Study on the formation mechanism of segregation in squeeze casting ZM5 magnesium alloy baffle

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Abstract: Squeeze casting, as a high-efficiency and green forming technology, has received extensive attention and application. However, due to the process characteristics of liquid metal solidification forming, the performance of castings is unstable, mainly manifested in the segregation defects produced on the castings. This study takes the ZM5 magnesium alloy partition as an example to explore the influence laws of pouring temperature and casting structure on the morphology and distribution of segregation bands. The results show that with the enrichment of Al element, the segregation band is a solute separation band, mainly existing in the form of $Mg_{17}Al_{12}$ phase, and the segregation band is mainly in the form of sheet and strip. Under different pouring temperatures and different casting positions, the variation trends of defect band distribution are not consistent. The stress mutation occurs near the wall thickness mutation of the melt, resulting in the fluctuation of the width and position of the defect band. In addition, during the pouring process, the pouring temperature and the cooling rate of the melt have a crucial impact on the formation of the defect band. This study explores the formation mechanism of segregation bands by optimizing the squeeze casting process parameters, which can effectively solve the problem of poor performance stability of squeeze casting castings caused by segregation defects.

Keywords: Squeeze casting; Magnesium alloy; Macroscopic segregation

1 Introduction

Squeeze casting is a high-efficiency and environmentally friendly forming technology for metallic materials. This technology has broad market prospects in the forming and manufacturing of structural components with high requirements for density, airtightness, surface quality and mechanical properties. Squeeze-cast aluminum alloy products have been applied in the automotive manufacturing field[1-3]. However, due to the problems of magnesium alloy's easy oxidation and combustion, easy occurrence of cold-separation defects and solute segregation, the performance of squeeze-cast magnesium alloy castings is not stable, especially the performance of indirect squeeze-cast products fluctuates greatly. Its performance stability cannot be compared with that of solid-state forged parts, and is even lower than that of low-pressure castings and die-castings. One of the reasons for the unstable performance is that squeeze casting belongs to the category of liquid forming, and the redistribution of solute during the solidification process cannot be completely avoided, and segregation is inevitable[4-10]. Breaking through this problem is the key to achieving high performance. Segregation is a phenomenon where alloy elements are unevenly distributed during crystallization, which is an inevitable result of solute redistribution. Segregation is a casting defect that affects the mechanical properties and solid solution strengthening ability of castings. Segregation phases are concentrated at the grain boundaries, and the fracture form is intergranular brittle fracture, resulting in poor plasticity of the castings and affecting their tensile strength and elongation[11-14].

According to the literature review, the existing research on alloy segregation mainly focuses on traditional AM and AZ series magnesium alloys and aluminum-silicon series aluminum alloys. ZM5 magnesium alloy, which does not contain rare earth metals and zirconium, is a relatively inexpensive magnesium alloy and has the advantages of good fluidity, weldability, and low hot cracking tendency[15-18]. However, current research on ZM5 magnesium alloy mainly concentrates on the improvement of its properties, while less attention has been paid to the microstructure characteristics and segregation phenomena of extrusion castings[19-22]. In addition, a systematic study on the distribution of multiple segregation bands in ZM5 magnesium alloy and the differences among various process parameters is still a relatively unexplored field.

In this study, an indirect squeeze casting process was employed to fabricate a ZM5 magnesium alloy partition. Microstructure characterization was conducted at different locations of the castings, and the effects of casting structure and process parameters on the distribution of segregation bands in squeeze cast ZM5 alloy were discussed.

2 Experimental procedures

Commercial ZM5 magnesium alloy was used as the experimental material, and its chemical composition is shown in Table 1. During the experiment, eight partition pieces with a diameter of 31 cm and a height of 9 cm were fabricated by a multi-functional semi-solid die casting machine. The 3D model of the partition piece is shown in Figure 1. Four groups of process parameters varying with the casting temperature were designed for comparison, and the key process parameters adopted are listed in Table 2. The same extrusion pressure and casting rate were used in all processes, and the same amount of protective gas was introduced. During the casting stage, the molten magnesium alloy was poured into the cylinder at a rate of 9.0 L/min, and then extruded into the mold at a speed of 100 cm/min. After the melt filled the cavity, a pressure of 450 T was maintained for 55 seconds. The casting solidified and formed, and the mold was opened to remove the casting. The process flow chart is shown in Figure 2.

Table 1. Mass fraction of each component of ZM5 spacer parts

Ingredient	Al	Zn	Mn	Fe	Ni	Cu	Si	Mg
Mark	7.5~9.0	0.2~0.7	0.15~0.5	≤0.004	≤0.008	≤0.008	≤0.05	tolerance

Table 2. Pouring process parameters of ZM5 partition parts

Group number	Pouring rate（L/min）	Mold temperature（℃）	Pouring temperature（℃）
A1	9.0	240	700
A2	9.0	240	720
A3	9.0	240	740
A4	9.0	240	760

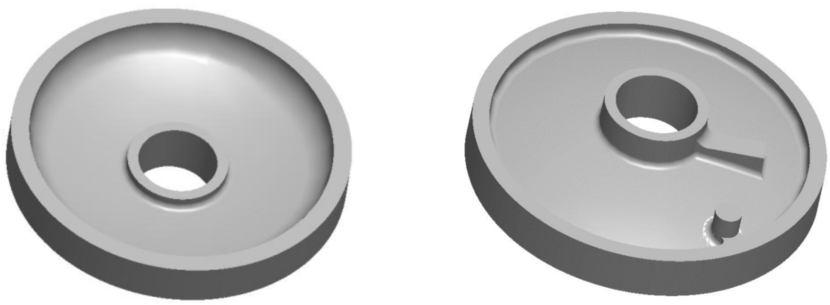

Figure 1. 3D model diagram of ZM5 partition component

Samples were extracted from different positions of the casting to study the influence of the casting structure on the distribution of defect bands, and samples were taken from the thin-walled and concave parts of the casting to study the evolution of segregation bands at different pouring temperatures, as shown in Figure 3. All samples were subjected to standard metallographic treatment and then etched with 4% nitric acid. Microstructure observation was carried out using an optical microscope (OM), a scanning electron microscope (SEM) equipped with an X-Max50 energy dispersive spectrometer (EDS). Electron backscatter diffraction (EBSD) experiments were conducted using a scanning electron microscope, and the crystal orientation information of the samples was calibrated. The distribution of segregation bands was quantitatively analyzed using Image Pro Plus 6.0 software.

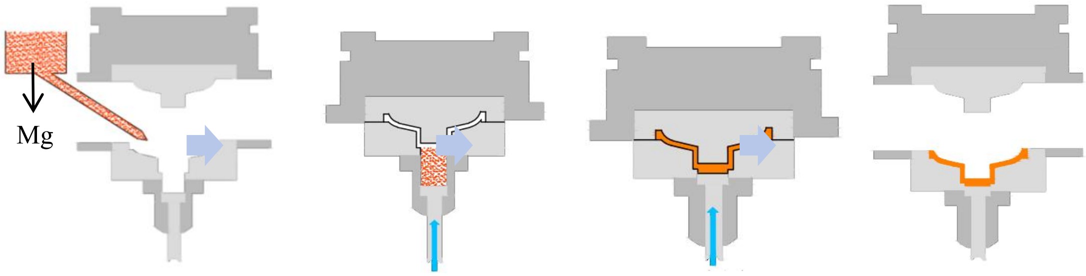

Figure 2. Process Flow Diagram of Indirect Extrusion Casting

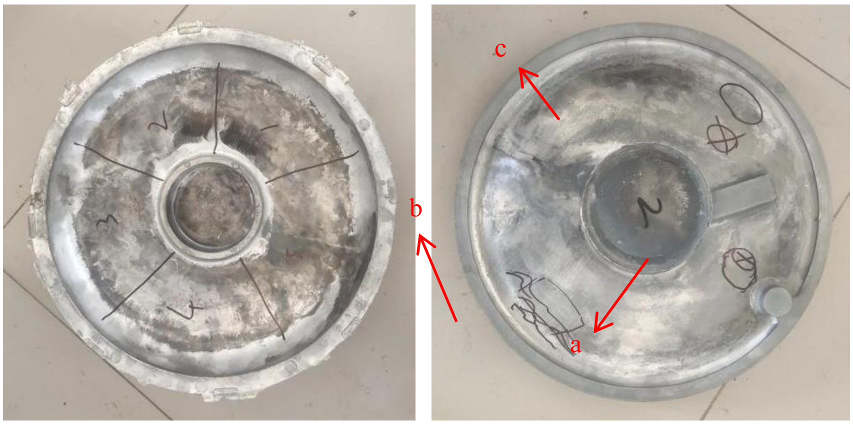

Figure 3. Finished drawing of ZM5 partition component and marking of sampling positions

3 Experimental results

3.1 The typical morphology and solute distribution of defect bands

The X-ray radiography images show the morphology of several typical segregation bands and the surrounding microstructure of the ZM5 alloy indirect extrusion casting sample (see Figure 4). As shown in the figure, there is a distinct aggregation of elements, with the white areas representing the α-Mg matrix and porosity, while the dark gray and black areas represent intermetallic phases and the wall thickness sections. The defects in the sample mainly present in three forms: the plate-like segregation defect near the thin-walled depression as shown in Figure 4(a), the block-like segregation defect near the slot as shown in Figure 4(b), and the strip-like segregation defect on the thin-walled plane as shown in Figure 4(c). The microstructure of the cross-sections at the above four locations was observed using a SEM device, and the microstructure of the corresponding four locations is shown in Figure 5. It can be seen from the figure that there is a significant difference in the morphology between the segregation bands and the surrounding microstructure, and the content of intermetallic phases in the segregation bands is much higher than that in the adjacent areas. At 500 times magnification, it can be clearly seen that the plate-like segregation band in Figure 5(a) does not run through the entire casting but exists in the middle interlayer, away from the upper and lower surfaces of the casting, occupying a certain longitudinal position and spreading laterally; Figure 5(b) shows the microstructure of the block-like segregation band, which is denser and has a larger solute segregation area compared to the plate-like and strip-like ones. Since it is a cross-sectional view, it indicates that the segregation spreads longitudinally from the interior to the surface of the casting; Figure 5(c) shows that the strip-like segregation band has a multi-layer distribution phenomenon and extends laterally in a linear manner. Through the observation of the microstructure images of the above four locations, it can be concluded that although the formation mechanism and intermetallic phase composition of the segregation bands at each location are similar, due to the different stress changes at each location, the diffusion directions of the segregation bands are also different, resulting in differences in the overall morphology of the segregation bands.

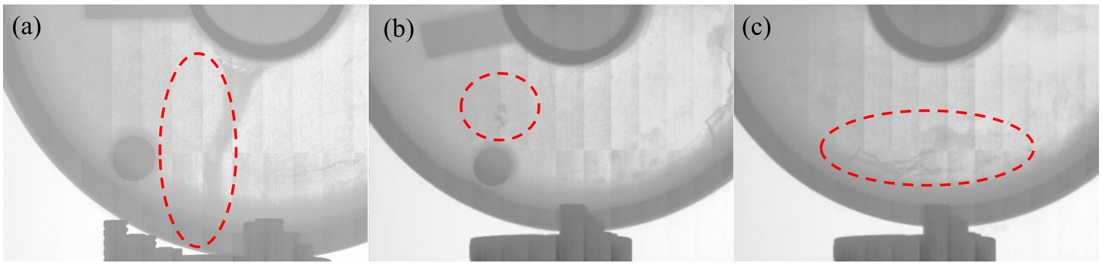

Figure 4. Common segregation band morphology in X-ray radiography of partition plate parts (a) Lamellar segregation band; (b) Massive segregation band; (c) Strip segregation band

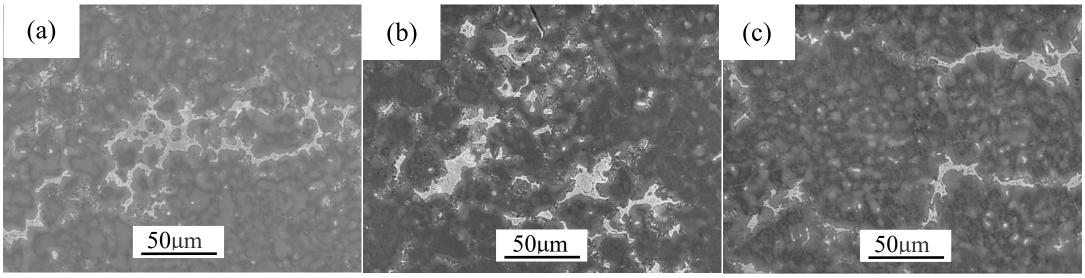

Figure 5. Microstructure of segregation areas in the partition plate Microstructure of lamellar segregation band; (b) Microstructure of blocky segregation band; (c) Microstructure of strip segregation band

Figure 6 shows the surface scanning electron microscope (SEM) image of the segregation band. The defect band is a solute segregation band enriched with Al elements. Due to the unique high-pressure feature of the squeeze casting process, the number of pores in the microstructure of the castings is significantly reduced. The core area of the casting usually has a large porosity in the form of shrinkage, as shown in Figure 6(a). However, there is no obvious accumulation of porosity within the defect band. That is to say, the defect band of the ZM5 alloy by squeeze casting is not a porosity band, but a segregation band with obviously non-uniform structure. It is worth noting that the particles inside the segregation band have the same shape, but differ in width, position and particle characteristics.

Elemental analysis was conducted at a point on the segregation band, and the results showed that the ratio of Mg to Al elements within the segregation band was approximately 17:12, indicating that the segregation phase mainly existed in the form of Mg17Al12. The content of Al element significantly increased at the segregation band and showed an opposite growth trend to that of Mg element. Zn element also accumulated in the segregation part, but there was no obvious relationship with Mg and Al elements.

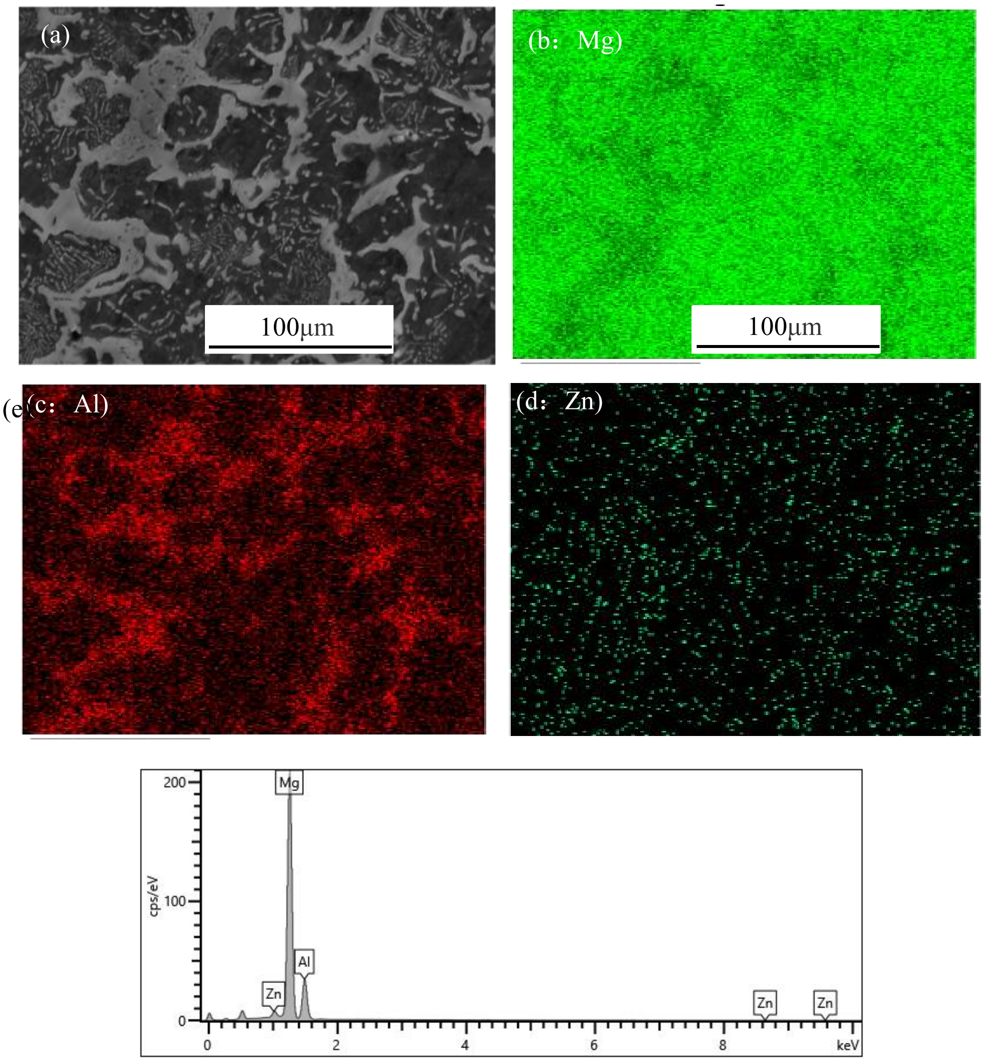

Figure 6. Scanning electron microscope (SEM) surface scan

(a) SEM image of the flake segregation band; (b) Mg element distribution in the segregation band; (c) Al element distribution in the segregation band; (d) Zn element distribution in the segregation band; (e) total element spectrum of the segregation band

3.2 Defect bands at different positions

Four pieces of the separator were selected under four sets of process conditions and marked as No. 1 to No. 4. Three positions were marked as a, b, and c on the castings (the sampling positions are shown in Figure 3). The segregation bands at different positions were studied. Figure 7 shows the X-ray radiographs of the four sets of separator castings. It can be seen from the X-ray radiographs that the normal area of the casting is a distinct bright ring-shaped area, while the area where the segregation bands exist is clearly black. The segregation morphologies in different areas of the samples are different, but the segregation morphologies at the same position are similar. The a position of the four separator castings in Figure 7 is near the concave area of the casting, and the segregation bands often appear as sheet-like, extending from the inner circle to the outer circle of the casting. The b and c positions of the separator castings are in the thin-walled area of the sample, and the segregation is mostly in irregular strip-like shapes. Multiple segregation bands are often present in one area, and the segregation morphology near the slot area is mostly block-like, existing as small black blocks separately. The microstructure was observed on the cross-sections of different areas using the SEM equipment (see Figure 8). At low magnification in Figures 8(a) and (b), it can be seen that the segregation part does not run through the entire casting but occupies a layer of the cross-section in the matrix and spreads along this direction. As the distance from the casting surface decreases, the segregation bands in the microstructure gradually disappear. Among them, the sheet-like segregation is the most obvious (as shown in Figure 8(c)), and the segregation bands divide the α-Mg matrix into upper and lower regions. The grain size of the intermetallic phase $Mg_{17}Al_{12}$ is significantly smaller than that of the α-Mg matrix, and the porosity is also significantly lower than that of the matrix part. In addition, comparing the results inside and outside the segregation bands, the grains inside the segregation bands are more compact and have well-developed dendrites, while the grain size of the defect bands outside is much larger than that inside the segregation bands.

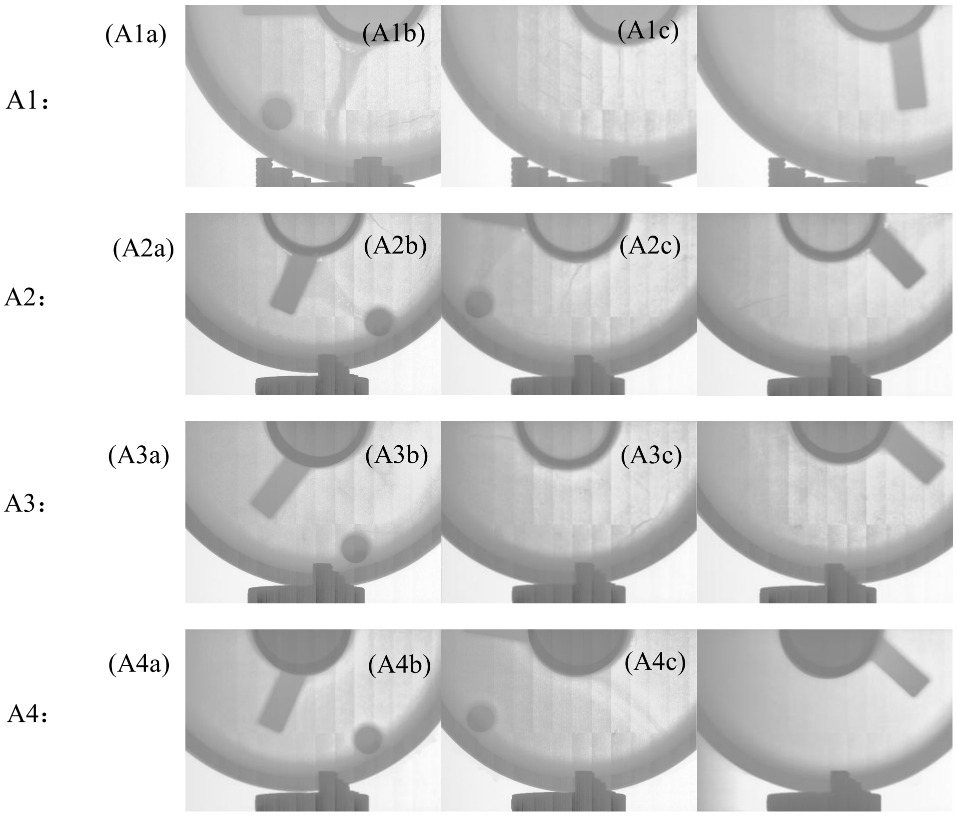

Figure 7. X-ray inspection results of the partition component

The pouring temperature of Group A1 is 700℃; that of Group A2 is 720℃; that of Group A3 is 740℃; and that of Group A4 is 760℃.

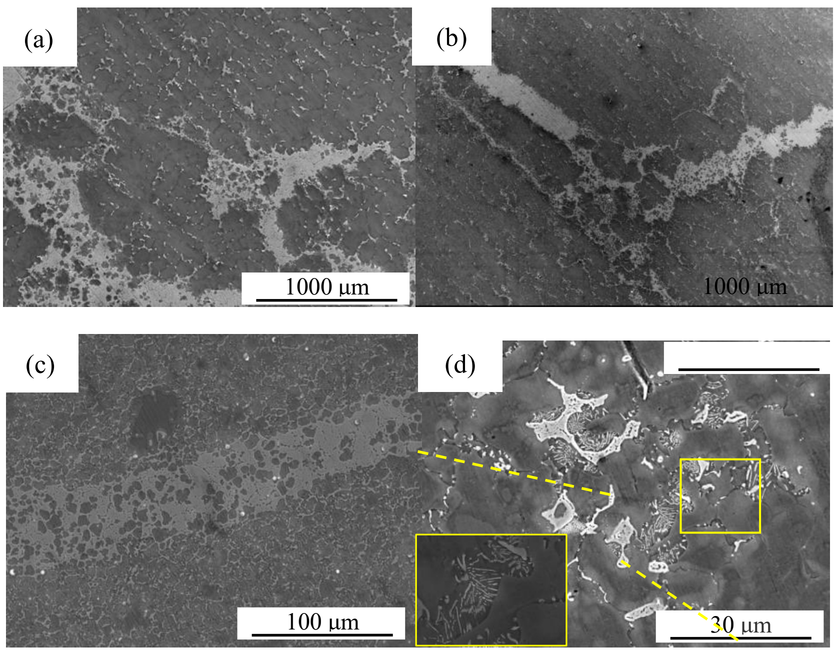

Figure 8. Microstructure diagram of the partition under SEM and (b) Microstructure images of flaky segregation bands at 37x magnification; (c) Comparison of segregation band structure and α-Mg matrix structure at 400x magnification; (d) Internal microstructure image of segregation band at 1000x magnification

3.3 Defective strips with different process parameters

During the squeeze casting process, the pouring temperature of the molten metal has a significant impact on the filling of the mold cavity and the subsequent solidification of the casting. For instance, existing research has found that the formation of porosity and ESCs in die castings is closely related to the pouring temperature. As is well known, the squeeze casting process is divided into two stages: pressurization and holding pressure. In this study, four sets of squeeze casting process parameters were designed and compared, with the pouring temperature increasing from A1 to A4. The changes in pouring temperature from A1 to A4 are shown in Table 2. The samples used to study the evolution of the defect band were taken from the same position a of the partition. As can be seen from Figure 9, as the pouring temperature decreases, the color of the segregation band in the casting deepens, and the contrast with the surrounding microstructure increases. However, when the pouring temperature is 740°C, the situation is different. The width and contrast of the segregation band experience a relatively significant decrease. When the temperature rises to 760°C, the segregation band slightly expands and intensifies. Combining the X-ray radiographs of the partition at various pouring temperatures in Figure 8, it can be seen that the segregation phenomenon in the A3 group at positions a, b, and c is significantly less than that in the other three groups, which also indicates that choosing a pouring temperature near 740°C has a significant effect on suppressing the occurrence of segregation phenomena.

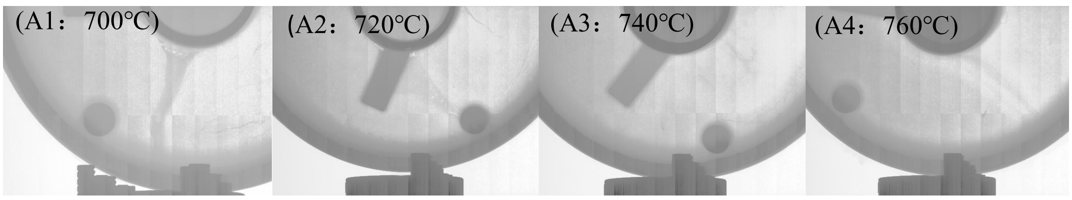

Figure 9. Morphological changes of segregation bands at the same position under different process parameters of the partitioning component

4 Discussion

This study separately discussed the variations in the distribution of segregation bands under different process parameters and different positions of the castings, but their changing trends were not consistent. When segregation bands appeared in the casting samples, their widths and positions varied greatly. Process parameters or casting structures might have different influences on the segregation bands. The cause of this difference can be traced back to the formation mechanism of the segregation bands. It is believed that during the solidification of the melt, in the semi-solid region where the grains are initially closely arranged, the grain network will collapse under a shear stress greater than what it can withstand. Then, the particles rearrange through mutual rotation and sliding, causing expansion, and the melt enters the semi-solid region, thus forming segregation bands. The reasons for the grain network being subjected to greater shear stress include the natural convection due to the solidification shrinkage of the alloy and forced flow under the influence of external forces[23]. In this case, the segregation band is a solute-rich band, and after the final solidification of the casting, the content of intermetallic phases is higher than that in the adjacent regions.

In ZM5 alloy, the nucleation and growth of α-Mg occur first when the melt temperature drops below the liquidus temperature (620℃). As the melt temperature decreases to 600℃, the solid fraction of the melt is approximately 50%. During the solidification process, due to the continuous supply of aluminum and the continuous nucleation and growth of α-Mg, the repulsion effect of solute on the growth of α-Mg and the absorption effect on the growth of $Mg_{17}Al_{12}$ lead to the coupled growth of α-Mg and $Mg_{17}Al_{12}$. Therefore, $Mg_{17}Al_{12}$ is the main intermetallic phase in ZM5 alloy.

Generally speaking, the formation of segregation bands requires two conditions: the existence of a semi-solid region with a solid fraction within a certain range and the application of shear stress to it. When the solid fraction of the melt is lower than the dendrite packing point fc, the melt behaves as a viscous fluid. When the solid fraction of the melt is higher than the maximum dendrite packing point fp, the melt undergoes overall deformation and exhibits behavior more similar to that of a solid. Within the solid fraction range of fc-fp, the grains in the melt gradually come into contact with each other, forming a grain network that can resist a certain degree of deformation. As the solid fraction increases, the grain network in the melt can withstand greater shear stress. Combining the distribution characteristics of segregation bands, it can be inferred that in the early stage of solidification, the surface layer first comes into contact with the cold mold, resulting in a higher degree of undercooling and a higher solidification rate, causing rapid cooling of the surface, while there is still a semi-solid region inside the casting. When the solid fraction of the surface layer approaches 1, until the end of the melt filling process, the solid fraction of the semi-solid region remains within the range of fc-fp. Due to the shear stress caused by the melt flow below the semi-solid zone, the grain network formed in the semi-solid zone gradually deforms. Once the shear stress exceeds the shear strength limit of the grain network, it collapses with the sliding and rotation of the grains. Therefore, the semi-solid zone expands, allowing mass and dendrites to enter this zone, ultimately forming segregation bands[24].

Regarding the influence of process parameters on the formation and distribution of segregation bands, a control variable experiment was designed with four groups of different pouring temperatures while keeping the mold temperature and extrusion rate constant to study the impact of pouring temperature and solidification rate on the generation of segregation. It is known that the formation mechanism of segregation bands is related to the formation time of the cooling layer and the semi-solid layer. The lower the pouring temperature, the smaller the temperature difference between the melt and the mold, and the gentler the solid fraction gradient in the semi-solid region. When the solid fraction at the surface reaches 1, there is still a significant stratification phenomenon in the semi-solid region, and the continuous flow of the melt generates shear stress. Therefore, the solute segregation is more severe at 700°C pouring than at 740°C pouring. However, when the pouring temperature is too high, the mold temperature will rise to approach the liquidus temperature of the melt, the surface solidification time will be prolonged, and the grain size will be too large, reducing the limit shear stress that can be withstood. The probability of segregation band formation at thin walls is higher than that at 700°C pouring. The experiment concluded that the least segregation occurs and the casting quality is best when the pouring temperature is around 740°C.

In view of the relatively irregular distribution of segregation bands, only the influences of process parameters and casting structure on the distribution of segregation bands were discussed.

5 Conclusion

(1) For ZM5 magnesium alloy, the segregation bands are solute segregation bands enriched with Mg and Al elements, mainly existing in the form of $Mg_{17}Al_{12}$ phase. The porosity within the segregation bands is significantly lower than that in the adjacent areas.

(2) Segregation bands occur in the middle area of the casting, far from the casting surface. At locations with sudden changes in wall thickness such as depressions and slots, there are sudden changes in stress, making segregation more likely to occur. The cause of segregation is directly related to the cooling rate. In the area close to the casting surface, the solidification rate is fast, and the precipitated phase is solidified without diffusion.

(3) Choosing the appropriate pouring temperature plays a significant role in suppressing segregation. If the temperature is too low, the cooling rate will be slow, making segregation more likely to occur. However, if the temperature is too high, it will lead to coarse grains in the casting, and temperature differences in areas with sudden changes in wall thickness and thin-walled areas, which also makes segregation more likely. In this experiment, when a pouring temperature of 740°C was used, the casting had fewer segregation phenomena and better performance.

(4) There are a large number of dendrites between the crystals inside the segregation band, which is related to the squeeze casting process. The continuous high pressure reduces the porosity inside the casting, and the second phase aggregates in large quantities, resulting in large-scale segregation.

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Conflicts of 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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