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ARTICLE

Friction Stir Channel Pressing of Carbon Nanotubes Reinforced 7075 Aluminum Alloy Composites

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Abstract: Friction stir channel pressing (FSCP) is a new solid-state method for producing metal matrix composite, which was invented by the authors based on the principles of friction stir welding and equal channel angular pressing. The carbon nanotubes (CNTs) reinforced 7075 aluminum alloy composites (CNTs/Al-7075) were fabricated by FSCP, with different volume percentages of CNTs (0%, 2% and 4%). The distribution of CNTs in Al-7075 matrix and the microstructures including fine grains and second-phase particles were analyzed by optical microscope, scanning electron microscope and transmission electron microscope. The solution and aging treatments were used for improving the microstructures and the mechanical properties of the CNTs/Al-7075 composites. The results show that the CNTs/Al-7075 composite with a uniform distribution of CNTs is fabricated by FSCP. The grain refinement of Al-7075 is realized by FSCP, and further finer grains are obtained by introduction of CNTs. The grains of CNTs/Al-7075 composite become finer with increasing the volume percentage of CNTs. The precipitation behavior of second-phase particles of the Al-7075 produced by FSCP and the CNTs/Al-7075 composite is improved by the solution and aging treatments, resulting in an increase in micro-hardness. The strengthening mechanisms of the CNTs/Al-7075 composite include fine-grain strengthening, dislocation strengthening, load transfer mechanism and second-phase strengthening, among which the second-phase strengthening plays a leading role.

Key words: friction stir channel pressing; CNTs/Al-7075 composite; microstructure; microhardness; strengthening mechanism

Carbon nanotubes (CNTs) have received more and more attention due to their unique structure and properties, which are considered as the reinforcement for fabricating the light-weight and high-strength composites[1]. Aluminium and its alloys are preferred choices for such fabrication due to their low density, high specific strength and modulus[2]. The 7075 aluminum alloy (Al-7075), as a kind of commonly used aluminum alloy, plays a significant role in automobile, aerospace and marine applications[3–4]. The fabrication of CNTs reinforced Al-7075 (CNTs/Al-7075) composites provides a new vision for further and wide use of Al-7075.

Powder metallurgy[5] and stirring-melt casting[6–7] have been used for preparing the CNTs reinforced aluminium alloy composite. Friction stir processing (FSP) is invented to improve the strength and ductility of material by making the grains finer and uniformly dispersed[8]. The CNTs reinforced aluminium alloys with different contents of CNTs have been also fabricated by FSP[9–11]. Compared to the above preparation technologies, the advantages of FSP for preparing CNTs reinforced aluminium alloy composite are as follows: (1) vacuum or protective gas is not required; (2) there is no high-temperature interface reaction due to the solid-state processing; (3) there is no problem of metal powder contamination in the preparation. However, a few processing passes are needed to achieve a uniform distribution of CNTs in aluminum alloy matrix, and to increase the interface contact

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area between CNTs and aluminum alloy matrix for promoting reaction [12]. The friction stir processing pass should not be excessive as it may cause extreme melting in the aluminum alloy matrix, resulting in the large cavities in stir zone and coarse microstructure[4]. Besides, the composite is only fabricated in the stir zone, also called as affected zone of stirring pin, but not in the whole aluminum alloy matrix. To overcome the drawbacks of FSP to produce the CNTs reinforced aluminium alloy composite, a new solid-state preparation method is expected.

Friction stir channel pressing (FSCP) is invented by the authors, which is based on the principles of friction stir welding[13–15] and equal channel angular pressing[16]. Fig. 1 shows the schematic of FSCP. Two prepared samples were pressed by the press blocks from both sides of the channel. The prepared sample was an aluminum alloy matrix embedded with CNTs. The frictional heat was generated when the rotating friction tool and the samples were contacted. The samples become soft and were mixed by the stirring effect of rotating friction tool. The mixed materials were pressed out of the squeeze mold as the press blocks continue to press the samples from both sides of the channel, resulting in a cylindrical bar of CNTs reinforced aluminum alloy composite.

In this study, different volume percentages of CNTs (0%, 2% and 4%) were embedded into Al-7075 matrix as prepared samples. These prepared samples were used to fabricate the CNTs/Al-7075 composite by FSCP. Then, the solution and aging treatments were used for the CNTs/Al-7075 composite. The distribution of CNTs in Al-7075 matrix, the grain refinement, the second-phase particles, and the microhardness were analyzed for the CNTs/Al-7075 composite. The strengthening mechanism of the CNTs/Al-7075 composite was discussed.

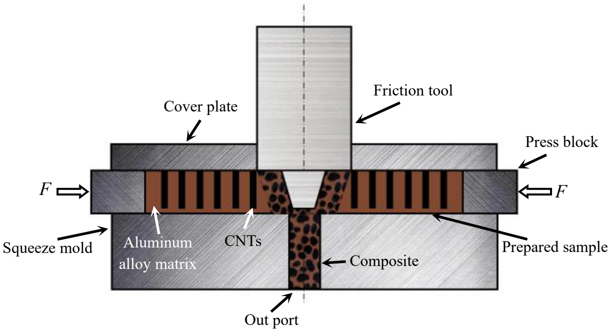

Fig.1 Schematic of friction stir channel pressing

1 Experiment

The Al-7075 (T6) commercial aluminum alloy with a desired size of 120 mm×12 mm×10 mm was prepared as the matrix. Table 1 shows the chemical composition of Al-7075. Some holes with 3 mm in diameter and 8 mm in depth were drilled on the surface of the Al-7075 matrix by a drilling machine, as shown in Fig. 2. The CNTs were supplied by a commercial supplier and no further purification was considered. The CNTs were filled in the holes of the Al-7075 matrix and compacted. Three volume percentages of CNTs were considered: 0vol%, 2vol% and 4vol%.

The friction stir channel pressing machine was self-designed, as shown in Fig. 3. The original samples prepared for fabricating the CNTs/Al-7075 composite were pressed by the press blocks from both sides of the squeeze mold under the cover plate. The pressing speed of the press block and the rotary speed of the friction tool were 0.28 mm/min and 315 r/min, respectively. The CNTs and the Al-7075 were mixed in the squeeze mold by the stirring effect of rotating friction tool. The mixed material of CNTs and Al-7075 was pressed out of the squeeze mold from the out port, resulting in a cylindrical bar of CNTs/Al-7075 composite.

Samples prepared with different processing parameters were defined in Table 2. The base material Al-7075 was defined as Sample B. Friction stir channel pressing of Al-7075 was defined as Sample F, which was then submitted to heat treatment, defined as Sample F-H. The solution treatment at 480 °C for 2 h and artificial aging at 120 °C for 24 h were

Table 1 Chemical composition of Al-7075 (wt%)

Zn	Mg	Cu	Ti	Fe	Si	Cr	Mn	Al
5.10–6.10	2.10–2.90	1.20–2.10	0.20	0.50	0.40	0.18–0.28	0.30	Bal.

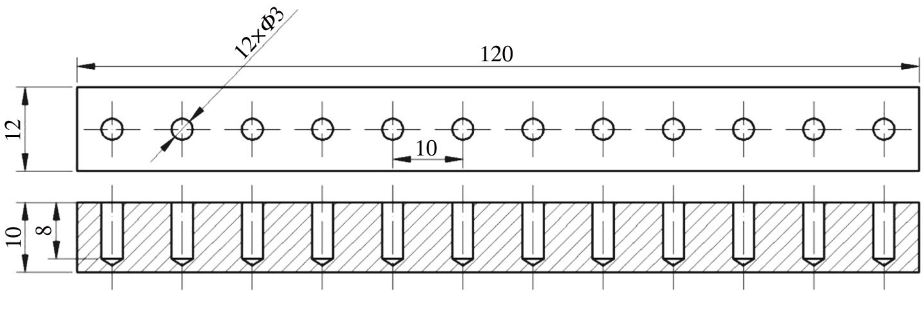

Fig.2 Original sample prepared for fabricating CNTs/Al-7075 composite

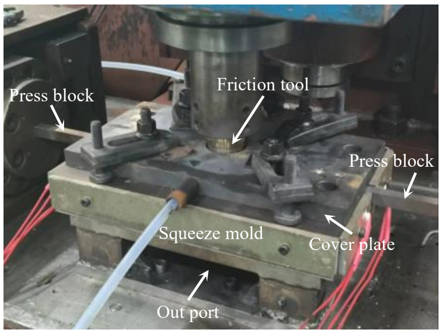

Fig.3 Self-designed friction stir channel pressing machine

considered. The 2vol% CNTs/Al-7075 composite and 4vol% CNTs/Al-7075 composite prepared by FSCP were defined as Sample F2C and Sample F4C, respectively. Sample F2C-H and Sample F4C-H indicate that the samples undergo the solution treatment and artificial aging.

The cross sectional microstructure of each sample was observed by optical microscope (OM) after etching with the Keller's reagent (1 mL HF+1.5 mL HCl+2.5 mL HNO_3+95 mL distilled water). The average grain size was measured by the linear pitch method according to GB/T 6394-2002. The FEI Quanta 200 Scanning electron microscope (SEM) was used to observe the second-phase morphology and energy spectral analysis was adopted. The JEM-2010 (HR) transmission electron microscope (TEM) was used to observe the microstructures. The TEM sample was cut along the cross section of the CNTs/Al-7075 composite with a precision cutting instrument. The plane spacing of second-phase particles was calculated by the software of Digital Micrograph. The HVS-100 microhardness tester was used to identify the microhardness of each sample.

2 Results and Discussion

2.1 Homogeneity of CNTs/Al-7075 composite

A cylindrical bar of 4vol% CNTs/Al-7075 composite is fabricated by FSCP. The length of the cylindrical bar can be up to 245 mm with 8.2 mm in diameter, and the surface is smooth except for few slight extrusion marks. There are no macroscopic defects in the cross-section macrograph of the 4vol% CNTs/Al-7075 composite, as shown in Fig.4a. Furthermore, the strong agglomeration interaction between CNTs cannot be observed. Fig.4b shows the SEM image of a local region in the cross section of 4vol% CNTs/Al-7075 composite, and no visible gaps or other defects can be found. The EDS maping for element C was carried out within an area of 2 mm×2 mm, as marked by the white frame in Fig.4b. It can be seen in Fig. 4c that the element C is distributed uniformly. The microhardness distribution is considered for verifying the homogeneity of 4vol% CNTs/Al-7075 composite. The testing area is a square of 5 mm×5 mm and the spacing of testing points is 0.5 mm, as shown in Fig.4a. It can be seen from Fig.4d that the microhardness value of the 4vol% CNTs/Al-7075 composite ranges from 127×9.8 MPa to 137×9.8 MPa, with an average value of 131×9.8 MPa. It can be inferred that the 4vol% CNTs/Al-7075 composite fabricated by FSCP is homogeneous, based on the analysis of cross-section macrograph, distribution of element C and microhardness distribution.

The uniform dispersion of CNTs in the aluminium alloy matrix is critical to the enhanced properties of composite, but due to the high content of CNTs, it is not easy to achieve uniform dispersion. According to Esawi et al[17], it is difficult to disperse the CNTs with a content greater than 2wt% by the powder metallurgy technology, and the improvement in tensile strength of the composite cannot be realized. Zhang et al[18] have used a higher energy input during FSP to obtain a better dispersion of 3.2vol% CNTs in Al matrix. Liu et al[12] have adopted five passes of FSP to achieve uniform dispersion of 6vol% CNTs in Al matrix. The dispersion of CNTs in FSCP is also based on the plastic material flow. The author will try to change the processing parameters to realize a higher content of CNTs for fabricating the composite in future study.

2.2 Grain refinement and microhardness

The microstructure of base material Al-7075 is shown in Fig. 5a, which presents irregular strip structure along the rolling direction. As shown in Fig.5b, homogeneous and fine equiaxed grains with an average grain size of 15 μm are obtained after FSCP, and several grains have a large size about 30 μm. Friction stir channel pressing is a technique based on the development of friction stir welding and equal channel angular pressing, which is attributed to the dynamic recrystallization of highly deformed grains due to the large plastic deformation during processing [19–20]. Fig.5c shows the microstructure of Al-7075 processed by FSCP after heat treatment. Its grains are equiaxed, while the grain size is smaller and more uniform, with an average size of about 8 μm. Due to the short processing time of FSCP and the rapid heat conduction of aluminum alloy, and the sharp decrease in temperature of material when it is extruded from the machine, some grains are not completely recrystallized, resulting in several large grains. These large grains have stored large deformation energy and the heat treatment provides a driving force for them to continue complete recrystallization, resulting in the further refinement of the grains after heat treatment.

Fig. 5d shows the microstructure of 2vol% CNTs/Al-7075 composite. With the introduction of carbon nanotubes, the

Table 2 Samples produced by different processing parameters

Sample	B	F	F-H	F2C	F2C-H	F4C	F4C-H
FSCP	No	Yes	Yes	Yes	Yes	Yes	Yes
Content of CNTs/vol%	0	0	0	2	2	4	4
Heat treatment	No	No	Yes	No	Yes	No	Yes

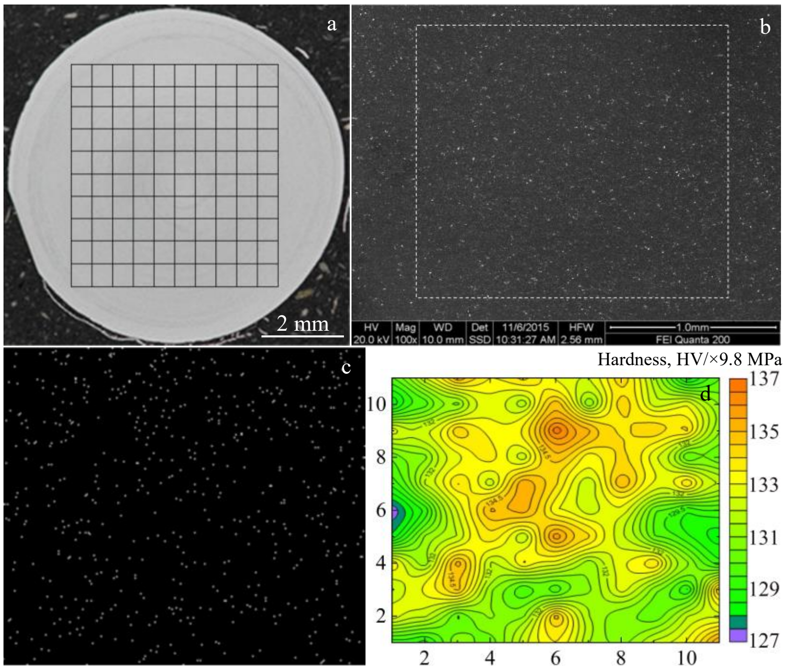

Fig.4 Homogeneity of 4vol% CNTs/Al-7075 composite: (a) cross-section macrograph, (b) SEM image of a local region in the cross section, (c) EDS mapping of element C inside the white frame marked in Fig.4b, and (d) microhardness distribution inside the black frame marked in Fig.4a

grains become finer than those in Fig.5b, with an average size of 8.5 μm. The grain size decreases to 3.0 μm with increasing the fraction of CNTs to 4vol%, as shown in Fig.5f. The CNTs may refine the grains of composite in two ways. Firstly, the CNTs become nucleation particles in the recrystallization process of composite during FSCP. When the grains begin to grow up, the CNTs as a special second-phase particle pin the grain boundary. The grain boundary is hard to migrate, which refines the grains of 4vol% CNTs/Al-7075 composite. Secondly, when the composite is subjected to deformation, the CNTs can hinder the movement of dislocations and eventually lead to the accumulation of dislocations. When the dislocations accumulate, sub-grain boundaries will be produced, which can finally form grain boundaries to further refine the grains. A further reduction in size is realized for the CNTs/Al-7075 composites after the heat treatment, as shown in Fig.5d and 5e.

2.3 Precipitation hardening behavior

Fig. 6a and 6b show TEM images of second-phase particles in grain and grain boundary of Sample B, respectively. It can be seen that rod-shaped particles and elliptical particles are distributed uniformly in the grain, and rod-shaped particles are distributed discontinuously in the grain boundary, with a large distance between adjacent particles. Fig. 6c and 6d show the enlarged images of the particles in grain and grain boundary. The diameter of rod-shaped particle is 20–30 nm and the length is 50–80 nm, while the diameter of elliptical particle is 40–80 nm. As shown in Fig.6a, the XRD patterns of Sample B indicate that α-Al and $MgZn_2$ are detected. The HRTEM images for the rod-shaped particle and elliptical particle are shown in Fig. 6e and 6f, respectively, in which two-dimensional lattice fringes are observed. The marked lattice of rod-shaped particle is 0.3951 nm and that of elliptical particle is 0.4475 nm, which are identical to the (101) and (100) facets of $MgZn_2$, respectively. Therefore, it can be inferred that these particles can be determined as $MgZn_2$ by combining the lattice measurements with the XRD results.

It can be seen from Fig. 6g and 6h that the second-phase particles in grains and grain boundaries of Al-7075 become larger after FSCP, and the quantity of second-phase particles is also decreased, compared with those in Fig. 6a and 6b. Therefore, the strengthening effect of second-phase particles in Al-7075 decreases after FSCP, resulting in a decrease in microhardness from 1587.6 MPa to 1107.4 MPa. The heat treatment T6 is considered to improve the microstructures of Al-7075 after FSCP. Fig. 6i and 6k show the microstructures after heat treatment and enlarged images for the second-phase particles are shown in Fig. 6j and 6l. It can be found that the second-phase particles become finer and are distributed more uniformly in the grains and grain boundaries than those in Fig. 6g and 6h. Therefore, the microhardness of Al-7075 produced by FSCP is increased obviously after heat treatment, from 1107.4 MPa to 1734.6 MPa.

Fig.7 shows the XRD patterns of Sample B, Sample F and Sample F-H. It can be found that $MgZn_2$ is the main precipitate in Al-7075. The characteristic peak of $MgZn_2$ cannot be detected in FSCP of Al-7075, which is caused by the dissolution of $MgZn_2$. There are more characteristic peaks of $MgZn_2$ in FSCP of Al-7075 after heat treatment than those in Al-7075.

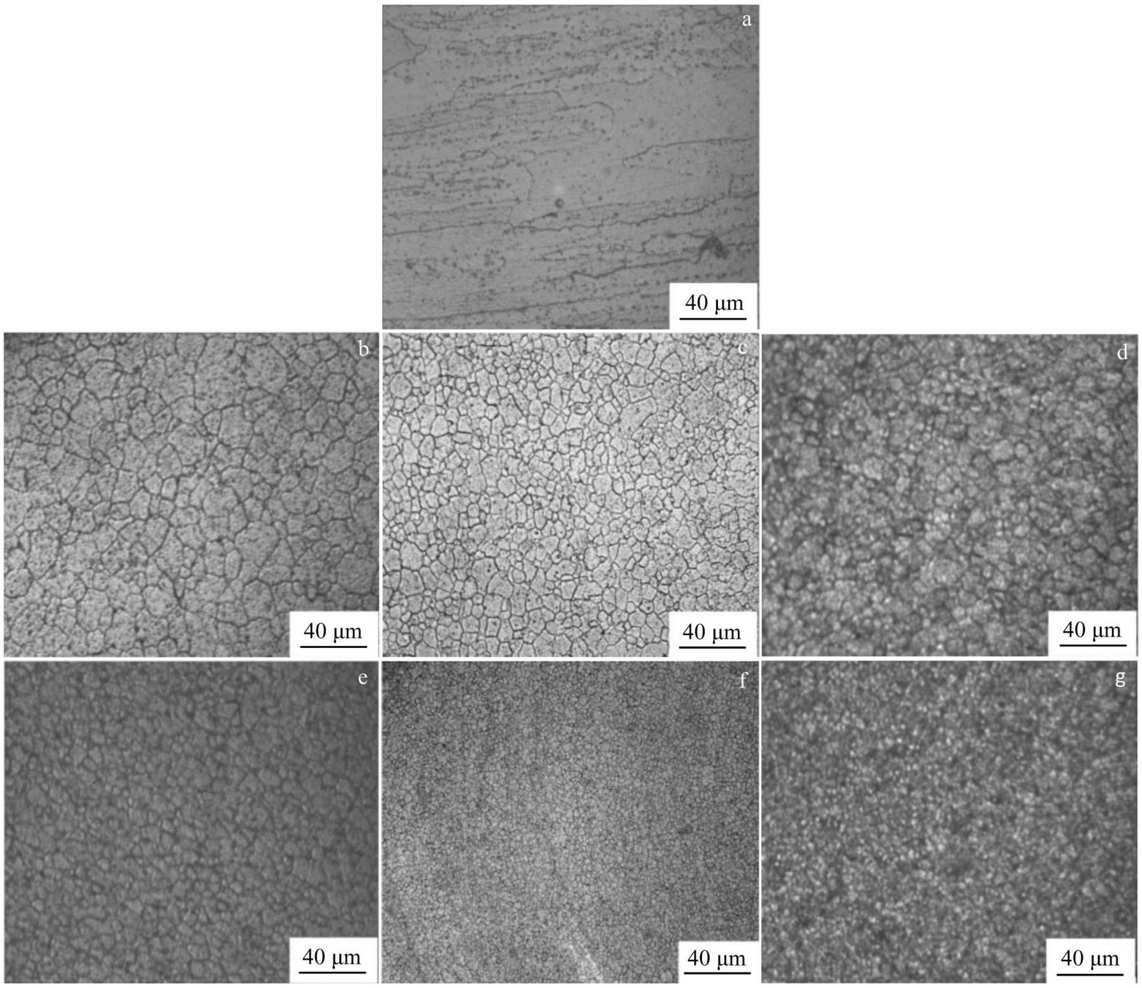

Fig.5 OM microstructures of Sample B (a), Sample F (b), Sample F-H (c), Sample F2C (d), Sample F2C-H(e), Sample F4C (f), and Sample F4C-H (g)

Fig. 8a shows TEM image of second-phase particles in grain of 4vol% CNTs/Al-7075 composite. There are a small number of rod-shaped particles with different sizes. Fig. 8b shows TEM image of second-phase particles in grain boundary of 4vol% CNTs/Al-7075 composite. The second-phase particles are coarsened. After heat treatment, the second-phase particles in grains or grain boundaries become finer and the quantity is increased, as shown in Fig.8c and 8d.

Fig. 9 shows the XRD patterns of 4vol% CNTs/Al-7075 composite and 4vol% CNTs/Al-7075 composite after heat treatment. The characteristic peak of $MgZn_2$ cannot be detected in 4vol% CNTs/Al-7075 composite, while it can be detected in 4vol% CNTs/Al-7075 composite after heat treatment. The characteristic peak of Al_4C_3 can be detected for two samples.

Friction stir channel pressing of Al-7075 is accompanied by the effect of heat, so the second-phase particles in grain and at grain boundary become coarse, and the quantity of second-phase particles is decreased. The solution and aging treatments can improve the microstructure, by which the second-phase particles become finer and are distributed more uniformly in the grains and at grain boundaries. The addition of CNTs leads to a decrease in the distribution uniformity of second-phase particles in the CNTs/Al-7075 composite, and second-phase particles become coarse. The solution and aging treatments can also make the second-phase particles become finer and more uniformly distributed in the grains and grain boundaries for the CNTs/Al-7075 composite. Therefore, the precipitation behavior of second-phase particles of the Al-7075 produced by FSCP and the CNTs/Al-7075 composite can be improved by the solution and aging treatments.

2.4 Interfacial bonding characteristics

The TEM is used to investigate the interfacial structure between Al-7075 matrix and CNTs. The interfacial bonding characteristics between Al-7075 matrix and CNTs are shown in Fig. 10. The CNTs retain the typical structure and the bonded interfaces between Al-7075 matrix and CNTs are well, where no visible gaps or other defects can be found. A strong interface between CNTs and aluminium matrix can transfer the load effectively, which is beneficial to the mechanical property of composite. According to Chen et al[21], the formation of Al_4C_3 between CNTs and aluminium

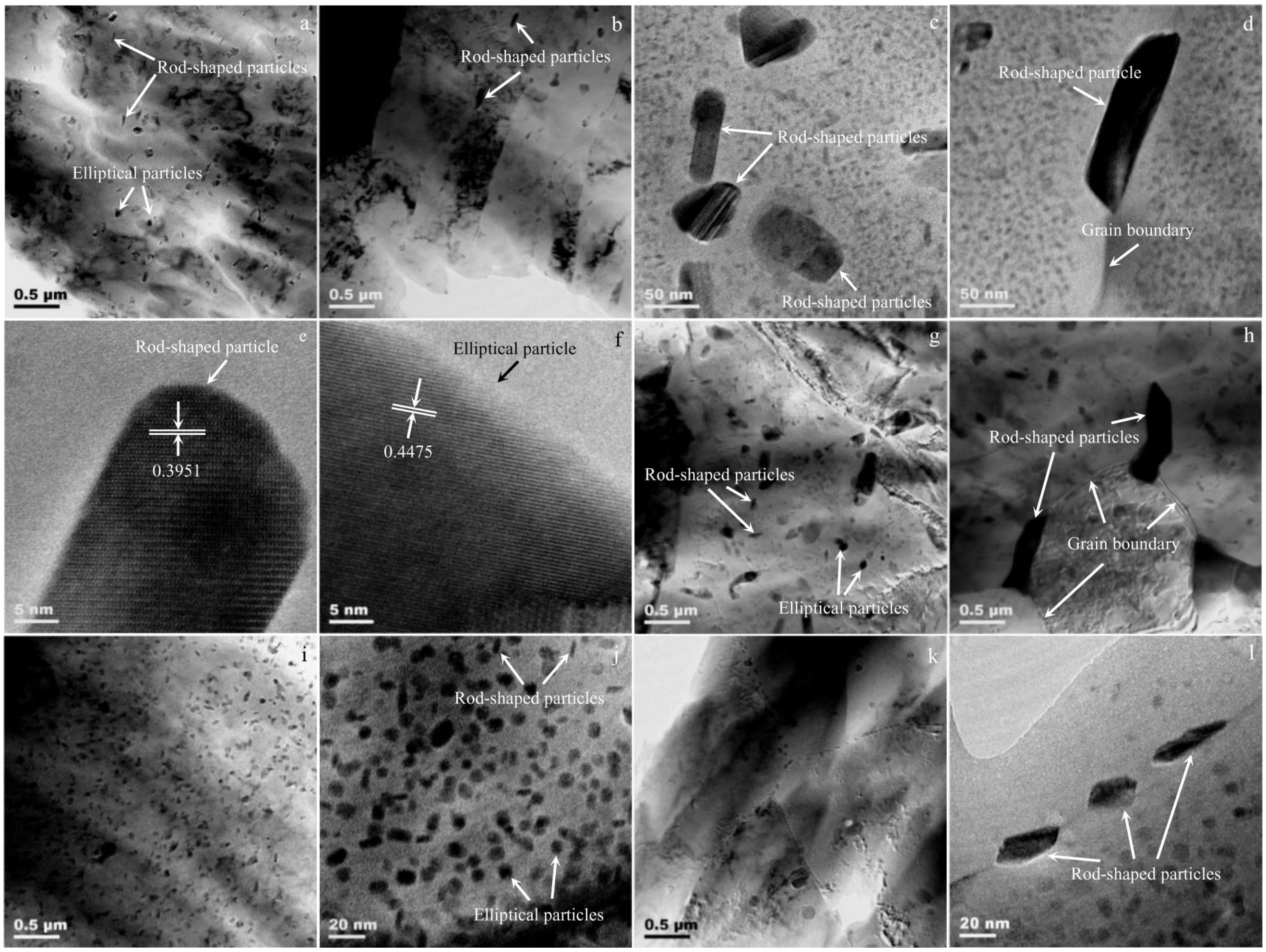

Fig.6 TEM images of second-phase particles in Sample B (a–d), Sample F (g, h) and Sample F-H (i–l); HRTEM images of second-phase particles in Sample B (e, f)

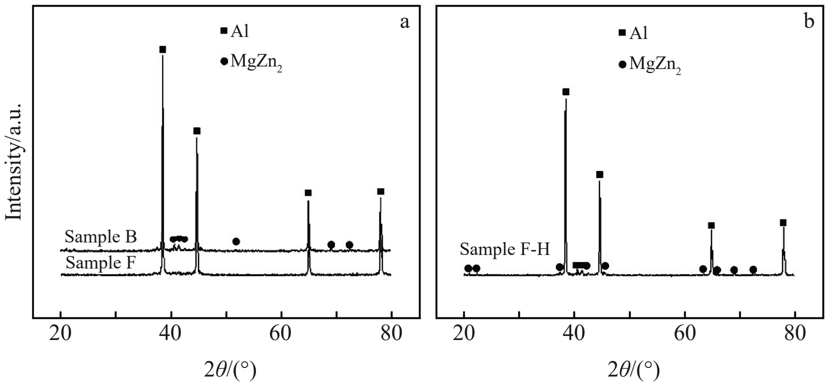

Fig.7 XRD patterns of Sample B and Sample F (a), as well as Sample F-H (b)

alloy matrix can be regulated by changing the sintering temperature, which is beneficial to the interfacial strength and load transfer efficiency. Zhou et al[22] found that the stress contrast around Al_4C_3 leads to the enhancement of the anchor effect from the Al matrix, which may hinder the local interfacial slippage and constrain the deformation of the Al matrix.

2.5 Strengthening mechanism in CNTs/Al-7075 composite

According to the Hall-Petch relation, grain refinement is beneficial to the yield strength, which is related to the microhardness. It can be found from Table 3 that the microhardness is increased with decreasing the grain size for the samples without heat treatment (Sample F, Sample F2C and Sample

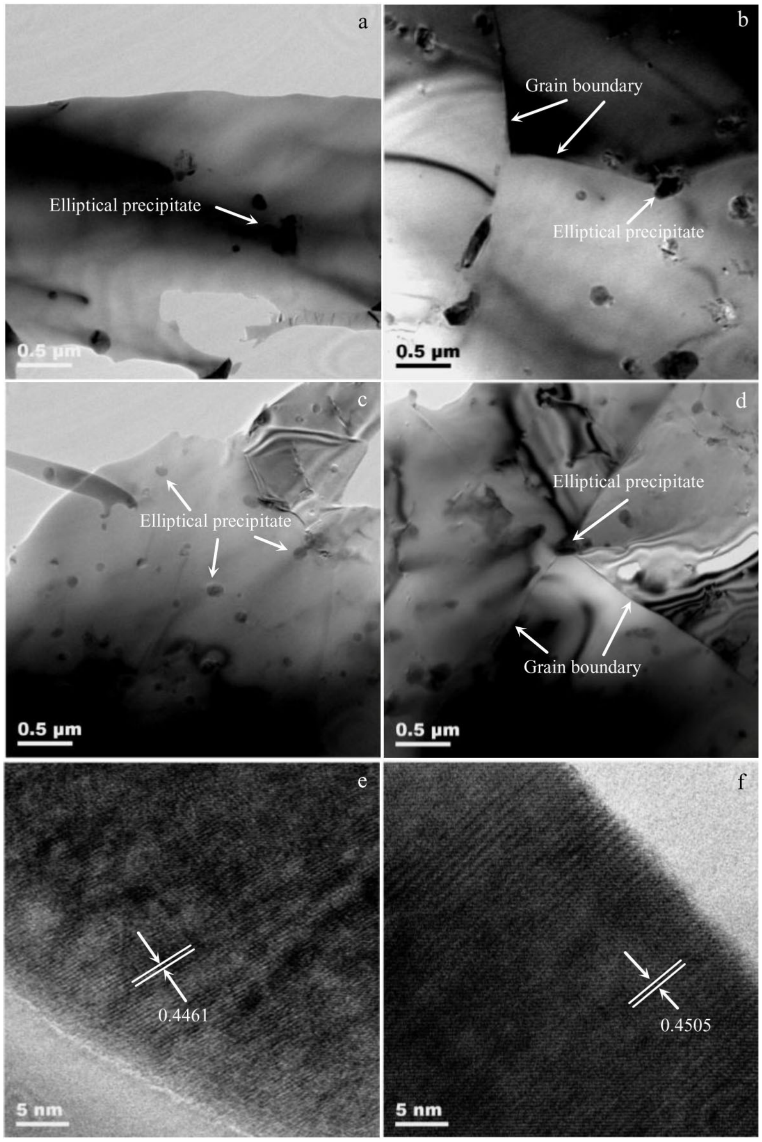

Fig.8 TEM images of second-phase particles in Sample F4C (a, b) and Sample F4C-H (c, d); HRTEM images of second-phase particles in Sample F4C (e, f)

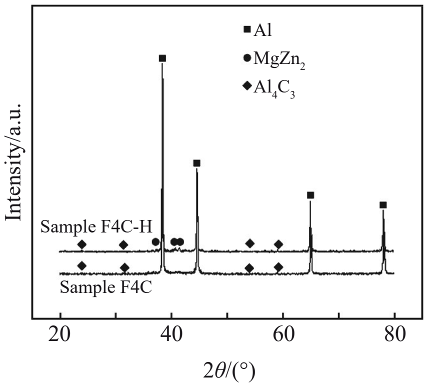

Fig.9 XRD patterns of Sample F4C and Sample F4C-H

F4C), and the same trend is shown for the samples with heat treatment (Sample F-H, Sample F2C-H and Sample F4C-H).

Table 3 Grain size and microhardness of different samples

Sample	B	F	F-H	F2C	F2C-H	F4C	F4C-H
Grain size/μm	-	15.0	8.0	8.5	5.5	3.0	1.5
Microhardness, HV/×9.8 MPa	162	113	177	124	183	135	190

However, comparing sample F and sample B, it can be found that equiaxed and uniform grains are obtained in sample F, but the microhardness of sample F is lower than that of sample B with irregular strip structure. The grains of Al-7075 are refined after FSCP, which is beneficial to the improvement of microhardness. However, the Al-7075 is a typical aluminum alloy reinforced by the second-phase particles, and its strengthening effect mainly comes from the dispersed second-

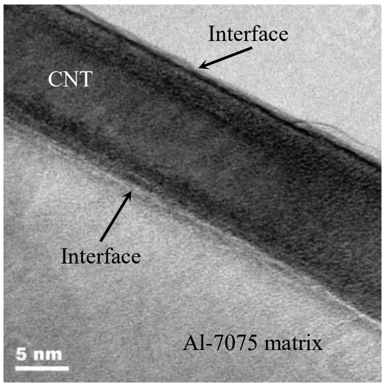

Fig.10 Representative TEM image of interface in CNTs/Al-7075 composite

phase particles. Furthermore, comparing sample F4C and sample F-H, it can be found that the microhardness is not increased with decreasing the grain size and introduction of CNTs. According to Nam[23], compared to the Al matrix, the increase in yield strength of CNTs/Al composite originates from a synergistic strengthening of the addition effect of CNTs and the grain refinement of composite, in which the addition effect of CNTs on the increase in yield strength is much higher than the effect of grain refinement. Here, it can be inferred that the effect of CNTs addition and the grain refinement of CNTs/Al-7075 composite on the increase in microhardness is much weaker than the effect of second-phase particles.

3 Conclusions

1) It is possible to fabricate a carbon nanotubes reinforced Al-7075 composite, with a uniform distribution of the carbon nanotubes by friction stir channel pressing.

2) The grain refinement of Al-7075 can be realized by friction stir channel pressing, and further finer grains can be obtained by the introduction of carbon nanotubes. The precipitation behavior of second-phase particles in the Al-7075 produced by friction stir channel pressing and the CNTs/Al-7075 composite can be improved by the solution and aging treatments.

3) The microhardness of the Al-7075 after friction stir channel pressing or the CNTs/Al-7075 composite is lower than that of the received Al-7075. The solution and aging treatments are effective to improve the microhardness.

4) The strengthening mechanism of CNTs/Al-7075 composite includes fine grain strengthening, dislocation strengthening, load transfer mechanism and second-phase particles strengthening, in which second-phase particles strengthening plays a leading role.

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搅拌摩擦通道挤压制备碳纳米管增强7075铝基复合材料

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摘　要：搅拌摩擦通道挤压是作者基于搅拌摩擦焊接和等通道转角挤压提出的一种固相状态制备金属基复合材料的新方法。采用搅拌摩擦通道挤压方法，通过添加不同体积分数的碳纳米管（CNTs）（0%、2%和4%），制备了碳纳米管增强7075铝合金基复合材料（CNTs/Al-7075）。通过光学显微镜、扫描电子显微镜和透射电子显微镜观察并分析了CNTs在Al-7075基体中的分布特征，以及复合材料的细晶组织和第二相颗粒特征。采用固溶和时效处理改善CNTs/Al-7075复合材料的组织和力学性能。结果表明，采用搅拌摩擦通道挤压方法可以制备CNTs分布均匀的CNTs/Al-7075复合材料，实现7075铝合金基体晶粒细化，通过引入CNTs增强相可获得更为细小的晶粒组织。随着CNTs体积分数增加，CNTs/Al-7075复合材料的晶粒更加细化。固溶和时效处理改善了搅拌摩擦通道挤压制备的7075铝合金和CNTs/Al-7075复合材料的第二相析出行为，使材料的显微硬度得到提高。CNTs/Al-7075复合材料的强化机制综合了细晶强化、位错强化、载荷传递和第二相强化，其中以第二相强化为主。

关键词：搅拌摩擦通道挤压；CNTs/Al-7075复合材料；显微组织；显微硬度；强化机制

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