Effects of hot extrusion deformation on microstructure and mechanical properties of the spray-deposition 17 vol.% SiCp/7055Al composite

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Abstract

In this study, the microstructure evolution and mechanical properties under hot extrusion of 17 vol.% SiCp/7055Al composite fabricated through the spray-deposition was investigated by employing the Microscope (SEM), the x-ray diffraction (XRD) and the electron back scattering diffraction (EBSD) under a range of deformation conditions. The extrusion temperature reached 400 °C, 450 °C, 500 °C, and the extrusion ratio was at 9, 13, 20 respectively. As revealed from the results the microstructure evolution and mechanical properties of the composite were affected by the extrusion and the precipitated phase. As indicated from the Optical microscope (OM) and the EBSD analysis, the structure variation of the hot extrusion deformation at 450 °C was mainly the dynamic recovery (DRV). However, the dynamic recrystallization (DRX) was dominating at the extrusion temperature of 500 °C and a ratio of 20. In the DRX process, the dislocation substructures appeared within the grains, to reduce low angle grain boundaries (LAGBs) progressively. The textures with {110} parallel to TD and the recrystallization textures {001} {100} and {124} {211} were characterized. As indicated from the tensile tests, the extrusion eliminated the preparation defects and accommodated the thermal expansion (CTE) mismatch between the matrix and the SiC particles, thereby improving the performance of the composite.

1. Introduction

Metal matrix composites (MMCs), especially the SiC particle-reinforced aluminum matrix composites (PAMCs), turn out to be the structural materials that exhibit high strength/weight ratios, wear resistance, low thermal expansion and elevated temperature strength [1–5]. PAMCs have been suggested as the promising materials in the structures of aerospace, automotive, defense and other engineering sectors [6–8]. On the whole, the preparation of composite materials significantly impacts the cost and performance of PAMCs [9, 10]. The main existing preparation methods of aluminum matrix composites comprise the stirring casting method, the powder metallurgy, the in situ reaction method, the infiltration method, the spray-deposition, etc [11–13]. Compared with additional manufacturing techniques, the spray-deposition method refers to a near-net-shape material preparation technology based on a rapid solidification [14, 15]. In the spray-deposition, the contact time between the matrix and the reinforcing particles is short, thereby leading to the minimal interfacial reaction products, and the composite material is characterized by a uniform particle distribution, a fine grain size (rapid solidification) and a stable performance, etc [15, 16].

However, the addition of ceramic particles can improve the performance of the materials and reduce the overall plastic deformation ability exhibited by the materials [17, 18], which is primarily because the ceramic particles hinder the dislocation and slippage of the matrix during the deformation. In addition, SiCp/Al
composites show several defects (e.g., interface bonding and holes in the preparation), so secondary forming has been commonly used as a vital strengthening method. Plastic processing techniques are the way to deform composite materials into components, and they act as the principal method to improve the microstructure and properties of composite materials [14, 19]. To be specific, the hot extrusion forming technology has been regarded as one of the critical forming technologies. As reported from the existing research, the extrusion deformation effectively eliminates the preparation defects, while causing the directional arrangement of the reinforcing particles to a certain extent, and the distribution is more uniform in the matrix [20–22]. However, the hot extrusion microstructure deformation on spray-deposited SiCp/7055Al composites has been rarely studied.

In this study, the 17 vol.% SiCp/7055Al composite was prepared through spray-deposition and then extruded. The present study aimed to determine the effects of extrusion temperature and the extrusion ratio on the microstructure and mechanical properties exhibited by the composite.

2. Materials and experimental procedures

In this study, the composite containing 17 vol% SiC particles was fabricated through the spray-deposition. The matrix alloy of the investigated SiCp/7055Al composite was Al-Zn-Mg-Cu alloy (7055 alloy), and the alloy chemical composition is listed in Table 1. The diameter of the reinforced SiC particles was 15–20 μm. The spray-deposition parameters included: atomization temperature of 1023–1123 K; atomizer pressure of 0.6–0.8 MPa; the diameter of the deposition disc, reaching 530 mm; the speed of the substrate, reaching 150–250 r min\(^{-1}\); the powder supply pressure of 0.1–0.2 MPa.

A 100t four-column hydraulic press was used to perform the extrusion experiments on the composite materials, and the positive extrusion dies were employed experimentally. The extrusion temperatures reached 400 °C, 450 °C and 500 °C, and the extrusion ratios were 9, 13 and 20, respectively. The samples for the extrusion were fabricated as Φ44 × 30 mm cylinder with the EDMPW2UP wire cutter. Prior to the extrusion, the surface of the samples was polished with an angle grinder to eliminate the surface oxide layer. Subsequently, the boron nitride spray was used to spray the surface of the samples to prevent the composite material from adhering to the mold at high temperatures and high-pressures. The samples were organized in the extrusion die for 2 h at a preset temperature, and the extrusion rate was 0.1 mm s\(^{-1}\).

The Leica DMI 3000 inverted optical microscopy was adopted to observe the structures of the deformed composite, the samples were etched with the modified Keller’s reagent. For the observation of the microstructures and the phase analysis, the SEM (ZEISS-6035) and Bruker D8 ADVANCE XRD were employed. The extruded material was tested by using the EBSD, and the samples measured were sequence prepared through the grinding, the mechanical polishing and the ion thinning in sequence. Furthermore, the Instron-5982 universal electronic tensile testing machine was adopted to perform ambient temperature tensile tests on samples exhibiting a range of extrusion parameters, and the tensile rate reached 0.2 mm min\(^{-1}\). Figure 1 illustrates the dimensions of the tensile specimen. To be specific, the test was repeated 3 times, and the average value was the value of the tests.

| Table 1. The chemical composition of Al7055 (wt%). |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Al              | Cu              | Mg              | Zn              | Si              | Fe              | Cr              | Ti              | Mn              | Zr              |
| Bal.            | 2.0–2.6         | 1.8–2.3         | 7.6–8.4         | ≤0.1            | ≤0.15           | ≤0.04           | ≤0.06           | ≤0.05           | 0.08–0.25       |

Figure 1. The sizes of tensile sample.
3. Results and discussion

3.1. Spray-deposition microstructure

Figure 2 presents the representative optical micrograph (OM) and the EDS spectrum of as-deposited composite. As indicated from the figure, the SiC particles were relatively homogeneous in the Al matrix, whereas an aggregation took place at the grain boundary (figure 2(a)). The SiC particles around the grain boundaries provided the nucleation points at the interface between the particles and the matrix during the solidification, and the grains grew along the surface of the SiC particles. Overall, the SiC particles were not homogeneously distributed since most of them were pushed at grain boundaries during the solidification [23].

The SEM images of the as-deposited composite can be seen in figures 2(b)–(d) present the EDS analysis results of the gray particles (Spectrum 1) and the cotton-shaped white particles (Spectrum 2). As clearly observed from these figures, the gray particles (Spectrum 1) represent the SiC particles, and the cotton-shaped white particles (Spectrum 2) were detected in Al, Cu, Zn and other elements. To be specific, the atomic ratio of Cu in spectrum 2 was significantly higher than the atomic ratio of Cu in the matrix alloy, which proved that the cotton-shaped white particles could be a copper-containing compound.

The atomized droplets were rapidly deposited during the spray-deposition. When the droplets moved the surface of the deposited billet, they were largely solid, liquid and semi-solid particles. During the atomization and the deposition, considerable super-cooled were easy to form. The mentioned super-cooled first formed small particles. After colliding with the droplets, some of them adhered to the surface of the droplets. Besides, some of them were embed into the interior of the droplets and turned out to be the heterogeneous cores [24]. As reported by the existing literature [25, 26], MgZn2 and Al2CuMg compounds existed in the Al-Zn-Mg-Cu alloys. However, according to figure 2(d), the accurate compound in the as-deposited composite could not be determined. For this reason, an in-depth testing and analysis should be conducted, and the compounds will be analyzed and determined more specifically in subsequent section.

3.2. Extrusion microstructure

Figure 3 illustrates the microstructures with extrusion temperatures of 400 °C, 450 °C, and 500 °C and the extrusion ratios of 9, 13 and 20, respectively. In addition, figures 3(a), (c), (e), (g) and (i) presents the microstructures transverse to the extrusion direction, and figures 3(b), (d), (f), (h) and (j) give the microstructures paralleling to the extrusion direction. According to figure 3(a) the fine grains were observed to be more equiaxial and homogeneous, and the SiC
particles were more uniformly distributed, which was due to shear force caused by the breaking phenomenon in a portion of the SiC particles. The distribution was consistent with the as-deposition of the grains and SiC particles, as showed in figure 3(c). Compared with figure 3(a), the grain size increased, considerable discontinuous granular particles appeared at the grain boundaries, and several fine recrystallized grains occurred at the grain boundaries. Figure 3(e) shows that the extruded micro-structures were recrystallized grains. Figures 3(b), (d) and (f) present the micro-structures paralleling to the extrusion direction at the extrusion ratio of 20 and the extrusion temperature distribution of 400 °C, 450 °C and 500 °C, respectively. At the extrusion temperature of 400 °C (figure 3(b)), the grain boundaries flowed in the matrix with the extrusion under a large shear stress, and the grains just elongated mechanically as impacted by the external force. With the increase in the extrusion temperatures (figure 3(d)), the grain boundaries turned out to be clear, and the grains were striped along the extrusion direction, while point-like grains appeared at the grain boundaries. With the extrusion temperature further increasing to 500 °C (figure 3(f)), the microstructures were recrystallized grains.

In figures 3(a), (b), during the extrusion at 400 °C, as impacted by the significant extrusion stress, the flow of the composite matrix caused the redistribution of the SiC particles. In the extrusion, the organization was

\[\text{Figure 3. OM images of f spray-deposition 17 vol}\%\text{ SiCp/7055Al composite under various conditions (Temperature/Extrusion ratio): (a), (b) 400 °C-20; (c), (d) 450 °C-20; (e), (f) 500 °C-20; (g), (h) 500 °C-9; (i), (j) 500 °C-13; (Extrusion Direction, ED; Transverse Direction, TD).}\]
further refined and homogenized as impacted by the external force. The original layer structures were disrupted, and the grains were significantly elongated under the extrusion and friction. After the extrusion, the alloy basically eliminated a small number of voids available in the as-deposition. When extruded at 450 °C (figures 3(c), (d)), with the increase in the temperature, the elements in the matrix diffused faster, and the grain boundaries of the second phase particles tended to be precipitated at the grain boundary and turned out to be clear [27]. The dislocation densities of subgrains and grain boundaries decreased with the increase in the deformation temperature, and the subgrains started to transform from the slender to the equiaxed. Grain boundaries migration increased with the increase in the deformation temperature, thereby up-regulating the rate of DRX [28, 29]. However, the precipitates and SiC particles in the composites exerted a strong nailing effect on the dislocation and subgrain boundaries, thereby seriously hindering the occurrence of recrystallization in the deformation of the matrix alloy. Accordingly, the structural variation of the hot extrusion deformation at 450 °C was primarily DRV. With the increase in the temperature, the recrystallized grain size gradually increased. For the mentioned reason, coarse and partially recrystallized grains could be observed in figure 3(c). When extruded at 500 °C (figures 3(e), (f)), with the increase in the deformation temperature, DRX predominated and the deformed microstructures were transformed into DRX grain microstructures.

Figures 3(e)–(j) shows the microstructure with different extrusion ratios at the extrusion temperature of 500 °C. According to the figures, the grain size of the material first increased and then decreased with the increase in the extrusion ratios. The grain size and aspect ratio in the extrusion direction (ED) and the transverse direction (TD) exhibited a significant anisotropy. At the extrusion ratio of 9 (figures 3(g), (h)), the grains were insignificantly elongated under the small extrusion. On the whole, the extruded composite materials eliminated a small number of voids available in the as-deposited materials [31]. Figure 3(i) and (j) show the microstructures at the extrusion ratio of 13, thereby demonstrating the grain changes were not insignificant compared with the grains at the extrusion ratio of 9. This was mainly because the increase in the extrusion ratio was not obvious, and the grain boundaries migration were not discernible with the increase in the extrusion ratio, and DRX was not formed. However, at the extrusion ratio of 20 (figures 3(e), (f)), the grain boundaries migration increased with the increase in the ratio, thereby increasing the rate of DRX. Thus, the DRX was dominant, and finally the uniform micro-structures were formed.

Figure 4 displays the grain size of the composite at various extrusion temperatures. As the extrusion temperature increased, the average grain size first slightly increased and then decreased, as showed in figures 4(b)–(d). The average grain size of the extruded material was 11.1 μm, 13.2 μm and 3.1 μm at 400 °C, 450 °C, and 500 °C, respectively. However, as showed in figure 4(a), the grain size of the as-deposited was
12.1 μm, which was slightly larger than that at the extrusion temperature of 400 °C. After the extrusion, the grains of the as-deposited composite were broken, and the grain boundaries were extended. Furthermore, the micro-structures were more compact and uniform than those of the as-deposited composites. The grain refinement during the high temperature deformation was attributed to the deformation inducer.

3.3. Phase analysis

Figure 5 presents the XRD analysis of 17 vol% SiCp/7055Al composite under a range of extrusion deformations. As shown from the results, the composition phase of the composite material (e.g., α-Al, SiC, MgZn2, and Al2CuMg) hardly varied under different deformation conditions. The composite material mainly contained MgZn2, while the Al2CuMg phase was not clear. However, the studies [32, 33] reported that at 400 °C and 450 °C, the primary (MgZn2) phase would be completely transformed into Al2CuMg. In this study, MgZn2 phase was not overall transformed into Al2CuMg phase at the extrusion temperature of 400 °C and 450 °C, which was attributed to the short extrusion time.

According to the EDS analysis in figure 6 and 2, the MgZn2 phase contains a large amount of Cu and Al. This was explained as Cu and Al atoms entered the MgZn2 phase to replace part of the Zn atoms, and finally they formed the Mg (Zn, Cu, Al)2 structures phase [34, 35]. Figures 6(b)–(g) give elemental surface scanning maps of Al, Zn, Cu, Mg, Si and C in the composite. As indicated from the mentioned maps, there was the segregation of Mg and Cu, in which Cu segregation took place around SiC particles, while Mg was mainly concentrated at the grain boundaries and around SiC particles. As impacted by the characteristics of spray forming technology, there was almost no macro-segregation in the prepared material, and the degree of micro-segregation was significantly low [32]. For this reason, the place where Mg and Cu elements gathered was the second phase produced during the deposition, i.e., MgZn2, Al2CuMg and MgZn2 extended phase (Al2Cu).

In the preparation of spray-deposition of composite materials, MgZn2 and a small amount of Al2CuMg phase were formed, in which the MgZn2 phase would undergo the solid solution and transformation at 430 °C–450 °C [32, 36]. In the present study, MgZn2 constantly existed in the composite materials since the material was shortly exposed to high temperatures. As revealed from the concentration of Mg and Cu elements around SiC particles and at grain boundaries, the micro-segregation of alloying elements remained at the grain boundaries.

3.4. Mechanical properties

Figure 7 presents the tensile strength and elongation of the pray-deposition 17 vol% SiCp/7055Al composite under a range of extrusion deformation conditions. As indicated from figure 7(a), the strength and elongation of the composite materials at the extrusion temperature of 500 °C and the extrusion ratio of 9, 13 and 20, respectively. At the extrusion ratio of 9, the tensile strength was improved from 329 Mpa in the deposited state
to 369 Mpa, and the elongation was reduced from 5.8% to 3.3%. At the extrusion ratio of 13, the tensile strength and elongation were 463 Mpa and 7.7%, respectively. However, the tensile strength and elongation were basically unchanged at the extrusion ratio was 20 compared with the extrusion ratio was 13.

The tensile properties of the extruded composites were better than those of the deposited composites (figure 7(a)), which was mostly because the extrusion eliminated the preparation defects and accommodated the CTE mismatch between the matrix and the SiC particles [37]. The dynamic recovery eliminates the residual stress and work hardening, which greatly improves the performance of the composite extruded at 500 °C and a ratio of 13. At the extrusion ratio of 20, DRX occurs and the grains were refined (figure 3), which further increases the elongation of the composites. The SiC particles were broken at high extrusion ratio, and the interface between SiC particles and matrix produces defects, which lead to the failure of the composite.
Generally, when the interface bonding of composite materials is too weak or too strong, the mechanical properties of the composite are unsatisfactory [18].

In figure 7(b), the strength and elongation of the composite materials at the extrusion ratio of 20 and the extrusion temperature of 400 °C, 450 °C, 500 °C, respectively. The tensile strength increased from 329 MPa to 409 MPa and the elongation increased from 5.8% to 9.2% at the extrusion temperature of 400 °C. While, the tensile strength and elongation of the composites decrease first and then grow with the increase in the extrusion temperature. The lower performance of the composite extruded at 450 °C was due to the DRV increase the grain size (figure 3), and the second phase particles were precipitated at the grain boundaries. The second phase particles further hinder the movement of dislocations and reduce the elongation.

Figure 8 presents the tensile fracture morphology exhibited the composite. As indicated from the figure, considerable SiC particles were deboned in the fracture of the composite materials in the as-deposited composite, and no fracture dimples were reported. With the increase in the extrusion ratio, the debonding phenomenon of SiC particles and the matrix tended to decrease, and the precipitation phase particles tended to
increase. At the extrusion temperature of 500 °C and a ratio of 20, there were numerous precipitated particles and dimples in the fractures. With the increased in the extrusion ratio, the DRX turned out to be ever more complete, and the plasticity of the composite increased. In the tensile deformation, the second phase particles would further hinder the dislocation movement, and with the increased in the deformation, the dislocations around the particles would form micro-cracks and finally form dimples. However, the elongation of the composite was low in the as-deposited and the extruded, and the fracture of the composite was a brittle fracture.

Figure 9. EBSD microstructure and misorientation angle distribution maps of SiCp/7055Al with extrusion ratio of 20 at different temperatures. (a), (b) 400 °C, (c), (d) 450 °C, (e), (f) 500 °C.
3.5. Grain structure evolution Study by EBSD

Figure 9 presents the EBSD microstructures and misorientation angle distribution maps of the composite with a ratio of 20 at different temperatures, in which different colors in the figure represent the different orientations of the grains. The gray regions represent the SiC particles, not resolved during the EBSD scanning. In figures 9(a)–(d), some sub-grains and recrystallized grains were observed, whereas the grains were primarily equiaxed with an amount fraction of LAGBs. With the increase in the extrusion temperature, the recrystallized grains increased, and the content of high-angle grain boundaries (HAGBs) increased, as showed in figure 9(e) and (f). As opposed to the mentioned, the result was inconsistent with the [38, 39], reporting that the content of LAGBs increased with the increase in the deformation. However, in theirs study, an increases proportion of HAGB was found at the extrusion temperature of 500 °C. This might be explained as the subgrains would be transited at the elevated temperature with considerable strain accumulated during the extruding deformation. Moreover, the DRX occurred, while the DRX produced microstructures were unstable at the elevated temperature. In the DRX process, relatively stable HAGBs were formed, while dislocation substructures might appear inside the grains, as an attempt to reduce LAGBs gradually.

Figure 10 presents the \{111\}, \{110\}, and \{100\} pole figure maps of the extruded composite at different temperatures, which illustrates the texture components and relative intensity. The textures with \{110\} \{111\} parallel to TD were observed in the composite extruded at 400 °C and 450 °C. The main textures of the composite extruded at 500 °C were recrystallization textures \{001\} \{100\} and \{124\} \{211\}, (figure 10(c)). Recrystallization refers to a work of nucleation and growth, which could be affected by thermal deformation conditions and the orientation of deformed grains. Accordingly, the recrystallized textures tended to be formed in the recrystallized material.
Figure 11 displays the kernel average misorientation (KAM) and the variations in mean KAM of the composite at a range of extrusion temperatures. The mean values of KAM could take account of the uniformity of plastic deformation. The higher the mean KAM, the greater the plastic deformation or defect density of the material would be. According to figure 11(b), KAM values of the composite at the as-deposition and extruded at 500 °C were lower than those at the extrusion temperature of 400 °C and 450 °C. The mean KAM value decreased from 0.77 at the extrusion temperature of 400 °C and 450 °C to 0.39 at the extrusion temperature of 500 °C, which was primarily due to the increase in grain boundaries and the fragmentation of SiC particles at the extrusion temperature of 400 °C and 450 °C. With the extrusion temperature increasing to 500 °C, the generation of DRX led to the reduction of dislocation and then decrease the mean KAM values.

4. Conclusions

In this study, the microstructures and mechanical properties of the spray-deposition 17 vol.% SiCp/7055Al composite during the extrusion were investigated. The following conclusions can be drawn:

(1) After the extrusion, the typical elongated grains with a small aspect ratio were identified in the microstructures. The second phase particles of the composite extruded at 450 °C tended to be precipitated at the grain boundaries. The structural change of hot extrusion deformation at 450 °C was largely the DRV. However, DRX dominates at the extrusion temperature of 500 °C and a ratio of 20.

(2) The composition phase of the composite hardly varied as compared with the as-deposited composite, mainly including \( \alpha \)-Al, SiC, MgZn2, and Al2CuMg, under different extrusion conditions. However, the micro-segregation of the alloying elements remained at grain boundaries.
(3) Brittle fracture acted as the main fracture mechanism of the extruded and deposited samples. Moreover, the extrusion eliminated preparation defects and accommodation the CTE mismatch between the matrix and the SiC particles. The dynamic recovery eliminated the residual stress and work hardening, thereby significantly improving the performance of the composite. To be specific, the tensile strength and elongation extruded of the composite at 500 °C and a ratio of 13 reached 459 Mpa and 7.9%, respectively.

(4) With the increased in the extrusion temperature, the content of HAGBs increased. In the DRX process, a relatively stable HAGBs were formed, while the dislocation substructures might appear inside the grains, as an attempt to reduce LAGBs gradually.

(5) The textures with \{110\} \{111\} paralling to TD were observed in the composite extruded at 400 °C and 450 °C. The main textures of the composites extruded at 500 °C were recrystallization textures \{001\} \{100\} and \{124\} \{211\}. The generation of DRX led to the reduction of dislocation and then decreased the mean KAM value of the composite extruded at 500 °C and a ratio of 20.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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