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Fabrication of SiC reinforced aluminium metal matrix composites through microwave sintering

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Abstract
In this study, SiC reinforced aluminum (Al) metal matrix composites were prepared by microwave sintering. The effects of SiC content, pressure and sintering temperature on the density, hardness and microstructure of the composites were investigated. It was found that the relative density of SiC/Al composites is 98.43% when the content of SiC is 5 Vol%. When the SiC content is 15 Vol%, the sintering temperature is 770 °C and the pressure is 250 MPa, the density and hardness of the composites SiC/Al composites are 96.14% and 130 HV, respectively. And the SiC particles can be uniformly dispersed in the Al matrix by microwave sintering.

1. Introduction

With the rapid development of lightweight materials, the requirements for material properties are becoming higher and higher, such as good high temperature resistance, good corrosion resistance, good fatigue resistance, high specific strength, good thermal conductivity and low thermal expansion coefficient [1–3]. At the same time, it requires low manufacturing cost, light weight, isotropy and high reliability. A single material can no longer meet the requirements of lightweight materials. In order to adapt to the new material requirements, the research of composite materials is a hot spot for researchers. Since the composite has the advantages of both the material and the reinforcing phase material. The performance of the material can be greatly improved. Therefore, it is extremely important to select a suitable matrix and a reinforcing phase.

Aluminum and aluminum alloys have many excellent physical and chemical properties, such as low density, good corrosion resistance, good ductility, good thermal conductivity and good electrical conductivity [4–6]. They are widely used in aviation, aerospace, electronics, electrical appliances and automobiles. However, due to the low hardness, poor wear resistance, high thermal expansion coefficient, and poor mechanical properties of aluminum or aluminum alloy, the application of aluminum or aluminum alloy in the industry is greatly limited. Adding SiC to aluminum and aluminum alloy can significantly enhance the mechanical properties and wear resistance of the composite and maintain the lightweight characteristics. So, it becomes an important research field.

At present, there are many methods for preparing SiC-reinforced Al-based composite materials [7–9]. The commonly used methods include powder metallurgy, stirred casting, pressureless infiltration, spray deposition, in situ synthesis, etc. However, these methods have certain limitations, and the traditional heating method has a high surface temperature and a large loss of sintering power. The microwave heating has the characteristics of overall rapid heating, and the microwave is passed through the coupling of the material itself (medium loss) to achieve densification of the material [10–13]. In particular, it has special advantages in the sintering of powder metallurgy, which can improve the distribution of temperature in the sintered embryo to reduce the sintering temperature, and shorten the reaction time. The manufactured product has excellent physics mechanical properties. In this paper, SiC/Al composites were prepared by microwave sintering. The effects of different

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silicon carbide content, pressure and sintering temperature on the density, hardness and microstructure of SiC/Al composites were studied.

2. Experiment

2.1. Experimental materials
In this experiment, SiC produced by Shanghai Maclean Biochemical Technology Co., Ltd. and was used as a reinforcement. The median diameter ($D_{50}$) of the original SiC powder was 0.997 μm. The matrix material used in the experiment was atomized pure aluminum powder, which was produced by Tianjin Zhiyuan Chemical Reagent Co., Ltd. (the purity was 99.0%). The median diameter ($D_{50}$) of the original pure Al powder was 33.113 μm.

2.2. Experimental methods and measurements
The press-formed sample was placed in a microwave vacuum sintering furnace for sintering. The schematic diagram of the structure of the microwave vacuum sintering furnace is shown in figure 1.

The press-formed sample was placed in the center of the crucible, and the sample was filled with a silicon carbide sheet. Place the crucible in the center of the mullite incubator, which is placed in the center of the rotating table (directly below the infrared thermometer). After the furnace door is closed, the vacuum is evacuated to a certain negative pressure, and Ar gas is filled into the furnace as a shielding gas. Then, the microwave is fed for sintering, the heating rate is controlled to be 35 °C–40 °C min$^{-1}$, heated to the set experimental temperature and kept warm, and the temperature is measured by an infrared thermometer. After the sintering is completed, the furnace is cooled and the sample is taken out.

The density of the sample after sintering was determined by Archimedes drainage method. The sintered sample was polished, and the microhardness of the sintered sample was measured by Vickers hardness tester. The microstructure of the sample were observed by scanning electron microscopy.

3. Results and discussions

3.1. Effect of SiC content on properties and microstructure of SiC/Al composites
It can be seen from figure 2 that the relative density of SiC/Al composites is 98.43% when the volume fraction of SiC is 5 Vol%. With the increase volume fraction of SiC, the relative density of SiC/Al composites decreases gradually due to the good plasticity and fluidity of aluminum, and the high hardness of silicon carbide. As the volume fraction of SiC increases and the flow of aluminum in the process, defects such as agglomeration and voids of local SiC particles appear in the green body, which leads to the decrease of the relative density of SiC/Al composites with the increase of SiC content.

It can be seen from figure 3 that the hardness of SiC/Al composites increases first and then decreases with the increase of SiC volume fraction. When the SiC volume fraction is 15 Vol%, an inflection point occurs and the hardness reaches a maximum. This may be due to the formation of a phase interface between the aluminum matrix and the SiC particle reinforcement. As the reinforcement particles increase, the phases interface increases, and the ability of the composite to withstand the load increases, so the hardness increases as the...
The volume fraction of SiC increases. However, increasing the content of SiC will lead to an increase in agglomeration. When the composite is subjected to an applied load, the stress will be generated at the interface of the reinforcement. Under the action of the stress, the composite begins to undergo microplastic yielding. These microplastic yielding mainly occurs in the pores formed by the reinforcement agglomeration and the sharp corners of the interface where the reinforcement and the matrix are combined. Therefore, under the action of external force load, the reinforcement agglomeration area is easy to form stress concentration, thereby losing the ability to transmit and bear external loads, thereby causing the material to be easily broken and the hardness decreased when subjected to load.

Figure 4 is a scanning electron micrograph of a SiC/Al composites with different SiC contents by microwave sintering at a sintering temperature of 770 °C under a pressure of 250 MPa. It can be seen from the figure 4 that an appropriate amount of SiC particles can be uniformly dispersed in the Al matrix. When the volume fraction of SiC reaches 20 Vol%, the reinforcement has local agglomeratio, resulting in the appearance of many voids, which leads to a decrease in the density of the composite. This is because the matrix particles are much larger than the reinforcement particles, and the composite powders are not completely uniformly mixed together during the mixing process. As the content of the reinforcement increases, the probability of segregation of the reinforcement increases.

Stone et al[15] found that the ratio of matrix particles and reinforcement particles affects the distribution of reinforcement particles in the matrix. When the particle sizes of the two powders are nearly the same, the more uniform the distribution of the reinforcement in the matrix; and the larger the particle size difference, the more
uneven the reinforcement distribution. Since the particle size of the matrix particles selected in the research is much larger than the particle size of the reinforcement particles. Therefore, the distribution of the reinforcing phase in the matrix is affected.

3.2. Effect of pressure on properties and microstructure of SiC/Al composites

When the SiC content is 15 vol% and the sintering temperature is 770 °C, the effect of pressure on properties and microstructure of SiC/Al composites was studied. As can be seen from figure 5, the relative density of SiC/Al composites increases firstly with increasing pressure. This is because during the pressing process, the powder particles are in the sliding stage, and as the particles are continuously displaced, the pores are continuously filled. When the pressure increases to 250 MPa, the relative density of the SiC/Al composites reaches a maximum. Continue to increase the pressure of the compact, the relative density of the SiC/Al composite is slightly reduced. This is because the compacts relative density reaches a certain value after the

Figure 4. Effect of SiC content on microstructure of SiC/Al composites (a) 5 Vol%; (b) 10 Vol%; (c) 15 Vol%; (d) 20 Vol%.

Figure 5. Effect of pressure on the relative density of SiC/Al composites.
sliding stage, and a certain compression resistance occurs between the particles. Continue to increase the pressure of the green compact can not reduce the porosity of the sample, and a certain work hardening phenomenon occurs after sintering. A certain recovery and recrystallize will occur. The relative density of SiC/Al composites has a certain downward trend [16, 17].

It can be seen from figure 6 that the hardness of the SiC/Al composites increases first and then decreases. When the pressure is less than 250 MPa, the hardness of the SiC/Al composite increases. This is because the porosity of the green compacts decreases as the pressure increases. The green compacts gradually becomes denser, and the bonding force between the particles increases the hardness of the SiC/Al composites. When the pressure reaches 250 MPa, the hardness of the SiC/Al composites reaches 130 HV. Work hardening occurs as increase the pressure. Excessive compacting pressure will cause work hardening of the green compacts, resulting in poor formability of the green compacts and uneven density distribution of the green compacts, which further leads to a decrease in the hardness of the SiC/Al composite material.

Figure 7 is a scanning electron micrograph of SiC/Al composites after microwave atmospheric pressure sintering at a sintering temperature of 770 °C and different molding pressures (100 MPa, 150 MPa, 200 MPa, 250 MPa, 300 MPa, 350 MPa). It can be seen from the figure that as the pressure of the green compact increases, the gap between the SiC particles and the Al matrix gradually decreases, and the phase interfaces of the two are continuously improved. With proper pressure, the SiC particles can be uniformly dispersed in the Al matrix, and the phase interface effect of the two is better. After analysis, it is found that the SiC/Al composite powder under the action of high temperature, the fluidity of the Al matrix powder is intensified. The atoms on the surface are diffused, and the phase interface between the SiC particles and the Al matrix is gradually increased, and the surface of the original matrix particles becomes the grain boundaries form a metallurgical bond. As the pressure of the green compact increases, the pores at the interface between the SiC particles and the Al matrix are gradually filled, and the density of the composite increases and the strength begins to increase. As the pressure of the green compact increases, the reinforcement agglomerates locally, and the reinforcement and the reinforcement overlap each other, resulting in the appearance of many pores, which leads to the decrease of the density of the composite and the mechanics of the composite.

3.3. Effect of sintering temperature on properties and microstructure of SiC/Al composites

When the SiC content is 15 vol% and the pressure is 250 MPa, the effect of sintering temperature on properties and microstructure of SiC/Al composites was studied. Figure 8 shows the change in the relative density of the sintered samples at different sintering temperatures. It can be seen from the figure 8 that the relative density of the SiC/Al composite increases with the increase of the sintering temperature. At the sintering temperature of 770 °C, the density of the SiC/Al composite reaches a maximum value of 96.14%. Continue to increase the temperature and the density of the SiC/Al composite begins to decrease. This is because of the selective heating of the microwave, the SiC particles are first heated by the microwave, and the heat generated by the SiC particles is transferred to the sample itself by heat conduction. When the sample temperature reaches a certain temperature, its heating mode is changed from heat conduction to volume heating. Thereafter, the Al particles around the SiC particles began to melt, and the interconnected pores of the particles began to shrink and close the pores, and the number of pores in the sample began to be greatly reduced, and the sintered sample began to
densify. However, as the temperature increases further, the SiC particles begin to aggregate in the liquid Al, resulting in uneven heating of the Al in the sample and thermal runaway [18], and resulting in a decrease in density.

Figure 9 shows the change in hardness of sintered samples at different sintering temperatures. It can be seen from the figure 9 that the hardness of SiC/Al composite increases with the increase of sintering temperature. When the sintering temperature reaches 770 °C, the hardness of SiC/Al composite reaches the maximum value. As the temperature rises, the hardness of the sample begins to decrease. This is because the microstructure of the sintered sample changes as the sintering temperature changes. At lower or too high temperatures, the microstructure of the sample is heterogeneous and there are more holes, so the hardness is lower. At the sintering temperature of 770 °C, the interface between SiC particles and Al in the sample is relatively tight, and the density of the sample is large, so the hardness is high.

Figure 10 shows the microstructure of the samples at different sintering temperatures. As can be seen from the figure 10, the microstructure of the sample is different at different sintering temperatures. When the
sintering temperature is 750 °C, the SiC particles absorb microwaves and are converted into heat which is absorbed by Al. Then Al around the SiC starts to melt. As the molten state of Al increases, the sintered sample begins to densify, as shown on the microstructure diagram at 750 °C. As the sintering temperature increases, the pores in the sample are gradually filled with aluminum, and the density of the sample begins to increase. When the sintering temperature reaches 770 °C, the density of the sample reaches the maximum. When the sintering temperature continues to rise to 790 °C, the SiC particles aggregate in the liquid Al, resulting in a more uneven microstructure of the sample [19].

Figure 11 is an XRD diffraction pattern of a 15 Vol% SiC/Al composite prepared at different sintering temperatures. Phase analysis revealed that when the temperature was 710 °C, the phases of the SiC/Al composite were mainly Al and SiC, and almost no other impurity phases were formed. When the sintering temperature rises to 750 °C, an interfacial chemical reaction begins between the Al matrix and the SiC particles, and an impurity phase Al4C3 is formed [20]. When the temperature reaches 790 °C, a large amount of impurity phases begin to form. The diffraction peak of SiC is basically unchanged, and the peak of Al is weakened. From the perspective of thermodynamics, in the
Al-SiC system, the interface chemical reaction between $\text{Al}_4\text{C}_3$ and SiC to form $\text{Al}_4\text{C}_3$ is shown in (1):

$$4\text{Al} + 3\text{SiC} = \text{Al}_4\text{C}_3 + 3\text{Si}$$  \hspace{1cm} (1)

Studies have shown that a small amount of $\text{Al}_4\text{C}_3$ phase helps to improve the interfacial wettability between the Al matrix and the SiC particles. However, as a brittle phase, the amount of $\text{Al}_4\text{C}_3$ will decrease the strength of the composite. And at the same time, a large amount of Al matrix and SiC particles will be consumed. Lin [21] and Lee [22] found that controlling process parameters, such as temperature, pressure, and cooling rate, can reduce the generation of impurity phases.

4. Conclusions

In this study, SiC/Al composites were prepared by microwave sintering. The optimal conditions are that SiC content is 15 vol%, the sintering temperature is 770 °C and the pressure is 250 MPa. It was found that the relative density and hardness of SiC/Al composites prepared under this optimal conditions are 96.14% and 130 HV, respectively. The microstructure is significantly improved by microwave sintering.

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