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ARTICLE

Mechanical Properties and Failure Behavior of 7075Al Matrix Composites Hybrid Reinforced by Carbon Fibers-FeCoCrNiAl High-Entropy Alloy Particles

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Abstract: To investigate the effects of double-scaled reinforcements on the tensile properties and failure behavior of aluminium matrix composites, Ni-Co-P coated carbon fibers reinforced 7075Al matrix composites (CF/Al), FeCoCrNiAl high-entropy alloy particles reinforced 7075Al matrix composites (HEA_p/Al) and Ni-Co-P coated carbon fibers and FeCoCrNiAl high-entropy alloy particles hybrid reinforced 7075Al matrix composites (CF-HEA_p/Al) were fabricated by vacuum hot pressing sintering under the pressure of 30 MPa at 580 °C for 10 min. The microstructure and the fracture morphologies of the composites were observed, and the tensile properties of composites were characterized. Results show that the yield strength (YS) and ultimate tensile strength (UTS) of CF-HEA_p/Al composites are firstly increased and subsequently decreased with increasing the CFs content from 0vol% to 40vol%, while the elongation is decreased. Owing to the hybrid reinforcing effects of Ni-Co-P coated carbon fibers and FeCoCrNiAl high-entropy alloy particles, the YS and UTS of CF-HEA_p/Al composites have larger improvement compared with those of CF/Al and HEA_p/Al composites, and the fracture morphology is characterized by mixed rupture modes of ductile rupture of 7075Al matrix and fiber pull-out and fracture.

Key words: carbon fibers; FeCoCrNiAl high-entropy alloy particles; hybrid reinforcing effects; mechanical properties; fracture behavior

Owing to the excellent mechanical performances and low density, aluminium matrix composites have broad applications in aerospace, marine ships, military equipment and other fields^[1-3]. According to the geometric shapes, the reinforcements can be divided into discontinuous reinforcements (such as Al₂O₃^[4], SiC^[5], HEA_p^[6] and CNTs^[7]) and continuous reinforcements (like metal fibers^[8], basalt fibers^[9] and carbon fibers (CFs)^[10]). Traditionally, the mechanical performances of composites reinforced by single reinforcement are far lower than their theoretical performance because of insufficient utilization of reinforcement effect, which only can be regulated through interface bonding state and distribution of reinforcements. The problems restrain the

depth optimization of performances and further development of aluminium matrix composites. Introduction of double-scaled or multiple reinforcements in composites can not only integrate the advantages of each component but also improve mechanical properties by synergy effects of the multiple strengthening mechanisms^[11]. As a new design and preparation concept of composites, the hybrid effects of double-scaled or multiple reinforcements on aluminium matrix composites are expected to obtain excellent comprehensive performances and thus to expand their application.

Currently, the hybrid effects on performances of aluminium matrix composites have been reported. Zhou et al^[12-13] selected carbon fibers as continuous reinforcements and ZrC particles

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as discontinuous reinforcements for the hybrid strengthening of aluminium matrix composites. By the comparison with alloy matrix and monolithic reinforcement reinforced composites, the tensile strength of hybrid composites has a significant improvement, which is attributed to the excellent strengthening effects including load transfer effect, Orowan strengthening, dislocation strengthening and others. Likewise, the SiC nanoparticles and carbon fibers are used as the hybrid reinforcing phases to reinforce pure Al^[14]. The hardness and tensile strength of SiC-CF/Al composites are larger than those of pure Al and SiC/Al composites, which is caused by the synergistic strengthening effects of Orowan strengthening of SiC and an effective load transfer between carbon fibers and Al matrix. Besides, the coefficient of thermal expansion of SiC-CF/Al composites is lower than that of SiC/Al composites^[15]. Interestingly, high-entropy alloy particles (HEA_p) are used as the discontinuous reinforcements for hybrid strengthening of metals matrix composites. For the HEA_p and SiC particles hybrid reinforced 7075Al composites, HEA_p can increase the dislocation density in Al matrix so that the initiation of microcracks at interface is restrained, which are the main reasons that the fracture ductility of hybrid composites is superior to that of SiC reinforced 7075Al composites^[16]. In comparison with the Al matrix composites reinforced by uniformly dispersed SiC and HEA_p, the SiC particles coated HEA_p reinforced Al composites possess the optimized properties^[17]. Also, HEA_p and B₄C hybrid reinforced 6061Al matrix composites have excellent tensile strength, hardness and impact strength compared with 6061Al^[18]. Hybrid of different reinforcements can build novel microstructural features of hybrid composites, which is solely responsible for the obtained mechanical properties^[19]. As the comparatively ideal reinforcements, carbon fibers and HEA_p are expected to be applied for hybrid strengthening of composite materials. Regrettably, the research on carbon fibers and HEA_p hybrid composites is rarely reported.

In this study, Ni-Co-P coatings modified carbon fibers and FeCoCrNiAl HEA_p were selected as double-scaled reinforcements to reinforce 7075Al matrix composites fabricated by vacuum hot pressing sintering technique with direct heating. Through the comparison of tensile properties of 7075Al matrix composites reinforced with carbon fibers, FeCoCrNiAl HEA_p and double-scaled reinforcements, effects of double-scaled reinforcements on coordination of mechanical properties of composites were discussed, which may provide a theoretical basis for the development and application of double-scaled reinforcement phases in metals matrix composites.

1 Experiment

7075Al powders (7075Al), carbon fibers (CFs) and FeCoCrNiAl HEA_p were selected as metal matrix, continuous reinforcement and discontinuous reinforcement, respectively. The fabrication process of Ni-Co-P coated CFs and HEA_p hybrid reinforced 7075Al matrix composites (CF-HEA_p/Al) is shown in Fig. 1, and described as follows. Firstly, 7075Al and HEA_p were mixed in a volume fraction of 9:1 using vacuum ball milling with the ball to powder ratio of 10:1 at 350 r/min for 6 h. Otherwise, CFs modified by Ni-Co-P coatings with the thickness of about 0.3 μm were prepared by palladium-free electroless plating process, and the experimental details were described in previous work^[20]. Secondly, Ni-Co-P coated CFs and HEA_p/Al mixed powders were packed into a graphite mold by the method of homogeneous overlap. Lastly, the samples were vacuum sintered at 580 °C under a pressure of 30 MPa for 10 min through direct-heating heat way, and then cooled in the furnace. HEA_p reinforced 7075Al composites (HEA_p/Al) with 10vol% HEA_p, CFs reinforced 7075Al composites (CF/Al) with 30vol% CFs, and CF-HEA_p/Al composites with 10vol% HEA_p and CFs of different content (10vol% , 20vol% , 30vol% and 40vol%) were obtained through the alteration of reinforcement volume fraction.

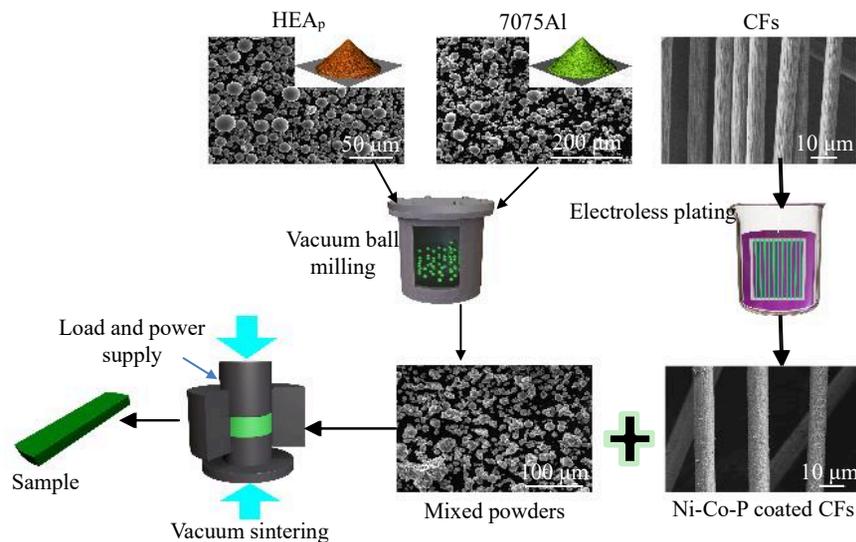


Fig.1 Schematic diagram of fabrication of CF-HEA_p/7075Al composites

The density values of different composites were measured by Archimedes' displacement principle. The tensile properties of composites were tested by the electronic universal testing machine (MTS-E44.304) with a crosshead displacement rate of 0.2 mm/min according to ASTM D3552-17. Tensile samples were polished by waterproof abrasive paper before testing, and the sizes are shown in Fig. 2. Microstructure, element distribution and fracture morphologies of composites were investigated using a scanning electron microscope (SEM, JSM6700F) equipped with energy dispersive spectrometer (EDS).

2 Results and Discussion

2.1 Microstructure of composites with different reinforcements

The microstructures of HEA_p/Al, CF/Al and CF-HEA_p/Al are shown in Fig. 3. Visibly, integrity of HEA_p and CFs morphologies reflects that reinforcements can maintain a complete intrinsic structure, and reinforcements have a homogeneous distribution in the Al matrix and there is no obvious defect at the interfaces between Al matrix and HEA_p and between Al matrix and Ni-Co-P coatings, which indicates that relatively strong interfacial bonding is formed between Al matrix and reinforcements. As seen from the elements mappings of CF-HEA_p/Al in Fig. 3c, element Fe, Cr, Ni and Co have no obvious interface interdiffusion with Al matrix because of high temperature stability of HEA_p. Also, the uniform enrichment distribution of element Ni, Co and P around CFs and regular mapping of C show that Ni-Co-P coatings not only form a stable intermediate layer but also inhibit the interface reaction. Interestingly, element Mg is concentrated around HEA_p and Ni-Co-P coatings. Elements distribution at interface is affected by enthalpy of mixing (ΔH_{mix}). Theoretically, the element Al may diffuse from matrix to FeCoNiCrAl to form FeCoNiCrAl_x ($x > 1$) owing to the obvious difference in the content of Al between matrix and reinforcements. Compared with FeCoNiCrAl_x ($x > 1$)^[21-23], the absolute value of ΔH_{mix} of HEA_p is less, which means that FeCoNiCrAl_x is difficult to form by the diffusion of Al to HEA_p. Also, the negative ΔH_{mix} implies binding force between elements of HEA_p so that Ni, Co, Fe and Cr have no diffusion to Al alloy matrix. In the fabrication of composites, reinforcements are surrounded by Al matrix deformed by thermo-mechanical coupling effects, and high dislocation density is generated, which promotes the diffusion of Mg,

while element Mg cannot diffuse into HEA_p because of the positive ΔH_{mix} between Mg and Co, Cr and Fe, which is the main reason for the formation of Mg accumulation zone around HEA_p. Similarly, the diffusion of Mg to Ni-Co-P coatings is restrained by the positive ΔH_{mix} between Mg and Co^[24]. In addition, the interdiffusions of elements at the interface between Al matrix and reinforcements are inhibited by the shorter holding time.

The interfaces between CFs and 7075Al matrix and between HEA_p and 7075Al matrix are shown in Fig. 4. As shown in Fig. 4a, the interface structure of CF is complete because of the protection from Ni-Co-P coatings, which is consistent with the microstructure and elements distribution of CF-HEA_p/Al composites shown in Fig. 3. In the sintering process, element Al diffuses into the coating and forms Ni-Co-Al intermetallic compound^[25]. The intermetallic compound layers not only restrain the diffusion of element Al to CFs but also reduce the difference of thermal expansion coefficient between CFs ($-0.74 \times 10^{-6}/\text{K}$) and 7075Al ($23.2 \times 10^{-6}/\text{K}$). As shown in Fig. 4b, more dislocations are generated near the interface between HEA_p and 7075Al matrix, which is caused by the distinct differences in thermal expansion coefficient between HEA_p ($13.5 \times 10^{-6}/\text{K}$) and 7075Al matrix.

2.2 Density of different composites

Density values of composites can directly reflect the content of cavity defects in composites. The theoretical density value of composites can be calculated by mixture rule of density, as shown in Eq.(1):

$$\rho_c = V_r \rho_r + V_m \rho_m \quad (1)$$

where ρ_c , ρ_m and ρ_r refer to the density of composites, metal matrix and reinforcements, respectively; V_r and V_m are the volume fraction of reinforcements and matrix, respectively. According to Eq. (1), the theoretical density values of different composites are calculated with the density values of HEA_p (6.72 g/cm³), 7075Al (2.82 g/cm³) and CFs (1.76 g/cm³). The theoretical density and measured density of CF-HEA_p/Al composites with different CFs contents are shown in Fig. 5a. With the increase in CFs content from 0vol% to 40vol% , theoretical density decreases with a gradient variation from 3.21 g/cm³ to 2.62 g/cm³, while measured density decreases with a larger variation from 3.19 g/cm³ to 2.36 g/cm³. Interestingly, differential value between theoretical density and measured density becomes larger with the increase in CFs content, which is up to 0.26 g/cm³ when the CFs content reaches 40vol%. The lower relative density

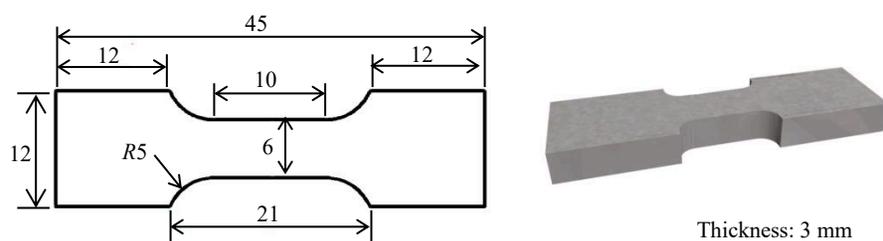


Fig.2 Size of tensile samples of composites

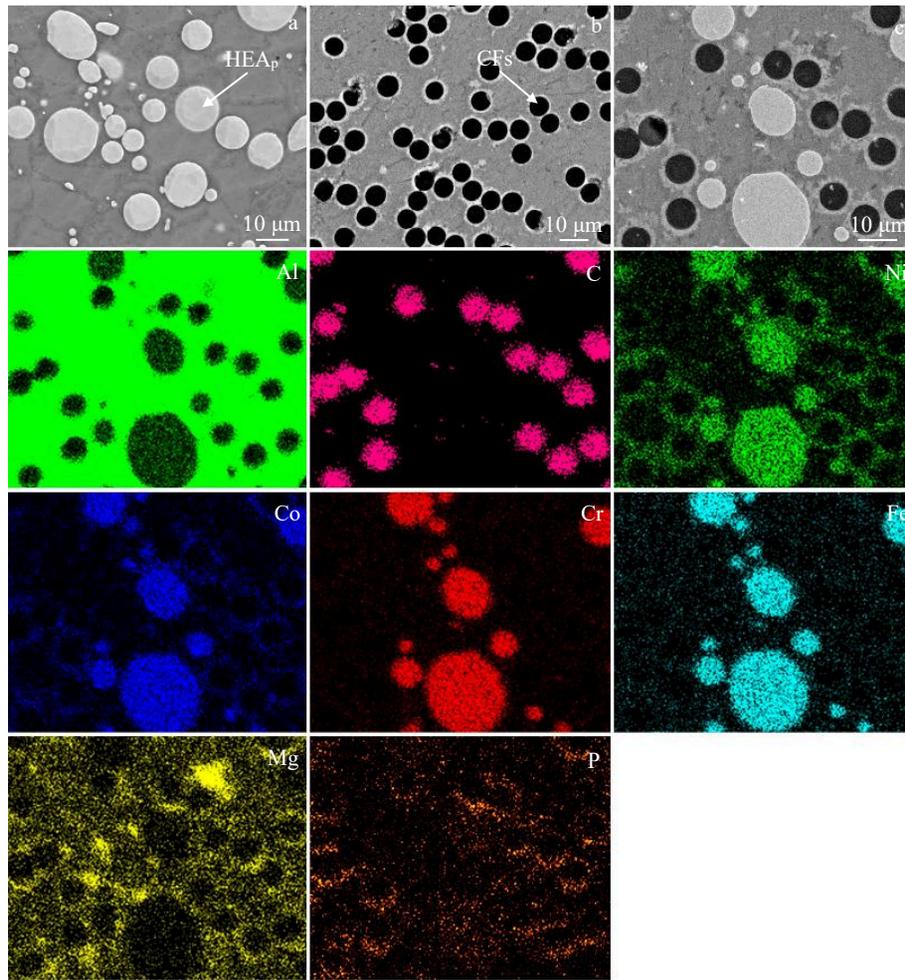


Fig.3 Microstructures of HEA_p/Al composite (a), CF/Al composite (b), and CF-HEA_p/Al composite (c), and the elements mappings of CF-HEA_p/Al composite

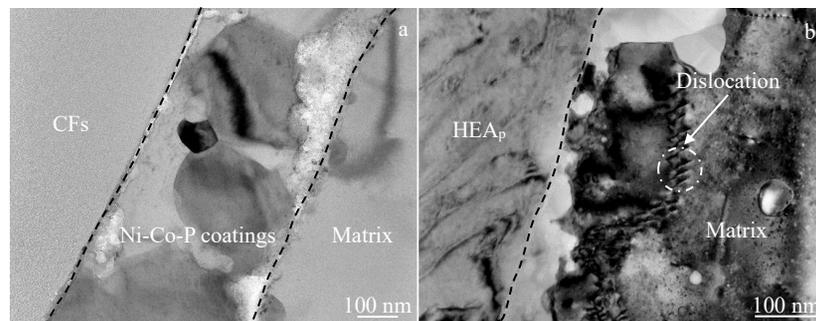


Fig.4 TEM images of interfaces between CFs and 7075Al matrix (a) and between HEA_p and 7075Al matrix (b)

reflects that the CF-HEA_p/Al composites with 40vol% CFs contain more cavity defects compared with other composites. As observed in Fig. 5b, the HEA_p/Al composites have the highest density of 3.19 g/cm³, while CF/Al composites and CF-HEA_p/Al composites have the lowest density of 2.58 g/cm³ and the moderate density of 2.62 g/cm³, respectively. The variation of measured density of composites is mainly determined by the content and type of reinforcements.

Compared with 7075Al matrix and CFs, the addition of HEA_p with higher density improves the density of composites, and the formation of dense interface between HEA_p and 7075Al matrix restrains the generation of cavity defects. Conversely, the addition of CFs with lower density decreases the density of composites. Therefore, the measured density of HEA_p/Al composites is the highest compared with that of CF/Al composites and CF-HEA_p/Al composites. In the preliminary

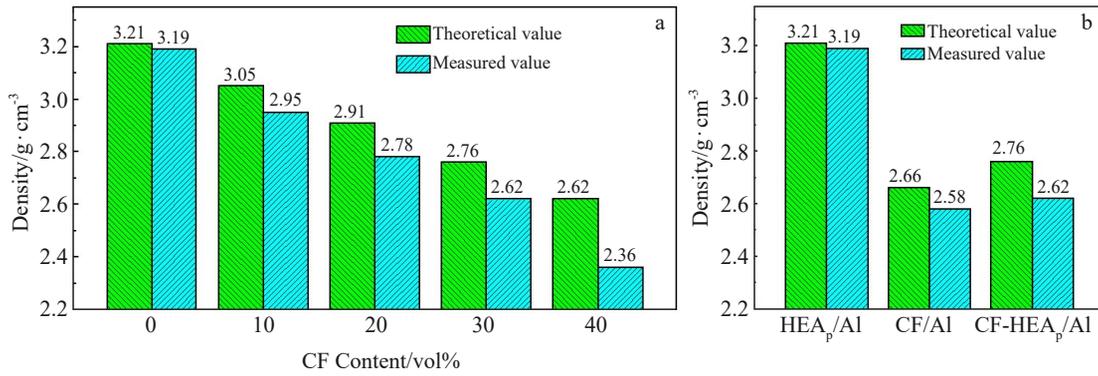


Fig.5 Theoretical density and measured density values of CF-HEA_p/Al composites with different CF contents (a) and composites reinforced by different reinforcements (b)

work^[25], the excessive volume fraction (>30vol%) of CFs reduces the filling ability of metals matrix powder so that cavity defects are generated among fibers, which is the main reason for the improvement in difference between measured density and theoretical density. The ratio of measured density to theoretical density is up to 95% when CFs content does not exceed 30vol%, which illustrates that the optimized CFs content is 30vol% in the fabrication process of CF-HEA_p/Al composites by ball milling-powder metallurgy process.

2.3 Tensile properties of different composites

The tensile properties of CF-HEA_p/Al composites with different CFs contents, are shown in Fig.6a. With the increase in CFs from 0vol% to 40vol%, yield strength (YS) and ultimate tensile strength (UTS) of CF-HEA_p/Al increase firstly and then decrease, and reach the maximum value of 427 and 705 MPa, respectively, when the CFs content is up to 30vol%, which are improved by 2.29 and 2.08 times compared with those of HEA_p/Al composites, respectively. Similarly, the elongation (EL) value is up to the maximum value of 7% when the CFs content is 10vol%, and the EL decreases with the improvement of CFs content from 10vol% to 40vol%. As shown in Fig.6b, the YS and UTS of CF-HEA_p/Al composites are 3.45 times and 5.73 times larger than those of 7075Al. By the comparison of tensile properties of HEA_p/Al, CF/Al and CF-HEA_p/Al composites (Fig. 6b), the YS and UTS of CF-HEA_p/Al composites are better than those of CF/Al and HEA_p/

Al composites, which implies that the hybrid strengthening effects of CFs and HEA_p are superior to single strengthening effects of CFs or HEA_p. Although YS and UTS of CF-HEA_p/Al composites are improved by 40% and 5.5% compared with those of CF/Al composites, respectively, the EL (3.2%) of the former is close to that of the latter.

Fig.7a shows the variation trend of specific strength of CF-HEA_p/Al composites with increase in CFs volume fraction. Clearly, the variation of specific strength is similar to that of the tensile strength, that is, increasing first and then decreasing. The largest specific strength value of CF-HEA_p/Al composites with 30vol% CFs is as high as 270 kN·m/kg, increased by 215.8% and 154.7% compared with that of 7075Al matrix and HEA_p/Al composites, respectively. Fig. 7b shows the variation of specific strength of 7075Al and composites reinforced by different reinforcements. The specific strength of CF-HEA_p/Al composites is the highest, those of CF/Al composites and HEA_p/Al composites are the second, and that of 7075Al matrix is the lowest.

As mentioned above all, the tensile properties of CF-HEA_p/Al composites reinforced by different reinforcements are superior to those of 7075Al matrix, which is attributed to the strengthening effect of different reinforcements. As discontinuous reinforcements, the HEA_p in composites mainly exerts two strengthening effects^[6]. One kind of strengthening effect is load transfer effect, i.e., load transfer from matrix to

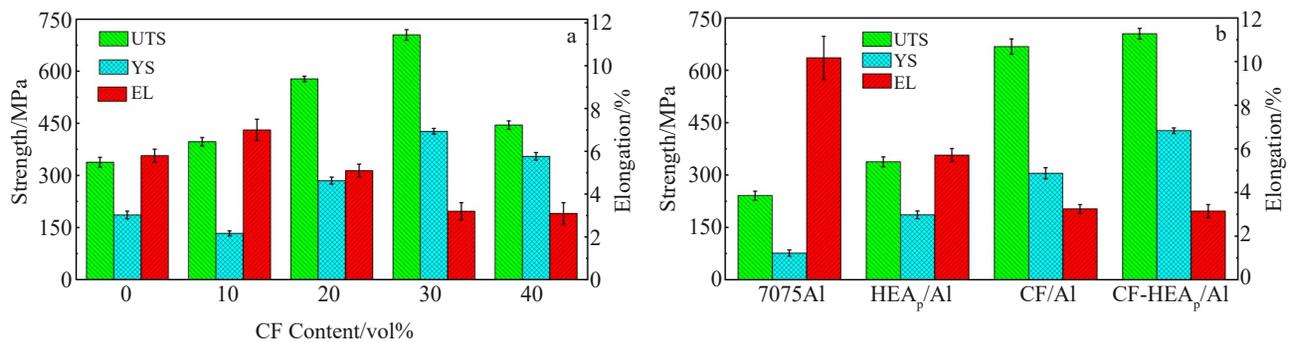


Fig.6 Tensile properties of CF-HEA_p/Al composites with different CF contents (a) and composites reinforced by different reinforcements (b)

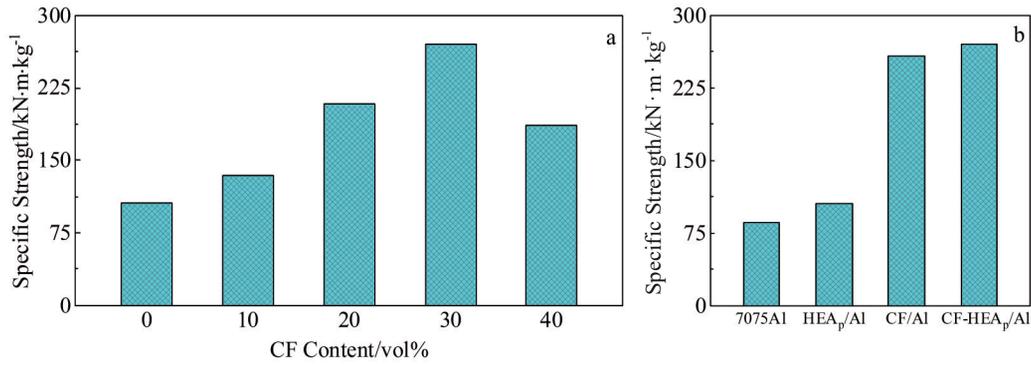


Fig.7 Specific strength of CF-HEA_p/Al composites with different CF contents (a) and composites reinforced by different reinforcements (b)

HEA_p through the metallurgical bonding interface, which is beneficial to improve the yield strength and elastic modulus of composites. The other one is matrix strengthening effect, which is caused by the improvement of dislocation density owing to the difference of thermal expansion coefficient between matrix and reinforcement, and is conducive to the improvement of yield strength. So, the tensile strength of HEA_p/Al composites is higher than that of 7075Al matrix. Unlike HEA_p, CFs play a major bearing role in composite, while the matrix plays the role in forming and load transferring^[20]. Because of the higher tensile strength of CFs, CF/Al composites exhibit excellent tensile strength compared with 7075Al and HEA_p/Al composites. The addition of CF and HEA_p not only strengthens the matrix but also causes the load-bearing component to change from the matrix to CFs. Therefore, CF-HEA_p/Al composites have the best tensile properties.

2.4 Failure behavior of different composites

The fracture morphologies of 7075Al, HEA_p/Al, CF/Al and

CF-HEA_p/Al composites are shown in Fig. 8. As shown in Fig. 8a, 7075Al has the typical ductile fracture characteristic of dimples. The addition of HEA_p changes the propagation of microcracks in composites. During loading, the microcracks initiate in the matrix firstly and then are propagated to the interface between reinforcement and matrix. The high density dislocations at interface improve the microcracks propagation resistance so that the microcracks propagation rate decreases^[6]. As the loading process continues, the microcracks propagation direction deflects at interface owing to the blocking effect of HEA_p with ultra-high strength, so that the microcracks are propagated around HEA_p. As shown in Fig. 8b, besides the dimples with small size generated by the failure of metal matrix, the pits with large size are generated by the delamination of HEA_p particles. For CF/Al composites, the interfacial bonding strength between CFs and matrix is ideal owing to the presence of the Ni-Co-P coatings^[25]. During the loading, individual CFs with low tensile strength fracture

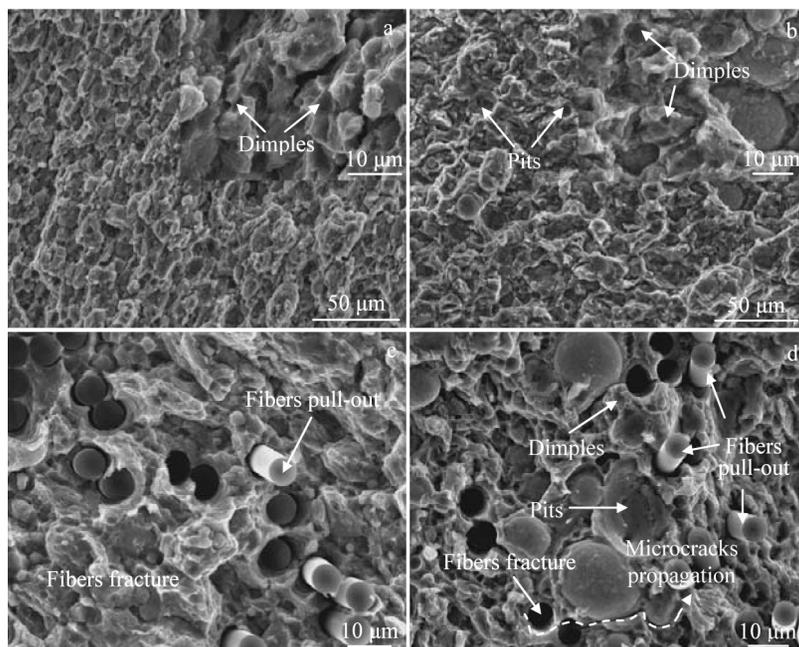


Fig.8 Fracture morphologies of 7075Al (a), HEA_p/Al composite (b), CF/Al composite (c), and CF-HEA_p/Al composite (d)

so that the loading of adjacent fibers increases sharply. When the stress carried by CFs reaches fiber's ultimate strength, pull-out of fibers occurs, which not only restrains the microcracks propagation through bridging to prevent the fracture of adjacent fibers but also prolongs the crack propagating path^[26]. The gradual failure of CFs ultimately manifests as the fracture of composites. As shown in Fig. 8c, fibers pull-out and fracture occurs simultaneously on the CF/Al composites besides the dimples on matrix. With the hybrid addition of CFs and HEA_p, strengthening of matrix caused by HEA_p restrains the propagation rate and changes the propagation path of microcracks in matrix, while bridging of fibers decreases stress concentration of adjacent fibers and deflects microcracks propagation direction, and the propagation path is marked around HEA_p and CFs. The propagation of microcracks in CF-HEA_p/Al composites consumes more energy than in HEA_p/Al and CF/Al composites because of the synergistic effect of HEA_p and CFs. As shown in Fig. 8d, the CF-HEA_p/Al composites exhibit all fracture characteristics such as dimples generated by failure of matrix, the pits generated by the delamination of HEA_p, fiber's pull-out and fracture.

3 Conclusions

1) FeCoCrNiAl high-entropy alloy particles (HEA_p) and Ni-Co-P coated carbon fibers (CFs) hybrid reinforced 7075Al matrix composites (CF-HEA_p/Al) with high relative density and dense interface are fabricated by vacuum hot pressing sintering at 580 °C, 30 MPa for 10 min.

2) The yield strength and ultimate tensile strength of CF-HEA_p/Al composites increase first and then decrease with the increase in CF content from 0vol% to 40vol%, and have higher values of 427 and 705 MPa, respectively, compared with those of 7075Al, HEA_p/Al and CF/Al composites, while the elongation is almost not reduced compared to that of CF/Al composites.

3) Owing to the hybrid addition of HEA_p and CFs, the fractures of CF-HEA_p/Al composites exhibit complex characteristics including fiber pull-out and fracture, dimples with small size and pits with large size.

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碳纤维-FeCoCrNiAl高熵合金颗粒混杂增强7075铝基复合材料的力学性能及断裂行为

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摘要: 为探究双相增强体对铝基复合材料拉伸性能和断裂行为的影响, 采用真空热压烧结工艺在580 °C, 30 MPa条件下保温10 min制备了FeCoCrNiAl高熵合金颗粒增强7075铝基复合材料(HEA_p/Al), Ni-Co-P镀层修饰碳纤维增强7075铝基复合材料(CF/Al)和FeCoCrNiAl高熵合金颗粒及Ni-Co-P镀层修饰碳纤维混杂增强铝基复合材料(CF-HEA_p/Al)。并对不同复合材料微观结构及拉伸性能进行分析表征及比较。结果表明: CF-HEA_p/Al复合材料的屈服强度(YS)与极限拉伸强度(UTS)随纤维含量的升高(体积分数由0至40%)呈现先增大后降低的变化, 延伸率则逐渐降低。鉴于Ni-Co-P镀层修饰碳纤维与FeCoNiCrAl高熵合金颗粒的混杂强化效应, CF-HEA_p/Al复合材料的YS和UTS较HEA_p/Al与CF/Al复合材料明显提高, 且其断口表现出基体韧性断裂及纤维拔出与断裂的多种失效特征。

关键词: 碳纤维; FeCoCrNiAl高熵合金颗粒; 混杂增强效应; 力学性能; 断裂行为

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