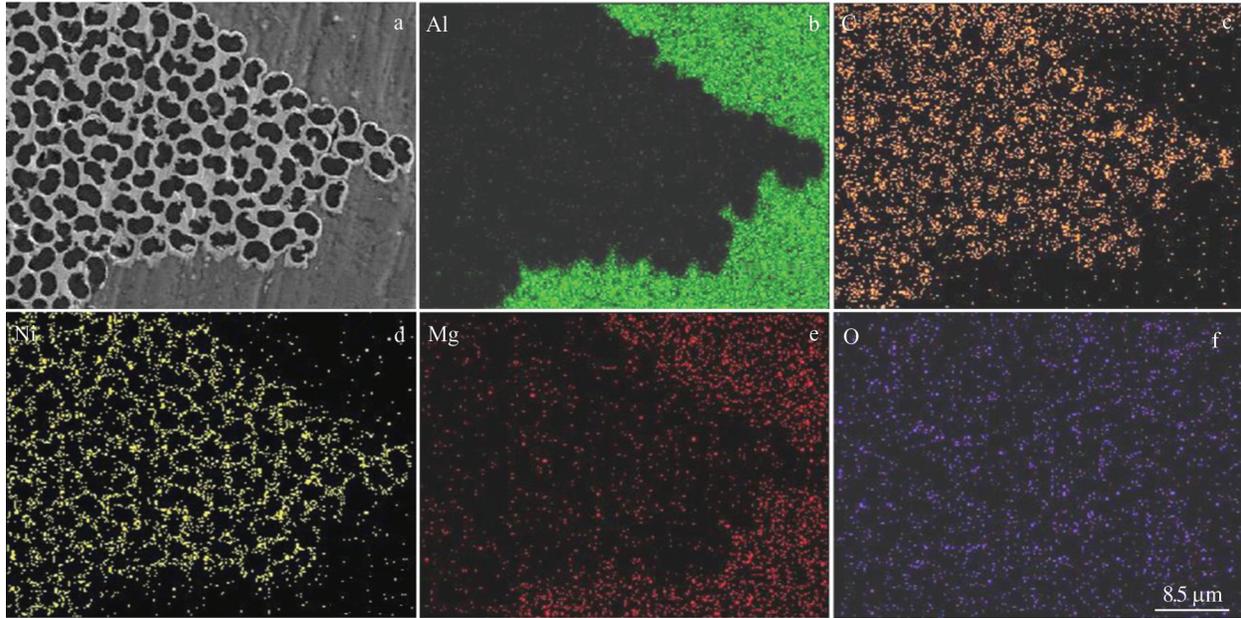


Fig.4 SEM microstructures (a) and EDS element distributions of Al (b), C (c), Ni (d), Mg (e), and O (f) in CFs/Al-10Mg composites at pouring temperature of 963 K

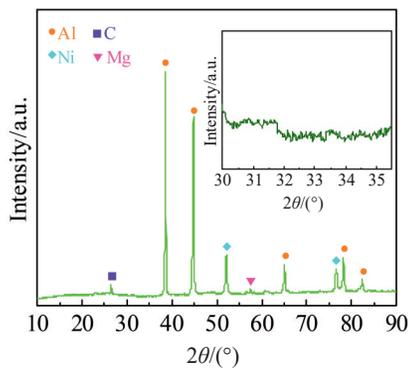


Fig.5 XRD pattern CFs/Al-10Mg composites at pouring temperature of 963 K

reinforced Al-10Mg alloy matrix composites. In addition, the Al_4C_3 brittle phase also has a significant impact on the mechanical properties of composites^[28]. Al_4C_3 can improve the tensile strength of CFs/Al-10Mg composites due to its brittle characteristic^[14] but can easily cause stress concentration^[13]. Wang^[29] and Wang^[30] et al found that the addition of Si and Mg alloying elements can effectively inhibit the formation of Al_4C_3 . Thus, 10wt% Mg was used in this research to inhibit the formation of Al_4C_3 . The difference in density of the molten Al and CFs is also a problem in twin-roll casting process. The continuous CFs are likely to float on the surface of molten Al-10Mg alloy^[20]. Therefore, a pre-tensioning device was used to ensure the uniform distribution of CFs.

2.2 Mechanical properties

Fig. 7a shows the tensile stress-strain curves of Al-10Mg alloy and CFs/Al-10Mg composites. The ultimate tensile strength (UTS) of CFs/Al-10Mg composites and Al-10Mg substrate is shown in Fig. 7b and 7c, respectively. UTS of the

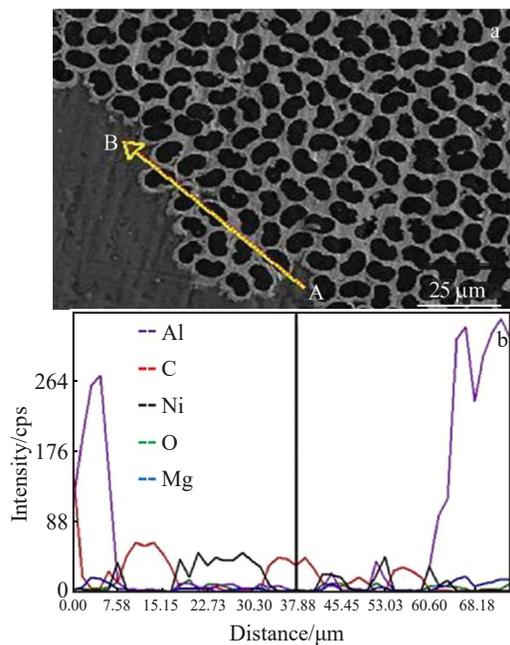


Fig.6 SEM microstructure (a) and EDS line scanning along line A-B (b) of CFs/Al-10Mg composites

composites is firstly increased and then decreased with increasing the pouring temperature. When the pouring temperature is 963 K, CFs/Al-10Mg composites have the highest tensile strength. Owing to the addition of continuous CFs, UTS of the composites is raised from 131 MPa to 185 MPa, increasing by 41.2% compared with that of the Al-10Mg substrate. However, the elongation of CFs/Al-10Mg composites is lower than that of Al-10Mg substrate. Due to different distribution characteristics of the continuous CFs, the stress

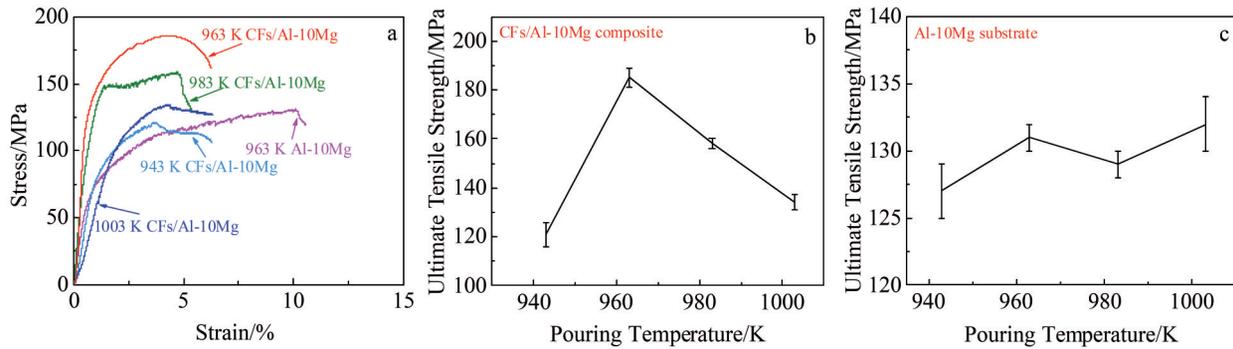


Fig.7 Tensile stress-strain curves of Al-10Mg substrate at 963 K and CFs/Al-10Mg composites at different pouring temperatures (a); UTS of CFs/Al-10Mg composites (b) and Al-10Mg substrate (c) at different pouring temperatures

changes among the composites. For CFs/Al-10Mg composites at the pouring temperature of 963 K, the stress-strain curve has an upward trend with a small deformation, and then a more rapid increase occurs until the specimen fails. It can be easily observed that both the addition of continuous CFs and the suitable pouring temperature have noticeable effects on the mechanical properties of the composites.

The interface between CFs and Al matrix plays a major role in the final properties of CFs/Al-10Mg composites. The interface microstructures of the composites prepared by different pouring temperatures are shown in Fig. 8. The Ni coating on the CFs surface improves the wettability and interface bonding of the composites. Fig. 8a and 8b show the interface microstructures of the composite at pouring temperature of 943 K: many pore defects appear after the rolling process, which is caused by the shrinkage behavior of Al-10Mg alloy matrix during solidification. As the pouring temperature increases to 963 K, no obvious defects can be observed at the interface, which is related to the fact that the increased pouring temperature delays the solidification process, as shown in Fig. 8c and 8d. Since Mg element can improve the wetting behavior during the solidification process, the Mg content in the composites was investigated,

and the results are shown in Fig.9 and Table 1. Point 1 is near CFs and Point 2 is far from CFs. It can be seen that Point 1 has a higher Mg content than Point 2 does, indicating the segregation of Mg. With increasing the pouring temperature to 983 K, some powdered CFs appear at the interface, as shown in Fig. 8e and 8f, which is attributed to the high temperature oxidation during the rolling process. With further increasing the pouring temperature to 1003 K, the oxidation phenomenon becomes serious between the Al-10Mg alloy matrix and the reinforcement, as shown in Fig. 8g and 8h. Therefore, the pouring temperature of 963 K is optimal.

2.3 Fracture morphology

The fracture morphologies of CFs/Al-10Mg composites after tensile tests are shown in Fig. 10. The fracture morphologies are different due to the interface bonding state between CFs and Al-10Mg alloy matrix. For the composites obtained at the pouring temperature of 943 K, the fracture morphology is mainly characterized by the lamellar structure, as shown in Fig. 10a and 10b. Some CFs are drawn out from CFs/Al-10Mg composites. Fig. 10c and 10d show the fracture morphologies of CFs/Al-10Mg composites obtained at pouring temperature of 963 K. There are many little dimples at the fracture surface, indicating the typical ductile fracture.

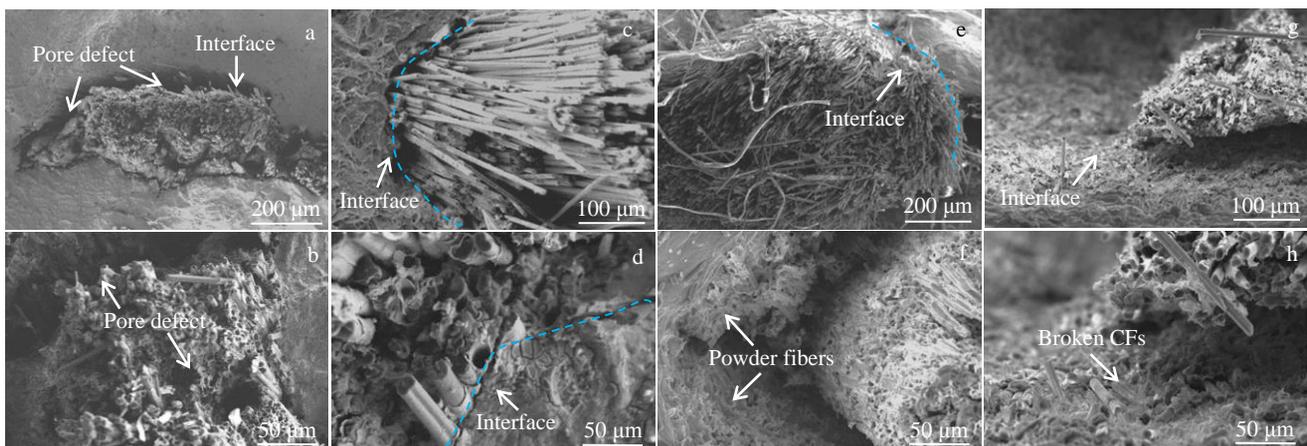


Fig.8 Interface morphologies of CFs/Al-10Mg composites at different pouring temperatures: (a, b) 943 K, (c, d) 963 K, (e, f) 983 K, and (g, h) 1003 K

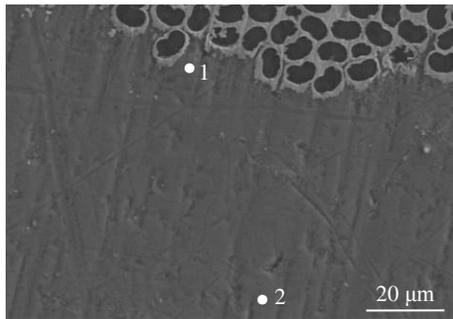


Fig.9 SEM point scanning positions of CFs/Al-10Mg composites

Table 1 Element contents of Point 1 and Point 2 in Fig.9 (wt%)

Point	Mg	Al	C	Ni	O
1	19.8	65.3	2.35	1.05	11.5
2	11.2	78.9	0.2	0.6	9.1

This may be attributed to the well bonding between CFs and Al-10Mg alloy matrix. The dimple depth is determined by the ductility of CFs/Al-10Mg composites. Thus, the mechanical properties of composites are determined by matrix (Al-10Mg), reinforcement (CFs), and interface bonding.

When the pouring temperature is 983 and 1003 K, the

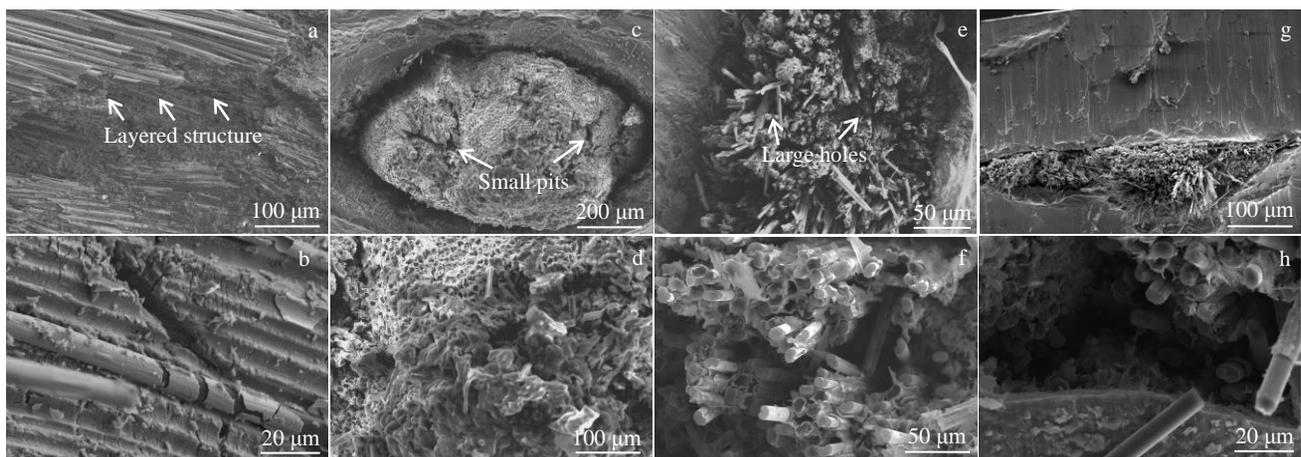


Fig.10 Fracture morphologies of CFs/Al-10Mg composites at different pouring temperatures: (a, b) 943 K, (c, d) 963 K, (e, f) 983 K, and (g, h) 1003 K

fracture morphologies of the composites are shown in Fig. 10e~10h. Many larger holes can be seen at the fracture surfaces due to the poor wettability between Al matrix and CFs^[31]. In this case, the plastic failure of Al-10Mg alloy matrix plays an important role in the fracture behavior of CFs/Al-10Mg composites.

According to the above analysis, due to the weak interface bonding, CFs fail at low stress and the crack is propagated along the boundaries, leading to the composite failure. Therefore, the appropriate pouring temperature for well wettability between CFs and Al matrix is necessary, and it can also improve the mechanical properties of the composites.

3 Conclusions

1) Ni-coated continuous carbon fibers (CFs)-reinforced Al-10Mg matrix composites can be prepared by twin-roll casting method, and the optimal pouring temperature is 963 K.

2) The addition of continuous CFs provides an obvious improvement in the tensile strength of the composites by 41.2%, compared with that of Al-10Mg alloy, because of the uniform distribution of continuous CFs and the good interface bonding between CFs and Al-10Mg alloy matrix.

References

- Xiao Hao, Lu Yonggen, Zhao Weizhe et al. *Journal of Materials Science*[J], 2014, 49(2): 794
- Edie D. *Carbon*[J], 1998, 36(4): 345
- Morgan P. *Carbon Fibers and Their Composites*[M]. Boca Raton: CRC Press, 2005
- Liu Zhenyi, Zhang Guoding, Li Hong et al. *Materials and Design*[J], 2005, 26: 83
- Baumli P, Sychev J, Budai I et al. *Composites Part A: Applied Science and Manufacturing*[J], 2013, 44: 47
- Miracle D B. *Composites Science and Technology*[J], 2005, 65(15-16): 2526
- Hufenbach W, Andrich M, Langkamp A et al. *Journal of Materials Processing Technology*[J], 2006, 175(1-2): 218
- Tjong S C. *Materials Science and Engineering R: Reports*[J], 2013, 74(10): 281
- Su Hai, Gao Wenli, Chang Mao et al. *The Chinese Journal of Nonferrous Metals*[J], 2010, 20(2): 8 (in Chinese)
- Ouyang Haibo, Li Hejun, Qi Lehua et al. *Journal of Materials Science*[J], 2008, 43(13): 4618
- Yang Yanjiao, Dai Mingjiang, Wen Chenbei et al. *Rare Metal Materials and Engineering*[J], 2017, 46(12): 3663
- Yang Zhiming, Wu Tengeng, Li Jinxu et al. *Rare Metal Materials and Engineering*[J], 2017, 46(4): 869
- Pippel E, Woltersdorf J, Doktor M et al. *Journal of Materials Science*[J], 2000, 35(9): 2279

- 14 Tham L M, Gupta M, Cheng L et al. *Acta Materialia*[J], 2001, 49(16): 3243
- 15 Etter T, Schulz P, Weber M et al. *Materials Science and Engineering A*[J], 2007, 448(1-2): 1
- 16 Li Daguang, Chen Guoqin, Jiang Longtao et al. *Materials Science and Engineering A*[J], 2013, 586: 330
- 17 Singh B B, Balasubramanian M. *Journal of Materials Processing Technology*[J], 2009, 209(4): 2104
- 18 Zhang Junjia, Liu Shichao, Lu Yiping et al. *Materials and Design*[J], 2016, 95: 89
- 19 Kusakabe K, Sha B K, Hayashi J et al. *Carbon*[J], 1996, 32(2): 179
- 20 Shirvanimoghaddam K, Hamim S U, Akbari M K et al. *Composites Part A: Applied Science and Manufacturing*[J], 2017, 92: 70
- 21 Zhang Junjia, Liu Jiaming, Lu Yiping et al. *Journal of Materials Processing Technology*[J], 2020, 281: 116 617
- 22 Zhou Jiming, Zheng Wuqiang, Qi Lehua et al. *Rare Metal Materials and Engineering*[J], 2015, 44(8): 1851
- 23 Zhang Dawei, Zhang Qi, Fan Xiaoguang et al. *Rare Metal Materials and Engineering*[J], 2019, 48(1): 44 (in Chinese)
- 24 Silvain J F, Heintz J M, Lahaye M. *Journal of Materials Science* [J], 2000, 35(4): 961
- 25 Zhang Junjia, Liu Shichao, Lu Yiping et al. *Journal of Materials Science and Technology*[J], 2017, 33(7): 623
- 26 Lalet G, Kurita H, Miyazaki T et al. *Materials Letters*[J], 2014, 130: 32
- 27 Zhang Shaofeng, Chen Guoqin, Pei Risheng et al. *Materials Science and Engineering A*[J], 2015, 647: 105
- 28 Steffens H D, Reznik B, Kruzhanov V et al. *Journal of Materials Science*[J], 1997, 32(20): 5413
- 29 Wang Chenchong, Chen Guoqin, Wang Xu et al. *Metallurgical and Materials Transactions A*[J], 2012, 43(7): 2514
- 30 Wang Xu, Jiang Daming, Wu Gaohui et al. *Materials Science and Engineering A*[J], 2008, 497(1-2): 31
- 31 Shi Xiaohong, Zeng Xierong, Li Hejun et al. *Rare Metal Materials and Engineering*[J], 2011, 40(3): 403

浇注温度对连续碳纤维增强 Al-10Mg 基复合材料组织和力学性能的影响

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摘要: 通过双辊铸轧工艺制备了连续碳纤维增强的铝镁 (10% Mg, 质量分数) 基复合材料, 其中碳纤维的体积分数是 6.8%。对不同浇注温度 (943、963、983 和 1003 K) 下获得的复合材料的组织和力学性能进行了研究。通过在碳纤维表面上电镀镍涂层以抑制脆性相 Al₄C₃ 的形成。结果表明: 碳纤维与铝镁基体具有相对较好的浸润性, 并且没有形成 Al₄C₃ 脆性相。随着浇注温度的升高, 复合材料的抗拉伸强度 (UTS) 先升高后降低。当浇注温度为 963 K 时, 复合材料的抗拉伸强度达到了 185 MPa, 比基体材料 (131 MPa) 提高了 41.2%。此外, 对复合材料的拉伸断面进行研究, 进一步证实了碳纤维与铝镁基体之间良好的界面结合。双辊铸造法是制备碳纤维增强铝镁基复合材料的有效方法, 且具有良好的研究前景。

关键词: Al-10Mg 复合材料; 连续碳纤维; 双辊铸轧; 断口形貌; 拉伸性能

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