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ARTICLE

Microstructures and Mechanical Properties of Tungsten Particle Reinforced 6061Al Composites Fabricated by SPS-Hot Rolling

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Abstract: Tungsten particle (W_p) reinforced 6061Al matrix composites with different volume fractions of W_p (1 vol～7 vol%) were fabricated by spark plasma sintering (SPS) followed by hot-rolling. The effects of W_p content on the microstructure, mechanical properties and electrical resistivity of the as-rolled composites were investigated. The results show that the W_p is homogeneously distributed in the metal matrix, achieving excellent metallurgical bonding. At the interface of W_p/6061Al, element solid solution and WAl_{12} intermetallics form during the fabrication process. The mechanical test results show that with the increase of W_p, relative density and ductility of the as-rolled composites decrease while the tensile strength first increases and then decreases. It is noteworthy that both high tensile strength and ductility are obtained by the composites with 1 vol% and 3 vol% W_p, being 192.85 MPa, 16.84% and 315.18 MPa, 11.93%, respectively. Besides, the fabricated W_p reinforced 6061Al matrix composites have excellent electrical conductivity and the increase of W_p content has little influence on the electrical conductivity.

Key words: spark plasma sintering; W_p/6061Al composites; microstructure; mechanical properties; electrical resistivity

Particulate reinforced aluminum matrix composites (PAMC) have wide application in aerospace, automotive, electronic and electric, manufacturing, and nuclear power industry due to their high specific strength and stiffness, low density, good thermal stability and wear resistance[1-5]. Traditionally, ceramic particles like B_4C[3], Al_2O_3[6], TiB_2[7] and TiC[8] are used for reinforcement since they have high strength, elastic modulus and wear resistance. However, the poor wettability of particles and matrix, brittle interfacial reaction products and the presence of porosity of these PAMC lead to the low ductility [9-11]. Therefore, the ceramic particles reinforced aluminum matrix composites cannot achieve high strength and ductility simultaneously. Besides, the poor electrical conductivity of ceramic particles also has a detrimental effect on the composites which can restrict the application of PAMC especially when used as aerospace materials [12].

In order to solve these problems, an alternative way is to use hard metal phases as reinforcements[13-14]. Compared with the ceramic reinforcements, the wettability of metal particles and Al matrix is much better and the closer coefficient of thermal expansion may offer a good combination of strength and ductility[13-15]. In addition, the metallic particles have better electrical conductivity than ceramic particles which can widen the range of application of PAMC. Tungsten is an attractive reinforcement due to its high

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melting point, strength, stiffness, hardness and good chemical stability, especially the better electrical conductivity than ceramic particles. However, the traditional fabrication methods such as stir casting and hot pressing sintering are not suitable for processing metal particles reinforced aluminum matrix composites, because the high processing temperature of these methods will cause the formation of brittle intermetallics, which can promote premature fracture[12]. Recently, researchers have used FSP to produce metal matrix composites due to its low processing temperature[16,17]. However, friction stir processing (FSP) is mainly used as secondary processing technique of composites prepared by casting, spray deposition and powder metallurgy, and is not up to the level of industrial application.

Spark plasma sintering (SPS) is a relatively new sintering method widely used to synthesize composites, nanocrystalline materials, and amorphous alloys. Compared with traditional sintering methods, SPS has many advantages like high heating rate, shorter sintering time and lower sintering temperature [18,19]. Besides, during the SPS process, spark plasma generated by direct pulse current can break the oxide layer on the particle surface and thus facilitate neck formation and rapid densification[3,20]. Especially when fabricating metallic materials, spark plasma can be formed more easily due to the good electrical conductivity [18]. Given the above mentioned advantages, using SPS to fabricate metal particles reinforced aluminum matrix composites can avoid the formation of intermetallics and acquire fully dense material.

In the present work, tungsten particle reinforced 6061Al matrix composite were processed by SPS and hot rolling. The aim of this work is to obtain the composites with high ductility when strength is improved. The microstructure and mechanical properties of the rolled composites were studied and the relationship between the microstructure and mechanical properties was discussed. Besides, the fracture mechanism and the electrical behavior of the composites were also analyzed.

1 Experiment

Powders of W_p and 6061Al alloy (Beijing Xingrongyuan Technology, Co., Ltd., China) were used as initial materials with a purity of 99.9%. The average particle size of W_p and 6061Al powders was 1.5 and 13 μm, respectively. And the SEM images of initial powders are given in Fig.1.

The composites containing 1 vol%, 3 vol%, 5 vol% and 7 vol% W_p (marked as 1WAl, 3WAl, 5WAl and 7WAl) were fabricated by SPS and hot rolling. Firstly, the powders were mixed evenly by ball-milling under argon atmosphere at a speed of 300 r/min for 5 h with a mass ratio of ball to power at 10:1. Secondly, the mixed powders were loaded into a graphite die of 30 mm in diameter with graphite foils to segregate powder and punches. Then, SPS (DR. SINTER SPS331-Lx, Japan) was used for sintering process. The samples were sintered at 580 °C under an uniaxial pressure of 40 MPa for 10 min in vacuum. The size of sintered samples was approximately 30 mm in diameter and 6 mm in thicknesses. Finally, the sintered samples were rolled from 6 mm to 2 mm after 4 passes at 500 °C. In addition, the pure 6061Al alloy sample was prepared by the same method for comparison.

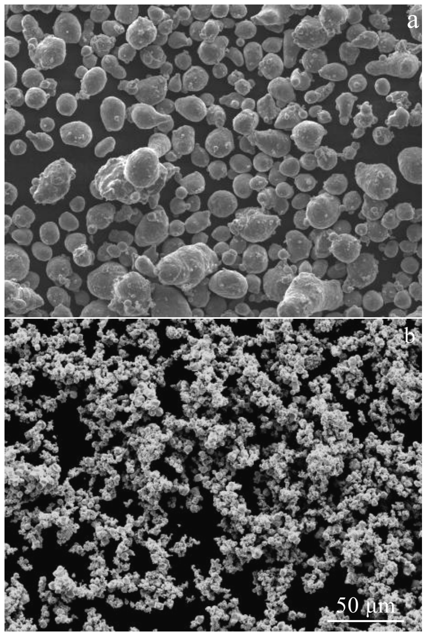

Fig.1 SEM images of 6061Al alloy (a) and W_p (b)

Archimedes principle was used to measure the relative densities of the as-rolled 6061Al and W/6061Al composites. The microstructure of all samples was evaluated by scanning electron microscopy (SEM, JSM-6700F) equipped with an energy-dispersive spectrometer (EDS). The phase analysis was performed in an X-ray diffractometer (XRD) using Cu Kα radiation. The tensile test was conducted at room temperature at a rate of 0.2 mm/min. The tensile samples were cut along the tensile axis parallel to the direction of rolling process. For each sample, three specimens were tested. Fracture surfaces after the tensile test were examined by SEM. Microhardness measurements were performed by a ZHVST-1000C tester under 100 g load for 15 s. Samples with a size of 10 mm×3 mm×3 mm from the as-rolled composites with different W contents and as-rolled 6061Al alloy were used for testing electrical resistivity by Namicro-3L (Joule Yacht).

2 Results and Discussion

2.1 Relative density and hardness

Table 1 shows the density of as-rolled 6061Al and as-rolled composites. The relative density was obtained by

Table 1 Density of as-rolled 6061Al alloy and W_p/6061Al composites

W_p content/vol%	Measured density/g·cm^{-3}	Theoretical density/g·cm^{-3}	Relative density/%
0 (6061Al)	2.696	2.700	99.85
1	2.834	2.865	98.92
3	3.154	3.194	98.75
5	3.473	3.528	98.44
7	3.790	3.857	98.26

the comparison of measured density and theoretical density, which also shows the porosity of the materials. All samples reach a comparatively high relative density after SPS and rolling, especially 6061Al (99.85%, almost fully dense). The relative density of W_p/6061Al composites decreases from 98.92% to 98.26% with the increase of W_p content (1 vol%~7 vol%), but all higher than 98%.

The high relative density of all composites can be explained as follows: (1) the special sintering mechanism of SPS leads to the higher densification of materials compared with traditional sintering methods[3]; (2) the rolling process can further improve the densification of the as-SPSed materials due to the large plastic deformation. The decrease of the relative density of composites is because little plastic deformation of W_p (the melting point: 3410 °C) occurs due to the low processing temperature (580 °C during SPS). Consequently, W_p is loosely distributed in the 6061Al matrix and with the increase of W_p content, the relative density of composites decreases.

Fig.2 shows the results of hardness tests of the as-rolled 6061Al and as-rolled composites. As can be seen that the hardness of all as-rolled composites is higher than that of the as-rolled 6061Al matrix (587 MPa). And with the increase of W_p content, the hardness of composites increases from 670 MPa (1WAl) to 870 MPa (7WAl). The high hardness value of W, homogeneous distribution of W_p in the matrix, and good interfacial bonding between W_p and matrix all result in the increase of microhardness. Besides, hard W particles imped the motion of the soft 6061Al matrix during hot rolling process, thus increasing the dislocation density in the Al matrix, and finally resulting in the hardness improvement [21].

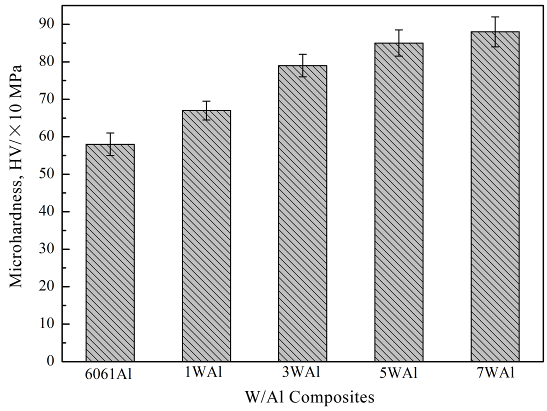

Fig.2 Hardness value of as-rolled 6061Al alloy and composites with different W_p contents

2.2 Microstructures and phases

Fig.3a shows the XRD patterns of as-rolled 6061Al alloy and its composites. According to the Al-W binary phase diagram shown in Fig.3b, tungsten has very limited solid solubility in aluminum so it is easy to form intermetallics like WAl_{12}, WAl_5, and WAl_4. As can be seen from Fig.3a, diffraction peaks of WAl_{12} intermetallics are observed in the composites of 3WAl, 5WAl and 7WAl, while in the composites of 1WAl, only W and Al diffraction peaks are observed obviously. The absence of WAl_{12} in 1WAl composites may be caused by the relatively low content of W_p, thus leading to little formation of intermetallics. Besides, the high heating rate, shorter sintering time and low sintering temperature of SPS process also restrict the formation of intermetallics [22].

Fig.4 shows the SEM images of as-rolled composites with different W_p content. It can be seen that the distribution of W_p is relatively homogeneous in 6061Al matrix of all composites. During rolling process, the flow of matrix is beneficial for the dispersion of W_p. However, the increase of W_p content causes particle aggregation in some areas as shown in Fig.4c and 4d with red arrows. The large difference of density between W_p and 6061Al alloy may cause difficulties for homogeneous distribution during ball-milling process. According to Fan's work [23], the direct contact of W_p is unfavorable to the mechanical property of material due to the absence of metallurgical bonding at W-W interface.

Fig.5 displays the SEM images of W/Al interface at higher magnification and corresponding EDS line scanning and EDS mapping. From the magnified image in Fig.5a, a good bonding at W/Al interface is achieved without any defects like micro pores and cracks. The line scanning results (Fig.5d, red line in Fig.5a) reveal that there is a certain diffusion layer of W and Al elements at W/Al interface. This demonstrates that the interface of W/Al contains element solid solution and possible Al-W intermetallics. Therefore, a favorable metallurgical bonding is achieved at the interface which improves the strength of the composites, since a better combination of reinforcement and matrix can help to transfer load from the 6061Al matrix to the hard W particles, thus increasing the yield strength of material[3]. The EDS mapping results (Fig.5b and 5c) show that at the gray-white area, the amount of Al element is greater than that of W element, which may cause the formation of intermetallics.

2.3 Mechanical properties

Fig.6 shows the tensile stress-strain curves of as-rolled

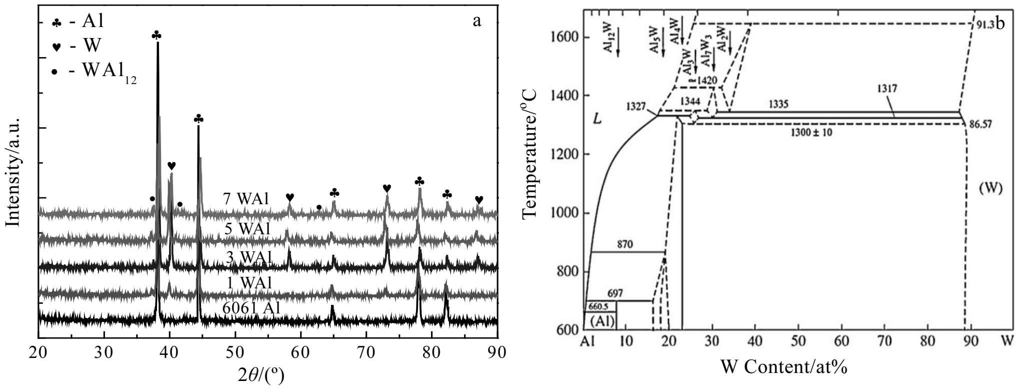

Fig.3 XRD pattern of as-rolled 6061Al alloy and its composites (a) and Al-W binary phase diagram (b)

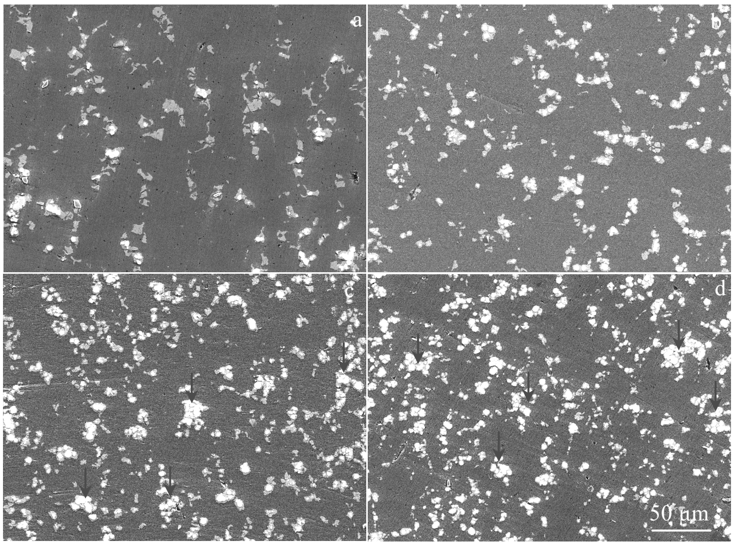

Fig.4 SEM micrographs of as-rolled composites with different W_p contents: (a) 1 vol%, (b) 3 vol%, (c) 5 vol%, and (d) 7 vol%

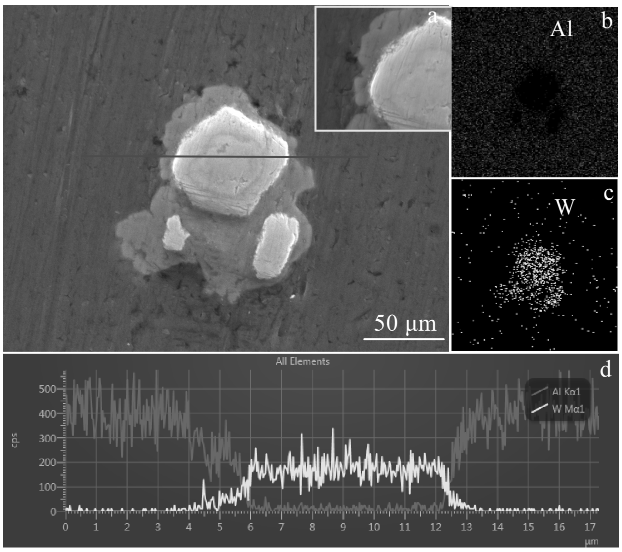

Fig.5 SEM image of the W/Al interface of the as-rolled composites (a); EDS mapping of Al (b) and W_p (c); main element profiles by EDS line scanning in detection shown in Fig.5a (d)

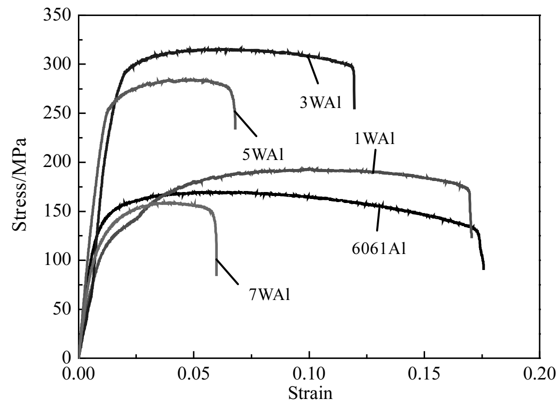

Fig.6 Tensile stress-strain curves of as-rolled 6061Al alloy and its composites

6061Al alloy and its composites and the mechanical properties are represented in Table 2. It can be seen that the yield strength (σ_{YS}: 0.2% proof stress) and the ultimate tensile strength (σ_{UTS}) of composites firstly increases, reaching 289.25 and 315.18 MPa (3WAl), and then decreases to 124.76 and 158.60 MPa (7WAl), respectively with the increase of W content. The UTS of composites with W content from 1 vol% to 5 vol% is all much higher than that of 6061Al alloy.

The higher strength of the as-rolled composites can be explained as follows. (1) Effect of grain refinement: the fabrication method SPS can achieve fast sintering with less holding time, which effectively confines the grain growth [3]; the subsequent hot-rolling process also further refines the grain size; based on the Hall-Patch equation [24], the decrease of grain size is favorable to the increase of strength. (2) The presence of W particles: (a) the large difference in the coefficient of thermal expansion between Al matrix (23.86×10^{-6} K^{-1}) and W_p (4.5×10^{-6} K^{-1}) leads to higher dislocation density around the W_p, and thus impedes the motion of dislocation; (b) the favorable interfacial bonding between W_p and Al matrix (shown in Fig.5a) is beneficial to the transfer of load from matrix to the reinforcements; (c) during the process of hot-rolling, the existence of W_p can restrict the movement of grain boundaries to a great extent and then lead to further grain refinement.

The elongation to fracture of composites shows a downtrend and is all lower than that of 6061Al alloy. However, it should be noted that a relatively high ductility of 1WAl and 3WAl is retained with less decrease compared to 6061Al alloy (17.16%), being 16.84 and 11.93%, respectively. Especially 3WAl, compared with 6061Al alloy, its UTS increases by 86.16% while its ductility decreases only by 30.88%. This is quite different from the conventional ceramic reinforced Al matrix composites.

For example, Chen et al[10] fabricated 30 vol% B_4C/6061Al composites with a UTS greater than 250 MPa while a elongation only less than 4%; Jiang et al[25] fabricated 7075Al matrix composites reinforced with nano-sized SiC particles by ultrasonic-assisted semisolid stirring, and the UTS of the composites can reach 492 MPa while the elongation is only 9.6%. The combination of high strength and good ductility of 1WAl and 3WAl fabricated in this work is mainly due to the better interfacial bonding compared to ceramic particles. Meanwhile, the advantages of SPS process can restrict the formation of brittle intermetallics to a great extent[22]. Thus, excellent interfacial properties increase the strength and ductility of the composites at the same time. The decrease of strength and ductility of 5WAl and 7WAl compared with 1WAl and 3WAl is mainly caused by the aggregation of W particles where higher W-W contiguity exits. Microcracks easily form and extend in these areas during tensile process, causing lower strength and ductility of the composites.

Fig.7 shows the SEM tensile fractographs of as-rolled 6061Al alloy and its composites with different W_p contents. The dimples of the as-rolled 6061Al alloy (Fig.7i) are very deep, which show a typical ductile fracture. The fracture surfaces of as-rolled W_p/6061Al composites with different W_p contents show similar characteristics including ductile and brittle fractures. Compared with composites containing 1 vol% and 3 vol% W_p (Fig.7a and 7b), the fractographs of composites with 5 vol% and 7 vol% W_p (Fig.7d~7g) show more aggregation of W particles, consistent with the distribution of W_p shown in Fig.4. The flatter fracture surfaces of the areas with W_p aggregation can explain the lower ductility of the composites with higher W_p content. The aggregation of hard W_p can cause severe stress concentration, together with the poor combination between W_p; microvoids and cracks more easily form in these areas, thus causing the decrease of strength and ductility (Fig.7f and 7h). It is known that the particle/matrix interfacial bonding is crucial to the mechanical property of material. During deformation process, large stress will concentrate at the interface of W/Al. If the interfacial bonding strength is high enough, the fracture will first occur at particles rather than the matrix or the interface. Fig.7c shows that even though the W_p is fractured, it will not debond from the matrix, indicating a strong interfacial bonding.

2.4 Electrical conductance properties

Fig.8 shows the electrical resistivity of as-rolled 6061Al and its as-rolled composites with different contents of W_p at

Table 2 Mechanical properties of as-rolled 6061Al alloy and W_p/6061Al composites

Material	6061Al	1WAl	3WAl	5WAl	7WAl
σ_{UTS}/MPa	169.31	192.85	315.18	283.95	158.60
σ_{YS}/MPa	142.33	160.18	289.25	251.36	124.76
Elongation/%	17.26	16.84	11.93	6.62	5.80

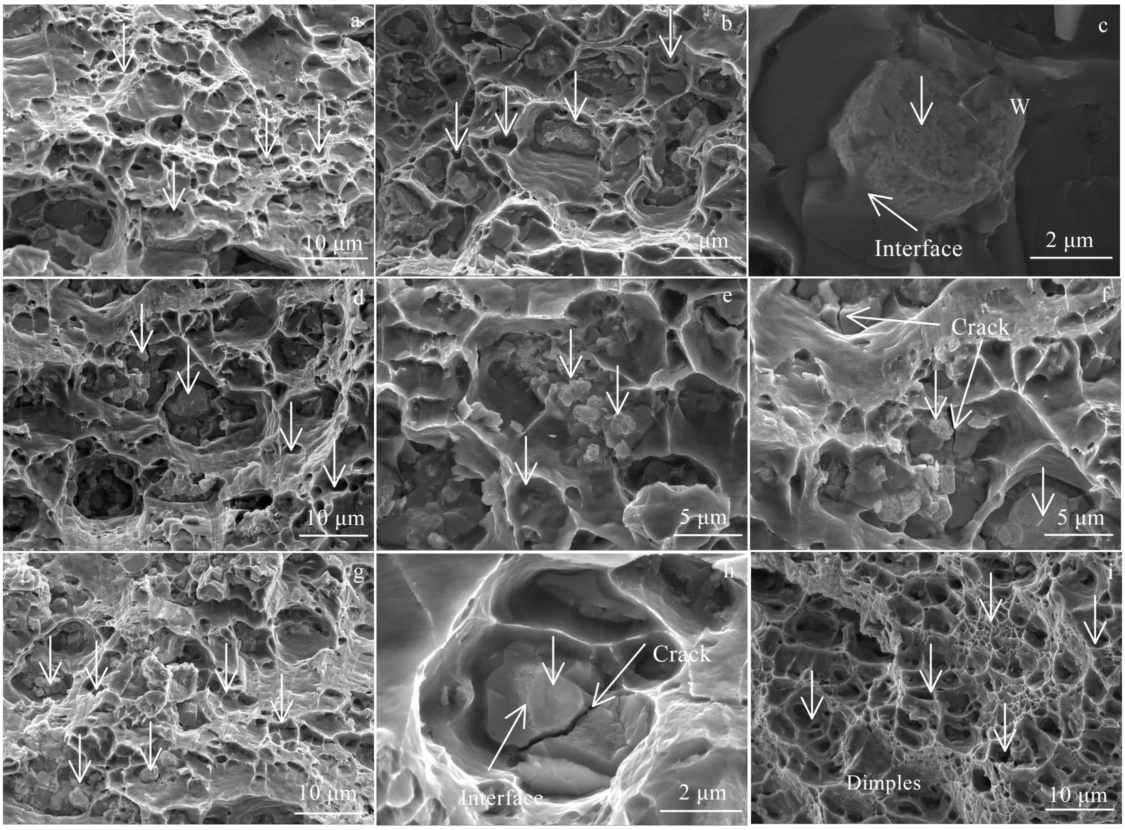

Fig.7 Tensile fractographs of as-rolled composites with different W_p contents: (a) 1 vol%, (b, c) 3 vol%, (d~f) 5 vol%, and (g, h) 7 vol%; (i) as-rolled 6061 Al alloy

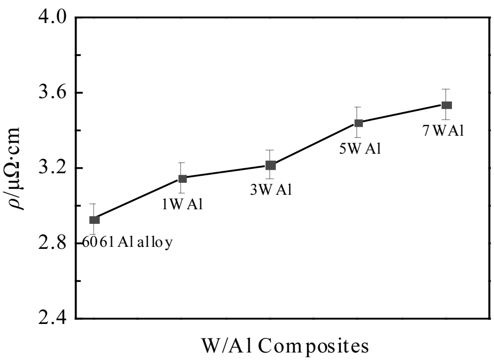

Fig.8 Electrical resistivity of as-rolled 6061Al alloy and its composites at room temperature

room temperature. The value of electrical resistivity of all as-rolled composites is higher than that of as-rolled 6061Al matrix, but all remains at a relative low level. The electrical conductance property of composite materials is mainly affected by the grain size and the particles in the matrix.

The fast heating rate and lower holding time during SPS process can effectively avoid grain coarsening of the 6061Al matrix. Besides, the large plastic deformation of 6061Al matrix during hot rolling also leads to the grain refinement[21]. By reducing the grain size, the total grain boundary resistivity can be increased, thus achieving higher electrical resistivity[12]. The presence of W_p can impede electric current in the composites due to its higher electrical resistivity (5.56 μΩ·cm). Thus, with the increase of W_p content, the electrical conductivity of composites decreases.

3 Conclusions

1) In the W_p/6061Al composites, W_p is homogeneously distributed in the 6061Al matrix and the interface between the W and Al is well bonded.

2) With the increase of W_p content, the strength of the as-rolled W_p/6061Al composites first increases and then decreases, while the ductility decreases. The strength and ductility of 7WAl decrease due to the W_p aggregation.

3) The electrical resistivity of the W_p/6061Al composites has a little increase compared to 6061Al matrix. The W_p/6061Al composites fabricated by SPS followed by hot-rolling show excellent electrical conductivity.

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基于 SPS-热轧法钨颗粒增强铝基复合材料的微观组织及力学性能研究

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摘 要：采用放电等离子烧结后加热轧制方法制备了不同体积分数钨颗粒增强 6061Al 基复合材料，钨颗粒含量为 1 vol%～7 vol%，研究了不同的钨颗粒含量对轧制态复合材料的微观组织、力学性能和电阻系数的影响。结果表明：钨颗粒均匀的分布在金属基体当中，颗粒界面之间实现了冶金结合。在制备过程中，钨颗粒与 6061Al 颗粒之间界面处产生固溶体和 WAl_{12} 金属间化合物；力学测试结果表明复合材料内部随着 W 颗粒体积分数的增加，轧制态复合材料的致密度和韧性降低，而抗拉强度呈现先增加后降低的趋势；在复合材料内部钨颗粒含量为 1 vol%和 3 vol%时，复合材料的抗拉强度和延伸率分别为 192.85 MPa, 16.84%和 315.18 MPa, 11.93%。此外，制备的钨颗粒增强 6061Al 基复合材料具有较好的导电能力，钨颗粒含量的增加对复合材料的导电性能影响较小。

关键词：放电等离子烧结；W_p/6061Al复合材料；微观组织；力学性能；电阻系数

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