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Effects of Sintering and Extrusion on the Microstructures and Mechanical Properties of a SiC/Al-Cu Composite

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This article studied the effects of sintering and extrusion on the microstructures and mechanical properties of SiC particle reinforced Al-Cu alloy composite produced by powder metallurgy method. It has been shown that both extrusion and increasing sintering temperature can significantly improve the strength and plasticity of the composite. The extrusion and increase of the sintering temperature can break up the oxide coating on the matrix powder surfaces, decrease the number of pores, accelerate the elements' diffusion and increase the density and particle interfacial bonding strength, thus significantly improve the mechanical properties of the composite. The strength and hardness of the composite increase and the elongation decreases with increasing the aging time at under-aged stage, while the strength and hardness start to decrease and the elongation starts to increase with increasing the aging time at over-aged stage due to the formation and growth of the secondary strengthening precipitates in the Al-Cu matrix.

Keywords extrusion, mechanical testing, metal matrix composites, powder metallurgy

1. Introduction

SiC particle reinforced aluminum alloy composites provide significantly enhanced mechanical properties over the corresponding matrix alloys, such as the high strength, modulus, wear resistance, and fatigue resistance. These composites are the typical candidates for engineering applications in aerospace, military, and civil manufacturing industries (Ref 1-4). In general, many techniques have been developed to fabricate the SiC particle reinforced aluminum alloy composites, including casting, powder metallurgy, molten metal methods, pressure infiltration, and spray deposition (Ref 5-9).

Among all the fabrication methods, the powder metallurgy (P/M) processing technique is attractive because it offers uniform distribution of the reinforcements, fine grained structures, and easy controlling of the microstructures (Ref 10). However, P/M processing technique normally employs lower temperature sintering, therefore reduces the diffusion rates and interfacial bonding strength (Ref 7). As shown by Lee et al. (Ref 11), for Al matrix composites reinforced with SiC particles the formation of Al_4C_3 phase by the reaction between the SiC reinforcement and the Al matrix is a problem in controlling the microstructures and mechanical properties, and this problem has also been pointed out by Shin et al. (Ref 12). Another problem of the P/M method is that the Al oxide coating on most commercially atomized powders can act as the barrier for the atom diffusion during sintering, especially solid phase sintering. Under that condition, extensive hot deformation after sintering, such as forging and extrusion, can significantly break the oxide coating and improve the interfacial bonding strength of the composites, and thus improve the density and mechanical properties.

The understanding of the relationships between the processing parameters and microstructures is very important to improve the mechanical properties of the composites. In the last several decades, a lot of researchers had explored the effects of the processing parameters on the microstructures and mechanical properties of the SiC reinforced aluminum alloy composites. Tham et al. (Ref 9) investigated the effect of the reinforcement volume fraction on the evolution of the reinforcement size during the extrusion of Al-SiC composites produced by disintegrated melt deposition technique. Yotte et al. (Ref 13) characterized the particle clustering in SiC reinforced Al-X2080 alloy matrix composites. Hong and Chung (Ref 14) explored the volume fraction of the reinforcement, vacuum hot pressing temperature, vacuum hot pressing pressure, extrusion temperature, and extrusion ratio on the mechanical properties of SiC whisker reinforced 2124 Al composites. Davis et al. (Ref 15) studied the effect of particle size and volume fraction on the mechanical properties of SiC particulates reinforced 2080 Al matrix composites. Rahmani Fard and Akhlaghi (Ref 8) explored the effect of extrusion temperature on the microstructure and porosity of A356/SiC_p composites. Song et al. (Ref 16) developed a constitutive model to calculate the effect of the volume fraction and granularity of the SiC particles, and aging time and temperature on the yield strength of Al-Cu-Mg and Al-Mg-Si alloy-based composites. The effects of volume fraction of SiC particles, die-pressing pressure, and extrusion on the microstructures and mechanical properties of pure Al-based composites were also studied by Song et al. (Ref 17, 18).

In this article, we studied the effects of sintering temperature and extrusion on the microstructures and mechanical properties of a SiC particle reinforced Al-Cu alloy composite. Scanning electron microscope (SEM) and mechanical testing were used

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to characterize the microstructures and mechanical properties of the composite.

2. Experimental

The matrix alloy used in this study is Al-3.47%Cu alloy (weight percentage) with the total impurities in the matrix being less than 0.2 wt.%. The Al-Cu powders were produced by inert gas atomization, and have an average size of about 60 μm after sieving. The SiC reinforcements are in the form of particulate with a diameter of about 77 μm. The aluminum powders were mixed with about 20 vol.% SiC particulates for 7 h using a powder rotator mixer. The ball to powder weight ratio was 1:4. The mixed powders were die-pressed at room temperature under a pressure of 200 MPa in a cylindrical steel die. The specimens were then heated in a vacuum furnace (pressure of 7×10^{-3} Pa) with a heating rate of 10 °C per minute to 470, 520, 570, and 610 °C, respectively. Then the specimens were sintered under a pressure of 25 MPa for 5 h. After sintering, the specimens were hot extruded to rods at 430 °C with an extrusion ratio of 9:1. The extruded rods were solution heat-treated for 6 h at 505 °C, followed by cold water quenching to the room temperature. Then the rods were artificially aged for various periods of time at a temperature of 177 °C.

The bulk densities of the composites before and after extrusion were measured by standard Archimedes method. The sizes of the SiC particles in all the specimens before and after extrusion are characterized using image analysis software (Image J, Bethesda, MD, USA). More than 500 SiC particles are analyzed during particle size measurements and average values are used. The HB hardness measurements were performed on all the specimens before and after extrusion, using a 2.5 mm diameter steel ball as the indenter with an applied force of 62.5 N for 30 s. The HB hardness measurements were also performed on all the aged samples to explore the relationships between the mechanical properties and aging time. The yield strength, tensile strength, and elongation of the composites under different aging times were tested by tensile testing. At room temperature, the dog-bone-shaped tensile specimens, having a gage size of 6 mm in diameter and 40 mm in length, were served in the tensile tests with a constant strain rate of 5×10^{-4} s^{-1} on an Instron 8802 testing machine. The yield stress was determined at the 0.2 pct offset. All the specimens have an axis along the extrusion direction. Each point of the yield strength values has been measured on three specimens and the average value was used. Differential scanning calorimetry (DSC) examination was performed on the matrix alloy to determine the thermal properties from 50 to 700 °C, with a heating rate of 10 °C per minute. The microstructures of the composites and the fracture surfaces of the tensile specimens before and after extrusion were studied using a FEI Nano230 field emission scanning electron microscope (SEM).

3. Results

3.1 Microstructures of the Composites Before and After Extrusion

Figure 1 shows the microstructures of the composite after sintering at different temperatures before extrusion. It can be seen that the SiC particles are distributed quasi-uniformly in the Al-Cu matrix with a little aggregation after sintered at the temperatures from 470 to 610 °C. At the same time, pores (arrowed) are generally observed in all the specimens. It can also be seen that the number of the pores is significantly decreased with increasing the sintering temperature. The bonding strength between the SiC particles and the matrix has largely been improved since high sintering temperature can accelerate the diffusion rate.

Figure 2 shows the microstructures of the composites after extrusion. It can be seen that the number of the pores in the matrix decreases with increasing the sintering temperature, similar to Fig. 1 before extrusion. The most important feature is that the number of the pores in the as-extruded composite is largely decreased compared to the composites before extrusion, no matter what is the sintering temperature. By comparing Fig. 1 and 2, we can clearly see that the size of the SiC particles is decreased after extrusion due to the breaking up of the particles during extrusion. The image analysis results showed that the sizes of the SiC particles are approximate 65 and 44 μm before and after extrusion, respectively. Compared to the original particle size of 77 μm, it has already been decreased after mixing, die-pressing, and sintering processes due to the breaking up of the particles. At the same time, it should be noted that the size of SiC particles has largely been decreased after extrusion. It is believed that extrusion can obtain a more uniform distribution of the SiC particles, decrease the number of the pores, decrease the size of the SiC particles, and improve the interfacial bonding strength, since extrusion can break up the oxide coating on the surface of the aluminum powders and enhance adhesion between the powders, as shown by Cöcen and Önel (Ref 6), Rahmani Fard and Akhlaghi (Ref 8), and Tham et al. (Ref 9). The reduction in the particle size was due to the fracture of large-sized particles as a result of the high stress concentration on the particles during the extrusion process.

3.2 Density Evolutions of the Composite Before and After Extrusion

Figure 3 shows the density evolution of the composite as a function of the sintering temperature both before and after extrusion. It can be seen from Fig. 3 that the density increases with increasing the sintering temperature both before and after extrusion. This agrees well with Fig. 1 and 2, in which the number of the pores decreases with increasing the sintering temperature. It can also be seen that no matter what is the sintering temperature, the density of the composites is higher after extrusion than before extrusion, indicating that extrusion can improve the density of the composite. It should be noted that the density of the composite after extrusion are very close to the theoretical density (note that the theoretical density of the composite in this work is 2.825 g/cm^3).

3.3 Hardness Evolution of the Composite Before and After Extrusion

Figure 4 shows the HB hardness evolution of the composite as a function of the sintering temperature both before and after extrusion. It can be seen that the hardness increases with the sintering temperature both before and after extrusion. From Fig. 1 to 3, we know that increasing the sintering temperature can decrease the number of the pores and enhance the interfacial bonding strength between the SiC particles and the

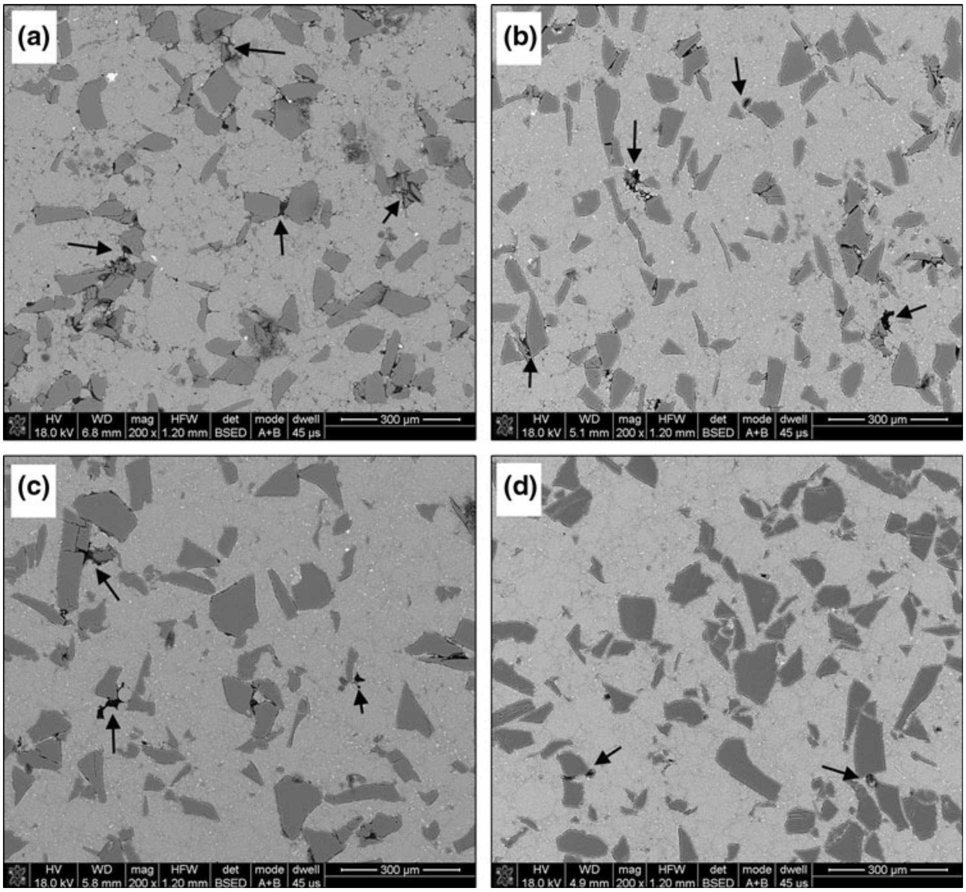

Fig. 1 Microstructures of the composite after sintering at different temperatures before extrusion. The sintering temperatures are (a) 470 °C, (b) 520 °C, (c) 570 °C and (d) 610 °C, respectively

Al-Cu matrix, thus inevitably improve the hardness of the composite. It can also be seen that no matter what is the sintering temperature, the hardness of the composites is higher after extrusion than before extrusion, indicating that extrusion can improve the hardness of the composite. It should be noted that the evolution of the hardness has also been observed previously (Ref 19). It should also be noted that the hardness of the corresponding Al-Cu alloy sintered at 570 °C are 54.5 and 67.5 MPa before and after extrusion, respectively; while the hardness of the SiC reinforced Al-Cu composites sintered at 570 °C are 70.6 and 72.8 MPa before and after extrusion, respectively. It can thus be concluded that the addition of SiC particles generates a higher resistance to the indentation deformation.

3.4 Hardness of the Composite as a Function of Aging Time

Figure 5 shows the HB hardness curves of the composites aged for different times at 177 °C. It can be seen that the hardness increases with increasing the sintering temperature no matter what is the aging time. Most importantly, the hardness increases initially with the aging time up to 20 h, after which the hardness starts to decrease with the aging time. It is generally accepted that the precipitation sequence in Al-Cu alloys can be expressed as: G.P. zone $\rightarrow \theta''$ phase $\rightarrow \theta'$ phase $\rightarrow \theta$ phase when the aging temperature is below 190 °C (Ref 20, 21). So the increase of the hardness of the composite at under-aged stage is attributed to the formation of the G.P. zone and θ'' phase, while the peak-aged hardness is attributed to the formation of the θ' phase. The decrease in the hardness starts with the formation of the stable θ (Al_2Cu) phase. The HB hardness of the Al-Cu alloy sintered at 570 °C and aged for 8, 20, and 24 h are 62.4, 76.3, and 70.4 MPa respectively, which these values are significantly lower than the hardness of the SiC particles reinforced Al-Cu composites sintered at 570 °C (see Fig. 5 for details). It can thus be concluded that the addition of SiC particles improves the hardness of the composites.

3.5 Tensile Properties of the Composite

Figure 6 illustrates the tensile mechanical properties of the composite. It can be seen from Fig. 6 that the yield strength and tensile strength of the composite increase with increasing the sintering temperature both before and after extrusion. However, it should be noted that the elongation of the composite increases initially with the sintering temperature up to 570 °C, after which it decreases. The increases in both the yield strength and the tensile strength with the sintering temperature from 470 to 610 °C are due to the decreased number of the pores and enhanced interfacial bonding strength of the composite sintered at high temperature. Figure 7 is the DSC curve of the matrix alloy. It can be seen from Fig. 7 that the melting point of the matrix is around 651 °C, while there is a small exothermic peak around 604 °C. This small exothermic peak is corresponding to the partial melting of intermetallic compounds along the grain

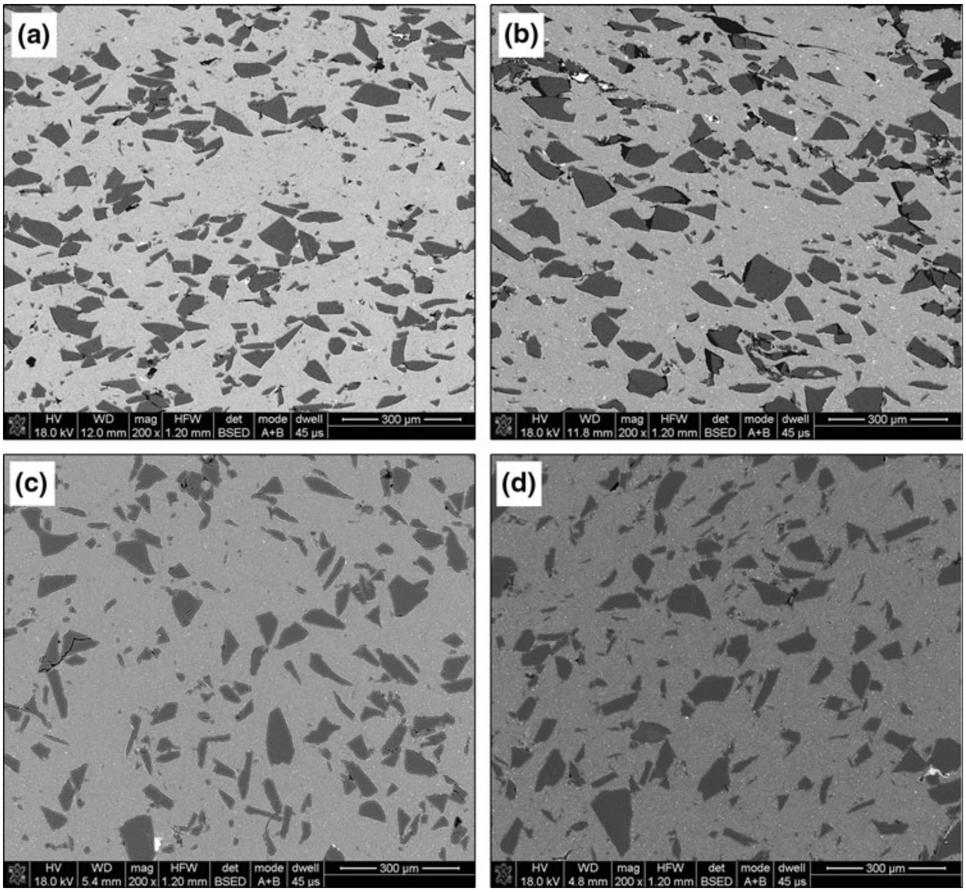

Fig. 2 Microstructures of the composite after sintering at different temperatures after extrusion. The sintering temperatures are (a) 470 °C, (b) 520 °C, (c) 570 °C, and (d) 610 °C, respectively

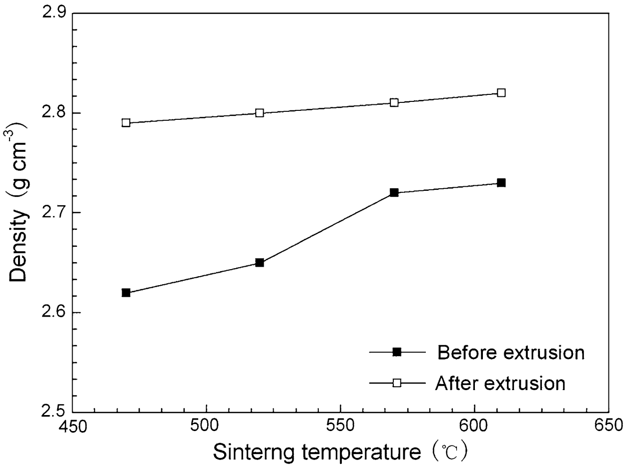

Fig. 3 Density evolution of the composite as a function of the sintering temperature

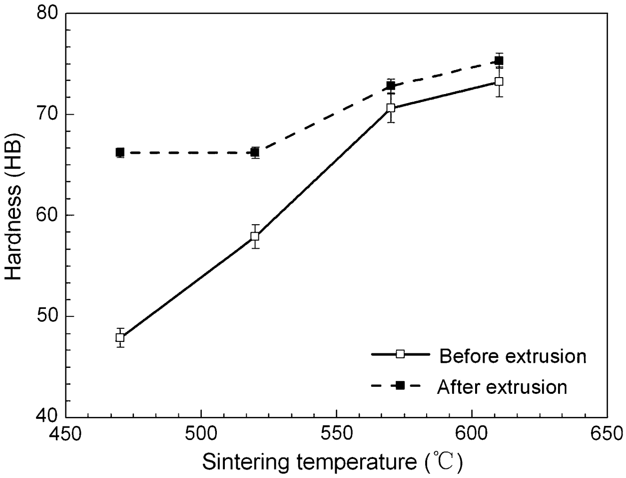

Fig. 4 HB hardness evolution of the composite as a function of the sintering temperature

boundaries. Thus, the decrease of the elongation of the composite sintered at 610 °C may be due to partial melting of intermetallic compounds with low melting point along the grain boundaries during the sintering process. Theoretically, for binary Al-3.47%Cu alloy, the Cu will be totally dissolved into the Al matrix at 548.2 °C. Therefore, the composite sintered at 570 and 610 °C should be super solid sintering, and precipitates will nucleate and grow in the matrix during cooling inside the furnace, and thus resulting in precipitate strengthening. However, it should be noted that in SiC particle reinforced Al-Cu alloy composites, the SiC is the main strengthening factor. The precipitates generated during cooling after sintering has neglect effect on the hardness and strength of the composites, compared to the SiC particles.

3.6 Fracture Surfaces of the Composite After Tensile Testing

Figure 8 shows the SEM fracture surfaces of the composite after tensile testing before extrusion. It can be seen from Fig. 8(a) and (b) that no obvious plastic deformation can be observed along the fracture surfaces when the sintering temperatures are 470 and 520 °C. On the other hand, spherical aluminum powders can clearly be observed, indicating the poor cohesion of the powders and thus the degraded mechanical properties. This agrees well with the microstructures observation in Fig. 1, the density testing in Fig. 3 and the hardness testing in Fig. 4 under the sintering temperature of 470 and 520 °C. It can also be seen from Fig. 8(c) and (d) that a mixed fracture mode including typical ductile dimples of the matrix and decohesion of the SiC particles from the matrix can be observed. Lots of the small dimples exist along the fracture surfaces generated by the plastic deformation of the matrix. Some large voids with the size similar to SiC particles can also be observed, generated by decohesion of the SiC particles from the matrix. It can thus be concluded that the fracture of the composites after sintering with the temperatures of 570 and 610 °C is dominated by the ductile fracture of the matrix, accompanied by decohesion of the SiC particles from the matrix.

Figure 9 shows the SEM tensile fracture surfaces of the composite after extrusion and aged to the peak-aged stage. It can be seen that all the fracture surfaces show a mixed fracture mode. In the Al-Cu matrix, the existence of the numerous small dimples along the matrix indicates a ductile fracture of the matrix, while many fractured SiC particles can also be observed

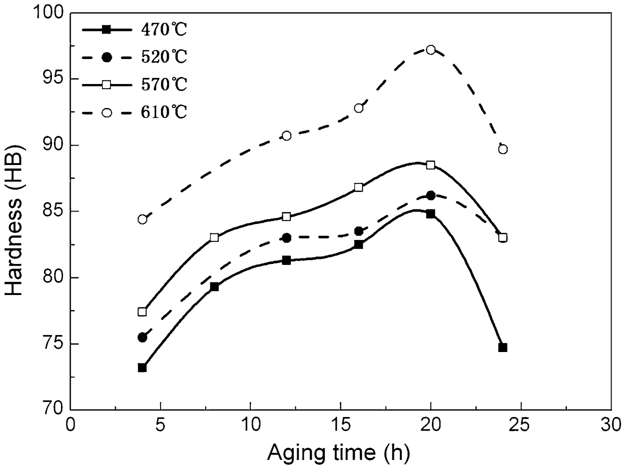

Fig. 5 The HB hardness as a function of the aging time for the composite under different sintering temperatures

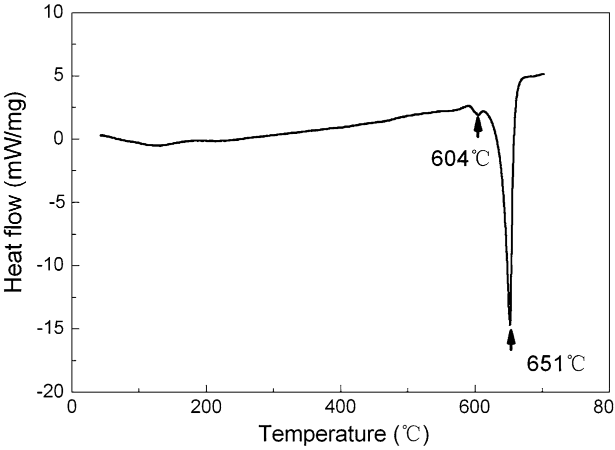

Fig. 7 The DSC curve of Al-Cu matrix

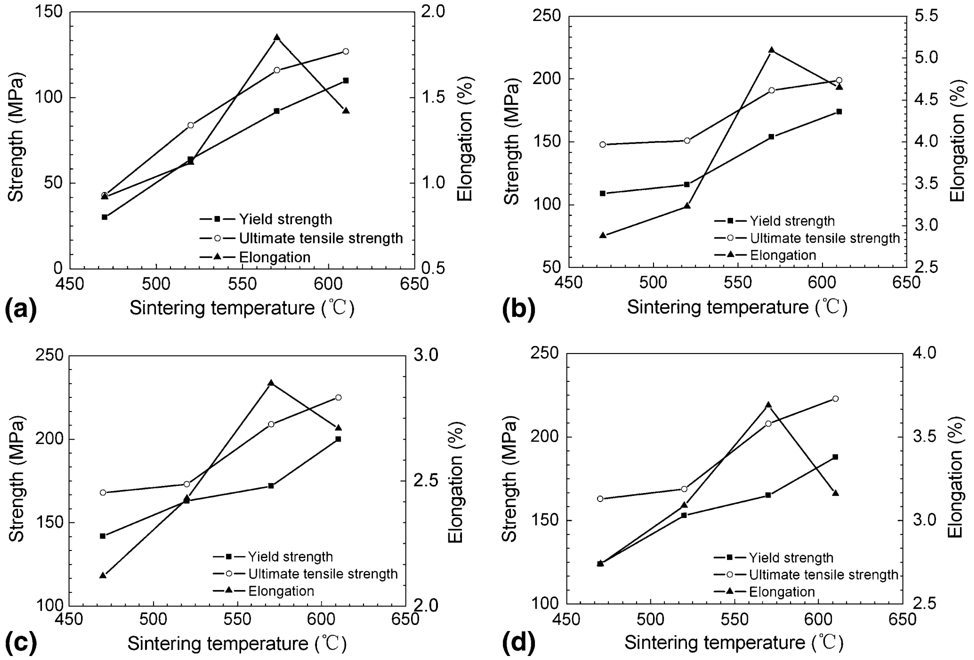

Fig. 6 Yield strength, ultimate tensile strength, and elongation evolutions as a function of the sintering temperature. (a) Before extrusion, (b) after extrusion and aging for 8 h, (c) after extrusion and aging for 20 h, and (d) after extrusion and aging for 24 h

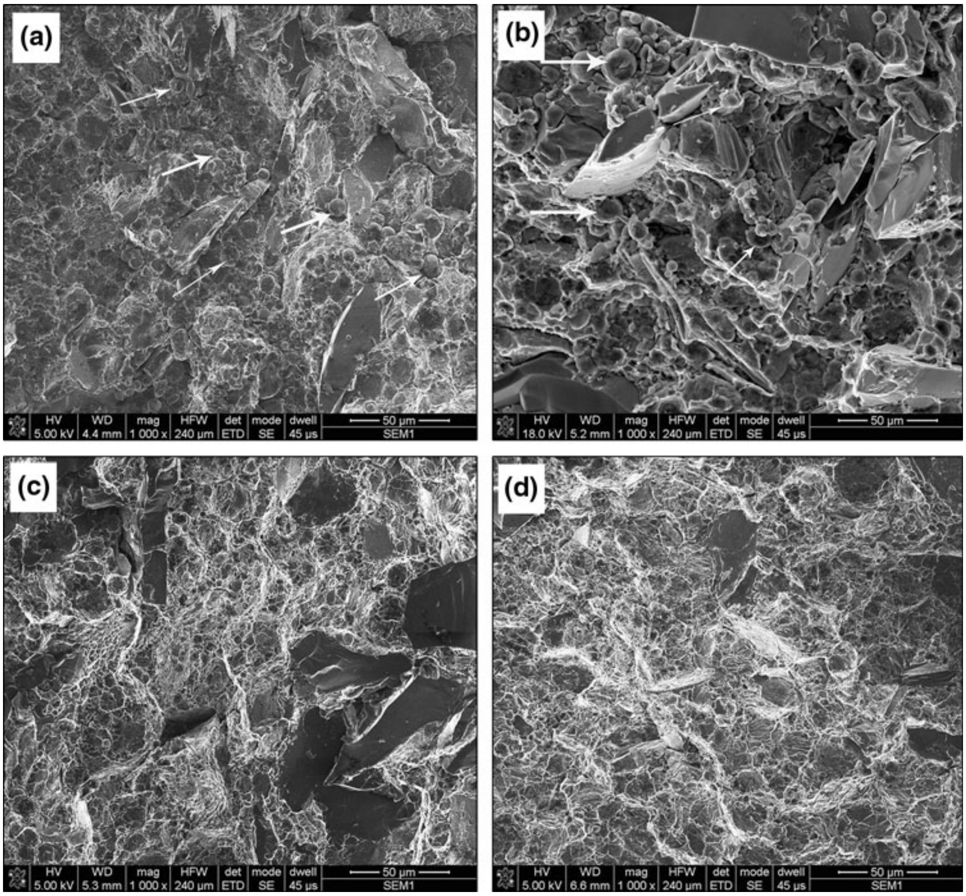

Fig. 8 SEM fracture surfaces of the composite after tensile testing before extrusion. The sintering temperatures are (a) 470 °C, (b) 520 °C, (c) 570 °C, and (d) 610 °C, respectively

along the fracture surfaces. This observation indicates that the fracture of the composites after extrusion is dominated by the ductile fracture of the matrix and the fracture of the SiC particles. It should be noted that all the fracture surfaces of the composites after extrusion and aged for different times show similar pattern, indicating the similar deformation and fracture mechanisms.

4. Discussion

4.1 Effects of the Sintering Temperature on the Microstructures and Mechanical Properties

For SiC particle reinforced aluminum alloy composites, the SiC particles are the main strengthening factor. During deformation, if the interfacial bonding between the SiC particles and the matrix is strong, the external applied stress will be transferred from the matrix to the SiC particles. The SiC particles will fracture when the local strain and dislocation density reach the critical value due to the high stress concentration, and thus, increase the strength and ductility of the composites. Otherwise, if the interfacial bonding between the SiC particles and matrix is weak, decohesion will happen before the fracture of the SiC particles at a rather low external applied stress and the corresponding mechanical properties will thus be substantially decreased. Thus, a strong interfacial bonding strength is very important to improve the mechanical properties of the composites.

It can be clearly seen that the sintering temperature has important effects on the microstructures and mechanical properties of the composite. As shown in Fig. 1, increasing the sintering temperature can substantially decrease the number of the pores and increase the interfacial bonding strength. It is generally accepted that solid phase sintering is a thermal activated process, and increasing the sintering temperature can accelerate the diffusion rate, which can effectively decrease the number of pores and improve the interfacial bonding strength. This will inevitably improve the density and mechanical properties of the composites, as shown in Fig. 3, 4, and 6.

Figure 8 also indicates that as the sintering temperature is too low (470 and 520 °C), the interfacial bonding between the particles is only the mechanical bonding since the temperature is too low to obtain enough element diffusion. Thus, spherical aluminum powders can clearly be observed along the tensile fracture surfaces in Fig. 8(a) and (b) (arrowed), which will deteriorate the mechanical properties. As the temperature increases to 570 and 610 °C, the mechanical properties of the composite have been substantially improved due to the fast diffusion rate during the high temperature sintering. As a result of this, ductile fracture is commonly observed, as shown in Fig. 8(c) and (d).

It can be seen from Fig. 6 that the yield strength and the ultimate tensile strength (UTS) increase with the sintering temperature from 470 to 610 °C, while the elongation of the

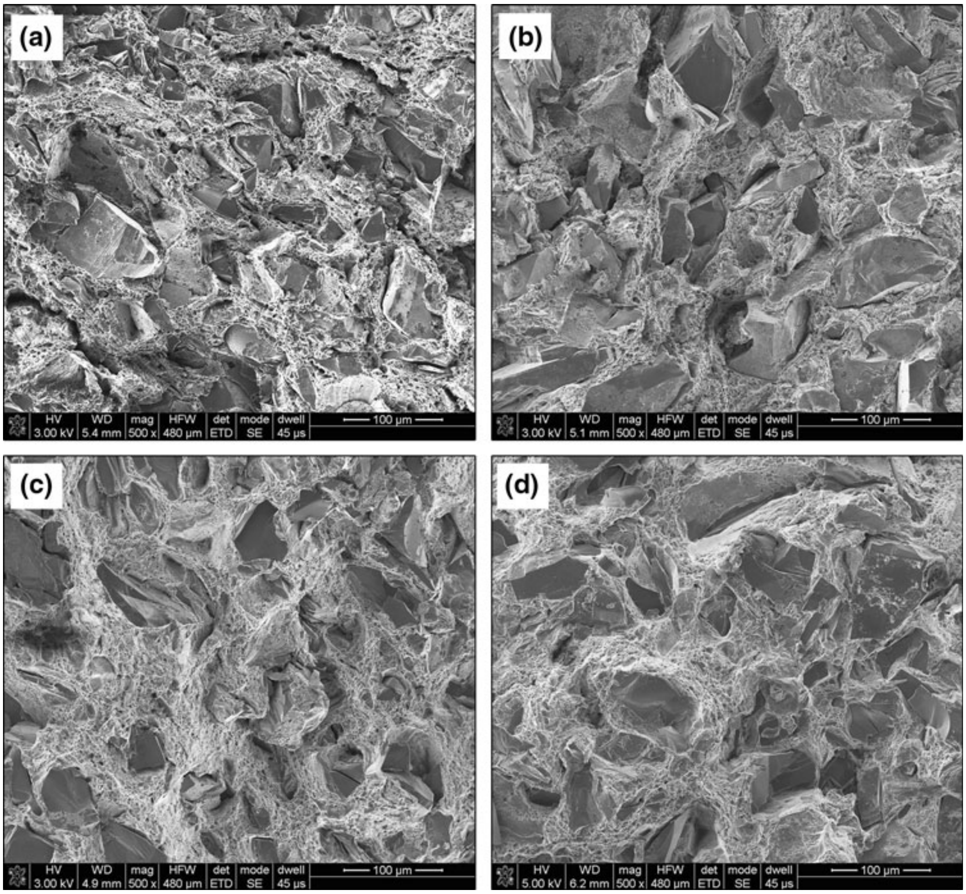

Fig. 9 SEM fracture surfaces of the composite after tensile testing. The specimens were extruded and then aged for 20 h. The sintering temperatures are (a) 470 °C, (b) 520 °C, (c) 570 °C, and (d) 610 °C, respectively

composite increases initially with the sintering temperature only up to 570 °C. The DSC curve (Fig. 7) showed that there is a small exothermic peak around 604 °C. This small exothermic peak is corresponding to the partial melting of intermetallic compounds along the grain boundaries. Sintering at 610 °C results in partial melting of these intermetallic compounds with low melting point and might generate micro-sized cracks in the matrix, which inevitably decreases the elongation of the composites since the cracks can easily propagate once the externally applied stress reaches a critical value.

4.2 Effects of Extrusion on the Microstructures and Mechanical Properties

It is generally believed that the oxide coating on the matrix powder surface is a severe barrier to the interfacial bonding of the particles through inhibiting the element diffusion, and will significantly reduce the ductility and fatigue strength (Ref 22). The combinations of the pressure and temperature during solid phase sintering result in shear between the particles and the fracture of the oxide coating on the Al-Cu particles, and thus improve the interfacial bonding strength. Hot extrusion is a more effective step in breaking up the oxide coating because of the high shear stress generated between particles, resulting in a better adhesion of interfacial bonding (Ref 23). Cöcen and Önel (Ref 6) found that hot extrusion can improve the mechanical properties due to the reduction in the reinforcement particle size, the absence of particle decohesion, and the improvement of the particle-matrix interfacial bond. Rahmani Fard and Akhlaghi (Ref 8) concluded that extrusion can improve the mechanical properties of the composites due to a more uniform distribution of the SiC particles, the break-up of particles agglomerates, the reduction in the porosity of the composites, and the improvement in the particle-matrix interfacial bonding. Tham et al. (Ref 9) also found that reinforcement particles were refined by extrusion.

From Fig. 1 and 2, it can be seen that extrusion substantially decreases the number of the pores, decrease the size of the SiC particles, and improve the interfacial bonding strength between the SiC particles and the matrix. As a result of these, the density and mechanical properties of the composite have been largely improved, as shown in Fig. 3, 4, and 6. Figure 9 shows that the fracture surfaces of the composite after extrusion include mainly ductile dimples of the matrix and particle fracture, indicating an enhanced interfacial bonding strength and mechanical properties.

From Fig. 3 and 4, it can be seen that the evolution trends in the density and hardness with the sintering temperature are slightly different, especially for the composites after extrusion. The density of the composites increased gradually with the sintering temperature, while the hardness of the composites increased dramatically when the sintering temperature increases from 520 to 570 °C. It should be noted that the density of the composites is only related to the porosity, while the hardness depends not only on the porosity, but also on the interfacial bonding strength of the composites (Ref 24). Thus, the density of the composites increased gradually with the sintering temperature since the porosity decreased gradually with the sintering temperature. However, for the composites sintered under the lower temperatures of 470 and 520 °C, the interfacial bonding strength is not as high as the composites sintered under the high temperatures of 570 and 610 °C. The enhanced interfacial bonding strength of the composites sintered at 570 °C help further improve the hardness of the composites.

It is believed that the high shear stress generated during the hot extrusion can also lead to the breakage of large-sized SiC particles and the formation of small-sized particles. In general, large-sized SiC particle will cause high stress concentration around the particles, and the microcracks caused by the large SiC particles will also be large, thus decrease the elongation of the composites. For a given volume fraction of the SiC particles, smaller particles result in a larger interfacial surface area between the matrix and the particles, and thus increase the loading being transferred to the SiC particles, which inevitably improve the mechanical properties of the composite.

4.3 Effect of Aging Time on the Mechanical Properties

It can be seen from Fig. 6 that the strength initially increases with aging time until reaching a maximum value, after which the strength starts to decrease. On the other hand, the elongation of the composite shows an inverse evolution trend with the aging time, initially decreasing with aging time before peak-aged stage, after which the elongation begins to increase with aging time. This phenomenon, which was also observed previously (Ref 16, 25), can be explained by the precipitation process of the Al-Cu matrix during aging. During aging, new precipitates nucleate and grow from the supersaturated matrix (Ref 26, 27). The increase in the strength and decrease in the elongation at the under-aged stage are due to the increase in the volume fraction of the precipitates (Ref 28). A high number of volume-fractioned precipitates will effectively inhibit the movement of the dislocations, generate more geometrically necessary dislocations and reach the critical dislocation density for fracture earlier during deformation, and thus increase the strength and decrease the elongation. Once the excess solute atoms are totally exhausted, the growth stage of the precipitates is terminated. At that time, the strength and the elongation of the composite reach the maximum and minimum values, respectively. Then the volume fraction of the precipitates remains constant, and the size and interspacing of the precipitates increase if the aging time continues to increase, through the growth of the larger precipitates and the dissolving of the smaller precipitates. The increase in the size and interspacing of the precipitates will decrease the strength and increase the elongation.

5. Conclusion

In this article, a SiC particle reinforced Al-Cu alloy composite was fabricated by powder metallurgy method. The effects of the sintering temperature and extrusion on the microstructures and mechanical properties of the composite were studied. Several conclusions have been drawn:

(1) Increasing the sintering temperature can significantly improve the density and mechanical properties of the composites by accelerating the elements diffusion, decreasing the number of pores, and microcrack initiators during deformation, and improving the interfacial bonding strength.
(2) The extrusion can break up the oxide coating on the matrix powder surfaces, decrease the number of pores, refine the particle size, and increase the interfacial bonding strength, and thus, improve the density and mechanical properties of the composite.
(3) At the under-aged stage during aging, the strength increases and the elongation decreases with aging time due to the nucleation and growth of the metastable precipitates in the matrix. At the over-aged stage, the strength decreases and the elongation increases with increasing aging time due to the increase in the size and interspacing of the precipitates with aging time.

Acknowledgments

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