



Microstructure, texture and mechanical properties of Mg-2.10Zn-0.58Ca-1.02Zr-10.34SiC_p (wt%) composites after being extruded at different speeds

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ABSTRACT

The Mg-2.10Zn-0.58Ca-1.02Zr-10.34SiC_p (wt%, SiC_p/ZX20K) composites were fabricated by stir casting and hot extruded at 400 °C with extrusion speed of 5 mm/s and 10 mm/s, respectively. The SiC particles, which initially exhibited a necklace-type distribution at the grain boundaries, transformed into a band-type distribution after hot extrusion. The SiC_p/ZX20K composites underwent complete recrystallization, resulting in a pronounced bimodal microstructure consisting of coarse secondary recrystallized grains in the SiC-poor region and fine grains in the SiC-rich regions. A basal plane fiber texture was achieved, with the {0001} plane oriented parallel to the transverse direction (TD). Moreover, grains with their <2-1-10> orientation parallel to the extrusion direction (ED) experienced preferred growth because those grains possessed characteristics such as larger initial size, lower storage energy, and a larger orientation difference from neighboring grains. The SiC_p/ZX20K composites extruded at 5 mm/s exhibited a yield strength (YS) of 218 MPa, with a decent elongation to failure (EL) of 4.8 % and an ultimate tensile strength (UTS) of 294 MPa which was 2.3 times greater than that of the as-cast state. Increasing the extrusion speed resulted in better overall mechanical properties and reduced the anisotropy in mechanical properties, due to the combined effects of texture, particle orientation and particle distribution.

1. Introduction

As the lightest structural materials, magnesium alloys have not gained widespread application due to their low strength, low elastic modulus and weak high-temperature stability. Fabricating particle reinforced metal matrix composites (PMMCs) by adding ceramic particles into magnesium alloys is an effective way to overcome these shortcomings [1,2]. Common processing techniques for PMMCs include powder metallurgy, stir casting, spray forming, in-situ synthesis, etc. [3]. Among these, stir casting is particularly advantageous for large-scale industrial production and allows for the fabrication of PMMCs with a high volume fraction of particles, making it a relatively economical preparation process. This method has been widely used in

the fabrication of SiC_p/ZM6, Mg/SiC, and Y2O3_p/ZG21 [4–6].

However, fabricating PMMCs by stir casting inevitably leads to porosities and other casting defects [7]. This processing technique often results in uneven particle distribution and clusters in PMMCs, which may lead to premature failure because of stress concentration [8]. The uneven distribution of particles can also negatively impact the wear resistance of PMMCs. Jolokhani et al. [9] found that with the uniform distribution of SiC particles, the wear resistance of Mg-0.5Zn-0.5Zr/SiC composites was improved. Hot extrusion can induce significant deformation in PMMCs, effectively eliminating casting defects and heterogeneous particle distribution. Meanwhile, hot extrusion can also result in dynamic recrystallization and refine grains and thereby enhancing mechanical properties [10].

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The microstructure, particle distribution and mechanical properties of as-extruded composites are influenced by extrusion temperature and extrusion ratio, which have been extensively investigated [11–14]. In contrast, adjusting extrusion speed provides a more flexible means of controlling the microstructure and mechanical properties of PMMCs in industrial production compared to adjusting extrusion temperature and extrusion ratio. Zhou et al. found that increasing extrusion speed reduced the yield strength in the extrusion direction (ED) during the hot extrusion process of MgAlCoCrFeNi alloys but improved the plasticity of the alloys [15]. The findings of Niu et al.'s [16] on SiC_p/AZ61 composites are also consistent with this phenomenon. Moreover, particle size plays an important role in determining the uniformity of particle distribution after extrusion. For example, Nie [17] and Wang [18] used similar process parameters to extrude SiC_p/AZ91 composites with reinforcement particle sizes of 60 nm and 10 μ m, respectively. The composites with larger particles exhibited more uniform distribution after extrusion.

However, current systematic studies on the effects of extrusion speed on the microstructure and mechanical properties of PMMCs have primarily focused on slow extrusion speeds, often below 2 mm/s. This limitation means that slow extrusion speeds have little effect on improving the uneven distribution of particles in composites, and their low processing efficiency limits practical industrial production [19,20]. Moreover, studies on the mechanical properties of as-extruded PMMCs have mainly focused on room temperature mechanical properties when loaded along the ED, while less attention has been paid to room temperature mechanical properties in the transverse direction (TD) [10]. However, given that many structural applications require load-bearing capabilities in multiple directions, understanding the anisotropic mechanical properties of PMMCs is crucial.

Furthermore, due to the significant difference in hardness between reinforcement particles and the magnesium alloy matrix, it is difficult to analyze the crystallography orientation using electron back-scatter diffraction (EBSD). Consequently, there is a lack of systematic analysis regarding the influence of particles on adjacent grains, which is crucial for understanding the performance and microstructure of PMMCs [16, 17].

In this study, a highly ductile Mg-Zn-Ca alloy was selected as the matrix [21,22] and SiC particles, which possess a significantly higher elastic modulus than the magnesium alloy, were chosen as reinforcement particles with an average particle size of 2 μ m. SiC_p/Mg-Zn-Ca-Zr composites ingot was prepared and hot extruded at two different extrusion speeds of 5 mm/s and 10 mm/s, which are much higher than speeds previously studied. This novel approach aims to evaluate the effect of higher extrusion speed on particle distribution and the resulting microstructure and mechanical properties of SiC_p/Mg-Zn-Ca-Zr composites. Moreover, a particular focus was placed on systematically investigating tensile properties in both ED and TD, providing new insights into the anisotropic behavior and highlighting the impact of extrusion speed on mitigating anisotropy in mechanical properties. Characterization methods such as EBSD were used to comprehensively investigate the effect of extrusion speed on particle distribution, microstructure, and mechanical properties of SiC_p/Mg-Zn-Ca-Zr composites. The influence of the reinforcing particles on surrounding grains and mechanical properties was also analyzed. This study aims to contribute to a deeper understanding of the mechanisms through which extrusion speed influences the properties of PMMCs and to offer practical insights for optimizing extrusion conditions in industrial production.

2. Experimental procedure

In this study, the SiC micro-particles with an average diameter of about 2 μ m and Mg-Zn-Ca-Zr alloy were chosen as the reinforcement and matrix, respectively. The composites ingot with a diameter of 260 mm was prepared using the stir casting process. The actual compositions of

the composites measured by inductively coupled plasma atomic emission spectrometry (ICP-AES) were determined to be Mg-2.10Zn-0.58Ca-1.02Zr-10.34SiC (named as SiC_p/ZX20K, wt%). The ingot was machined into two rods, each with a diameter of 120 mm and a length of 320 mm for the extrusion process, and then homogenized at 430 °C for 12 h. The rods were heated to 400 °C and maintained at this temperature for 3 h to ensure uniform and stable temperatures for both the rods and the die. Afterward, the rods were extruded to form extrudates with a width of 50 mm and a thickness of 12 mm, using an extrusion ratio of 19:1 and extrusion speeds of 5 mm/s and 10 mm/s (named as E5 and E10), respectively. The two extrudates were cooled by forced air after extrusion. The three directions of the extrudates named ED, TD, and normal direction (ND) are shown in Fig. 1.

After grinding with abrasive papers with nos. 1000, 2000, 3000, 5000 and polishing by 1.5 μ m polishing paste, the microstructure of SiC_p/ZX20K composites was observed using optical microscope (OM) and scanning electron microscope (SEM, Philips XL30ESEM-FEG), semi-quantitative analysis of the composition of each phase, as well as element distribution was conducted with an energy dispersive spectrometer (EDS) equipped with the SEM. The samples were analyzed by X-ray diffraction (XRD) using Cu K α radiation at a scanning speed of 4 °/min to determine phase composition. After mechanical polishing and ion etching, the crystallographic orientation information before and after hot extrusion was obtained by EBSD (Quattro 5), with a step size of 1 μ m, and the EBSD data was analyzed by the OIM software. Transmission electron microscope (TEM, FEI Talos F200X) was used to investigate the microstructure of SiC_p/ZX20K composites which had been ion milled by Gatan 690. The observation surface used for the microstructure characterization before extrusion was located at the center of the rods and perpendicular to their axis. After hot extrusion, the microstructures of E5 and E10 were observed on the plane of ED-TD located at one-third of the thickness from the original surface.

The window technique adopted by Wang et al. [23] was used to describe the distribution of SiC particles. The SEM images shown in Fig. 1(b-c), depicting specimens extruded at different speeds, were divided into 10 subsets perpendicular to the ED or 15 subsets perpendicular to the TD, respectively. The frequency of each area fraction was calculated by dividing the occurrence times of the area fraction by the total number of subsets. The distribution of SiC particles in each direction was determined using three randomly selected SEM images taken at 200x magnification, and the ImageJ software was used to calculate the area fraction of SiC particles in each subset.

Due to the size limitations of the as-extruded SiC_p/ZX20K composites, tensile tests at room temperature (RT) of ingots were conducted using rod-shaped specimens with a parallel segment length of 30 mm and a diameter of 5 mm. Dog-bone-shaped specimens were used to measure the tensile properties of E5 and E10. The parallel segment length of the dog-bone-shaped specimens was 20 mm, with a width of 4 mm and a thickness of 2 mm. The ultimate tensile strength (UTS), yield strength (YS), and elongation (EL) after fracture were obtained according to the standard GB/T 228.1–2010. Cylindrical specimens with a diameter of 8 mm and a height of 10 mm were used to test the RT ultimate compressive strength (UCS) based on the standard GB/T 7314–2017. Tensile tests were loaded along the ED and TD, respectively, while compression tests were loaded along the ED, TD, and ND. The Vickers hardness of E5 and E10 was measured on the plane of ED-TD with a load of 500 g and a holding time of 10 s.

3. Results

3.1. The microstructure of as-cast SiC_p/ZX20K composites

The microstructure of the as-cast SiC_p/ZX20K composites is shown in Fig. 2. Fig. 2(a) reveals a coarse dendritic structure consisting of α -Mg grains, with SiC particles arranged in a necklace-type pattern along the grain boundaries. This distribution suggested that the SiC particles were

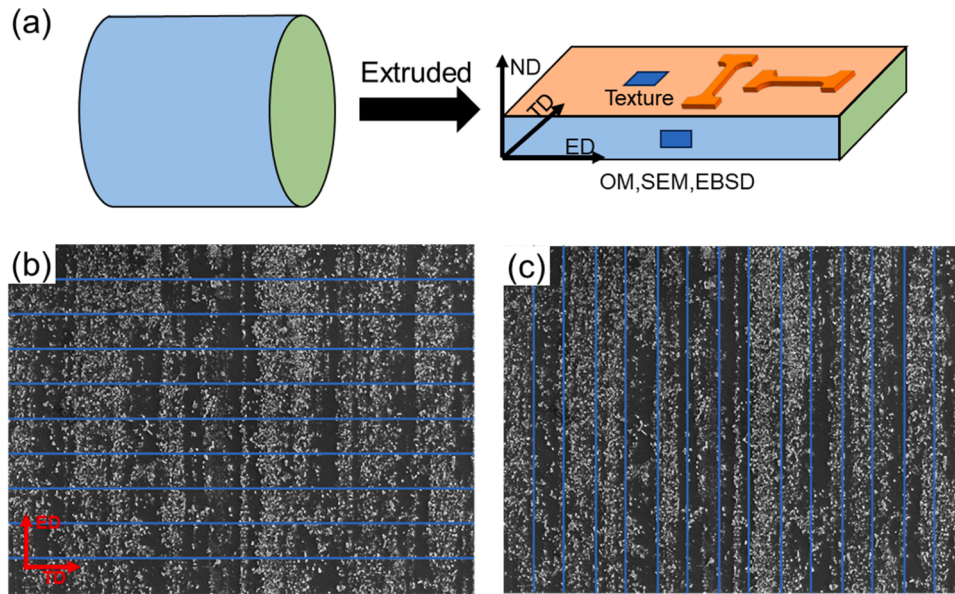


Fig. 1. (a) Schematic diagram for extrusion processes and the microstructure, mechanical properties characterization of E5 and E10; (b-c) different ways to divide subsets in SEM images perpendicular to the ED (b) and the TD (c).

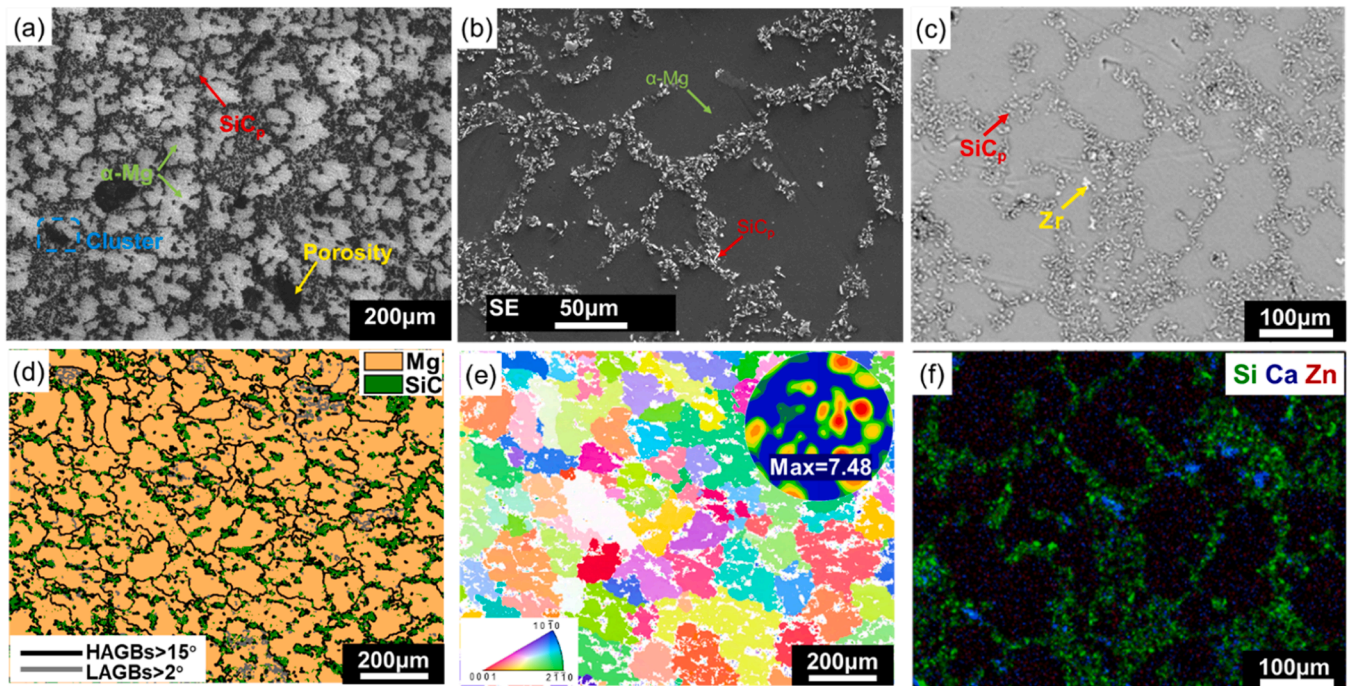


Fig. 2. Microstructure of as-cast $\text{SiC}_p/\text{ZX20K}$ composites. (a) OM image, (b, c) SEM images detected by (b) secondary electron (SE) and (c) back scattered electron (BSE), (d) phase map, (e) inverse pole figure (IPF) map and (f) EDS image of (c).

displaced to the grain boundaries by the liquid-solid interface as $\text{SiC}_p/\text{ZX20K}$ composites solidified. This can be attributed to the differences in thermal conductivity and thermal diffusivity between the particles and the matrix. However, as shown in Fig. 2(b), despite evident clustering in certain areas, the SiC particles along the grain boundaries remained discretely distributed, providing the potential to achieve uniform particle distribution through subsequent thermal deformation processing. During the stirring process, air was entrained into the composites melt. Additionally, due to the presence of van der Waals force, air was adsorbed on the surface of the SiC particles before being mixed with the matrix [24]. These two factors led to the presence of porosities in the

$\text{SiC}_p/\text{ZX20K}$ composites, which is a drawback of the stir casting process. Furthermore, there were some segregations of Zr, with a content of 1.02 wt% in the $\text{SiC}_p/\text{ZX20K}$ composites, as indicated by the white areas in Fig. 2(c).

The phase map obtained by EBSD in Fig. 2(d) further confirmed that the microstructure of the as-cast $\text{SiC}_p/\text{ZX20K}$ exhibited a dendritic structure, with most of the SiC particles (marked in green) distributed along the grain boundaries. The trace amounts of low-angle grain boundaries (LAGBs) in the dendrites indicated the absence of substructure or low thermal stress. The IPF map shown in Fig. 2(e) provides the crystallographic orientation information of the Mg phase in the as-cast

SiC_p/ZX20K composites. The uniform color distribution of the grains indicated that the as-cast SiC_p/ZX20K composites exhibited a random texture, which was confirmed by the (0001) pole figure (PF) in the upper right corner of Fig. 2(e). In the subsequent sections, the white areas in Fig. 2(f) represent the positions of SiC particles, originally marked in green in Fig. 2(d). Fig. 2(f) presents the EDS surface scanning results of Fig. 2(c), where green, blue, and red indicate the presence of Si, Ca, and Zn, respectively. Discrete Ca-rich phases were observed attached around the SiC particles. However, the eutectic phase of the SiC_p/ZX20K composites didn't form a continuous network pattern at grain boundaries like Mg-1.95Zn-0.75Ca reported by Luo et al. [21]. The main reason for this phenomenon was the interface between the particle and matrix, where the eutectic phase precipitated on particle surface, reducing the interfacial energy of the system [25]. It is evident that the addition of reinforcement particles significantly altered the morphology, size, and distribution of eutectic structures in the Mg-Zn-Ca alloy, consistent with the findings reported by Wang et al. [26].

By combining the EDS, XRD and phase diagram calculation results, the composition of the eutectic phase in the SiC_p/ZX20K composites was analyzed. Three representative areas were selected in Fig. 3(a) for composition analysis using EDS, with the results shown in Fig. 3(b–d). Area 1 corresponded to the Zr-rich phase, Area 2 represented the SiC particles, and Area 3 was the Ca-rich phase. The content of Zn in Area 3 was even lower than the other two areas, and the Zn element distribution in Fig. 2(d) also indicated that there was no obvious enrichment in the SiC/ZX20K composites. Combining the XRD results in Fig. 3(e) and the Mg-rich corner of the Mg-Zn-Ca ternary phase diagram calculated by Pandat software in Fig. 3(f), it was concluded that the Ca-rich phase was Mg₂Ca.

3.2. The microstructure of as-extruded SiC_p/ZX20K composites

The SiC particles distribution of E5 and E10 after extrusion is shown in Fig. 4. The necklace-type particle distribution in the as-cast SiC_p/ZX20K composites transformed into a band-type distribution parallel to the ED, as shown in Fig. 4(a–b, d–e). SiC-rich regions and SiC-poor

regions alternate along the TD, indicating an uneven particle distribution. Fig. 4(c,f) illustrates that the SiC particles underwent rotation during the extrusion process. As a result, the long axis of these particles shifted from a random orientation to an alignment in which the particles' long axis became parallel to the ED. This phenomenon was more pronounced for particles with a larger aspect ratio. Due to their significantly higher hardness compared to the magnesium alloy matrix, the SiC particles could not deform in coordination with the matrix, which led to cracks forming in some particles during deformation, as indicated by the red arrows. The direction of crack propagation was random, and in some cases, cracks fully propagated, splitting the original particle into two separate parts. Although the SiC particles appeared relatively concentrated in the band-type distribution shown in Fig. 4(a,d), the SEM images in Fig. 4(c,f) demonstrate that most of the SiC particles were separated by the easily deformable matrix, and the particle segregation phenomenon was significantly mitigated.

EBSD was used to analyze the microstructure and crystallographic orientation of the SiC_p/ZX20K composites before and after extrusion, as shown in Fig. 5. As seen in Fig. 5(a, b), the texture of the as-homogenized SiC_p/ZX20K composites remained random with coarse grains. In Fig. 5(a), many grains contain {10–12} tensile twins, whose morphology is depicted in Fig. 5(c) through TEM characterization. Due to magnesium's thermal expansion coefficient of $27.9 \times 10^{-6} \text{ K}^{-1}$, thermal stress is generated during both the heating and cooling processes of homogenization [27]. The SiC particles, which have a thermal expansion coefficient significantly different from that of the matrix, contributed to stress concentration and also restricted dislocation slip [28]. Therefore, {10–12} tensile twinning, which has a relatively low activation energy, became an important mechanism for releasing thermal stress. As illustrated in Fig. 5(d–e), dynamic recrystallization occurred during hot extrusion, leading to a reduction in average grain size. However, the microstructure exhibited two distinct grain morphologies: fine grains with an average grain size of approximately 9 μm , and coarse grains with an average grain size of approximately 55 μm . The fine recrystallized grains were found around and along the SiC-rich regions, with some LAGBs present within these grains. In contrast, the coarse grains were

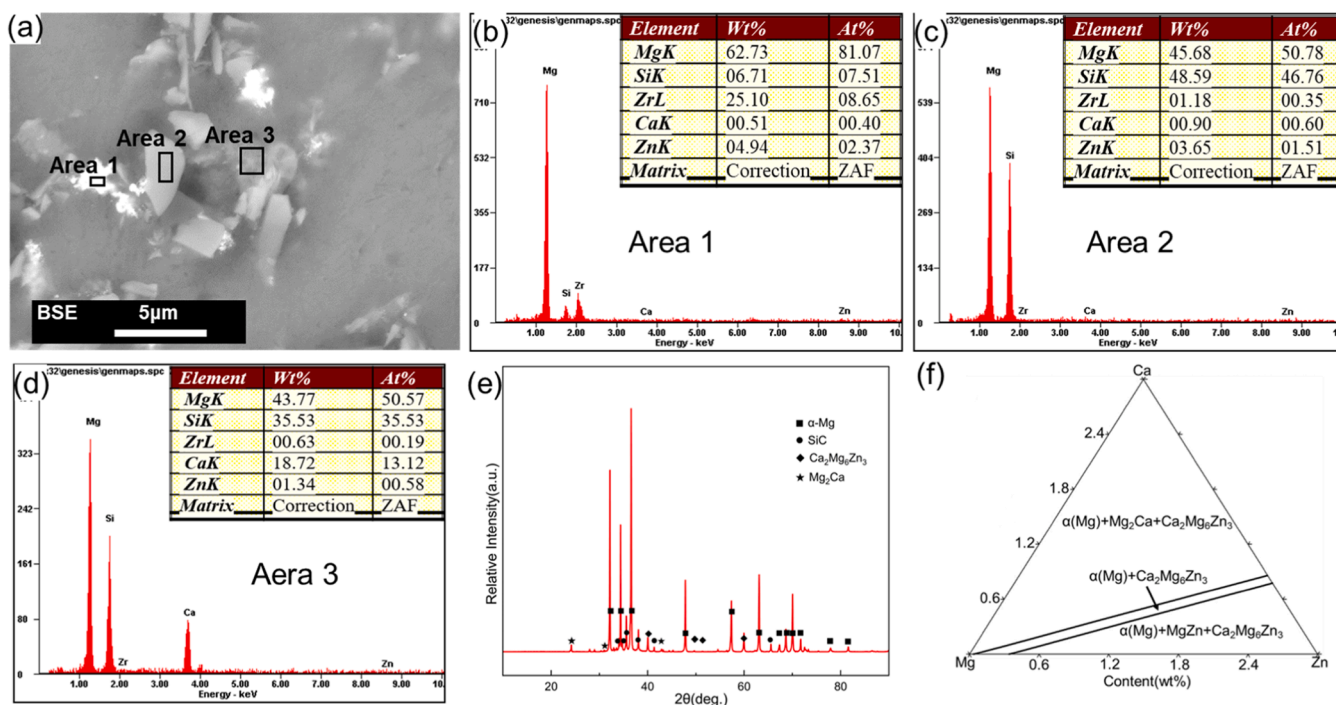


Fig. 3. Phase analysis of the as-cast SiC_p/ZX20K. (a) microstructure by SEM, (b–d) EDS maps of (a), (e) X-ray diffraction result, (f) Mg-rich corner in ternary phase diagram about Mg-Zn-Ca alloy.

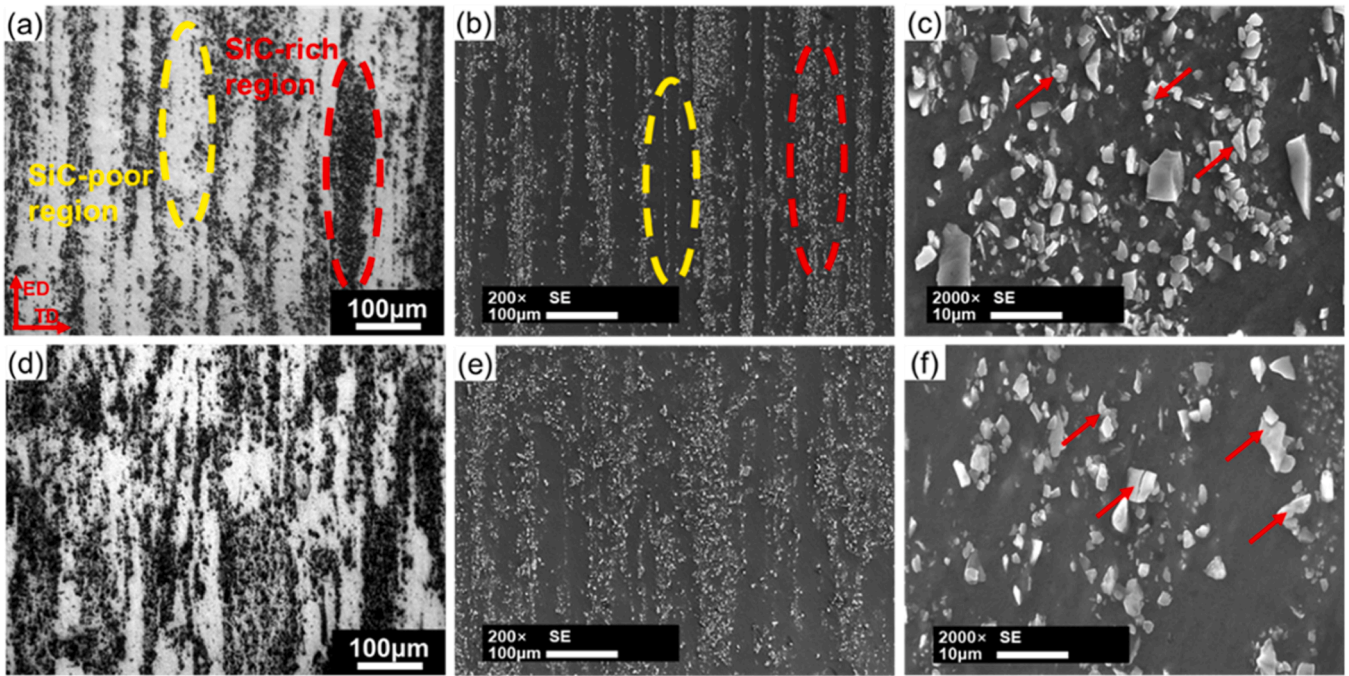


Fig. 4. Particles distribution of as-extruded SiC_p/ZX20K at different extrusion speeds. (a-c) E5, (d-f) E10, (a, d) OM, (b-c, e-f) SEM images.

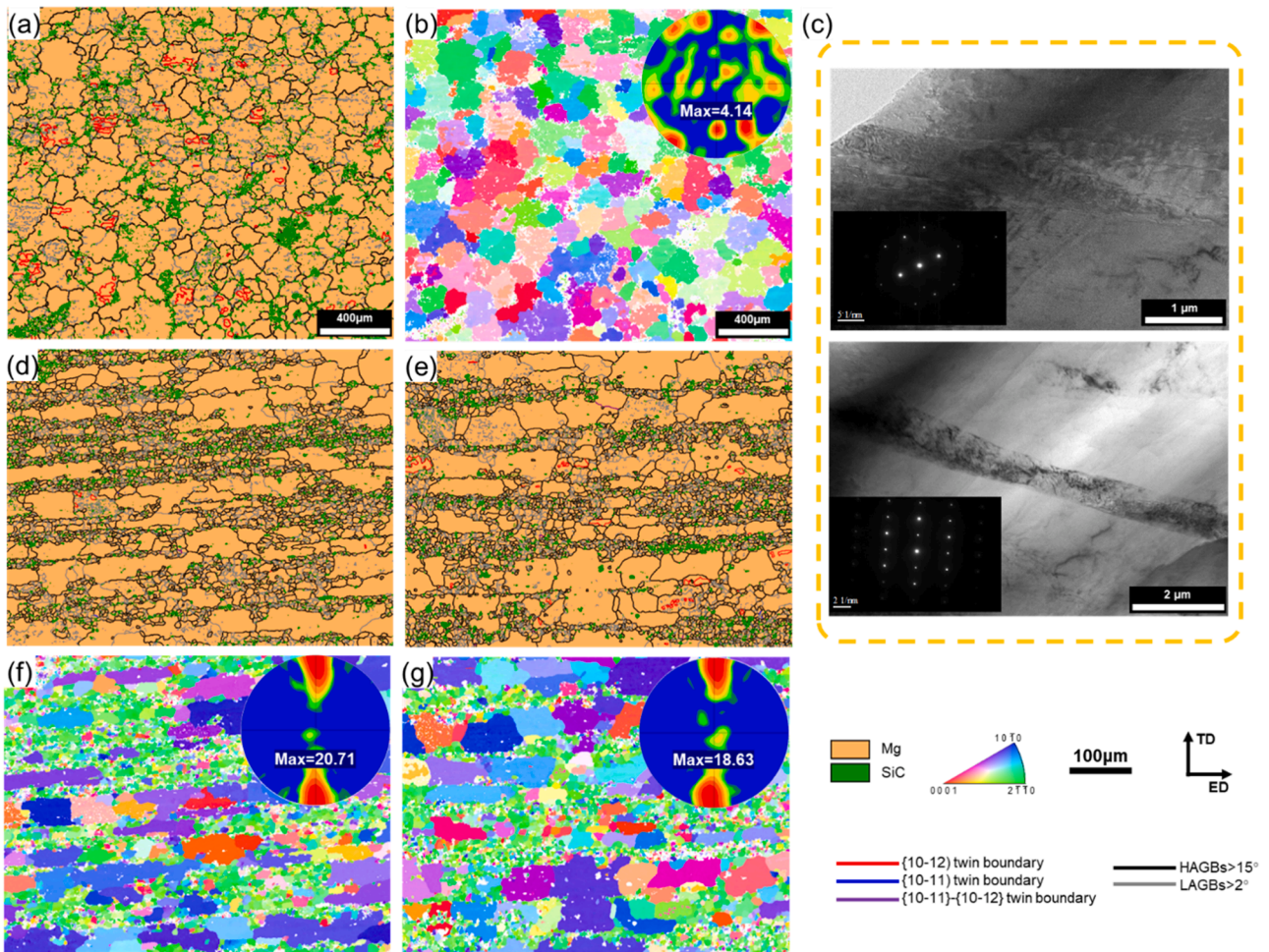


Fig. 5. Microstructures of (a-c) As-homogenized, (d, f) E5 and (e, g) E10 observed by (a, d, e) phase maps, (b, f, g) IPF maps and (c) TEM.

present in the SiC-poor regions and elongated along the ED. Comparing the IPF maps and (0001) PFs in Fig. 5(b,f,g), the texture evolved into a fiber texture with the {0001} plane parallel to the TD after extrusion, which is the typical texture formed after the extrusion of magnesium alloys such as Mg-Sn-Si-Ca, Mg-Zn-Ca, and Mg-Al-Zn [29–31]. The texture intensity of the two specimens extruded at different speed was similar. However, grains with their c-axis parallel to the ND represented a higher proportion in the E10, as shown by the (0001) PF in Fig. 5(f–g).

3.3. Mechanical properties

Fig. 6 shows the mechanical properties of the SiC_p/ZX20K composites loaded in different directions at room temperature both before and after extrusion. The tensile properties are presented in Fig. 6(a), and the label E5ED represents the specimens that reflect the microstructure and mechanical properties of E5 in the ED. Similarly, E5TD, E10ED, E10TD are labeled following the same rule. The YS and UTS of the as-cast SiC_p/ZX20K composites were 92 MPa and 125 MPa, respectively, while the EL was only 1.0 %. Compared to the room temperature tensile properties of a similar matrix alloy (Mg-2Zn-0.5Ca-0.5Zr) studied by Tong et al. [32] in the as-cast state (YS=93 MPa, UTS=117 MPa, EL=4.1 %), the addition of SiC particles reduced the EL of SiC_p/ZX20K composites significantly, indicating that particle clusters and internal casting defects contributed to a reduction in plasticity. After extrusion, the tensile properties of the SiC_p/ZX20K composites improved significantly, with enhanced plasticity. Both the YS and UTS of E5 and E10 loaded along the ED and TD were notably higher than those of as-cast state, and even significantly higher than those of as-extruded Mg-Zn-Ca alloys [33–35], with the maximum increase in YS reaching 137 %. The effect of extrusion speed on the room temperature tensile properties varied depending on the loading direction. When the loading direction was parallel to the

ED, the YS and UTS of E5 were slightly higher than those of E10, with a lower EL. This is consistent with the findings of Liu et al. [36], who reported that increasing the extrusion speed reduced the YS in the ED while increasing the EL. However, when loaded parallel to the TD, the YS and UTS of E5 were significantly lower than those of E10, with a similarly lower EL. This suggested that increasing the extrusion speed could comprehensively improve the room temperature tensile properties of the composites in the TD. The room temperature compressive properties are shown in Fig. 6(b), where the label UCS_{ED} represents the UCS loaded parallel to the ED, and UCS_{TD}, UCS_{ND} are labeled similarly. As shown in Fig. 6(b), the UCS along different loading directions were significantly improved after extrusion compared to the as-cast SiC_p/ZX20K composites, with the UCS of E5 and E10 being similar. When compressed parallel to the ED, the UCS increased by nearly 200 MPa from 324 MPa to 520 MPa (E10). There was a noticeable anisotropy in UCS, with UCS_{ED} > UCS_{TD} > UCS_{ND}. By adding SiC reinforcements to the Mg-Zn-Ca-Zr alloy, the Vickers hardness of the SiC_p/ZX20K composites increased significantly from 53.4 HV [32] to 76.4 HV. After extrusion, the Vickers hardness further increased by more than 12 HV, as shown in Fig. 6(c).

4. Discussion

4.1. Effects of hot extrusion on the distribution of SiC particles

The effect of extrusion speed on the distribution of SiC particles was evaluated using the window technique. The statistical results are shown in Fig. 7(a–d). It can be seen in Fig. 7(a) that the area fraction span of the SiC particles distribution in E5ED ranged from 13 % to 19 %. However, the frequency of the average area fraction was not the peak one. Instead, there was a local minimum value within this interval, resulting in a

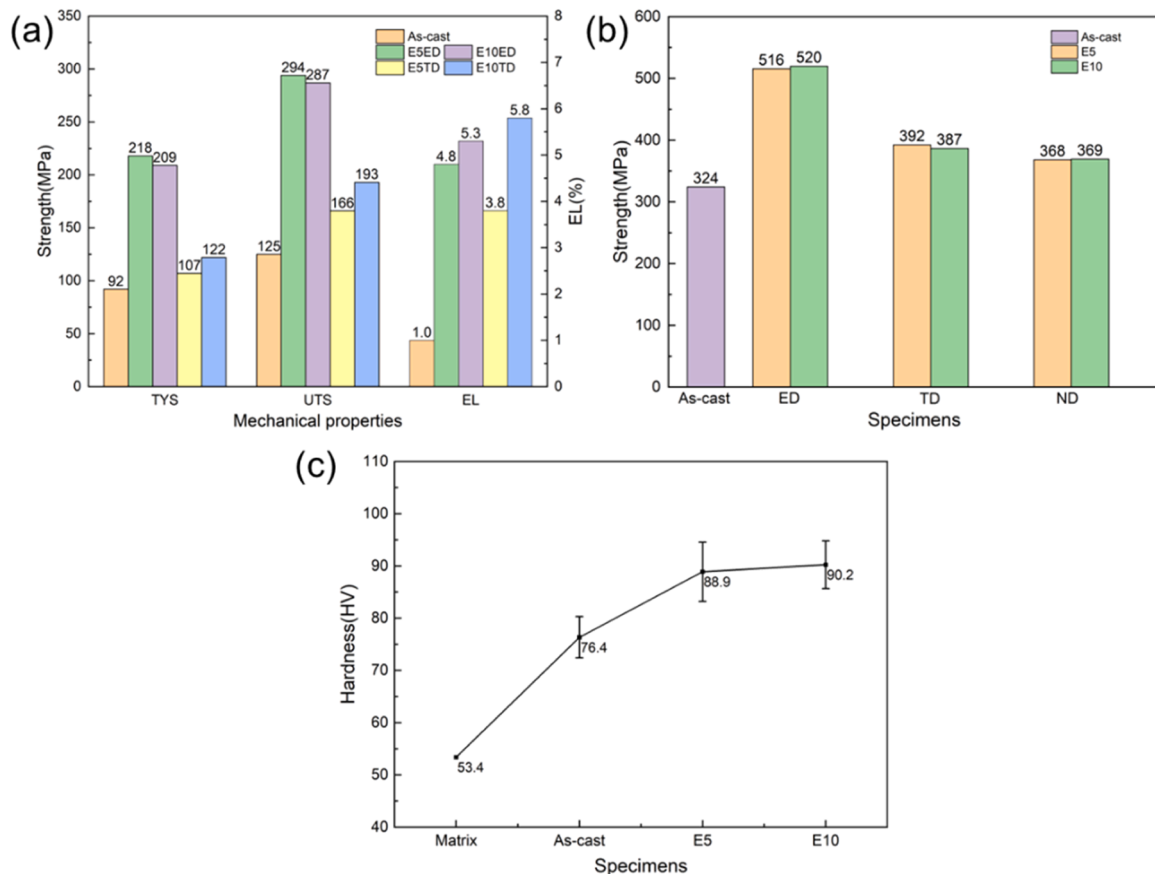


Fig. 6. Mechanical properties of SiC_p/ZX20K at room temperature. (a) YS, UTS and EL of tension, (b) UCS of compression, (c) Vickers hardness.

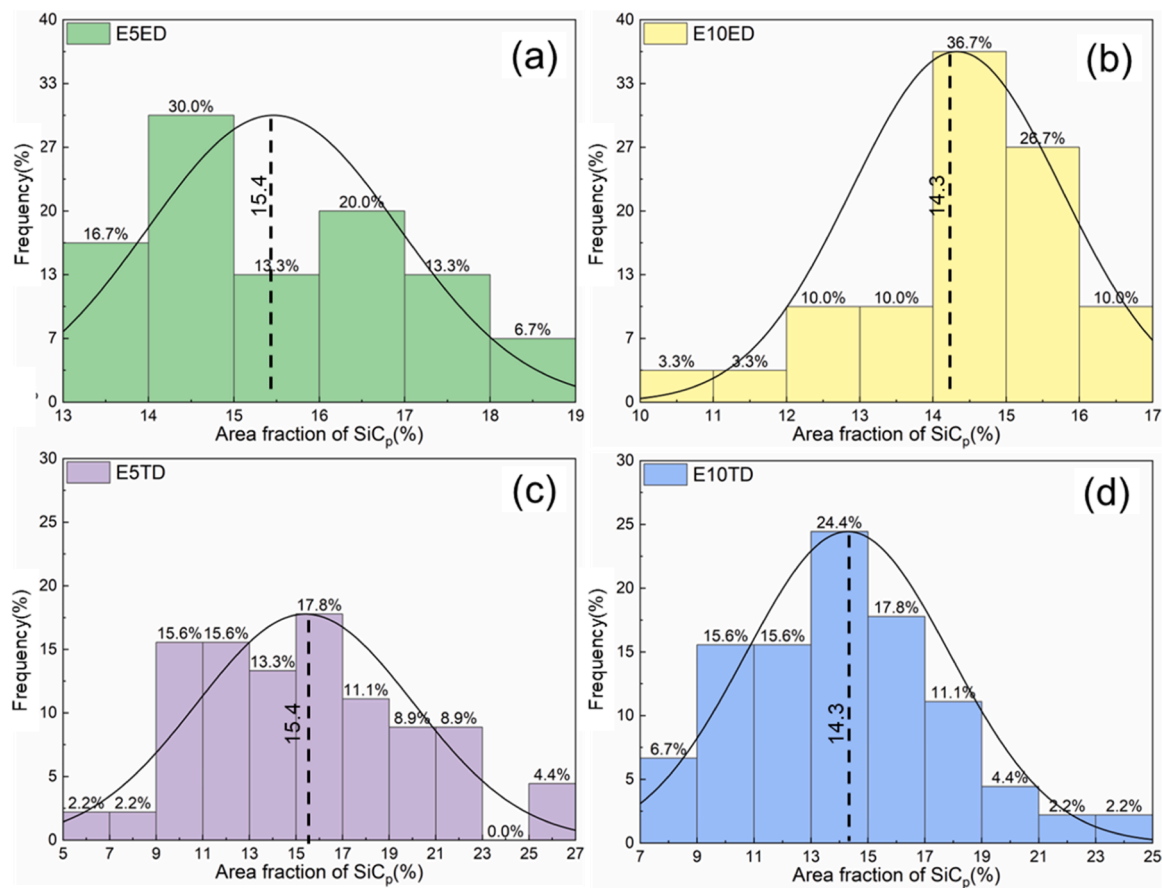


Fig. 7. Frequency of area fraction of SiC_p per subset about different directions. (a-b) ED of E5 and E10, (c-d) TD of E5 and E10.

bimodal distribution in the intervals of 14–17 %. The frequency of area fraction in the 14–17 % interval was only 63.3 %, indicating an uneven particles distribution. As shown in Fig. 7(b), the peak frequency of the area fraction in E10ED fell within the 14–15 % interval, which was consistent with the calculated average value, although the span of the area fraction expanded to 10–17 %. The total frequency of the interval containing the average area fraction and its two adjacent intervals was 73.3 %, which was higher compared to 63.3 % in E5ED. And the frequency of the area fraction in the 10–12 % interval was only 6.6 %, indicating a more uniform distribution along the ED in E10 compared to E5. Comparing Fig. 7(c,d), the particles distribution in E5TD was extremely heterogeneous, with the area fraction ranging from 5 % to 27 %. The maximum and minimum values deviated significantly from the average value. The frequency of area fraction in the 21–27 % interval was as high as 13.3 %. In contrast, the range of the area fraction of SiC particles in E10TD was compressed to 7–25 %, and the peak frequency at the average area fraction was 24.4 %, which was higher than that for E5TD. Moreover, the total frequency of the interval containing the average area fraction and its four adjacent intervals was 84.5 % considerably higher than the 66.7 % observed for E5TD. This indicated that the frequency of extremely low or high area fractions along the TD was relatively low in E10ED, leading to a more uniform particle distribution. From the above comparison, it can be seen that a higher extrusion speed is beneficial for improving particle distribution.

During the extrusion process, the decrease in cross-sectional area forced the softened matrix to fill the spaces between the necklace-type distributed SiC particles, ultimately leading to the breakup of particle clusters and the elimination of the necklace-type distribution. The extrusion temperature increased due to the frictional heat generated by the higher extrusion speed, which improved the fluidity of the matrix [37]. Simultaneously, the necklace-type distribution transitioned to a

band-type distribution, and the SiC particle clusters were eliminated.

4.2. Effects of hot extrusion on the matrix

Hot extrusion can refine grains by inducing recrystallization, and the effect of extrusion speed on recrystallization grain size can be analyzed by observing grain size distribution maps in Fig. 8(a–c). It could be seen that after extrusion, the average grain sizes of E5 and E10 were significantly decreased compared to those after homogenization treatment. In Fig. 8(b,c), it could be observed that the grain sizes of E5 and E10 exhibited a bimodal distribution, with E10 having a slightly larger grain size compared to E5. In E5, the area fraction of fine equiaxed recrystallized grains with a grain size of less than 20 μm was approximately 50.8 %, and the average grain size was 9.53 μm . In contrast, the fraction of such grains in E10 decreased to 45.44 %, with an average grain size of 9.20 μm which was slightly smaller than that of E5. Fig. 8(d,e) shows the phase maps of the magnesium phase with grain sizes smaller than 20 μm and the SiC particles. It could be seen that the fine equiaxed recrystallized grains were mainly located near the band-type distributed SiC particles. It is known that precipitated phase can exert a Zener drag effect on the surrounding grain boundaries in Mg alloys [38,39], which hinders grain growth by limiting grain boundary migration. In this study, the SiC particles can also play a similar role in pinning the grain boundaries, which will be discussed in detail later.

Many studies [40,41] have demonstrated an inverse relationship between the dynamically recrystallized (DRX) grain size (d_{DRX}) and the Zener-Hollomon parameter (Z), defined as $Z = \dot{\epsilon} \exp(Q/RT)$, where $\dot{\epsilon}$ is the strain rate, Q is the activation energy for lattice diffusion of Mg, R is the gas constant, T is the deformation temperature, A is a constant, and m is the grain size exponent. It can be seen that during deformation process, the d_{DRX} decreases with increasing strain

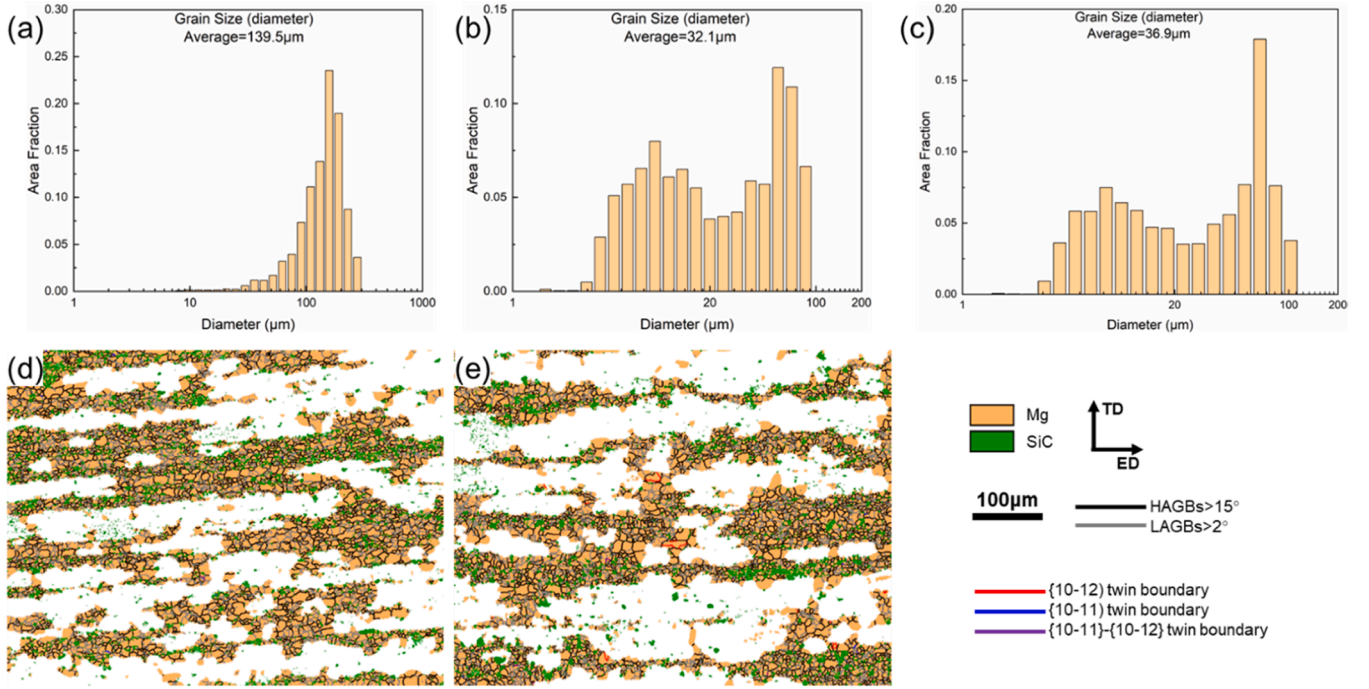


Fig. 8. (a–c) Grain size distribution of (a)As-homogenized, (b) E5 and (c) E10, (d, e) subsets of phase maps of (b) E5 and (c) E10.

rate or decreasing deformation temperature. There is a conversion relationship between the extrusion speed (V_R) and the strain rate ($\dot{\epsilon}$) as follows [42]:

$$\dot{\epsilon} = \frac{6D_B^2 V_R \ln ER}{D_B^3 - D_E^3} \quad (1)$$

Where ER , D_B , and D_E represent the extrusion ratio, diameter of the

extruded rods, and rectangular equivalent diameter of the extrudates, respectively. It can be observed that the extrusion speed is directly proportional to the strain rate during the deformation process. In other words, the d_{DRX} decreases with increasing extrusion speed. This also explains why the grain size in E5 was larger than in E10 for grains smaller than 20 μm .

The coarse grains larger than 20 μm in Fig. 8(b,c) were mostly

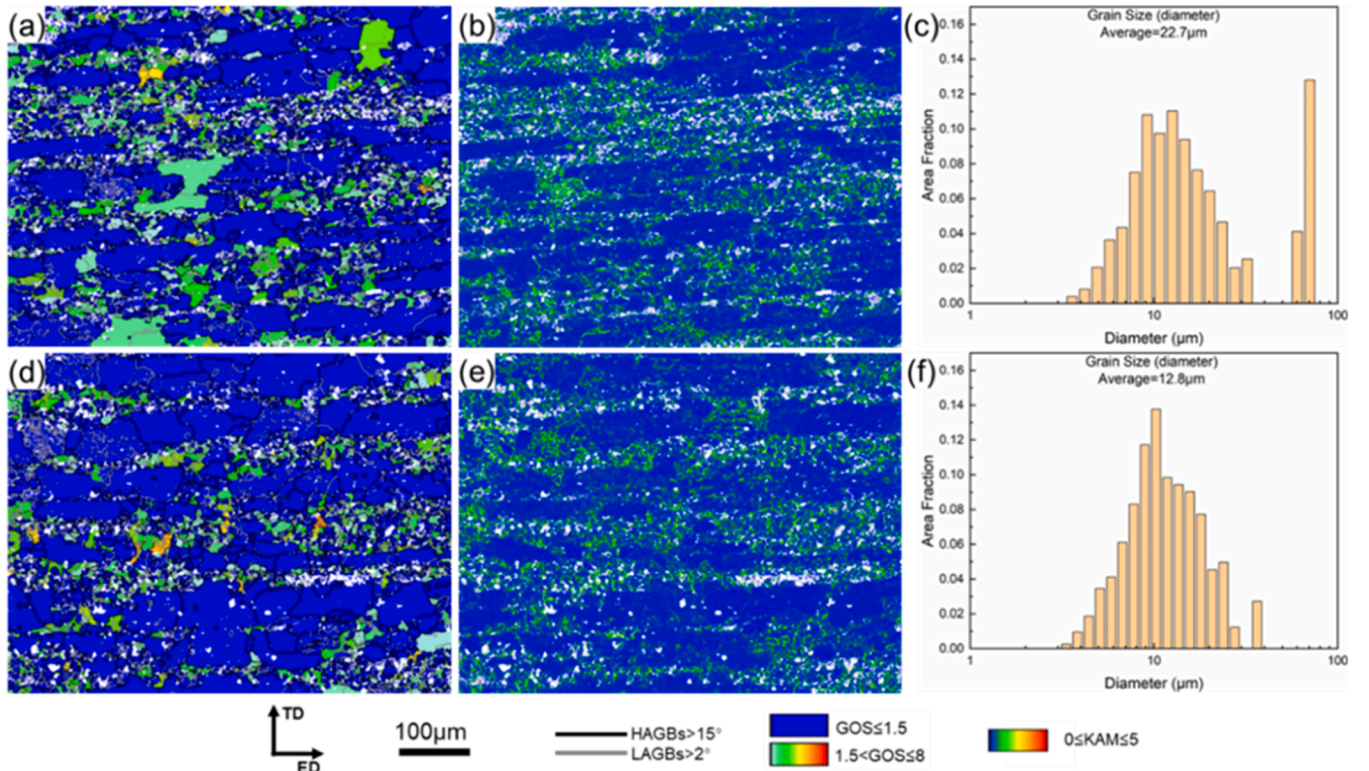


Fig. 9. Grain distribution statistics of E5(a–c) and E10 (d–f). (a, d) GOS maps, (b, e) KAM maps, (c, f) grain size distribution.

located in the SiC-rich regions shown in Fig. 5(e,f). For grains larger than 20 μm , the average grain sizes in E5 and E10 were 54.10 μm and 59.76 μm , respectively. Grain Orientation Spread (GOS) maps were used to determine whether the grains larger than 20 μm in Fig. 8(b,c) were grown recrystallized grains or deformed grains, as shown in Fig. 9(a,d). The GOS of a grain describes the average misorientation between each pixel within the grain and its average orientation. It is one of the methods used to distinguish deformed and recrystallized grains [43,44]. In this study, grains with $\text{GOS} \leq 1.5$ were considered as recrystallized grains and were shown in blue. Among the subset of grains larger than 20 μm in E5, the proportion of grains with $\text{GOS} \leq 1.5$ was 81.6 %, while in E10, the proportion was 88.4 %. Therefore, it could be approximately assumed that both E5 and E10 had undergone complete recrystallization after extrusion, and the grains larger than 20 μm were recrystallized grains. Li [45] observed a similar phenomenon in the recrystallized microstructure of AZ31 extruded at high temperatures, attributing it to significant grain growth during the cooling process, referred to as secondary recrystallization. Secondary recrystallization does not involve nucleation, and its driving force is the reduction of interfacial energy [46]. As shown in Fig. 5(e,f), the grain boundaries of coarse secondary recrystallized grains are often concave, and smaller grains enclosed within them are engulfed as grain boundary migration occurs. In this study, the SiC particles in the particle-depleted regions hindered grain growth, with the limiting grain size being inversely proportional to the volume fraction of particles. During the extrusion process, the distribution of SiC particles gradually changed, forming particle-depleted regions with reduced SiC particle volume fraction, which weakened the hindrance effect. Consequently, the grains in these regions could overcome the obstacles and grow abnormally, resulting in secondary recrystallization. In the Mg-3Gd-3Al-1Zn alloy [47], abnormal grain growth also occurred during annealing at 400°C due to the discrete distribution of intermetallic compounds.

The influence of SiC particles on the surrounding grains could be more intuitively observed and confirmed through EBSD analysis. It was worth noting that many fine grains with a $\text{GOS} > 1.5$ were present near the SiC particles represented by white areas, especially in the SiC-rich regions. By comparing the grain size changes before and after extrusion, it could be determined that these were recrystallized grains. Guo et al. [48] also observed a similar phenomenon in the hot-rolled microstructure of Mg-Al-Zn alloy. Fig. 9(b, e) show the Kernel Average Misorientation (KAM) maps for E5 and E10, which characterize the average misorientation between each pixel and its neighboring pixels. This method was often used to analyze residual stress within a deformed microstructure [49,50]. In this study, the residual stress was primarily concentrated near the SiC particles, and its distribution was consistent with the band-type distribution of SiC particles. However, the residual stress was significantly reduced in SiC-poor regions, and the proportion of grains with $\text{GOS} > 1.5$ was also greatly reduced.

The grain size distributions of grains with a $\text{GOS} > 1.5$ in E5 and E10 are shown in Fig. 9(c, f). The average grain size in E10 was 12.8 μm , which was significantly smaller than that in E5. The fact that the GOS of the fine recrystallized grains near the particles was greater than 1.5 indicated that the recrystallized grains formed during early deformation underwent a second further deformation during the later stages of extrusion. During hot extrusion at high temperatures and low strain rates, the initially formed recrystallized grains can continue to deform, leading to an increase in intra-grain misorientation [51]. The strain rate for E5 was lower than that for E10, which explains why the proportion of deformed recrystallized grains in E5 was 23.1 %, compared to 16.1 % in E10 after extrusion. Consequently, the grain size of E5 was also significantly larger than that of E10. Therefore, the SiC particles not only restricted grain growth in the particle-rich regions through pinning effects but also led to the formation of softer regions, which were more prone to further deformation.

As shown in the IPF maps in Fig. 5(f–g), fine grains and coarse grains in E5 and E10 exhibited different crystallographic orientations. Taking

E10 as an example, with its bimodal distribution of recrystallized grain sizes, the IPF map showed two distinct colors representing two different crystallographic orientations, as illustrated in Fig. 10. For instance, fine grains 'a' and 'b' belonged to one category, while coarse grains 'c' and 'd' belonged to another.

The (0001) PF and the IPF parallel to the ED of E10 were compared between fine grains ($\leq 20 \mu\text{m}$) and coarse grains ($> 20 \mu\text{m}$) in Fig. 10. The IPF showed that the texture type of E10 was a $\langle 10\text{--}10 \rangle\text{--}\langle 2\text{--}1\text{--}10 \rangle$ double fiber texture. The $\langle 10\text{--}10 \rangle\text{//ED}$ component was the dominant texture in the fine grains, but there still existed a weak $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ texture component. In contrast, the primary texture component of the coarse grains was the $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ texture. Wang et al. [52] obtained a partially recrystallized structure in a Mg-Zn-Ca alloy by low-temperature extrusion, where the fine recrystallized grains exhibited a $\langle 10\text{--}10 \rangle\text{//ED}$ orientation. Cano-Castillo, G et al. [53] found that the grain size increased by increasing extrusion speed, which was also accompanied by a change in grain orientation from $\langle 10\text{--}10 \rangle\text{//ED}$ to $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ in a Mg-Zn-Ca alloy. Therefore, we can infer that the recrystallized grains underwent preferential growth, meaning that grains with a $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ orientation had growth advantages.

The reason why grains with a $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ orientation had growth advantages can be inferred by analyzing the microstructure and orientation information. In the extruded $\text{SiC}_p/\text{ZX20K}$ composites, due to frictional heat generation and the Zener drag effect, both fine recrystallized grains close to the initial state and coarse grown grains were retained simultaneously. So it was possible to study the microstructure of the initial recrystallized grains and the grown recrystallized grains simultaneously. In the IPF of fine grains in E10 (Fig. 10), two texture components were observed, $\langle 10\text{--}10 \rangle\text{//ED}$ and $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$, with the former being the main texture component. Grains with two texture components were extracted separately from E10 and labeled as E10a ($\langle 10\text{--}10 \rangle\text{//ED}$) and E10b ($\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$). The corresponding IPF maps are shown in Fig. 11(a,d), and the area ratio of E10a to E10b was about 4.2:1. The average grain size of E10a was about 8.8 μm , with 31.2 % of the grains being larger than 10 μm . In contrast, E10b had a larger average grain size of 9.4 μm , with 38.0 % of the grains being larger than 10 μm . Therefore, the grains in E10b, which had an initial size advantage, could preferentially grow and maintain this growth advantage during subsequent growth processes [54]. In Fig. 11(b,e), the proportion of grains with a GOS greater than 1.5 in E10a was 35.2 %, while it was only 13.8 % in E10b. The KAM maps in Fig. 11(c,d) also confirmed this difference indicating that the storage energy in E10b was lower than that in E10a. During subsequent grain growth, grains with lower storage energy tend to grow preferentially and absorb grains with higher storage energy [55]. Moreover, as shown in Fig. 11(a), grains with similar orientation of E10a were adjacent to each other. However, in Fig. 11(d), the grains in E10b were distributed in an isolated manner, with most of them surrounded by grains from E10a (which is more easily observed in Fig. 10). The orientation difference between grains in E10b and their adjacent grains was larger, and the grain boundaries between them were high-angle grain boundaries (HAGBs), which had fast migration speed during grain growth [56]. Therefore, the grains with a $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ orientation exhibited characteristics such as larger initial size, lower storage energy, and a greater orientation difference from adjacent grains. These features provided them with growth advantages, resulting in a transformation of the texture type of the grown recrystallized grains to a $\langle 2\text{--}1\text{--}10 \rangle\text{//ED}$ fiber texture.

4.3. Effects of hot extrusion on mechanical properties

When the loading direction was parallel to the ED and TD after extrusion, the YS and UTS exhibited obvious difference, indicating anisotropy. When the extrusion speed was 5 mm/s, the ratio of YS between the ED and TD was 2.04, which decreased slightly to 1.71 when the extrusion speed increased to 10 mm/s. It is well known that the main deformation mechanism of magnesium alloys at room temperature is

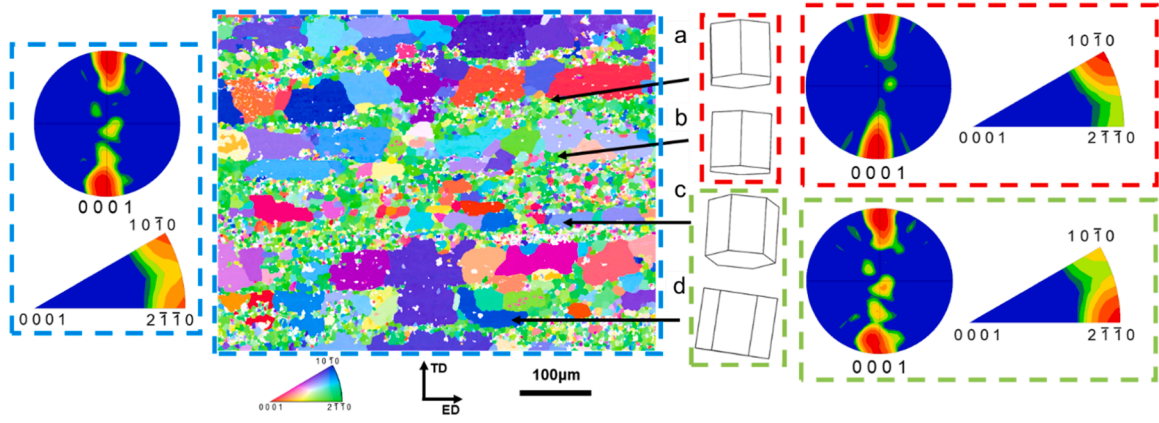


Fig. 10. The examples of two kinds of grains with different crystallographic orientations in E10.

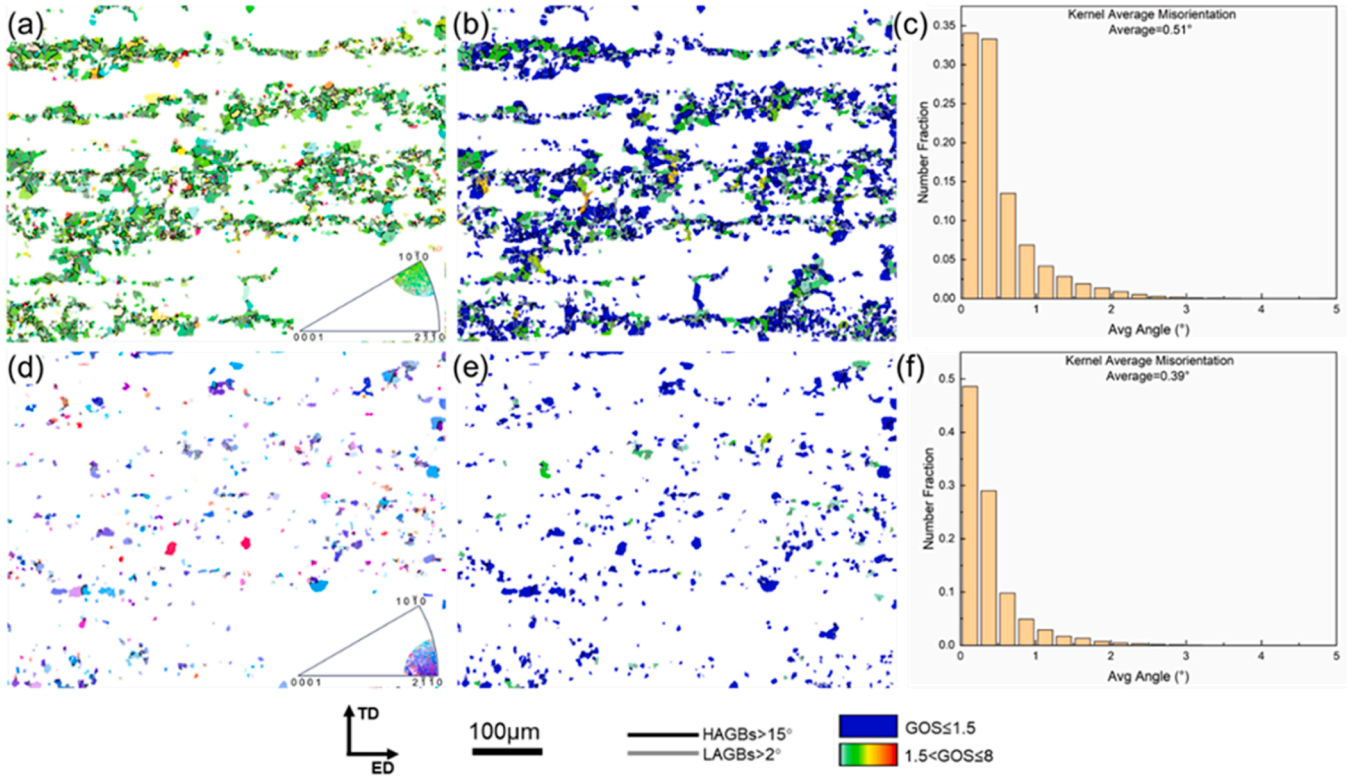


Fig. 11. Preferred growth analysis of E10a (a-c) and E10b(d-f). (a, d) IPF maps, (b, e) GOS maps, (c, f) KAM distribution.

basal slip, with a slip system of $(0001) \langle 11\bar{2}0 \rangle$. The critical resolved shear stress (CRSS) for basal plane slip in magnesium alloys is given by:

$$\text{CRSS} = \sigma_s \cos \varphi \cos \lambda \quad (2)$$

Where σ_s is the initial yield strength required to initiate basal plane slip, φ is the angle between the loading direction and the normal of the slip plane, and λ is the angle between the loading direction and the slip direction. The product of $\cos \varphi$ and $\cos \lambda$ is known as the Schmid factor (SF) [57]. For a given CRSS, there is an inverse relationship between σ_s and SF. Therefore, the SF for basal plane slip is often used to analyze the anisotropy of mechanical properties. As shown in Fig. 12(a,b), there was a significant difference in SF between the two tensile loading directions parallel to the ED and TD. Taking E5 as an example, when loaded along the ED, the SF was only 0.08, which was significantly smaller than the value of 0.26 when loaded along the TD. This would result in higher strength and lower plasticity. The SF distribution maps for E5 were

drawn separately tensile force loading parallel to the ED and TD, with the colors mapped to the (0001) PFs as shown in Fig. 12(c,d). Due to the formation of basal texture in $\text{SiC}_p/\text{ZX20K}$ composites after extrusion, the (0001) basal plane was approximately parallel to the ED. When the tensile direction was parallel to the ED, $\cos \varphi$ was close to 0, resulting in small SF values in Fig. 12(a). Consequently, a higher initial yield strength was required to initiate basal slip. If the tensile loading direction was parallel to the TD, and the normal direction of the (0001) basal plane was parallel to the TD, the slip direction would be perpendicular to the tensile loading direction, resulting in $\cos \lambda = 0$. If the normal direction of the basal plane was parallel to the ND, the slip plane would be parallel to the tensile loading direction, leading to $\cos \varphi = 0$. Therefore, only these two crystallographic orientations were hard orientations. When loaded parallel to the TD, the SF values were uniformly distributed in the range of 0–0.5, with an average value of 0.26. As a result, the initial yield strength required to initiate basal slip was significantly

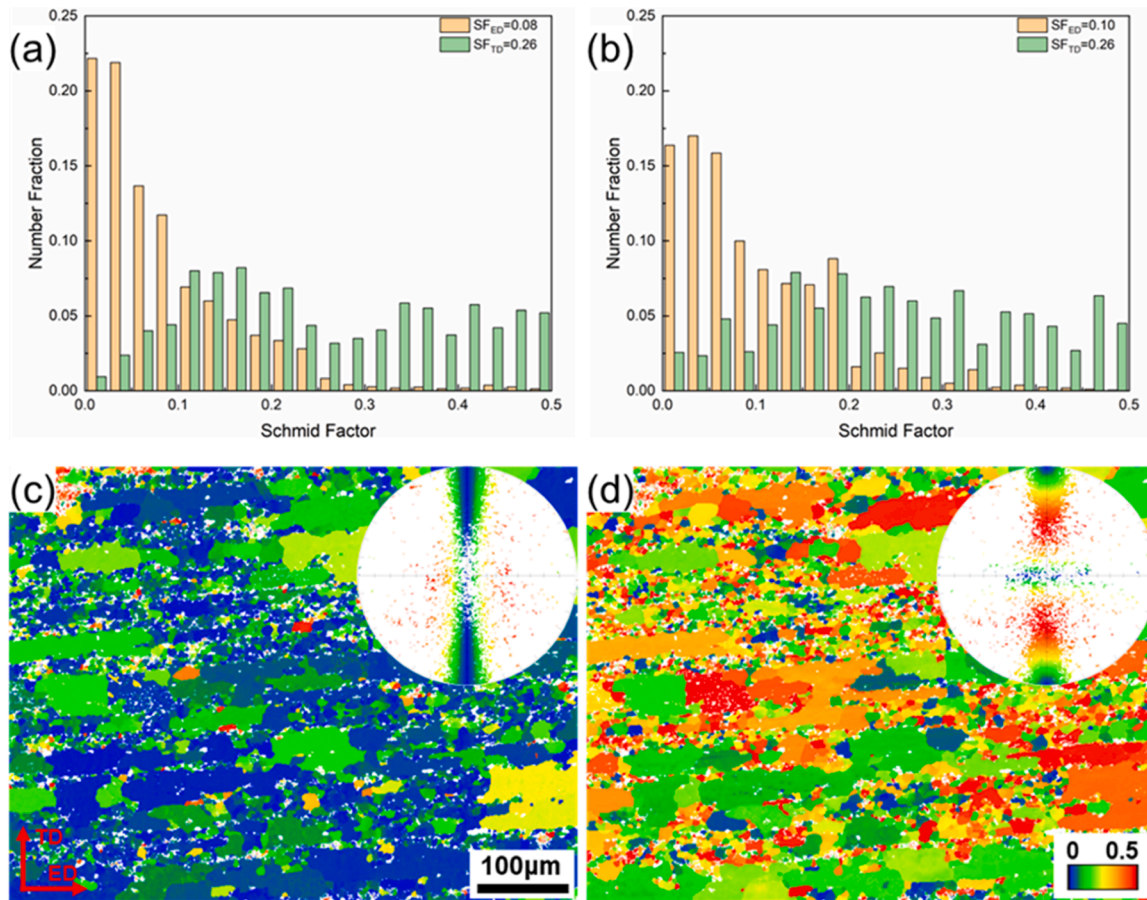


Fig. 12. Statistics of SF for basal slip in (a) E5, (b) E10 and distribution of E5's SF when loading direction was parallel to ED(c), TD(d).

reduced.

In addition to crystallography, the mechanical properties of SiC_p/ZX20K composites were also influenced by the orientation of the particles. For example, due to the uneven distribution of SiC particles along the TD in E5, the plasticity in this direction was still lower than that in the ED. Ganesh et al. [58] found that the anisotropy of particle orientation significantly impacts the mechanical properties of particle-reinforced aluminum-based composites, and this effect becomes more pronounced as particle content increases. Through finite element analysis of single-crystal magnesium containing particles of different shapes and orientations, Zhang et al. [59] concluded that rod-shaped particles are more effective in enhancing compressive and tensile properties when their axial direction is perpendicular or parallel to the loading direction, or when the axial direction of platelet-shaped reinforcements is perpendicular to the loading direction. In this study, the long axes of most SiC particles were aligned parallel to the ED, as shown in Fig. 4. Consequently, the anisotropy of SiC particles orientation results in anisotropy in the mechanical properties of the composites. Therefore, the observation that $UCS_{ED} > UCS_{TD} > UCS_{ND}$ further demonstrates that the anisotropy of particle orientation affects mechanical properties.

However, despite having similar SF values and SiC particles orientation, E5 and E10 exhibited significantly different degrees of anisotropy in room temperature tensile yield strength. The increase in extrusion speed resulted in a larger average grain size in E10 compared to E5. According to the theory of fine grain strengthening, the YS of E10 should be lower compared to E5, regardless of loading direction (parallel to ED or TD), assuming similar texture strength. However, an anomalous situation occurred when loading parallel to the TD, which was attributed to the distribution of SiC particles. Increasing the extrusion speed led to

a more uniform distribution of SiC particles in the TD, thereby improving both the EL and YS compared to E5. This was also the reason why the anisotropy in yield strength of E10 was weaker than that of E5.

In summary, the crystallographic orientation of the matrix and the orientation of SiC particles in the composites changed during the extrusion process, resulting in the anisotropy in mechanical properties. Additionally, the difference in distribution uniformity of SiC particles, caused by different extrusion speeds, further influenced the degree of anisotropy of mechanical properties.

5. Conclusions

In this study, SiC_p/ZX20K composites were prepared by stir casting followed by hot extrusion at speeds of 5 mm/s and 10 mm/s at 400 °C. Their microstructure and mechanical properties were characterized, and the main findings are as follows:

(1) In the as-cast SiC_p/ZX20K composites, a fine and dispersed Mg₂Ca eutectic phase was present, and the SiC particles were distributed in a necklace-type pattern at the grain boundaries with a few clusters. Its hardness was significantly improved compared to the matrix alloy, although plasticity remained poor.

(2) Hot extrusion effectively eliminated defects such as porosities and particle clusters, and increasing the extrusion speed further improved the uniformity of SiC particle distribution. Moreover, a basal plane fiber texture with the {0001} plane parallel to the TD was formed, with preferential growth for grains whose $\langle 2-1-10 \rangle$ orientation was aligned with the ED. The band-type distribution of SiC particles restricted the migration of adjacent grain boundaries, hindering grain growth. Secondary recrystallization occurred in grains within the SiC-poor regions.

(3) After hot extrusion, the mechanical properties of the SiC_p/ZX20K composites were significantly improved. The yield strength along the ED during tension increased by 137 % and reached to 218 MPa compared to the as-cast state, and the plasticity was also significantly enhanced. Increasing the extrusion speed resulted in better overall mechanical properties and reduced the anisotropy of mechanical properties.

CRedit authorship contribution statement

Rongshi Chen: Validation, Supervision, Funding acquisition, Conceptualization. **Xihai Li:** Writing – original draft, Data curation. **Hong Yan:** Writing – review & editing, Supervision, Funding acquisition. **Boyu Liu:** Supervision. **Zhiwei Shan:** Supervision. **Yanping Wang:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data Availability

Data will be made available on request.

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